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6. NAME OF INSTITUTION	7. REPORT PERIOD		8. REPORT NO.
	A. FROM	B. TO	
Engineering Experiment Station Georgia Institute of Technology	1 Feb. 65	1 Aug. 65	1

9. PROJECT TITLE

Frictional Properties of Cotton Fibers

10A. SIGNIFICANT FINDINGS (*If any*)

None

10B. SUMMARY OF PROGRESS (*Give concise summary of progress for this report period.*) (*If additional space is required, use AS Form 140A*)

The purpose of this research is to establish the frictional characteristics of cotton fibers and to determine how these characteristics may change as cotton fibers are processed from the boll to the yarn.

Two bales of Empire WR Cotton of approximately 1.05" Fibrograph and 4.1 micrograms/inch Micronaire were selected and characterized as to convolutions per-inch, crimp, dimensions, and surface features as determined by optical and electron microscopy. Fiber lengths varied over the range < 1/2" to 2-1/2"; cross sections, somewhat elliptical, exhibited long diameters slightly less than 0.001" and short diameters of 0.0002" to 0.0004". Convolutions were in the range of 69 to 86 per-inch with the majority in the 3/4" to 1-1/8" lengths being about 85 per-inch. Crimp was 16.5 per-inch over the same fiber length range. Micrographs exhibited non uniform half convolution lengths and adjacent measures were 0.0058" (85 conv/in) and 0.0084" (60 conv/in) signifying non uniform helices, i.e., adjacent fibers could not mesh even if of the same average number of convolutions per-inch. Optical micrographs and electron stereo micrographs displayed fibrallar surfaces with troughs between adjacent fibrils. Fibril diameters were 0.000020" to 0.000040".

An apparatus for measuring fiber friction was designed and constructed. It consisted of a balance arm capable of moving a fiber very slowly across a second fiber attached to the needle of a servo-controlled galvanometer. An electro-optical system and power supply maintained the galvanometer at the null position. A counter torque was supplied to the galvanometer coil by means of the galvanometer's fixed magnetic field and an electric current proportional to any force tending to displace the galvanometer needle, such as the sliding force between the aforementioned fibers. A potential proportional to the torque-restoring current, and hence to the frictional force, was supplied to an XY plotter which thus presented the frictional data in graphical form. The normal force between the fibers was measured with a balance.

Frictional data for 10 nylon, 150 cotton, and 10 dewaxed cotton specimens, contituting 340 data plots, were obtained. Utilizing the simple coefficient of friction formula

$$\mu = \frac{\text{sliding force}}{\text{normal force}}$$

SIGNATURE OF PRINCIPAL INVESTIGATOR IN CHARGE	12. DIRECTOR OF RESEARCH INSTITUTION
Richard B. Belser	W. C. Whitley

FORM 140
IE 1963

SUMMARY OF PROGRESS (Continued)

values of μ for the above specimens were 0.40 for nylon, 0.39 (3/4"), 0.43 (1"), and 0.44 (1-1/4") for cotton, and 0.41 for dewaxed cotton. Static coefficients in the same order were approximately 0.60, 0.78, 0.86, 0.88, and 0.72. If the normal force was varied the coefficient of friction varied, i.e., for 1" fibers a normal load of .5-12.5 mg produced a coefficient of about 0.43 but for a normal load of 26 mg the coefficient decreased to 0.30. The values for nylon cited are in line with those reported in the literature. The error is assumed to be ± 5% but may be slightly more. Measurement and the maintaining of the normal force are the greatest source of error presently.

Of particular importance was the character of the plotted data which was distinctly different for the three types of fibers, nylon, natural cotton, and dewaxed cotton. The data for nylon exhibited less deviation from the average force than the data for the other fibers and a rapid stick-slip behavior, 7 per mm; the data for natural cotton exhibited fewer small stick-slips, 3 or 4 per mm, and much larger excursions ± 100% from the mean; the data for dewaxed cotton were between those of natural cotton and nylon in stick-slip and excursion behavior but occasionally exhibited very large excursions of + 250%.

Since the coefficients of sliding friction for all the fibers were so similar, considering the present possible error, it is apparent that the characters of the data plots and the coefficients of static friction are the more important features of the data measured. The ability of the frictional instrument, as developed, to display these three parameters of the data simultaneously and rapidly is an outstanding feature of the apparatus. Further development of it is expected to improve its discrimination, accuracy, and reliability to the stage that it fulfills the friction measuring requirements of this research.

Infrared spectrographs were made of cotton fibers and of the wax extracted from them. Typical cotton wax spectra were obtained and relatively strong absorption bands for the wax were found at 3.42, 3.50, 5.80, 6.82, 7.90. in the region 8-10, 12.5, and 13.9 microns. The band at 5.80 μ was observed in the spectra of dewaxed cotton or rayon but the intensity of it was insufficient for quantitative data. Infrared reflectance spectra of cotton wax was also obtained, but the reflectance attachment on hand was not well suited to the problem. An attachment furnished by a second manufacturer is to be examined.

X-ray diffraction investigations of fibers have progressed through the literature search and apparatus arrangement stages.

Fifteen personnel have been associated with this work during the period. These include 8 physicists or physics students, 5 professors or students of Textile Engineering, one chemist, and one mechanical engineer. Seven of the 15 were students. Two theses for the degree of Master of Science in Textile Engineering have been published, one covering the preliminary work on cotton selection and characterization and the second on the frictional apparatus and fiber frictional data obtained with it.

REPORT NO. 1 (SEMIANNUAL REPORT)
GEORGIA TECH PROJECT NO. A-843

1 February 1965 to 1 August 1965

FRICTIONAL PROPERTIES OF COTTON FIBERS

By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

PREPARED FOR
UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
SOUTHERN UTILIZATION RESEARCH AND
DEVELOPMENT DIVISION
NEW ORLEANS, LOUISIANA

1 August 1965

Engineering Experiment Station
and
School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station
and
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REPORT NO. 8 (SEMIANNUAL REPORT)

FRICTIONAL PROPERTIES OF COTTON FIBERS

By

R. B. Belser and J. L. Taylor

Grant No. 12-14-100-7661(72)
United States Department of Agriculture

1 February to 1 August 1965

Placed by
United States Department of Agriculture
Agricultural Research Service
Southern Utilization Research and Development Division
New Orleans, Louisiana

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ABSTRACT

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values of μ for the above specimens were 0.40 for nylon, 0.39 (3/4"), 0.43 (1"), and 0.44 (1-1/4") for cotton, and 0.41 for dewaxed cotton. Static coefficients in the same order were approximately 0.60, 0.78, 0.86, 0.88, and 0.72. If the normal force was varied the coefficient of friction varied, i.e., for 1" fibers a normal load of 11.5 - 12.5 mg produced a coefficient of about 0.43, but for a normal load of 26 mg the coefficient decreased to 0.30. The values for nylon cited are in line with those reported in the literature. The error is assumed to be $\pm 5\%$, but may be slightly more. Measurement and the maintaining of the normal force are the greatest source of error presently.

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15 were students. Two theses for the degree of Master of Science in Textile Engineering have been published, one covering the preliminary work on cotton selection and characterization and the second on the frictional apparatus and fiber frictional data obtained with it.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influence of crimp, convolution, cross section, surface texture and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

Subsequently the information obtained will be applied to the development of improvements of the strength, handling, and other desirable and economic features of cotton yarns.

II. INTRODUCTION

Cotton has served as a useful fiber since early Egyptian times, and through the ages much skill has been developed by man in processing the fiber to a finished product. Even in very early periods of textile fabrication recognition of differences in fiber properties existing because of variety of the cotton, staple length, effects of climate and weathering were observed. With the coming of the mechanization of ginning and the textile industry in the 18th century the need for thoroughly mixing batches of cotton to obtain blending and uniformity of the resulting yarn and fabric were recognized. Hence the early textile manufacturers resorted to a statistical solution for overcoming the individual variations between cotton fibers and to manufacture a useful and economic product.

In more recent years scientists have developed the necessary instruments for examining more critically the cotton fiber. First came the optical microscope which allowed reasonably exact delineation of fiber shape and dimensions. More recently advanced optical microscopy, electron microscopy, infrared spectroscopy, x-ray diffraction, and modern chemistry have allowed more critical examination of the physical and chemical structure of the fiber. Concurrently during these later years the development of synthetic fibers and efforts to duplicate or improve the properties of these fibers lead to a more careful study of the natural fibers and to the development of scientific groups devoted largely to fiber and polymer chemistry and physics. As a result of these various endeavors the importance of the shape, surface texture, and surface condition of the fibers was recognized and emphasized. Efforts were begun on measuring frictional characteristics of the individual fibers and have been continued for over 40 years. This

problem, however, was complicated for the natural fibers by the great variation of the individual fibers themselves, the complexity and time consumption of the delicate measurements, and some misunderstanding as to the nature of friction itself.

At the present time continued improvements in instrumentation, measurement techniques, and in the understanding of friction have contributed to the feasibility of a thorough study of the frictional properties of cotton and other fibers, and some studies of this nature have been completed recently or are now in course.

A superior approach to the problem of fiber friction is one in which various facets of the characteristics of a particular fiber batch may be studied concurrently by a single research team. Thus, data may be accumulated by the several methods of measurement such as interfiber friction, electron microscopy, infrared spectroscopy, and x-ray diffraction for a particular fiber sample; and the resultant data may be evaluated in the light of the supporting information furnished by the several methods. At the same time improvement of past or current measurement techniques of interfiber friction appear feasible and desirable.

The research outlined in this report covers progress in establishing and using facilities for measurements of the characteristics of a selected specimen of cotton by a frictional technique, by optical and electron microscopy, by infrared spectroscopy, and by x-ray diffraction methods.

III. EXPERIMENTAL WORK

A. GENERAL

The methods of selection and the characterization of the cotton specimen for this study are explained, and the establishment and use of facilities for measurement of the properties of cotton by frictional methods, micrography, infrared spectroscopy, and x-ray diffraction are outlined. Particular emphasis has been placed on the development of the fiber friction measuring apparatus and on perfection of infrared spectroscopy techniques for the study of cotton fibers and the wax coating of the cuticle.

B. SELECTION AND CHARACTERIZATION OF THE COTTON SPECIMEN

1. Selection of the Specimen

In preliminary conferences concerning possible research on the frictional characteristics of cotton fibers the need of a control specimen for the experimental work was apparent. A graduate student of the A. French Textile School, Mr. Thomas R. Boys, was assigned the problem of selection, characterization, and procurement of the necessary fiber as a portion of his thesis program for the M.S. Degree[1].

As a result of conferences between the staff of the Textile School, members of the United States Agricultural Department, and through the cooperation of the staff of the University of Georgia Agricultural Experiment Station at Experiment, Georgia, Empire WR was selected as a variety of cotton typical of the Southeastern Section of the United States.

2. Procurement of Cotton Specimens Nos. 1 and 2

The cotton selected actually consisted of two specimens: (1) A typical bale of Empire WR picked in 1962 and selected for careful characterization by Mr. Boys and (2) A similar specimen, gathered in 1964, on which procedures of growing, gathering, and ginning were clearly established.

The first bale was grown at Experiment, Georgia in 1962. The fiber was machine picked, ginned at Harrelson, Georgia and stored in bale at Experiment, Georgia. The cotton was removed from the bale in May, 1964, fluffed, and stored at 70°F and 68% relative humidity (RH) for subsequent measurement.

The second bale to be used as a standard specimen was grown two miles west of Experiment, Georgia during the 1964 growing season. The temperatures for the season were normal but the rainfall was slightly above average. After an essentially normal* cultivation period the cotton was hand-picked on 26 October 1964 and ginned at Locust Grove, Georgia on 28 October 1964. A 465 pound bale was formed at the gin and transported to the Georgia Institute of Technology for storage at 70°F and 68% relative humidity.

3. Characterization of Cotton Specimen No. 1

As is well known the mature cotton fiber consists of a thin convoluted ribbon about 1 mil in width and 0.25 mil in thickness. The fiber length may vary from < 0.5 inches to > 2.5 inches. However, the staple or average length is usually > 0.75 inches for cotton used in fabrics. The staple length of the Empire WR is slightly greater than 1 inch. A micrograph of lengths of typical cotton fibers is shown in Figure 1.

* See thesis of T. R. Boys if more complete details are desired.

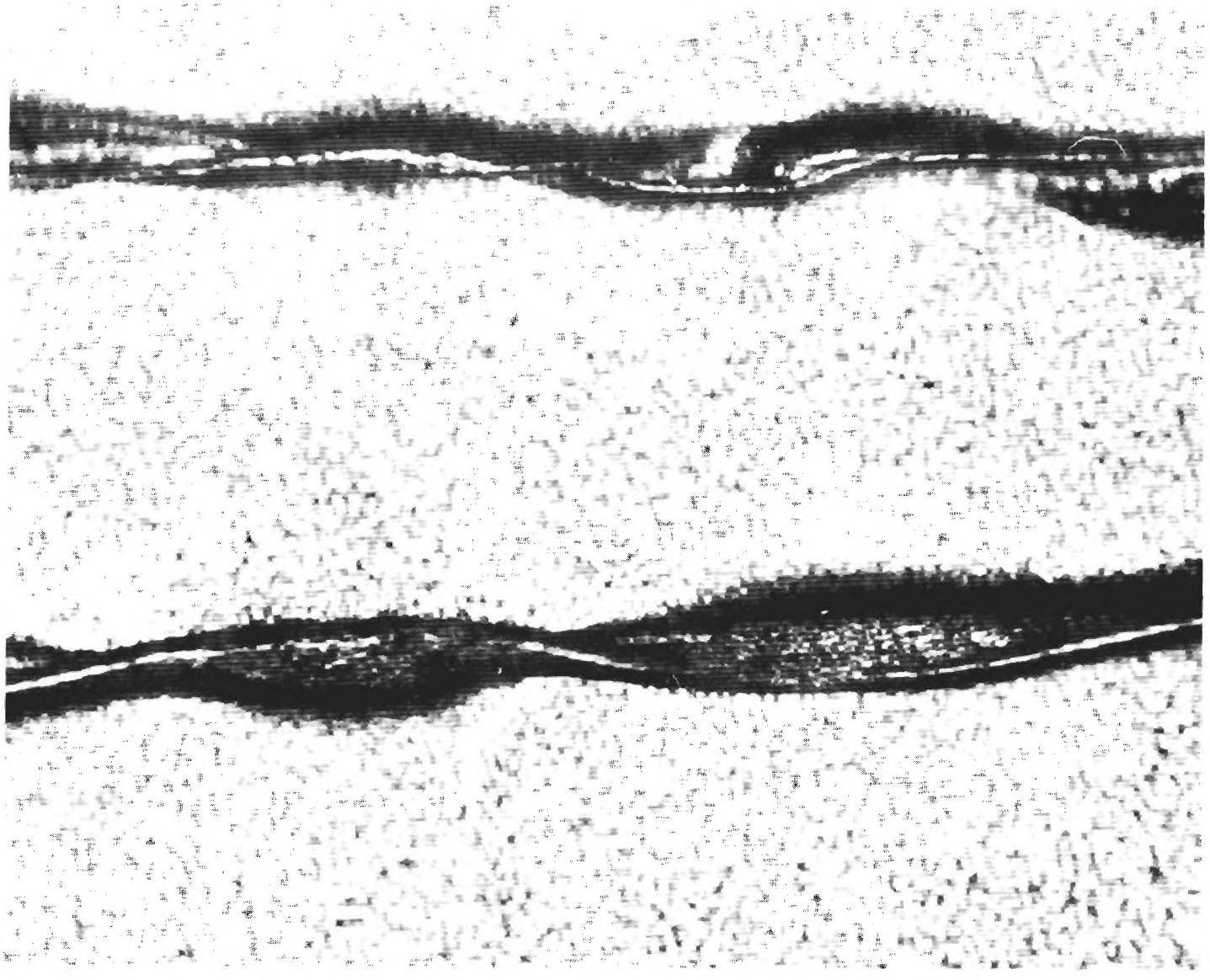


Figure 1. Photomicrograph of lengths of cotton fibers from Specimen No. 1. (chromium shadowed, 850x).

The first bale of Empire WR was conditioned at 70°F and 68% RH for one week after storage and then measured for average fiber length and fiber weight by means of a Spinlab Digital Fibrograph and a Sheffield Micronaire respectively. The fiber length was 1.04 inches (2.5 per cent span length) and the Micronaire gave a weight of 4.02 micrograms per inch of fiber.

By means of a binocular optical microscope (E-Leitz Ultropak) and a stereomicroscope the convolutions and crimp (as defined by Boys) per unit length of representative fibers were measured. Groups of 25 fibers of each 1/8" length interval were selected. The fibers were measured to ±1/32 inch and were subsequently gently mounted dry between two glass microscope slides for examination of the number of convolutions per inch. Results of the tabulation of this data are plotted in Figure 2. A maximum of 86 convolutions per inch was measured for fibers of 3/4" length and this number decreased only slightly out to a length of 1-1/8".

Crimp occurring was of two types, sharp angular bends, which were not counted in this study, and a second of much more regular curvature. Crimp of the latter type was an undulation of a series of characteristic one-half wave lengths of approximately 1/32", 2/32", or 3/32". Observation of this type of crimp was obtained by fastening a piece of doubly faced adhesive tape to a glass slide and then laying a fiber upon the adhesive surface, letting it rest where it made contact. Observation was then made with steromicroscope at 10x. Ten specimens of each fiber length were examined and a plot of average data is shown in Figure 3.

The maximum crimp is shown for the 1/2" fiber at approximately 19.5 crimps per inch. However, the number of crimps per inch is relatively

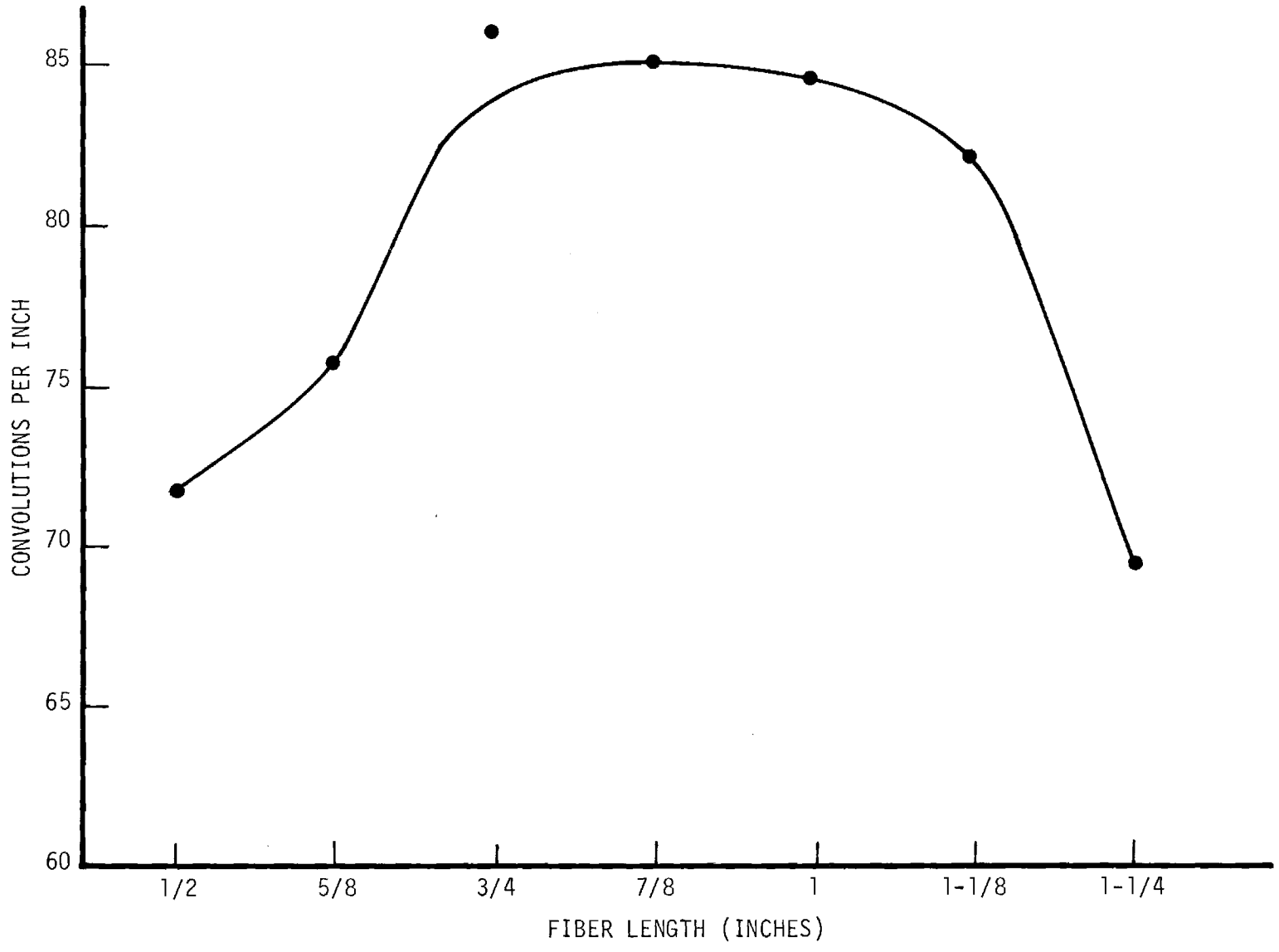


Figure 2. Convolutions per inch as a function of staple length of cotton Specimen No. 1.

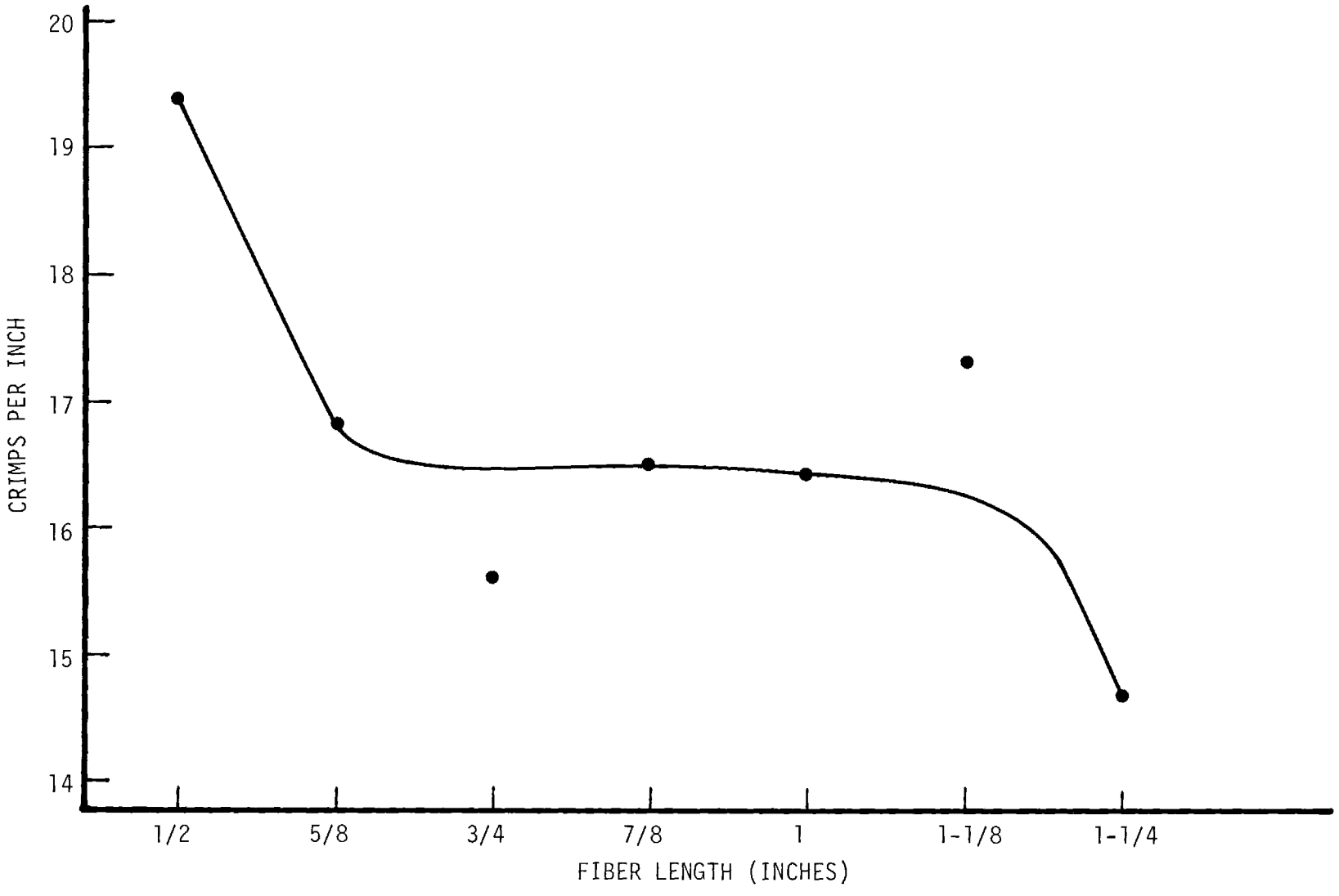


Figure 3. Crimps per inch as a function of staple length of cotton Specimen No. 1.

uniform over fiber lengths of 5/8" to 1-1/4" varying less than ±1 crimp per inch from 16.5 crimps per inch.

A thorough study of crimp has been conducted by M. Shiloh under a Contract of the Agricultural Research Service of the USDA. A portion of this work has been published in papers and the final report is expected to be available shortly[2, 3, 4].

4. Characterization of Cotton Specimen No. 2

Specimen No. 2, being hand picked was much cleaner than specimen No. 1. Due to a time limitation Mr. Boys was only able to perform Fibrograph and Micronaire measurements of this specimen. These were 1.056" compared with 1.043" for the 1962 bale and 4.11 microgram/inch compared to 4.02 microgram/inch, respectively. In view of the hand picking one might expect a slightly longer fiber length for specimen No. 2; the slightly greater weight per inch might be partly explained by the greater storage time of specimen No. 1. In any event the differences are so small as to be essentially negligible and for all practical purposes the characterization of bale No. 1 should fit bale No. 2.

C. EXAMINATION OF COTTON FIBERS BY OPTICAL AND ELECTRON MICROSCOPY

1. General

Optical microscopy was employed to count the convolutions and crimp of cotton specimen No. 1 as outlined in Section B. Optical and electron microscopy were used to characterize the fiber characteristics of cotton specimens Nos. 1 and 2 during the period of this report.

2. Examination of Cotton Fibers by Optical Microscopy

Representative cotton fibers of lengths of 1/2", 3/4", 1", 1-1/4", 1-1/2" were selected from specimens 1 and 2. A single fiber from specimen

No. 2 of 2-1/2" length was also found.

The fibers were mounted on small sections of glass photographic plate, the emulsion of which had been previously softened in water. The fiber was pressed gently into the emulsion with a cover glass, the cover glass was removed, and the emulsion was allowed to dry. The substrate and mounted fibers were placed in a vacuum chamber, which was then evacuated. Chromium was evaporated onto the fibers at an incident angle of about 45° to shadow the surface of the fibers. The fibers were then photographed at powers from 200 to 500 x using a Leitz Metallux microscope and a 35 mm camera. The resulting negatives were printed at magnifications of the original fiber of 1000 to 2500 x. Extensive visual examinations were also made by steromicrography.

Micrographs representative of the types obtained for fibers of 3/4", 1", and 2-1/2" lengths are shown in Figures 4 - 7. Although some differences between fibers of the various lengths examined were observed a survey of fibers of statistical significance has not been completed except as reported in Section III B, preceding. Immediately observable were the convoluted ribbon shape of the mature fiber, fiber surface texture, approximate fiber width and thickness, convolution length, the fibrils in the surface, and a reversal of the fibrils.

For instance, referring back to Figure 1 , the lower fiber, we can observe a half convolution occurring in approximately 0.0058" or at a rate of approximately 86 convolutions per inch. This number is in good agreement with the average number of convolutions per inch for 1" fibers of cotton specimen No. 1 as shown in Figure 2. However, the adjacent half convolution is much longer or approximately 0.0084" giving 60 convolutions per inch. The average for the two together is about 73 convolutions per inch. The characteristic of non uniform half convolution lengths is quite obvious in many of

the photographs as may also be observed in Figures 4 and 7. Figures 5 and 6 display fibers of 1" length from specimens 1 and 2 respectively. Both of these particular photomicrographs display half convolution lengths of > 0.0075 inches i.e., < 67 convolutions per inch. However, since the adjacent convolutions are not visible it is probable that we are observing a long half convolution at high power. These photographs and Figure 7 were selected principally to show the surface detail obtained by the optical method employed. In particular in Figure 7 the characteristic spiral wound substructure of fibrils is here visible and furthermore in the long half convolution of the fiber of Figure 8 a reversal of the winding direction of the fibril is observed.

Widths of the fiber ribbons are found to be about 0.001", or slightly less in several instances, by measurement from the photomicrographs. Thicknesses were found to be in the range 0.0002" to 0.0004". However, accurate measurements of width and thickness require good microsections, a problem to be discussed next.

Sectioning was attempted with a Bausch and Lomb optical microtome and a small Schwarz fiber microtome. Although both of these methods gave some usable sections none were considered of good enough quality for the type of measurements desired. On the other hand a high degree of irregularity of cross section shape was obvious, and crude measurements of dimension confirmed those cited above for fiber widths and thicknesses. Improved methods of sectioning are now under study.

3. Examination of Cotton Fibers by Electron Microscopy

Surface textures of cotton fibers of various lengths were observed by electron microscope replica techniques. The procedures used in replication are outlined below.

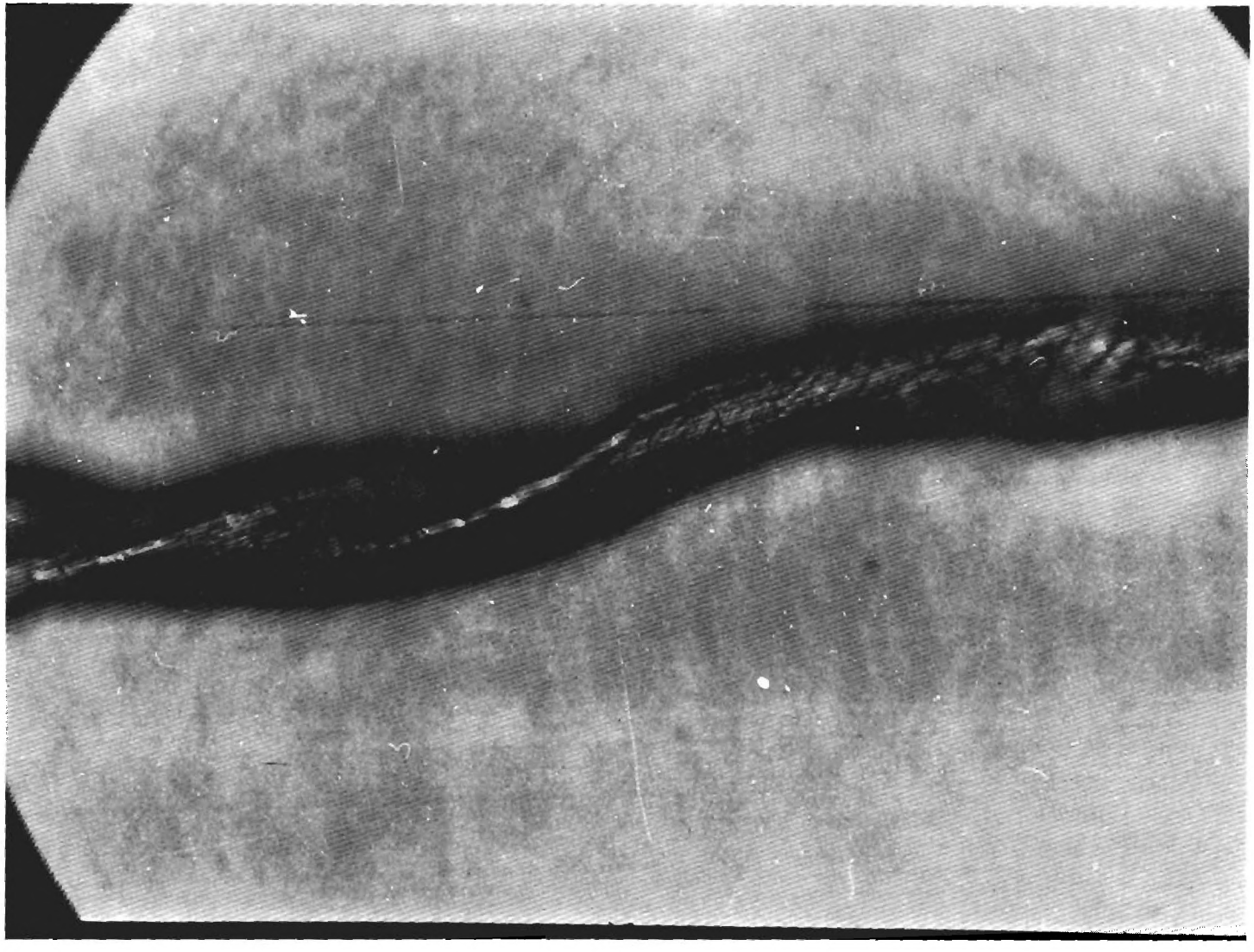


Figure 4. Photomicrograph of typical cotton fiber of 3/4" length, Specimen No. 2. (760x).

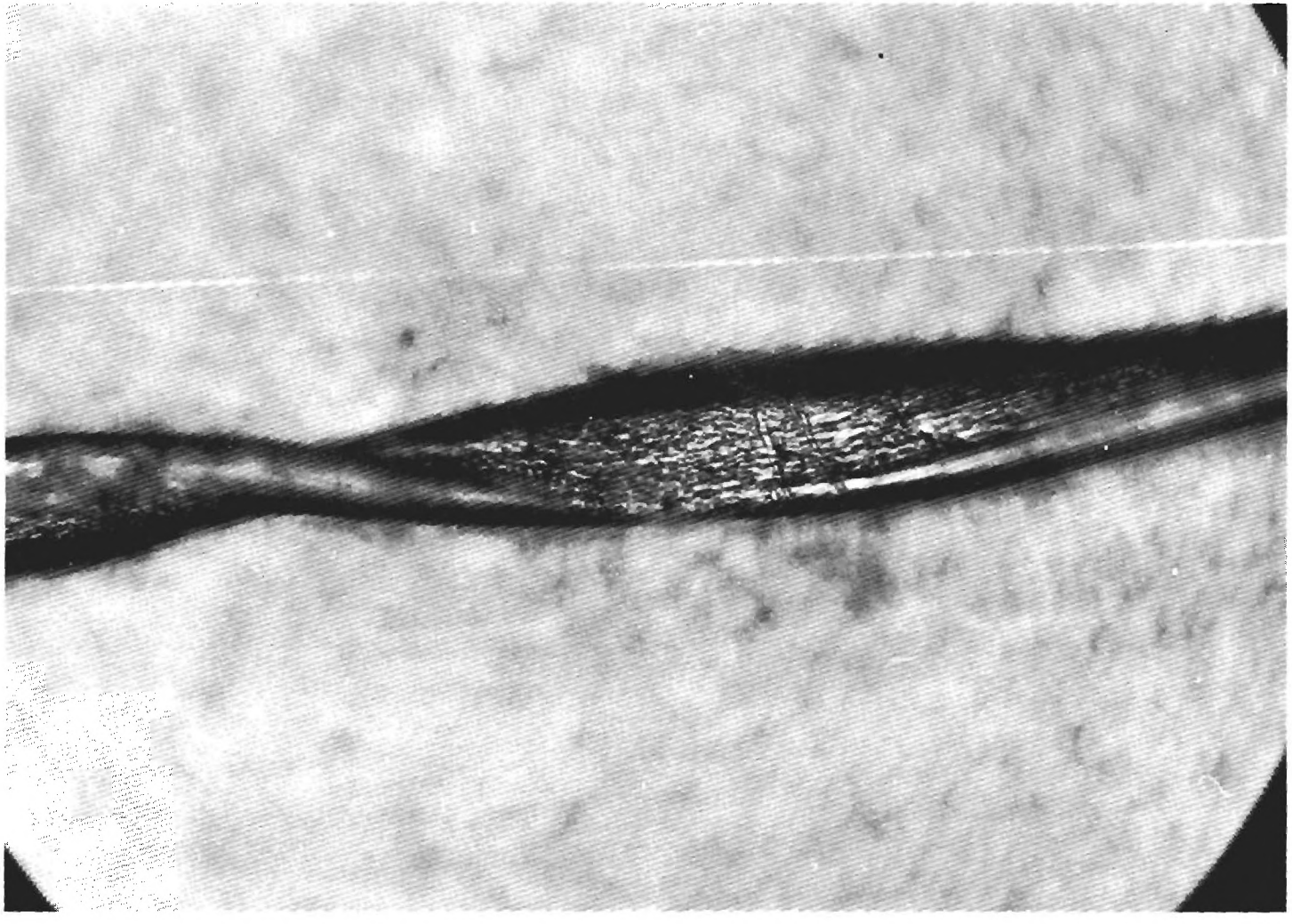


Figure 5. Photomicrograph of typical cotton fiber of 1" length, Specimen No. 1. (795x).

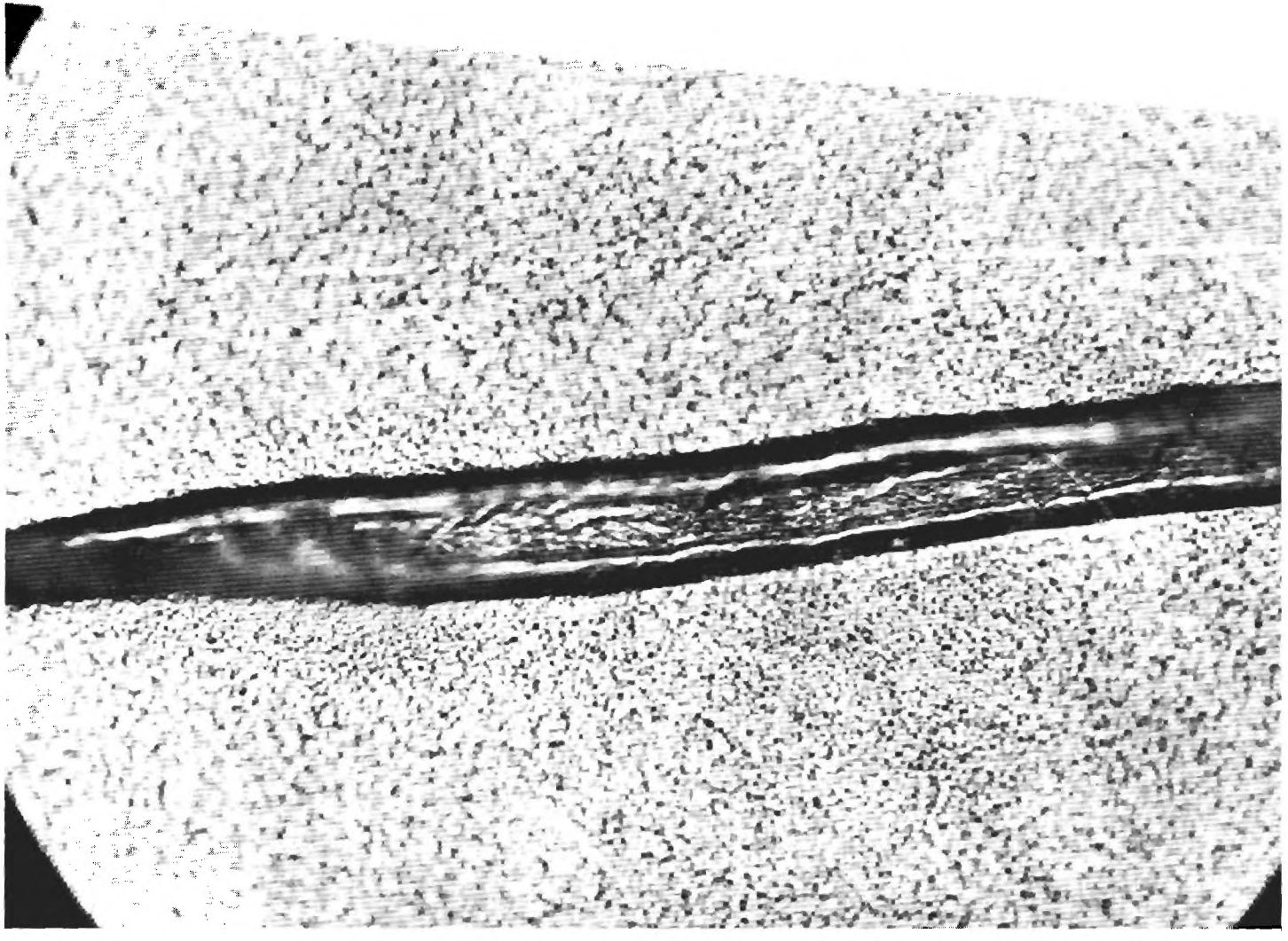


Figure 6. Photomicrograph of typical cotton fiber of 1" length, Specimen No. 2. (810x).

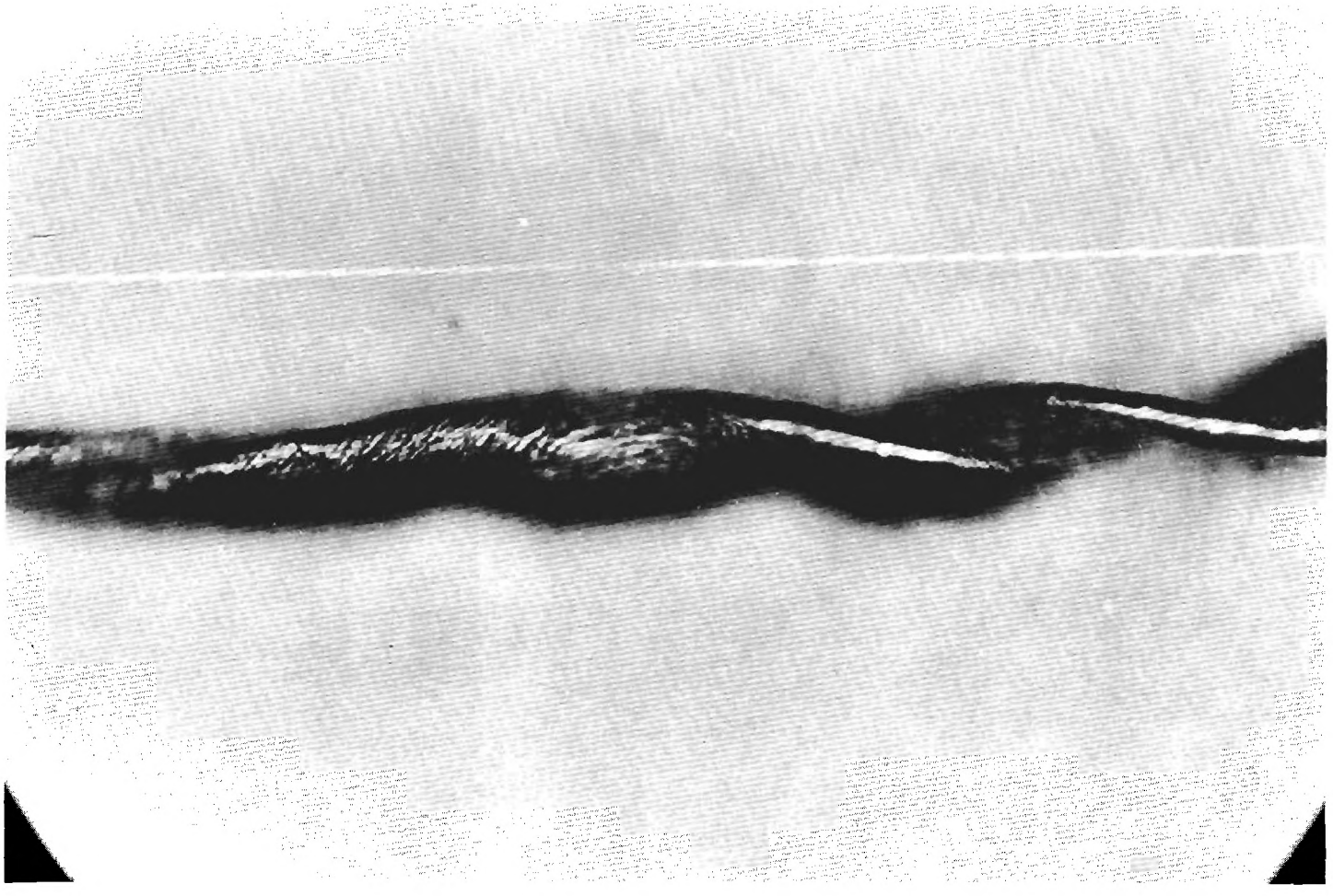
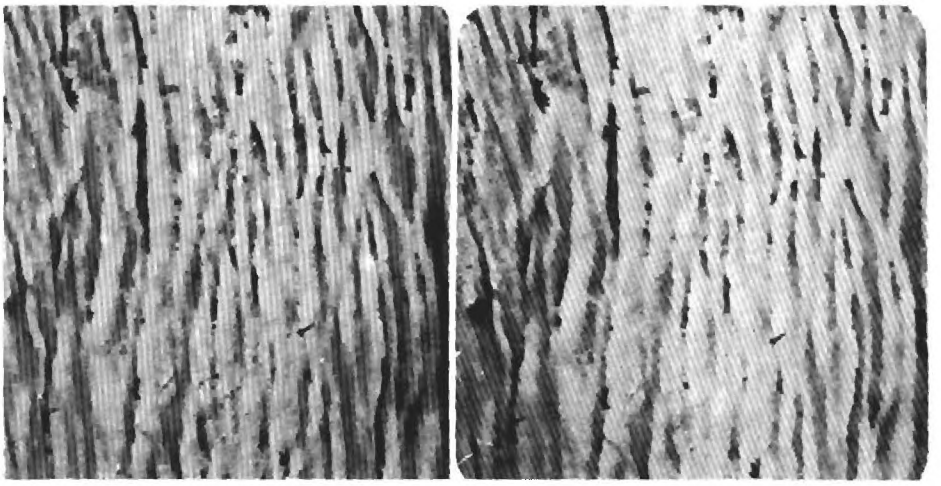


Figure 7. Photomicrograph of typical cotton fiber of 2-1/2" length, Specimen No. 2. (845x). (Note fibrils and reversal near center section.)

A solution of polystyrene in xylene with 0.5% dibutyl pthyalate was made. This solution was placed on a glass slide and allowed to dry. The slide was then heated on a hot plate to soften the plastic. The fiber was placed upon the plastic and pressed into it with another glass slide. The fiber was then stripped off leaving an impression in the plastic. This impression was shadowed with platinum and subsequently coated with carbon. The polystyrene base was dissolved in ethylene dichloride and the carbon replica picked up on an electron microscope grid. Electron micrographs were made in the normal manner at powers of 600x, 880x, and 3200x respectively and stereomicrographs were made by tilting the replicas approximately $\pm 6^{\circ}$ from the beam axis for successive micrographs.

Stereomicrographs typical of those obtained are printed in Figures 8A and 8B. Figure 8A is a stereomicrograph of a 1-1/4" fiber from specimen No. 1. Here it may be observed that the surface resembles a plowed field of successive valleys and peaks. These undoubtedly represent the surface contours of the outer fibrils. Examination of a series of these stereomicrographs gave widths from peak to peak in the range 0.6 to 1 micron or 0.000024" to 0.000040". Hence, a fibril diameter must be of about this dimension. Optical micrography gave rough dimensions of about 0.000020" to 0.000040" also for fibrils of the 2-1/2" fiber from specimen No. 2. Hence, approximate agreement is observed here by either method and the range is definitely established.

In Figure 8B may be seen a stereo-electron micrograph of a reversal of a 2-1/2" fiber from specimen No. 1. The black streaks in the print are artifacts caused by tearing of the replica. These artifacts have been traced to use of the dibutyl pthyalate solvent and the percentage of this solvent in the



A. Striated Surface indicating fibril widths in the range 0.000024" to 0.000040". (9550x).

B. Reversal of wind of fibrils. (2620x).

Figure 8. Electron steromicrographs of fibers of Empire W R Cotton of 1-1/4" and 2-1/2" lengths. (Note striated surfaces and reversal in B).

xylene-polystyrene medium will be reduced or omitted in subsequent replications.

4. Summary of Observation of Cotton Fibers by Optical and Electron Microscopy

Examination of fibers of Empire WR Cotton with optical microscopes and by means of enlarged micrographs in the range 200 to 2600 diameters have delineated shape, surface texture, and structural characteristics of the fibers. The fibers have been shown to be shaped like thin convoluted ribbons having widths of about 0.001" or less and thicknesses generally in the range 0.0002" to 0.0004". Convolution rates for statistically significant counts appear to vary over the range 69 to 86 in the center of the fiber dependent to a degree on fiber length, but estimates of convolution frequency from a single half convolution are inaccurate since successive half convolutions may change greatly in length. The irregularity of the half convolution lengths point to diversity of the fibers and a high improbability of complete mesh of convolutions of contiguous fibers even of similar lengths or of similar statistical convolution counts.

Relatively poor cross sections, thus far obtained, have likewise pointed to a diversity of cross sectional shapes, and departure from the ideal of a ribbon section of essentially a rectangle with rounded corners. However, the fact that sections were made for a group of fibers without control as to position with respect to the convoluted zone accounts for some of the variation observed here. Properly descriptive sections would be made at the central zone of a half convolution and requires sectioning of a single fiber at discrete positions along its length. This examination method is planned as our sectioning techniques are improved.

Optical micrographs exhibited also the substructure of the fiber consisting of fibrilswound helically about the axis and the collapsed central lumen. The fibrils were of diameters of approximately 0.000020" to 0.000040" or of the order of 25 to 50 per fiber width. Reversals of direction of the helix were observed and occurred in one instance in the center of a long half-convolution. The surfaces of all fibers showed a texture as a result of the fibrils, and hence were ridged by a series of hills and valleys as one traversed from the top of one fibril to the next. The angle the fibrillar helix made with the axis of the fiber was not visible in most micrographs, but was clearly established as about 45° for one fiber (Figure 7).

Electron stereomicrographs of replicas of fiber surfaces exhibited with better resolution the results obtained by the optical methods; the texture of the surface of the fibers appeared similar to that of a ploughed field with successive essentially parallel furrows and crests. Dimensions from crest to crest were in the range 0.000024" to 0.000040", the same value observed optically. A reversal of the direction of the wind of the fibrils was also observed in an electron micrograph (Figure 8B).

The micrographs have thus characterized the shape, dimensions, surface, and substructure of fibers of Empire WR Cotton and have exhibited a high diversity principally in shape from fiber to fiber. Although possibly significant differences between fibers of various lengths may exist these were not characterized sufficiently well by the small numbers of each fiber length examined to date to furnish dependable data. No appreciably consistent differences between fibers of cotton specimens Nos. 1 and 2 have been observed by methods of microscopy.

D. MEASUREMENT OF THE FRICTIONAL PROPERTIES OF COTTON FIBERS

1. Introduction

Sliding friction means the force of opposition offered to the sliding of one surface over another; rolling friction is the opposition offered to the rolling of one body on another; and viscous friction is the force of opposition offered to the passage of a body through a liquid or a gas. In general, in this discussion we will be concerned principally with sliding friction and viscous friction.

A working knowledge of the macroscopic properties of friction is interwoven into the history of man from the dawn of his existence. The development of fire-sticks, stone cutting and polishing techniques, lubricants, and the wheel before the dawn of recorded history bear witness to this knowledge. Not until the time of Aristotle, however, do we find extant writings which deal with friction. These were followed much later by works of Leonardo da Vinci who displayed a keen insight concerning friction and its quantitative evaluation. Amontons[5] (1699) first delineated the early classic laws of sliding friction; namely,

a. The frictional force is proportional to the load and for most surfaces is equal to one-third of the load; and

b. The friction is independent of the size of the bodies.

Amonton's statements were checked and generally confirmed during the same year (1699) by de La Hire[6] who ascribed the reason for friction to adhesion between asperities on the contacting surfaces.

In modern times, using advanced experimental apparatus and methods, Bowden, Tabor, and coworkers have published a series of papers on the frictional properties of matter. Their findings have been summarized in two

books, which furnish the most comprehensive treatment presently available on friction.

They have shown that a metal plate placed upon another is held apart from its mate by asperities protruding from the contiguous surfaces and constituting a very small proportion of the total area. Since the pressure exerted at each asperity is high enough to cause plastic flow the asperities adhere or bond to the adjoining sheet, and the area of real contact varies with the load dependent on the shear strength of the metal. A force applied to the upper plate tangent to the interface causes the bonded junctures to grow, and the static friction force reaches a maximum just before the junctures shear. As slipping continues junctures are remade, grow, and are sheared causing the "stick-slip" behavior frequently observed.

The behavior described applies principally to contaminated surfaces where the number of asperities is relatively small. For these cases the value of the coefficient of friction $\mu < 1$, and may be as small as 0.15 for two surfaces of hard steel. In these cases the expression $F = \mu W$ can be applied, where F = force applied to weight, W, tangential to the interface between the two objects of concern and just sufficient to cause sliding to begin between the two surfaces (static coefficient) or to continue at a uniform velocity (sliding coefficient); and μ = the coefficient of friction $= F/W$. On the other hand, if the metal surfaces are outgassed in vacuo the metals may seize and the value of μ may exceed 50.

In the case of polymers at low sliding speeds frictional behavior can also be explained in terms of the adhesion mechanism. Since junction growth does not appear to occur to any appreciable extent the simple theory of friction is better suited to polymers than to metals. The major factor affecting

the friction between polymers is the area of contact. This depends on the geometry of the surface and on the scale of surface roughness as well as on the load. Because these materials are visco-elastic it also depends on the time of loading and on the speed of sliding. At constant sliding speeds, however, if these are not too large, it is possible to describe the friction of polymers in very simple terms based on the simple adhesion theory of friction. If the adhesion component of friction is small, an appreciable part of the resistance to sliding may arise from internal hysteresis losses within the deformed polymer.

At higher speeds the behavior is more complicated and the friction can no longer be described in terms of the simple adhesion theory. The friction depends markedly on both speed and temperature and reflects the visco-elastic properties of the polymer. Hence, the variation will change from polymer to polymer and varies with polymer structure; it is greatest for polymers having the greatest chain flexibility and least for cross linked polymers of the most rigid structure.*

2. Friction Between Textile Fibers

Friction between textile fibers in processing and in a yarn, in its simplest form, can be considered as sliding friction between solids. However, the geometry of the fibers, their viscc-elastic properties, and coatings such as the natural wax on cotton complicate measurements of fiber friction. The small size of the cotton fiber leads to additional complications in mounting, and the application and measurement of small forces.

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* This short discussion on friction is based principally on the work of F. P. Bowden and D. Tabor, "The Friction and Lubrication of Solids, Part II", Oxford Press (1964).

Another point for consideration is that in a yarn the strength of the yarn is in part a function of the static friction between fibers, i.e., the force resisted before sliding of one fiber across another begins.

The principal initial problem in the study of friction between fibers was the design and development of an apparatus capable of measurement of small forces and to accurately portray the variation of force as the surface of a single fiber is drawn over the surface of another. The high degree of variation in the size and shape of cotton fibers, discussed in Section III, C-2, pointed to the need also of an instrument which would allow many accurate measurements to be made in a relatively short time in order to obtain statistically significant data. The development of an instrument to fulfill these needs is discussed in the succeeding Section D-3.

3. Development of Apparatus for the Measurement of Fiber Friction

a. Apparatus Used by other Investigators.

A search of the literature of devices for the measurement of fiber friction was conducted. This revealed that successful instruments were based principally on four concepts. These were: (1) the fiber bundle method; (2) the fiber twist method; (3) the torque principle; and (4) the stick-slip technique.

Adderley[8] in 1922 described a fiber bundle method in which two bundles of aligned fibers were mounted on glass and pressed together. A single fiber pressed between the two bundles was extracted and the force for extraction was measured. This measurement was performed for many species of cotton and for many different fiber lengths. A relation between the force required to obtain slip and convolutions per unit length was obtained. However, no measurement of the coefficient of friction as such was determined.

In the fiber twist method two fibers are twisted together a known number of turns. One fiber is connected to a fixed arm and the second to a movable one or to a variable weight. The force to remove the movable fiber from the pair is measured or the number of twists required to prevent its removal is determined. B. G. Hood and J. Lindberg and N. Gralen* have described instruments using this principle. Although a number correlating to frictional differences was obtained in each case the translation of this to a coefficient of friction was not attempted.

An application of the torsion method is described by J. C. Guthrie and P. H. Oliver[9]. In this apparatus a fiber was mounted on one end of a horizontal arm extending perpendicularly from a vertical torsion wire. A mirror in a vertical plane was affixed to this assembly near the axis of the wire. A second fiber perpendicular to the first, with both fibers in a horizontal plane, was dragged across the first by a horizontal arm rotating about a vertical pivot offset some distance from the axis of the torque suspension. The load between the fibers was adjusted by a weight placed on the arm attached to the torque wire.

When the second fiber was drawn across the first the torque displacement was measured by the angular displacement of a light beam reflected from the mirror. The frictional force was the restoring torque exerted by the wire and could readily be calculated from the known parameters of the aparatus. With this apparatus values representing both static and kinetic friction were obtained. The results, however, were generally expressed in graphic form of a plot of essentially the frictional force versus the force exerted in a normal direction between the surfaces of the fibers. Values obtained for rayon were of the order of 0.22 for the static coefficient of both 1.5 and 3 denier rayon

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* See references 12 and 15 in Bibliography A.

and 0.19 and 0.21 respectively for the kinetic value. These values appear low. The angle between the axes of the fibers in a horizontal plane could also be varied and data was presented over the range 10° to 90° included in the acute angle. However, results were inconclusive as to a change in the coefficient of friction with respect to a change in the angle between the fibers.

The fourth frictional method of interest, the stick-slip method, is that described in Bowden and Tabor[7] (page 226) and previously described in a paper by Pascoe and Tabor[10]. In this case a polymer fiber or rod is suspended horizontally and a fiber of similar material, mounted on a small driven carriage, is dragged across the under surface of the other fiber near its end. The upper fiber is pressed down on the lower fiber thus flexing the upper fiber in a vertical plane. The load may be determined from the bending of the upper fiber and the frictional force from the deflection of the end of this fiber in a horizontal plane as the lower fiber is dragged across it. Using this equipment the coefficient of friction of many polymer fibers were determined over a very large load range. Values for nylon, polythene, telflon and P. V. D. C. were obtained over the load range 10^{-6} grams to 10 grams, or a total range of 10^{7}. Variations in the coefficient of friction, μ, were found to occur with load and the expression $\mu = kW^{-B}$ was suggested as the correct one for the data found, where B was in the range 0.2 to 0.3 and k is an undesignated constant. This was shown from a plot of log μ versus log load, the slope of the resulting curves being negative and in the suggested range.*

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* See reference 7, page 227.

For a nylon fiber of 0.042 mm in diameter (0.0016") and a load of 10 mg the coefficient of friction was found to be approximately 0.5. At lesser loads of about 1 mg it became > 1 and at greater loads of about 100 mg it decreased to < 0.4.

b. The Design of a Servo-Controlled Fiber Friction Apparatus

Based on the preceding studies and an investigation of other references listed in the Bibliography, the torque method similar to the one described by Guthries and Oliver appeared to be the principle best adapted to the task of measuring the friction between cotton fibers. Because of the many measurements to be made and rapid variations in the characteristic stick-slip relations it appeared desirable to incorporate into the instrument an electrically recording output system to facilitate data collection. In discussing this matter with members of our staff it was suggested by Mr. Billy R. Livesay, Research Physicist, that a servo-mechanism used for measuring small torques on metal film specimens placed in a magnetic field might be adaptable to the measurement of small torques applied to the system by fibers. An examination of the apparatus then available revealed the feasibility of this suggestion, and material was obtained for constructing an apparatus for measuring fiber friction. Its design is outlined below.

The frictional forces between individual cotton fibers are in the neighborhood of a few milligrams for the practical range of normal forces. The microscopic nature of surfaces is such that highly non-uniform forces are experienced when a fiber is drawn across a stationary object, which may be a second fiber. An instrument responsive to rapidly changing forces is therefore required to obtain accurate information about the resulting frictional characteristics of two such surfaces. An electromagnetic servo-system with

the required sensitivity and response characteristics has been adapted to make these measurements. The principle of this system[11, 12] has been used in other types of physical measurements, but it is believed that this is the first time it has been applied to the measurement of frictional forces.

A D' Arsonval galvanometer of the type used in Honeywell portable potentiometers was found to be quite suitable for this application. A fine gold ribbon suspension provides a frictionless pivot about the vertical axis, but is sufficiently rugged to support the loads used in these measurements. A mirror is fixed to the lower end of the coil and a pointer extending horizontally about two inches is attached at the top of the coil. Forces applied horizontally and normal to the pointer produce a torque about the pivot axis. This torque may be balanced by an electric current of the proper magnitude and direction in the coil to produce a counter-torque which prevents deflection of the pointer. The magnitude of the applied force is thereby calculable from the required current.

The short response time needed for the measurement of rapidly changing frictional forces is obtained by using a photoelectric device to detect small deflections of the galvanometer from its null position. A tiny beam of light reflected from the galvanometer mirror onto a small dual photodiode generates a differential emf when the light beam is made to fall more on one section of the diode than on the other. The output of the photo diode is then fed into a high gain D.C. differential amplifier which in turn supplies the required correcting current to the galvanometer coil.

Figure 9 displays the principal apparatus for measuring fiber friction. This consists of a mechanical balance and fiber driver, the galvanometer and servo-system, and an XY plotter. A close-up view of the fiber driving mechanism and galvanometer is shown in Figure 10.

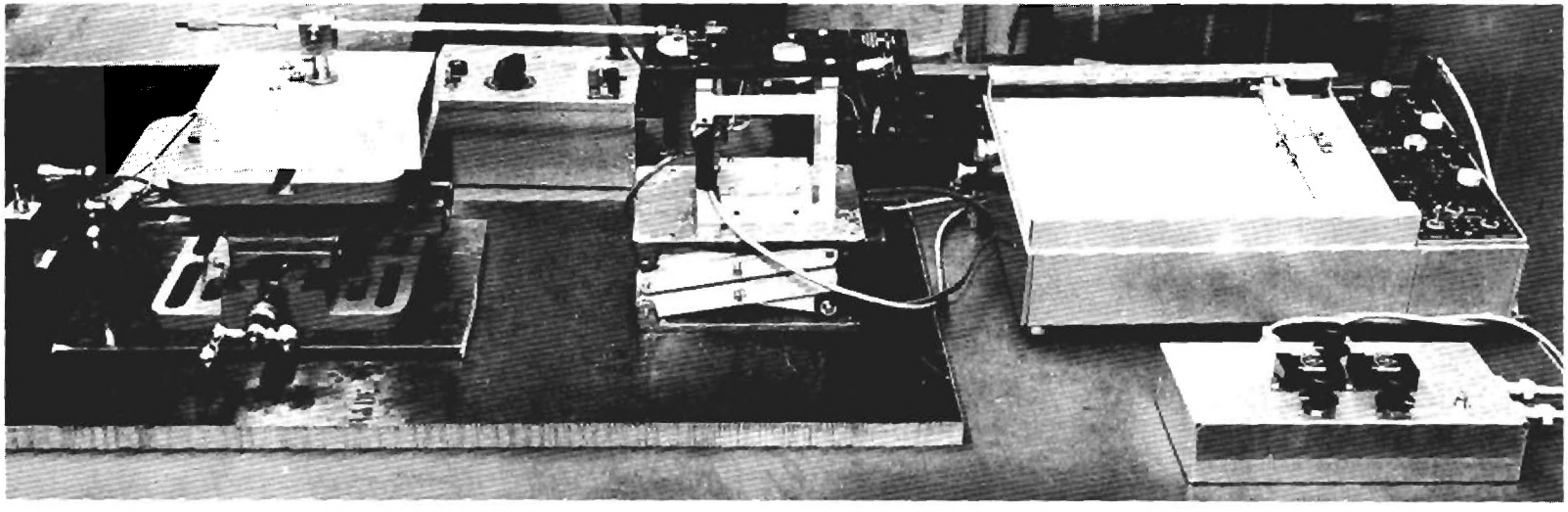


Figure 9. Apparatus for measuring fiber friction.

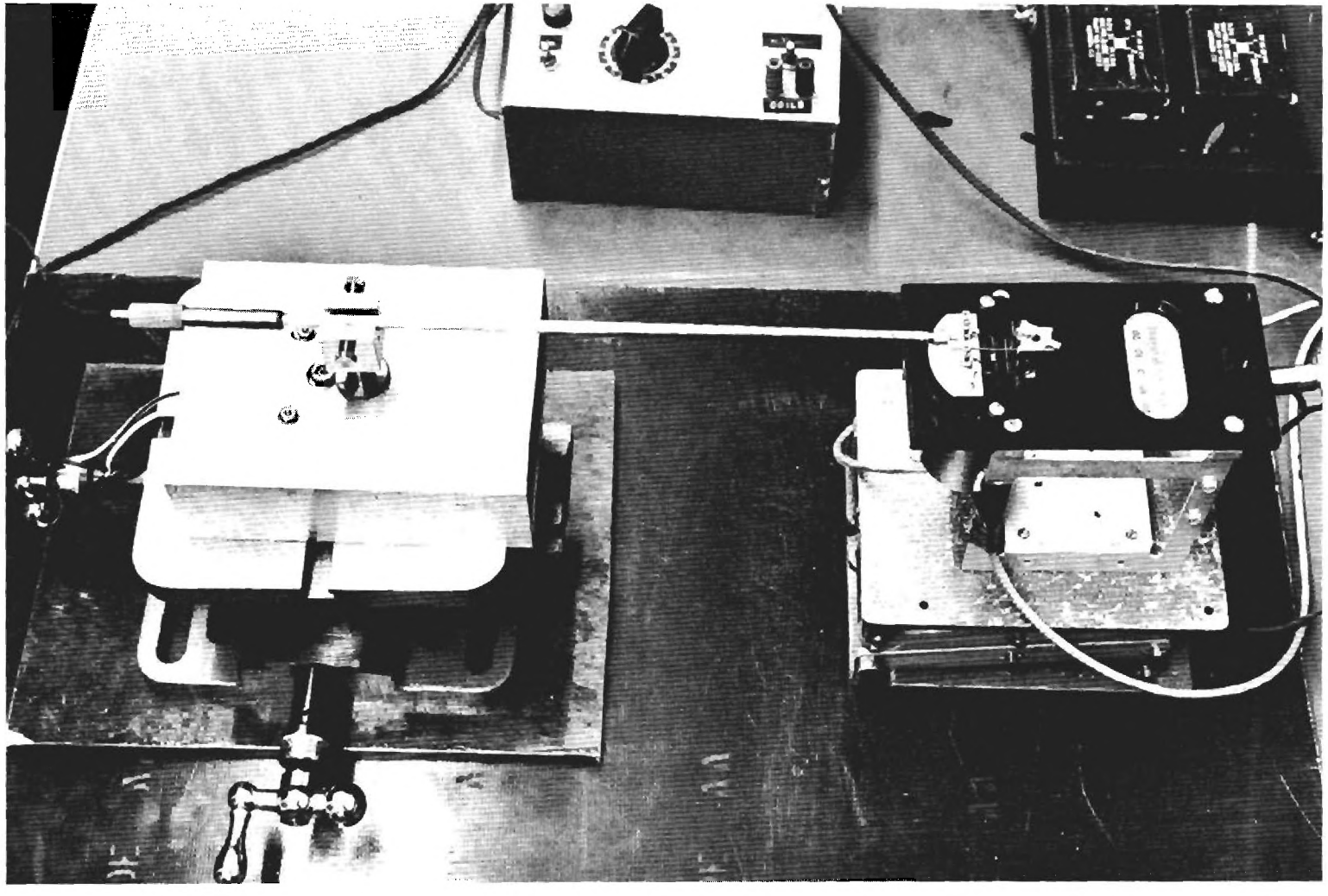


Figure 10. Fiber drive mechanism and servo-controlled galvanometer.

The fiber driver consists of a slender, tubular, balance arm supported by sapphire bearings and rotated about a vertical axis by a shaft immediately beneath and attached to the bearing support. The vertical shaft is rotated by a synchronous motor through a reduction gear system at a rate of 1/75 rpm.

A fiber to be studied is suspended horizontally and normal to the shaft in an adjustable holder under a tension of a few milligrams at one end of the balance arm. Normal force adjustments are made with a micrometer type screw sleeve at the other end of the balance arm. The length of the balance arm from the pivot to the fiber was 10 inches so that for fiber lengths of one inch or less the variation of position of the applied force on the lower arm of the galvanometer pointer, was very small.

A second fiber (or other material) is mounted on the galvanometer pointer in a bow type holder fastened to the pointer by sealing wax. The holder supplies sufficient tension to maintain a taut suspension. Most of our measurements have been made with a balance arm shaft speed of 1/75 rpm which means the fiber moves about 0.8 inch per minute.

Other comments pertinent to the instrument are noted below. The light source is a No. 80 grain of wheat lamp and the diode is a TI No. LS 221 null indicator. The servo-amplifier is a Burr-Brown model 1509 differential amplifier. The instrument is calibrated by rotating the galvanometer to the horizontal and hanging standard milligram weights from the pointer at the point of normal fiber contact.

c. Fiber Mounting and Measuring Technique

Inasmuch as both tension and normal force may have a pronounced effect on the frictional forces exerted, a consistent fiber mounting technique was required. A method was designed to mount a stationary fiber on the

galvanometer needle parallel to it. A special holder was also constructed for mounting a second fiber on the end of the balance arm at any tension desired.

For the stationary fiber, a piece of eight-thousandths phosphor bronze was used and fabricated into a U-shaped holder. The spring was held in a special vise when each fiber was mounted making reproducible values of tension possible. Initially, several glues were tried as methods to fix the fiber to the spring, but were too slow in hardening for practical application. Therefore, small amounts of sealing wax were used and an excellent technique was acquired for making very rapid mounting possible. The spring mount was fixed on the galvanometer needle with sealing wax. This method was very satisfactory since a small quantity of wax was sufficient and, a minimum mass was added to the needle.

The holder for the fiber on the balance arm was fabricated from brass and stainless steel stock as shown in Figure 11. Minimum dimensions were used to prevent increasing the total mass of the arm and thus prevent excessive friction in the bearings. The holder was designed to handle any length fiber by incorporating movable clamps which operated in a small slot. Therefore, one-half inch fibers could be mounted, leaving a one-fourth inch length for examination. The inside surfaces of each clamp were faced with rubber, bonded by adhesive to the metal. The fiber mount was attached to the balance arm by means of an attached steel pin, inserted along the axis of the arm and locked by a cap screw.

For mounting each fiber, the holder was first removed from the arm and placed so the clamps were arranged along a vertical axis. One end of the selected fiber was secured in the upper clamp. A one-half gram weight was

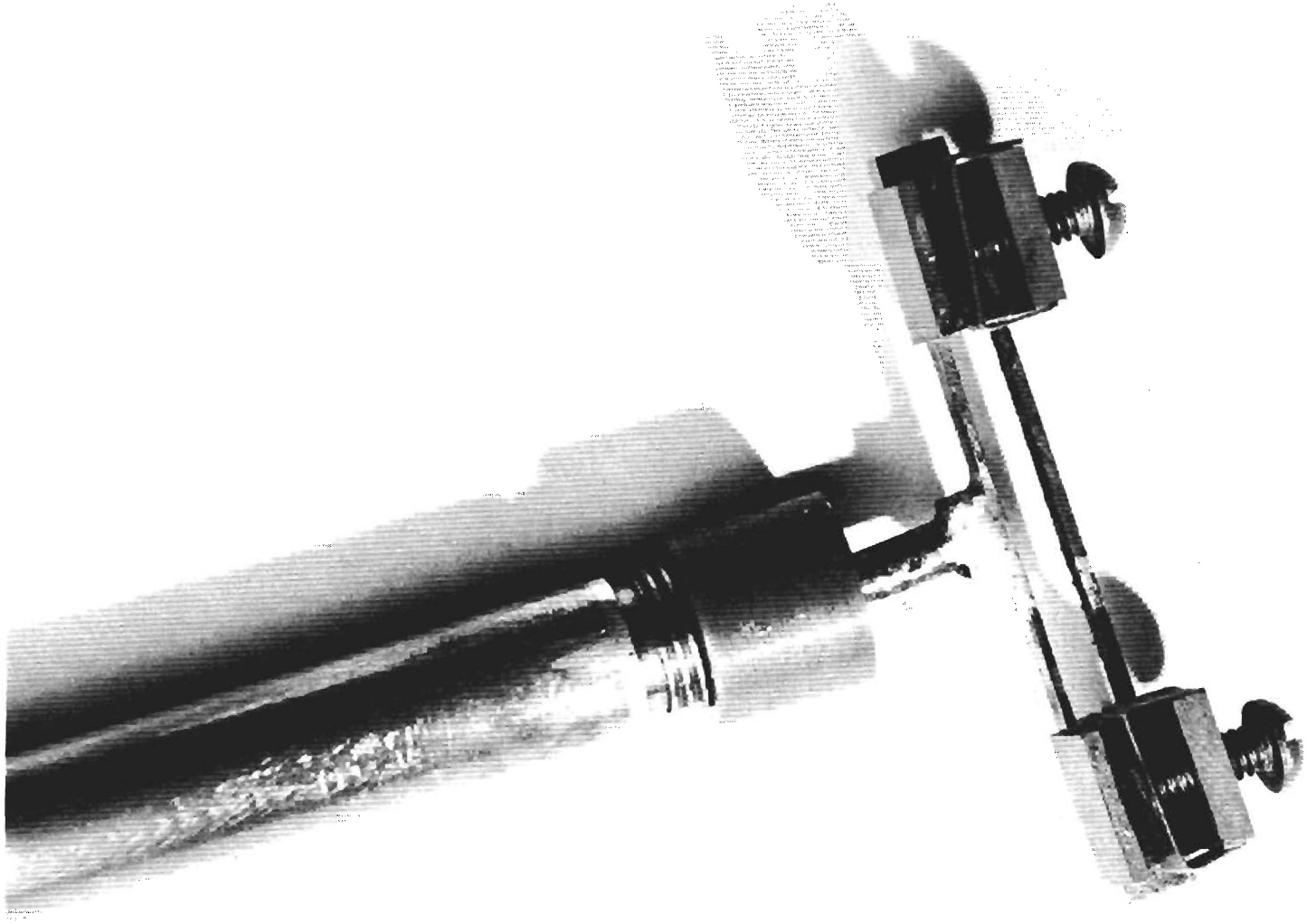


Figure 11. Fiber mount on driven balance arm.

clamped to the other end of the fiber thus aligning the fiber along a vertical axis. The other end of the fiber was fixed by tightening the second screw. Hence, a consistent one-half gram tension was applied to each fiber during alignment and clamping.

The fiber mounted on the needle was not changed with each new fiber tested, but was changed after measurements of five fibers were completed. No change in data was noticed as a result of this procedure instead of changing the stationary fiber for every experiment. However, fibers of corresponding lengths were used in all measurements in both fiber holders.

d. An Apparatus and Procedure for Measuring Fiber Clinging Power

An additional apparatus for measuring fiber friction, based on the principle of fiber clinging force as described by Adderley,[8] was constructed. This measured the force necessary to pull a fiber from between two bundles of parallelly aligned fibers. Two pistons of 7/32" diameter operated in annular ring cylinders facing each other and on the same axis. One annular ring cylinder was fixed with respect to the base and the other was movable. Each piston was faced with an adhesive bonded rubber pad. Fiber pads were formed by aligning fibers closely together across each rubber facing to a depth of several fibers; the fibers were fastened into position with Duco cement applied well away from the surfaces of interest.

A fiber was placed in the center and aligned with the other fibers, but with one end slightly extruding. The two pads were brought into contact and one piston was fixed. The second was forced against the fixed one in a direction parallel to the axis of the two holders by one quarter turn of a screw fixed to the base support. The end of the fiber to be tested was attached with sealing wax to the servo-controlled galvanometer indicating needle, which was mounted horizontally and rotated about a vertical axis. The

fiber clamping apparatus was now moved away from the galvanometer needle in a direction lying in a horizontal plane and perpendicular to the needle, until the fiber slipped. The current flowing through the galvanometer was recorded up to and at the moment of slip on the XY recorder thus allowing the calculation of the force to require slip.

4. Measurements of Friction of Fibers of Nylon, Cotton and Dewaxed Cotton

a. Introduction

Upon completion of the apparatus described in Section 3b it was used to measure friction between various textile fibers. In the early stages various loads between the upper and lower fibers were used. Generally these ranged between 11.5 and 28.5 milligrams, the heavier loads being used at first. The heavier loads appeared less adapted to the particular instrument than the lighter ones and a load of 11.5 mg was the final one adopted.

b. Procedure for Measurement

Using the apparatus and procedures described in paragraphs III D 3b and c frictional measurements of individual fibers were made. The individual mounts were detached from the balance arm and galvanometer respectively and loaded with fibers of the same variety in the manner previously outlined. The mounts were then replaced.

The balance arm was aligned precisely with the galvanometer needle by means of its milling vise mount and the fiber to fiber contact point was adjusted to a precise position indexed on the galvanometer needle. This index mark indicated the calibration point for the needle lever arm. The galvanometer was then lowered by means of a jack stand support. The balance arm was adjusted to a precise level. The normal force was adjusted by the micrometer sleeve to a desired value while the arm was supported by a Mettler

balance arranged to maintain the arm at the level position. The balance was removed and the galvanometer now raised back into position so that the fibers touched at the indexed point and the balance arm was level. Any final adjustments necessary in alignment were made while the balance arm was slightly raised by hand until the readjusted position appeared correct when contact was made.

The contact was normally established near one end of the driven fiber but not too close, since in the latter case a horizontal component of the normal force, caused by a slight deflection of the fiber axis, could cause a slight error added to the frictional force component. The XY plotter was adjusted to the proper zero and scale and the drive mechanism of the recorder was started to establish the zero; a few seconds later the fiber drive motor was started. When the moving fiber had completed a run of approximately 1/2" it was lifted from the fiber fixed to the galvanometer needle.

The fiber mount on the galvanometer needle was shifted a very small amount, about 1/32" or less and the run repeated. The moving fiber was changed and two more runs were made. The moving fiber was changed after each two runs but the fiber on the galvanometer needle only every 10 runs.

Five fibers of a type were measured and then five of another. The fibers on the moving arm and on the needle were always matched pairs of the same length or type. Calibration of the indication of force on the XY recorder was made periodically by hanging a small weight on the galvanometer needle at the point where the movable fiber crossed the needle while the galvanometer was placed on its side. The weight then tended to rotate the needle as would the moving fiber and the displacement of the XY recorder as

a result of the restoring current could be measured. Small errors undoubtedly were introduced by the slight displacement of the fiber force from the axis of the needle and by rotation of the galvanometer onto its side. However, these were small compared to other sources of error discussed subsequently. At the gain used in most instances a force of 7.7 milligrams on the needle displaced the recording pen one-inch on the XY plotter. In some cases a slightly larger gain was used in which cases 5.0 milligrams displaced the recording needle one-inch in the ordinate direction.

The swept zone of the moving fiber was from 1/2" to 3/4", generally near 1/2". The time drive on the recorder was 12"/minute and a 20 second sweep gave a four inch abcissa on the recorder. The rate of sweep of the moving fiber was thus approximately 1/40" per second or 0.62 mm/second.

c. Measurement of Friction between Relatively Smooth Fibers, Nylon

The generally featureless surface of nylon monofilament compared to that of cotton fiber and the availability of a number of coefficient of friction measurements in the literature provided a means of testing the newly designed friction apparatus. The friction between single specimens of nylon monofilament of 0.0008" (19 microns) diameter were measured in the manner outlined. Values for the coefficient of friction were determined from the data plot by integrating the area, determining the mean deflection from zero and calculating the coefficient of friction from this value, the deflection constant per inch, and the normal force.

Data for typical runs are shown in Figure 12. Here it will be noted that the XY plot obtained indicates a typical stick-slip behavior. Fluctuations are occurring at approximately a rate of 20 to 25 per one-inch traverse on the chart or at a rate of 7-8 per mm of fiber travel.

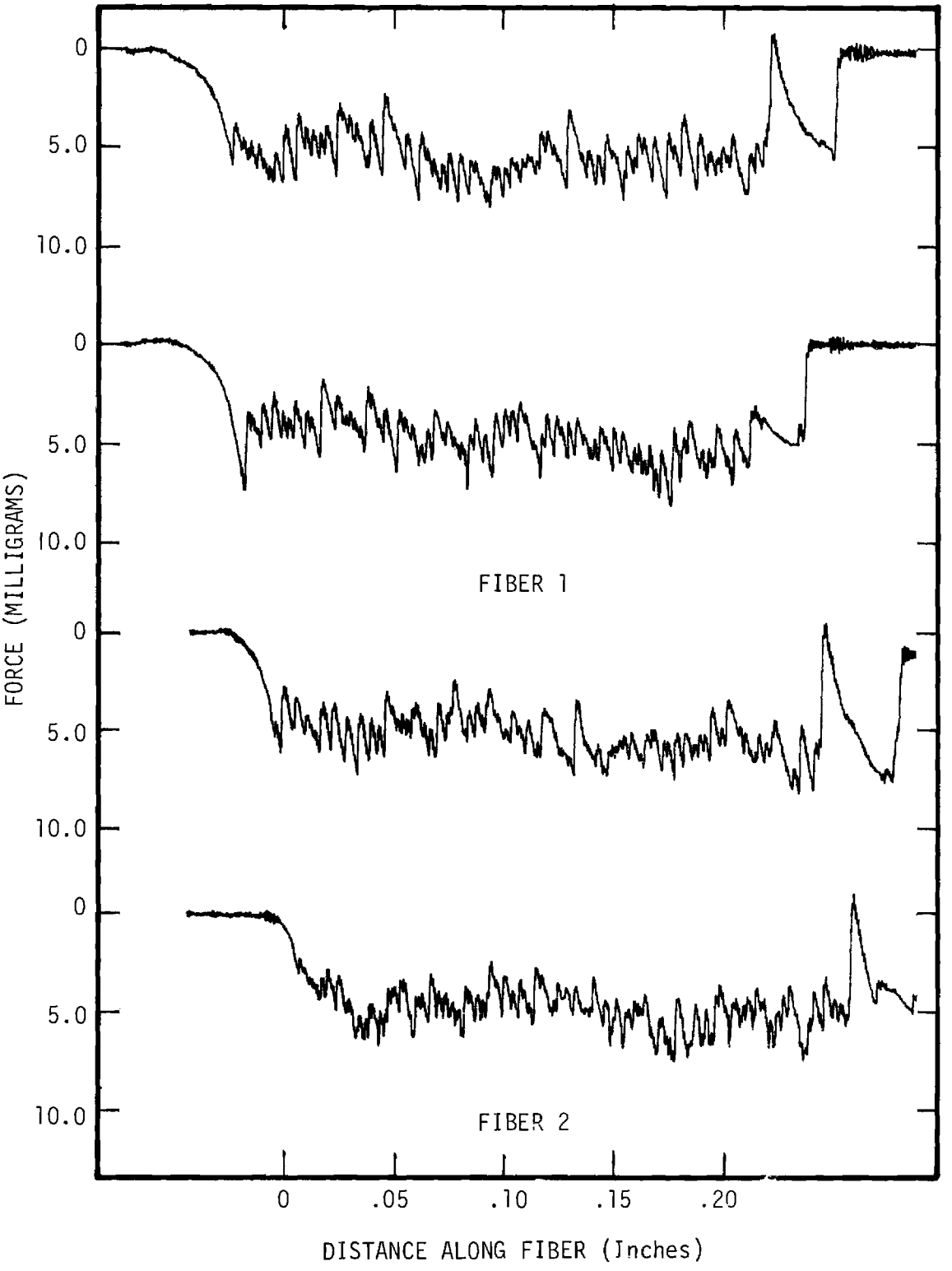


Figure 12. Frictional force data for representative nylon fibers of 0.0008 inch diameter, 11.5 mg. normal force.

Maximum fluctuations over the first 3/4 of the travel distance are of the order of ±0.30" from a mean value of 0.50" to 0.60" and in some cases a good bit less.

Calculations of the coefficient of friction of the ten fibers were made from the series of curves, and values are shown in Table 1. In these cases loads were held constant at 11.5 milligrams as nearly as the capability of the instrument allowed. It will be seen that the average value of the coefficient of friction calculated by the straightforward formula

$$\mu = F/W$$

is 0.40. The extremes were 0.28 and 0.56. The value F was found from the curve by obtaining the average displacement of the data plot from zero and multiplying by the force factor 7.7 mg/in of displacement.

The value 0.40 checks with a value of 0.40 given by M. Harris [13] for nylon and one of approximately 0.50 given by Bowden and Tabor[7] for a fiber of 42 microns diameter and a load of 10 milligrams. Interpolation from values given for 19 micron fibers to 10 mg loads also gave a coefficient of approximately 0.5. The data of Bowden and Tabor exhibited a considerable variance of μ with load and with fiber diameter for nylon filaments. It also appears that the static coefficient of friction was the one given in the reference data although this is not clear.

The principle variance from fiber to fiber in our data for nylon may be associated partly with load variability during a cycle which will be discussed later and partly with the intrinsic surface variance of the fiber, or possibly its handling history. Since these measurements were fairly preliminary the check with previous existing data appears quite good.

TABLE 1

Coefficients of Friction of Ten Specimens of Nylon Fibers on Nylon

Test Number	Average Force (mg.)	Normal Force (mg.)	Friction Coefficient
1	4.95	11.5	0.43
2	4.62	11.5	0.41
3	5.29	11.5	0.46
4	3.91	11.5	0.34
5	3.22	11.5	0.28
6	4.14	11.5	0.36
7	3.91	11.5	0.34
8	6.44	11.5	0.56
9	4.37	11.5	0.38
10	4.84	11.5	0.42
Average	4.57	11.5	0.40

d. Measurement of Friction between Natural Cotton Fibers

Fifty specimens each from the fiber lengths 3/4", 1" and 1-1/4" were obtained from the standard bale of Empire WR Cotton and the friction between like fibers was measured. These particular measurements were made in an air conditioned room where the temperature was normally about 25°C. No control of humidity existed except that provided by the air conditioner and this was not measured in these particular experiments. It is generally about 60%, however. All measurements were conducted in an enclosure of plexiglas which restricted air flow or rapid fluctuations of temperature if they occurred. In order to restrict differences that might occur due to the difference in time at which measurements for various fiber lengths were made, five fibers of one length were tested, then five of the next length, etc., until 50 fibers of each type had been tested in a period of approximately 10 days.

A measurement for a typical cotton fiber of each length is shown in Figures 13, 14 and 15 and the data obtained for all fibers are reported in Table 2. Unfortunately, constant loads were not used throughout this experiment, but were varied over the range 11.5 mg to 28.5 mg. The heavier loads were used at first, but after excessive bouncing of the balance arm occurred the load was reduced through a series of stages to 11.5 mg which was used during about one-half of the runs.

As may be seen from the Figures 13 to 15 the character of the natural cotton data is different from that of the smooth fiber of nylon. The stick-slip variations are at lower frequency, approximately half that for nylon, i.e., for cotton 3 or 4 per mm of traverse of the fiber in contrast to 7 per mm for nylon. Superimposed over these minor fluctuations are much larger

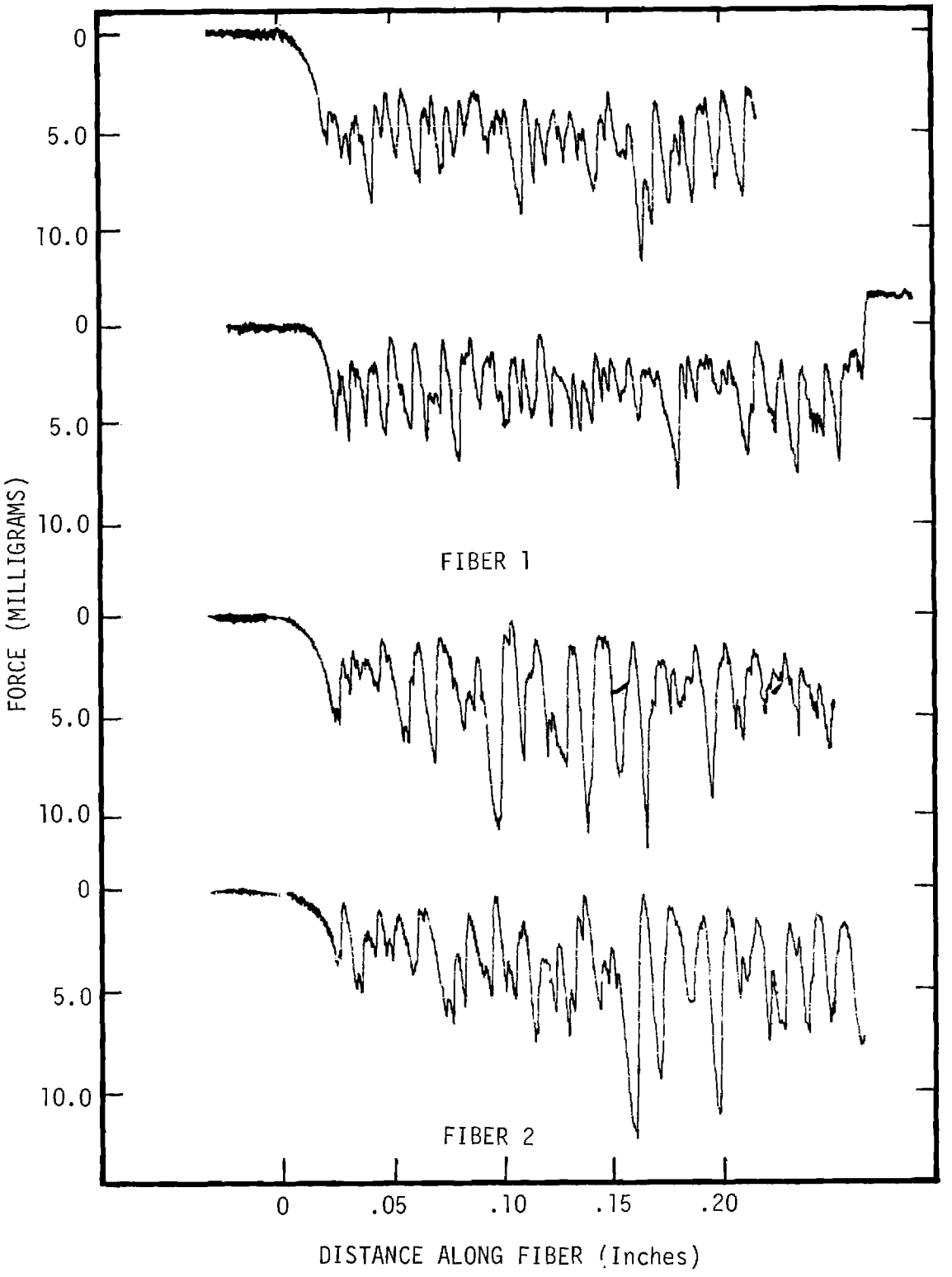


Figure 13. Frictional force data for representative fibers of 3/4 inch Empire W R cotton, 12.5 mg. normal force.

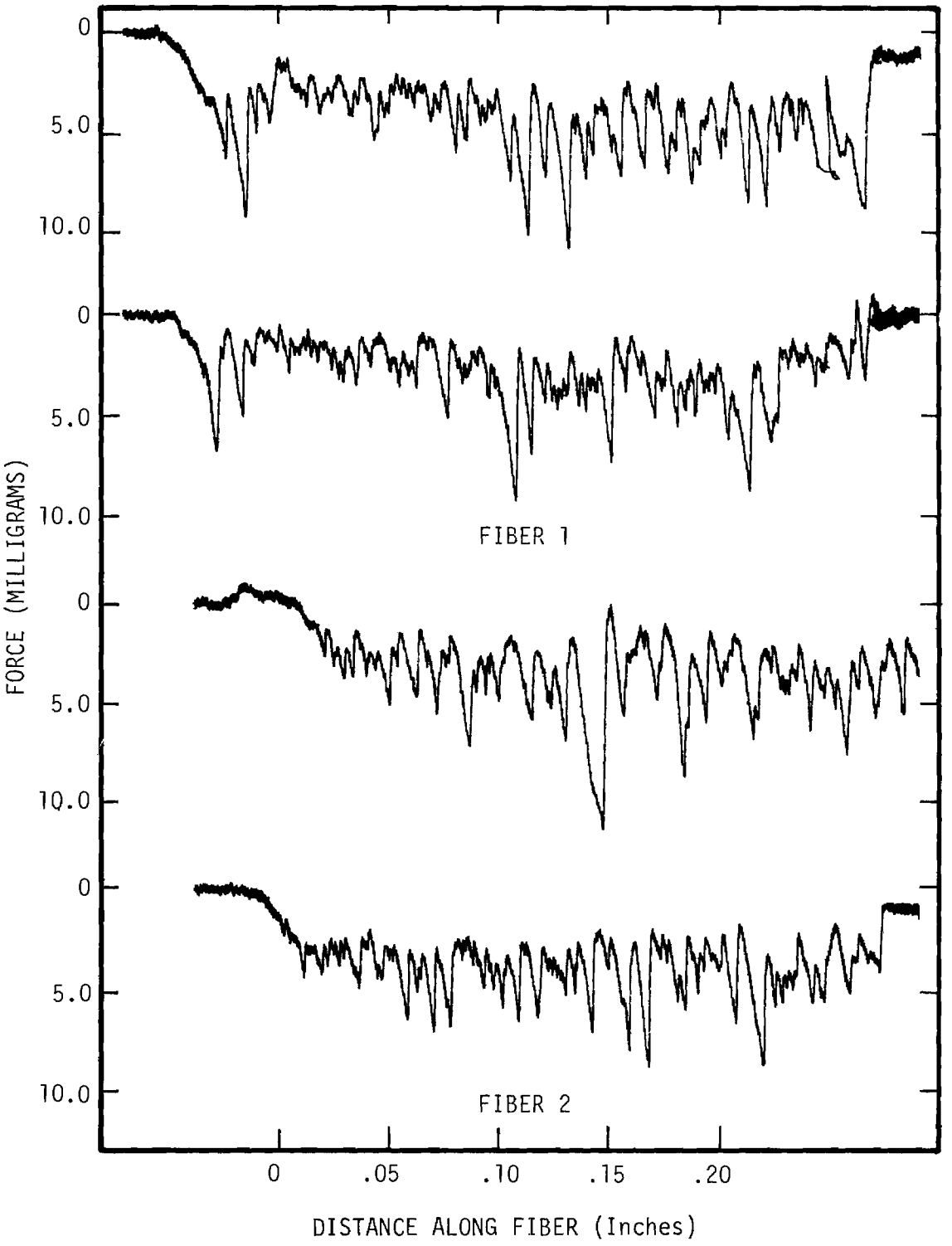


Figure 14. Frictional force data for representative fibers of 1 inch Empire W.R cotton, 11.5 mg. normal force.

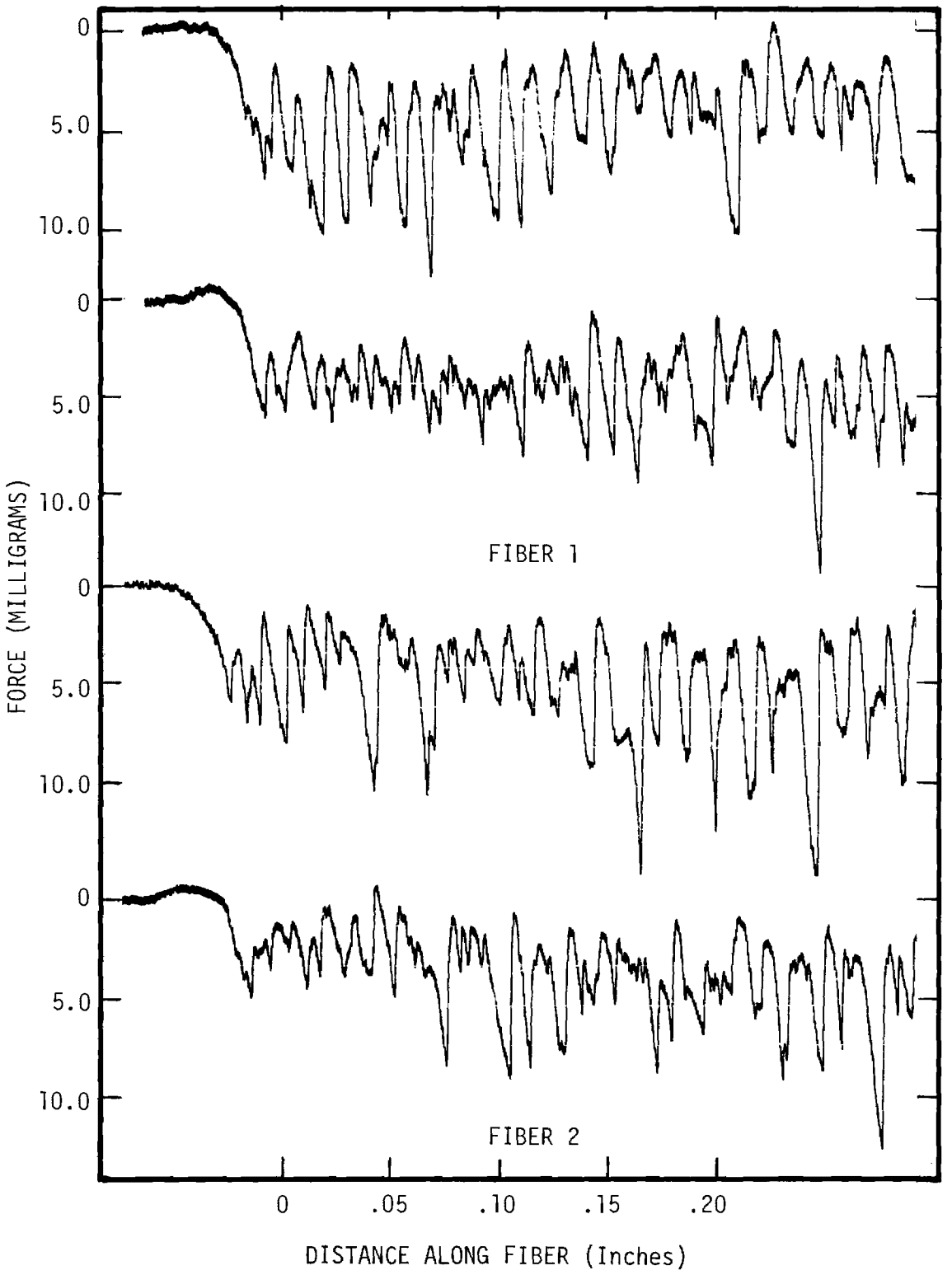


Figure 15. Frictional force data for representative fibers of 1-1/4 inch Empire W R cotton, 12.5 mg. normal force.

TABLE 2

Computed Average Coefficients of Friction

Empire WR Cotton Fibers of 3/4", 1", and 1-1/4" Lengths

Test Number	Fiber Length (Inches)			Test Number	Fiber Length (Inches)		
	3/4	1	1-1/4		3/4	1	1-1/4
1	0.47	0.13	0.14	26	0.34	0.31	0.38
2	0.40	0.13	0.30	27	0.33	0.24	0.24
3	0.49	0.14	0.31	28	0.64	0.34	0.18
4	0.40	0.15	0.22	29	0.48	0.27	0.18
5	0.44	0.17	0.25	30	0.52	0.32	0.23
6	0.39	0.18	0.23	31	0.37	0.23	0.42
7	0.46	0.25	0.18	32	0.52	0.25	0.24
8	0.46	0.26	0.13	33	0.36	0.38	0.51
9	0.28	0.29	0.09	34	0.38	0.30	0.41
10	0.27	0.32	0.09	35	0.38	0.30	0.46
11	0.37	0.33	0.51	36	0.34	0.28	0.50
12	0.25	0.54	0.43	37	0.55	0.29	0.40
13	0.23	0.36	0.28	38	0.40	0.27	0.50
14	0.31	0.42	0.47	39	0.42	0.50	0.43
15	0.53	0.49	0.57	40	0.56	0.51	0.40
16	0.25	0.32	0.50	41	0.52	0.43	0.53
17	0.32	0.38	0.62	42	0.28	0.34	0.34
18	0.26	0.37	0.41	43	0.34	0.36	0.50
19	0.59	0.55	0.40	44	0.46	0.41	0.61
20	0.35	0.35	0.44	45	0.42	0.52	0.57
21	0.23	0.38	0.47	46	0.44	0.36	0.55
22	0.31	0.29	0.54	47	0.40	0.51	0.42
23	0.40	0.35	0.32	48	0.35	0.52	0.50
24	0.29	0.21	0.29	49	0.41	0.36	0.53
25	0.35	0.26	0.37	50	0.51	0.33	0.57
Average					0.40	0.33	0.37

excursions of about one or two per mm of traverse of the fiber. The average displacement of the ordinate on the data for a typical fiber was 0.60" and the maxima and minima varied from this by ±0.50" to ±0.60", i.e., there was approximately a displacement variation of ±100% from the average compared to nylon at about ±50%. The curves of occasional fibers of cotton were of much less variation, however, and sometimes very large displacements occurred at particular sites which repeated on the second run. These were +200% or more. The character of the data changed somewhat with load, a slightly higher rate of variation occurred at higher loads. Excessive bouncing also appeared to occur under these loads although the load did not go to zero and only the stick-slip rate may have increased. In some cases large fluctuations increased markedly at high loads (28.5 mg).

As may be seen from Table 2, coefficients of friction obtained by the straightforward statistical integration of the curves and the value

$$\mu = F/W$$

gave the values of μ as 0.40, 0.33 and 0.37 for fibers of 3/4", 1", and 1-1/4" lengths respectively. All data, irrespective of load, were used and some values are obviously very low (as low as 0.09). The highest value registered was 0.62. It is quite apparent that the dispersion was unacceptably large, but how much may be ascribed to the resettability of the apparatus has not yet been determined.

Considering now only typical 11.5 mg or 12.5 mg loads we have for the various fibers the data in Table 3. In these data curves indicating change of recorder zero, other irregularities, or loads greater than 12.5 mgs were discarded. The zero change, possibly due to misalignment of the fiber carrier from a vertical plane, appeared to be a real source of error in a

number of cases as it could add an increment of 20% or more to the average force displacement calculation.

By correcting the data as indicated and using the lower loads in Table 3 we obtain the values 0.39, 0.43 and 0.44 for the coefficient of friction of fibers of 3/4", 1", and 1-1/4" lengths respectively. These are greatly different from the previously cited data and are believed to be more accurate for the load range indicated.

Also by reviewing all of the data of Table 2 for data of high and low loads, and using only data which discarded questionable zero return values, the data of Table 4 were obtained. Here we observe for 1" fibers the coefficient of friction of 0.43 for loads of 11.5 to 12.5 mg (taken for 19 specimens) and 0.30 for loads of 26 mg.

It is thus apparent that data obtained at various loads cannot be lumped into a statistical average of the coefficient of friction for cotton and that the values cited for average coefficients of friction in Table 1 must be discarded. The values obtained from Table 3 appear to be more nearly valid. Whether the difference shown between the various fibers is real or not cannot be stated at this time pending further development of the friction measuring instrument. It is interesting to note that the average of the three values is 0.42 and that the variation from the mean of the three is only slightly more than $\pm 5\%$.

e. Fiber Friction of Dewaxed Cotton

Specimens of Empire WR Cotton dewaxed with cold chloroform for purposes of infrared absorption spectra study were obtained and friction measurements were made for these fibers as for the preceding specimens. Data was obtained for 10 fibers (2 runs each) and the values obtained are

TABLE 3

Selected Data Exhibiting Coefficients of Friction of Empire WR Cotton for Various Fiber Lengths Under Loads of 11.5 - 12.5 mg.

No.	3/4" Value of μ	1" Value of μ	1-1/4" Value of μ
1.	0.40	0.49	0.41
2.	0.35	0.51	0.47
3.	0.37	0.34	0.50
4.	0.31	0.36	0.41
5.	0.53	0.43	0.40
6.	0.32	0.41	0.44
7.	0.35	0.52	0.54
8.	0.31	0.36	0.32
9.	0.29	0.52	0.42
10.	0.34	0.36	0.51
11.	0.33	0.33	0.28
12.	0.48	0.54	0.46
13.	0.52	0.36	0.50
14.	0.37	0.42	0.40
15.	0.52		0.50
16.	0.36		0.43
17.	0.38		0.40
18.	0.38		0.53
19.	0.40		0.34
20.	0.40		0.50
21.			0.42
22.			0.50
23.			0.37
24.			0.38
25.			0.53
	0.39	0.43	0.44

TABLE 4

Data Comparing Coefficients of Friction of Empire WR Cotton Fibers of 1" Length at Different Loads

Test Number	Load 11.5 - 12.5	Test Number	Load 26 mg.
1	0.49	1	0.35
2	0.32	2	0.38
3	0.38	3	0.29
4	0.37	4	0.35
5	0.55	5	0.21
6	0.51	6	0.26
7	0.34	7	0.31
8	0.36	8	0.24
9	0.43	9	0.34
10	0.41	10	0.27
11	0.52	11	0.32
12	0.36	12	0.23
13	0.51	13	0.25
14	0.52	14	0.38
15	0.36	15	0.30
16	0.33	16	0.30
17	0.54	17	0.28
18	0.36	18	0.29
19	0.42	19	0.27
Average	0.425	Average	0.295

shown in Table 5. A data plot for a typical fiber is shown in Figure 16.

A particular characteristic of these curves was a high rate of stick-slip incidence as for nylon, about 20 per inch on the data plot or essentially 7 per mm of fiber length. The incidence of large fluctuations was approximately 5 per inch of plot or 1.7 per mm of fiber length. Fluctuations from the mean were approximately ± 0.40" from a mean of 0.60", or essentially $\pm 67\%$. However, occassional peaks went much higher as seen in Figure 16, to $+200\%$ or more. Examining the data of Table 5 we find a coefficient of friction value for 10 specimens as being 0.39. Reexamination of the recorder sheets indicates that the value 0.23 is probably in error due to poor zero registry. Discarding this value, we obtain an average coefficient of friction of 0.41 for fibers of 1" length.

This value is insignificantly different from values obtained for fibers on which natural wax was present. On the other hand, the character of the data plot, as described above and displayed in Figure 16, is somewhat different as to rate of stick-slip occurrence, rate of major fluctuation occurrence, and low variation of the fluctuations from the main displacement over a large part of the curve. However, this variation occasionally did exhibit very large fluctuations and generally speaking the character of the plotted data was between that of nylon and natural cotton except for these large excursions. It was noted that these particular dewaxed specimens handled differently from the natural cotton and may have been damaged by the extraction treatment.

The low average coefficient of friction is surprising and the source of the large excursions has not been determined. The coefficient of static friction may not be determined from this value alone, but from seven maxima

TABLE 5

Coefficients of Friction of Specimens of Dewaxed Empire WR Cotton Fibers

Test Number	Average Force (mg.)	Normal Force (mg.)	Friction Coefficient
1	4.37	11.5	0.38
2	4.14	11.5	0.36
3	4.83	11.5	0.42
4	5.40	11.5	0.47
5	4.49	11.5	0.39
6	5.18	11.5	0.45
7	4.95	11.5	0.43
8*	2.64	11.5	0.23
9	5.86	11.5	0.51
10	3.34	11.5	0.29
Average	4.52	11.5	0.39*

* Average of nine specimens, omitting No. 8, is 0.41 for the coefficient of friction. This latter value appears to be more nearly valid.

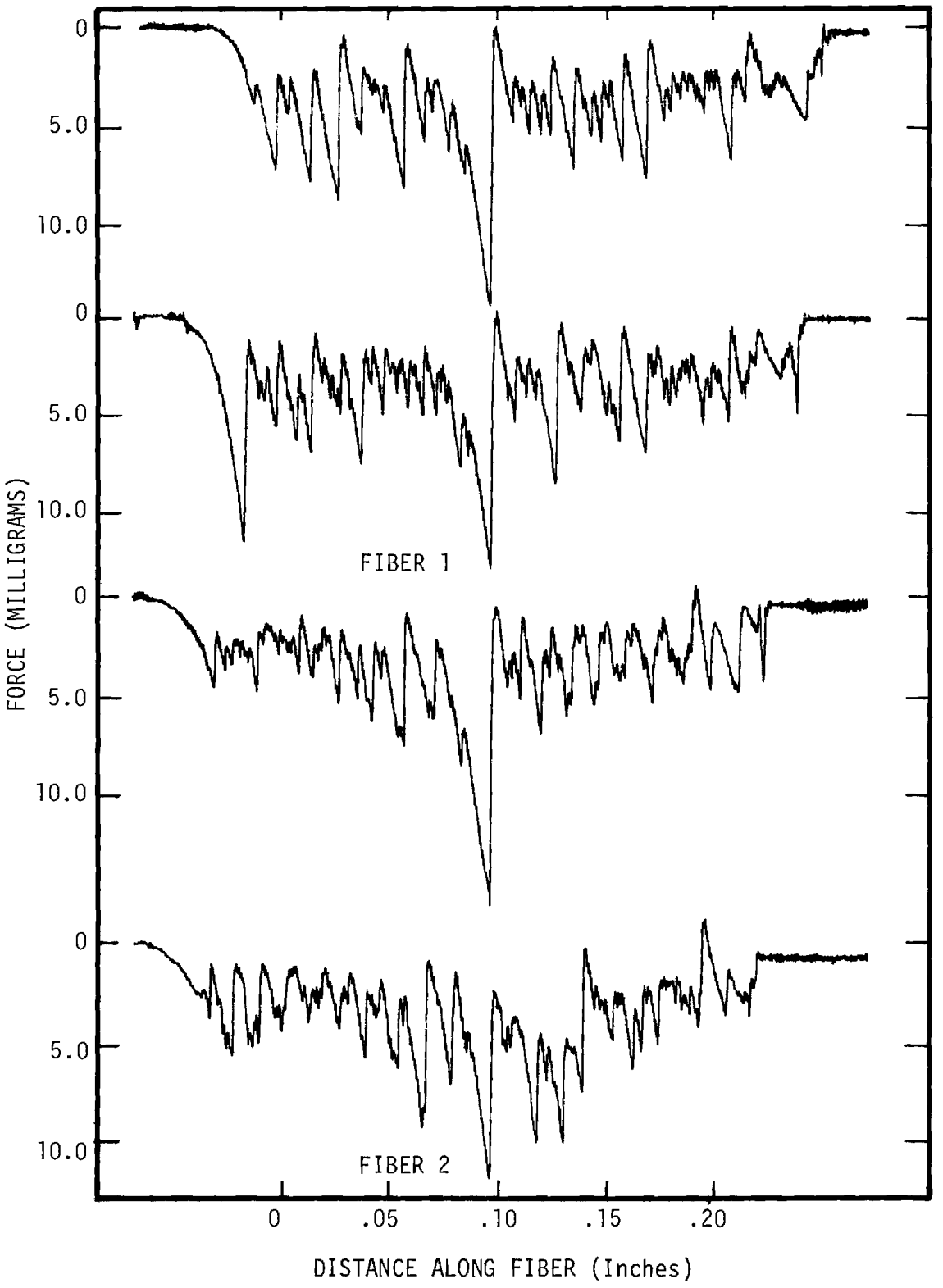


Figure 16. Frictional force data for representative fibers of 1 inch dewaxed Empire W R cotton, 11.5 mg. normal force.

including the large one it becomes 0.72 and omitting the large one it becomes 0.64. For the very large excursion it is 1.34*. The possibility of a twist or tangle at the large excursion exists and the need for microscopic examination during the experiment is apparent here.

Large excursions did occur, however, in about five out of the ten dewaxed fibers examined. Since the experiments constitute a rather limited sampling of dewaxed fibers, the results recorded must be considered as only pointing to tentative behavior, which can only be confirmed by a much more statistically significant sampling.

f. Coefficient of Static Friction

In many textile processes the coefficient of static friction may be more important than the coefficient of sliding friction. From examination of the data obtained for the various fibers, the maximum positive fluctuation can be used for obtaining a value for the static coefficient as is shown below.

		Coefficient of sliding Friction μ_k	Fluctuation	Coefficient of Static Friction μ_s
Cotton Empire WR	Nylon	0.40	50%	0.60
	3/4" staple	0.39	100%	0.78
	1" staple	0.43	100%	0.86
	1-1/4" staple	0.44	100%	0.88
	Dewaxed staple	0.41	75% to 200%	0.72 to 1.34 (max)

* See next paragraph for discussion of coefficient of static friction.

More precise values can be obtained by a more extensive reanalysis of the data on hand, and this analysis will be completed in the near future. The lower value of the average static coefficient of friction for the dewaxed cotton than for that of the natural cotton is a significant difference, but is not yet explicable. The extremely high value also for occassional maxima is important if this proves to be a normal behavior for dewaxed fibers.

g. Measurements of Cotton Fiber Clinging Power

Utilizing the apparatus described in Section D 3d the clinging power of 20 fibers of Empire WR was measured by withdrawing the fibers successively from the pads adjusted as nearly as practicable to the same pressure between pads. The data obtained are summarized in Table 6. There is a considerable dispersion between values, but the average value is 18.3 mg. This is approximately four times the sliding frictional force or twice the static frictional force between single crossed cotton fibers under a load of 11.5 mg. The range of withdrawal forces is from 11.2 to 28.1 mg or -7.1 mg to + 9.8 mg from the average withdrawal force. This amounts to essentially a ± 50% fluctuation from the average withdrawal force.

We do not yet have data showing the effects of increasing force between the fiber pads or for a known force; however, modifications to the apparatus to incorporate measurements of the force between the pads is planned. Subsequently data showing effects of force changes, fiber lengths, etc., will be obtained. It will be interesting to note whether an increase in force between the fiber pads reveals a decrease in the coefficient of friction for the fibers when their surfaces are being moved across each other with axes essentially parallel, as has been noticed for motion with axes at right angles with the servo-controlled friction apparatus in the preceding experiments.

TABLE 6

Frictional Force Developed Between a Single Cotton Fiber and a Parcel of Parallel Cotton Fibers

Test Number	Recorder Pen Deflection (Inches)	Force (mg.)
1	2.90	22.4
2	1.45	11.2
3	1.95	15.0
4	2.95	22.7
5	3.75	28.9
6	3.50	27.0
7	2.45	18.8
8	2.80	21.6
9	1.80	13.9
10	1.80	13.9
11	3.40	26.2
12	1.80	13.9
13	1.75	13.5
14	2.00	15.4
15	1.50	11.6
16	1.90	14.6
17	2.50	19.2
18	3.65	28.1
19	1.75	13.5
20	2.05	15.8
Average	2.38	18.3

Max 28.9
Min 11.2
Median 15.4 15.8
15.6
Average 18.3
Fiber contact zone 7/32" linear dimension

5. Summary

It has been stated by Bowden and Tabor that friction between polymers at small velocities can be based with little error on the classical friction law of Amonton's, namely $F = \mu W$. However, over a large range of loads it was found that μ varied according to the expression $\mu = kW^{-B}$ where k is an undesignated constant and B has a range of 0.2 to 0.3. At higher sliding velocities it was pointed out that hysteresis losses in the polymers might be expected to become an appreciable factor in friction measurements.

A frictional apparatus, utilizing a balance mechanism for application of a normal force or load and a servo-controlled galvanometer and an XY plotter for measuring the tangential force, has provided a sensitive means of measuring frictional forces of the order 10^{-5} gram. Using this device fiber to fiber frictional measurements were made of nylon, Empire WR Cotton of staple lengths 3/4", 1", and 1-1/4", and the same cotton dewaxed by solvents.

For normal forces of about 11.5 milligrams the value of μ for nylon fibers of 0.0008" (19 microns) diameter was found to be 0.40 and for Empire WR Cotton the values were 0.39, 0.43, and 0.44 for fibers of lengths 3/4", 1", and 1-1/4" respectively. If the load on the cotton fiber was increased to 26 milligrams the frictional coefficient was reduced to about 0.30 in one series of measurements. For specimens of dewaxed cotton a coefficient of friction of 0.41 was determined.

Of considerable interest was the character of the plots of frictional force variation with fiber traverse. Those of nylon exhibited a high rate of stick-slip force fluctuation. These varied from the mean about $\pm 50\%$.

For nylon and dewaxed cotton the stick-slip fluctuations were about 7 per mm of fiber traversed.

The stick-slip rate for cotton was about one-half that for nylon and frictional force fluctuations were ±100 per cent of the mean value. This means that the coefficient of static friction for cotton fibers was about 0.80 compared to 0.60 for nylon. For dewaxed cotton an average variation obtained was about ±75% or 0.72 for μ_s. However, in a number of fibers very large excursions were observed as high as +250% representing a μ_s value of 1.34. The limited data obtained for dewaxed cotton thus far permits only preliminary observations at this time.

The character of the recorded data plots of the frictional measurements furnished significant information concerning frictional behavior of the various fibers; and in early measurements with this particular instrument the character of the data plots appears of more significance than the mean values of coefficient of friction determined for the respective fibers. As is fairly obvious the present frictional instrument requires some refinement and further development to eliminate sources of error now known. These include some variation in friction of the balance-arm bearing and loss of calibration of the normal load; change in zero registry during the course of a run is thought to be caused by slight misalignment of the fiber holder from a vertical plane. Improvement of these features is expected to lead to improved data and more accurate and consistent measurements of the frictional behavior of fibers.

E. INFRARED ABSORPTION SPECTRA OF COTTON AND COTTON WAXES

1. Introduction

The frictional properties of cotton fibers are related in part to materials which are intrinsic to the natural fiber surface such as cotton

wax. Hence, the presence, absence, or change in the constitution of this wax or of any other coatings which may be accidentally acquired or intentionally applied in processing, are undoubtedly of interest to a study of fiber friction. One of the potentially valuable methods for examining such coatings is infrared spectroscopy; hence, this method was utilized to provide pertinent information for our investigation of fiber friction. In addition, infrared spectroscopy has many other applications to textile investigations related to fiber structures, fiber coatings, and fiber chemistry. However, the principle objective of the current study was to develop sufficently skills in the infrared spectroscopy of textile products to make use of this excellent tool in the frictional studies of cotton fibers.

2. Literature Survey

a. General

Search of published research work on the infrared spectroscopy of cotton, cellulose and other textile materials has been made to determine the work already accomplished in this field and possible applications of spectroscopy to textile research. The search was conducted at Price Gilbert Memorial Library.*

Chemical Abstracts (1947-1964), Textile Technology Digest, Summary of Current Literature, and The Journal of the Textile Institute (1950 - March 1965) were examined under the following topics:

Arnel (cellulose triacetate)	Polyester
Cellulose	Dacron
Cellulose acetate	Rayon

* A selected bibliography of the literature examined appears in the Appendix.

Cotton	Spectra
Cotton waxes	Ultra violet
Polyacrylonitriles	Infrared
Orlon	Spectrometry
Polyamides	Viscose
Nylon 6	
Nylon 66	

A total of 123 articles was found and a card file of references was prepared. These were then studied in detail insofar as pertinent to the planned work.

b. Techniques of Fiber Preparation

The techniques of application and preparation of fiber samples for IR spectroscopy cited in the literature have been studied. These methods fall into the following general categories.

(1) Ground fibers dispersed in an alkali halide pressed pellet (KBr, KCL, AgCl).

Method:

(a) Grind fiber in Wiley mill;

(b) Grind together approximately 150 to 300 mg dry halide and 1 to 3 mg fiber in a small mortar.

(c) Evacuate sample in die.

(d) Apply pressure slowly to 50 tons/in^2 maintaining pressure 5 to 10 minutes.

(e) Remove pellet.

(2) Direct pressing of fibers to obtain a thin film

(a) Continuous filament synthetic

The continuous filament is wound on a fork to obtain a

parallel arrangement which is then pressed.

(b) Natural (short staple) fibers

Thin films of these fibers may be obtained directly by grinding and pressing. This method is extremely tedious, and specimens giving reproducible results are very difficult to prepare.

(3) Immersion techniques

The fiber sample may be ground or left intact. The fiber, suspended in an immersion medium (nujol, etc.), is sandwiched between plates transparent to IR radiation (KBr or NaCl pellets).

(4) Miscellaneous techniques

(a) Micro techniques

IR radiation is focused on a single fiber.

(b) Reflectance techniques

This technique was not used as a common method of analysis of natural polymers (thin films were used).

(c) Dispersion of polymer into colloidal state

This method can be used on the synthetic polymers; however, physical structure is drastically changed.

(5) Comments

Most of the references on techniques of sample preparation and on cellulose and cotton have been carefully examined. The references on cellulose and cotton show that infrared spectral data may be used in determining physical and chemical modification, crystallinity, and fiber axis orientation in cellulose I, II, III, and IV. Most of these investigations were conducted in the 2 - 15 μ region with NaCl optics. A number of authors noted that greater resolution than that of NaCl optics

and more thorough study of the near infrared would be desirable in the study of cellulose. Micro-techniques were mentioned as being well suited for the study of fibers.

c. Summary

The reference literature delineated the techniques and the usefulness of infrared spectroscopy in textile research investigations; and the equipment on hand appears adequate for the initial phases of this investigation.

3. Experimental Work

a. General

On completion of the literature search the infrared group concentrated on developing techniques using existing equipment in the study of cotton and on determining what additional equipment and/or sampling methods could be used. The specific goals were to develop competence in obtaining spectra of cotton fibers and of cotton wax.

b. Apparatus

The following equipment is currently being used in the study:

(1) Perkin Elmer Model 221 double beam infrared spectrophotometer (NaCl optics),

(2) Matched pairs of liquid sample cells,

(3) Equipment necessary for producing KBr disks,

(4) Wiley Mill for preparing cotton samples,

(5) Soxhlet and other extraction equipment for obtaining wax samples, and

(6) A precision balance (used in wax study).

Equipment being considered for future use includes:

(7) Perkin Elmer Reflectance Attachment,

(8) Wilks Double Beam Multiple Internal Reflection Attachment, and a

(9) Specially designed apparatus for extracting cotton wax at room temperature at reduced pressure.

c. Infrared Spectra of Empire WR Cotton and Extracted Cotton Waxes

The work done to date has been based on and has agreed with previous infrared studies of cotton as revealed by our recent literature search. Spectra of cotton fibers and various synthetics have been obtained using the KBr pellet method. Spectra have been obtained using the KBr and liquid cells of cotton wax extracted with[14] both hot and cold solvents. Several methods and solvents have been tested to determine the most desirable extraction procedure.

(1) A hot Soxhlet extraction using 95% ethanol was performed[15]. 76% of the wax was obtained in a twenty-four hour period.

(2) "Hot" chloroform extraction (at the boil) was run. The spectra of material obtained by methods (1) and (2) differed significantly, probably due to alcholysis of the esters present in the wax.

(3) To minimize the chance for changes in the composition of the waxes, a "cold" chloroform extraction (at room temperature) was run. Samples were taken at various times to establish that total extraction had occurred. These spectra were similar to those obtained by hot chloroform extraction; however, this is not a guarantee that the waxes are similar. The weight of the wax obtained from each aliquot indicated that complete extraction would require 28 days or more as shown in Table 7.

TABLE 7

Weight Versus Time Data for Wax Extracted at Room Temperature in 27 Days from Empire WR Cotton Fibers by Static Chloroform Technique

Time (hours)	Amt. of Wax (mg./25 ml. sample)	Comment
12.5	1.73	This experiment revealed two things:
22.5	1.82	(1) The sampling techniques used must be greatly improved so that more accurate data can be obtained.
40.3	1.83	
63.8	2.66	
67.8	1.77	(2) The time required for total wax removal by static room temperature extraction is inconveniently large for our work.
69.8	2.12	
135.8	2.60	
143	2.03	
523	2.76	
648	4.26	

(4) Cold extractions with chloroform and hexane have been started. Extraction with methylisobutylketone is going to be conducted.

(5) Glassware has been made to adapt a Soxhlet for operation at reduced pressure, thus allowing rapid cold extractions.

(6) A moisture regain study of the standard cotton sample was made (moisture regain 13.9%), and methods were adapted for handling of samples dried in a vacuum desiccator. All weighings are being made in the bone dry state since humidity and temperature control are impractical.

Representative spectra of the following samples among others were obtained and are displayed in Figures 17 to 19.

(1) Cotton (Standard Bale) Not Dewaxed

(2) Cotton (Standard Bale) Dewaxed

(3) Viscose (Commercial Rayon)

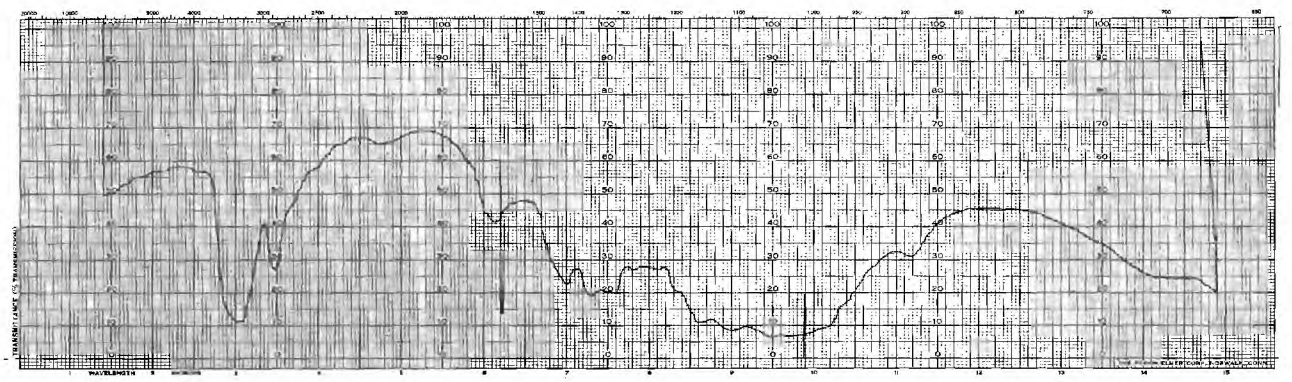
(4) Cotton Wax - extracted with hot ethanol

(5) Cotton Wax - extracted with hot chloroform

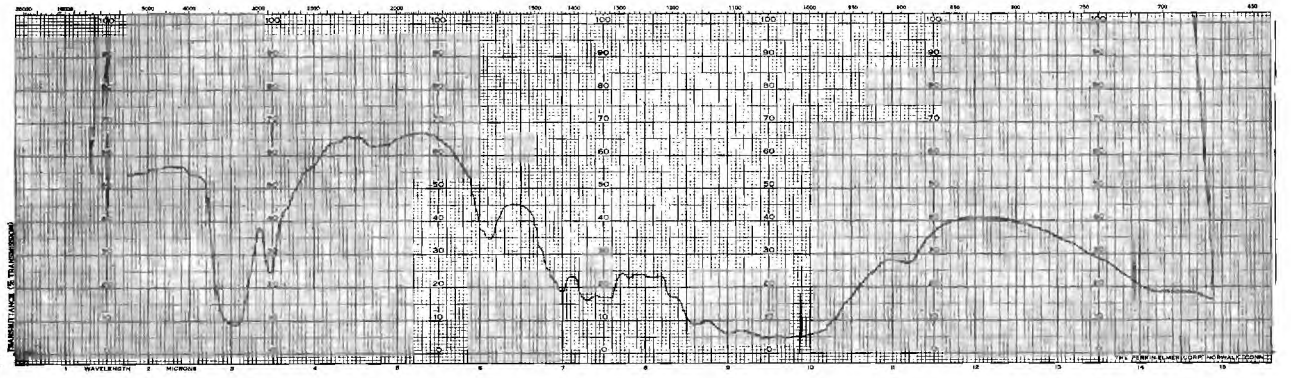
(6) Cotton Wax - extracted with cold chloroform

These spectra were quite reproducible and are representative of some 35 spectrometer recordings made.

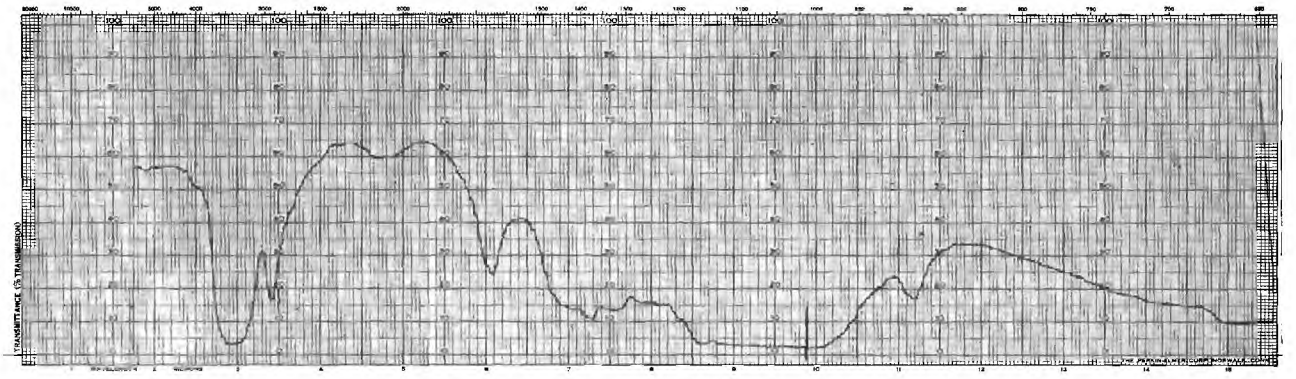
Of particular interest is a comparison of the data of Figures 17, 18 and 19. Here it will be observed from Figures 18 and 19 that cotton wax in heavy concentrations has a number of strong bands of 3.45 μ, 5.83 μ, 7.25 μ, 7.95 μ and several bands in the region 8-10 microns. Some of these are coincident with the strong bands of the cotton itself. However a band in the wax spectrum appearing at 5.80 microns is at a position not masked on the cotton spectrum. Examination more closely of this band at 5.80 μ on



A. Natural Cotton

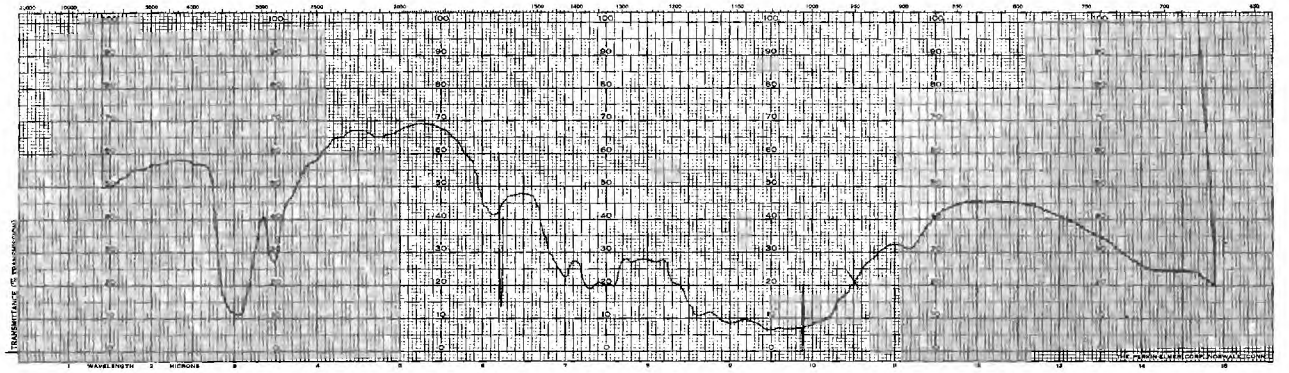


B. Dewaxed Cotton

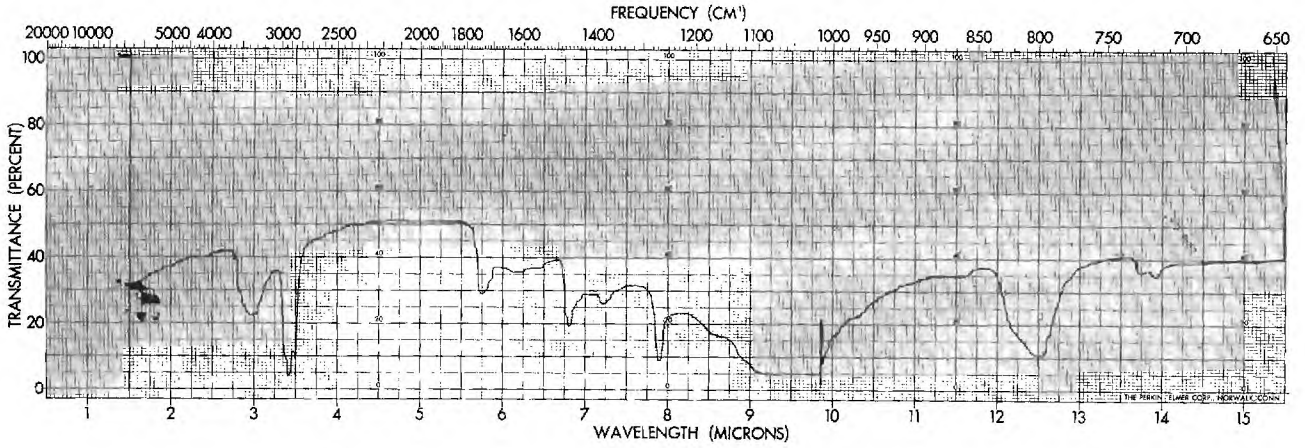


C. Rayon

Figure 17. Comparison of spectrum of natural Empire W R Cotton with dewaxed cotton from the same bale and with rayon.

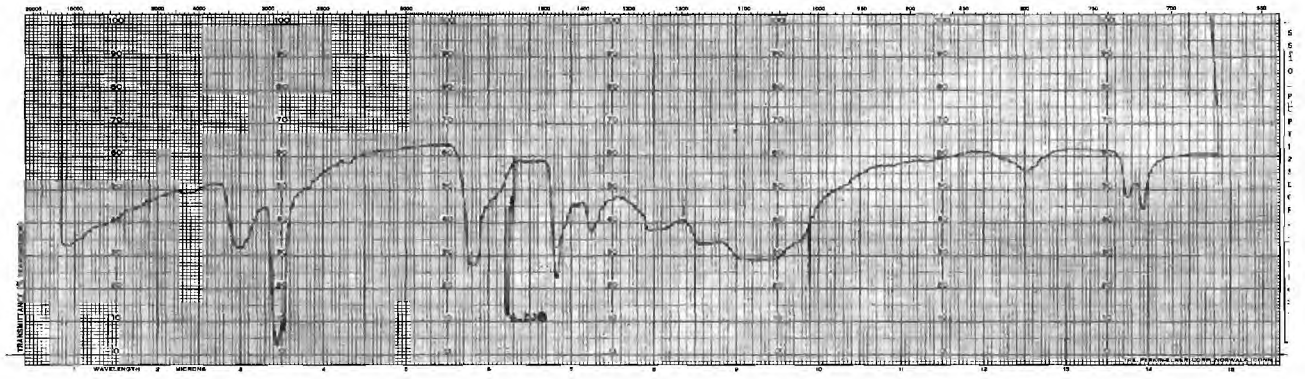


A. Natural Cotton

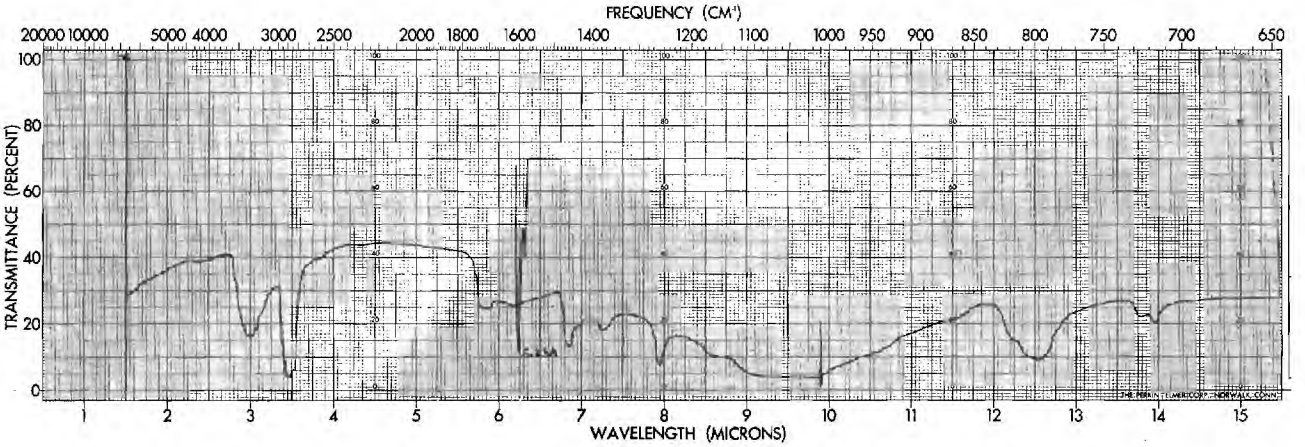


B. Cotton wax from cold chloroform extraction

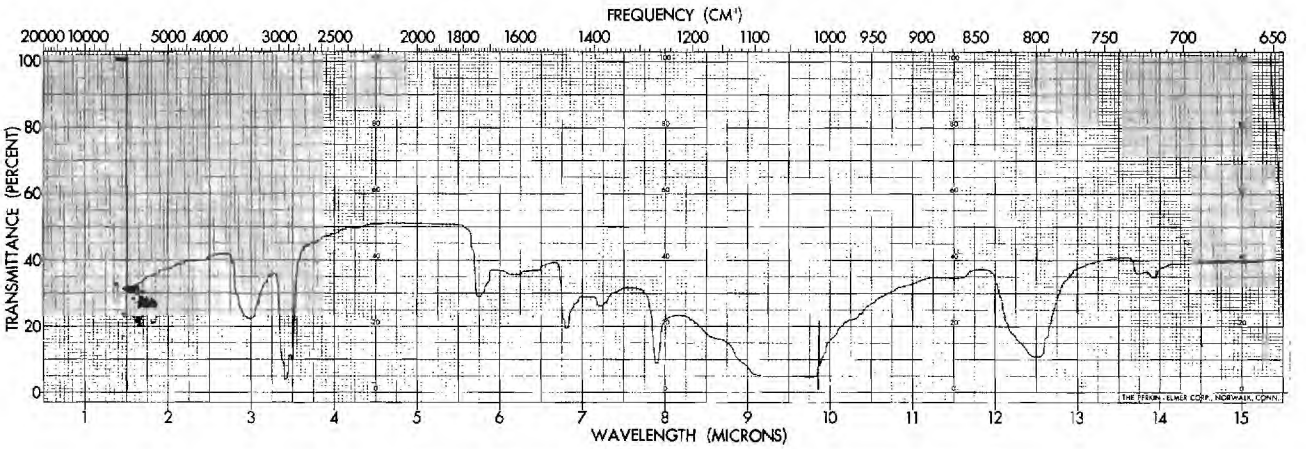
Figure 18. Comparison of spectrum of natural Empire W R Cotton with that of wax extracted from a specimen of the same cotton with cold chloroform.



Wax extracted with hot ethanol



Wax extracted with hot chloroform



Wax extracted with cold chloroform

Figure 19. Comparison of spectra of cotton waxes extracted by various methods.

the natural and dewaxed cotton reveals a small dip in the natural cotton spectrum not visible in the dewaxed cotton. This appears to provide one position which may be examined profitably at higher amplification to reveal the presence of the cotton wax. There also appears to be a region in the 8-10 micron band where appreciably more absorption occurs in the case of the natural cotton. However, the relative weakness of the 5.80 μ band and the uncertainty of the meaning of the 8-10 μ absorption, in the current sample preparation methods for direct transmission spectra of the cotton fibers, gives little hope of actual successful quantitative studies by the transmission method outlined of any wax degradation which may occur. Hence, information of significant value with regard to the presence of degradation of cotton wax appears unlikely by the techniques used thus far.

The other spectra furnished in Figure 19 delineate the data obtained for the waxes obtained by hot and cold extraction methods. The cold extraction method was believed to remove principally the outer wax rather than the additional material which may be deep in the interior of the fiber or in the lumen. This spectrum appears in Figures 18 and 19. It will be noted, however, that the cold extraction with chloroform resembles closely that using hot chloroform; the specimen extracted with the cold liquid actually has the greatest absorption in the cases displayed.

The spectrum made from the hot ethanol extract resembles quite closely those for wax extracted by chloroform, although absorption bands were more accentuated in this particular specimen.

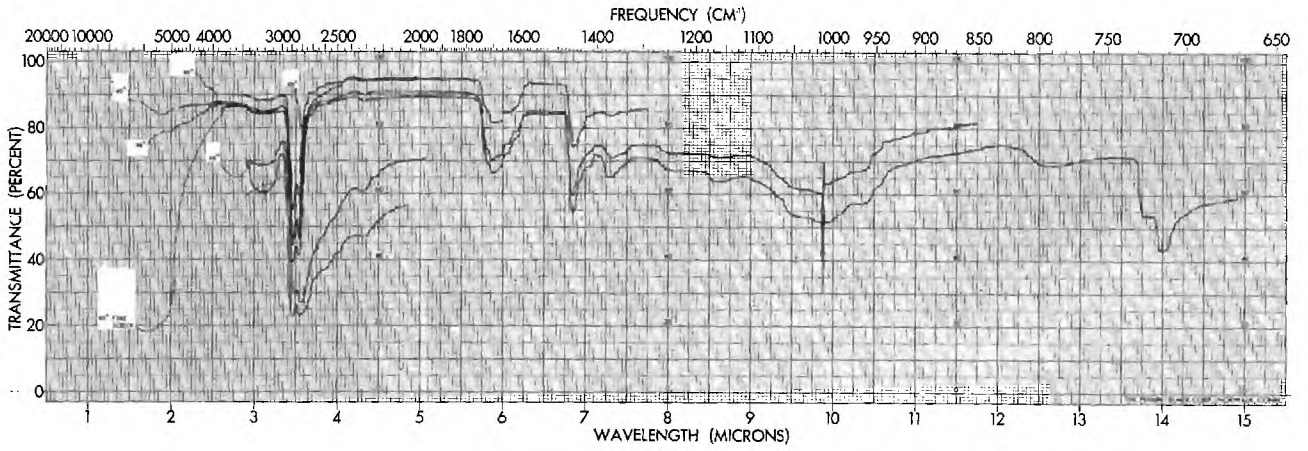
The spectrum of rayon is included in Figure 17 to indicate the close resemblance of this regenerated cellulose fiber to that of the natural cotton. The wax absorption band at 5.80 μ is missing in the rayon spectrum.

d. Studies by Means of a Reflection Attachment

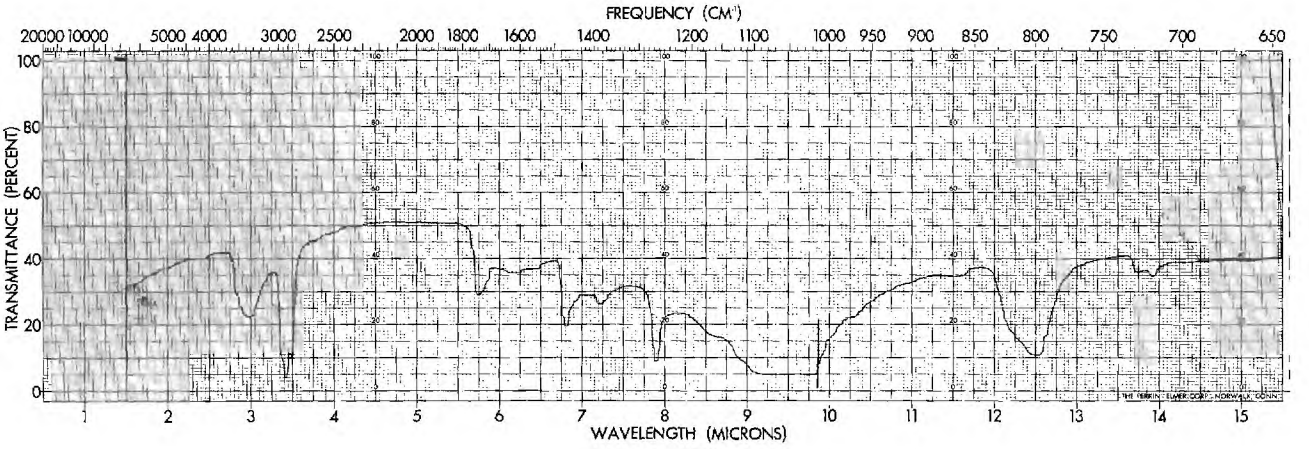
A reflection attachment provided by the manufacturer of the IR instrument was used to make a number of spectra of extracted cotton wax and of several organic liquids. A typical example is shown in Figure 20 of a reflection spectrum obtained for cold-chloroform-extracted wax compared to that of the patternof the wax obtained by transmission. It will be observed that the bands of greatest absorptivity are clearly observed at the proper sites in the reflection spectra. The absorption varies greatly with the angle of incidence, and an angle of 46° is the proper one.

In this spectrum the wax was in highly concentrated form smeared on the teflon backing plate of the reflectance optic. Reflectance absorption spectra of organic liquids made in the same manner possesed fewer and less intense absorption bands than transmission spectra of the same liquids, only the more intense bands being registered. It did not appear from the experiments that a single reflectance would give information on cotton waxes superior to that obtained by the transmission method. The critical reflection angle and the susceptibility of the instrument of a maladjustment by slight shocks were also undesirable features.

A second reflection attachment is under consideration for evaluation. Being based on the principle of multiple internal reflection it is believed that it holds some prospect for application to the analysis of surface coatings of cotton fibers. The reflection attachment, ideally, has a feature of primary interest in that it allows examination of fibers without macerization and pelletization. For this reason alone it is worth further investigation and evaluation. It has other values as well with regard to examination directly of the undisturbed fiber in yarns and fabrics.



Reflectance Spectrum.



Transmission Spectrum

Figure 20. Comparison of reflectance and transmission spectra of cotton wax extracted with cold chloroform.

e. Summary

Excellent progress has been made in developing skills in the infrared spectroscopy of fibers, and typical spectra of natural cotton, dewaxed cotton, and cotton waxes have been obtained and partially analyzed. An absorption band appearing at 5.80 microns appears to be a characteristic band which will identify the presence or absence of wax as this band is present in the spectrum of the natural cotton, but not in the spectra of dewaxed cotton or of rayon. The weakness of the band, however, in the transmission pattern does not indicate the probability of significant information being obtained from it concerning slight degradation of the wax of natural cotton due to weathering or minor processing steps.

An evaluation of an infrared reflectance attachment indicated that the design of this particular accessory limited information gained to that concerning only the stronger absorption bands and could not be expected to give information significant to the study of cotton waxes or wax degradation. A reflectance attachment using a multiple internal reflectance system holds promise of greater sensitivity for the examination of fiber surface coatings, and its possible procurement is being investigated.

F. X-RAY DIFFRACTION STUDIES OF COTTON FIBERS

The x-ray diffraction study of fibers was delayed somewhat by lack of personnel and equipment available for application to the problem immediately upon the initiation of the research on cotton fibers. However, work was commenced on June 15, 1965.

The initial effort was devoted to a study of the literature and of the techniques employed in x-ray diffraction studies of fibers.

The application of x-rays to fibers was explored along with the methods used in determining the structure of special fibers (notably collagen). Problems presented by other fibers and some of the previous approaches taken in the attempt to analyze them were examined. Discussions were found of some of the experimental difficulties peculiar to low-angle scattering (beam collimation, resolution and sensitivity needed) and of the varied laboratory techniques employed to overcome them. A brief summary of this information was written and filed for subsequent use.

A more formal research of the literature was then undertaken. The Chemical Abstracts Index yielded a number of papers on x-rays, and on their application to fibers, cellulose, and cotton in particular. Upon locating the abstracts, some 150 of these were collected. After a close inspection of these abstracts, papers dealing with problems and results of previous experiments directed along the same general line as our own research were examined. The field was narrowed further by inspection of these articles to about sixty papers.

A careful study of these papers is currently being made, and necessary equipment is being readied for the experimental work.

IV. CONCLUSIONS AND RECOMMENDATIONS

The research program on the frictional properties of cotton fibers has progressed satisfactorily with respect to cotton procurement, fiber characterization by statistical and microscopic examination, and the experimental work on friction measurement and infrared spectrophotometer studies of cotton fibers and cotton waxes. The x-ray diffraction work is in its beginning stage.

The optical and electron microscopy work progressed favorably except for sectioning techniques, and correct instructions and equipment are now in our possession, or on order. Establishment of good sectioning methods will provide us with better information concerning fiber diameters, structures, and variations for correlation with frictional measurements.

A fiber friction measuring instrument, the design of which incorporates an electrical servo-controlled force balancing device for tracking and determining frictional force fluctuations, has been built and has provided a quantity of interesting data on the friction of fibers of nylon, cotton staple lengths 3/4", 1", and 1-1/4", and of dewaxed cotton. Friction coefficients derived from the average force exerted during a run were in the range of 0.39 to 0.44 for all these materials. However, the coefficient of static friction derived from the peak forces occurring was much larger for the material cotton than for nylon, 0.80 versus 0.60. The coefficient of sliding friction for the dewaxed cotton, surprisingly, was in the same range as the natural cotton. However, the coefficient of static friction was somewhat less except for a number of large excursions which have not yet been interpreted.

The data plots of frictional force versus position along a fiber had

much character with respect to rate of stick-slip fluctuations, maximum force, and minimum force and additional study of these parameters is expected to reveal new and important information.

Improvements in the instrument, especially with respect to the adjustment and maintenance of calibration of the normal force applied, are necessary to improve the accuracy and consistency of measurements. Considering the present probable error in the coefficients determined thus far for average sliding friction, at least ±5 per cent, it is not believed the cited values have much significance except that for all fibers tested they were very similar. On the other hand the character of the curves and the coefficients of static friction were significantly different, and these factors may become the chief areas allowing descrimination between fibers subjected to textile processing stages. The fact that the developed instrument does display this discriminatory capability is rewarding and may become of considerable significance in this study.

The work in infrared spectroscopy advanced to the state that excellent and reproducible infrared absorption spectra were obtained for cotton, dewaxed cotton and extracted cotton waxes.

A minor absorption band at 5.80 μ was the only one of considerable strength in the wax spectrum which was not coincident with major bands in the cotton spectrum. The presence or absence of this band indicates the presence or absence of cotton wax. However, it is too weak to be utilized for other than very crude quantitative measurements. Evaluation of a reflectance attachment for the spectrophotometer yielded similarly unrewarding results. However, a second reflectance attachment utilizing a multiple internal reflectance principle with respect to the specimen holder and specimen is expected to give better results. In general, the progress in infrared

spectroscopy was considered excellent for the period.

The x-ray diffraction work commenced somewhat late, but the experimental program is now underway and useful data will be forthcoming shortly.

The student personnel employed in the program have performed excellently in this interdisciplinary effort and the progress made reflects the degree of their endeavor.

It is recommended that the work continue as outlined initially. The program is somewhat ahead of the expected schedule, but the friction measuring apparatus needs considerable modification to achieve its maximum potential. A maximum effort will be exerted to this end. Other areas will progress at a normal pace except the x-ray diffraction work which will be emphasized to bring it abreast of progress in the other phases.

V. PROGRAM FOR THE NEXT PERIOD

Work during the next period will be primarily a continuation of the research outlined in this report. Special emphasis will be placed on improved sectioning of fibers for viewing by optical and electron microscopy; the electron microprobe will be evaluated as a tool for fiber study; similar emphasis will be placed on refinement of the present friction measuring apparatus and delineation of its capabilities.

Upon completion of the necessary apparatus refinements additional studies of frictional characteristics of fibers will be undertaken. Checks of previous data will be made and studies of effects of fiber processing will be commenced.

The infrared spectroscopy work will be continued and a reflection attachment using multiple internal reflectance will be employed and evaluated.

The x-ray diffraction study of fibers will be advanced to the degree of competence necessary for the employment of the x-ray diffraction technique as a useful tool in this work.

Additional theses for the Masters Degrees will be commenced in the areas of fiber friction, infrared spectroscopy of fibers, and the microscopy of fibers.

VI. PERSONNEL

The individuals employed on this research during the period of this report are listed below.

Individual	Title	Area of Research
Richard B. Belser	Research Associate Professor	Project Director
James L. Taylor	A French Textile School, Director	Associate Project Director
William L. Hyden	Professor, Textile Engineer	Associate Project Director
John L. Brown	Director, Analytical Instrumentation Laboratories	Optical and Electron Microscopy
James L. Hubbard	Assistant Research Physicist	Optical and Electron Microscopy
James A. Knight	Research Professor, Chemistry Head, Radioisotopes Laboratory	Infrared Spectroscopy
Rick A. Porter	Graduate Assistant (Textiles and Chemistry)	Infrared Spectroscopy
Marvin P. Smoak	Student Assistant (Physics)	Infrared Spectroscopy
R. A. Young	Diffraction Laboratories, Director	X-ray Diffraction
Harry W. Ellis	Student Assistant (Physics)	X-ray Diffraction
Billy R. Livesay	Research Physicist	Friction Apparatus
Leland K. Jordan	Graduate Assistant (Physics)	Friction Apparatus
Thomas E. McBride	Graduate Assistant (Textiles)	Friction Apparatus and Fiber Microscopy
Lester D. Dozier	Assistant Research Scientist (Mechanical Engineer)	Friction Apparatus

In general, all work was performed on a part time basis except that of Mr. Dozier who was employed May 24, 1965 as a full time assistant to give continuity to the program of development of the fiber friction measuring instrument and its application in the research program.

REFERENCES

1. T. R. Boys, "An Evaluation of Factors Affecting the Frictional Properties of a Selected Cotton Fiber Sample," M.S. Thesis, Georgia Institute of Technology, p 47, 1964.

2. M. Shiloh and E. Alexander, "Investigation of Crimp in Cotton Fibers," Final Report, Contract No. FG-Is-103-60, UR-A10-(20)-5, Agricultural Research Service, USDA April 1965, 150 pages and Appendix, (Prepared by Institute for Fibers and Forest Product Research, Jerusalem, State of Israel).

3. M. Shiloh, J. Goldstein, D. Mejzler, and E. Alexander, "A Modification of the Method for Defining and Measuring Crimp of Textile Fibers," Textile Research Journal 31, 999-1006 (1961).

4. E. Alexander, M. Lewin, Y. Litav, H. Peres, and M. Shiloh, "The Crimp of Cotton Fibers," Textile Research Journal 32, 888-908 (1962).

5. G. Amontons, Memoires de l 'Academie Royale, p 206 (1699).

6. P. de la Hire, Histoire de l 'Academie Royale, p 104 (1699).

7. F. P. Bowden and D. Tabor, "The Friction and Lubrication of Solids," Parts I Clarendon Press (1950), Part II Oxford University Press (1964), pages 214-241.

8. A. Adderley, "The Clinging Power of Cotton Hairs," Textile Institute Journal XIII, 249-255 (1922).

9. J. C. Guthrie and P. H. Oliver, "Interfiber Friction," Journal of Textile Institute XLIII, T579-T595 (1952).

10. M. W. Pascoe, PhD Thesis, University of Cambridge (1955); and M. W. Pascoe and D. Tabor, Research 8, S15 (1955).

11. R. F. Penoyer, Rev. Sci. Instr. 30, 711 (1959).

12. F. B. Humphrey and A. R. Johnston, Rev. Sci. Instr. 34, 348 (1963).

13. M. Harris, "Handbook of Textile Fibers," Harris Research Laboratories, Inc., Washington, D. C., pp 183-186 (1954).

14. J. H. Kettering, "Solvent Extraction of Cotton Wax," Textile Research Journal 16, 627-634 (1946).

15. C. M. Conrad, "Determination of Wax in Cotton Fiber, A New Alcohol Extraction Method," Industrial and Engineering Chemistry 16, 745 (1944).

APPENDIX

BIBLIOGRAPHY
(Selected)

A. FRICTIONAL MEASUREMENTS OF COTTON FIBERS

1. Adderley, A., "The Clinging Power of Single Cotton Hairs," Textile Institute Journal, XIII, 249-255 (1922).

2. Balls, W. L., Studies of Quality in Cotton. London: Macmillan, (1928).

3. Bartlett, G. W., Smith, T. M. and Thompson, H. A., "Stick-Slip," Modern Textiles Magazine XXIV, 51 (1953).

4. Bartlett, G. W., Smith T. M. and Thompson, H. A., "Frictional Properties of Filament Yarns and Staple Fibers as Determined by the Stick-Slip Method," Textile Research Journal XXIII, (1953).

5. Bowden F. and Leben, L., "The Nature of Sliding and the Analysis of Friction," Proceedings of the Royal Society CLXIX, 371-391 (1938).

6. Chakrabarti, M. and Saha, A. N., "Lubricating Power of Oils on Jute," Textile Manufacturer LXXXI, 434-435 (1955).

7. Chakravarty, A. C., "Some Observations on Kinetic Friction of Jute," Textile Research Journal XXX, 592-597 (1960).

8. du Bois, W. F., "Frictional Measurements on Fibrous Materials," Textile Research Journal XXIX,451-466 (1959).

9. Fort, T. and Olsen, J. S., "Boundary Friction of Textile Yarns," Textile Research Journal XXXI, 1007-1011 (1961).

10. Frishman, D., Smith, A. L. and Harris, M., "Wool Fiber: Friction Measurement," Textile Research Journal XVIII, 475-480 (1948).

11. Guthrie, J. C. and Oliver, P. H., "Inter-Fiber Friction," Journal of Textile Institute XLIII, T579-T595 (1952).

12. Hood, B. G., "Frictional Properties of Textile Fibers," Textile Research Journal XXIII, 495-505 (1953).

13. Howell, H. G., "Inter-Fiber Friction," Journal of Textile Institute XLII, 521-533 (1951).

14. King, C., "Some Frictional Properties of Wool and Nylon Fibers," Journal of Textile Institute XLI, T135-T144 (1950).

15. Lindberg, J. and Gralen, N., "Frictional Between Single Fibers: II Frictional Properties of Wool Fibers Measured by the Fiber-Twist Method," Textile Research Journal XVIII, T227-T240 (1947).

16. Mercer, E. H. and Makinson, K. Rachel, "Textile Fibers: Frictional Properties," Journal of Textile Institute XXXVIII, T227-T240 (1947).

17. Morton, W. E. and Hearle, J. W. S., Physical Properties of Textile Fibers, Butterworth, London (1962).

18. Postle, L. J. and Ingham, J., "Measuring of Inter-Fiber Friction in Silvers," Journal of Textile XLIII, 43 (1952).

19. Stranton, T. E., Friction. London: Longmans (1923).

BIBLIOGRAPHY
(Selected)

B. Studies of Fibers (Principally Cotton) and Cotton Waxes by Infrared or Other Spectrophotometric Methods.

1. Techniques

1. Akselrod, V. S., "Pressed Samples for IR Spectrometry," Optics and Spectrose USSR 8, No. J, 379-380 (1960). (C.A. 40:4097).

2. Burgess, S. and Spedding H., "Sample Preparation of Fibers for Identification by IR Spectroscopy," Chem & Ind (London), 1166-1167 (1961).

3. Cannon, C. G., "Applications of Infrared Spectroscopy to Textile Problems," Chemistry & Industry No. 2, 29-33 (1957).

4. Cleverley, B. and Heamann, R., "Infrared Spectra of Textile Materials: Identification of Fibers and Pure Fabrics," J Appl Chem 11, 344-351 (1961).

5. Crook, A. and Taylor, P. J., "Simple Mulling Technique for the Preparation of Samples for Infrared Spectroscopy," Chem & Ind (London), 95 (1958) (C.A. 52:5976 fig).

6. Elliott, A., Hanby, W. E., and Malcolm, B. R., "The Near Infrared Absorption Spectra of Natural and Synthetic Fibers," Brit J Appl Phys 5, No. 11, 377-381 (1954).

7. Ermolenko, I. M. and Gusev, S. S., "On the Method of Measuring the Infrared Spectra of Cellulose Materials, "Vysokomolekulyarnge Soedmeniya 1, No. 3, 466-473 (1959). (J Poly Sci 39, No. 135, 565 (1959).

8. Hacshalylo, M., "Preparation of Compounds for IR Spectrometry," Anal Chem 26, 1410-1412 (1954). (C.A. 49:1431f).

9. Higgins, H. G., "Use of KCl Disks in the IR Examination of Fibrous Cellulose and Other Solid Materials," J Chem 10, 496-501 (1957). (C.A. 52:3517d).

10. Jeffries, R., "An Infrared Study of the Deuteration of Cellulose and Cellulose Derivatives," Polymer 4, 375-389 (1963).

11. Kiel, E. G. and Gusveller, L., "Development of Infrared Spectroscopy in Textile Research. Part I. An Introduction to IR Spectroscopy," Tex. 23, No. 1, 50-65 (1964).

12. Malcom, B. R., "The Mounting of Fibers and Organic Crystals for Spectroscopy in the Near Infrared Region," J Sci Instr 35, No. 11, 423-424 (1958). (BCIRA 39:1876 (1959).

13. Mann, J., "Infrared Spectroscopy and Fibers," Skinner's Silk and Rayon Record 30, 1262-1265 (1956).

14. Nicholls, S. F., "Analysis of Oil Stains on Cotton by Infrared Spectrophotometry," J Textile Inst 52, T530-T532 (1961).

15. Niskino, Y., et al., "A Device for Measuring the IR Absorption Spectra of Fibers," Japan Analyst 4, 174-175 (1955). (C.A. 50:1415g).

16. Polyak, A. B., "A Method of Preparing Fibrous Cellulosic Materials for IR Studies," Izvest. Vyssnikh Ucheb. Zavedeniis Iesnoi Zhur 3, No. 1, 152-154 (1960). (C.A. 54:25787f).

17. Quynn, R. G., "Infrared Microspectroscopis Study of Orientation in Fibers," Textile Research Institute, Princeton, N. J., p 146 (1955).

18. Sadtler, P., "Textile Analysis by IR Technique," Am Dyestuff Reptr 37, No. 17, 555-558 (1948). (C.A. 43:3201c).

19. Shablygin, M. V. and Mikhilov, N. V., "Immersion Method for the Determination of Infrared Absorption Spectra of Fibers in Polarized Light," Khim. Volokna. 6, 51-55 (1963). (C.A. 60:6970d).

20. Wharton, M. K., "Identification of Fiber Blends by IR Spectroscopy," Am Dyestuff Reptr 50, No. 14, 33-36 (1961). (C.A. 55-18116gf).

21. Zhbankov, R. G., "Methods of Obtaining IR Spectra of Fibrous Materials," Zavodsk. Lab. 29, No. 12, 1438-1441 (1963). (C.A. 60:3618h).

22. Zhbankov, R. G., "IR Spectra of the Cellulose Transparent Films Obtained by Pressing Cellulose Fibers under High Pressure," Vistsi Akadinmi Navuk Belaruskai S.S.R. Seryya Fizika-Tekhnichyykk Navuk, No. 1, 15-24 (1956). (C.A. 51:3989g).

2. Spectra of Cotton and Cellulose

23. Fraser, R. D. B., "Use of Fiber Specimens for Infrared Absorption Measurements," J Opt Soc Amer 48, 1017-1018 (1958).

24. Gusev, S. S. and Ermolenko, I. N., "Measurement of IR Absorption of Natural Cellulose Fibers," Zavodsk Lab 30, No. 2, 181-183 (1964). (C.A. 60:14656b).

25. Higgins, H. G., Stewart, C. M. and Harrington, K. J., "Infrared Spectra of Cellulose and Related Polysaccharides," J Polymer Sci 51, 59-84 (1961). (Shirley Institute 41:3456 (1961).

26. Hurtubise, F. G., "Applications of IR Spectroscopy to Cellulose Chemistry," Can Textile J 76, No. 12, 53-60 (1959). (C.A. 53:14501i).

27. Hurtubise, F. G. and Krassig, H., "Classification of Fine Structure Characteristics in Cellulose by Infrared Spectroscopy - Use of Potassium Bromide Pellet Technique," Anal Chem 32, 177-181 (1960).

28. Liang, C. Y. and Marchessault, R. H., "Infrared Spectrum of Cellulose: Structure and Conformation of Native and Mercerized Cellulose," Proc Cellulose Conference 2, 68-79 (1959).

29. Mann, J. and Marrinan, H. J., "Crystalline Modification of Cellulose Part II. A Study with Plane-polorized Infrared Radiation," J Polymer Sci 32, 357-370 (1958).

30. McCall, E. R., Miles, S. H., Thripp V. W. and O'Connor, R. T., "Differential Infrared Spectroscopy of Chemically Modified Cottons," Appl Spectroscopy 18, No. 3, 81-84 (1964).

31. Nelson, M. L. and O'Connor, R. T., "Relation of Certain IR Bands to Cellulose Crystallinity and Crystal Lattice Type, Part II. A New IR Ratio for Estimation of Crystallinity in Cellulose I and II," J Appl Polymer Sci 8, 1325-1341 (1964).

32. Nelson, M. L. and O'Connor, R. T., "Relation of Certain Infrared Bands to Cellulose Crystallinity and Crystal Lattice Type, Part I. Spectra of Lattice Types I, II, and III and of Amorphous Cellulose," J of Appl Polymer Sci 8, 1311-1324 (1964). (C.A. 59:299g).

33. Nikitin, V. N., "Application of the IR Absorption Spectrum to the Investigation of Cellulose and its Derivatives," Vestnick Leningrad Univ. 5, No. 3, 33-44 (1950). (C.A. 48:9059c).

34. Nikitin, V. N., "Study of Cellulose and Its Derivatives by Means of IR Absorption Spectra," Zhur. Fiz. Khim. 23, 775-785 (1949).

35. O'Connor, R. T., DuPre, E. F. and McCall, E. R., "Applications of Infrared Absorption Spectroscopy to Investigations of Cotton and Modified Cottons, Part II. Chemical Modifications," Textile Research J 28, 542-554 (1958).

36. O'Connor, R. T., DuPre, E. F. and Mitcham, D., "Applications of Infrared Absorption Spectroscopy to Investigations of Cotton and Modified Cottons, Part I." Textile Research J 28, 382-392 (1958).

37. O'Connor, R. T., McCall, E. R. and Mitcham, D., "Infrared Spectra of Cellulose Modifications," Am Dyestuff Reptr 49, 214-222 (1960).

38. Ran, J. H., "The Infrared Spectra of Textile Fibers. Parts I, II, III," Melliand Textilberichte 44, No. 10, 1098-1102; No. 11, 1197-1200; No.12, 1317-1320 (1943). (Shirley Institute 44:1156 (1964).

39. Saksena, B. D., Agarwalt, K. C. and Jauhri, G. S., "Infrared Spectrum of Cellulose in Relation to its Structure," J Polymer Sci 62, 347-357 (1962).

40. Smith, J. K., "Structural Study of Cellulose Fibers," J Polymer Sci Pt. C, No. 2, 499-513 (1963). (C.A. 59:4082g).

41. Sobue, H., "Study of Accessibility, Degree of Crystallinity, and Fine Structure of Cellulose Fiber by IR Absorption Method," Kogyo Kagaku Zasshi 60, 320-323 (1957). (C.A. 53:5667c).

42. Stepanov, B. I. and Znbankov, R. O., "Application of IR Spectroscopy in the Investigation of Cellulose," Zavodsk Iab 29, (6), 696-699 (1963). (C.A. 59:2999g).

43. Treiber, E., "Light Absorption of Cellulose and its Derivatives," Kolloid Z. 130, 39-63 (1953). (C.A. 47:6133c).

44. Tsuboi, M., "IR Spectrum and Crystal Structure of Cellulose," J Polymer Sci 25, 159-171 (1957). (C.A. 51:13576e).

3. Spectra of Waxes

45. Kettering, J. H., "Solvent Extraction of Cotton Wax," Textile Research Journal 16, 627-634 (1946). (C.A. 4:1449i).

46. Sadtler, Philip, "IR Technique as Applied to Waxes and Polishes," Proc Chem Specialties Mfgs. Assoc,190-193 (1950). (C.A. 45:6403h).

47. Solopova, A. I., "A New Method for Determining Fatty Waxes and Coloring Matter in Cotton Fibers," Khlopko Vodstvo 13, No. 4, 30-31 (1963). (Shirley Institute 43:2616 (1963).

48. Suchecki, S. M., "Solvent Dewaxing of Cotton," Textile Inds 125, No. 126, 23-25 (1961). (C.A. 55:17018g).

4. Miscellaneous

49. Gore, R. C., "Review of Fundamental Developments in Analysis: Infrared Spectroscopy," Anal Chem 32, 238R-240R (1960).

50. Hirt, R. C., "Ultra violet Spectrophotometry: Review of Fundamental Developments in Analysis," Anal Chem 32, 225R-229R (1960).

51. Liang, C. Y., "IR Study of Polymers," Am Chem Soc Div Org Coatings and Plastics Chem Reprints 22, No. 2, 1-5 (1962). (C.A. 61:1953g).

52. Marrinan, H. J., "A Study by IR Spectroscopy of Hydrogen Bonding in Cellulose," J Appl Chem 4, 204-211 (1954). (C.A. 48:12401e).

53. Morrison, R. D., "New Photomicrographs Included in Current Method of Fiber Identification," Am Dyestuff Reptr 52, (22), 867-886 (1963). (C.A. 60:1877h).

54. Ono, Y., "Growth Process of Cellulose Fibers. I. Growth Process of Cotton Fibers," Seni-i Gakkaishi 11, 520-526 (1955). (C.A. 51:6790i).

55. Thompson, H. W., "Study of Macromolecules by IR Spectroscopy," J Chem Soc, pp 289-297 (1947). (C.A. 41:5020i).

56. Sabue, H., "IR Absorption Spectra of Silk, Wool, and Cellulose by KBr Tablet Method," Kogyo Kazaku Zasshi 60, 88 (1957). (C.A. 53:2629h).

57. Shigorin, D. N., et al., "Investigation of the Physical Structure of Synthetic Polyamides by the Method of IR Spectral Absorption," Doklody Akad. Nauk. S.S.S.R. 97, 711-714 (1954). (C.A. 49:13655d).

REPORT NO. 2 (SEMIANNUAL REPORT)
GEORGIA TECH PROJECT NO. A-843

1 August 1965 to 1 February 1966

FRICTIONAL PROPERTIES OF COTTON FIBERS

By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

PREPARED FOR
UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
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REPORT NO. 2 (SEMIANNUAL REPORT)

FRICTIONAL PROPERTIES OF COTTON FIBERS

By

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ABSTRACT

The purpose of this research is to establish the frictional characteristics of cotton fibers and to determine how these characteristics may change as cotton fibers are processed from the boll to the yarn.

Examination of specimens of Empire WR cotton were continued by means of optical and electron microscopy, infrared spectroscopy, x-ray diffraction techniques, and by frictional and physical measurements of the individual fibers. Sections of fibers of 1-inch cotton were examined by microscopy; the median ratio of the major to the minor axis was found to be 2.8. The average width thickness and wall thickness of the fibers were 24.4, 9.3, and 4.2 microns respectively. Measurements of the ratio, the sum of the major axis plus the minor axis/4 times the wall thickness, gave 2.0 as a median value. This ratio expresses a quantitative relation between the fibrous portion and the lumen area of the fiber. Electron micrographs of fiber surfaces exhibited high variability for the surface features of a single fiber but did not identify characteristics particular to a fiber length group.

Infrared spectrographs of cotton and other fibers were prepared by the KBr pellet transmission method, by the Wilks doublebeam reflectance technique, and from specimens prepared by a fiber press technique developed here. Comparable spectra were obtained by each method. The fiber press method appears to be a significant new method of preparing specimens; the prepared specimens have the additional feature of precise orientation which is of interest from the standpoint of polarized optical and infrared examination and possibly in x-ray diffraction studies. Evaluation of the infrared technique as a tool in the current research program is continuing.

A survey of the literature on x-ray diffraction studies of fibers has been completed, and necessary apparatus for the research program has been assembled. Initial calibration runs of the apparatus have been completed, and preliminary diffraction data of fiber specimens are being obtained currently.

Modifications of the frictional apparatus were made to improve the bearing surfaces of the balance arm and the measurement accuracy of the normal force between fibers being examined. Three balance arms possessing widely different moments of inertia were used for measurements. These were

not found to agree with respect to the coefficients of sliding friction determined by each at low normal forces; and it appeared that each may have a different intrinsic lower limit of normal force below which it can not be successfully employed. This limit is related to the oscillation period of the arm about its pivot. For the arm of greatest moment of inertia employed, 12,000 gm cm^2, the limit is about 20 mg. With this arm, at 20 mg of normal force, values of the coefficient of sliding friction of 1-1/4 inch fibers of Empire WR cotton were about 0.26 compared to 0.295 obtained previously at 26 mg normal force and 0.29 for 15 denier nylon monofilament at 20 mg compared to 0.40 at 11.5 mg normal force. Furthermore no appreciable change in these values over the normal force range 20 to 40 mg were registered with the present high inertia arm. The source of the difference appears to have been principally in binding in the bearings of the original apparatus at low normal force values, which has now been eliminated. However, frictional behavior at low normal forces is the region of greatest pertinence to the problem and a device incorporating an electromagnetic method of normal force application, and thus intrinsically damped, is now under construction.

Specimens of three species of cotton before and after ginning have been procured, and initial measurements of the effects of ginning have been undertaken. Fibrograph measurements of seed and ginned cotton specimens revealed reductions of 3 to 14 percent over a series of span lengths for the various species. The changes in fiber mean length were 9 to 14 percent.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influence of crimp, convolution, cross section, surface texture and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

Subsequently the information obtained will be applied to the development of improvements of the strength, handling, and other desirable and economic features of cotton yarns.

II. INTRODUCTION

The interest in and problems associated with the measurement of the friction of cotton fibers have been discussed in the preceding report. This report presents further development of the frictional measurement apparatus and its employment. Progress in fiber microscopy, infrared spectroscopy, and x-ray diffraction are reported. Studies of the effects of ginning on cotton have been initiated.

III. EXPERIMENTAL WORK

A. GENERAL

Further development of the frictional apparatus has revealed that the measurements of the coefficient of sliding friction obtained for cotton fibers with a particular apparatus may be different from those obtained with another. Hence, a further investigation of the characteristics of the measuring apparatus itself has been required. Concurrently development has been continued on the techniques of microscopy infrared spectroscopy, and x-ray diffraction as applicable to fiber measurements. As an initial phase in research on the effects of processing on the cotton fiber the effects of ginning are being studied.

B. EXAMINATION OF COTTON FIBERS BY OPTICAL AND ELECTRON MICROSCOPY*

1. Purpose

The purpose of this research is to correlate surface features and cross sectional features of cotton fibers with frictional data on these fibers. This report is an analysis of optical cross sections and electron micrographs of surface replicas.

2. Introduction

During the period of this report methods of preparation of cross sections of cotton fibers have been studied and a method has been developed which gave cross sections suitable for the desired dimensional measurements. The measurements have been statistically analyzed in part. Replication techniques have been employed for surface studies of the fibers and for comparison of surface features of fibers of the lengths, 3/4", 1", and 1-1/4".

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* Contributed by: James L. Hubbard, Assistant Research Physicist and Donald L. House, Graduate Student, A. French Textile School.

3. Apparatus and Procedures

a. The Hardy Microtome and its Employment

For the purpose of preparing cross sections of cotton fibers for examination by optical methods a Hardy microtome was obtained.* This microtome consists of two rectangular metal plates which are held together and aligned in the same plane by means of two brass guides on each side of one of the plates. One plate has on one end a centrally located slot parallel to the long side of the plate. Into this slot is fitted a brass tongue which projects from the second plate. A bundle of fibers is placed in the slot and the two pieces fitted together so that the brass tongue presses the bundle tightly into the slot. The bundle is of proper size when the tongue presses it tightly enough so that it will just barely move when pulled through the slot. The ends of the fibers protruding from the slot are cut with scissors as closely as possible to the plate. The remaining protruding ends are then coated with a mixture of 50% Neg-O-Lac and 50% Acetone. After the Neg-O-Lac has hardened the ends of the fibers are cut with a razor blade flush with the surface of the plate. The feeder mechanism is provided for adjusting the thickness of the cross sections. This consists of a screw fed plunger which fits into the slot. The bundle is pushed up a very small amount, barely noticeable under a stereo-microscope, coated with Neg-O-Lac, and an initial thick section is cut using a fresh razor blade. Subsequent sections are then made for examination by turning the plunger screw through 1/3 to 1/2 of one of the marked divisions on the head of the screw, coating the protruding ends with Neg-O-Lac and cutting with a new edge of a razor blade. These sections are in the range 7 to 10 microns in thickness.

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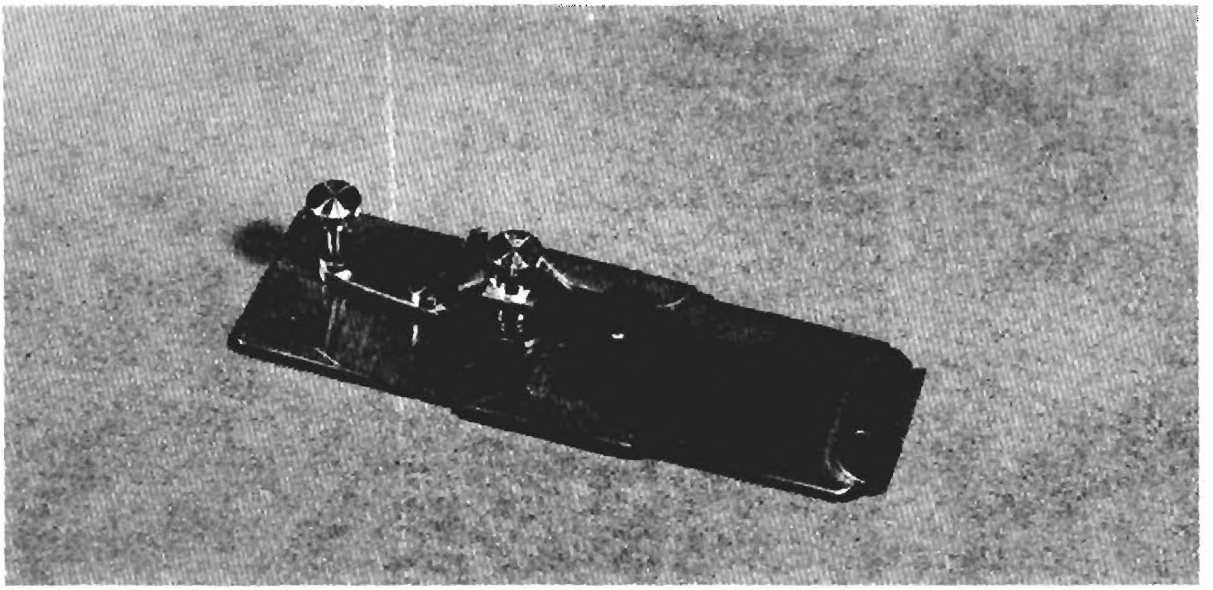
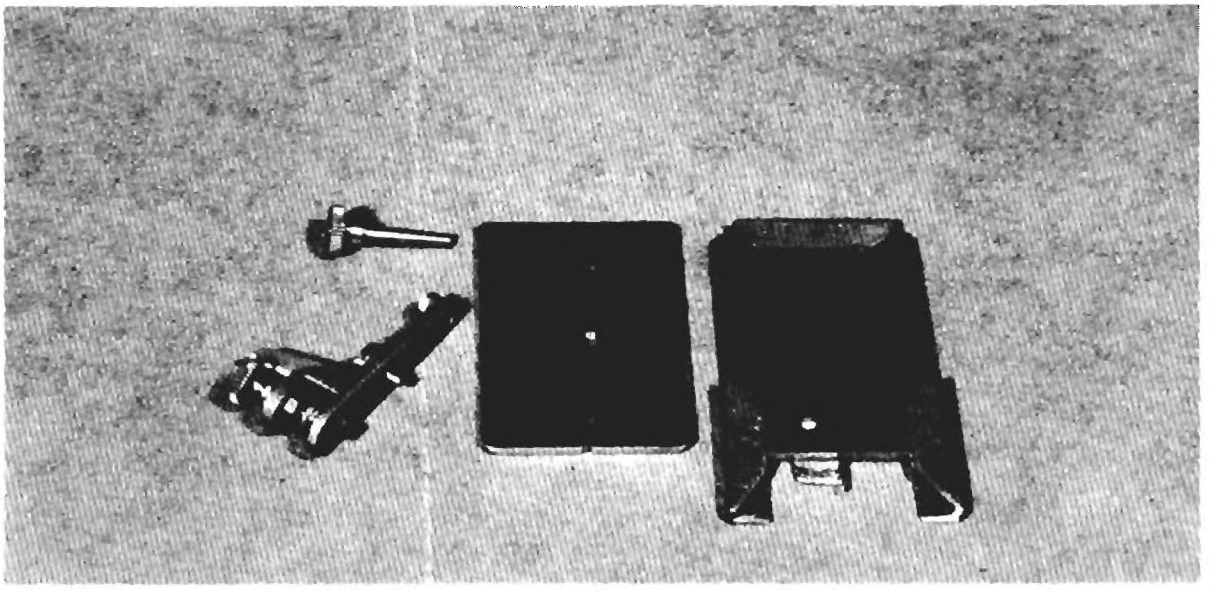
* See Figure 1a, page 5.



A. ASSEMBLED AND IN THE INVERTED POSITION



B. DISASSEMBLED SHOWING VARIOUS PARTS

Figure 1a. Views of the Hardy Microtome.

Considerable skill and practice is required for the cutting of a section. The blade used for cutting must be held firmly against the plate during the cut, at an angle of about 20° to it and at an angle of about 40° to the axis of the slot, so that it will not ride up over the bundle, and the sliced section will be smooth and uniform. As the cut is made the blade is slid along its axis to give a slicing action. The steel used in the particular Hardy microtome was much softer than the razor blades used and it was difficult to cut sections without scarring the surface of the microtome plate. This made it very difficult to cut subsequent usable sections without first repolishing the plate. Therefore, the plate containing the slot, over which the razor blade passed as a section was cut, was duplicated using a piece of 440 C stainless steel. This piece was then hardened by heat treatment to Rockwell C 62. This is harder than the razor blades used and sections can be cut without the problems arising from a scarred surface.

b. Technique for Preparing Section of a Single Fiber

A new technique has been developed for isolating small numbers of fibers for the purpose of examining their cross-sections. It is possible using this technique to view one fiber both longitudinally and cross-sectionally with an optical microscope.

Using the small brush, paint 3 or 4 layers of the Neg-O-Lac on a glass microscope slide making a strip 1-1/4 - 1-1/2" long and 1/4" wide. Allow 15 minutes between each layer for drying. Before the last layer dries completely, lay the fiber to be examined on the Neg-O-Lac with its longitudinal axis parallel to the longitudinal axis of the Neg-O-Lac.

After the fiber has been placed on the Neg-O-Lac 3 or 4 more layers of Neg-O-Lac are painted on the fiber. The solidified Neg-O-Lac and the embedded

fiber are then examined under an optical microscope. At powers of 150x or higher the convolutions of the fiber are distinctly visible and are easily counted. Also it is possible to select a particular area of the fiber to be cross-sectioned.

The embedded fiber is then removed from the slide, the particular section to be examined is cut away from the rest of it, and the desired portion is placed in the slot of the Hardy microtome. (Note: The reason that the number of layers of Neg-O-Lac is not specified above is that the thickness of the package should be such that it will fit perfectly in the slot. This thickness, of course, varys with the amount of Neg-O-Lac used for each coating.)

c. Preparation of Surface Replicas for Electron Microscopy

Replicas of the surface of cotton fibers were made using a two stage replica technique designed to give a positive replica of the surface. A solution of approximately 20% polystyrene in xylene was placed on a glass microscope slide and alowed to dry, forming a thin film of polystyrene. A few cotton fibers of measured length were placed on the polystyrene, another glass slide placed on top of these, and the entire sandwich was placed on a hot plate at a medium temperature (150° C). As the polystyrene softens, the fibers are pressed into it by pressing down on the top glass slide. The sandwich is quickly removed and pressure kept on the top slide until the polystyrene has hardened. The slides are separated and the fibers lifted from the polystyrene with the aid of sharp tweezers and a low power stereomicroscope. The plastic impressions are then placed in a vacuum system where they are coated with a thin layer of carbon evaporated from a position normal to the surface of the film. The film is removed from the vacuum and the areas containing the fiber impressions are cut into 1/8" squares. Copper microscope grids (100 mesh) are

placed in a petri dish on a few layers of filter paper and the plastic replicas placed carbon-side-down on the grids. The filter paper is saturated with xylene, the dish covered and left for 24 hours to dissolve the polystyrene. The grids on which now lie thin carbon film replicas of the cotton fibers are removed, dried, and placed back in the vacuum system for shadowing. During the shadowing process a thin film of a platinum is evaporated from a position on a line making about a 30° angle with the surface of the grids. The finished replica is removed from the vacuum and placed in the electron microscope for observation and photography.

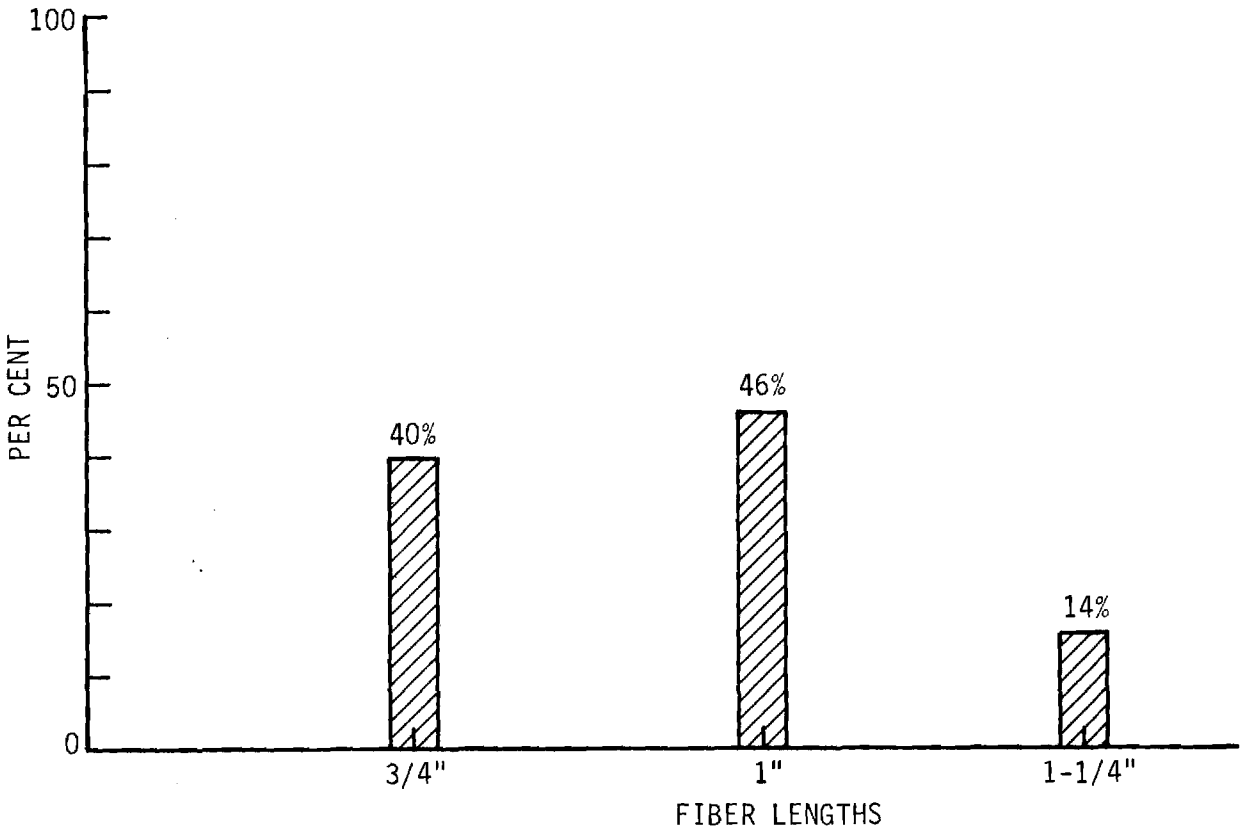
4. Experimental Work

a. Fiber Selection

The cotton examined was obtained from a specially selected bale of Empire WR cotton previously characterized.* Fibers from this bale were separated into groups of 3/4", 1", and 1-1/4" respectively with values in each group of the dimension indicated ±1/8". The distribution found for the various fibers examined are shown in Figure 1B. These numbers are only approximate because of the wide distribution of lengths included in each group but represent the distribution of groups of 200 to 500 fibers examined.

It will be noted that approximately 40% of the fibers fall in the 3/4" group, 46% in the 1" group, and 14% in the 1-1/4" group. Evidence of non-random selection is indicated in the relatively large percent of long fibers obtained. Normally this would only be 2 or 3%. A further analysis has been obtained by weighing the fibers as noted. The fiber weights per average unit length are given as 3.74, 3.10 and 2.88 micrograms per inch for the 3/4", 1",

* T. R. Boys, "An Evaluation of Factors Affecting the Frictional Properties of a Selected Cotton Fiber Sample," Thesis for M.S. degree, Georgia Institute of Technology 1964.



		WEIGHT 100 FIBERS	WEIGHT 1 FIBER	WEIGHT/in.	WEIGHT/cm
FIBER WEIGHTS:	3/4"	0.28 mg	0.0028 mg	0.00374 mg	0.00147 mg
	1"	0.31 mg	0.0031 mg	0.0031 mg	0.00121 mg
	1-1/4"	0.36 mg	0.0036 mg	0.00288 mg	0.00113 mg

Figure 1b. Length and weight distribution of cotton fibers examined by optical and electron microscopy.

and 1-1/4" lengths respectively. These are also recorded as 1.47, 1.21 and 1.13 micrograms per centimeter respectively.

b. Optical Microscopy

Due to the difficulty encountered in the use of the original Hardy microtome until a harder cutting table was fabricated, usable sections were obtained for the 1" fiber length only during this period. Figure 2 displays typical photomicrographs of cross sections of fibers in the 1" range. Dimensions of the cross sections were taken from these micrographs. Three measurements were made on each fiber; the major axis, the minor axis, and the wall thickness. These dimensions shall be referred to as M, m, and W respectively. In order for these measurements to be valid statistically it was necessary to measure over 200 fibers; 226 fibers were actually measured.

From these data were calculated two sets of factors to be used to compare these data with future data. The first set of data was calculated using the equation $R = M + m / 4W$. This factor is related to the maturity of the fiber. Since $\frac{M + m}{4W}$ could be related to the average radius of the fiber and W is the wall thickness, a fiber with wall thickness equal to its radius, that is having no lumen, will have an R value of 1. The R value is a measure of the wall thickness in relation to the overall size of the fiber. Figure 3 is a plot of these data versus the distribution of fibers.* 1

The second set of data was calculated using $S = M/m$. The S is relative to the shape of the cross section, S equalling 1 for a round cross section and larger for elliptical shapes. A graph of this factor versus frequency of

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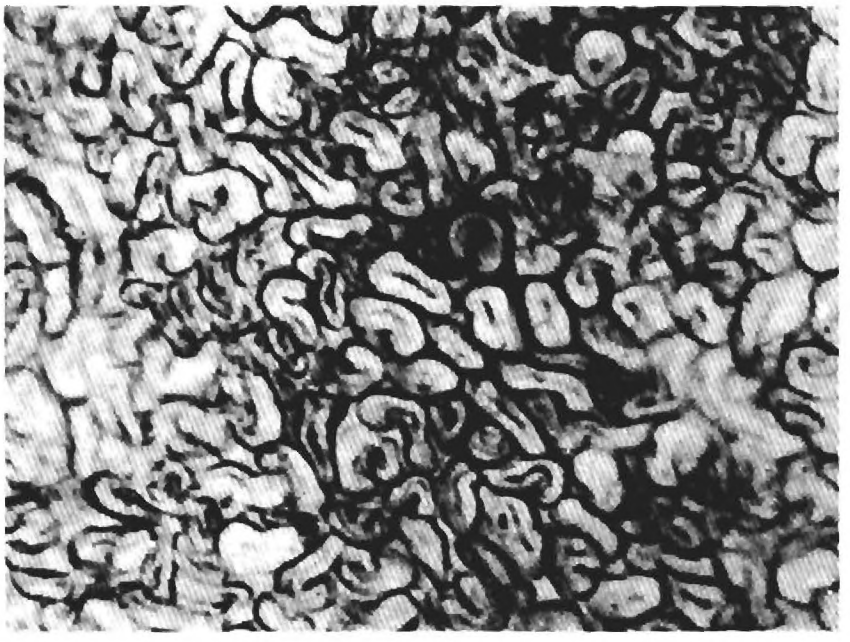
*Note: These plots are made on logarithmic-probability paper. This possesses the special features of converting skewed gaussian distribution to a straight line plot and simplifying calculation of the mean diameter and standard deviation.

A. 540x



B. 540x

Figure 2. Photomicrograph of representative cross-sections of 1-inch Empire WR cotton fibers.

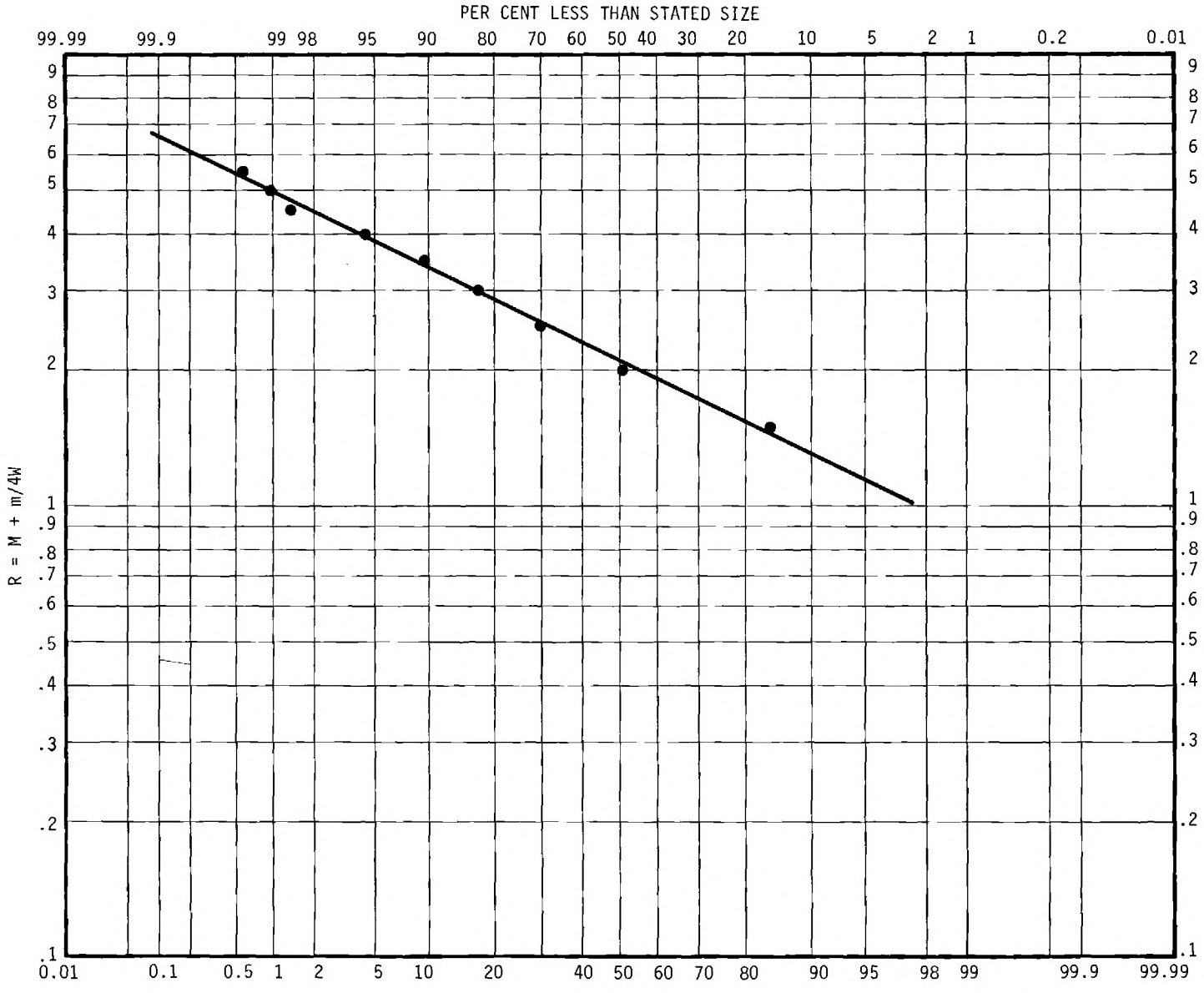


Figure 3. Distribution of 1-inch Empire WR cotton fibers in accordance with radius factor, $R = \frac{M+m}{4W}$.

occurrence among its fibers is shown in Figure 4.

Examination of Figure 3 shows that for the fibers examined the extremes of the factor $R = \frac{M + m}{4W}$ are R = 1.5 and 5.5 respectively whereas the median value is 2.0. As previously indicated a round fiber with no lumen would have an R value of 1. It is evident that all fibers have a significant lumen area, in some cases very large.

Examination of Figure 4 reveals that the shape factor S = M/m varies over the range 1.5 to 8.5 with the median value at 2.8. This means that the median fiber width is essentially 2.8 times its thickness. Referring back to Semi-annual Report No. 1 of this report we find that values slightly less than 0.001" were given for fiber widths and thicknesses are in the range 0.0002" to 0.0004". This gives the shape factor span as 2.5 to 5 for the few fibers examined and these fall into the distribution range of 10 to 60% of fibers. For the series of measurements for this report the average width and thickness measurements were 24.4 μ and 9.3 μ respectively; the average wall thickness was 4.2 μ.

Figure 5 reveals the number of convolutions per inch (for a 360° turn) as varying over the range 66 to 100 for fibers of the type examined whereas the median value is at 84 convolutions per inch. This plot was made from data taken from T. R. Boys and reported as Figure 2 of Report No. 1 of this research.

c. Electron Microscopy

Figures 6, 7, and 8 are platinum shadowed-carbon replicas of typical surface areas of 3/4", 1", and 1-1/4" fibers respectively. The fibrils and general irregularity of the surfaces can be readily observed. Both relatively smooth and rough areas can be found along the same fiber;

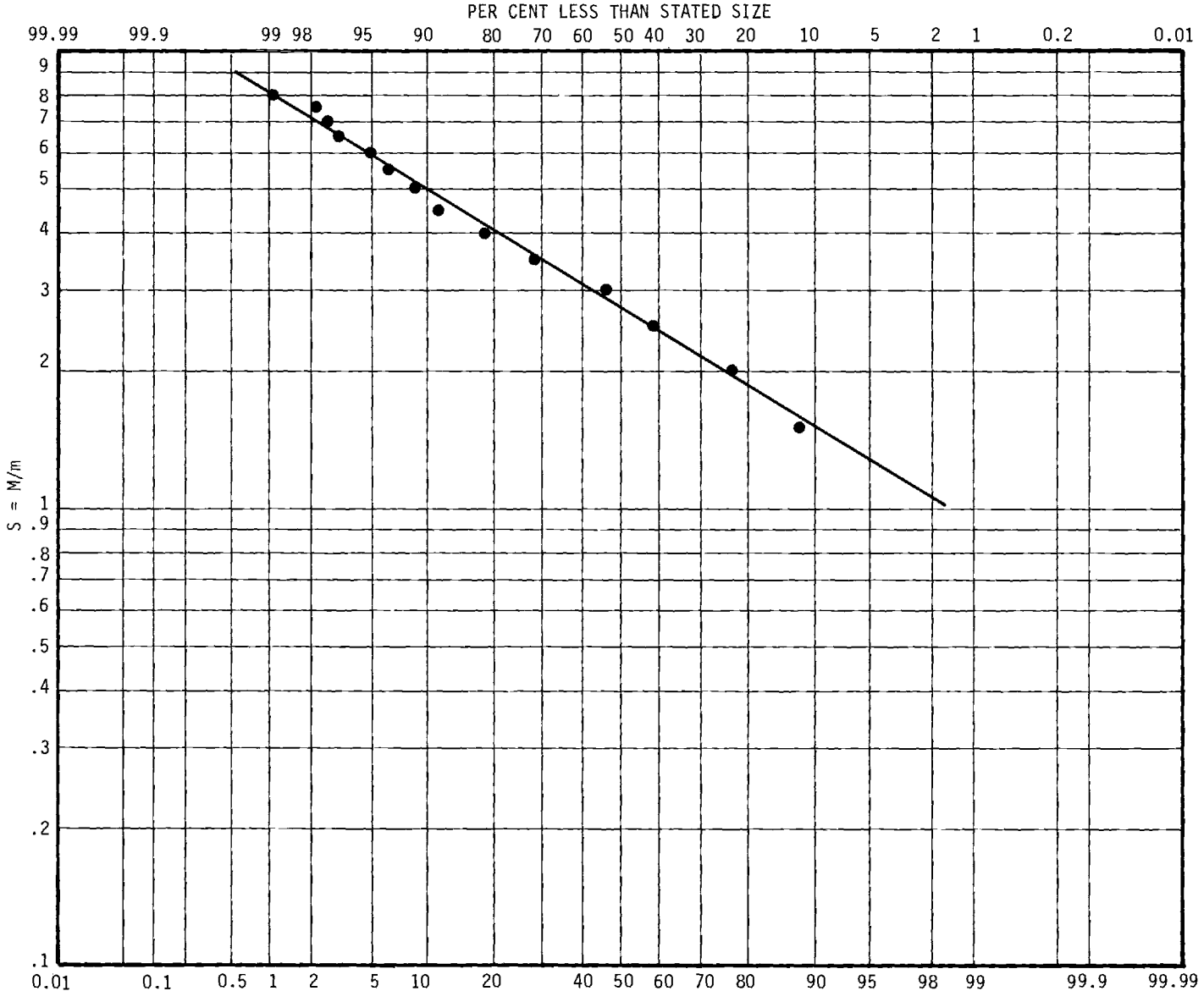


Figure 4. Distribution of 1-inch Empire WR cotton fibers in accordance with shape factor, $S = \frac{M}{m}$.

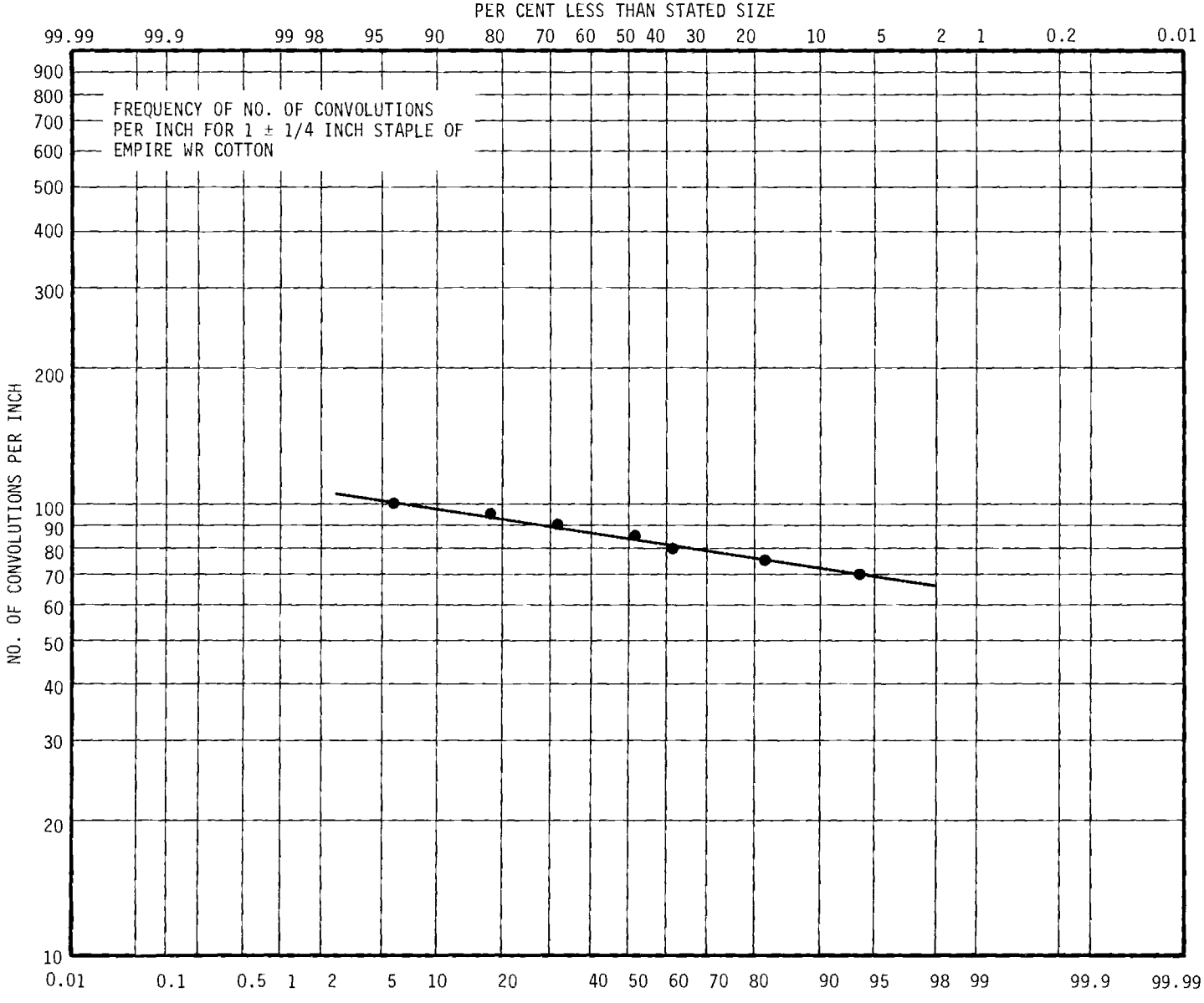
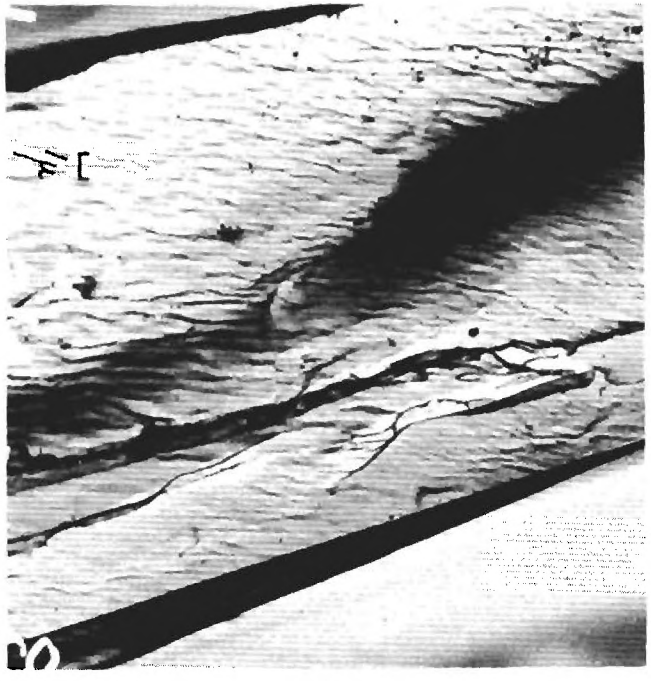
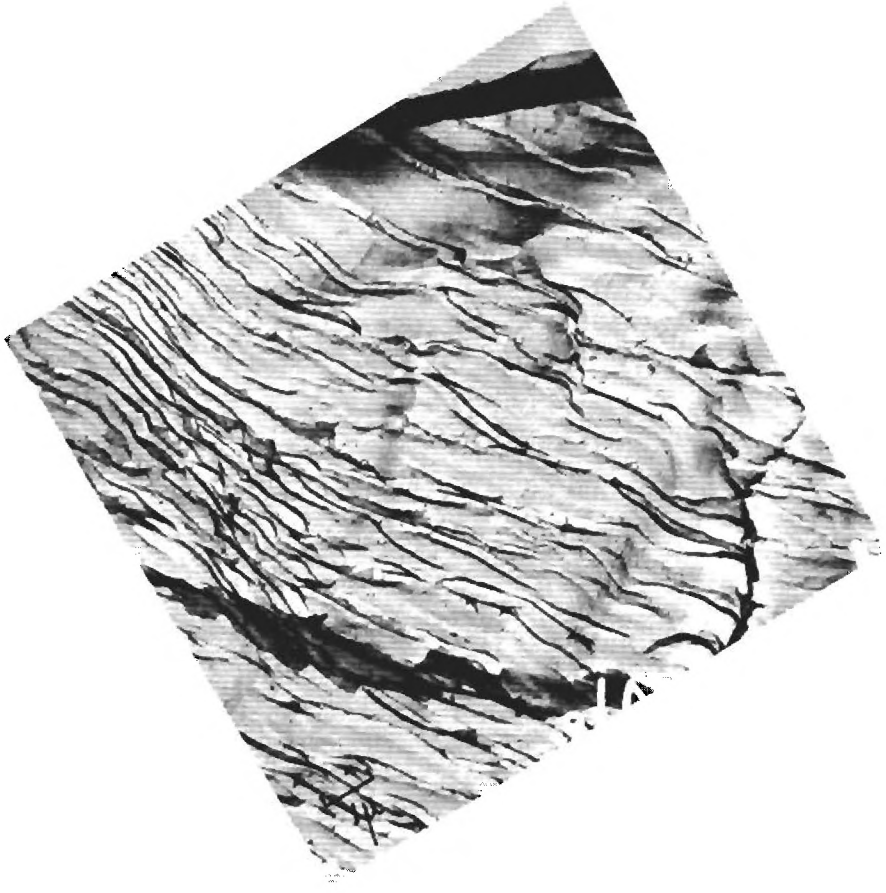


Figure 5. Distribution of (1"±1/4") Empire WR cotton fibers in accordance with number of convolutions per inch.

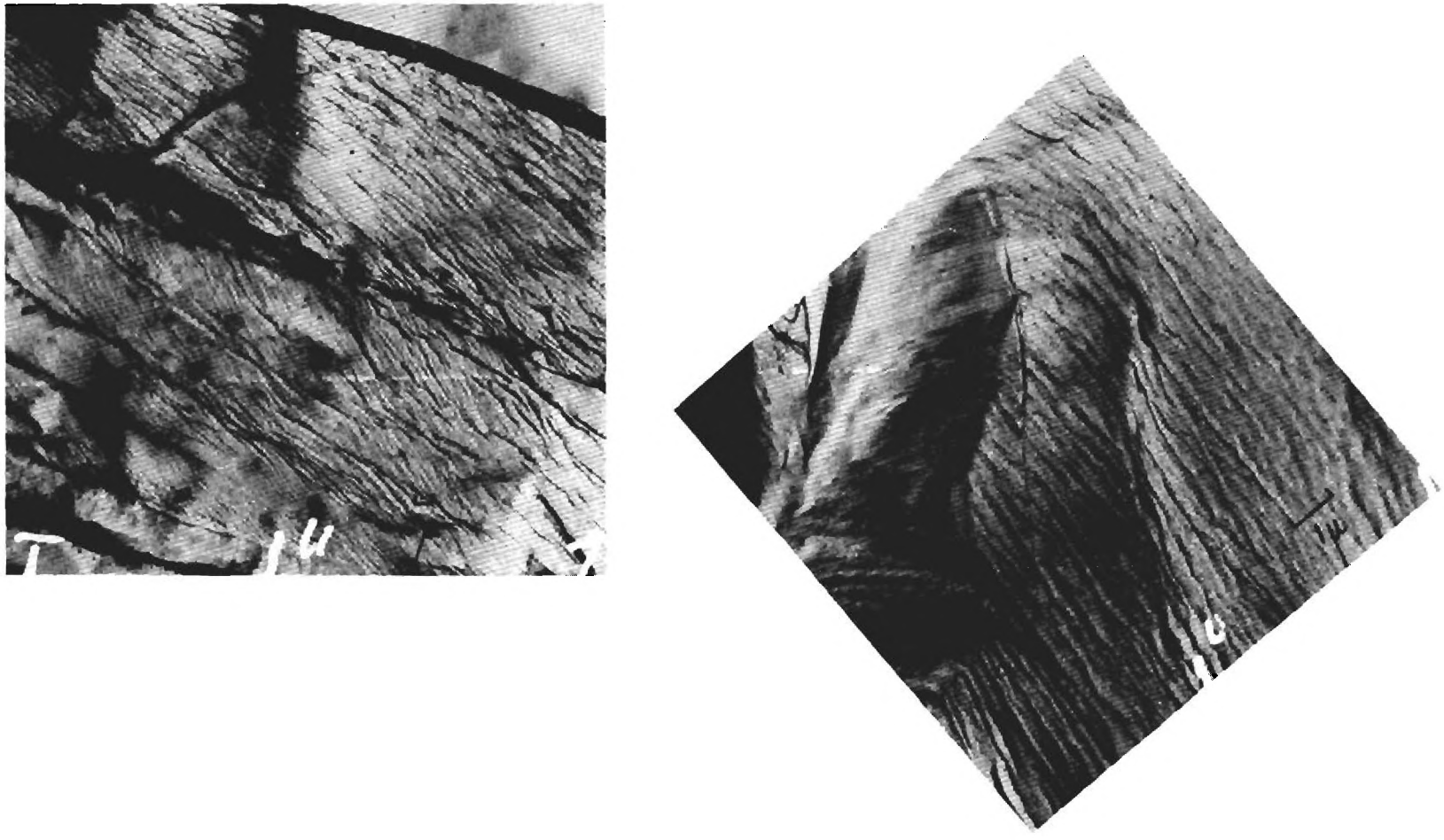


A. 3600x



B. 7500x

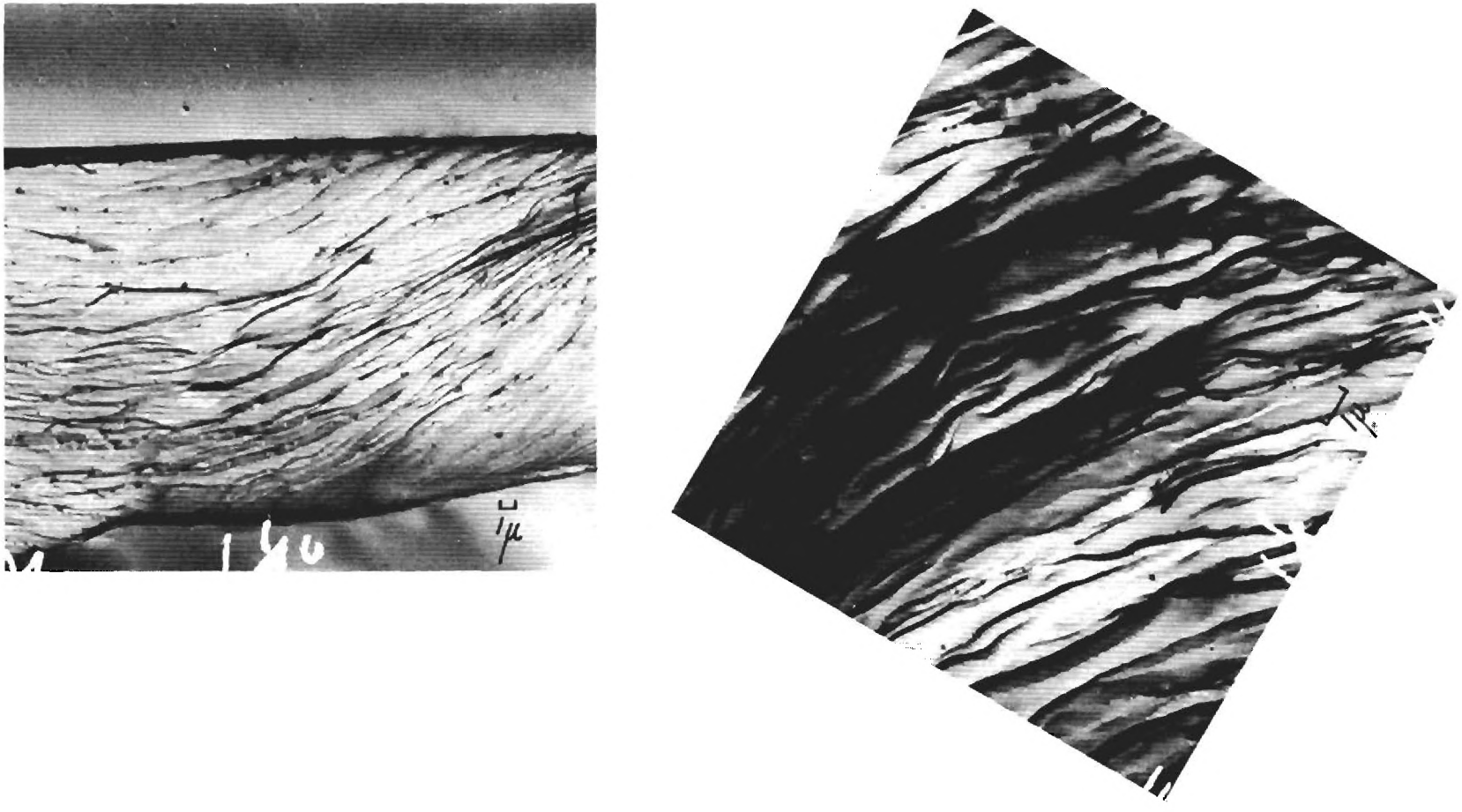
Figure 6. Electron micrographs of replica of a typical 3/4-inch Empire WR cotton fiber.



A. 2640x

B. 7500x

Figure 7. Electron micrographs of replica of a typical 1-inch Empire WR cotton fiber.



A. 2640x

B. 6000x

Figure 8. Electron micrographs of replica of a typical 1-1/4-inch Empire WR cotton fiber.

however, no characteristic particular to a given fiber length was observed. An optical micrograph of the surface of a 1" fiber showing a few ridges running across the fiber perpendicular to the direction of the fibrils is shown in Figure 9. Figures 10 A and 10 B are electron micrographs of a similar banded zone. In Figure 10 A the fibrils are uninterrupted by the ridge whereas in Figure 10 B the furrows, denoting fibril direction, are stopped by the ridge and new furrow lines begin on the ridge. Hence, the surface fibrils appear to be interrupted and restarted in this zone.

Figure 11 is a micrograph exhibiting the reversal of the direction of the fibrils along the fiber. The small thin peaks apparently protruding from the fiber in this and some of the other electron micrographs are believed to be artifacts due to the plastic replication technique.

The electron micrographs thus far have pointed to the characteristic roughness of the cotton fiber, the fibrillar structure, and the occurrences of interruptions and reversals. However, a sufficient number of observations has not yet been made in order to distinguish surface characteristics that may be particular to a given length of fiber.

C. FRICTIONAL APPARATUS AND MEASUREMENTS

1. General

Frictional measurements made during the first period indicated a need for better bearings for the frictional lever arm and a need for rapidly and consistently monitoring the applied normal force immediately before and after each fiber friction measurement. The old lever arm was remounted and two new ones were constructed; a balance was arranged to give rapid normal force readings. The calibration of this new equipment revealed that each balance

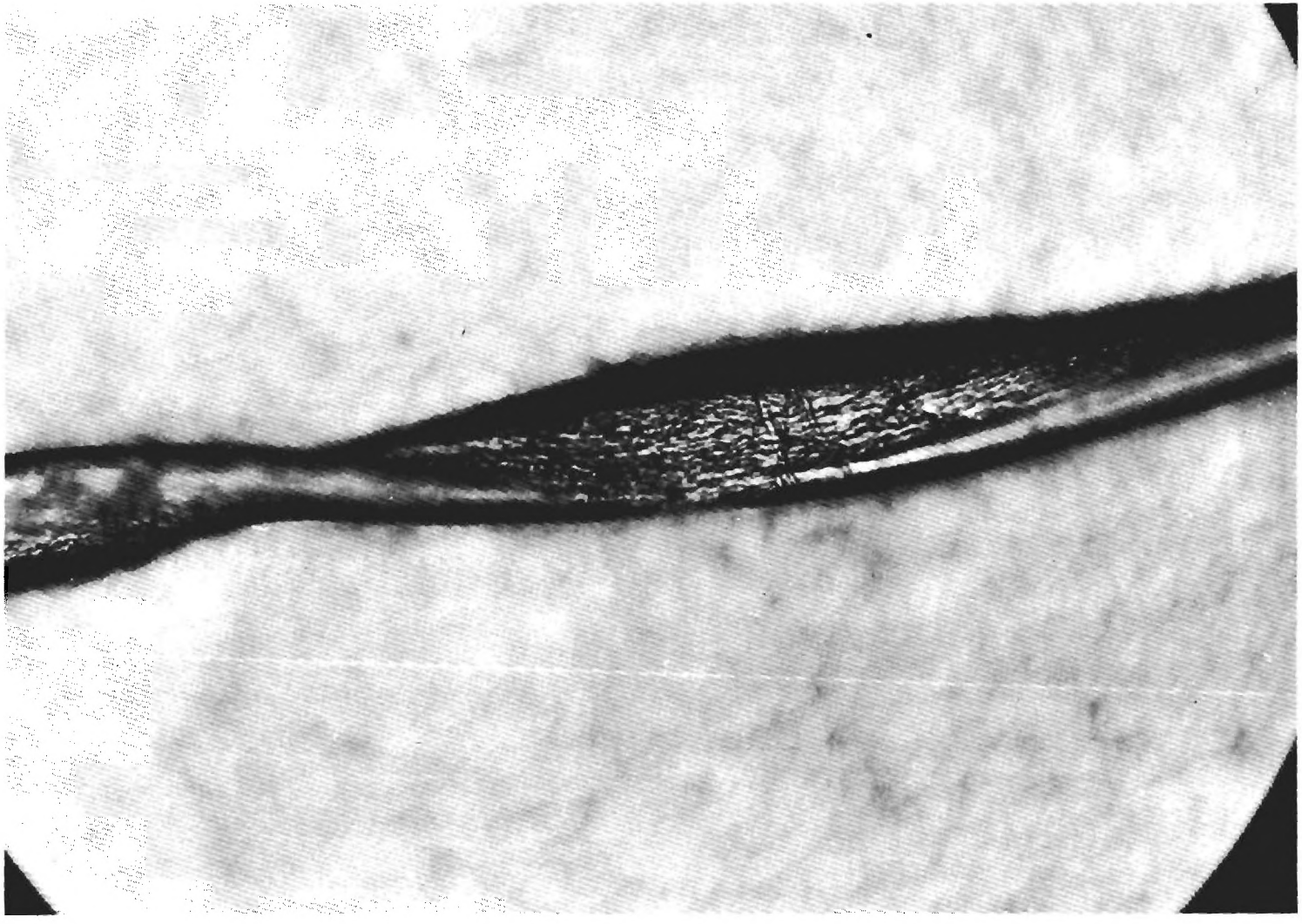
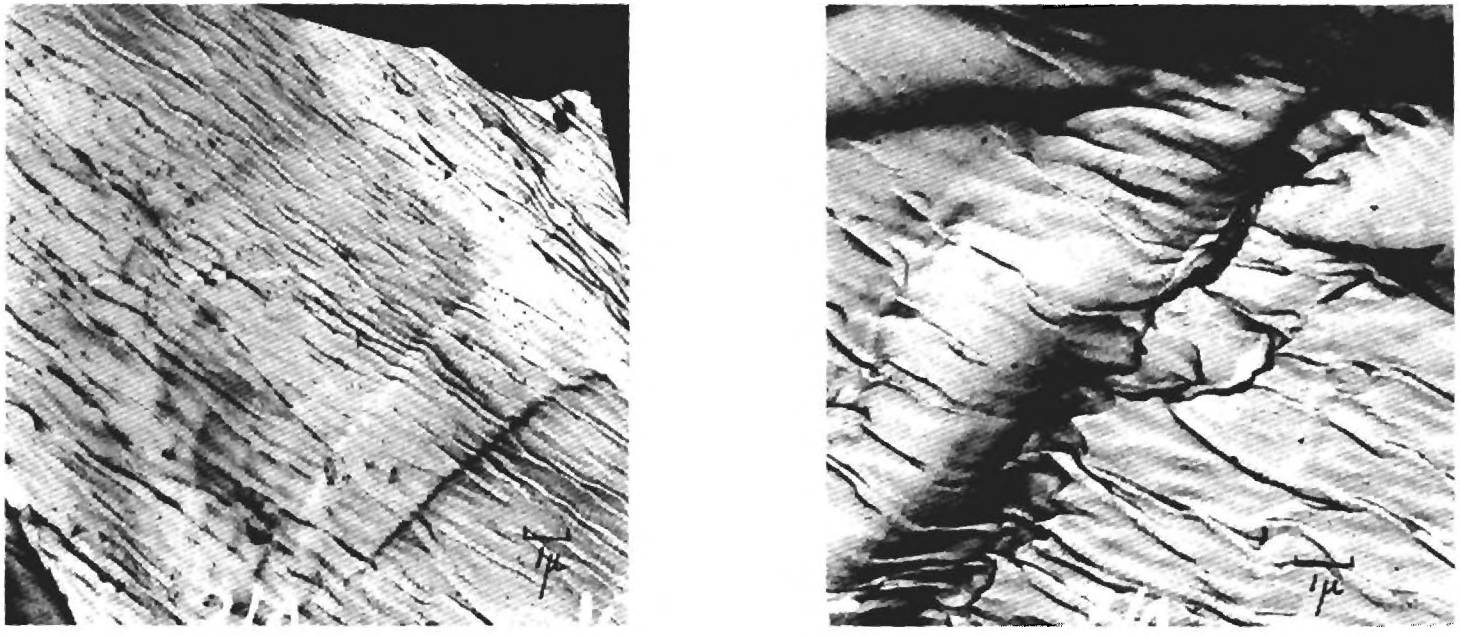


Figure 9. Photomicrograph of typical 1-inch Empire WR cotton fiber exhibiting banded zones across width.



A. 6000x

B. 7500x

Figure 10. Electron micrographs of replicas of 3/4-inch Empire WR cotton fibers exhibiting banded zones running across surface. Note discontinuity of fibrils in B.

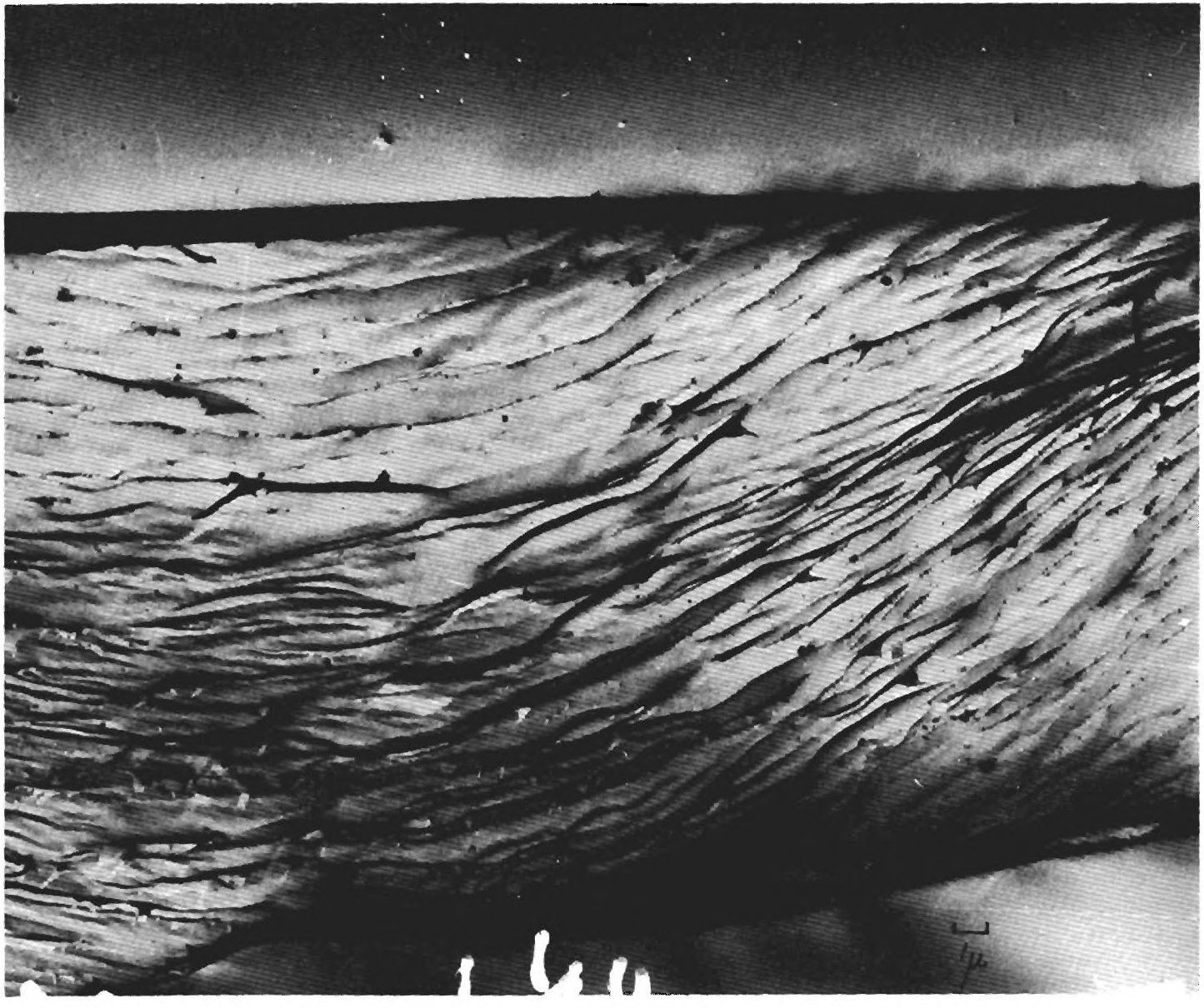


Figure 11. Electron micrograph of replica of 1-1/4-inch Empire WR cotton fiber exhibiting reversal of fibrillar winding direction.

arm gave data characteristic to itself only, and that data obtained with one were not necessarily comparable to those taken by another. An extensive series of fiber friction measurements were made to calibrate these instruments and to analyze their friction measurement capabilities. These measurements are outlined below.

2. Apparatus Modifications

The original apparatus (Figure 9, Report No. 1) was disassembled to replace the fulcrum pin and bearings. This arrangement consists of a hardened steel pin (#33 pocket watch staff) mounted in sapphire bearings. The original pin was found to be scarred and the bearings were replaced and reversed in order to obtain the maximum bearing surface. The bearings were demountable in order that this arm or another with the same suspension configuration could be used to replace it.

Two additional arms were constructed. One was constructed of 1/8" aluminum rod, 29 cm long, suspended at a position 23.9 cm from the fiber holder. The short end had a brass counterbalance affixed to it and the long end was indexed to accept a wire rider for adjusting the normal force. The second similar lever of 3/16" rod was 31.8 cm long suspended at a point 22.9 cm from one end. The mass of the three arms were 74.2 grams, 39.6 grams and 23.4 grams respectively and the moments of inertia about the pivot were calculated to be approximately 12,000, 3,000, and 1,150 gm cm^2 respectively.

A new fiber holder was also constructed of a brass block with a groove in each end for aligning the fiber. The span length of this holder was only 0.5".

A stand was constructed for a chainomatic balance, as shown in Figure 12. This places the balance directly over the fiber-holding end of the respective frictional arms. A chain suspended from a counterweight, replacing the left pan

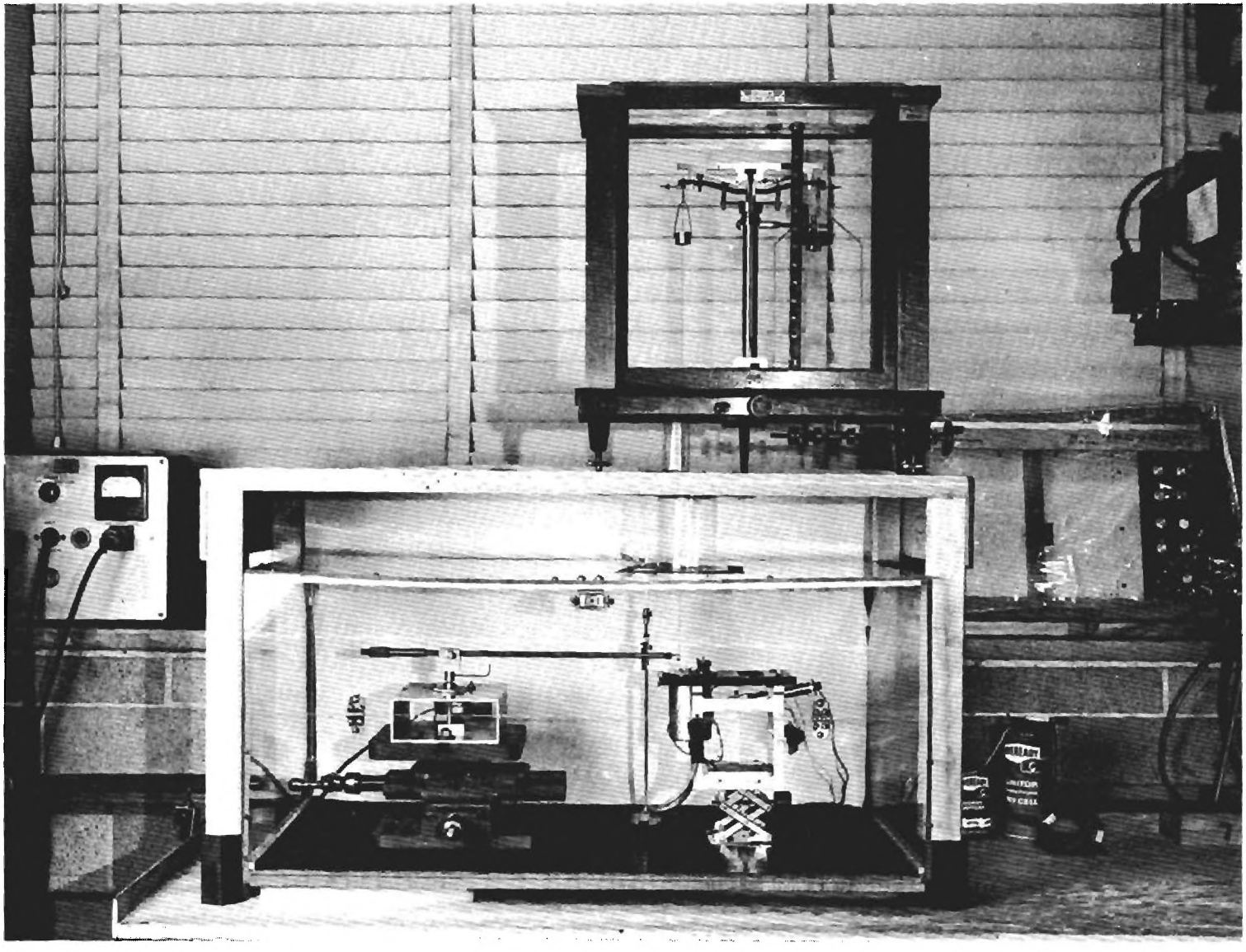


Figure 12. View of frictional measuring apparatus and chainomatic balance for measurement of normal force.

of the balance, extends down precisely to the level of the balance point of the respective lever arm where it can be engaged with a hook provided on the arm. A close-up view of this arrangement is shown in Figure 13. Repeatability of the normal force setting is ±0.0002 grams.

3. Measurements

When the instrument was reassembled after these modifications using the 1/8" rod as the frictional arm, the measurements of the coefficient of friction of cotton fibers were found to be much lower than the values previously obtained with the monel tube arm, i.e. approximately 0.2 compared with 0.4. A long series of measurements were then undertaken to see if any variables had been introduced that would account for this change.

One of the significant effects became apparent at low normal forces between the fibers. When values of the normal forced were reduced below about 15 mg, the coefficient of friction dropped whereas previously we had experienced a larger value, 0.42, at 11.8 mg than we had at 26 mg, 0.29. It thus became necessary to define the characteristic behavior of each arm.

On further experiment it was demonstrated that the moment of inertia, I, of each arm may be quite important. If we consider the expression for the period of a compound pendulum:

$$T = 2\pi \sqrt{\frac{I}{K}} = 2\pi \sqrt{\frac{I}{mg\ell}} \quad \text{(for small amplitudes)}$$

where

T is the period,

I is the moment inertia,

and K is a constant,

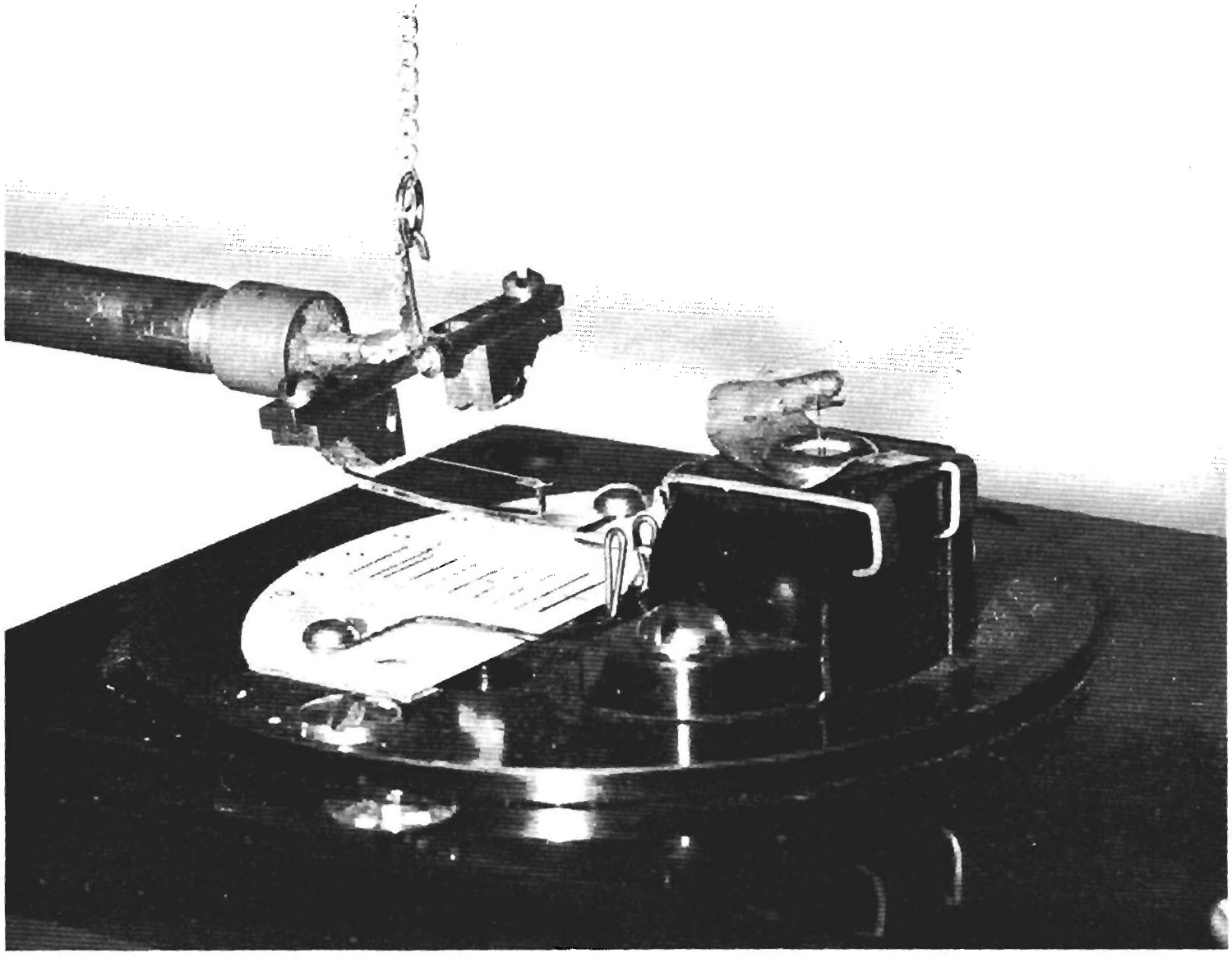


Figure 13. Close up of frictional arm exhibiting attachment of chain to balance.

we see that T is directly proportional to the $\sqrt{I}$.

For small amplitudes $K = mg\ell$

where m is the mass of the compound pendulum (the frictional arm)

g is the acceleration of gravity

and ℓ is distance from the center of gravity to the point of suspension.

Now, when the arm is perfectly balanced its center of gravity and the point of suspension are the same; hence, the distance ℓ is zero and T becomes infinite. For very small forces of a few mg, ℓ is very small and T is very large. Hence, we can expect long periods of oscillation of the arm at low values of the normal force. On the other hand, if I is large the force to displace the beam from the contact position is large and we might expect good tracking of fiber on fiber. The importance of this tracking is that if the beam be bounced by an asperity, the time for return to the correct normal force may be considerable, and assumption that the normal force is constant may introduce a major error into the calculations for the coefficient of friction. Secondly, a smooth surface is less likely to bounce the arm than a rough one. The significance of the factors discussed will be brought out in subsequently reported data.

During this period frictional data have been obtained for several hundred fibers; however, due to the reasons discussed these have been principally conducted for calibrating the instrument and its characteristics. Examples of these are reported.

Figure 14 depicts typical curves for 1-1/4" cotton fiber made at a normal load of approximately 20 mg for each of the three lever arms. It will be observed that the values of μ for each instrument are 0.25, 0.21, and 0.18, respectively, for the monel rod, the 3/16" aluminum rod, and the

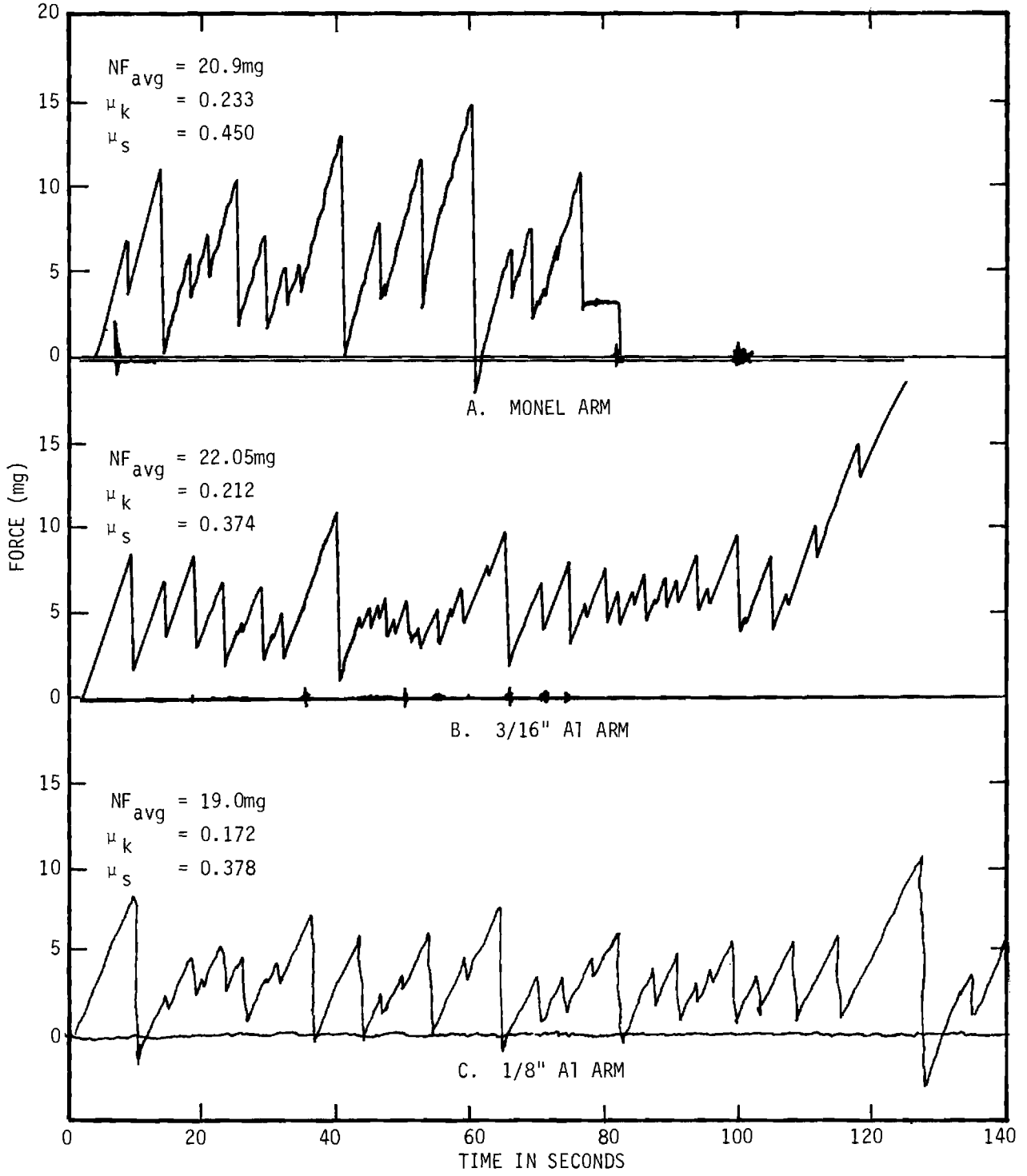


Figure 14. Comparison of frictional data plot of 1-1/4-inch Empire WR cotton fibers made with three different frictional arms at about 20 mg normal force.

1/8" aluminum rod. Similarily in Figure 15 are characteristic curves made for 15 denier nylon filament using the same frictional arms. Here we observe again a disparity in the coefficient of friction values of 0.35, 0.21, and 0.25 respectively.

Since the shape and mass of the monel arm gave it the higher moment of inertia, and this appeared to be an important factor in maintaining fiber to fiber contact, this arm was used in further measurements. Of particular interest was the behavior of the arm over a reasonably large load range such as 10 mg to 50 mg normal force. In Figure 16 are plotted the coefficient of sliding friction data obtained for this arm versus normal force for a single fiber pair of cotton and of nylon over the range 6 to 40 mg.

It will be noted that the peak value of the coefficient of sliding friction was found at about 18 mg. Furthermore, each type of fiber gave a different behavior as the normal force approached zero. For cotton the coefficient of friction dropped to a lower value along a curve that was extrapolated to intercept the zero force axis at about 0.16 whereas for nylon the coefficient of friction dropped to a value of 0.10 at 5.7 mg and appeared to be going to drop to near zero at 2 or 3 mgs. This behavior suggested that the rough surface of the cotton still caused snagging at low loads whereas on the smooth nylon adsorbed gas may have introduced a lubrication effect which resulted in the rod going into a periodic oscillation rather than measuring a correct value of sliding force.

The data for the nylon fiber is shown in Figure 17 to indicate the type of curve obtained at low values of normal force. A similar curve for cotton is shown in the Figure. The differences in the general character of the curves is readily observed.

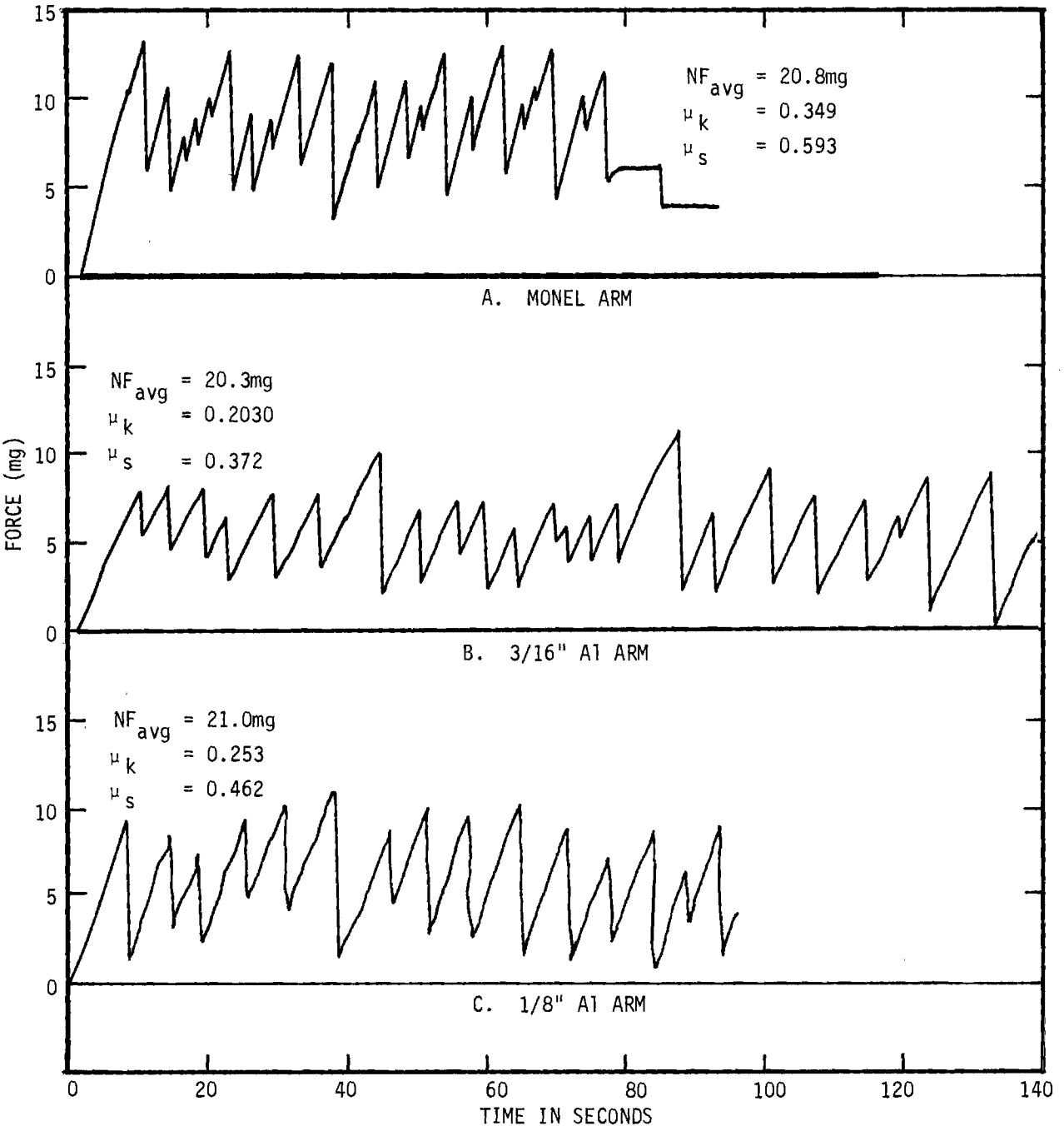


Figure 15. Comparison of frictional data plots of 15 denier nylon fibers made with three different frictional arms at about 20 mg normal force.

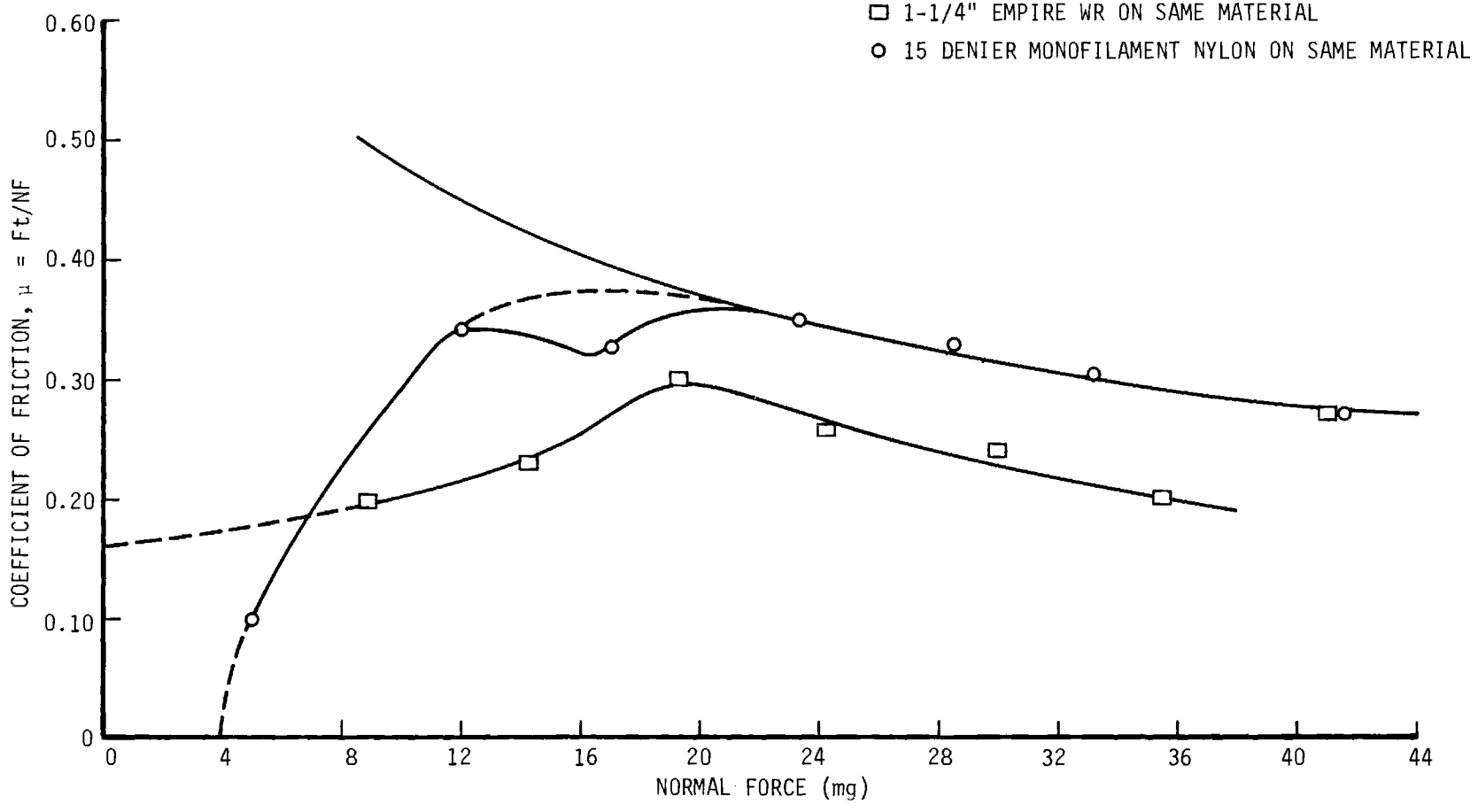


Figure 16. Plot of coefficients of friction versus normal force obtained for single fibers of 1-1/4-inch Empire WR cotton fiber and 15 denier nylon.

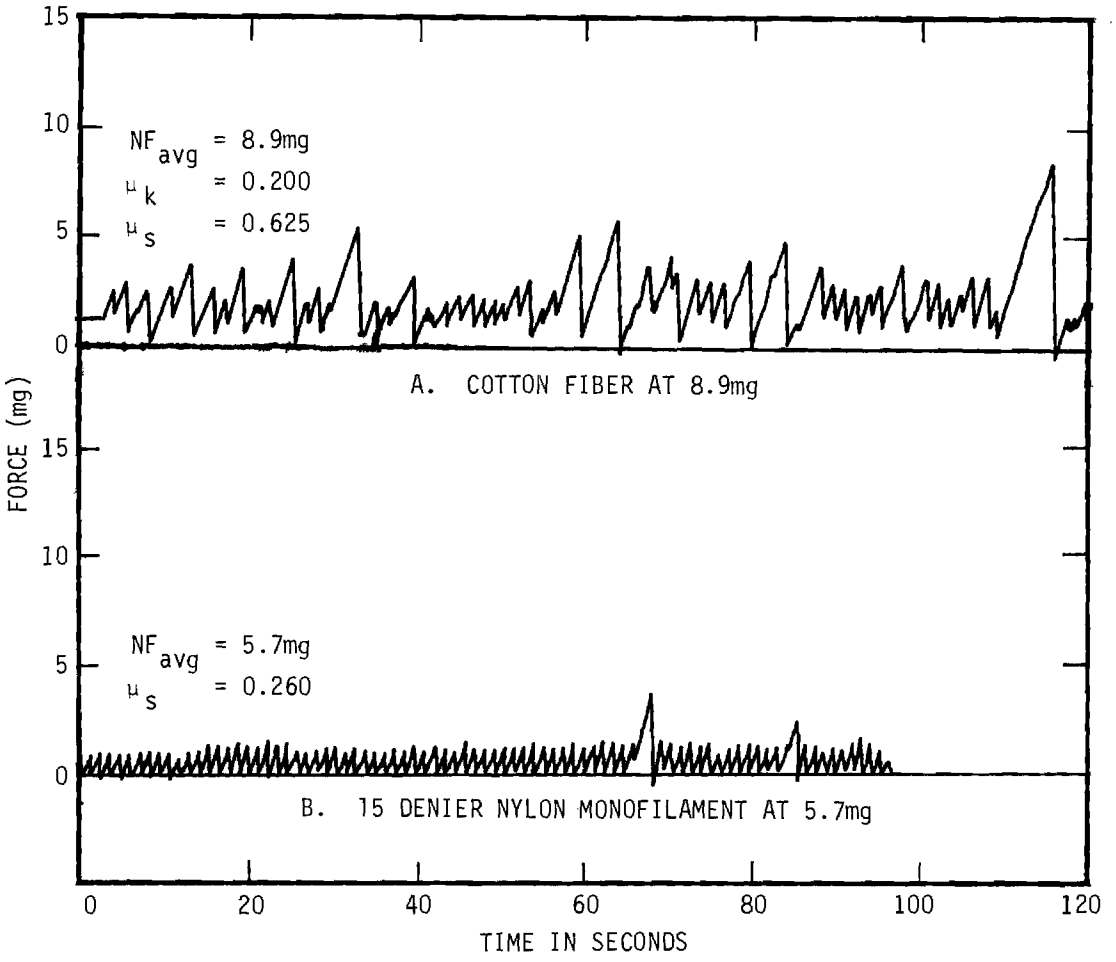


Figure 17. Comparison of frictional data obtained for 1-1/4-inch Empire WR cotton fiber and 15 denier nylon fiber at low normal forces, 8.9 mg and 5.7 mg respectively.

Using the preferred normal force of about 20 mg a series of new cotton fibers were examined. The coefficient of friction data are listed in Table 1 for three runs on each of three fibers. It will be seen that the average value for the coefficient of sliding friction is 0.245 and for static friction is 0.449. The range of variation was from 0.18 to 0.29 and 0.39 to 0.52 respectively. The ratio of μ static / μ kinetic is 1.83. Similar results for nylon are shown in Table 2. Coefficients of sliding friction over the range 0.26 to 0.34 are exhibited, the average value being 0.29. The μ_s value for nylon was found to be 0.458 with a ratio of $\mu_s/\mu_k = 1.58$.

An additional series of experiments were run to determine the value μ_k using the large inertia arm (K-monel tube) for more fibers over the load range 10 to 50 mg. A plot of these values is shown in Figure 18. It is evident here that there really does not exist a large variation with load and that the data of Figure 16, taken from experiments with a single fiber, are misleading. On the other hand, experiments made at very low normal forces, $<$ 10 mg, display a large scatter and a better method of maintaining normal force at a known value are required for investigation much below forces of 20 mg.

We would like to eliminate the problem of uncertain normal force and are going to build a unit in which the normal force will be electromagnetically applied. This unit has the desirable features of being self damping and of allowing continuous monitoring of the normal force.

4. Other Data

During the course of the early part of this period when the frictional measuring apparatus was being rebuilt several experimental runs on

TABLE 1

REPRESENTATIVE MEASUREMENTS OF COEFFICIENTS OF FRICTION FOR 1-1/4 INCH EMPIRE WR COTTON FIBER, HIGH INERTIA (K-MONEL) ARM.*

	μ_k		μ_s
	0.278		0.523
	0.248		0.465
	0.291		0.474
	0.253		0.453
	0.252		0.394
	0.241		0.442
	0.182		0.413
	0.251		0.463
	0.206		0.415
Average Value	0.245	Average Value	0.449

$$\text{Ratio } \frac{\mu_s}{\mu_k} = \frac{0.45}{0.25} = 1.8$$

*Normal force = 20 mg ±1 mg; three successive measurements on each of three fibers.

TABLE 2

REPRESENTATIVE MEASUREMENTS OF COEFFICIENTS OF FRICTION FOR 15 DENIER NYLON MONOFILAMENT, HIGH INERTIA (K-MONEL) ARM*

	μ_k		μ_s
	0.327		0.489
	0.303		0.468
	0.296		0.469
	0.335		0.491
	0.268		0.428
	0.275		0.421
	0.271		0.477
	0.289		0.453
	0.259		0.428
Average Value	0.291	Average Value	0.458

$$\text{Ratio } \frac{\mu_s}{\mu_k} = \frac{0.46}{0.29} = 1.58$$

*Normal force = 20 mg ±1 mg; three successive measurements on each of three fibers.

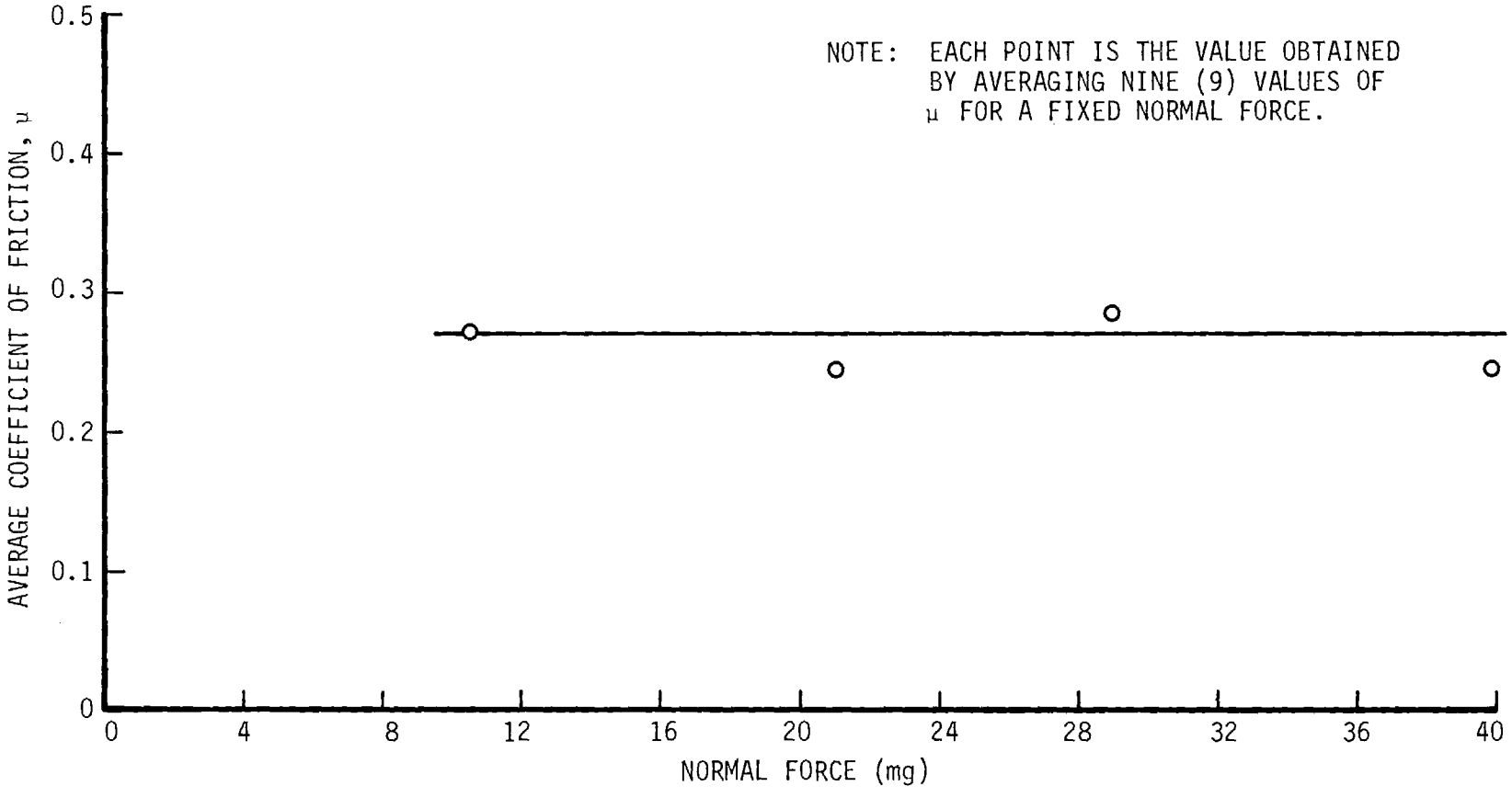


Figure 18. Plot of average coefficients of friction versus normal force for 1-1/4-inch Empire WR cotton fibers.

metal wires of 10 mil diameter were made. Characteristic data are exhibited in Figures 19, 20, and 21 for gold, silver and aluminum respectively. Pertinent data are shown on each graph. These were run with the 1/8" aluminum arm at a normal force in the range 16 to 20 mg. Coefficients of sliding friction in the range 0.18 to 0.50 were observed as shown in Table 3 and μ for static motion (taken from peak values) was > 1 and sometimes much higher implying frictional welding.

Of particular interest is the character of the curves which display very rapid and high peaked force fluctuations. These curves are quite different from those of both cotton and nylon. They were run at higher feed rates of about 52 mm/minute compared with 4 mm/minute currently employed in the fiber friction measurements. The possible changes in the data that may have been introduced by changes in feed rate have not been investigated to date.

5. Discussion

It is evident from the data presented, and from previous data reported by others, that measurement of either the coefficient of static or kinetic friction for fibers on an absolute basis is most difficult. However, the construction of an instrument which gives values that allow comparison between fibers, types of fibers, and effects of fiber processing has been accomplished by several investigators.

In order to reassess expected absolute values of coefficients of friction for various fibers, we have reviewed again selected references on fiber friction measurements in the light of our own measurement experiences. In most cases investigating scientists have recognized the difficulty in computing

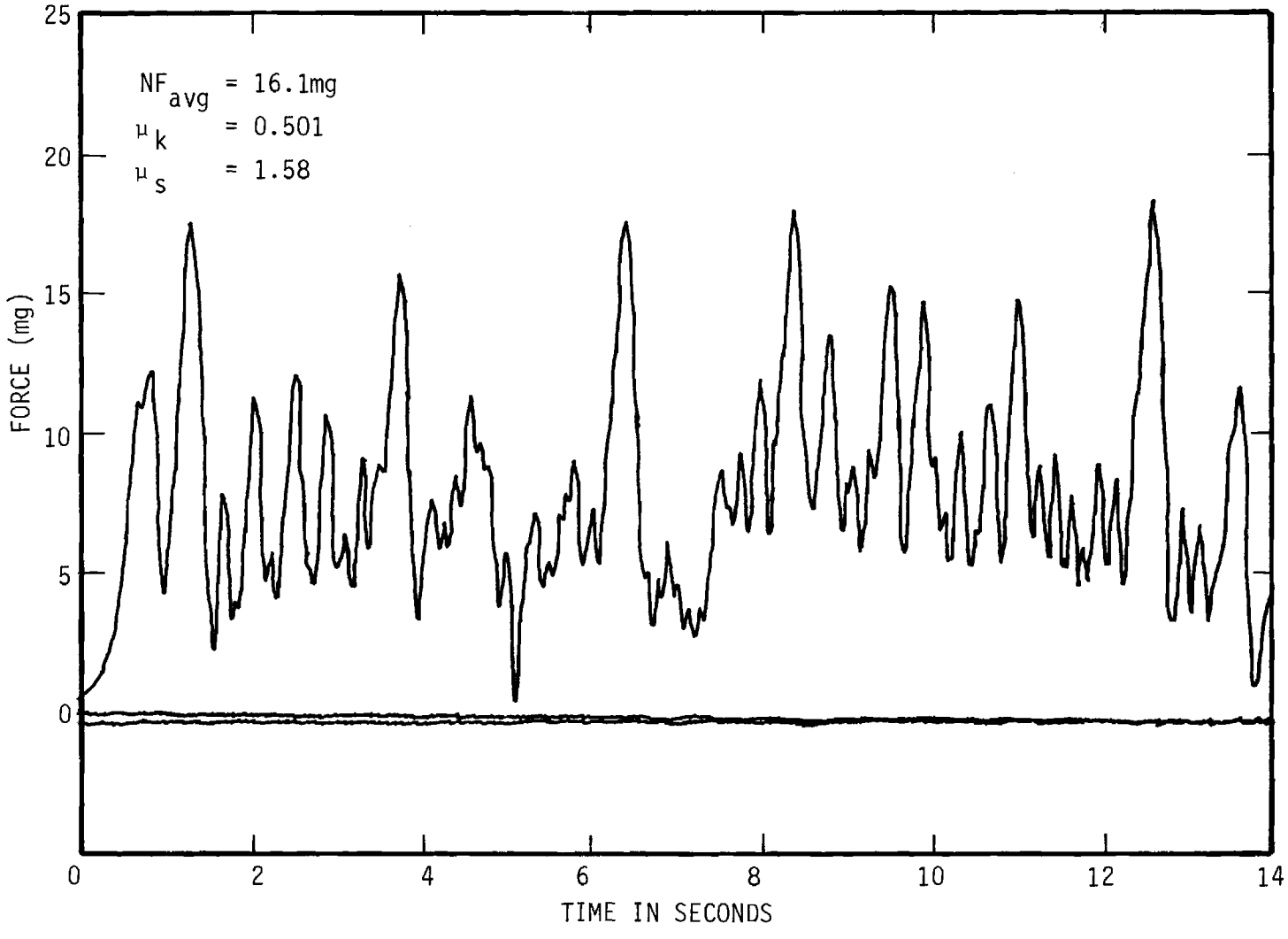


Figure 19. Frictional data plot for a 10 mil gold wire sliding against a similar wire.

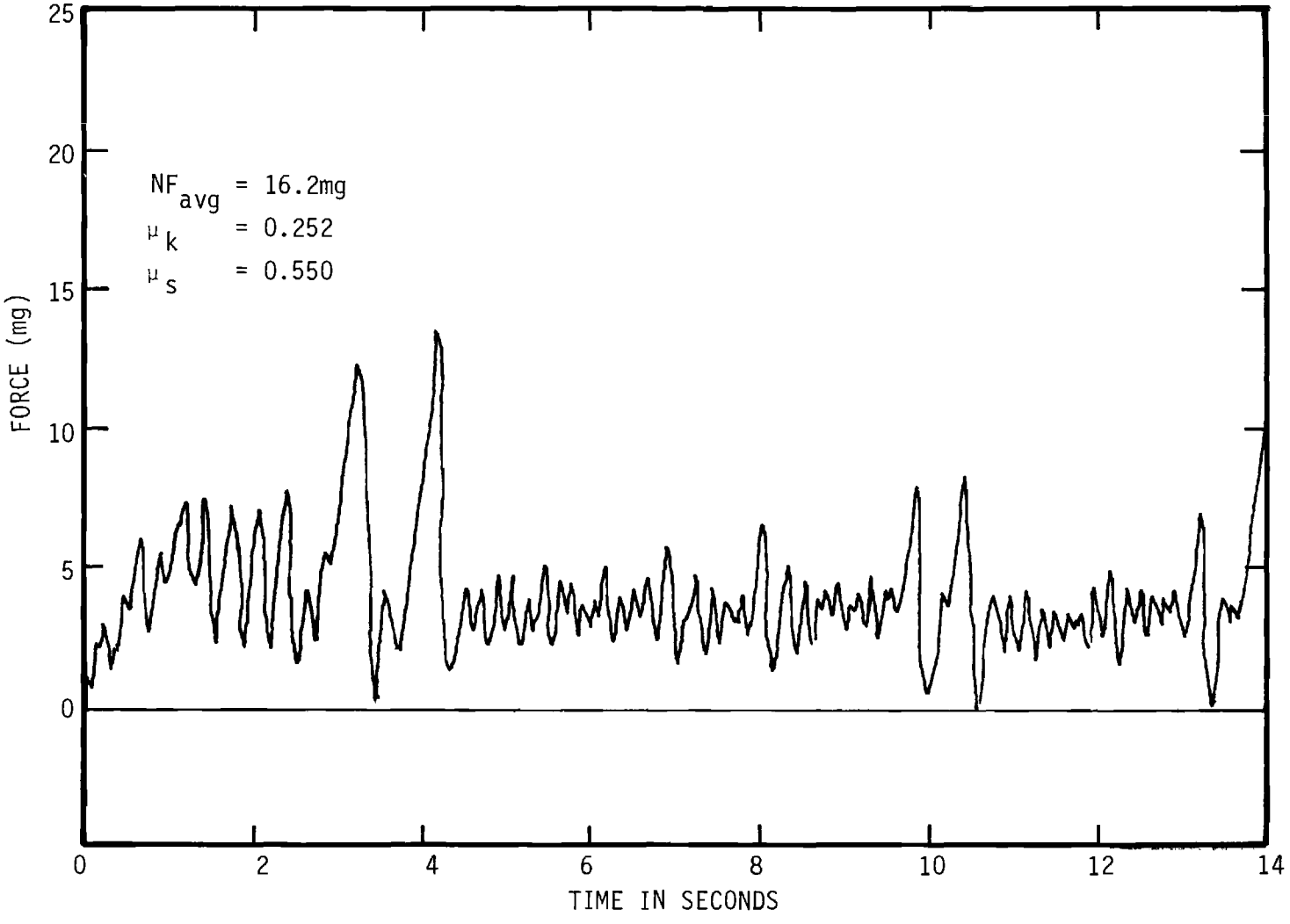


Figure 20. Frictional data plot for a 10 mil silver wire sliding against a similar wire.

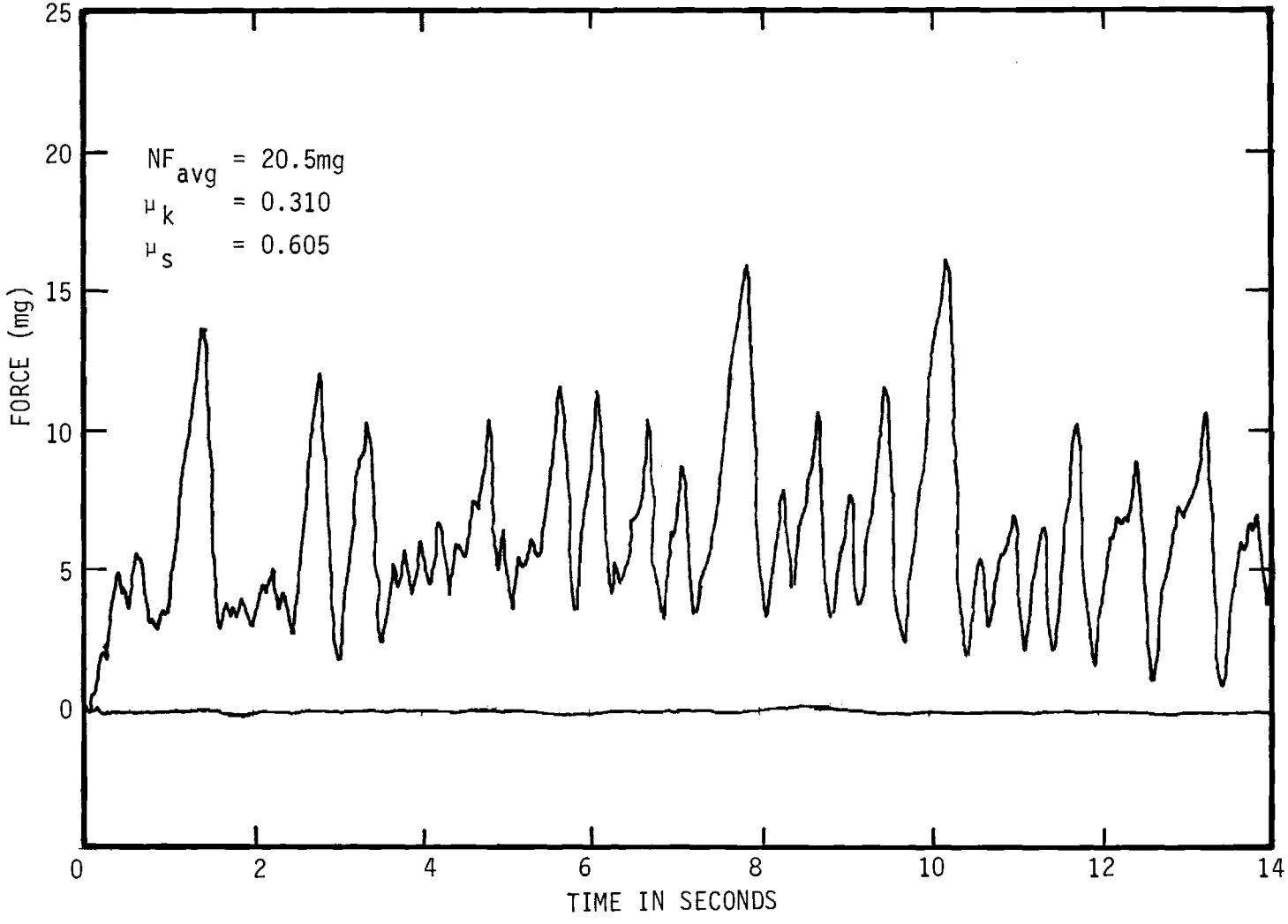


Figure 21. Frictional data plot for a 10 mil aluminum wire sliding against a similar wire.

a satisfactory value for μ_k. They have stated the normal force and the force to initiate sliding (coefficient of static friction) and on occasion the force averaged over a series of stick slip intervals. In no case, however, are we certain that the force was averaged over an entire time interval of measure; and, in fact, most measurements seem to have been obtained by averaging the maxima and minima of the stick slip variations.

Now it may be readily observed from examining the preceding Figures 14-15 that the distance along the abscissa (which is a function of the travel time of the recorder) is much greater on the force increase cycle, from the bottom of a slip to the next peak, than it is for the slip cycle which is almost a vertical line. Lyons and Scheier[2] have pointed out some of the vagaries of the slip cycle in a recent paper, but they failed to point out the much more important fact that the slip cycle time is an insignificant part of the total cycle time, only a few percent at most. The average force for the entire period of a number of stick slip cycles requires integration of force with respect to time; in this case the average force becomes the area under the curve divided by the abscissa above which the area is taken.

The work of Gralen and Olafsson,[3] of Guthrie and Oliver,[4] and of Scheier and Lyons[5] are the only papers that we have found that used devices giving analog traces of the frictional force. The work of the first two used a pencil of light reflected from a mirror on a torsion wire which was deflected through an angle proportional to the frictional force. These two groups also used balance arms which required a value of about 3 mg to overcome friction in the balance arm bearing, in turn requiring a correction to the normal force by this amount. This procedure appears somewhat dubious, especially at low normal forces, < 20 mg.

Scheier and Lyons[5] did use a device having an electrical analog output somewhat similar to ours. However, the force indicator arm moved through an arc on the force increase cycle and was restored by torsion during the slip cycle. In our case the force indicator arm remains stationary except for minute vibrations; there is no mechanical restoring system with an intrinsic period but a nearly instantaneously acting electromagnetic restoring couple.

Let us now examine the results of fiber friction measurements. Gralen and Olafsson[3] report for viscose in the range of our interest:

Normal force	$\mu_{kinetic}$	μ_{static}	Ratio μ_s/μ_k
17 mg	0.180	0.302	1.68
47	0.156	0.282	1.80
67	0.144	0.276	1.92

By restoring the 3 mg correction factor previously mentioned at 17 mg $\mu_k = 0.36$ and $\mu_s = 0.475$ giving a μ_s/μ_k ratio of 1.32, probably too low.

Gralen and Olafsson[3] examined the effect of angle between fibers over the range 10° to 90° and found no definite effect. A theoretical discussion concerning the effect of sliding velocity was also presented. It was reported that sliding velocities up to 1.5 cm/sec should make little difference in measurements obtained; effects of surface treatments were reported. They also discussed the problem of adhesion or cohesion when the value of normal force applied from an external source approaches zero, and suggested an equation for the tangential force to cause sliding, $F = \mu N + \alpha S$ where μ is the coefficient of friction, N is the normal force, α is the force of adhesion and S is the area of contact.

Guthrie and Oliver[4] used an apparatus very similar to that of Gralen and Olafsson[3]. Of particular interest is the fact that they reported an increase in the frictional force of approximately 50% for an increase of fiber tension from 400 mg to 1600 mg. At 125 mg normal force this amounts to a change in μ_k from 0.12 to 0.19 for viscose rayon. However, their data was taken at low tensions, about 500 mg; typical values cited were

	Normal finish	Extracted
F_k/R (125 mg)	0.141	0.154
F_s/R (125 mg)	0.216	0.228
Ratio $\frac{F_s}{F_k}$	1.53	1.48

Slightly higher coefficients were found for fibers extracted to remove finish. Studies of the effect of denier revealed no consistent changes that could be ascribed to this cause. Guthrie also reviewed the angle of contact measurements and concluded that experiments conducted at 90° between the fibers were representative. Effects caused by delustering, dyeing, and processing for dyeing were observed. Generally speaking values of μ were increased by these treatments but much more by some dyes than by others.

Scheier and Lyons[5] skirted making direct reports on values for μ they obtained except in one instance. A value of μ_s for nylon 66 against a razor blade slider was found to be approximately 0.70 for several variously treated fibers over the range 5 to 30 mg normal force. Other values for μ_s for several fibers can be estimated from graphical data in the same general range of force. Gralen and Olafsson[3] found a ratio of μ_s/μ_k of about 1.80

between rayon and rayon. The value 0.70/1.80 then gives 0.39 as a rough approximation of μ_k for nylon against a razor blade extrapolated from Scheier and Lyons[5] data. Our value for μ_s/μ_k for nylon was 1.58 and 0.70/1.58 = 0.44. However, we found no value of μ for nylon against steel other than the one given by Scheier and Lyons. The value we reported previously for nylon on nylon was 0.40. This was much higher than the value 0.29 reported by us in Table 2.

Examining the various data it is readily observed that variations in equipment, balance arm inertia, balance arm bearings, normal force, fiber tension, and less obvious factors may seriously affect the measurement of fiber friction. Furthermore, data reports are difficult to evaluate because of obscureness on the part of the authors concerning vital details of an experiment. The values reported by Gralen and Olafsson[3] and Guthrie and Oliver[4] appear somewhat low; and the reports of Scheier and Lyons[5] were insufficient to evaluate. Our own values are low compared with those initially obtained but higher than those of the first two groups of investigators.

With the normal force applied electromagnetically and simultaneously damped as previously noted it is expected that measurement consistency and repeatability will be improved and that friction measuring instrumentation can be duplicated so that one may obtain essentially the same measurement values for a fiber on duplicate instruments.

6. Summary

It has been shown that the coefficient of friction of fibers as measured by various devices may vary according to a number of parameters related to the construction of the device and to procedures used in obtaining

and reducing data. A survey of typical data from other sources reveals little that is directly comparable to the present work. Investigations by others of the effects of fiber tension, fiber denier, the effect of the angle between the fibers, and of the effects of changes in normal force have given some guide lines of the conditions that may be expected in these areas. The fiber tension appears to be one of major significance and may account in part for the large differences in measurements of the coefficient of friction observed between the measurements conducted in the first and second period. μ_k for cotton was found to be about 0.25 in contrast to 0.42 in the first period, and that for nylon 0.29 compared with 0.40. The ratios of μ_s/μ_k for the two were about 1.8 and 1.6 respectively compared to the previously reported values of about 2.0 and 1.5. The endeavors in defining the various factors affecting the measuring instrument's response and accuracy are expected to yield an improved and satisfactory frictional measurement apparatus in the near future.

D. INVESTIGATIONS OF COTTON FIBERS BY INFRARED TECHNIQUES*

1. Introduction

Research during this period has been principally directed toward the development of a new method of whole fiber specimen preparation for infrared examination and toward perfecting procedures for the evaluation and employment of infrared reflectance equipment and techniques. Additional examinations have been made of cotton waxes, cotton, and other fibers.

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*This section contributed by M. P. Smoak, Student Assistant (Physics), W. E. Kirkland, Graduate Assistant (Textiles), and J. A. Knight, Research Professor (Radioisotopes Laboratory).

2. Extraction of Cotton Wax and Determination of its Infrared Spectrum

a. General

Three extraction techniques and four solvents were used to remove and concentrate cotton wax for determination and comparison of its infrared spectrum. Empire WR cotton was obtained from the project standard bale and cleaned by passing it twice through a Shirley Analyzer. Spectra were taken of the extracted wax in both a solution and a paste phase. Both KBr and reflectance spectra were taken of the wax paste.

b. Experimental

(1.) Extraction Methods and Solvents

(a.) Hot Soxhlet Extractions

The initial extractions were performed using a Soxhlet apparatus with ethanol and chloroform as solvents. The rates of extraction and the percent wax obtained agreed with the results of Kettering.[6]

(b.) Room Temperature, Static, and Dynamic Extractions

Room temperature extractions were made to eliminate the effects of heat on the wax and its spectrum. In these, the cotton was immersed in the solvent and allowed to stand, or was stirred. This method is useful in obtaining one gm or larger quantities of wax but the time required for complete wax removal proved to be excessive for routine work. The solvents used for these extractions were chloroform and n-hexane.

(c.) Modified Soxhlet Extractions

Soxhlet extractions were made using a cold-finger condenser which allowed reduction of the pressure inside the extraction apparatus. By reducing the pressure and using a dry ice-acetone mixture in the

condenser, efficient solvent cycling was obtained with the solvent at room temperature. Teflon gaskets were used to seal the glass joints, and a water bath was used to keep the solvent in the boiling flask at room temperature. As shown in Figure 22, the efficiency of this method compared favorably with the hot Soxhlet extractions. Chloroform, n-hexane, and methyl isobutyl ketone were used as solvents in these extractions.

(2.) Infrared Techniques

(a.) Wax Solution Spectra

Spectra were obtained using a wax-chloroform mixture in the sample cell and chloroform in the reference cell. This method is useful since chloroform is an effective solvent for extracting cotton wax and, if so used, the extracted samples may be placed directly into cells for analysis. However, the information available in regions of high chloroform absorption is limited.

(b.) Wax Film on a Halide Disk

Paste wax, with all traces of solvent removed under vacuum, was spread as a film on a KBr disk and the spectrum was taken. This is a quick, simple method and gave excellent spectra.

(c.) Wax-KBr Pressed Disks

Satisfactory wax-KBr pressed disks were not obtained although several methods of preparing them were tried. In all cases, the disks crumbled on removal from the die. This problem persisted when wax concentration was drastically reduced. Therefore, this method was unsatisfactory for wax study.

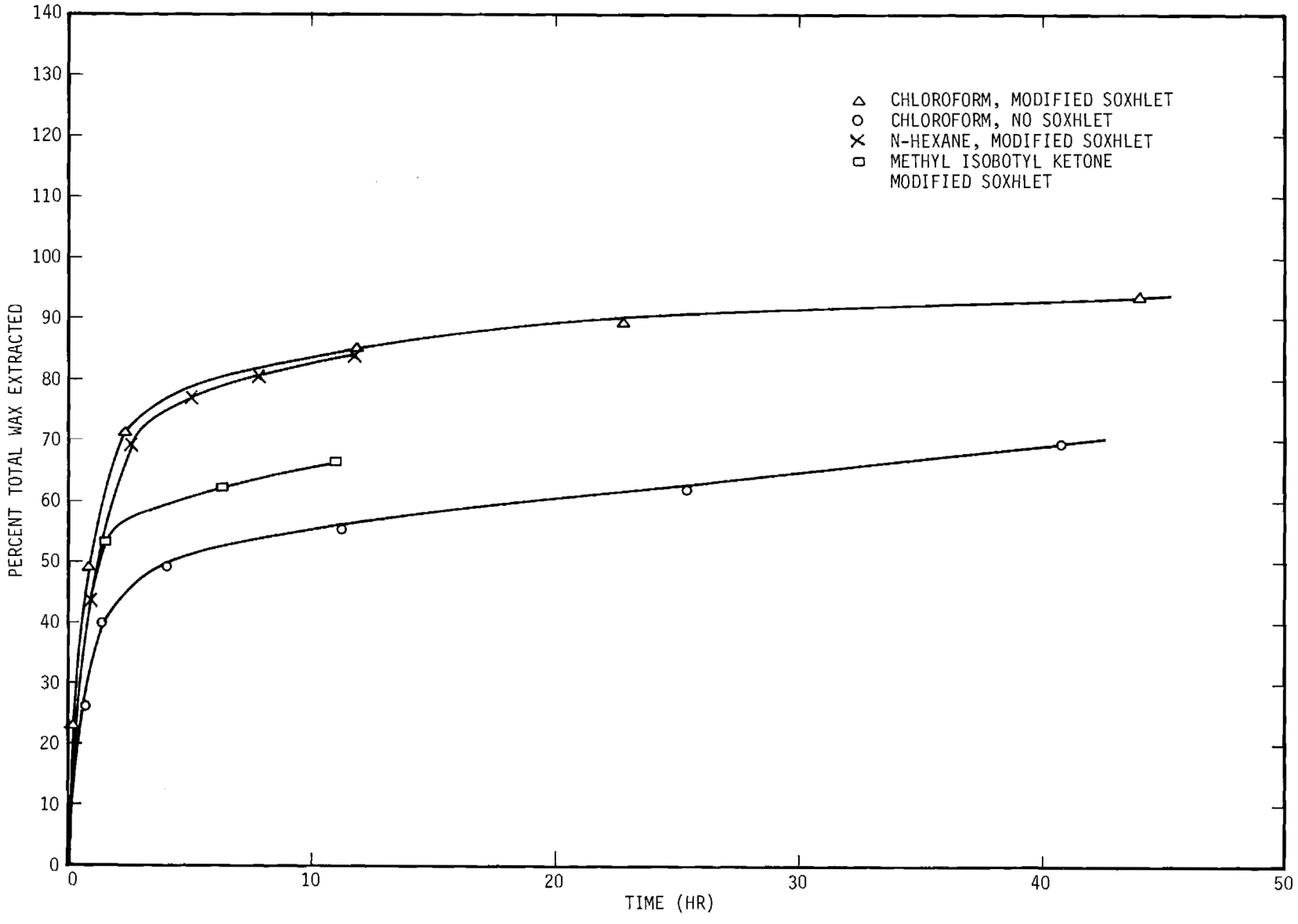


Figure 22. Data indicating percent cotton wax extracted versus time for three extraction methods.

(d.) Reflectance Spectra

Spectra were obtained using the Wilks Multiple Internal Reflectance Device described in a later section of this report. For these, the wax was spread as a film on the KRS-5 sample plate. As explained later, this technique is well suited for wax studies.

(3.) Data Obtained

The infrared spectra of a number of samples of extracted wax are given in Figures 23 A, B and C and Table 3 summarizes the work on the wax extractions with some comments about the spectra.

c. Summary

The extraction data and corresponding spectra indicate that wax may be satisfactorily removed from cotton by any of the methods discussed in the preceding section 2b. The solvents tested were satisfactory except for slight spectral changes when ethanol was used. Therefore, the choice of which method or solvent to use should be determined by the object of the extraction. For example, to obtain a large quantity of cotton wax, the static, room temperature method would be preferable. To completely de-wax cotton quickly, the hot or modified Soxhlet method could be used. In either case, the choice of solvent is relatively open; all those tested were satisfactory.

3. Internal Reflectance Devices in the Study of Cotton and Other Textile Fibers

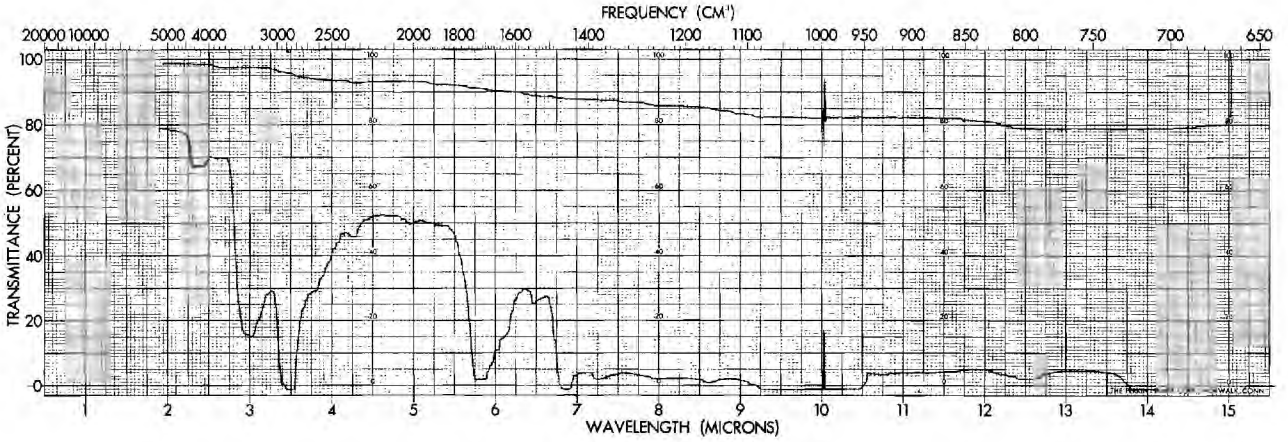
a. General

The infrared group has recently considered the possible use of internal reflectance spectroscopy in the study of textile materials. The initial study of the literature indicated that the technique should prove

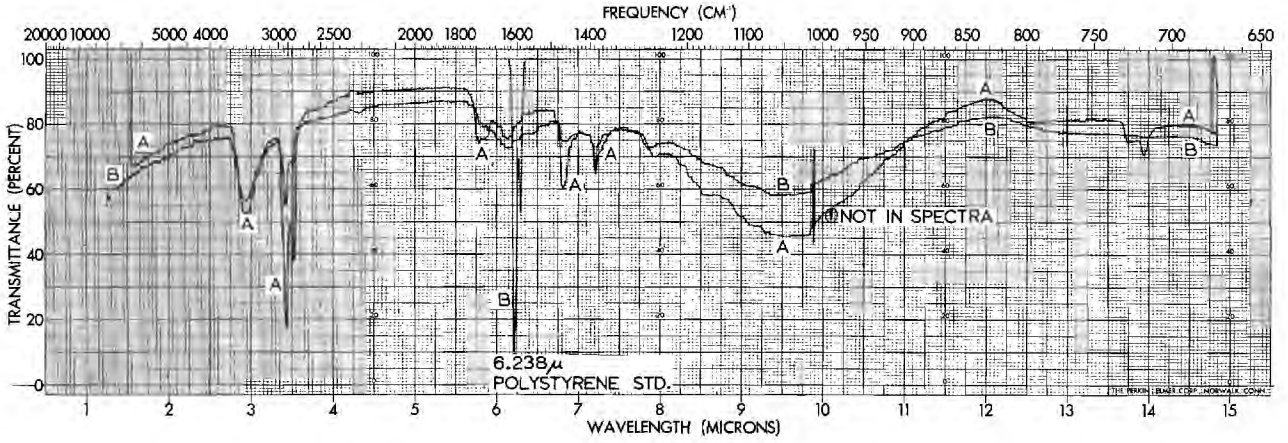
TABLE 3

SUMMARY OF WORK ON WAX EXTRACTIONS

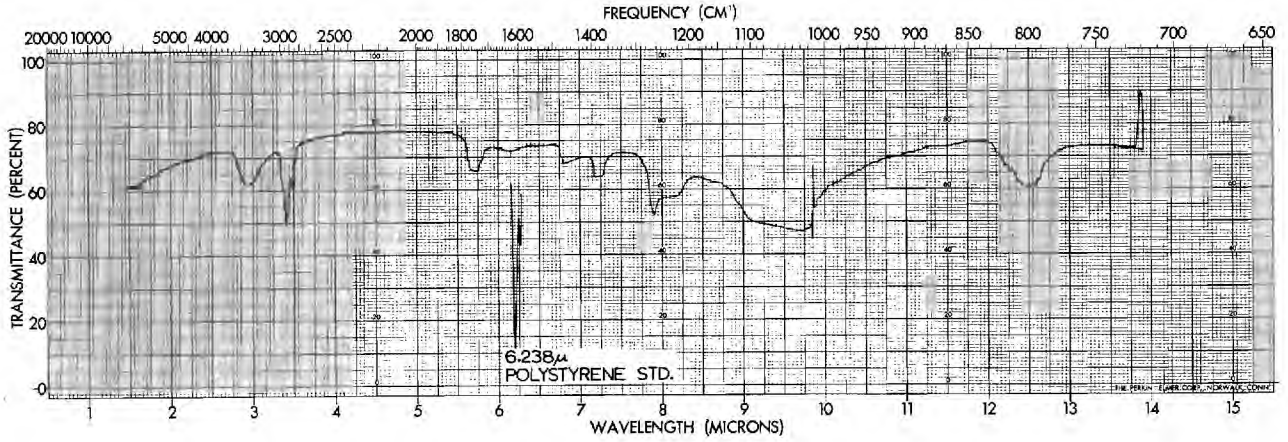
Extraction Technique	Solvent	Infrared Method	Comments about the Spectra
Hot Soxhlet	Chloroform	Transmission: Wax film on KBr disk	See Fig. 19, Proj. A-843, Report #1 Spectra taken showed major absorption bands around 2.95, 3.4, 3.5, 5.8, 6.85, 7.25, 7.95, 12.5, 13.75, and 13.95 microns.
Hot Soxhlet	Ethanol	Transmission: Wax film on KBr disk	See Fig. 19, Proj. A-843, Report #1 These spectra were similar to those obtained from chloroform extracted wax with small changes in the 8-15 micron region.
Static, Room Temperature	Chloroform	Reflectance: Wax film on KRS-5 reflectance crystal	See Fig. 23A. This spectrum is similar to the KBr spectra in the 2 to 7 micron region and shows bands at 2.35, 4.25 and 6.5 microns. Not observable in those spectra due to low concentration of wax.
Modified Room Temperature Soxhlet	Chloroform	Transmission: Wax film on KBr disk	See Fig. 23B. The spectra shown are: (A) wax removed during the first half hour of the extraction, and (B) wax removed during the sixth hour. While these spectra are similar, there are noticeable changes, particularly in the ratio of the 2.95 and 3.4 micron bands.
Modified Room Temperature Soxhlet	n-hexane	Transmission: Wax film on KBr disk	See Fig. 23C. All bands are the same as in the other spectra; intensity differences probably are due to the smaller amount of sample used.
Modified Room Temperature Soxhlet	Methyl isobutyl ketone	Transmission: Wax film on KBr disk	Not shown. All absorption bands appeared at the same wavelengths as in Fig. 23B. Higher concentration of wax used for this spectra failed to show bands at 2.35, 4.25 and 6.5 microns as observed with the very concentrated reflectance sample, Fig. 23A.



A. REFLECTION FROM WAX PASTE, STATIC CHLOROFORM EXTRACTION



B. KBr PELLET, RT CHLOROFORM EXTRACTION



C. KBr PELLET, RT HEXANE EXTRACTION

Figure 23. Comparison of infrared spectra of cotton wax specimens extracted at room temperature: (A) static chloroform; (B) chloroform using sohxlet; (C) hexane using sohxlet and low pressures.

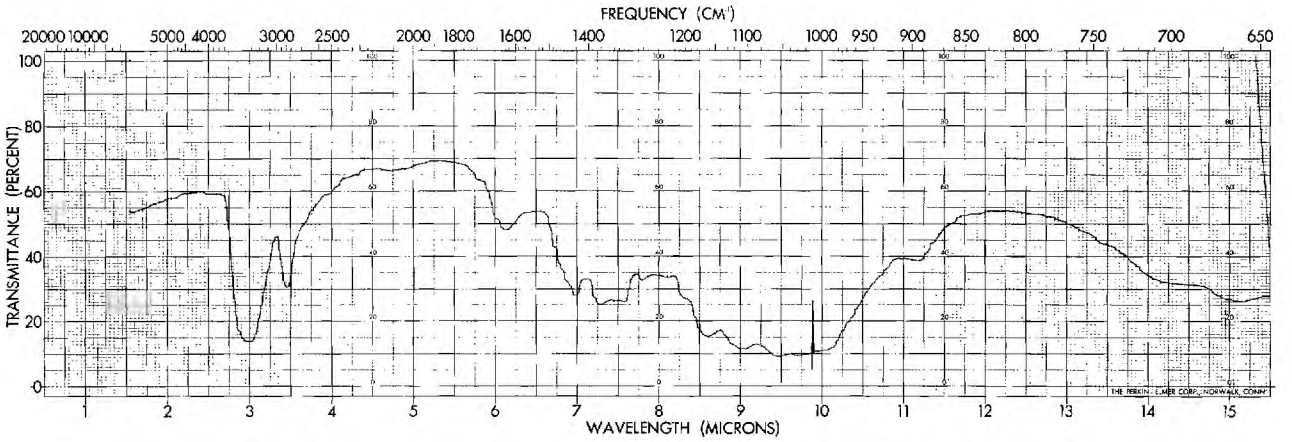
useful in obtaining the spectra of both fibers and surface coatings which might affect their frictional properties. A single beam, attenuated total reflectance accessory, obtained for testing, proved unsatisfactory because the radiation was reflected only once from the sample interface. Sufficient attenuation of the sample beam could not be obtained during this single reflection to produce satisfactory spectra.

After this experience, a double beam, multiple internal reflectance attachment[7] was considered next. The principle employed in this instrument has been explained in detail;[8] basically, the radiation is internally reflected many times from the sample-crystal interface, effectively multiplying the amount of sample beam attenuation due to absorption and thus yielding a satisfactory, reproducible spectrum of the sample. The double beam design provides reference beam compensation and allows differential reflectance spectra to be taken. This device has recently been acquired.

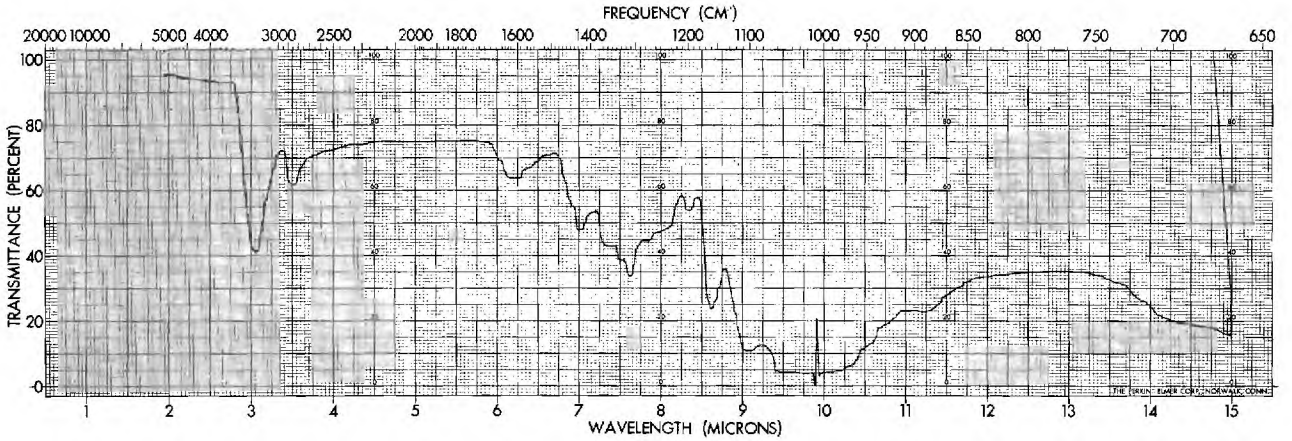
b. Experimental

Initial work with the attachment has yielded satisfactory, reproducible spectra of cotton and other textile fibers. Figures 24, 25 and 26 present typical spectra obtained; Table 4 summarizes these and references KBr spectra for comparison.

The reflectance spectra were obtained using a 1 mm crystal, KRS-5, as the specimen holder with the spectrometer slit program set for 2X. To assure reproducible results, the specimen holder plate was tightened with a torque wrench to 6 inch-pounds.

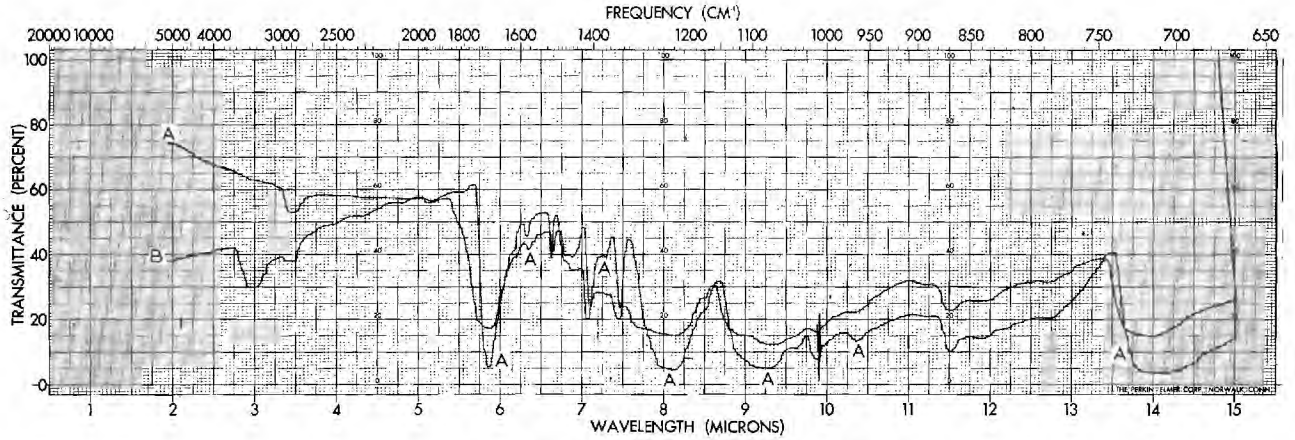


A. TRANSMISSION SPECTRA, KBr PELLET

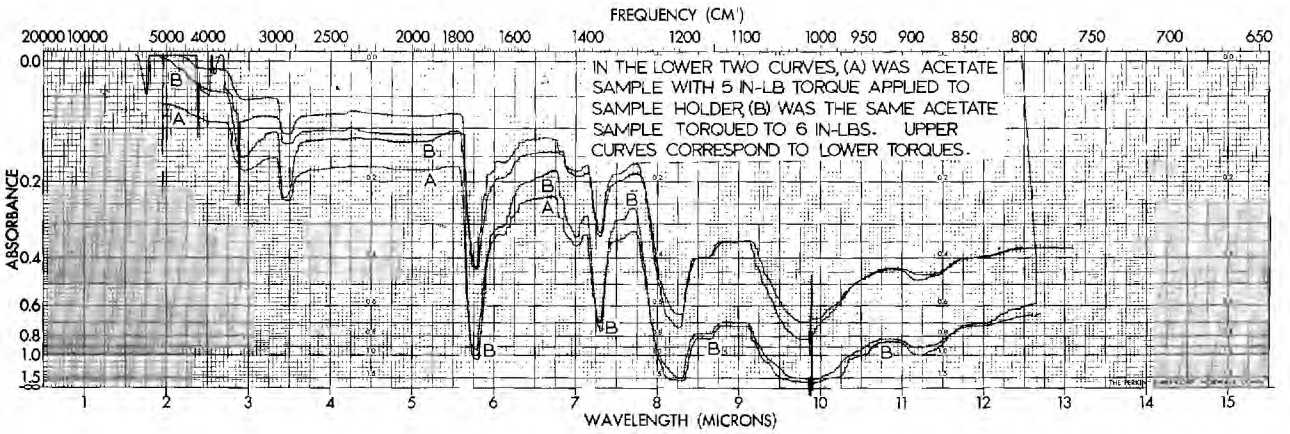


B. WILKS REFLECTANCE ATTACHMENT

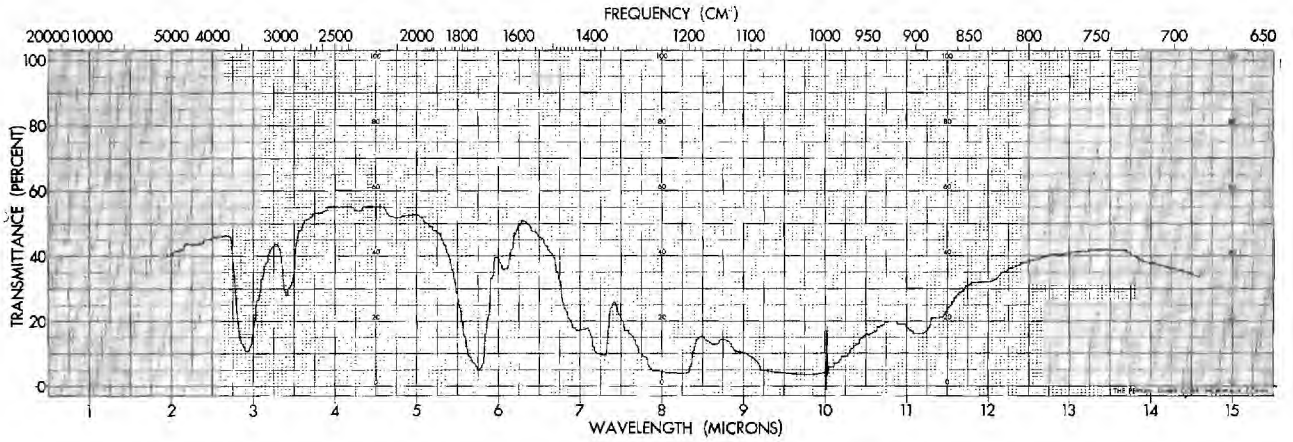
Figure 24. Comparison of infrared spectra of Empire WR cotton obtained by KBr transmission method and by the Wilks reflectance apparatus.



A. DACRON, REFLECTANCE AND KBr

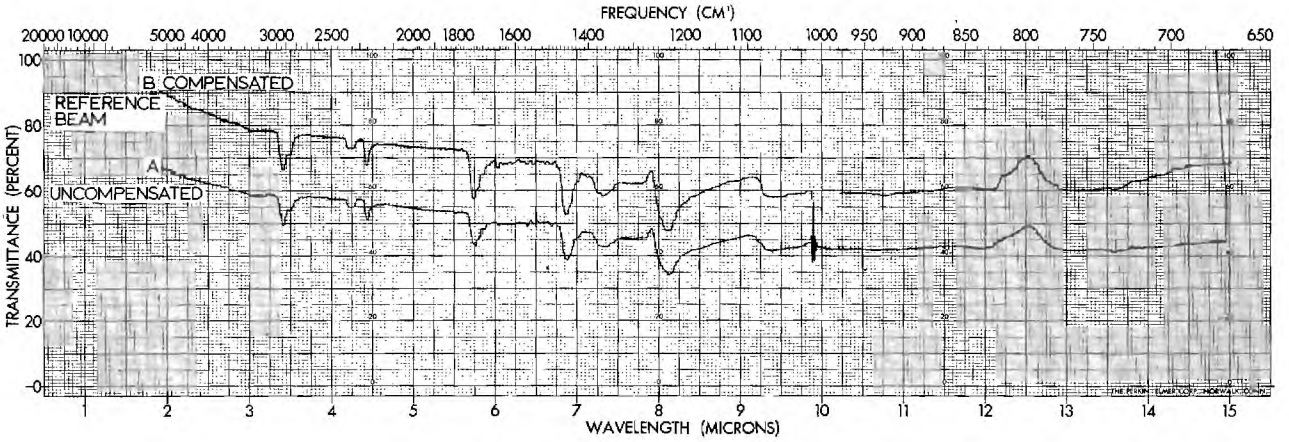


B. ACETATE, REFLECTANCE

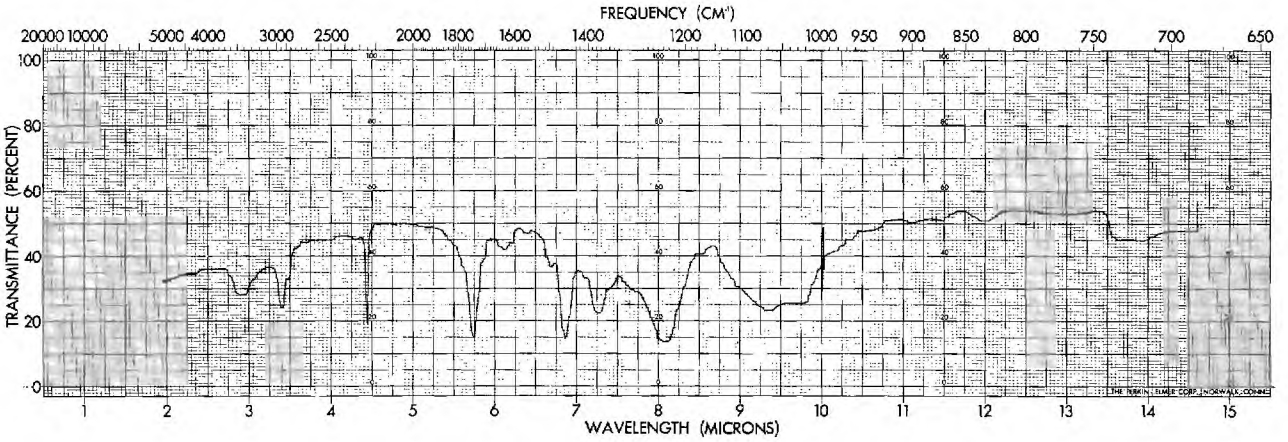


C. ACETATE, KBr PELLET

Figure 25. Infrared reflectance spectra of Dacron and Acetate Rayon.



A. ACRILAN, REFLECTANCE



B. ACRILAN, KBr PELLET

Figure 26. Infrared reflectance spectra of Acrilan.

TABLE 4

INFRARED SPECTRA OBTAINED BY REFLECTANCE AND KBr PELLET METHODS

Fiber Sample	Reflectance Spectra	KBr Spectra
Cotton	Figure 24	Fig. 24 A
Dacron	Figure 25 Spectra A	Fig. 25, Spectra B
Acetate	Figure 7	Fig. 25
Acrilan	Figure 26	Fig. 26

c. Discussion

The spectra obtained by reflectance compare satisfactorily with those obtained using the KBr pressed disk technique and in some cases are superior. Caution should be exercised, though, in interpreting these spectra. Potts[9] has pointed out that spectra obtained by reflectance may appear to be distorted. This distortion is due to sudden changes in the sample's index of refraction at absorption bands and gradual change in the index over the 2 to 15 micron region. The first of these causes the absorption band to appear broadened on the long wavelength side; the gradual change causes bands in the long wavelength region to appear to be stronger than corresponding transmittance recorded bands. In addition to these considerations, slight contamination of the reflectance crystals can produce noticeable distortion of the one-hundred percent line. All of these will greatly affect attempts of quantitative analysis, but appropriate corrections should allow satisfactory quantitative results.

Since reflectance spectra are characteristic of the surface of the sample, this technique is well suited for the study of coatings and surface contamination. Accessories which allow differential reflectance spectra to be obtained have been acquired and future work is anticipated in this area.

d. Summary

Equipment for recording reflectance spectra has been obtained and the spectra thus obtained compares favorably with KBr transmission spectra. Table 4 summarizes the spectra obtained of cotton and other fibers. Reflectance spectra are reproducible and, with proper care, may be used for quantitative work. Coatings and other surface properties may be studied using reflectance techniques, and future work in these areas is anticipated.

3. Pressed Fiber Sample Technique

a. Introduction

As is the case with most solids, textile fibers are not, as such, suitable samples for direct infrared analysis. The present methods for obtaining the infrared spectra of fibers are:

(1) the KBr pressed disk technique

(2) the Nujol mulling technique

(3) the cast film technique.

All have obvious disadvantages. In the first two, a substance other than the sample is introduced and the superimposed spectra of the two results. Due to adsorbed water and impurities, KBr may show some absorption. The third method, while obtaining a spectrum of the pure sample, may introduce structural changes which may correspondingly change the spectrum.

An alternate method has been suggested[10] whereby the sample would be prepared by forming a thin, smooth, closely packed, film-like layer of fibers by pressing. This method, as described, is applicable to continuous filament synthetics. Although "films" of short staple fibers have been obtained by chopping the fibers before pressing,[11] they are difficult to produce and are extremely fragile. A device developed recently on this project eliminates these difficulties and is being used to prepare "fiber film" samples of cotton and other short staple fibers.

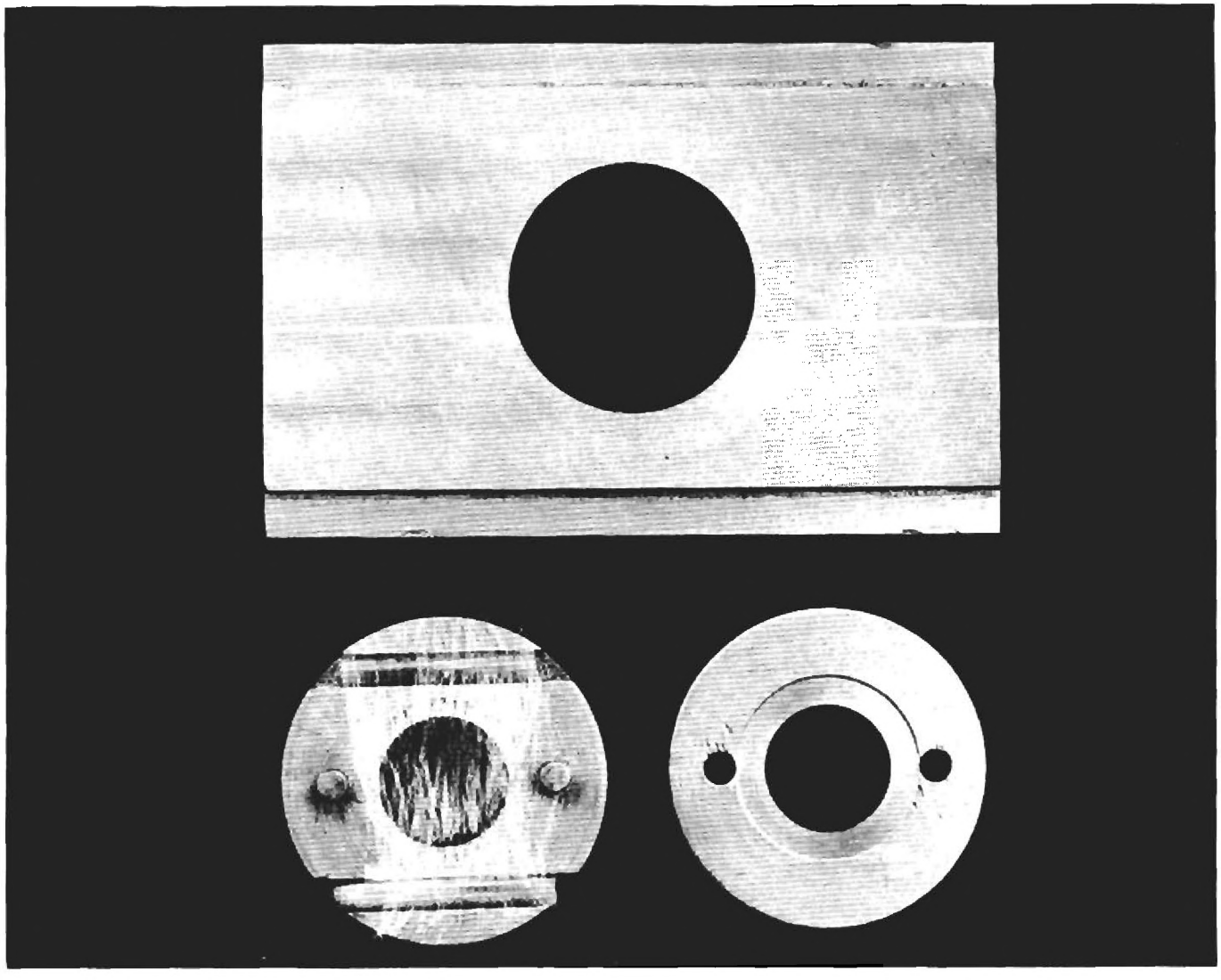
b. Description

The device, which is basically a holder for maintaining a thin, close packed, parallel fiber arrangement before, during and after pressing, is shown in Figures 27, 28, 29, and 30. As shown in Figure 30, the holder is designed for use with a Perkin-Elmer KBr die assembly, but could be easily modified for use with other pressing equipment. The three parts of the device are: (a) the holder which maintains the fiber arrangement both during pressing and while the spectrum is being recorded; (b) the spacer which places the fiber layer at the proper level in the KBr die, i.e. the level of the lower mirrored surface; and (c) the adapter which fits the spectrometer cell holder and holds the spacer-holder assembly in the spectrometer.

c. Procedure

The steps in using the devices are as follows:

(1) Place the adapter on a flat table, the barrel of the KBr die on the adapter, and the KBr die plunger inside the barrel so that it passes through the adapter and rests on the table. The adapter here serves only as a spacer. When the holder is placed atop the barrel, the plunger



A B

Figure 27. Parts constructed to adapt KBr pellet press to a fiber press. These consist of the adapter and the fiber holder parts A and B. Note placement of cotton fibers.

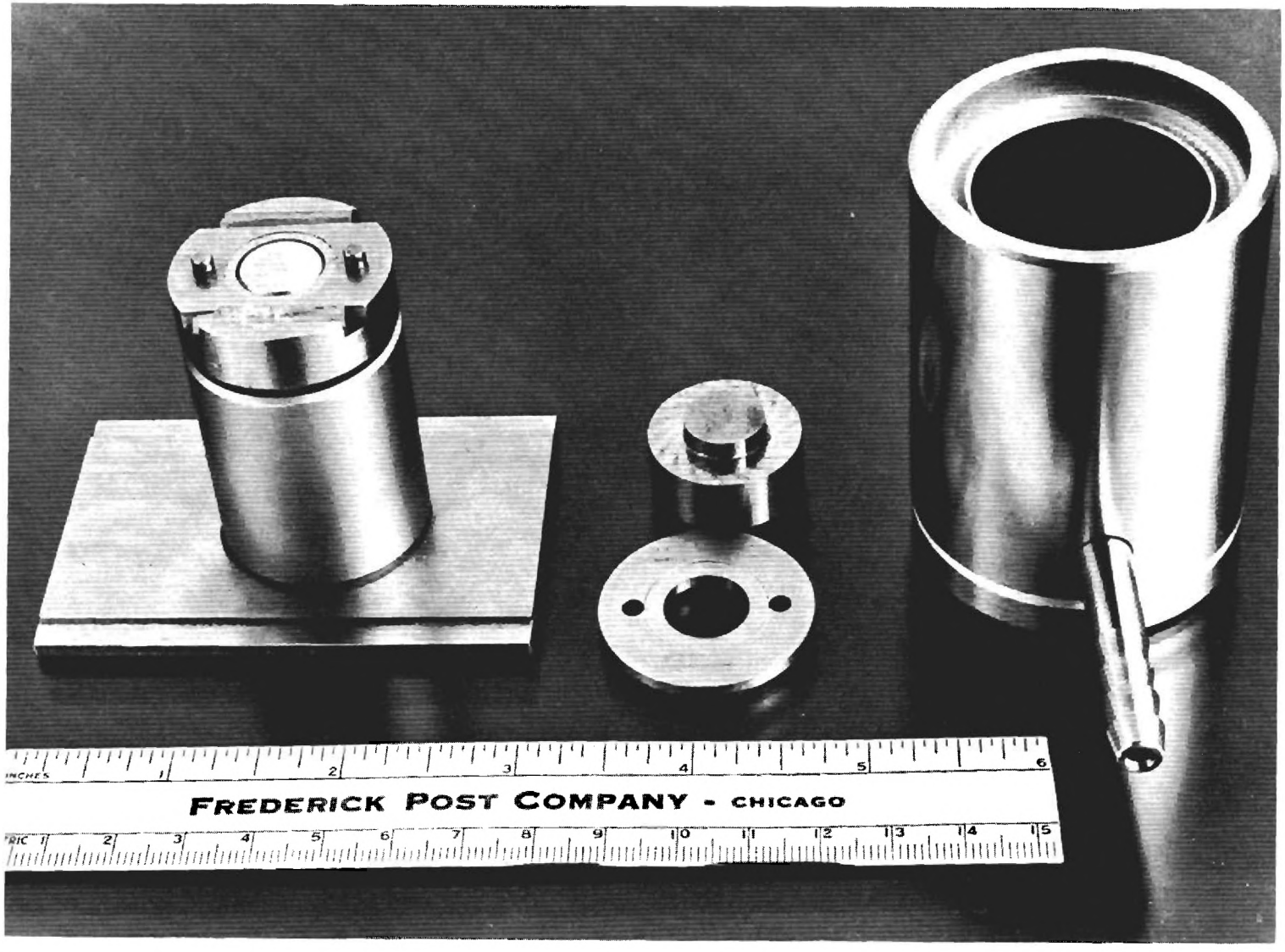


Figure 28. Complete press partially assembled showing use of adapter in assembling in inverted position.

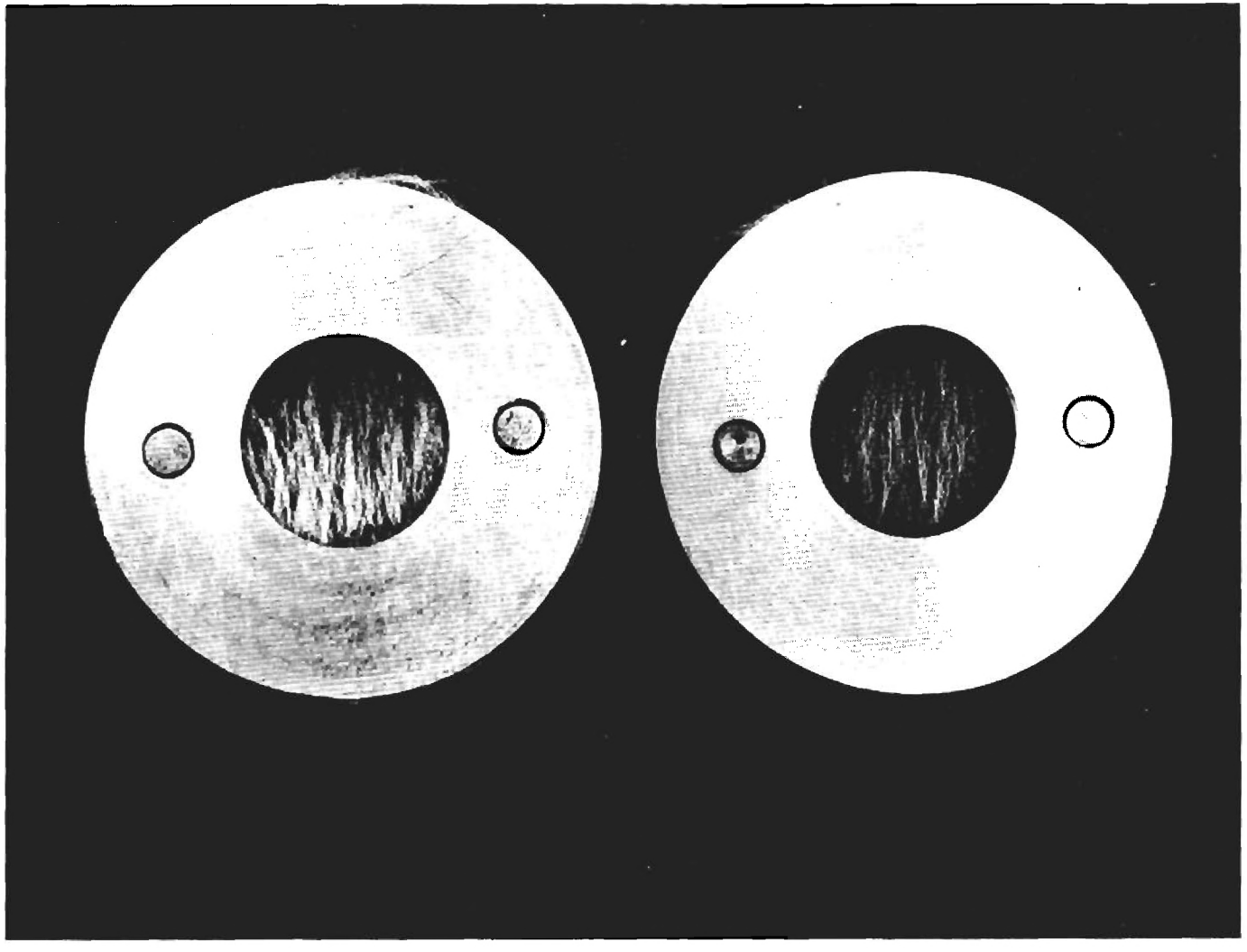


Figure 29. Fiber specimen before and after compression.

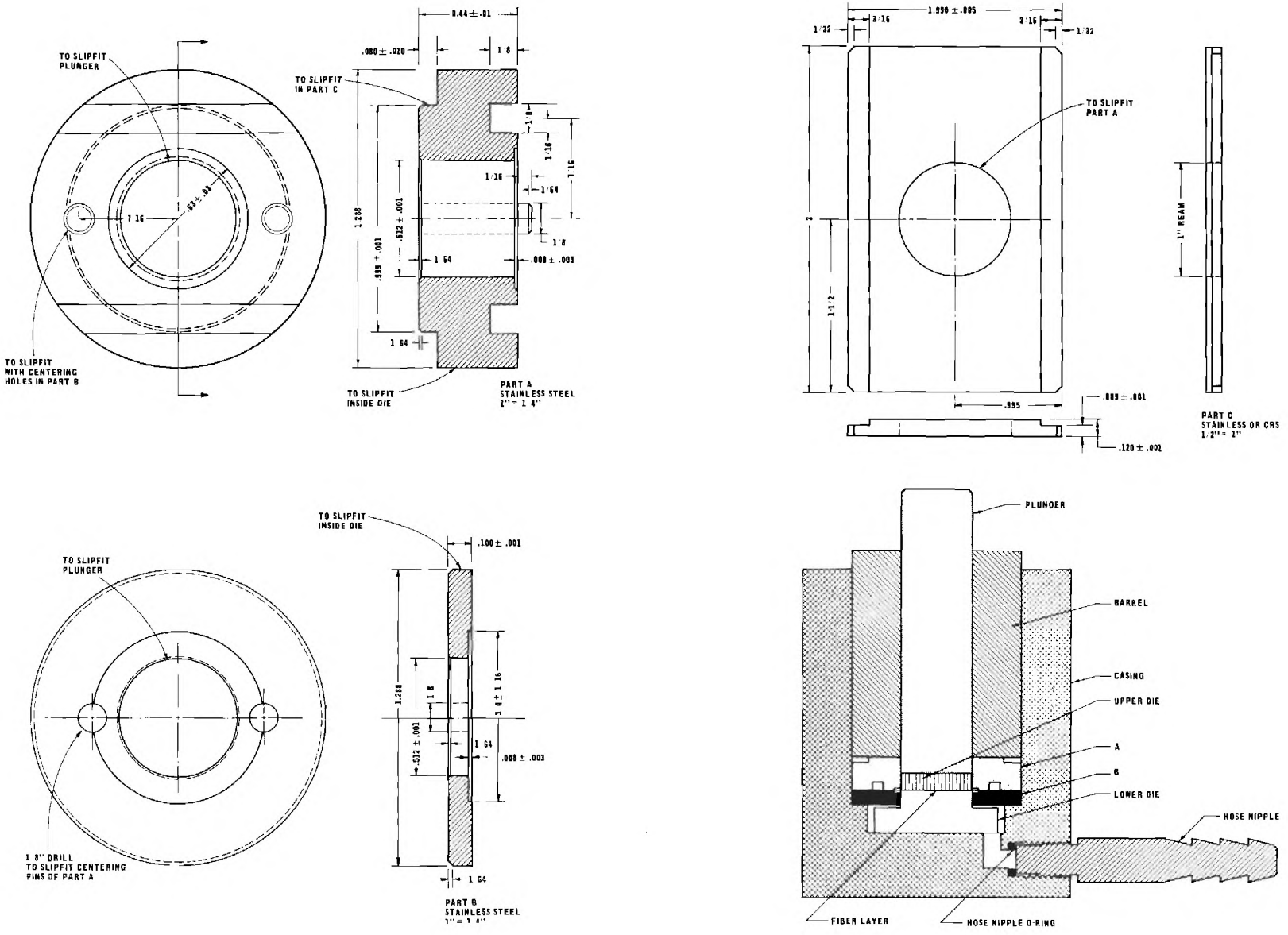


Figure 30. Dimensional drawing of fiber press parts and cross-section of assembled press.

should be even with the surface of the holder as shown in Figure 28. If not, use a suitable thickness of cardboard instead of the adapter. The plunger later acts as a support for the fibers.

(2) Beginning with a small bundle of fibers, a metal comb is used to comb out the short fibers until only one inch or longer fibers remain. These should now be fairly parallel, and their arrangement should be an evenly distributed, thin layer approximately 3 to 5 fibers thick and wide enough to cover the hole in the holder. See Figure 27.

(3) The fibers are then secured between the slots in the holder so that a thin uniform layer exists over the hole in the center. Several methods of securing the ends of the fibers have been tried; the best method seems to be a small cylinder of scotch tape, rolled with the adhesive side out and inserted into the slot, so that the fibers are held when pressed down lightly onto the tape. Clamping with a piece of cork or rubber after the fibers are in place tends to create uneven areas. Since very few fibers are used, the tape remains sticky enough for use with several samples and need not be removed until it fails to hold satisfactorily. After one end is fastened, the fibers may be smoothed slightly with a piece of velvet or a finger tip to assure an even distribution and then fastened at the other end.

(4) Place the spacer, Part B, on the holder with the counter-sink down. The counter-sinks on parts A and B provide for slight movements of the fibers while being placed in the KBr die casing.

(5) Place the lower die (Perkin-Elmer part) in the spacer hole so that the mirrored surface rests on the fibers which are supported by the plunger.

(6) Invert the KBr die casing and carefully slide it down on the barrel, holder and lower die.

(7) Slide the entire assembly to the edge of the table so that the plunger may be held from beneath. Hold the plunger and invert the entire assembly.

(8) Remove the plunger, insert the upper die, and replace the plunger.

(9) Apply pressure of 9 to 11 tons for 1 to 5 minutes.

(10) Remove the plunger and then the barrel. The holder with the upper die inside can now be seen.

(11) Using a suitable device, such as a large pair of tweezers, prevent the holder from moving while inverting the casing and allowing the upper die to drop out. Caution: Be sure the die drops onto a soft cloth to prevent scratching the mirrored surface.

(12) Partially insert a suitable size cork into the hole in the holder for use as a handle and pull upward lightly to remove the holder and film. The film is quite fragile and may be broken by air currents if the cork is not used.

(13) Place the adapter in the spectrometer cell holder and slip the fiber holder into the adapter. The spectrum may now be taken as usual. Best results are obtained when some method of reference beam compensation is employed since the average I_o for the samples is usually less than 50%.[12]

d. Discussion

The technique described above is quite simple once mastered, and sample preparation time is well under ten minutes including pressing.

Spectra of cotton and other fibers have been obtained and compared with KBr spectra as indicated in Table 5 and Figures 31 and 32.

Further work is planned to develop and improve the fiber-press technique and to investigate areas where it may be especially useful.

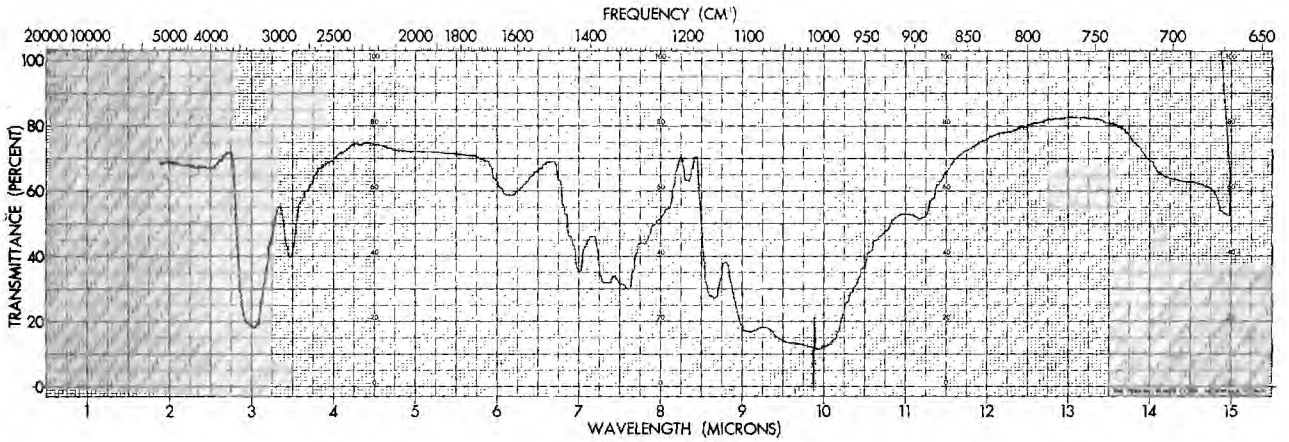
TABLE 5

INFRARED SPECTRA OBTAINED FOR FIBER PRESS SPECIMENS

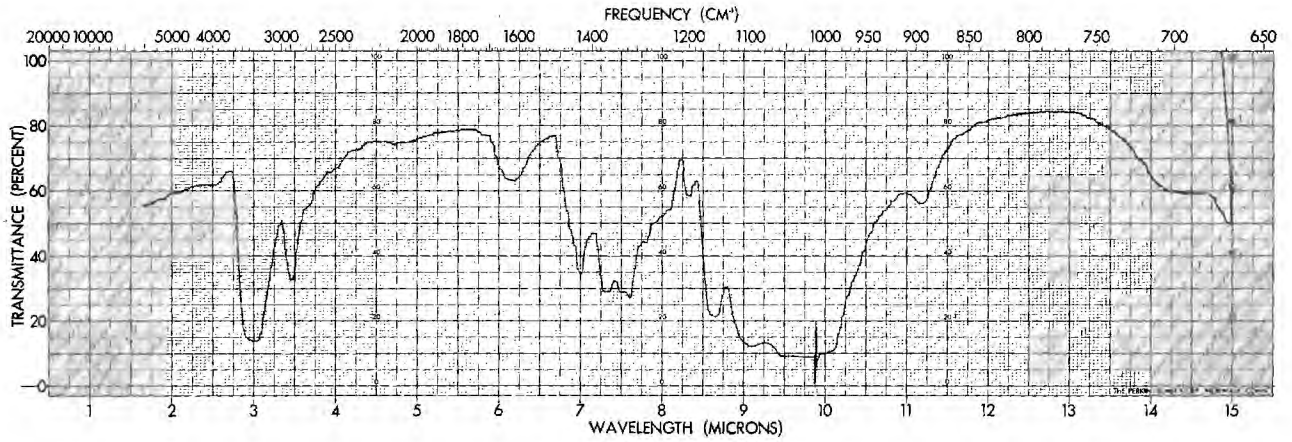
Sample	Fiber Press Spectrum	KBr Spectrum
Cotton	Figure 31A	Fig. 18A, Proj. A-843 Report #1
Cotton (De-waxed)	Figure 31B	Fig. 17B, Proj. A-843 Report #1
Viscose	Figure 31C	Fig. 17C, Proj. A-843 Report #1
Acetate	Figure 32A	Fig. 25C
Triacetate	Figure 32B	
Acrilan	Figure 32C	Fig. 26B

e. Summary

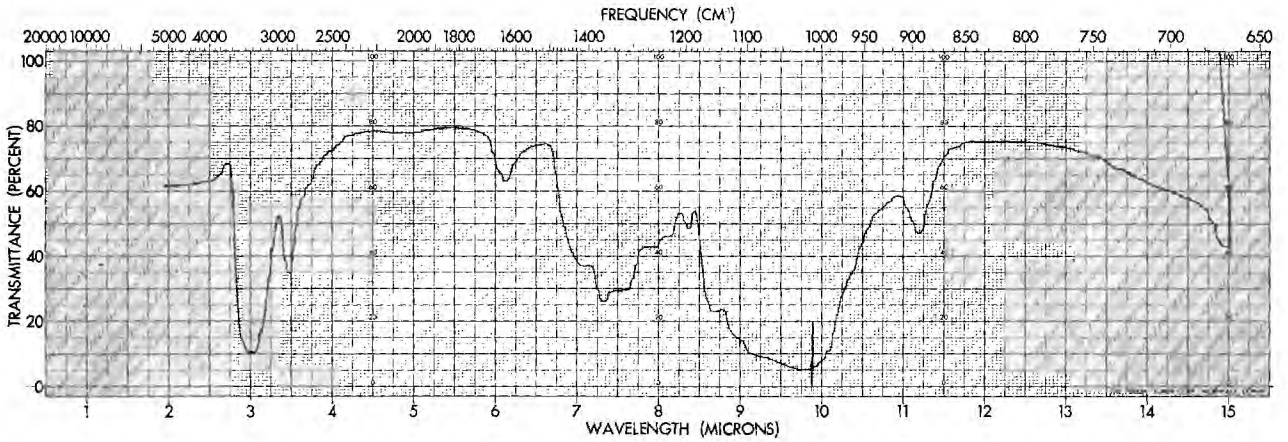
The fiber-press holder assembly described above allows direct spectral analysis of short staple fibers. Sample preparation involves only pressing the fibers under pressures comparable to those used in preparing KBr disks and thus should cause a minimum change, if any, in the sample's spectra or structure. Spectra obtained by this method compare favorably with those obtained using other techniques. The ease of sample preparation, the assurance of a spectrum of only the fiber, and the quality of the spectra obtained indicate that this technique may prove useful in many future studies.



A. COTTON

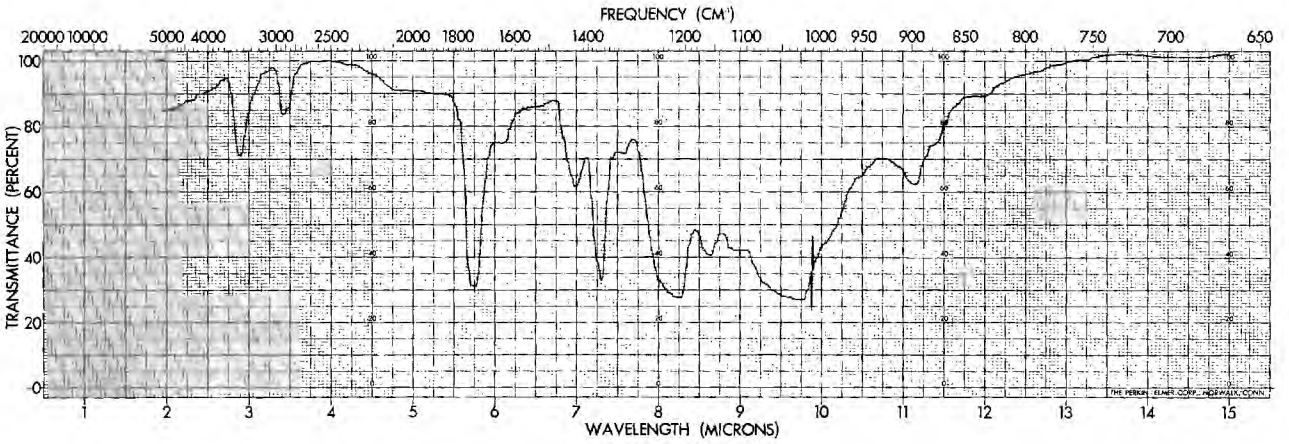


B. DEWAXED COTTON

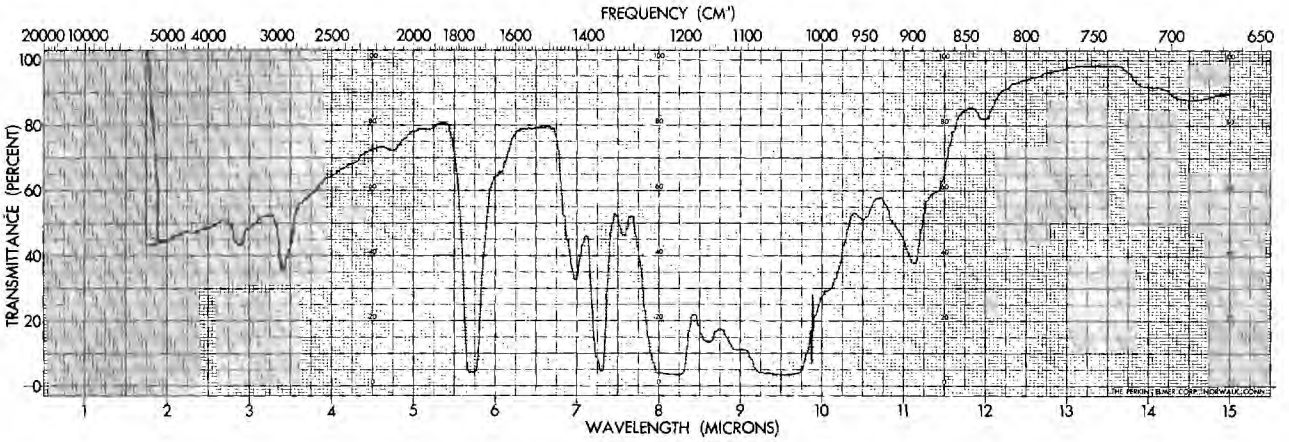


C. VISCOSE

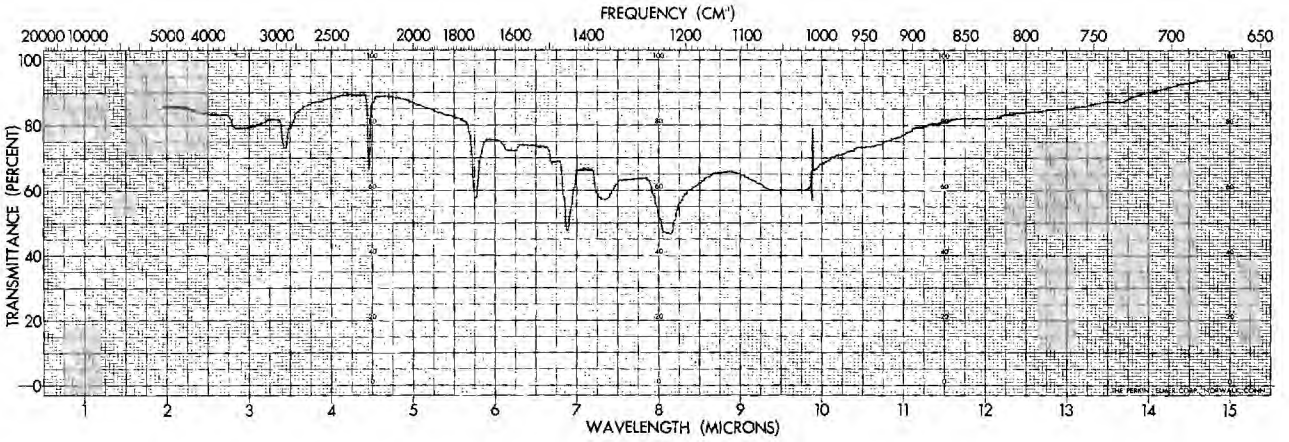
Figure 31. Infrared spectra obtained for pressed fiber specimens of: (A) cotton; (B) de-waxed cotton; (C) viscose.



A. ACETATE



B. TRIACETATE



C. ACRILAN

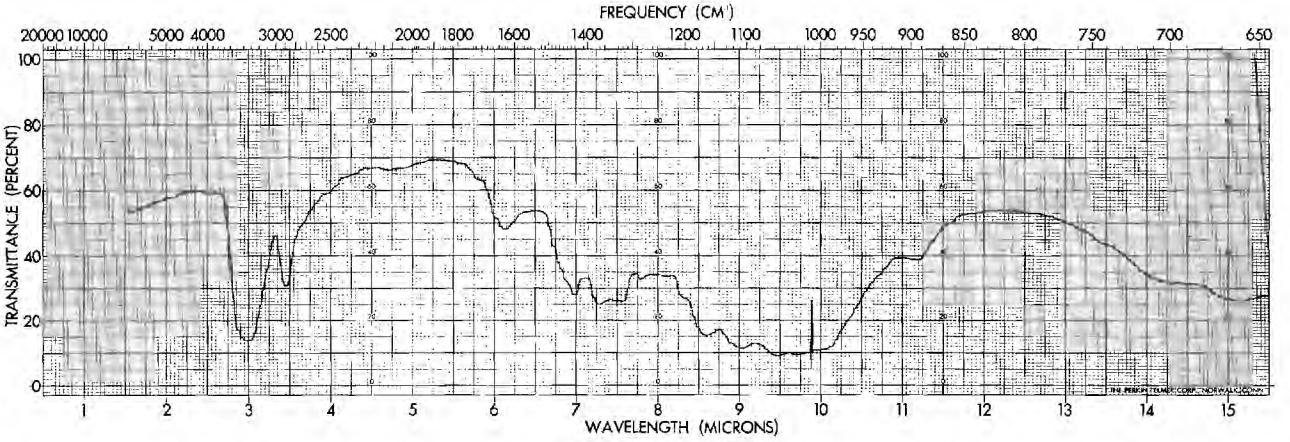
Figure 32. Infrared spectra obtained for pressed fiber specimens of: (A) Acetate Rayon; (B) Triacetate; (C) Acrilan.

4. Summary

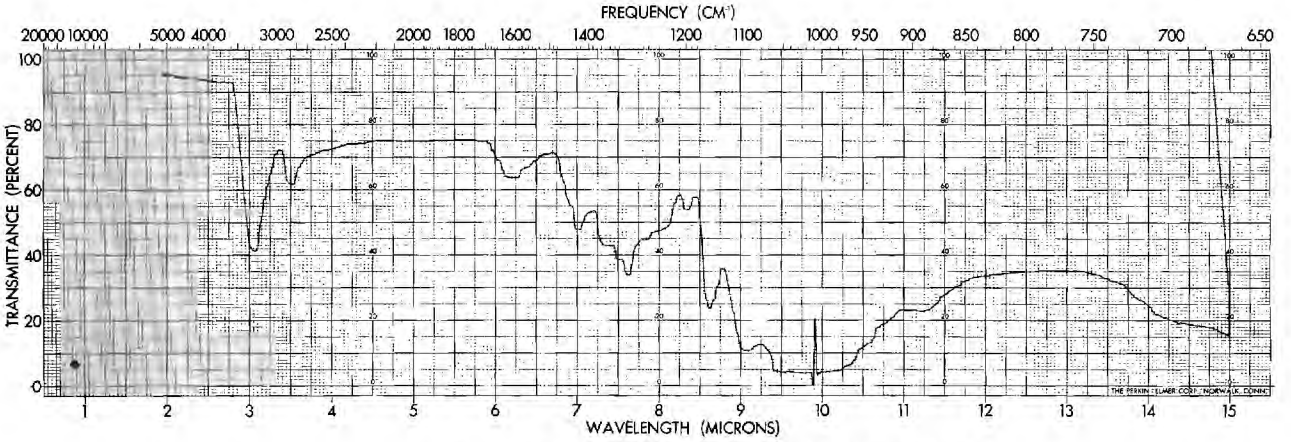
During the research period cotton wax extraction methods were further developed and room temperature extraction of the wax by chloroform and n-hexane, by the low pressure sohxlet method, were found to give high wax yields in reasonable time periods.

A Wilks doublebeam reflectance attachment for the Perkin-Elmer 1R 221 Spectrometer was obtained and techniques for preparing reproducible spectra were developed. Representative spectra of Empire WR cotton, cotton wax, dacron, acetate rayon, and acrilan were compared to transmission spectra. In general the spectra obtained are readily recognizable although reflectance spectra have been pointed out by Potts[12] to sometimes appear distorted as a result of changes in the index of refraction. These may cause the absorption bands to be broadened and intensified. Contamination of the reflectance crystal may also result in distortion of the 100 percent reflection line. Hence, use of these data are hindered for quantitative measurements without very careful interpretation and employment of appropriate correction factors.

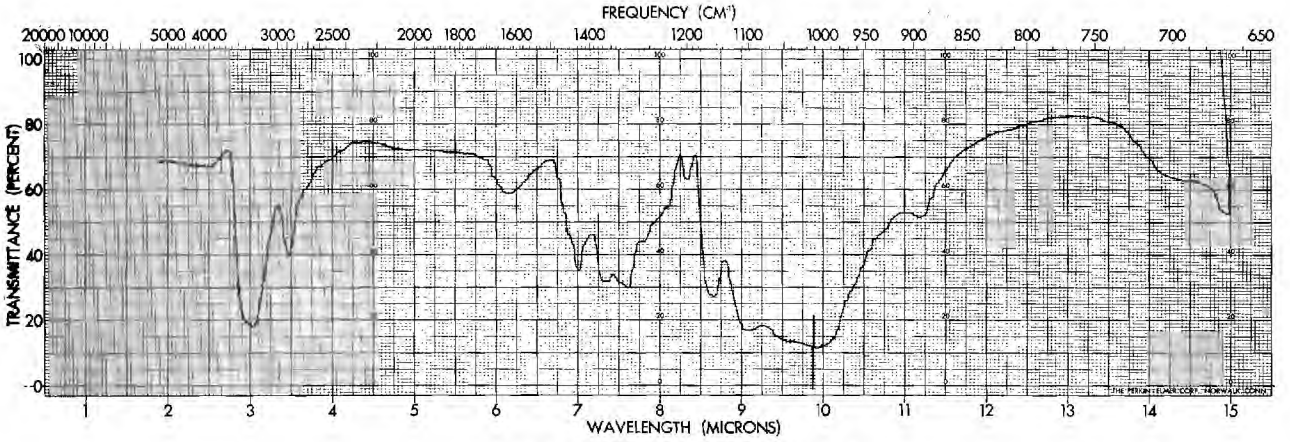
An apparatus has been developed for the pressing of a fiber arrangement directly into a specimen for infrared spectral examination. This technique has the advantage of using a series of lengths of whole fibers without maceration or pollution with a liquid or solid carrier. Representative spectra for Empire WR cotton, de-waxed cotton, viscose, acetate, triacetate, and acrilan have been presented. A comparison of cotton spectra obtained by the KBr transmission technique, by the reflectance method, and with a fiber press specimen are exhibited in Figure 33. As has been shown, spectra exhibit



A. KBr PELLET



B. WILKS REFLECTANCE ATTACHMENT



FIBER PRESS SPECIMENS

Figure 33. Comparison of infrared absorption spectra of Empire WR cotton obtained from KBr Pellet, reflectance, and fiber press specimens.

excellent discrimination and magnitude of absorption bands, and this instrument appears to be suitable for general adoption in infrared examination of fiber materials. This specimen also has the characteristic of the fibers being parallelly arranged, leading to an oriented arrangement which may present interesting polarized infrared information.

E. INVESTIGATIONS OF COTTON FIBERS BY X-RAY DIFFRACTION TECHNIQUES*

1. General

Research during the period has been principally directed toward a thorough search of the literature concerning the applications of x-ray diffraction techniques in the study of fibers and to the procurement, preparation, and preliminary operation of the necessary x-ray diffraction equipment and accessories.

2. Examination of the Literature

A search of the literature has been conducted, and some seventy papers were collected and filed. This collection is being continually revised and updated through checks on the more recent periodicals, and at present consists of over eighty papers. A study was made of each of these papers, and a comprehensive summary written on the theoretical as well as practical aspects of the application of x-rays to fiber research, as learned through the literature.** X-ray methods were found to provide a useful tool, and in many

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*Contributed by: H. W. Ellis, Student Assistant (Physics) and R. A. Young, Professor of Physics and Head of Diffraction Laboratories of the Engineering Experiment Station.

**The summary and its accompanying Bibliography are incorporated as Appendix B of this report.

cases have been singularly advantageous in exploring the microscopic structure of fibers, due to the sensitivity of the diffraction pattern to small changes in the fibers. In particular, the experimental findings of the authors indicated that much useful work could be done in the field of correlating x-ray data with physical parameters such as strength, water absorption capacity, crimp, and resistance to chemical action. There are few definitive works in correlating these quantities, though Dr. A. N. J. Heyn[13] has established and confirmed several times the relation between micelle orientation and tensile strength. (See Appendix B .) There is scattered evidence that other such relationships exist, and our work is directed toward establishing and finding ways to use these relationships to trace the effect of handling, storing, and treating cotton upon its structure, and thereby, upon its quality.

3. Apparatus

The apparatus used by us in this research includes a "Small Angle Scattering Apparatus" manufactured by the Rigaku-Denki Company, Tokyo, Japan. This instrument may be used with either a slit or pinhole optical system, and allows use of either counters or film as a means of x-ray detection. It is, at present, being used in conjunction with a GE x-ray tube with a copper anode. Also available for this work is a wide-angle diffractometer, which is at present used with a Philip's x-ray tube. The usual Hull-Debye-Sherrer and flat plate x-ray cameras are also available. Cotton fibers to be examined are to be taken principally from the specimen bale of Empire WR cotton described in Section IIIB of the 1st Semiannual Report.

4. Experimental Work

Although film has been employed for some qualitative results, in

order to record accurate quantitative data it is necessary to use a scintillation counter and an electronic counting system. Unfortunately, an existing shortage of equipment has prevented the installation of a much-needed automatic data-recording step-scan system. It has been necessary to use equipment employed by other groups on a temporary basis, during periods when it was not required by the specific group, and pending its recall. The continual changing of counting apparatus has made it impossible to quantitatively compare various sets of data. In lieu of possession of such a system, which could be employed twenty-four hours a day, data is being recorded manually at a much slower rate.

Recently, the temporary availability of an automatic data recording device made possible the discovery and elimination of a problem which had caused considerable difficulty. The particular x-ray tube which had been used in the experiments since the outset was found to emit radiation in an uneven pattern (non-uniform target illumination). The illumination pattern changed over periods of several hours, causing time variation in intensity which invalidated our data. Acquisition of a new x-ray source was required. The replacement tube has been obtained and is better, but it , too, is still suspect. Analysis of these difficulties continue while an attempt is being made to make some experimental progress through manual operations.

5. Summary

A literature search has been conducted to determine the methods of x-ray diffraction examination of fibers best suited to the current research on the frictional characteristics of cotton fibers. Correlations observed by others between x-ray diffraction studies of fibers and their physical properties

indicate the applicability of x-ray diffraction techniques to the problem. Necessary apparatus has been assembled and initial fiber diffraction patterns have been made. A calibration problem related to a non-uniform target illumination by the original x-ray tube was detected and corrected. However, unavailability of automatic data recorders for this work, except on an occasional basis, has impeded progress. Data is currently being obtained manually. Useful data will be obtained during the next period.

F. EFFECTS OF GINNING ON THE PROPERTIES OF COTTON FIBERS*

1. General

For more than 50 centuries cotton fiber was pulled from the seed by hand. Shortly after the advent of the gin for performing this task mechanically, about 1793, cotton became the principal textile fiber used by man. The purpose of the gin is to remove the cotton fiber from the trash and seed in the original seed cotton. It performs, in addition, the tasks of drying and compressing the cotton into bales.

During the ginning process the character of the cotton is somewhat altered. It is the purpose of this investigation to examine the changes in the cotton occurring as a result of ginning.

2. Survey of the Literature**

a. Introduction

Effects of ginning may be considered from the standpoint of

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*Contributed in part by Arthur M. Goldfarb, Graduate Assistant in Textile Engineering.

**See Bibliography, Appendix .

possible changes with regard to fiber length distribution, the percentage of broken fibers, fiber lengths and shape, fiber strength, moisture regain, the composition of the cuticle, and the crystal structure of the fiber. Changes in fiber friction, spinning character, feel, color and other less definable properties may also occur as a result of ginning. Current pertinent data in the literature are reported below.

b. Mechanical Effects of Ginning

Approximately 80 references have been examined to determine the parameters of which have been most affected by ginning. Lord, E.[14], has investigated (1959-1960) the change in fiber length distribution for cotton ginned by a roller gin at Kiraka in the Mengo area of Uganda. The incidence of whole, once and twice broken fibers was also determined. It was found that the mean length of the Uganda cotton was reduced from 29.0/32 to 24.9/32 giving a value of $\frac{\text{L ginned}}{\text{L unginned}} = 0.86$. Fiber breakage incidence was recorded for 300 fibers examined as:

no breakage	73%
tip but broken base	14%
base but broken tip	10%
broken base and tip	3%

Hence breakage occurred in 27% of the fibers.

Forces required for the removal of fibers from the seed were also measured. These were found to be in the range 0.1 gram to 3.5 grams. Breaking loads on the other hand were found to be in the range 0.1 gram to 7.0 grams over a 15 mm span at a loading rate of 90 grams/tex/minute. It is pointed out that the removal force overlaps the breaking force for approximately 109 of 150 fibers (73%) and it is stated that approximately 16% of the fibers were broken during removal from the seed leaving a basal part attached; a

slightly higher percentage of the remaining basal parts broke again on removal. Prakash and Iyengar[15] reported that the work required to remove a fiber from the seed was in the range 50 to 100 ergs.

Lord also states that there has been a strong complaint that short fiber content of United States Cotton has increased in recent years and that this has been ascribed by many as weakening of the fiber in hot air driers used to remove moisture from immature and damp cotton. Fiber strength measurements did not indicate a weakening, and he suggests rather that the bonding strength of the cotton to the seed may be increased by the heat cycle leading to an increase in breakage of the fiber during its removal from the seed. However, no measurements were reported to substantiate an increased bonding strength of the fiber to the seed.

c. Effects of Gin Drying Temperature

In addition to the effect suggested by Lord that the lint may be bonded more firmly to the seed during drying, other effects have been determined. Leitgeb and Wakeham[16] confirm the statement of Lord that the fiber strength is not appreciably changed by drying but that length distribution does change as was also reported by Waters and Phillips[17].

Berriman[18] reported that temperatures exceeding the range 230° F to 250° F changed irreversibly the fiber shape and the number of convolutions per unit length. Temperatures below this could change shape but the change was reversible on cooling.

Moisture regain rate was reported by Nelson[19] to decrease as a result of gin drying. This was reduced to less than 1/3 the amount of unheated cotton for the same time and to 1/10 that of unheated cotton for cotton flash heated to 322° F for 60 seconds.

Fineness was found essentially unchanged by Nelson et al.,[19] and Leitgeb and Wakeham[16]. On the other hand fiber density was reported to undergo a slight increase, and crystallinity as measured by x-ray diffraction was found essentially unaffected although a small change in crystal orientation was observed by Nelson et al.[19]

Berriman[20] reported changes in the character of the wax cuticle as a result of excessive temperatures.

Other effects noted were principally related to yarn strength properties which were reported to suffer when the cotton was subjected to excessive gin drier temperatures[17, 21, 22, 23, 24]. This effect has been recorded and reported by many observers by plotting gin drier temperatures versus ends-down per thousand spindle hours, skein breaking strength, single strand breaking strength, ends-down in weaving per unit time, and similarly fiber dependent parameters. However, changes in specific fiber properties, apparently responsible for the overall weakening, were not identified.

d. Miscellaneous Information

Other items of interest concerning ginning are that for a cotton variety of the types being studied a normal weight of about 1500 pounds of seed cotton produces 500 pounds of fiber, for one bale, and 1000 pounds of seed. The seed in turn may be subdivided into 92 pounds of linters (very fine short fibers), 157 pounds of oil, 450 pounds of cotton seed meal (cake), 233 pounds of hulls and 68 pounds of waste. The seed value at the gin, which usually buys it, runs in the vicinity of 1/6 the value of the cotton (1954). The linters are used in stuffing mattresses, for coarse yarns, for paper products, and for manufacture of explosives;

the oil is used in cooking oils, margarines, and other related areas; the cake and hulls are used principally in high protein feeds for livestock[25].

e. Summary

The preceding paragraphs have confirmed the fact that the process of ginning may change fiber length distribution, principally by breakage of the fibers, and that gin drying temperatures may affect fiber shape, density, moisture rate regain, orientation of the crystallites and the wax cuticle. Some of these properties of fibers or others, that may be deleteriously affected, also affect the properties of yarn spun from fibers heated excessively during gin drying (above 230° - 250° F) with the result that yarn spun from the cotton is relatively weak and subject to excessive breakage and ends-down during subsequent processing.

3. Experimental Work

During November 1965, 10 pound specimens of the variously harvested cotton varieties, Empire WR, Dixie King, and Carolina Queen, were obtained before and after ginning from each of one or two of three gins visited. These gins were within 25 miles of Experiment, Georgia. The types of cotton, the place of origin, and details of harvesting or processing are listed in Table 6.

Specimens of each cotton before and after ginning are illustrated in Figure 34. Enlargements of the detail of the cleanest and dirtiest cottons (Dixie King and Carolina Queen respectively) before and after ginning, are shown in Figures 35 and 36, and a typical commercially ginned Empire WR specimen

TABLE 6

DETAILS OF SEED AND GINNED COTTON

OBTAINED NEAR EXPERIMENT, GEORGIA, November 1965

1. Empire WR
   a. mechanically harvested
   b. obtained from Georgia Experiment Station, Experiment Georgia
   c. stock ginned on a model saw gin (no heaters or lint cleaners)

2. Empire WR
   a. mechanically harvested
   b. obtained from M. M. Brown Company, Locust Grove, Georgia
   c. ginning equipment - 1 lint cleaner, 1 dryer
      (1) stock heated to 180°F

3. Dixie King
   a. mechanically harvested
   b. obtained from M. M. Brown Company, Locust Grove, Georgia
   c. ginning equipment - 1 lint cleaner, 1 dryer
      (1) stock heated to 180°F

4. Carolina Queen
   a. hand harvested
   b. obtained from Redwine Brothers, Tyrone, Georgia
   c. ginning equipment - 1 tower dryer, 1 lint cleaner
      (1) stock heated to 290°F

5. Carolina Queen
   a. mechanically harvested
   b. obtained from Redwine Brothers, Tyrone, Georgia
   c. ginning equipment - (see above)
      (1) stock heated to 320°F

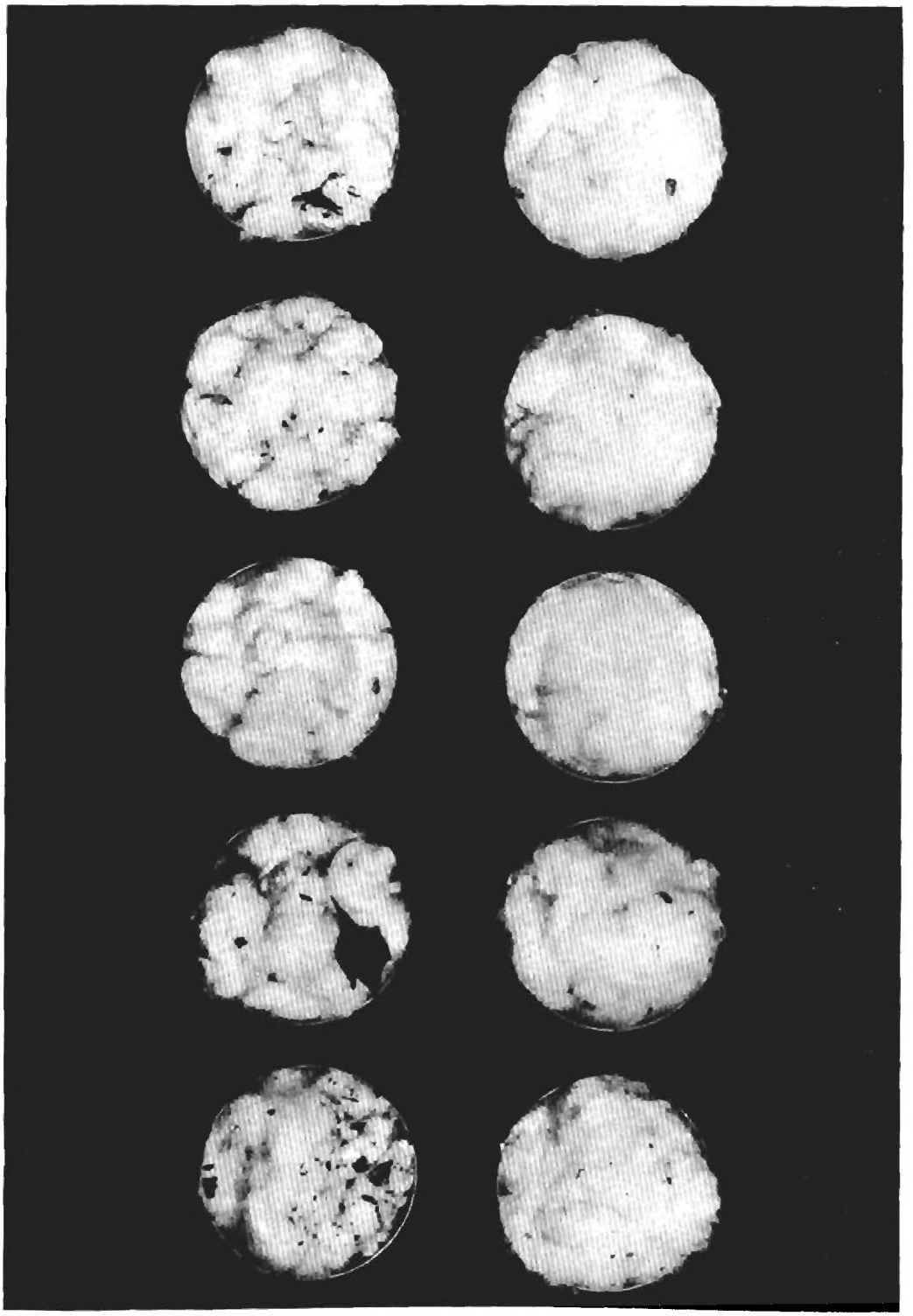

Figure 34. Specimens of hand and mechanically harvested cottons before and after ginning near Experiment, Georgia: (A) Empire WR mechanically harvested, model saw ginned; (B) Empire WR, mechanically harvested, saw ginned; (C) Carolina Queen, hand harvested, saw ginned; (D) Carolina Queen, mechanically harvested, saw ginned; (E) Dixie King, mechanically harvested, saw ginned.

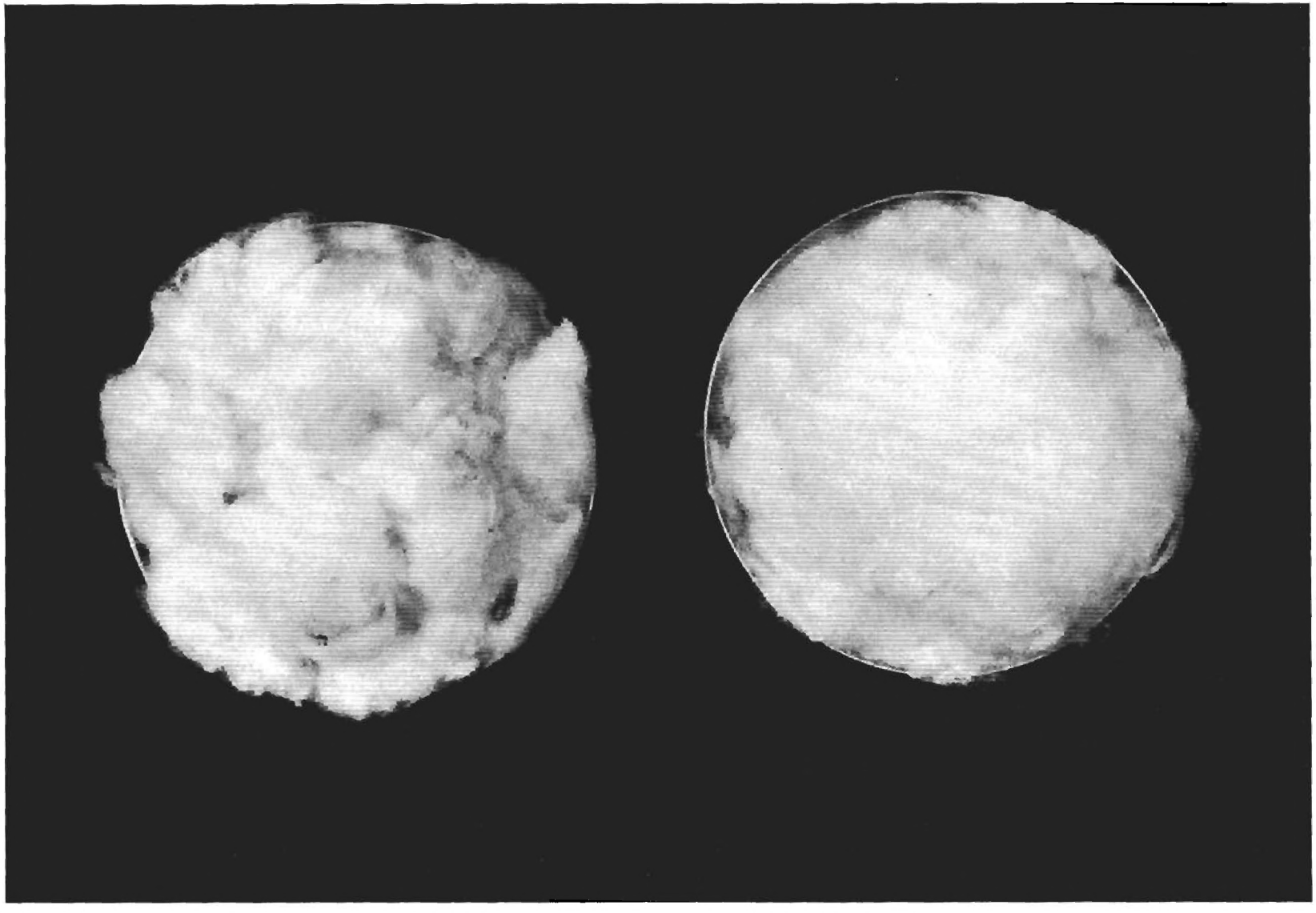

Figure 35. Specimen of hand harvested Carolina Queen cotton saw ginned at Tyrone, Georgia. This was cleanest specimen before and after ginning.

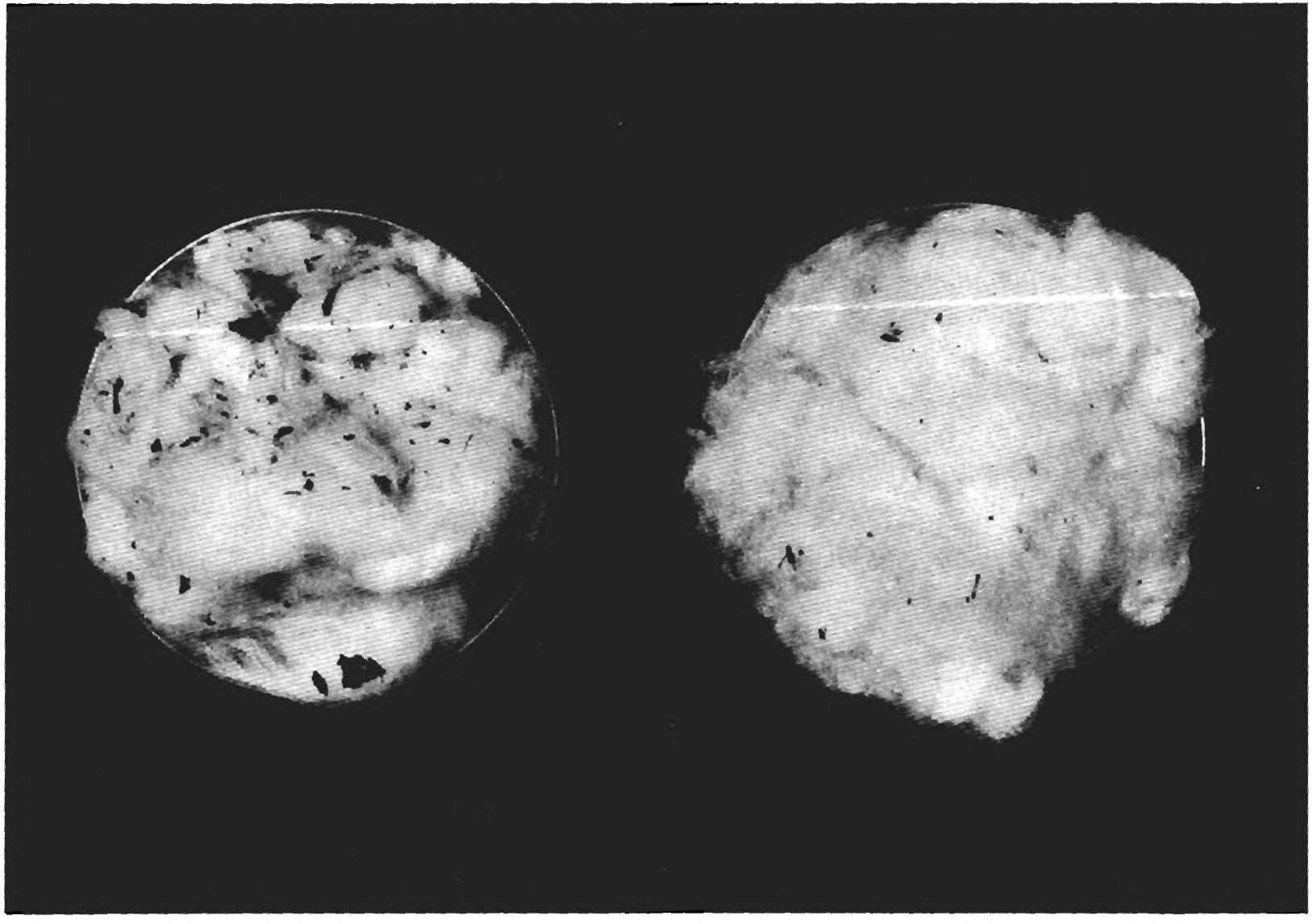

Figure 36. Specimen of mechanically harvested Dixie King cotton saw ginned at Locust Grove Georgia. This was trashiest specimen before and after ginning.

is shown in Figure 37. It will be noted that the hand harvested specimen, Carolina Queen of Figure 35, has much less initial and final trash than any other specimen. The second best is the Empire WR, mechanically harvested and ginned at Locust Grove, Georgia. This is shown in Figure 37.

Specimens of each cotton were defibered by hand for comparison with the ginned specimens. Even the linters were removed, which may affect the results slightly, as this does not occur in ginning. The weight of cotton versus that of the seed was not obtained although it is assumed that it would run about the normal ratio of 1 cotton/2 seed.

Attempts were made to run fiber length classifications on the Suter Webb sorter, but unreproducible results were obtained. Examination of the instrument revealed a need for new combs, and necessary repairs are in course.

Measurements of fiber length distribution were then made on the Fibrograph and the resultant data are illustrated in Figures 38, 39, and 40. Data taken from the graphs are listed in Table 7 for comparison purposes. Percent change for the various parameters measured are indicated in the section at the bottom of the table. Reductions in mean length of 10 to 14 percent are indicated as a result of ginning. The fiber from the Locust Grove gin, processed at 180° F, is very similar for both Empire WR and Dixie King fibers although the Dixie King was somewhat better as seed cotton and therefore suffered a slightly greater change. The Empire WR ginned at Experiment, Georgia possessed a little longer fiber initially and ended up a little longer than the others even though it suffered as much or slightly more reduction in fiber length than the cottons processed in the Commercial gin at Locust Grove, Georgia.

TABLE 7

Comparison of Data on Fiber Length Distribution of Seed and Ginned Cotton from three Sources.

Seed Cotton

	Empire WR Experiment, Ga. (inches)	Empire WR Locust Grove (inches)	Dixie King Locust Grove (inches)
66.7% Span Length	0.434	0.334	0.395
50.0% Span Length	0.611	0.506	0.521
2.5% Span Length	1.224	1.102	1.136
Mean Length	1.02	0.85	0.89
U.H. Mean Length	1.23	1.05	1.12

Ginned Cotton

	(inches)	(inches)	(inches)
66.7% Span Length	0.405	0.343	0.346
50.0% Span Length	0.535	0.462	0.459
2.5% Span Length	1.141	1.068	1.076
Mean Length	0.91	0.77	0.77
U.H. Mean Length	1.12	1.01	1.04

Per Cent Length of Ginned Cotton Versus Seed Cotton

	%	%	%
66.7% Span Length	93.3	102.0	87.5
50.0% Span Length	87.5	91.4	88.0
2.5% Span Length	93.3	97.3	94.8
Mean Length	89.3	90.3	86.3
U.H. Mean Length	91.1	96.3	93.0

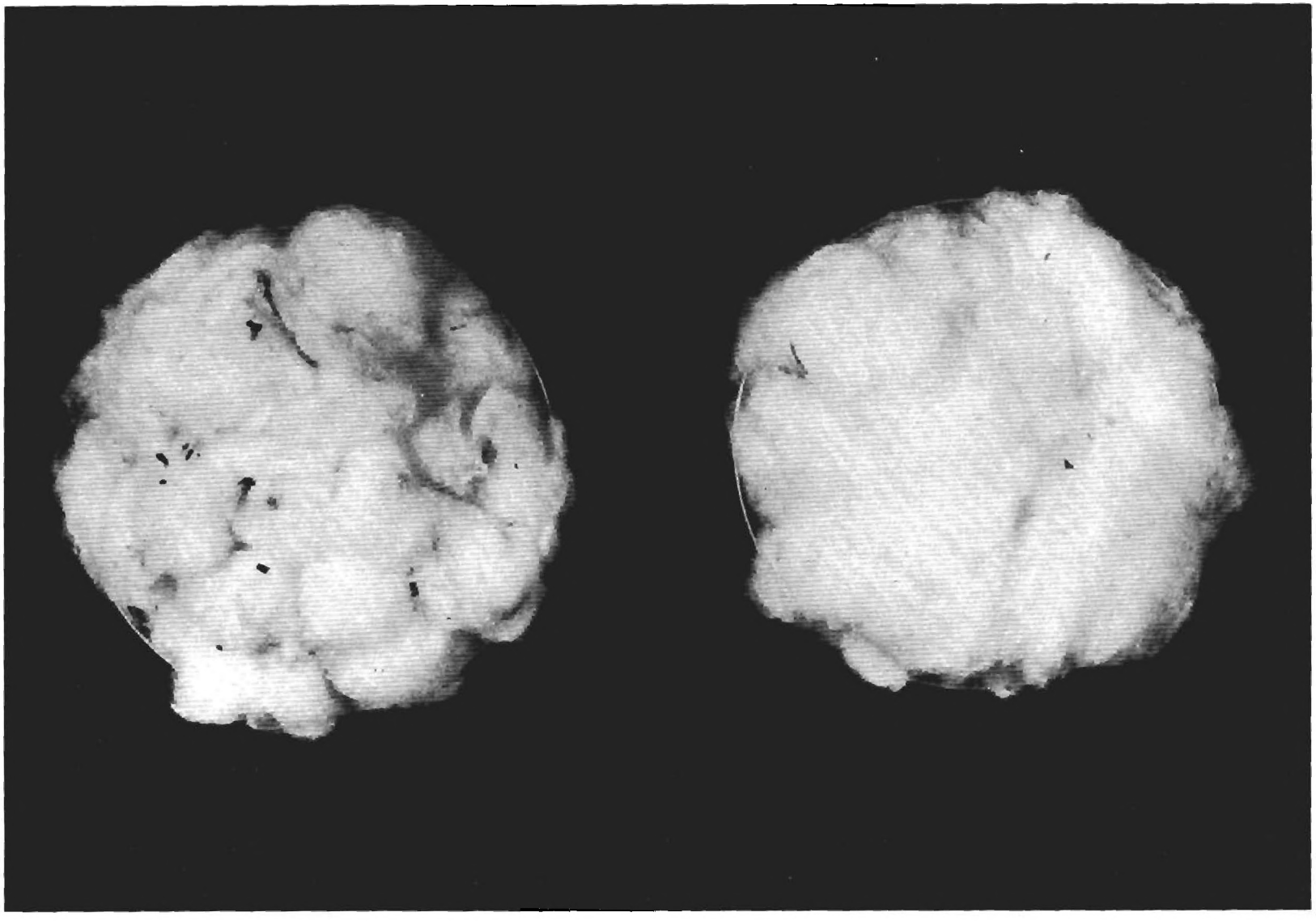

Figure 37. Specimen of mechanically harvested Empire WR cotton saw ginned at Locust Grove Georgia. This was second cleanest cotton and is the species being examined in the current work (hand harvested, however).

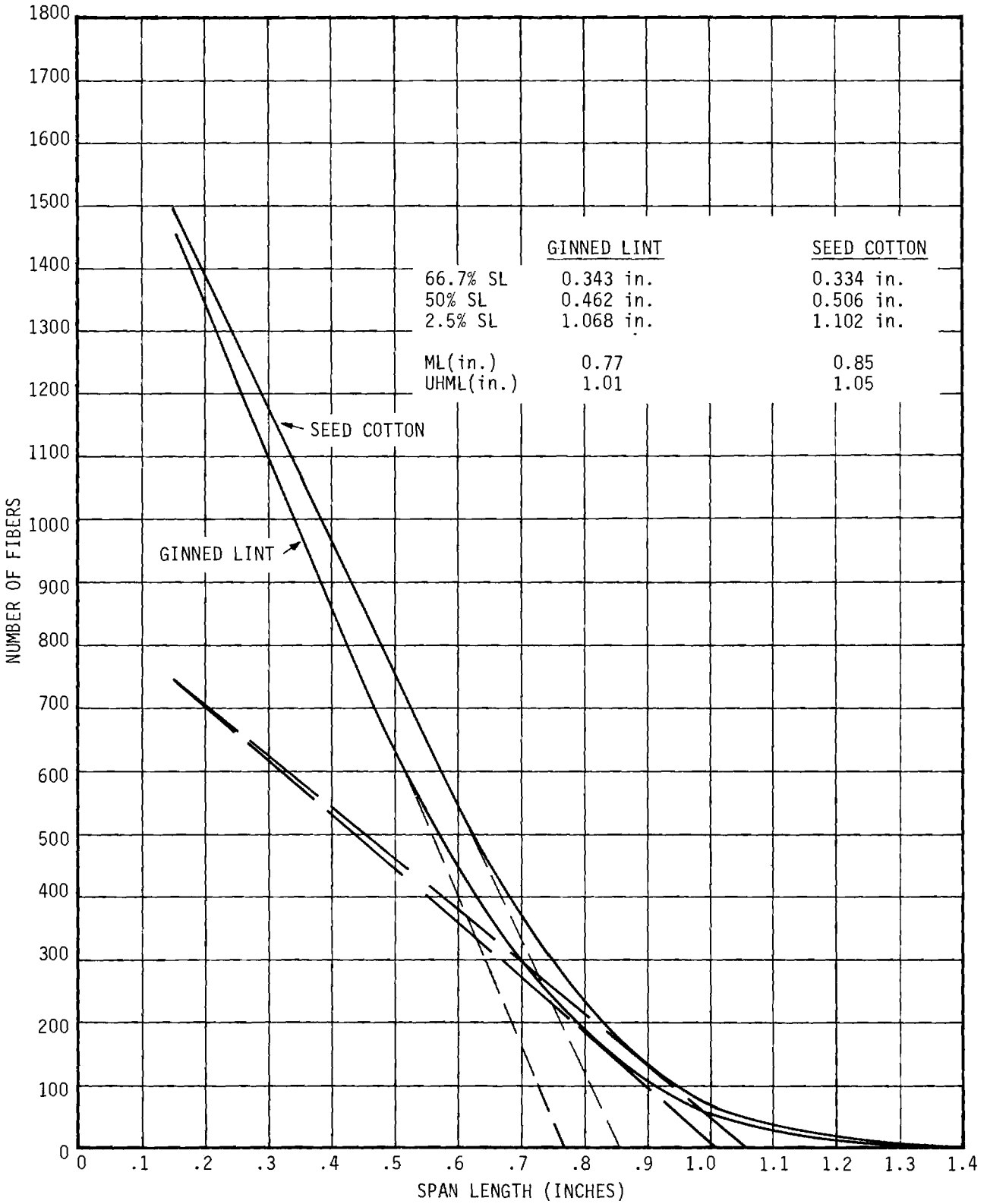

Figure 38. Comparison of Digital Fibrograph fiber-length distribution data for Empire WR cotton from the gin at Locust Grove, Georgia, before and after ginning.

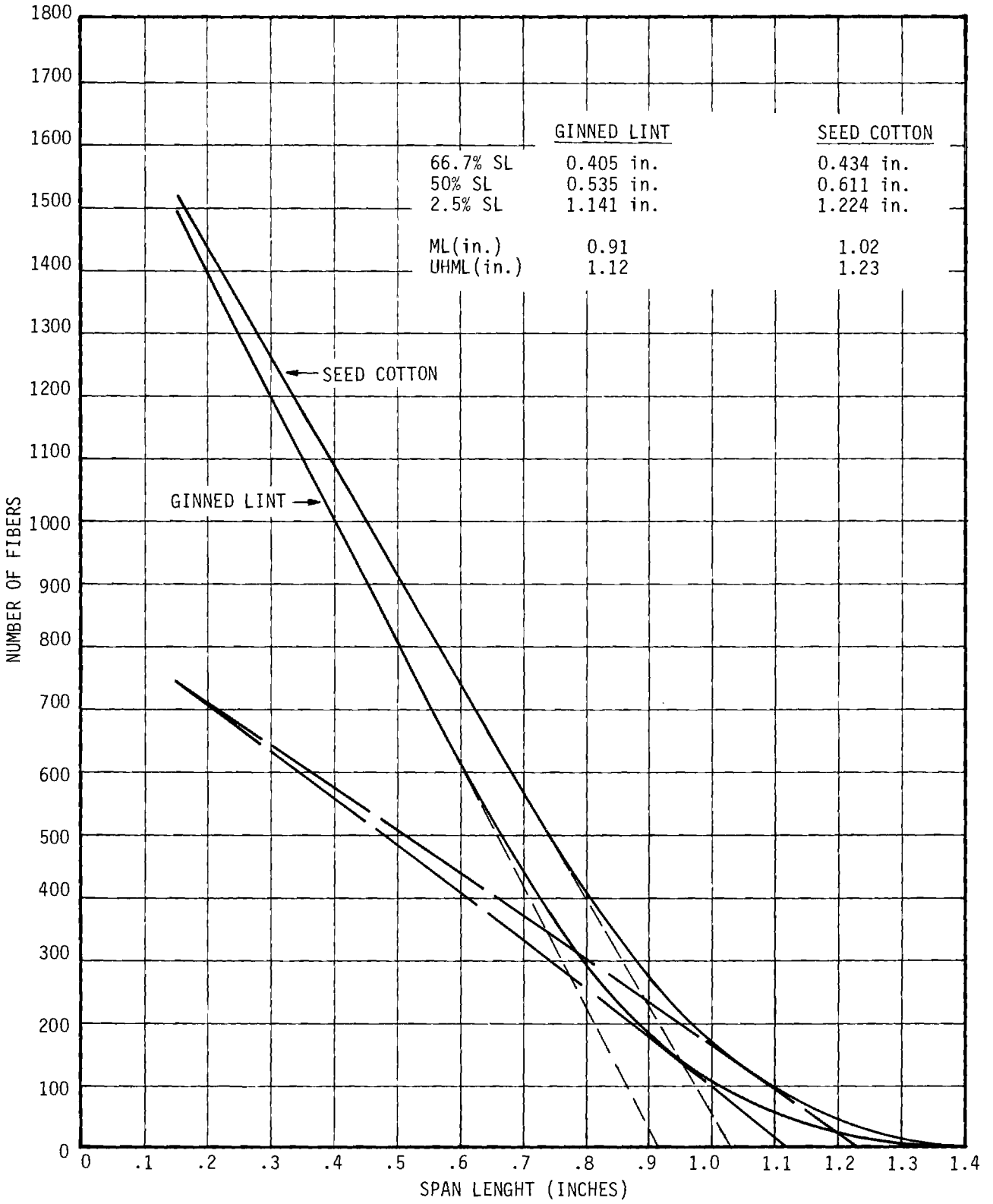

Figure 39. Comparison of Digital Fibrograph fiber-length distribution data for Empire WR cotton from the gin at Experiment, Georgia, before and after ginning.

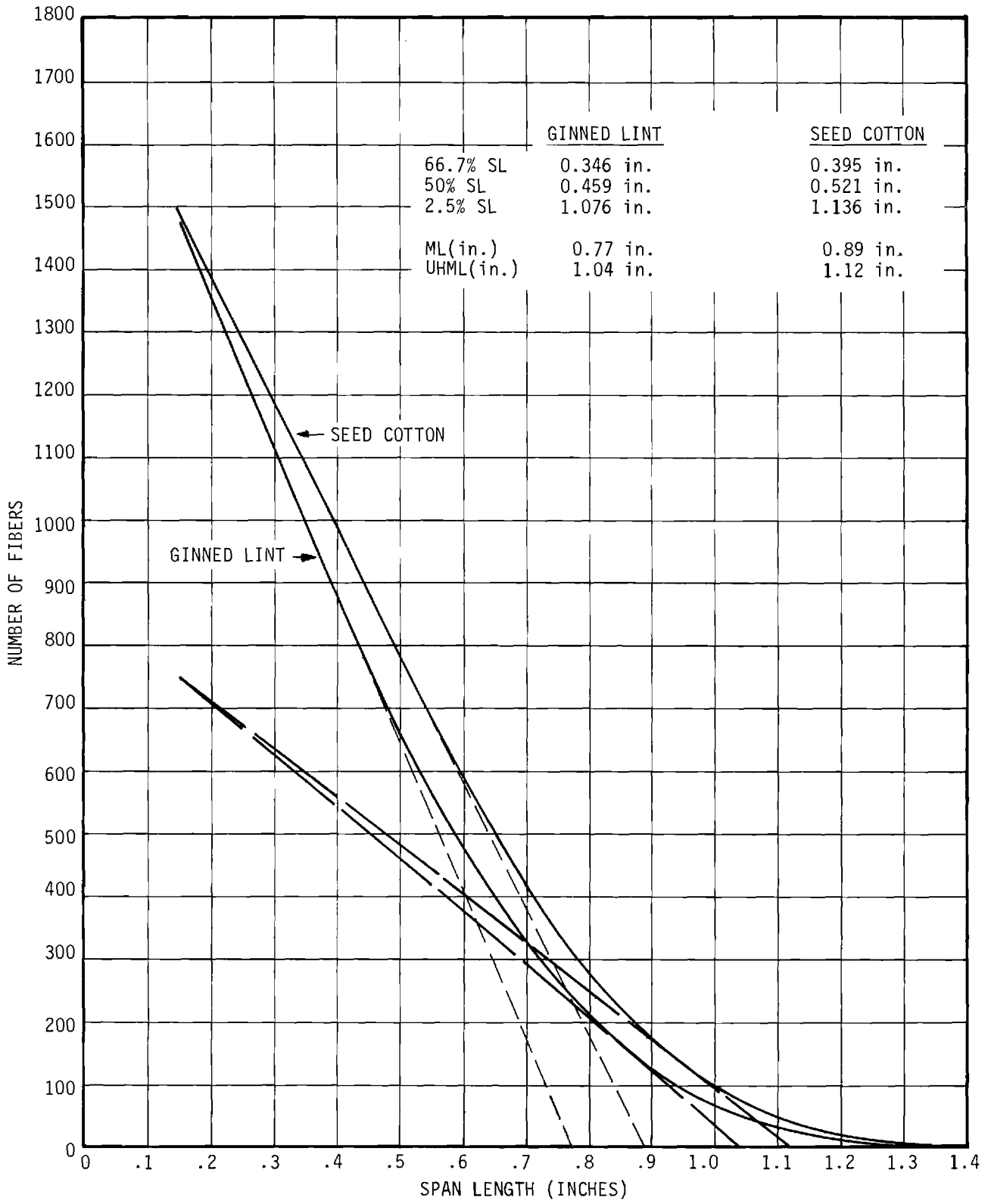

Figure 40. Comparison of Digital Fibrograph fiber-length distribution data for Dixie King cotton from the gin at Locust Grove, Georgia, before and after ginning.

4. Summary

The literature concerning the effects of ginning on the cotton fiber reports that fiber length distribution of the fiber of seed cotton removed by hand is changed by ginning, resulting in a reduction of mean length and an increase in the number of broken fibers. Temperatures employed in fiber drying frequently result in changes in shapes of the fibers (numbers of convolutions per unit length and length) and a reduction in rate of moisture regain. Changes in the wax cuticle have also been reported as well as increased in orientation of the crystallites of the fibers. Much literature reports weakening of yarns spun from fiber heated to high temperatures during ginning ($> 240^{\circ}F$). However the strength of the fiber itself has not been reported to suffer appreciably.

Cotton specimens of Empire WR, Dixie King and Carolina Queen were obtained from one or more of three gins and fiber cleanliness and length distribution before and after ginning have been examined. Mean length of ginned specimens (as measured by the Fibrograph) were found to be reduced by 10 to 14 percent, in reasonable agreement with data reported in the literature.

IV. CONCLUSIONS AND RECOMMENDATIONS

The research program on the frictional properties of cotton fibers progressed favorably with respect to optical, frictional measurement, infrared and x-ray diffraction investigations. Unexpected difficulties were encountered, however, in constructing friction measuring instruments which would reproduce equivalent measurements on similar fibers. An investigation of the effects of ginning on cotton was begun and is now in course.

Sectioning techniques for fibers have been developed and photomicrographs have furnished data for size and shape analyses. The median fiber width was found to be 2.8 times its thickness for 1" cotton fibers. A second factor describing the ratio of the sum of the major and minor axes to four times the wall thickness was found to give a median value of 2.0. Electron micrographs of replicas of 3/4", 1", and 1-1/4" fibers displayed rough surface features, banded zones, and reversals. Surfaces topography varied considerably along a single fiber; no features, however, were found to be particular to a single length.

Modification of the frictional measuring instrument, a better normal force measurement, and construction of two new frictional arms led to measurements of μ_k and μ_s for cotton and nylon below those previously obtained. The moment of inertia of the two new arms was low and appeared partly responsible for the lower values obtained with them. Fiber tension appears also to be critical. However, in most measurements made here, initially and in the second period, the tension has been maintained at 500 mg. None of the arms now available appear to be capable of giving reliable measurements of μ_s or μ_k under 15 mg although this appears to be a very

interesting part of the data curve. Some measurements made at normal forces of 5 to 8 mg indicate very different behavior of cotton and nylon in this region. With the modified instrument values for the coefficient of friction for 1-1/4" Empire WR cotton were found to be about 0.25 for μ_k and 0.45 for μ_s. For 15 denier nylon monofilament these values were 0.29 and 0.46 respectively. Ratios of μ_s/μ_k were 1.8 and 1.6 respectively. Investigations of reports by other investigators revealed no data directly applicable to our problem although the μ_k of viscose was reported as lower. Indications of incorrect data evaluations were also noted. In order to be able to operate at normal forces less than 20 mg an electromagnetically operated frictional arm is planned. This will have the advantage of being self-damping.

The Wilks infrared reflectance attachment was evaluated and found to be a very useful attachment. Reflectance spectra were obtained for Empire WR cotton, cotton wax, acetate rayon, dacron, and acrilan and compared to these obtained by transmission methods. Discrimination was excellent and amplitudes were large. Qualitative similarities are readily apparent; however, experienced investigators have counseled caution in quantitative interpretations because of effects related to index of refraction changes occurring under reflection conditions. A fiber press was developed which allows pressing of sections of whole fiber, parallelly arranged, into a specimen suitable for direct infrared transmission studies. This specimen has high purity and is prepared without maceration or pollution. In addition it possesses orientation features which may be of great interest for investigation with polarized optical or infrared radiation. Spectra have been prepared for Empire WR cotton, de-waxed cotton, viscose, acetate, triacetate, and acrilan. Spectra reveal excellent discrimination and amplitude. The fiber

press appears suitable for general adoption in the infrared examination of fiber materials.

X-ray investigations have progressed through the literature search, equipment assembly, and shakedown stages. Data useful in analyses of fibers can now be expected.

Specimens of Empire WR, Carolina Queen, and Dixie King cotton were procured before and after ginning. Effects of ginning on the fiber are being studied. Preliminary data on fiber length distribution has shown reductions of the mean length to be in the range 10 to 14 percent and in the 2.5 percent span length 3 to 7 percent.

V. PROGRAM FOR THE NEXT PERIOD

Work during the next period will be principally a continuation of the work outlined in this report. In particular, a friction arm allowing application of the normal force by electromagnetic means is expected to limit varying normal force due to oscillation of the arm and to allow investigation of the region of low normal forces.

The infrared techniques have been developed to the stage they can now be applied directly to the investigations of differences between differently processed fibers if these are distinguishable. They will be so applied.

The x-ray diffraction techniques will be applied to fiber structure analyses. Fiber press specimens from the infrared technique will be examined for possible usefulness as x-ray diffraction specimens.

Investigations of the effects on cotton fiber of ginning and carding will be completed.

Five theses for the M.S. Degree in Textile Engineering will be completed covering facets of the program.

VI. PERSONNEL

The individuals employed on this research during the period of this report are listed below.

Individual	Title	Area of Research
Richard B. Belser	Research Associate Professor	Project Director
James L. Taylor	A French Textile School, Director	Associate Project Director
William L. Hyden	Professor, Textile Engineer	Associate Project Director
John L. Brown	Director, Analytical Instrumentation Laboratories	Optical and Electron Microscopy
James L. Hubbard	Assistant Research Physicist	Optical and Electron Microscopy
James A. Knight	Research Professor, Chemistry Head, Radioisotopes Laboratory	Infrared Spectroscopy
Rick A. Porter*	Graduate Assistant (Textiles and Chemistry)	Infrared Spectroscopy
Marvin P. Smoak	Student Assistant (Physics)	Infrared Spectroscopy
R. A. Young	Diffraction Laboratories, Director	X-ray Diffraction
Harry W. Ellis	Student Assistant (Physics)	X-ray Diffraction
Thomas E. McBride**	Graduate Assistant (Textile School)	Friction Apparatus and Fiber Microscopy
Lester D. Dozier	Assistant Research Scientist (Mechanical Engineer)	Friction Apparatus

*Porter is completing a thesis in dye chemistry and completed his work period under the project September 20, 1965.

**McBride completed the M.S. Degree in Textile Engineering August 31, 1965 and is now employed at Fiber Industries, Inc., Greenville, South Carolina.

Individual	Title	Area of Research
James P. Bryant	Graduate Assistant (Textile School)	Frictional Properties of Cotton Fibers
Arthur M. Goldfarb	Graduate Assistant (Textile School)	Effects of Ginning on Properties of Cotton Fibers
W. Eudon Kirkland	Graduate Assistant (Textile School)	Infrared Investigations of Cotton Fibers
Donald L. House	Graduate Assistant (Textile School)	Optical and Electron Microscopy of Cotton Fibers
Howard R. Levy	Graduate Assistant (Textile School)	Effects of Opening and Carding on Cotton Fibers

In general, all work was performed on a part-time basis except that of Mr. Dozier who is employed on a full time basis to give continuity to the fiber friction measurement program.

REFERENCES

1. J. M. Dallavalla, "Micromeritics," Pitman Publishing Corp., New York-London, pp. 46-57 (1943-1948).

2. W. J. Lyons and S. C. Scheier, "Velocity Dependence during the Stick-Slip Process on the Surface Friction of Fibrous Polymers," Journal of Applied Physics 36, 2020-2023 (1965).

3. N. Gralen and B. Olafsson, "Measurement of Friction Between Single Fibers," Textile Research Journal 17, 488-496 (1947).

4. J. C. Guthrie and P. H. Oliver, "Inter-Fibre Friction of Viscose Rayon," Journal of the Textile Institute 43, T579-T594 (1952).

5. S. C. Scheier and W. J. Lyons, "Measurement of the Surface Friction of Fibers by an Electro-Mechanical Method," Textile Research Journal 35, 385-394 (1965).

6. J. H. Kettering, "Solvent Extraction of Cotton Wax," Textile Research Journal 16, 627 (1946).

7. Instruction Manual, Wilks Model 12 Double Beam Multiple Internal Reflectance Attachment, Wilks Scientific Corp., South Norwalk, Conn.

8. P. A. Wilks, "Instrumental Considerations for the Application of ATR to the Analysis of Coatings," Proc. of the Amer. Chem. Soc., Div. of Organic Coatings and Plastics Chem. 25, No. 2, 357 (1965).

9. W. J. Potts, "ATR: Uses and Abuses," Proc. of the Amer. Chem. Soc., Div. of Organic Coatings and Plastics Chem. 25, No. 2, 364 (1965).

10. Zhbonkou, "I. R. Spectra of Cellulose," Vesti. Akad. Novak Belaruskai S.S.R. Seryya Fizika-Tekh. Navuk, No. 1 (1956); see Chem. Abstract Vol. 51, 3989.

11. S. Burgess, "Sample Preparation of Fibers for Identification by I. R. Spectroscopy," Chem. & Ind., London, p. 1161 ff (1961).

12. W. J. Potts, Chemical Infrared Spectroscopy, John Wiley and Sons, Inc., New York, N. Y., p. 204 ff (1963).

13. A. N. J. Heyn, "Small-Angle X-ray Scattering by Cellulose Fibers--Experimental Study of the Orientation Factor in Model Filaments and Rayons," J. Am. Chem. Soc. 72, 2284-5 (1950).

14. E. Lord, "Some Observations on Fiber Breakage in the Ginning of Cotton," Text. Inst. J., Trans., T69-T78 (1963).

15. Jai Prakash and R. L. N. Iyengar, "Work Done in Pulling Fibers from the Cotton Seed Under Impact Conditions in Relation to Fiber Properties and Ginning Behavior," Text. Res. J. XXXIV, 444-55 (May, 1964).

16. D. J. Leitgeb and H. Wakeham, "Cotton Quality and Fiber Properties," Text. Res. J. XXIV, 1047-57 (Dec., 1954).

17. W. T. Waters and J. Phillips, "Who Killed Cotton Quality?" Text. Ind. 125, 56-62 (July, 1961).

18. L. P. Berriman, "Overdrying Cotton at Gin Can Change Fiber Shape," Textile Ind. 125, 25, 114 (Sept., 1961).

19. M. L. Nelson, et al., "Effects of Heat - Drying on the Physical and Chemical Properties of Cotton," Text. Res. J. XXIX, 260-9 (March, 1959).

20. L. P. Berriman, "Heat Induced Changes in Fiber Friction Characteristics," 15th Annual Cotton Research Clinic, Pinehurst, North Carolina, (February 4, 1964).

21. Anonymous

22. S. T. Burley, Jr., "A Method for Determining the Effects of Various Gin Treatments on the Spinnability of Cotton," Text. Res. J. XXIX, 696-99 (Sept., 1959).

23. J. L. Delany, "Effect of Heat and Ginning Operations on Spinning and Weaving," Text. Res. J. XXVII, 175-6 (Feb., 1957).

24. J. L. Delany, "Gins Cause Much of Our Trouble - Delany," Text. Ind. 121, 136-8 (Feb., 1957).

25. Encyclopedia Britannica 6, 584 (1962 edition).

APPENDIX

APPENDIX A

BIBLIOGRAPHY

1. INVESTIGATIONS OF COTTON FIBERS BY INFRARED TECHNIQUES

1. Fagot, C. C., "Infrared Identification of Synthetic Thermoplastic Fibers and Resins," Appl. Spectroscopy 19, No. 1, 30-31 (1965).

2. Fahrenfort, Spectrochemica Acta 17, 698-709 (1961).

3. Nelson and Mares, "Accessibility and Lateral Order Distribution of the Cellulose in the Developing Cotton Fiber," Textile Research J., p. 592 (July, 1965).

4. Novak, I. I. and Vettegre, V. I., "Effect of Orientation on the Crystallinity of Kapron," Vysokomol. Sold 62, No. 6, 1027-1029 (1965).

5. Patil, Dweltz and Radhakrishnan, "Studies on Decrystallization of Cotton," Textile Research J., p. 517, (June 1965).

6. Wilks, P. A. and Iszard, M. R., "The Identification of Fibers and Fabrics by Internal Reflectance Spectroscopy," Wilks Scientific Corp., (1964).

BIBLIOGRAPHY

2. INVESTIGATION OF THE EFFECTS OF GINNING ON COTTON FIBERS

1. Anderson, James H. et al. "The Effects of Drying and Ginning Practices on Cotton Fiber Properties and Spinning Performance," Technical Bulletin 1002, South Carolina Agricultural Station, 28 (May, 1961).

2. Andrews, F. R., et al. "Heating, Cleaning, and Mechanical Processing Effects on Cotton," Text. Res. J. XXXIII, 609-16 (August, 1963).

3. Anon. "Will Spinners Take Action on Gin-Damaged Cotton?", Textile Industries 118, 145-61 (Feb., 1954).

4. Berriman, Lester P. "Effects of Heat on the Quality of Cotton," Textile Bull. 88, 49-54 (March, 1962).

5. Berriman, Lester P. "Overdrying Cotton at Gin Can Change Fiber Shape," Textile Ind. 125, 25, 114 (Sept., 1961).

6. Burley, Samuel T., Jr. "A Method for Determining the Effects of Various Gin Treatments on the Spinnability of Cotton," Text. Res. J. XXIX, 696-99 (Sept. 1959).

7. Cardozier, V. R. Growing Cotton, 310-51 (New York, 1957).

8. Collings, Gilbeart H., The Production of Cotton, 144-54 (New York, 1926).

9. Delany, J. L. "Effect of Heat and Ginning Operations on Spinning and Weaving," Text. Res. J. XXVII, 175-6 (Feb., 1957).

10. Delany, J. L. "Gins Cause Much of Our Trouble - Delany," Text. Ind. 121, 136-8 (Feb., 1957).

11. Farquhar, Ruth L., et al. "Measuring Changes Produced by the Heating of Cotton Cellulose," Can. Text. J. 23, 51-6 (Feb. 10, 1956).

12. Grant, James N., et al. "Heating, Cleaning, and Mechanical Processing Effects on Cottons," Text. Res. J. XXXIII, 839-48 (Oct., 1963).

13. Hart, W. J., et al. "The Effect of an Excessive Gin-Drying Temperature of Cotton on Yarn Quality and Mill Processing," Text. Res. J. XXV, 415-21 (May, 1955).

14. Hessler, L. E. and H. Workman. "Heat Induced Chemical Changes in Cotton Fiber," Text. Res. J. XXIX, 487-92 (June, 1959).

15. Leitgeb, D. J. and H. Wakeham. "Cotton Quality and Fiber Properties," Text. Res. J. XXIV, 1047-57 (Dec., 1954).

16. Lord, E. "Some Observations on Fiber Breakage in the Ginning of Cotton," Text. Inst. J., Trans., T69-T78 (1963).

17. Nelson, Mary L., et al. "Effects of Heat-Drying on the Physical and Chemical Properties of Cotton," Text. Res. J. XXIX, 260-9 (March, 1959).

18. Orr, Rollin S., et al. "Degradation of Cotton Fibers and Yarns by Heat and Moisture," Text. Res. J. XXIV, 399-406 (May, 1954).

19. Prakash, Jai and R. L. N. Iyengar. "Work Done in Pulling Fibers from the Cotton Seed Under Impact Conditions in Relation to Fiber Properties and Ginning Behavior," Text. Res. J. XXXIV, 444-55 (May, 1964).

20. Waters, W. T. and J. Phillips. "Who Killed Cotton Quality?" Text. Ind. 125, 56-62 (July, 1961).

21. Wolfgang, William G. and Ruby. An Introduction to Textile Fibers, 12-21 (1960, n.p.).

APPENDIX B

1. SUMMARY OF LITERATURE SEARCH ON THE APPLICATION OF X-RAYS TO FIBERS--PARTICULARLY CELLULOSE

by Harry Ellis, Crystal Physics Branch

Introduction

The following is a summary of both the theoretical and practical aspects of the application of x-ray techniques to cotton fiber study, as found in the literature. Each section is preceded by a brief discussion of pertinent background material. Particular attention is given to the correlation of x-ray data with other physical properties of the fiber, as this is our major field of interest.

The application of x-rays to fibers is a relatively new field, and the bulk of the work that has been done has come since 1945. This work covers a very broad field encompassing both high and low angle scattering, exploring, respectively, submicroscopic and macroscopic qualities of the fibers studied. It will be advantageous to divide these explorations into various specific parts which will be dealt with singularly.

High Angle Scattering

Scope

There are four main qualities of fibers which may be determined by the wide angle Bragg scattering: (1) nature of the crystal structure--to some extent; (2) percentage of crystalline material; (3) average size of crystallites; and (4) degree of orientation. These are independent for natural materials (Ward, 1950) except in the extreme values. They are treated separately.

Nature of the Crystal Structure

In attempting to justify the term "lattice parameters" in connection with fibers, it can only be said that the existence of high angle Bragg reflections indicates some degree of long-range periodicity which was early assumed to be due to "crystallites" or "micelles" which were arranged along the fiber axis. The action of certain chemical reagents with these fibers had already suggested a structure partially crystalline and partially amorphous. Thus, in the early days of fiber x-ray experiments, the fiber model was a long, thin arrangement of amorphous substance with crystallites (or micelles) existing at intervals (not necessarily periodic) along the axis, the intervening distances being occupied with amorphous (disordered) material. The high angle Bragg reflections arose from the scattering by the crystalline regions. In comparing fibers with crystals for the purpose of determining the lattice parameters (assuming that a lattice exists), we must describe a fiber as "part way" between a crystal and a powder. A fiber pattern is equivalent to that of a crystal rotated in all positions about one axis whereas a powder pattern is equal to that of a crystal rotated in all positions about all axes (Stokes, 1955). In all the fiber x-ray experiments to be described the x rays are assumed to be incident to the fiber perpendicular to its long axis.

A complete determination of the crystal structure cannot be made from x-ray data alone (Stokes, 1955), the successful explanations of crystal structure have been made by incorporating chemical and electron microscopy evidence along with x-ray results (Howsman, 1950; Bunn, 1949).

Degree of crystallinity

One factor which may be determined more definitely by high angle Bragg diffraction is the crystalline to amorphous ratio. In absolute terms this would be percentage of mass in the fiber which existed in crystalline form, but in practice it is defined as the ratio of the amount of intensity scattered by periodic scatterers to the total scattered intensity.

This parameter is explored thoroughly by Hermans and Weidinger in several papers since 1946. Beginning by developing a method for measuring accurately the absolute intensity scattered by the amorphous regions as opposed to the crystalline regions, and to account for all superfluous scattering by air, sample holder, cement, etc. (Hermans and Weidinger, 1946, 1948). A process of determining the degree of crystallinity of cellulose was completed, and determination made on several samples (Hermans and Weidinger, 1949, 1951). Cotton, flax and ramie were found to posess a degree of crystallinity of 70 per cent, as were most rayons. Physical treatment varied the degree of crystallinity and some cellulose with degree of crystallinity as low as 10 per cent was found.

The methods of Hermans and Weidinger were rather complex and involved many absolute intensity measurements. An attempt was made (Wakelin, 1959) to obtain a simple method suitable for "routine" quantitative evaluation of the degree of crystallinity in cellulose. This method involved the use of an amorphous standard (completely amorphous sample of ground cotton) and a crystalline standard, and the comparison with the intensity-angle diagrams of cellulose. Two methods for statistically comparing these plots were developed. Segal (1959) develops an even simpler method also involving the use of an amorphous standard obtained by grinding the cotton in a ball mill. The peaks almost completely disappear, and a broad, diffuse halo appears at an angle corresponding to a Bragg spacing 4.8 Å, assumed to be due to amorphous scattering. The maximum height of the 002 peak was used as indicative of the scattering of crystalline material, and a "crystallinity index" defined as

$$\text{C. R.} = \frac{I_{002} - I_{\text{amorphous}}}{I_{002}} \times 100$$

is obtained. Ant-Wourrienn (1962) sets forth yet another method for obtaining the degree of crystallinity being nearly identical with that of Segal's. These experimenters used different methods so a comparison of numbers is useless, however, Herman's and Weidinger's techniques are accepted as the most accurate (and most difficult), and in the 1949 paper, an attempt was made to derive an estimation of the absolute crystallinity based on their measurements. Numbers of 70 per cent for native cellulose, and 40 per cent for regenerated cellulose were obtained, which are in rough agreement with numbers found by others.

Average size of crystallites

The previously mentioned model for the fiber, i.e., crystallites existing at intervals along the axis separated by amorphous regions was challenged by Hosemann. In 1962 in a paper treating lattice distortions and the occurence of diffuse scattering on the film, he divided distortions into two types. In the lattice distortions of the first type the particles (atoms or molecules) lie in the right positions but each has a different shape or size. Another case of the first type is when the atoms (though all the same size and shape) suffer a small displacement from their ideal positions. A strong background scattering is evident in both situations. It is mentioned that as distortions of the first kind appear, the Bragg peaks lose intensity as the background intensity grows. This is in concurrence with the concept of "scattering power" as explained by Heikens (1959 a and 1959 b) and Hermans and Weidinger (1958). The scattering power per unit volume is related to its density, thus the total intensity scattered depends upon the mass and must be conserved for a given sample mass.

In distortions of the second type no attempt is made to define an "ideal" position and all long range order is lost. One deals with each atom and associates it only with its unit cell, each cell being of a different size. Each cell is defined by three vectors, a, b, and c, and the degree of order is simply the degree to which each of these vectors conform to a standard. This is a concept of the paracrystal. In amorphous substances each of these vectors can be approximated by a straight line (on a graph of length of the vector vs the number of cells with that length). In a perfect crystal, all of the a vectors

would be identical, and the graph would be a vertical line. For most crystals the graph is approximately a Gaussian distribution. Hosemann thus obtains a general mathematical method of describing any material as to its degree of crystallinity by simply specifying the distribution of each of the unit cell vectors. Obviously, the old model of crystallite embedded in an amorphous region is inadequate in the face of a paracrystalline model; sharp boundaries become doubtful and definite crystallite size becomes meaningless. Nevertheless, a parameter which may be called the "average size of the ordered regions" may be calculated from line broadening as will be described.

The method of obtaining the average size of the crystallites in high angle work is by the use of line broadening which employs the formula

$$t = \text{thickness of crystallite} = \frac{R\lambda}{B \cos \theta}$$

where B is the breadth of the line in degrees, 2θ is the scattering angle, λ the x-ray wavelength, and R a constant.

Not a great deal of work has been done on this technique of determining the size of the crystallite regions in fibers. Ruscher in 1958 treated this method from the standpoint of crystallites separated by amorphous regions and sought to make corrections necessary for its valid use. He arrives at a complex formula which may be repeatedly used to make better approximations and takes into account the photometer slit width, the considerations of $k\alpha 1$ and $k\alpha 2$ radiation, the sample radius, and the natural spectral line width. The necessity of subtracting the scattering due to amorphous region when finding the width of the Bragg interference peak presents some difficulty, and the fact that, even at its best, the size determination can be made only in one dimension causes more doubt. These problems are, however, well understood by us and may be overcome by special techniques.

Defining order as opposed to disorder becomes arbitrary in the light of the paracrystalline theory, and the value of R varies with the definition of "crystalline". Nevertheless, by adjusting R and making comparisons based on fixed values, this method may provide a relative quantitative measure of crystallite size.

Orientation

This is a subject which has been dealt with by many authors, being subjected to analysis by both high and low angle diffraction. The most used technique of obtaining an "average" orientation in high angle work is to measure the angular width of the 002 Bragg peak, since it is the most intense interference in native cellulose (Creely, 1956), and the distribution of intensity around the Debye-Scherrer rings.

Since a fiber pattern is equivalent to that of a crystal rotated about one axis, the relps (reciprocal lattice points) corresponding to the crystalline lattices within the fibers trace out circles which intersect the Ewald sphere twice at points. If all the crystals were oriented parallel, their relps would coincide and a pattern consisting of rows and columns of spots would result. If, however, some crystals were oriented at some angle α to the common (average) orientation, their relps would not coincide and the (already finite) relps become adjacent. The arrangement of relps for a fiber whose micelles are oriented at angles between plus and minus α is an arc. Thus, the relps become arcs and as the crystal is rotated about the long axis, a portion of the surface of a sphere is traced out (Figure 1).

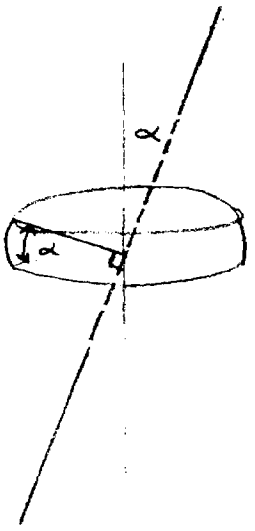

Figure 1

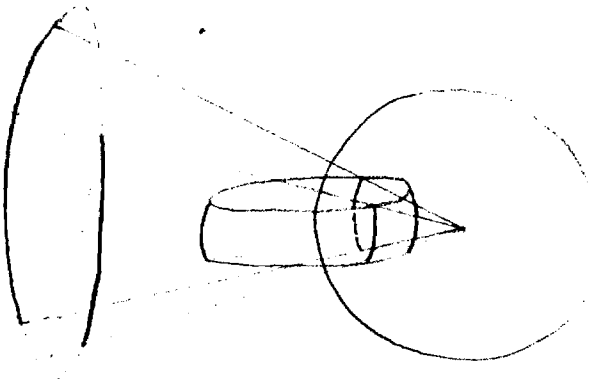

Figure 2

Diffraction occurs at the intersection of this sphere with the Ewald sphere, i.e., at two arcs on the circle (Figure 2). We get a portion of a powder pattern: two arcs of a circle. The film would show two arcs of a ring centered about the main beam.

low orientation
between ∞ and

high orientation

Figure 3

The fraction of fibers at any one orientation should be proportional to the relative intensity at that angle of arc away from the central spot which defines the "average" orientation. An empirical method was developed by Sisson and Clark (1933) for deriving the intensity distribution by the darkening of the film about the 002 arc. The angle δ between the arc center and the point at which the scattered intensity is one-half that at the arc center is used as a measure of the relative degree of orientation. Another method similar to this is used by Rebenfeld (1957) and by Weiss(1961),and uses, as a measure of orientation,"the x-ray angle" which is defined to be the angle between the equator of the photograph and a line drawn from its center to a point on the 002 peak where the diffracted intensity is 60 per cent its maximum. A method using detection counters is explained by Creely (1956) and is much easier and simpler in determining δ. Meridith (1951) sets forth three different methods for

experimentally obtaining a degree of orientation, all closely related to the method of Sisson and Clark and using the 002 arc. Tsien in 1949 had mathematically derived an expression for the relative number of crystallites at any orientation in cellulose as a function of the angle from the average orientation. He mathematically overcomes the problem of separating the closely spaced 002 and 101 arcs, and emphasized that this must be done experimentally by good resolution when using the 002 arc-length technique.

Correspondence of physical parameters

From the very first there has been an attempt to correlate the x ray data obtained with some physical property of the fibers. Several such correlations have been found and some attempts at a feasibility argument as an explanation have been made.

In exploring the degree of crystallinity of many fibers it has been found (Ward, 1950; Howsman, 1950) that those displaying a large degree of crystallinity are less active chemically--also the greater the crystallinity the less the moisture regain and the greater the density. The plots of these variables vs the degree of crystallinity yield straight lines. Ward gives a plausibility argument based on average intermolecular distances. Assuming this parameter to be greater in amorphous than in crystalline material (as indicated by density observations), the water or chemical reagent molecules would more easily fit among the disordered molecules than among the ordered. This is supported by the fact that reagents with very small molecules react more easily with the crystalline material.

The effect of varying degrees of orientation is discussed by several authors (Howsman, 1950; Bose, 1946; Kasai, 1964). Bose found that the orientation of

cellulose fibers could be greatly increased by stretching the fibers, and that this resulted in the fiber's having a greater tensile strength. Rebenfeld, in 1957 compared the x ray angle of various cotton samples with mechanical properties of the fibers and reaches a similar conclusion. Howsman states that increasing orientation results in increasing the tensile strength, Young's modulus, and the rigidity, while decreasing the plasticity, crease resistance, and chemical reactivity. Kasai made a study of the necking portion of cold-drawn polyethylene, i.e., a sample which contained unstretched and greatly stretched regions, and various degrees of stretching in-between. The orientation of the fibers increased with increasing stretch making a strong case for a stretch-orientation dependence. Ward (1950) agrees with Bose's observations of the increase of tensile strength of cellulose with orientation and adds that another parameter dependent upon this variable is the double refractivity of the sample.

High angle Bragg reflections were the first type of x ray analysis to be applied on fibers. Much information has been obtained by their use, and much more remains to be discovered with the development of new techniques of experimentation and analysis of data. The foregoing was only a very brief summary of the great amount of work, and makes no mention of instrumentation, sample preparation, or procedure. Howsman's summary contains a catalog of this information as well as other related topics.

Small Angle Scattering

Scope

There are, in general, two types of small angle scattering: small angle Bragg diffraction, which is just a special case of ordinary Bragg scattering (at small angles corresponding to long periodicity spacings), and small angle

diffuse scattering which was investigated by Guinier in 1939 and does not depend on periodicity at all. Both have been applied to fibers.

Small angle Bragg reflections

Fibers are small in two directions and very large (relatively) in the third. One application of x ray scattering at small angles is to detect periodicities existing in this long axis. In the old model of fibers (chains of crystals connected by amorphous regions) periodicities arose when the crystallites appeared at something like regular intervals, causing a difference in electron density to occur periodically. The diffraction spots would appear as arcs above and below the main beam, centered on the meridian (assuming main beam perpendicular to fiber axis). In theory, the structure of a fiber may be determined in this way (Stokes, 1955) since such parameters as size of the crystallites (from line broadening) and relative number of chains at any orientation (from the distribution of intensity about the arc) may be determined as in high angle work. In practice, however, the diffracted spots are often lost in the main beam, and obtaining good resolution combined with the necessity for monochromatization is one of the main problems in small angle work. MacArthur (1945) discusses the problems concerned with obtaining resolution and monochromatization with slits and pinholes and of focusing the beam. Hagström has treated this problem and has eliminated much of the error by the use of absorption filters together with various slit systems. Although these slits (or pinholes) give high resolution for very small openings, the loss of intensity is tremendous. A method is described for using plane crystals (to obtain monochromatization and parallel rays), cylindrically ground crystals (monochromatized and linearly focused rays) and spherically ground crystals (monochromatization and a point focus).

Kiessig (1960) derived a table of optimum sample to counter distances corresponding with various pinhole and slit sizes. Because of the difficulty in achieving satisfactory results a very small experimental variable may result in complete disruption of the data. Nelson (1963) examines the instrumental variability to be expected in the routine runnings of cellulose diffractograms and the effect of such variables as sample size, effect of cement needed to hold the fiber together, and effects of sample preparation. He found the diffraction peak height varied up to two per cent (this is for high angle, however, the result would not be less for low angle) for runs on successive days, nothing being changed by the experimenter. The need for a sample holder made of some material other than steel (such as aluminum) was also discussed together with the necessity for having samples which weigh 50 mg or greater. 50 mg seems to be the point above which weight makes no difference in the diffractogram, below which the pattern varies with weight. The concept of scattering power again must be considered and a sample must contain sufficient mass to scatter an observable amount of intensity. A small amount of cement was found not to affect the pattern.

A word might be said here concerning these crystallites. The type of transition of the molecules from the crystallite area to the amorphous areas is not settled. Although Hosemann's theory, previously described, has revolutionized thought in this area, others maintain that continuous transitions between these regions are unlikely. Hermans stated in a paper (1954 a) that "smooth transitions between the crystalline and non-crystalline portions seemed to be out of the question." Kiessig (1958) uses the concept of well of potential

energy associated with each particle, which has its trough at a certain distance from each molecule, and argues that when the molecules come within a certain distance they will "snap" into position, i.e., reach stable equilibrium, forming a crystal lattice,and making a sharp transition probable. Nevertheless, whether the transitions are sharp with definite crystalline and amorphous regions, or whether transitions are continuous and the definition of crystal is arbitrary (as in the paracrystalline concept), there are definitely regions with more order than the surroundings, with a consequently higher density which will thus give rise to Bragg scattering if they occur periodically.

Unfortunately, most fibers do not exhibit a very strict periodicity of these crystallites along their axes. Collagen gives excellent periodicity, and yields itself to almost complete analysis, but cellulose appears to have only one observable peak at a spacing of about 80 Å (Hermans 1954 b; Kiessig 1958). The increase of the sharpness of the reflections upon treatment with acid is attributed to the fact that the acid removes much amorphous material while leaving most of the crystalline material intact, increasing the difference in density between the two regions. This apparent selectivity in the action of the acid is one basis for the sharp boundary argument. There is a possibility, of course, that other periods in cellulose are so long that separation from the main beam is impossible. Stokes (1955) discusses the limits of spacing which can be determined by the x ray method and obtains a maximum spacing about 1000 Å although special apparatus may extend this limit.

Small angle diffuse scattering

Small angle diffuse scattering was introduced as an experimental tool by Guinier in 1939. Unlike Bragg diffraction it is not dependent upon periodicity for its application, and can therefore be used for exploration of non-periodic scatterers. An excellent resume of the subject is presented by Kratky (1948), who divided small angle diffuse scattering into dense and dilute portions. Much controversy has arisen over the interpretation of these types of scattering, and has not yet been settled. Briefly, dilute systems are systems in which the scattering particles may be considered to scatter radiation independently. One particle is the ideal dilute system, and a dilute system composed of n particles would behave as n independent scatterers. A dense system is one in which interparticle interference must be taken into account, and factors of relative particle position as well as number, size, and shape of the particles must be considered. For a single particle, small angle diffuse scattering depends not upon periodicity but only upon the differences in electron density within the material. As a function of the angle h, the intensity scattered is

$$I(h) = I_e \overline{F^2(h)} \qquad \text{where}$$

I_e = intensity scattered by one free electron at that angle,

$h = 4\pi \sin \theta/\lambda$

2θ= scattering angle

and

$$F(h) = \int_v [p(r)-p_o]e^{-ih\cdot r} dv$$

$p(r)$ = electron density as a function of the radial distance r from any particle

p_o = the average overall density of the irradiated material.

Thus the scattered intensity at any given angle is proportional to the difference in electron density between the region doing the scattering and the overall average density of the irradiated material, integrated over the volume of the region of different density. Specific expressions for the intensity scattered from particles of different geometrical shapes are given in Kratky (1948) and Guinier (in Volume III of "International Tables for X ray Crystallography"). Cylindrical particles, and systems of cylindrically symmetric particles are dealt with specifically by Oster and Riley (1952), Burge (1959) and Stuart (1959), and Milberg (1963). The amplitude scattered from an infinitely long cylindrically symmetric system is

$$F = \frac{2J_1(hR)}{hR}$$

where $J_1(hR)$ is a first order Bessel function. For systems of independent scatterers (dilute systems) this intensity is merely multiplied by the number of particles (it is understood that particle means a region of different electron density as described above). More complex equations exist for systems of non-independent scatterers (Heyn, 1955 e)

$$I = N\,\phi(hR)\left[1 + \int_0^\infty 2\pi r\,(\rho(r) - \rho_0)\,J_0(hR)dr\right]$$

where

N = no. of particles

$h = \frac{4\pi \sin\theta}{\lambda}$

2θ = scattering angle

$\phi(kR) = \frac{2\,J_1\,(hR)}{hR}$

$J_0(hR)$ = zero-order Bessell function

There are three quantities which lend themselves easily to analysis by small angle diffuse scattering. They will be treated separately as they have been applied to cellulose.

Orientation of micelles

This was the first parameter to be determined by the use of small angle diffuse scattering. The low angle pattern of most fibers consists of two lines along the equator stemming away from the main beam which, together with the absence of any lines extending radially along the meridian, suggests a fiber-like structure. For fibers whose micelles are arranged in a spiral-like configuration at some angle β to the fiber axis, the small angle pattern is an "x," and the angle between the arms on each side of the meridian is 2β.

high orientation low orientation

Figure 4

The amount of scattering between these limits, indicates their degree of orientation of the crystallites (Figure 4).

The micelles themselves are believed to be perpendicular face-oriented lamellar units. The method by which this conclusion was reached is described by Roy and Dan (1965), who described the expected pattern for several possible micellar shapes.

For cellulose, whose micelles are generally parallel to the fiber axis, the degree of disorientation may be seen in the amount of intensity scattered

at angles other than zero to the equator. Heyn studies the pattern of cellulose in several papers (1948, 1949 a, 1949 b), and finds decreasing orientation in the following order: hemp, flax, jute, roselle, and ramie. As in high angle investigation the orientation has been found to increase with stretching and tensile strength with orientation.

Size of microcrystallites

In 1939 Guinier developed an approximate formula for the intensity vs angle curve at very small angles which applied to dilute systems. The curve was approximated by an exponential and contained a parameter which was associated with the mass of the scattering heterogenity and was found in actuality to be the radius of gyration. For particles of uniform size and shape, the approximation is:

$$\ln I = - h^2R^2 + (\text{constant})$$

where

I = scattered intensity

R = radius of gyration of the particles

$h = \frac{4\pi \sin \theta}{\lambda}$

For particles of different geometrical shape, R would be related to the actual dimensions of the particles by various constants. A plot of ln I vs h^2 would give a straight line, whose slope would be related to the radius of gyration R. Therefore it would be possible to find this parameter unambiguously from the small angle scattering curve by measuring the slope as h approaches zero. (The approximation improves with decreasing angle, and approaches exactness at h = 0. The slope should be taken at this point rather than an average over the curve (Guinier 1955).)

The ideal case considered above (i.e., uniformity of size and shape) is not usually attained in reality. When a system of particles of uniform shape but different size is treated, it is seen that each particle tends to give a curve whose slope is related to its own particular radius of gyration. The ln I vs h^2 slope is then an indication of the distribution of radii of gyration, its linearity being a measure of the uniformity of particle size.

When no assumption can be made concerning the general geometrical shape of the particles, another variable must be taken into account. The determination of the radii of gyration would then yield very little information about the particles, since R depends on shape as well as size and density, and spheres and cylinders of the same density and volume would give different slopes. To sum up, the only parameter which may be determined unambiguously from the Guinier method is the average radius of gyration (its average being weighted in favor of those particles with the greater R) which depends upon density, volume, and shape. If assumptions may be made concerning the distribution of any two of these, the third distribution may be estimated.

In developing his approximation formula, Guinier made no allowances for interparticle interference. Some controversy has arisen about the possibility of applying Guinier's formula to dense systems of particles, and of determining whether a system is dense or dilute. In the early work on cellulose a controversy existed between Hosemann, who considered cellulose to be a dilute system and suitable to Guinier analysis, and Kratky, who advocated the assumption of interparticle interference in cellulose fibers. In later times, the argument has been carried on by Heyn on the first part, and Hermans and Weidinger on the second.

Both parties equate dilute systems with disorder and dense systems with systems in which the particles are structurally regular (Heyn 1950 a; Hermans and Weidinger 1954 b). Heyn has found that swelling cellulose fibers with a suitable swelling agent (water or various concentrations of NaOH, depending on the fiber and found by trial and error) destroys the regularity in the structure, and that cellulose fibers so treated yield straight line ln I vs h^2 plots. Mathematically, the scattering from non-independent systems is

$$I = N \Phi(hR) \left[1 + \int_0^\infty (\rho(r) - \rho_0) J_0(hR) dr \right]$$

(see page 15 for terminology)

The radial density function determines the contribution of interparticle interference, and if a regularity exists in the electron density, maxima and minima will occur, interparticle interference will be important, and the system is dense. If a random distribution exists, the density function will be a straight line equal to the average overall density, and the equation above collapses to that for a dilute system: $I = NQ^2(hR)$. Swelling was found by Heyn to be a treatment which would destroy this regularity, thus changing cellulose (previously a dense) into a dilute system.

Hermans and Wiedinger (1954 b) disagree, and argue that they have failed to obtain straight Guinier plots from cellulose with swellings up to 600 per cent. They further state that the appearance of maxima and minima depend on a small variation in regularity, and conclude that the arrangement of spots of increased local density, which are the crystalline regions in cellulose, are of a surprisingly high regularity, not to be removed by swelling. The controversy has not been settled, but now appears to perhaps be irrelevant.

In more recent papers (1958), Hermans and Weidinger have been concerned with absolute measurements of intensity and with the development and application of the concept of scattering power, which, as stated earlier, is proportional to the mass. A careful measurement of intensity leads to the formula

$$S \equiv \text{Scattering Power per Unit Volume} = (\rho_1 - \rho_2)^2 w_1 w_2$$

for a two phase system where w_1 is the volume fraction of material with density ρ_1. By calculating the scattering power per unit volume for cellulose, and comparing with the observed scattered intensity from air-dry cellulose, it is found that the scattered intensity cannot be explained in terms of the difference in density between the crystalline and amorphous regions. The needed density difference corresponds to the actual average overall density of the material. From the obtained data Hermans concludes that the air-dry fiber scattering curves are mainly determined by voids which exist in the fiber. These voids constitute a dilute system, to which Guinier analysis may be applied.

For water-swollen fibers, the scattering can be considered to be due to the presence of crystallites within a homogeneous body of water and amorphous material (their density is comparable). In another paper (1960) Hermans and Weidinger treat several other fibers and find that their scattering is what would be expected due to the differences in electron density between crystal and amorphous regions. These assertions about the origins of the scattering in cellulose are in direct opposition to the previous theory: that air-dry cellulose was dense and saturated cellulose was dilute. If the 'void' theory is confirmed, it will open up entire new areas to exploration by x rays. No new work has been reported, but it seems reasonable to ask about the influence of various physical treatment and chemical action upon these voids.

Electron density distribution

The third quantity dealt with is electron density distribution. It will be remembered that the intensity scattered from non-independent systems is

$$I = N \phi(hR) \left[G(hr) + 1 \right]$$

where

$$G(hr) = \int_0^\infty 2\pi r \, (\rho(r) - \rho_0) \cdot J_0(hR) dr$$

The contribution to this intensity function from interparticle interference is G(hr).

By use of the Hankel inversion theorem,

$$2\pi(\rho(r) - \rho_0) = \int_0^\infty h \, G(hr) \, J_0(hr) dh$$

so that the radial distribution of electron density may be determined (Heyn, 1955 b). The problem then lies in determining G(kr), the intensity distribution caused by interparticle interference. If one accepts Heyn's assertion that swelling creates a dilute system in cellulose, then by exposing samples of dry and swollen fibers, and subtracting the latter from the former, the radial distribution of electron density from any particle could be found.

Using the low-angle scattering apparatus, and employing a proportional counter rather than films, the most available data concerns the two subjects: size of crystallites and orientation of the crystallites. There is very much that is not clear in cellulose, as (hopefully) made obvious by the foregoing summary, and more knowledge needs to be found concerning the cause of low angle scattering (voids or crystallites) and the effects of swelling to various degrees by soaking with liquids of various types. It is hoped that some advances may be made in this direction in the present experiment.

BIBLIOGRAPHY
(Selected)

2. APPLICATION OF X-RAYS TO FIBERS - PARTICULARLY CELLULOSE

1. Ant-Wuorinen, O. and A. Visapaa. Norelco Reptr. 9, 47-52 (1962).

2. Arend, A. G. "Practical X-ray Examination of Textile Fibers," Textile Recorder 60, 24-26 (1942).

3. Bonart, R. and R. Hosemann. "Paracrystalline Character of Long-Period Interferences," Fortschr. Mineral 38, 59-61 (1960).

4. Bonart, R. "The Possibility of a Hypothesis-Free Analysis of Axially Symmetrical Fiber Diagrams," Kolloid-Z 152, 57-61 (1957).

5. Bose, C. R. and N. Ahmad. Indian J. Phys. 20, 105-110 (1946).

6. Bunn, C. W. "The Study of Fibers by X-ray Diffraction Method," Fiber Science, J. M. Preston, ed., 198-222 (1949).

7. Burge, R. E. "X-ray Scattering by Bundles of Cylinders," Acta Cryst. 12, 285-9 (1959).

8. Creely, J. J., L. Segal, and H. Ziifle. "Determination of the Degree of Crystallite Orientation in Cotton Fibers by Means of the Recording X-ray Diffraction Spectrometer," Textile Research J. 26, 789-97 (1956).

9. Fraser, R. D. B., T. P. MacRae, and H. C. Freeman. Acta Cryst. 12, 171 (1959).

10. Frisch, H. L. and F. H. Stillinger. "Contribution to the Statistical Geometric Basis of Radiation Scattering," Journal of Chem. Phys. 38, 2200-2207 (1963).

11. Go, V. Y., S. Nagata, and J. Kakinoki. Bull. Chem. Soc. Japan 13, 198-210 (1938).

12. Gross, J. "Collagen," Scientific American, (May, 1961).

13. Gjonnes, J. and N. Norman. "The Use of Half Width and Position of the Lines in the X-ray Diffractogram of Native Cellulose to Characterize the Structural Properties of the Samples," Acta Chemica Scandinivica 12, 2028-2033 (1958).

14. Hagstrom, S. and K. Seigbahn. "A Small-Angle X-ray Scattering Apparatus Using a Spherically Bent Crystal," J. Ultrastructure R. 3, 401-419 (1960).

15. Hearle, J. W. S. "The Structure and Mechanical Properties of Fiber," J. Textile Inst. Proc. 53, 449-464 (1962).

16. Heikens, D. (A) and D. Heikens, P. H. Hermans, and A. Weidinger (B). J. Polymer Sci. 35, 139-43, 145-65 (1959).

17. Hermans, P. H. and A. Weidinger. Rec. Trav. Chem. 65, 620-3 (1946).

18. Hermans, P. H. "Ratio of Crystalline-Amorphous Region in Celluloise Fiber," J. Chem. Phys. 44, 135-40 (1947).

19. Hermans, P. H. and A. Weidinger. J. Appl. Phys. 19, 491-506 (1948).

20. Hermans, P. H. and A. Weidinger. "X-ray Studies on the Crystallinity of Cellulose," J. Polymer Sci. 4, 135-44 (1949).

21. Hermans, P. H. Makromol Chem. 6, 25-29 (1951).

22. Hermans, P. H. and A. Weidinger. Makromol Chem. 13, 30-9 (1954).

23. Hermans, P. H. and A. Weidinger. J. Polymer Sci. 14, 405-7 (1954).

24. Hermans, P. H. and A. Weidinger. "Experimental Determination of the Correction for the Lorentz Factor in Small-Angle Photographs of Highly Oriented Cellulose Fibers," Z. Elektrochem 58, 924-5 (1954).

25. Hermans, P. H., A. Weidinger, and D. Heikens. "Recent Contributions of X-ray Low-Angle Diffraction to the Investigation of Cellulose Fiber Structure," Pulp Paper Mag. Can. 59, 137-41 (1958).

26. Hermans, P. H. and A. Weidinger. Makromol Chem. 39, 67-78 (1960).

27. Hermans, P. H. and A. Weidinger. "Relative Intensities of the Crystalline X-ray Lines in Cellulose Fibers," J. Polymer Sci. 50, No. 153, 510-512 (1961).

28. Hesslev, Lyle E. "The Relationship Between Cotton Fiber Development and Fiber Properties," Textile Research Journal, 38-43 (January 1961).

29. Heyn, A. N. J. J. Am. Chem. Soc. 70, 3138-9 (1948).

30. Heyn, A. N. J. J. Textile Research 19, 163-72 (1949).

31. Heyn, A. N. J. J. Am. Chem. Soc. 71, 1873-4 (1949).

32. Heyn, A. N. J. J. Am. Chem. Soc. 72, 5768-70 (1950)

33. Heyn, A. N. J. "Small-Angle X-ray Scattering by Cellulose Fibers-- Experimental Study of the Orientation Factor in Model Filaments and Rayons," J. Am. Chem. Soc. 72, 2284-5 (1950).

34. Heyn, A. N. J. J. Polymer Sci. 14, 403-4 (1954).

35. Heyn, A. N. J. J. Appl. Phys. 26, 519-26 (1955).

36. Heyn, A. N. J. J. Appl. Phys. 26, 1113-20 (1955).

37. Heyn, A. N. J. "Crystalline State of Cellulose in Fresh and Dried Mature Cotton Fiber from Unopened Bolls as Studied by X-ray Diffraction," J. Polymer Sci. 3 (Part A), 1251-1265 (1965).

38. Hoseman, R. "The Theory of Small-Angle Scatterins," Z. Elektrochem 60, 189-90 (1956).

39. Hoseman, R. Polymer 3, 349-92 (1962).

40. Howsman, J. A. Phys. Methods in Chem. Anal. 1, Walter G. Berl, ed., 108-165 (1950).

41. Kasai, N. and M. Kakudo. "Fine Texture in Necking Portion of Cold-Drawn Polyethylene," J. Polymer Sci. 2, 1955-76 (1964).

42. Kast, W. and L. Flaschner. Kolloid-Z 111, 6-15 (1948).

43. Kast, W. "The Indications of X-ray Diagrams of Cellulose Fibers," Kunststoffe 39, 46-7 (1949).

44. Kast, W. "X-ray Structure Studies of Cellulose Fibers," Melliand Textilber 31, 83-8 (1950).

45. Kast, W. "X-ray Investigation of the Structure of Cellulose Fiber II," Melliand Textilber 32, 442-6 (1951).

46. Kast, W. Z. Electrochem 57, 525-30 (1953).

47. Kenney, Arthur W. "The Structure of Cellulose and Its Derivative by the X-ray Diffraction Method," Tech. Assoc. Papers 25, 722-3 (1942).

48. Kiessig, Heinz. "X-ray Investigation of the Long Period in Cellulose," Das Papier 12, 117-22 (1958).

49. Kiessig, Heinz. "X-ray Low-Angle Investigations of Native and Regenerated Cellulose Fibers," Norelco Reptr. 7, No. 3, 99-103 (1960).

50. Kratky, O. Z. Elektrochem 57, 42-50 (1953).

51. Kratky, O. and G. Porod. Z. Elektrochem 58, 918-23 (1954).

52. Kratky, O. and H. Sembach. "X-ray Small-Angle Scattering Measurements on Cellulose," Angew. Chem. 67, 603-6 (1955).

53. Kratky, O. and G. Porod. "Small-Angle Scattering by Cellulose," Z. Elektrochem 60, 188-190 (1956).

54. Kratky, O. and G. Miholic. Monatsh. 94, 151-61 (1963).

55. Legrand, Charles. "A New Method of Recording X-ray Diagram of Fibers," Compt. Rend. 237, 573-5 (1953).

56. Legrand, C., A. Bonnemayre, A. Guinier, and P. Antzenberger. Nature 175, 1045 (1955).

57. Luzzati, V. "Interpretation des Mesures Absolues de Diffusion Centrale des Rayons X en Collimation Ponctuelle ou Lineaire: Solutions de Particules Globulaires et de Bâtonnets," Acta Cryst. 13, 939 (1960).

58. MacArthur, Ian. Proc. Leeds. Phil. Lit. Soc., 243-249 (July 1945).

59. Matano, Chujiro. "X-ray Diagram of Fiber Structure," Proc. Phys. Math. Soc. Japan 21, 14-20 (1939).

60. Meredith, R. J. Textile Inst. 42, T275-90 (1951).

61. Milberg, M. E. "Application of the Simplified Cylindrical Distribution Function: Orientation in Linear Polyethylene," J. Appl. Phys. 34, 722 (1963).

62. Nelson, Mary L. and E. F. Shultz. "X-ray Diffractograms of Cotton: Effect of Sample Weight and Other Variables," Textile Research J. 33(7), 515-20 (1963).

63. Nowotny, Hans. "X-ray Structure Investigation in Fiber Research III. The Application of X-ray Specta in Fiber Research," Melliand Textilber 22, 614-19 (1941).

64. Orr, R. S. et al. "The Role of Spiral Structure in Untreated and Treated Cottons," Textile Research J., 302-310 (April 1961).

65. Oster, Gerald and D. P. Riley Acta Cryst. 5, 272-6 (1952).

66. Plotze, E. and H. Person. "Crystal Orientation in Cellulose Fibers," Z. Physik. Chem. B45, 193-200 (1940).

67. Porod, G. Makromol. Chem. 35, 1-25 (1960).

68. Rebenfeld, L. and W. P. Virgin. "Relation Between the X-ray Angle of Cottons and Their Fiber Mechanical Properties," Textile Research J., 286-289 (April 1957).

69. Roy, S. C. and S. Das. "Small-Angle X-ray Scattering of Cellulose I. Shape and Extent of Scattering in Some Vegetable Fibers," J. of Appl. Polymer Sci. 9, 3427-3437 (1965).

70. Ruscher, Christian. "X-ray Determination of Particle Size," F. Asertorsch U. Textiltech 9, 485-7 (1958).

71. Segal, L. et al. Textile Research J. 29, 786-93 (1959).

72. Sisson, W. A. and G. L. Clark. "X-ray Method for Quantitative Comparison of Crystallite Orientation in Cellulose Fiber," Industrial and Chemical Engineering, Analytical Edition 5, 296-300 (1933).

73. Stern, F. Trans. Faraday Soc. 51, 430-41 (1955).

74. Stokes, A. R. "Theory of X-ray Fiber Diagram," Progr. in Biophys. and Biophys. Chem. 5, 140-67 (1955).

75. Stuart, I. M. "Calculation of Scattering Intensity from a Cylindrically Symmetric System," Acta Cryst. 12, 71-2 (1959)

76. Synecek, V. and M. Simerska'. "Rapid Graphical Detn. of Radial Intensity Distribution of Small-Angle Scattering of X-rays from Measured Data," Czechoslov J. Phys. 10, 240-6 (1960).

77. Trillat, J. J. and C. Legrand. "Recent X-ray Techniques Suitable for the Study of Fibers," Bull. Inst. Textiles France 96, 7-25 (1961).

78. Tsien, P. C. "A New Method of Evaluation of the Crystalline Orientation of Cellulose Fibers from X-ray Data," Textile Research J. 19, 330-41 (1949).

79. Vineyard, George H. "Patterson Transforms and the Interpretation of X-ray Scattering from Fibers," Acta Cryst. 4, 281 (1951).

80. Virgin, W. P. and H. Wakeham. "Cotton Quality and Fiber Properties," Part IV, Textile Research J., 177-191 (March 1956).

81. Wakelin, J. H., H. S. Virgin, and E. Crystal. J. Appl. Phys. 30, 1654-62 (1959).

82. Ward, Kyle, Jr. J. Textile Research 20, 363-71 (1950).

83. Weiss, L. C. et al. "Moduli of Cotton Fibers and Yarns in Relation to X-ray Angles," Textile Research J., 787-793 (September 1961).

REPORT NO. 3 (SEMIANNUAL REPORT)
GEORGIA TECH PROJECT NO. A-843

1 February 1966 to 1 August 1966

FRICTIONAL PROPERTIES OF COTTON FIBERS

By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

PREPARED FOR
UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
NEW ORLEANS, LOUISIANA

1 August 1966

Engineering Experiment Station
and
School of Textile Engineering

GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

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United States Department of Agriculture
Agricultural Research Service
Southern Utilization Research and Development Division
New Orleans, Louisiana

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ABSTRACT

The purpose of this research is to establish the frictional characteristics of cotton fibers and to determine how these characteristics may change as cotton fibers are processed from the boll to the yarn.

Five theses for the M.S. degree in Textiles, conducted concurrently with this work, have allowed extensive studies of the microscopy, infrared spectroscopy, and frictional character of fibers, principally cotton; and specific studies of the effects of ginning, opening, picking and carding on the cotton fiber length distribution, strength, and frictional character.

Further developments in the frictional measuring apparatus have been made and a second apparatus, employing an electromagnetically controlled normal force as well as a servo-controlled frictional force output, has been constructed. This equipment has extended the normal force range capability to a lower limit of 2 mg from a previous limit of approximately 20 mg.

In the microscopy work sections and surface features of 10 specimens of Georgia cotton, 14 specimens from a cotton collection provided by Mr. J. N. Grant of USDA, SURDD, and 14 man made fiber specimens were presented. From the micrographs or observations denier was found to vary from approximately 0.9 to 2.2 for the various species and convolutions over the range 62 to 95 per inch. Crimp varied over the range 7.4 to 16.6 per inch. Maturity coefficients of 0.74 to 0.79 were found. Increases in convolutions per inch, indicative of fiber shortening were observed on heating in a gin dryer simulator. Heating to 280°F for 30 seconds caused residual (over 24 hours later) changes of 6 percent and, to 340°F, 27 percent.

In the infrared research the KBr disc, ATR (attenuated total reflectance) and Fiber Press methods of specimen preparation were compared. Spectra of cotton, viscose rayon, a polyester (Dacron), an acrylic (Creslan), and nylon were examined. The KBr method, at this stage of development, gave the more reliable quantitative results. The fiber press technique appears to be especially valuable for rapid specimen preparation and for studies including polarized radiation; the ATR method is especially useful for examination of surface properties of fibers or fabrics.

Spectra of cotton fiber before and after ginning displayed insignificant differences. Cotton subjected to heating at 392°C for an extended period exhibited a strong band at 5.8 μ which was not discernible in the ginned cotton. Cotton milled to 20, 40, and 60 mesh respectively exhibited little change in crystallinity index (by the method of O'Connor), 0.68, 0.69, 0.69, respectively compared with 0.71 reported by O'Connor for cotton of a different variety milled to 20 mesh. On the other hand the cotton subjected to ball milling for 12 hours was reduced to a low crystallinity index of 0.24.

Spectra prepared by the fiber press method and examined by polarized radiation exhibited excellent discrimination of absorption bands and splitting of several bands into composite bands. For instance a band at 6.15 μ was composed of a strong band at 6.05 μ and lesser bands on each side. The ATR and fiber press technique need further investigation to exploit their full potential.

In the friction research, it was found that an arm of high moment of inertia, a normal force of about 20 mg, and slow fiber sweep rates were desirable (< .07 mm/sec). Whereas effects of normal force changes within the range 20 to 40 mg were small, effects of varying fiber tension were large. The μ_k value of cotton varied over the range 0.36 at 125 mg tension (applied to fiber at moment of attachment) to 0.24 at 1150 mg. Fibers heated to various temperatures between 25°C and 220°C exhibited a small increase in μ_k, in the range 0.29 to 0.33. Coefficients of friction for seed cotton, cotton after ginning and after opening, picking and carding were 0.26, 0.28, 0.29, 0.31 and 0.32, respectively.

A friction measuring instrument, using electromagnetically applied normal force values, has also been developed for use at normal forces of 2 to 20 mg. Measurements with this unit exhibit excellent promise. Significant differences in the character of the analog plot of the frictional force occur for fibers of different material; the character of the curve for a specific fiber of cotton is similar on successive runs. The character is related to the shape of the particular fiber as well as the material factor. The μ_k for viscose rayon is very similar to that for cotton.

Specimens of Empire WR, Dixie King, and Carolina Queen cottons grown in Georgia were examined before and after mechanical ginning. Reductions in fiber mean length of 2.5 to 15 percent were observed. The force of withdrawal of fibers from the seed gave an average value of 1.23 grams and

the distribution of the withdrawal forces compared to the distribution of fiber breaking strengths at 6.4 mm span length gave an overlap of 27.5 percent. Convolution counts of fibers made before and after simulated gin drying (30 seconds in hot air) gave count increases, retained after 24 hours, of 6 percent at 266°F and 27 percent at 340°F.

Investigations of the effects of opening, picking and carding on the physical properties of the fibers were examined. Fiber mean lengths were found to be 0.848 (before opening), 0.693, 0.703, and 0.878 inches for the successive stages indicating a fiber shortening effect followed by removal of the short fibers. The micronaire values were 4.00, 4.02, 4.19, and 4.37 micrograms/inch indicating some increase in fiber size or damage. The Pressley Bundle Strength Test gave values of 24.01, 24.14, 25.20, and 26.31 grams/tex. Since measurements of single fiber strengths gave no overall improvement in strength the Pressley Bundle method indicated a strength increase due to loss of weak fibers. Elongation was reduced by processing. The Congo Red dye test indicated an increase in damaged fibers from 16 percent after ginning to 32 percent after carding. The coefficient of kinetic friction for fibers increased from 0.28 to 0.32. Changes also occurred in the coefficient of static friction and character of the analog plot which appear more significant than the change of kinetic coefficient.

In x-ray diffraction studies of fibers good agreement with crystallinity changes observed by infrared absorption methods were found for fibers undergoing milling in the Wiley Mill or ball milling. Insignificant changes in crystallinity resulting from ginning were noted.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influence of crimp, convolution, cross section, surface texture, and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

II. INTRODUCTION

The importance of fiber friction and the problems associated with its measurement have been discussed in the preceding reports of this series.[1] This report presents information concerning the development of a second frictional measurement apparatus and the effects on the friction between fibers resulting from the tensile force with which the respective fibers are mounted, the normal force between the fibers, and temperature cycling the cotton fibers. In addition some measurements have been obtained for cotton in the condition before ginning, after ginning, and after opening, picking and carding.

Theses[2,3,4,5,6] have been prepared by five candidates for the M.S. degree in Textiles or Textile Engineering in conjunction with this work and have covered the topics, fiber microscopy, fiber friction, infrared spectroscopy of fibers, effects of ginning on the cotton fiber, and the effects of opening, picking and carding on the cotton fiber. Results obtained in the various theses are being submitted to the sponsor concurrently.*

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* Copies of these thesis are also available from the library of the Georgia Institute of Technology through interlibrary loan service.

III. EXPERIMENTAL WORK

A. GENERAL

In the study of the friction of cotton fibers one upon another the effects of the tensile force with which the fibers are mounted and the normal force between the fibers are of importance. Although some data on these effects have been reported previously by others,[7,8] the possible importance of the effects to the measurements in this research required their delineation for the present instrument. In addition, indications had already been obtained, that data obtained with the present instrument exhibited large scatter below normal forces of 20 mg. A new instrument was designed and built to cover the normal force range 2 to 20 mg.

Concurrently with these investigations further work was conducted in the fields of optical and electron microscopy, infrared spectroscopy, and x-ray diffraction with respect to the characterization of cotton and other fibers. The effects on the physical properties of the cotton fiber of ginning and of opening, picking and carding were examined.

B. EXAMINATION OF COTTON FIBERS BY OPTICAL AND ELECTRON MICROSCOPY*

1. Introduction

The purpose of this research was to characterize and catalogue a large number of cotton species and other fibers by optical and electron microscopy in order to furnish information that might be applicable to the frictional measurement problem. A second objective was to analyze data with respect to fiber parameters obtained by methods of microscopy for the purpose of fiber identification, denier determination, fiber maturity measurement, studies of effects of temperature cycling, and fiber process damage estimates. The methods and results of the various examinations are discussed below.

---

* The data herein were extracted largely from a thesis for the M.S. degree in Textiles by Donald L. House, completed September 1966.

2. Apparatus

The apparatus and principal procedures used have been described in Semiannual Reports Nos. 1 and 2 of this contract and in more detail in the thesis of D. House.[2] In general these follow common practice with only minor modifications. Special apparatus used during the period will be described with relation to the particular experiment.

3. Fibers Examined

The longitudinal forms and the cross sections of 17 cotton species, 14 man-made fibers, and wool, as listed in Table 1, have been examined and catalogued in the thesis of House. Surface replicas have also been examined by electron microscopy and these features of each fiber have been displayed in some 40 illustrations of the thesis noted.

Illustrations of a selection of various cotton fibers are shown in Figures 1, 2, 3, 4, 5, 6, 7, and 8 of this report.

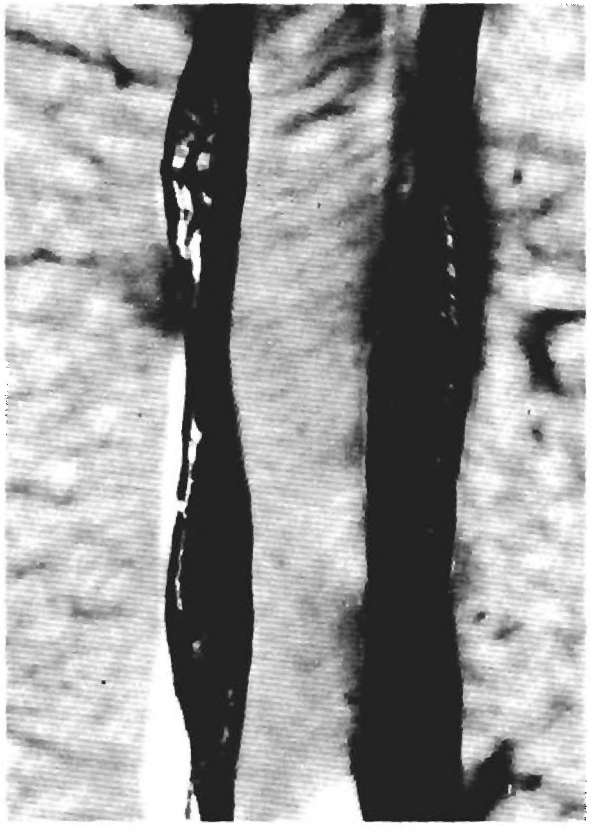
4. Fiber Denier Determined from Cross Section Models

Measurements of the median major and minor axes of the cotton fibers of the various species were made, a diagram model of the cross section of each fiber was constructed; and the average cross section for each variety was determined from the model with a planimeter. Utilizing the cross sectional area, a unit length, and the density of cellulose, the denier of each variety of cotton was calculated and compared with that obtained by Orr et al.,[9] for the same cotton.* The data obtained by Orr et al., employing the weight method, for denier versus the fiber upper quartile length are exhibited in Figure 9. The decrease in denier with fiber length has long been recognized. The grouping by principal species is also apparent. The data obtained by the cross section method in this research is shown in Figure 10. The check between the denier calculated from the data obtained by micrography and by weight is reasonably good in most cases, with higher scatter for the micrography data.

* Furnished through the courtesy of Mr. J. N. Grant, SURDD, USDA, New Orleans, Louisiana.

Table 1. Fiber Specimens Examined

Specimen Number	Fiber Name
1.	Empire WR Hand Ginned (Experiment, Ga.)
2.	Empire WR Ginned Lint (Experiment, Ga.)
3.	Empire WR Hand Ginned (Locust Grove, Ga.)
4.	Empire WR Ginned Lint (Locust Grove, Ga.)
5.	Dixie King Hand Ginned
6.	Dixie King Ginned Lint
7.	Carolina Queen Hand Ginned
8.	Carolina Queen Ginned Lint
9.	Carolina Queen Hand Ginned (Hand Picked)
10.	Carolina Queen Ginned Lint (Hand Picked)
11.	Wilds 5
12.	Delfos
13.	Mexican Big Boll
14.	Rowden
15.	Pima S-1 (Silver)
16.	St. Vincent Sea Island (Silver)
17.	SXP
18.	Bobshaw (1951 crop)
19.	Hopi Acala 54
20.	Pima S-1
21.	Deltapine 15
22.	Bobshaw (1955 crop)
23.	Stoneville
24.	Deltapine (1955 crop)
25.	Dacron Polyester
26.	Celanese Dull Triacetate
27.	Avisco Rayon
28.	Avisco Bright Rayon
29.	Avisco Dull Rayon
30.	Avisco Bright Fiber 40 Rayon
31.	Enka Rayon
32.	Dupont Semi-Dull Nylon Polyamide
33.	Herculon Polypropylene
34.	Kodel Polyester II Semi-Dull
35.	Kodel Polyester IV Semi-Dull
36.	Eastman Verel Modacrylic Bright
37.	Eastman Verel Modacrylic Dull
38.	Dupont Orlon 72 Acrylic
39.	Wool-unscoured
40.	Wool-scoured



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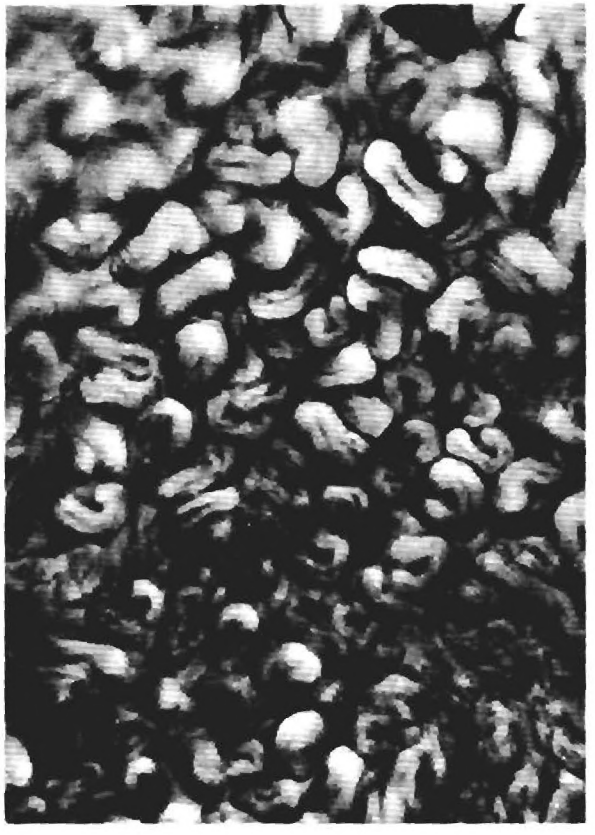

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Figure 1. Optical and Electron Micrographs of Hand Ginned, Empire WR Cotton (Experiment, Georgia): a. Fiber Form; b. Sections; c., d. Replicas of Surface.

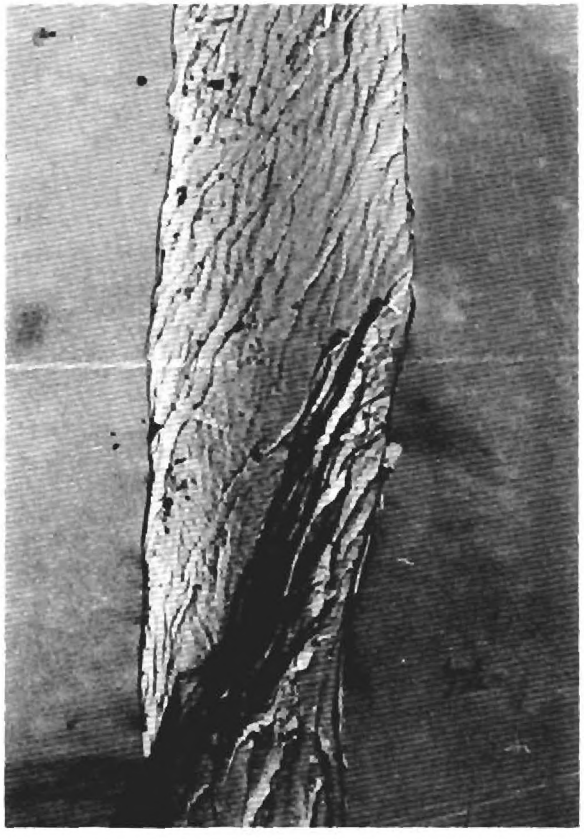

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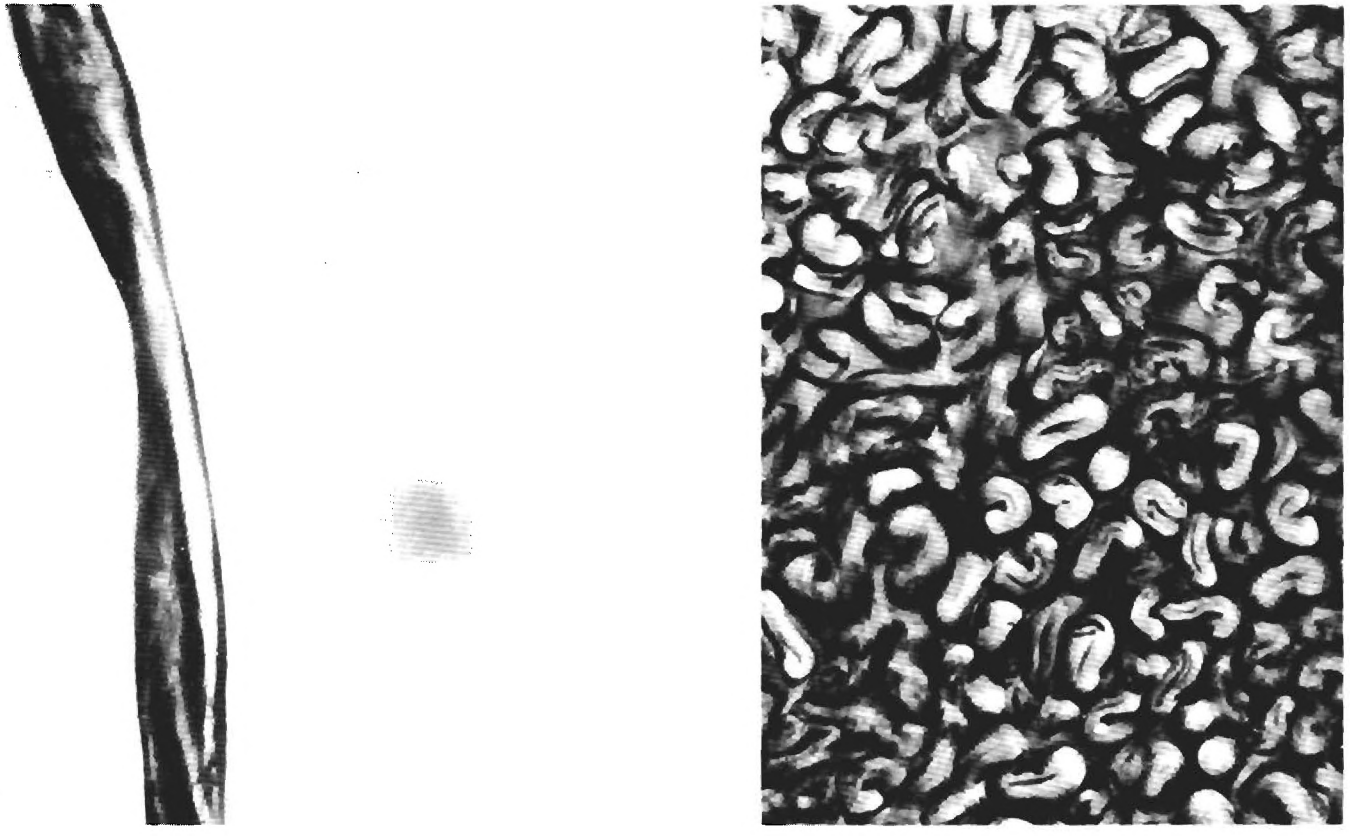
Figure 1. Continued, Optical and Electron Micrographs of Hand Ginned, Empire WR Cotton (Experiment, Georgia): a. Fiber Form; b. Sections; c., d. Replicas of Surface.



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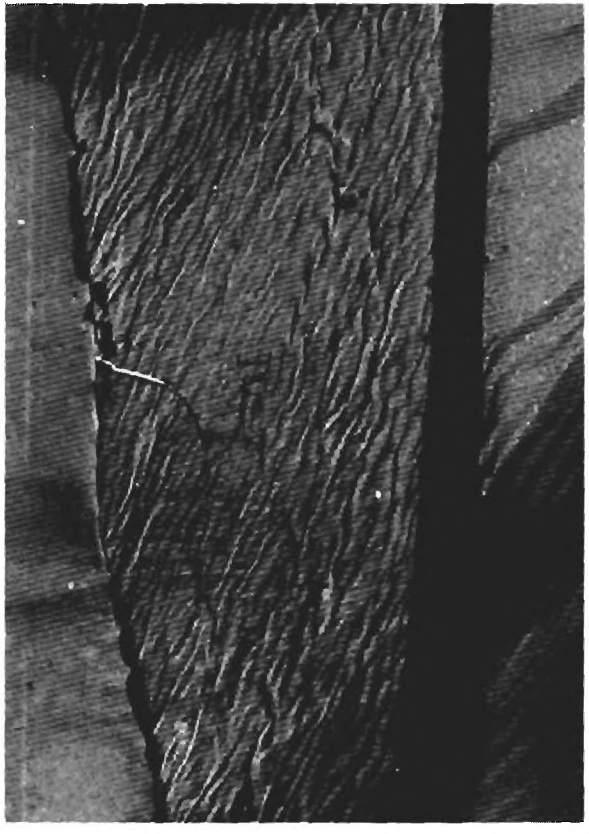
(b) 560X

Figure 2. Optical and Electron Micrographs of Mechanically Ginned, Empire WR Cotton (Experiment, Georgia): a. Fiber Form; b. Sections; c., d. Replicas of Surface.



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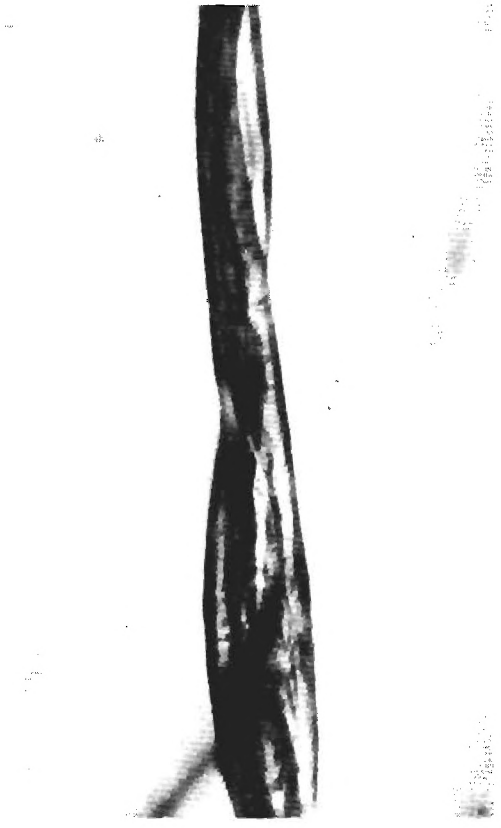

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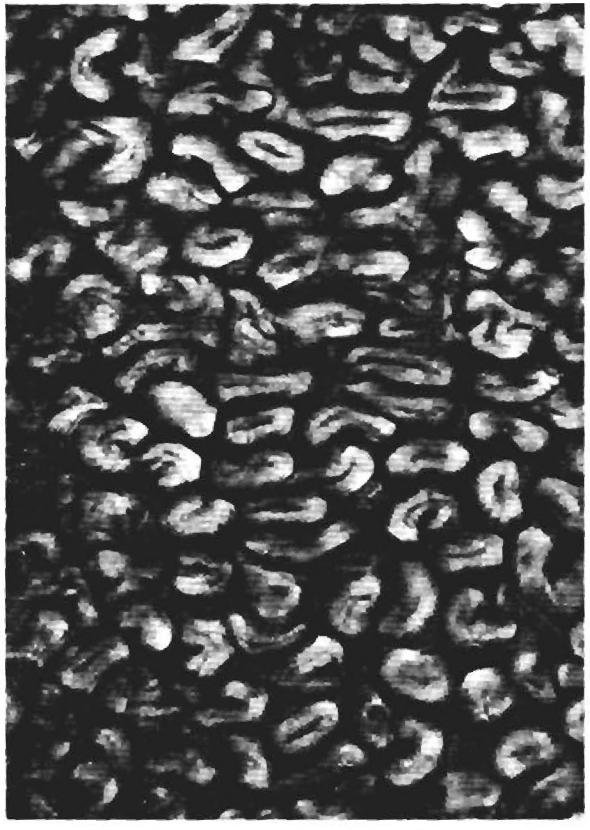

(b) 560X

Figure 3. Optical and Electron Micrographs of Hand Ginned Dixie King Cotton (Locust Grove, Georgia): a. Fiber Form; b. Sections; c., d. Replicas of Surface.

(c) 3,800X

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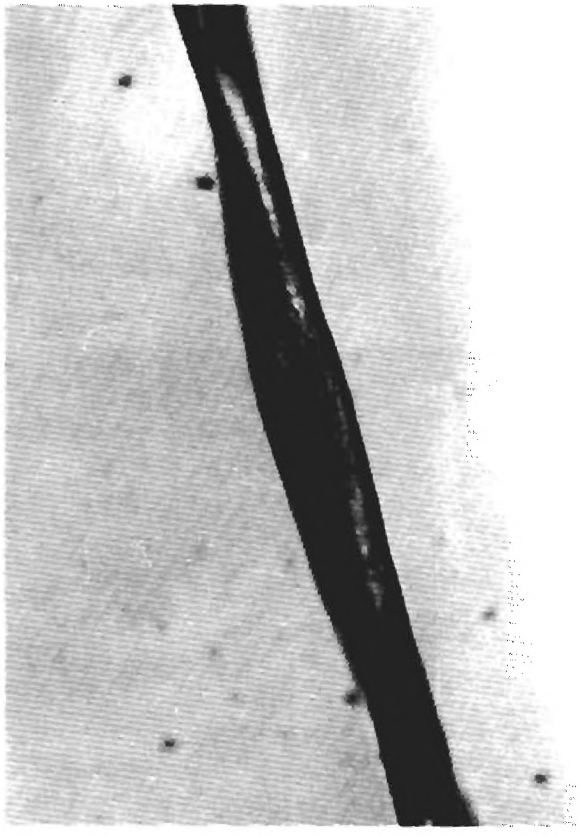
Figure 3. Continued, Optical and Electron Micrographs of Hand Ginned Dixie King Cotton (Locust Grove, Georgia): a. Fiber Form; b. Sections; c., d. Replicas of Surface.



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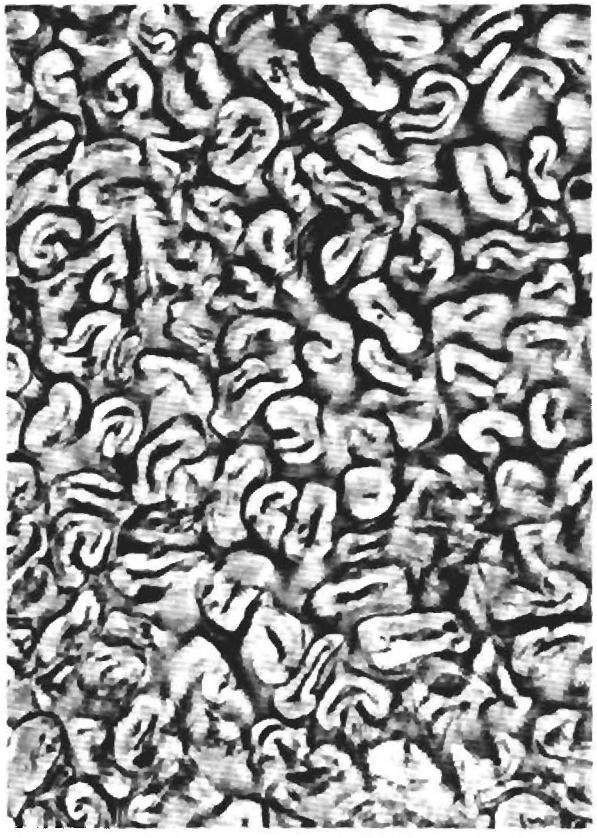

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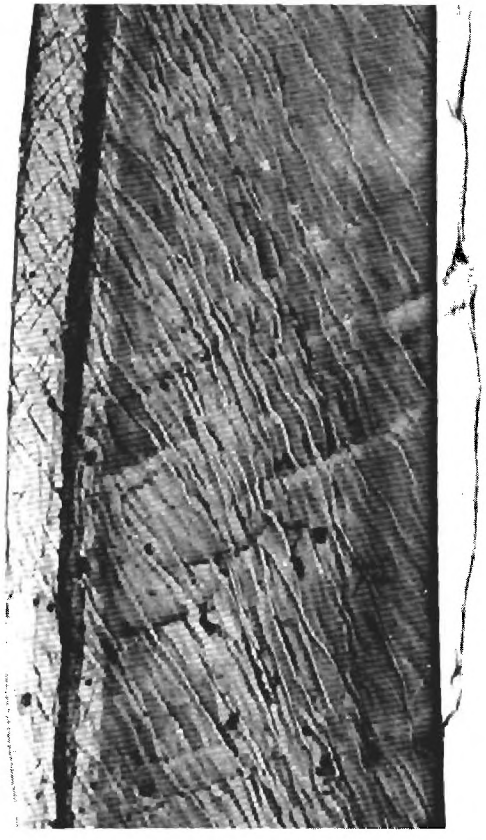
Figure 4. Optical and Electron Micrographs of Mechanically Ginned Dixie King Cotton (Locust Grove, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.



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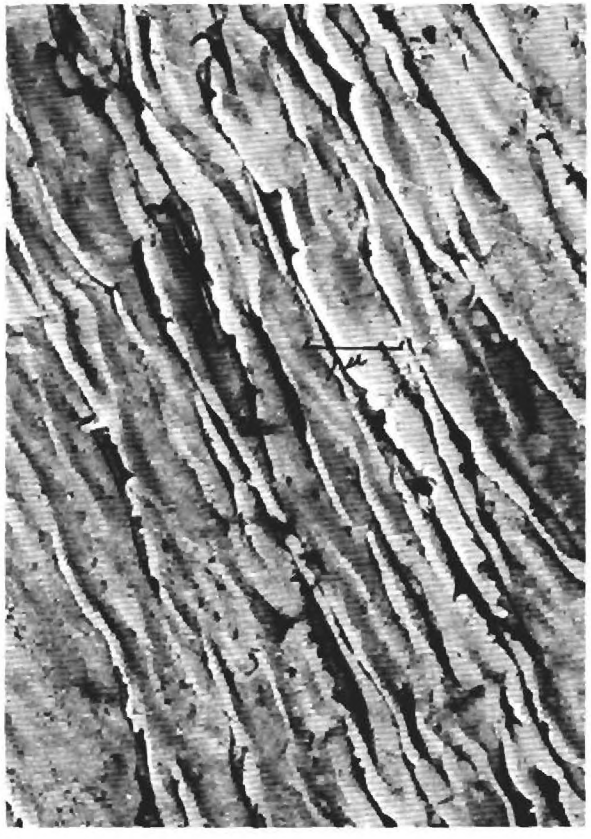

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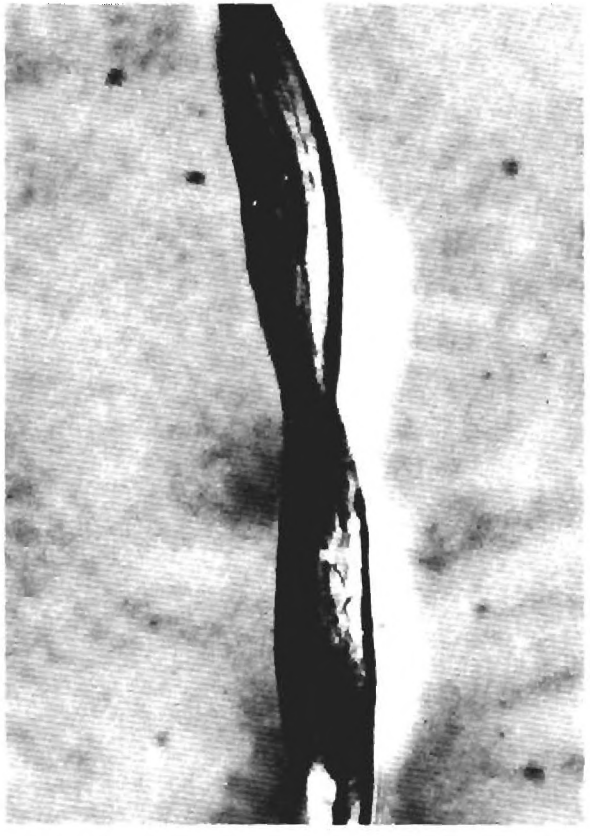
Figure 4. Continued, Optical and Electron Micrographs of Mechanically Ginned Dixie King Cotton (Locust Grove, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.



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(b) 560X

Figure 5. Optical and Electron Micrographs of Hand Ginned Carolina Queen Cotton (Tyrone, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.

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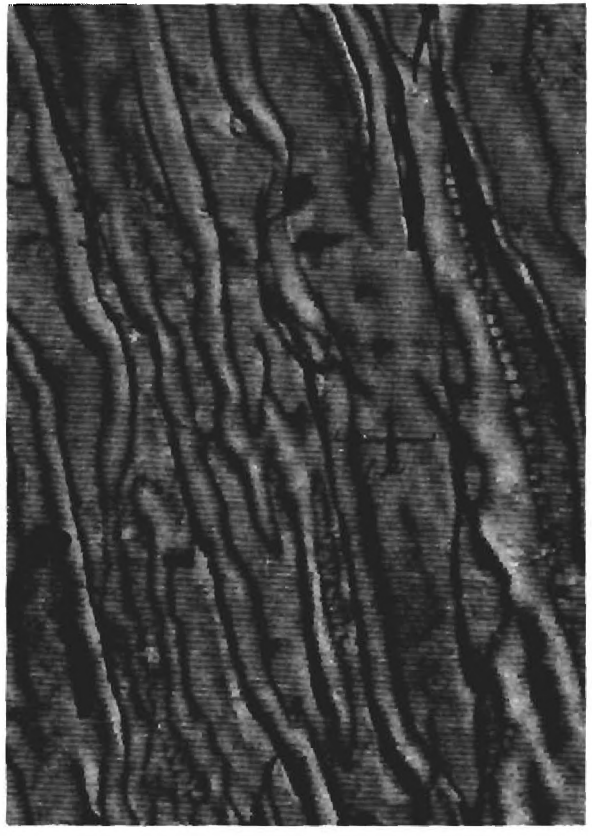

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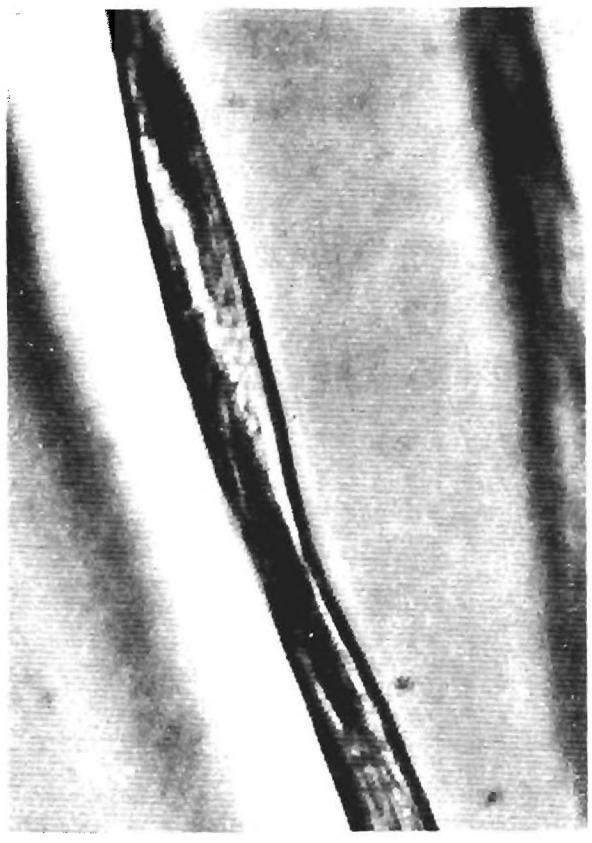
Figure 5. Continued, Optical and Electron Micrographs of Hand Ginned Carolina Queen Cotton (Tyrone, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.



(a) 560X

(b) 560X

Figure 6. Optical and Electron Micrographs of Mechanically Ginned Carolina Queen Cotton (Tyrone, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.

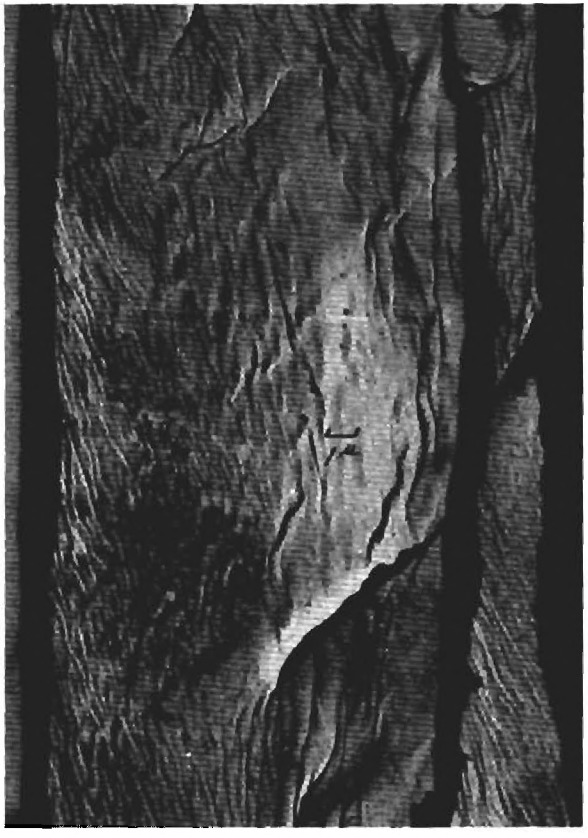


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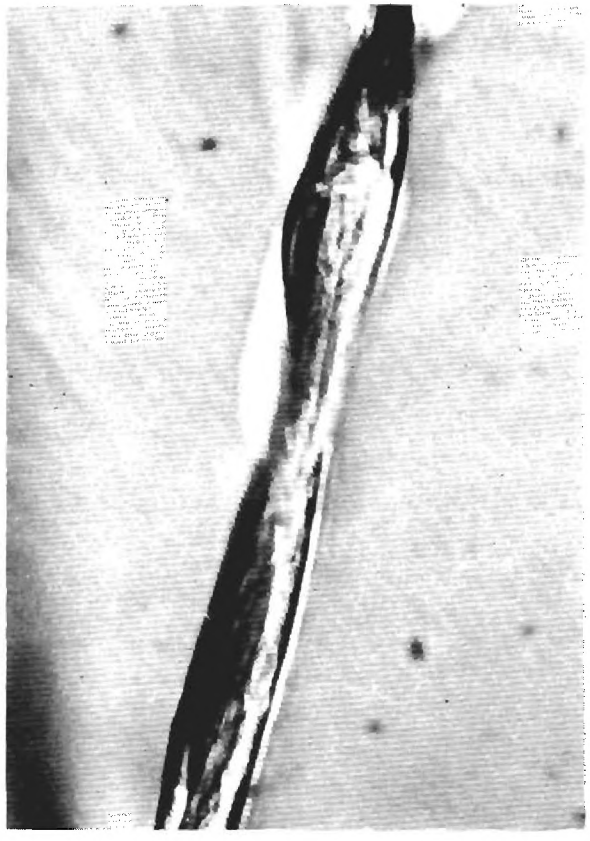
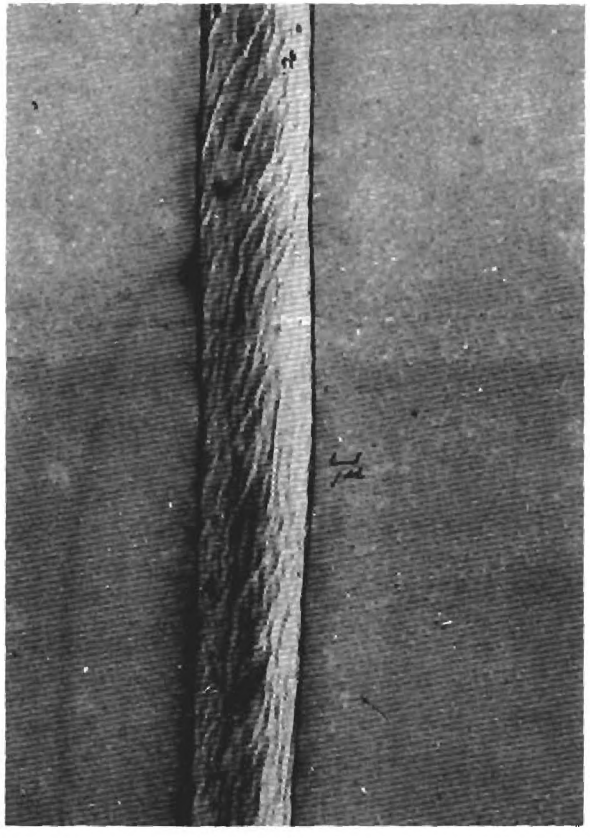
Figure 6. Continued, Optical and Electron Micrographs of Mechanically Ginned Carolina Queen Cotton (Tyrone, Georgia): a. Fiber Form; b. Section; c., d. Replicas of Surface.



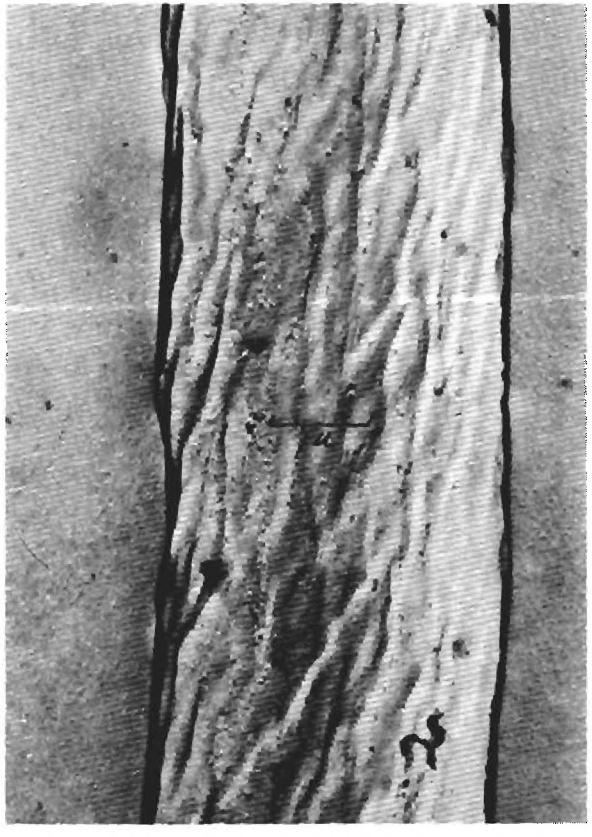
(a) 560X

(b) 560X

Figure 7. Optical and Electron Micrographs of Delfos Cotton, Supplied by J. N. Grant, USDA: a. Fiber Form; b. Section; c., d. Replicas of Surface.

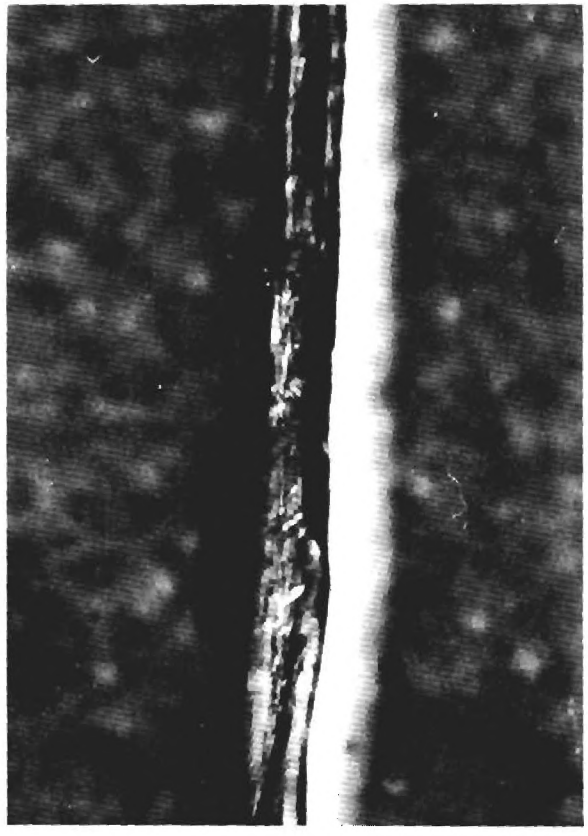


(c) 3,800X



(d) 12,800X

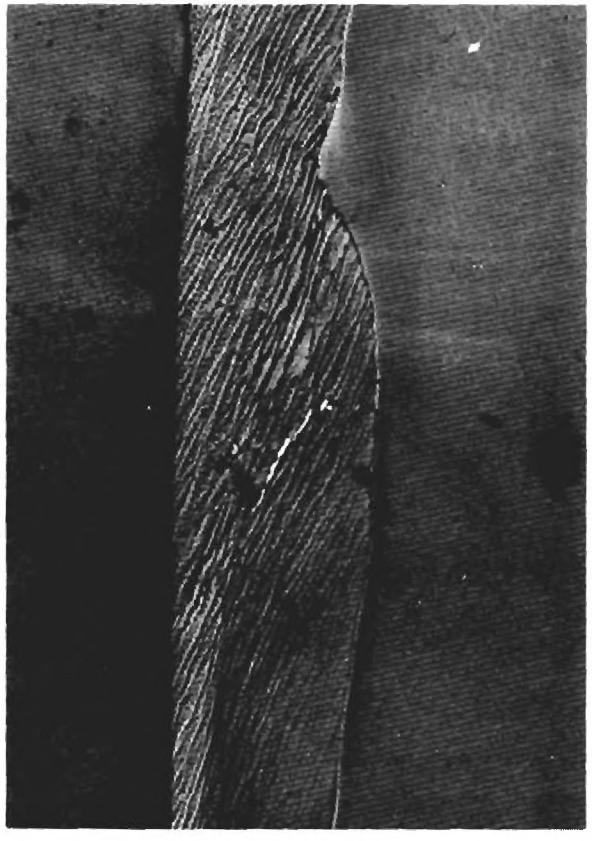
Figure 7. Continued, Optical and Electron Micrographs of Delfos Cotton, Supplied by J. N. Grant, USDA: a. Fiber Form; b. Section; c., d. Replicas of Surface.



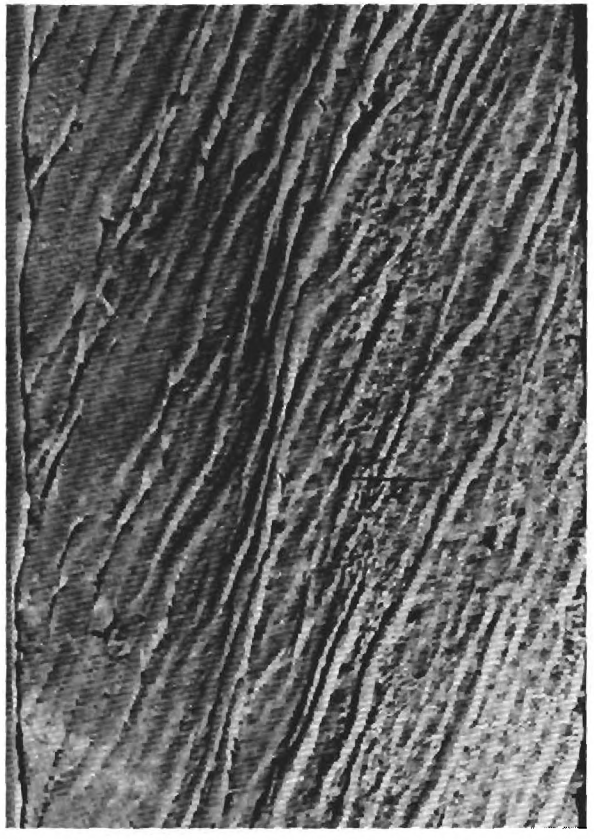
(a) 560X

(b) 560X

Figure 8. Optical and Electron Micrographs of Pima S-1 Cotton (Sliver), Supplied by J. N. Grant, USDA: a. Fiber Form; b. Section; c., d. Replicas of Surface.



(c) 3,800X



(d) 12,800X

Figure 8. Continued, Optical and Electron Micrographs of Pima S-1 Cotton (Sliver), Supplied by J. N. Grant, USDA: a. Fiber Form; b. Section; c., d. Replicas of Surface.

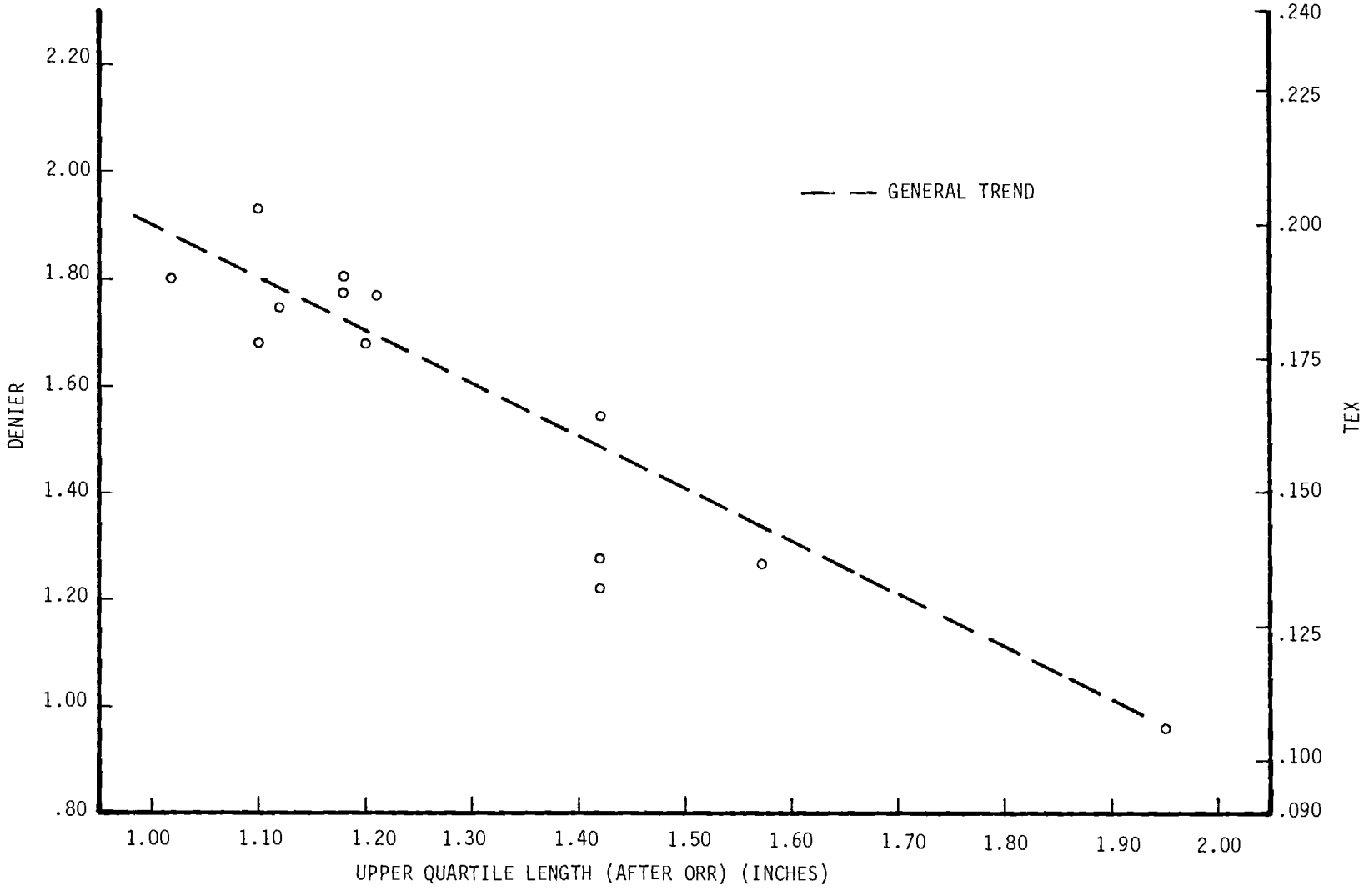


Figure 9. Fineness Versus Staple Length (Upper Quartile) for Various Cottons after Data by Orr et al.[9]

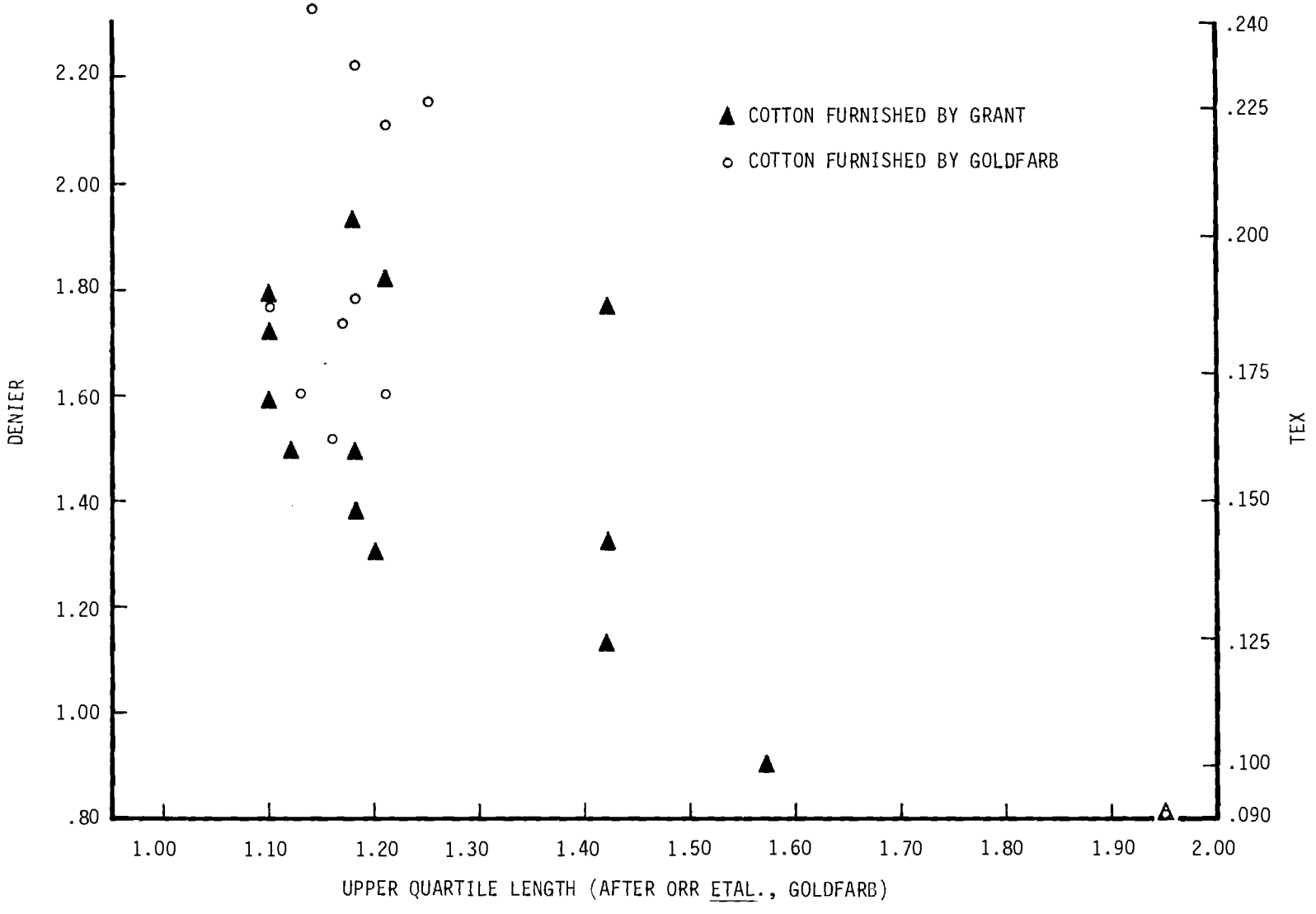


Figure 10. Calculated Fineness Versus Staple Length (Upper Quartile) for Various Cottons Using Quartile Length Data by Orr et al.[9] and by Goldfarb[5].

5. Man-Made Fibers

Examination of man-made fibers revealed many cross sectional shapes other than circular. Examples of Avisco Rayon, Enka Rayon, Verel Bright Modacrylic, Orlon 72 Acrylic, and Nylon are shown in Figures 11, 12, 13, 14, and 15. Some of these shapes should possess interesting frictional characteristics, especially if a circular section can be compared with a dumbbell or other configuration of the same material.

6. Wool

Sections and surface studies were also made of an unscoured and scoured wool. Figure 16 displays a typical photostudy of unscoured wool. The scales are readily visible in both the optical and electron micrographs.

7. Fiber Maturity Study

By the method described by Stoves[10] specimen of Empire WR, Carolina Queen, and Dixie King Cotton were treated and examined for dead and immature fibers. As a result of this treatment the mature fibers became cylindrical, the dead fibers remain convoluted and the partially mature fibers display characteristic in between the two extremes.

Using the equation

$$MC = (M + 0.6H + 0.4II),$$

where

MC = Maturity Coefficient

M = Percent Mature

H = Percent Half Mature

I = Percent Immature

reported by Pillay and Shankaranavayana[11] the maturity index was calculated for hand ginned specimens of Empire WR, Dixie King and Carolina Queen Cotton as shown in Table 15.* These data illustrate that the maturity coefficients of the cottons examined were in the range 0.74 to 0.79. The one mechanically harvested specimens, Carolina Queen, exhibited the lowest maturity coefficient. However, this may only be a coincidence.

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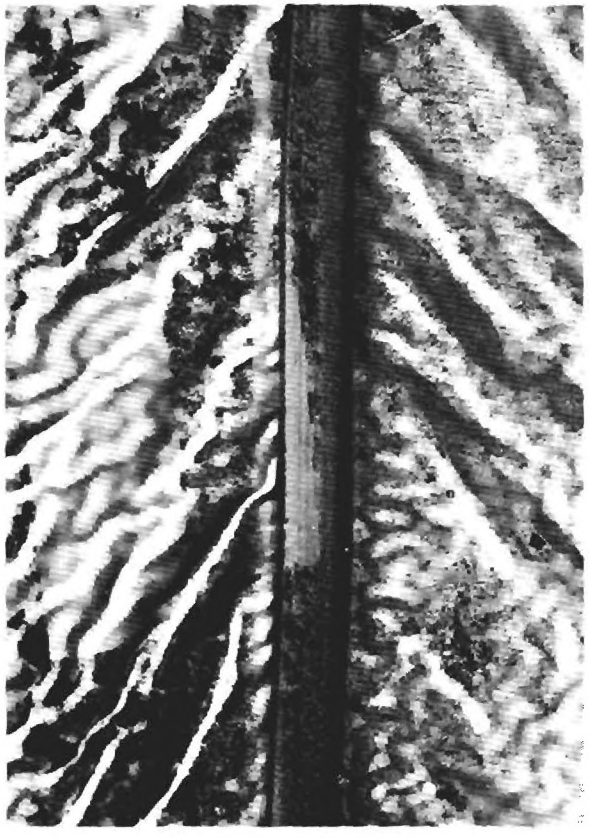
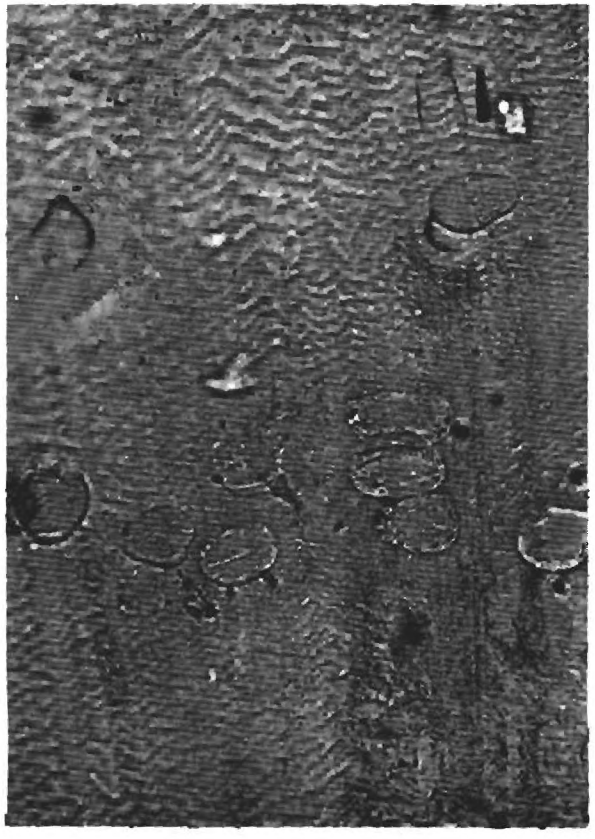
* See Appendix.

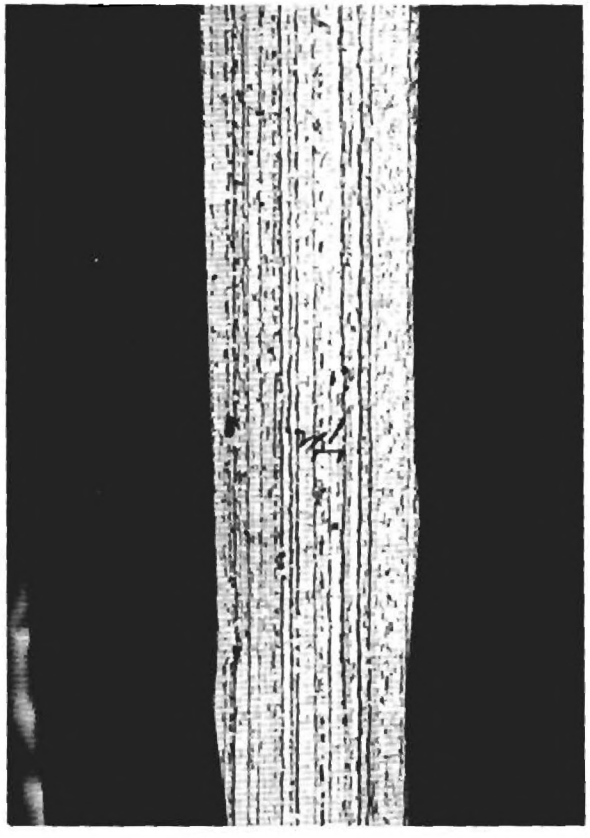


(a) 560X



(b) 560X

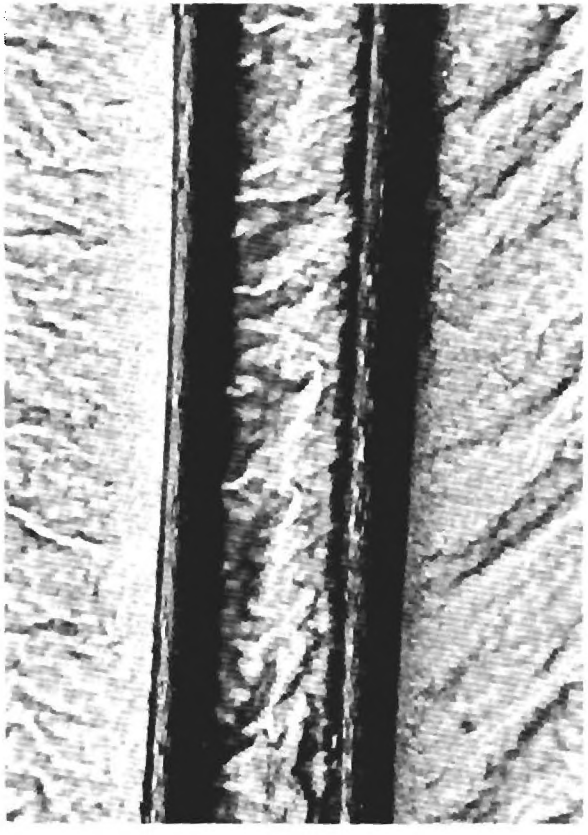
Figure 11. Optical and Electron Micrographs Avisco Bright Fiber 40 Rayon: a. Fiber Form; b. Section; c., d. Surface Replicas.



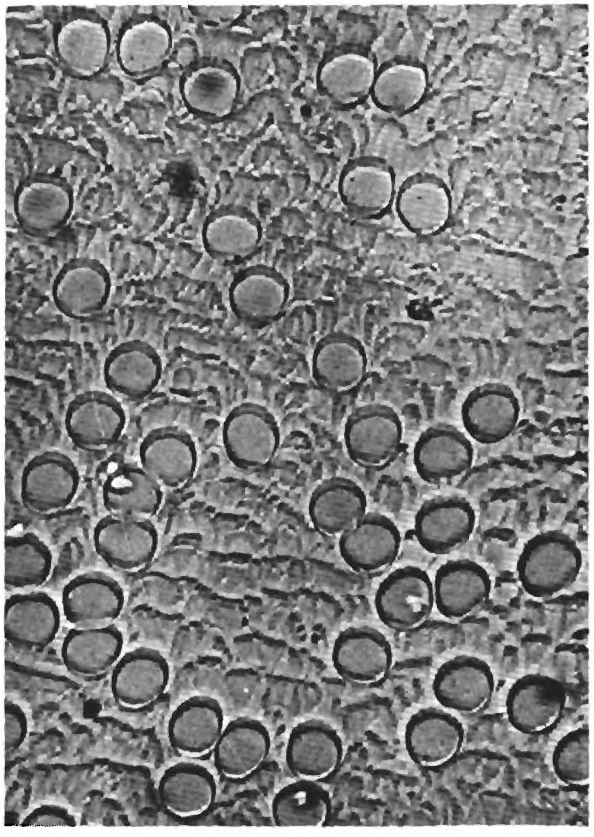
(c) 3,800X

(d) 12,800X

Figure 11. Continued, Optical and Electron Micrographs Avisco Bright Fiber 40 Rayon: a. Fiber Form; b. Section; c., d. Surface Replicas.

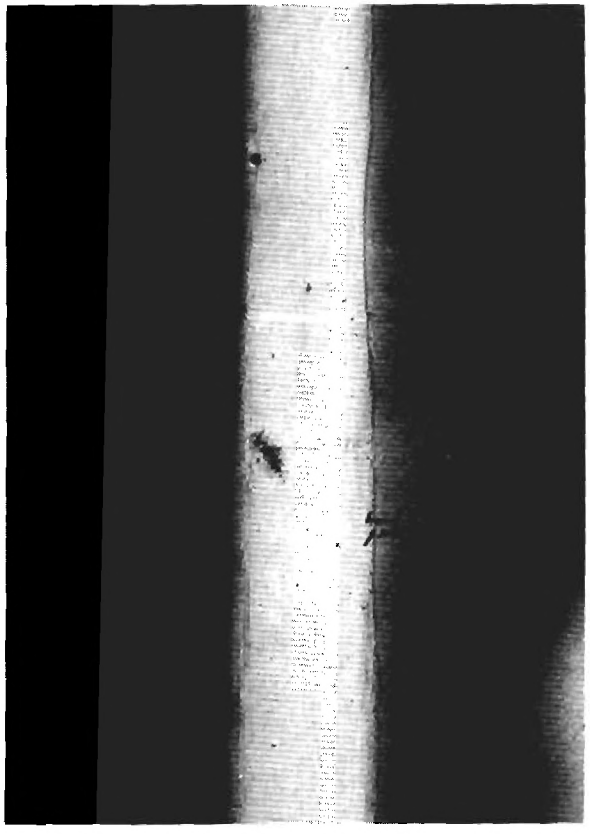


(a) 560X

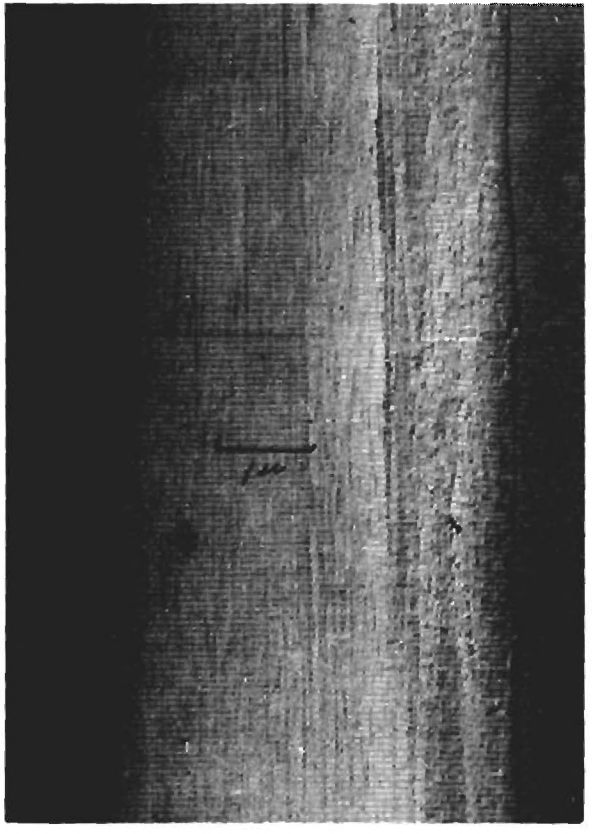


(b) 560X

Figure 12. Optical and Electron Micrographs of Enka Rayon: a. Fiber Form; b. Sections; c., d. Surface Replicas.

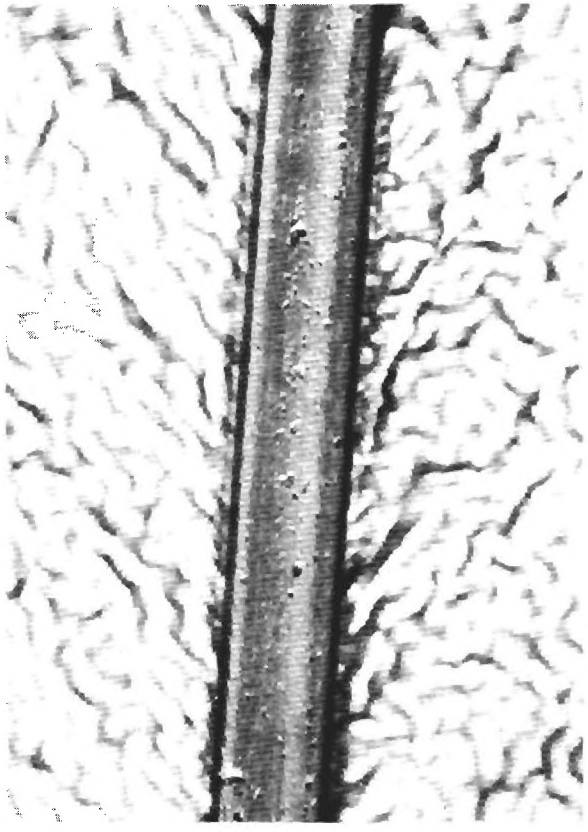


(c) 3,800X

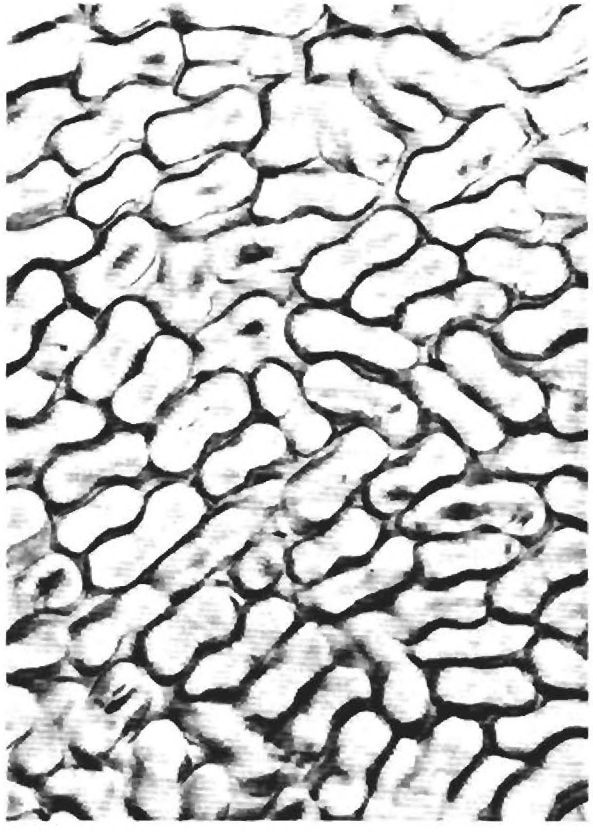


(d) 12,800X

Figure 12. Continued, Optical and Electron Micrographs fo Enka Rayon: a. Fiber Form; b. Section; c., d. Surface Replicas.

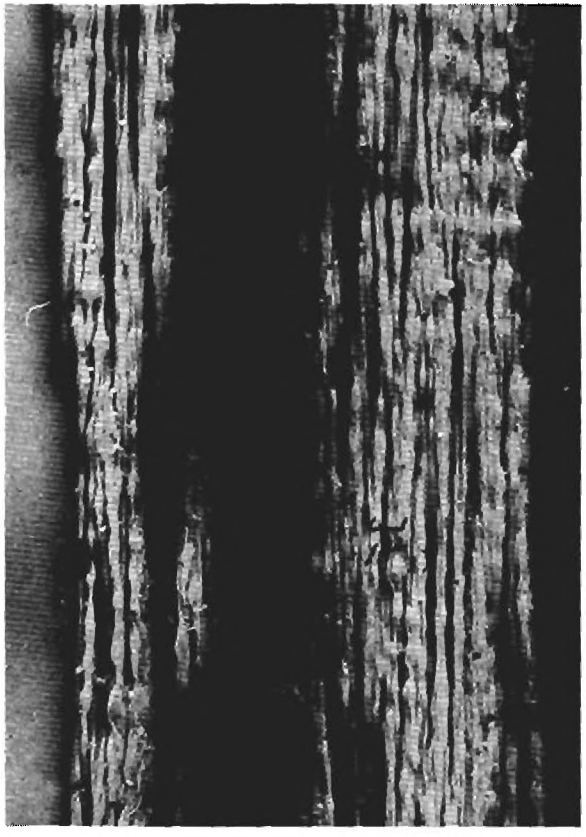


(a) 560X



(b) 560X

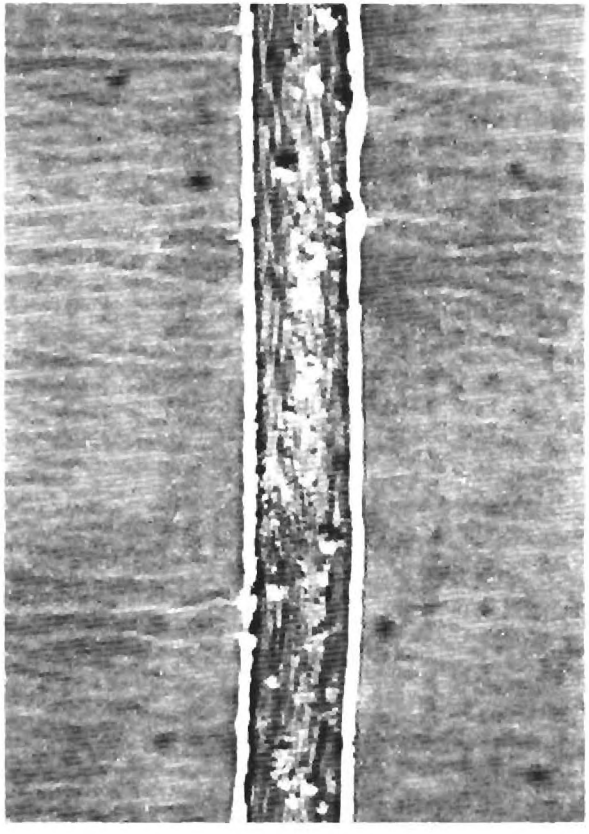
Figure 13. Optical and Electron Micrographs of Bright Verel Modacrylic: a. Fiber Form; b. Sections; c., d. Surface Replicas.



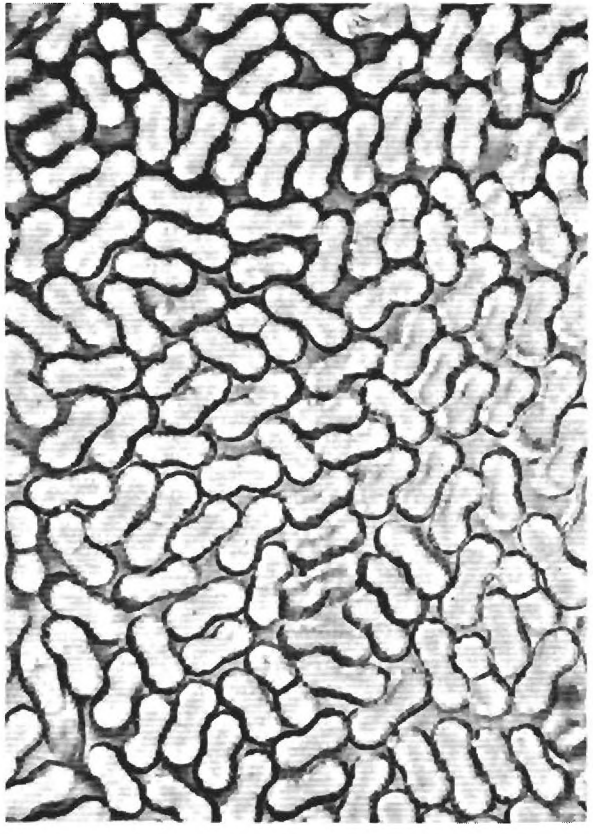
(c) 3,800X

(d) 12,800X

Figure 13. Continued, Optical and Electron Micrographs of Bright Verel Modacrylic: a. Fiber Form; b. Section; c., d. Surface Replicas.



(a) 560X



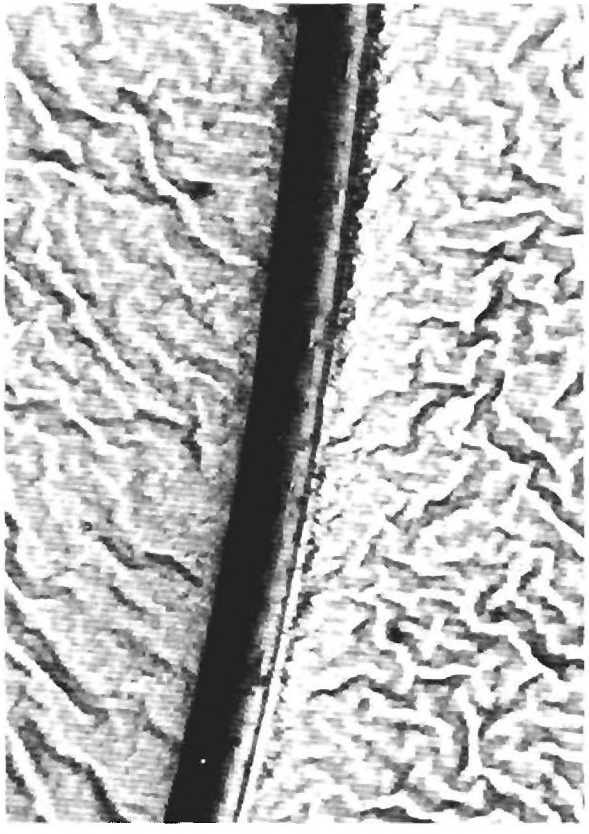
(b) 560X

Figure 14. Optical and Electron Micrographs of Orlon 72 Acrylic: a. Fiber Form; b. Sections; c., d. Surface Replicas.

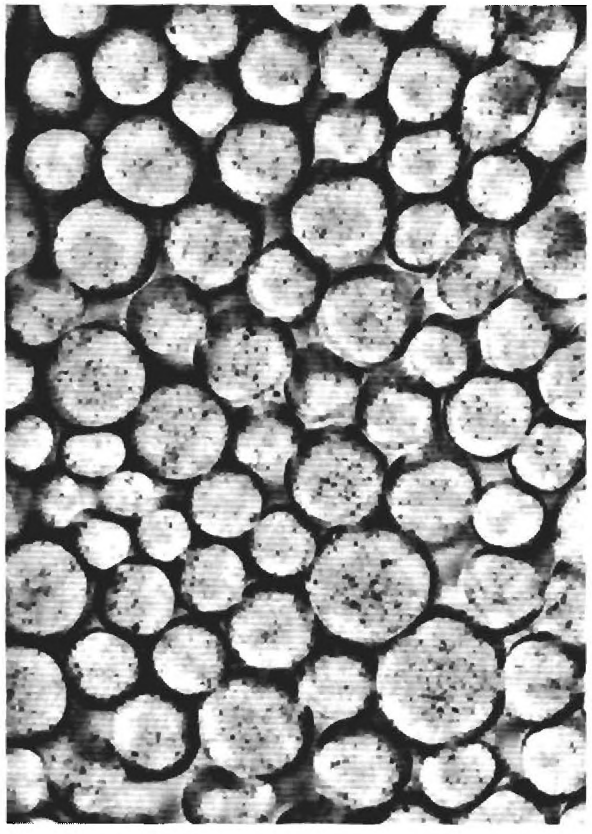
(c) 3,800X

(d) 12,800X

Figure 14. Continued, Optical and Electron Mocrographs of Orlon 72 Acrylic: a. Fiber Form; b. Sections; c., d. Surface Replicas.



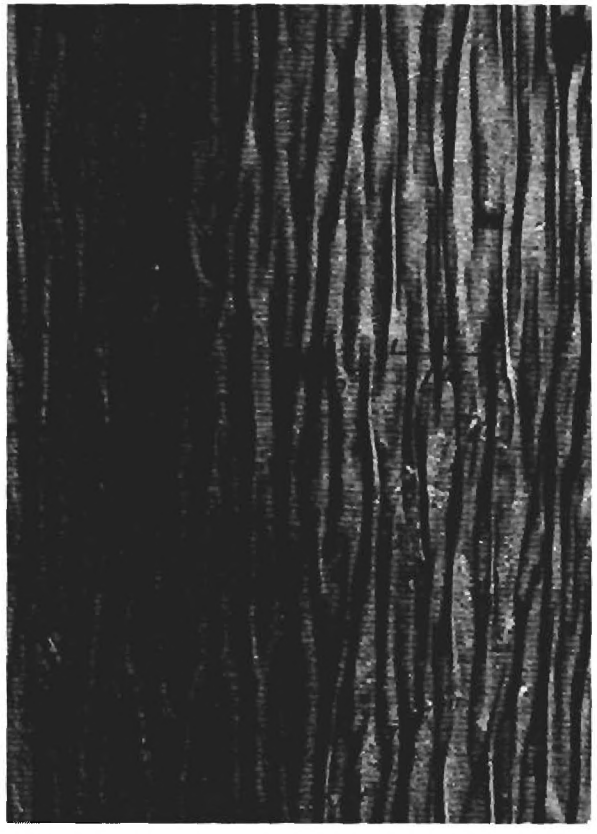
(a) 560X



(b) 560X

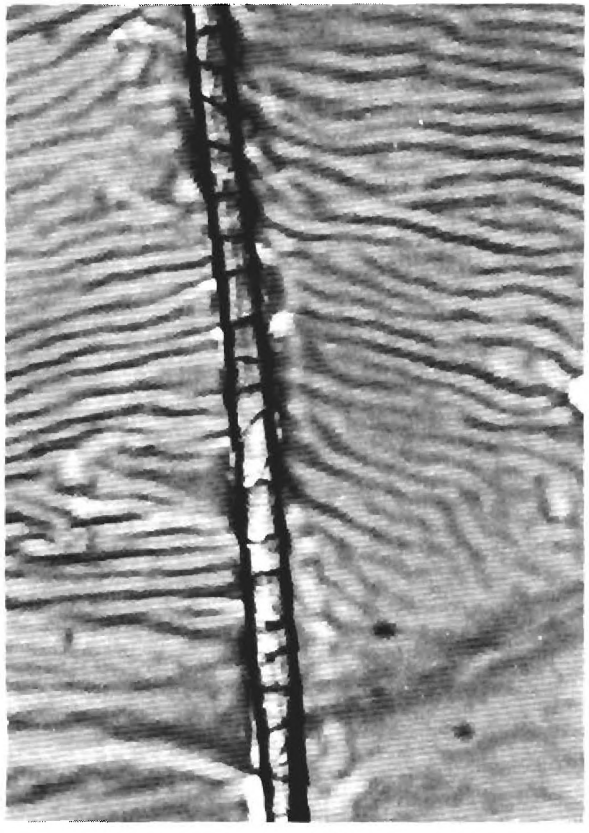
Figure 15. Optical and Electron Micrographs of Semi-dull du Pont Nylon: a. Fiber Form; b. Sections; c., d. Surface Replicas.

(c) 2,900X

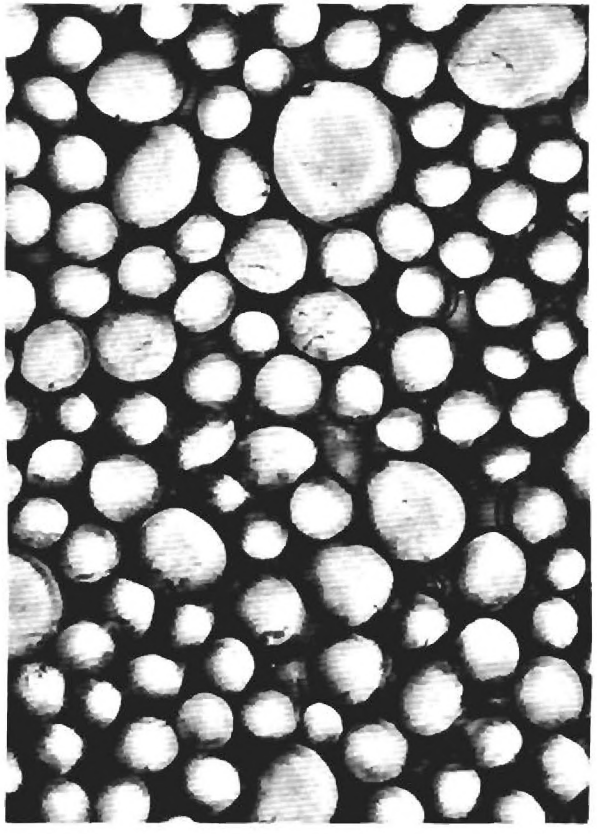


(d) 12,800X

Figure 15. Continued, Optical and Electron Micrographs of Semi-dull du Pont Nylon: a. Fiber Form; b. Sections; c., d. Surface Replicas.



(a) 560X



(b) 560X

Figure 16. Optical and Electron Micrographs of Unscoured Wool: a. Fiber Form; b. Sections; c., d. Surface Replicas.

(c) 2,900X

(d) 12,800X

Figure 16. Continued, Optical and Electron Micrographs of Unscoured Wool: a. Fiber Form; b. Sections; c., d. Surface Replicas.

8. Effects of Temperature Cycling on Convolution Count

Using the method of Betrabet[12] convolution counts over the 5 mm central region of cotton fibers were made before and after cycling to various temperature plateaus in a simulated gin drier constructed from a hot air gun and a pyrex tube as shown in Figure 17.* One end of the fiber was fastened to a glass slide by means of a silicone rubber adhesive, and convolution counts were made by means of a microscope before heating, 15 minutes after heating to the particular temperature plateau for 30 seconds, and 24 hours subsequently. Figure 18 depicts the changes observed for the median convolution count of approximately 75 fibers examined for each data point. It will be noted that increases were observed in the convolution count for all fibers heated and examined 15 minutes later (possibly due to drying effects at the lower temperatures) but only for those heated above 120°C (248°F) existed after 24 hours. The temperature of this semipermanent change agrees well with that reported by Berriman.[13] Unfortunately changes in total length for these fibers were not measured. If we can assume the convolution count increase is the result of fiber shrinkage a change in length up to 27% might be expected. Changes of this nature are related to high temperatures frequently used in the ginning process and indicate semi-permanent changes in the convolution count, surface configuration, and the length.[14] These reports imply that the cross sectional dimension is changed. However, this parameter was not examined.

The changes observed and suggested are sufficient to cause changes in character of the fiber which are of importance in textile processing.

Heating to 200°C, for only 2 minutes, caused darkening and apparent partial degradation of the fiber.

9. Summary

A catalogue of fiber photographs has been prepared and published as a thesis. Uses of cross sectional data to determine denier and the relation of denier to fiber upper quartile length has been exhibited. Maturity

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* See also paragraph F,3,a, Section III.

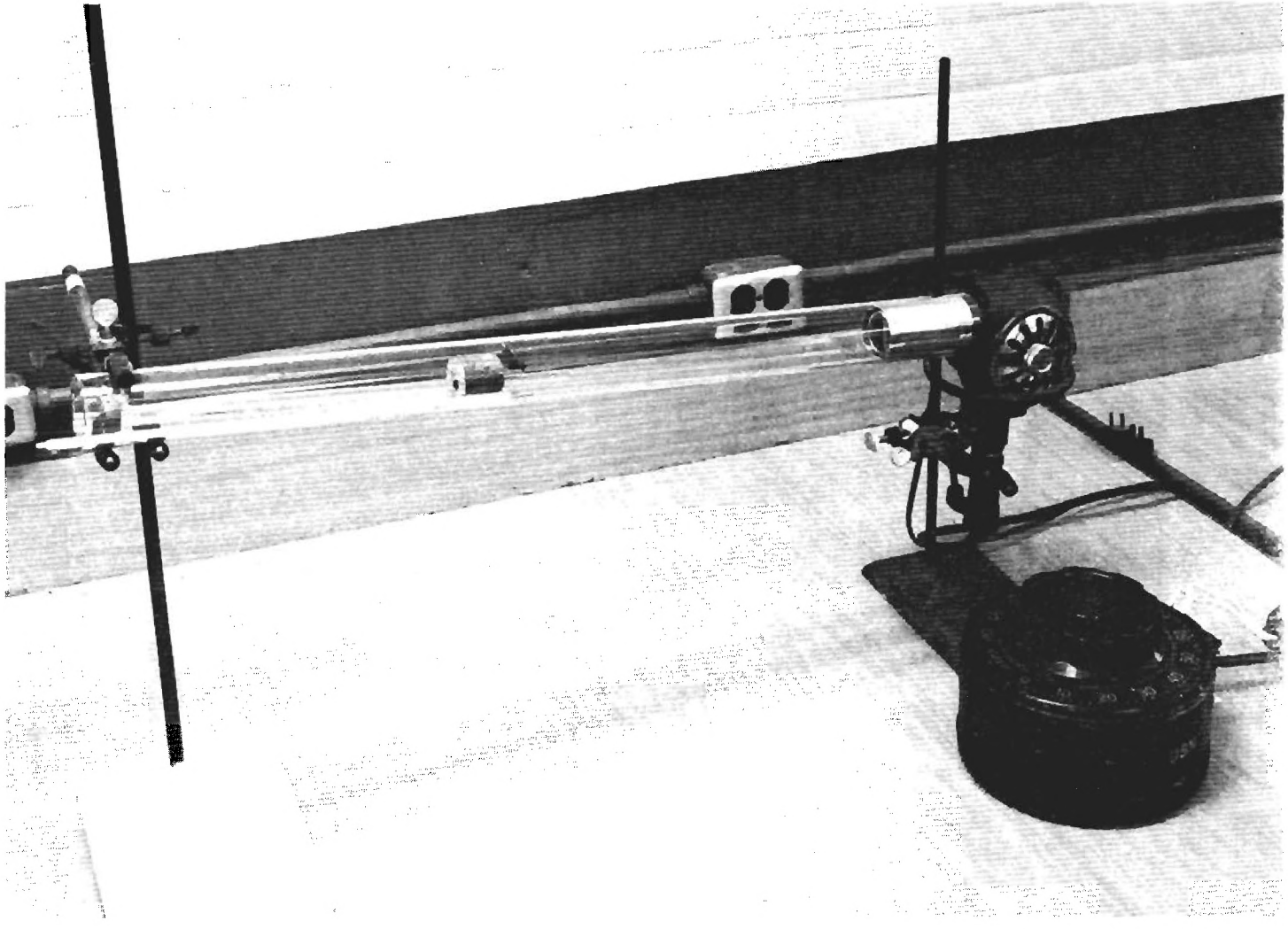

Figure 17. Gin Dryer Simulator.

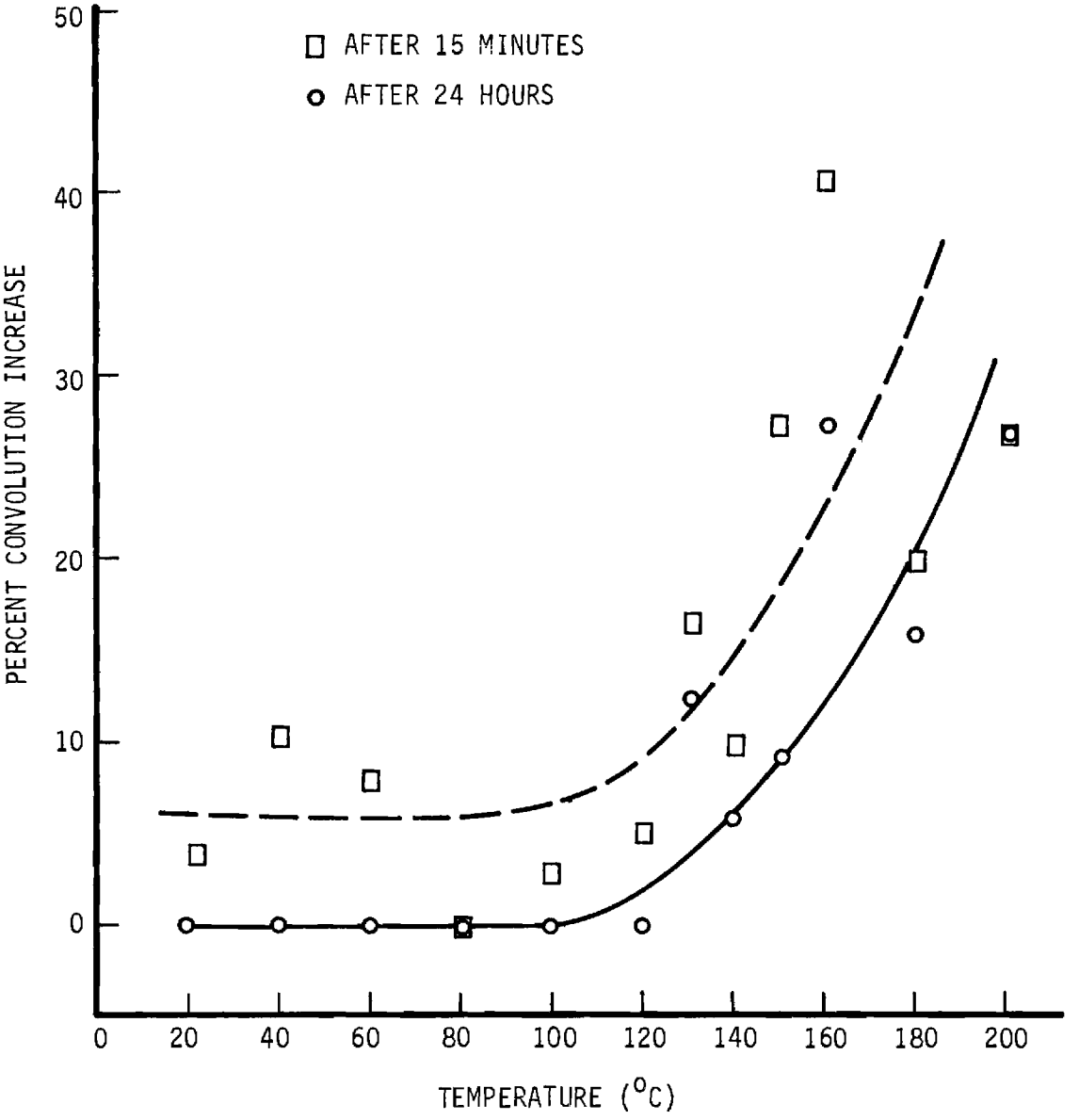

Figure 18. Convolution Count Changes Versus Temperature to which Cycled for Empire WR Cotton after 15 Minutes and after 24 Hours at Room Temperature Respectively.

indices of Empire WR, Carolina Queen, and Dixie King cottons were found to be in the range 0.74 to 0.79, the lower value being for a mechanically harvested specimen of Carolina Queen cotton. Temperature cycling Empire WR cotton fiber to temperature plateaus in the range 50° to 200°C, in order to simulate treatment received in gin driers, revealed that a semipermanent change in convolution count occurred after heating the fibers to 120°C (248°F) for 30 seconds. This action is indicative of change in surface configuration and possibly of length and diameter. All of these possible actions are undesirable and can be expected to affect the processing of the cotton deleteriously. Hence, it is undesirable from the standpoint of the fiber dimension change to employ temperatures above 240°F in gin driers.

C. FRICTIONAL APPARATUS AND MEASUREMENT

1. General

The frictional apparatus described in Semiannual Report No. 2, has been employed for measurements of the effects on the coefficient of friction of the tensile force exerted on fibers during the mounting process and for additional studies on the effects of the normal force between the contiguous fibers. Some studies of the effects on the coefficient of friction of temperature cycling cotton fibers in a gin dryer simulator were also conducted.

A second frictional apparatus, useful in the normal force range 2 to 20 mg, has been constructed. This unit is provided with an electromagnetically applied normal force control and a servo-balanced frictional force measurement system. Preliminary experiments conducted with the latter apparatus are reported.

2. Analysis of Frictional Motion by Cinemaphotography and Stereomicroscopy

Both black and white and color motion pictures were made of the operation of the principal frictional apparatus employing the high inertia (12,000 gm cm^2) arm.

At first, the camera was run at about 600 frames per second but 64 frames per second was found to be a sufficient rate. Films were made of the behavior of the instrument while employing pairs of cotton and nylon fibers, respectively, at fiber traverse rates of 0.11 and 1.02 mm/sec. The "stick-slip" motion was readily observable and was more exaggerated at the higher traverse rate. In the stick-slip motion the lower fiber, on the servo-controlled galvanometer arm, was deflected a small amount from its zero position by the traversing fiber. The photo-diode sensor detected the amount and generated a restoring counter torque by means of its amplifier. When the counter torque equaled the shear strength of the contact area the fiber fixed to the galvanometer arm returned rapidly to zero. The distance of the slip was dependent on the rate of drive and the magnitude of the stick, but appeared to be up to about 1.5 mm on occasion. This, of course, for cotton means that the slip may traverse several convolution lengths each time, i.e., up to 5 or 6.

Examination of bounce of the arm indicated that this was very low at low fiber traverse rates but was much more severe at the higher traverse rates. When it was severe, slips always appeared to occur during the upward bounce which appreciably reduced the normal force between the fibers. For most measurements at the normal force (20 mg) and traverse rate (0.11 mm/sec) employed tracking of the fibers appeared to be excellent.

Observations made under similar circumstances by use of a stereomicroscope confirmed the behavior described above. The traversing fiber was also observed to twist back and forth on its axis as the convoluted ribbon moved across the fixed fiber. For one severe stick a small particle of spattered wax (from the wax mounting) was observed on the fiber.

The motion picture* and stereomiscroscopic examinations of the fiber to fiber frictional behavior presented graphic examples of the stick-slip action and a better insight into the operational dynamics of the current instrument.

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* A copy of the motion picture was furnished SURDD, USDA, New Orleans, Louisiana, and it was also exhibited at the May 1966 meeting of the Fiber Society in Williamsburg, Virginia.

3. Effects of Fiber Tensile Mounting Force

Guthrie and Oliver[7] reported an increase in the frictional force of approximately 50% for an increase in the tensile force employed in mounting fiber pairs of viscose rayon. An increase of 50% occurred over the range 400 mg to 1600 mg of tensile force. The maximum tension employable is dependent on fiber strength at the long gauge or span to that normally used, > 0.5" for friction measurements. The increase discussed would represent a change in μ_k from about 0.12 to 0.19 for viscose rayon at 125 mg normal force according to the data of Guthrie and Oliver.

In order to examine the effects of tension more thoroughly measurements of the coefficient of friction of 15 Empire WR cotton fiber pairs were made at the Georgia Institute of Technology for fibers mounted with tensile forces of 125, 425, 825 and 1125 mg, respectively.

Forces were estabilished by fastening one end of the fiber to its mount with sealing wax. With the fiber in the vertical position, and parallel to the mount base, a weight of proper amount was attached to the fiber. Its end was then fastened in position with Duco cement which was allowed to dry while the weight was attached.* Three sets of data were obtained for each fiber pair. Typical data for one run are shown in Table 2 and the data obtained for all runs are plotted in Figure 19 to exhibit the variation.

Here it will be observed that the value of μ_k, measured at a normal force of 20 mg, decreased from 0.356 to 0.236 as the tensile force was increased from 125 to 1150 mg. This action is contrary to the action previously reported by Guthrie et al. Similarly the values of μ_s decreased from 0.647 to 0.483.

---

* Actually these numbers do not represent the final tension; only that established before cementing. A correct number can only be obtained by maintaining a tension measuring device in series between the fiber and the mounting posts. The tension will also be changed considerably on the application of the normal force between the fibers. However, it may be estimated that this change will be less than 200 mg for the normal force range 20 to 40 mg.

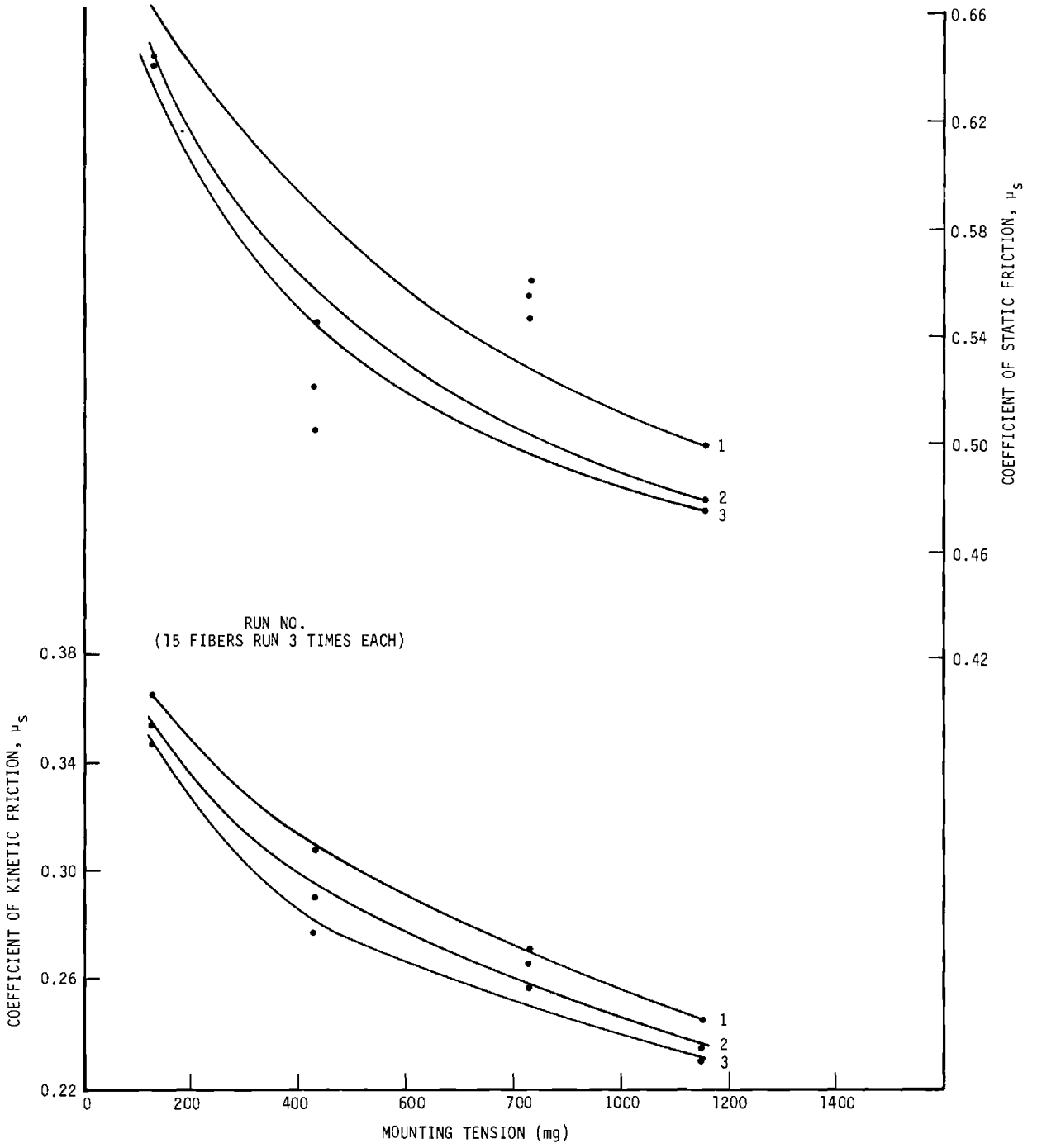

Figure 19. Variation of Coefficients of Kinetic and Static Friction of Empire WR Cotton Fibers with Tensile Force Employed for Mounting.

Table 2. Friction Versus Tension Data for Empire WR Cotton Fibers

Part I: 425 Milligrams Tension

Fiber Number	Coefficient of Kinetic Friction (μ_k)			Coefficient of Static Friction (μ_s)		
	a	b	c	a	b	c
1	.278	.248	.291	.523	.465	.474
2	.253	.252	.241	.453	.394	.442
3	.182	.251	.206	.413	.463	.415
4	.262	.270	.264	.493	.443	.458
5	.303	.321	.258	.546	.578	.485
6	.314	.258	.257	.513	.466	.508
7	.450	.505	.390	.781	.837	.581
8	.421	.348	.352	.639	.615	.677
9	.402	.324	.308	.677	.628	.547
10	.355	.333	.333	.620	.571	.594
11	.268	.257	.266	.522	.514	.547
12	.284	.261	.258	.496	.478	.503
13	.310	.294	.256	.529	.485	.475
14	.210	.197	.222	.429	.398	.400
15	.322	.252	.251	.528	.463	.452
Average	.308	.291	.277	.544	.520	.504
Grand Average		.292			.523	
μ_s/μ_k	1.76	1.78	1.83	Average	1.80	

Normal Force = 20 ± 1 mg.

Table 2. Friction Versus Tension Data for Empire WR Cotton Fibers

Part II: Data Summary for Various Tensions

Tension (mg.)	μ_k (Avg.)	μ_s (Avg.)	μ_s/μ_k (Avg.)
125	.356	.647	1.82
425	.292	.523	1.80
825	.265	.552	2.15
1150	.236	.483	2.08

*Fiber was fastened at one end to holder. A weight equivalent to the desired value of tension was suspended from the other end of the fiber which was in a vertical position. The fiber was fastened at the loose end by cementing (Duco usually) to the fiber support. The true tension after mounting is not known but will be examined subsequently.

An interesting point here is that the ratio μ_s/μ_k increased from 1.8 to > 2.1 at tension of 825 mg and higher. Examination of Figure 20 exhibiting photomicrographs of cotton fibers at various tensions exhibits the general straightening out of the fiber. However, at convolutions or reversals a sharp asperity now appears with little or no approaching incline. This results in snagging and the higher stick maxima observed.

An analysis of the problem reveals the following: the contiguous area of the fibers decreased as the tnesile force is increased and at low tensions the traversing fiber is considerably displaced from its axis; hence, it is continually climbing a small incline. One might, then, expect a decrease in the coefficient of friction as the fiber tension is increased, in agreement with the result obtained. The effect of the convolutions, however, is to increase the amplitude of sticks, but only for relatively short time periods. Hence we have the phenomena of high coefficient of static friction and lower coefficient of kinetic friction.

4. Effects of Successive Measurements with the Same Fiber Pair

During the course of the measurements made above and some other measurements reported subsequently the friction between fifteen fiber pairs was measured on three successive runs. In almost every instance repeated runs resulted in lowering of the coefficients of both static and kinetic friction by small increments.

Typical plots obtained from the tension data previously discussed are shown in Figure 21 for successive runs. It will be noted that decreases of approximately five percent occurred for each successive run and that an essentially uniform slope of decrease occurred in 3 of the 4 instances.

If the median value of μ_k is used for all 45 measurements in Table 2 our result is essentially 5% low. The value of μ_k at 425 mg, adopted as standard, is 0.308.

A subsequent measurement with the same pair of fibers was made for 13 data plots. A tabulation of these data are shown in Table 3. Here a comparison of data plots of Figure 22 taken on the 1st and 13th runs exhibit the permanence of major features. The actual values of μ_s and μ_k obtained for successive measurements are different but variations from the median

Table 3. Coefficients of Static and Kinetic Friction for 13 Successive Measurements of an Empire WR Cotton Fiber Pair*

Measurement No.	Coefficient of Kinetic Friction, μ_k	Coefficient of Static Friction, μ_s	Ratio μ_s/μ_k
1	0.252	0.510	2.02
2	0.272	0.458	1.69
3	0.263	0.406	1.55
4	0.261	0.403	1.55
5	0.234	0.388	1.66
6	0.242	0.388	1.60
7	0.274	0.374	1.38
8	0.261	0.408	1.57
9	0.241	0.415	1.72
10	0.262	0.417	1.59
11	0.268	0.416	1.56
12	0.245	0.392	1.60
13	0.272	0.430	1.58
(198A to 198M)			
Average	0.257	0.416	1.62

* Hand Ginned Cotton

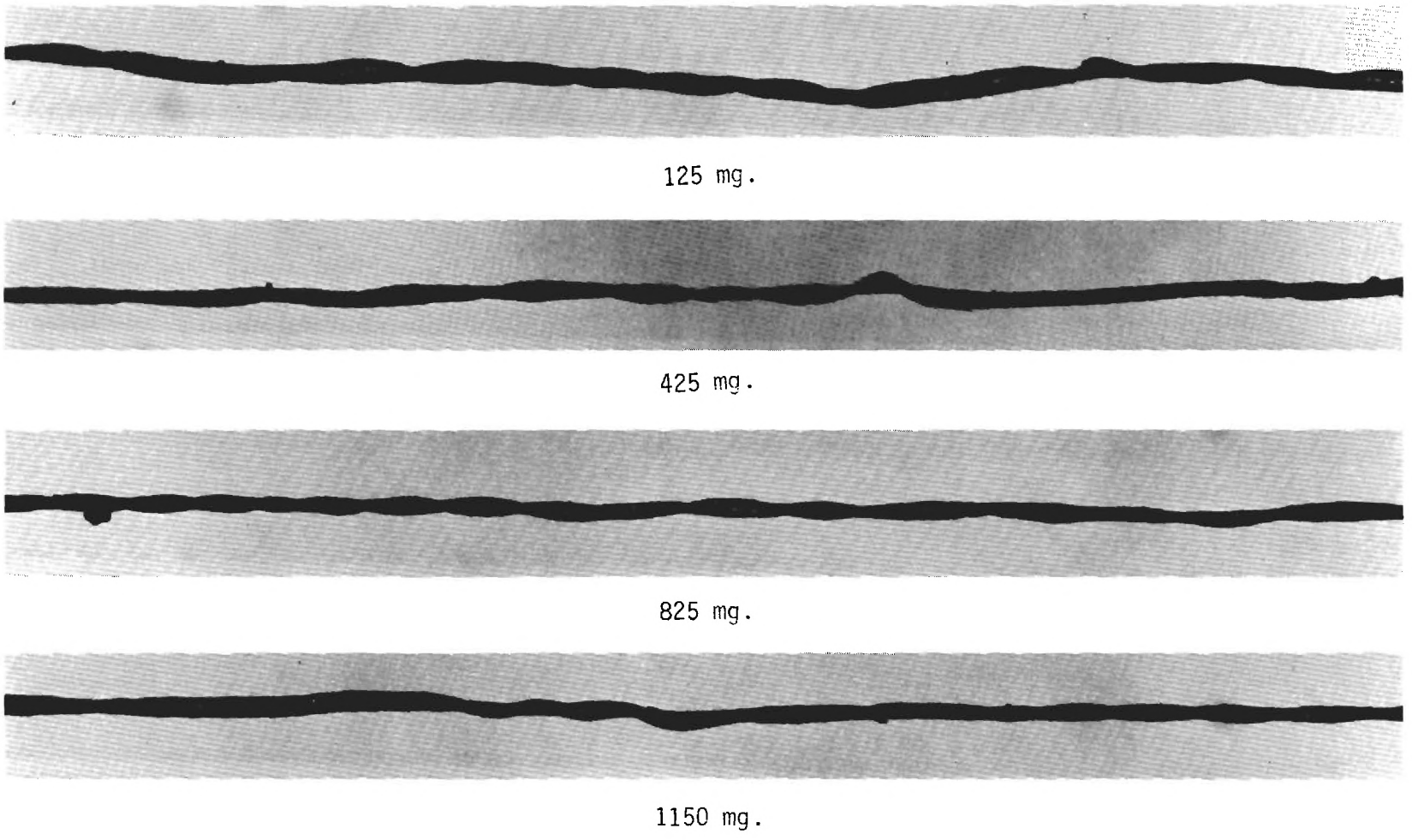

Figure 20. Photomicrographs of Empire WR Cotton Fibers at Mounting Tensions of 125, 425, 825, and 1150 mg.

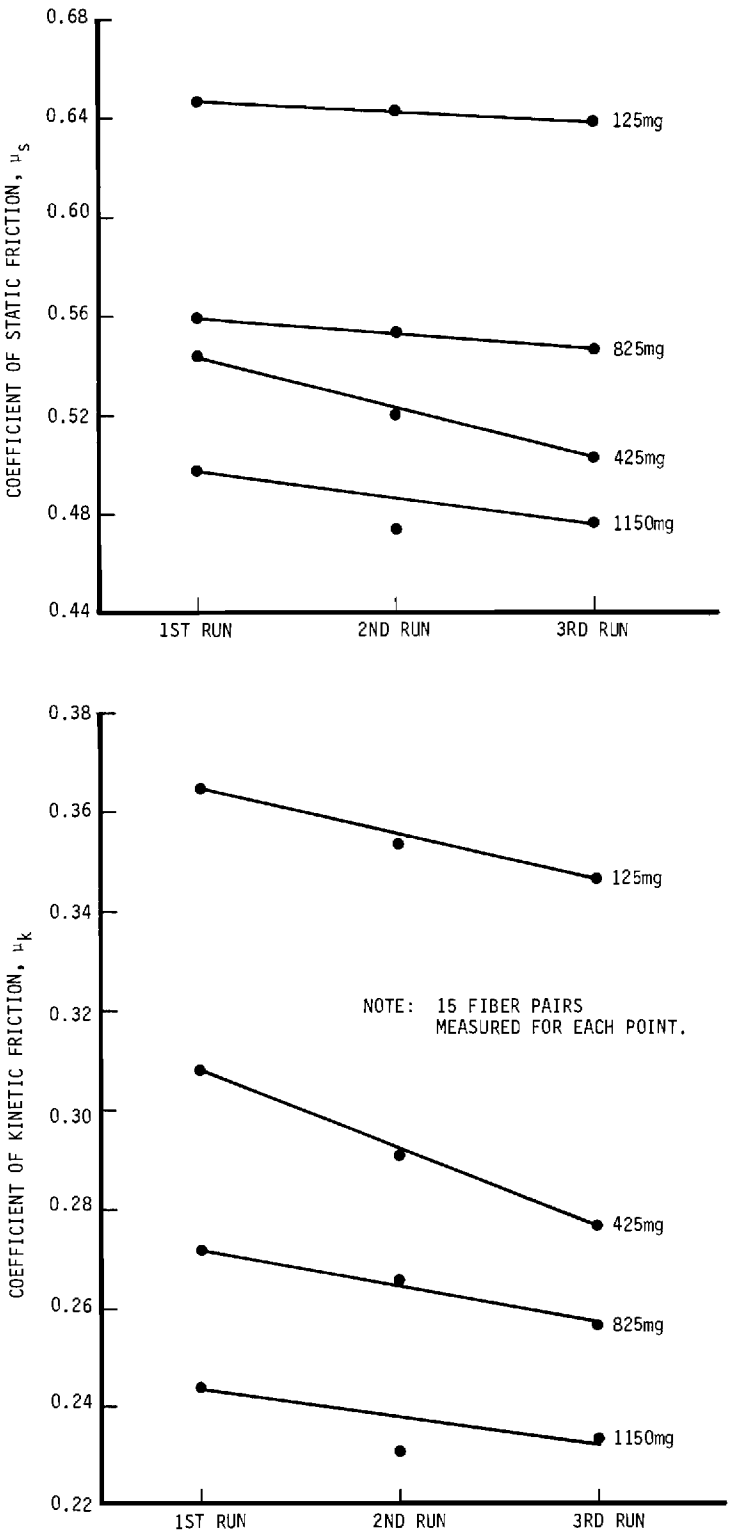

Figure 21. Variation of Coefficients of Kinetic and Static Friction of Empire WR Cotton Fibers with Tension and on Three Successive Runs with the Same Fiber Pairs.

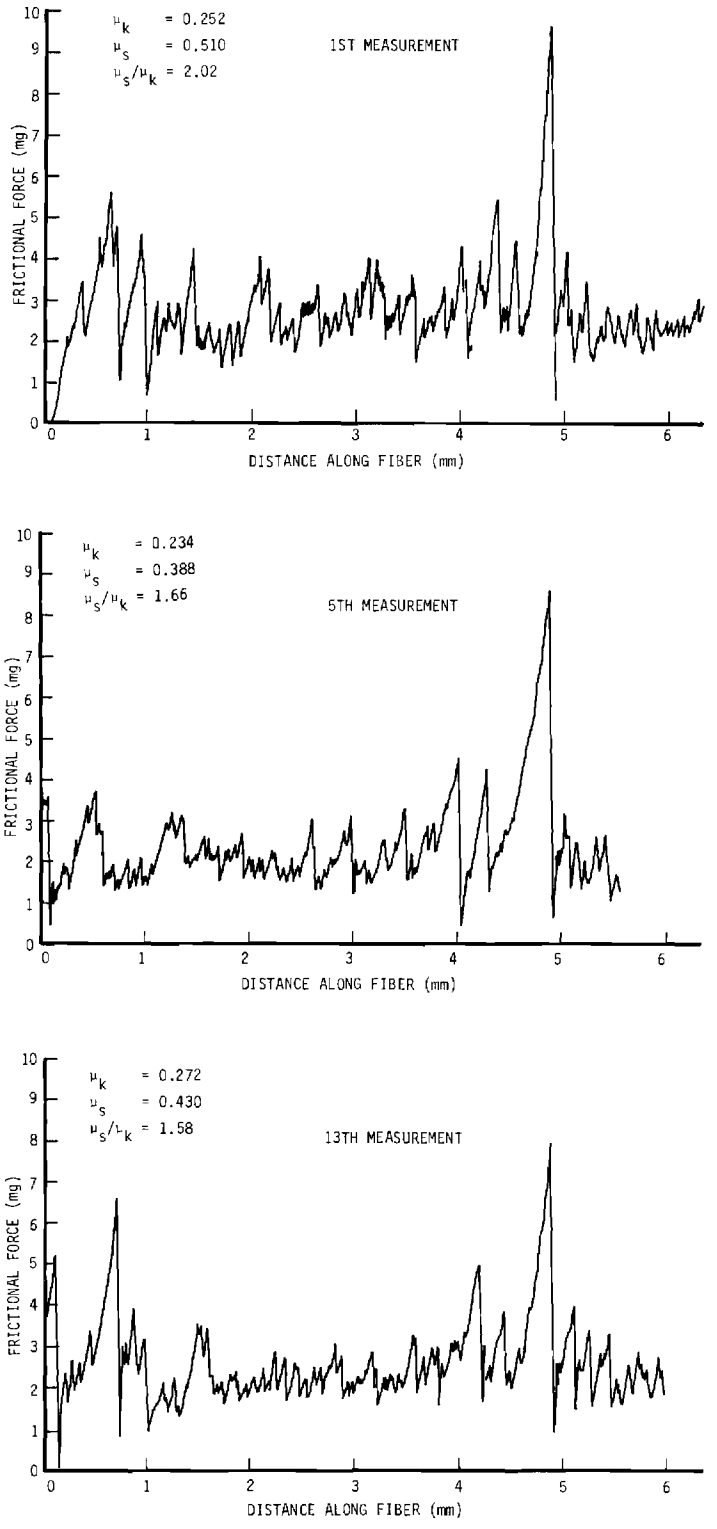

Figure 22. Friction Data Plots of 1st, 5th, and 13th Successive Measurements for an Empire WR Cotton Fiber Pair.

value for μ_k are only +0.017 and -0.023. The value μ_s changed markedly during the first three runs. Thereafter it fluctuated to +0.014 and -0.042 from the median value. Data obtained from successive measurements of the same fiber pair are generally biased negatively.*

5. Effects on Coefficient of Friction of Temperature Cycling Cotton Fiber

Since cotton fiber is subjected to temperature cycling during the drying process accompanying ginning, the effect of temperature cycling of the fiber on its frictional character was examined.

Specimens of cotton were heated in an oven to temperatures of 70°, 120°, 170°, and 220°C and the frictional parameters of typical fibers were measured. Nine fibers were measured three successive times at each temperature. A summary of the measurements is given in Table 4 and Figure 23.

It will be observed that there are small increases in the coefficients of kinetic and static friction as the drying temperature is increased. A large increase occurs at the 170°C (338°F) step.

Again small changes (generally reductions) are observed on successive runs with the same fiber but overall consistency is quite good. The ratio μ_s/μ_k is interesting in its constancy with a considerable reduction at the 220°C level, a temperature high enough to scorch the fiber. The value of 2.07 is high for 70°C and all thereafter. However, a number of large stick peaks was a characteristic of these curves. This fact is brought out by the higher μ_s/μ_k ratio. This higher value contrasts with the ratio of approximately 1.80 obtained for cotton in the tensile test series at 425 mg and 25°C. Hence, it appears that even low temperature heating (70°C) has affected the μ_s value of the cotton and the μ_s/μ_k ratio. Again we observe the relative probable importance of μ_s value in fiber behavior

6. Effects of Fiber Processing on Friction of Cotton Fibers

Frictional measurements have now been obtained for cotton fibers as seed cotton, hand ginned; ginned cotton from the standard bale; and after the successive steps opening, picking and carding.

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* Although this practice has proven undesirable it has been used heretofore because of difficulties encountered in fiber mounting and in obtaining a desirable quantity of measurements within a reasonable period of time.

Table 4. Variation with Simulated Drying Temperature of Frictional Coefficients of Empire WR Cotton

	μ_k				μ_s			
Run No.	1	2	3	Ave.	1	2	3	Ave.
*25° C	0.308	0.291	0.277	0.292	0.544	0.520	0.504	0.523
70° C	0.292	0.289	0.275	0.285	0.606	0.586	0.578	0.590
120	0.300	0.300	0.278	0.293	0.603	0.613	0.569	0.595
170	0.315	0.321	0.317	0.318	0.656	0.653	0.635	0.648
220	0.340	0.313	0.327	0.327	0.645	0.602	0.639	0.629

Ratio μ_s/μ_k

	1	2	3	Ave.
*25°	1.77	1.79	1.81	1.80
70	2.07	2.03	2.11	2.07
120	2.02	2.05	2.04	2.03
170	2.08	2.04	2.00	2.04
220	1.90	1.93	1.95	1.92

*These are values taken from Table 3, and were not run at same time.

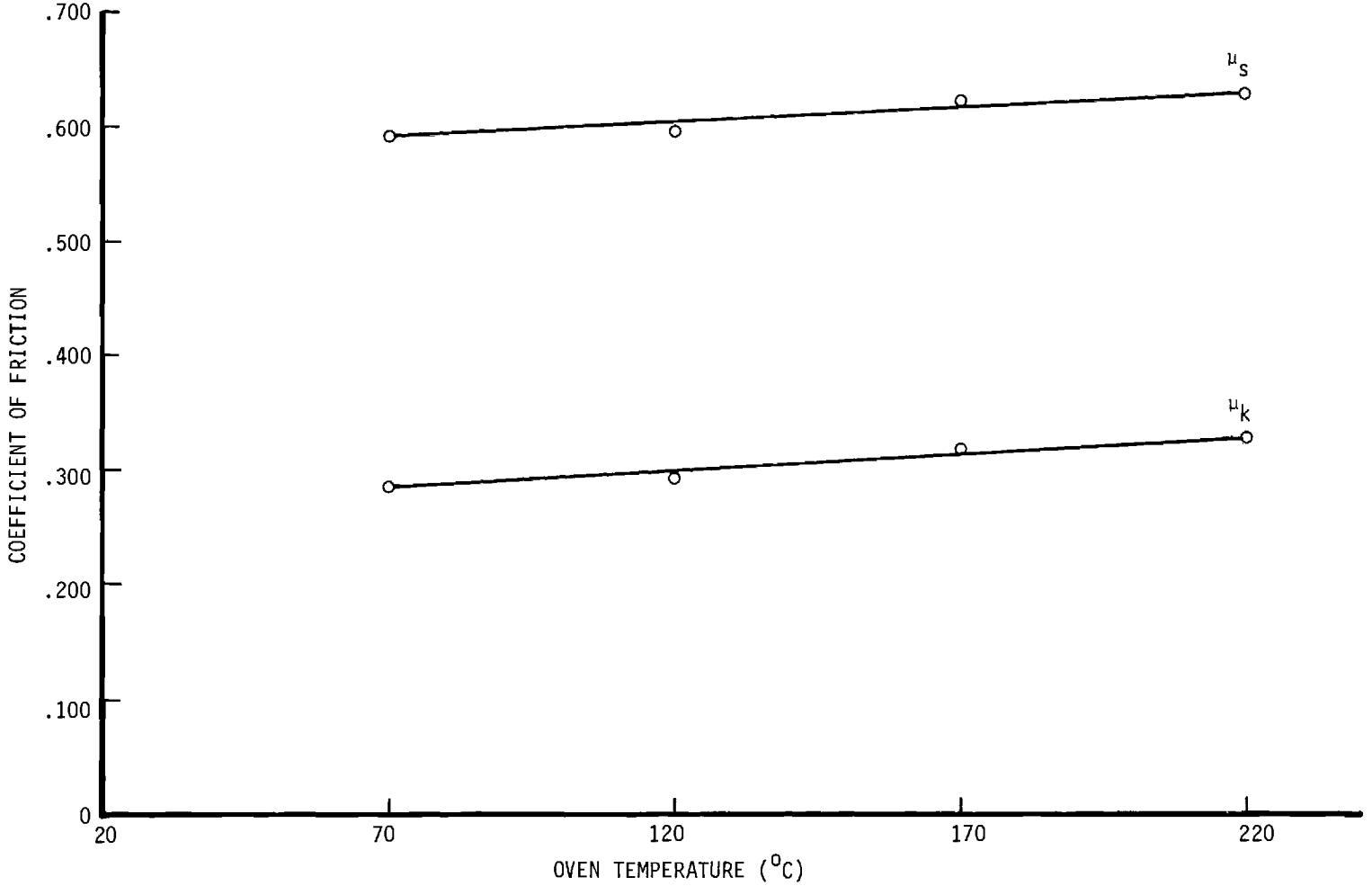

Figure 23. Variation of Coefficients of Static and Kinetic Friction of Empire WR Cotton Fibers Temperature Cycled to Selected Temperature Plateaus.

The value for hand ginned cotton is 0.262 as recently measured and for ginned cotton 0.292 as deduced from Bryant[3] tension data. Levy[6] has obtained the data for opening, picking, and carding.* However, he used in some cases a slightly higher tension than used for other measurements, 500 mg instead of 425 mg possibly contributing to a somewhat lower value for μ_k of ginned cotton than that reported by Bryant (0.275 compared to 0.292). The data are shown in Figure 24 and Table 5.

It will be noted in Figure 24 that there is a definite increasing trend in both the coefficients of static and kinetic friction as a result of processing. This increase is about 20 percent in both cases from the stage hand-ginned cotton to the carded fiber.

The leveling off of the static coefficient here is probably not real as in reviewing data curves obtained by Levy it was found that many high peaks were not included in the data because of fiber snagging. However, the snagging is a real behavior due to fiber damage. Hence, fiber damage is indicated by the frictional data in confirmation of reports of damage observed by the Congo Red Test (Section III, G, 3); and the μ_s values for the latter stages of the fiber processing are probably higher than those indicated in Figure 24.

The ratio μ_s/μ_k increased from 1.77 (hand ginned) to 1.82 (ginned and opened) and then decreased to 1.73 (picking and carding). The number 1.80 is surprisingly constant for cotton. The value 1.73 appears low and may be the result of dropping off the major peaks as indicated above.

7. Coefficients of Friction for Wet Cotton and Aluminum-Coated Cotton

A few fibers of cotton were saturated with water, mounted, and the friction between fiber pairs measured. Initial measurement for μ_k were about the same 0.263 but high peaks gave a high value for μ_s, 0.590 and the ratio of μ_s/μ_k was quite high, 2.30. As the water partially dried, the maxima, hence μ_s, greatly increased giving values of 0.620 and 0.710 respectively. Further work on this study appears desirable.

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* These measurements were performed by Graduate Research Assistant Howard E. Levy; see Section III, G, 2 this report. All measurements by Levy and Bryant use the three measurements per fiber pair method, resulting in a probable low reading of about five percent.

Table 5. Variation of Coefficients of Friction of Empire WR Cotton as a Result of Processing

Processing Stage	Mean Value	Standard Deviation	Percent Coefficient of Variation
Before Opening	.275	.017	6.29
After Opening and Cleaning	.289	.059	20.5
After Picking	.307	.074	24.3
After Carding	.317	.022	6.86

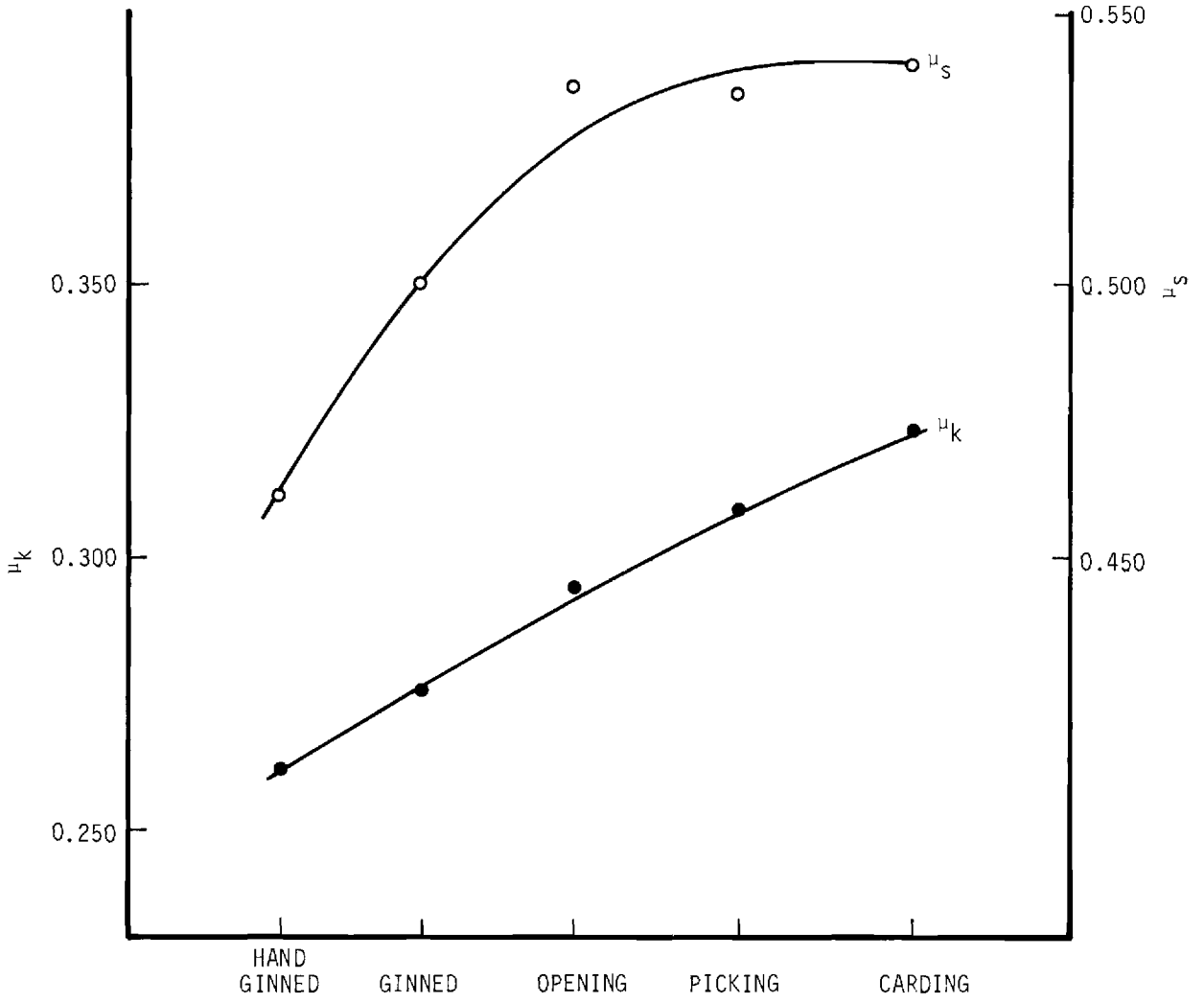

Figure 24. Variation of Coefficients of Static and Kinetic Friction of Empire WR Cotton Fibers as a Result of the Process Stages of Ginning, Opening and Cleaning, Picking, and Carding.

In a second experiment some cotton fiber from the standard bale was placed in a vacuum chamber; the chamber was evacuated; and a very thin film of aluminum (100 to 150 Å) was evaporated onto the fiber. The fiber thus partially coated, was removed and its frictional character was measured. Data obtained are shown in Table 6. The values of μ_k and μ_s were greatly increased to 0.370 and 0.634, respectively and μ_s/μ_k was 1.71. The character of the curves was also somewhat changed. It is known at this time how much of the effect was due to the aluminum and how much to the effect of the evacuation and drying. Cotton subjected to vacuum drying but not coated with aluminum will be examined subsequently.

8. Coefficients of Friction of Other Fibers

The coefficients of friction of a few fibers of rayon and nylon have been measured for comparison purposes. These measurements have been made in sufficient quantity only to give some idea as to the coefficients to be expected and the characters of the respective curves.

Data obtained are exhibited in Table 7. It will be noted, somewhat surprisingly, that coefficients of kinetic friction are higher than for cotton and the coefficients of static friction are slightly higher. The ratio of μ_s/μ_k are nearly the same for rayon, 1.73, but lower for nylon, 1.61. Some values obtained for rayon at higher tensions, 825 mg, were markedly lower, 0.254 for μ_k and 0.484 for μ_s, giving 1.90 which appears high for the ratio μ_s/μ_k.

9. New Low Normal Force Fiber Friction Apparatus

Using the same principles of the friction apparatus described in our first progress report a new apparatus for measuring friction at low normal forces has been developed. The new instrument is basically the same as the original device but design improvements allow important parameters to be varied remotely and the low force sensitivity has been increased. The normal force, or load, may now be varied continuously between about 2 and 25 mg to an accuracy better than 0.1 mg. The drive speed may be set at any value desired and is reversible.

The previous apparatus (Figure 12 of Semiannual Report No. 2) employed a long counter-balanced arm with a micrometer screw adjustment for obtaining

Table 6. Coefficients of Friction of Empire WR Cotton After Partial Coating with Evaporated Aluminum*

	Coefficients of Friction					
	Kinetic			Static		
(Run No.)	1	2	3	1	2	3
Spec. No.						
178	0.448	----	0.485	0.806	----	0.743
179	0.407	0.416	0.385	0.707	0.694	0.654
180	0.286	0.346	0.242	0.501	0.602	0.530
181	0.362	0.359	0.358	0.594	0.600	0.546
Average	0.376	0.374	0.361	0.652	0.632	0.618
Overall Average		0.370			0.634	

	Ratio μ_s/μ_k		
(Run No.)	1	2	3
178	1.67	----	1.62
179	1.74	1.67	1.70
180	1.75	1.74	2.19
181	1.64	1.67	1.53
Average	1.70	1.69	1.76
Overall Average		1.72	

*Cotton was coated in vacuum by evaporation of aluminum at a pressure of 10^{-6} torr. Film thickness estimated at 150 angstroms but surfaces not uniformly coated.

PART I.

Table 7. Coefficients of Friction of Rayon, Nylon, and Other Fibers

Rayon on Rayon (3 Den Brt, Tension 530 mg)

(Run No.)	Kinetic 1	Kinetic 2	Kinetic 3	Static 1	Static 2	Static 3	Ratio μ_s/μ_k 1	Ratio μ_s/μ_k 2	Ratio μ_s/μ_k 3
Spec. No.									
175	0.308	0.326	0.324	0.506	0.498	0.486	1.64	1.53	1.50
176	0.286	0.299	0.279	0.590	0.565	0.590	2.06	1.89	2.11
177	0.425	0.372	0.322	0.692	0.600	0.567	1.63	1.63	1.76
Average	0.340	0.332	0.308	0.596	0.554	0.548	1.78	1.68	1.79
Grand Average		0.327			0.566			1.75	

Rayon on Rayon (3 Den Brt, Tension 825 mg)

184	----	0.264	0.272	----	0.536	0.523	----	2.03	1.92
185	0.254	0.228	0.220	0.447	0.421	0.438	1.76	1.85	1.99
Average	0.254	0.246	0.246	0.447	0.503	0.480	1.76	1.94	1.96
Grand Average		0.249			0.477			1.91	

Nylon on Nylon (15 Den Brt, Tension 825 mg)

186	0.380	0.353	----	0.596	0.510	----	1.57	1.44	----
187	0.386	----	0.335	0.703	----	0.605	1.82	----	1.81
188	0.302	0.309	0.296	0.457	0.462	0.451	1.52	1.49	1.52
Average	0.356	0.331	0.316	0.585	0.486	0.528	1.64	1.47	1.67
Grand Average		0.331			0.533			1.62	

PART II.

Table 7. Typical Values of the Coefficients of Friction for Various Textile Fibers

Investigator	Fiber	Method	Normal Force (mg)	Tension (mg)	μ_k	μ_s	μ_s/μ_k
Bryant	Cotton	Fiber/Fiber	20	425	0.292	0.523	1.8
Morrow	Cotton	Fiber/Pads			0.22		
Mercer and Makinson	Cotton	Fiber/Fiber	170-180			0.57	
Viswanathan	Cotton	Fringe/Fringe	30000			0.587	
Bryant	Viscose	Fiber/Fiber	20	425	0.304	0.539	1.8
Howell	Viscose	Fiber/Fiber	5.7	1000		0.460	
Mercer and Makinson	Viscose	Fiber/Fiber	170-180			0.190	
Gralen and Olofsson	Viscose	Fiber/Fiber	17		0.180	0.302	1.68
Gralen and Olofsson	Viscose	Fiber/Fiber	47		0.156	0.282	1.80
Gralen and Olofsson	Viscose	Fiber/Fiber	67		0.144	0.276	1.92
Guthrie and Oliver	Viscose	Fiber/Fiber	125		0.141	0.216	1.53
Viswanathan	Viscose	Fringe/Fringe	30000			0.546	
Bryant	Nylon	Fiber/Fiber	20	425	0.338	0.549	1.62
Howell	Nylon	Fiber/Fiber	15.4	1000		0.37	
Mercer and Makinson	Nylon	Fiber/Fiber	170-180			0.23	
Gralen and Olofsson	Nylon	Fiber/Fiber			0.400	0.470	1.18
Viswanathan	Nylon	Fringe/Fringe	30000			0.621	

* See Thesis of Bryant for references in this Table.

a desired load. The long arm was necessary to achieve an effectively linear fiber drive since the motion was generated by rotation about the vertical axis of the balance arm. Our experience showed that this arrangement required tedious adjustments to obtain a desired fiber load and our precision was less than ± 1 mg in the initial form. Below 20 mg the heavy, undamped, arm was often excited into a vertical mode, or bouncing, which generated obvious problems in interpretation of frictional force data.

Our new instruments exhibited in Figure 25 uses a simple electromagnetic system to apply the fiber load. A sensitive but rugged meter movement was mounted on a milling vise with the rotational axis of the meter in the horizontal plane. A short, light arm, with a fiber holder at one end, was constructed from non-magnetic cupro-nickel alloy tubing and attached to the coil of the meter movement. A second length of tubing attached to the opposite side of the coil is used as a rough counterbalance adjustment. Fine balancing is obtained with the meter's normal zero adjustment mechanism. With the meter mechanically balanced, a current through the coil will produce a force at the end of the fiber holder directly proportional to the coil current. Calibration of the loading device is conveniently and precisely accomplished by hooking a previously weighed fine wire segment over the fiber and reversing the current through the coil. We find our reproducibility of normal loads to be better than ± 0.1 mg and the load-current curve is linear over the range of interest, 2 to 20 mg. We are therefore able to use the applied current as an electrical signal which is proportional to the fiber load. The construction of the meter movement is such that small tendencies to "bouncing" are quickly damped.

As mentioned above, the loading device with the traversing fiber attached is mounted on a milling vise. The milling vise is driven by a variable speed motor through reduction gears to provide any desired linear fiber drive rate.

The drive direction may be reversed by reversing the motor. A tachometer mounted at the motion output is used to give an electrical signal proportional to the fiber drive speed.

The improvements described above have made it possible to obtain reliable fiber friction data with loads as low as 2 mg. In addition, for a typical fiber driven at a given speed we are able to obtain directly an x-y recorder plot of the frictional force vs the normal force, or load. The resulting curves have displayed no eccentricities from the expected stick-slip pattern in preliminary measurements. In addition, we can produce such curves at various desired drive speeds, and with the drive reversed to study the influence of drive parameters. A further discussion of the data obtained is presented in subsequent paragraphs.

10. Friction Measurements at Low Normal Forces

The apparatus described in paragraph 9 preceding has very delicate fiber mounting assemblies. In order to explore its capabilities initial measurements and adjustments were made with fine metal wires (2 mils in diameter).

Subsequently a glass fiber was used for the lower or fixed fiber. Its diameter was 19 μ. Data were obtained for cotton and nylon against the glass fiber at normal forces of 2 and 5 mg respectively. Tensions were established at 425 mg. Traverse speed was 0.1 mm/sec. The glass fiber was rinsed with acetone after each run. The data are shown in Table 8.* Typical frictional data plots for fibers at 2 and 5 mg normal force are shown in Figures 26 and 27.

It is interesting to note here that the value of μ_k for cotton against glass is 0.398 and 0.344 at 2 and 5 mg of normal force respectively and that μ_s is 0.902 and 0.589. Ratios of μ_s/μ_k are 2.27 and 1.73 respectively. If these values are plotted on a graph along with data at 20 mg for cotton on cotton, it is surprising that the latter points appears to lie on or near the projected curve as shown in Figure 28. These results check well with previous experiments discussed by Bowden and Tabor[8] for nylon instead of cotton.

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* The nylon against glass data are in the Appendix as Table 16.

Table 8. Coefficients of Friction of Cotton Fibers Against Glass and Nylon at Low Normal Forces

Cotton on Glass at 2 mg Normal Force

(Run No.)	μ_k 1	2	3	μ_s 1	2	3	μ_s/μ_k 1	2	3
Specimen									
1	0.379	0.407	0.378	0.86	0.88	0.90	2.27	2.16	2.38
2	0.457	0.338	0.367	0.98	0.84	0.89	2.14	2.48	2.42
3	0.434	0.414	0.410	0.93	0.91	0.93	2.14	2.20	2.27
Average	0.423	0.420	0.385	0.92	0.88	0.91	2.18	2.28	2.36
Grand Average		0.409			0.90			2.21	
				5 mg Normal Force					
1	0.395	0.402	0.390	0.648	0.656	0.632	1.64	1.63	1.62
2	0.343	0.372	0.352	0.568	0.596	0.552	1.66	1.60	1.57
3	0.280	0.296	0.266	0.562	0.562	0.528	2.00	1.90	1.98
Average	0.339	0.357	0.336	0.593	0.605	0.604	1.77	1.71	1.72
Grand Average		0.344			0.601			1.75	
				Cotton on Nylon 2 mg Normal Force					
1	0.470	0.466	0.408	0.962	0.953	0.803	2.04	2.04	1.97
2	0.325	0.320	0.288	0.699	0.701	0.703	2.15	2.19	2.44
3	0.353	0.319	0.366	0.873	0.912	0.970	2.47	2.91	2.65
Average	0.383	0.368	0.354	0.845	0.855	0.825	2.22	2.38	2.35
Grand Average		0.368			0.842			2.30	
				5 mg Normal Force					
1	0.564	0.554	0.528	1.00	1.02	0.82	1.78	1.85	1.55
2	0.488	0.487	0.485	1.05	1.05	1.06	2.15	2.16	2.19
3	0.456	0.425	0.444	0.96	0.95	1.00	2.10	2.23	2.25
Average	0.503	0.489	0.486	1.00	1.01	0.96	2.01	2.08	2.00
Grand Average		0.492			0.99			2.03	

SUMMARY

	μ_k	μ_s	μ_s/μ_k
Cotton on Glass (2mg)	0.409	0.90	2.21
Cotton on Glass (5mg)	0.344	0.601	1.75
*Cotton on Nylon (2mg)	0.368*	0.842*	2.30
*Cotton on Nylon (5mg)	0.492*	0.99*	2.03

*The inversion of the values of μ_k and μ_s for cotton on nylon at the two different loads appears to be real. However, rechecks on these measurements are currently being made.

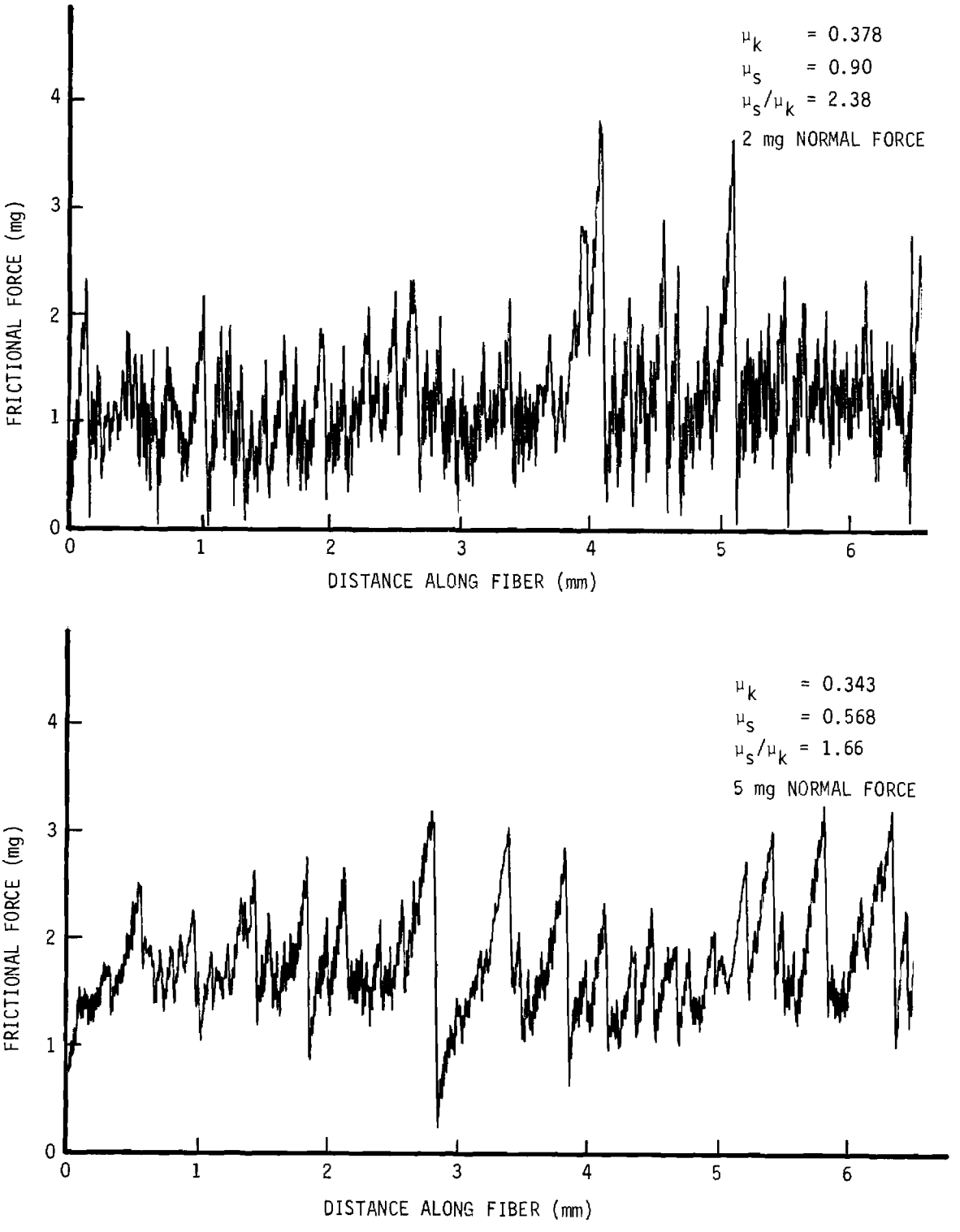


Figure 26. Frictional Data for a Cotton Fiber on a Glass Fiber at Normal Forces of 2 mg and 5 mg.

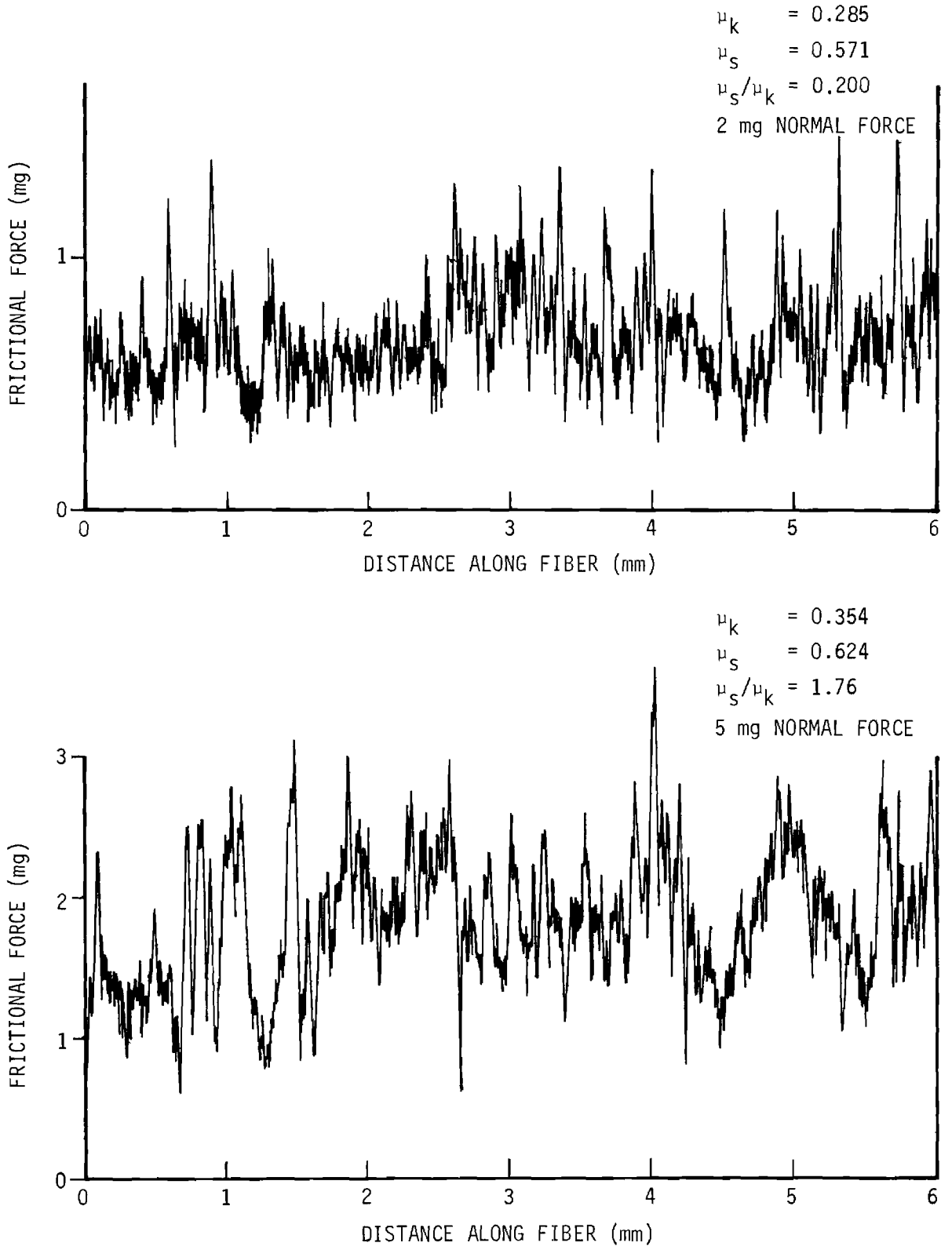


Figure 27. Frictional Data for a Nylon Fiber on a Glass Fiber at Normal Forces of 2 mg and 5 mg.

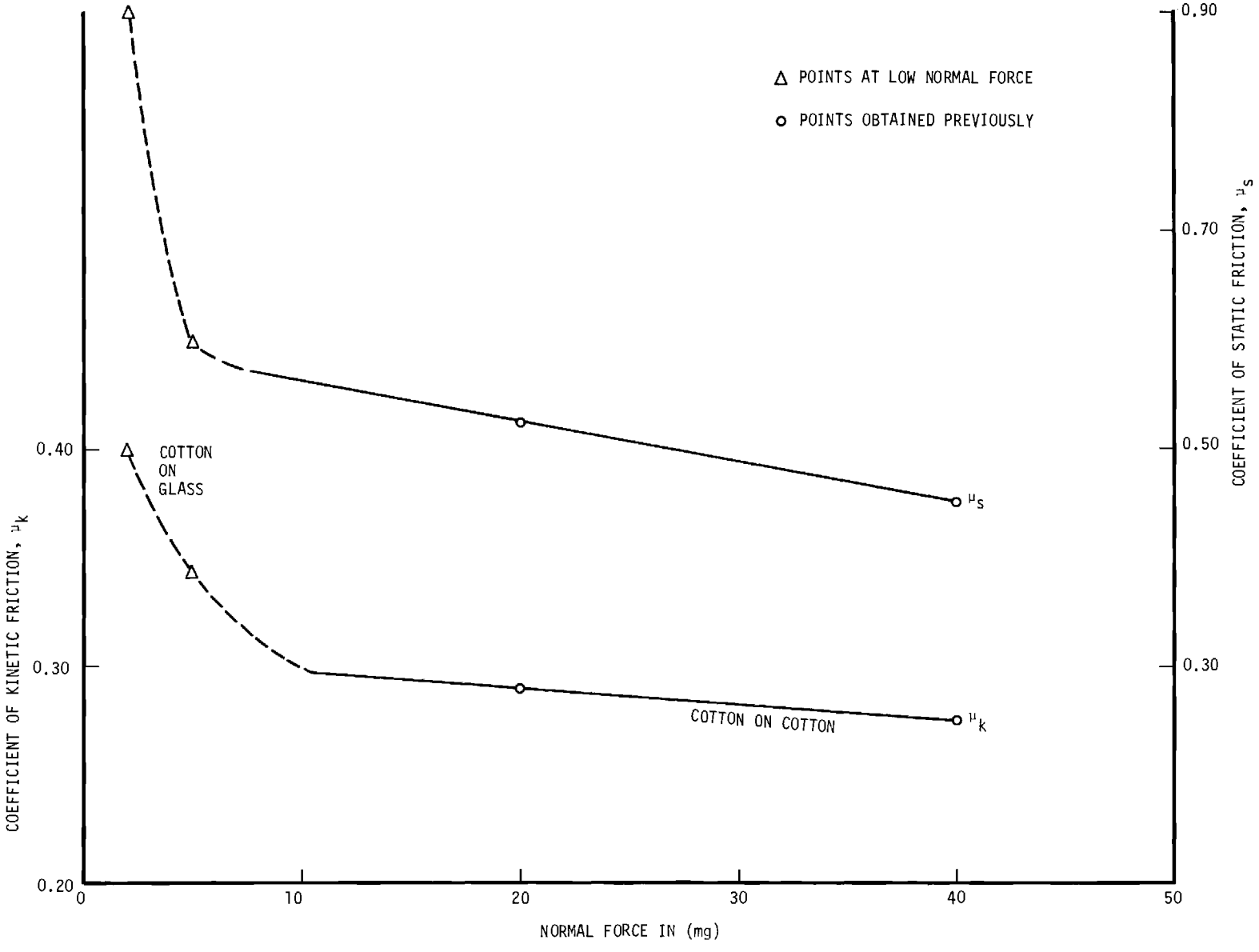


Figure 28. Data Indicating Variation of Coefficients of Kinetic and Static Friction with Normal Force for Cotton.

11. Coefficients of Friction of Metallic Fibers*

As noted previously, for the preliminary experiments with the low normal force apparatus fine metal wires were employed. Frictional data was obtained for the metals gold, aluminum and tungsten at normal forces of 2, 4, 8 or 16 milligrams. A typical example of frictional data is shown in Figure 29 for the metal tungsten. In these data also a new method of obtaining the coefficient of static friction was utilized. In this method a statistical plot of all stick maxima was made as shown in Figure 30. The median was then determined from the plot. Data for the various metals are shown in Table 9. And ratios of μ_s for the ten maxima to those obtained by the statistical plot are shown. Calculations for μ_k values have not yet been made.

It will be noted that there are large differences between the values μ_s obtained for the several metals with gold exhibiting much the higher frictional coefficient. The oxidized surfaces of the aluminum and tungsten and the hardness of the latter undoubtedly contribute to the lower value found for these metals.

12. Character of Frictional Data Curves

It has been noted previously that the character of frictional curves is specific to the fiber material and configuration. For instance the sticks per cm of the frictional data plot are greater for rayon and nylon than for cotton but peak heights and μ_s/μ_k ratios are normally greater for cotton than the other textile fibers examined. In repeated runs of one cotton fiber on a second the character of the traversing fiber is repeated with small changes such that both the static and kinetic coefficients are slightly reduced for the 2nd and 3rd runs. However, reductions subsequent to the third consecutive run, in limited experiments, do not appear to be significant. Principal peaks persist throughout as much as 13 consecutive runs; and parts of a cotton fiber are obviously quite different from other parts insofar as the frictional plot are concerned.

If one examines character at very slow fiber traverse rates and slow pen traverse rates, the stick maxima are so frequent and so narrow as to

* This work was performed principally by an undergraduate student in Physics, Mr. J. Taylor, now an undergraduate in Physics at the California Institute of Technology.

Table 9. Coefficients of Friction of Gold, Aluminum, and Tungsten At Low Normal Forces

Substance	Normal Force mg	Static Friction† Median Value	Static Friction Greater Ten Values	Ratio Greater Ten / Median	Remarks
Au on Au	2	0.470	1.05	2.23	0.002" wire
Au on Au	4	0.420	0.95	2.27	
Au on Au	8	0.425	0.89	2.09	
Al on Al	2	0.115	0.25	2.21	0.002" wire
Al on Al	2	0.137	0.285	2.08	
Al on Al	4	0.165	0.234	1.42	
Al on Al	8	0.122	0.230	1.88	
Al on Al	16	0.068	0.109	1.60	
W on W	2	0.265	0.450	1.70	0.0005" wire
W on W	8	0.243	0.435	1.79	
W on W	16	0.280	0.355	1.27	

† The median value of the coefficient of static friction is higher than μ_k by 25 percent or so.

*Data from First Apparatus at 15 to 25 mg NF

	μ_k	μ_s	μ_s/μ_k
Au on Au	0.368	1.15	3.14
Ag on Ag	0.337	0.526	1.56
Al on Al	0.287	0.62	2.16

*This work conducted on original apparatus at higher normal forces.

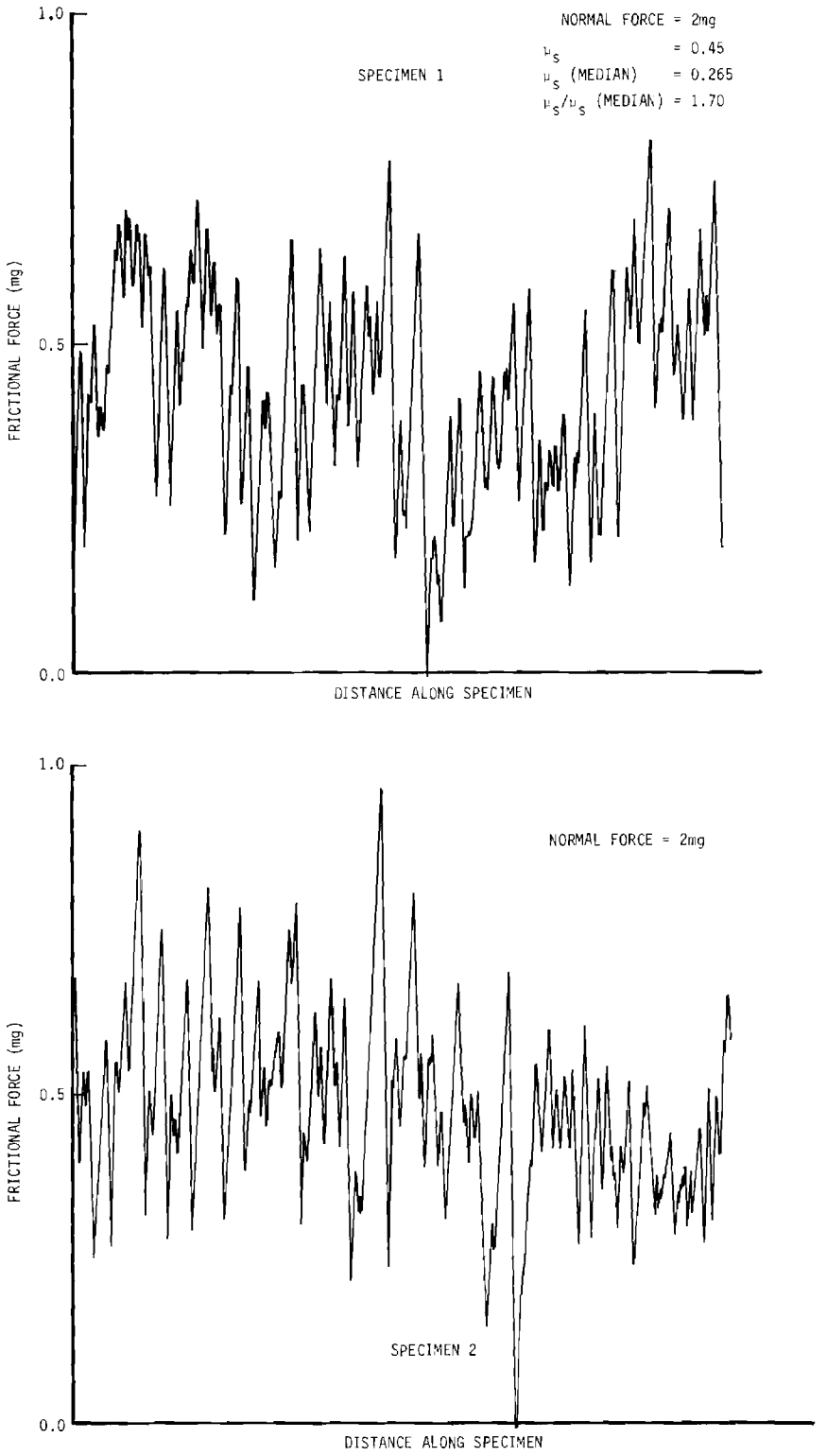


Figure 29. Frictional Data for a Tungsten Wire against a Second Tungsten Wire.

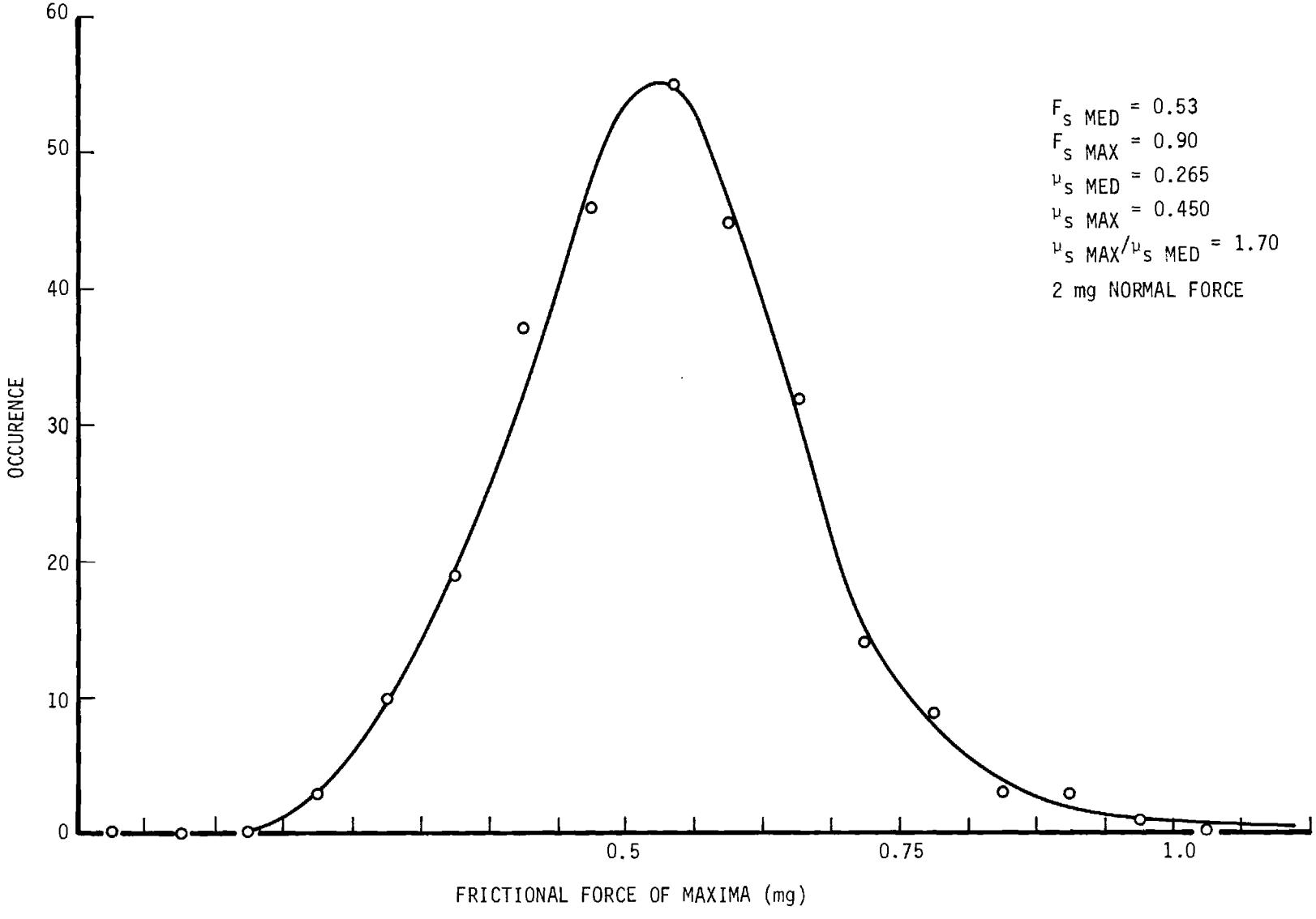


Figure 30. Magnitude of Static Coefficient of Friction Obtained from All Frictional "Sticks" by Plotting Frequency Versus Frictional Force Distribution for a Pair of Tungsten Wires.

be difficult to compare and interpret, especially after reproduction from the original data plot. However, by increasing the pen traverse rate of the x-y plotter by five (from 0.1"/second to 0.5"/second) the difference in character, as shown in Figure 31, is readily observed. However, the rapid pen traverse only allows examination of a very small portion of the fiber (less than 1.6 mm at a traverse rate of fiber of 0.1 mm/second) and the stick maxima are cut off by the rapid pen traverse.

The data curves obtained for cotton fibers after subjection to various processing stages frequently exhibit very high peaks. These have been observed as a result of heating and through the stages opening and cleaning, picking and carding. Since there is a correlation with fiber damage in these stages as indicated with the Congo red test method, it appears that peaks are associated with damage. A typical data plot of a cotton fiber after picking is shown in Figure 32. This peak reoccurred on 3 successive runs.

From the data observed and reported it is evident that much is to be learned from the character of the frictional data plots for the various fibers. The low normal force frictional apparatus should give additional information concerning curve character and its interpretation. It should also allow examination of fiber features not heretofore descernible because of the lack of an apparatus with a suitable discriminating capability.

13. Comments on Frictional Measurements

The frictional measurement data are subject to a number of variables such as tension and normal force which are difficult to control precisely. However, familarity with the method of measurement by the investigators and improvements in instrumentation are giving relatively accurate and repeatable results. These measurements thus enable one to observe changes in the frictional properties of cotton fibers resulting from various mechanical processing stages to which the fibers are subjected in progressing from the cotton boll to the yarn.

D. INVESTIGATIONS OF COTTON FIBERS BY INFRARED TECHNIQUES

1. General

Research conducted by infrared techniques during the current period has been reported in detail in a thesis for the M.S. degree in

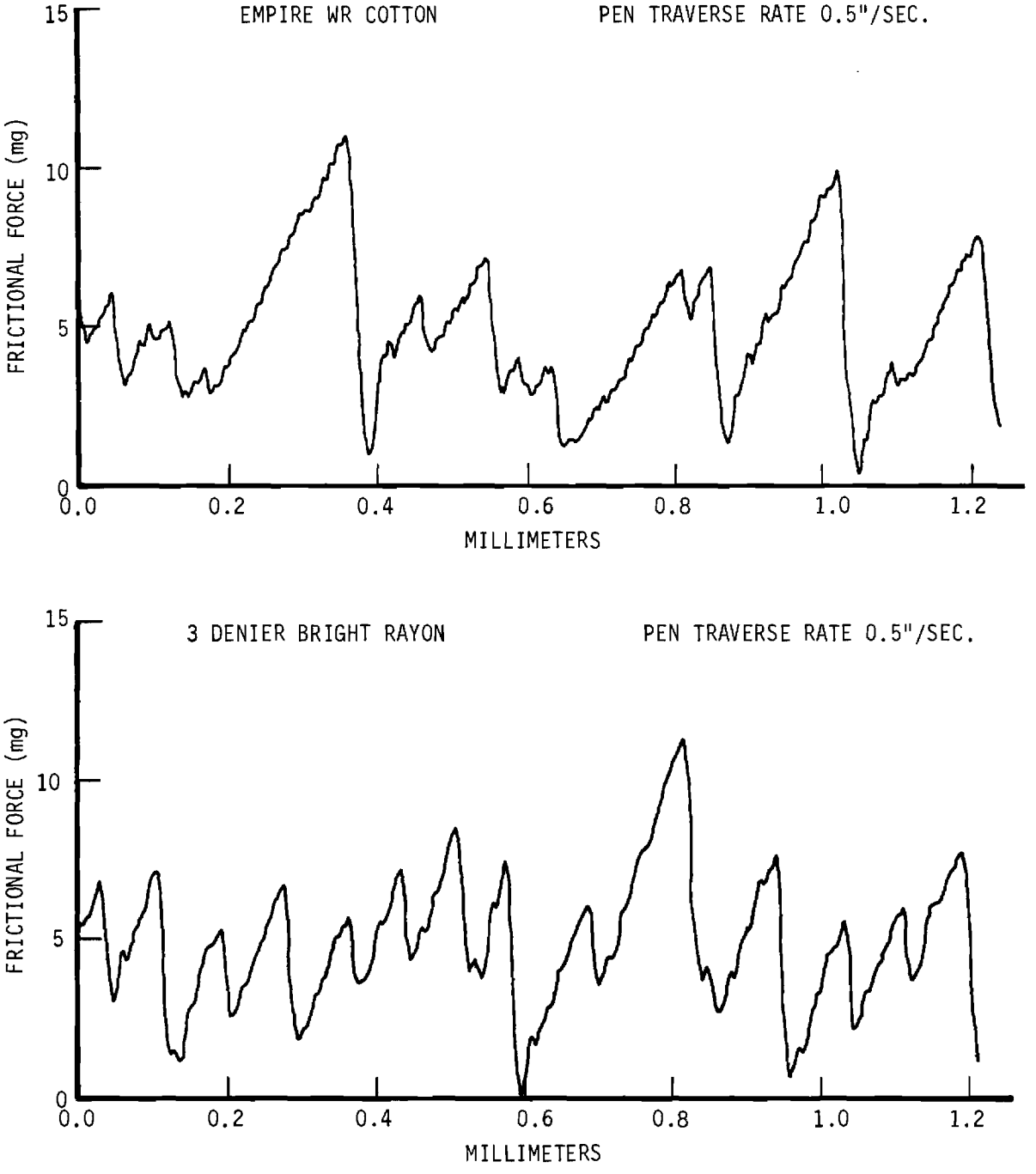


Figure 31. Comparison of Character of Frictional Force Data Plots for Rayon and Cotton at High Plotter Pen Speeds (0.5"/Sec.).

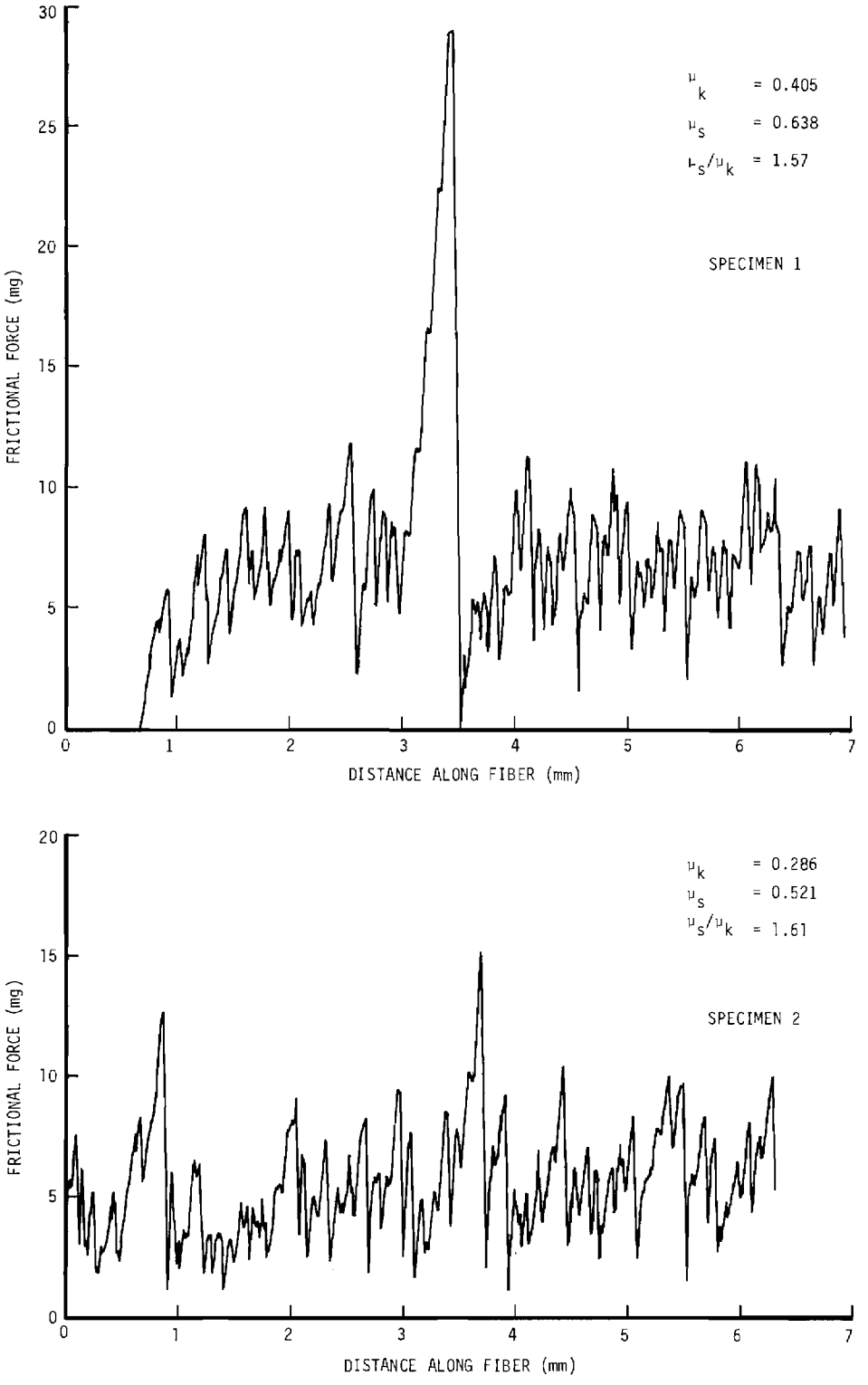


Figure 32. Typical Frictional Data Plots of Empire WR Cotton Fiber after Processing through the Picking Stage.

Textiles prepared by Mr. W. E. Kirkland,[4] Graduate Research Assistant. The title of the thesis is "An Investigation of Techniques to Determine Infrared Spectra of Textile Fibers." Copies of this thesis are being submitted to the sponsor concurrently with this report.

A summary of the work performed by Mr. Kirkland is reported below.

2. Summary of Infrared Investigations

The purpose of this research was to develop and evaluate techniques of textile fiber preparation for infrared spectra analysis. Additional purpose were to examine changes in the spectra of cotton fibers resulting from selected textile processing stages and to collect and compare spectra of a number of various textile fibers prepared by the selected techniques.

The KBr disk, attenuated total reflectance (ATR), and fiber press methods were employed. Effects of fiber concentration, preparation procedure, and particle size (of ground specimens) were examined. By the ratio of absorption of 7.3 μ/3.45 μ bands, a method suggested by O'Connor, both the methods and variables were evaluated.

For cotton spectra the KBr method proved to be best for quantitative results; however, all methods gave excellent qualitative spectra. The KBr method required milling of the fibers to a small size whereas the other did not. Preparation of a fiber press specimen was simple and rapid and did not introduce a second agent. It had an additional advantage of controlled fiber orientation which is necessary in polarized infrared investigations. The ATR method, when carefully controlled, gave excellent spectra related principally to surface properties of the fibers. However, it was subject to exact pressure control, sample contact variables, and damage to the optical element unless great care was used. The latter two methods were superior to the KBr method in that better band resolution was displayed in the 7 - 10 μ region.

By use of each of these techniques spectra of cotton, viscose rayon, and acetate rayon, a polyester (Dacron), an acrylic (Creslan), nylon (except fiber press), and polypropylene (Herculon) were examined. Excellent qualitive spectra were obtained by each method except for nylon by the fiber press method. Nylon fibers did not adhere to each other under the maximum pressures employed (approximately 120,000 psi).

By use of one or more of these methods spectra of cotton specimens were examined before and after ginning, before and after heating to 200°C, after milling to pass various mesh sizes, and after ball milling. Crystallinity indices of the fibers determined by O'Connor's absorption ratio method, were measured.

Spectra of specimens of three varieties of cotton after ginning exhibited insignificant differences from those of unginned cotton. Cotton subjected to heating to 200°C exhibited a new strong band at 5.8 μ. This strong band was not observed in the ginned cotton although some of it had been heated to 165°C for a few minutes. Crystallinity indices of cotton subjected to milling to 20, 40, and 60 mesh exhibited indices of 0.68, 0.69, and 0.69, respectively, compared to a value of 0.71 reported by O'Connor for a 20 mesh specimen of a different cotton. These values indicate a low value of crystallinity for this Empire WR cotton, Georgia grown, but little change resulting from the milling treatment. They also compared favorably with similar data obtained by an x-ray diffraction technique. The ball milled cotton (15 hours) exhibited a crystallinity index of 0.24, having changed to a predominantly amorphous material, in agreement with x-ray data.

3. Examination of Cotton by Polarized Infrared Radiation

The fiber specimens prepared by the press, using aligned whole fibers, are especially useful for examination by polarized infrared radiation. Hence a polarizer for the IR 221 was procured from the manufacturer (Perkin-Elmer). The polarizing optical element consists of a silver chloride plate upon which are deposited a grid of 2880 lines/mm by means of the vacuum evaporation of gold. The resultant grid provides excellent transmission and polarization over the spectral range 2.5 to 25 microns. Approximately 80 percent relative transmission is achieved resulting in an actual transmission level of 42 - 45 percent, essentially the same as that of the silver chloride plate upon which the grid is formed.

Spectra of cotton fiber prepared by the fiber press method were examined by polarized infrared radiation. Bands at several positions, appearing as single bands normally, were observed to be composed of two or more bands. In particular a band at 6.15 μ was found to be a strong band at

6.05 μ with weaker bands on each side of it. A typical spectrum of cotton made with polarized radiation is shown in Figure 33.

4. Conclusions

Each of the techniques have been found to have useful application to textile technology; and variables related to each technique have been defined. Examples of spectra of seven fibers obtained by each method have been exhibited in the thesis by Kirkland, and applications of the various methods to a study of the effects of textile processing on cotton fibers have been reported. Further investigations utilizing the fiber press technique and polarized infrared radiation should give useful fiber structure information.

E. INVESTIGATIONS OF COTTON FIBERS BY X-RAY DIFFRACTION TECHNIQUES

1. General

X-ray diffraction techniques have been used to examine the effects on cotton crystallinity of milling cotton fibers in a Wiley Mill to 20, 40, and 60 mesh, of ball milling them for a period of 15 hours, and for examining the crystallinity of cotton specimens before and after ginning.

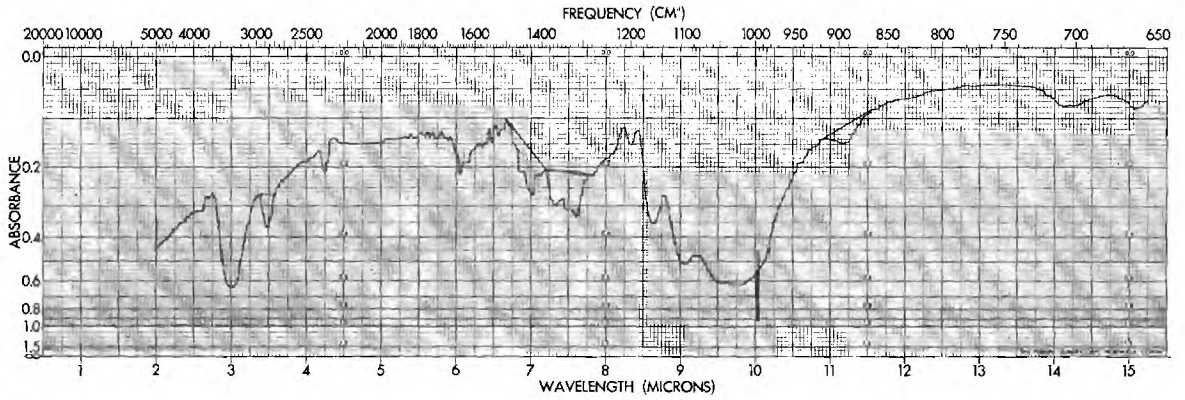
2. Effect on Crystallinity of Milling Cotton in A Wiley Mill

a. Procedure

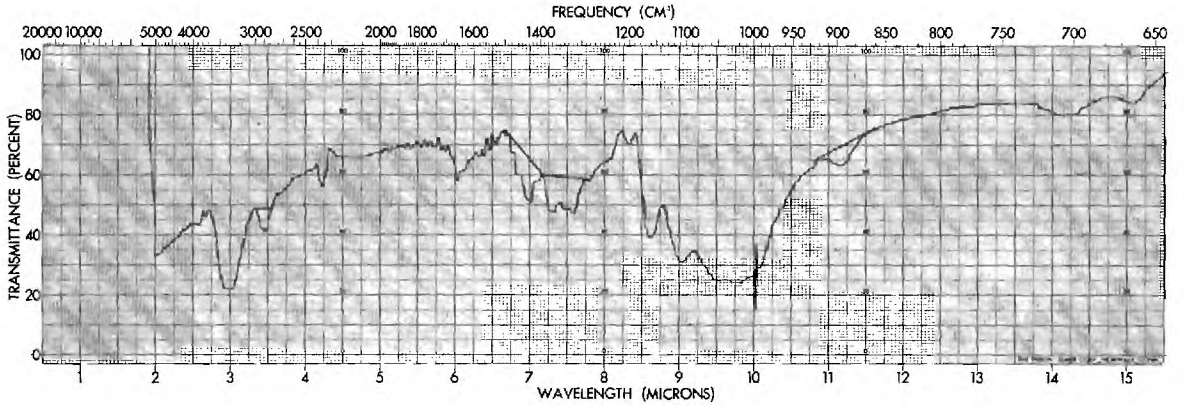
Samples of 1964 crop cotton, mechanically ginned, were chopped to pass a 20, 40, and 60 mesh screen in a Wiley Mill. X-ray powder diffraction patterns were run on each type. There were seven samples taken of each type and for each sample there was calculated a "crystallinity ratio;" C.R.:

$$C.R. = \frac{I_{002} - I_{am}}{I_{002}}$$

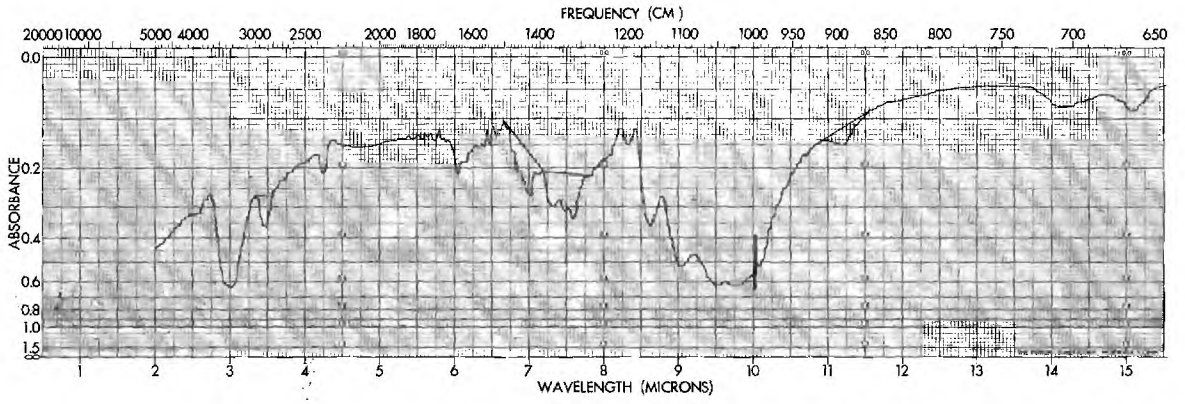
where I_{002} is the intensity of the 022 peak (at about 22.6°) and I_{am} is the intensity of the minimum at about 19° where there are no peaks, an intensity characteristic of amorphous scattering (hence, I_{am}).



A. At 0°



B. At 45°



C. At 90°

Figure 33. Spectra of Empire WR Cotton Fibers Obtained Using a Fiber Press Specimen and Polarized IR Radiation.

The data are shown in Table 10, along with the mean values and the associated standard deviations, σ, defined as

$$\sigma = \sqrt{\frac{\sum(\Delta x)^2}{n-1}} \quad *$$

where Δx is the number $|x_o - x|$, x_o the mean value, and n, the number of samples (7 in this case).

The electronic settings were essentially the same throughout, with the PHA set each day. The same sample-holder was used, and patterns of this empty holder showed no scatter in the regions of interest.

The crystallinity ratio of the cotton subjected to ball milling for 15 hours was 0.18.

b. Conclusions

The data are insufficient to permit us to determine what, if any, is the difference in C.R. between the twenty and forty, and the forty and sixty mesh cotton. A larger number of samples would possibly allow determination of a more definite difference.

There does appear to be a slight increase in the crystallinity ratio of the sixty mesh as compared to the twenty, since we do get a $\delta_{20,\ 60} > (\sigma_{20} + \sigma_{60})$. The difference is small, however, and a larger number of samples would be needed to establish a definite number for its magnitude.

3. Effects of Ginning on the Crystallinity of Seed Cotton

Crystallinity measurements were made on three types of cotton: (1) 1964 crop cotton which had been mechanically ginned; (2) 1965 crop cotton from the same soil which had also been mechanically ginned, and (3) identical 1965 crop seed cotton which was hand-ginned. Ten samples were taken of each type, and all samples were chopped by a Wiley Mill to pass a 20 mesh screen. For each sample an x-ray diffraction pattern was run, and a crystallinity ratio (C.R.) was calculated:

---

* The term n-1 rather than n is used to give an additional allowance for the small number of measurements made.

Table 10. Comparison of Crystallinity Ratios of Cotton Milled to 20, 40 and 60 mesh in a Wiley Mill

$$\text{Crystallinity Ratio} = \frac{I_{002} - I_{am}}{I_{002}}$$

SAMPLE	20 Mesh	40 Mesh	60 Mesh
AA	0.784	0.802	0.807
A	0.792	0.795	0.797
B	0.765	0.797	0.803
C	0.781	0.789	0.808
D	0.769	0.782	0.804
E	0.783	0.799	0.792
F	0.786	0.794	0.797
Average Value of C.R.	0.780	0.794	0.801
Standard Deviation σ	0.010	0.007	0.006

RESULTS

Differences:	$\delta_{20,40} = 0.014 \pm 0.017$	$1.8\% \pm 2.1\%$
	$\delta_{40,60} = 0.007 \pm 0.013$	$0.9\% \pm 1.6\%$
	$\delta_{20,60} = 0.021 \pm 0.016$	$2.6\% \pm 2.0\%$

Note: By the analysis of variance there was a significance difference between the effects of the number of mesh at 95% confidence level. By Duncan's multiple range tests, there was no difference between the means of 40 mesh and 60 mesh at 95% significance level.

$$C.R. = \frac{I_{002} - I_{am}}{I_{002}}$$

where I_{002} is the intensity of the (002) diffraction peak (dependent on the density of crystalline material in the sample) and I_{am} is the intensity of the minimum at about 19^{o} (dependent on the total mean density of the sample). A pattern made of the empty sampleholder showed no scatter in the region of interest. The electronic settings were maintained constant throughout.

The results are shown in Table 11 along with the standard deviations σ:

$$\sigma = \sqrt{\frac{\sum(\Delta x)^2}{n-1}}$$

where $\Delta x = (x_o - x)$; x_o is the mean value of the C.R. for a particular type, and n is the number of samples. The probable error shown is the sum of the standard deviation of the values involved.

The sum of the standard deviations is larger than the measured differences both between the mechanically ginned 1964 and 1965 cotton, and between the mechanically and hand-ginned 1965 cotton. There was inappreciable difference in crystallinity among the three types of cotton tested in this experiment.

F. COTTON FIBER PROPERTIES AS AFFECTED BY GINNING*

1. General

An investigation of the effects of ginning on the physical properties of cotton fibers has been reported in detail in the thesis of Mr. A. Goldfarb[5] Graduate Research Assistant, submitted concurrently with this report. A summary of this thesis and a few of the more pertinent facts are discussed in the following paragraphs.

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* A large part of the text of this section has been obtained from the thesis of Mr. A. Goldfarb submitted as a part of the requirements for the award of Master of Science in Textiles (Georgia Institute of Technology, August 1966).

Table 11. Comparison of Crystallinity Ratios of Hand and Mechanically Ginned Cotton

Sample	(α) 1966 Mech. Ginned	(β) 1965 Mech. Ginned	(γ) 1965 Hand Ginned
III	.778	.814	.789
IV	.807	.803	.800
V	.800	.800	.789
VI	.782	.784	.774
VII	.806	.817	.772
VIII	.790	.801	.803
IX	.803	.800	.803
X	.789	.800	.795
XI	.781	.799	.782
XII	.780	.823	.793
Mean	.792	.804	.790
Standard Deviation (σ)	.011	.011	.012
	$\delta_{\alpha,\beta} = .012 \pm .022$	$1.5\% \pm 2.8\%$	
	$\delta_{\beta,\alpha} = .014 \pm .023$	$1.8\% \pm 2.9\%$	

Note: A subsequent statistical analysis of these data showed there was a significant difference between means of these data at 95% significance level but not at the 99% level. By Duncan's multiple range tests, there was no difference between specimens α and γ at 95% level. But significant differences were observed between α and β, and γ and β at the same significance level.

2. Effects of Ginning on the Cotton Fiber

Specimens of three cottons, Empire WR, Carolina Queen and Dixie King, grown and ginned in central Georgia, were obtained in the seed cotton and ginned cotton form. Examinations or measurements were made of fiber appearance, fiber length distribution, force and energy required to withdraw individual fibers from the seed, fiber tensile properties, and changes in the seed cotton fiber resulting from simulating gin drying conditions. Micrography was used for extensive examination of the fibers in pertinent phases of the study.

By construction and use of special apparatus for measurement of fiber withdrawal and breaking forces, it was possible to measure and to show that the average breaking force of individual fibers was 4.24 g using a 6.35 mm gage length and that the average fiber withdrawal force from its seed was 1.23 g. However, the occurrence distribution of these forces showed a 27.5 percent overlap, indicating that many fibers would normally be expected to break during the ginning process. Measurement of fiber withdrawal forces after heating to successively higher temperatures in the range of 22° to 200°C indicated that a maximum increase of about 25 percent occurred at approximately 150°C (302°F), which is lower than the drying temperature employed by many gins. Measurements by microscopy exhibited increases in convolutions per unit length of up to 27 percent, implying fiber shrinkage in agreement with reports in the literature.

Fiber length distribution measurements conducted for seed cotton and ginned cotton by both the Digital Fibrograph and by the array method on the Suter-Webb sorter indicated fiber shortening over all span lengths as a result of the ginning process. This varied, to a degree in accordance with species, harvesting method, gin drying temperature, and the mechanical features intrinsic to the gin employed. Reductions in the mean length ranged from 2.5 to 15 percent and the results were in accord with the overlap of fiber withdrawal and breaking force distributions previously discussed, and with increases in the overlap resulting from heating.

3. Special Apparatus Employed in Ginning Effect Studies

a. Gin Dryer Simulator

An oven capable of simulating a gin dryer was constructed as shown in Figure 17. It consisted principally of a hot-air blower

and a 70 cm length of Pyrex tube (4.75 cm inside diameter). Air from the blower was forced into the tube which was maintained in a position inclined 7.5° from the horizontal. The temperature was controlled by means of a Variac connected to the heater element of the hot-air blower. The specimen support consisted of a length of one cm (outside diameter) Pyrex tube, supported near the axis of the larger tube with cork stoppers, one end of which had a metal clamp attached. A mercury thermometer capable of registering over the range of 0 to 200°C indicated the temperature near the position of the specimen. Temperatures over the range of 22°C to 200°C were readily produced and maintained to ± 1°C with this apparatus. Higher temperatures could have been attained but were not needed.

The desired temperature was obtained in the oven by adjustment of the Variac and allowing the oven to attain a steady state. The fiber specimens, cemented at one end to a glass slide, were inserted into the oven and allowed to remain for 30 seconds. The specimens were then removed for examination. More details of the procedure were discussed in Section III,B,7.

b. Apparatus for Measuring Withdrawal Forces and Tensile Strengths of Cotton Fibers

A force measuring instrument was devised from a Servo Torque-Balance Reaction Dynamometer (Model 114A manufactured by McFadden Electronics Company, South Gate, California) and its amplifier, a Mosely X-Y Recorder (Model 135) and a small one r.p.m. electric motor driving a one inch diameter roll. The assembled apparatus can be seen in Figure 34.

A drawing of the dynamometer is shown in Figure 35 and the instrument is described below:

> The Model 114A Servo Torque-Balance Reaction Dynamometer is an extremely sensitive system for measuring . . . low torques . . . . It consists of a rotary air-bearing supported platform, a servo controller, and an interconnecting cable.
>
> When a torque is generated by a mounted test specimen it is applied to the air-bearing supported platform tending to cause angular displacement. Any displacement is sensed by the position transducer which sends a signal to the controller

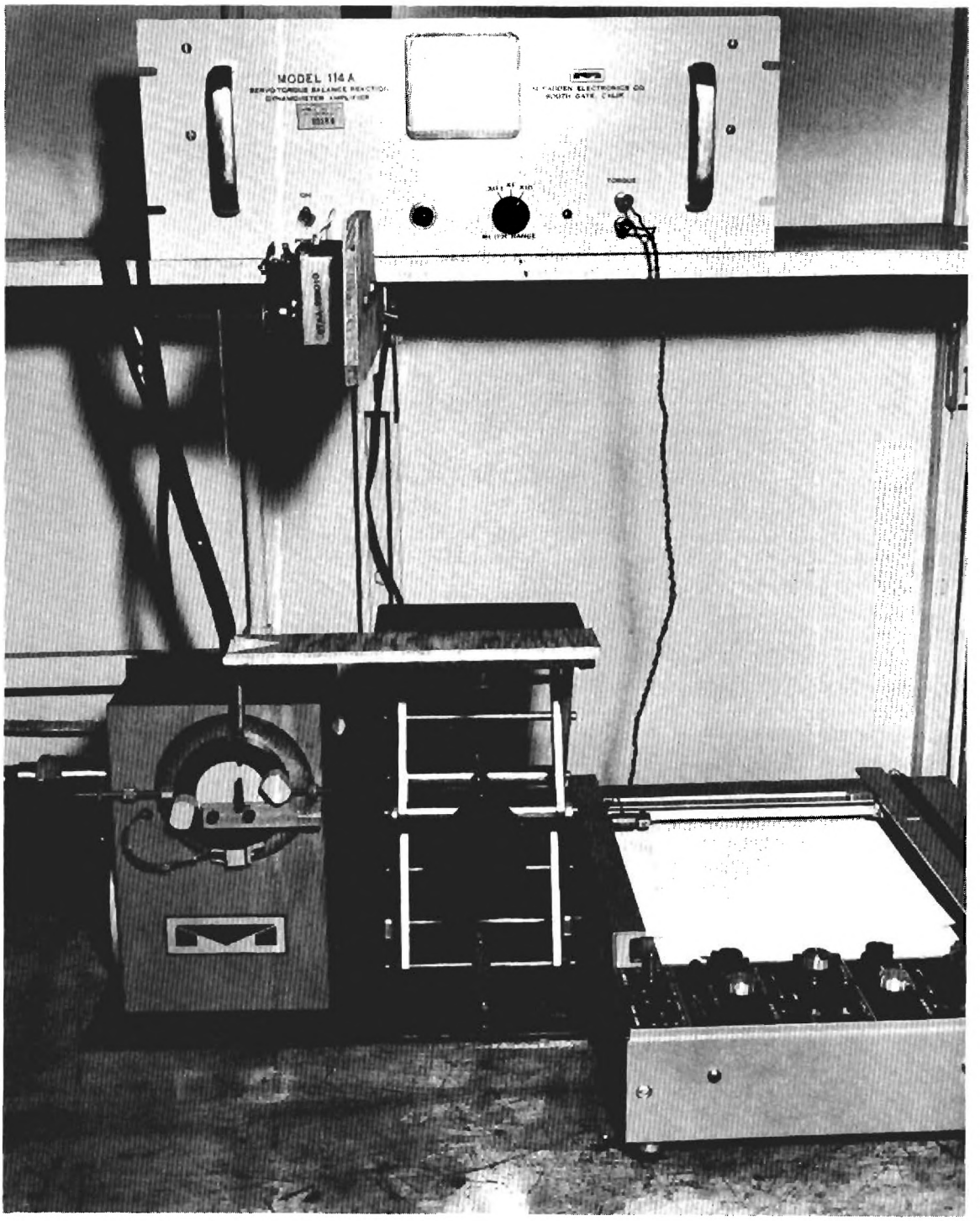

Figure 34. Apparatus Employing Servo-Torque Balance Dynamometer for Measurement of Fiber Tensile Strengths.

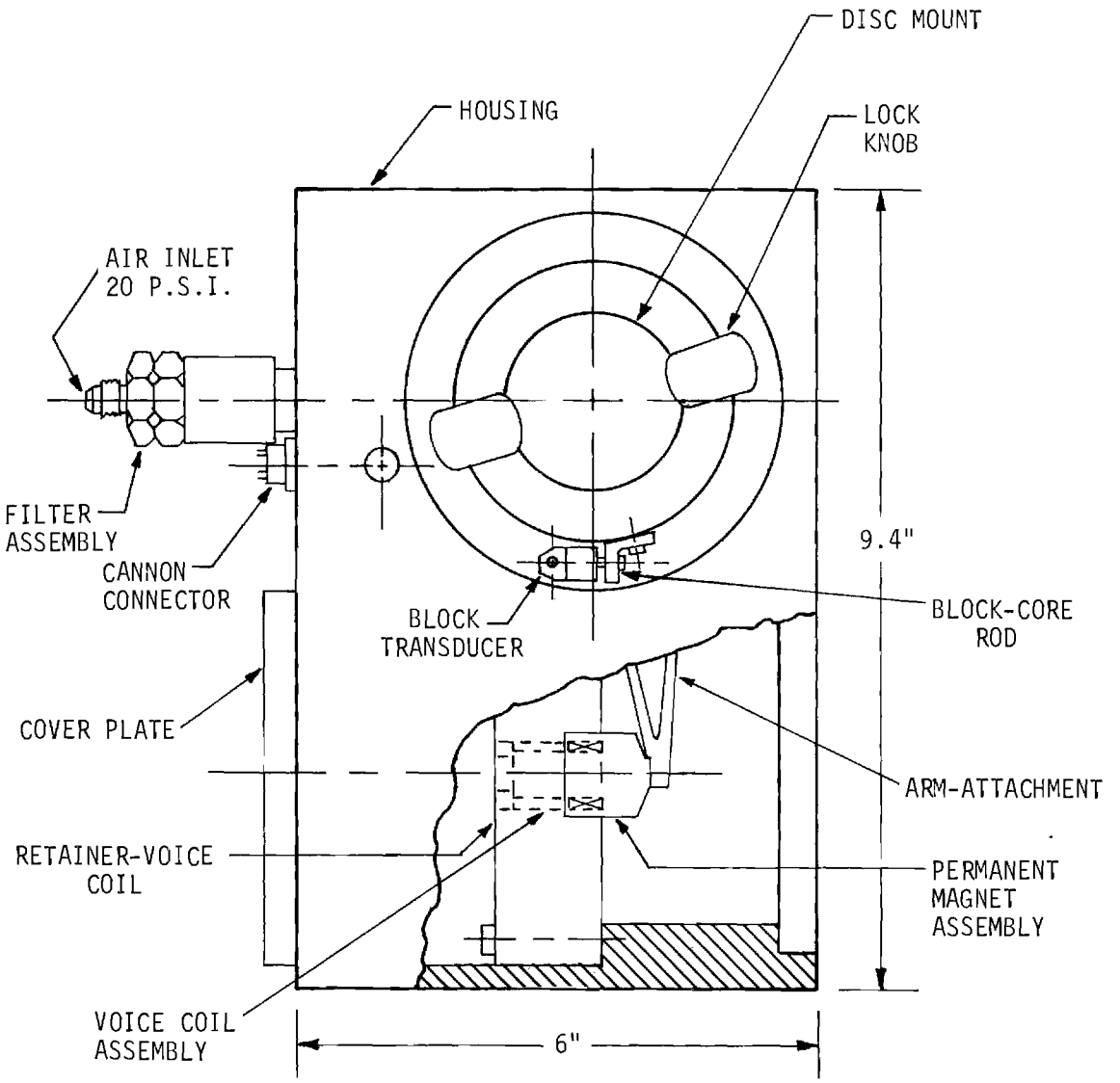

Figure 35. Drawing of Servo-Torque Balance Reaction Dynamometer.

> amplifier to supply current to the torquer. This provides a reaction torque exactly equal and opposite to that . . . (applied to the lever arm). Integration in the forward portion of the servo loop assures essentially zero steady state angular displacement.[15]

An output voltage proportional to the restoring current to the torquer is supplied to a set of terminals to which an x-y recorder may be connected. The recorder thus plots an analog trace of the force applied to the lever. The instrument may be calibrated by attachment of small weights to the lever arm at the position of interest and reading the force displacement on the x-y plotter.

For measurement of the withdrawal forces of a fiber from its seed, a pin was mounted at the end of the dynamometer lever-arm in order to facilitate mounting of the seed. The windlass arrangement utilized a length of braided polyester cord which supported a fiber clamp. The clamp passed through a narrow opening cut in a wedge of Teflon. This limited movement of the clamp normal to the direction of translation.

When a seed was impaled by the pin, a fiber clamped, and the motor started, the fiber was pulled upward at a constant rate of π in./min. or 1.33 mm/sec. The servo sensed the force applied to the lever arm maintaining it near its zero position. A potential representing the torque restoring current was taken off the terminals mentioned and fed to the x-y recorder. During the operation cycle, a record was thus obtained of the force of withdrawal of the fiber and the time (or distance) through which the force acted. The apparatus arrangement for these measurements is shown in Figure 36.

Essentially, the same apparatus was also employed to measure the forces required to break cotton fibers. Since these forces were expected to be of a higher magnitude than the previous measurements, the effective length of the torque lever was shortened by applying the force to a point which was closer to the pivot of the lever arm. An alligator clip was attached to the lever arm for use as the lower jaw. The upper jaw consisted of a similar clip, and was suspended from the windlass as before.

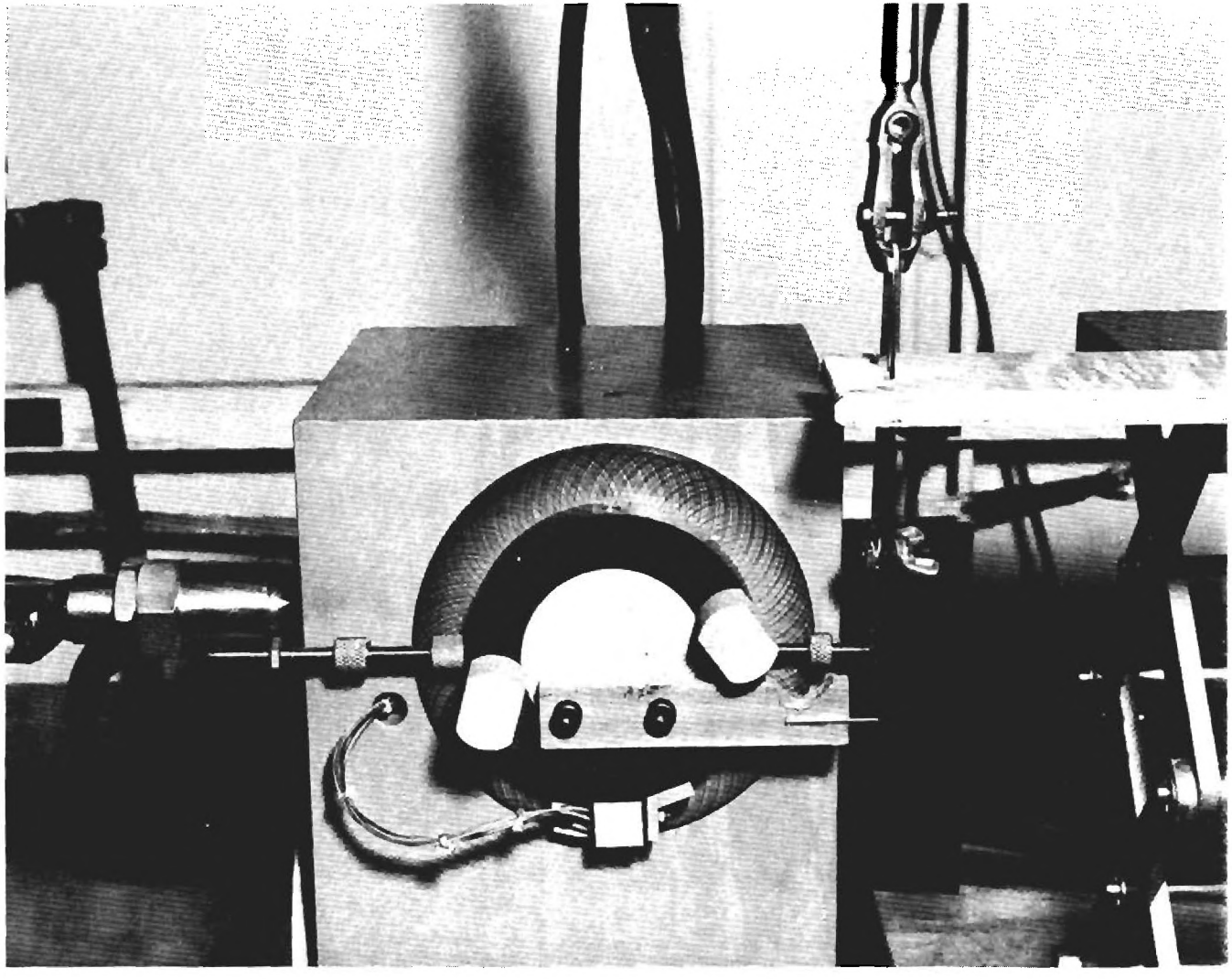

Figure 36. Dynamometer Modified for Measurement of Fiber Withdrawal Force.

The cord was passed through a hole cut in a wedge of Teflon to restrict undesired lateral motion.

The upper jaw was pulled upward at the same constant rate as for previous measurements (1.33 mm/sec). The force to break the fiber was sensed by the lever arm and recorded in the same manner as before. The apparatus arrangement for these measurements is shown in Figure 37.

With this device, the stress-strain curve was automatically plotted for each fiber. Force, elongation, and energy measurements could readily be taken from the curves. Accuracy of ± .01 g-force in the range of 0 to 10 g was easily obtainable and the sensitivity could readily be improved by simple adjustments if desired.

4. A Comparison of Cotton Fiber Attachment Forces to Fiber Strengths

a. General

The structure of the cotton seed, the attachment of the individual fibers to the seed, and the overlap of fiber removal forces with tensile strengths of the fibers are of pertinence to the ginning problem. These have been examined in some detail.

b. Structure of the Cotton Seed

In order to understand the attachment of the cotton fiber to the seed, the cotton seed was examined. Figure 38 displays a micrograph of a typical cotton seed in longitudinal section. Pertinent portions of the seed structure are labeled. A typical cotton seed cross section may be seen in Figure 39. A close up of a fiber root structure (870x) is shown in Figure 40. This micrograph revealed that the fiber had its only contact with the epidermal layer, and was not attached to any of the inner layers.

A micrograph of the cotton seed after mechanical saw-ginning revealed that a considerable amount of short fiber remained on the seed. It was previously reported that this was about 9.2% of the total weight of the seed.[1] Hand ginned cotton seed was found to contain considerably less fiber. Many fibers appeared to be broken off close to the seed surface, while a lesser amount appeared to be pulled out from within the seed. A comparison of the seeds after being ginned in both ways may be seen in Figures 41 and 42.

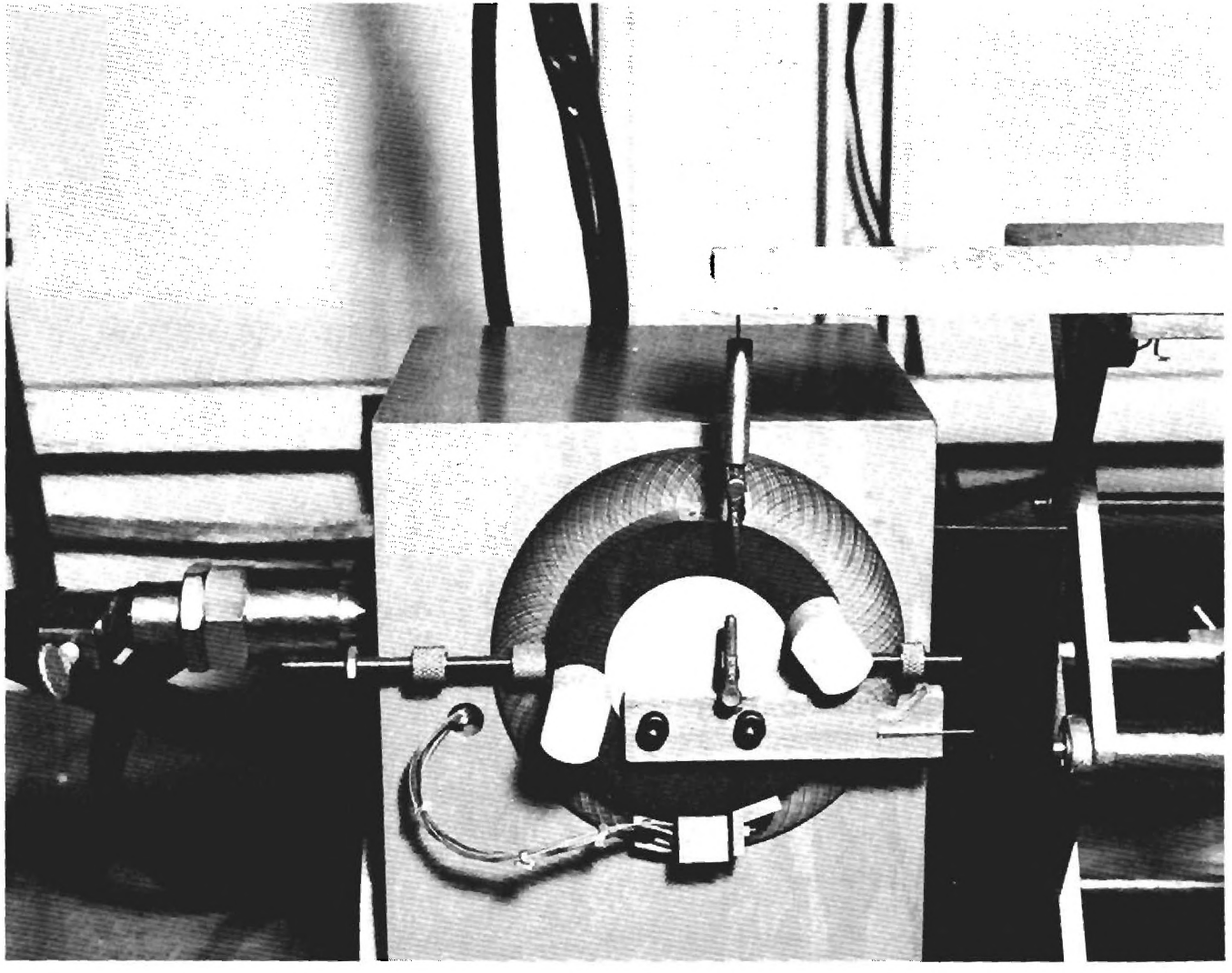

Figure 37. Dynamometer Modified for Measurement of Fiber Tensile Strength and Elongation.

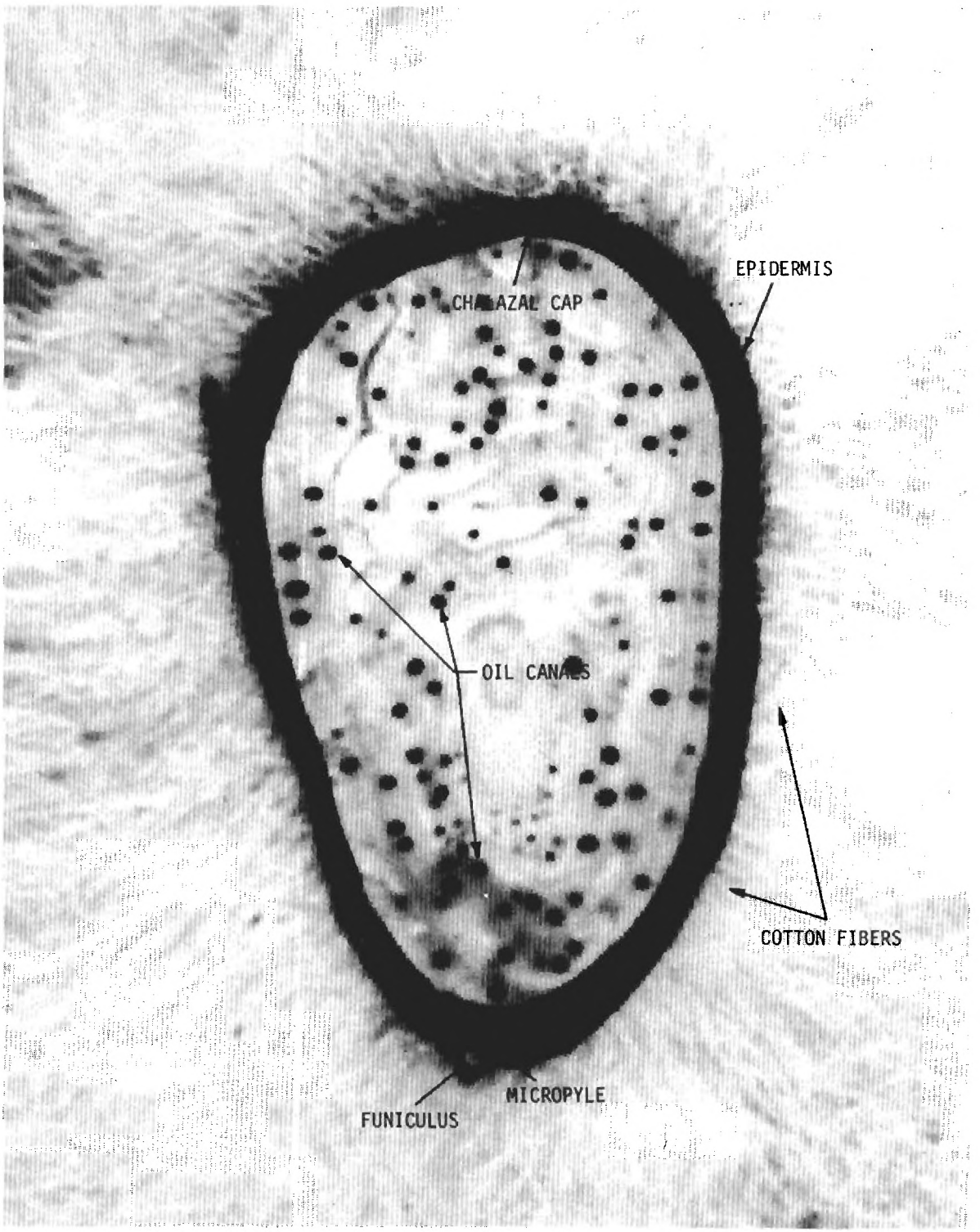

Figure 38. Empire WR Cotton Seed, Longitudinal Section (22x).

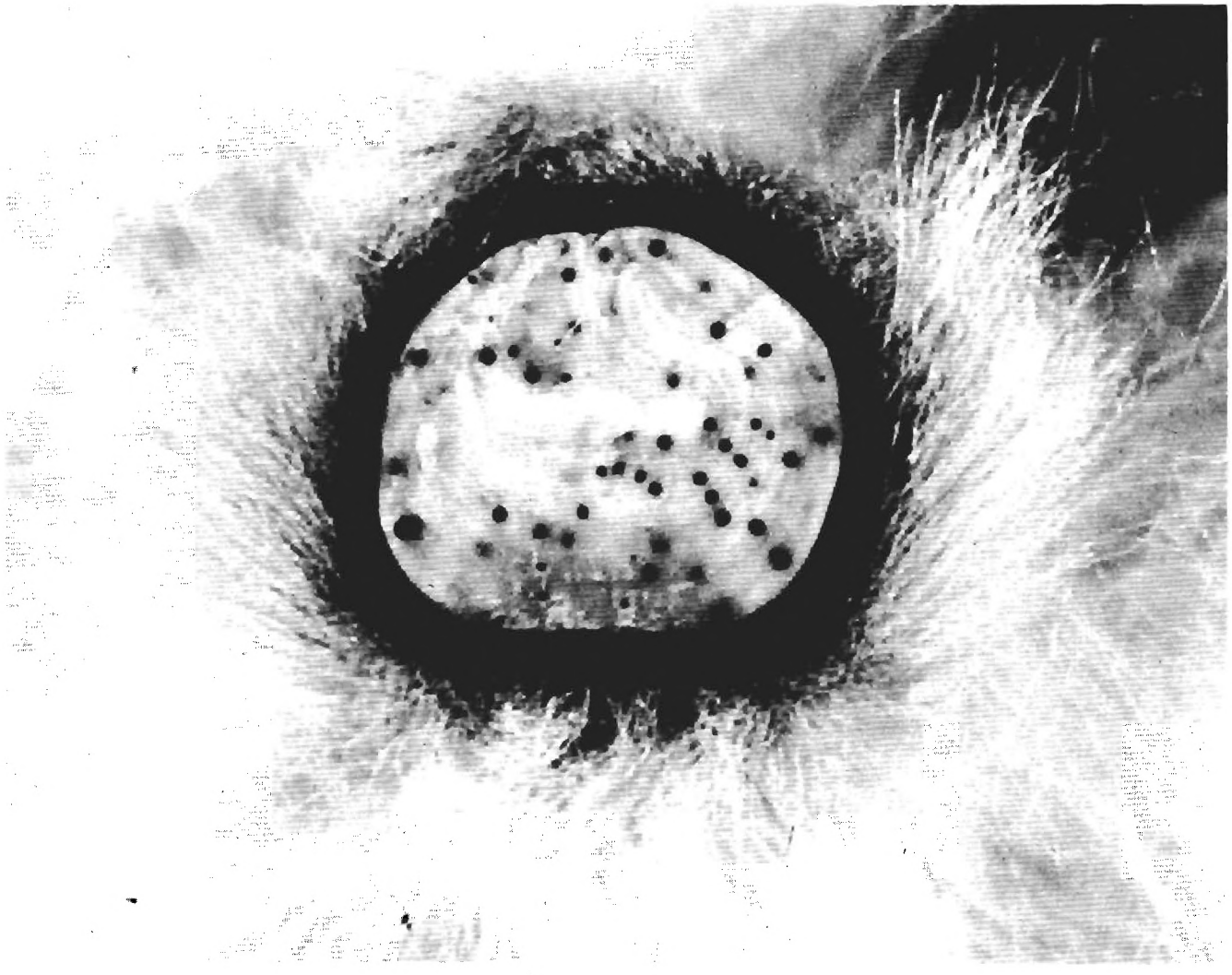

Figure 39. Empire WR Cotton Seed, Cross Section (22x).

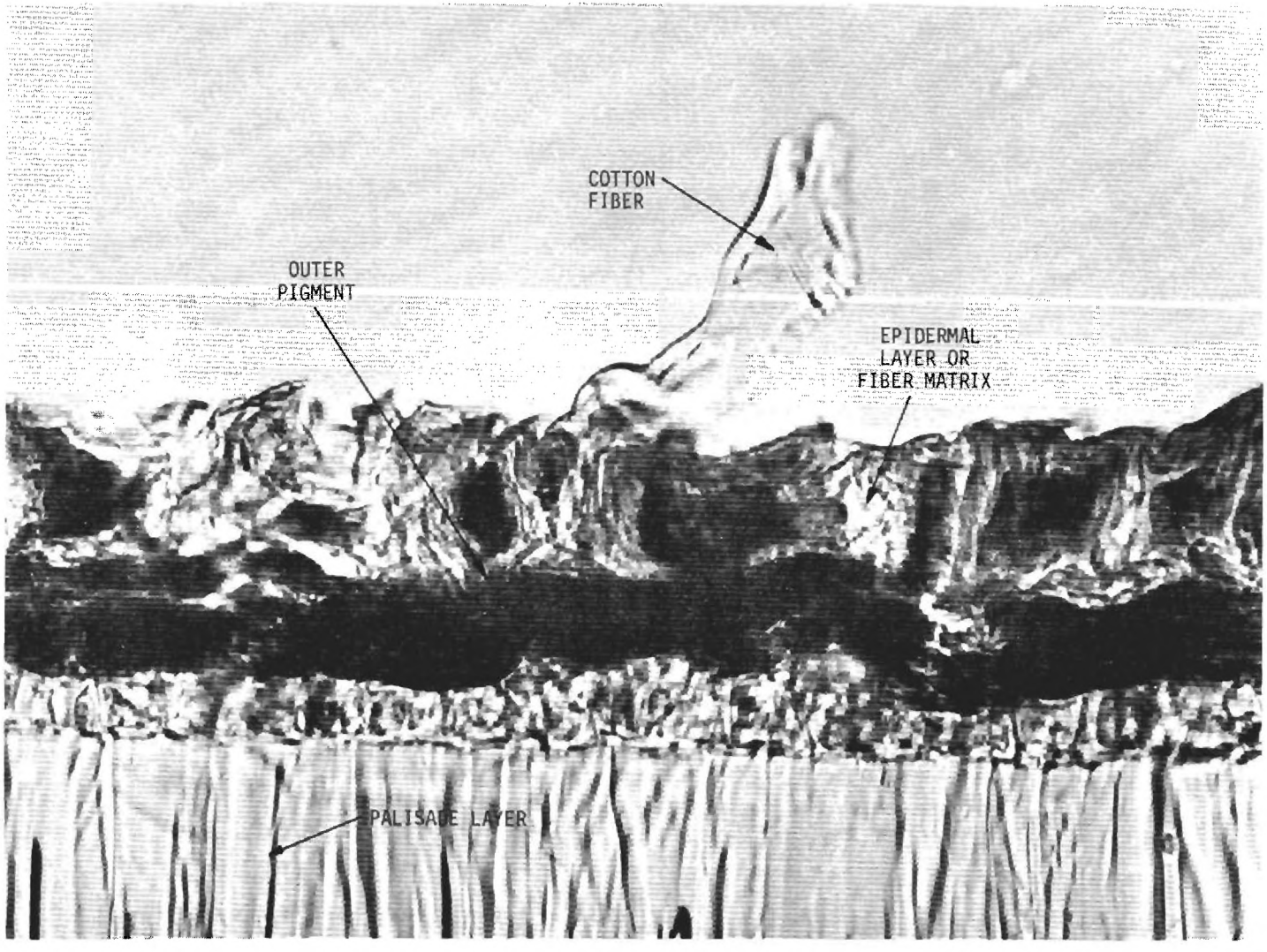

Figure 40. Empire WR Cotton Seed Displaying Root Structure and Epidermal Layer (870x).

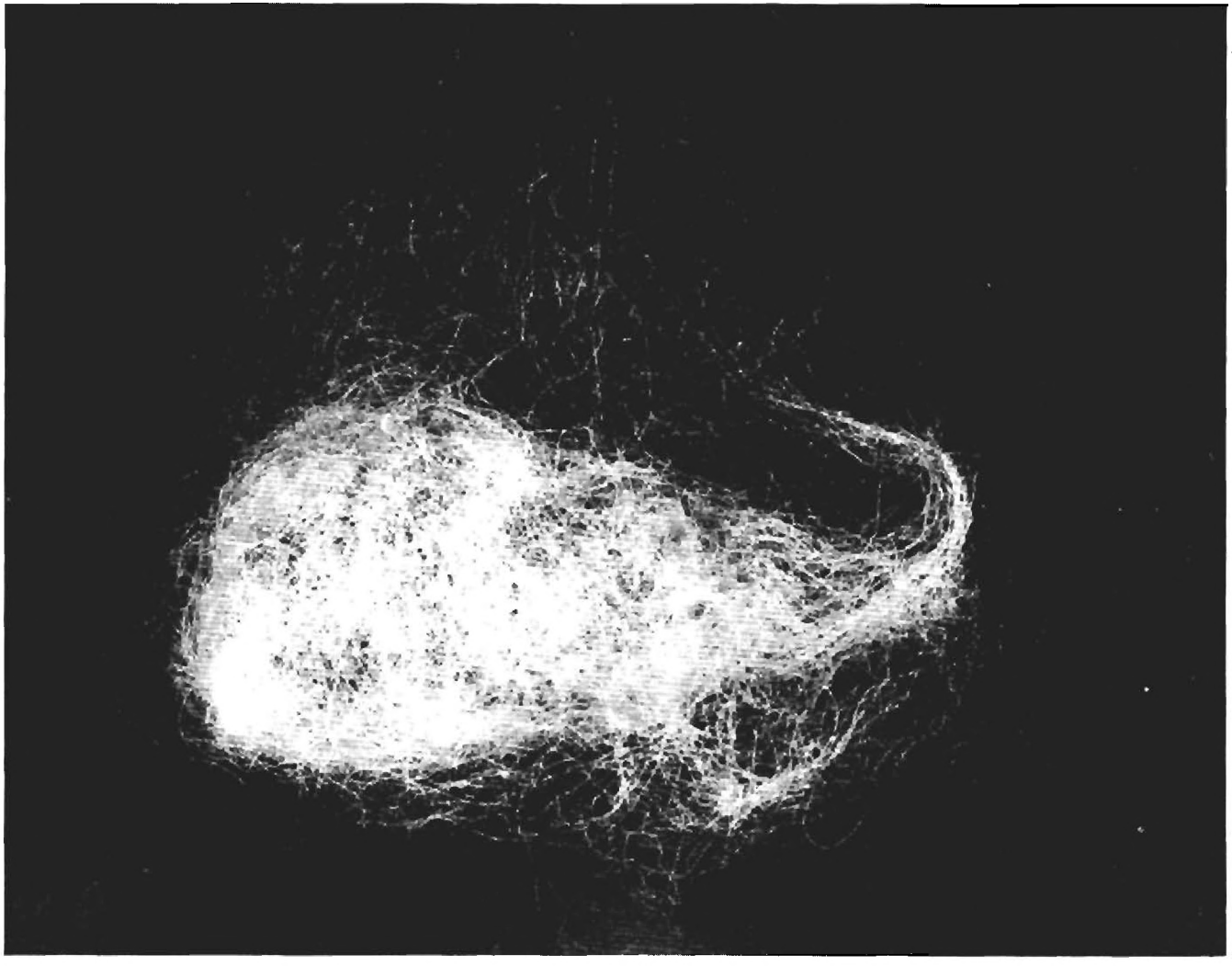

Figure 41. Typical Empire WR Cotton Seed after Mechanical Ginning (9x).

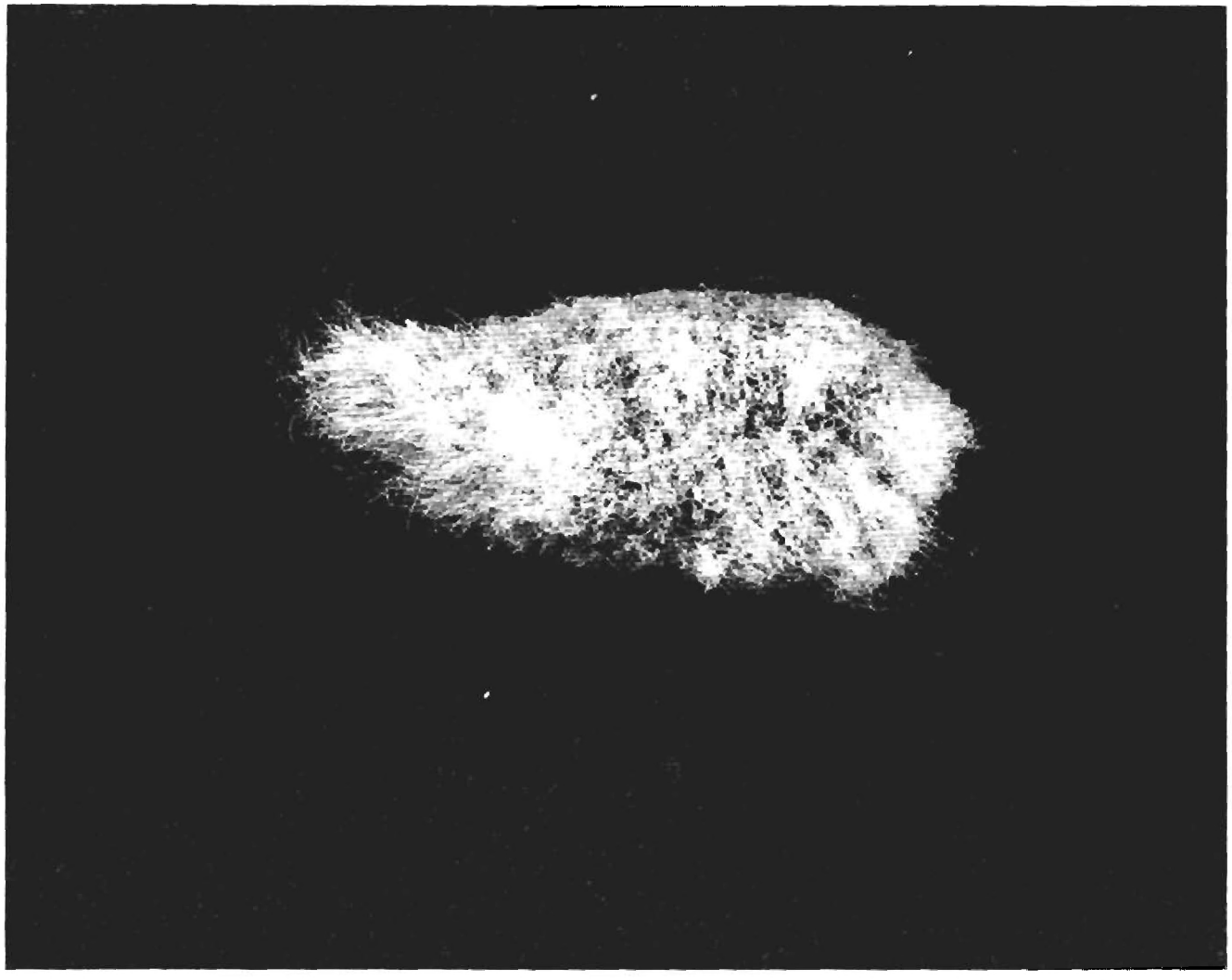

Figure 42. Typical Empire WR Cotton Seed after Hand Ginning.(9x).

c. Fiber Withdrawal Forces from Seed Before and After Heat Cycling and Fiber Breaking Forces

Measures of the forces required to remove cotton fibers from the seed were compared to the forces required to break cotton fibers at room temperature (22°C). Eighty measurements were made for each parameter. These data are summarized in Table 12. It was found that an average of 1.23 g was required to withdraw fibers from seed, and that the average fiber breaking force was 4.24 g. The ratio of the forces (F_B/F_W) was 3.45. Typical fiber withdrawal and fiber break measurement curves may be compared in Figures 43 and 44.

A comparison of the distributions of force of withdrawals and breaking forces may be seen in Figure 45. From the distributions, it was noted that 27.5% of the fiber breaking forces overlapped the fiber withdrawal forces.

The percent coefficients of variation (standard deviation x 100/mean) were calculated for the withdrawal forces and breaking forces and found to be 40.6% and 51.9% respectively for the eighty measurements taken of each. Using these data and an assumed total error of 10%, the statistical significance levels of both parameters were calculated by the following formula:

$$t = \left[\frac{n \cdot (\%E)^2}{(\%CV)^2}\right]^{1/2}$$

where: n = number of tests
E = total error
CV = coefficient of variation
t = value of Student's t-distribution

Determined confidence levels for fiber withdrawal forces and fiber breaking forces were 97% and 90% respectively.

Table 12. Summary of Forces of Withdrawal of Cotton Fiber from the Seed and of Fiber Strengths Before and After Heating

Test	Number of Tests	Temp.(°C)	Force (g)	Force %CV	Significance Level at %10 error
Fiber Break	80	22	4.24	51.9	90%
Fiber Withdrawal	80	22	1.23	40.6	97%
Fiber Withdrawal	20	50	1.17	29.6	85%
Fiber Withdrawal	20	100	1.40	31.7	82%
Fiber Withdrawal	20	150	1.54	40.2	72%
Fiber Withdrawal	20	200	1.36	26.6	89%

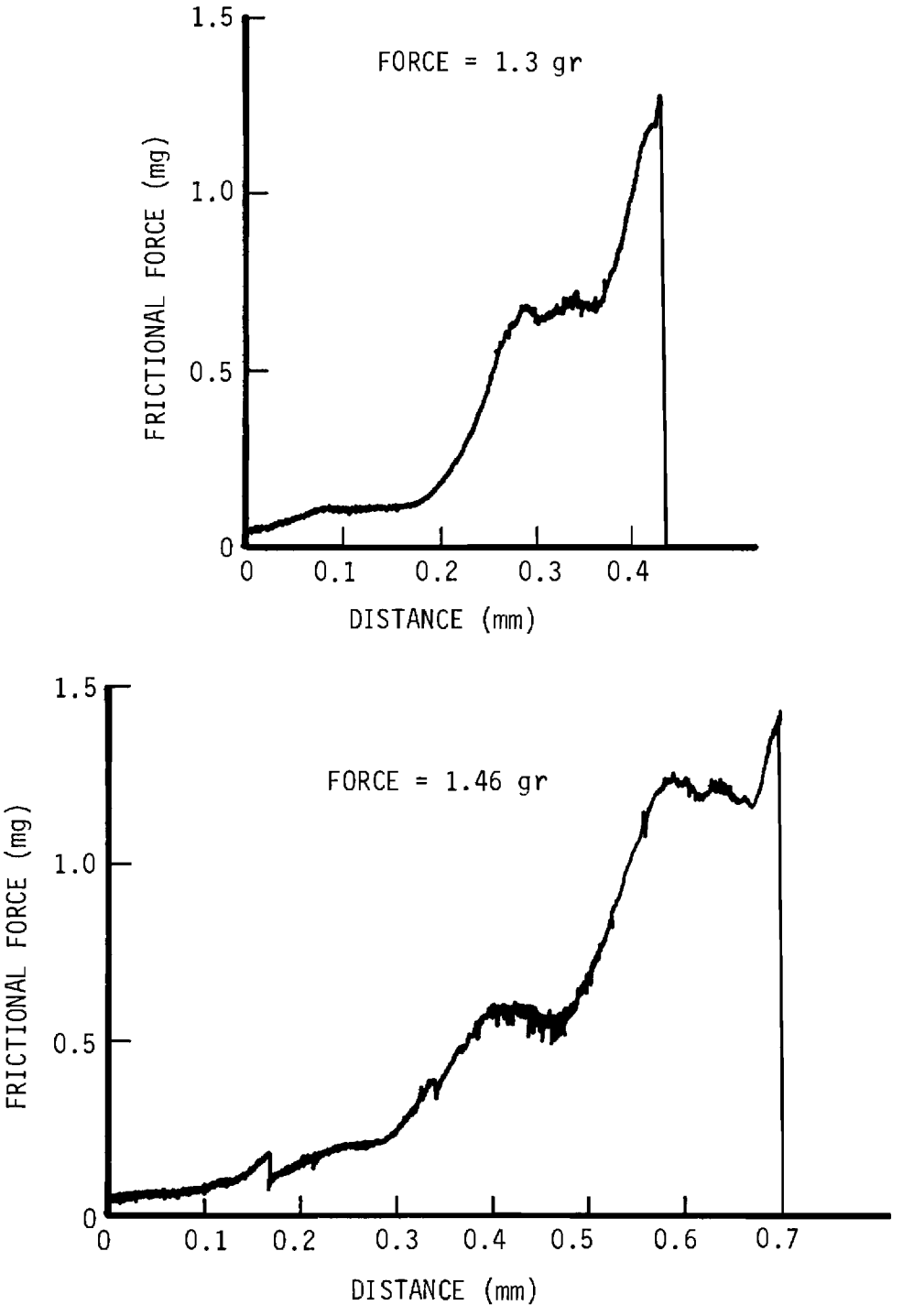

Figure 43. X-Y Plot of Typical Withdrawal Forces Required for Removal of Cotton Fibers from the Seed (Empire WR).

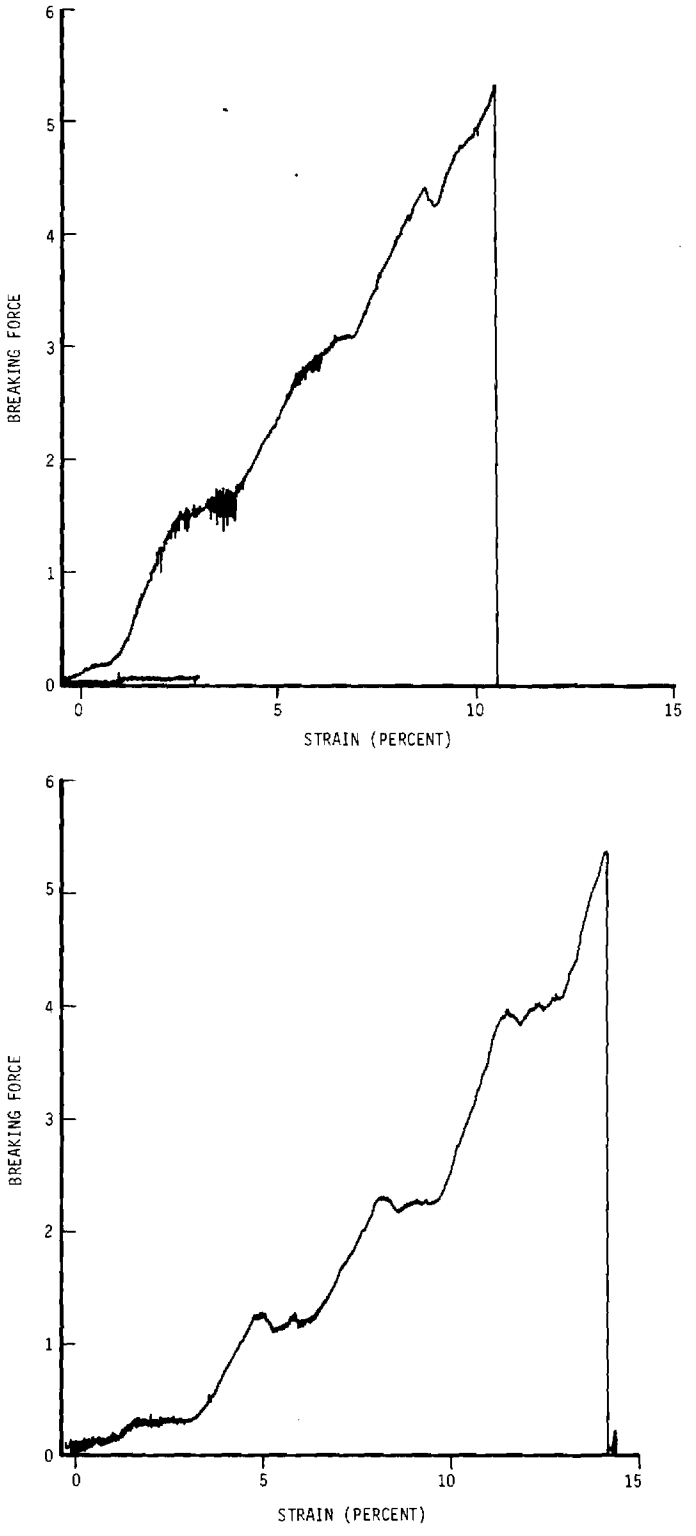

Figure 44. X-Y Plots of Tensile Forces Required to Break Typical Empire WR Cotton Fibers.

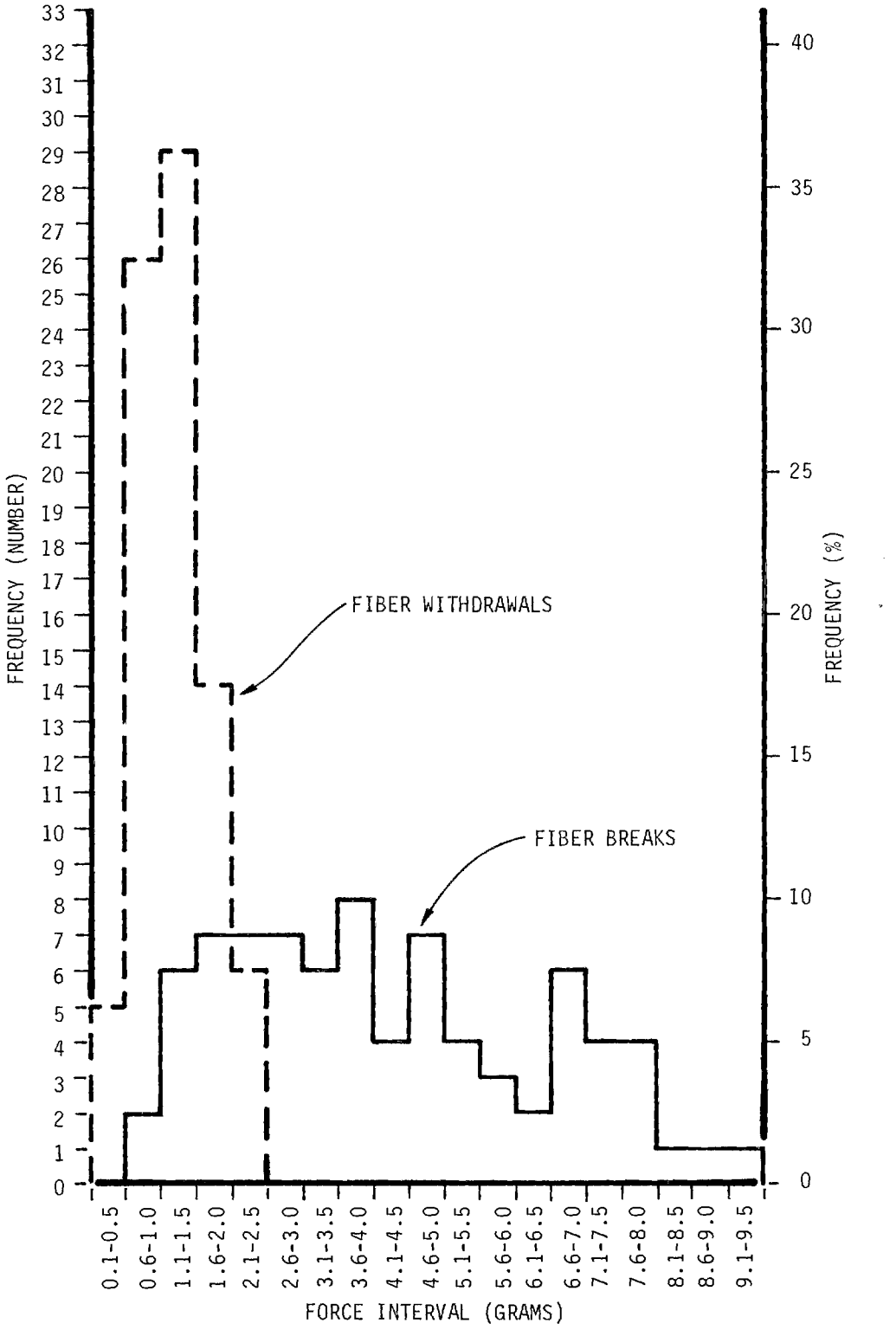

Figure 45. Intercept of Force Distributions of Fiber Withdrawal Force and Tensile Strengths for Empire WR Cotton.

Since the seed cotton is subjected to temperature cycling during the drying phase of ginning, the fiber is normally withdrawn from the seed while still warm. The temperature at which the cotton is ginned may vary from gin to gin and from cotton batch to cotton batch according to the ginner's estimate of drying requirements. In order to study this effect, the cotton was temperature cycled at 50°C, 100°C, 150°C, and 200°C for 60 seconds, and fiber extraction forces were measured 24 hours* later.

Measurements of the fiber withdrawal forces with respect to temperature for 20 fibers at each temperature level revealed that heating seed cotton to 50°C did not cause any changes, but heating to 100°C and 150°C caused increases of fiber attachment forces of 13.8% and 25.2% respectively. Heating to 200°C caused severe damage (as was evidenced by the yellowing of the fiber) and only a 9.55% increase in attachment force. Significance levels were calculated for results of each temperature as before.

A summary of results of the fiber withdrawal force and tensile force measurements was shown in Table 12. The effect of temperature on fiber attachment force is graphically presented in Figure 46.

5. Fiber Withdrawal Energies from Seed Before and After Heat Cycling and Energies of Fiber Ruptures

Measures of the energies required to remove cotton fibers from the seed were compared to the energies to break cotton fibers. These data are reported in Table 13. It was found that an average energy of 64.9 ergs was expended in withdrawing single fibers from seed, and the average energy expended in rupturing fibers was 250.3 ergs. The ratio of the energies (E_B/E_W) was 3.90. These values correlated well with those reported by Prakash and Iyengar[16] and Smith and Pearson.[17]

A comparison of the energy distributions may be seen in Figure 47. From the distributions, it was noted that 51.2% of the fiber breaking energies overlapped the fiber withdrawal energies.

Since the curves obtained from force measurements were used to calculate energies, the same number of tests as before were used for calculations. Coefficients of variation and significance levels were computed also as before.

* A shorter time interval would have been more desirable. However the data presented still indicated a trend which appears of importance.

Table 13. Summary of Energies Required for Withdrawal of Cotton Fiber From the Seed Before and After Heating and for Fiber Rupture

	Number of Tests	Temp.(°C)	Energy (ergs)	Energy CV	Significance Level at 10% error
Fiber Withdrawal Measurements					
Fiber Withdrawal	80	22	64.9	66.8	82%
Fiber Withdrawal	20	50	90.5	52.1	58%
Fiber Withdrawal	20	100	62.6	53.7	57%
Fiber Withdrawal	20	150	92.8	75.4	44%
Fiber Withdrawal	20	200	90.9	71.3	45%
Fiber Rupture Measurements					
Fiber Rupture	80	22	250.3	78.3	75%

$$\text{Ratio: } \frac{\text{Fiber Rupture Energy } (22^\circ C)}{\text{Fiber Withdrawal Energy } (22^\circ)} = 3.78$$

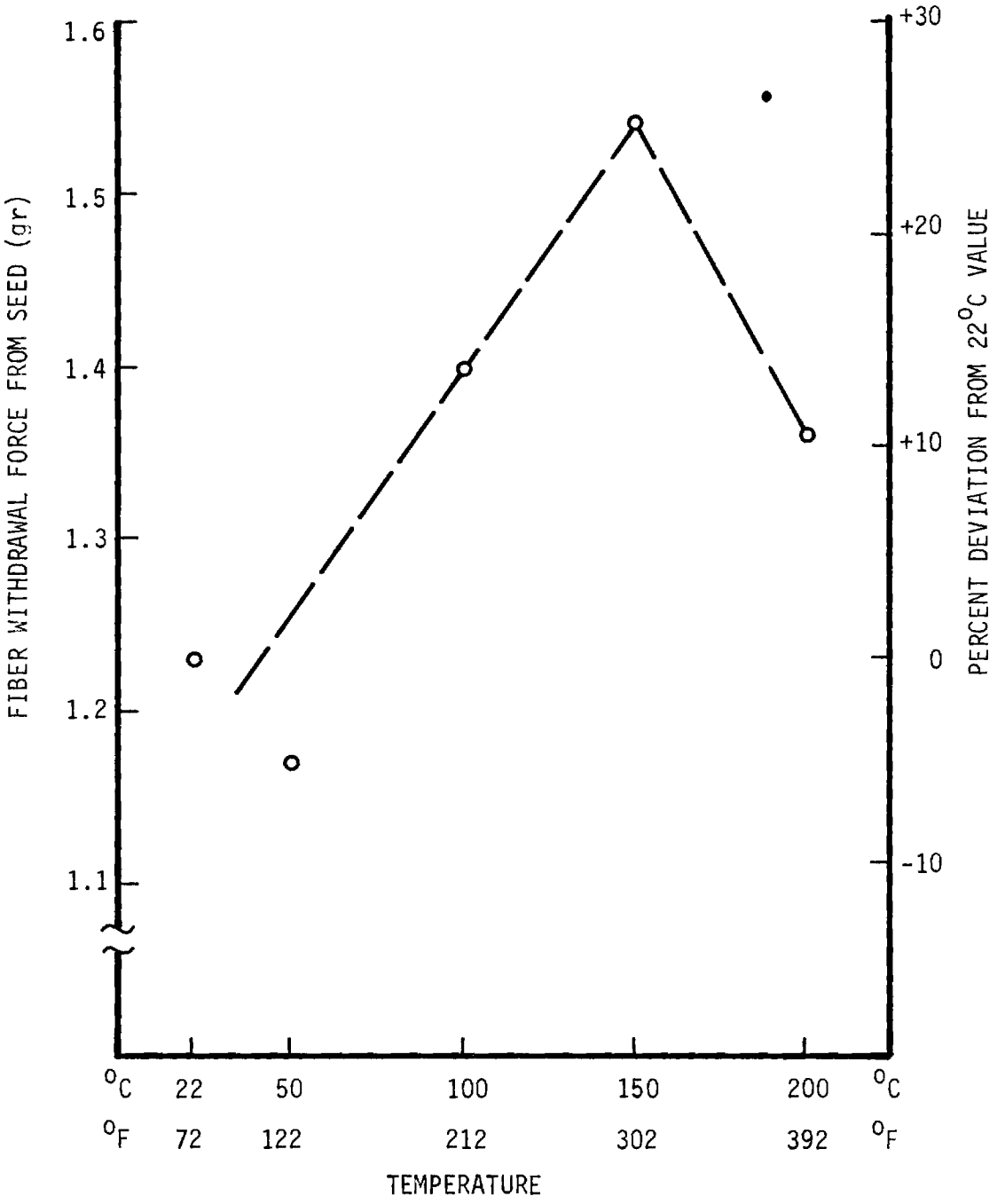

Figure 46. Plot of Variation of Fiber Withdrawal Forces Versus Temperatures to which Cotton Empire WR Seed Had Been Previously Heated.

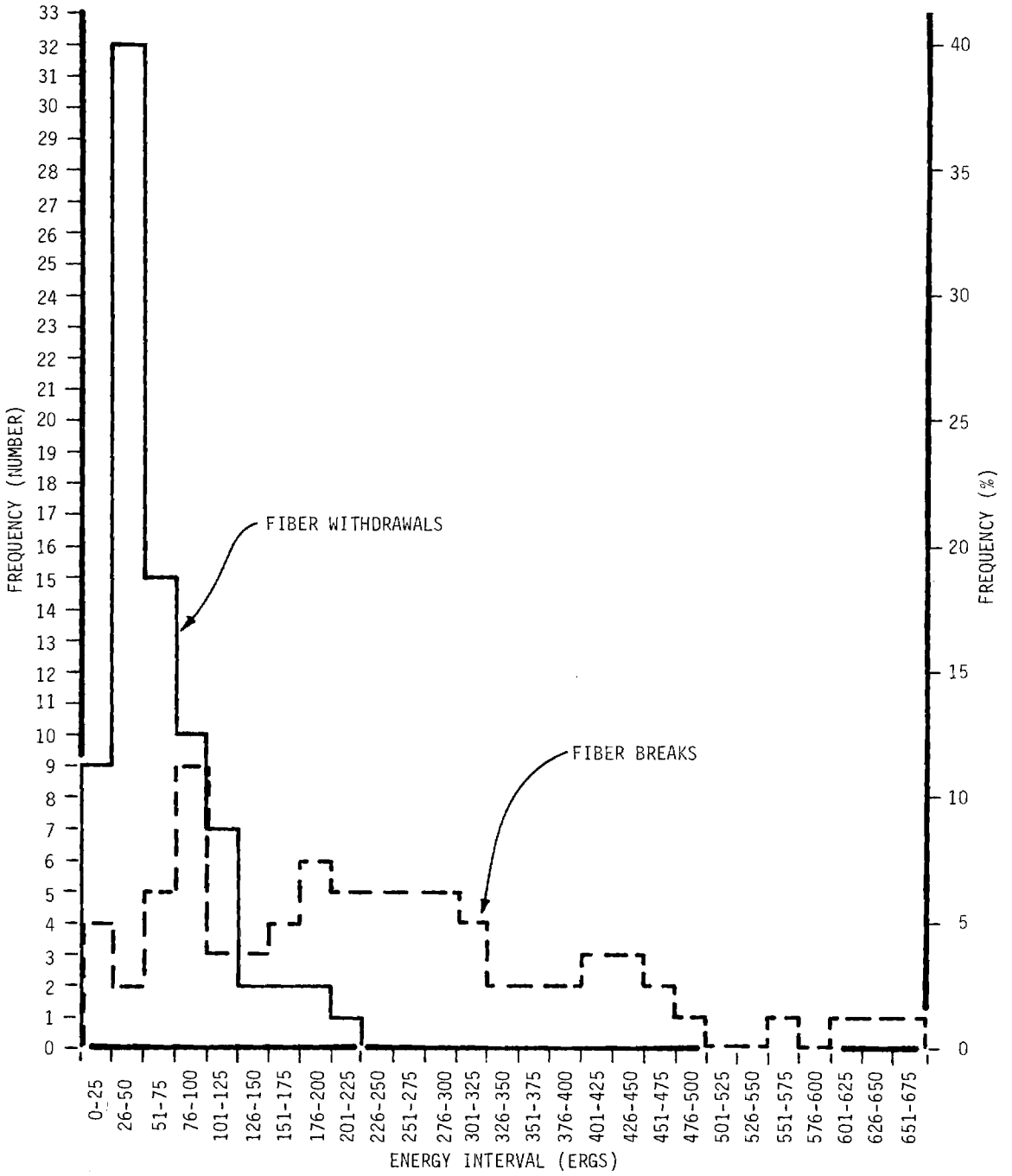

Figure 47. Intercept of Energy Distributions of Fiber Withdrawals and Fiber Tensile Breaks for Empire WR Cotton.

Expended energies during fiber extraction from seed were also calculated from measurements taken after heating the seed cotton. These are summarized in Table 13. More energy was required with increased temperatures. The relationship of expended energy vs temperature is shown in Figure 48.

The variability within samples of the energy computations was found to be considerably higher than for force parameters. Hence, the statistical significance of the data was not as great as for force measurements.

The increased amount of energy required for fiber extraction because of heating is shown to be as high as 43% which is of some consequence for the ginner since more power is required for ginning. The amount of power required to gin a bale of cotton (500 pounds) was calculated, assuming that the average weight per cotton fiber was 4.0 μg (4×10^{-6} g).

$$\text{Number of Fibers in a Bale of Cotton} = \frac{500 \text{ lb} \times 454 \text{g/lb}}{4 \times 10^{-6} \text{ g/fiber}} = \underline{5.67 \times 10^{10} \text{ fibers}}$$

$$\text{Power Consumed per Bale in Extracting Fiber from Seed} = 64.9 \text{ ergs/fiber} \times 5.67 \times 10^{10} \text{ fibers} \times 2.78 \text{ KWH/erg} \times 10^{-14} = \underline{0.102 \text{ KWH}}$$

Although power requirements for extraction of fibers from seeds (per bale) is of the order of 0.1 kilowatt-hours, the total power consumed is considerably higher since a great deal of power is used only for the movement of the machinery. The exact amount of power consumed is largely dependent on the efficiency of the particular gin equipment.

6. Effects of Heat on Convolutions of Cotton Fibers

Measurements of fiber convolution changes as a result of heating were made and are the same as those discussed in Section III, B, 7, since this work was performed jointly by House and Goldfarb. See Table 14.

Table 14. Percent Increase in Convolution Count of Empire WR Cotton After Heating

Temperature (°C)	% Increase After 15 Minutes	% Increase After 24 Hours
22	3.6	0.0
40	10.7	0.0
60	8.3	0.0
80	0.0	0.0
100	3.0	0.0
120	5.0	0.0
130	16.7	12.5
140	8.6	5.7
150	27.4	9.1
160	40.9	27.2
180	20.0	16.0
200	26.7	26.7

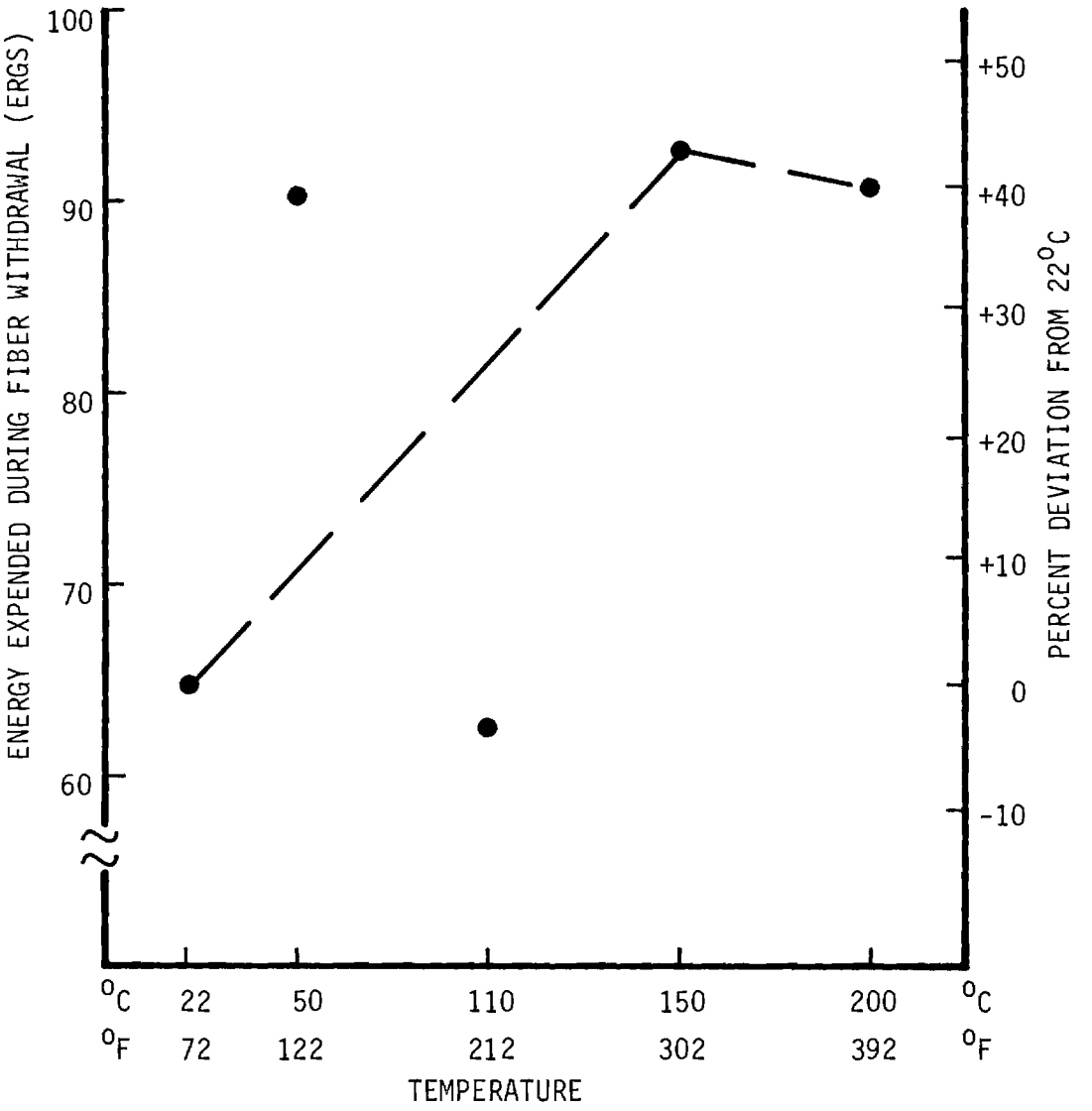

Figure 48. Plot of Variation of Fiber Withdrawal Energies Versus Temperature to Which Cotton Empire WR Seed Had Been Previously Tested.

7. Effects of Ginning on Fiber Length Distribution

The examination of staple length distribution properties from Digital Fibrograph fibrograms, as reported in Semiannual Report No. 2 of this Grant, revealed that the mean length of ginned cotton was 9.4% to 15.6% shorter than corresponding seed cotton. Upper half mean length measurements showed reductions of 3.8 to 9.7%. The 50% span lengths were reduced from 8.7 to 17.6%, and 2.5% span lengths lessened from 3.0 to 7.4%.

Data from the Suter Webb Arrays, indicated a lesser degree of fiber length shortening as a result of ginning. Decreases in mean length ranged from 3.03 to 7.85%. Generally, considerably more short fibers (less than 5/8") were found in ginned lint than seed cotton while examining the weight-length distribution graphs (Tables 17 to 26 and Figures 28 and 32 of Goldfarb's thesis).

It was interesting to note that both Carolina Queen seed cotton specimens (manually harvested and mechanically harvested) had almost identical Fibrograph length properties. After ginning, however, the mechanically harvested sample was considerably shorter than the hand picked specimen.

A summary of the various length measurements is given by Goldfarb.

8. Comments

Of particular interest to the study of ginning of Empire WR cotton was the measurement of the fiber withdrawal force by employment of a new and sensitive force measurement method, the servo-torque dynamometer described in Section III, F, 3b. The average value of the withdrawal force for the fibers of seed Empire WR cotton was found to be 1.23 gms * and to increase to 1.54 gms at 150°C (302°F). The apparatus was modified to replace the windlass withdrawal method by a translational system using a motor driven, worm gear of 7 threads per inch to provide withdrawal. A recheck of the withdrawal force for 100 fibers gave precisely the same value of 1.23 gms for the unheated seed cotton. A typical withdrawal is shown in Figure 43 exhibited previously.

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* The values of the fiber withdrawal force obtained for Deltapine Cotton by Smith and Pearson was 1.10 gms and for other cottons of similar types the values ranged up to 1.25 gms.[17]

In addition, measurements were made of fiber breaking forces and elongation. Typical tensile data plots are shown in Figure 44. Here it will be noted that the curves possess much detail with respect to what appears to be slip or some other phenomena relating to fiber behavior. In Figure 44 (lower) it will be noted that a large part of the elongation of 14% is accounted for by the slip, leaving a balance of 7.7% as a reasonable net value of elongation. A similar curve but with less extensive slip is shown in Figure 44 (upper). The average value of the breaking strength of 37 fibers was 5.01 gm somewhat larger than the value reported by A. Goldfarb. The servo torque dynamometer appears to have excellent applicability as a force measuring instrument in textile studies.

The hot air gun gin dryer simulator described also appears to have a number of uses in temperature cycling of textile fibers. The fiber form and length changes described are in general agreement with data reported by Berriman.

The effects of ginning on the friction of cotton fibers have been discussed in Section III, C, 7. It is apparent that the various fiber processing stages, including temperature cycling and the ginning action do contribute to an increase in fiber friction. It is apparent as well that the character of the curves will display differences that may show up in the ratio μ_s/μ_k and occasional very high frictional peak heights indicative of fiber damage.

G. EFFECTS OF OPENING, CLEANING, PICKING AND CARDING ON COTTON FIBERS

1. General

The effects on the physical properties of the cotton fiber of the processing stages opening, cleaning, picking, and carding have been examined by Mr. Howard R. Levy, Graduate Research Assistant, in a thesis for the Masters Degree of Science in Textiles (September 1966). The complete thesis has been submitted concurrently with this report.

2. Summary of Experimental Work and Results

Eighty pounds of Empire WR cotton were processed through the stages of opening, cleaning, picking, and carding at the A. French Textile

School, Georgia Institute of Technology. Random samples were taken after each processing stage; and the strength, elongation, fineness, length distribution, fiber-to-fiber friction and the surface characteristics of the fibers were evaluated.

The Pressley fiber strength test, the Instron and the Servo Torque-Balance Dynamometer were used to evaluate the changes in the tenacity of the fibers. The Pressley Bundle measurement indicated a small increase (10 percent) in strength over the stages opening through carding, whereas the other instruments, based on single fibers, indicated little change.

The Instron and the Servo Torque-Balance Dynamometer were also used to measure the elongation of the individual fibers. The elongation of the fibers as indicated by the Instron apparatus revealed a decrease in the percent elongation. Whether this is statistically real or not is uncertain. However, it is logical to expect that it may be real as many of the fibers are undoubtedly partially stretched during processing.

When the micronaire of the stock that had not been opened was compared with that of the opened stock, and with that processed through the stages of picking and carding, it was found that a significant difference existed between the samples. The micronaire of the stock increased from 4.00 micrograms per inch before opening, to 4.01 micrograms per inch after opening, to 4.19 micrograms per inch after picking, to 4.37 micrograms per inch after carding. This steady increase in the micronaire of the cotton indicates that the finer immature fibers are being removed as the cotton proceeds through the textile processes or that fiber damage is seen as an increase in size.

The mean length of the fibers as measured by the Digital Fibrograph was 0.848 inch before opening and cleaning, 0.693 inch after opening and cleaning, 0.703 inch after picking and 0.878 inch after carding. The fibers were processed through the opening and cleaning line which contained various types of beaters. Since the fibers were held by spikes while the beaters struck them, some fibers were broken, causing a decrease in the mean length due to the opening and cleaning processes. During the picking process, the mean length increased by 0.01 inch. This increase in mean length, if it is significant, can be attributed to the small amount of

waste removed as short fibers by the picker. The large increase in mean length after carding from 0.703 inch to 0.878 inch is believed to occur because of the large amount of waste and short fibers removed at the card.

The coefficient of kinetic friction exhibited successive increases through the steps 0.275, 0.289, 0.307, and 0.317 during the various stages of textile dry processing.

The number of fibers damaged, as indicated by the Congo Red test, after each process indicated a large upward trend. Eighteen percent of the fibers were damaged before opening, 24 percent were damaged after opening and cleaning, 40 percent were damaged after picking and 35 percent were damaged after carding.

All of these measurements point to successive damage to the fiber as it proceeds through the various stages. However, the removal of shorter, broken, or immature fiber, especially at the carding stage, resulted in a final product of strength and mean length equivalent or greater than in the beginning with a loss in total weight value being the principal sacrifice.

3. Comments

The study of the changes in the physical properties of cotton fibers resulting from the stages opening, cleaning, picking, and carding have resulted in three measurements of principal interest. These are the change in fiber mean length through the various fiber processing stages, the changes in frictional behavior, and the increase in fiber damage.

A summary of data describing the mean length change is shown in Figure 49. It is evident that a large decrease in fiber mean length must be accompanied by considerable fiber breakage. The increase in length after carding implies that the short fibers were largely discarded at this stage. Unfortunately the carding waste was not examined in this experiment.

The changes in frictional behavior of the fibers at the various stages were exhibited in Figure 24 and discussed in Section III, C, 6. It is evident here that each processing stage affects the fiber in such a manner as to increase the coefficients of kinetic and static friction and to change the character of the frictional analog. Very high stick peaks occur and

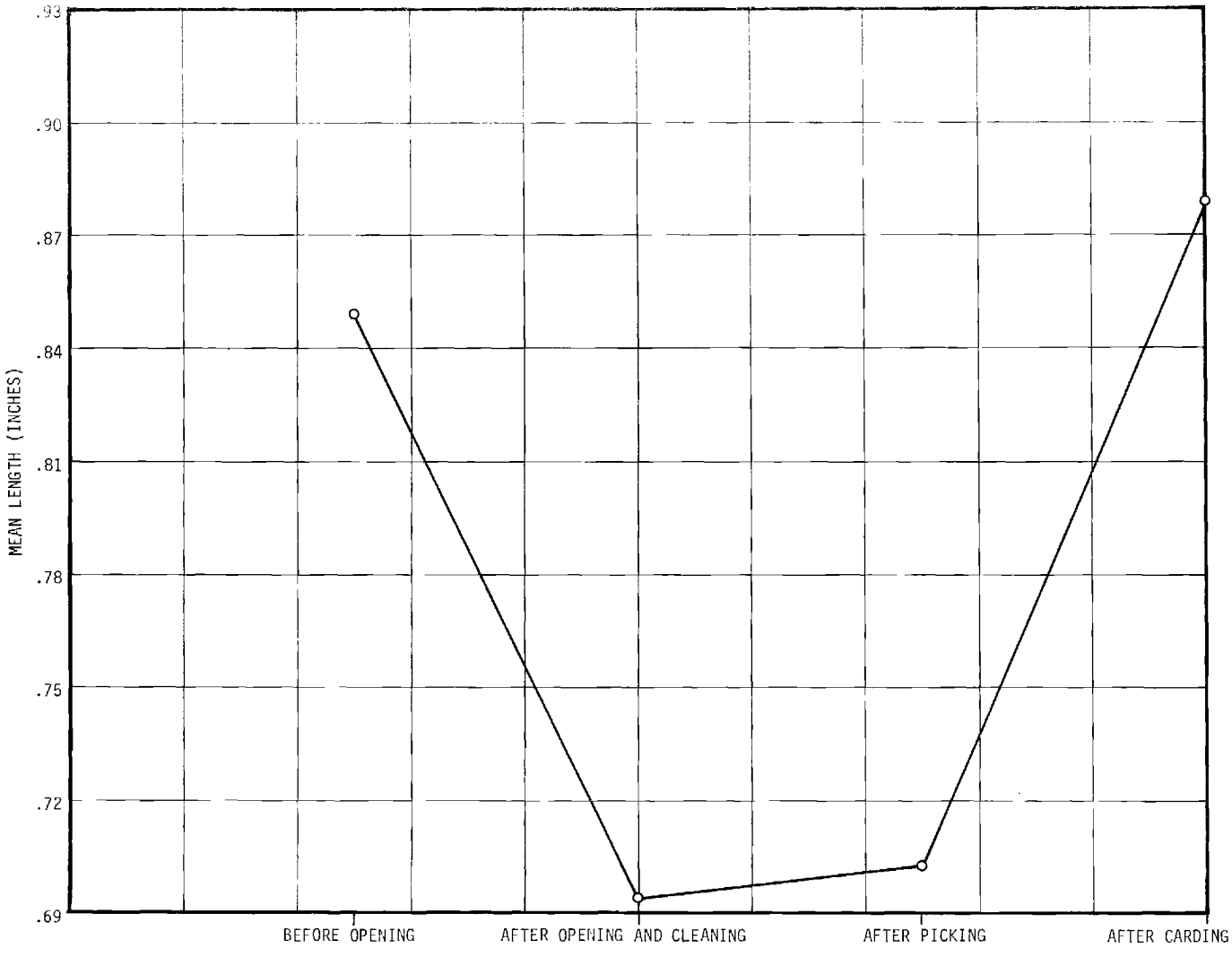

Figure 49. Variation of Mean Length of Empire WR Cotton Fiber Processed through the Stages Opening and Cleaning, Picking, and Carding.

these are undoubtedly due to fiber damage as suggested by the preceding discussion on fiber length changes and as indicated by the Congo Red method of fiber damage examination.

The evidence determined from the Congo Red examination is exhibited in Figures 50 and 51. From the micrograph it is readily observed that the damaged fiber portion would become a frictional snag. A snag of this type would account for the increase in large peaks occurring on the frictional plots of the fibers after picking and carding. In addition the Congo Red examination indicated that 40 percent and 32 percent of fibers exhibited damage after picking and carding respectively. Clegg[6] indicated about 24 percent damaged after picking and 35 percent after carding. Hence, we have evidence to exhibit that about 1/3 of the fibers have been damaged, resulting in changes that affect the fiber behavior in subsequent processing.

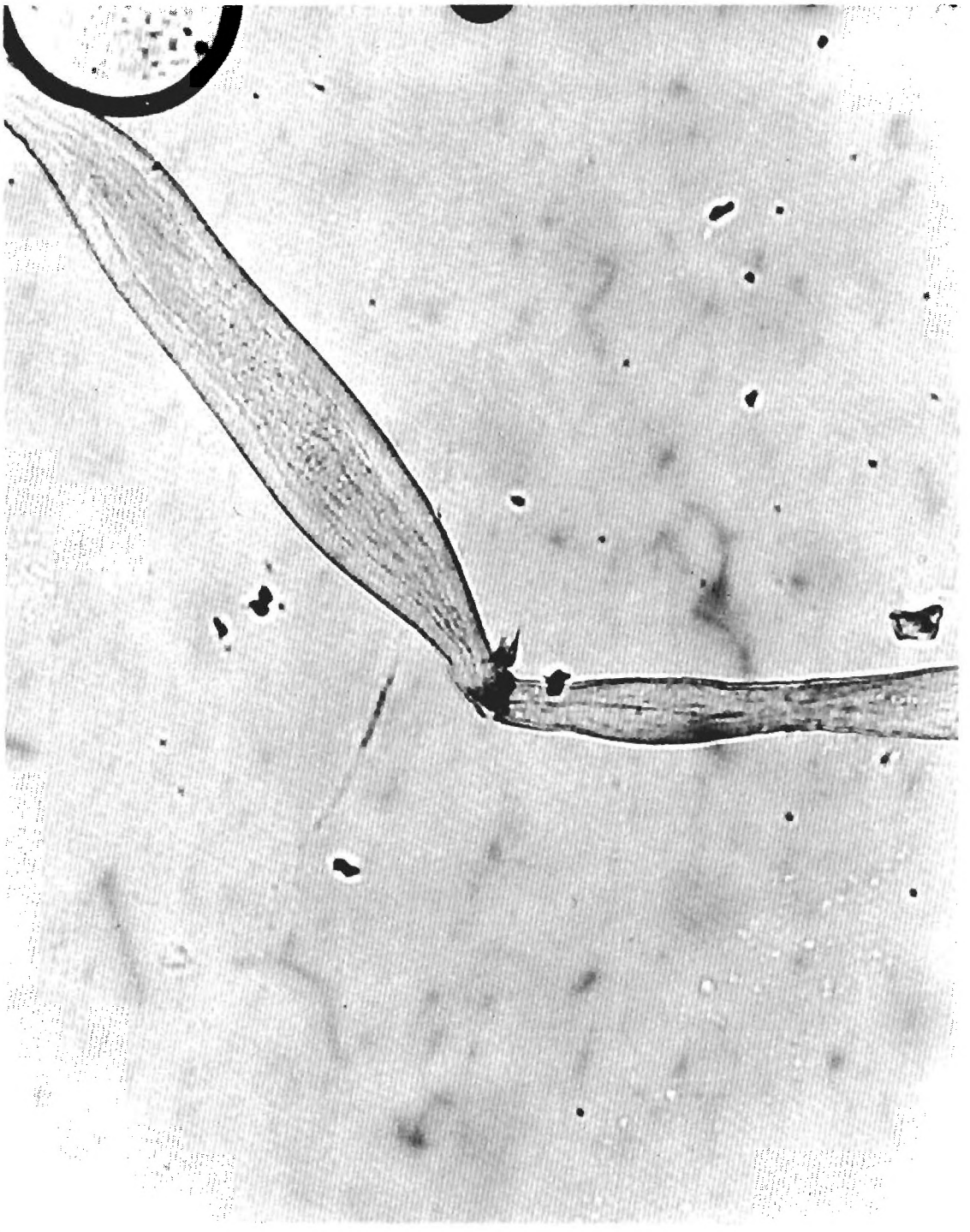

Figure 50. Photomicrograph of Empire WR Cotton Fiber Exhibiting Damage Resulting from Processing; Detected by the Congo Red Method.

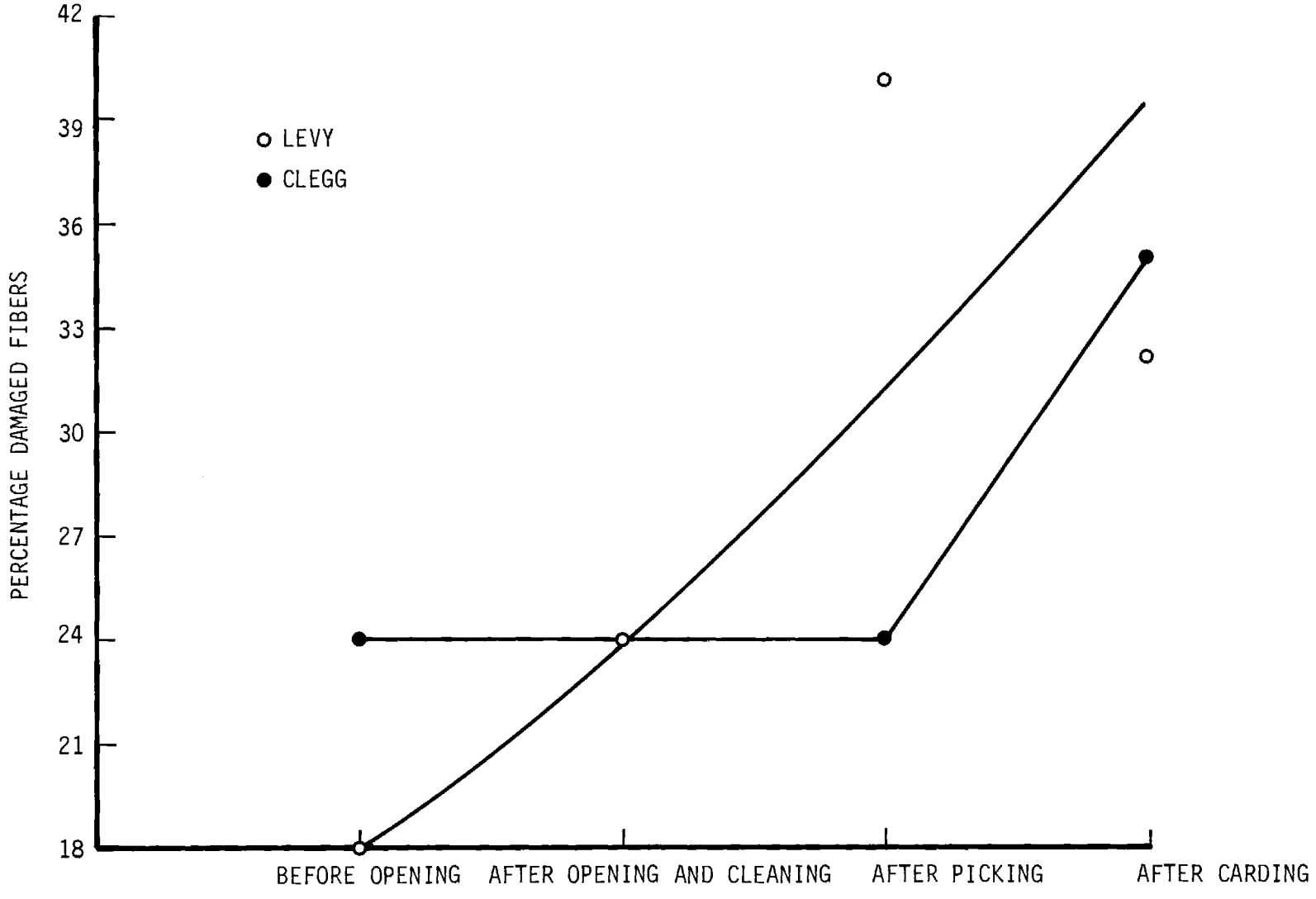

Figure 51. Variation in Number of Damaged Fibers Occurring as a Result of Processing through the Stages Opening and Cleaning, Picking, and Carding; Detected by the Congo Red Method.

IV. CONCLUSIONS

Improvements in frictional measurement techniques and apparatus and better control of fiber tension and normal force between fibers enabled us to make valid and repeatable measurements of the coefficients of static and kinetic friction of cotton and other fiber materials. A new apparatus, employing an electromagnetically applied normal force, extended our normal force range below the previous limit of 20 mg to 2 mg. The coefficients of static and kinetic friction for cotton, at 20 mg normal force, were found to decrease from 0.356 to 0.236 and 0.647 to 0.483 respectively as the tension at mounting was increased from 125 mg to 1125 mg. For cotton subjected to temperature cycling to temperature plateaus in the range 25° to 220°C the friction was found to increase from a value of 0.285 (for ginned cotton) to 0.327. For processed cotton the values 0.262, 0.275, 0.294, 0.308, and 0.322 were observed for the coefficients of kinetic friction for hand ginned, mechanically ginned, opened and cleaned, picked, and carded cotton respectively. In particular, the character of the friction data plots were observed to reflect various treatments indicative of fiber damage. Large frictional stick maxima were frequently observed and these resulted in an increase in the ratio μ_s/μ_k. A similar result was noted for the cotton subjected to temperature cycling, to wetting, and to coating with a very small amount of vacuum evaporated aluminum.

Frictional data for a cotton fiber against a glass fiber and nylon against glass indicated increases in the coefficients of kinetic and static friction as the normal force was decreased from 5 mg to 2 mg. The values obtained for cotton (against glass) were 0.344 and 0.398, 0.589, and 0.902, respectively giving μ_s/μ_k ratios of 1.73 and 2.27, respectively.

Other experiments reported in detail in five theses[2,3,4,5,6] extended the work in examination of fibers by optical and electron microscopy, infrared spectrometry, and other physical test methods. In particular extensive investigations were made of the effects of fiber processing on fiber appearances, dimensions, strength, elongation, friction, and damage as the fiber was processed from the boll through carding. The mean length of the fibers of Empire WR cotton was found to shorten from about 0.85 to 0.70 from the

ginned cotton through picking. However, after carding the mean length increased to 0.88, indicating removal of short fibers at this stage and a loss of yield. Congo Red dye tests indicated an increase in fiber damage from 18 percent after ginning to 40 percent after picking, which damage was reflected in the coefficients of friction as previously noted.

Other developments of interest were: a catalog of optical and electron micrographs of 17 different cottons and 14 man-made fibers; a sensitive torque dynamometer method of measuring the force of withdrawal of fibers from the cotton seed, tensile strengths, and elongation of fibers; and a fiber press technique for preparation of specimens for examination by infrared spectroscopy. The latter were especially suitable for examination with polarized radiation and exhibited higher discrimination in the 7 to 10 micron region. Use of the polarizer allowed splitting of prominent bands in the spectrum into lesser bands. For instance a band at 6.15 μ was found to consist of a principal band at 6.05 μ with lesser bands on either side.

Further applications of the instruments and techniques described are expected to give extensive further information concerning frictional behavior and fiber properties during the ensuing period.

V. PROGRAM FOR THE NEXT INTERVAL

The program for the next interval is to further analyze and consolidate the extensive data now on hand and to extend the fiber to fiber friction measurements to the low normal force ranges, 2 to 5 mg. Investigation of adaptation of an electron microprobe to electron reflection microscopy will be made. Other areas such as x-ray diffraction and infrared spectroscopy will receive lesser emphasis. Frictional measurements of cotton fibers after successive cotton processing stages will be reviewed and investigations of changes occurring as a result of spinning will be commenced.

VI. PERSONNEL

The principal individuals employed on this research and the area of their interest are listed below.

Individual	Title	Area of Research
Richard B. Belser	Research Associate Professor	Project Director
James L. Taylor	A. French Textile School, Director	Associate Project Director
William L. Hyden	Professor, Textile Engineering	Associate Project Director
John L. Brown	Director, Analytical Instrumentation Laboratories	Optical and Electron Microscopy
James L. Hubbard	Assistant Research Physicist	Optical and Electron Microscopy
James A. Knight	Research Professor, Chemistry, Head, Radioisotopes Laboratory	Infrared Spectroscopy
Marvin P. Smoak	Student Assistant (Physics)	Infrared Spectroscopy
Lester D. Dozier	Assistant Research Scientist (Mechanical Engineer)	Friction Apparatus
Kenneth W. Stephens	Student Assistant (Physics)	Infrared Spectroscopy
R. A. Young	Diffraction Laboratories Director	X-ray Diffraction
Harry W. Ellis	Student Assistant (Physics)	X-ray Diffraction
James P. Bryant	Graduate Assistant (Textile School)	Frictional Properties of Cotton Fibers
Arthur M. Goldfarb	Graduate Assistant (Textile School)	Effects of Ginning on Properties of Cotton Fibers
W. Eudon Kirkland	Graduate Assistant (Textile School)	Infrared Investigations of Cotton Fibers

Individual	Title	Area of Research
Donald L. House	Graudate Assistant (Textile School)	Optical and Electron Microscopy of Cotton Fibers
Howard R. Levy	Graduate Assistant (Textile School)	Effects of Opening and Carding on Cotton Fibers
Billy R. Livesay	Research Physicist	Fiber Friction and Tensile Properties
Joseph H. Taylor	Student Assistant (Physics)	Fiber Friction
Hong Ki Chin	Graduate Assistant (Textile School)	Fiber Friction
Chin Taik Kwon	Graduate Assistant (Textile and Chemical Engineering)	Fiber Friction and Tensile Properties

In general, all work was performed on a part-time basis except that of Mr. Dozier who was employed on a full time basis to give continuity to the fiber friction measurement program.

LIST OF REFERENCES

1. R. B. Belser and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Reports Nos. 1 and 2, USDA-SURDD Grant No. 12-14-100-7661(2), August 1965, February 1966.

2. D. L. House, "Optical and Electron Microscopy of Cotton Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

3. J. P. Bryant, "An Investigation of the Factors Which Influence the Frictional Properties of Textile Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

4. W. E. Kirkland, "An Investigation of Techniques to Determine Infrared Spectra of Textile Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

5. A. M. Goldfarb, "Cotton Fiber Properties as Affected by Ginning," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

6. H. R. Levy, "The Effects of Opening, Cleaning, Picking, and Carding on the Physical Properties of Empire WR Cotton Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

7. J. C. Guthrie and P. H. Oliver, "Interfiber Friction," J. Text. Inst. 43, T579-595 (1952).

8. F. P. Bowden and D. Tabor, "Friction and Lubrication of Solids," Oxford University Press, Cambridge, Part II (1964) pp. 214-241.

9. R. S. Orr, A. W. Burgis, L. B. De Luca, and J. N. Grant, "The Role of Spiral Structure in Untreated and Treated Cottons," Text. Res. J. 31, 4, 302 (1961).

10. J. L. Stoves, Fibre Microscopy, National Trade Press, London, 3 (1957).

11. K. P. R. Pillay and K. S. Shankaranavayana, "Variations in the Properties of Cotton Fibers with Length," Text. Res. J. 31, 515 (1961).

12. S. M. Betrabet, K. P. R. Pillay, and R. L. N. Iyengar, "Structural Properties of Cotton Fibers, Part I," J. Sci. Indus. Res. 19A, 91 (1960).

13. L. P. Berriman, "Overdrying Cotton at Gin Can Change Fiber Shape," Text. Indus. 125, 25 (September 1961).

14. H. Wakeham, "Cotton Fiber Length Distribution - An Important Quality Factor," Text. Res. J. 25, 260 (March 1959).

15. Anonymous, "Model 114A Servo Torque-Balance Reaction Dynamometer," Instruction Manual, McFadden Electronics Company, South Gate, California.

16. J. Prakash and R. L. N. Iyengar, "Work Done in Pulling Fibers From the Cotton Seed Under Impact Conditions in Relation to Fiber Properties and Ginning Behavior," Text. Res. J. 34, 444 (May 1964).

17. W. S. Smith and N. L. Pearson, "A Method of Measuring the Strength of Attachment of Cotton Fibers to the Seed and Some Results of Its Application," Report of USDA Agricultural Marketing Service and Bureau of Plant Industry, Washington, D. C., February 1941.

APPENDIX

Table 15. Maturity Study of Five Cotton Specimens

Cotton Name	Number Fibers Examined (Percentage) 1/2 Mature	Mature	Total	Maturity Coefficient*
Empire WR Hand Ginned (Experiment, Ga.)	45 (5.93)	342 (45.02)	372 (49.05)	759 (100.00)	.785
Empire WR Hand Ginned (Locust Grove, Ga.)	38 (5.25)	321 (44.48)	363 (50.25)	722 (100.00)	.793
Dixie King Hand Ginned	30 (5.92)	329 (53.84)	252 (41.24)	611 (100.00)	.759
Carolina Queen Hand Ginned (Mech. Harvested)	33 (5.68)	327 (56.28)	221 (38.04)	581 (100.00)	.740
Carolina Queen Hand Ginned (Hand Picked)	11 (2.68)	207 (50.36)	193 (46.96)	411 (100.00)	.783

* Maturity Coefficient = M + 0.6H + 0.41I,

where

M = percent mature fibers

H = percent half mature fibers

I = percent immature fibers

See Section III B 7 for additional information

Table 16. Coefficients of Friction of Nylon Fiber Against Glass at Low Normal Forces

	μ_k			μ_s			μ_s/μ_k		
(Run No.)	1	2	3	1	2	3	1	2	3
Specimen			2 Milligrams Normal Force						
1	0.271	0.313	0.255	0.560	0.571	0.515	2.07	1.83	2.02
2	0.321	0.291	0.274	0.628	0.590	0.543	1.95	2.03	1.98
3	0.297	0.292	0.259	0.624	0.651	0.566	2.10	2.23	2.18
Average	0.296	0.299	0.263	0.604	0.604	0.541	2.04	2.02	2.06
Grand Average		0.286			0.583			2.04	
			5 Milligrams Normal Force						
1	0.254	0.252	0.278	0.676	0.656	0.632	2.66	2.60	2.27
2	0.341	0.354	0.382	0.625	0.624	0.668	1.83	1.76	1.75
3	0.161	0.171	0.236	0.435	0.476	0.516	2.71	2.78	2.18
Average	0.252	0.259	0.299	0.579	0.585	0.605	2.29	2.26	2.02
Grand Average		0.270			0.590			2.18	

REPORT NO. 4 (SEMIANNUAL REPORT)
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1 AUGUST 1966 to 1 FEBRUARY 1967

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By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

PREPARED FOR
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SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
NEW ORLEANS, LOUISIANA

1 FEBRUARY 1967

Engineering Experiment Station
and
School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

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Placed By

United States Department of Agriculture
Agricultural Research Service
Southern Utilization Research and Development Division
New Orleans, Louisiana

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ABSTRACT

The purpose of this research is to establish the frictional characteristics of cotton fibers and to determine how these characteristics may change as cotton fibers are processed from the bulk to the yarn.

High- and low-draft cotton fibers have been investigated by methods of microscopy and frictional measurement in order to determine differences intrinsic to the fibers. The high-draft fibers, by cross-sectional measurement, electron microscopy, and electron scanning microscopy, were found to be flatter fibers than the low-draft ones and of generally smoother surfaces. However, the convolutions at the zone of rotation possessed a high degree of twist per unit length, resulting in a rapid increase in the slope gradient of the surface of the fiber at each convolution. The low draft fibers, on the other hand, displayed a somewhat rougher surface but a slower twist rate in the vicinity of the convolution and a lower surface slope gradient.

Frictional measurements of the high-draft fibers produced large data scatter. Snagging of many fibers produced large peaks in the frictional analog plots, some off scale. The kinetic coefficient of friction, for the first pass on the fibers was 0.27 compared to 0.24 for the low-draft and the static friction was 0.86 compared to 0.76. The off scale data were not included and would have caused a greater difference than shown.

Some 300 frictional measurements were made for fiber pairs at normal forces of 2 to 20 mg. Fiber pairs examined were cotton against cotton, nylon and glass, and nylon against cotton, glass, and nylon. Coefficients of kinetic friction obtained were in the range 0.16 to 0.37 and of static friction 0.48 to 1.07. Friction decreased from high values at normal forces of 2 mg to the lower values at 20 mg. Some fibers were run against others with

no externally applied normal force, except the weight of the fiber and cohesion. Frictional forces of 0.5 to 1.5 mg were commonly measured. High stick effects of convolutions in cotton and crimp in nylon were obvious. In addition, single fibers were drawn from cotton card sliver and roving indicating forces of 11.6 and 7.5 mg respectively. It is evident that very high coefficients of friction exist for fiber to fiber contacts of the cotton during the processing stages. Investigations of cotton by microscopy indicated convolutions or other asperities are responsible for large peaks in the frictional curves. The evidence suggests that interlock of these between fibers allow motion only at the zones of no interlock or low friction unless the rate of loading is high, in which event slippage at asperities occurs. A second pass of a fiber across the servo-controlled element results in some changes of the data curve, but principal peaks usually remain only slightly changed in amplitude, shape, or position. The frictional data curves depict characteristics of the material, asperities, and damage to the fiber. The frictional values obtained at very low normal forces are informative but some doubt exists as to the absolute values obtained due to some difficulties in establishing the plot zero, and possible vibration effects. These matters are being more exhaustively investigated in concurrent thesis research.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influence of crimp, convolution, cross section, surface texture, and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

II. INTRODUCTION

The development of the necessary apparatus for the measurement of fiber friction and preliminary information concerning the effects on the friction between two fibers of the fiber material, normal force between the fibers, fiber tension, temperature, and fiber processing have been discussed in previous reports of this contract.[1] In addition, during the current period five theses dealing with related topics[2, 3, 4, 5, 6] have been submitted. Concurrently four additional theses[7, 8, 9, 10] are under way and will be submitted prior to February 1, 1968.

During the period covered by this report, in addition to the extensive effort required for completion and publication of the five theses submitted, work has been concentrated on the evaluation of the apparatus for measurement of friction at normal forces in the range 2 to 20 mg and in some studies made of the friction between fibers where no normal force other than cohesion or the weight of a single fiber were applied. Related to both of these efforts fiber shape studies by optical microscopy, and electron stereo- and scanning microscopy have been made for a number of fibers. X-ray diffraction and infrared investigation of fibers were reduced pending assignment of incoming students or graduate student assistants for these areas.

III. EXPERIMENTAL WORK

A. GENERAL

The experimental work has consisted principally of further work with the friction apparatus for measurement at very low normal forces and a study of the micro features of cotton fibers as related to the xy plot produced by the friction measuring instrument. Micrographic and frictional studies have been made of cotton fibers of high and of low draft performance.

B. APPARATUS AND PROCEDURES

The apparatus and procedures used have been given extensive coverage in the preceding Semiannual Reports (1, 2 and 3) of this series. Any additions to or deviations from these will be discussed in the description of the particular experiment.

C. INVESTIGATIONS OF FIBERS BY OPTICAL AND ELECTRON MICROSCOPY

1. General

Optical microscopy has been used for the examination of cotton fibers of high and low draft performance and for the delineation of micro surface features as related to the friction measurement plot of a specific fiber and feature, respectively. The fibers have been further examined by additional information concerning the fiber shapes and surfaces by electron replica, replica stereo-microscopy and by scanning microscopy.

2. Cross-Sections of Cotton Fibers of Low and High Draft Performance

Specimens of cotton fiber (supplied by Mr. J. N. Grant of USDA, New Orleans) were examined for maturity and shape by cross-sectioning and measuring the cross-sectional dimensions of the fibers. Using the technique described in Semiannual Report No. 2 (page 10)[1] distribution curves

of the values $R = (M + m)/4w$ and $S = M/m$ versus percent of fibers less than the value plotted were prepared. R and S are values of the ratios defined where M is the major axis of the section, m is the minor axis of the section, and w is the wall thickness of the fiber. R is related to maturity since a larger value is obtained as the size of the lumen increases. S is related to shape or the ratio of width of the fiber to its thickness. This also would appear to increase with maturity but may also vary with species (as may R to some degree).

Typical cross-sections of a high and of a low draft cotton specimen are exhibited in Figure 1. Typical distribution curves for each are shown in Figures 2 and 3.

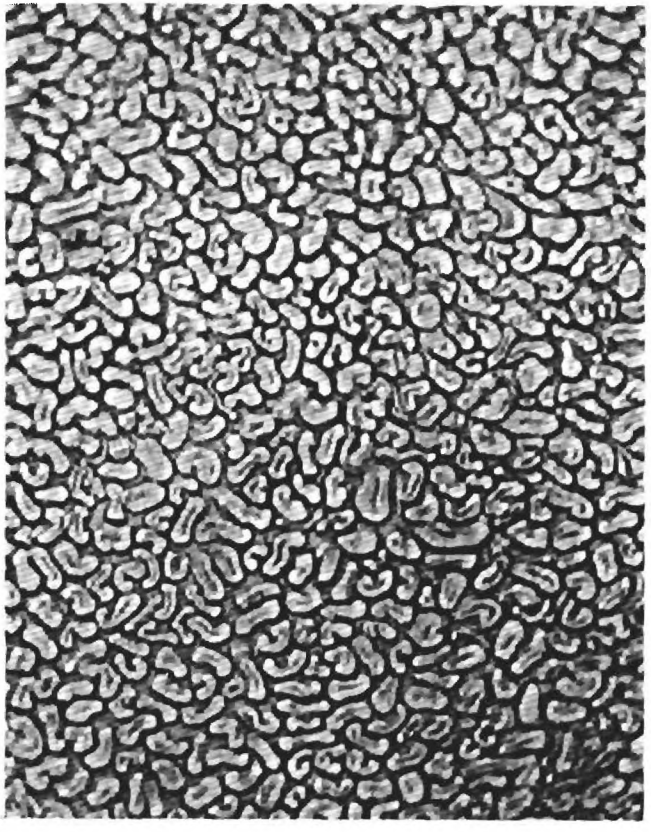
A tabulation of the median values of the measurements are given in Table 1. Here it will be noted that the average R and S values compare as follows:

	R	S
Low Draft	2.33	3.13
High Draft	2.73	3.27

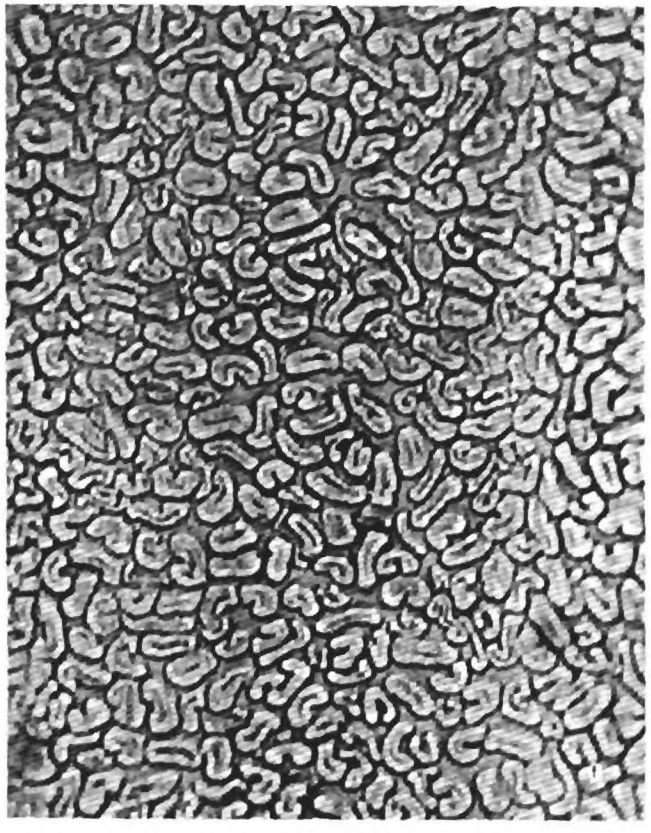
These results appear to indicate that the high draft variety is cotton of greater maturity than the low draft or that it is a species possessing a wider and thinner ribbon as its characteristic fiber.

3. Electron Micrographs of High and Low Draft Cotton Specimens

Specimens of the same cottons discussed above were examined by replica techniques to determine the surface character of typical fibers of both the high draft and low draft performance. Typical micrographs for each type are displayed in Figures 4 and 5. Examination of a selected sample of some 12 specimens indicated in general a slightly smoother surface for the high draft cotton, although occasional exceptions were noted.



(a) High Draft



(b) Low Draft

Figure 1. Micrographs of cross-sections of high- and low-draft cotton fibers (345X).

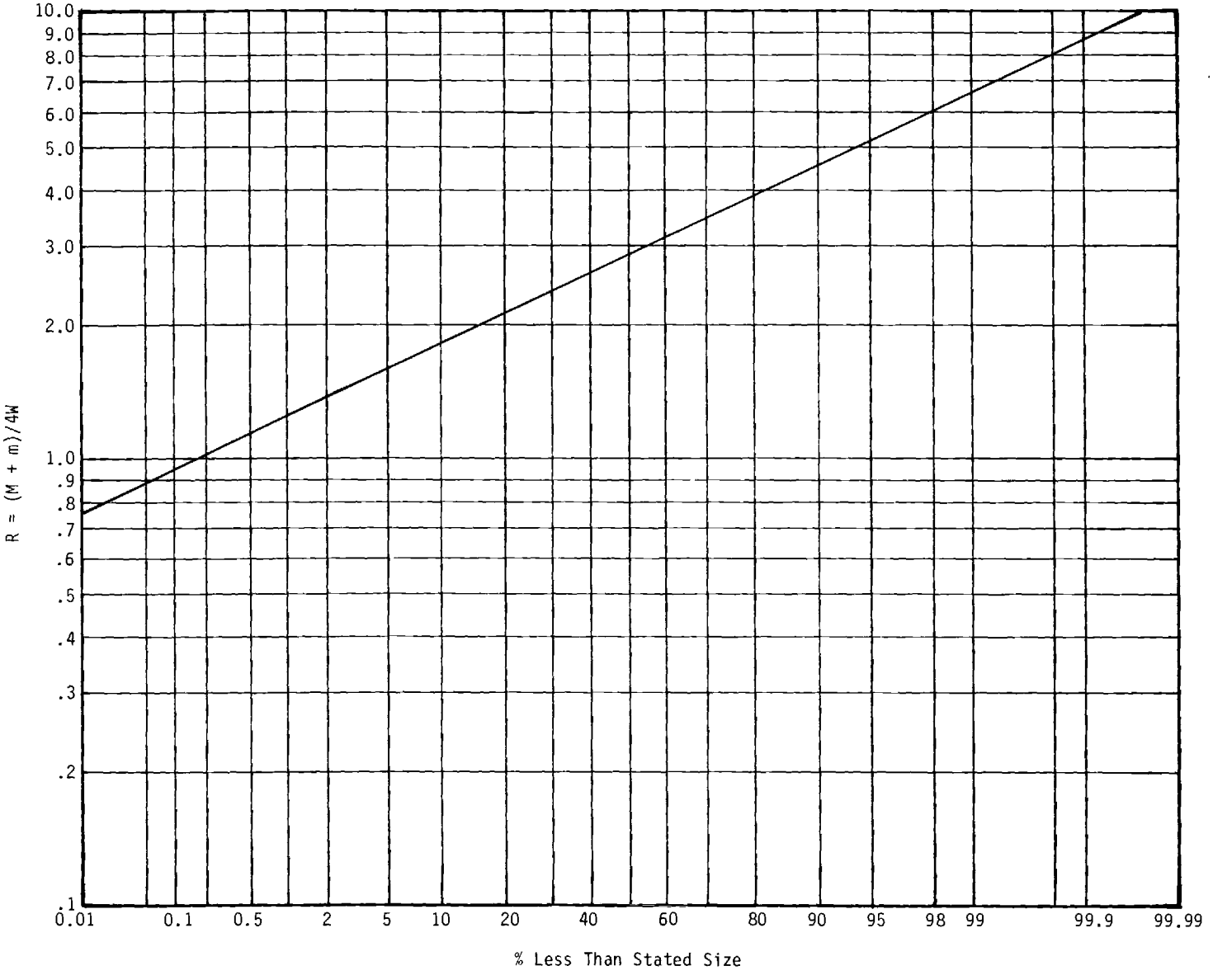


Figure 2. Typical distribution curve for value R = (M + m)/4w, of high-draft cotton fiber cross-sections.

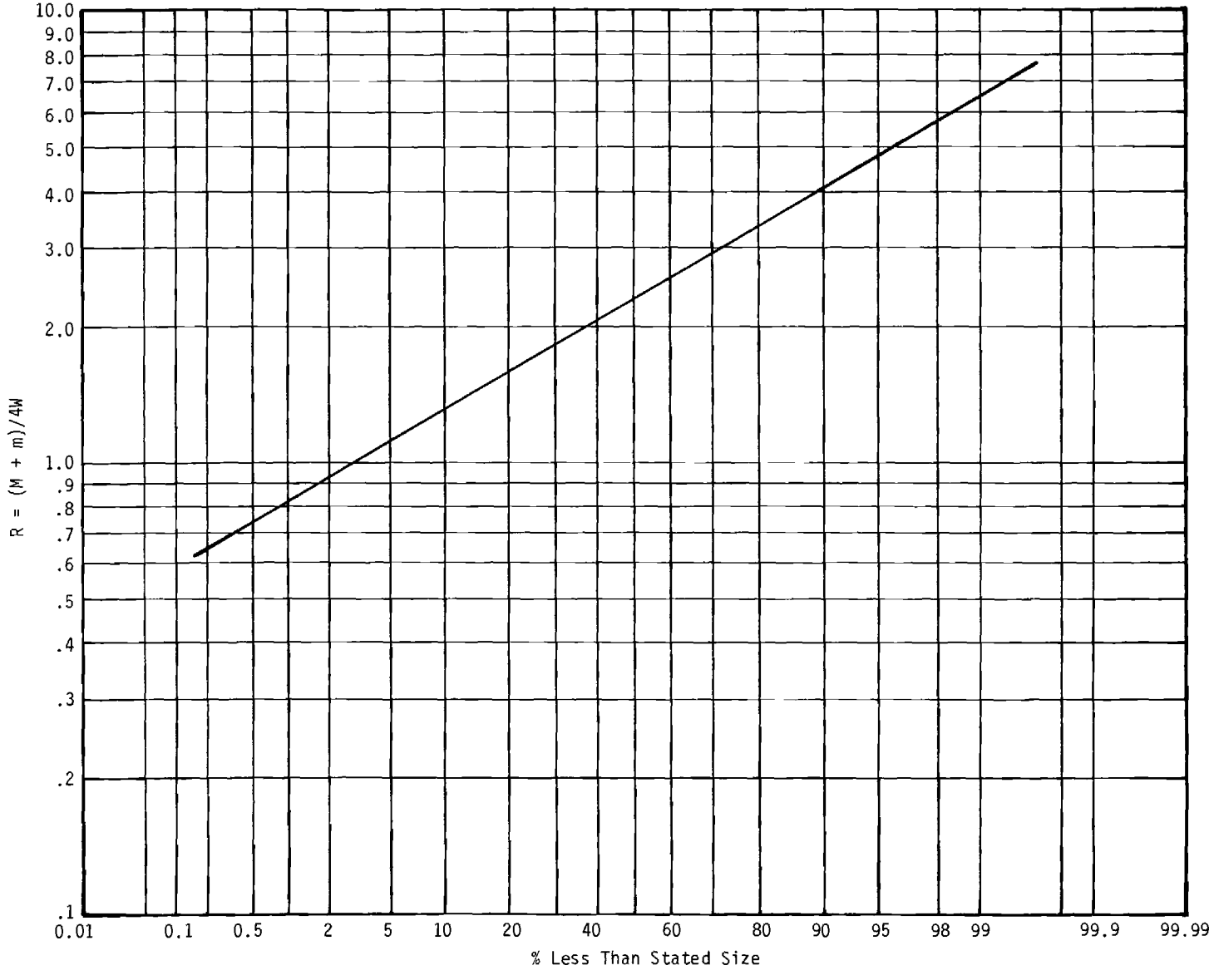


Figure 3. Typical distribution curve for value R = (M + m)/4w, of low-draft cotton fiber cross-sections.

TABLE 1

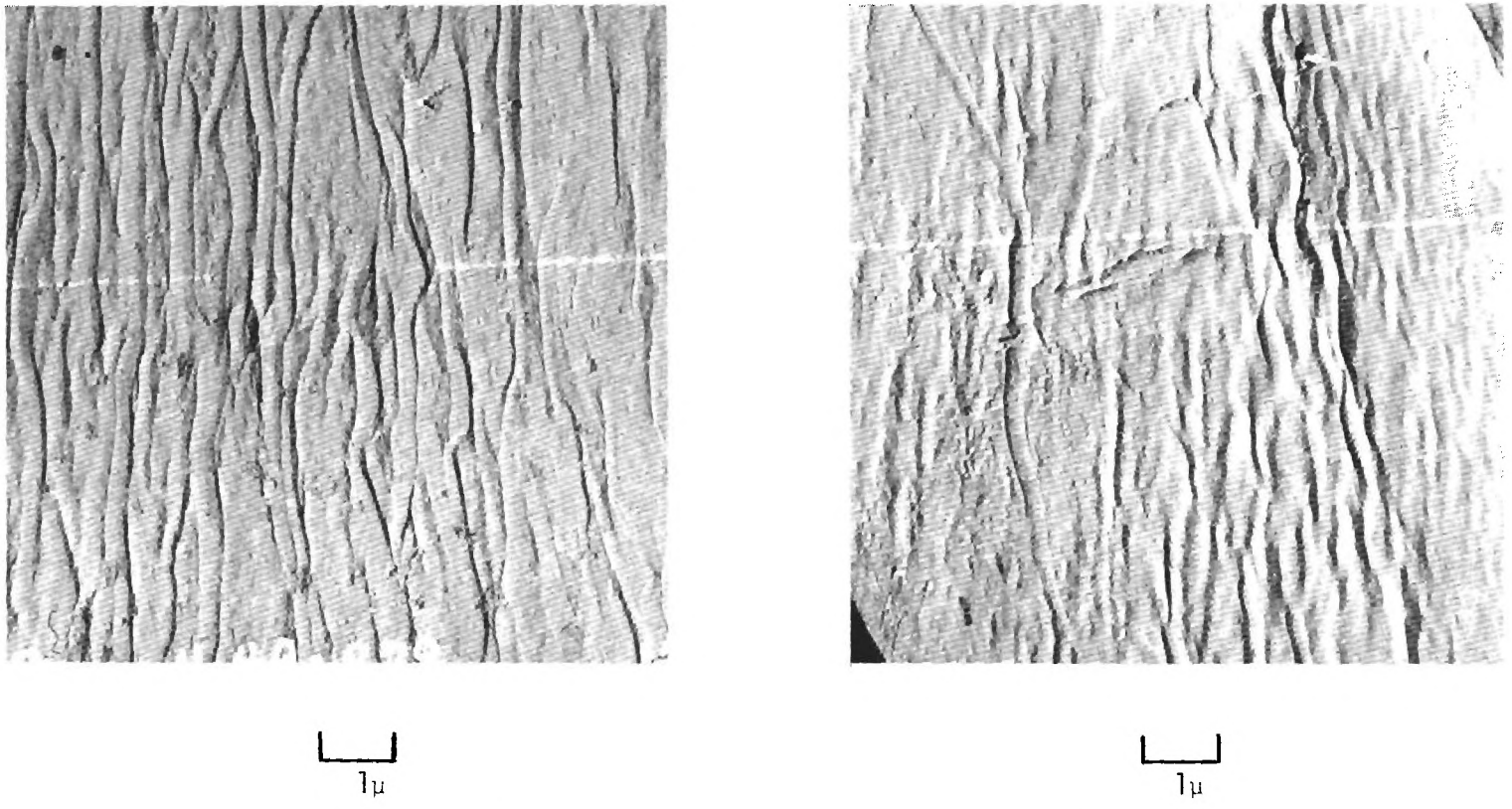
Comparison of R and S Parameters
of High- and Low-Draft Cotton Fibers

S Values M/m

Specimen	Range S	Median	Range Covered
Low-Draft Cotton			
942	2.0 to 8.0	3.3	10% to 98%
933	2.0 to 7.0	3.1	14% to 98%
867	2.0 to 8.0	2.95	16% to 98%
High-Draft Cotton			
879	2.0 to 8.0	3.2	12% to 96%
892	2.0 to 10.0	3.6	10% to 98%
824	1.0 to 8.0	3.0	2% to 96%

R Values $= \frac{M + m}{4W}$

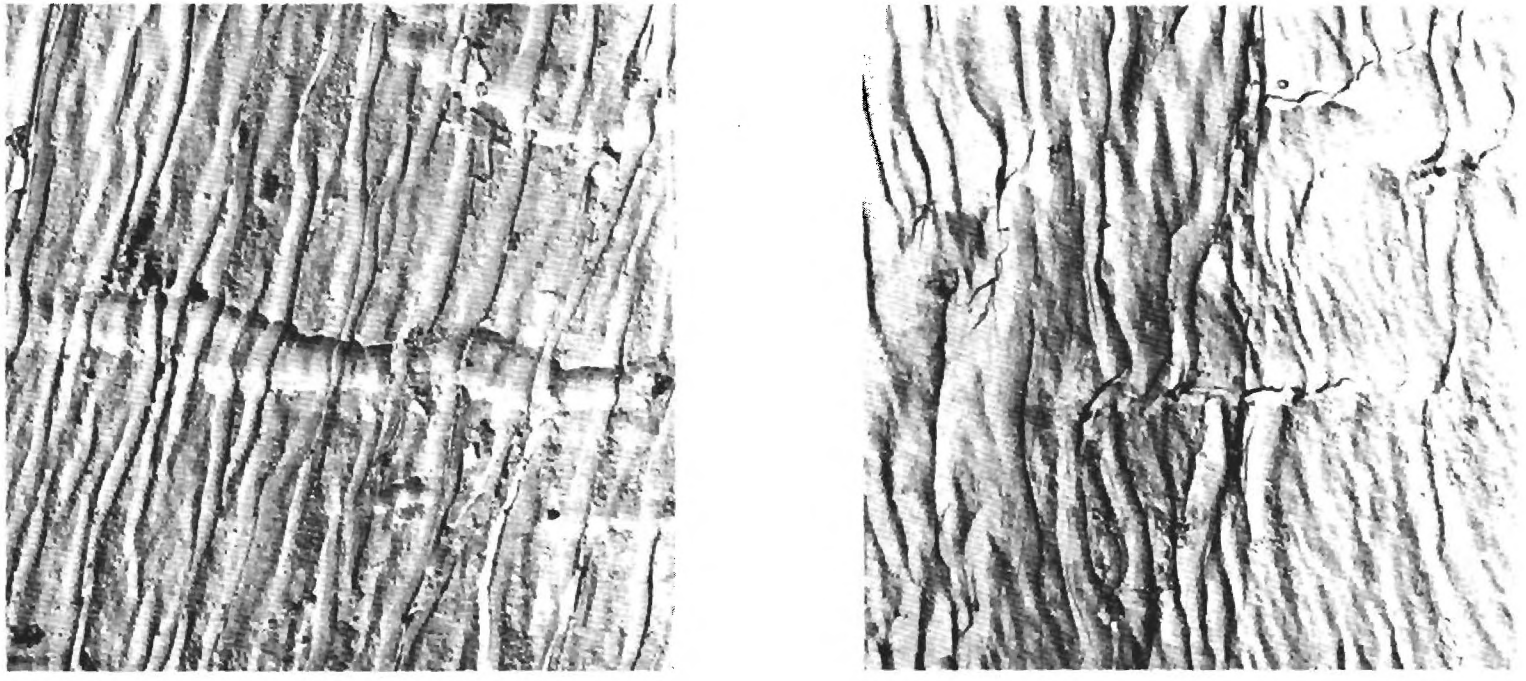
Specimen	Range	Median	Range Covered
Low-Draft Cotton			
942	2.0 to 5.0	2.4	26% to 98%
933	2.0 to 4.0	2.3	40% to 90%
867	2.0 to 4.0	2.4	30% to 92%
High-Draft Cotton			
879	2.0 to 4.0	2.9	18% to 84%
892	2.0 to 8.0	2.75	24% to 98%
824	2.0 to 5.0	2.6	34% to 97%



(a) Specimen H49-879

(b) Specimen H58-824

Figure 4. Typical electron micrographs of replicas of surfaces of high-draft cotton fibers (10,300X).



(a) Specimen L52-942

(b) Specimen L61-933

Figure 5. Typical electron micrographs of replicas of surfaces of low-draft cotton fibers (10,300X).

A similar examination made by the stereo replica technique in which the replica is tilted in the beam approximately 3 degrees to either side of its usual position of 90 degrees to the beam displayed similar information as indicated in Figures 6 and 7 for typical specimens.

An opportunity was also presented due to the courtesy of a textile manufacturer* to examine a few of these fibers with a scanning electron microscope. This microscope allows a very large range of magnification with definition slightly less than the replica method but with a tremendous depth of focus. Little fiber preparation is required and the actual object itself is examined.

Scanning electron micrographs of the high and low draft specimens are displayed in Figures 8, 9, 10 and 11. Although only a few specimens of the cottons were examined, at the lower powers the high draft cotton appears to present a more crimpy and convoluted appearance. The degree of surface roughness was not examined for a sufficient number of specimens to draw a conclusion from the few specimens examined. However, the depth of focus and the tremendous detail displayed in the micrographs is apparent.

4. Conclusions

Both optical and electron microscopic examinations of the high and low draft cotton fibers indicated differences in the two types of fibers. The optical examination applied statistically to an examination of the cross-sections indicated that the ratio $M + m/4w$** was greater for the high draft cotton in the proportions 2.73/2.33 and that there was also a slight difference in the ratio M/m in the same direction indicating that the high draft cotton was a flatter ribbon. Electron microscopy indicated a smoother

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* The Deering Milliken Company of Spartanburg, South Carolina.

** $\frac{\text{major axis + minor axis}}{4 \text{ wall thickness}} = R$

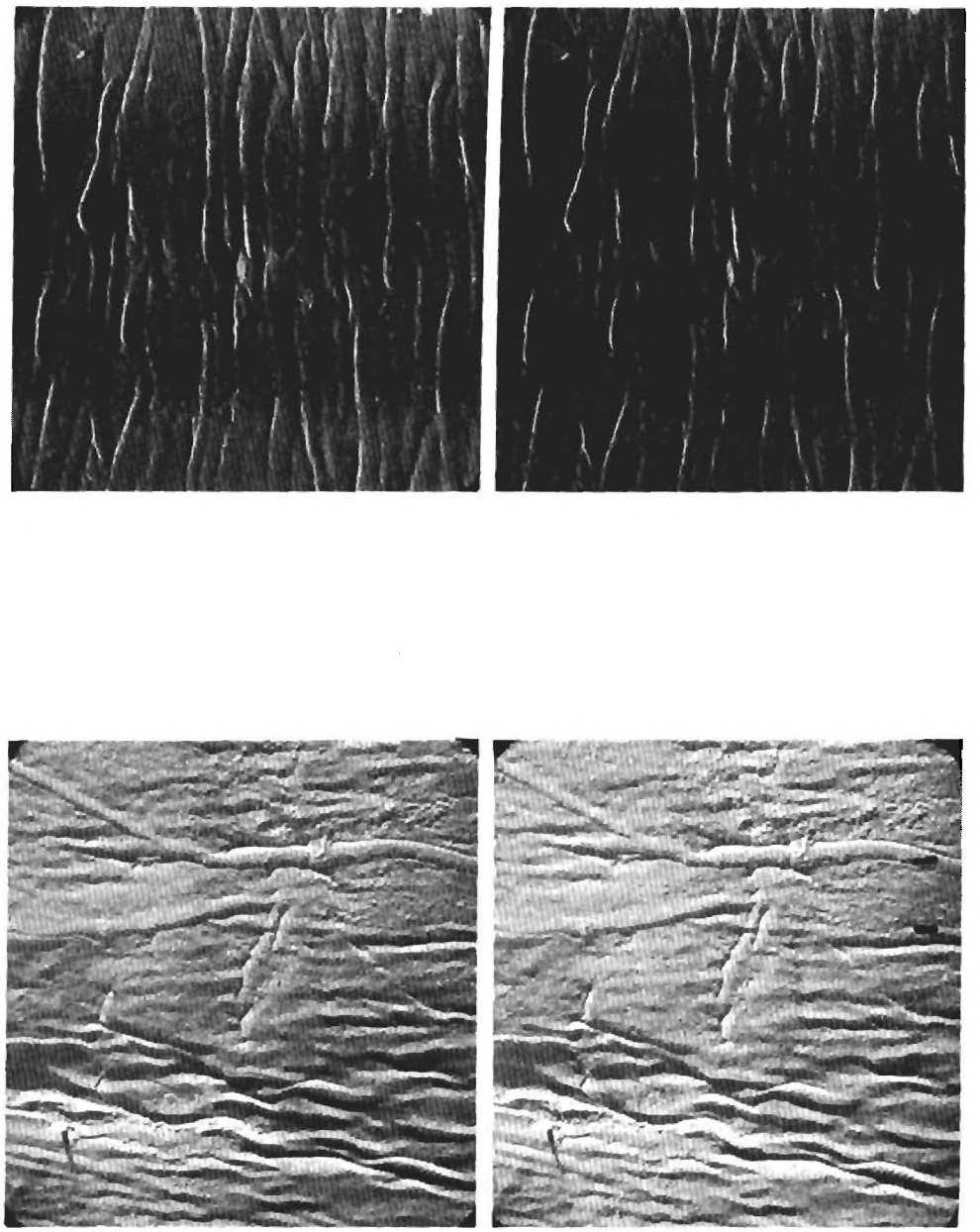


Figure 6. Typical electron stereomicrographs of replicas of the surface of a high-draft cotton fiber (9,600X).

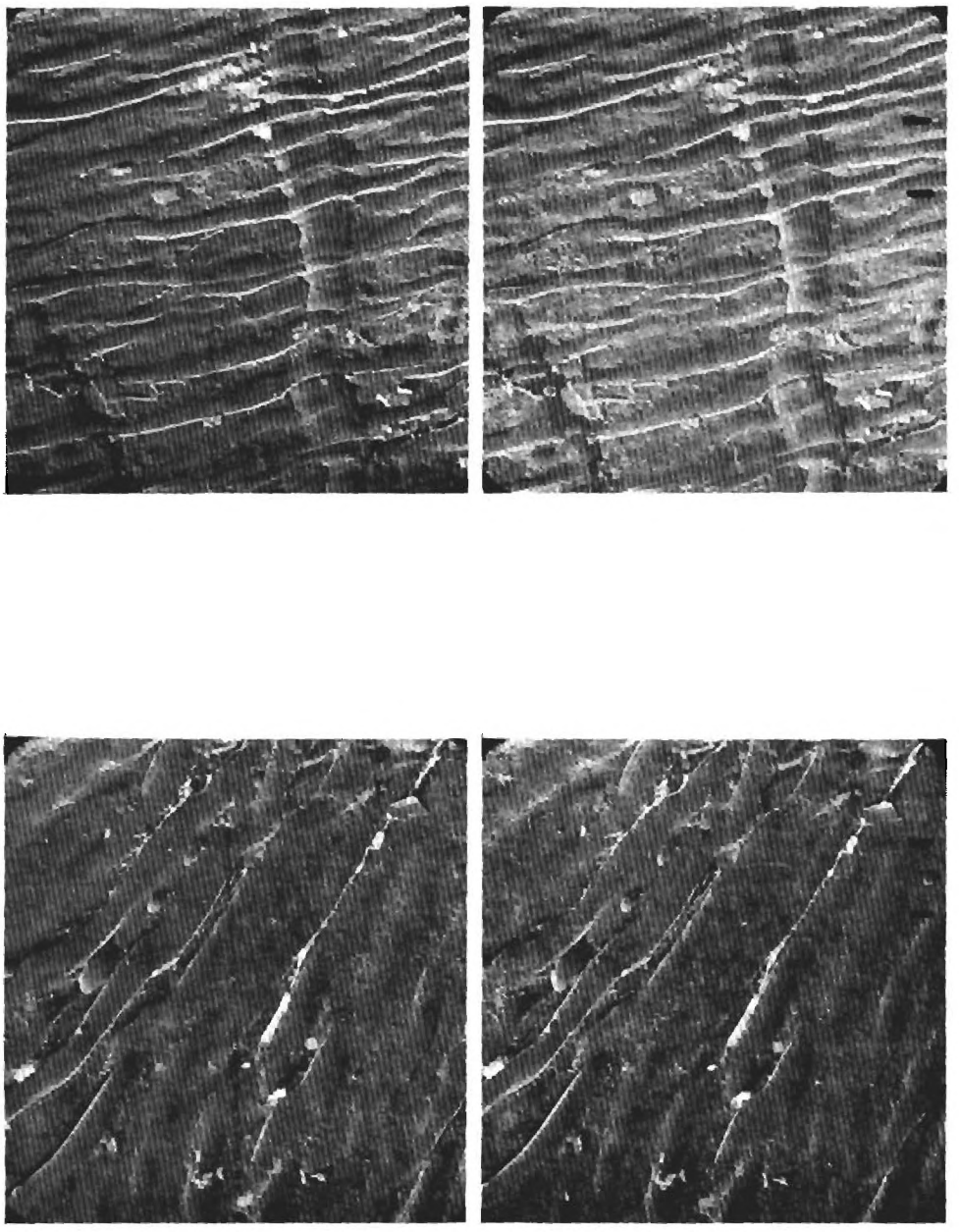
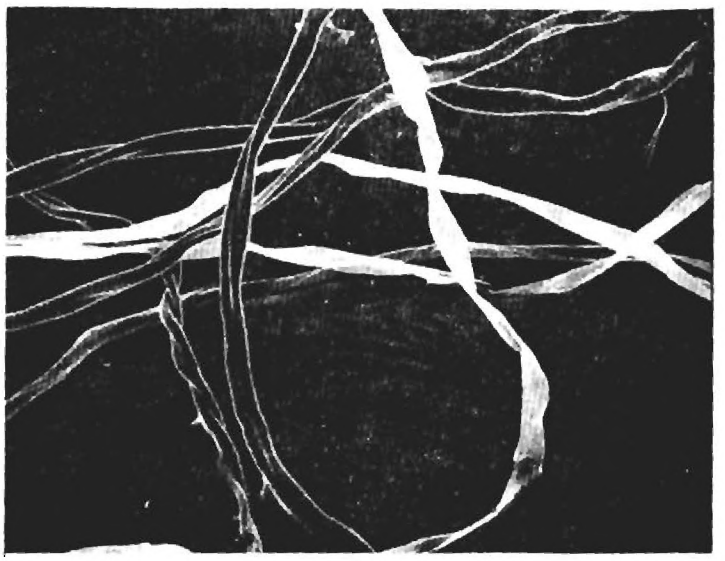
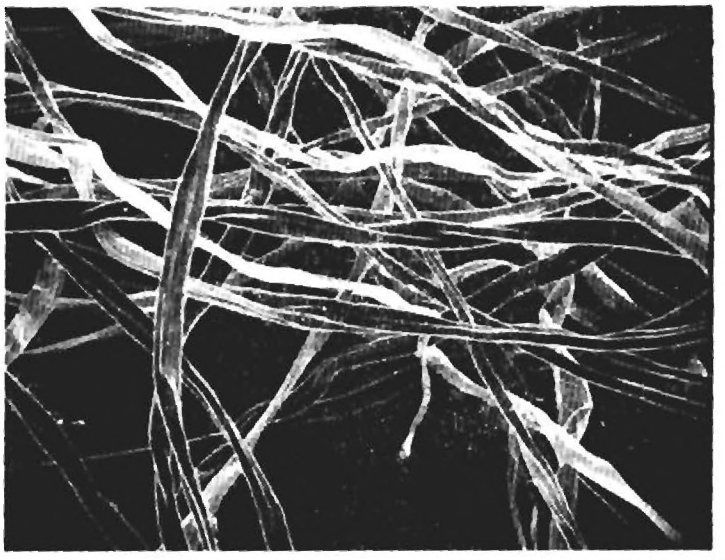


Figure 7. Typical electron stereomicrographs of replicas of the surface of a low-draft cotton fiber (9,600X).



(a) High Draft (180X)



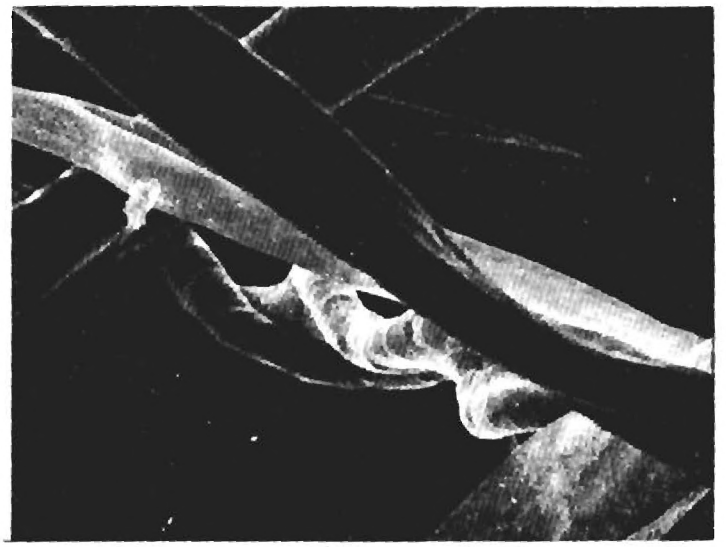
(b) Low Draft (200X)

Figure 8. Scanning electron micrographs of high- and low-draft cotton fibers at low magnification (180X, 200X).

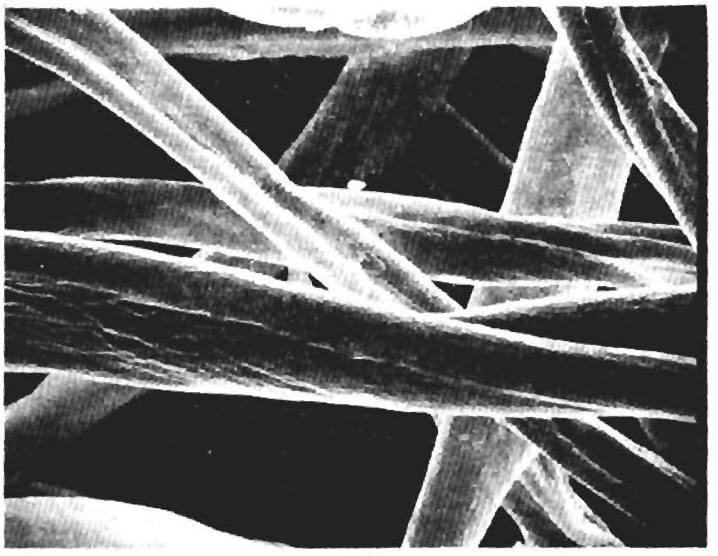
(a) High Draft (180X)

(b) Low Draft (500X)

Figure 9. Additional scanning electron micrographs of high- and low-draft cotton fibers at relatively low magnifications (180X, 500X).



(a) High Draft



(b) Low Draft

Figure 10. Scanning electron micrographs of high- and low-draft cotton fibers at a higher magnification (1000X).

(a) Convoluted Zone (2000X)

(b) Straight Zone (5000X)

Figure 11. Scanning electron micrographs of low-draft cotton fibers at high magnifications showing large depth of focus and excellent detail.

surface for the high draft cotton as well; and from a very small sample, the scanning electron microscope indicated greater crimp and convolutions occurred in the high draft specimen. Indications are that these are two different varieties but insufficient evidence, or possibly experience, has yet been gained to identify the specific varieties. Fiber length distribution and convolution counts have not been made at this time; those operations should cast further light on the variety identification. Frictional properties of the fibers will be discussed in Section C, following.

D. FRICTIONAL APPARATUS AND MEASUREMENTS

1. General

The frictional apparatus employed has been comprised of t instruments,* one for use in the range 20 to 50 mg normal force[1] and a second one[1] for use in the range 2 to 20 mg. The first instrument has been used to check on data obtained by Levy[5] on the changes in frictional properties of cotton as a result of textile processes and for investigation of the cotton of high and low draft performance. The second instrument h been given extensive further tests and used for examination of friction between cotton and glass, nylon and glass, and cotton and nylon. The variation of frictional properties of other materials over the normal force range 2 to 20 milligrams has been investigated.

2. Variation of Friction with Cotton Processing

In the last report of this series (Semiannual No. 3) data compiled by Levy was displayed in Figures 24 and 32 of that report indicating that both the kinetic and static coefficients of friction increased with fiber processing through the successive stages ginning, opening, picking and carding. Furthermore, very large peaks occurring on the frictional graph

* These have been completely described in Semiannual Reports 1 and 3, respectively, of this Grant.

displays were indicative of fiber damage during the various processing stages. Measurements made on a large number of fibers of Levy's cotton specimens indicated that the changes noted by Levy were real and accurate within the capability of the instrument used.

3. Matching of Frictional Graphs with Fiber Features

Because of the stick-slip nature of the frictional process it has been shown previously by us (in high speed movie film) that a stick for cotton may occur at a single fiber feature followed by a slip during which little or no fiber contact may exist for a number of successive features. The slip may be induced by bobbing of the frictional arm due to vibration and low inertia of the arm (where the normal force is applied by a small change in the center of gravity of the frictional arm).

A series of frictional curves were made for the high and low draft cottons previously discussed. Optical micrographs were made of the fibers along the entire length of the fiber utilized, and features from a marked starting point were examined in relation to the frictional curve. The fiber of principal interests is the traversing fiber which is the one examined in this case. Figures 12 and 13 display typical graphs and micrographs obtained for high draft specimens. Convolutions or reversals appeared to be the prime stick points for cotton. A slip would then occur over several convolutions until the lower fiber (on the galvanometer needle) returned to essentially the zero position. This distance could be as much as several convolution lengths, or say 0.1 cm or 0.04 inches. On occasion a feature of significant displacement from the axis of the fiber appeared. In such an instance a large stick was indicated by the plotter; the pen was displaced to its maximum position and remained until the force was sufficient to cause a slip. In the case of the feature of Figures 13 and

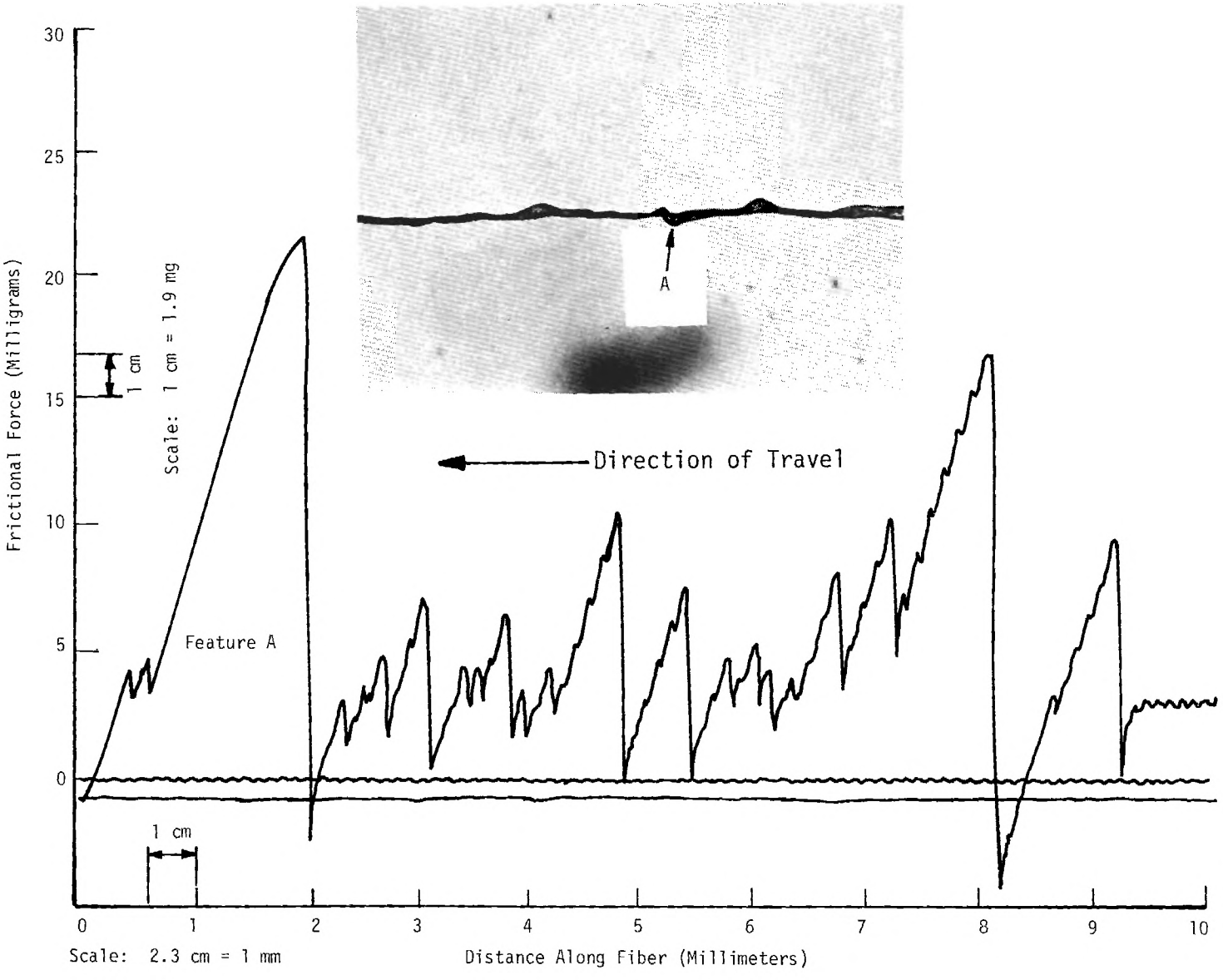


Figure 12. Frictional graph and micrograph of high-draft cotton fiber indicating friction "stick effect" and feature responsible.

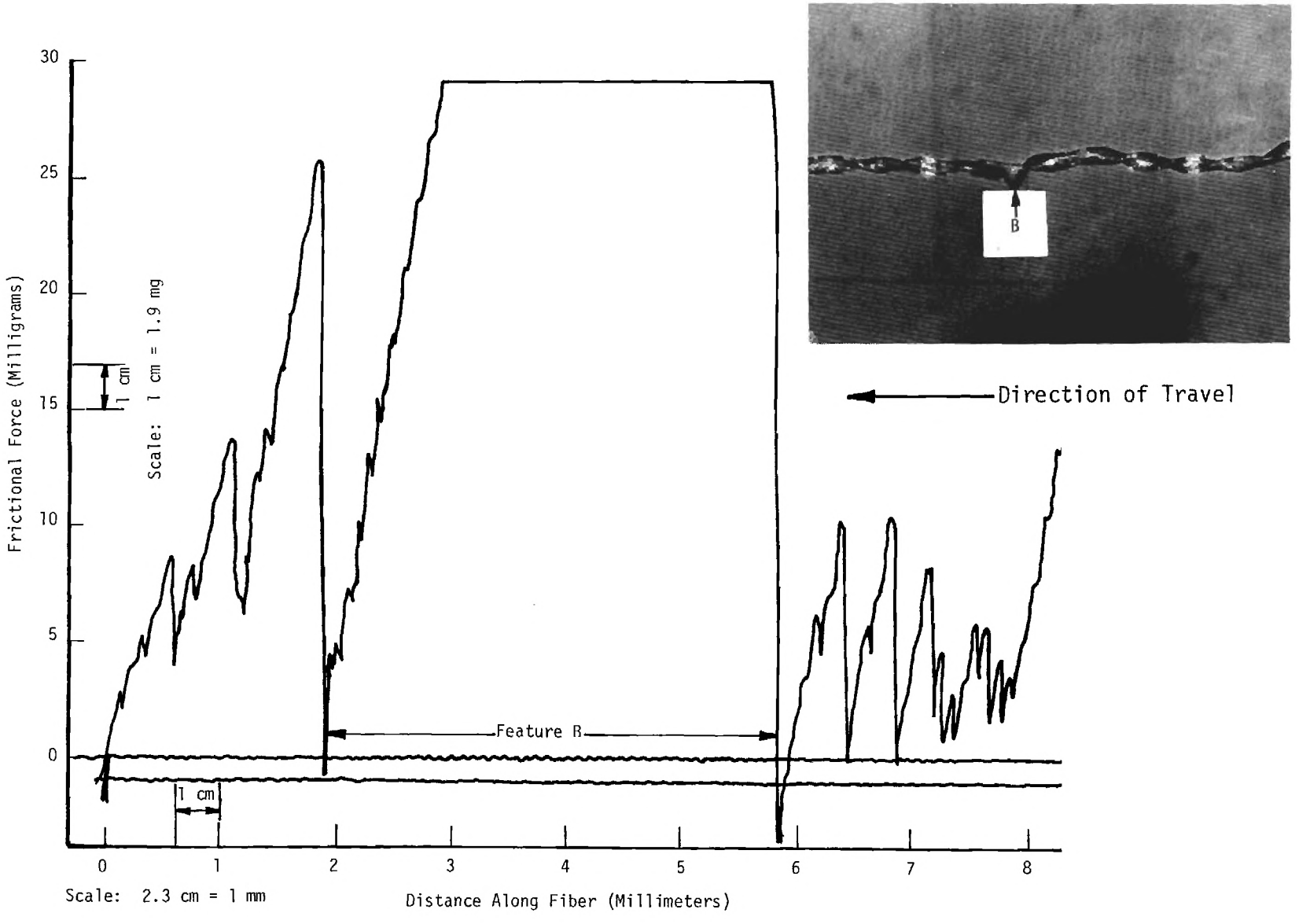


Figure 13. Frictional graph and micrograph of second high-draft cotton fiber displaying friction peak and features responsible for various peaks.

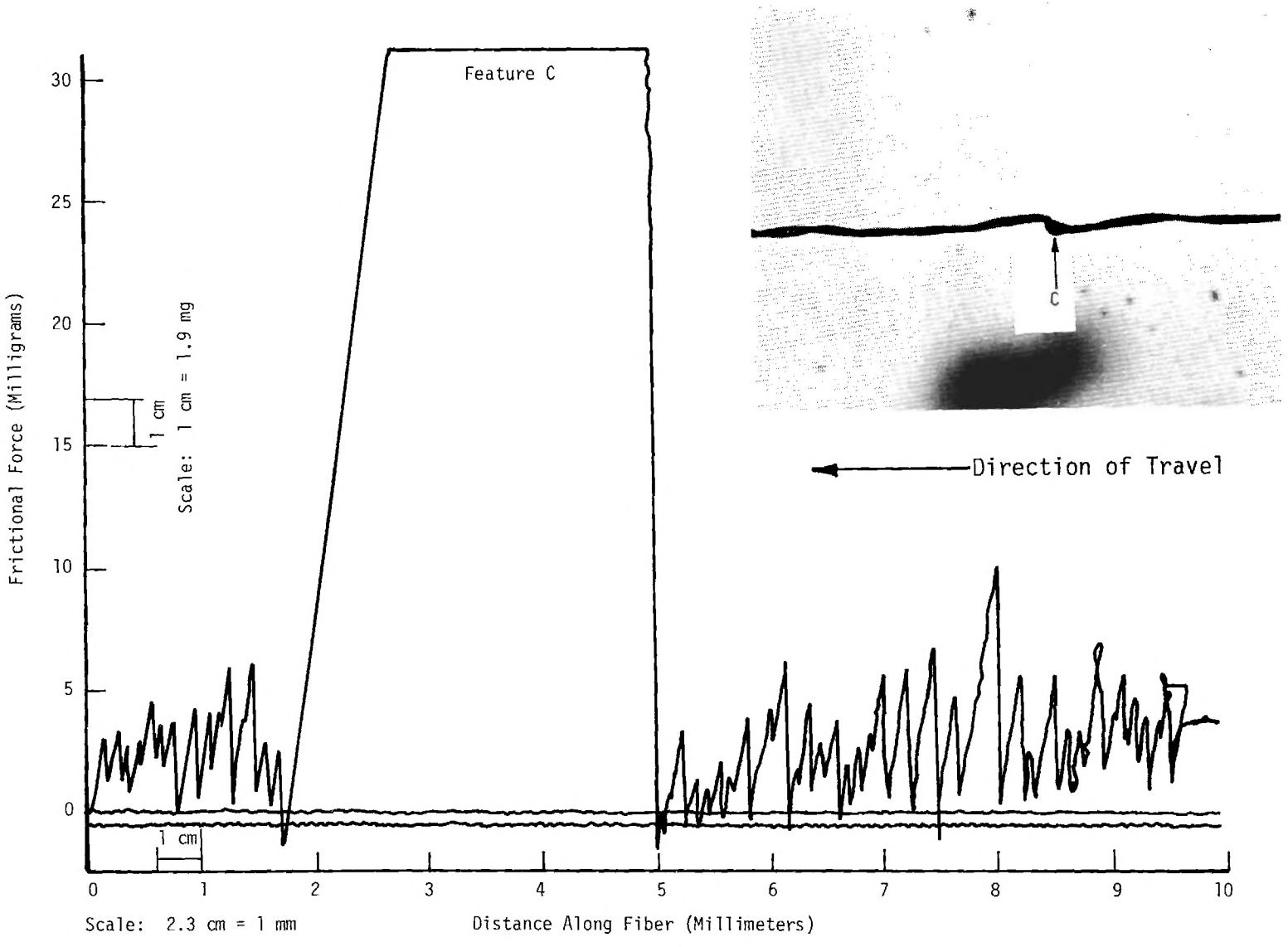


Figure 14. Frictional graph and micrograph of a cotton fiber having an unusual protuberance giving a very high stick.

14 the estimated force was 20 mg, on the order of the normal force employed. Such forces may easily stretch, damage, or conceivably break the traversing or the stationary fiber. However, no breaks were observed in a limited number of examinations. Features exhibiting such large displacements have been a rarity in previous examinations but have been observed in the high draft cotton and appear more commonly in cotton subjected to processing stages such as picking. Fig. 15 displays a peak, D, beyond C of Fig. 14, masked by C.

4. Frictional Measurements of High and Low Draft Cotton

The micro features of high and low draft cottons have been discussed in some detail in the preceding pages of this report. Accompanying these studies some 120 frictional measurements have been made and the frictional plots have been analyzed. Typical friction data for the two types are exhibited in Figures 16 and 17.

Data for a group of high draft fibers (42 measurements) are exhibited in Table 2. It is to be noted here that the data are arranged in three columns for μ_k, three for μ_s, and three for the ratio μ_s/μ_k. These are the relative measurements, A, B, and C (1st, 2nd, 3rd) for the same pair of fibers. In most cases, although there is a successive decrease in the values A, B, C, the checks are fairly close. These indicate a small amount of ironing out of kinks and crimps in the traversing fiber on successive trips, possibly compensated in part by some stretching and reduction of tension.

Data for a group of low draft fibers are indicated in Table 3. These data indicate that the μ_k value for the low draft fiber is lower than that of the high draft fiber in the first pass and very little different thereafter. The μ_s value is also slightly higher.

If now we examine the actual measurement distribution of the μ_k values for each variety as shown in Figures 18 and 19 we observe that the values for the high draft fibers are widely scattered whereas the values for the

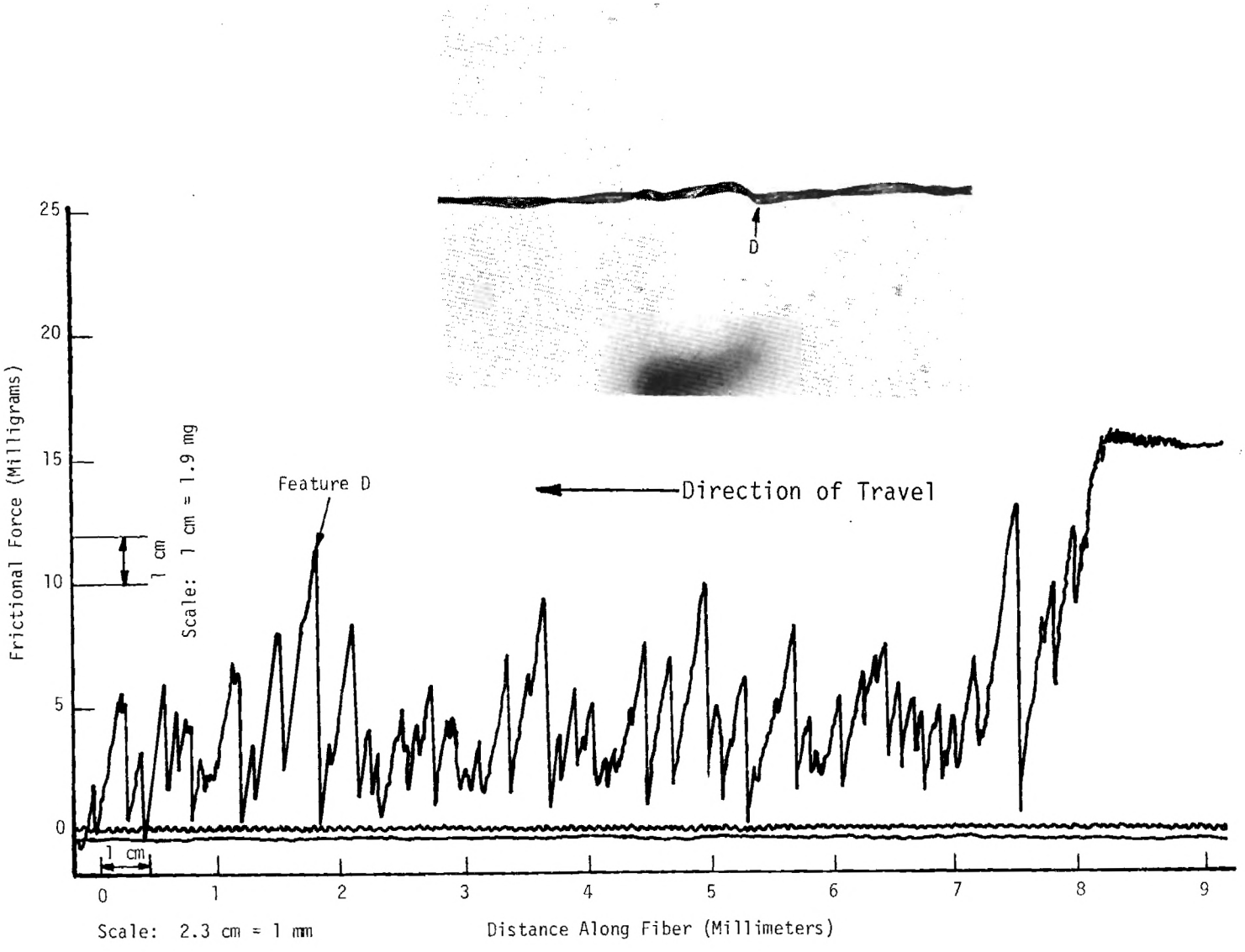


Figure 15. Frictional graph and micrograph of the cotton fiber of Figure 14 at a section beyond Feature A.

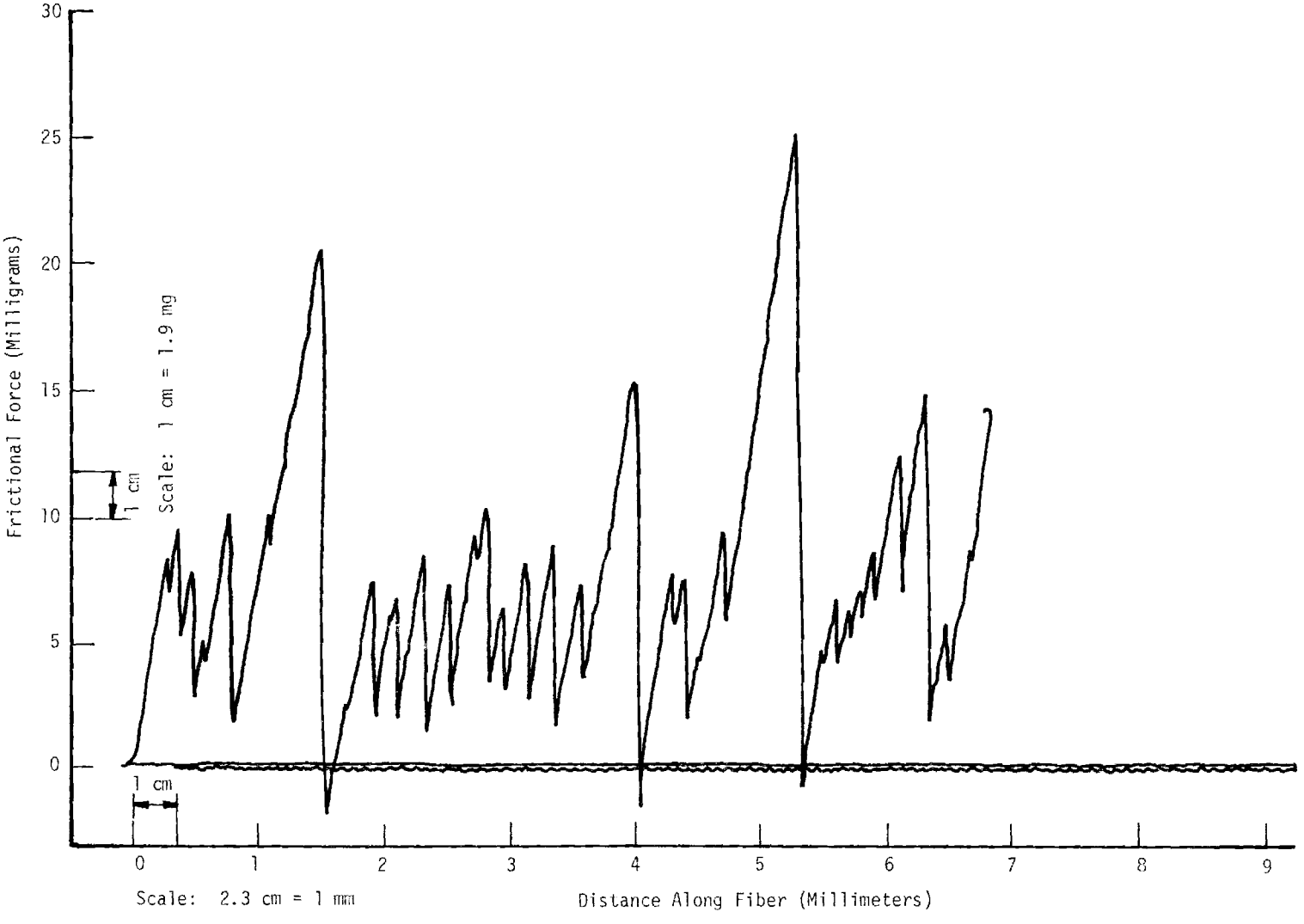


Figure 16. Typical frictional data for high-draft cotton fiber. Note large peak.

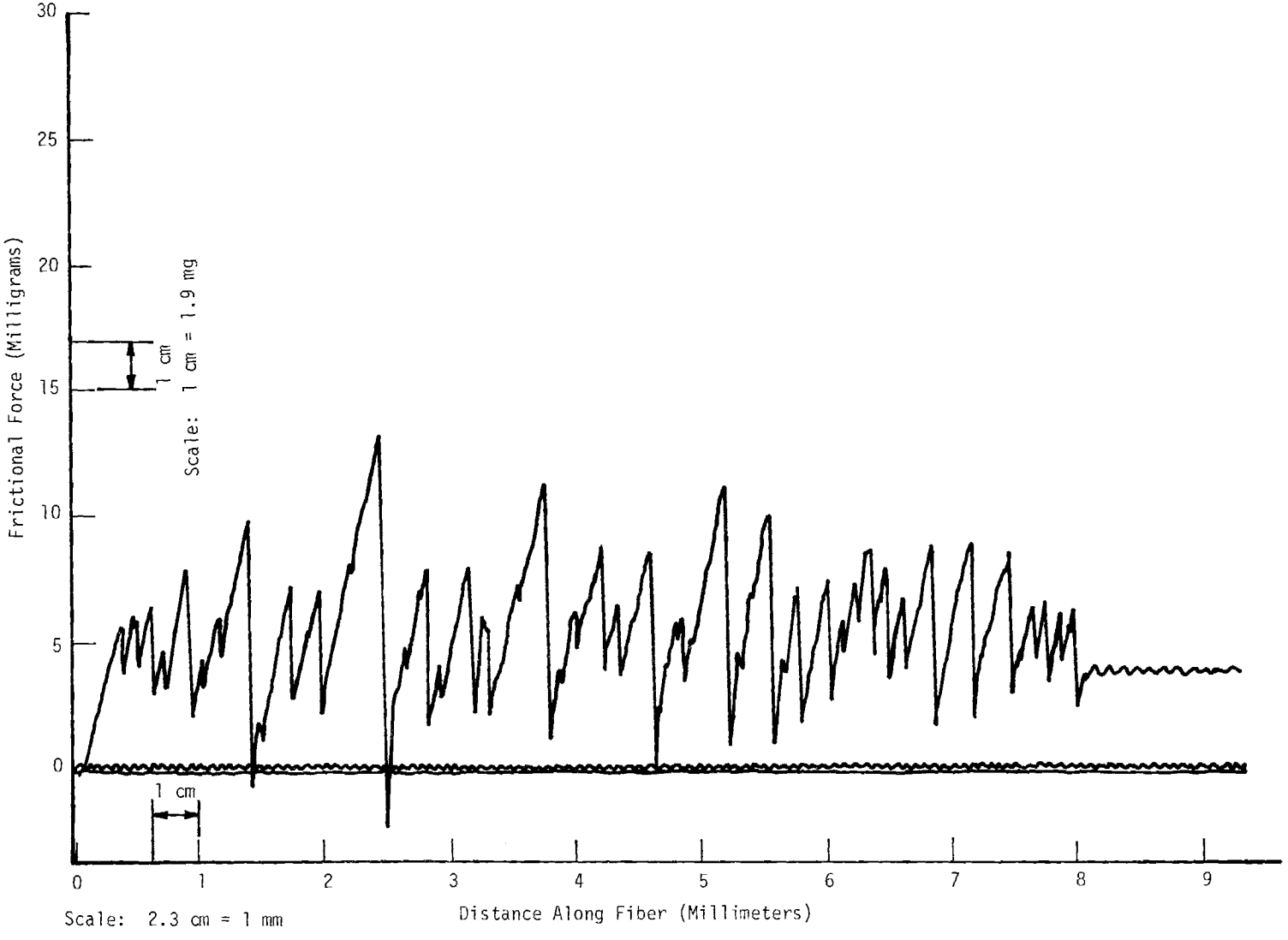


Figure 17. Typical frictional data for low-draft cotton fiber. Note more uniform peak heights.

TABLE 2

Frictional Data for High Draft Cotton Fibers

Specimen	μ_k			μ_s			μ_s/μ_k		
	A	B	C	A	B	C	A	B	C
47	0.290	0.222	0.216	0.52	0.486	0.444	1.79	2.19	2.06
55	0.237	0.252	0.264	0.495	0.532	0.502	2.09	2.11	1.90
56	0.253	0.310	0.241	0.470	0.471	0.435	1.86	1.53	1.81
57	0.338	0.318	0.344	0.578	0.625	0.615	1.71	1.97	1.78
58	0.332	0.401	0.314	0.605	0.732	0.620	1.82	1.83	1.97
59	Omit -- very large peak, snagging.								
60	0.260	0.222	0.288	0.479	0.449	0.511	1.85	2.02	1.77
61	0.347 (one very large peak)	0.322	0.272	0.567 (one very high peak)	0.553	0.497	1.63	1.72	1.83
62	0.332	0.324	0.302	0.632	0.637	0.587	1.88	1.97	1.94
63	0.220	0.202	0.277	0.465	0.458	0.485	2.13	2.38	1.75
64	0.266	0.242	0.228	0.484	0.443	0.427	1.82	1.83	1.87
65	-----	0.202	0.217	-----	0.386	0.376	----	1.91	1.73
66	0.156	0.167	0.168	0.387	0.370	0.389	2.48	2.22	2.32
79	0.320	0.237	0.171	0.605	0.565	0.410	1.89	2.34	2.39
80	0.178	0.163		0.425	0.367		2.40	2.27	
Averages	0.271	0.256	0.255	0.517	0.505	0.484	1.91	2.02	1.93
Grand Average	0.261			0.502			1.95		

TABLE 2 (Continued)

High Draft Cotton

Highest Peaks

Specimen	A_1	A_2	B_1	B_2	C_1	C_2	Normal Force
	cm		cm		cm		
47	15.3	7.3	12.4	6.6	10.4	4.6	20.8
55	8.3	6.5	11.3	5.8	10.4	6.3	20.6
56	8.4	5.4	5.9	4.8	7.0	4.6	19.3
57	7.7	6.7	11.9	7.1	11.5	6.4	20.3
58	12.8	10.5	16.1	13.4	7.5	7.4	20.0
59	Too large to measure -- estimated 30 cm						
60	9.2	5.9	8.2	6.2	11.3	7.7	20.5
61	17.0	6.1	17.5	5.8	13.5	5.7	20.0
62	11.8	7.7	11.5	7.4	8.6	7.1	20.0
63	5.8	5.7	6.0	5.4	6.4	5.3	19.5
63′	5.6	4.4	6.6	5.7	6.4	5.6	19.5
64	8.1	6.1	9.5	5.6	6.4	5.2	20.3
65	----	---	6.7	5.2	6.0	5.5	20.6
66	8.0	4.8	7.0	4.5	6.7	4.4	20.0
79	10.7	9.7	8.5	7.0	6.1	5.3	20.0
80	4.7	4.5	5.7	5.2	5.2	5.2	20.2
81	5.3	4.8	5.6	5.4	5.2	4.9	19.9
82	7.9	6.2	7.6	6.8	9.1	7.0	20.4
85	10.4	6.7	11.2	7.6	10.7	6.8	19.7
86	6.9	6.2	6.0	5.0	6.5	5.9	20.0
87	9.0	4.7	8.1	5.6	9.3	7.2	19.7
88	8.2	5.2	6.4	4.5	6.7	5.1	19.7
89	5.5	4.8	6.3	5.3	4.7	4.4	20.3
103	4.6	4.5	4.6	4.5	5.0	4.4	19.8
104	10.1	6.9	11.1	7.6	10.1	7.5	
105	10.8	8.0	10.8	5.0	10.0	5.5	
Averages	8.9	5.9	9.3	5.3	8.8	5.8	20.0

TABLE 3

Frictional Data for Low Draft Cotton Fibers

Specimen No.	μ_k			μ_s			μ_s/μ_k		
	A	B	C	A	B	C	A	B	C
67	0.236	0.289	0.250	0.537	0.591	0.505	2.26	2.04	2.02
68	0.219	0.222	0.200	0.441	0.445	0.451	2.02	2.00	2.26
69	0.296	0.338	0.355	0.654	0.640	0.655	2.21	1.89	1.85
70	0.210	0.239	0.264	0.409	0.485	0.465	1.95	2.03	1.76
71	0.241	0.246	0.291	0.483	0.476	0.481	2.00	1.93	1.65
72	0.245	0.224	0.209	0.485	0.383	0.376	1.97	1.71	1.80
73	0.143	0.141	0.173	0.337	0.326	0.362	2.46	2.32	2.10
74	0.252	0.233	0.251	0.517	0.511	0.536	2.04	2.20	2.14
75	0.223	0.214	0.229	0.432	0.412	0.452	1.94	1.93	1.97
76	omit - very large peaks								
77	0.391	0.379	0.441	0.630	0.596	0.642			
78	0.240	0.266	0.266	0.386	0.483	0.481	1.61		
90	0.226	0.179	0.228	0.516	0.479	0.503	2.28	2.58	2.21
91	0.238	0.293	0.266	0.510	0.570	0.599	2.14	1.96	2.26
92	0.189	0.183	0.186	0.413	0.406	0.422	2.18	2.22	2.27
93	0.222	0.193	0.227	0.479	0.462	0.465	2.16	2.27	2.05
94	0.228	0.258	0.247	0.513	0.573	0.543	2.26	2.22	2.19
95	0.314	0.358	0.368	0.633	0.763	0.77	2.02	2.13	2.08
96	0.251	0.273	0.255	0.546	0.551	0.564	2.18	2.02	2.21
97	0.286	0.273	0.305	0.628	0.620	0.680	2.18	2.27	2.23
Averages	0.243	0.252	0.262	0.496	0.510	0.514	2.04	2.02	1.97

TABLE 3 (Continued)

Low Draft Cotton

Highest Peaks

	A_1	A_2	B_1	B_2	C_1	C_2	NF_A	NF_B	NF_C
67	7.4	7.0	8.7	7.3	7.8	7.0	20.4	20.4	20.4
68	6.7	5.2	6.4	5.9	6.5	6.3	19.7	19.7	19.7
69	10.7	9.0	11.9	8.4	11.9	10.0	19.6	19.6	19.6
70	5.1	4.9	6.7	5.7	7.6	6.5	20.2	20.2	20.2
71	6.6	6.5	6.5	5.7	6.1	5.9	21.7	21.7	21.7
72	8.4	8.1	6.4	5.9	7.5	5.4	20.7	20.7	20.7
73	4.8	4.2	4.2	3.9	5.1	4.9	20.5	20.5	20.5
74	10.0	7.5	10.6	7.0	7.7	7.4	20.2	20.2	20.2
75	5.3	5.2	5.2	4.8	6.7	5.9	20.0	20.0	20.0
76	Omit because of very large peak.								
77	12.4	11.8	14.4	6.8	15.1	12.8	20.6	20.6	20.6
78	11.3	4.6	11.1	6.3	10.2	9.8	20.0	20.0	20.0
90	9.6	6.9	5.6	5.5	6.8	6.1	20.0	20.0	20.0
91	8.8	7.0	8.1	8.0	8.0	8.0	20.0	20.0	20.0
92	5.7	5.2	7.0	4.8	5.6	4.7	20.0	20.0	20.0
93	6.4	5.9	5.7	5.2	6.5	5.5	20.0	20.0	20.0
94	7.4	6.1	7.5	6.3	7.2	6.5	20.0	20.0	20.0
95	8.0	6.7	11.4	10.3	9.4	8.6	20.0	20.0	20.0
96	7.4	6.3	7.5	6.6	7.5	6.7	19.6	19.6	19.6
Average	7.9	6.6	8.1	6.4	8.0	7.1	20.2 Average		

$$\mu_s \text{ first peak} = \frac{1.90}{20.2} \times 8.0 = 0.76$$

$$\mu_s \text{ second peak} = \frac{1.90}{20.2} \times 6.7 = 0.63$$

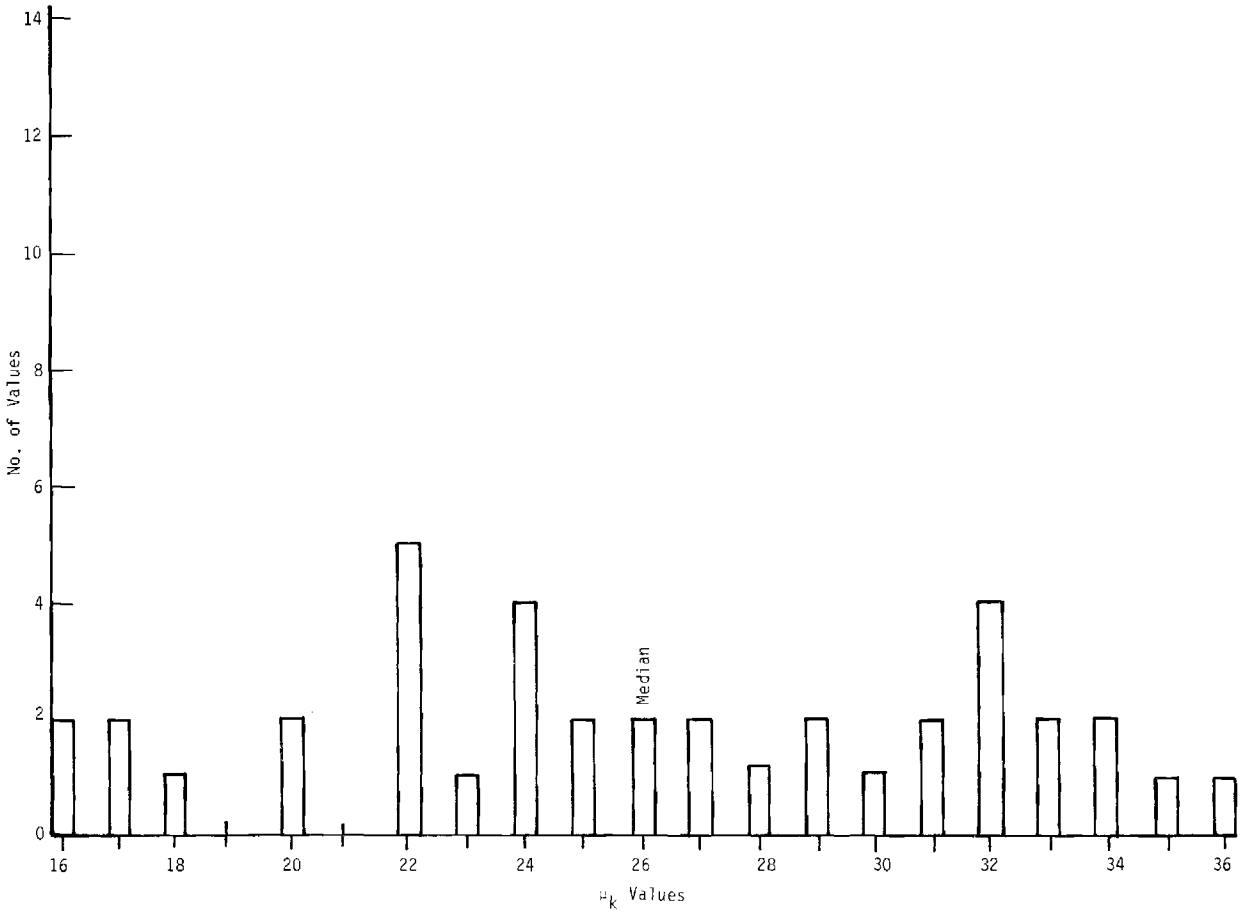


Figure 18. Distribution of μ_k values measured for high-draft cotton.

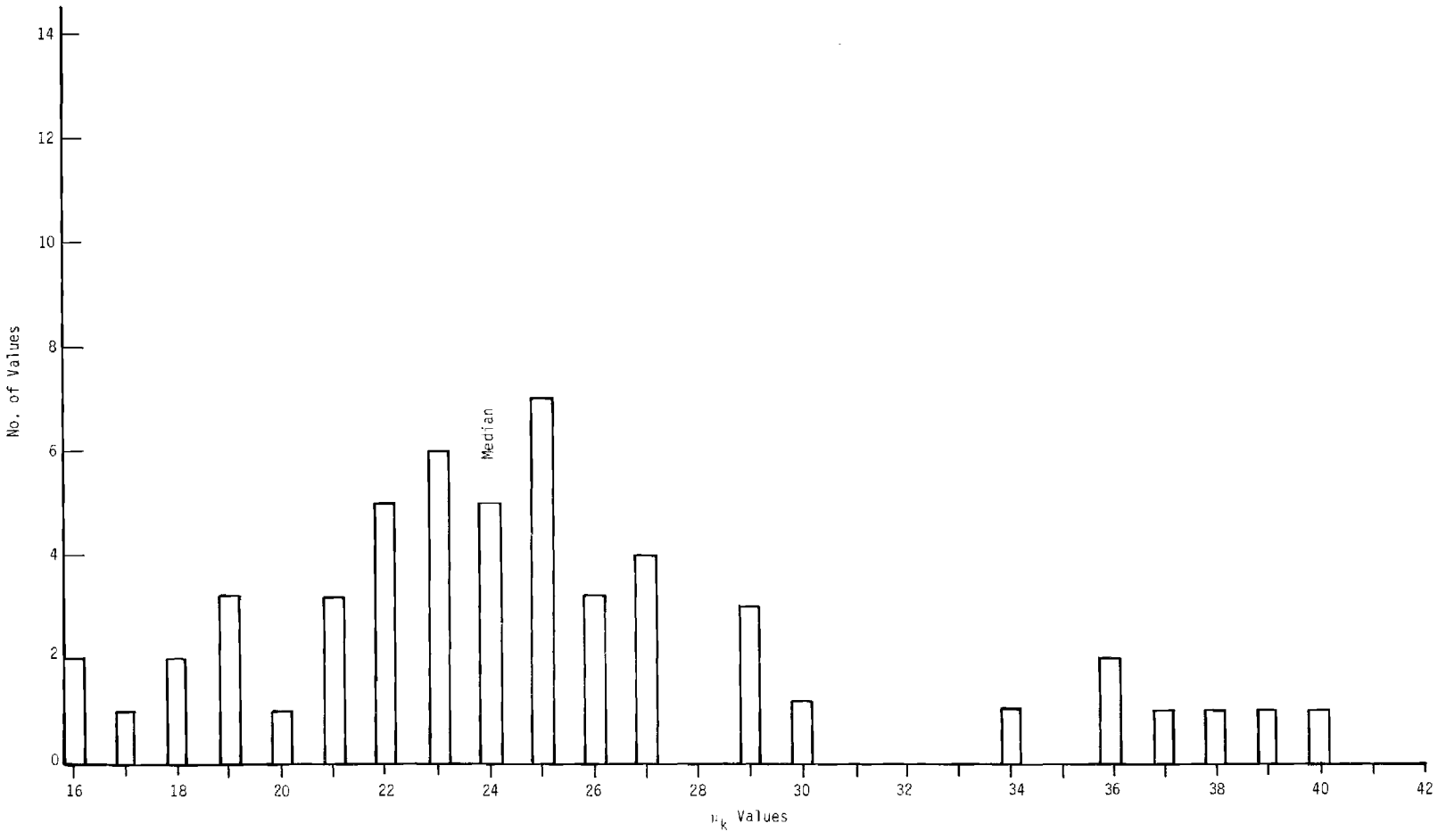


Figure 19. Distribution of μ_k values measured for low-draft cotton.

low draft are more closely grouped about the median. The frictional data of the high draft type displayed frequent very large peaks, some going off scale. Hence the character of the curves was considerably different. Tabulation of the two highest peaks gave μ_s values of 0.86 and 0.54 respectively.* These are 3.3 and 2.0 times μ_k respectively. Since the scanning electron micrographs and the optical sections indicate the high draft fibers also have a different shape, i.e., a flatter ribbon with prominent convolutions, it appears that this shape factor causes the primary difference between the two sets of data and also the high dispersion of the high draft data.

The smoother appearance of the high draft fiber as indicated by electron microscope replica methods may have also contributed to a frictional difference correlating with a report of Scardini[11] that he observed higher cohesion measurements for man-made fibers of low surface roughness. Hence, the high-draft cotton possesses three features which affect its intrinsic behavior, i.e., a flatter fiber, a more convoluted one, and a smoother fiber surface. The convolutions interlock to give high stick friction and the smooth and flat surfaces may allow more area of interfiber contact at contact points. These effects combine to give a fiber requiring settings for high draft conditions in processing.

E. FRICTIONAL MEASUREMENTS AT LOW NORMAL FORCES

1. General

Utilizing the low normal force apparatus described in the last report, friction measurements were made for a series of fiber pairs at normal forces in the range 2 to 20 mg. The fiber pairs cotton-glass, nylon-glass, and nylon-cotton were examined. A few experiments were performed with no normal force applied except the weight of the traversing fiber alone which was attached at one end only to the traversing mechanism. Experiments

* These may be compared with 0.76 and 0.63 for the low draft cotton.

were also performed on the withdrawal of a single cotton fiber from card sliver and from roving.

2. Measurements at Normal Forces of 2, 5, 10 and 20 mg for Various Fiber Pairs

Approximately 300 frictional measurements were made of the fibers cotton against glass, nylon against glass, and cotton on nylon at normal force levels of 2, 5, 10, and 20 mg. Typical data plots for cotton against glass at the various levels are shown in Figures 20, 21, 22, and 23. A frictional curve for nylon against glass at 2 mg is shown in Figure 24. A summary of the various experiments and number of specimens examined is given in Table 4. It will be noted that in general there is a successive reduction in the μ_s and μ_k values for both cotton and nylon against glass as the normal force is increased and that the values for both cotton and nylon against glass are very similar. The value for cotton against nylon is somewhat higher. The various data are plotted in Figure 25 for ready comparison.

3. Measurements of the Friction of Fibers with No Normal Force Externally Applied

It has previously been shown that reductions in tension[3] and normal force (as discussed above) result in increases in the coefficient of friction between fibers. In order to examine this effect further a cotton fiber attached at only one end was drawn across a fiber on the servo controlled galvanometer. Withdrawal was made by a worm gear driven vise at a rate of 0.1 mm/sec. A typical frictional curve for cotton on cotton is indicated in Figure 26. The maximum forces indicated for the cotton fibers examined were in the ranges 0.25 mg to 1.0 mg. Since the fiber weight (hence normal force) was only a few micrograms (at most) it is evident that the values of either μ_s or μ_k would be relatively large.

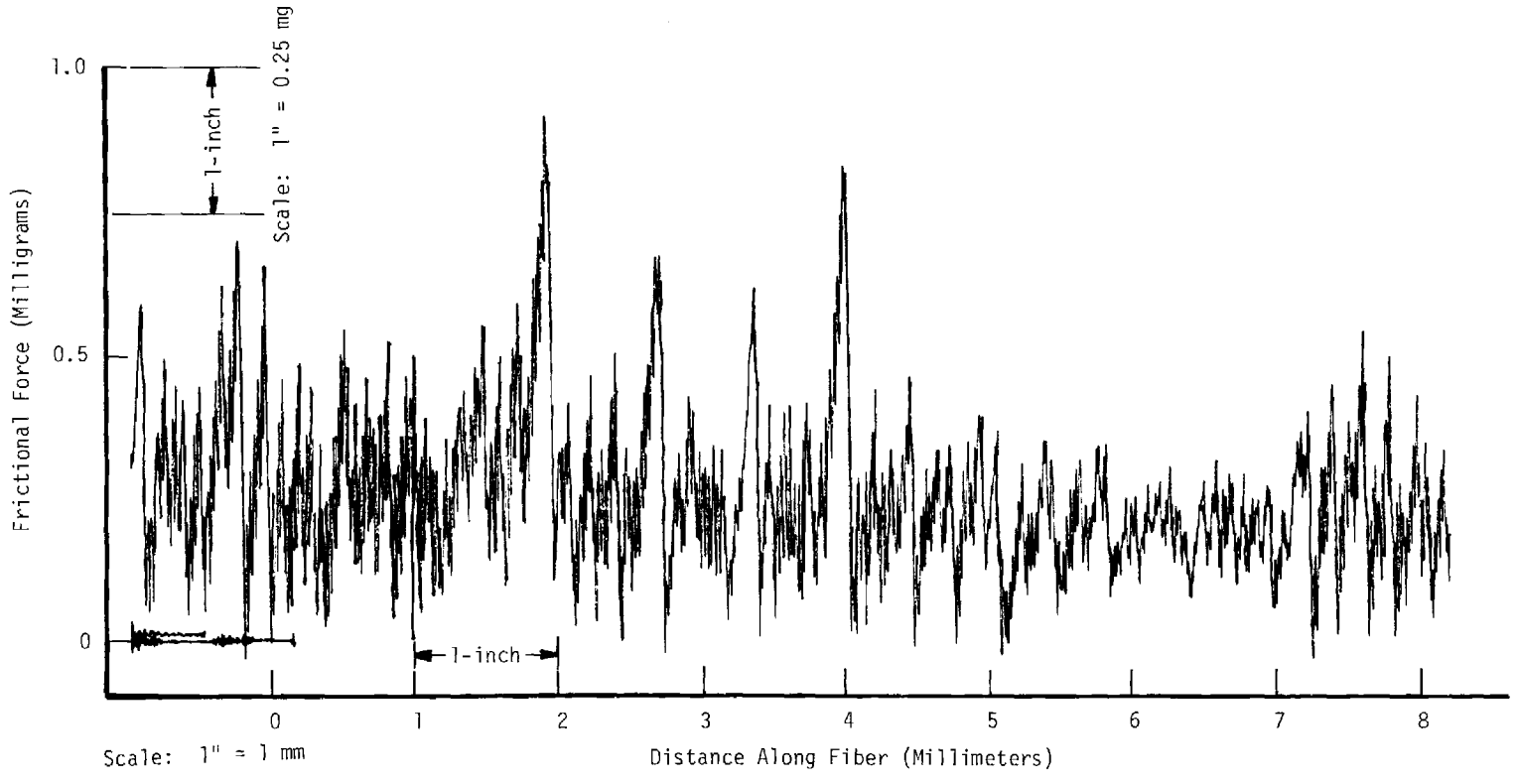


Figure 20. Frictional data plot of cotton fiber against glass at 2 mg normal force.

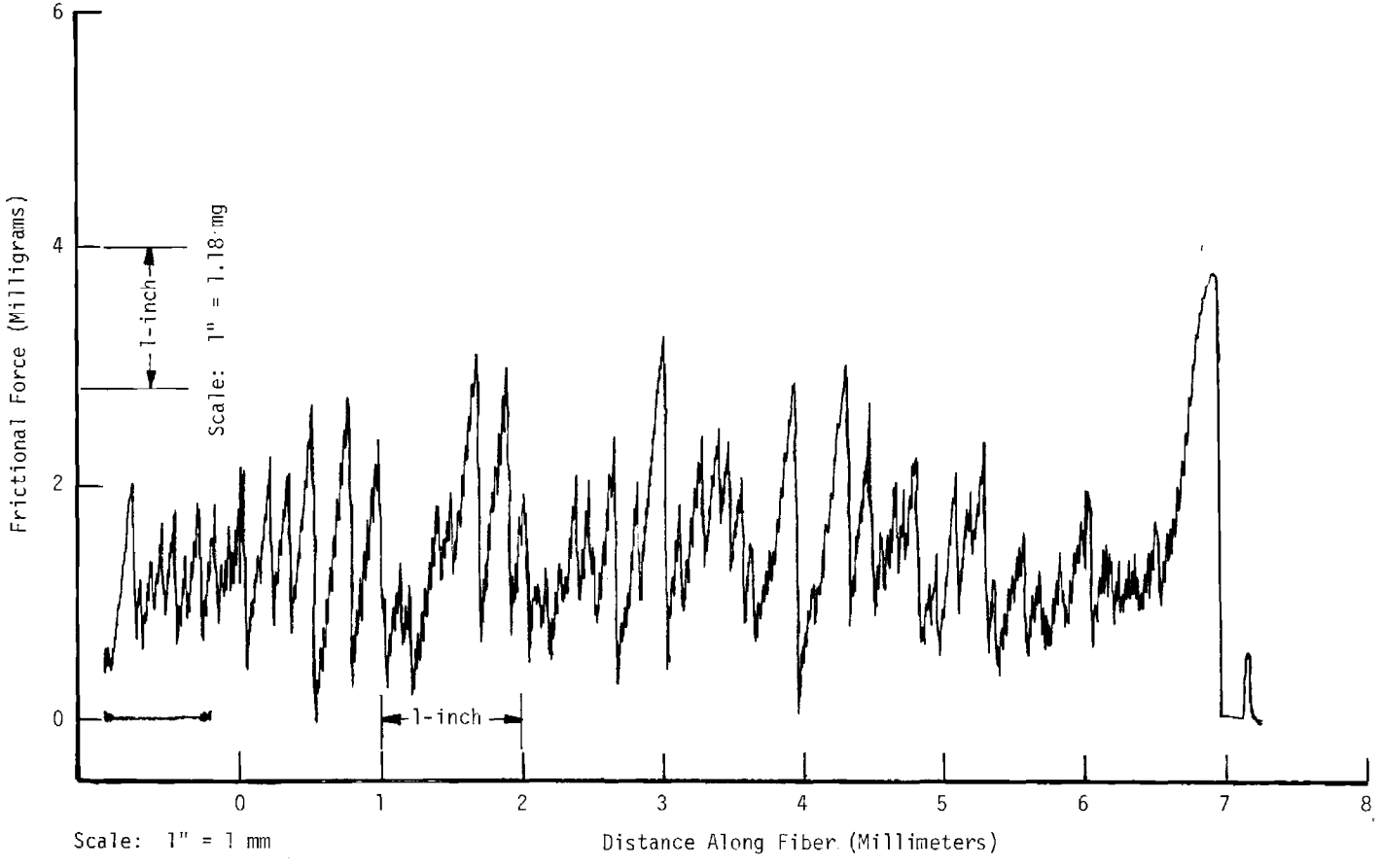


Figure 21. Frictional data plot of cotton fiber against glass at 5 mg normal force.

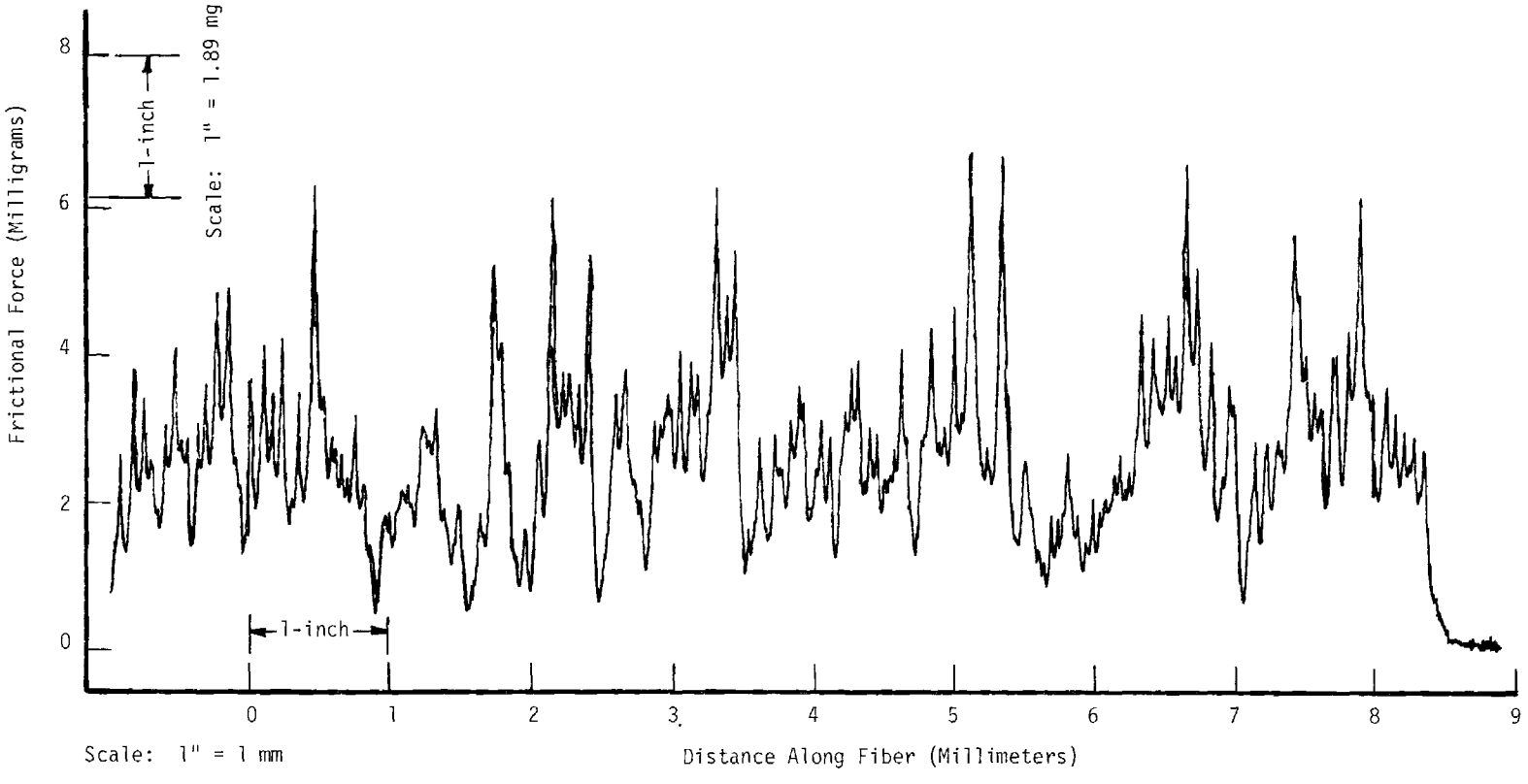


Figure 22. Frictional data plot of cotton fiber against glass at 10 mg normal force.

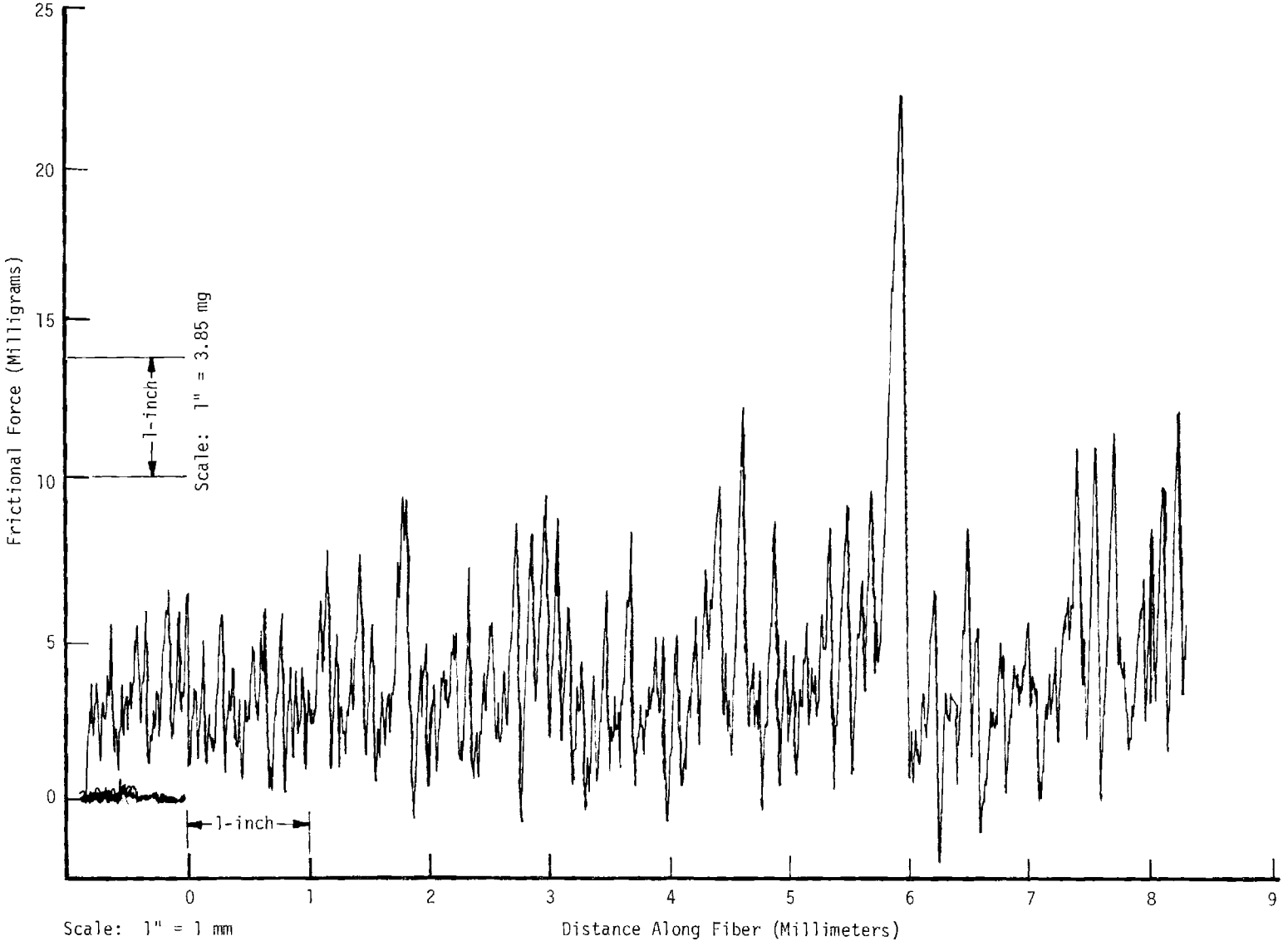


Figure 23. Frictional data plot of cotton fiber against glass at 20 mg normal force.

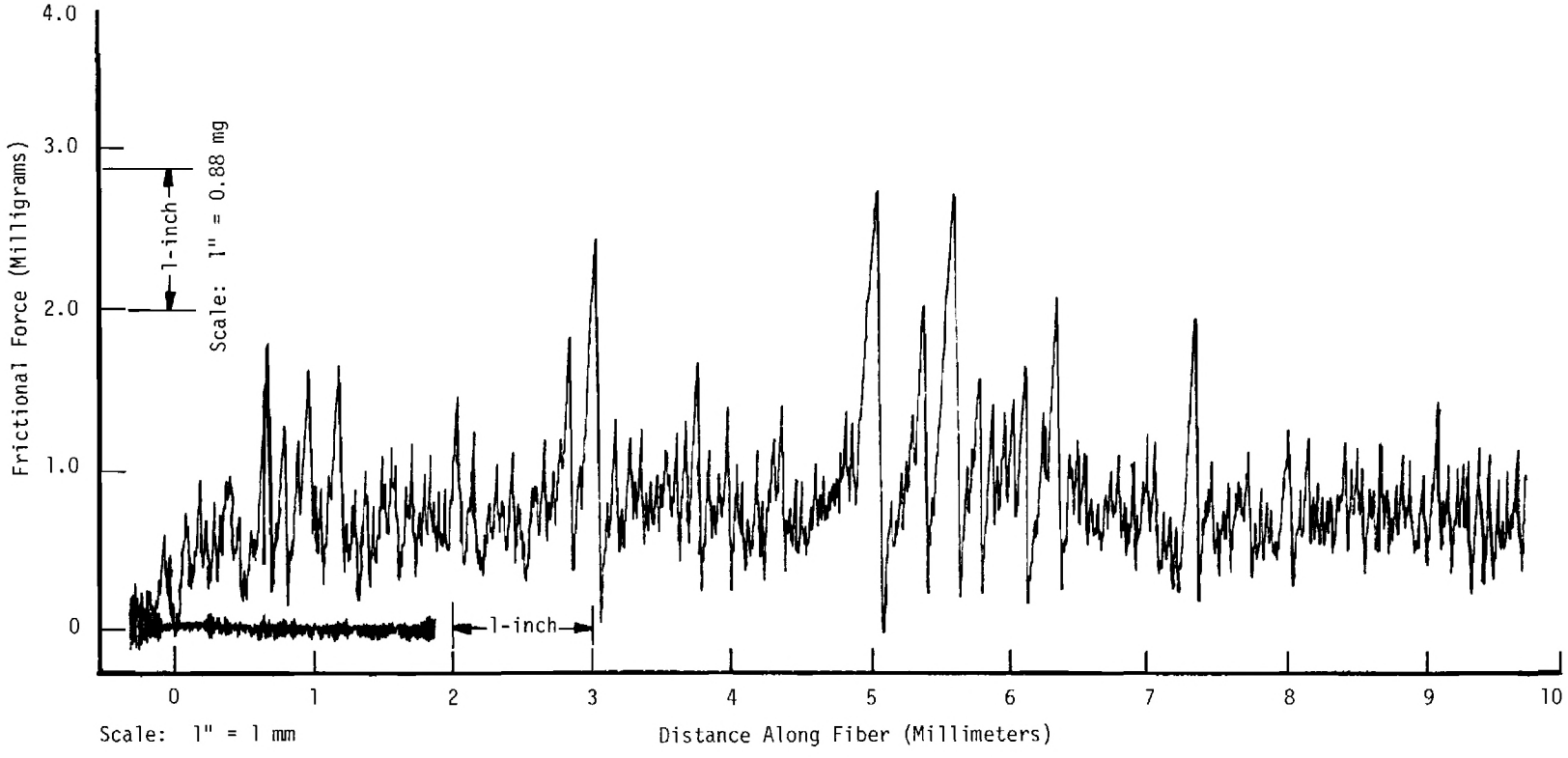


Figure 24. Frictional data plot of cotton fiber against nylon at 2 mg normal force.

TABLE 4

Frictional Data at Low Normal Forces for Various Fiber Pairs

Cotton on Glass

Materials	Normal Force	No. of Runs	μ_s	μ_k	μ_s/μ_k
Cotton on glass	0	1	0.92 mg (maximum force recorded)		
Cotton on glass	2	9	0.902	0.398	2.27
Cotton on glass	2	20	1.097	0.248	4.48
Cotton on glass	2	9	0.587	0.286	2.19
Cotton on glass	5	9	0.589	0.344	1.73
Cotton on glass	5	20	0.674	0.180	3.78
Cotton on glass	10	20	0.598	0.196	3.07
Cotton on glass	10	9	0.558	0.151	3.73
Cotton on glass	10	20	0.508	0.185	2.76
Cotton on glass	19.5	20	0.512	0.148	3.55
Cotton on glass	19.5	9	0.553	0.186	2.99
Average Values for All Cotton on Glass					
Cotton on glass	2 mg	38	0.93	0.293	3.17
Cotton on glass	5 mg	29	0.67	0.232	2.89
Cotton on glass	10 mg	49	0.56	0.183	3.07
Cotton on glass	19.5 mg	29	0.53	0.160	3.32

TABLE 4 (Continued)

Frictional Parameters

Cotton on Nylon

Materials	Normal Force	No. of Runs	μ_s	μ_k	μ_s/μ_k
Cotton on Nylon	0	4	1.8 mg (maximum force recorded)		
Cotton on Nylon	2	9	0.842	0.368	2.32
Cotton on Nylon	2	20	1.160	0.361	3.25
Cotton on Nylon	5	9	0.632	0.223	2.78
Cotton on Nylon	5	20	0.700	0.266	2.62
Cotton on Nylon	10	20	0.671	0.252	2.68
Cotton on Nylon	19.5	20	0.587	0.224	2.67
Cotton on Nylon	19.5	9	0.632	0.261	2.43
		Nylon on Cotton			
Nylon on Cotton	0	3	1.12 mg (maximum force recorded)		
		Average Values Cotton on Nylon			
Cotton on Nylon	2	29	1.06	0.364	2.92
Cotton on Nylon	5	29	0.68	0.253	2.69
Cotton on Nylon	10	20	0.671	0.252	2.68
Cotton on Nylon	19.5	29	0.60	0.235	2.56

TABLE 4 (Continued)

Frictional Properties of Nylon on Glass

Materials	Normal Force	No. of Runs	μ_s	μ_k	μ_s/μ_k
Nylon on glass	2	9	0.587	0.286	2.19
Nylon on glass	5	9	0.585	0.260	2.10
Nylon on glass	10	40	0.483	0.203	2.38
Nylon on glass	10	9	0.467	0.259	1.81
Nylon on glass	10	10	0.535	0.189	2.88
Average Values for All Nylon on Glass					
Nylon on glass	2	9	0.587	0.286	2.19
Nylon on glass	5	9	0.585	0.260	2.10
Nylon on glass	10	59	0.490	0.209	2.34

TABLE 4 (Continued)

Frictional Parameters

Nylon on Nylon

Materials	Normal Force	No. of Runs	μ_s	μ_k	μ_s/μ_k
Nylon on Nylon (Crimped)	0	1	1.8 mg (maximum force recorded)		
		Cotton on Cotton			
Cotton on Cotton	0	7	1.63 mg (maximum force recorded)		
		Summary of Data Recorded for No Applied Normal Force*			
Cotton on Glass	0	1	0.92 mg (maximum force recorded)		
Cotton on Nylon	0	4	>1.8 mg (maximum force recorded)		
Cotton on Cotton	0	7	1.63 mg (maximum force recorded)		
Nylon on Cotton	0	3	1.12 mg (maximum force recorded)		
Nylon on Nylon (Crimped)	0	1	>1.8 mg (maximum force recorded)		
Glass on Nylon	0	2	0.68 mg (maximum force recorded)		

* Only normal force employed was weight of fiber, a few micrograms (3 to 6 μgm), plus force of cohesion.

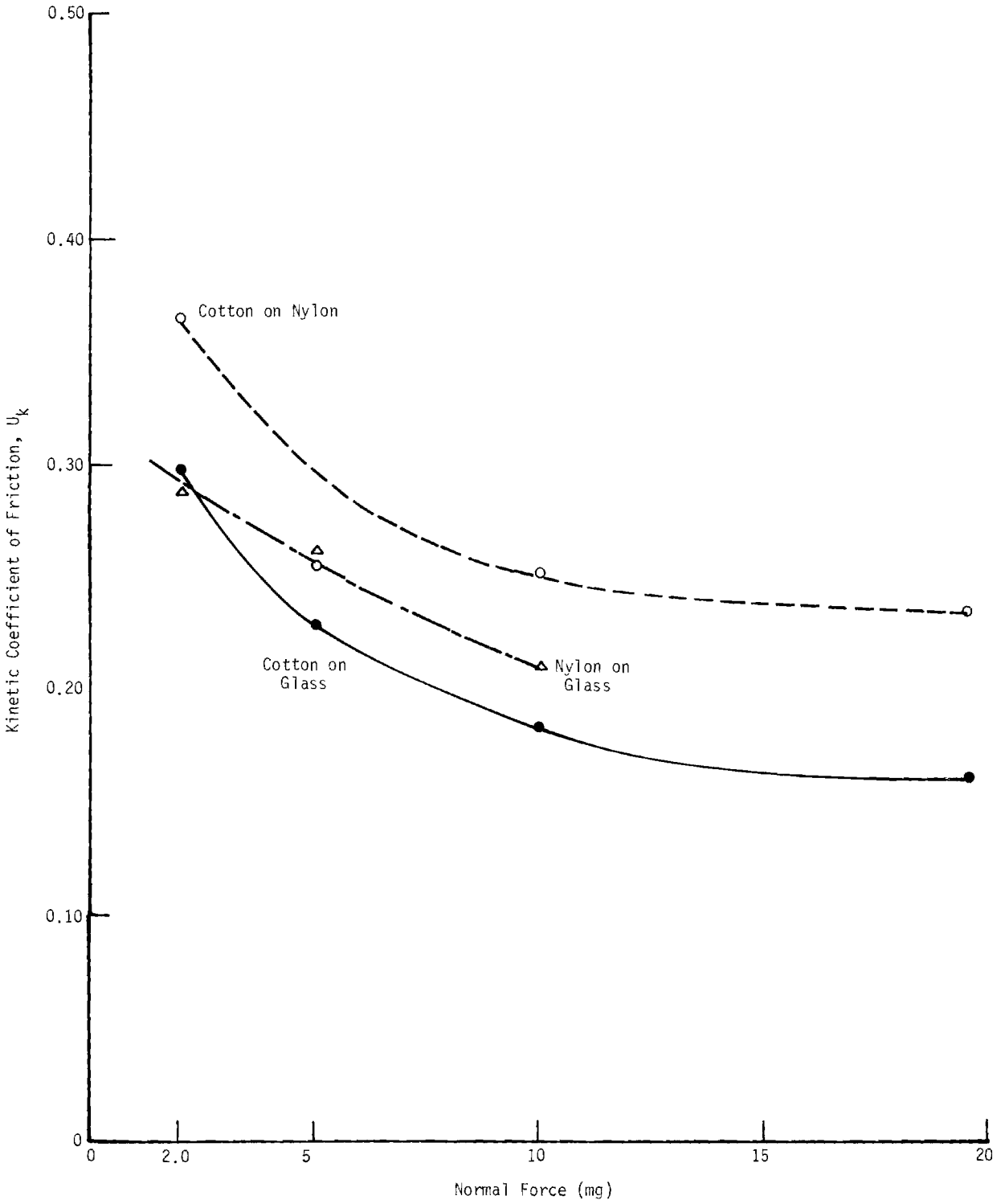


Figure 25a. Variation of friction with normal force of cotton against glass and against nylon. (Coefficient of kinetic friction).

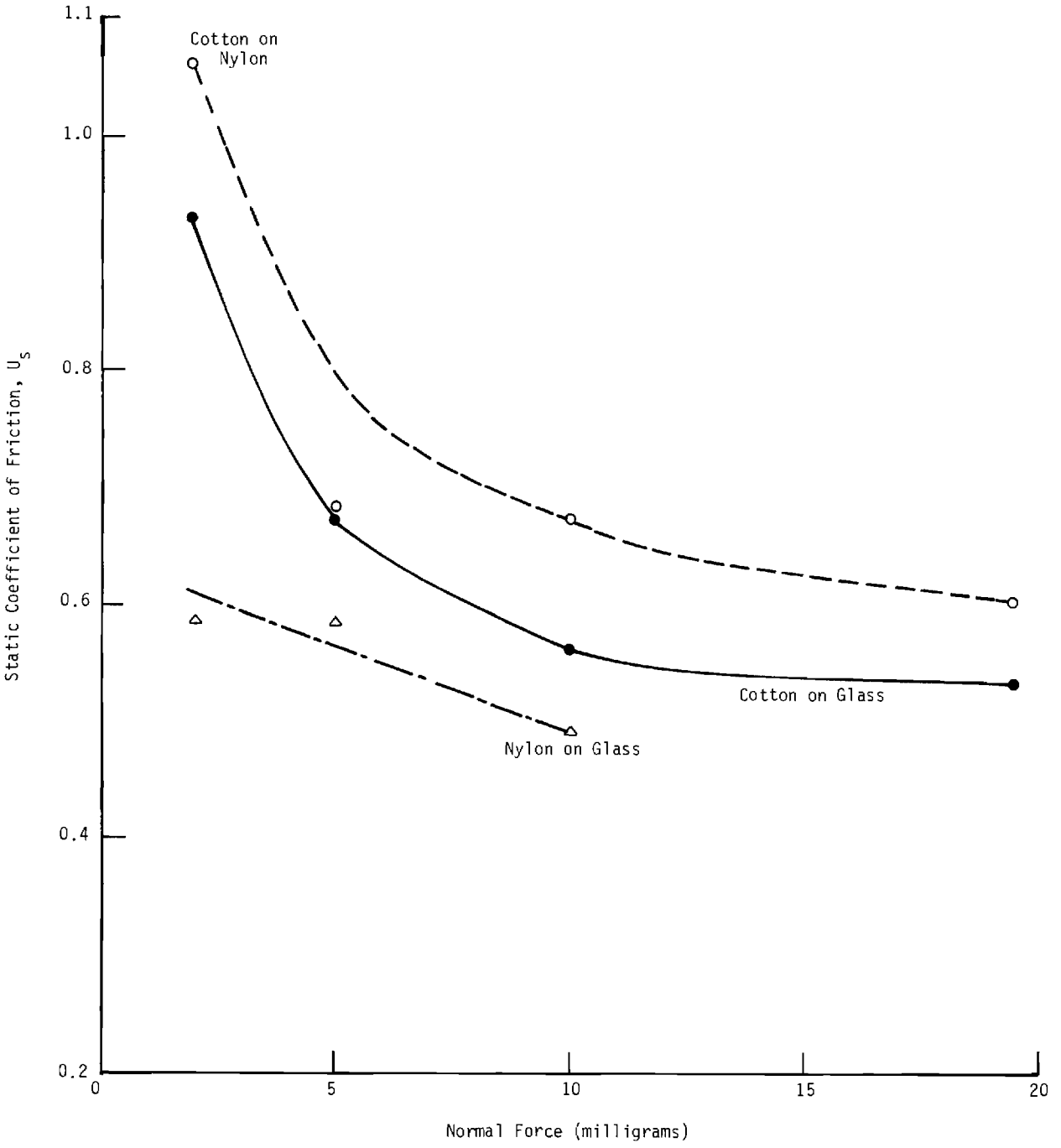


Figure 25b. Variation of friction with normal force of cotton against glass and against nylon. (Coefficient of static friction).

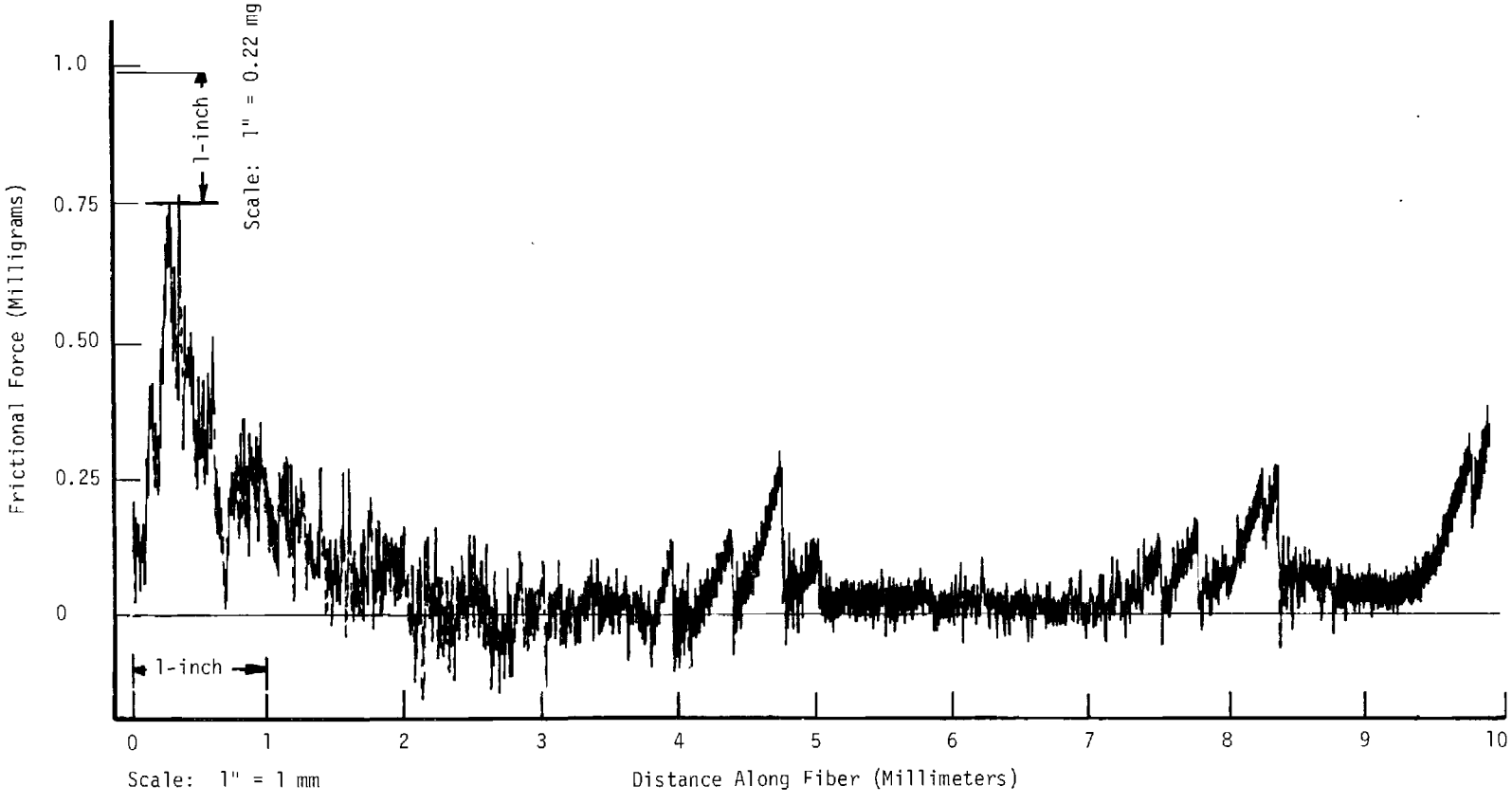


Figure 26. Frictional data plot for cotton fiber, supported only at one end, as it was drawn across a cotton fiber attached to the friction recording instrument.

Data for crimped nylon against nylon are presented in Figure 27. It is evident in Figures 26 and 27 that convolutions in cotton and crimp in nylon give large frictional forces. However, if one then examines the data for nylon on cotton in Figure 28 and glass on nylon in Figure 29 it is also apparent that friction between two smooth surfaces is also very large, of the same relative magnitude and the principal change observed is in the character of the curves. This is particularly true for glass on nylon where the saw tooth peaks characteristic of cotton are replaced by many high sticks of relatively short time interval.

4. Forces to Withdraw Cotton Fibers From Card Sliver and From Roving

A Servo Torque-Balance Reaction Dynamometer,* its amplifier, and an x-y recorder (Model 7000A), in conjunction with a worm driven fiber holder, were utilized to perform measurements of the forces to withdraw a single cotton fiber from a fiber bundle of card sliver or of roving.

The card sliver (4.2 gram/yd or 64.9 grains/yd) was mounted at the clamp, which was movable at the speed of 2.38×10^{-2} cm/sec, and a single fiber at the other end of the sliver was clamped by a pin vise using a vacuum technique. The pin vise was attached to the dynamometer lever arm. The fiber withdrawal force was recorded during the withdrawal of the card sliver by the motor driven worm gear. Five measurements were made at four different places in the sliver. This action was repeated for the roving (0.3 gram/yd or 4.5 grains/yd).

The span length was 2" for card sliver and 1 3/4" for the roving.

From the data plots, the maximum and average withdrawal forces and the energy needed to pull the respective fibers out of sliver and roving were calculated. A typical plot is shown in Figure 30.

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* Described in Semiannual Report No. 3 of this Grant.

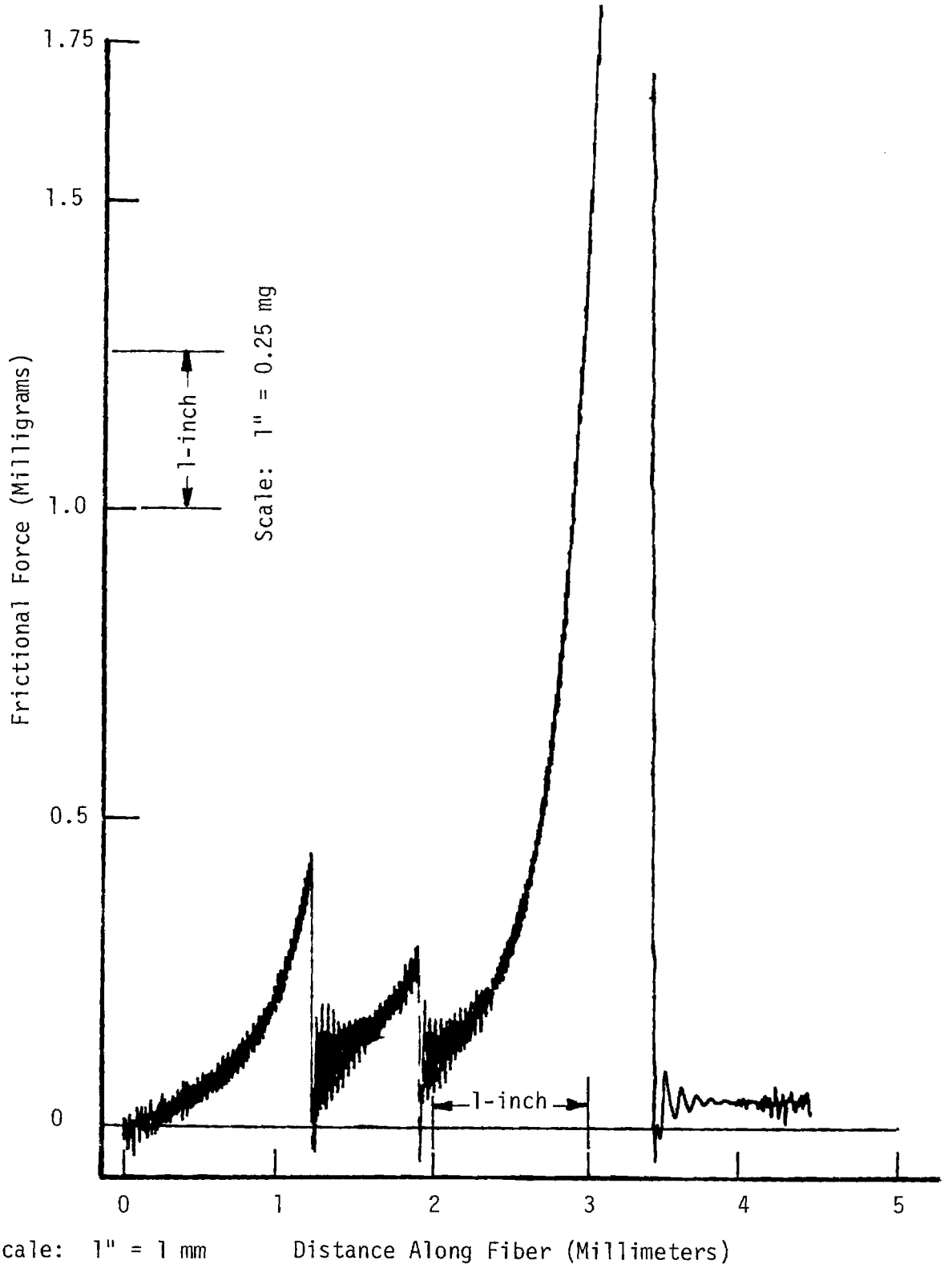

Figure 27. Frictional data plot for crimped nylon fiber, supported only at one end, as it was drawn across a nylon fiber attached to the friction recording instrument.

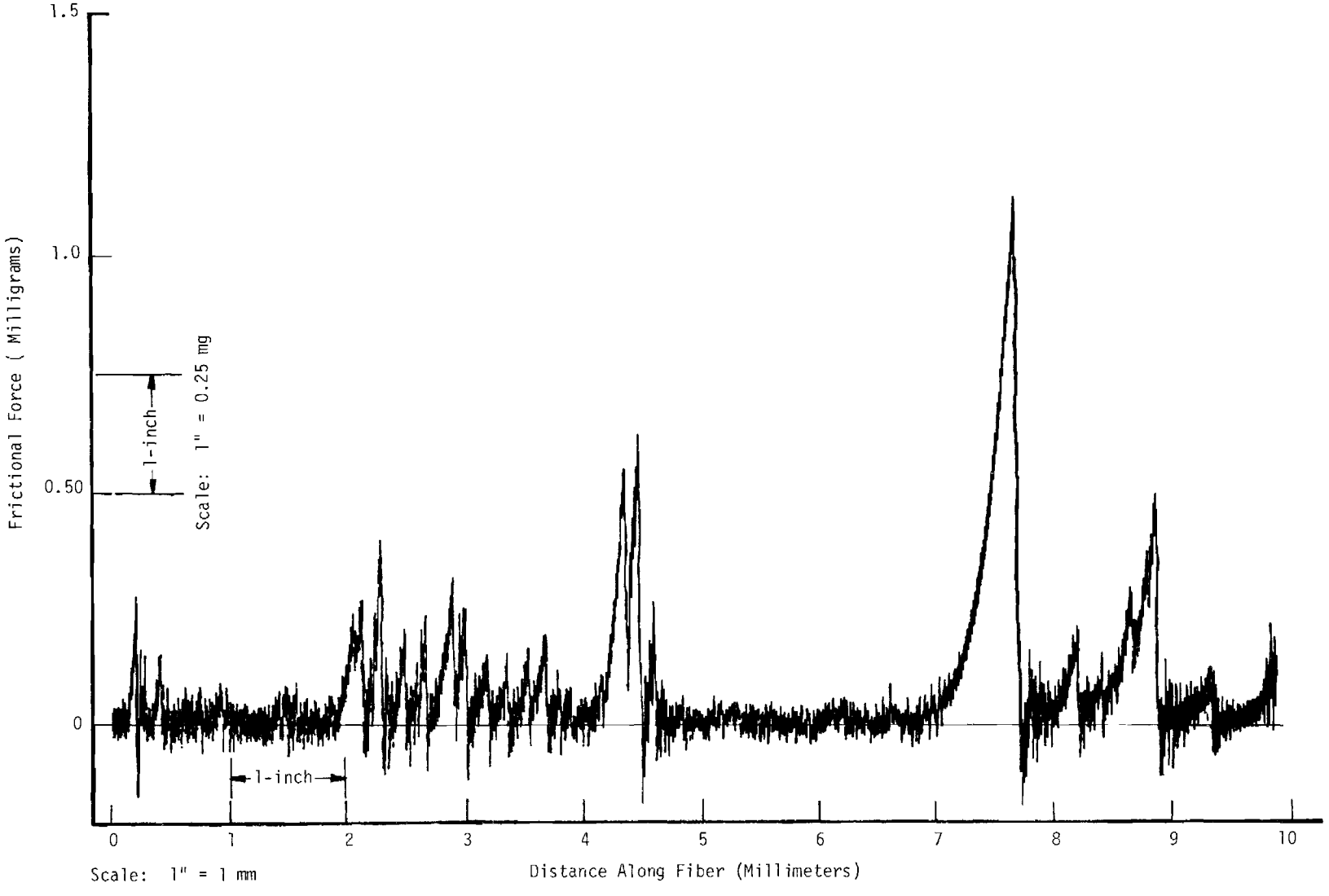

Figure 28. Frictional data plot for nylon fiber, supported only at one end, as it was drawn across a cotton fiber attached to the friction recording instrument.

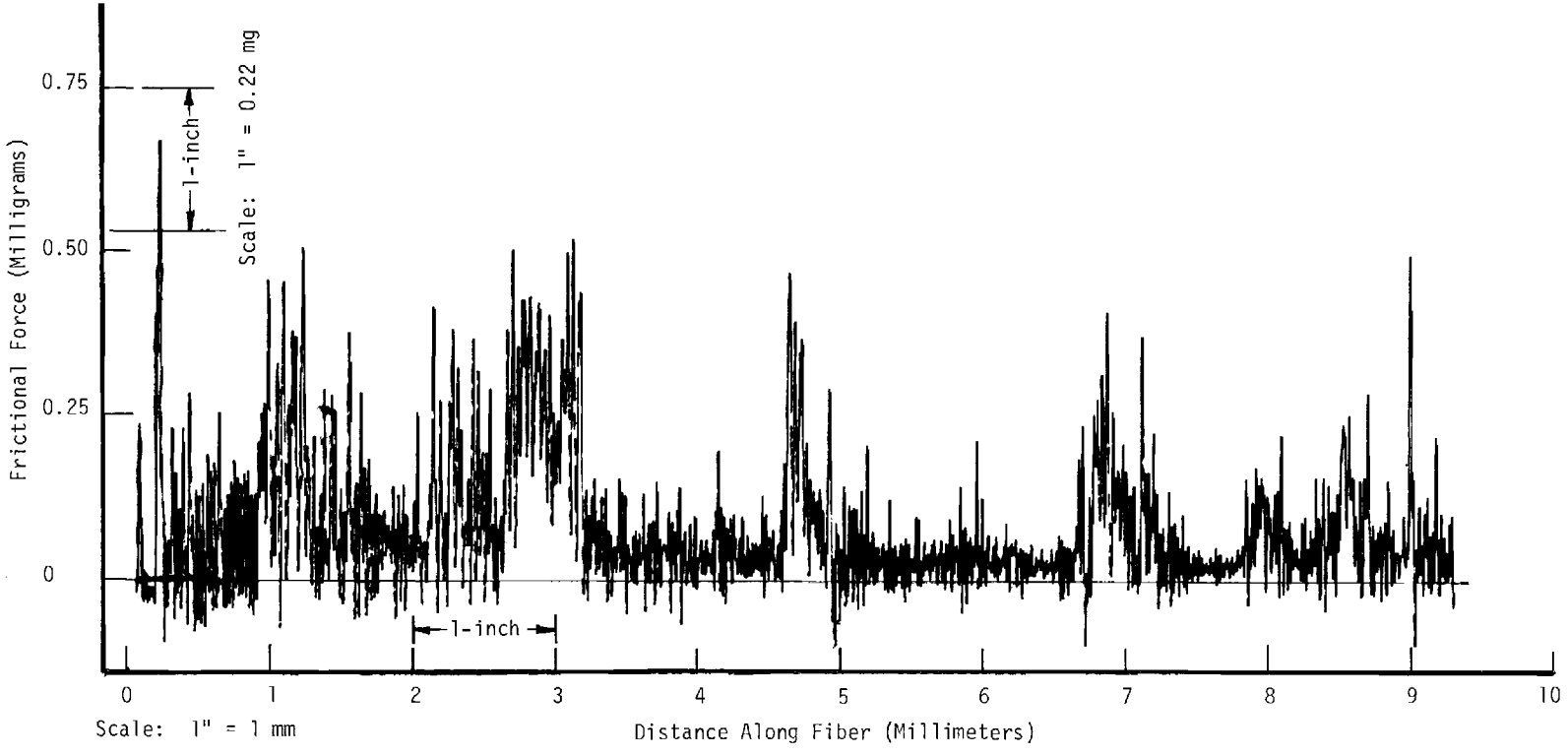

Figure 29. Frictional data plot for glass fiber, supported only at one end, as it was drawn across a nylon fiber attached to the friction recording instrument.

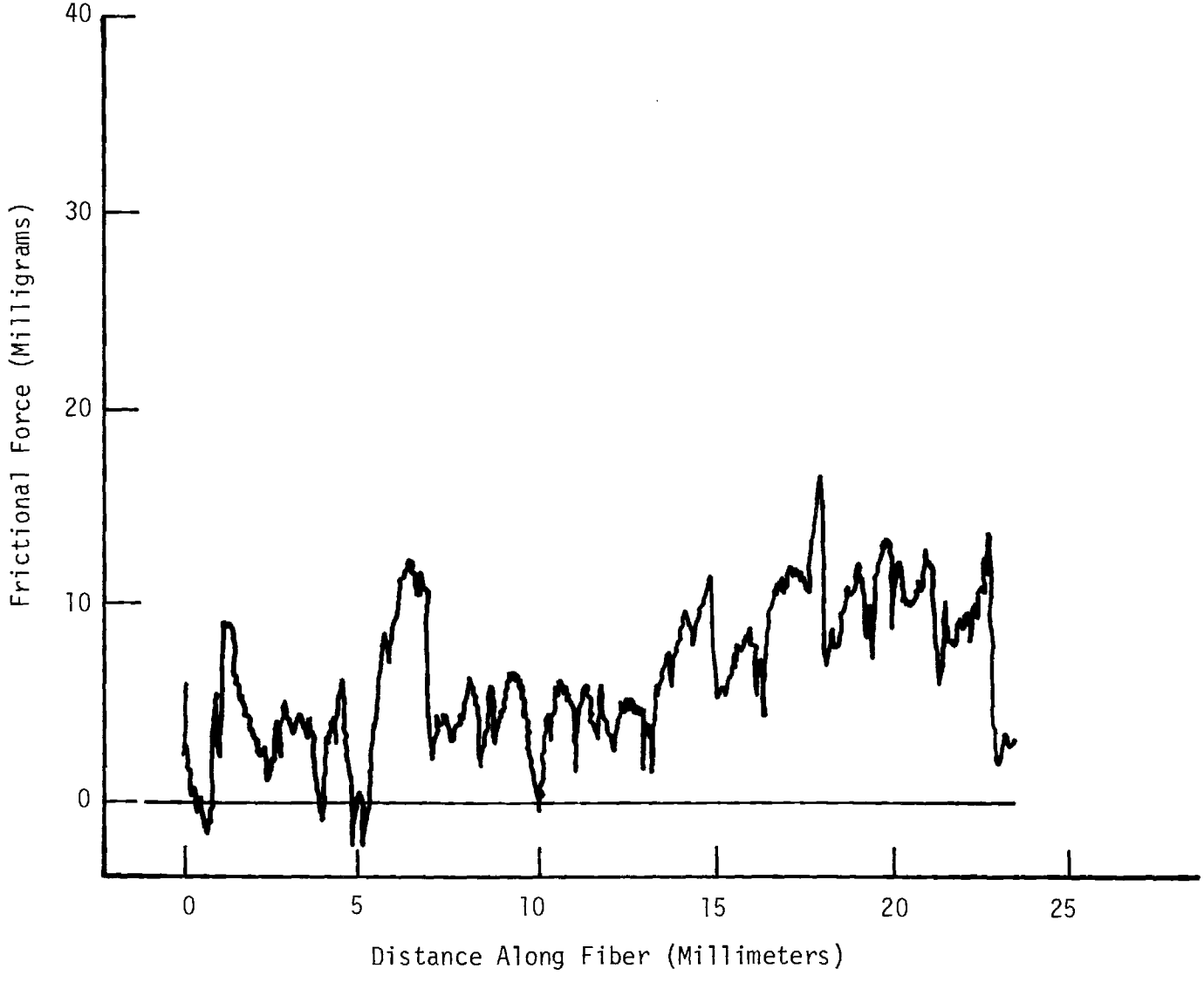

Figure 30. Frictional data plot exhibiting withdrawal force and energy to remove a fiber from 1-3/4" span length of cotton roving.

The results are displayed below:

	Card Sliver	Roving
maximum withdrawal force (mg)	42.2	24.9
average withdrawal force (mg)	11.6*	7.5*
energy (ergs)	25.3*	12.7*

* 20 measurements

The withdrawal force is the average force exerted over the time required to withdraw the fiber whereas the energy is this average force times the time for withdrawal of the fiber. The total energy is the sum of the energy required to withdraw the fiber from the sliver or roving and of the energy needed to straighten the fiber. On the other hand, since the force needed to straighten the fiber is relatively very small the measured values of force and energy represent those applied to overcome fiber-to-fiber friction. Although the frictional force of an individual fiber against a single fiber may be very small, relatively large forces are required to pull the fiber out of a bundle of fibers. Since the fibers are not parallel in the sliver or roving and each fiber is contacting many fibers these values are significantly large since as we have already seen forces of 1 mg to draw a single fiber across another with no external normal force are not uncommon. In some cases, several additional fibers were withdrawn by the single fiber. These were entangled near the end of the fiber being withdrawn.

It appears that the differences in the withdrawal force and energy of card sliver and roving are caused by the differences in fiber parallelization of the stages. Hence, the frictional force values obtained for card sliver contains a force to overcome random fiber placement and entanglement whereas for roving the measurement must represent a more nearly true frictional measurement. Based on the 1 mg force discussed for a single

fiber (no normal force applied) it appears that 6 or 7 fibers are in contact with each fiber. This is a reasonable number from the geometric standpoint.

F. OTHER EXPERIMENTS

Data obtained by x-ray diffraction and infrared spectroscopy techniques during the current period have been limited and will be included in the next Semiannual Report (August 15, 1967).

Four graduate students have been assigned problems related to this research. These include a more extensive evaluation of the low normal force friction instrument, an investigation of the frictional changes in Empire WR cotton as it is processed from the card to the yarn, a similar investigation of changes in pima cotton during manufacturing into yarn in a regular textile mill, and further infrared investigations of cotton fibers, including use of polarization techniques and studies of the effects of deuteration of the cotton on its spectra.

Preliminary data on the studies will be available for the next report, and the theses will be completed by December 1967.

G. COMMENTS

The character of the frictional data plots continues to prove to be a significant feature of friction measurement. Effects of shape and fiber damage are visible as is evident in the cases of the high draft cotton and of crimped nylon against nylon. However, there is also a character specific to smooth surfaces, for instance, glass fiber on nylon. This consists of relatively high excursions at a rapid rate. In the case of a crimped, damaged, or convoluted fiber, the snagging or interlocking of the protuberances with one or more fibers causes saw tooth excursions in the plot. At the completion of the excursion, or stick, a translated cotton fiber

(on 90 degree arrangements) may skip a number of convolutions. Very large sticks contribute to large variation in the values of both the kinetic and static friction and account for a wide scatter of values as is indicated for high draft cotton. The large slips, after sticks, also mean that only a small portion of the fiber surface is actually examined. For smooth surfaces a higher μ_k value may be evident; and a lower value of the ratio μ_s/μ_k may result. However, since the principal measurement effort thus far has been devoted to cotton, insufficient data on smooth surface materials have been obtained for proper knowledge of their behavior.

The low normal force instrument indicates a trend to increased frictional force as the normal force is reduced. However, some inaccuracies due to improper zero registration or vibration have cast doubt on the accuracy of some of the data; and the values appear to be low compared to a theoretical absolute value. An extensive effort is being made to realize the full potential accuracy of the instrument down to a normal force of less than 2 milligrams and this work is currently the subject of a thesis investigation.

Using the instrument, in its present state, has indicated the high relative values of friction between fibers in contact under the conditions in which only the fiber weight of the translated fiber and forces of cohesion act as the normal force. Forces of 1 mg registered for single-fiber cross contacts indicate high frictional forces exerted between fibers at low tension and low normal force, as expected. Effects of convolutions or crimp in the fiber were especially apparent here.

With the torque dynamometer the frictional force to withdraw a fiber from a bundle proved to be large, of the order of 7 mg for withdrawal from roving. This large force suggests the fiber is in contact with the equivalent of about seven fibers under condition of low tension and low normal force.

IV. CONCLUSIONS

The high draft cotton specimen examined differs from the low draft cotton specimen in fiber shape, surface character, uniformity and friction coefficient. The high draft fiber is a flatter ribbon than the low draft one, with a high degree of twist at the convolution. The fiber surface, examined by electron replica, stereoscopic, and scanning microscopy, was smoother in the between-convolution zones. The scanning electron microscope and electron stereo-microscopy proved to be valuable aids in examining fiber shapes and surfaces.

The frictional data plots of the high draft cottons exhibited very high peaks, some completely off scale. These peaks are indicative of extreme fiber irregularity or damage and correlate with the accentuated appearance of the convolutions. The fiber irregularity is pointed out by the scatter of the distribution of the values of kinetic (μ_k) and static (μ_s) frictional coefficients of the high draft cotton fibers. These values are in turn affected by the high peaks, just discussed, which contribute to irregularity of the measured coefficients. The low draft cotton, on the other hand, displayed fewer high peaks in the frictional curves and more typical distribution of measured values. The differences in frictional coefficients obtained are principally related to the differences in shape outlined, to the surface differences (to a smaller degree), and to the large degree of nonuniformity of the high draft fibers.

Frictional forces between cotton and other fibers such as glass and nylon, obtained at normal forces in the range 2 to 20 mg, give values of frictional coefficients not greatly different than those for cotton on

cotton. These coefficients increase as the normal force decreases. However, instrumental difficulties at low normal forces, still prevent high confidence in the absolute value of these coefficients. Vibration of the instrument and recorder zero determination may contribute to considerable error on occasion. The sensitivity of the instrument is such, however, that repeatability of measurements on a single fiber are excellent and the character of the frictional plot is intrinsic to both the material and the particular fiber.

Utilizing the instrument it is possible to pull an unsupported fiber across a fiber fixed to friction recording instrument and to obtain frictional forces greater than one milligram, in the absence of any applied normal force other than the weight of the fiber (a few micrograms). Shape factors due to convolutions in cotton and crimp in nylon are readily visible. Smooth fiber surfaces such as nylon on glass give many closely spaced peaks whereas cotton and nylon crimps give saw-tooth peaks usually relatively quite far apart.

The servotorque dynomometer served as a very sensitive method of measuring the removal forces of a single fiber from card sliver and from roving. Forces of the magnitude of approximately 12 and 7 milligrams respectively were measured. The reduction between the two stages is ascribable principally to fiber parallelization. The value of 7 mg indicates that the equivalent of approximately seven fibers are in contact with each fiber in the latter phase. In the first phase, lack of parallelization contributes to entanglement and multiple fiber extraction.

Further studies of frictional curve character are expected to give excellent interpretability of the frictional phenomena observed and of changes in fibers occurring in textile processing.

V. PROGRAM FOR THE NEXT INTERVAL

Investigations of fiber topography versus frictional data will be continued and more extensive research in the low normal force region will be carried out. Effects of processing of the cotton from the carding stage to the yarn will be examined for both a Pima cotton and the Standard Bale (Empire WR). Parallel work on microscopy, x-ray diffraction, and infrared spectroscopy will be continued to the degree found necessary or desirable. Data on four thesis initiated in January, covering these general areas, will be available for the last two Semi Annual Reports. These will cover extensively friction at low normal forces, changes in friction occurring as a result of cotton processing through manufacturing, and additional work on infrared studies of cotton and other fibers.

VI. PERSONNEL

The principal individuals employed on this research and the area of their interest are listed below.

Individual	Title	Area of Research
Richard B. Belser	Research Associate Professor	Project Director
James L. Taylor	A. French Textile School, Director	Associate Project Director
John L. Brown	Director, Analytical Instrumentation Laboratories	Optical and Electron Microscopy
James L. Hubbard	Assistant Research Physicist	Optical and Electron Microscopy
James A. Knight	Research Professor, Chemistry, Head, Radioisotopes Laboratory	Infrared Spectroscopy
J. Conrad Meaders	Assistant Research Scientist (Physics)	Friction Appratus
Kenneth W. Stephens	Student Assistant (Physics)	Infrared Spectroscopy
R. A. Young	Diffraction Laboratories Director	X-ray Diffraction
Harry W. Ellis	Student Assistant (Physics)	X-ray Diffraction
Donald H. Gunther, Jr.	Graduate Assistant (Textile School)	Frictional Properties of Cotton Fibers at Low Normal Forces
Edwin D. Cromer	Graduate Assistant (Textile School)	Effects of Processing on the Properties of Empire WR Cotton
H. Lamar Hicks	Graduate Assistant (Textile School)	Infrared Investigations of Cotton Fibers
Billy R. Livesay	Research Physicist	Fiber Friction and Tensile Properties

Individual	Title	Area of Research
Hong Ki Chin	Graduate Assistant (Textile School)	Fiber Friction
Chin Taik Kwon	Graduate Assistant (Textile and Chemical Engineering)	Fiber Friction and Tensile Properties
Larry B. Whitworth	Graduate Assistant (Textile School)	Effects of Processing on Properties of Pima Cotton Fibers in a Manufacturing Plant

In general, all work was performed on a part-time basis except that of Mr. Meaders who was employed on a full time basis to replace Mr. Dozier in order to give continuity to the fiber friction measurement program.

LIST OF REFERENCES

1. R. B. Belser and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Reports Nos. 1, 2, and 3, USDA-SURDD Grant No. 12-14-100-7661(2), August 1965, February 1966, August 1966.

2. D. L. House, "Optical and Electron Microscopy of Cotton Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

3. J. P. Bryant, "An Investigation of the Factors Which Influence the Frictional Properties of Textile Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

4. A. M. Goldfarb, "Cotton Fiber Properties as Affected by Ginning," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

5. H. R. Levy, "The Effects of Opening, Cleaning, Picking, and Carding on the Physical Properties of Empire WR Cotton Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

6. W. E. Kirkland, "An Investigation of Techniques to Determine Infrared Spectra of Textile Fibers," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, 1966.

7. Donald H. Gunther, Jr., "An Evaluation of Fiber Friction at Low Normal Forces," Thesis for the M.S. degree in Textile Engineering, Georgia Institute of Technology, (December 1967).

8. E. D. Cromer, "Change in the Physical Properties of Empire WR Cotton Fibers after Drawing, Roving, and Spinning," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, (December 1967).

9. Larry B. Whitworth, "Alteration of Physical Properties of Long Staple Cotton by Combing, Drawing, and Roving," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, (September 1967).

10. H. Lamar Hicks, "Evaluation of Cotton Crystallinity by Infrared Fiber Press and Deuteration Methods," Thesis for the M.S. degree in Textiles, Georgia Institute of Technology, (December 1967).

11. F. Scardino, "The Influence of Fiber Geometry on the Cohesion of Cotton Card Slivers," Notes on Research, No. 162, Textile Research Institute, Princeton, N. J., (January - February 1967).

A-843

REPORT NO. 5 (SEMIANNUAL REPORT)
GEORGIA TECH PROJECT NO. A-843

1 FEBRUARY 1967 TO 1 AUGUST 1967

FRICTIONAL PROPERTIES OF COTTON FIBERS

By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

PREPARED FOR
UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
NEW ORLEANS, LOUISIANA

1 AUGUST 1967

Engineering Experiment Station
and
School of Textile Engineering
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

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Grant No. 12-14-100-7661(72)
United States Department of Agriculture

1 February 1967 to 1 August 1967

Placed By
United States Department of Agriculture
Agricultural Research Service
Southern Utilization Research and Development Division
New Orleans, Louisiana

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ABSTRACT

Extensive measurements of the coefficients of friction of cotton and nylon in the normal force range 1 to 20 mg have proven the measurement method accurate. Anomalous earlier data for cotton were found to be related to the instrument arm's bouncing below normal forces of 7 mg. This was induced by the shape factor in cotton and was not present for cylindrical nylon fibers. Accurate measurements for the latter extended down to a normal force of 1 mg. Coefficients of friction of cotton increase as normal force decreases from 20 mg to 7 mg, registering μ_s values of 0.38 and 0.72 respectively and μ_k values of 0.20 and 0.26. Values for nylon increased in the expected manner as the force was decreased from 20 to 1 mg, registering values for μ_s of 0.72 and 1.64 respectively and 0.36 and 0.66 for μ_k.

Measurements of the μ_s and μ_k values of cotton and nylon at fiber traversing velocities of 0.135, 0.270, and 0.540 inches/minute at 5 and 10 mg normal forces indicated increases of 20 to 50% in μ_s but little for μ_k.

Measurements of the friction and work to remove a single cotton fiber from an unrestricted card and roving specimen indicated respective forces required of 5 to 113 mg and 6 to 70 mg. Average energies of withdrawal were 26 and 13 ergs respectively. The force according to measurements acted strongly over 1/2 to 2/3 of the mean fiber length. In general, values for roving specimen were about 2/3 the values for card specimen. Measurements of forces required for drawing single cotton or nylon fibers across a similar fiber with only the forces of cohesion and fiber weight acting as normal forces gave resisting forces up to about 0.75 mg. Hence, the μ_s value for a 5 microgram fiber would be equivalent to about 150. Since the friction coefficients increase at a rapid rate as the normal forces are reduced and as interfiber velocity increases, it is evident that in usual textile processing stages friction coefficients are at a high level. The great accentuation of these parameters as normal force decreases indicates the need for extensive measurements of the friction coefficients of fibers at low normal forces in order to delineate fiber behavior.

Fiber shape asperities and fiber damage zones are responsible for

many high sticks among cotton fibers and appear to establish essentially the μ_s value as reported in this research. Processing of fibers in textile machinery results in straightening, twisting, swaging, breaking, or other damage to the fibers. The straightening tends to reduce the friction but the damage greatly increases static friction. The static friction in turn is the agent causing fibers in masses to travel as small fiber bundles with slips occurring only at the zones of lower friction. It appears that vibration applied to the fibers may reduce this tendency.

Coefficients of static and kinetic friction at 10 mg normal force were measured for Acrilan, Dacron, Orlon, Viscose, and Dynel in addition to cotton and nylon giving μ_s values of 0.49, 0.51, 0.53, 0.55, and 0.67 and μ_k values of 0.17, 0.24, 0.27, 0.34, and 0.36, respectively. These values may be compared to cotton at 0.54 and 0.26 and nylon at 0.80 and 0.45 at the same normal force.

Investigations of crystallinity determination of cotton and other cellulose fibers were made using fiber press specimens, fiber deuteration techniques, and infrared spectroscopy. Deuteration substitutes the OD bond for the OH bond principally in the amorphous phase. Relative heights of the 3μ OH band and the 4μ OD band are used for determination of fiber crystallinity. Average measurements indicated a crystallinity of about 0.76 for Empire WR cotton.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influences of crimp, convolution, cross section, surface texture, and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

II. INTRODUCTION

The development of the apparatus to measure friction between fibers at low and at very low normal forces have been described in previous reports[1,2] and in one thesis.[3] A second thesis describing the further development of the very low normal force instrument will be completed in December 1967.[4]

During the period covered by this report, extensive further studies of the effects of the shape factor on the cotton fiber have been carried out. The results of these studies, in turn, have been used to interpret frictional data obtained in two theses being prepared concurrently on the effects of processing through the stages of combing, drawing, roving, and spinning.[5,6] These latter are to be completed by December 1967.

Concurrently, other investigations by microscopy, X-ray diffraction, and infrared spectroscopy have been pursued. Considerable progress in the determination of the crystallinity of cotton by deuteration of the cotton and infrared absorbtion measurements of a band at 4.0 microns has been made.[7]

These various investigations have contributed toward the solution of the frictional behavior of cotton but, in particular, the friction measurements at very low normal forces have given much insight into the behavior of fiber assemblies in textile processing and the influence of the shape factor on the behavior of the cotton fiber. Evidences of this behavior and a preliminary explanation of probable fiber actions in processed fiber assemblies are presented.

III. EXPERIMENTAL WORK

A. GENERAL

Studies of the microstructure of cotton have continued and extensive studies of frictional effects for various fibers at low normal forces have been completed. Two theses on the effects of textile processing on the physical properties of cotton fibers are nearing completion and extensive data from these are becoming available for examination and discussion. Progress on infrared investigation of fibers has led to the development of a deuteration technique for examining the crystallinity of the cellulose within the fiber. The method is reported herein.

B. APPARATUS AND PROCEDURES

The apparatus and procedures used, with the exception of the deuteration equipment, have been given extensive coverage in previous Reports No. 1-4 of this series.[8] The deuteration apparatus is described in Section III, E, 2.

C. INVESTIGATIONS OF FIBERS BY OPTICAL AND ELECTRON MICROSCOPY

1. General

Microscopy has been used during the period of this report principally to examine fiber shape phenomena. Experiments were specifically aimed at characterizing features which were responsible for large frictional sticks and for observing changes in fibers occurring as a result of processing through the stages of carding, combing, roving, and

spinning. Damage was further examined by the Congo Red technique[9] aided by optical microscopy. Changes in surface texture as a result of processing were examined by replication and electron microscopy of the replicas.

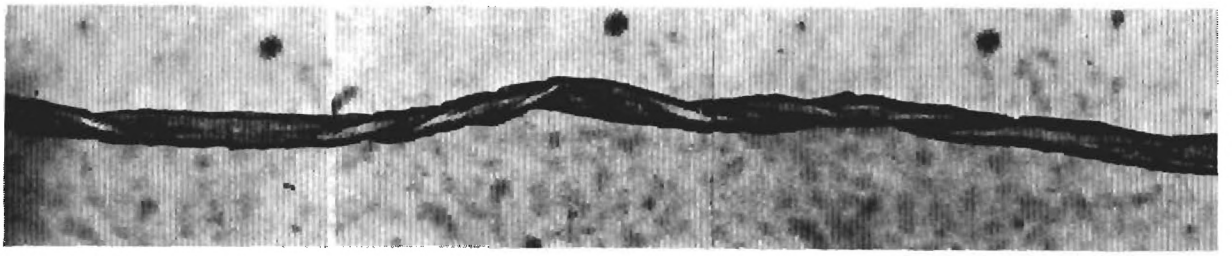
2. Optical Micrographs

a. Unusual Fiber Configurations

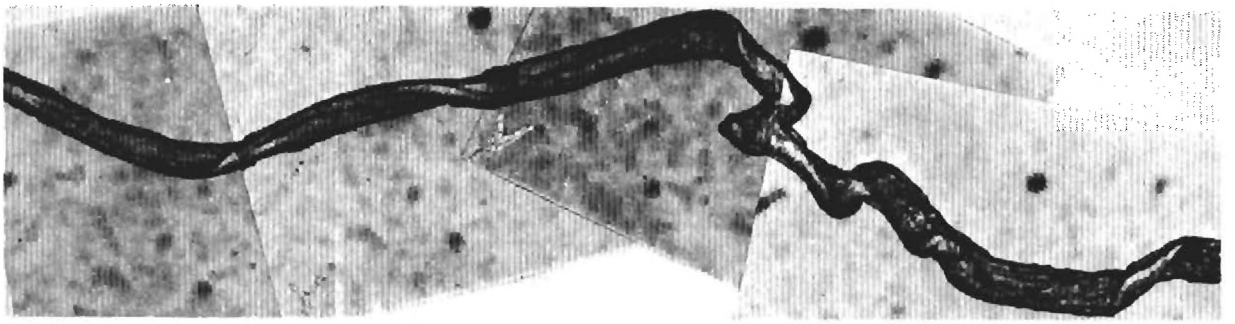
During frictional studies, occasional fibers have shown tremendous shape irregularity resulting in large excursions of the fiber frictional analog curves. A paper by H. J. Denham,[10] written in 1923, presents an excellent coverage of some of the fiber features of the type observed. Figure 1 exhibits several portions of such a fiber at about 225x. The irregularity of the fiber is noteworthy and it may be expected that the frictional analog of this fiber would display many large peaks such as that in Figure 2 exhibiting the accompanying fiber feature which caused the major peak.

b. Effects of Fiber Processing

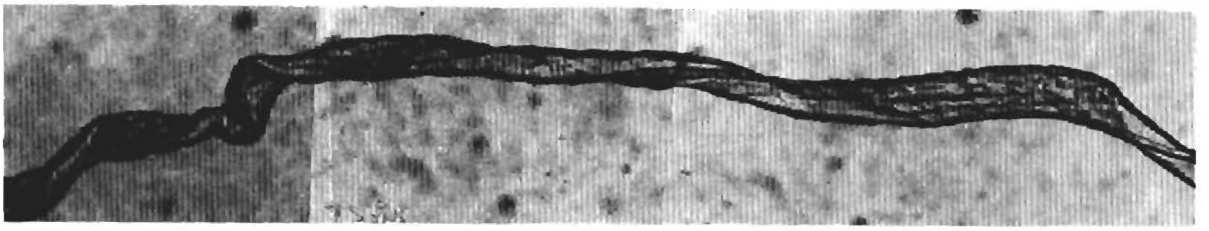
Among a number of fibers examined after the various processing stages, very sharp frictional peaks appear indicating that some feature of the fiber may have been accentuated or that a portion of the fiber was damaged. Figures 3 and 4 exhibit typical fibers from the Pima-Menoufi raw cotton stock entering the mill of a commercial manufacturer. Fibers which were damaged in subsequent processing are exhibited in Figure 4. The fiber in Figure 4(b) is from the comber noil rejected by the combing process. It will be observed that this fiber has been twisted; and for



A



B



C

Figure 1. Portions of a cotton fiber exhibiting types of irregularities observed in many fibers.

Note: This fiber has been subjected to processing up through the roving stage and appears to have been damaged by twisting and swaging.

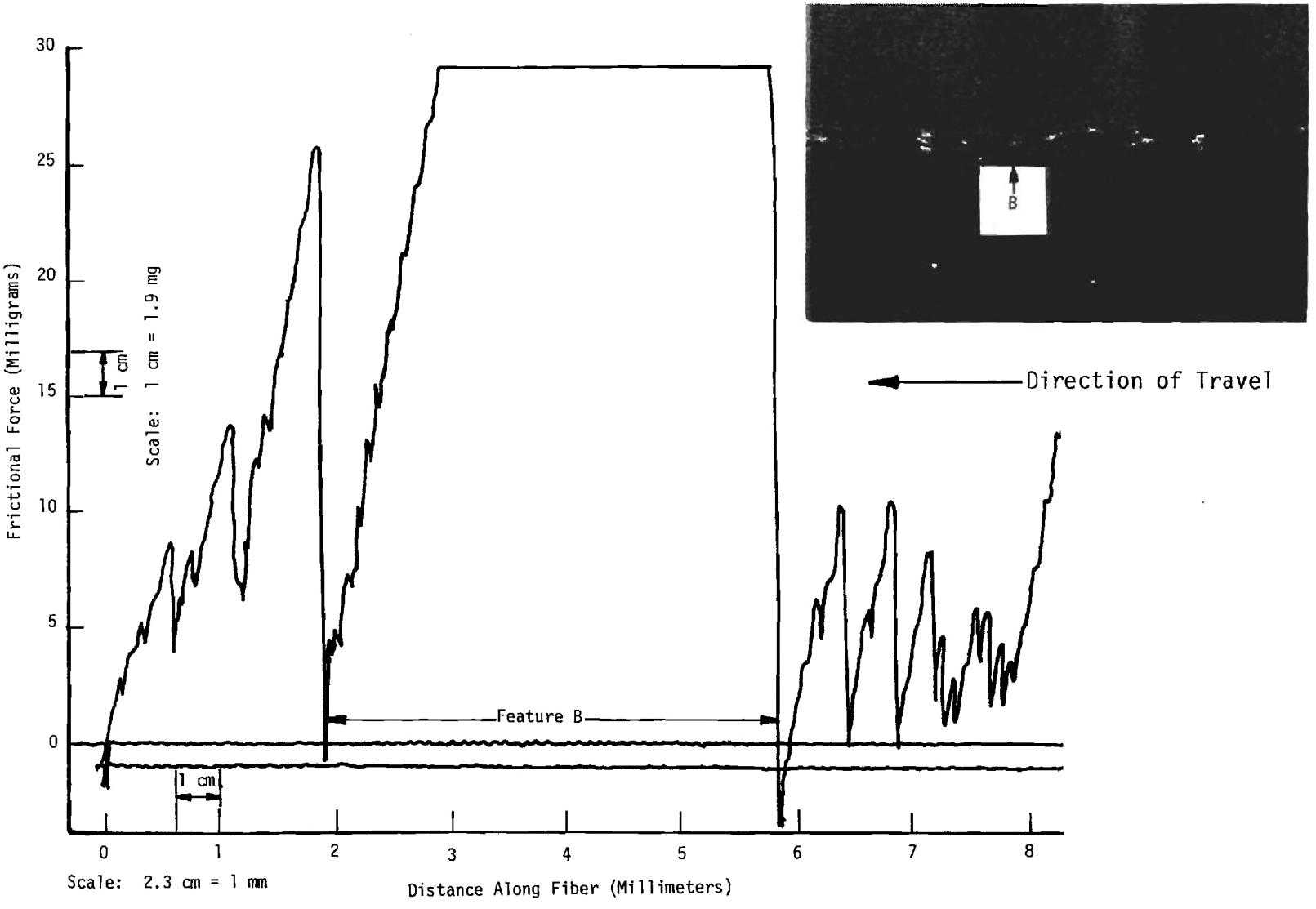
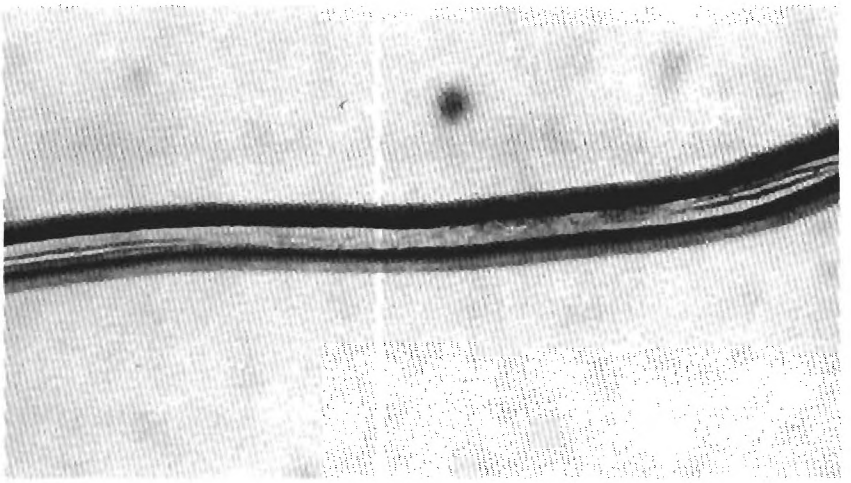
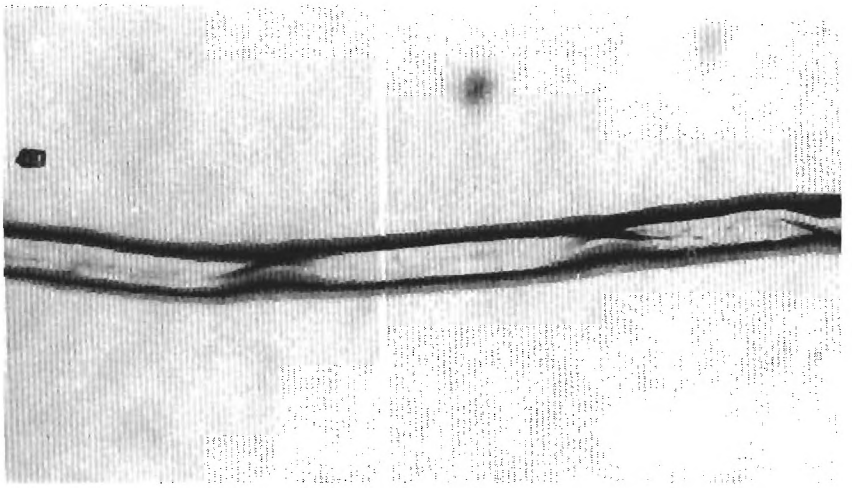


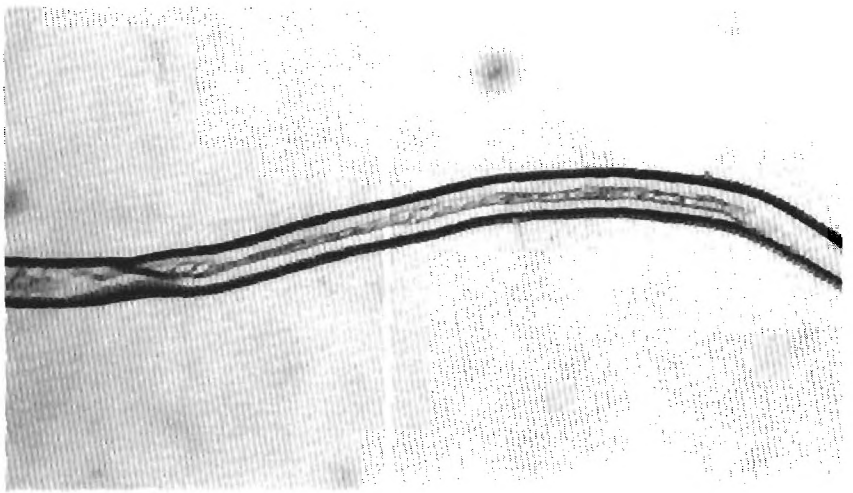
Figure 2. Frictional graph and micrograph of high-draft cotton fiber displaying large frictional peak and feature responsible for it.



A. PIMA-GRADE #2
(230X)



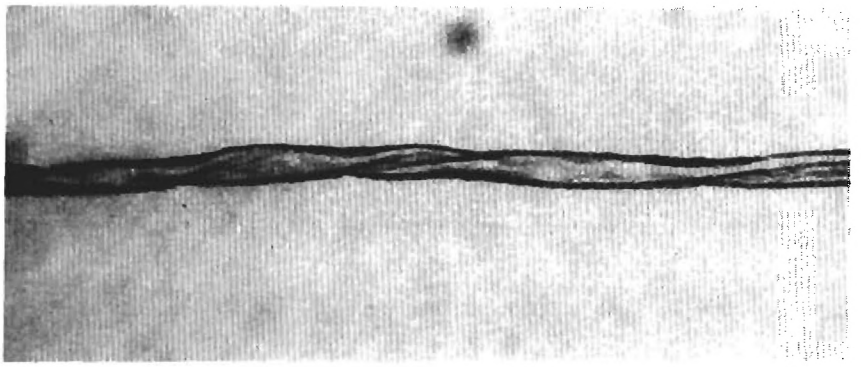
B. EGYPTIAN MENOUFI
(230X)



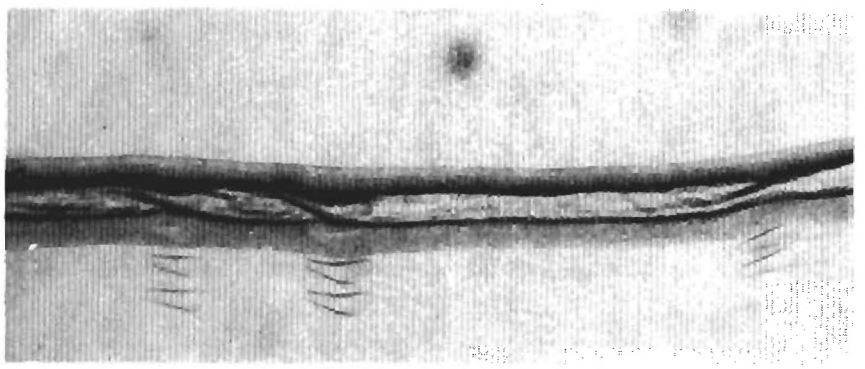
C. EGYPTIAN MENOUFI
(230X)

Figure 3. Typical cotton fibers from raw stock input of commercial manufacturer.

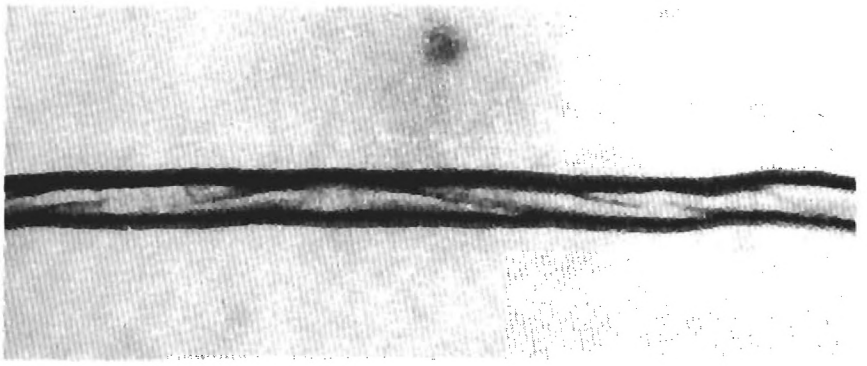
A. LAP (230X)



B. COMBER NOIL (230X)



C. BREAKER DRAWING (230X)



D. FINISHER DRAWING (230X)

Figure 4. Cotton fibers from various processing stages of commercial manufacturer exhibiting typical damage effects observed.

a fiber from the breaker drawing stage [Figure 4(c)], one convolution has apparently been forced closer to a second one. Twisting and convolution distortion appear to be a common result of the processing normally incurred.

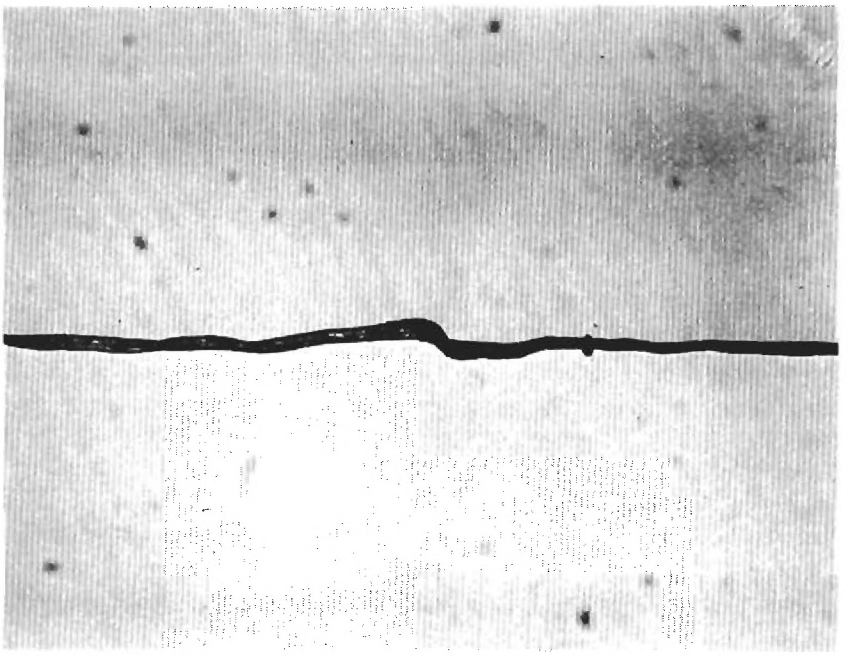
Denham, cited previously, noted that certain convolutions or other features of the cotton fiber are movable under the influence of humidity changes. The above Figures indicate what appears to be movement of a convolution under a swaging action applied to the fiber as a result of processing. Hence, portions of the fiber flow viscoelastically to a new position. Features of the nature of those exhibited tend to snag in the present frictional apparatus giving very high peaks and what appears to be a somewhat biased frictional coefficient. Other examples of this will be discussed in Section D, 3 of this Chapter.

A fiber was examined before and during application of a Congo Red solution (20% NaOH) with a stereomicroscope. The fiber almost immediately exhibited a swelling effect and a general smoothing of the fiber. Peak-to-valley amplitude was greatly reduced. Before and after micrographs are shown in Figure 5. Section D, 4, b of this chapter discusses changes occurring in the frictional behavior of the fiber.

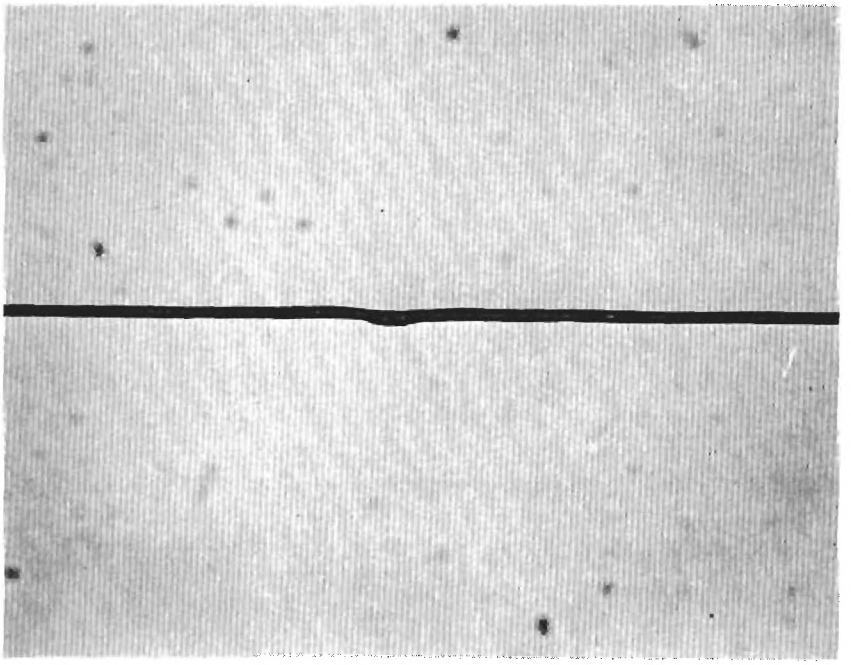
3. Electron Micrographs

Although extensive electron micrographs of fiber surfaces after processing have been made, these are still undergoing analysis and will be discussed in the Final Report.

Some work has been performed on adapting the Acton electron microprobe analyzer into a scanning electron microscope. Some micrographs of fibers were obtained. However, resolution was relatively poor due to the large



A. BEFORE (100X)



B. AFTER (100X)

Figure 5. Cotton fiber before and after treatment with Congo Red solution (up to 20% NaOH).

electron beam diameter of the microprobe, approximately one micron.

An effort to procure funds for a scanning electron microscope is now underway.

D. FRICTIONAL MEASUREMENTS

1. General

Frictional measurements have been directed principally toward examining the effects of the shape factor of the cotton fiber on the character of the frictional plot and upon the coefficients of friction derived therefrom. Although preliminary data have been obtained from the gravity loaded frictional beam at 20 mg, more extensive data have been obtained with the electromagnetically loaded beam at normal forces as low as 1 mg. In addition, the withdrawal force measurements of single fibers from card and sliver specimens have been made. Although a preliminary discussion of these data was given in the last report,[8] they are more extensively reported here since the information obtained has a bearing on the processing behavior of fibers now becoming more clearly illuminated as a result of the recent frictional measurements at low normal forces.

2. Forces to Withdraw Cotton Fibers from Card Sliver and From Roving*

A Servo Torque-Balance Reaction Dynamometer,** its amplifier, and an x-y recorder (Moseley Model 7000A), in conjunction with a worm

*Repeated in part from Semiannual Report No. 4, 1 February 1967.

**Described in Semiannual Report No. 3 of this Grant.

driven fiber holder, were utilized to perform measurements of the forces to withdraw a single cotton fiber from a fiber bundle of card sliver or of roving.

The card sliver (4.2 gram/yd or 64.9 grains/yd) was mounted at the clamp, which was movable at the speed of 2.38×10^{-2} cm/sec, and a single fiber at the other end of the sliver was clamped by a pin vise using a vacuum technique. The pin vise was attached to the dynamometer lever arm. The fiber withdrawal force was recorded during the withdrawal of the card sliver by the motor driven worm gear. Five measurements were made at four different places in the sliver. This action was repeated for the roving (0.3 gram/yd or 4.5 grains/yd).

Twenty specimens each were extracted from the respective specimens of card sliver and roving. The values obtained are listed in Tables 1 and 2. Typical curves showing some of the types of behavior observed are illustrated in Figures 6, 7, 8, and 9.

From examination of the tables, it will be immediately noted that a large range of maximum forces exerted are obtained. For the card specimens this amounts to a range of 14 mg to 113 mg. It should be noted specifically that these numbers represent forces where no normal force other than cohesion is exerted between the fibers of the bundle. Furthermore, these forces are much greater than the usual static or kinetic frictional force even at 20 mg normal force between fibers. It is quite apparent that we are, in reality, dealing with fiber bundles in most instances and static frictional effects of high magnitudes.

The average force during the time of withdrawal from the card specimens, which means the average force exerted during the principal

TABLE 1

Force and Energy Data Obtained on Extracting Single Fibers from Cotton Card Sliver Specimen

Specimen	Maximum Force mg	Average Force mg	Total Energy ergs/cm	Distance cm	ergs/cm
A-1	82.5	20.5	35.0	1.70	20.5
A-2	67.6	23.9	75.6	3.15 (entangled)	23.9
A-3	38.4	9.5	34.0	3.58	9.5
A-4	24.3	5.6	14.8	2.64	5.6
A-5	22.2	8.7	8.0	0.92	8.7
B-1	5.1	2.3	5.3	2.30	2.3
B-2	27.6	7.9	9.6	1.21	7.9
B-3	21.0	4.5	7.1	1.58	4.5
B-4	13.8	3.3	6.0	1.82	3.3
B-5	19.3	5.0	9.9	1.98	5.0
C-1	40.2	10.0	15.8	1.58	10.0
C-2	50.0	16.1	18.1	1.12	16.1
C-3	22.6	6.2	13.9	2.24	6.2
C-4	20.7	3.2	3.6	1.12	3.2
C-5	42.4	12.4	10.2	0.83	12.4
D-1	46.6	10.4	31.2	3.00	10.4
D-2	111.7	29.8	74.8	2.50	29.8
D-3	37.8	12.4	33.0	2.67	12.4
D-4	113.0	30.2	79.8	2.64	30.2
D-5	36.3	10.4	23.7	2.28	10.4
Averages	42.2	11.6	25.5	2.21	11.6

TABLE 2

Force and Energy Data Obtained on Extracting Single Fibers from Cotton Roving Specimen

Specimen	Maximum Force mg	Average Force mg	Total Energy ergs/cm	Distance cm	ergs/cm
A-1	58.0	25.1	73.2	2.94	25.1
A-2	8.6	1.0	1.9	1.90	1.0
A-3	40.0	10.8	23.6	2.18	10.8
A-4	57.3	21.6	10.1	0.47	21.6
A-5	40.5	7.1	7.25	1.02	7.3
B-1	28.5	3.8	4.1	1.08	3.8
B-2	29.2	7.7	14.9	1.94	7.7
B-3	11.6	3.5	2.7	0.77	3.5
B-4	17.0	6.1	4.3	0.71	6.1
B-5	7.9	2.4	1.5	0.64	2.4
C-1	21.8	4.1	6.9	1.69	4.1
C-2	23.2	4.0	8.2	2.05	4.0
C-3	15.3	4.8	8.6	1.79	4.8
C-4	15.2	4.0	2.5	0.63	4.0
D-1	6.7	1.8	2.8	1.56	1.8
D-2	38.9	8.1	12.0	1.48	8.1
D-3	8.4	2.0	2.5	1.25	2.0
D-4	12.9	4.5	8.2	1.82	4.5
D-5	70.3	21.0	44.0	2.09	21.0
Averages	26.9	7.5	12.6	1.68	7.5
Comparison of Card and Sliver Average Data					
Average Card Sliver Values	42.2	11.6	25.5	2.21	11.6
Average Roving Value	26.9	7.5	12.6	1.68	7.5
Ratios Card/Sliver Values	1.57	1.55	2.03	1.31	1.55
Percentages ($\frac{\text{sliver}}{\text{card}}$ x 100)	64	65	49	77	65

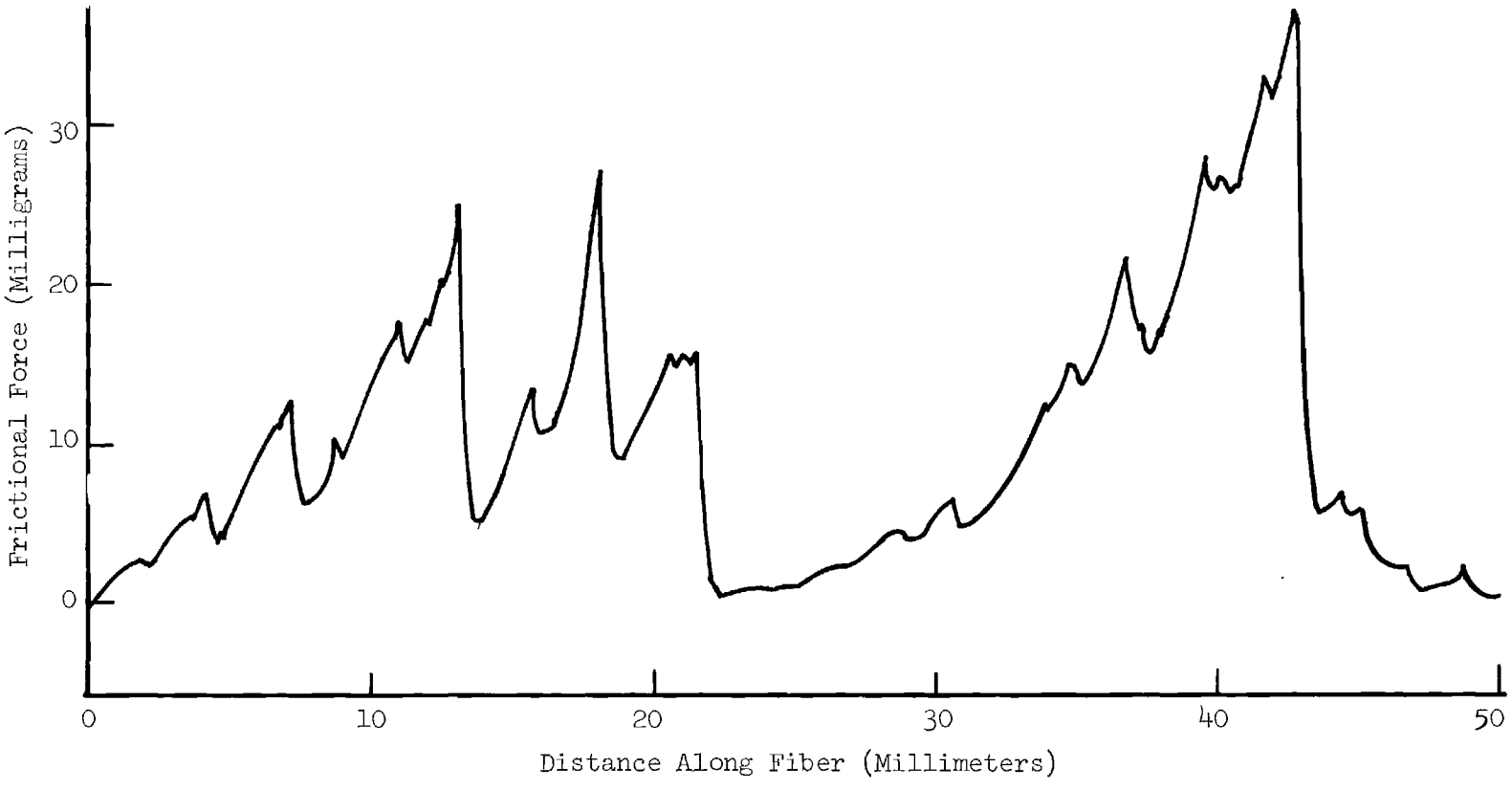


Figure 6. Analog plot of force required to withdraw a single cotton fiber from card specimen (A-3).

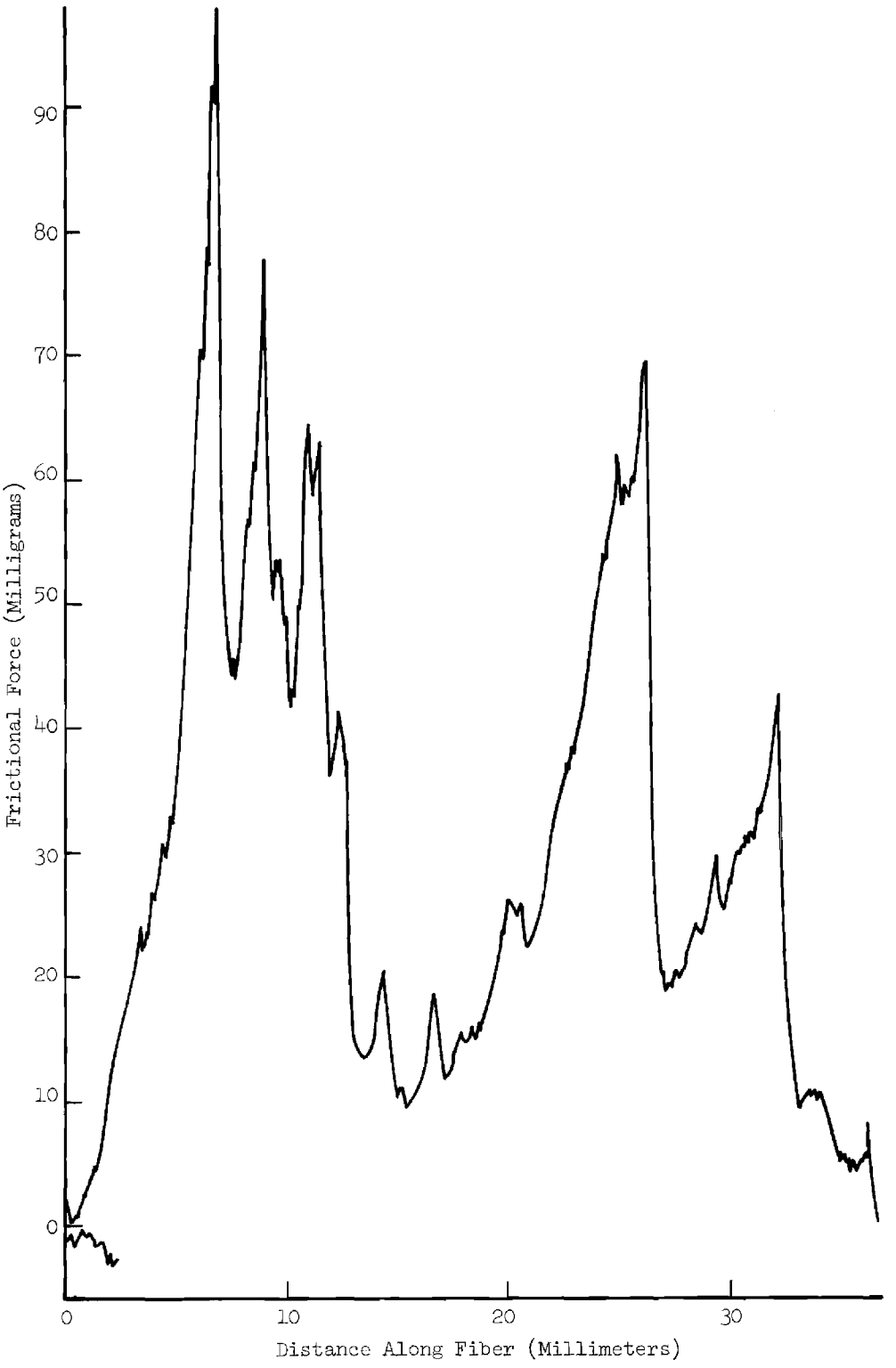


Figure 7. Analog plot of force required to withdraw a single cotton fiber from card specimen (D-4).

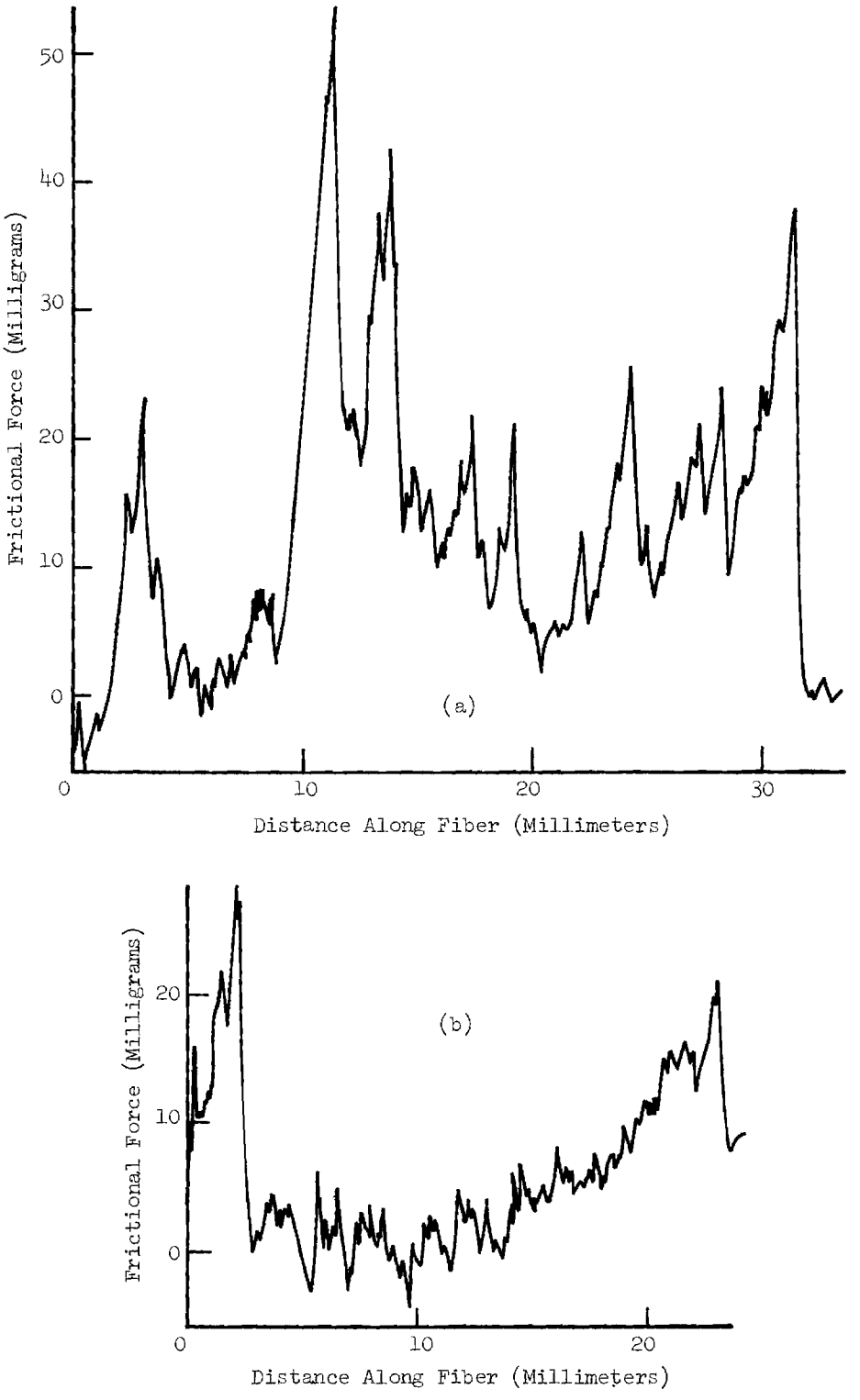


Figure 8. Analog plots of forces required to withdraw a single fiber from cotton roving (A-3, C-1).

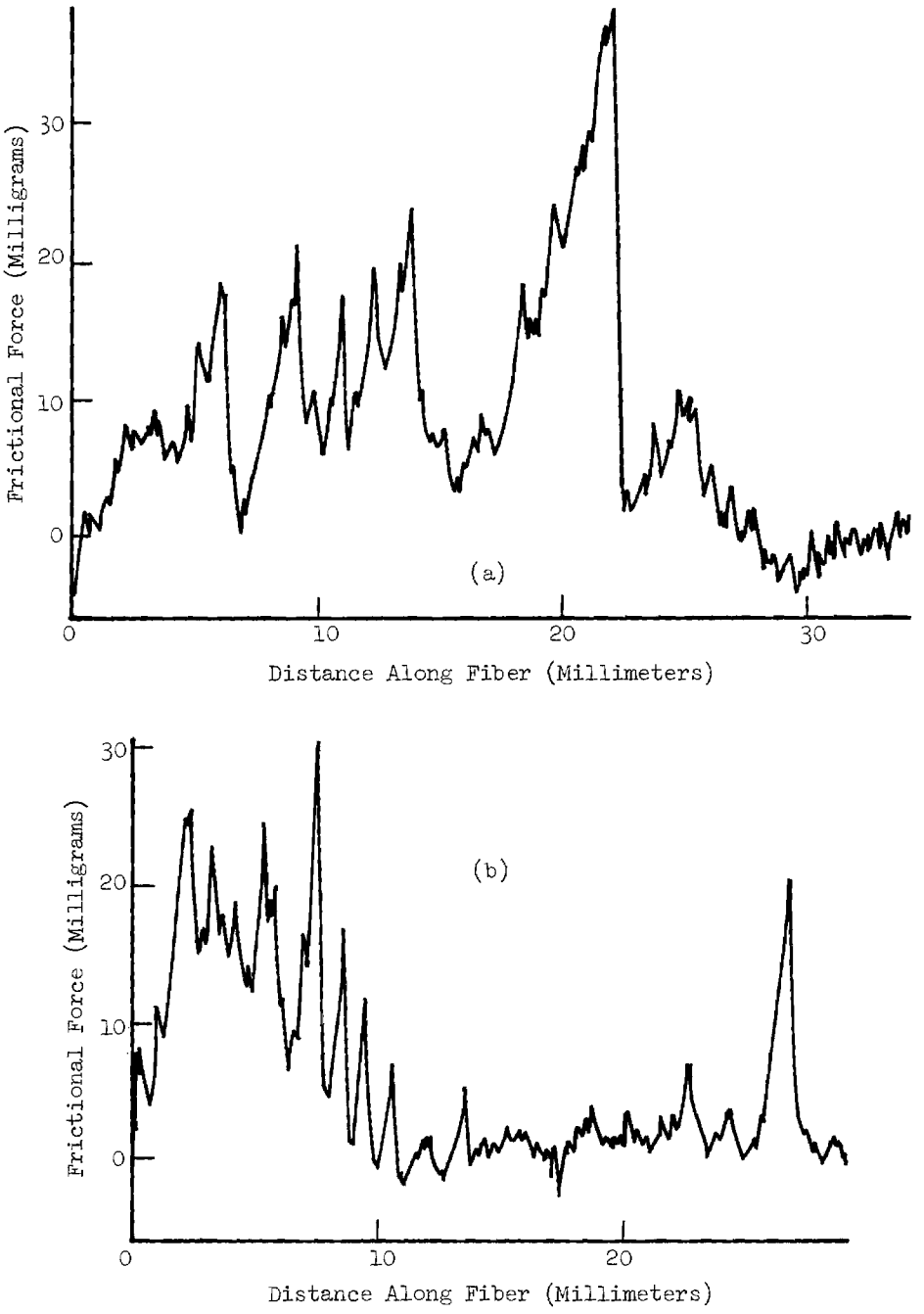


Figure 9. Analog plots of forces required to withdraw a single fiber from cotton roving (B-2, C-2).

contact between the fibers, was 11.6 mg but ranged over the values 2.3 mg to 30.2 mg. These latter numbers also represent essentially the work performed in removing a fiber per cm of its withdrawal since an erg is a dyne-cm or 1/980 gm x 1 cm; hence, essentially a mg cm as an approximation.

It is noteworthy here also that the withdrawal distance ranges from 0.92 cm to 3.58 cm with an average of 2.21 cm.

The measurements for roving gave essentially similar results except that most values were reduced to about 2/3 the values obtained for the fibers from the card specimen. Maximum force ranges were 6.7 mg to 70.3 mg with the average 26.9 mg. The average force ranged from 1.0 mg to 25.1 mg with resulting energy/cm reductions. The withdrawal distance was reduced to 1.68 cm.

The various data are compared at the foot of Table 2. It is quite evident that there is a fairly uniform reduction of the various force, energy, and withdrawal distance parameters in a ratio of approximately 3/2 as the fiber goes from the card to the roving stage. Parallelization of the fiber appears to be the principal factor to which the reduction can be ascribed.

It is also apparent that frictional forces at low normal forces are large; forces are sufficient to drag a series of fibers one over the other for a distance of 1 to 3 cm. The forces are active and large over about 2/3 of a fiber length in the card and 1/2 its length in the roving.

It is evident that in the drawing process, the fiber must move principally in small bundles, slipping over each other at points of lowest stick (or static coefficient of friction). This performance will be discussed more thoroughly in Sections D, 3 and D, 4 subsequently.

3. Friction Measurements at 20 mg Normal Force

Friction measurements with the gravity loaded beam have been continued principally at a normal force of 20 mg. Extensive measurements have been made for cotton specimens extracted from the various processing stages from carding to the yarn. These have included specimens taken from a Pima-Menoufi blend during manufacturing into yarn by a commercial manufacturer. Specimens were taken after combing, drawing, roving, and spinning. Comber noils were also examined.

Specimens of Empire WR cotton have been examined for frictional properties after processing from carding to the yarn with equipment of the A French Textile School. Specimens from the stages of carding, drawing, roving, and spinning were studied. The data obtained in these various measurements are being reported extensively by Whitworth[5] and Cromer[6] in their respective theses.

As shown in Figure 10, from data by Whitworth, only small changes in frictional coefficients resulted from the effects of processing in the Pima-Menoufi cotton blend study. There appeared to be a small reduction in the values of μ_k and μ_s as a result of processing beyond the carding stage. However, the data scatter is such that only the general trend can be established. In reality, the measurements are somewhat biased by the fact that severely damaged fibers (giving very high static peaks) frequently appear to have been eliminated from the study because of the excessively high values of μ_k and μ_s obtained for such fibers. Fibers of this character appear to constitute some 15% of the fibers examined and their inclusion would result in probable increasing values of μ_s and μ_k with processing

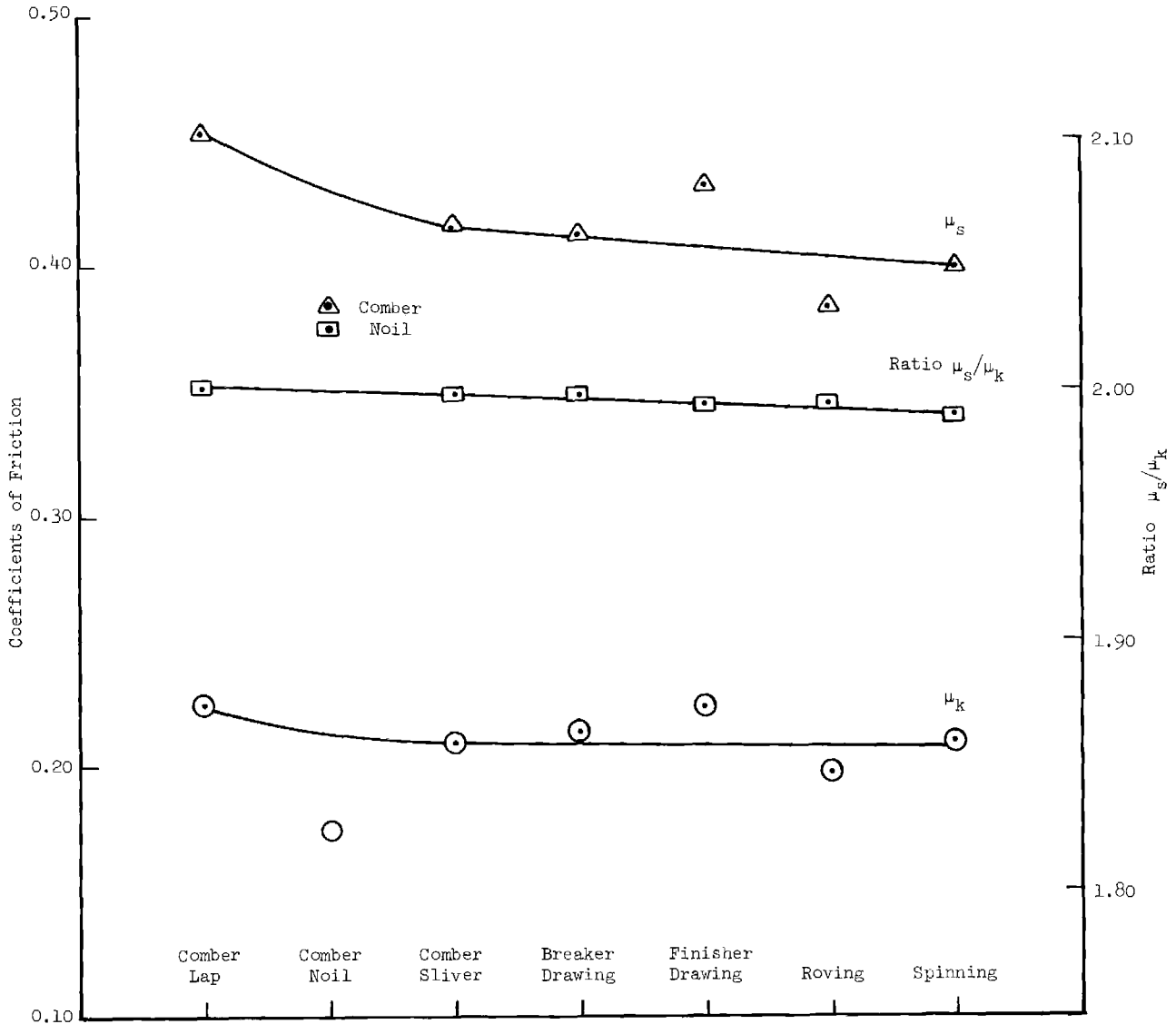


Figure 10. Changes in coefficients of kinetic and static-friction of a Pima-Menoufi blend of cotton as a result of processing in a commercial textile plant.

similar to that observed earlier by Levy.[9] In examining specimens up to carding.

The work of Cromer has not yet been tabulated but will be available for inclusion in the Final Report. He has made an effort to include damaged fiber effects in his frictional data.

Referring back now to Figure 4(c), it was observed by microscopy that a convolution appears to have been swaged along the fiber until a convoluted zone has approached very closely to a second convoluted zone. An example of a flow behavior of this nature has also been found with the frictional apparatus. In Figures 11, 12, and 13 are displayed three frictional data plots made in succession for a single Empire WR cotton fiber taken after the roving stage. Observe the three peaks A, B, and C in succession. Peak A, a very large peak, shrinks with each pass of the fiber, whereas Peaks B and C grow. It is evident that some flow of the shape of the fiber must occur under the pressure exerted at the fiber-to-fiber interface. An estimate of this pressure can be made by assuming the contact area is $(10^{-3}$ in$)^2$ or 10^{-6} sq in. The pressure then becomes $0.020/10^{-6}$ or 20 kilograms per sq in (44 lbs psi). This would be an assumption that the entire widths of 2 fibers are in contact. However, the area might be as small $(10^{-4}$ in$)^2$. Instead of 44 psi, this would mean 4400 psi. This would probably be a sufficient pressure to cause flow in the fiber as observed. Hence, it is probable that pressures of this order of magnitude are exerted at the interface.

On closer observation of a number of friction data curves, similar peak migrations have been observed, and it is apparent that flow and stretching of fibers are occurring on successive frictional passes of a

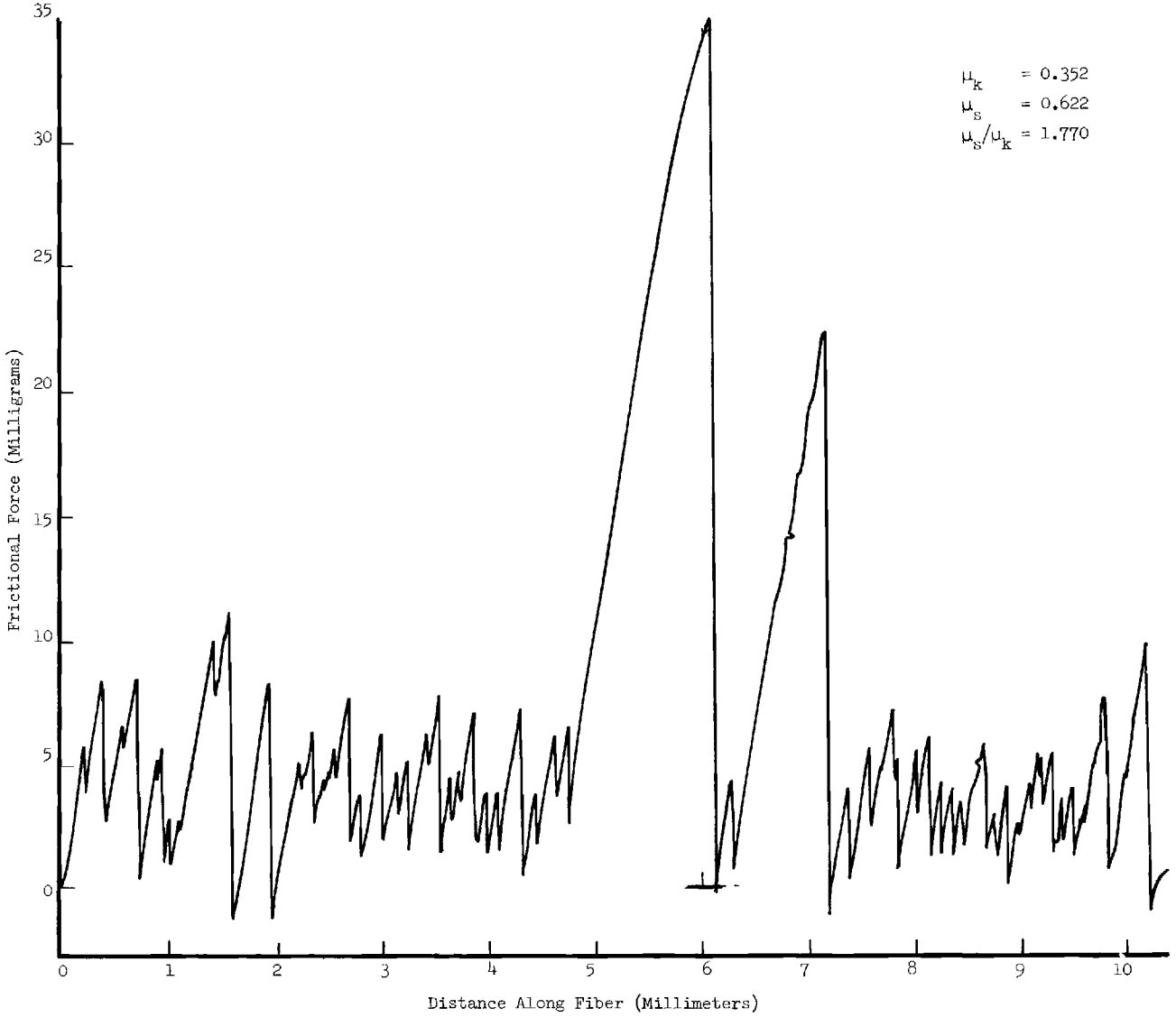


Figure 11. Frictional data plot of Empire WR cotton fiber from roving specimen, 20 mg normal force, first pass in frictional apparatus.

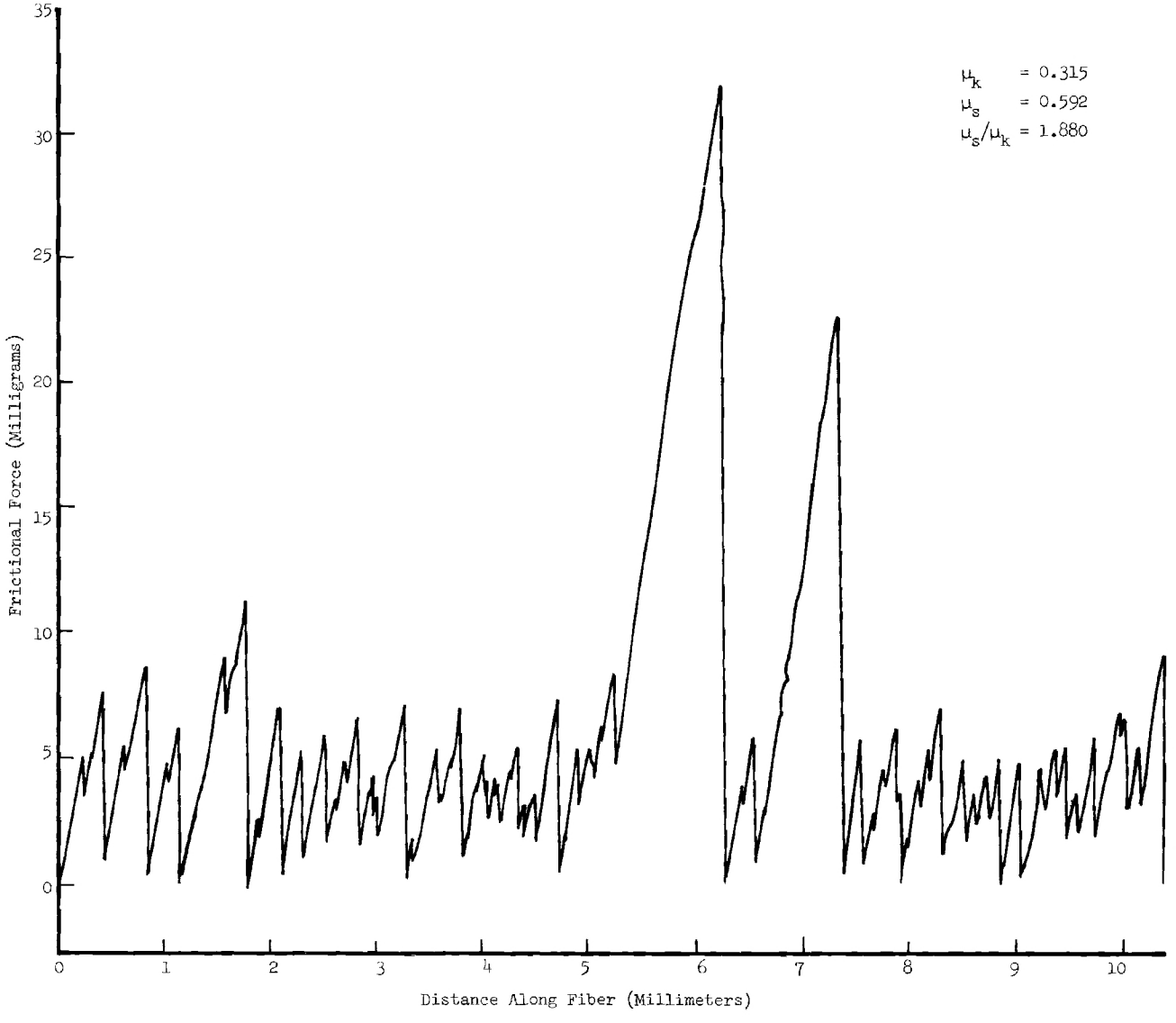


Figure 12. Frictional data plot of Empire WR cotton fiber from roving specimen, 20 mg normal force, second pass in frictional apparatus.

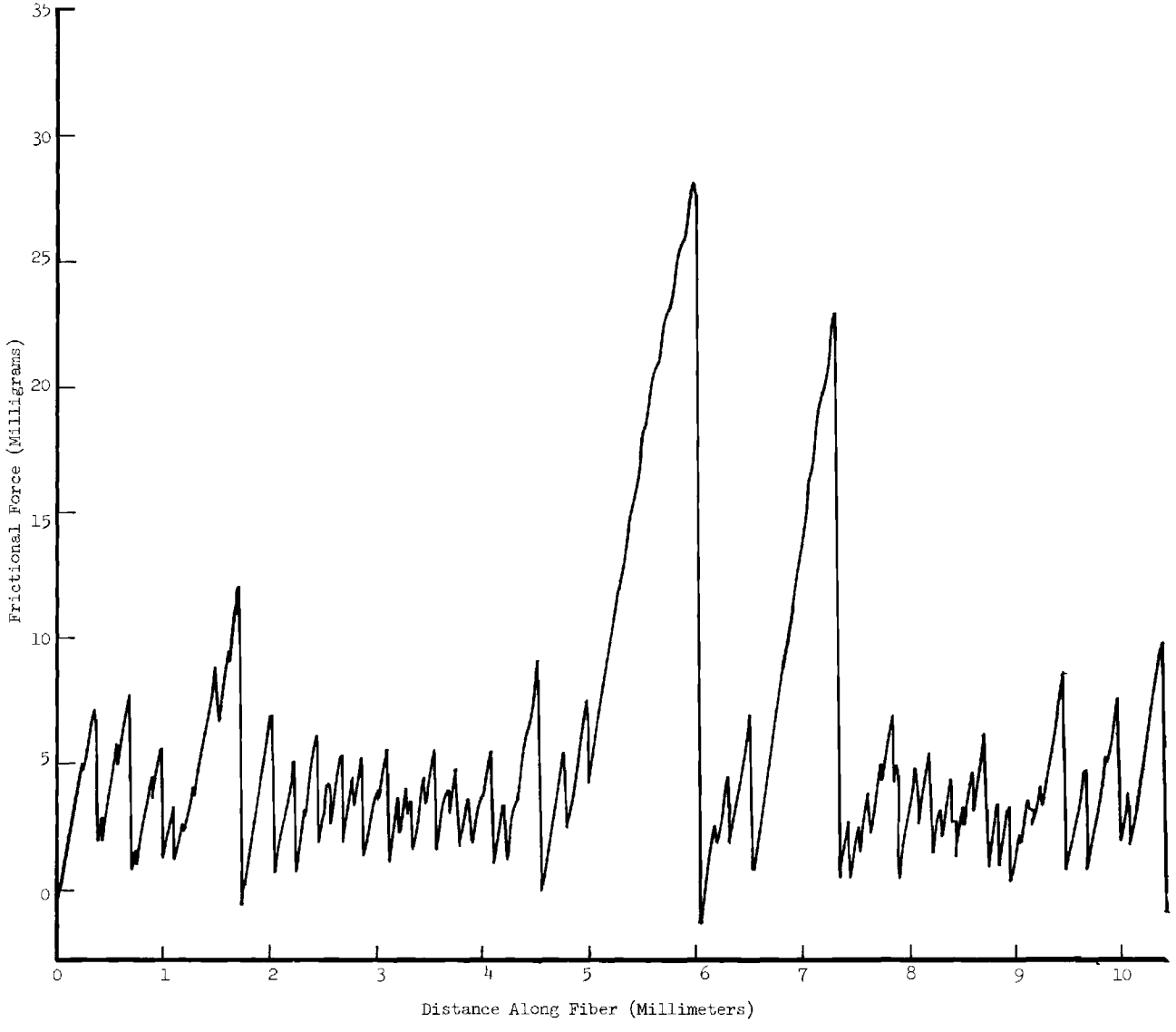


Figure 13. Frictional data plot of Empire WR cotton fiber from roving specimen, 20 mg normal force, third pass in frictional apparatus.

single fiber. This action then leads to the conclusion that some textile processing stages must cause similar behavior in the fibers in more exaggerated form because of the higher forces and velocities commonly employed in these stages. It also suggests that normal forces higher than those employed previously in this work should result in larger changes between data plots for curves run on the same fiber successively.* The possibility also exists that only very small changes as a result of successive passes will be observed at very low normal forces. We are in a position to examine this result in the subsequent Section D, 4.

Another example of fiber shape change was recorded in an experiment in which a cotton fiber was mounted and its frictional data plot obtained. The fiber was then treated with a drop of Congo Red dye solution (containing about 20% of NaOH). After a drying interval of 30 minutes, its frictional curve was rerun. The two data plots are exhibited in Figures 14 and 15. A large change in the character of the curves is observed as the peak heights are markedly reduced; the μ_k value is somewhat reduced. However, the principal changes are observed in the μ_s value and in the ratio of μ_s/μ_k which are reduced from 0.59 and 2.10 to 0.40 and 1.69 respectively.

Observation of the fiber under the stereomicroscope revealed that the fiber became swollen and smoother in appearance after the treatment as shown in Figure 5 preceding; obviously, smaller peak-to-valley heights existed for the convolutions or other asperities of the fiber after the treatment than existed before it. This result is in agreement with that

*A preliminary examination of friction data plots of fibers made at higher normal forces (40 and 60 mg) revealed greater changes between successive plots than at 20 mg normal force.

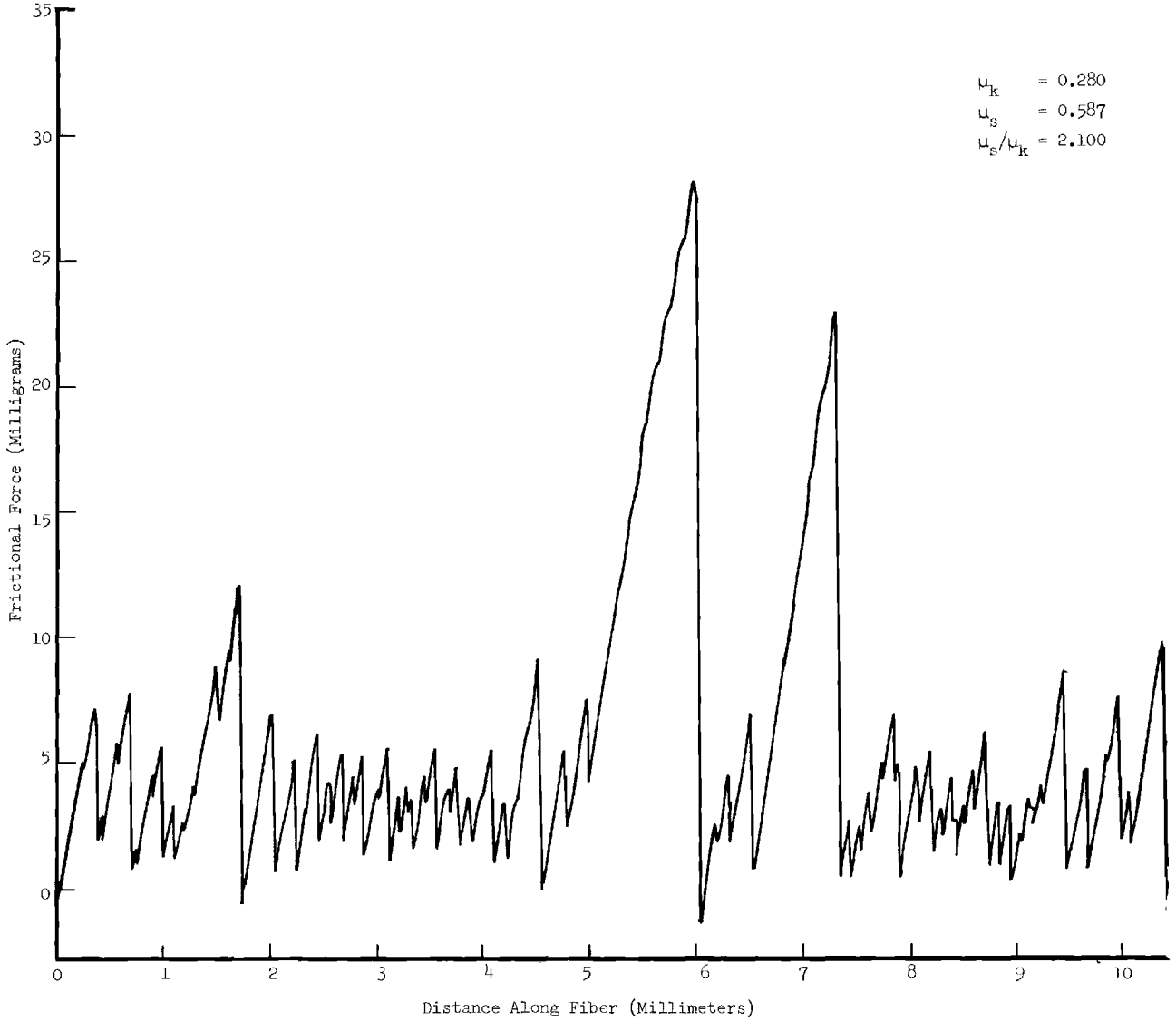


Figure 14. Frictional data plot of Empire WR cotton fiber before treatment with Congo Red dye solution.

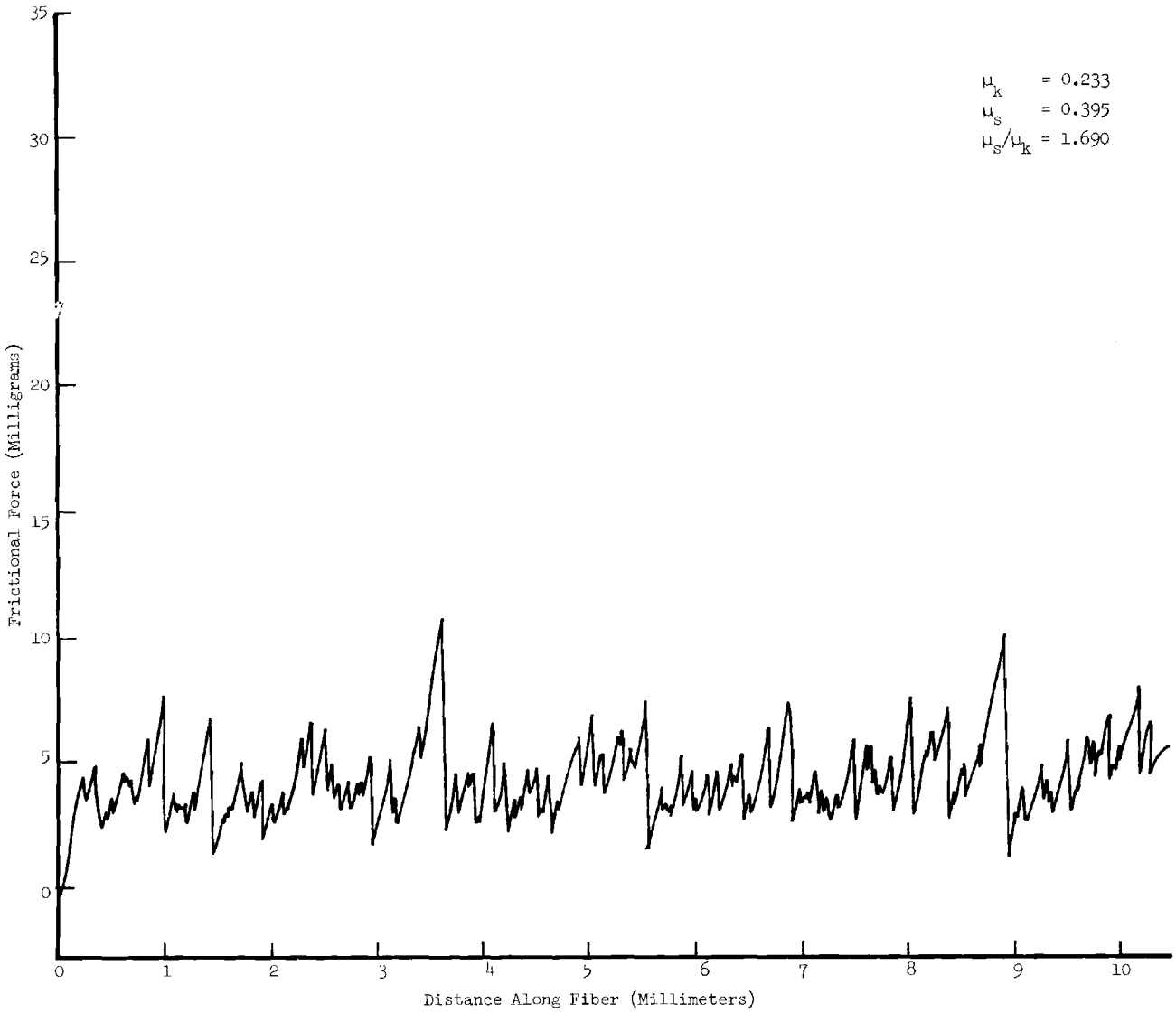


Figure 15. Frictional data plot of Empire WR cotton fiber after treatment with Congo Red dye solution (drying time about 30 minutes).

depicted by the frictional curves. The ability of the frictional measurements to depict shape changes of a fiber by character changes of the data plot are clearly indicated.

4. Frictional Measurements at Low Normal Forces

a. General

The instrument for measurement of friction at low normal forces was described in Semiannual Report No. 3 and typical data obtained were presented in that report and in the following Report No. 4. This instrument has the capability of application of the normal force by an electromagnetic means as well as the servo analog output capability of the gravity loaded beam. In theory, it is capable of operating down to normal forces of about 1 mg as compared to the approximate minimum of 20 mg for the gravity loaded frictional instrument. In addition, it has as an accessory, an automatic integrator allowing rapid data analysis.

In spite of the apparent capability of the instrument and a large amount of data taken with it, some uncertainty of the zero line on the data charts and the unknown degree of susceptibility of the instrument to vibration and inertia effects limited confidence in the results obtained with it. These data often appeared to disagree with frictional theory, with measurements with the gravity loaded frictional instrument, and with respect to some measurements made with the instrument itself.

An endeavor was made in a thesis by Mr. Donald H. Gunther, Jr.[4] to completely explore the vagaries of the instrument and to resolve the apparent difficulties. The data for this thesis have now been essentially

completed, and the thesis will be published in December 1967. An outline of some of the important results of this work follow and the data are analyzed in respect to the overall frictional behavior of the cotton fiber in textile processing.

b. Effects of Changes in Normal Force on the Coefficients of Friction

In the preceding report (No. 4) some effects of the normal force changes on the coefficients of static and kinetic friction were reported over the range 2 to 10 mg. Also in an earlier report (No. 2) a similar study with the gravity loaded instrument was reported. In each of these, some low coefficient of friction values obtained for cotton at the lower normal force ranges were in disagreement with theories of friction and measurements presented by Tabor[11] for nylon and some other fibers (but not for cotton which has not previously been examined). Tabor showed for nylon that large increases in frictional coefficients occurred as the normal force approached zero. The discrepancy in measurements between Tabor's values and those obtained here with the gravity loaded beam could only be accounted for by ascribing it to an intrinsic fault of the instrument. For the gravity loaded instrument, the inertia of the arm and some obvious bouncing effects observed in high speed motion pictures of measurements seemed to account for the problem. For the electromagnetically loaded instrument, the fault and the sometimes erratic nature of the results were not so easily resolved. Fortunately, the new data have resolved the problem and added greatly to our comprehension of the overall frictional behavior of the cotton fiber. The supporting data are presented below.

Frictional measurements were made for cotton and nylon fibers over the normal force range 1 to 20 milligrams. Approximately 10 measurements were made at each level. Although the number of measurements was somewhat fewer than desirable, the large coverage of material prevented a large number of measurements from being made at each level. Figure 16 exhibits the plot of the data obtained for the values of μ_k and μ_s for the respective fibers. It will be noted that whereas the nylon curves follow the direction of the theoretical curve, the cotton curve approaches a peak at about 6 or 7 mg and decreases as the normal force decreases.

In Figure 17 (from Report No. 2) we see a similar behavior of the cotton and nylon curves occurring at about 15 mg. Since the previously observed behavior was correctly ascribed to the moment of inertia and dependent vibration period of the instrument, it is now evident that the shape factor of cotton has caused a similar behavior in the electromagnetically instrument, i.e., some bounce or time delay of the fibers in making contact after slips, have made the kinetic coefficient no longer valid at normal forces below about 7 mg. This behavior was not interpretable until the concurrent measurements of the performance of cotton and of the performance of cylindrically shaped nylon fibers were compared under as nearly identical experimental conditions as possible.

It is thus evident that a symmetric and cylindrically shaped fiber of relatively smooth surface will furnish with this instrument frictional data at low normal forces closely matching the theoretical frictional behavior curve, but that a fiber of complex shape such as the cotton fiber misleads the same instrument at some minimum normal force. For this instrument, the limit is about 6 or 7 milligrams, although on occasion

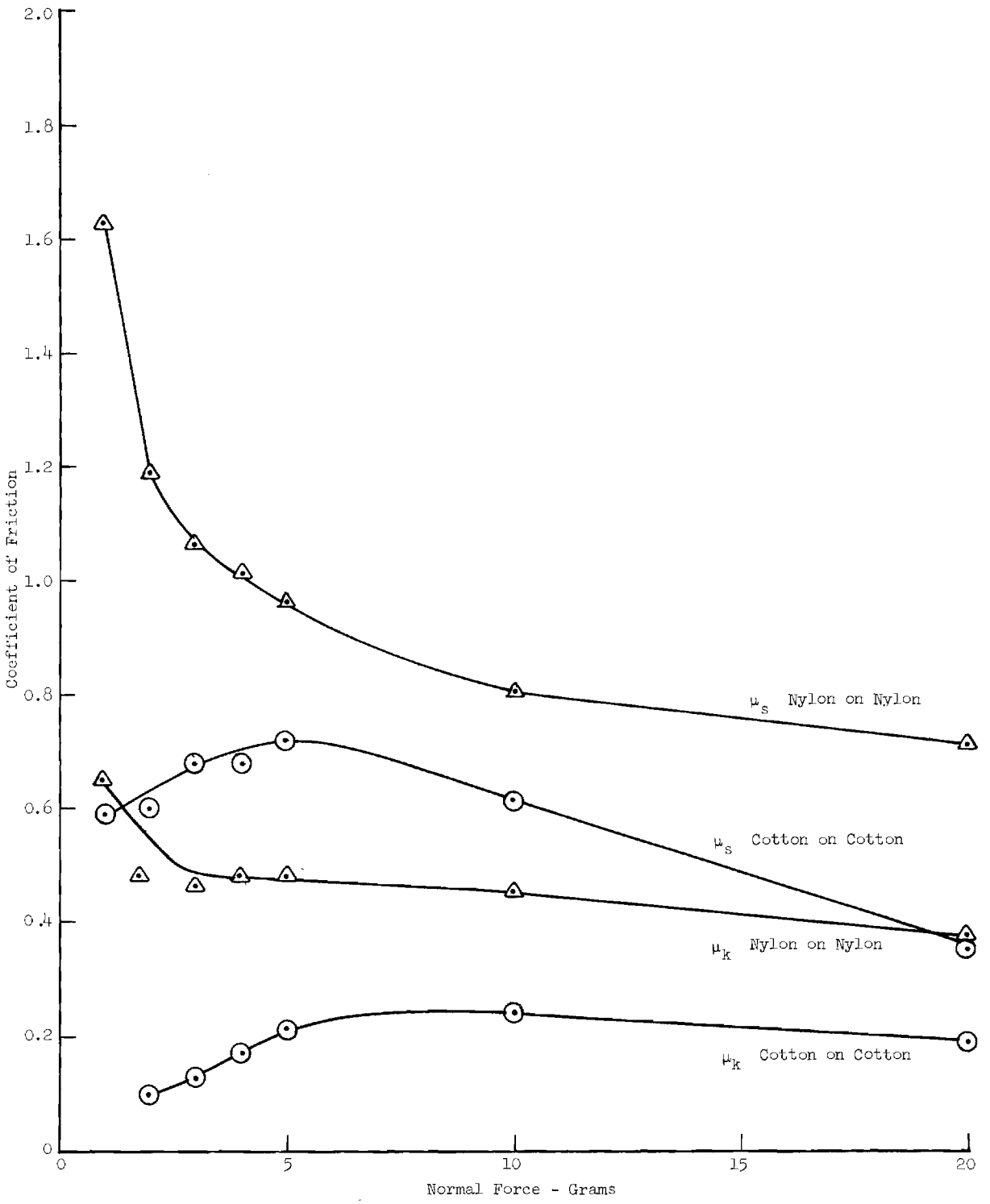


Figure 16. Variation of coefficients of kinetic and static friction of cotton and nylon at low normal force.

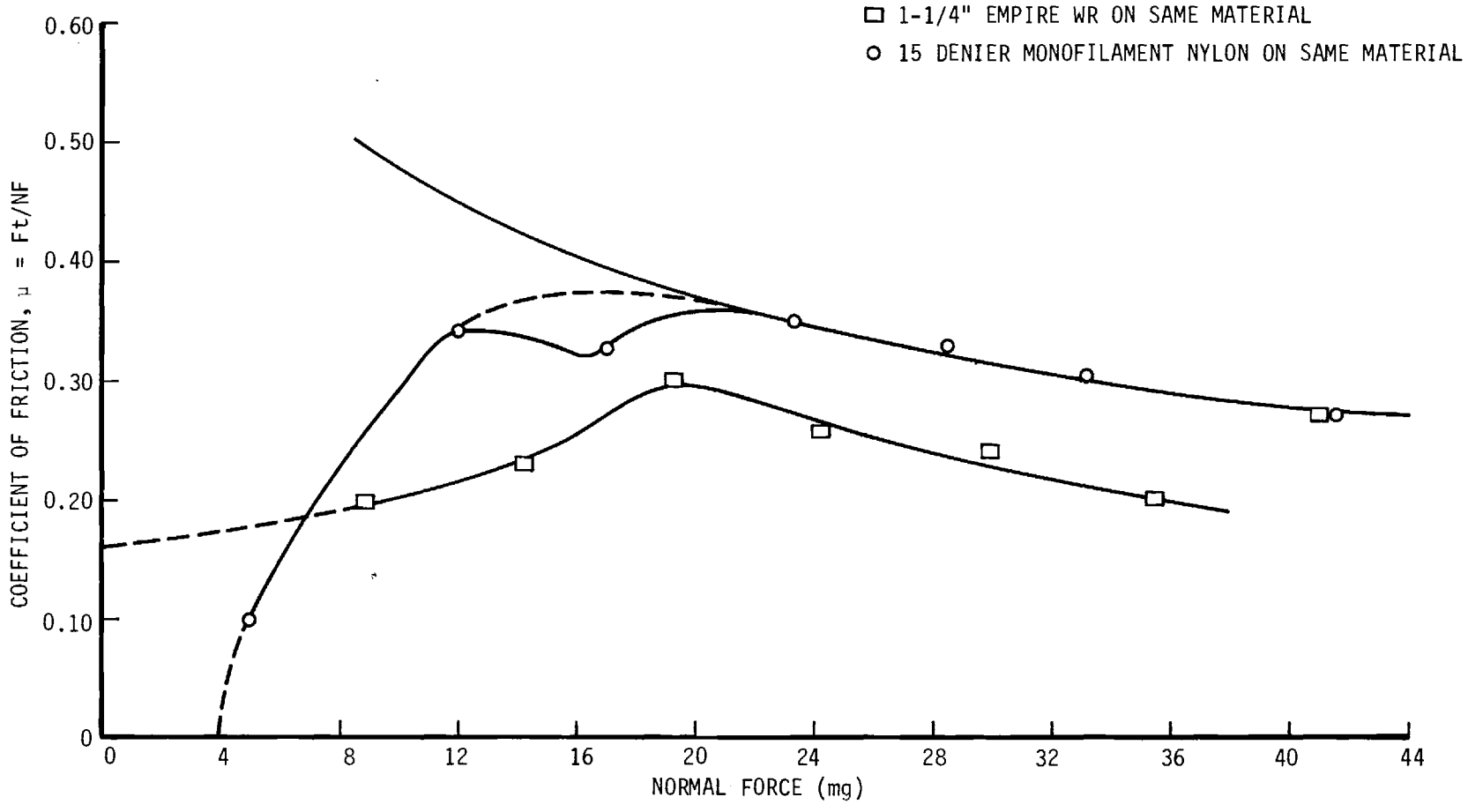


Figure 17. Variation of coefficients of kinetic friction with normal force for single ($1\frac{1}{4}$") Empire WR cotton fiber and for 15 denier nylon.

(perhaps with some fibers) it may be somewhat lower. At the same time, the measurements indicate the great importance of the shape factor of cotton in its frictional behavior.

It is, of course, noteworthy that here again the μ_k and μ_s values for cotton are lower than for the smoother nylon.

Another point of interest is indicated in the ratios of μ_s/μ_k. These also increase as the normal force decreases as shown in Figure 18. However, the increase in the ratio of μ_s/μ_k for cotton is much greater than in the case of nylon. This behavior is in part due to the lack of capability of the instrument in measuring the μ_k value of cotton below a normal force of about 7 mg; and the steep ascending slope of the μ_s/μ_k plot for cotton as the normal force is diminished, reflects the incorrect values of the true μ_k below this level of normal force. This behavior is obviously related to the irregular shape of the cotton as previously noted. The shape factor increasingly has made its presence known in these measurements, and will be further emphasized as the other experiments discussed subsequently.

c. Effects of Traversing Velocity

The traversing velocity of the fiber mounted on the gravity loaded frictional beam has been maintained at about 0.11 mm/sec (0.26"/min) during the majority of its use and except for the earliest measurements performed by McBride[3] in which the traversing rate was approximately 0.32 mm/sec (0.75"/min).

The traversing velocity of the fiber mounted on the electromagnetically loaded (low normal force) beam was 0.1 mm/sec during most of its use. Hence, the velocities of the two systems during the predominant part of

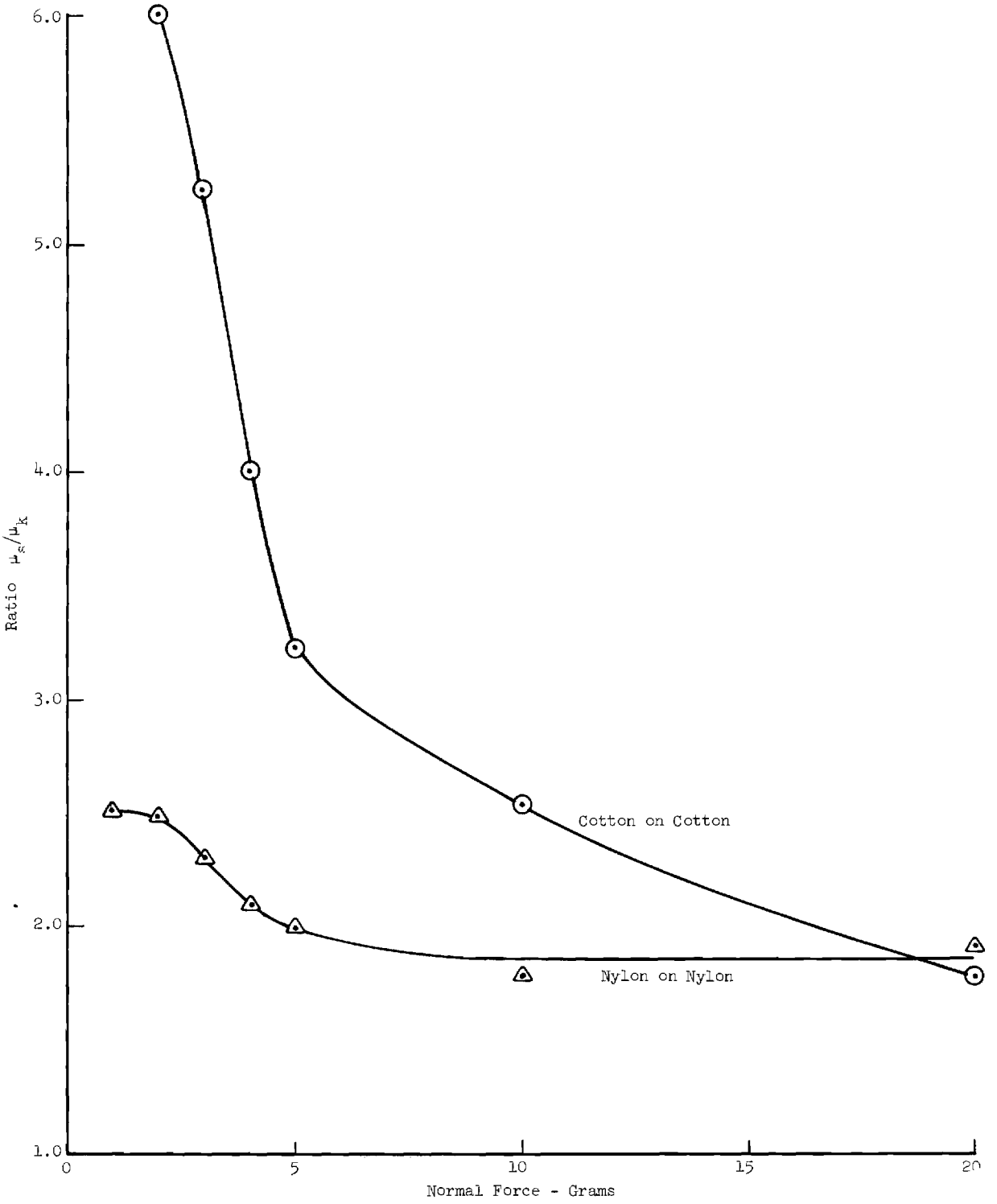


Figure 18. Variation of ratio μ_s/μ_k with normal force for cotton and nylon.

their use has been approximately 0.25"/min as compared to the 0.75"/min used in the original version.

Since traversing velocities have been varied considerably by various experimenters in the fiber friction field, and textile processing occurs at high velocities, a close look at the effects of velocity change on the respective coefficients of friction appeared desirable in order to place our own measurements in proper perspective. A series of cotton and nylon fibers were examined at velocities of 0.135 inch/min, 0.270 inch/min, and 0.540 inch/min, and at normal forces of 5 mg and 10 mg. The results are exhibited in Figure 19 for cotton and in Table 3. It will be noted that there is a marked increase in the coefficient of static friction of cotton with increased velocity. The rate of change is about the same at normal forces of 5 and 10 mg. However, the rate of change is small in the case of the kinetic coefficient of friction. The variation of the ratio μ_s/μ_k with velocity is shown in Figure 20.

It is interesting to note here that the lower traversing velocities and higher normal forces used subsequent to McBride's work probably accounts in part for the somewhat lower values of μ_s and μ_k obtained for cotton in the work of Bryant[12] and in our Technical Reports 2, 3, and 4, respectively, compared to the values as McBride reported.

Similar measurements of the variations of μ_s, μ_k, and μ_s/μ_k for nylon on nylon were made at normal forces of 5 and 10 mg and the data obtained are exhibited in Figures 20 and 21. It will be noted that the μ_s value exhibits a slight linear upward trend with increase in velocity and the μ_k values slight positive and negative trends at 10 mg and 5 mg respectively. The μ_s/μ_k ratio exhibits an upward trend. The trend of the μ_k values are so

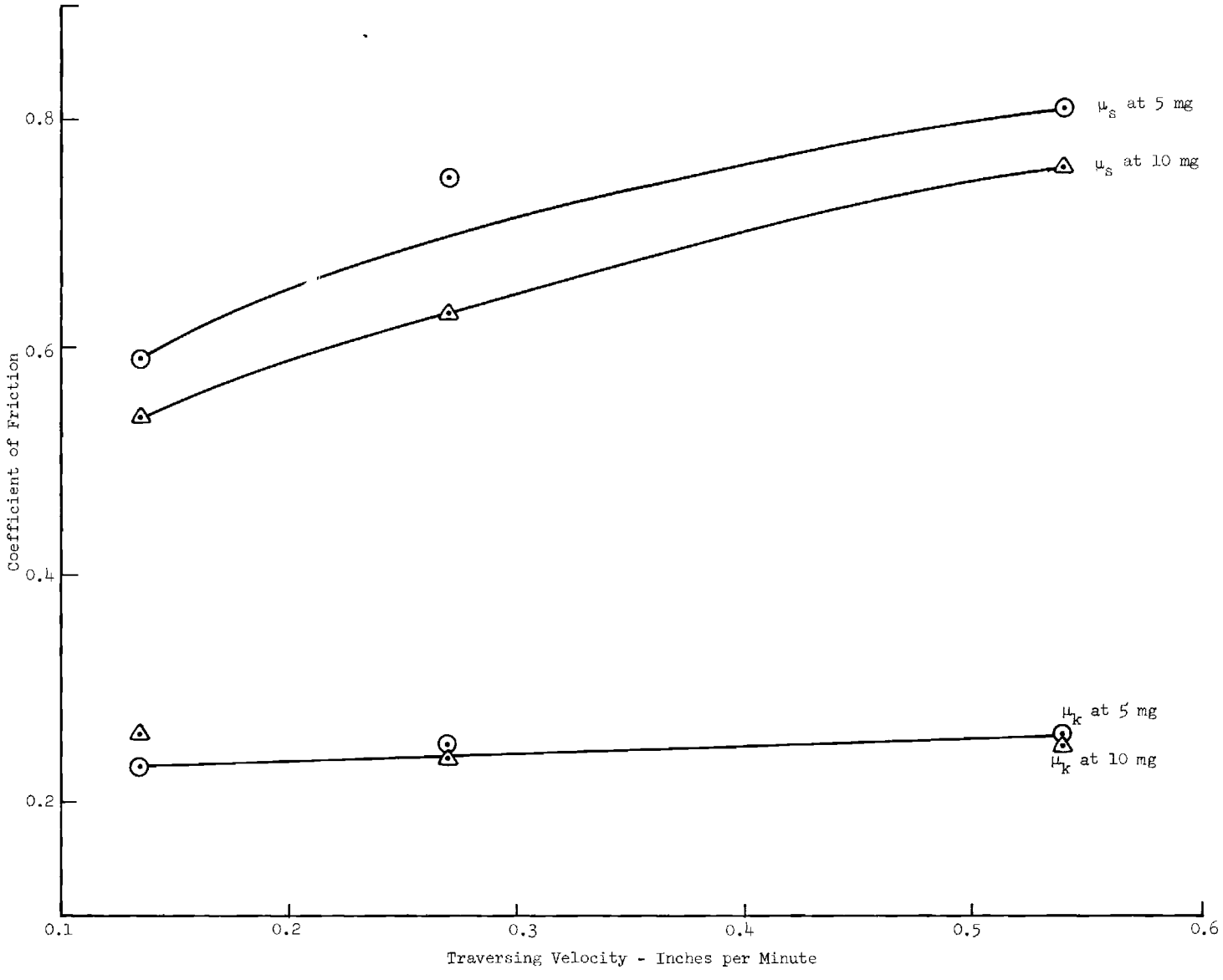


Figure 19. Coefficients of friction of cotton on cotton versus fiber traversing velocity.

TABLE 3

Variation of Frictional Parameters of Cotton and Nylon with Fiber Traversing Velocity

Relative Speed (in/min)	μ_s	μ_k	μ_s/μ_k
Summary Chart - Cotton 5 mg			
.135	.59	.23	2.93
.270	.75	.25	2.96
.540	.81	.26	3.17
Summary Chart - Nylon 6 5 mg			
.135	.91	.49	1.89
.270	.93	.48	2.01
.540	.95	.45	2.17
Summary Chart - Cotton 10 mg			
.135	.54	.26	2.14
.270	.63	.24	2.61
.540	.76	.25	3.05
Summary Chart - Nylon 6 10 mg			
.135	.73	.44	1.66
.270	.80	.45	1.78
.540	.87	.46	1.89

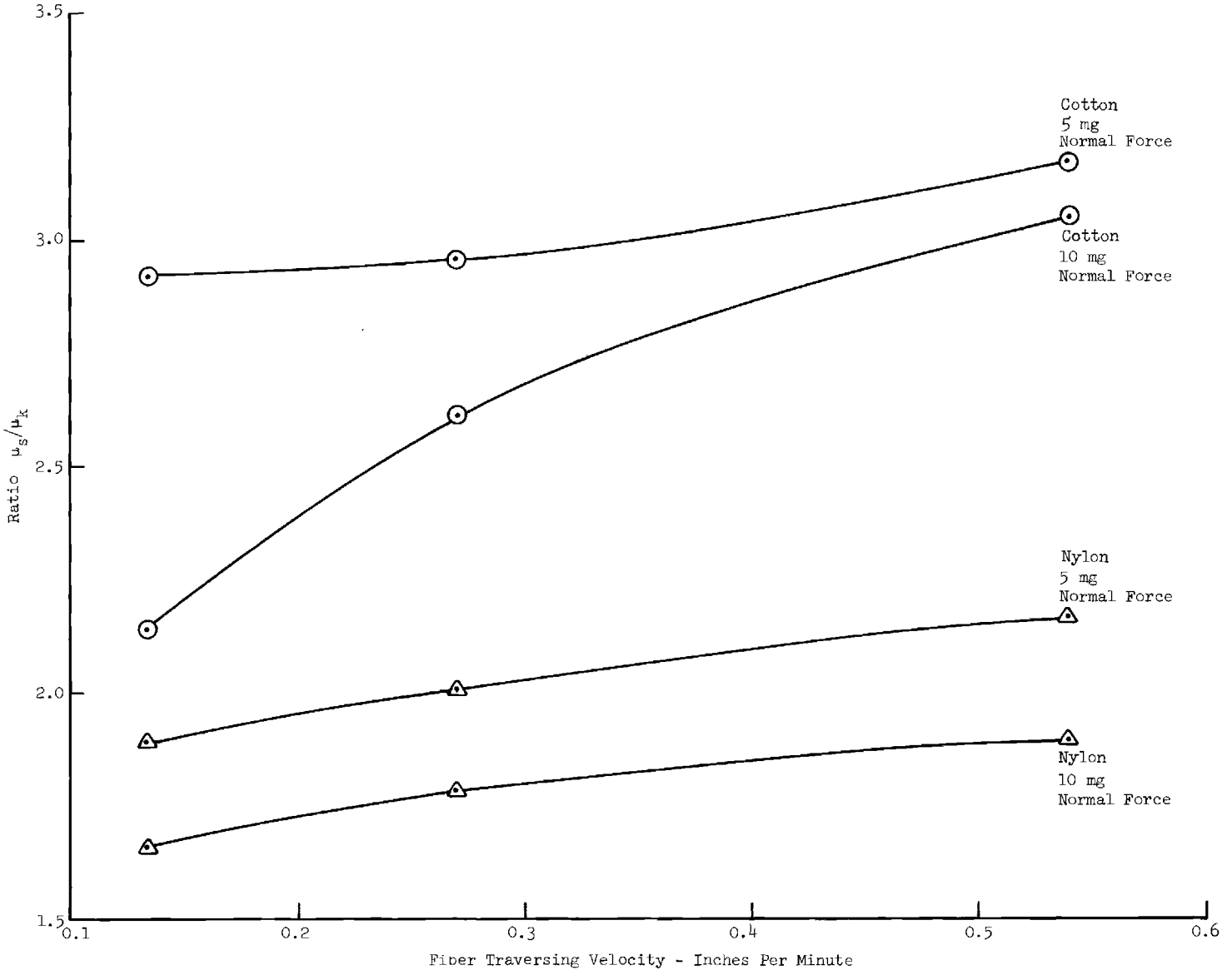


Figure 20. Variation of ratio μ_s/μ_k of cotton and of nylon versus fiber traversing velocity.

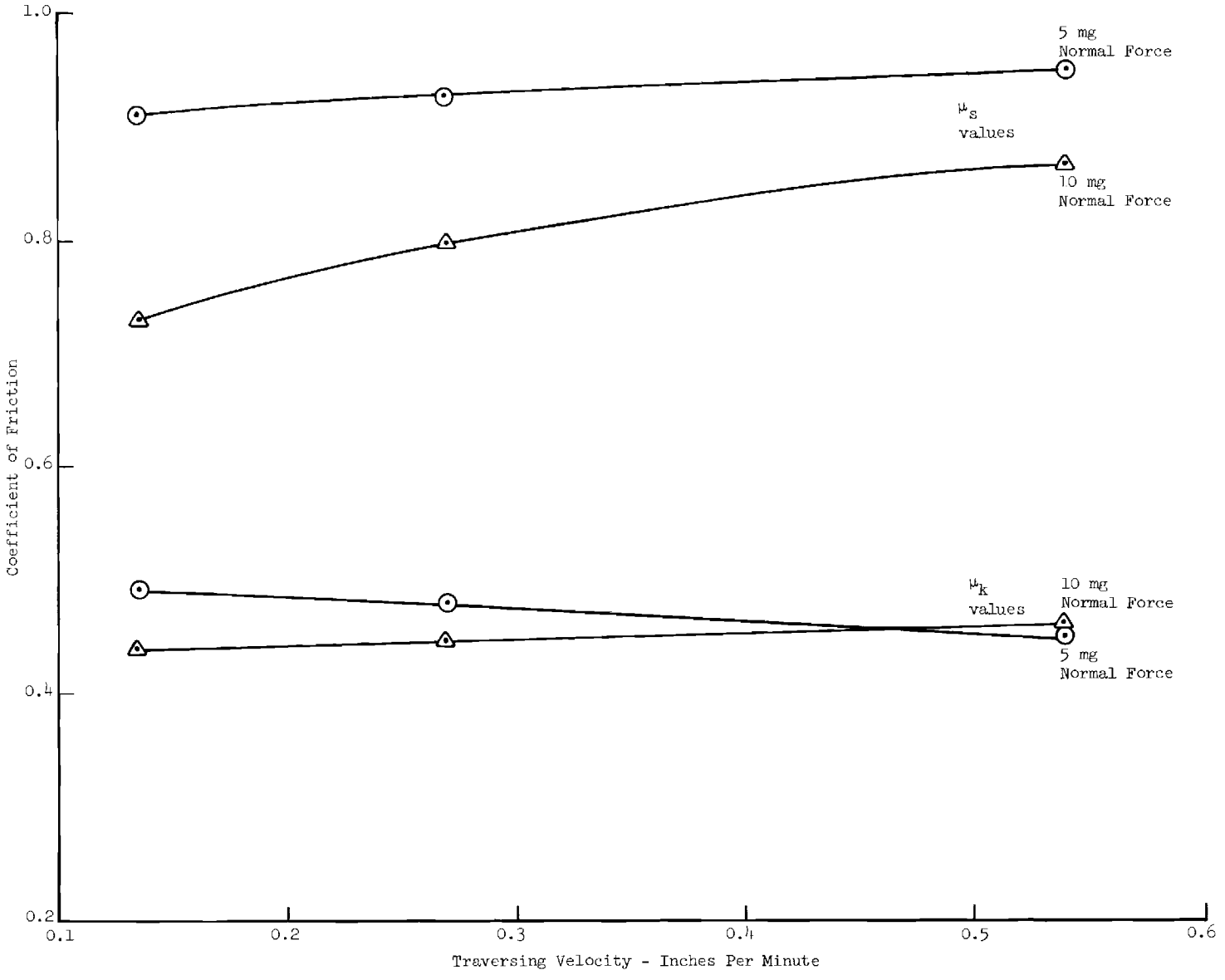


Figure 21. Coefficients of friction of nylon on nylon versus fiber traversing velocity.

small as to suggest they are within the limit of error and a larger number of measurements would be necessary to verify if a definite trend actually exists.

d. Frictional Parameters of Various Fibers

In order to assess better the frictional parameters of the cotton fiber, a series of measurements were made of the frictional behavior of a series of fibers of other materials. The measurements were conducted at a normal force of 10 mg, a traversing rate of 6.86 mm/min, and a tension of 425 mg. The measurements, except for cotton, represented usually the average of 9 measurements of each parameter. The specimens were obtained from supplies available from the A French Textile School of the Georgia Institute of Technology, but the history of each specific speciman, except the cotton, has not been established. The nylon was of a semi-gloss cylindrical nylon 6 type. The cotton was Empire WR from the standard bale used throughout this research. Micrographs of the other specimens are in preparation.

The measurements of μ_k and μ_s are exhibited in Figure 22 and Table 4 for comparison in ascending order of the coefficient values. The ratios of μ_s/μ_k are plotted in Figure 23, in the order used in Figure 22. Here it will be noted that the μ_s/μ_k values form a descending pattern.

These data are presented principally to show the frictional coefficient range presented by various fiber materials. Secondly, they point to higher μ_k values obtained for smooth fibers such as nylon and viscose compared to the rougher cotton fiber. The static coefficients of friction of cotton and rayon were very similar. Two points that remain

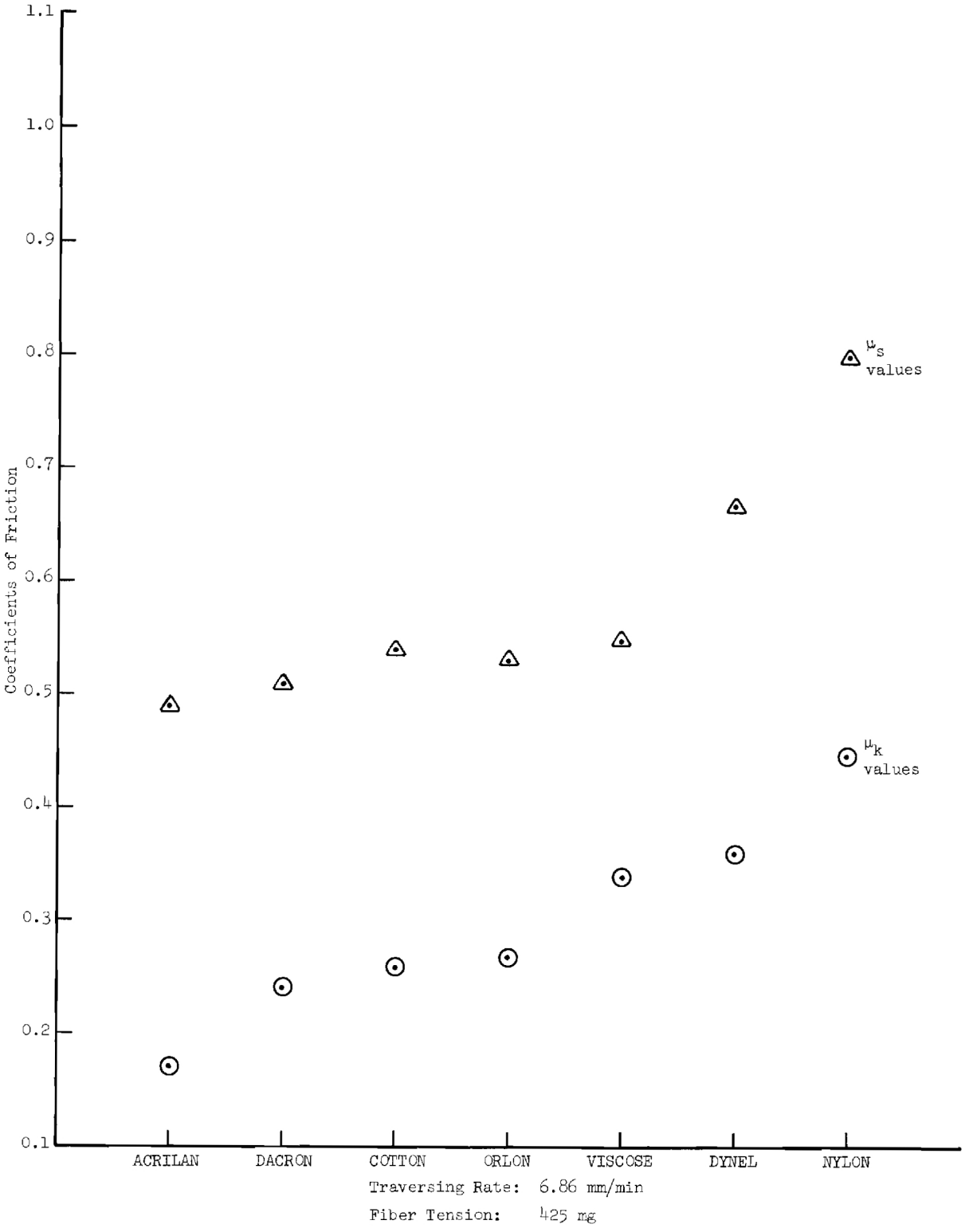


Figure 22. Coefficients of friction of various fibers at 10 mg normal force.

TABLE 4

Frictional Parameters for Various Fibers*

	μ_k	μ_s	μ_s/μ_k
Acrilan	0.17	0.49	2.95
Dacron	0.24	0.51	2.17
Orlon	0.27	0.53	1.97
Viscose	0.34	0.55	1.72
Dynel	0.36	0.67	1.86
Nylon	0.45	0.80	1.78

*Normal force 10 mg
Tension 425 mg
Traversing velocity = 0.270"/min =
6.86 mm/min =
0.114 mm/sec.

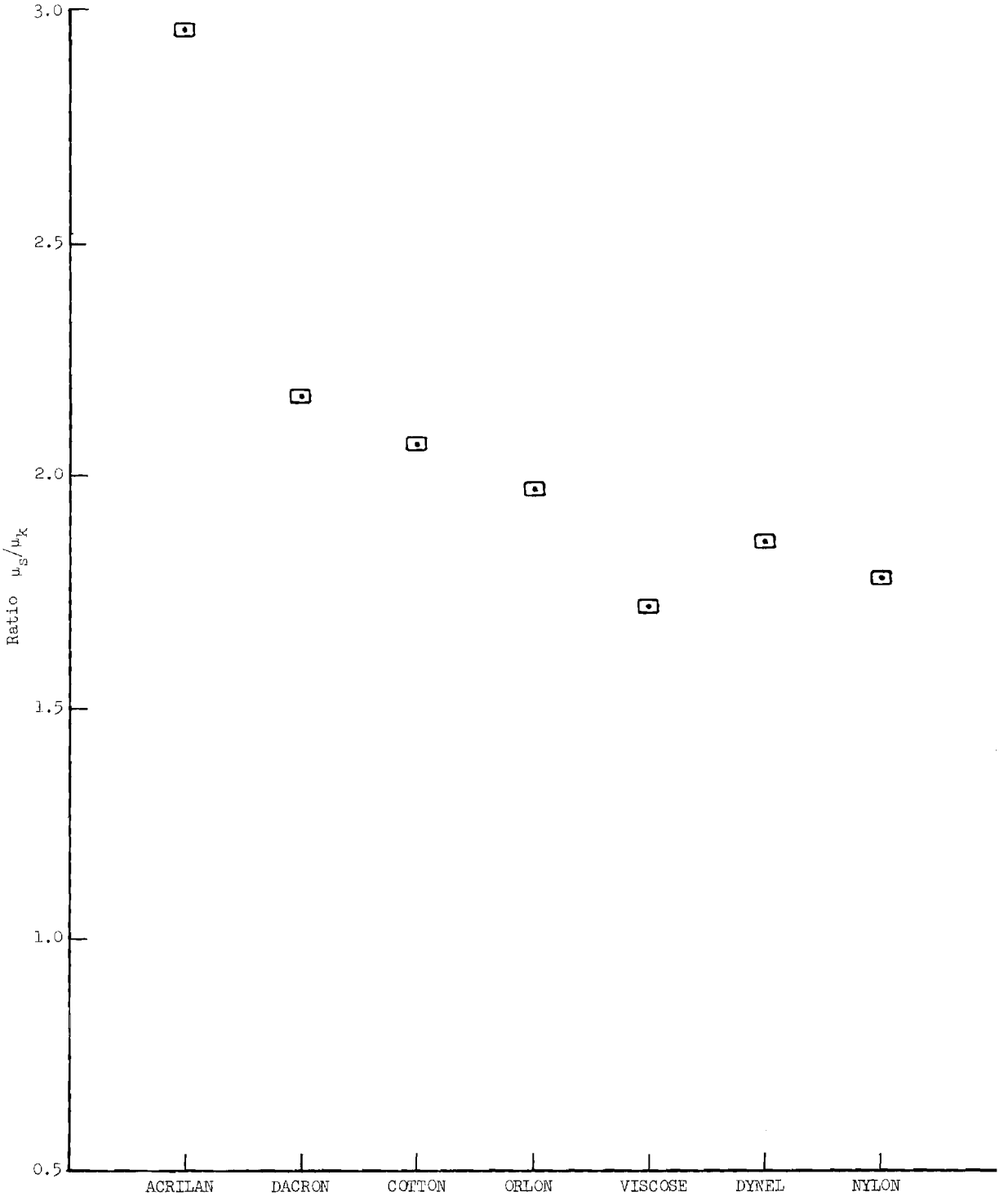


Figure 23. Ratio μ_s/μ_k for various fibers plotted in the same order as Figure 22.

to be assessed are the actual contact area of the fibers and the shape effect. Larger contact zones reduce overall pressure and should have the same effect as reducing normal force, which action results in an increased coefficient of friction. The nylon 6 fiber used was larger in cross section (15 denier) than the other fibers. Another matter which needs consideration is the relative shear strengths of the fibers since the stick portion of the frictional analog plot should be related to this property as reported by Bowden and Tabor.[11]

The matter of shape and the materials of various fibers needs to be examined in greater detail and will be undertaken in subsequent thesis research.

5. Comments

It is evident from the data presented, that for comparison of frictional data of fiber materials by various investigators, every detail of the investigation must be defined. These details include the apparatus; the fiber tension; the traversing velocity; the fiber material, size, and shape; the normal force; and the ambient conditions of temperature and humidity. Finally, the method of measuring friction must be considered and the method of interpreting the data obtained. From the analog curve and comparisons of the peak half-heights with the average curve height, obtained from area integration, it is clear that these two methods do not agree. The correct system is the area integration method and the determination of the average force acting over the distance represented by the abscissa. In macroscopic friction, the friction consists of millions of tiny point contacts, of the order of magnitude of the area presented by

two cross fibers (10^{-6} to 10^{-8} in^2) or smaller, acting successively or simultaneously. The frictional force at any instant is the average of the different phases of each stick slip acting at the moment. The large number of these acting simultaneously results in a constant average force for a constant sliding velocity. However, each point must act just as the two fibers. The work performed in moving a block at constant velocity is

$$\text{Work} = \text{force} \times \text{distance} = \mu_k \times \text{normal force} \times \text{distance},$$

$$\text{and } \mu_k = \frac{\text{work}}{\text{normal force} \times \text{distance}} .$$

On the frictional graph, after suitable calibration, work is represented by the area between the curve trace and the zero force line. Hence,

$$\mu_k = \frac{\text{area} \cdot (\text{calibration factor})}{\text{normal force} \cdot \text{distance along x}} = \frac{\text{average frictional force}}{\text{normal force}} .$$

The calibration factor is expressed in mg/cm for the displacement of the ordinate direction of the recorder, and is varied according to the normal force being employed. In the low normal force range, the factor used has frequently been 0.25 mg/cm. At 20 mg normal force, a factor of 1.9 mg/cm has usually been employed.

The method described appears to be the one valid one for calculating the coefficient of friction. Other methods of averaging the stick peak-to-valley distances do not take account of the time factor during which each force pulse acts and fails to represent over a short length of fiber the true μ_k value. In one comparison check we made, we found the values

obtained from stick-slip half-heights averaged approximately 20% higher than the μ_k values obtained from integrating the area under the frictional curve. The method of stick-slip height averaging is tedious and there is a tendency to skip many of the smaller stick-slips, which may account for the somewhat larger coefficient obtained by this method in which a considerable portion of the travel distance may be ignored. Where no analog plot is obtained, the peak-valley observations are all that may be available. This fact emphasizes the desirability of the analog plot method of frictional measurement. The character of the curve also presents much additional information concerning fiber characteristics and properties as previously discussed.

E. INFRARED ABSORPTION SPECTRA OF DEUTERATED FIBERS*

1. Introduction

The work conducted by the infrared spectroscopy group during the last period has consisted primarily of an investigation into the use of the fiber-press device in deuterium-exchange studies with fibers and the determination of fiber crystallinities. The work has been divided into three phases:

(1) The design and development of the required equipment for the deuteration experiments,

(2) The formulation of techniques of deuteration based on previously reported methods and original research conducted on the project, and

*This work was contributed principally by Kenneth W. Stephens and H. Lamar Hicks, graduate students in Nuclear Engineering and Textiles, respectively, and Dr. James A. Knight, Jr., Research Professor, Head Radioisotopes Laboratory, Georgia Institute of Technology.

(3) The application of the deuteration methods to the investigation of fiber crystallinities.

The first two of the phases have been concluded, and the third is nearing completion. The fibers used were cotton, ramie, and rayon.

2. Equipment

Most of the work which has been reported by others concerning deuterium-exchange studies of cellulose has used cast viscose films as the sample medium. Although liquid-phase and vapor-phase methods have been used successfully with viscose films, the vapor phase was chosen for this project because the pressed-fiber specimens disintegrate when placed in a liquid.

The technique of obtaining the D_2O vapor is a modification of that used by Marrinan and Mann.[13]

As shown in Figure 24, the deuterium oxide vapor is obtained by passing nitrogen gas first through a phosphorous pentoxide drier, and then through a fritted-glass bubbler submerged in heavy water. The mercury manometer is calibrated to indicate nitrogen flow rates directly in ml/min. Since atmospheric water vapor would interfere with the deuteration, a mercury back-trap is used to prevent the entry of the water vapor through the exit sides of the cells.

The sample and reference cells shown in Figure 25, which are identical except for the bushing which holds the sample holder in its cell, were designed for easy access to the sample and the salt windows. The cells are matched so that the percent transmittance of one cell differs from that of the other by no more than two percent. Compensation in the reference beam of the spectro-photometer is achieved by means of wire grids.

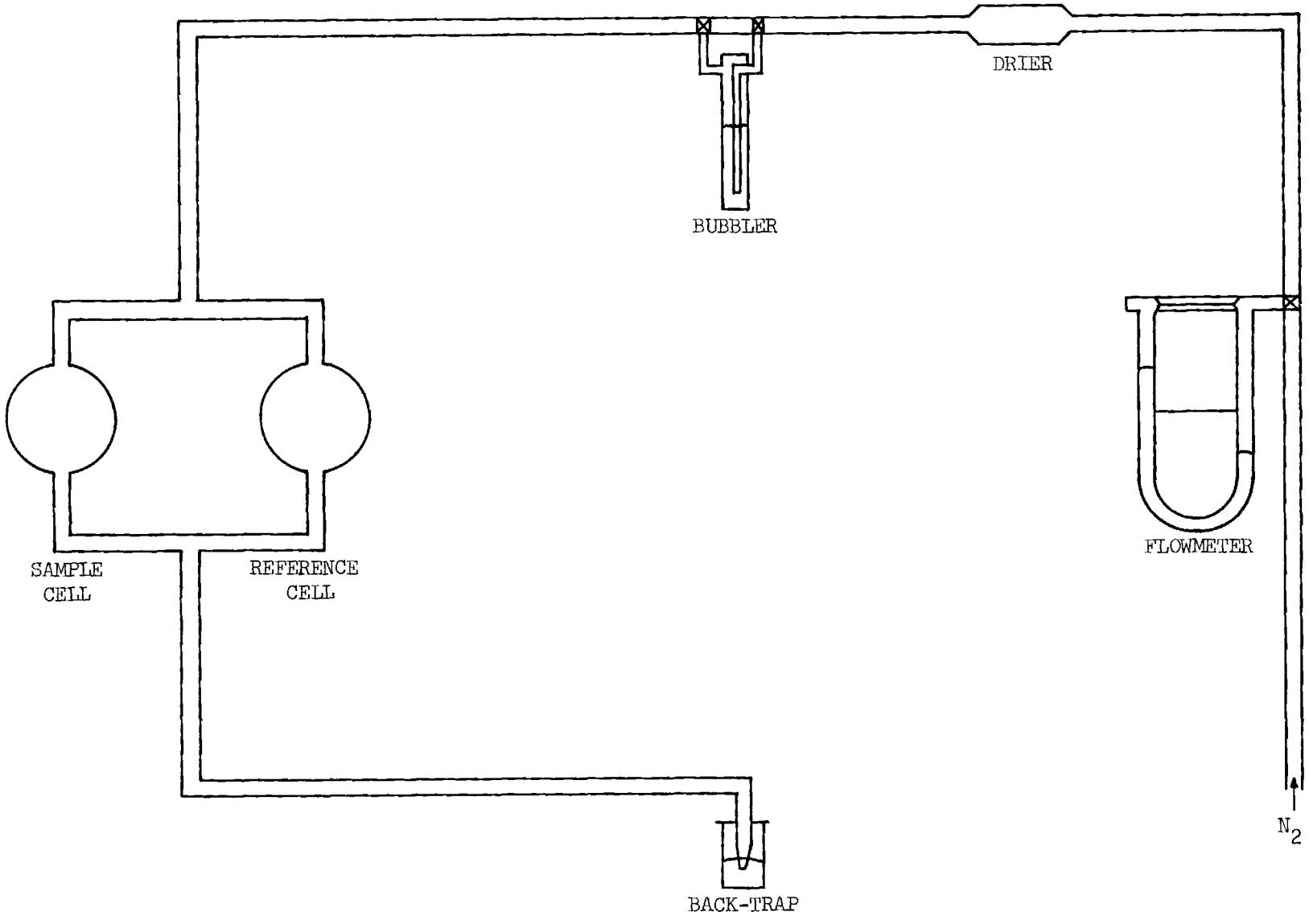


Figure 24. Preparation and flow of D_2O vapor in deuteration of fiber specimens.

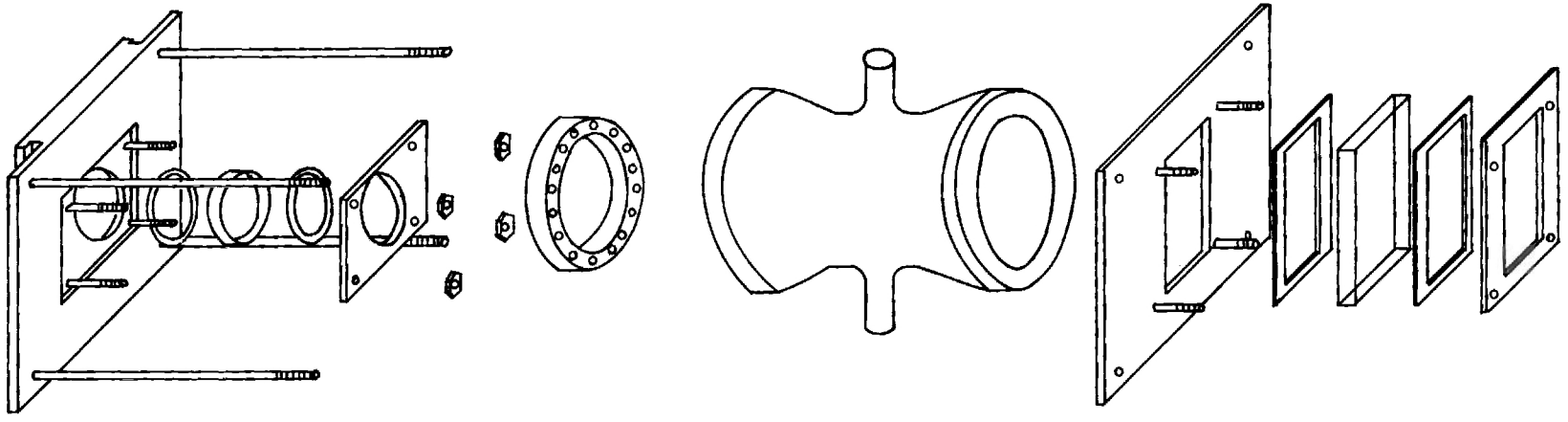


Figure 25. Exploded view of deuteration cell.

3. Deuteration Techniques

a. Pre-Deuteration Drying

The pressed sample must be thoroughly dried before the deuteration is begun, because the rapid exchange of deuterium atoms with the hydrogen atoms in absorbed normal light water results in an infrared band which erroneously implies that the hydrogen atoms in the cellulose have exchanged.

The drying times reported in the literature range from a few minutes to several days; the chief reasons for the differences are the particular sample weights employed and the varied criteria of dryness. The band at 6.1μ is known to be due to HOH rocking in absorbed water; consequently, the decrease in the intensity of this band during drying is an excellent measure of the dryness of the sample.

In practice, the dry nitrogen is passed through the cells while the spectrophotometer plots the height of the band as a function of time. When the rate of decrease of the band height becomes very small, the sample is considered dry enough to deuterate. Although the time required for drying depends on the particular sample, two hours is generally sufficient. Because of the sample blooming, which will be considered in a subsequent section, the above method of obtaining the drying time is not applicable to cotton; however, two hours has been found to be a suitable time for cotton also.

b. Deuteration

After the pre-deuteration drying has been completed, the dry nitrogen is bubbled through the heavy water until the 4μ band, which is

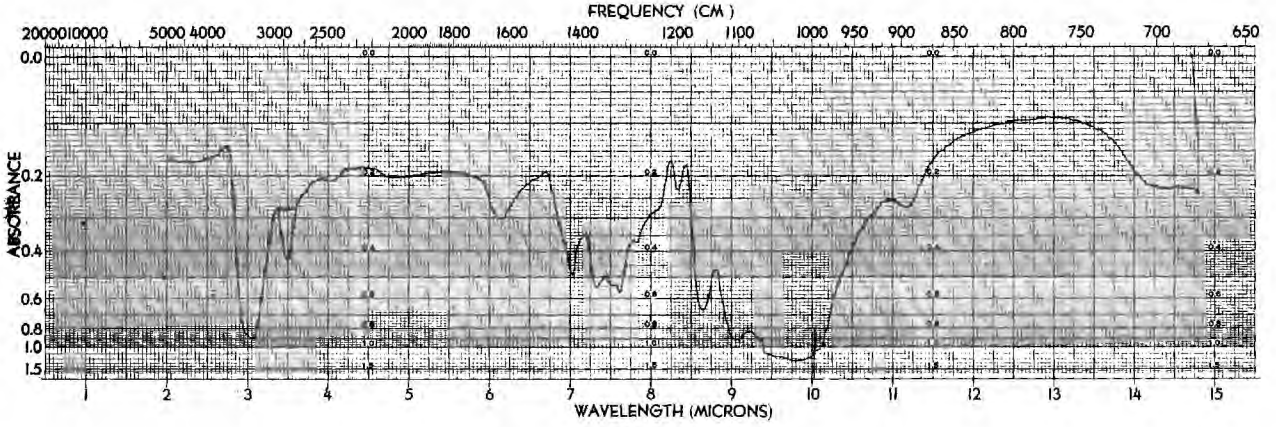
due to OD stretching, reaches its maximum. Most of the work reported listed four hours as the time required for deuteration of the amorphous regions; however, the deuteration of the fiber-press samples has been found to occur in shorter periods, a typical value being two hours. The usual flow rate of the nitrogen is 800 ml/min. The time the sample remains in the deuteration atmosphere, rather than the flow rate of the nitrogen, has been observed to determine the extent of deuteration. Further work is planned to observe the part the flow rate plays in the deuteration.

c. Post-Deuteration Drying

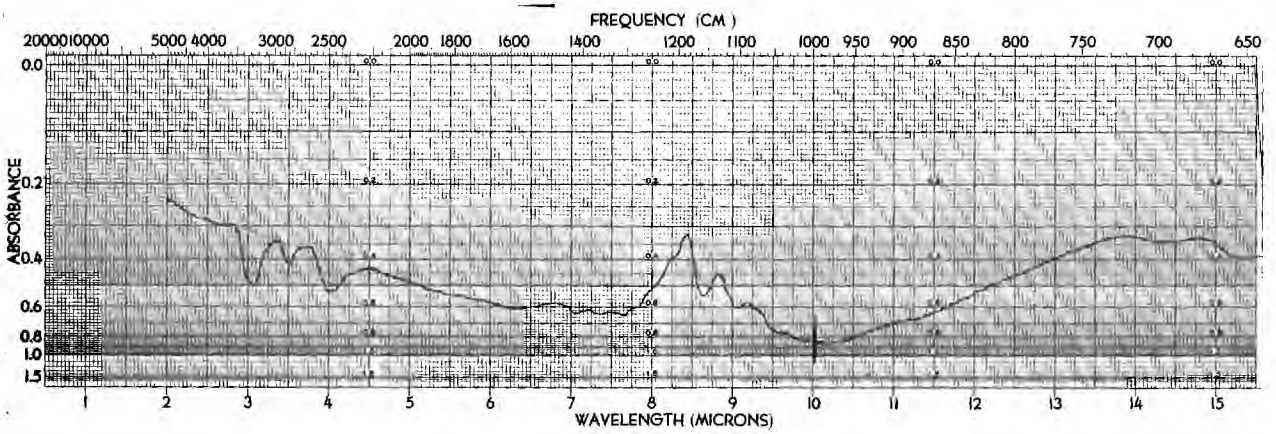
The sample must be dried after the deuteration because there are OD vibrations from the heavy water as well as from the exchange of deuterium atoms for the hydrogen. The length of drying is determined by the method used in the pre-deuteration drying, i.e., the sample is dried until the 4μ band fails to decrease. The post-deuteration drying is completed in cotton, viscose, and ramie within a time of one hour.

d. Deuterated Cotton Specimen

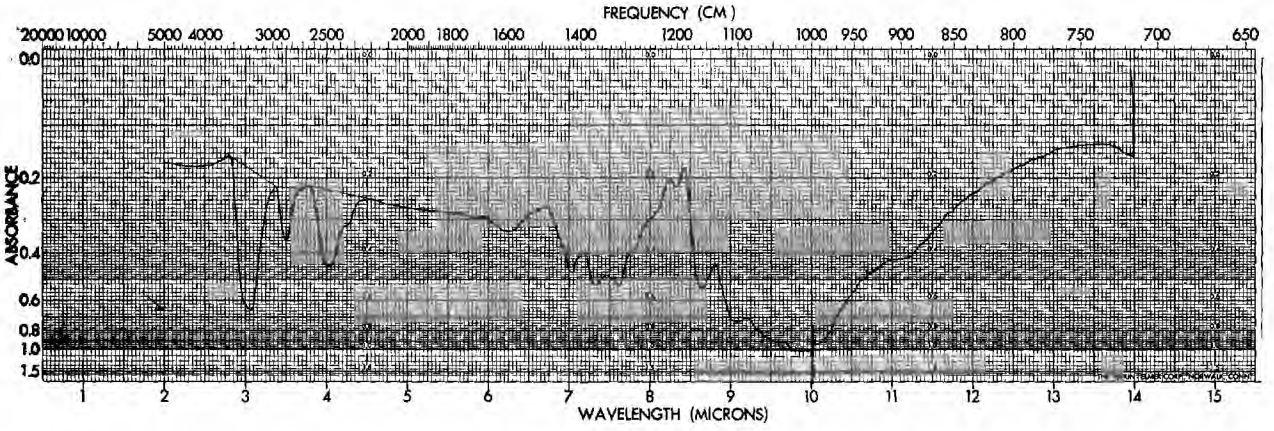
In Figure 26 are observed spectra displaying the contrast between the infrared absorption of an undeuterated and deuterated cotton specimen. Comparing the top and bottom spectra in the figure, one sees that significant changes in the spectra have occurred. Note especially the new band appearing at a wavelength of approximately 4 microns. The significance of the various changes will become clear as the discussion proceeds.



A. Undeuterated cotton before initial drying



B. Deuterated cotton (subjected to some blooming: Index 1.4)



C. Deuterated cotton (deuterated before pressing)

Figure 26. Comparison of infrared absorption spectra of cotton specimen before and after deuteration.

4. Crystallinity

a. Introduction

The generally accepted concept of cellulosic materials is that they can be considered as being composed of crystalline (ordered arrangement of hydrogen bonds) and amorphous (less ordered) regions. Deuterium-exchange techniques have been used for a number of years in the study of the extent of each region. Frilette, Hanle, and Mark[14] found that the deuteration reaction was very fast and was virtually completed in four hours. Moreover, they concluded that the part of the cellulose which does not react is crystalline. Rowan and Plyler[15] were able to deuterate small fractions of the crystalline regions using elevated temperatures and reactions running as long as 100 hours; however, at laboratory temperatures the fraction of crystalline regions which actually exchange is so small that the amorphous regions may be considered to be the only part of the cellulose which is affected. This preferential deuteration of the amorphous parts of cellulose enables the experimenter to separate the bands due to the two regions since the OD absorption frequency is different from that of the OH.

b. Interpretation of Spectra of Deuterated Samples

Marrinan and Mann[13] observed that upon deuteration, the 3μ OH band decreased into four distinct bands corresponding to the different ways in which the OH's are bonded; however, the spectra made in the course of this project (Figure 26) show only a reduction in the height of the 3μ band, as do those of Higgins, Stewart and Harrington[16] who referred to the four

residual bands observed by Marrinan and Mann[13] without making any attempt to explain reasons for the discrepancy.

The 4μ band (Figure 26), which is due to bonded OD stretching, has been found to increase proportionately as the 3μ band decreases. The relationship between the changes in the two bands can be used to calculate the percentage of crystalline material.

c. Methods of Obtaining Crystallinity Ratios

Marrinan and Mann[17] deuterated viscose films, resubstituted the hydrogen using light water, and measured the refractive index of the resubstitution water to determine the amount of deuterium exchanged. The percentage of amorphous material was calculated from the weight of the cellulose, the weight of the H_2O, and the amount of deuterium which exchanged.

If Beer's Law holds, the absorbance of a band at wavelength λ is related to the concentration of the sample by

$$\text{absorbance} = \log_{10} (I_o/I) = k_\lambda \, c \, \ell \quad ,$$

where I_o is the intensity of the radiation incident on the sample, I is intensity of the radiation transmitted, k_λ is the extinction coefficient per mole fraction, and ℓ is the pathlength of the radiation through the sample. Thus,

$$\frac{\log_{10} (I_o/I)_{OD}}{\log_{10} (I_o/I)_{OH}} = \frac{k_{OD} \, C_{OD}}{k_{OH} \, C_{OH}} \quad ,$$

where

$$C_{OD} + C_{OH} = 1 \quad .$$

From their measurements of absolute crystallinity, using the refractive index method, Marrinan and Mann[17] concluded the ratio $k_{OD}/k_{OH} = 1.11$ for cellulose. Therefore, the two equations in two unknowns can be solved for C_{OD}/C_{OH}.

Sepall and Mason,[18] in studying starches, assumed that the value of 1.11 found by Marrinan and Mann holds for the various forms of cellulose. There is no apparent reason why the value of this constant should not hold for fibrous cellulose.

Another possible method of obtaining crystallinity estimates employs only the 3μ band. Since the band is due to both crystalline and amorphous regions, the same band after deuteration is due to crystalline OH's only. Therefore,

$$\frac{A_{OH} \text{ after deuteration}}{A_{OH} \text{ before deuteration}} = \frac{A_{OH} \text{ crystalline}}{A_{OH} \text{ crystalline} + A_{OH} \text{ amorphous}} \quad ,$$

where A is absorbance.

d. Comments

Work is presently being conducted using these techniques.* The accuracy of these methods will be checked against x-ray diffraction data, and a comparison of the values obtained by the use of these techniques on the same samples will be made.

---

*The average crystallinity for Empire WR cotton was found to be 0.76.

Because of the experimental difficulties involved in the use of the refractometer technique of Marrinan and Mann, that approach is not feasible for this research; however, the other methods appear promising for future work. Consideration will be given to the establishment of a relationship between certain other ratios, used by Nelson and O'Connor[19] in work with the KBr pellet technique, and the aforementioned deuteration methods for crystallinity determinations.

5. Blooming

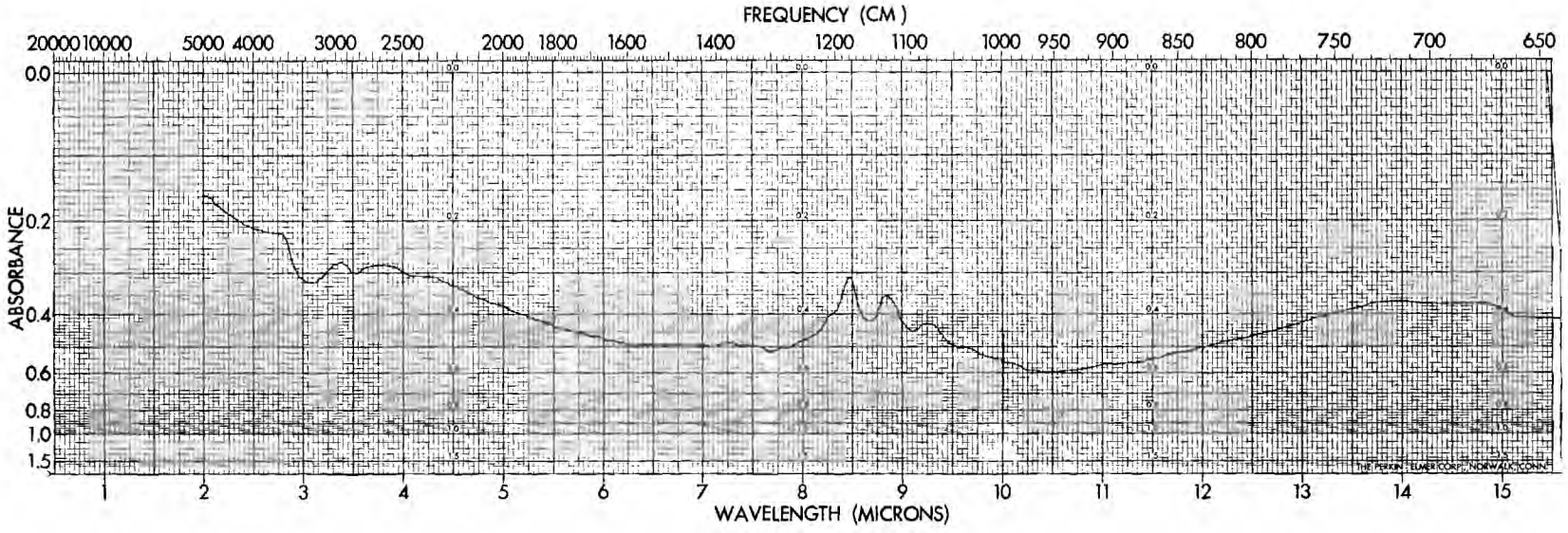
a. Introduction

Immediately after pressing, the fiber films are very smooth and plane, but certain of them lose their smoothness as the fibers expand. This effect has been named "blooming". The predominant characteristic of the spectrum of a bloomed sample is the loss of resolution, which is caused by the increased scattering of the impinging infrared beam. This effect can be observed in part in Figure 26(b), contrasted to Figure 26(c), and in Figure 27(a).

b. Blooming Index

So that blooming could be studied quantitatively, a blooming index was devised. As a direct result of blooming there is a decrease in band heights throughout the spectrum; thus, a band which is not affected by moisture changes in the samples was chosen as a blooming indicator. The blooming index is defined as

$$B.I. = \frac{A_1}{A_2} - 1 \quad ,$$



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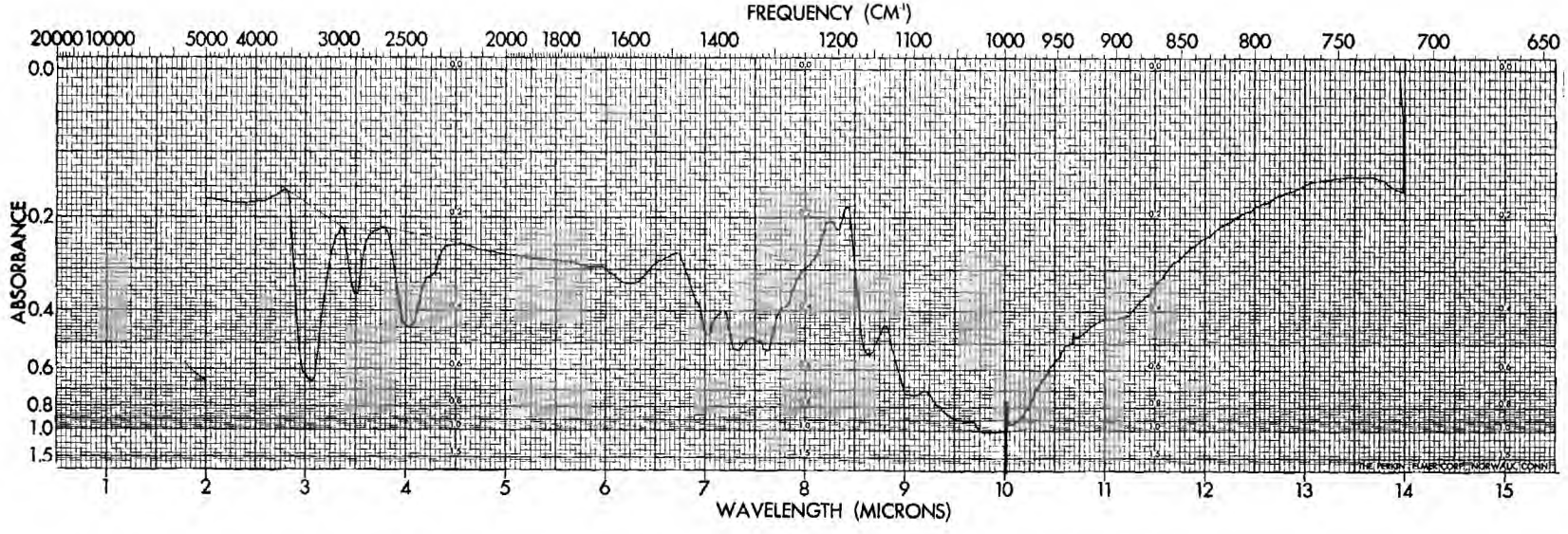


B

Figure 27. Effects on infrared spectra of blooming of the cotton specimen.

where A_1 and A_2 are the absorbances of the 3.45μ band (due to CH and CH_2 variations) before and after blooming respectively. Notice that if no blooming has occurred, the index will equal zero.

An overall reduction in band heights, such as that caused by blooming, may also arise if there are holes in the film; therefore, the blooming index is meaningful only when the initial spectrum is of acceptable quality.

Observation of the blooming index for each of the three fibers revealed that there is severe blooming in cotton samples, whereas the blooming in the ramie and rayon is negligible. The value of the blooming index is typically 0.2 for both ramie and rayon, while the cotton usually has a value as large as 6.0 and in some cases 10.0 or 12.0. A blooming index of 6.0 is indicative of a spectrum which is virtually worthless. When the entire spectrum is squeezed into a small range of absorbance (this is common in blooming), the error involved in band measurement becomes as large as the quantity being measured. In general, the spectra are not seriously affected if the blooming index is less than 0.5.

c. Remedies for Blooming

There are two obvious methods for reducing the effects of blooming. The first of these, re-pressing, was tried on a sample with a blooming index of 6.0. After re-pressing, the blooming index had been reduced to 0.6, and the spectrum was correspondingly improved. The fact that many of the films break when re-pressed implies that such treatment might significantly alter the physical structure. Comparison of absorbance ratios[19] for the re-pressed sample revealed the following:

	$\frac{7.30\mu}{3.45\mu}$	$\frac{7.0\mu}{11.2\mu}$
Before re-pressing	1.76	3.20
After re-pressing	1.25	2.80

Whether these significant changes are due to the treatment, or only to variations in the light-scattering properties of the sample, has not been determined. However, it is believed to be principally a reduction in light scattering after re-pressing.

A second and more feasible method of minimizing the blooming problem is that of deuterating before pressing; the usual procedure is to press the films before deuteration. The fibers are placed in the holder exactly as if they were to be pressed, but the holder is inserted into the deuteration cell for treatment. When the post-deuteration drying is completed, the holder is quickly placed into the heated die for pressing, after which time it is replaced in the cell for the running of spectra. The exposure of the sample to the atmosphere, for the short time in which the sample is in neither the cell nor the die, does not cause appreciable resubstitution of hydrogen since the time necessary for rehydrogenation in the laboratory atmosphere is on the order of several hours.

Figures 26(c) and 27(b) display spectra made after the cotton sample was first deuterated and then pressed and exhibit the resulting increase in spectral quality. The other spectrum in the H52 series [Figure 27(a)] shows the results of H_2O resubstitution and drying, which induced the usual blooming. Obviously, no spectrum could be taken before the deuteration in this case, and the blooming between the spectrum after pressing and the one after resubstitution had a value of 6.0

In summary, blooming is of no consequence for viscose or ramie but is severe in the case of cotton. For the latter fiber, press specimens must be deuterated before pressing for obtaining useful IR spectra.

6. Related Work

a. Weight Study

Twenty fiber specimens of cotton were pressed and weighed using the newly-acquired Mettler microbalance and a device designed to cut the films from the holder (see Figure 28). The weights are given below:

	Weight in Grams
1.	0.001610
2.	0.001971
3.	0.002159
4.	0.001690
5.	0.001495
6.	0.001453
7.	0.001102
8.	0.001521
9.	0.001205
10.	0.001165
11.	0.001400
12.	0.001205
13.	0.001342
14.	0.001145
15.	0.001125
16.	0.001310
17.	0.001329
18.	0.001195
19.	0.001185
20.	0.001075
Mean Value =	0.001384
Mean Deviation =	0.000222 gm
Standard Deviation =	0.000284 gm

The above weights illustrate the consistency with which the samples

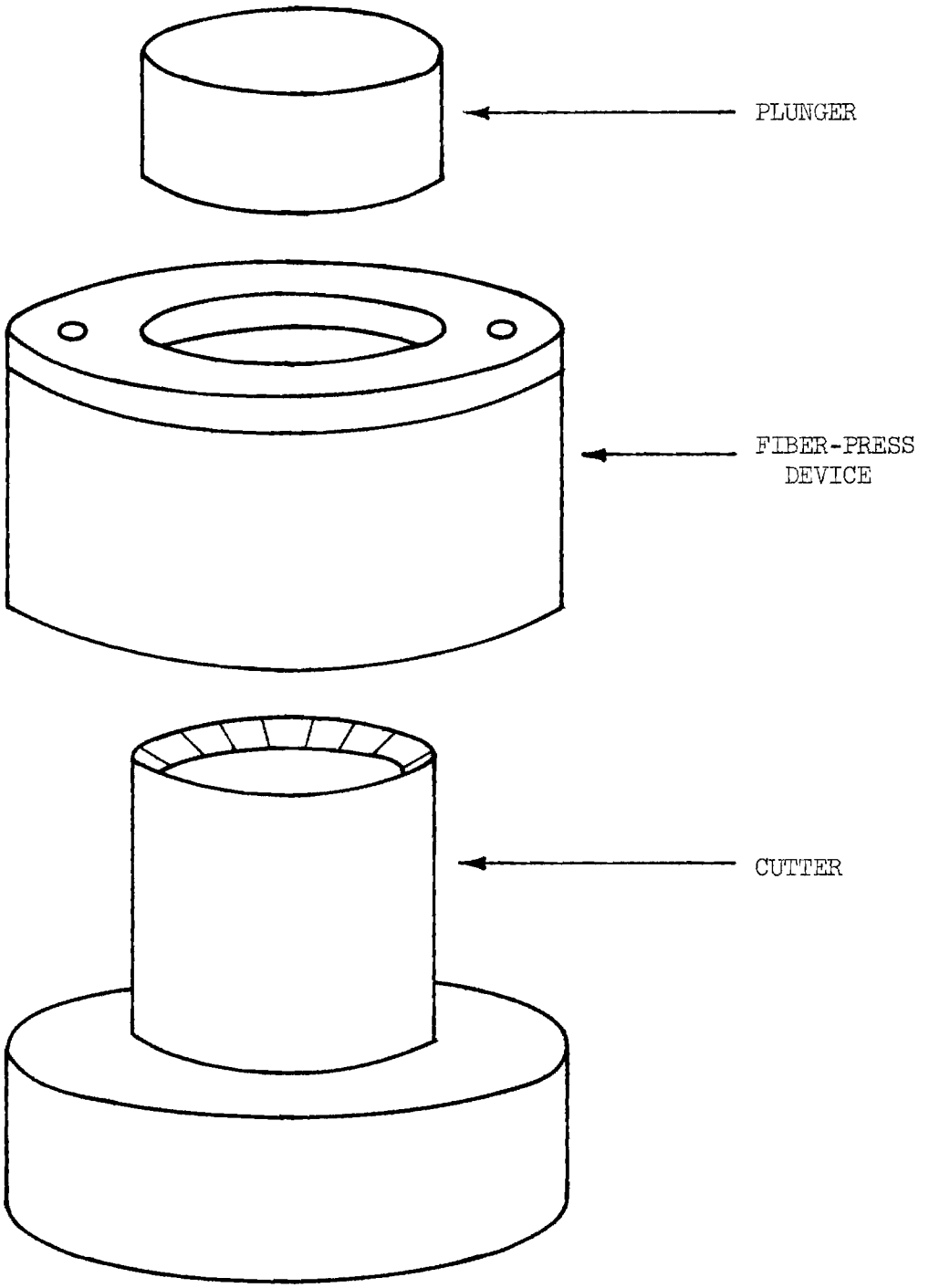


Figure 28. Drawing of cutter to remove fiber press specimens from holder.

can be made. The effects of sample weight (thickness) on absorption ratios will be examined in the near future.

b. Spectral Measurements During Deuteration Studies

Table 5 lists spectra made during deuteration studies accomplished during the period of this report. Typical spectra obtained for untreated and deuterated rayon and for ramie are exhibited in Figures 29 and 30, respectively.

The prominent absorption band at about 4 microns occurs in spectra of both of these fibers after deuteration. A number of other significant changes may also be observed. In rayon, the bands at 3μ and 6.10μ are greatly diminished and the minimum of the latter is slightly shifted to approximately 6.27μ. This latter band is markedly diminished during the drying process before the deuteration of cotton, rayon, and ramie. As pointed out previously, the decrease in the intensity of this band during drying is an excellent measure of the dryness of the specimen. When the rate of decrease of the height of this band becomes very small, the sample is considered dry enough to deuterate.

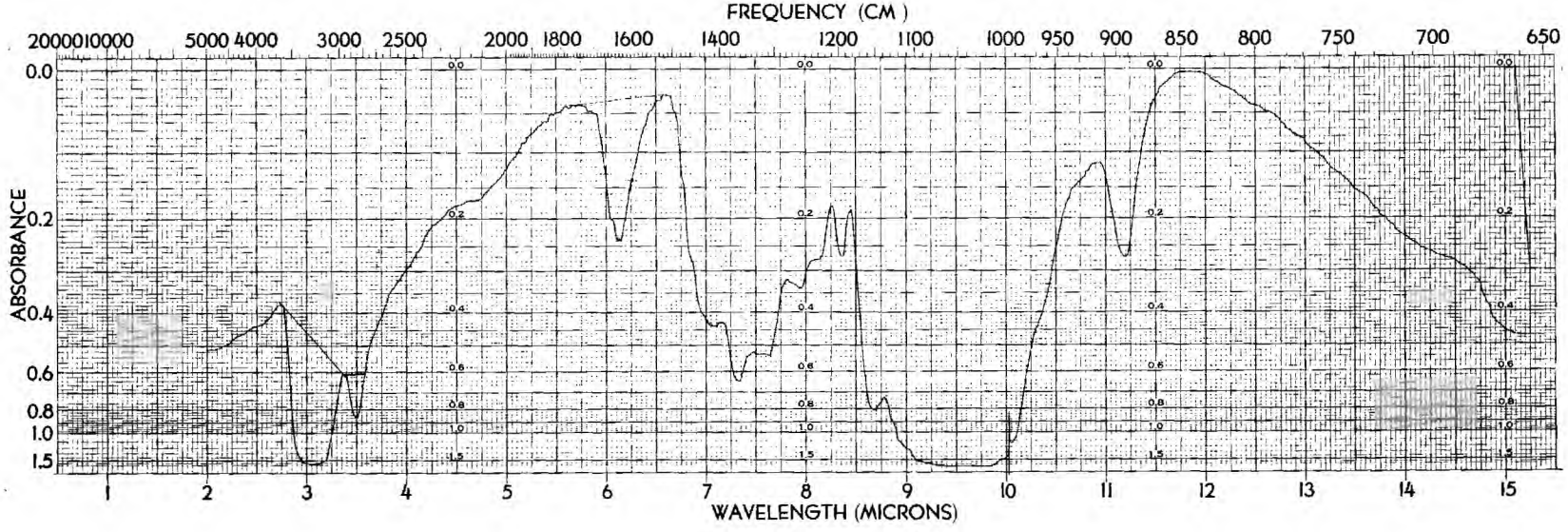
7. Summary

Equipment and methods for the preparation and deuteration of fiber specimens have been outlined, and typical spectra of cotton, viscose, and ramie before and after deuteration have been presented. The relative strength of the band at 6.1μ may be used to determine the dryness of the specimen before deuteration. The relationship between the changes in the 3μ

Table 5

List of Spectra Made During Fiber Deuteration Studies

Spectrum Number	Description
H1-5	Standard cotton
H6	Unknown cotton
H7-13	Practice spectra
H14	NaCl and glass
H15 A	Deuterated cotton
B	Deuterated cotton 48 hours later
H16 (A-D)	Deuteration study of cotton
H17 (A-D)	Drying study of cotton and re-pressed cotton
H18	Rayon
H19	Card sliver from the standard cotton
H20	KBr of the card sliver
H21	D_2O vapor
H22	D_2O liquid
H23 (A-D)	Deuteration study of cotton
H24	Combed pima cotton
H25 (A-C)	Deuteration study of cotton
H26	KBr of combed pima cotton
H27 (A-C)	Deuteration study of cotton
H28 (A-E)	KBr of the card sliver
H29	KBr of cellobiose
H30	Deuterated cotton
H31	Deuterated rayon
H32	Ramie
H33	KBr of ball-milled cotton
H34	KBr of ramie (40 mesh)
H35	Ramie
H36	KBr of ramie (20 mesh)
H37	KBr of ramie (40 mesh)
H38	KBr of ramie (40 mesh)
H39	KBr of ramie (20 mesh)
H40 (A-E)	Drying study of cotton
H41 (A-D)	Deuteration study of cotton
H42 (A-D)	Deuteration study of rayon
H43 (A-E)	KBr of ramie (60 mesh)
H44 (A-C)	KBr of ramie (20 mesh)
H45 (A-B)	KBr of ramie (40 mesh)
H46 (A-E)	Deuteration study of ramie
H47 (A-D)	Deuteration study of cotton
H48 (A-C)	Drying study of ramie
H49 (A-D)	Deuteration study of ramie
H50	Test spectrum
H51 (A-T)	Weight study of fiber films
H52 A	Cotton after post-deuteration pressing
B	Cotton after resubstitution and blooming



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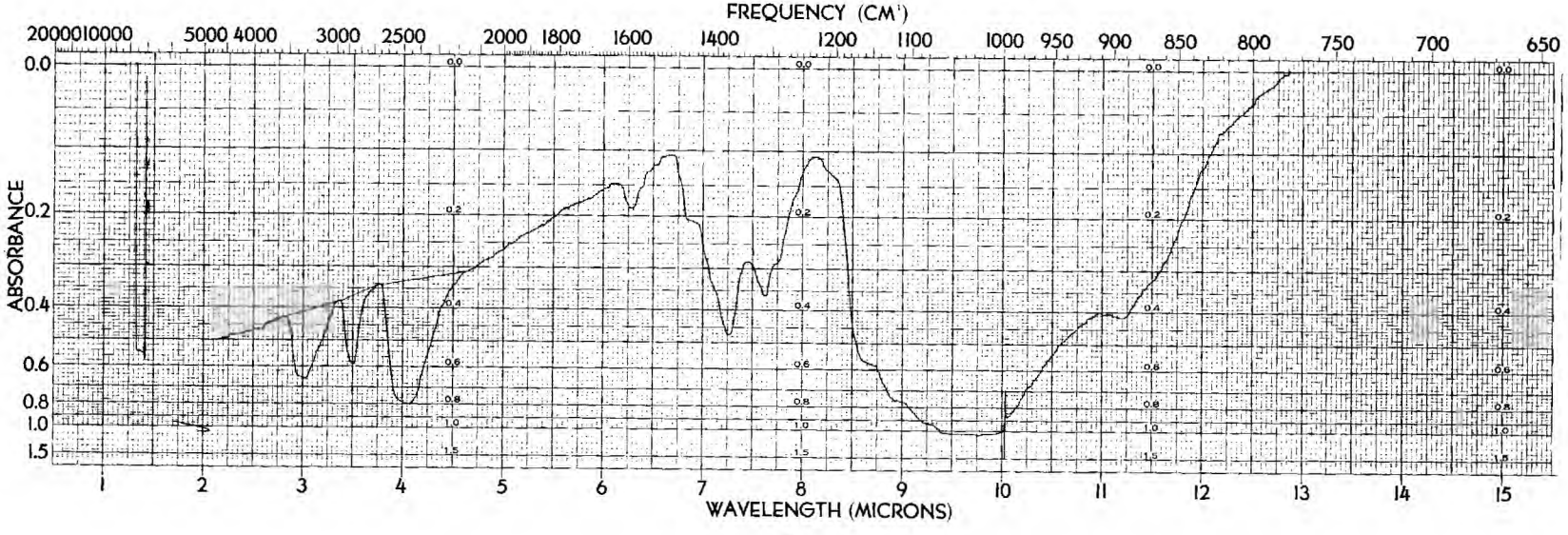
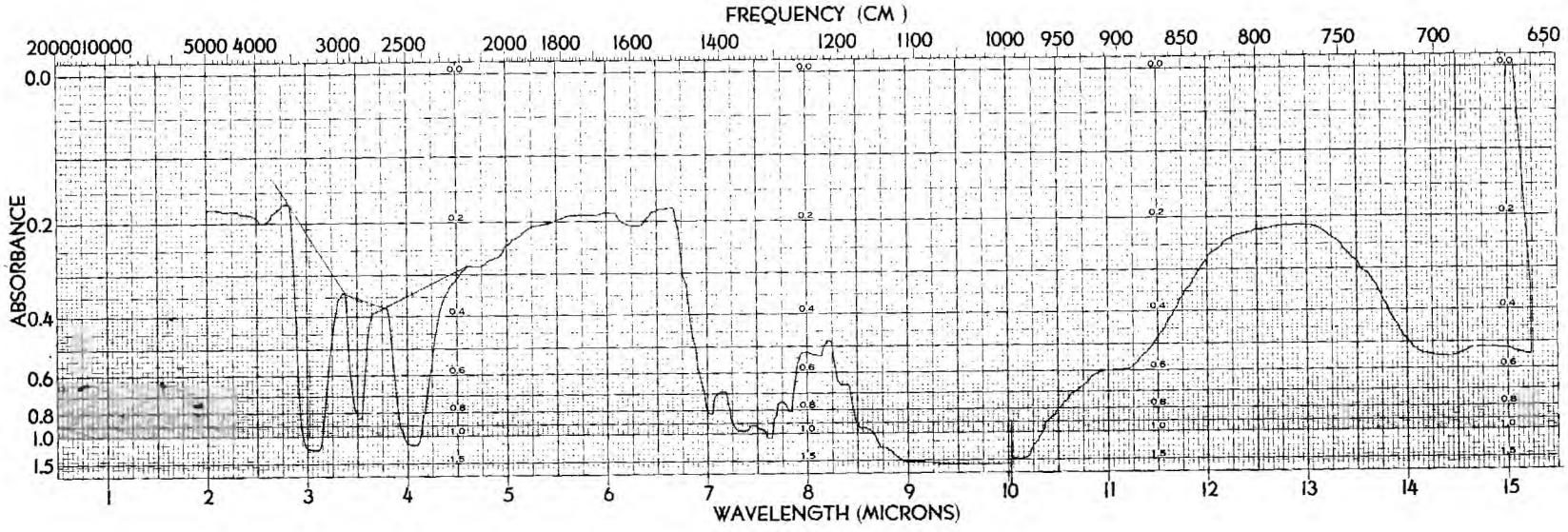
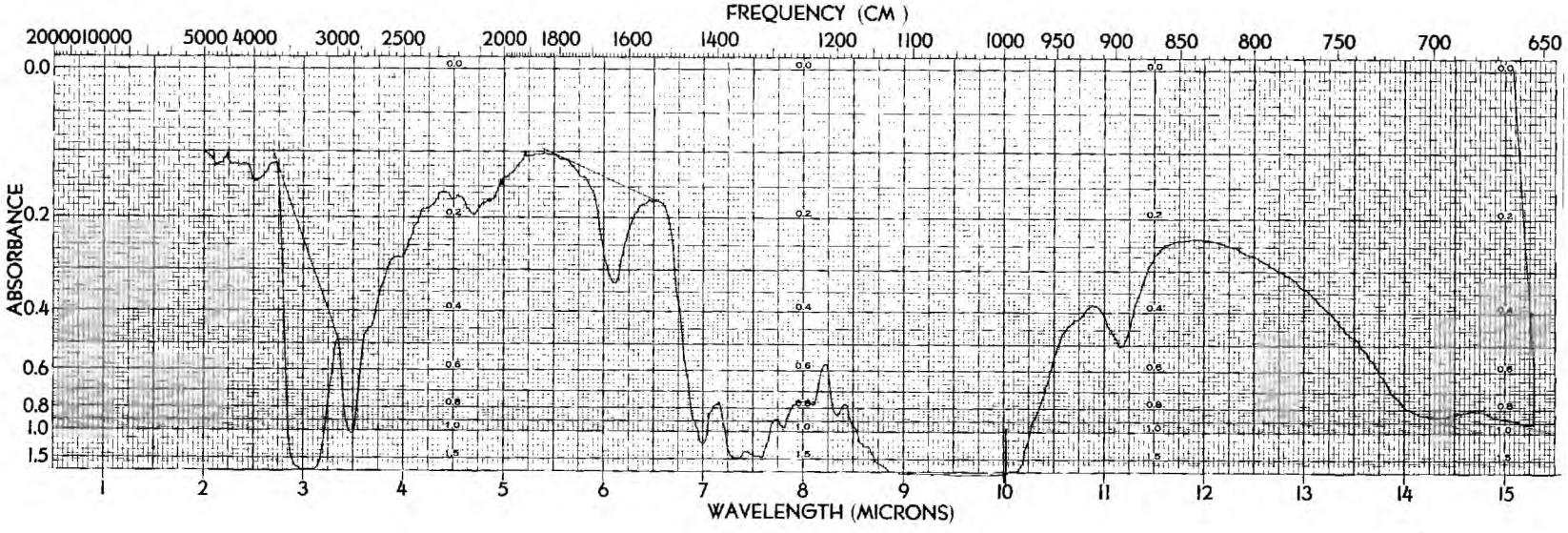


B

Figure 29. Infrared spectra of viscose before and after drying and deuteration.



A



B

Figure 30. Infrared spectra of ramie before and after drying and deuteration.

band (diminishing) and the 4μ band (increasing) may be used to determine the crystallinity of a fiber composed principally of a cellulose material.

Crystallinity determinations of a number of fiber specimens are now in progress and will be reported in Semiannual Report No. 6 (Final) to be submitted about 1 February 1967.

IV. CONCLUSIONS

The interfiber friction of textile fibers increases as interfiber velocity and as temperature* increase, and as fiber tension* and interfiber normal force decrease. Hence, normal conditions under which textiles are processed tend to maximize frictional effects.

Single fibers withdrawn from card sliver and from roving specimens withdraw with each fiber several other fiber and register withdrawal forces ranging between 5 and 113 mg active over 1/2 to 2/3 of a fiber length. Hence, fibers in fiber masses move as small fiber bundles under forces transferred by high "stick" zones as registered on the frictional analog curve. Interfiber slip occurs at zones of lower friction.

Cylindrical fibers such as some nylon specimens enable accurate frictional measurements to be made with present instrumentation down to approximately 1 mg normal force. For convoluted fibers such as cotton, however, the shape factor superimposed upon the fiber's intrinsic properties degrade the operation of the electromagnetically loaded instrument at about 7 mg and the gravity loaded one at about 17 mg. Fiber shape zones and fiber damage zones are responsible for many high sticks among cotton fibers and appear to establish, essentially, the μ_s value as defined in this work.

Processing of cotton fibers results in twisting, swaging, and other damage to the fiber. These effects frequently increase the localized heights and areas of stick peaks and zones respectively of the frictional

---

*Variation of fiber friction with temperature and tension were reported in previous reports of this Grant.

analog curve. However, fiber straightening and swaging effects also tend to reduce the overall area under the frictional analog curve. If damaged fibers are selectively (by the experimenter) omitted from measurements because of apparent large deviations of the data from the median value, the effects of processing through a commercial textile mill on the coefficients of friction of the fibers appear to be small. However, the increasing number of damaged fibers with processing and an effort to procure sufficient data to validly include frictional data from damaged fibers in the measurements should indicate an increase in the overall value of frictional coefficients with processing.

Measurements of the coefficients of friction of acrilan, dacron, cotton, orlon, viscose, dynel, and nylon, at 10 mg normal force, gave values of μ_s ranging from 0.49 to 0.80 and μ_k ranging from 0.17 to 0.45 in ascending order. Values for three or more of these materials, however, lay on a plateau with cotton at about 0.54 and 0.26 respectively.

Frictional data, at this point, because of the predominant part the μ_s values play in fiber frictional behavior and the behavior of the two measuring instruments under vibratory conditions, indicate that the imparting of a vibratory motion to a fiber mass should increase the frequency of slips and reduce the interfiber friction. Hence, increased fiber processing velocities and greater yarn uniformity might be expected as a result.

Techniques of preparing deuterated cotton fiber specimens by the fiber press technique for examination by infrared spectrometry have been outlined, and a method of determining the crystallinity of cotton from the relative infrared absorption of the 3.0 and 4.0 micron bands has been presented.

V. PROGRAM FOR THE NEXT INTERVAL

Experiments and theses now in course will be completed. Additional studies of interfiber friction at low normal forces and of the effects of fiber shape or fiber processing on the frictional analog curves will be undertaken.

Infrared evaluations of fiber crystallinity by the deuteration technique will be summarized.

Data for the final report will be collected, evaluated, and summarized. The report will be completed and submitted.

VI. PERSONNEL

The principal individuals employed on this research and the area of their interest are listed below.

Individual	Title	Area of Research
Richard B. Belser	Research Associate Professor	Project Director
James L. Taylor	Director, A. French Textile School	Associate Project Director
John L. Brown	Director, Analytical Instrumentation Laboratories	Optical and Electron Microscopy
James L. Hubbard	Assistant Research Physicist	Optical and Electron Microscopy
James A. Knight	Research Professor, Chemistry, Head, Radioisotopes Laboratory	Infrared Spectroscopy
J. Conrad Meaders	Assistant Research Scientist (Physics)	Friction Apparatus
Kenneth W. Stephens	Student Assistant (Physics)	Infrared Spectroscopy
R. A. Young	Director, Diffraction Laboratories	X-ray Diffraction
Ronald W. Dorman	Graduate Research Assistant (Nuclear Engineering School)	X-ray Diffraction
Donald H. Gunther, Jr.	Graduate Assistant (Textile School)	Frictional Properties of Cotton Fibers at Low Normal Forces
Edwin D. Cromer	Graduate Assistant (Textile School)	Effects of Processing on the Properties of Empire WR Cotton
H. Lamar Hicks	Graduate Assistant (Textile School)	Infrared Investigations of Cotton Fibers
Billy R. Livesay	Research Physicist	Fiber Friction and Tensile Properties

Individual	Title	Area of Research
Hong Ki Chin	Graduate Assistant (Textile School)	Fiber Friction
Larry B. Whitworth	Graduate Assistant (Textile School)	Effects of Processing on Properties of Pima Cotton Fibers in a Manufacturing Plant

In general, all work was performed on a part-time basis except that of Mr. Meaders who is employed full time in order to give continuity to the fiber friction measurement program.

LIST OF REFERENCES

1. R. B. Belser and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 1, USDA-SURDD Grant No. 12-14-100-7661(72), August 1965.

2. R. B. Belser and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 3, USDA-SURDD Grant No. 12-14-100-7661(72), August 1966.

3. Thomas E. McBride, "Development of an Instrument to Measure Friction of Textile Fibers," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, (August 1965).

4. Donald H. Gunther, Jr., "An Evaluation of Fiber Friction at Low Normal Forces," Thesis for the M. S. Degree in Textile Engineering, Georgia Institute of Technology, (December 1967).

5. Larry B. Whitworth, "Alteration of Physical Properties of Long Staple Cotton by Combing, Drawing, and Roving," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, (September 1967).

6. E. D. Cromer, "Change in the Physical Properties of Empire WR Cotton Fibers after Drawing, Roving, and Spinning," Thesis for the M. S. Degree in Textile Engineering, Georgia Institute of Technology, (December 1967).

7. H. Lamar Hicks, "Evaluation of Cotton Crystallinity by Infrared Fiber Press and Deuteration Methods," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, (December 1967).

8. R. B. Belser and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report Nos. 1-4, USDA-SURDD Grant No. 12-14-100-7661(72), 1965-1967.

9. H. R. Levy, "The Effects of Opening, Cleaning, Picking, and Carding on the Physical Properties of Empire WR Cotton Fibers," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, (September 1966).

10. H. J. Denham, VII, "The Structure of the Cotton Hair and It's Botanical Aspects," ii, The Morphology of the Wall, Shirley Institute Memoirs II, pp. 61-88, (1923).

11. F. P. Bowden and D. Tabor, "Friction and Lubrication of Solids," Oxford University Press, Cambridge, 1964.

LIST OF REFERENCES (Continued)

12. James P. Bryant, "An Investigation of the Factors Which Influence the Frictional Properties of Textile Fibers," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, (September 1966).

13. H. J. Marrinan and J. Mann, Journal of Applied Chemistry 4, (1954).

14. V. J. Frilette, J. Hanle, and H. Mark, Journal of American Chemical Society 70, p. 1107, (1948).

15. J. W. Rowan and E. K. Plyler, Journal Research National Bureau of Standards 44, p. 313,(1950).

16. H. G. Higgins, C. M. Stewart, and K. J. Harrington, Journal of Polymer Science 51, p. 65, (1961).

17. H. J. Marrinan and J. Mann, Transactions of Faraday Society 52, p. 89, (1956).

18. O. Sepall and S. G. Mason, Canadian Journal of Chemistry 39, p. 1934, (1961).

19. M. L. Nelson and R. T. O'Connor, Journal of Applied Polymer Science 8, p. 1325, (1964).

FINAL REPORT
GEORGIA TECH PROJECT NO. A-843

1 FEBRUARY 1965 TO 1 FEBRUARY 1968

FRICTIONAL PROPERTIES OF COTTON FIBERS

By R. B. Belser and J. L. Taylor

GRANT NO. 12-14-100-7661(72)
UNITED STATES DEPARTMENT OF AGRICULTURE

Prepared For
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SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
NEW ORLEANS, LOUISIANA

1 FEBRUARY 1968

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REPORT NO. 6 (FINAL REPORT)

FRICTIONAL PROPERTIES OF COTTON FIBERS

by
R. B. Belser and J. L. Taylor

Grant No. 12-14-100-7661(72)
United States Department of Agriculture

1 February 1965 to 1 February 1968

Placed By
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Agricultural Research Service
Southern Utilization Research and Development Division
New Orleans, Louisiana

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ABSTRACT

The purpose of this research is to measure the friction between contiguous cotton fibers and to evaluate the effects of the respective parameters of the fiber which establish its frictional characteristics.

An electrically operated servo-controlled force measuring instrument was developed and applied to the measurement of the frictional forces between crossed cotton fibers in the normal force range 1 to 40 mg. Special adaptations of the instrument were utilized to measure the friction of a fiber, using only the weight of the fiber as the normal force, and to extract a single fiber from a fiber tuft. The instrument and its accessories furnished an electrical analog of the frictional forces to an X-Y plotter providing a graphic display of the forces. These data could be analyzed either automatically or subsequently by mechanical methods to give the desired frictional data.

The instrument was used to measure the friction of over one thousand fiber pairs, principally of Empire WR cotton, but the measurements included those of other cottons, Nylon, Orlon, Viscose, Dacron, silk, glass, and metal. The analog plots were used to determine the values of the kinetic coefficient of friction, μ_k, and of the static coefficient of friction, μ_s, and to study the character of the plot which was intrinsically related to the fiber material and to the individual fiber. The μ_k value was determined by use of the value of the average force obtained by integrating the data plot over a selected length (6 to 10 mm) of the fiber traversed. The μ_s value was defined arbitrarily as the average force of the ten highest sticks over a similar length. The ratio μ_s/μ_k was found to be a useful number which reflected changes of fiber character and experimental conditions.

The measured value of the friction of cotton fibers was found to be affected by experimental conditions controllable by the experimenter and by features intrinsic to the fiber. In the first category were fiber mounting tension, normal force between contiguous fibers, traversing velocity, relative humidity, fiber treatment or processing, and temperature history of the fiber. The effect of the ambient temperature was not

examined. In the second category were fiber size, cross-sectional shape, and longitudinal shape, including crimp, convolutions, reversals, or growth abnormalities. In short, any element which affected the area of the fiber to fiber contiguous zone of contact, or the time of continuous contact during a traverse, or the intrinsic physical properties or surface condition of the fiber affected the measured frictional coefficients. Measurements by various experimenters cannot be properly compared without duplication as nearly as possible of conditions which are controllable by the experimenter. In general, these conditions have not previously been delineated or controlled for cotton.

The principal properties of cotton which differentiate its behavior from that of a cylindrical fiber such as nylon is its normally smaller size dimension, its cross-sectional shape, and its longitudinal shape. The smaller size, the constantly varying area of the contact zone, and the intrinsic roughness established by the longitudinal shape, reduce the average area of contact and the time of contact during a traverse, resulting in lower measured values of its coefficients of friction than those of nylon. At the same time, the large interlocking asperities give many stick peaks of high force and energy. Whereas at 20 mg of normal force between crossed fibers μ_k and μ_s values may be 0.250 ± 0.050 and 0.48 ± 0.10, respectively, the free fiber may register numbers in the range > 25 or > 50 respectively. The μ_s value especially is accentuated by increases in traverse velocity, temperature cycle treatment, and humidity; and the stick peaks appear to be primarily responsible for the mode of fiber travel by fiber to fiber snagging and fiber clusters. Processing reduces the frictional parameter values by fiber straightening and fiber selection, but introduces frictional peaks by fiber damage. Straightening and selection appear to predominate over increases due to damage although a bias in measurement exists here because of problems in mounting severely damaged fibers.

If all polymeric fibers examined were normalized to the same size and to a cylindrical shape there apparently would be only small differences among them in basic frictional parameters in agreement fundamentally with work outlined by Pascoe and Tabor.[12] The frictional parameters now accorded each natural fiber are intrinsically established by the fiber's

natural size, shape, and surface texture. The relative significance of these factors and the manner in which they affect the friction of the cotton fiber have been delineated and compared to changes observed for nylon fibers of duokelion, quasi-triangular, trilobal, tetrakelion, and circular cross section. Fibers of cross-sectional shape other than circular generally exhibited smaller frictional parameters because of reductions of the contiguous contact zone and the relative time of contact during a fiber translation in contrast to those registered by a cylindrical fiber under similar conditions.

ACKNOWLEDGEMENTS

Appreciation is expressed to the United States Department of Agriculture, Southern Utilization Research and Development Division, New Orleans, Louisiana for its support of this work. The cooperation and advice of Mr. James N. Grant, the technical administrator for the project, was most gracious and greatly appreciated.

Support from the Agricultural Research Station of the United States Department of Agriculture in Experiment, Georgia in obtaining the cotton specimens and in measuring fiber distribution data of selected cotton specimens was of great value. In particular, the cooperation and advice of Dr. H. A. Peacock, Research Agronomist, was appreciated.

Sincere thanks are also expressed to each of the below named individuals for his contribution to this research effort:

Individual	Title*	Contribution
Mr. B. R. Livesay	Res. Physicist	Friction Measuring Instrument
Dr. J. A. Knight, Jr.	Res. Professor & Hd. Radioisotopes Lab.	Infrared Spectroscopy
Dr. R. A. Young	Res. Professor & Hd. Diffraction Labs.	Crystallinity of Cotton
Mr. J. W. McCarty	Res. Assoc. Prof.	Properties of Fibers
Mr. James L. Hubbard	Res. Physicist	Microscopy of Fibers
Mr. J. Conrad Meaders	Asst. Res. Engr.	Friction Measurements
Mr. Lester D. Dozier	Asst. Res. Scientist	Friction Instrumentation
T. R. Boys	Graduate Asst., Textiles	Cotton Specimen Characterization
James P. Bryant	Graduate Asst., Textiles	Cotton Fiber Friction
Hong ki Chin	Graduate Asst., Textiles	Fiber Friction
Edwin Dwain Cromer	Graduate Asst., Textiles	Effect of Cotton Processing
Harry W. Ellis	Student Asst., Physics	Cotton Crystallinity (x-ray)

*At the time of the contribution.

Arthur M. Goldfarb	Graduate Asst., Textiles	Effects of Ginning
Donald H. Gunther, Jr.	Graduate Asst., Textiles	Friction at Low Normal Force
Henry Lamar Hicks	Graduate Asst., Textiles	Cotton Crystallinity (Infrared)
Jonathan O. Huff	Graduate Asst., Textiles	Friction vs Fiber Shape
Donald Lee House	Graduate Asst., Textiles	Microscopy of Fibers
William E. Kirkland	Graduate Asst., Textiles	Infrared Spectroscopy of Fibers
Chin T. Kwon	Graduate Asst., Textiles	Fiber Friction
Howard R. Levy	Graduate Asst., Textiles	Effects of Cotton Processing
Thomas E. McBride	Graduate Asst., Textiles	Friction Instrument
Rick A. Porter	Graduate Asst., Textiles	Fiber Press for IR Specimen
Eugene V. Rushing, Jr.	Student Asst., Textiles	Spider Silks
Marvin P. Smoak	Student Asst., Physics	Infrared Spectroscopy (Fiber Press)
Ken W. Stephens	Student Asst., Physics	Infrared Spectroscopy (Deut. & Crystallinity)
Joe Taylor	Student Asst., Physics	Friction of Metals
Clyde E. Turner, Jr.	Student Asst., Physics	Friction Measurements
Larry B. Whitworth	Graduate Asst., Textiles	Effects of Cotton Processing

Sincere thanks are also expressed to Mrs. Patricia Hambrick for her many endeavors in the preparation and typing of this manuscript and of several associated theses, and to Dr. W. L. Hyden, Professor of Textile Engineering now retired from the Georgia Institute of Technology,* whose enthusiasm and interest was of great assistance in the early stages of this work.

*After retirement (1966), Dr. Hyden accepted an appointment as Head of the Department of Natural Sciences at the Baptist College of Charleston, S.C.

I. PURPOSE

The purpose of this research is to investigate the frictional properties of cotton fibers and to delineate the respective influences of the shape and of the surface texture of the fibers on the friction between contiguous fibers. The ultimate objective is to evaluate the relative influences of crimp, convolution, cross-section, surface texture, and surface condition of a fiber on the friction of the fiber and to relate these parameters to the behavior of the fiber during the various processing stages from the cotton boll to the yarn.

II. INTRODUCTION

When this work was begun (1965), a method of measurement of fiber friction had not yet been selected and relatively small amounts of data on the friction of cotton fibers existed. However, in the interim period, two instruments have been developed here and quantities of fiber friction data have been collected. The developments have been reported extensively in Semiannual Reports Nos. 1 through 5 of this Grant and in twelve theses for the M. S. Degree in Textile Engineering or Textiles, submitted to the United States Department of Agriculture as they were completed. The voluminous nature of the data does not allow a complete recapitulation of them in this report but particularly pertinent sections will be summarized and fitted into appropriate positions in the report structure.

Concurrently, developments in work conducted under the leadership of Dr. K. L. Hertel of the Agricultural Research Station at the University of Tennessee and under Dr. W. J. Lyons and Frank Scardino of the Textile Research Institute have paralleled our work and made available data from other methods of fiber friction measurement which have aided in the interpretation of the fiber frictional data procured by us.

This report will present the frictional measurement results as they now exist and interpret fiber frictional behavior during processing in terms of these and other supporting data.

III. DEVELOPMENT OF AN INSTRUMENT FOR MEASURING FIBER FRICTION

A. THE NATURE OF FRICTION

Friction may be defined as the force resisting the motion of matter across the surface of a solid. In this work we are concerned principally with friction between surfaces of solids.

The existence of friction has been recognized by man since his very earliest stages. Its effects have aided and hindered him in his pursuit of life and control of his environment. Early developments included fire-sticks, lubricants, and wheel and axle bearings which go back to the dawn of recorded history. Considering the size of many early architectural achievements it is apparent that more understanding of the control of friction was practiced than our records indicate.

Leonardo da Vinci, in 1500 A.D., appears to have had an extensive understanding of the nature of sliding and rolling friction and proposed ball, roller, tapered roller, and tin-rich alloy bearings according to some recent copies of his notes found in the Madrid Codex (reported in Burlington Magazine [English], January 1968).

In studying the nature of friction, da Vinci,[1] Amontons,[2] and Coulomb[3] contributed to the presently accepted laws of friction for a macroscopic body moving across a second at a uniform velocity. These state essentially that:

(1) The frictional force, F, is proportional to the normal load, N, such that the coefficient of friction, μ equals F/N, remains constant for a given pair of bodies; and

(2) The frictional force is independent of the area of contact between the two bodies.

When the body is started in motion from rest, the force required to start it is greater than that to keep it moving. We thus have a static coefficient of friction, μ_s, and a kinetic one, μ_k, as pointed out by Coulomb.

The mechanisms of friction have been discussed by many authors. Early theories were based principally on the work of Coulomb,[3] who espoused a surface roughness theory based on interlocking of asperities. More recent work by Bowden and Tabor[4] and some of their associates have lead to a surface interaction theory which involves adhesion and welding over microzones. In fact, the works of Bowden and Tabor and their co-workers have treated most of the current frictional problems including fiber friction.

An analysis of the very early studies of friction shows that they dealt with the problem as related to friction between macroscopic zones of surfaces. To these, because of the relatively large area statistically, the general laws of friction developed by Coulomb appear to apply. However, if one of the respective zones is reduced essentially to a point, we may no longer follow these rules. Bowden and Tabor have best described the results here with a number of different approaches.[4] These have been further discussed by Howell, Mieszkis and Tabor at some length.[5]

It is evident that with fibers one is working with the friction of one or a few asperities and that the area of contact between the respective surfaces is of paramount importance to the frictional measurements. Reviewing some of the data provided by the cited authors we find that Howell used the expression $F = KW^n$ where n is less than unity (1953)[6] and W is the load. This expression was satisfactorily employed by

Lincoln[7] (1952) and Huntington[8] (1957) in their work.

If we adhere to the contact weld-shear theory of friction as expounded by Bowden and Tabor and the shear, s, is constant for a given substance, then the area, $A = k_1 W^n$. Lincoln found that the friction between a nylon sphere and surface was $F = kW^{2/3}$ in the range 1 gram to 100 grams. This is in agreement with Hertz's solution for the elastic deformation of a spherical surface. A measurement was also made optically for the sphere against a glass flat, verifying the validity of the exponent, 2/3.

Howell and Mazur[9] (1953) measured the friction between crossed fibers in the load range 0.3 to 400 mg and found the relation $F = kW^n$ where n was 0.8 for drawn nylon and 0.9 for undrawn nylon and 0.96 for cellulose acetate.

In some additional experiments using fibers wound about a cylindrical rod and pressed against an optical flat a value of $n = 0.73$ satisfied all fibers examined where the loading time was 15 seconds. Subsequent studies lead to the conclusion that for pure elastic contact the true area of contact is proportional to W^n where n lies between 2/3 and 1.

A further analysis made for a sphere on a plane surface has been made by Lodge and Howell[10] and leads to the conclusion that the area of contact was proportional to $W^{8/9}$ and $R^{2/9}$ where R is the radius of the sphere.

Using a principle outlined by Tabor,[11] this work was extended to a study of a hard spherical indenter against a plastic surface. The area was shown to vary as $W^{0.74}$ for nylon and as $R^{0.52}$ where R is the radius of the sphere. From these data Pascoe and Tabor[12] assumed that for

crossed cylinders

$$F = C_1 \, s \, W^{2/m} \, D^{-2(2-m)/m} \quad ,$$

where m is the slope of the plot log W versus the diameter of the impression of the spherical indenter discussed by Tabor. For undrawn nylon m = 2.7 and

$$F = C_1 \, s \, W^{0.74} \, D^{0.52} \quad .$$

Then

$$\mu = C_1 \, s \, W^{(2-m)/m} \, D^{-2(2-m)m} \quad ,$$

dividing by W^m to incorporate the area variable

$$\mu = C_1 \, s \, W^{-0.26} \, D^{0.52}$$

for crossed nylon fibers where m is 2.7 and C_1 is 1.4. The validity of these expressions was confirmed from data measured with the apparatus described in Section III.B.

It is evident from this discussion and the data presented subsequently that in fiber friction we are dealing with point contacts of small size. Hence, we are working in a zone where the area of contact is very important with respect to the frictional force measured. Since it has been shown that the frictional force measured varies both as a function of the normal force applied and the radius of the fiber it is evident that data can only be compared by maintaining these features constant or by extrapolation using the expression provided by Pascoe and Tabor[12] to fit the material used.

Unfortunately, variables in textile fibers, especially cotton, cannot always be controlled properly nor can fibers be procured often in a desired size, shape, or purity. Thus, much of the data in this research

was obtained without the capability of control of the radius of curvature (or size) variable and, some control was lacking during early measurements due to lack of realization of the importance of the size factor. Where comparisons bringing in the size factor have been possible they have been discussed in relation to the preceding paragraphs of this section.

B. APPARATUS USED BY OTHER INVESTIGATORS

A search of the literature of devices for the measurement of fiber friction was conducted. This revealed that successful instruments were based principally on four concepts. These were: (1) the fiber bundle method, (2) the fiber twist method, (3) the torque principle, and (4) a stick-slip technique described by Bowden and Tabor.

Adderley[13] in 1922 described a fiber bundle method in which two bundles of aligned fibers were mounted on glass and pressed together with a single fiber pressed between the two bundles. The single fiber was extracted and the force for its extraction was measured. This measurement was performed for many species of cotton and for many different fiber lengths. A relation between the force required to obtain slip and convolutions per unit length was obtained. However, no measurement of the coefficient of friction as such was determined.

In the fiber twist method two fibers are twisted together a known number of turns. One fiber is connected to a fixed arm and the second to a movable one or to a variable weight. The force to remove the movable fiber from the pair is measured or the number of twists required to prevent its removal is determined. B. G. Hood[14] and J. Lindberg and N. Gralen[15] have described instruments using this principle. Although a number correlating

to frictional differences was obtained in each case the translation of this to a coefficient of friction was not attempted.

An instrument capable of giving an analog plot of frictional forces was desired for this work. Mercer and Makinson[16] used the principle of Bowden and Leben[17] to record an analog of frictional forces exerted as a fiber, supported by a bow, was traversed across a second. The second fiber was supported by a second bow which was attached to a small section of clock spring to which was affixed a small mirror. The traversing fiber was supported by a balance arm of light construction and suitably pivoted on jewel bearings. The normal force was applied by the repulsion of a magnetized needle, attached to the balance arm and suspended as the core of the solenoid, when a suitable current was supplied to the solenoid. The balance arm was damped from free oscillation by a vane suspended from the pivot point and immersed in oil. The arm assembly was moved for traversing by a hydraulic mechanism driving the entire assembly along a horizontal path perpendicular to the stationary fiber.

When a friction measurement was made at traverse rates of 0.01 mm/sec to 0.1 mm/sec, a light beam impinging on the mirror was deflected in accordance with the frictional force applied by the traversing fiber to the fiber attached to the spring. The trace of the reflected light beam was recorded on a moving photographic film.

This instrument employed a friction analog recording system, a magnetically applied normal force, a balance-beam damping system, and a hydraulic drive mechanism for translating the beam assembly. All of these were extremely useful features.

Mercer and Makinson measured the friction coefficient of wool fibers

in both directions (tip-to-root, root-to-tip) and the effect on friction of varying the normal force over the range 10 mg to 200 mg. The friction coefficients at 20 mg were approximately 0.56 and 0.90 with and against the scales respectively and increased rapidly as the normal force was reduced. At 200 mg, the values were 0.23 and 0.45 respectively.

An application of the torsion method to friction measurement is described by J. C. Guthrie and P. H. Oliver.[18] In this apparatus a fiber was mounted on one end of a horizontal arm extending perpendicularly from a vertical torsion wire. A mirror in a vertical plane was affixed to this assembly near the axis of the wire. A second fiber perpendicular to the first, with both fibers in a horizontal plane, was dragged across the first by a horizontal arm rotating about a vertical pivot offset some distance from the axis of the torque suspension. The load between the fibers was adjusted by a weight placed on the arm attached to the torque wire.

When the second fiber was drawn across the first, the torque displacement was measured by the angular displacement of a light beam reflected from the mirror. The frictional force was equal to the restoring torque exerted by the wire at any moment and could readily be calculated from the known displacement of the arm and other parameters of the apparatus. With this apparatus values representing both static and kinetic friction were obtained. The results obtained by the authors were expressed in graphic form of a plot of essentially the frictional force versus the force exerted in a normal direction between the surfaces of the fibers. From these data values obtained for the coefficient of friction of rayon were calculated to be 0.22 for the static coefficient of both 1.5

and 3 denier rayon and 0.19 and 0.21 respectively for the kinetic value when normal forces of approximately 125 mg were used. The angle between the axes of the fibers in a horizontal plane could also be varied and data was presented over the range 10° to 90° included in the acute angle. However, results were inconclusive as to a change in the coefficient of friction with respect to a change in the angle between the fibers.

The fourth frictional method of interest is that described in Bowden and Tabor[4] and previously described in a paper by Pascoe and Tabor.[12] In this case, a polymer fiber or rod is suspended horizontally and a fiber of similar material, mounted on a small driven carriage, is traversed across the under surface of the other fiber near its end. The upper fiber is pressed down on the lower fiber thus flexing the upper fiber in a vertical plane. The load may be determined from the bending of the upper fiber and the frictional force from the deflection of the end of this fiber in a horizontal plane as the lower fiber is dragged across it. Using this equipment the coefficient of friction of many polymer fibers were determined over a very large load range. Values for nylon, polythene, teflon, and PVDC were obtained over the load range 10^{-6} grams to 10 grams, or a total range of 10^{7}. Variations in the coefficient of friction, μ, were found to occur with load. The expression of $\mu = kW^{-B}$ was suggested as the correct one for the data found, where B was in the range 0.2 to 0.3 and k is an undesignated constant. This was shown from a plot of log μ versus log load, the slope of the resulting curves being negative and in the suggested range.*

For a nylon fiber of 0.042 mm in diameter (0.0016") and a load of 10

*See reference 4, p. 227.

mg, the coefficient of friction was found to be approximately 0.5. At lesser loads of about 1 mg it became > 1 and at greater loads of about 100 mg it decreased to < 0.4.

C. THE DESIGN OF A SERVO-CONTROLLED FIBER FRICTION APPARATUS

1. Introduction

Based on the preceding studies and an investigation of other references listed in the Bibliography, the torque method similar to the one described by Guthrie and Oliver[18] appeared to be the principle best adapted to the task of measuring the friction between cotton fibers. Features of the instrument of Mercer and Makinson[16] also appeared to be desirable. Because of the many measurements to be made and rapid variations in the characteristic stick-slip relations it appeared that an electrical recording method should be incorporated into the equipment to facilitate data collection. Mr. Billy R. Livesay, a research physicist on our staff, suggested that a servo-mechanism designed by him for measuring small torques on metal film specimens placed in a magnetic field might be adaptable to the measurement of small torques applied to one fiber by a second traversing across it. An examination of the apparatus then available revealed the feasibility of this suggestion, and material was obtained for constructing an apparatus for measuring fiber friction. Its design is outlined below.

2. Design of the Original Apparatus

The frictional forces between contiguous individual cotton fibers when one is placed in motion are a few milligrams for normal forces of the same order of magnitude. The microscopic nature of surfaces

is such that highly non-uniform forces are experienced when a fiber is drawn across a stationary object, which may be a second fiber. An instrument responsive to rapidly changing forces is therefore required to obtain accurate information about the resulting frictional characteristics of two such surfaces. An electromagnetic servo-system with the required sensitivity and response characteristic has been adapted to make these measurements. The principle of this system[19, 20] has been used in other types of physical measurements, but it is believed that this is the first time it has been applied to the measurement of frictional force. The initial instrument was constructed by J. McBride[21] under the tutelage of Mr. Livesay.

A D'Arsonval galvanometer of the type used in Honeywell portable potentiometers was found to be quite suitable for this application. A fine gold ribbon suspension provides a frictionless pivot about the vertical axis, but is sufficiently rugged to support the loads used in these measurements. A mirror is fixed to the lower end of the coil and a pointer, extending horizontally about two inches, is attached at the top of the coil. Forces applied horizontally and normal to the pointer produce a torque about the pivot axis. This torque may be balanced by an electric current of the proper magnitude and direction in the coil to produce a counter-torque which prevents deflection of the pointer. The magnitude of the applied force is thereby calculable from the required current.

The short response time needed for the measurement of rapidly changing frictional forces is obtained by using a photoelectric device to detect small deflections of the galvanometer from its null position. A tiny beam of light reflected from the galvanometer mirror onto a dual

photodiode generates a differential emf when the light beam is deflected to illuminate one section of the diode more than the other. The output of the photodiode is then fed into a high gain DC differential amplifier which in turn supplies the required correcting current to the galvanometer coil.

The first apparatus constructed by us for measuring fiber friction consisted of a mechanical balance and fiber driver, the galvanometer and servo-system, and an XY plotter. A close-up view of the fiber driving mechanism and galvanometer is shown in Figure 1.

The fiber driver consists of a slender, tubular, balance arm supported by a steel shaft and sapphire bearings and rotated about a vertical axis by a shaft immediately beneath and attached to the bearing support. The vertical shaft is rotated by a synchronous motor through a reduction gear system at a rate of 1/75 rpm or at another selected rate. The fiber to be studied is suspended horizontally and normal to the shaft in an adjustable holder under a tension of about 425 milligrams at one end of the balance arm. Normal force adjustments are made with a micrometer type screw sleeve at the other end of the balance arm. Settings were calibrated with an accurate balance system. The length of the balance arm from the pivot to the fiber was 10 inches so that for fiber lengths of one inch or less the variation of position of the applied force on the lower arm of the galvanometer pointer during one traverse was very small.

A second fiber (or other material) is mounted on the galvanometer pointer in a bow type holder fastened to the pointer by sealing wax or a cement. The holder supplies sufficient tension to maintain a taut suspension. Most of our early measurements with this instrument were made with

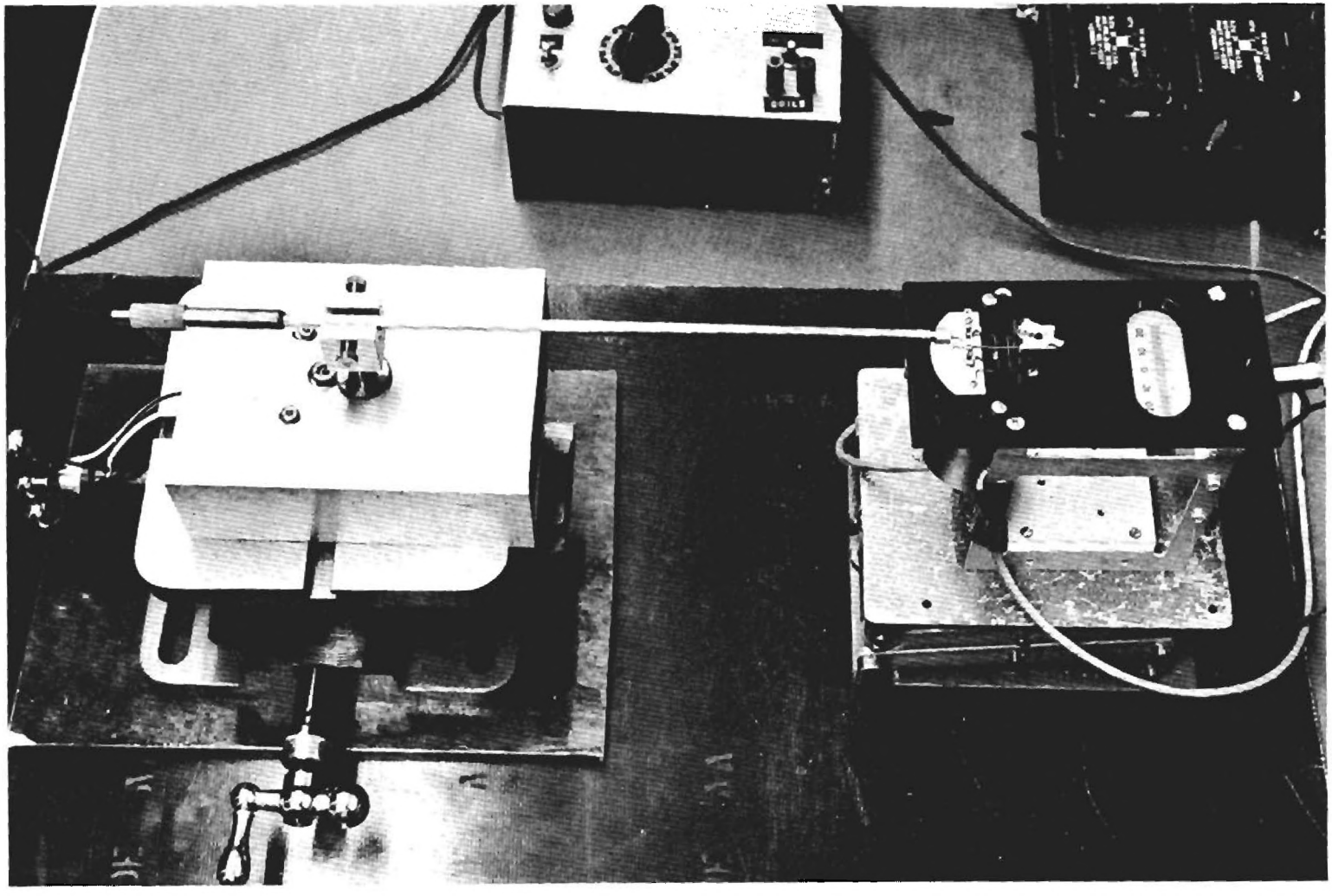


Figure 1. Fiber Drive Mechanism and Servo-Controlled Galvanometer.

balance arm shaft speed of 1/75 rpm which means the fiber moved about 0.8 inch per minute (0.33 mm/sec).

Other comments pertinent to the instrument are noted below. The light source is a No. 80 grain of wheat lamp and the diode is a TI No. LS 221 null indicator. The servo-amplifier is a Burr-Brown Model 1509 differential amplifier. The instrument is calibrated by rotating the galvanometer from a vertical to a horizontal position and hanging standard weights from the pointer at the point of normal fiber contact. Calibrations were made in both possible directions and corrections were made for the needle weight.

3. Modification of the Original Apparatus

The original apparatus was disassembled after six months use to replace the fulcrum pin and bearings. This arrangement consists of a hardened steel pin (No. 33 pocket watch staff) mounted in sapphire bearings. The original pin was found to be scarred and the bearings and shaft were replaced. The bearings were made demountable in order that this arm or another with the same suspension configuration could be used to replace it.

Two additional arms were constructed. One was constructed of 1/8" aluminum rod, 29 cm long, suspended at a position 23.9 cm from the fiber holder. The short end had a brass counterbalance affixed to it and the long end was indexed to accept a wire rider for adjusting the normal force. The second similar lever of 3/16" rod was 31.8 cm long suspended at a point 22.9 cm from one end. The masses of the three arms were 74.2 grams, 39.6 grams and 23.4 grams respectively and the moments of inertia about the pivot were calculated to be approximately 12,000, 3,000, and 1,150 gm cm^2 respectively. The arm with high inertia was found to give more consistent results and was selected for use in making the required

friction measurements.

A new fiber holder was also constructed of a brass block with a groove in each end for aligning the fiber. The span length of this holder was only 0.5".

A stand was constructed for a chainomatic balance, as shown in Figure 2. This places the balance directly over the fiber-holding end of the respective frictional arms. A chain suspended from a counter-weight, replacing the left pan of the balance, extends down precisely to the level of the balance point of the respective lever arm where it can be engaged with a hook provided on the arm. A close-up view of this arrangement is shown in Figure 3. Repeatability of the normal force setting is ±0.0002 grams with this arrangement. Normal forces were generally measured before and after a frictional measurement and the average value used as the normal force. Changes of a few tenths of a milligram were usually experienced.

4. Low Normal Force Fiber Friction Apparatus

Using the same principles of the friction apparatus described previously a new apparatus for measuring friction at low normal forces was developed. The new instrument is basically the same as the original device but design improvements allow important parameters to be varied remotely and the low force sensitivity has been increased. The normal force, or load, may now be varied continuously between about 2 and 25 mg to an accuracy better than 0.1 mg. The drive speed may be set at any value desired and is reversible.

The improved instrument exhibited in Figure 4 uses a simple

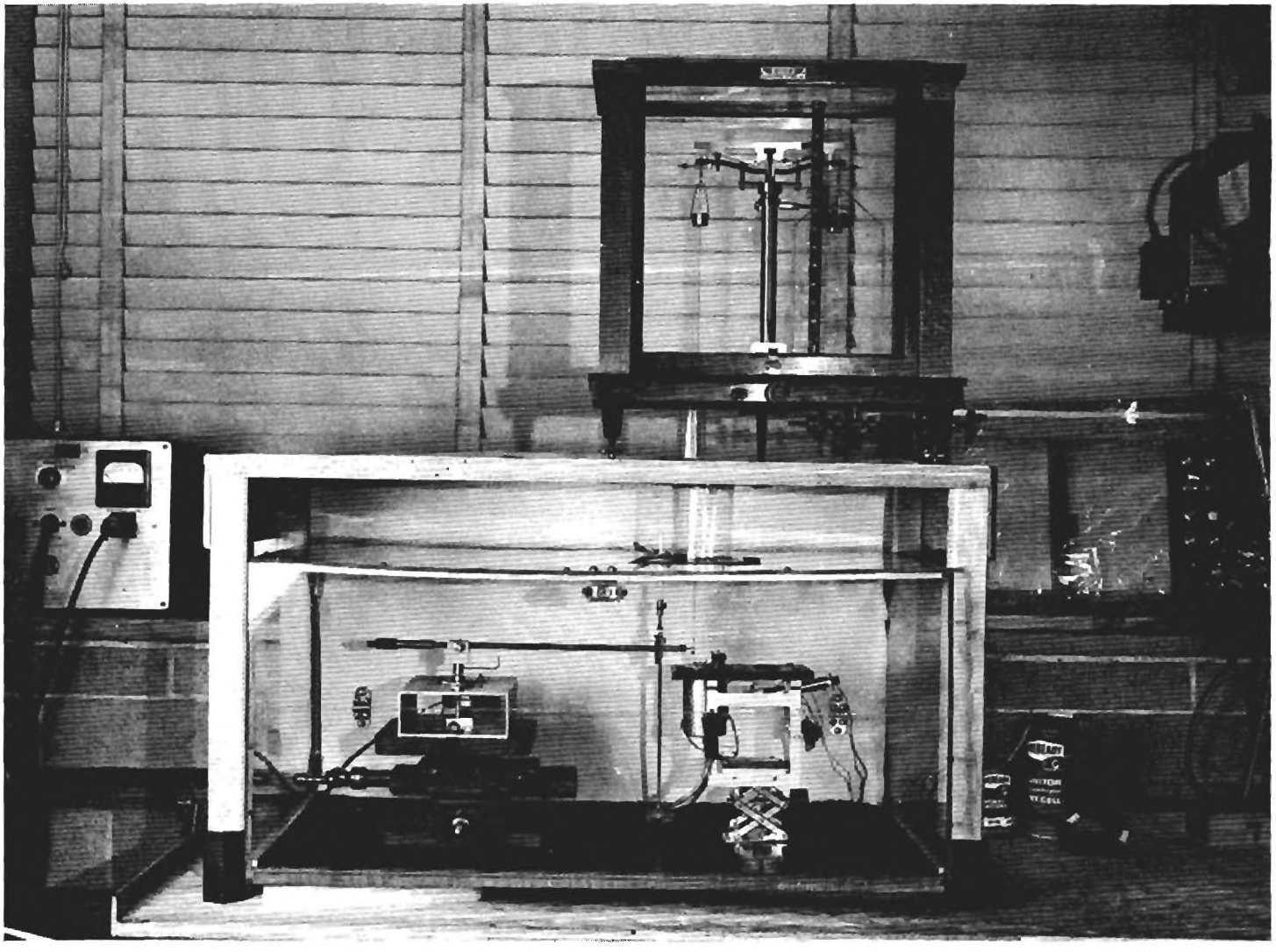


Figure 2. View of Friction Measuring Apparatus and Chainomatic Balance for Measurement of Normal Force.

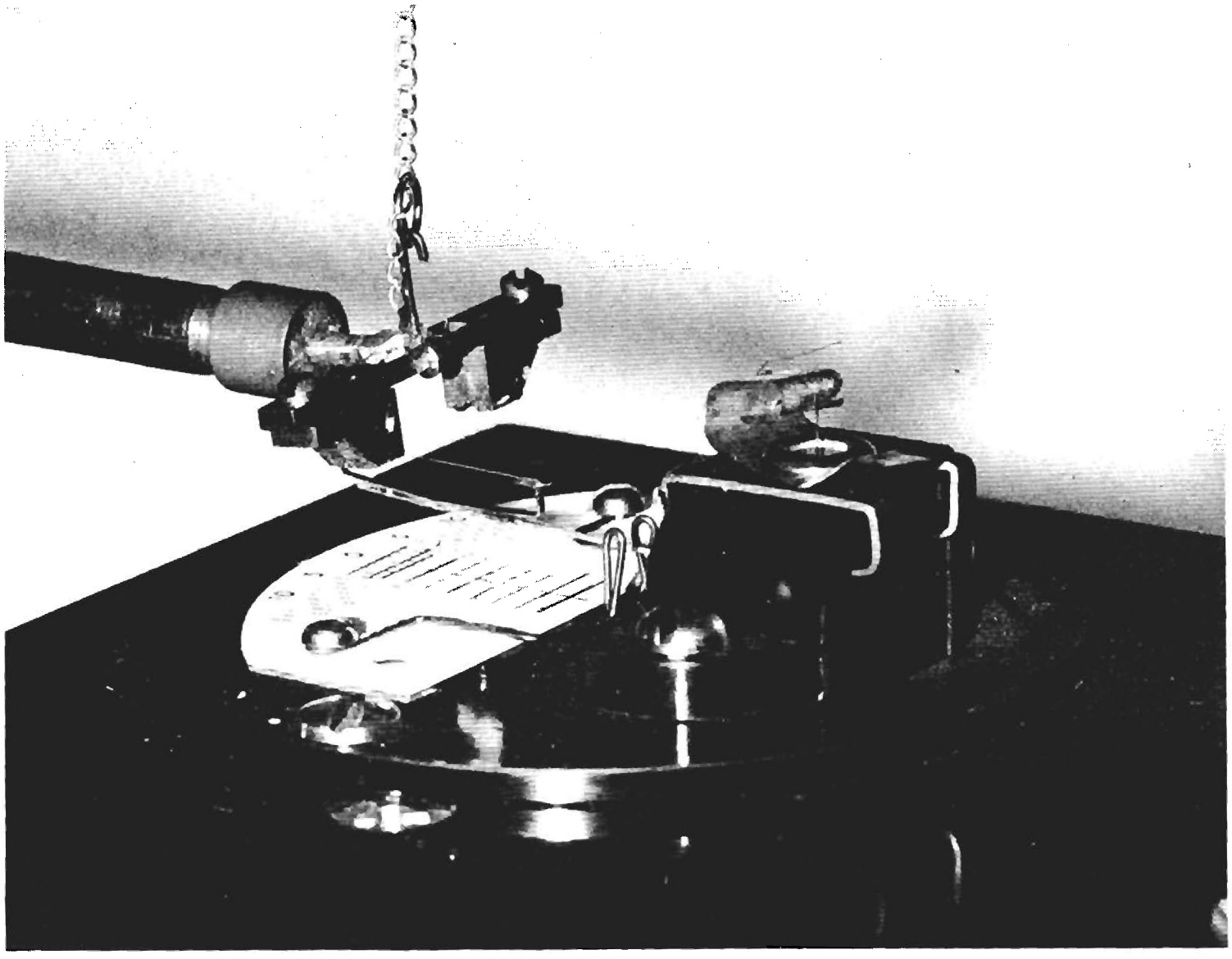


Figure 3. View of Cross Fiber Arrangement and Support Chain to Balance.

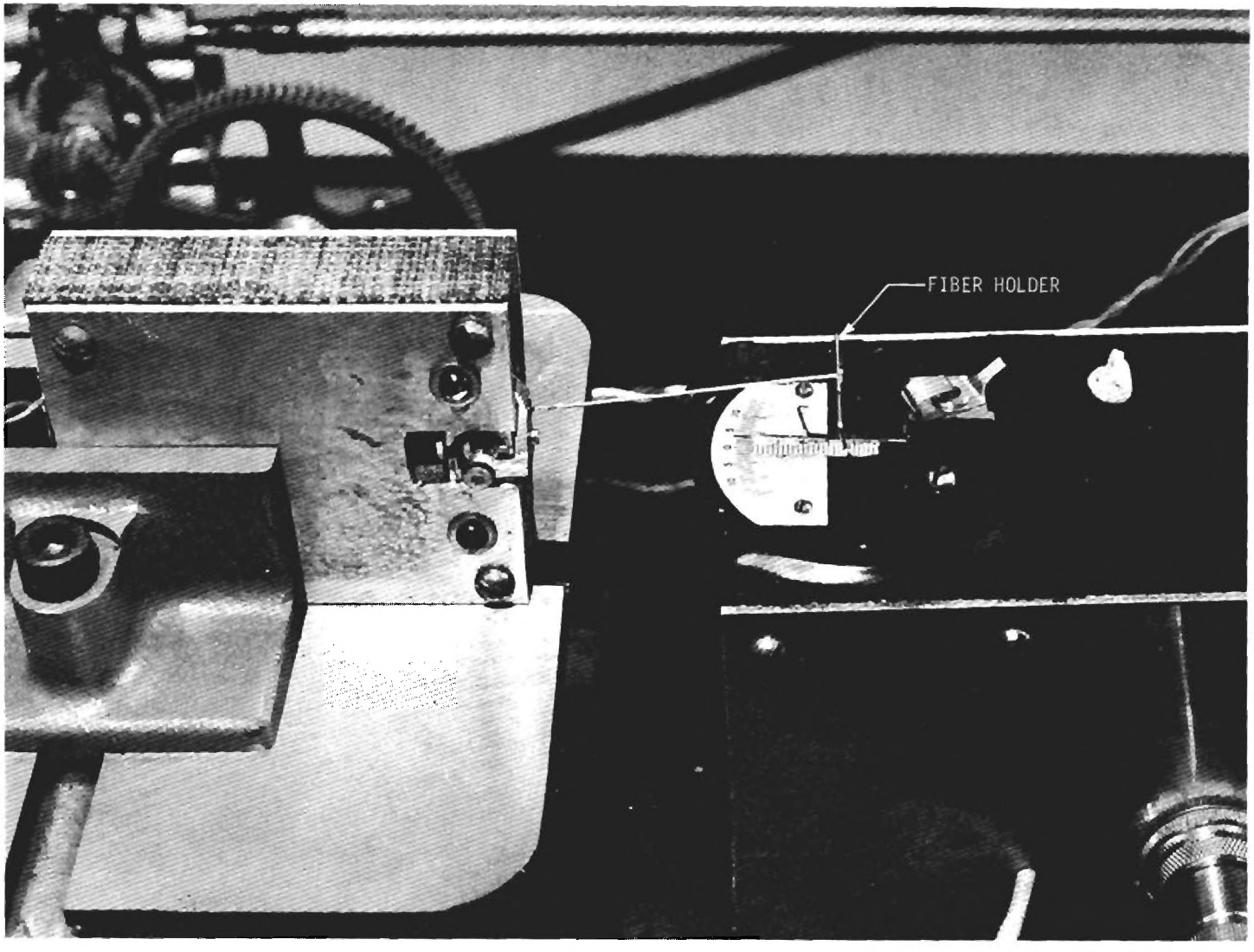


Figure 4. Instrument to Measure Frictional Force at Low Normal Forces, Electromagnetically Applied.

electromagnetic system to apply normal force between the fibers. A sensitive but rugged meter movement was mounted on a milling vise with the rotational axis of the meter in the horizontal plane. A short, light arm with a fiber holder at one end was constructed from non-magnetic cupro-nickel alloy tubing and attached to the coil of the meter movement. A second length of tubing attached to the opposite side of the coil is used as a rough counter-balance adjustment. Fine balancing is obtained with the meter's normal zero adjustment mechanism. With the meter mechanically balanced, a current through the coil will produce a force at the end of the fiber holder directly proportional to the coil current. Calibration of the loading element is conveniently and precisely accomplished by hooking a previously weighed fine wire segment over the fiber and reversing the current through the coil. We find our reproducibility of normal loads to be better than ± 0.1 mg and the load-current curve is linear over the range of interest, 2 to 20 mg. We are therefore able to use the applied current as an electrical signal which is proportional to the fiber load. The construction of the meter movement is such that small tendencies to "bouncing" at low normal force levels are quickly damped.

The loading device with the traversing fiber attached is driven by a variable speed motor, with the helical screw arrangement of the milling vise support and reduction gears to provide any desired linear fiber drive rate.

The drive direction may be reversed by reversing the motor. A tachometer mounted at the motion output is used to give an electrical signal proportional to the fiber drive speed.

This apparatus has made it possible to obtain reliable fiber friction

data for normal force values as low as 2 mg. For a typical fiber driven at a known velocity in contact with a second on the sensing arm, the XY recorder plots directly data concerning the frictional force versus the normal force. Data over a range of selected drive speeds, and with the drive reversed may easily be procured.

A further improvement to the instrument to incorporate an integrator was made by Gunther,[22] and subsequently the entire instrument was overhauled and improved by Huff.[23] These modifications are described in detail in the respective theses. The principal modifications by Huff were to improve integration and calibration and to establish precision of operation. As a result, a fiber could be traced in a forward direction, then reversed, reversing at the same time to a -X, -Y direction on the plotter, thus giving almost a mirror image of the forward traverse friction curve. This enabled one to study directional effects of friction in fibers and viscoelastic flow within the fiber. The apparatus as employed by Gunther[22] is exhibited in Figure 5.

D. FIBER MOUNTING PROCEDURE

Several types of fiber mounts were used. These generally were "U" type mounts of beryllium copper or cupro-nickel tubing, applied to the servo-controlled galvanometer needle or to the low normal force instrument, and of machined brass for attachment to the moving balance arm of the initial instrument. The span of the fiber was 0.5" for the machined brass unit and somewhat more for the tubular supports.

The fibers required a magnifying lens or stereomicroscope for observation during mounting. An end of the fiber was cemented to one arm of the mount; the cement was dried; and the mount placed with its

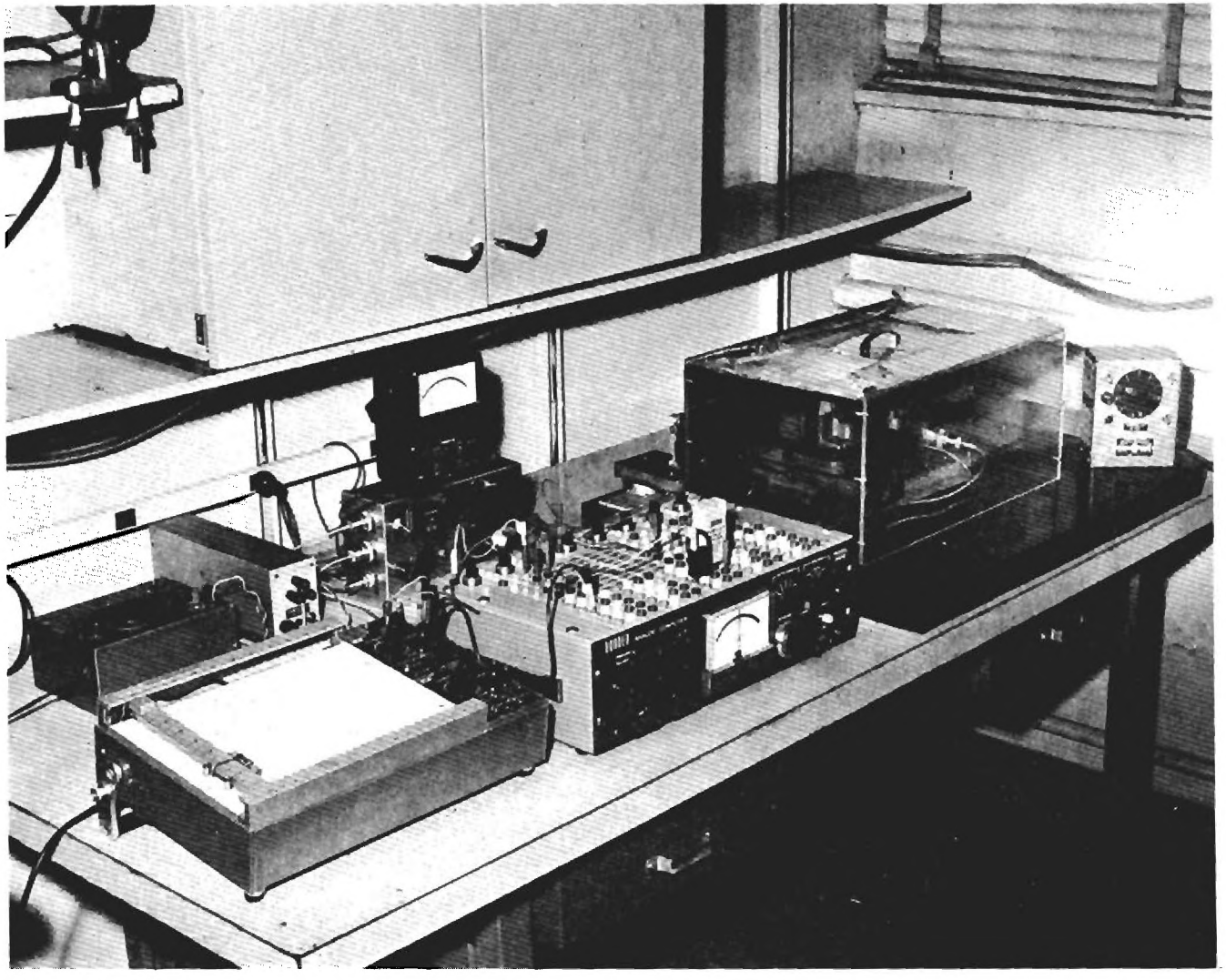


Figure 5. Fiber Friction Apparatus as Employed by Gunther.

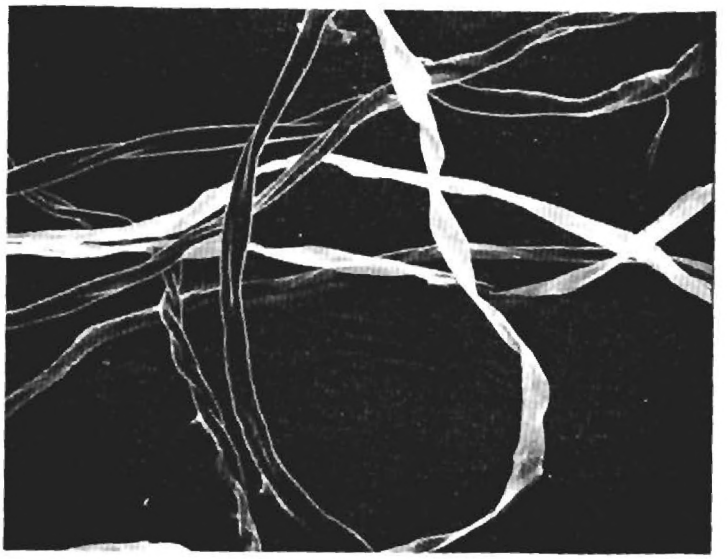
axis vertical. A small weight (425 mg usually) was attached to the free end of the fiber. The fiber and mount were adjusted to obtain suitable contact at the unfastened end and cement was applied. Sealing wax was used in some of the early measurements but Duco cement was employed subsequently because heat applied during sealing was found to introduce undesirable variables. In addition, maintaining a constant known fiber tension was found to be important.

The removable mounts for the galvanometer needles were attached to the respective needles by sealing wax or cementing. All of the final phases of the work employed Duco cement. The mounts on the driven members were attached mechanically to the balance beam.

E. THE FRICTIONAL DATA AND ITS INTERPRETATION

With the instruments described, analog data plots of the frictional forces encountered in traversing a single fiber across a second at right angles to it are readily obtained. By calibration of the instrument with respect to the plotter the frictional forces may be determined from the plot. The normal force between the fibers may be preset at a desired value over the range of about 2 to 60 mg. Although only the range 2 to 20 mg is presently feasible with the electromagnetically loaded instrument.

Scanning electron micrographs of cotton fibers of types identified as high draft and low draft are shown in Figure 6. These exhibit the complex convoluted ribbon character of the cotton fiber. Typical data plots for fiber pairs of cotton, rayon, and nylon, respectively, are shown in Figure 7. It will be noted that each curve presents data indicating a stick-slip frictional process and reflects in part the character of the cotton fiber. The frequency of the stick-slips is



(a) High Draft (180X)

(b) Low Draft (200X)

Figure 6. Scanning Electron Micrograph of High and Low Draft Cotton Fibers at Low Magnification (180x, 200x).

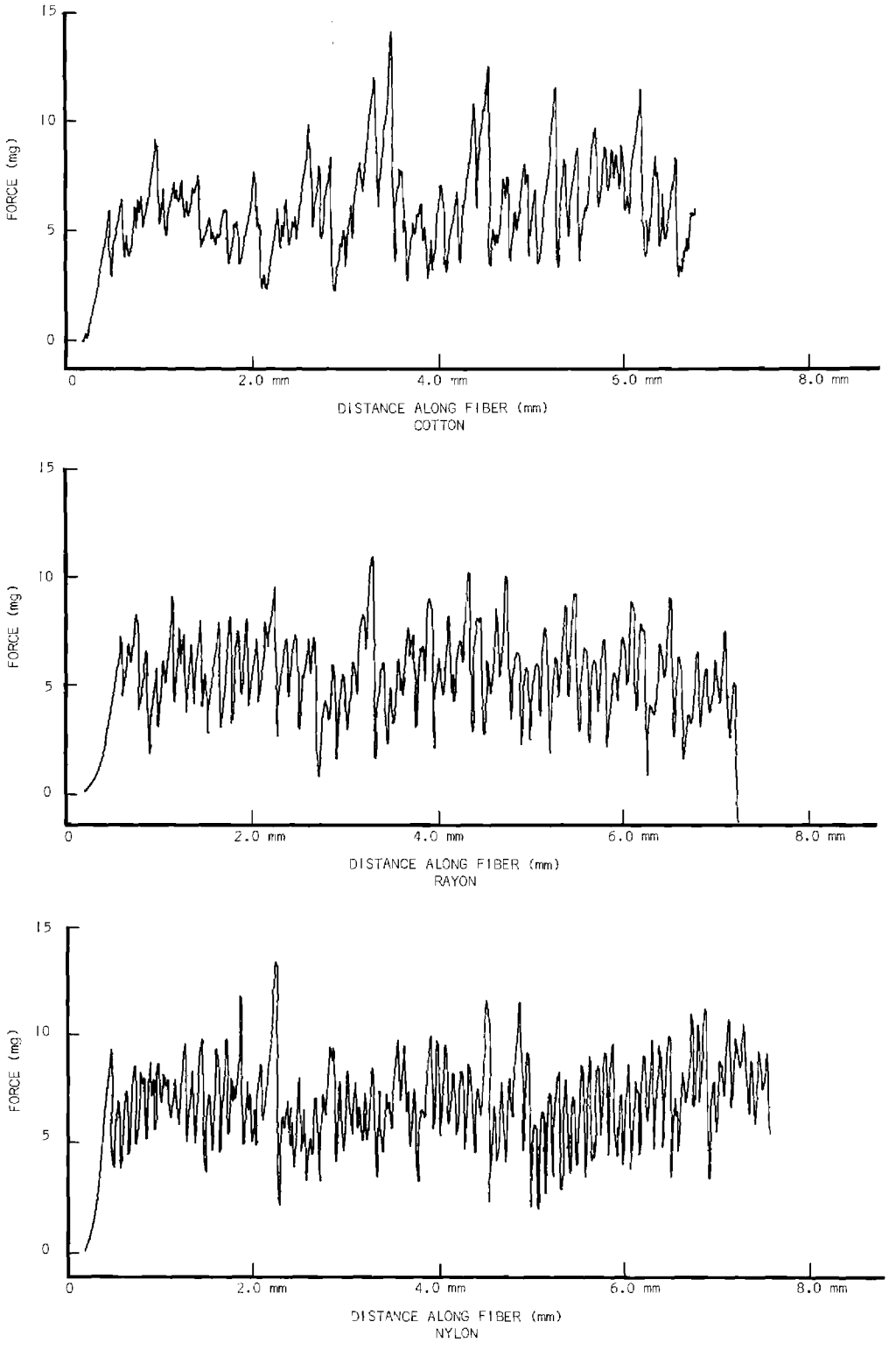


Figure 7. Typical Frictional Data Plots of Cotton, Rayon, and Nylon Fibers Showing Character Exhibited by These Fibers (20 mg NF).

greater for nylon and rayon than for cotton. Hence, we immediately perceive that the curve has a specific character intrinsic to a given fiber material.

For clarity in understanding the frictional data, examine Figure 8. If one integrates the area under the curve with a planimeter or an electronic integrator one can determine the average ordinate displacement which is proportional to the frictional force. By use of the calibration constants of the instrument and plotter combination, the frictional force may be calculated. The kinetic coefficient of friction,

$$\mu_k = \frac{\text{Frictional Force}}{\text{Normal Force}} \quad .$$

The static frictional force is defined as the force which must be overcome in order to start an object at rest in motion across the surface of another object. In the data plots just examined it is evident that point frictional contacts slide in a stick-slip manner, i.e., continuous sliding does not normally occur for a point contact but sliding takes place as a series of small sticks and slips. The static coefficient, then, must be represented by the maxima of the curve and the average of these would give its value.

In fiber friction for essentially free fibers, however, the fibers may cling to each other at a single high stick and travel together for some time before being pulled apart. Hence, the high sticks must be important. In our work we have arbitrarily taken the ten high sticks for the traversed zone usually, 6 to 10 mm on the fiber, as a measure of the static frictional force for a given fiber. Thus, we have defined the coefficient of static friction for our purposes as

$$\mu_s = \frac{\text{Static frictional force determined from ten highest sticks}}{\text{Normal force}} \quad .$$

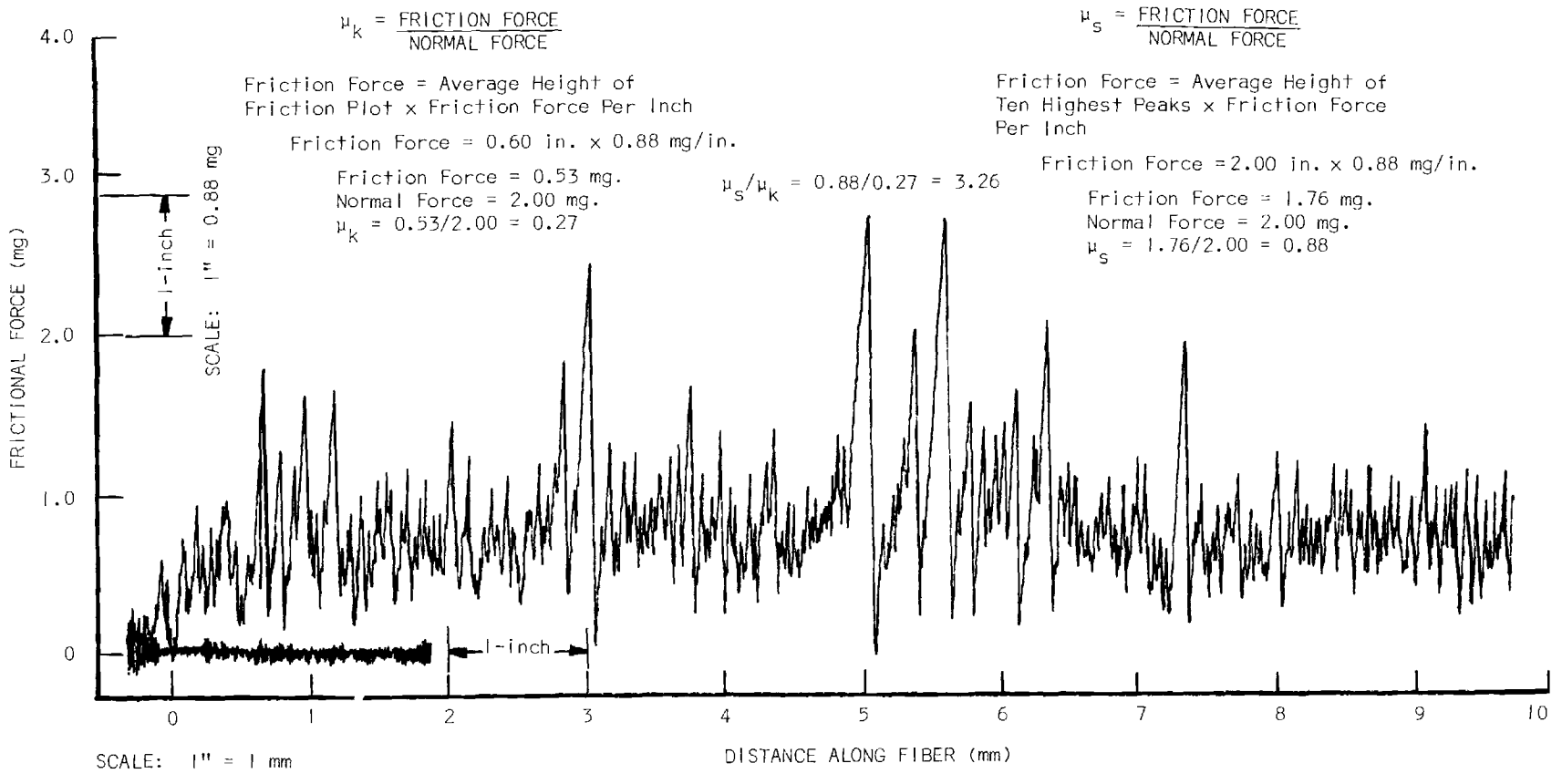


Figure 8. Typical Data Plot of Cotton on Nylon at Low Normal Force (2 mg) Showing Calculation of μ_s, μ_k, and μ_s/μ_k.

It may be shown that, if all the static peaks are used to determine the μ_s value, it approaches the value of μ_k as the frequency of the stick-slips increased.

Other investigators have used the average of all the maxima, or the average of the half-heights between all the maxima and minima as a measure of the frictional force. It may be shown on any one data plot that each of these methods give a somewhat different value of the frictional force dependent on the material being measured. These values would normally be higher than those obtained by integration of the curve for the average frictional force but lower than the static frictional force determined from the ten higher peaks.

One of the problems in comparing our frictional data with those of previous investigators is the variation of the method employed by each for obtaining the frictional force. However, the integration method satisfies the equation, work equals force times distance, and is the only one that does. Therefore, it is the correct method.

In frictional contacts of macroscopic areas many points are undergoing stick-slip cycles simultaneously but out of phase. Because of the large number of points and the phase differences of each stick slip cycle, a statistical average value of kinetic friction is normally obtained. Similarly with fibers if we integrate frictional force over a sufficient length of fibers or a sufficient number of short lengths we will get an essentially macroscopic value of friction for a fiber of the type selected.

If we now reconsider the values of μ_s and μ_k for fibers, as employed by us and as defined previously, we will find that a ratio μ_s/μ_k may be established which appears to be fairly characteristic of a fiber type.

We also find that its value varies with normal force and other conditions of the fiber. Hence, it may also give information of value. It has been employed by us as a parameter of interest in interpreting frictional data of fibers. Interpretations assisted by its use will be discussed subsequently.

IV. FIBER MATERIALS EXAMINED

A. INTRODUCTION

Although the principal emphasis during the course of this research was on cotton, it was soon discovered that the variable shape factor of cotton introduced many difficulties in understanding its frictional properties. Therefore Nylon 6 of circular and of other cross-sectional shapes was obtained. Concurrently, with the assistance of student assistants performing special problems assignments or graduate student theses, we were able to obtain measurements of a number of other fiber materials. All of these studies contribute to an understanding of fiber friction as related to cotton and are therefore reported in summary version with appropriate references.

B. SELECTION AND CHARACTERIZATION OF THE COTTON SPECIMENS

1. General

In conferences between the staff of the Textile School, members of the United States Agricultural Department, and through the cooperation of the Staff of the University of Georgia Agricultural Experiment Station at Experiment, Georgia, Empire WR was selected as a variety of cotton typical of the Southeastern Section of the United States.

2. Procurement of Cotton Specimens Nos. 1 and 2

The cotton selected consisted of two specimens: (1) a typical bale of Enpire WR picked in 1962 and selected for careful characterization by Mr. T. R. Boys, a graduate student at Georgia Tech[24] and (2) a similar specimen, gathered in 1964, on which procedures of growing, gathering, and ginning were clearly established.

The first bale was grown at Experiment, Georgia, in 1962. The fiber was machine picked, ginned at Harrelson, Georgia, and stored in bale at Experiment, Georgia. The cotton was removed from the bale in May, 1964, fluffed, and sorted at 70° and 68% relative humidity (RH) for subsequent measurement.

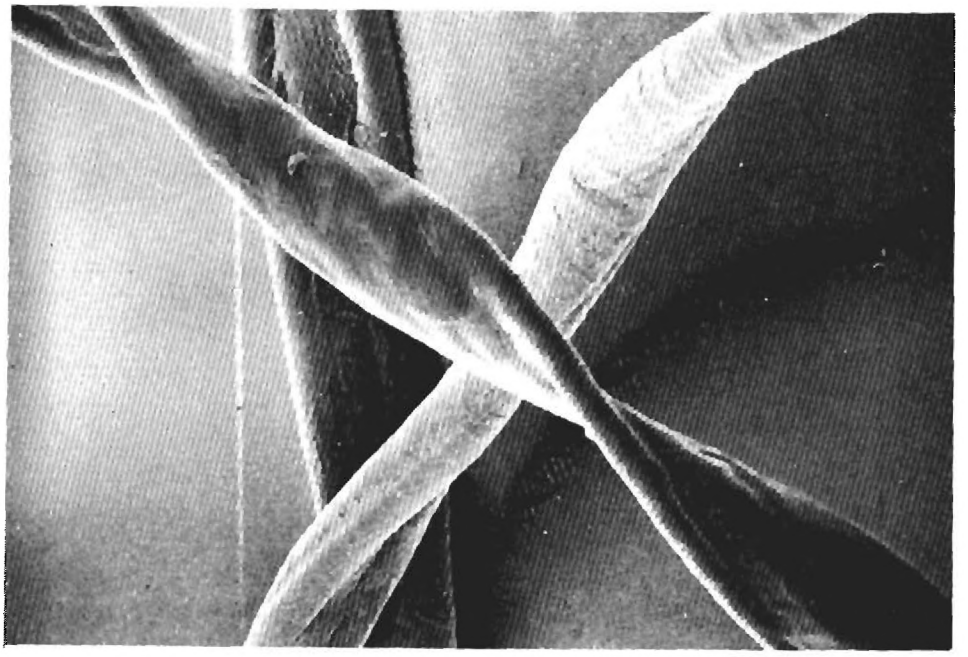
The second bale to be used as a standard specimen was grown two miles west of Experiment, Georgia, during the 1964 growing season. The temperatures for the season were normal but the rainfall was slightly above average. After an essentially normal cultivation period the cotton was hand-picked on 26 October 1964 and ginned at Locust Grove, Georgia, on 28 October 1964. A 465 pound bale was formed at the gin and transported to the Georgia Institute of Technology for storage at 70°F and 68% relative humidity.

3. Characterization of Empire WR Cotton

A scanning electron micrograph of fibers of Empire WR Cotton is shown in Figure 9A and of other cotton fibers in Figure 6 preceding. As may be seen the fiber is a thin convoluted ribbon. Its section dimensions are approximately 0.001 x 0.0003" and its length varies from about 0.4" to 2.5". However, the mean length of the cotton under discussion is slightly over one inch.

Since the fiber is made of fibrils of a very much smaller size (0.000030" diameter) its surface is rough and ridged and is further complicated by the presence of a wax coating. A high power scanning electron micrograph of a typical surface is shown in Figure 9B. Extensive additional micrographs of cotton and man-made fibers appear in a thesis by D. House.[25]

Mr. T. R. Boys,[24] as part of his thesis, characterized the cotton



A. (819x)

B. (5,250x)

Figure 9. Scanning Electron Micrographs of Empire WR Cotton Fibers.

obtained for the frictional experiments. Bale No. 1 (1962) possessed a fiber length of 1.04 inches (2.5 per cent span length) and a micronaire weight of 4.02 micrograms per fiber inch. Convolutions per inch were found to be in the average range 83 to 86 for fibers of 3/4 to 1-1/8 inches in length. Shorter and longer fibers ranged down to about 70 convolutions/inch. Crimps, of regular curvature rather than sharp kinks, were found to be approximately 16.5 ± 1 per inch over fiber lengths of 5/8" to 1-1/4".

4. Characterization of Cotton Specimen No. 2

Specimen No. 2, being hand-picked, was much cleaner than Specimen No. 1. Due to a time limitation, Mr. Boys was only able to perform fibrograph and micronaire measurements of this specimen. These were 1.056" compared with 1.043" for the 1962 bale and 4.11 microgram/inch compared to 4.02 microgram/inch, respectively. In view of the hand-picking one might expect a slightly longer fiber length for Specimen No. 2. For all practical purposes the characterization of Bale No. 1 appeared to fit Bale No. 2.

C. OTHER COTTON SPECIMENS EXAMINED

Goldfarb[26] measured changes in specimens of Empire WR, Carolina Queen, and Dixie King Cotton resulting from ginning but did not measure frictional changes.

Whitworth[27] examined a Pima-Menoufi blend of cotton and measured changes in friction resulting from processing through the stages drawing, roving, and spinning. Mr. James N. Grant of the United States Department of Agriculture (SURDD, New Orleans) supplied us with specimens of cotton designated high draft and low draft respectively. The frictional properties of these materials were measured.[28]

D. NYLON FIBERS OF SPECIALLY SHAPED SECTIONS

The materials evaluated in this investigation by J. O. Huff[23] were 15 denier Nylon 6 fibers of five respectively different cross-sectional shapes obtained from a private source.

Nylon 6 is a linear polymer made from the α, ω-six carbon amino-acid. The Nylon 6 polymer is shown chemically as

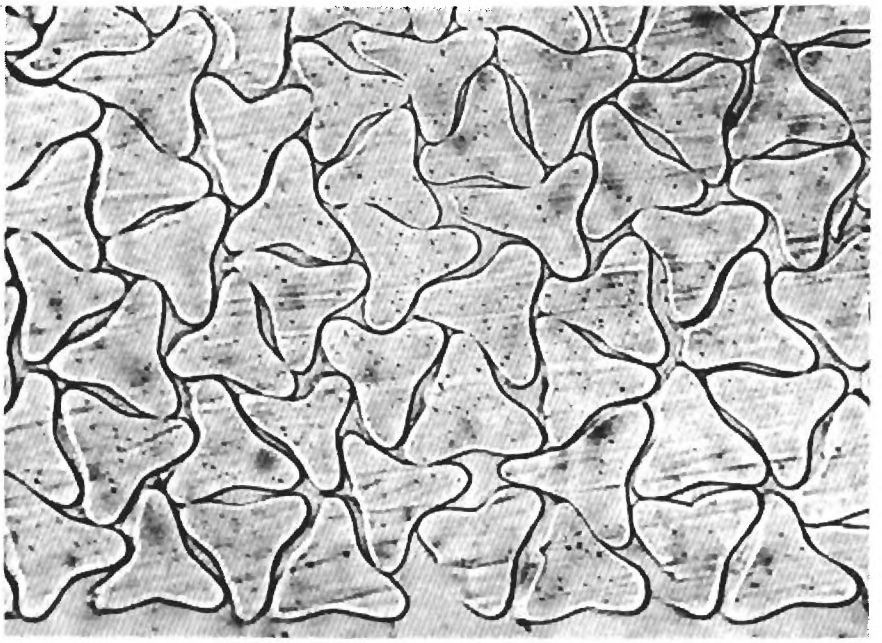
$$H\ [-\ NH(CH_2)_5\ CO\ -]_n\ OH \quad ,$$

where n is approximately 200. Nylon 6, also known as caprolactam, is a melt spun fiber. The Nylon 6 fibers evaluated contained 0.22 per cent titanium dioxide. The surface finish was removed from the fibers before measurement with a cold bath of 1, 1, 1 trichlorethane for two minutes.

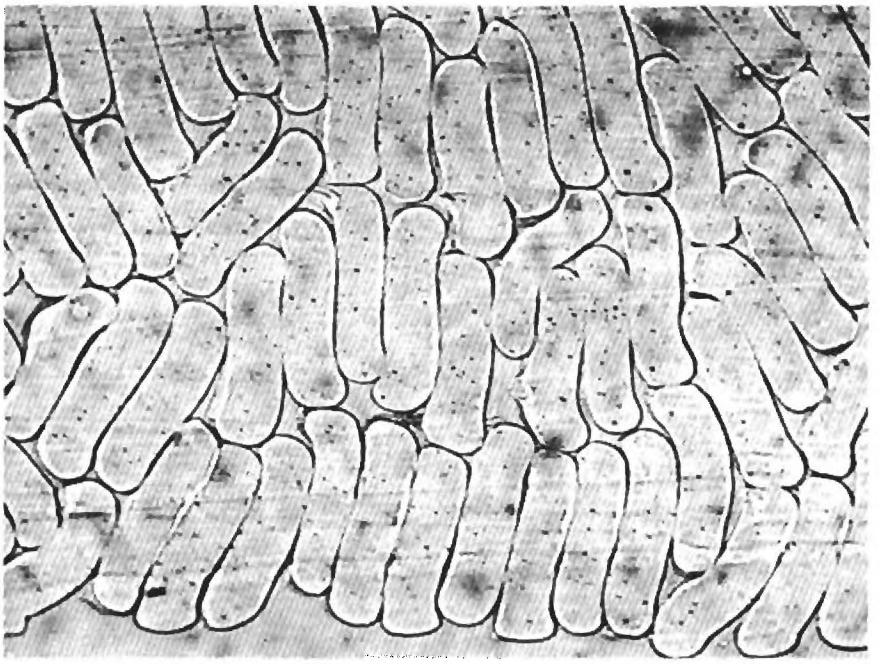
The cross-sections of the fibers measured for frictional properties were cylindrical, duokelion, trilobal, quasi-triangular, and tetrakelion. Illustrated in Figures 10A and 10B respectively, are typical examples of fibers with duokelion and trilobal sections. By controlling other variables to the degree feasible and changing the cross-sectional shape, it was possible to evaluate the influence of the shape on the frictional properties of fibers of Nylon 6. From these data a better understanding of the effect of fiber section shape on the frictional properties of other fibers may be deduced by analogy, and a clearer understanding of the possible effect of the shape of cotton on its friction is presented.

E. OTHER FIBERS EXAMINED

Because of the assistance rendered by graduate students in preparing their theses and undergraduate students completing special problems, it



(a)

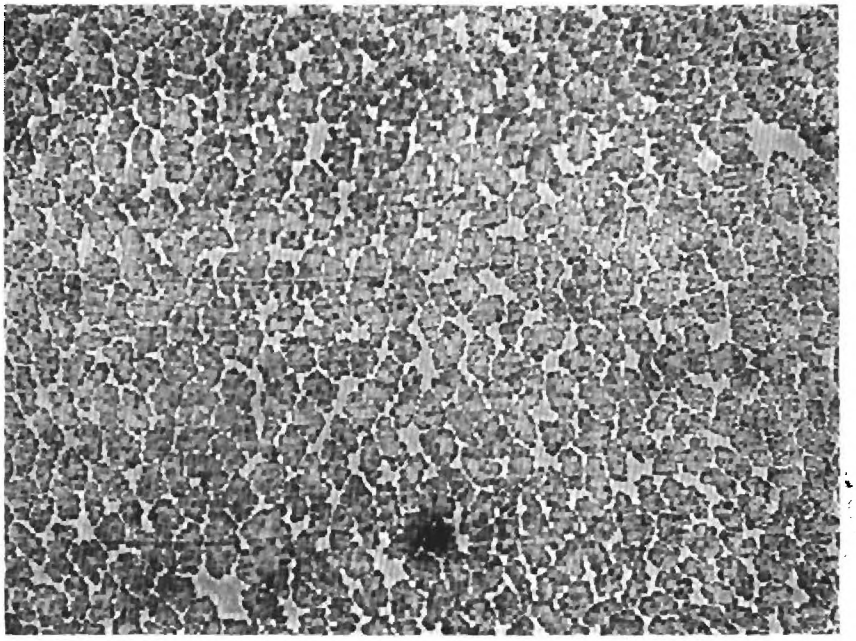


(b)

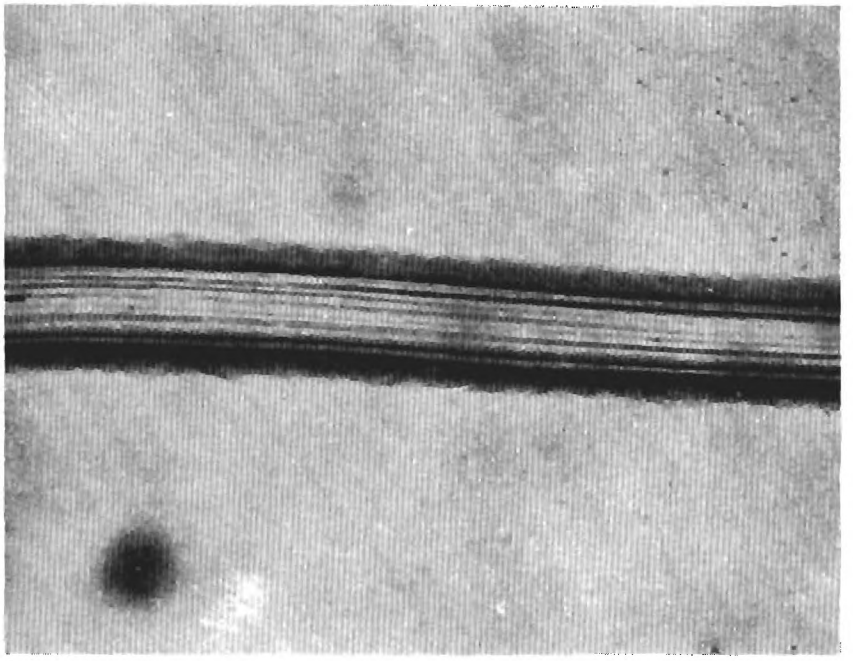
Figure 10. Optical Micrographs of Cross Sections of Trilobal and Duokelion Nylon (320x).

was possible to examine the frictional properties of a number of other fibers. Bryant[29] examined Empire WR Cotton, Viscose, and Nylon. Gunther[22] examined Acrilan, Dacron, Orlon, Dynel, Nylon, and Viscose in addition to Empire WR Cotton. Micrographs of fiber sections of Viscose and Dynel are shown in Figures 11 and 12.

Specimens of spider silk (A. benjaminus) and silkworm silk (B. mori) were examined by Rushing.[30] Spider silk specimens appear to be of circular section whereas silkworm silk is triangular or trapezoidal in section and is very variable in size and shape. Zefchrome (polyacrylonitrile) fibers were examined by Simmons[31] and by Wakelyn.[32] Simmons examined friction effects before and after spinning the fibers and therefore observed processing effects. Wakelyn examined the effects of various antistat coatings on the fibers he examined.

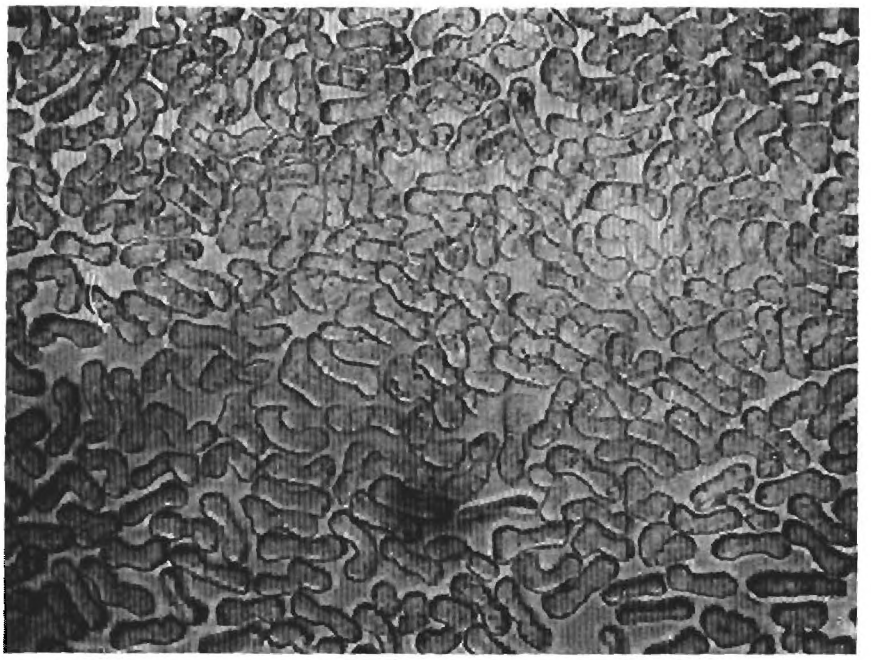


(a) CROSS-SECTION 380X

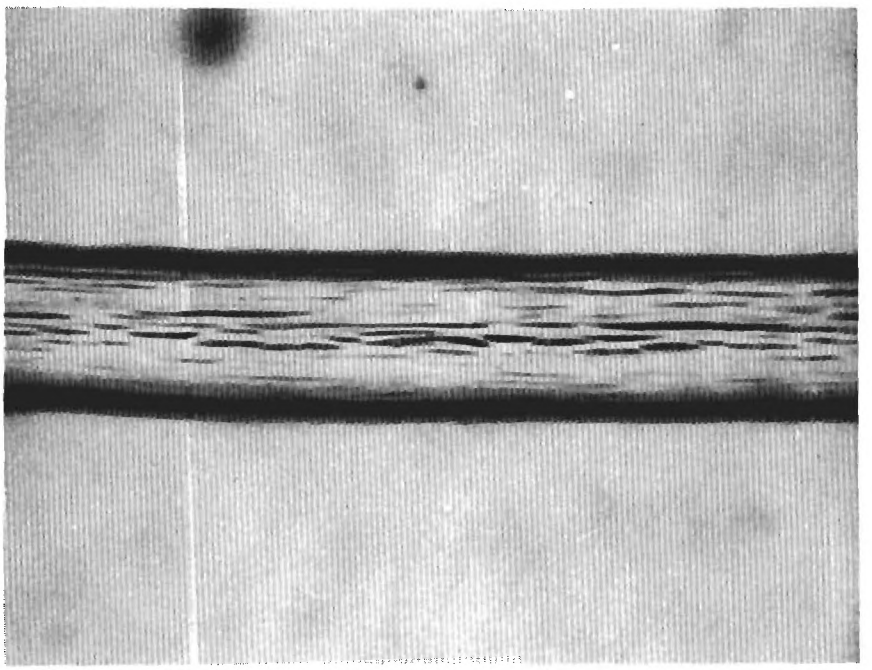


(b) SURFACE 427X

Figure 11. Micrographs of Cross Sectional and Longitudinal Shapes of Viscose Fiber (380x), (427x).



(a) CROSS-SECTION 380X



(b) SURFACE 427X

Figure 12. Micrographs of Cross Sectional and Longitudinal Shapes of Dynel Fibers (380x), (427x).

V. PROCEDURES FOR FRICTION MEASUREMENT

A. GENERAL

Utilizing the apparatus previously described for measuring friction at low normal force procedures were evolved which would allow full utilization of the apparatus for this investigation. The methods developed and the procedures used in obtaining measurements are discussed in the subsequent paragraphs.*

B. SELECTION AND PREPARATION OF FIBER SPECIMENS

The majority of the frictional measurements were made for fibers of cotton or of nylon. The cotton used was the same Empire WR cotton collected by T. R. Boys and described in the preceding chapter. The Nylon 6 used was 15 denier monofilament fiber. Other fibers examined to a lesser degree included Acrilan, Dynel, Orlon, Viscose, Dacron, Zefchrome, Silk, and Spider Silk.

In an effort to eliminate as many sources of error as possible, a consistent fiber mounting technique was employed as outlined in Chapter III.D.

C. PROCEDURES FOR MEASURING COEFFICIENTS OF FRICTION OF FIBERS

Once the fiber pair under investigation had been mounted, the measuring procedure was initiated.

The electronic equipment was switched on to allow a warm-up period of about ten minutes. The normal force was adjusted as previously described by regulating the amount of current flowing through the coil. The traverse

*These methods apply essentially to the earlier version of the instrument as well except that a planimeter was used for curve integration with that instrument.

speed of the upper fiber was adjusted to the desired setting. The sensitivity of the XY recorder was selected to keep the frictional plot within the limits of the chart.

The normal force adjustment was made carefully to avoid damage to the fiber or oscillations of the record pen. Adjustment of the restoring force to the lower fiber mount attached to the D'Arsonval galvanometer was also necessary to achieve the correct degree of "stiffness" or resistance to the movement of the upper fiber once relative motion commenced.

A short period of time was allowed for the equipment to settle down since the delicate nature of the experiment made it sensitive to any external forces or vibrations. The pen on the XY recorder was then set in motion and a base line indicating zero frictional force was established on the chart paper. The pen was then returned to the starting position.

The drive motor was switched on and the relative motion between the fiber pair commenced. Simultaneously the XY recorder sweep and the integrator were switched on. It was essential that both of the latter be started at the same time if the integrator reading was to be accurate. The fiber was allowed to complete its traverse and the XY recorder and the integrator were then switched off together. The drive motor was turned off and the current controlling the normal force was reversed, thus raising the upper fiber. The completed chart was removed from the XY recorder and replaced with a new one. The old chart was then marked to indicate the measurement conducted and the integrator reading was recorded. The initial friction measurement was now complete.

The fiber drive motor was reversed and the fibers returned to their initial starting position. At least two runs were completed for each

fiber pair and for certain tests three runs were made. The average values obtained for these runs were reported. The same procedure was followed for the subsequent runs.

The restoring force of the servo apparatus was calibrated by hanging small, known weights upon the fibers and observing the respective deflection from zero of the XY recorder at a specific sensitivity. Knowing the weight, position, and amount of deflection, a ratio indicating force per unit of deflection was computed. This value was then used in the computation of both the kinetic and static coefficients of friction.

After the stick-slip friction plots had been made under the desired conditions, the static and kinetic coefficients of friction and their ratio were calculated (see Figure 8) as discussed in the preceding chapter.

D. PROCEDURE FOR MEASURING FRICTIONAL FORCES WITH NO EXTERNAL NORMAL FORCE APPLIED

One variation from the previously discussed experimental procedure occurred during a study of friction under conditions of no externally applied normal force. A cotton fiber attached only at one end was drawn across a fiber mounted on the servo controlled galvanometer. The lower fiber was mounted as previously described. The upper fiber was not attached to the normal force apparatus but rather to an arm attached to a worm gear driven milling vise. The fiber withdrawal rate was 0.1 mm/sec. The only normal force involved here was the weight of the upper fiber which amounted to a few micrograms. With the exception of the differences in mounting of the upper fiber and the normal force application, the experiment was conducted as previously described. No precise calculations of the static and kinetic coefficients of friction could be made, but

measurements of maximum forces and energy of withdrawal could be obtained and values of μ_s and μ_k could be approximated. A second version of this method withdrew a fiber tuft from a single fiber attached to a servo controlled torque dynamometer. This method gave large forces, on occasion, and will be discussed independently.

VI. CAPABILITIES OF THE FRICTION MEASURING INSTRUMENTS

A. INTRODUCTION

The early phases of this research revealed the capabilities of the friction measuring instrument. Improvements in it gradually brought its capabilities to a sensitivity necessary to examine fiber behavior at very low normal forces, the condition under which fibers are handled in textile processing up to the yarn. These developments and familiarity with the quantities of plotted data obtained gradually led to a resonable understanding of fiber frictional behavior which is outlined herein. In this chapter we will discuss the capabilities of the instrument in order to present a more logical basis for the evaluation of the subsequent friction data by the reader.

B. FRICTION DATA AND ITS REPEATABILITY FOR A SINGLE FIBER

1. General

The characters of the analog plots of fiber friction between fibers of cotton, nylon, and cotton on nylon have been displayed in previous figures (7 and 8) and the differences in the data characteristics of the particular material have been noted. Let us now examine the character as it pertains to the repeated measurements of a single fiber. In this instance it must be remembered that we are examining essentially only the friction of the undersurface of the traversing fiber as it slides across the top of the fiber attached to the servo controlled galvanometer needle.

2. Effects of Successive Measurements of the Same Fiber Pair

Early in the research it was noted that data plots of a specific

fiber resembled one another. Many fibers were measured more than once to avoid mounting a new fiber between every measurement which is a time consuming task. However, invariably small changes in the friction of a cotton fiber resulted. In an effort to determine the consistency of the change and the consistency of the fiber character the friction of the same single fiber against a second was measured for 13 successive times. Figure 13 displays the data for the 1st, 5th, and 13th measurements and Table 1 gives the data for all measurements. This particular cotton was hand ginned. The principal peaks exhibited in the three measurements remain little changed and in essentially the same positions. The table indicates scatter in the successive values of μ_k and a general down trend in the μ_s value from the 1st to the 7th measurements. A comparative view of the data may be obtained by plotting the values of μ_s, μ_k, and μ_s/μ_k in accordance with the order of measurement. These data appear in Figure 14. It is apparent that there is a marked reduction in the value of μ_s for each successive pass for the first three passes and a smaller one thereafter until the seventh pass after which the value has essentially stabilized at the value of the third pass, 20 per cent below the first value. The quantity of μ_k, on the other hand, changes only a small amount which could be attributed to scatter about a value of 0.265. However, there is some evidence of a minimum at the 5th or 6th measurement. The μ_s/μ_k ratio displays a minimum at about the 3rd or 4th measurement then stabilizes. This is also about 20 per cent below the initial value.

It is definite from these and other measurements that principal features of a cotton fiber continue to give peaks after successive traverses. These peaks are particular to a given fiber. Only the first traverse

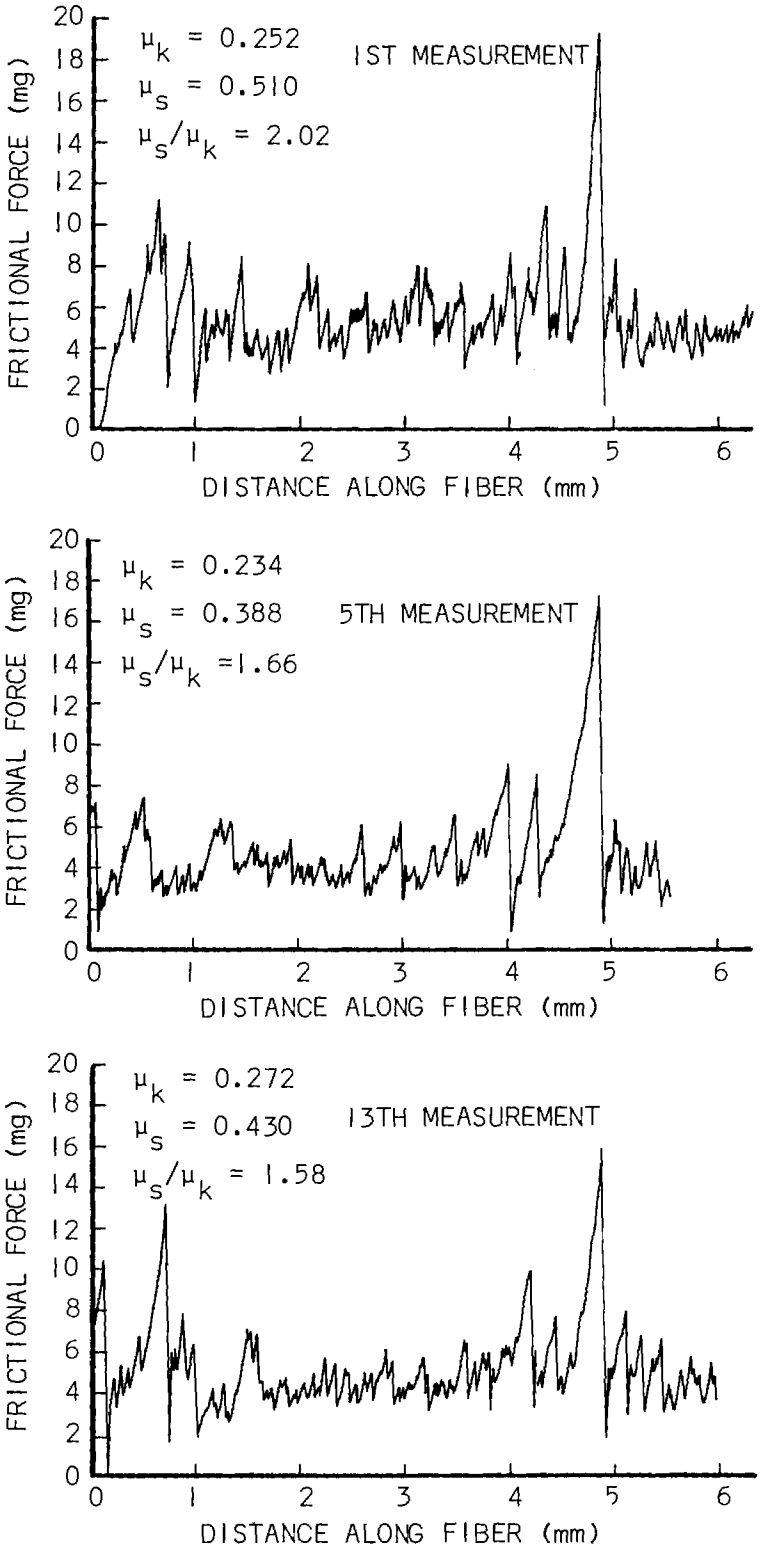


Figure 13. Friction Data Plots of 1st, 5th, and 13th Successive Measurements for an Empire WR Cotton Fiber Pair.

Table 1. Coefficients of Static and Kinetic Friction for 13 Successive Measurements of an Empire WR Cotton Fiber Pair*

Measurement No.	Coefficient of Kinetic Friction, μ_k	Coefficient of Static Friction, μ_s	Ratio μ_s/μ_k
1	0.252	0.510	2.02
2	0.272	0.458	1.69
3	0.263	0.406	1.55
4	0.261	0.403	1.55
5	0.234	0.388	1.66
6	0.242	0.388	1.60
7	0.274	0.374	1.38
8	0.261	0.408	1.57
9	0.241	0.415	1.72
10	0.262	0.417	1.59
11	0.268	0.416	1.56
12	0.245	0.392	1.60
13	0.272	0.430	1.58
(198A to 198M)			
Average	0.257	0.416	1.62

*Hand Ginned Cotton

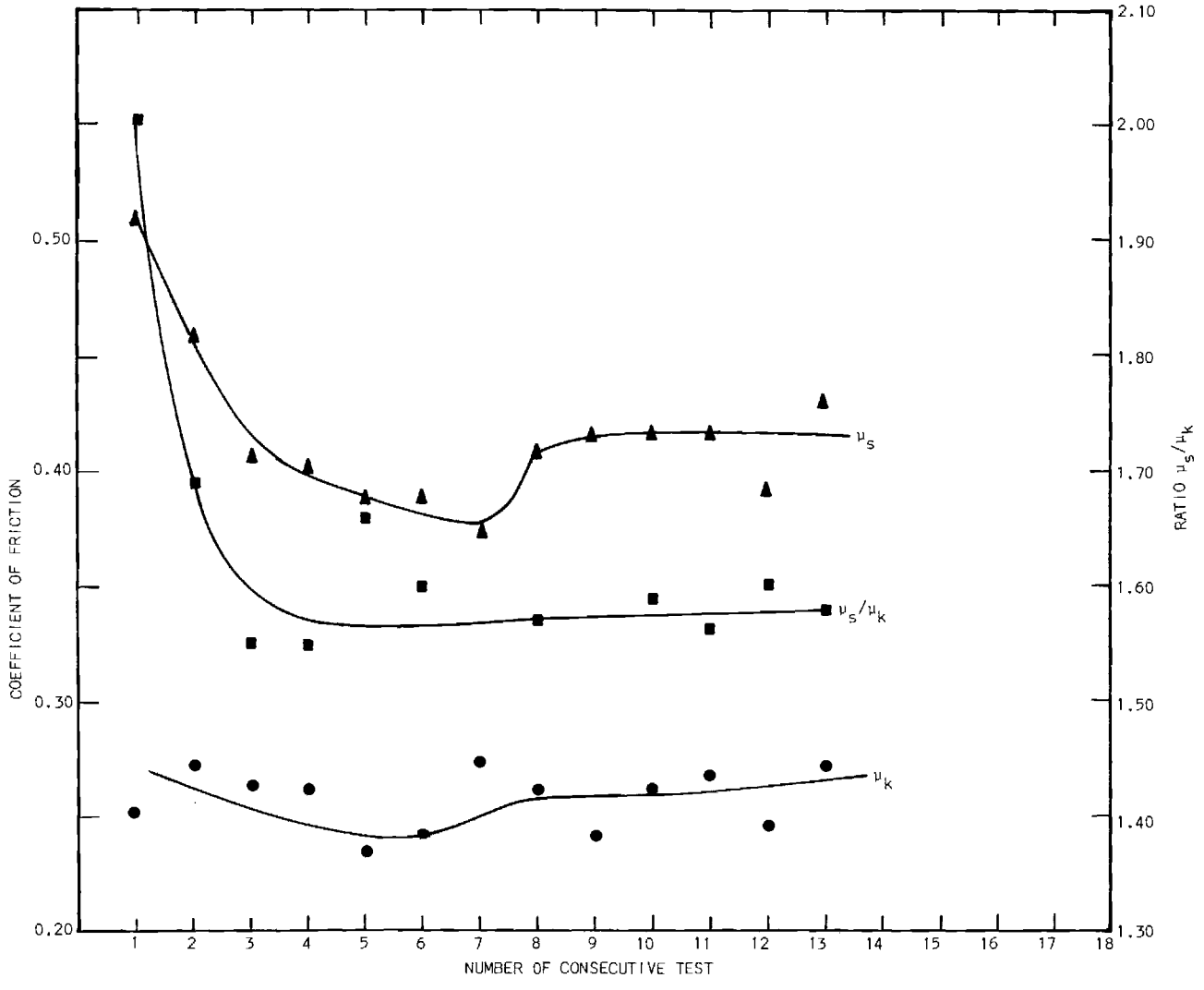


Figure 14. Variation of Frictional Parameters of Empire WR Cotton Fiber in 13 Successive Traverses Across a Second Fiber.

gives the true nature of the virgin fiber. Successive traverses indicated approximately 10 per cent reduction per pass in the values of μ_s and μ_s/μ_k for this fiber for two or three traverses. Other measurements of many other fibers have indicated that the average changes observed for μ_s and μ_k per pass are generally smaller, of the order of 5 per cent per pass, for the first three traverses.

A decrease of frictional coefficients for this type of fiber contact is normally observed on successive traverses indicating that processing may be expected to lower frictional forces of the fibers.

C. COMPARISON OF FRICTION DATA WITH MICROGRAPH OF COTTON FIBER

Some cotton fibers exhibit very high "stick" friction peaks. An examination of a series of such fibers was made in which a micrograph of a specific fiber was compared with the respective friction data plot of the fiber. Examples of this type of study are shown in Figures 15 and 16.

It will be noted that very large sticks may occur at certain features which appear to be reversals of the fiber convolutions in these particular fibers. Similarly large sticks have been observed for damaged fibers and for fibers displaying surface deformities. The particular cotton illustrated here is cotton designated as "high draft" cotton.*

It is obvious that large sticks of this type give a biased frictional measurement. On the other hand, a certain statistical percentage of such fibers is always present and the high sticks must play an important part in fiber travel through various textile processing stages.

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*Supplied by Mr. James N. Grant, USDA, SURDD, New Orleans, La.

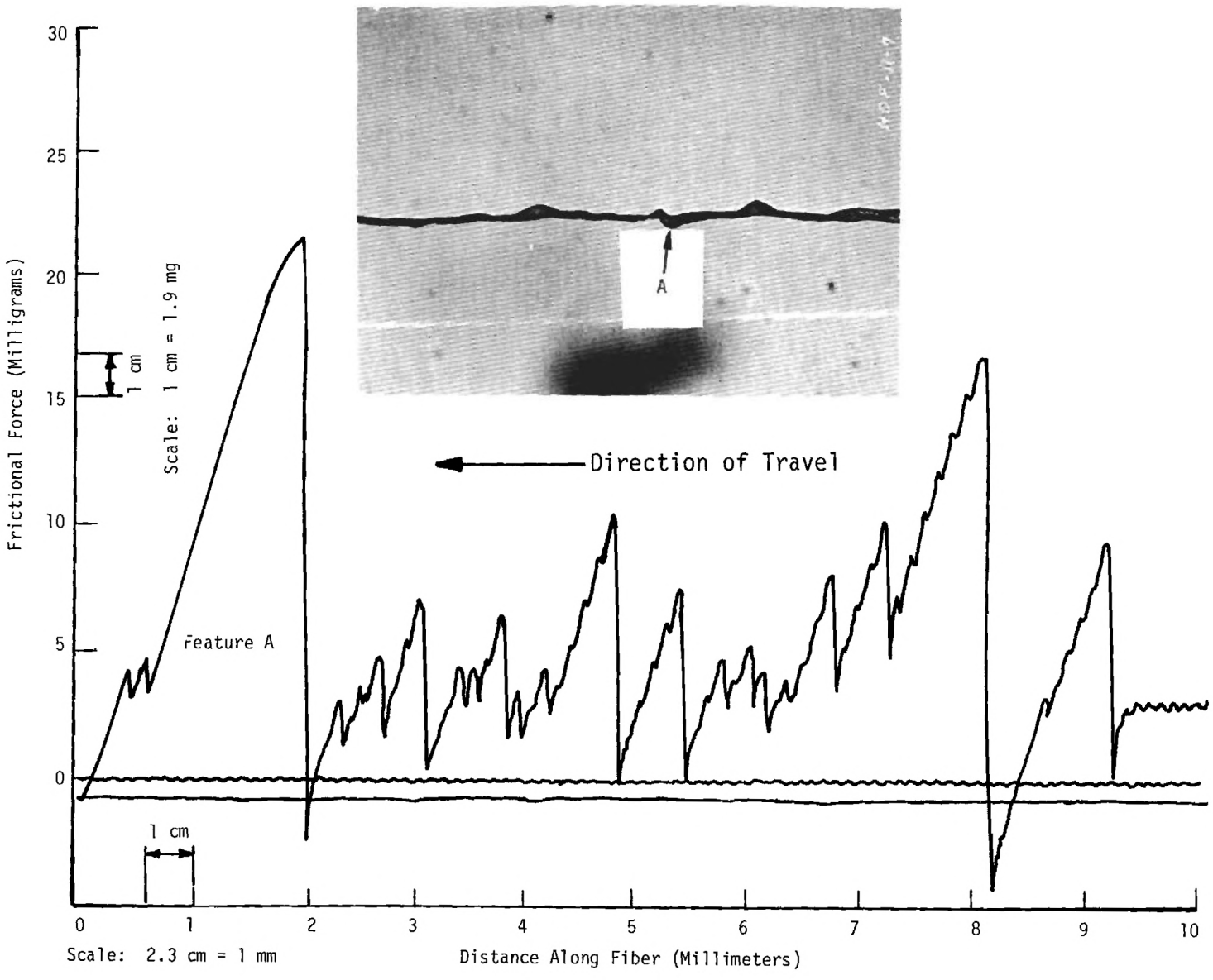


Figure 15. Frictional Graph and Micrograph of High Draft Cotton Fiber Indicating "Stick Effect" and Feature Responsible.

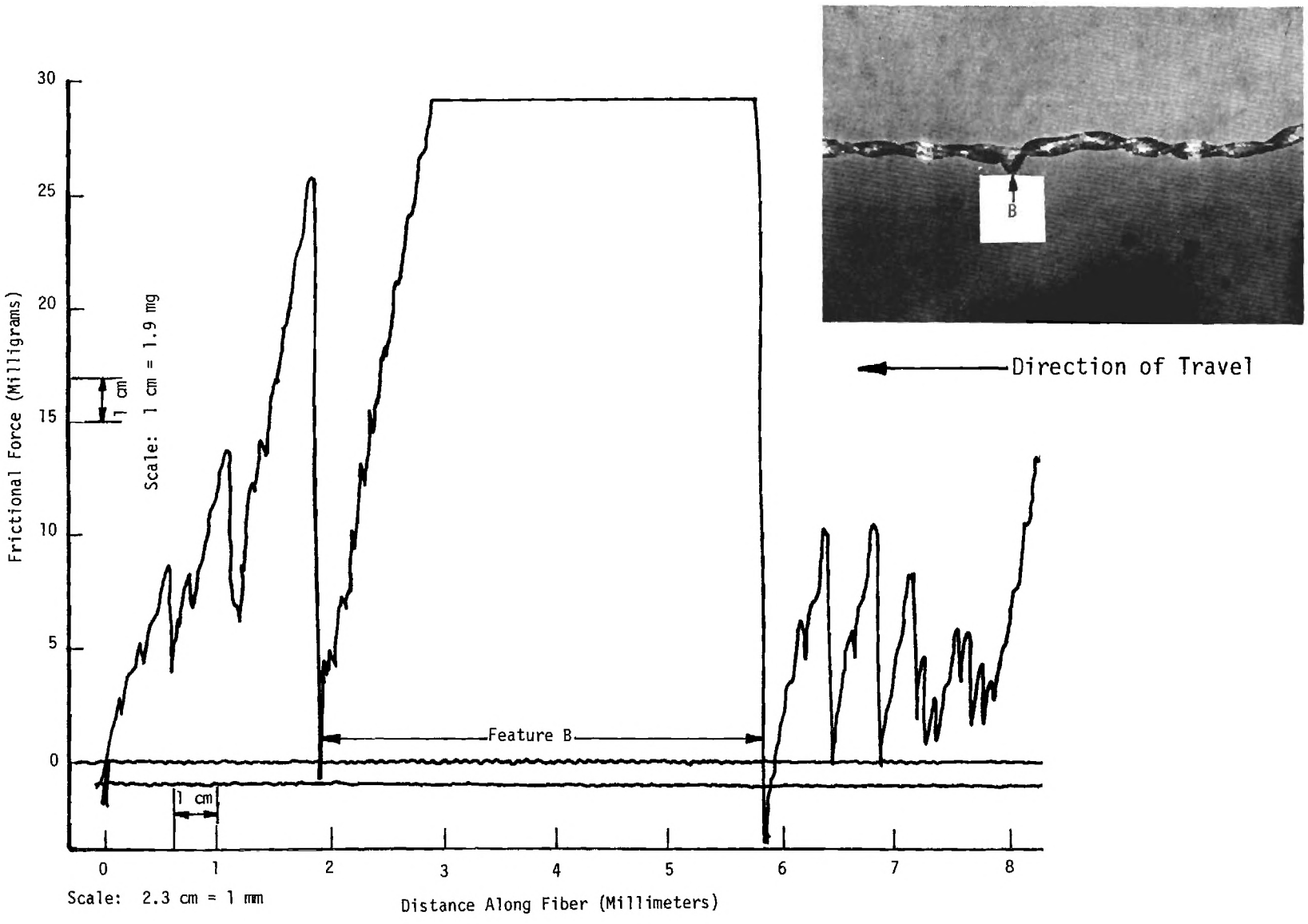


Figure 16. Frictional Graph and Micrograph of High Draft Cotton Fiber Displaying Friction Peak and Features Responsible for it.

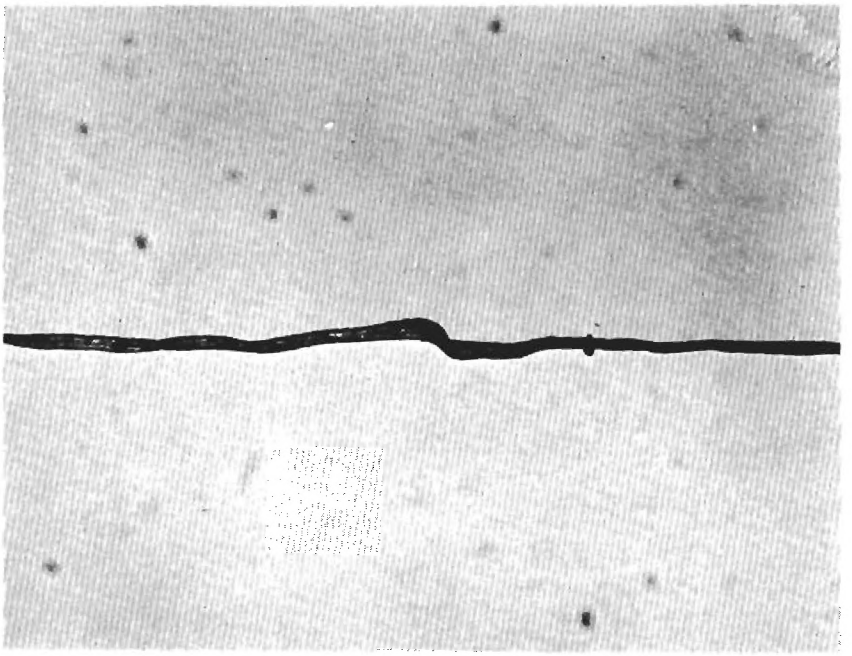
D. EXAMPLE OF FRICTION DATA CHANGE WITH INDUCED FIBER SHAPE CHANGE

A method of detection of fiber damage is the Congo Red test. This employs a Congo Red dye solution containing about 20 per cent NaOH. While damage studies were being conducted for processed fibers a single fiber mounted on the friction apparatus was treated with one drop of the Congo Red solution. The fiber was allowed to dry for 30 minutes after treatment. Photographs of the fiber before and after treatment are shown in Figure 17. The fiber examined under a stereomicroscope during the application of the solution began swelling immediately. The surface became smoother and the peak to valley amplitude became less. Its friction data before and after treatment are displayed in Figures 18 and 19. The marked change in the data are obvious: the large peaks have disappeared. The values of μ_s, μ_k, and μ_s/μ_k have been lowered significantly. The ability of the frictional instrument to depict shape changes of the fiber by character changes of the data plot are clearly indicated.

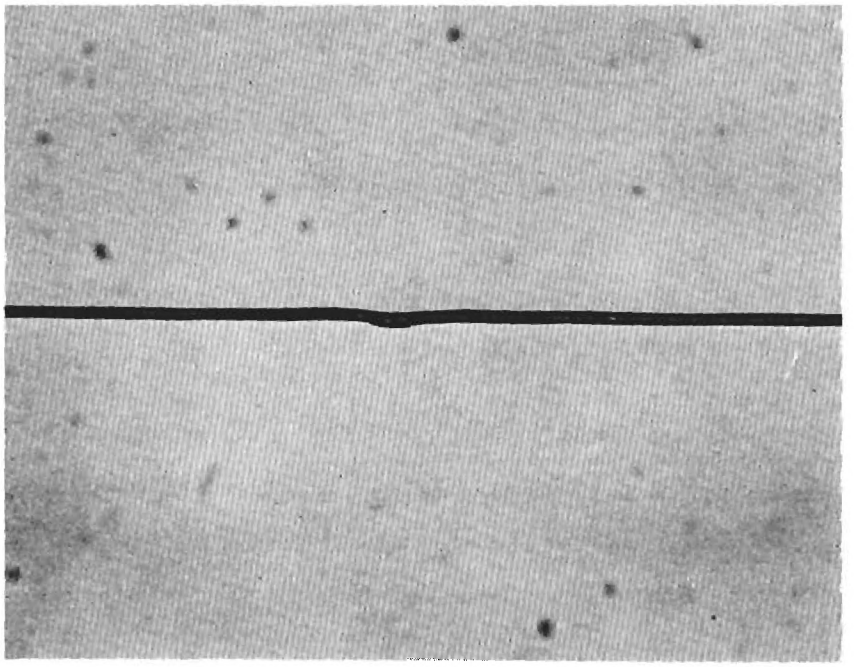
E. MEASUREMENT CAPABILITY AT VERY LOW FORCES

In the initial instrument reduction of normal forces below about 20 mg lead to excessive bouncing and lack of fiber to fiber contact during the slip phase of friction measurements. The modified instrument, however, exhibited good contact capability down to approximately 1 mg with smooth fibers such as cylindrical nylon. As the normal force was reduced, the character of the curves became very detailed as shown in Figure 20 for cotton on glass.

It is possible also with this instrument to draw a fiber across a second with only the weight of the fiber, Van der Waal, and cohesive or adhesive forces acting in lieu of an external normal force. As shown in



A. BEFORE (100X)



B. AFTER (100X)

Figure 17. Cotton Fiber Before and After Treatment with Congo Red Solution (up to 20% NaOH) (100x).

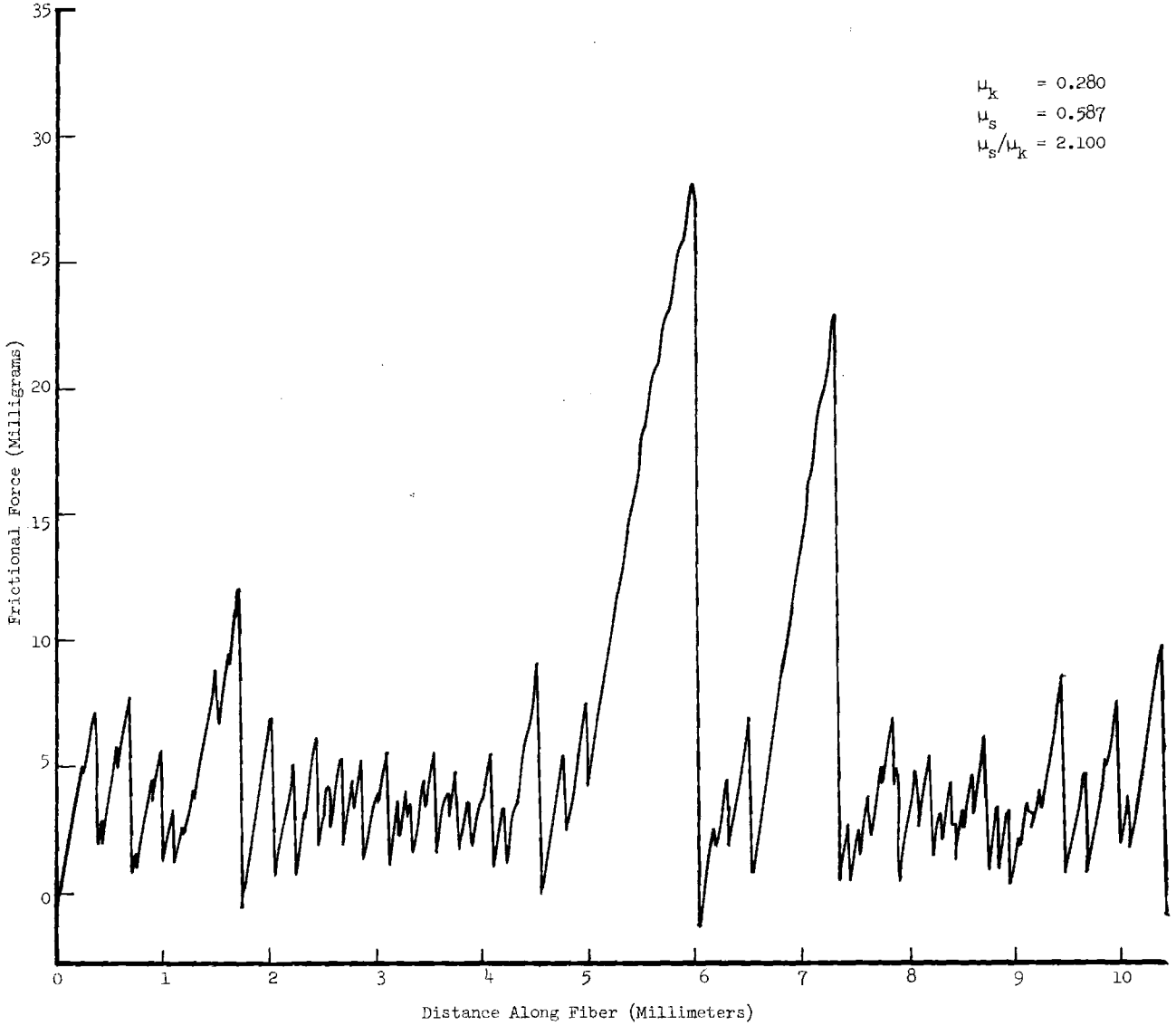


Figure 18. Frictional Data Plot of Empire WR Cotton Fiber Before Treatment with Congo Red Solution.

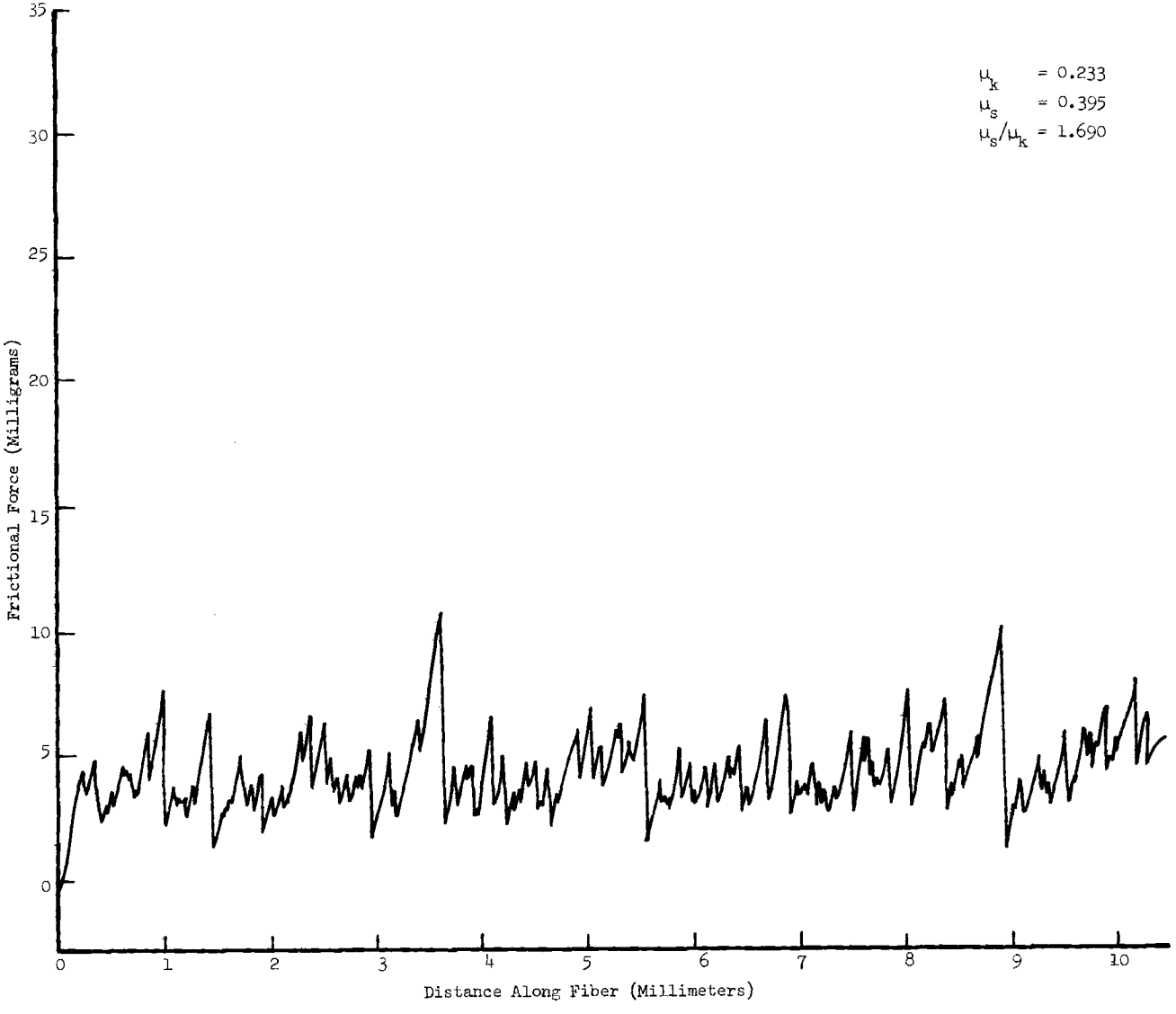


Figure 19. Frictional Data Plot of Empire WR Cotton Fiber After Treatment with Congo Red Dye Solution (drying time 30 minutes).

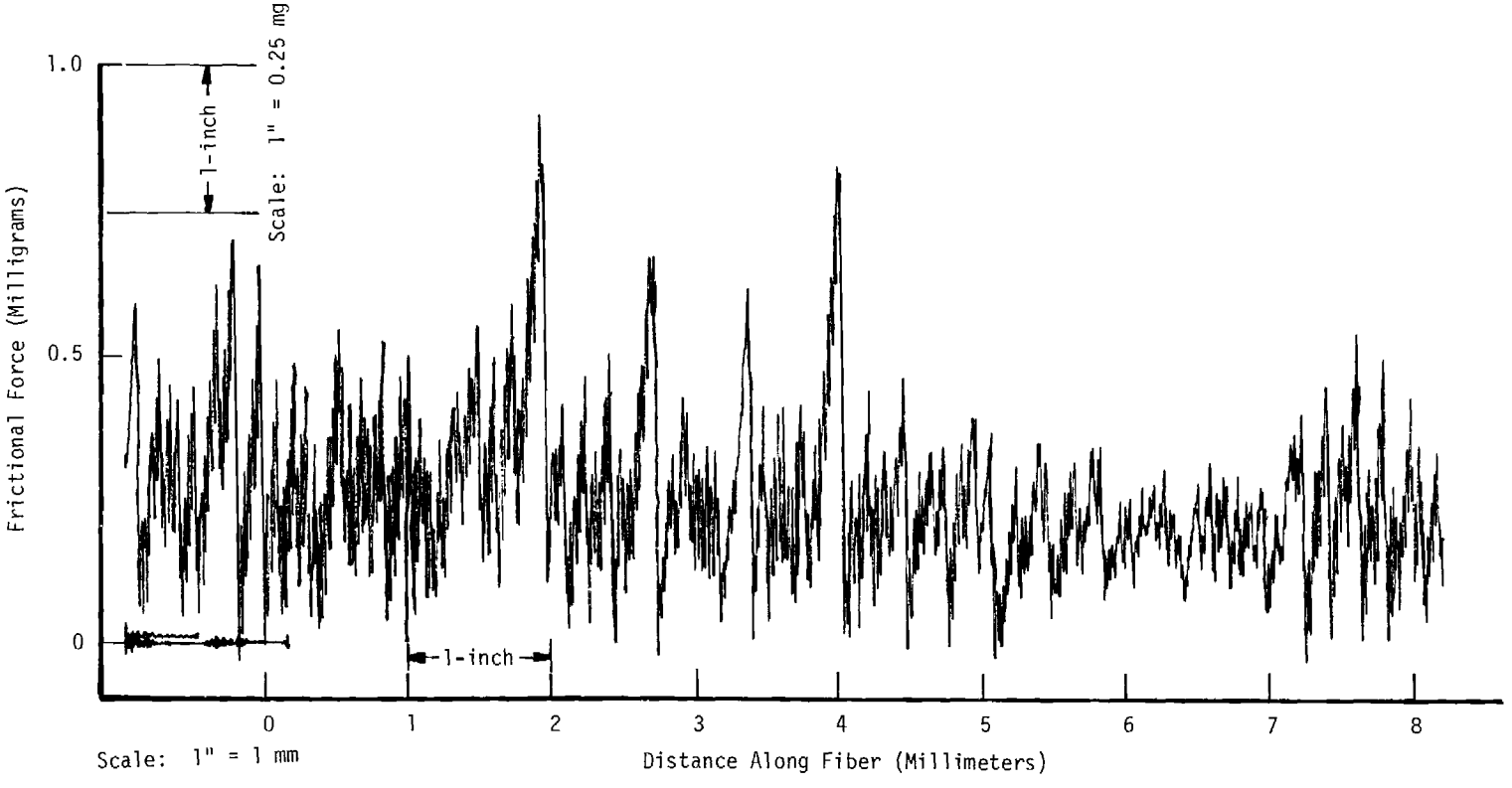


Figure 20. Frictional Data Plot of Cotton Fiber Against Glass Fiber at 2 mg Normal Force.

Figure 21, for a glass fiber drawn across a nylon fiber frictional forces as high as 0.50 mg may be experienced and these are not greatly different in magnitude than for the cotton fiber on glass exhibited in the preceding Figure 20. An interesting curve character is also experienced when a crimped nylon fiber supported only at one end is drawn across a second nylon fiber supported by the bow on the galvanometer arm as shown in Figure 22. The effects of the crimp on the nylon data are quite evident. A rough calculation of μ_s, if we assume the normal force is approximately the weight of the fiber of glass on nylon (0.000006 gm), in the data of Figure 21, gives a value of 83 and μ_k would be about 25.

A final method of measuring frictional forces at low normal forces was achieved by withdrawing a tuft of fibers at a rate of 2.38×10^{-2} cm/sec from a single fiber attached to a torque dynamometer.* The torque dynamometer operates with a torque restoring servo system, has nearly friction free air supported bearings, and has a large range of force measurement capability. Since forces involved in fiber extraction from a tuft are much larger and more variable than the single fiber pair forces, the dynamometer was employed. Its electrical output provides analog plots of forces involved in removing cotton fibers from cotton seeds, breaking fibers, or extracting single fibers from tufts. Examples of the data output obtained when a single fiber was withdrawn from tufts of cotton roving are shown in Figure 23. It is obvious here that large frictional forces are involved in extracting a cotton fiber from a tuft of cotton.

*Servo Torque Balance Reaction Dynamometer (Model 114A manufactured by McFadden Electronics Company, South Gate, California), described in detail by Goldfarb.[26]

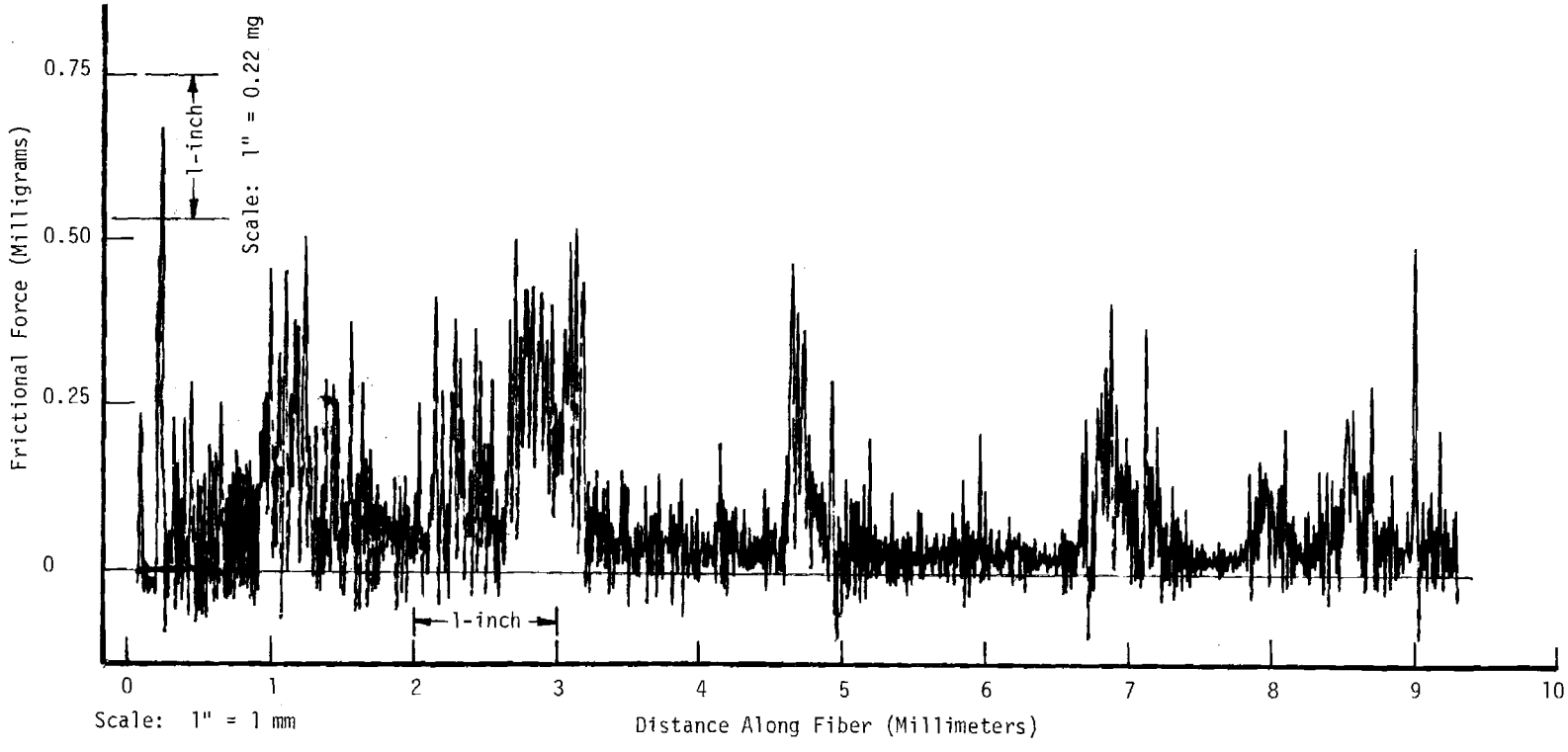


Figure 21. Frictional Data Plot for Glass Fiber, Supported Only at One End, as It was Drawn Across a Nylon Fiber Attached to the Frictional Recording Instrument.

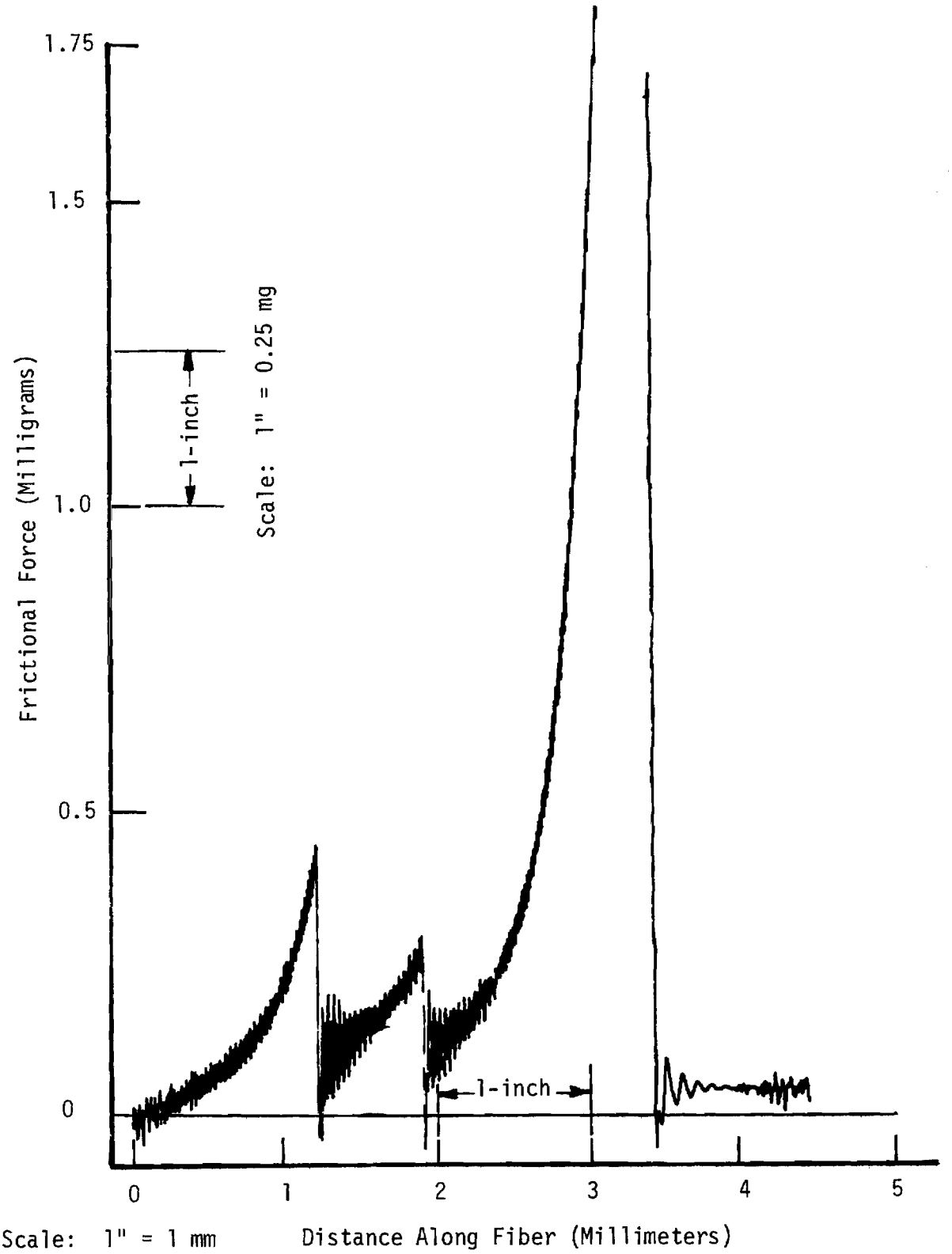


Figure 22. Frictional Data Plot for Crimped Nylon Fiber, Supported Only at One End, as It was Drawn Across a Nylon Fiber Attached to the Friction Recording Instrument.

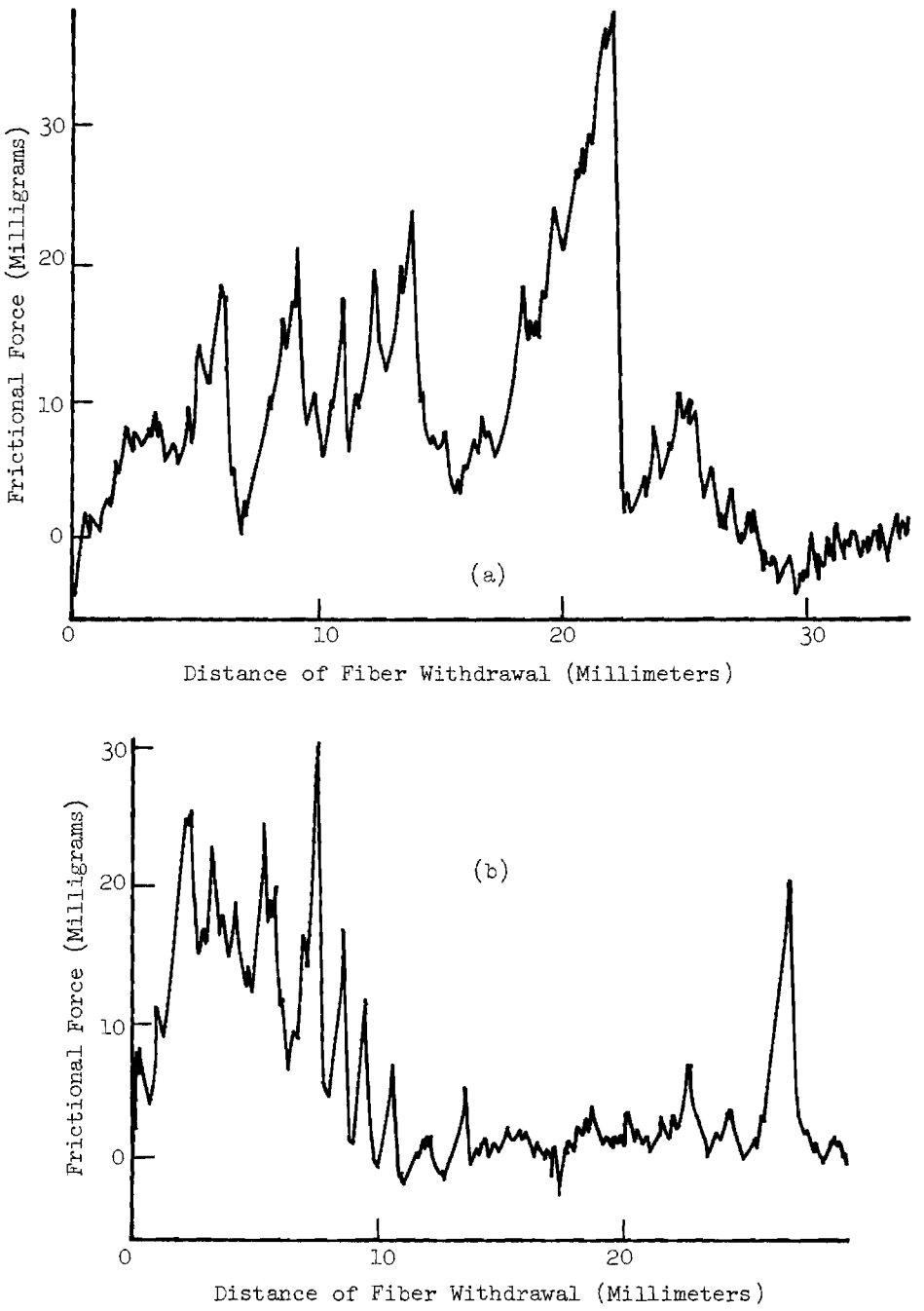


Figure 23. Analog Plots of Forces Required to Withdraw a Single Fiber from Cotton Roving.

F. SUMMARY

It is apparent from the foregoing data that the frictional instruments employed are able to provide significant information concerning fiber friction to the point that the "fingerprint" of a single fiber of a single material may be recognizable and to the point that the fiber may be essentially unconstrained. Since the discrimination is excellent the principle task is to interpret the quantities of data obtained.

Data are also dependent on variables which may or may not be controlled to the desired degree. However, before any consistent frictional data could be obtained it was necessary to explore to some degree the effects of these variables. A discussion of this effort is reported in the next chapter.

VII. MEASUREMENTS OF EXPERIMENTAL FACTORS AFFECTING FIBER FRICTION OF COTTON

A. GENERAL

During the course of this work friction data have been recorded for several varieties of cotton, Viscose, Nylon, Acrilan, Orlon, Dacron, Dynel, silk, ramie, and wires of several metals. These measurements comprised more than one thousand data plots. About 700 of these were made with the first improved gravity loaded instrument at normal force of 20 mg and the balance were made on the low normal force instrument generally in the 2 mg to 10 mg region.

Near the beginning of the work it was found that it would be necessary to establish precise procedures in fiber mounting and instrument operation. As a result, measurements were made to establish variations in friction measurements resulting from changing fiber tension during mounting, the normal force used, the temperature to which the fiber was heated during processing, the velocity of fiber traverse, and the humidity at which the fiber was measured. The measuring instrument was not in a constant humidity room. However, humidities were recorded in most instances and an effort was made to measure the effects on the fiber friction of changing humidity of a crudely controlled ambient. Most measurements were made approximately in the range 60% relative humidity ± 5 per cent and a temperature of 25°C ± 2°C.

B. EFFECT OF FIBER TENSILE MOUNTING FORCE

The frictional measurements presented herein were made and reported principally by Bryant in his thesis[29] and in Semiannual Report No. 3 of this grant.[33] Measurements were made on the gravity loaded friction

instrument at 20 mg normal force.

Guthrie and Oliver[18] reported an increase in the frictional force of approximately 50% for an increase in the tensile force employed in mounting fiber pairs of viscose rayon. An increase of 50% occurred over the range 400 mg to 1600 mg of tensile force. The maximum tension employable is dependent on fiber strength at the long gauge or span length normally used, ~ 0.5" for friction measurements made here. The increase discussed would represent a change in μ_k from about 0.12 to 0.19 for viscose rayon at 125 mg normal force according to the data of Guthrie and Oliver.

In order to examine the effects of tension more thoroughly, measurements of the coefficient of friction of 15 Empire WR cotton fiber pairs each were made for fibers mounted with tensile forces of 125, 425, 825, and 1125 mg, respectively. Three measurements were made for each fiber pair. Hence, 180 total measurements were made.

Forces were established by fastening one end of the fiber to its mount with sealing wax. With the fiber in the vertical position, and parallel to the mount base, a weight of proper amount was attached to the fiber. Its end was then fastened in position with Duco cement which was allowed to dry while the weight was attached.* Typical data for one run are shown in Table 2 and the data obtained for all runs are plotted in Figure 24 to exhibit the variation.

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*Actually these numbers do not represent the final tension; only that established before cementing. A correct number can only be obtained by maintaining a tension measuring device in series between the fiber and the mounting posts. The tension will also be changed considerably on the application of the normal force between the fibers. However, it may be estimated that this change will be less than 200 mg for the normal force range 20 to 40 mg.

Table 2. Friction Versus Tension Data for Empire WR Cotton Fibers

Part I: 425 Milligrams Tension

Fiber Number	Coefficient of Kinetic Friction (μ_k)			Coefficient of Static Friction (μ_s)		
	a	b	c	a	b	c
1	.278	.248	.291	.523	.465	.474
2	.253	.252	.241	.453	.394	.442
3	.182	.251	.206	.413	.463	.415
4	.262	.270	.264	.493	.443	.458
5	.303	.321	.258	.546	.578	.485
6	.314	.258	.257	.513	.466	.508
7	.450	.505	.390	.781	.837	.581
8	.421	.348	.352	.639	.615	.677
9	.402	.324	.308	.677	.628	.547
10	.355	.333	.333	.620	.571	.594
11	.268	.257	.266	.522	.514	.547
12	.284	.261	.258	.496	.478	.503
13	.310	.294	.256	.529	.485	.475
14	.210	.197	.222	.429	.398	.400
15	.322	.252	.251	.528	.463	.452
Average	.308	.291	.277	.544	.520	.504
Grand Average		.292			.523	
μ_s/μ_k	1.76	1.78	1.83	Average	1.80	

Normal Force = 20 ± 1 mg

(Continued)

Table 2. Friction Versus Tension Data for Empire WR Cotton Fibers

Part II: Data Summary for Various Tensions

Tension (mg)	μ_k (Avg)	μ_s (Avg)	μ_s/μ_k (Avg)
125	.356	.647	1.82
425	.292	.523	1.80
825	.265	.552	2.15
1150	.236	.483	2.08

*Fiber was fastened at one end to holder. A weight equivalent to the desired value of tension was suspended from the other end of the fiber which was in a vertical position. The fiber was fastened at the loose end by cementing (Duco usually) to the fiber support. The true tension after mounting is not known but will be examined subsequently.

(Concluded)

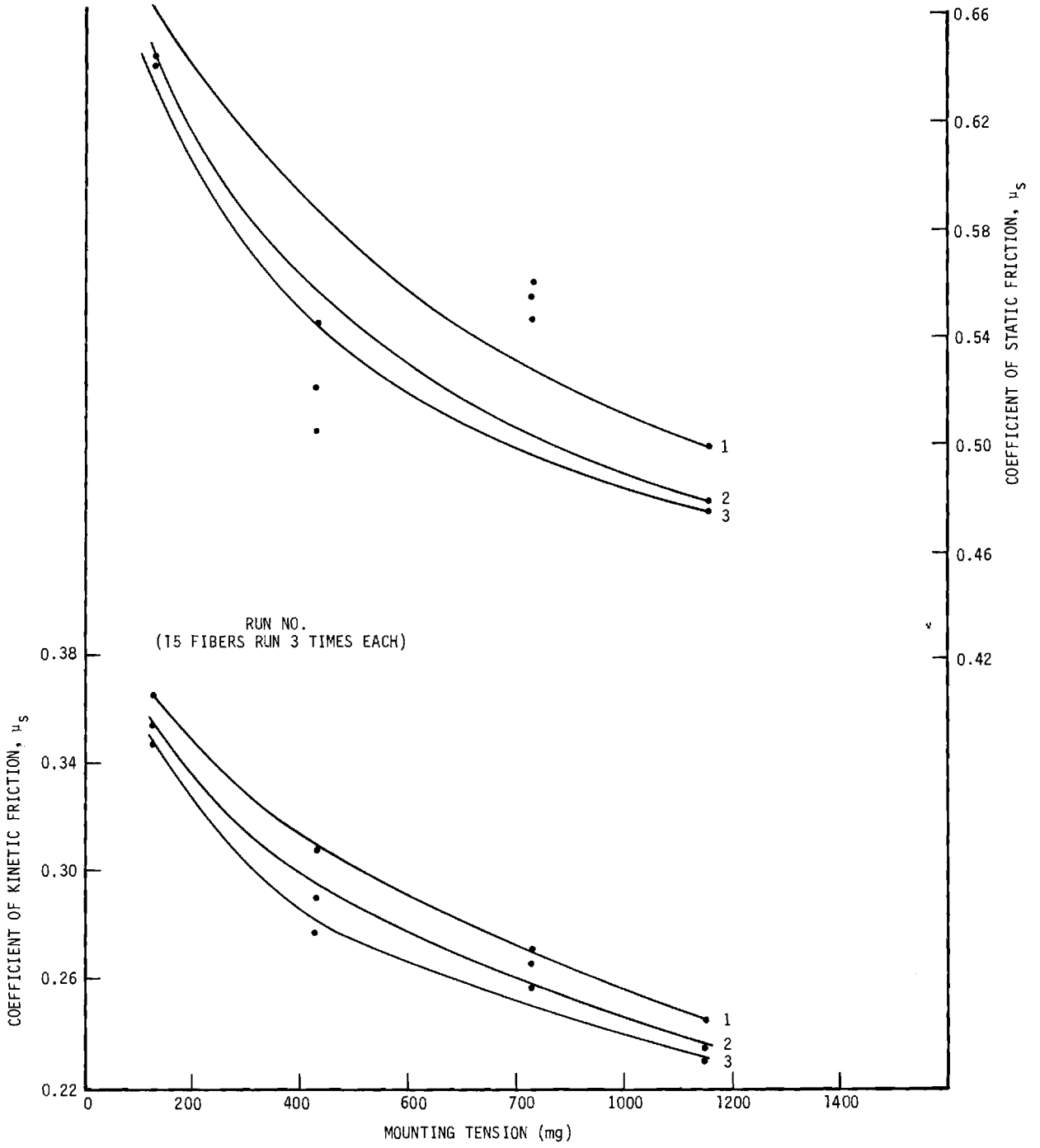


Figure 24. Variation of Coefficients of Kinetic and Static Friction of Empire WR Cotton Fibers with Tensile Force Employed for Mounting.

Here it will be observed that the value of μ_k, measured at a normal force of 20 mg, decreased from 0.356 to 0.236 as the tensile force was increased from 125 to 1150 mg. This action is contrary to the action previously reported by Guthrie, et al, for viscose rayon.* Similarly, the values of μ_s decreased from 0.647 to 0.483.

An interesting point here is that the ratio μ_s/μ_k increased from 1.8 to > 2.1 at tension of 825 mg and higher. Examination of Figure 25 exhibiting photomicrographs of cotton fibers at various tensions exhibits the general straightening out of the fiber. However, at convolutions or reversals a sharp asperity now appears with little or no approaching incline. This results in snagging and the higher stick maxima observed.

An analysis of the problem reveals the following: the contiguous area of the fibers decreased as the tensile force is increased and at low tensions the traversing fiber is considerably displaced from its axis; hence, it is continually climbing a small incline. One might, then, expect a decrease in the coefficient of friction as the fiber tension is increased, in agreement with the result obtained. The effect of the convolutions, however, is to increase the amplitude of sticks, but only for relatively short time periods. Hence, we have the phenomena of relatively higher coefficient of static friction and lower coefficient of kinetic friction.

C. EFFECT OF THREE SUCCESSIVE FRICTION MEASUREMENTS OF THE SAME FIBER PAIR

We have previously discussed in Chapter VI.B the effect of successive measurements of the same fiber. In the case of measurements made for the

*Since the materials and configurations of the viscose and cotton fibers are different it cannot be yet reported that the data of Guthrie, et al, are incorrect. We are discussing only cotton fibers here.

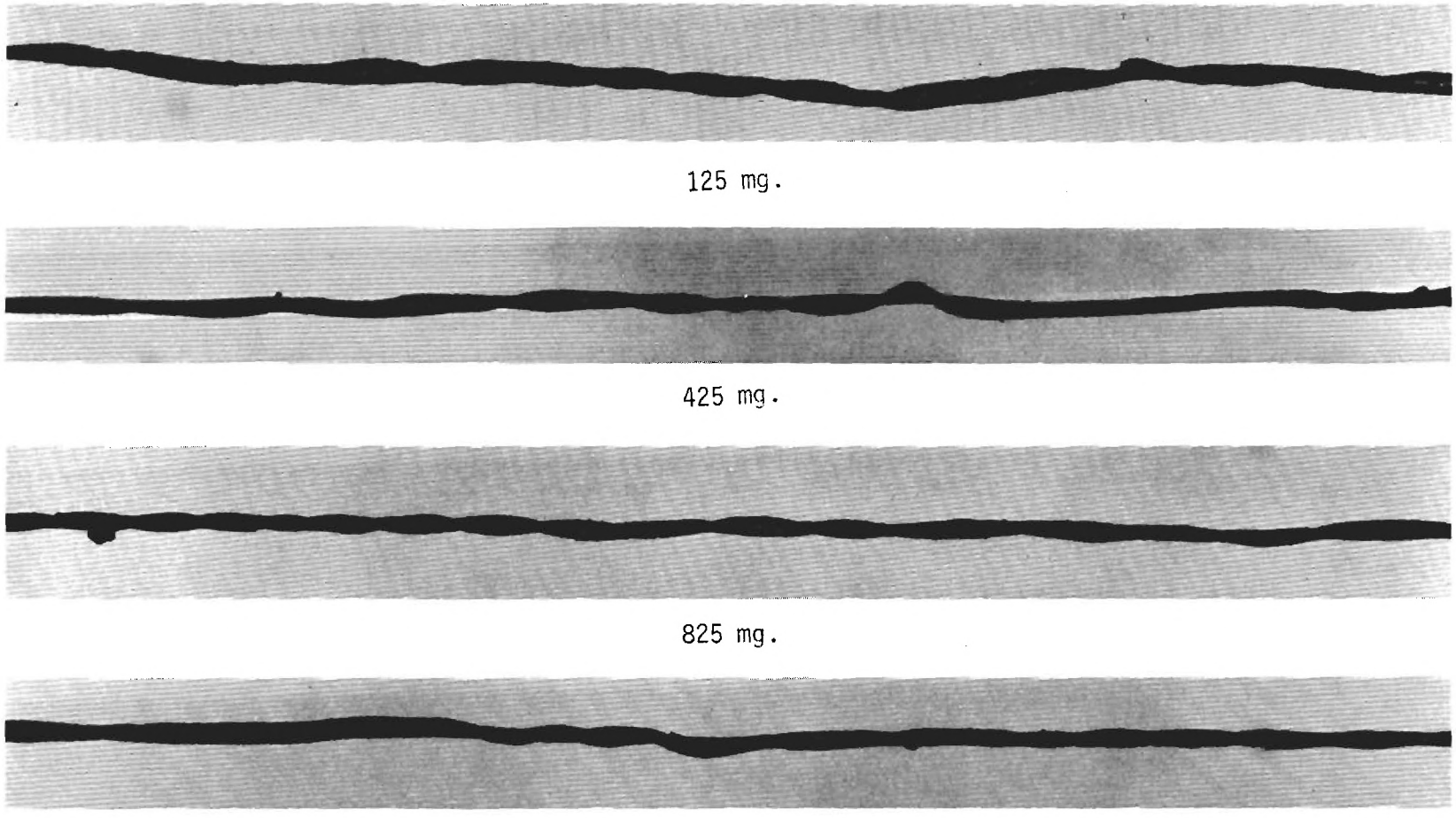


Figure 25. Photomicrographs of Empire WR Cotton Fibers at Mounting Tensions of 125, 425, 825, and 1150 mg.

60 fiber pairs discussed in Section B above, three measurements were made for each fiber pair. These data, as reported in Table 2 and Figure 24, can be averaged and plotted according to the order of the run for the fiber pair (1st, 2nd, or 3rd).

Another method of reporting these data is shown in Figure 26. Here the slope of the plot indicates the change in the coefficient of friction per run and the vertical ordinate between successive plots indicates the effect of tension change.

The anomaly of the inversion of the expected position of the μ_s values for 425 mg and 825 mg cannot be readily explained. However, it is known that the μ_s value, as defined, is quite variable and a poor sample may have given the result obtained. The principal trends in the coefficients of friction due both to tension and successive measurement of the same fiber pair, however, are clearly established. A change in μ_s and μ_k of a few per cent each run due to successive runs of the same fiber pair appeared to result. This was somewhat variable with the fiber, the tension, and the load and was in the range 1 to 5 per cent generally. At the 425 mg level normally used for tension and at a load of 20 mg, a slope of approximately five per cent was experienced for the values of both μ_s and μ_k. Hence, if three values were measured for each fiber pair, essentially a five per cent negative bias of friction coefficients would result for the average of all the measurements. At low normal forces, the change in coefficients due to successive measurements is generally less than the values cited or unobservable. This behavior will be discussed further in Chapter VII.C.

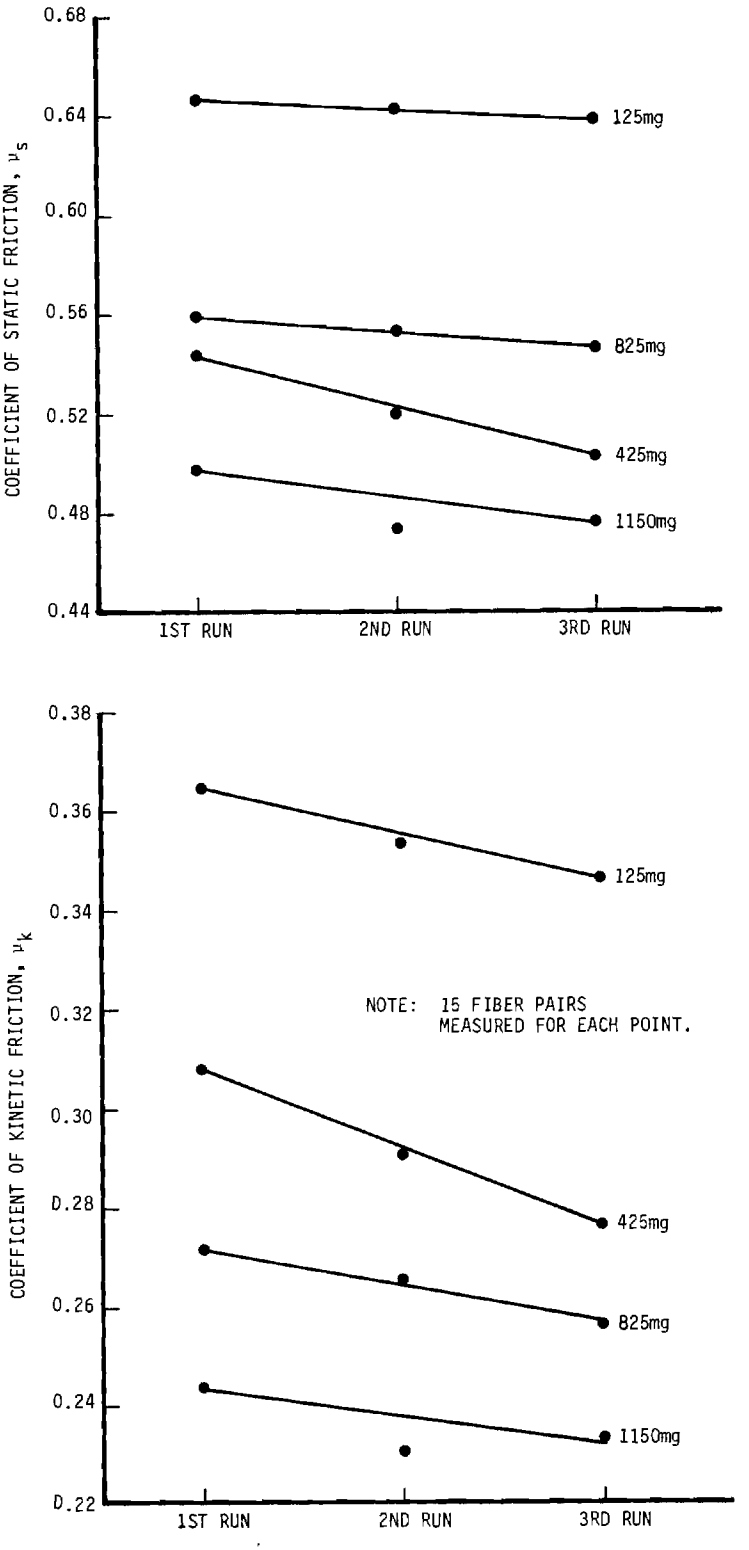


Figure 26. Variation of Coefficients of Kinetic and Static Friction of Empire WR Cotton Fibers with Tension and on Three Successive Traverses with the Same Fiber Pairs.

D. EFFECTS OF TEMPERATURE CYCLING COTTON FIBER ON ITS COEFFICIENT OF FRICTION

Since cotton fiber is subjected to temperatures in the range 80°C to 160°C during the drying process accompanying ginning,[26] the effect of temperature cycling of the fiber on its frictional character was examined.

Specimens of cotton were heated in an oven to temperatures of 70°C, 120°C, 170°C, and 220°C and the frictional parameters of typical fibers were measured. Nine fibers were measured three successive times at each temperature. A summary of the measurements is given in Table 3 and Figure 27.

It will be observed that there are small increases in the coefficients of kinetic and static friction as the drying temperature is increased.

Again small changes (generally reductions) are observed on successive runs with the same fiber but overall consistency is quite good. Especially fibers heated to 120°C or above exhibited little change in values of μ_s or μ_k with successive runs indicating an effect rigidizing the fiber shape. The ratio μ_s/μ_k is interesting in its constancy with a considerable reduction at the 220°C level, a temperature high enough to scorch the fiber. The value of 2.07 is high for 70°C and all thereafter. However, a number of large stick peaks was a characteristic of these curves. This fact is brought out by the higher μ_s/μ_k ratio. This higher value contrasts with the ratio of approximately 1.80 obtained for cotton in the tensile tests series at 425 mg and 25°C. Hence, it appears that even low temperature heating (70°C) has affected the fiber and its intrinsic value of μ_s and μ_k and the μ_s/μ_k ratio. The relative importance of the μ_s value in studying fiber behavior is indicated.

Table 3. Variation of Frictional Coefficients of Empire WR Cotton with Simulated Drying Temperatures

Run No.	μ_k				μ_s				μ_s/μ_k			
	1	2	3	Avg	1	2	3	Avg	1	2	3	Avg
*25°C	0.308	0.291	0.277	0.292	0.544	0.520	0.504	0.523	1.77	1.79	1.81	1.80
70	0.292	0.289	0.275	0.285	0.606	0.586	0.578	0.590	2.07	2.03	2.11	2.07
120	0.300	0.300	0.278	0.293	0.603	0.613	0.569	0.595	2.02	2.05	2.04	2.03
170	0.315	0.321	0.317	0.318	0.656	0.653	0.635	0.648	2.08	2.04	2.00	2.04
220	0.340	0.313	0.327	0.327	0.645	0.602	0.639	0.629	1.90	1.93	1.95	1.92

*These are values taken from Table 2, and were not run at same time.

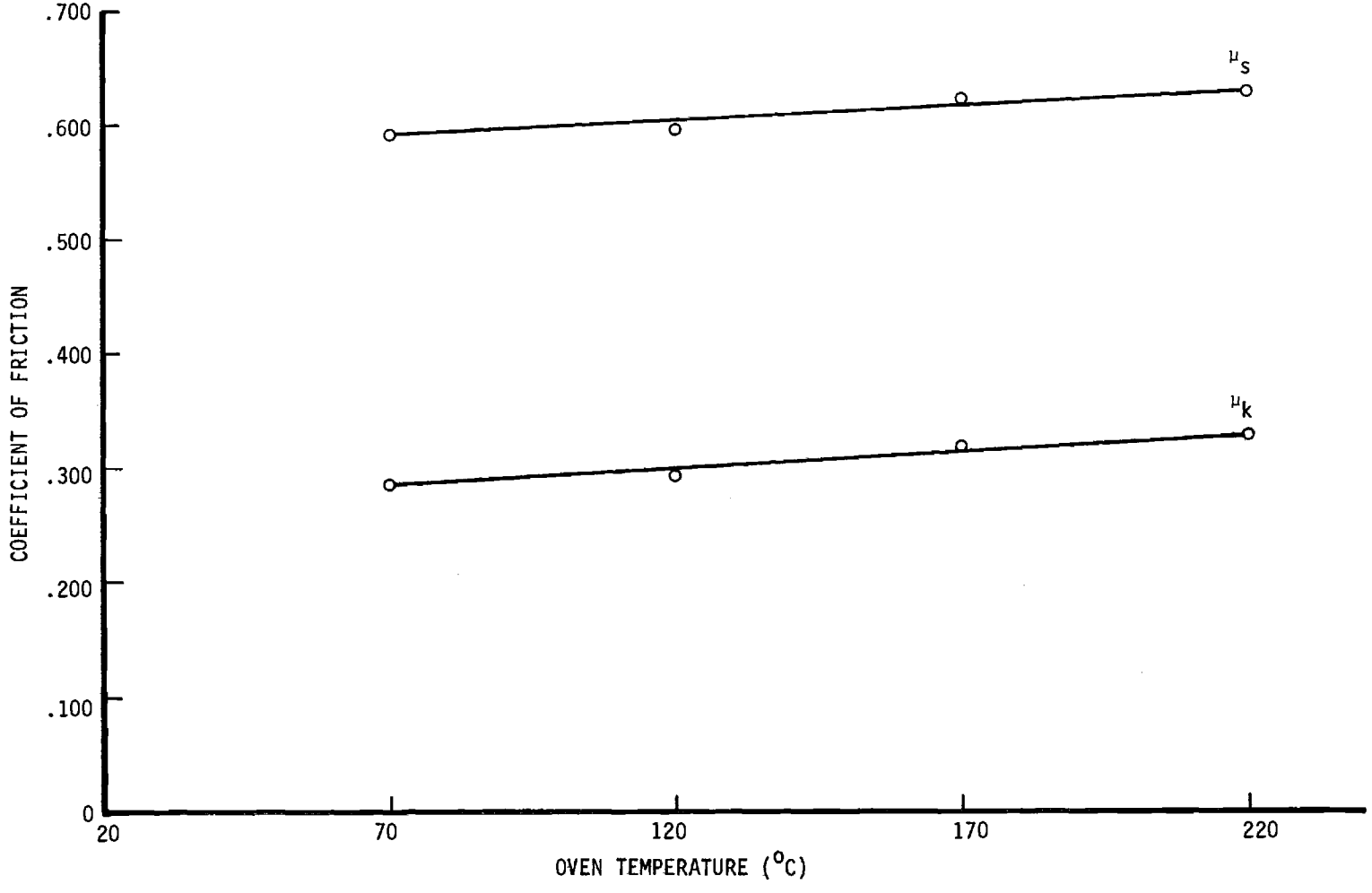


Figure 27. Variations of Coefficients of Static and Kinetic Friction of Empire WR Cotton Fibers Cycled to Selected Temperatures.

E. EFFECTS OF TRAVERSING VELOCITY ON FIBER FRICTION

The traversing velocity of the fiber mounted on the gravity loaded frictional beam has been maintained at about 0.11 mm/sec (0.26"/min) during the majority of its use and except for the earliest measurements performed by McBride[21] in which the traversing rate was approximately 0.32 mm/sec (0.75"/min).

The traversing velocity of the fiber mounted on the electromagnetically loaded (low normal force) beam was 0.1 mm/sec during most of its use. Hence, the velocities of the two systems during the predominant part of their use has been approximately 0.25"/min as compared to the 0.75"/min used in the original version.

Since traversing velocities have been varied considerably by various experimenters in the fiber friction field, and textile processing occurs at high velocities, a close look at the effects of velocity change on the respective coefficients of friction appeared desirable in order to place our own measurements in proper perspective. A series of cotton and nylon fibers were examined at velocities of 0.125 in/min, 0.270 in/min, and 0.540 in/min, and at normal forces of 5 mg and 10 mg. The results are exhibited in Figure 28 for cotton and in Table 4. It will be noted that there is a marked increase in the coefficient of static friction of cotton with increased velocity. The rate of change is about the same at normal forces of 5 and 10 mg. However, the rate of change is small in the case of the kinetic coefficient of friction. The variation of the ratio μ_s/μ_k with velocity is shown in Figure 29.

It is interesting to note here that the lower traversing velocities

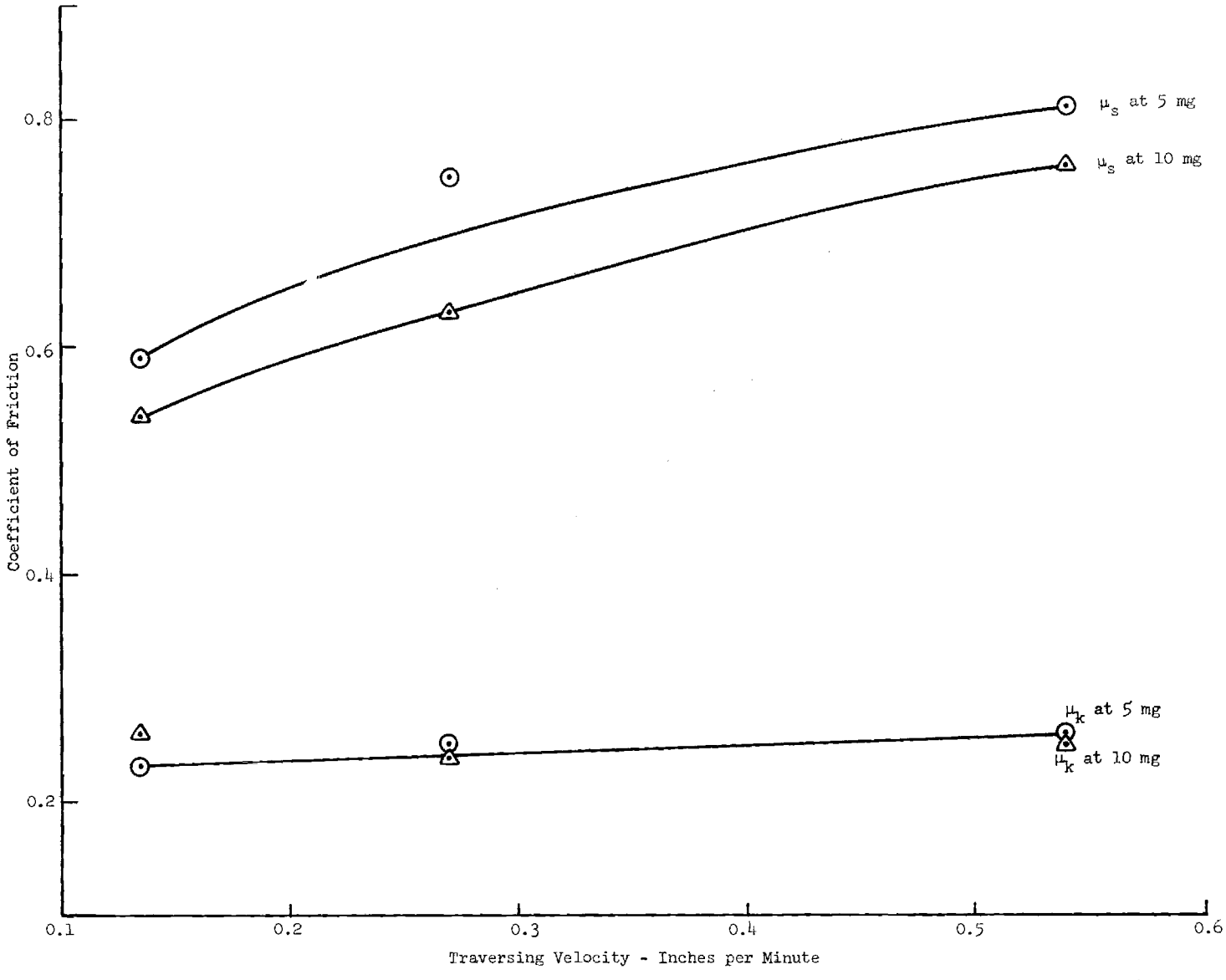


Figure 28. Coefficients of Friction of Cotton on Cotton Versus Fiber Traversing Velocity.

Table 4. Variation of Frictional Parameters of Cotton and Nylon with Fiber Traversing Velocity

Relative Speed (in/min)	μ_s	μ_k	μ_s/μ_k
	Summary Chart - Cotton 5 mg		
.135	.59	.23	2.93
.270	.75	.25	2.96
.540	.81	.26	3.17
	Summary Chart - Nylon 6 5 mg		
.135	.91	.49	1.89
.270	.93	.48	2.01
.540	.95	.45	2.17
	Summary Chart - Cotton 10 mg		
.135	.54	.26	2.14
.270	.63	.24	2.61
.540	.76	.25	3.05
	Summary Chart - Nylon 6 10 mg		
.135	.73	.44	1.66
.270	.80	.45	1.78
.540	.87	.46	1.89

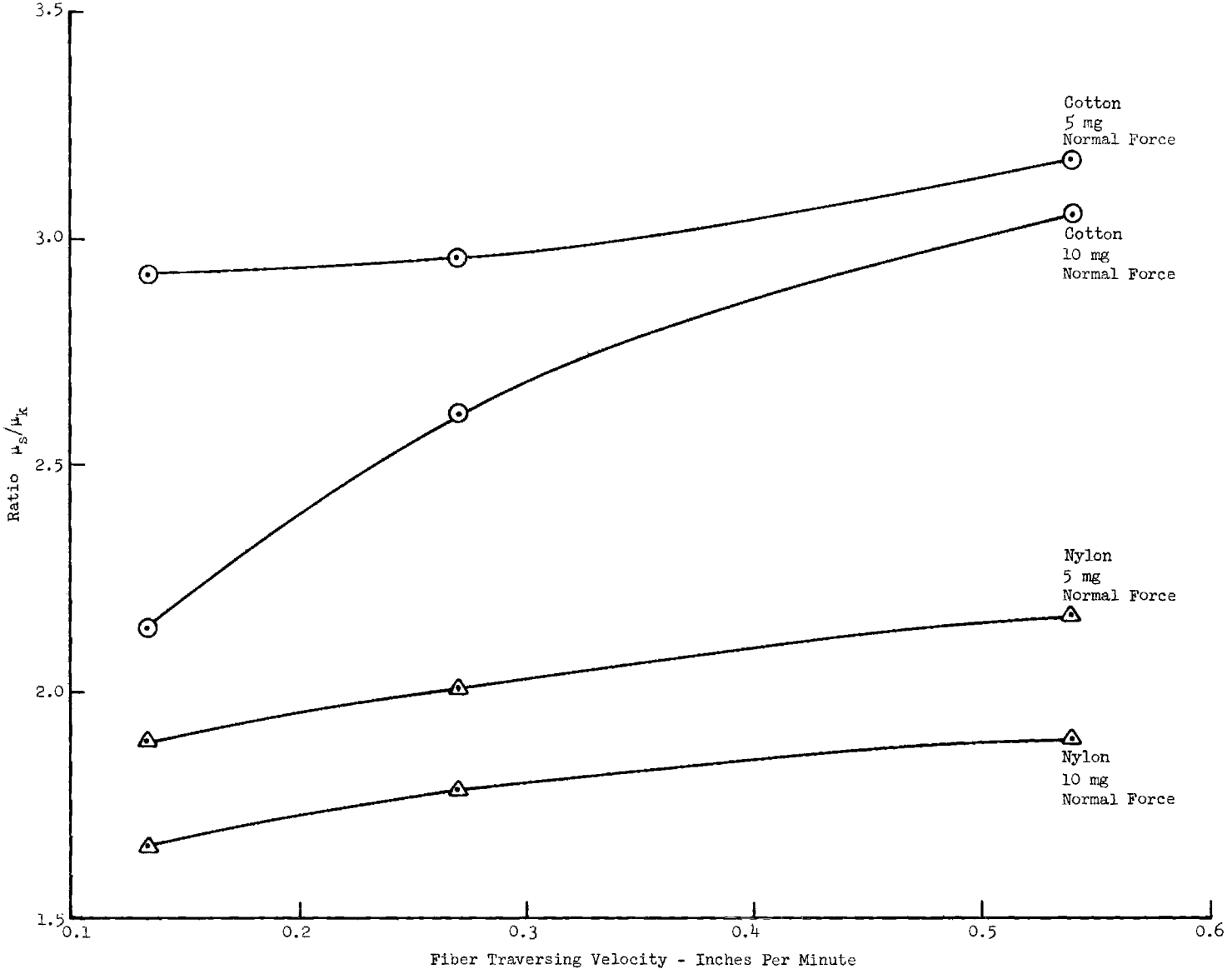


Figure 29. Variation of Ratio μ_s/μ_k of Cotton and of Nylon Versus Fiber Traversing Velocity.

and higher normal forces used subsequent to McBride's work probably accounts in part for the somewhat lower values of μ_s and μ_k obtained for cotton in the work of Bryant[29] and in our Technical Reports 2, 3, and 4,[34, 33, 28] respectively, compared to the values that McBride reported.*

Similar measurements of the variations of μ_s, μ_k, and μ_s/μ_k for nylon on nylon were made at normal forces of 5 and 10 mg and the data obtained are exhibited in Figure 30 and in Figure 29 preceding. It will be noted that the μ_s values exhibit positive trends at both 5 and 10 mg. The μ_s/μ_k ratio exhibits an upward trend. The trend of the μ_k values is negative at 5 mg and positive at 10 mg but is small enough to suggest that the values are within the limit of error and that a larger number of measurements would be necessary to verify if a definite trend actually exists.

F. EFFECTS OF VARIATION OF NORMAL FORCE ON FIBER FRICTION

1. General

The instrument for measurement of friction at low normal forces was described in Chapter III.C.4 of this report. Typical data obtained have been presented in previous reports and in Chapters III and VI of this report. The instrument has the capability of application of the normal force by an electromagnetic means as well as the servo-analog output capability of the gravity loaded beam. For cylindrical fibers, it is capable of operating down to normal forces of about 1 mg as compared to the approximate minimum of 20 mg for the gravity loaded frictional

*Some of McBride's data were taken at about 12 mg normal force which was below the sensitivity of the early instrument which had developed binding in the balance bearings. The data taken at 26 gm normal force was very nearly correct.

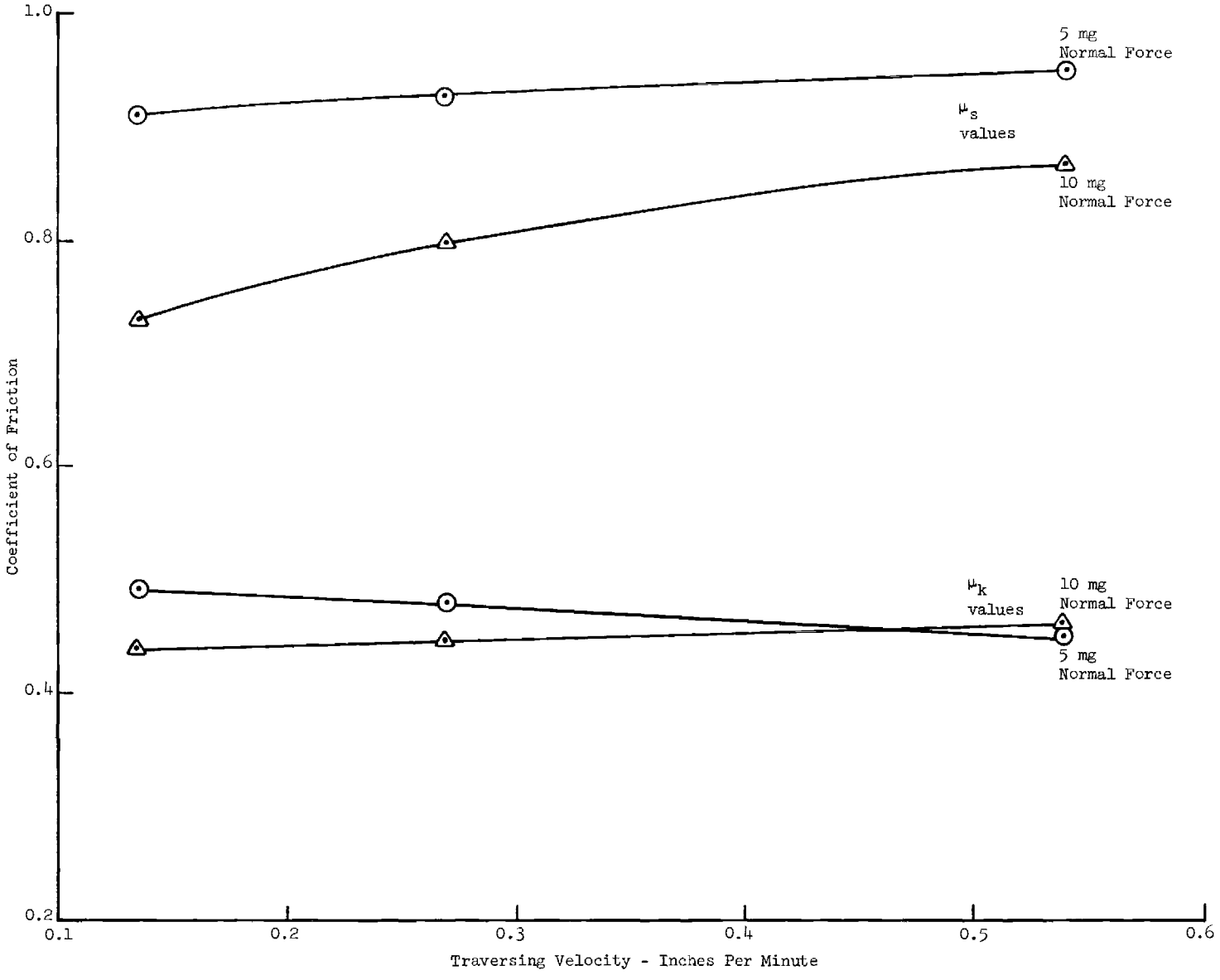


Figure 30. Coefficients of Friction of Nylon on Nylon Versus Fiber Traversing Velocity.

instrument. In addition, it has as an accessory, an automatic integrator allowing rapid data analysis.

In spite of the apparent capability of the instrument and a large amount of data taken with it, some uncertainty of the zero line on the data charts and the unknown degree of susceptibility of the instrument to vibration and inertia effects limited confidence in the results obtained with it in the first series of measurements made with it. These data often appeared to disagree with frictional theory, with measurements with the gravity loaded frictional instrument, and with respect to some measurements made with the instrument itself.

An endeavor was made in a thesis by Mr. Donald H. Gunther, Jr.[22] to completely explore the vagaries of the instrument and to resolve the apparent difficulties. An outline of some of the important results of this work follow and the data are analyzed in respect to the overall frictional behavior of the cotton fiber in textile processing.

2. Measurement of Changes in Normal Force on the Coefficients of Friction of Cotton and of Nylon

In Technical Report No. 4 of this research,[28] some effects of the normal force changes on the coefficients of static and kinetic friction were reported over the range 2 to 10 mg. Also in an earlier report (No. 2)[34] a similar study with the gravity loaded instrument was reported. In each of these, low coefficient of friction values obtained for cotton at the lower normal force ranges were in disagreement with theories of friction and measurements presented by Pascoe and Tabor[12] for nylon and some other fibers (but not for cotton which has not previously been examined extensively). Pascoe and Tabor showed for nylon that large

increases in frictional coefficients occurred as the normal force approached zero. The discrepancy in the shape of the curve obtained by plotting μ_s versus normal force for cotton compared to the shape found by them for nylon and other fibers could only be accounted for by ascribing it to an intrinsic fault of the instrument. For the gravity loaded instrument, the inertia of the arm and some obvious bouncing effects observed in high speed motion pictures of measurements seemed to account for the problem. For the electromagnetically loaded instrument, the fault and the sometimes erratic nature of the results were not so easily resolved. Fortunately, the new data have resolved the problem and added greatly to our comprehension of the overall frictional behavior of the cotton fiber. The supporting data are presented below.

Frictional measurements were made for cotton and nylon fibers over the normal force range 1 to 20 milligrams. Approximately 10 measurements were made at each level. Although the number of measurements was somewhat fewer than desirable, the large coverage of material prevented a large number of measurements from being made at each level. Figure 31 exhibits the plot of the data obtained for the values of μ_k and μ_s for the respective fibers. It will be noted that whereas the nylon curves follow the pattern of the theoretical curve as outlined by Pascoe and Tabor the cotton curve approaches a peak at about 6 or 7 mg and decreases as the normal force decreases.

In Figure 32 (from Technical Report No. 2) we see a similar behavior of the cotton and nylon curves occurring at about 15 mg. Since the previously observed behavior was correctly ascribed to the moment of inertia and dependent vibration period of the instrument, it is now evident that

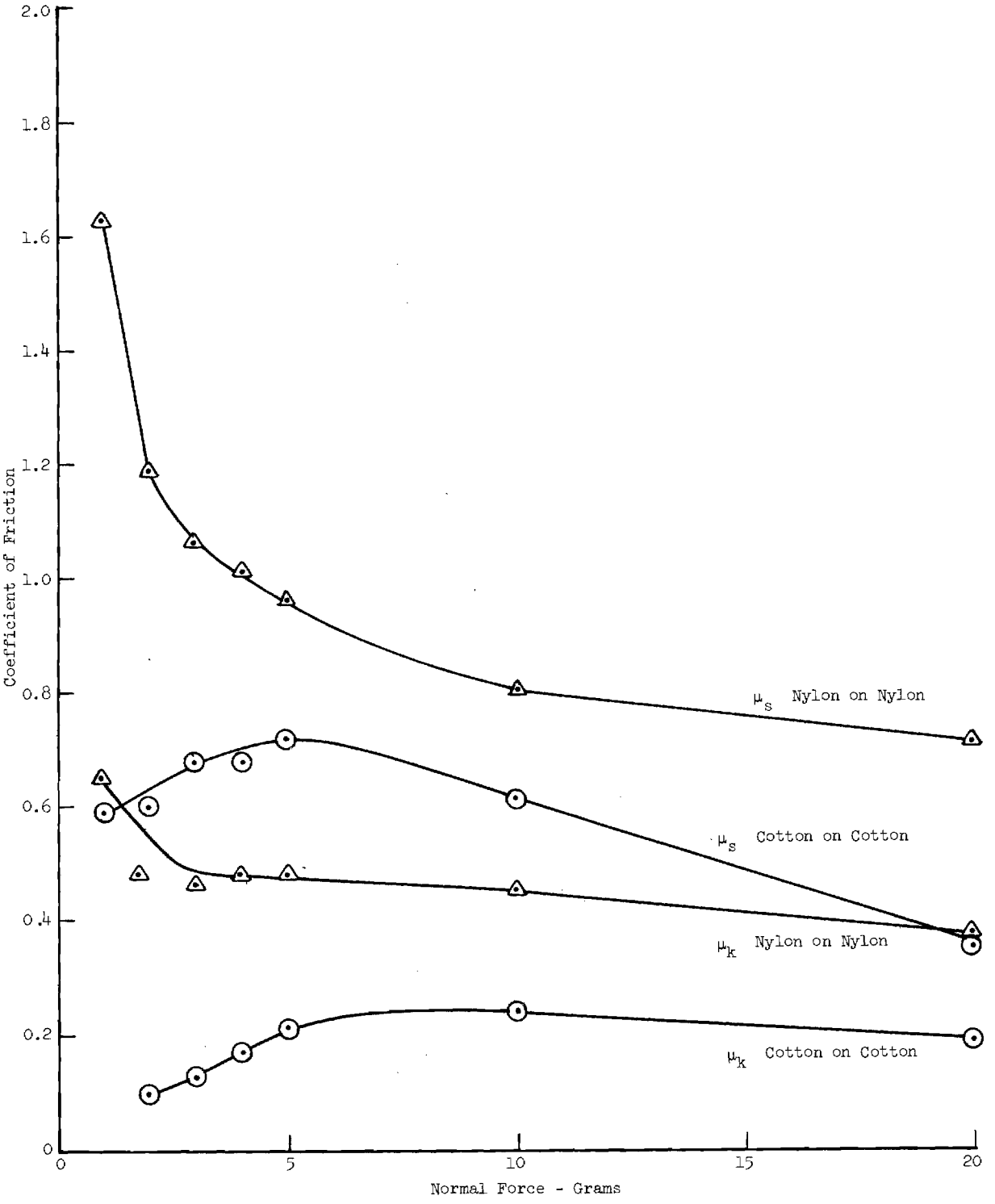


Figure 31. Variation of Coefficients of Kinetic and Static Friction of Cotton and Nylon at Low Normal Force.

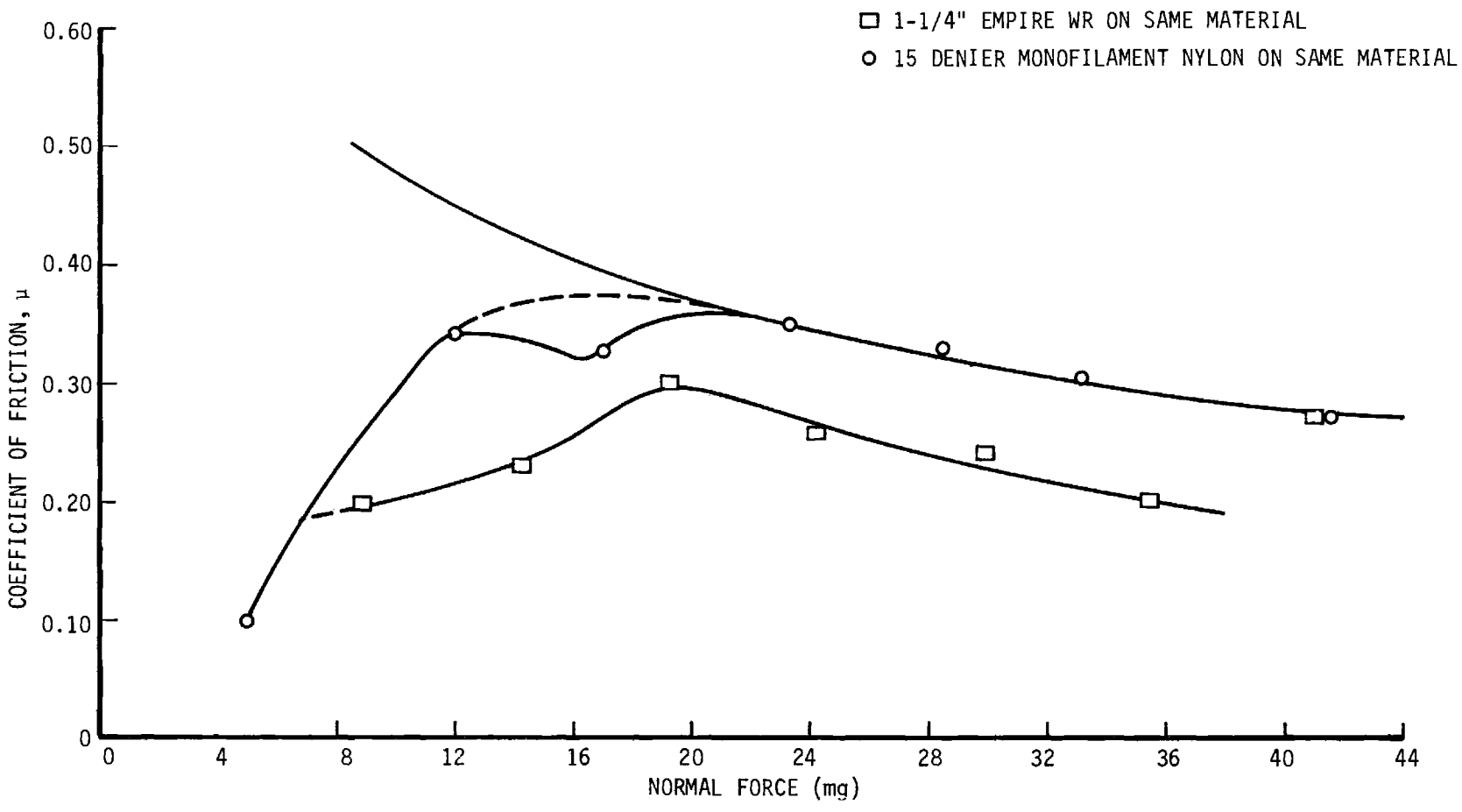


Figure 32. Variation of Coefficients of Kinetic Friction with Normal Force for Single (1¼") Empire WR Cotton Fiber and for 15 Denier Nylon.

the shape factor of cotton has caused a similar behavior in the electromagnetic instrument, i.e., some bounce or time delay of the fibers in making contact after slips, have made the kinetic coefficient no longer valid at normal forces below about 7 mg. This behavior was not interpretable until the concurrent measurements of the performance of cotton and of the performance of cylindrically shaped nylon fibers were compared under as nearly identical experimental conditions as possible.

It is thus evident that a symmetric and cylindrically shaped fiber of relatively smooth surface will furnish with this instrument frictional data at low normal forces closely matching the frictional behavior of Pascoe and Tabor, but that a fiber of complex shape such as the cotton fiber misleads the same instrument at some minimum normal force. For the electromagnetically loaded instrument, the limit is about 6 or 7 milligrams, although on occasion (perhaps with some fibers) and at lower traversing velocities it may be somewhat lower. At the same time, the measurements indicate the great importance of the shape factor of cotton in its frictional behavior.

It is, of course, noteworthy, that here again the μ_k and μ_s values for cotton are lower than for the smoother nylon.

Another point of interest is indicated in the ratios of μ_s/μ_k. These also increase as the normal force decreases as shown in Figure 33. However, the increase in the ratio of μ_s/μ_k for cotton is much greater than in the case of nylon. This behavior is in part due to the lack of capability of the instrument in measuring the μ_k value of cotton below a normal force of about 7 mg; and the steep ascending slope of the μ_s/μ_k plot for cotton as the normal force is diminished, reflects the incorrect

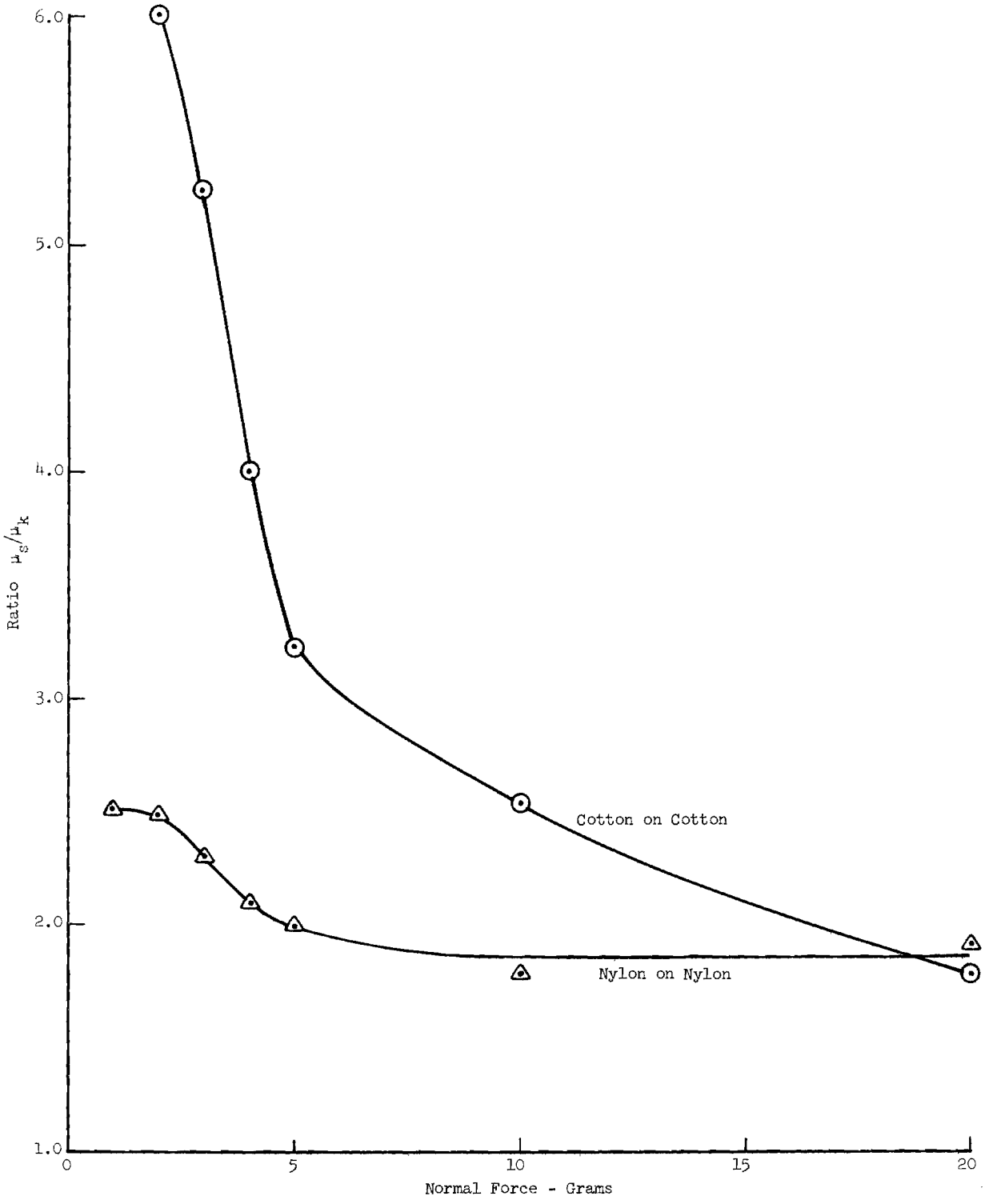


Figure 33. Variation of Ratio μ_s/μ_k with Normal Force for Cotton and Nylon.

values of the true μ_k below this level of normal force. This behavior is obviously related to the irregular shape of the cotton as previously noted. The relatively higher μ_s values may also be important in explaining the mode of fiber travel in fiber processing. Moreover, each stick peak displays a decidedly greater integrated energy total than sticks of any other fiber examined. These high energy values suggest again that the cotton fibers move individually through processing by the snagging action of the high stick phases for adjacent fibers.

3. Effects of Measurement of Fiber Friction With No Externally Applied Normal Force

As discussed in Chapter VI.E and as illustrated in Figures 21 and 22 of that Chapter, it is possible with the friction instrument used to measure friction between two fibers using only the weight of the upper fiber and its cohesive or adhesive force to the lower fiber (a total of a few micrograms) as the normal force. In such instances it is evident that the frictional forces involved are large with respect to the normal force and calculated coefficients of static friction may be as high as the range 80 to 100. Likewise, calculated values of μ_k may be 30 or more.

High adhesion of smooth fibers such as glass on nylon, as shown previously in Figure 21, are observed and factors involving shape for crimped nylon drawn across nylon, as shown previously in Figure 22, and cotton on cotton and nylon on cotton, as shown in Figures 34 and 35 respectively, are exhibited. It is thus evident that smooth cylindrical fibers display high frictional forces at even very low normal forces and that effects of fiber shape are superimposed upon the normal behavior to give friction data and effects that are different in character and magnitude.

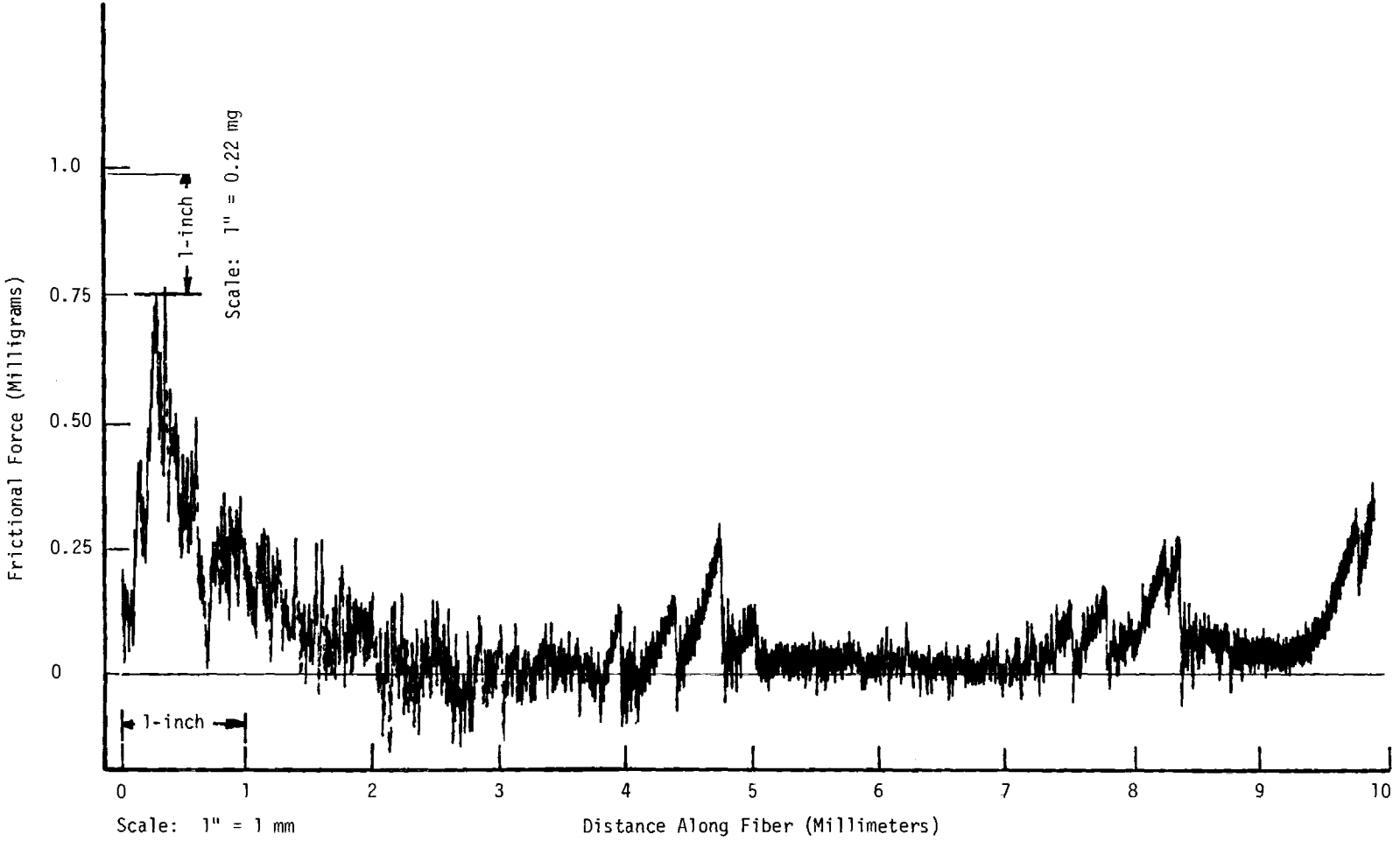


Figure 34. Frictional Data Plot for Cotton Fiber, Supported Only at One End, as It was Drawn Across a Cotton Fiber Attached to the Friction Recording Instrument.

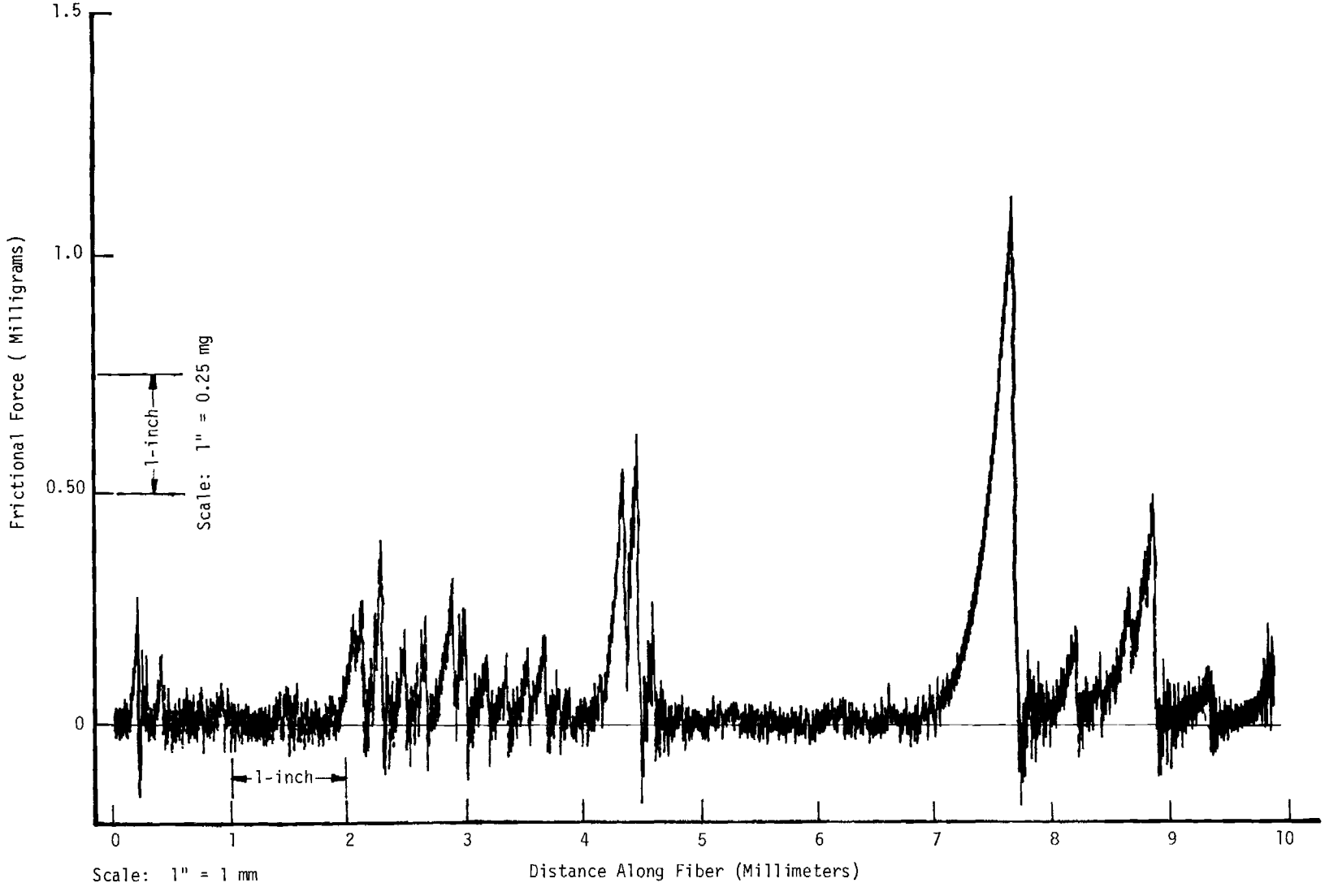


Figure 35. Frictional Data Plot for Nylon Fiber, Supported Only at One End, as It was Drawn Across a Cotton Fiber Attached to the Friction Recording Instrument.

Another method of making measurements of fiber friction at low normal forces is to measure the force of withdrawal required to remove a single fiber from a fiber tuft as also discussed in Chapter VI.E and displayed in Figure 23 of that Chapter.

Another example of data is exhibited in Figure 36. These data are for the extraction of a single fiber from a cotton card sliver specimen. Frictional forces as high as 100 mg are experienced, and the average frictional force of 30.2 mg and the average energy of withdrawal per cm 29.6 ergs are quite high. Undoubtedly, the high forces are partially due to entanglement but they are also intrinsic to fiber behavior in the normal bulk fiber state. Measurements of single fiber extraction forces for cotton card sliver and roving indicated average forces for approximately 20 fibers each of 11.6 mg and 7.5 mg respectively. The reduction occurring in the processing to roving was to a frictional force value 65 per cent of the amount measured for the card sliver.

If we consider that each fiber makes quite a few contacts with the other fibers in the bundle and that we have already seen that the frictional force for drawing a single virtually unconstrained fiber across a second may be 0.5 to 1 mg, we can see that the removal of a single fiber from a bundle might easily give forces ten to 20 times as high as for a single fiber. It is also possible to argue that since the integrated value of the force for the single fiber is more like 0.1 mg than 0.5 mg (the maximum stick value) we can argue that each cotton fiber averages 50 to 100 fiber to fiber contact points while in a reasonably parallelized bundle. If the fibers be more regularly shaped or smoother one may expect a higher number of contact points, a higher total area of

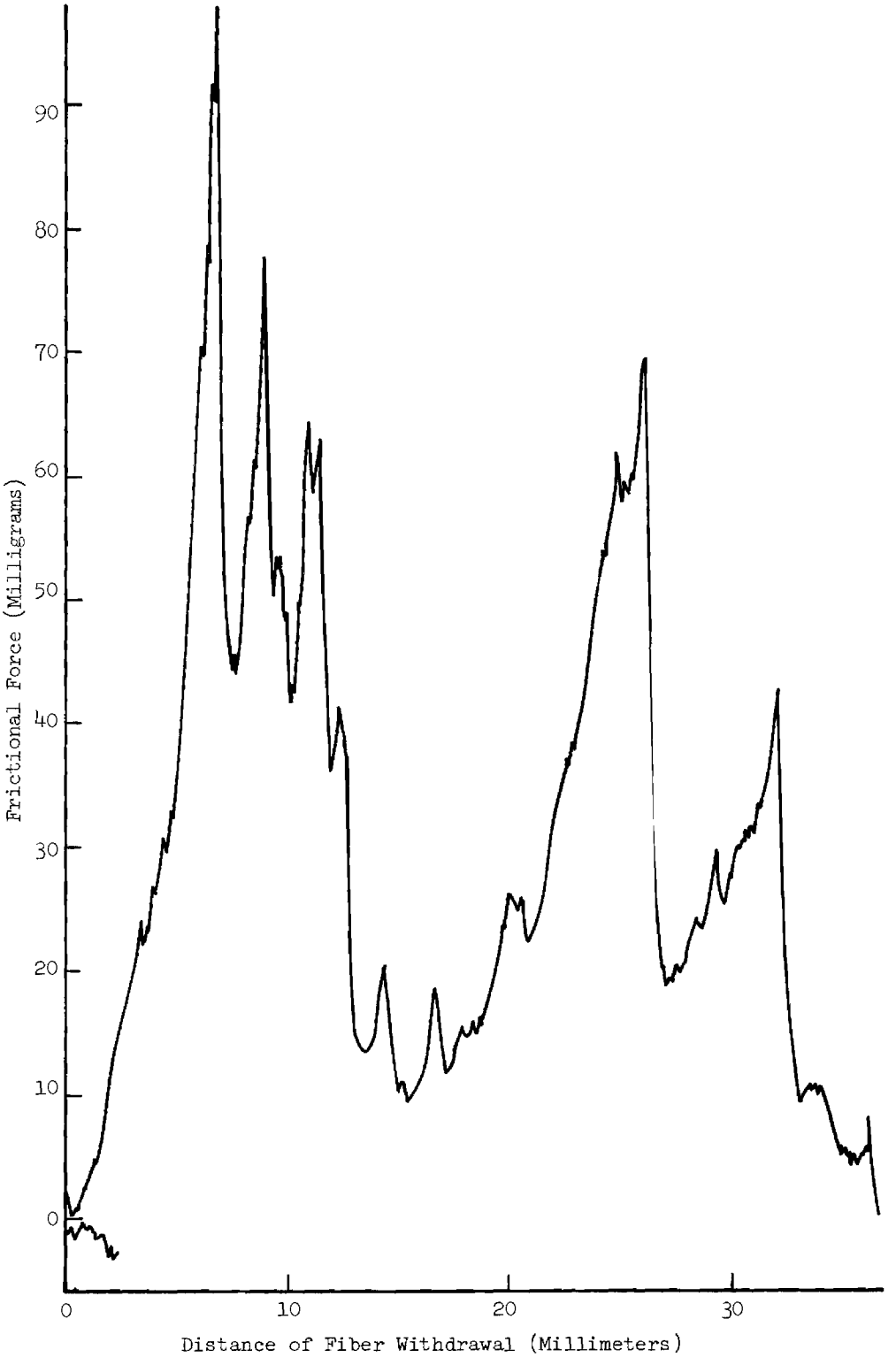


Figure 36. Analog Plot of Force Required to Withdraw a Single Cotton Fiber from Card Specimen (D-4).

contact, and a higher total frictional force. Hence, we are led again to the need for examining the effect of the shape of the fiber on its frictional properties. We must also note the exceptionally high integrated energy within a single peak. This suggests again that the mechanism of fiber motion is related to the high energy required to overcome snagging peaks. Hence, fibers in contact with these may rarely slip by leading to travel in clumps or bundles. A study of shape as applied to nylon fibers is discussed in Chapter VII.B.

G. EFFECTS OF RELATIVE HUMIDITY ON FRICTION OF COTTON FIBERS

In these studies of friction constant temperature and humidity conditions were not available in the laboratories in which the friction measurements were conducted. However, it was possible to establish and to crudely control a desired condition for short periods of time. In addition, records were made of relative humidity and temperature for most of the data obtained.

By collecting appropriate friction data from the data series on Empire WR cotton, hand ginned, made with the gravity loaded friction instrument, it was possible to plot the data to indicate the general trend of the coefficients of friction with relative humidity. These data are shown in Figure 37. Data points are average values for 10 to 20 data curves.

Since data for Empire WR cotton was not available at 45% relative humidity and some for the high draft cotton supplied by Mr. James N. Grant of the United States Department of Agriculture was, the data for the high draft cotton were plotted at this position as is noted in the figure.

The data clearly indicate an increase in the coefficients of fiber

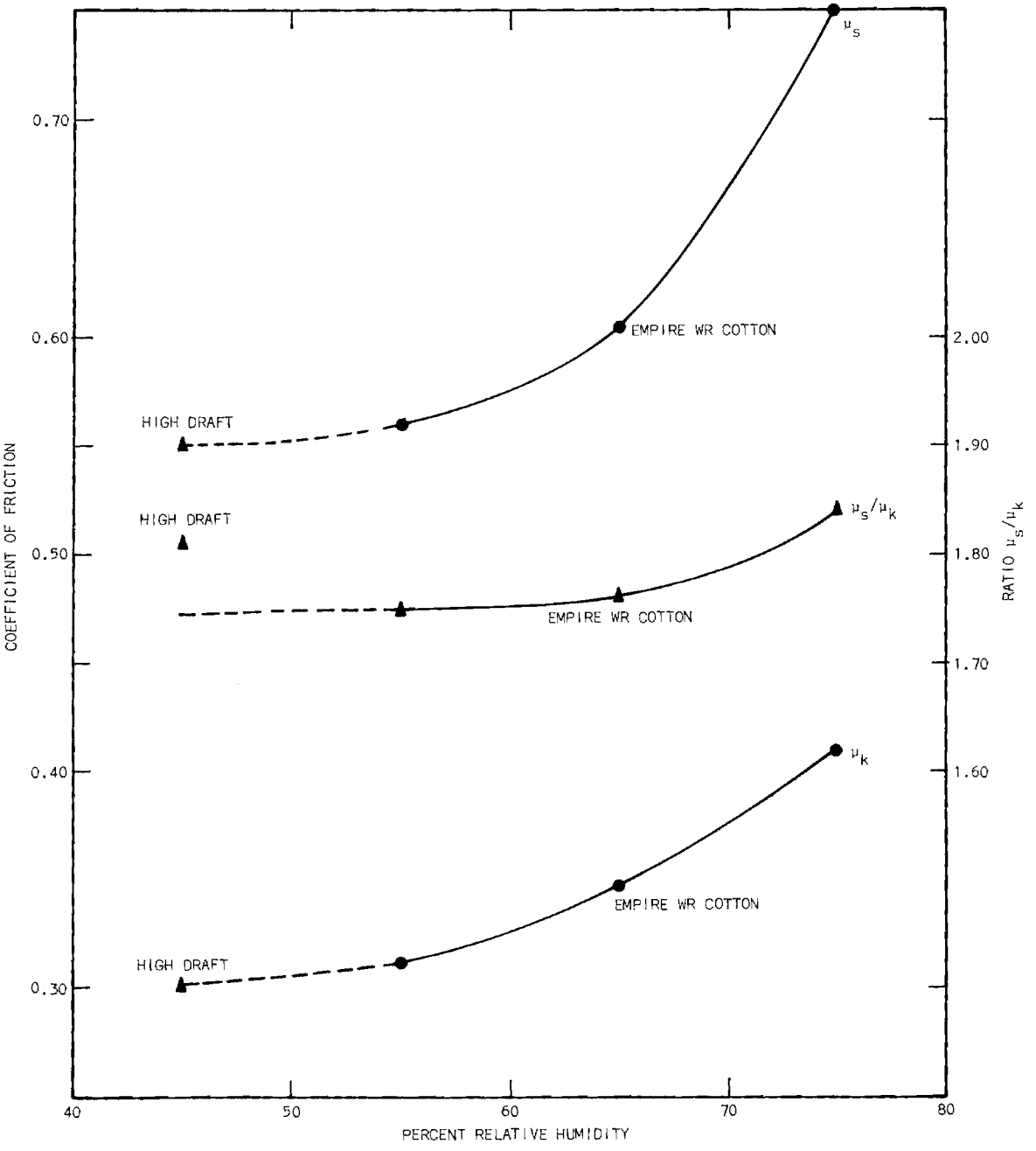


Figure 37. Coefficients of Friction Versus Relative Humidity for Empire WR Cotton (Hand-Ginned).

friction with relative humidity. The rate of change of the slope of the plotted curves becomes marked at 65% relative humidity and even in the selected range 60 ± 5% the total change in friction coefficient is approximately 10% or ± 5%. These data indicate that for precision measurements of the coefficient of friction of cotton they must be conducted under controlled humidity conditions. Measurements conducted at 75% instead of 65% may be high by 25% or more. The value of μ_s varies somewhat more than μ_k but the μ_s/μ_k ratio does not vary markedly, holding close to the 1.80 values suggested from our earliest measurements of cotton.

H. COMMENTS

It is evident from the foregoing measurements that measurement of fiber to fiber friction and the resulting frictional coefficient in a precise quantity requires precise control over a number of experimental factors. Duplicate measurements by several investigators require duplication of the experimental conditions and a similar method of data interpretation. Heretofore, for data reported experimental conditions have varied markedly one from the other and vital details concerning these have frequently been omitted. In addition, methods for interpreting data have not been clarified or have been limited by the absence of analog plots of the frictional data. Instrument limitations or variables have also entered into the problem to an unknown degree.

In this research we have attempted to define the variables and to point to trends resulting from a change of a single variable. We have shown that the measured coefficient of friction of cotton changes with changes in fiber tensile mounting force, traversing velocity, normal

force, relative humidity, and temperature to which the fiber has been cycled. Changes were also observed as a result of successive passes of the same fiber over the fiber on the measuring element. Changes in the coefficient of friction resulting from ambient temperature changes were not measured as a separate parameter but should be. Changes in fiber fineness are also important and the effect is discussed in Chapter VIII.D.

Variations in relative humidity during the course of the measurements imposed a greater scatter in the data than is desirable and reduced the absolute accuracy of any cited measurement. On the other hand, the trends developed by changes of each of the parameters discussed are rather definitely established regardless of some variation in relative humidity from time to time.

The improved instrument, its capability at low normal forces, its capability of delivering analog plots of frictional forces, and the repeatability of the curve character of a single fiber have given us much new information on fiber friction. Very similar instruments appear to deliver some offset in absolute friction coefficient values determined, but the relative values and interpretations derived from them appear to be correct. It is probable that very careful attention to all construction, experimental, and fiber material details will eliminate offsets between like instruments. Since cotton was largely used for those measures, fiber variability alone can cause large offsets unless a very large number of fibers are measured under the same conditions.

This brings us to one of the major problems with understanding frictional properties of fibers through measurements made only on cotton. The effect of shape of the fiber on its coefficients of friction is quite

significant and measurements cannot be properly interpreted until the degree of this effect and its direction can be designated. Thus, we were forced to turn to a fiber the shape of which could be controlled in order to examine the shape variable. This has been done with Nylon 6 and the results will be discussed in the next Chapter.

Another point that comes to mind is that we may be interested in friction at the two ends of the feasible normal force scale, the lowest possible normal force for fiber behavior in processing and the highest possible for fiber behavior in the yarn. Thus far we have looked at the effects at the lowest possible normal forces that we can arrange; and we find very interesting effects with an implication that the coefficient of static friction may be 100 or more in the fiber tuft stage.

Finally, the measurements with respect to effects of tension, relative humidity, and temperature cycling, were only measured with respect to cotton. The effects of tension and relative humidity, especially, might be considerably different for other materials; therefore, the trends outlined are not necessarily transferrable to fibers at large. There thus remains a considerable area of information here yet to be explored.

VIII. MEASUREMENTS OF OTHER FACTORS AFFECTING THE FRICTION OF FIBERS

A. INTRODUCTION

We have discussed in the preceding Chapter the parameters concerning friction measurements of fibers which may be controlled by the experimenter and which are a part of the experimental equipment design or the measurement conditioners. We will now discuss the effects of the factors that may be intrinsic to the fibers, such as its shape or material, and changes that may be wrought upon the fiber by processing, surface treatments, or other conditions which the fiber may experience during its existence.

B. EFFECT OF FIBER SHAPE

1. General

When one considers the problems associated with very small contact zones between spherical or cylindrical objects of small radii, it is clear that the area of contact is a function of the respective radii as pointed out by Pascoe and Tabor and discussed in Chapter III.A. Likewise, it is a function of normal force for elastic bodies and of normal force and time for viscoelastic materials such as many fibers.[4, 5, 12]

If now we consider a fiber such as a cotton fiber, a thin convoluted ribbon, and traverse this across a cylindrical fiber at right angles, the cotton fiber will exhibit a continually varying radius of contact to the cylindrical fiber at the contiguous zone. If it is also traversed across the second fiber at some appreciable velocity (0.1 mm/sec) the stick slip effect is readily observable as has been indicated and the number of

sticks made per unit of traversed fiber length will be less than for a second cylindrical fiber of an equivalent radius (or radii), i.e., for the second cylindrical fiber the total area of swept contact zone would be greater than in the case of the convoluted ribbon.

From the behavior of the measuring equipment and the data obtained for the friction of cotton fibers against cotton and other materials, it was apparent that the effect of the shape of the fiber on its friction must be studied with a material which may be given a desired range of cross-sectional shapes.

2. Material

A manufacturer supplied us with five varieties of 15 denier nylon fibers. Although fibers with no delustering agent would be desirable, the available fibers contained 0.22 per cent titanium oxide as this agent. Fibers of the cross-sectional shapes, circular, duokelion, quasi-triangular, trilobal, and tetrakelion, were obtained. Two of these were illustrated in Figure 10 of Chapter IV.

3. Measurements

The measurements discussed in this section were made by Huff[23] and are outlined in greater detail in his thesis (May 1968). Due to somewhat limited time, the accuracy of the modified low normal force friction measuring instrument, and the consistency of the measurements made with the latter, only 6 fibers of each type were measured against a cylindrical fiber mounted on the servo-controlled galvanometer. Each fiber was measured three successive times (except two times for cylindrical fibers) and measurements were made at normal force levels of 10 mg. Subsequently,

measurements of three fibers of each cross-sectional shape were made at 2 mg normal force.

Data plots typical of each fiber shape are shown in Figures 38 through 42. The data obtained at 10 mg normal force are shown in Figure 5. The data for μ_s, μ_k, and μ_s/μ_k are compared in summary of the table and in Figures 43 through 45.

4. Analysis of Data

The largest quantity of data was measured at a normal force of 10 mg; we will use the data of Table 5 and Figures 43 through 45 as the basis for the general argument. Here it is observed that the cylindrical fiber pair exhibited an average value of μ_s, μ_k, and μ_s/μ_k higher than any other fiber pair. Secondly, successive measurements of the same fiber gave essentially the same average value of μ_s, μ_k, and μ_s/μ_k. The other fiber shapes against the cylindrical shape gave definitely lower values of average μ_s, μ_k, and μ_s/μ_k, than for cylindrical against cylindrical. Furthermore, the average values for successive measurements with the same fiber pair again indicated successive lowering of measured values of μ_s and μ_k indicating shape changes or viscoelastic flow with successive passes of the traversing fiber. This is in essential agreement with the behavior of cotton. Furthermore, the μ_s value obtained for all shapes except cylindrical was about the same as values obtained for cotton (0.52). However, the μ_k values were greater than for cotton resulting in a lower μ_s/μ_k ratio in the range 1.52 to 1.79 at 10 mg. The value 1.79 was for the quasi-triangular fiber and the only shape exhibiting a μ_s/μ_k ratio greater than 1.67 at this normal force.

The similar series of measurements for fewer fibers at 2 mg normal

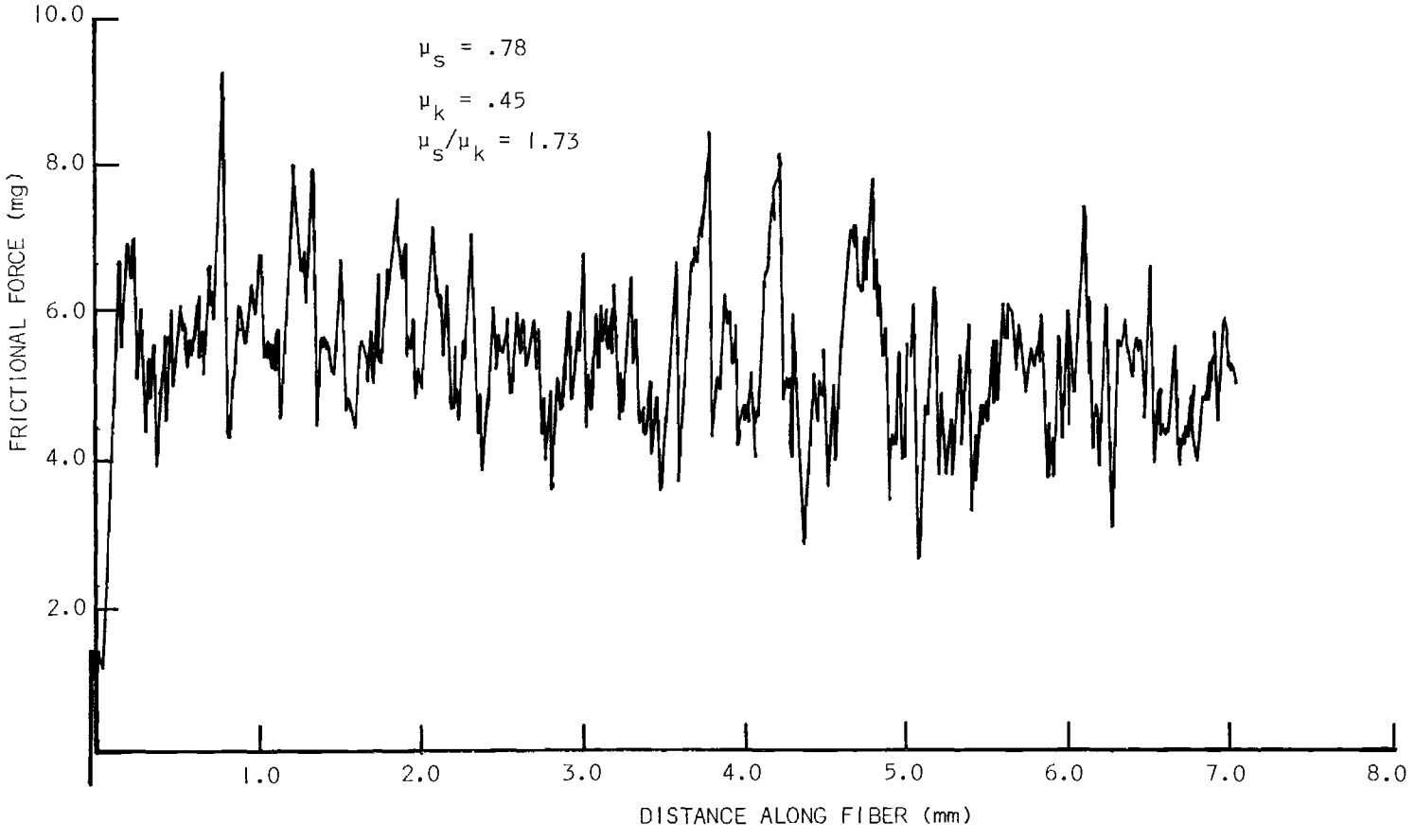


Figure 38. Typical Frictional Plot for 15 Denier Cylindrical Nylon 6 Fiber Against a Similar Fiber.

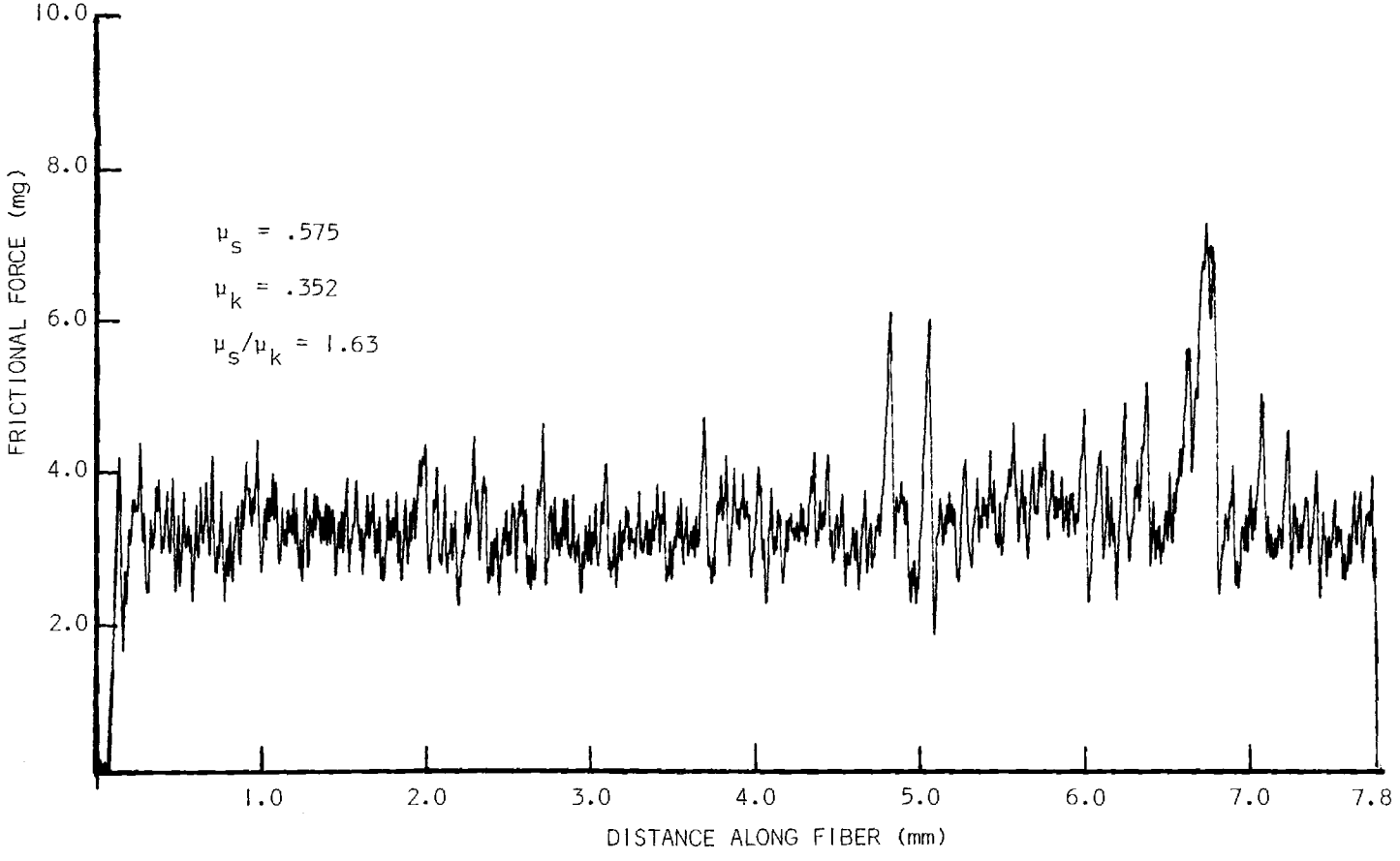


Figure 39. Typical Frictional Plot for 15 Denier Duokelion Nylon 6 Fiber Against a Cylindrical Fiber.

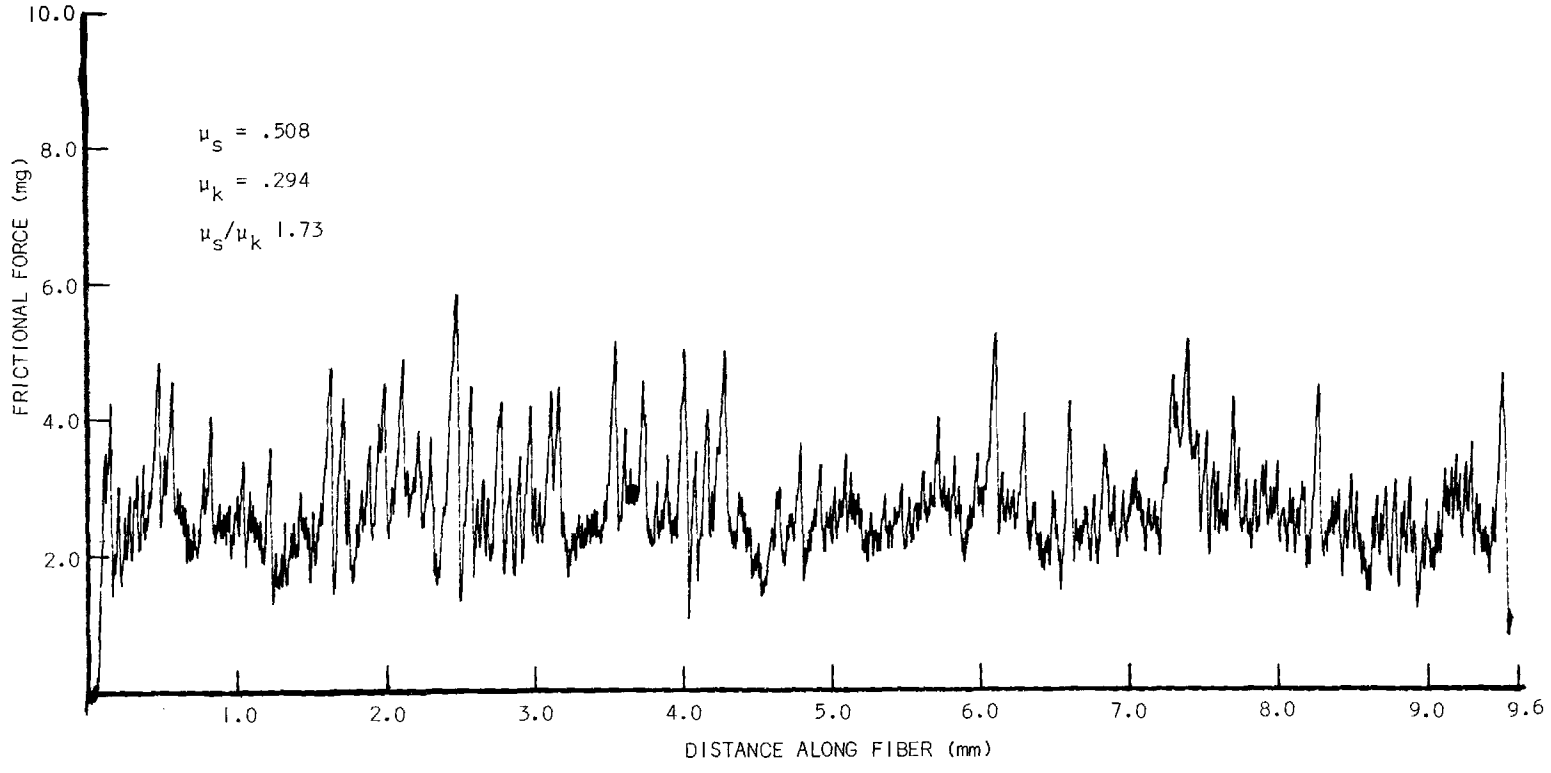


Figure 40. Typical Frictional Plot for 15 Denier Quasi-Triangular Nylon 6 Fiber Against a Cylindrical Fiber.

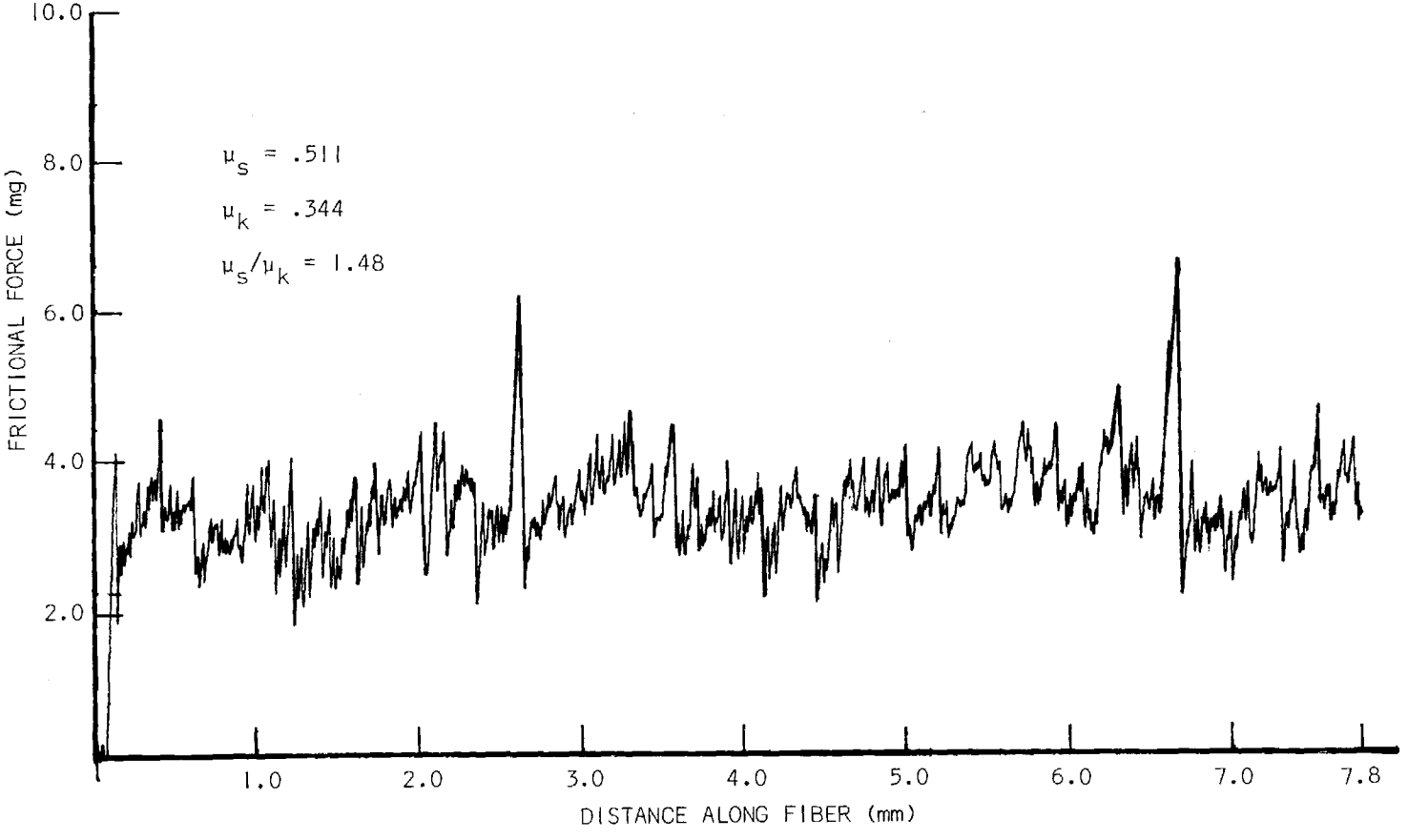


Figure 41. Typical Frictional Plot for 15 Denier Trilobal Nylon 6 Fiber Against a Cylindrical Fiber.

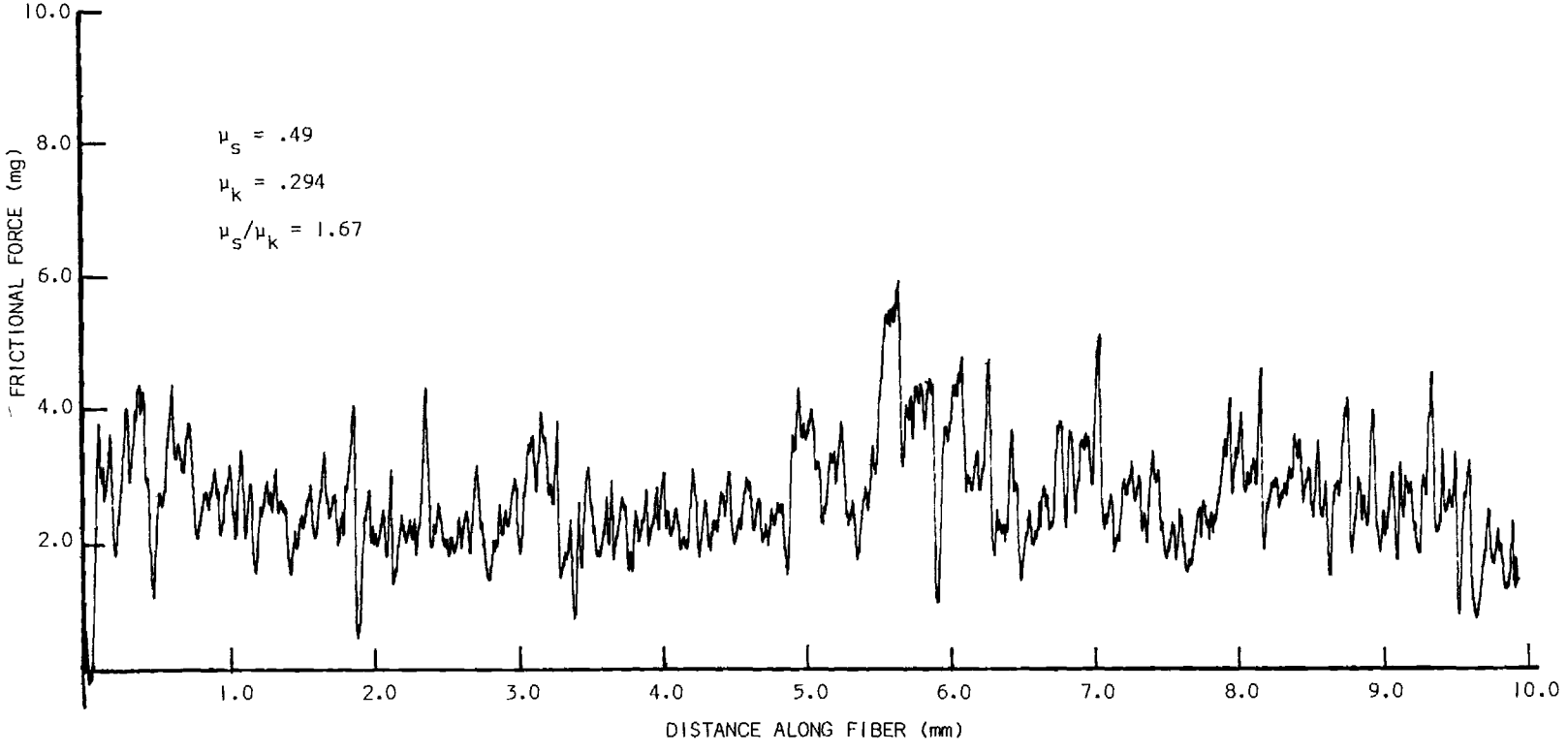


Figure 42. Typical Frictional Plot for 15 Denier Tetrakelion Nylon 6 Fiber Against a Cylindrical Fiber.

Table 5. Friction Versus Fiber Cross-Sectional Shape for 15 Denier Nylon Fibers at Low Normal Force

Fiber Number	Coefficient of Static Friction (μ_s)			Coefficient of Kinetic Friction (μ_k)		
	a	b	c	a	b	c
Nylon 6 Cylindrical on Nylon 6 Cylindrical (10 mg NF)						
1	.76	.77	*	.43	.42	*
2	.77	.76	*	.49	.47	*
3	.71	.71	*	.36	.36	*
4	.68	.67	*	.36	.37	*
5	.63	.62	*	.39	.39	*
6	.71	.70	*	.40	.41	*
Average	.71	.71		.40	.40	
Grand Average		.71			.40	
μ_s/μ_k			1.78			
Nylon 6 Duokelion on Nylon 6 Cylindrical (10 mg NF)						
1	.61	.57	.53	.41	.37	.36
2	.52	.52	.50	.34	.33	.33
3	.59	.58	.48	.36	.35	.33
4	.58	.57	.53	.34	.35	.34
5	.55	.52	.51	.34	.37	.35
6	.49	.48	.36	.34	.34	.34
Average	.56	.54	.50	.36	.35	.34
Grand Average		.53			.35	
μ_s/μ_k			1.52			
Nylon 6 Quasi-Triangular on Nylon 6 Cylindrical (10 mg NF)						
1	.59	.53	.43	.34	.32	.25
2	.67	.60	.66	.32	.29	.34
3	.39	.38	.37	.21	.22	.21
4	.51	.46	.45	.28	.22	.21
5	.57	.57	.57	.34	.33	.32
6	.56	.55	.51	.32	.33	.29
Average	.55	.52	.50	.30	.29	.27
Grand Average		.52			.29	
μ_s/μ_k			1.79			

*Values not obtained

(Continued)

Table 5. Friction Versus Fiber Cross-Sectional Shape for 15 Denier Nylon Fibers at Low Normal Force

Fiber Number	Coefficient of Static Friction (μ_s)			Coefficient of Kinetic Friction (μ_k)		
	a	b	c	a	b	c
Nylon 6 Trilobal on Nylon 6 Cylindrical (10 mg NF)						
1	.51	.47	.45	.34	.33	.32
2	.52	.50	.46	.34	.32	.33
3	.65	.60	.62	.42	.42	.37
4	.55	.51	.51	.36	.33	.33
5	.55	.48	.49	.36	.33	.30
6	.50	.48	.46	.31	.32	.29
Average	.55	.51	.50	.36	.34	.32
Grand Average		.52			.34	
μ_s/μ_k			1.53			
Nylon 6 Tetrakelion on Nylon 6 Cylindrical (10 mg NF)						
1	.55	.53	.49	.31	.31	.29
2	.49	.44	.39	.29	.31	.24
3	.59	.55	.52	.33	.31	.30
4	.47	.44	.46	.31	.28	.26
5	.54	.52	.50	.31	.29	.26
6	.53	.53	.53	.31	.30	.31
Average	.53	.50	.48	.31	.30	.28
Grand Average		.50			.30	
μ_s/μ_k			1.79			

(Continued)

Table 5. Summary of Variation of Frictional Coefficients with Cross-Sectional Shape of Nylon 6 Fibers at Normal Forces of 2 mg and 10 mg

Cross-Sectional Shape	Coefficient of Static Friction (μ_s)		Coefficient of Kinetic Friction (μ_k)		μ_s/μ_k	
	2 mg	10 mg	2 mg	10 mg	2 mg	10 mg
Cylindrical	1.42	0.71	0.61	0.40	2.32	1.78
Duokelion	0.81	0.53	0.46	0.35	1.76	1.52
Quasi-Triangular	1.17	0.52	0.67	0.29	1.75	1.79
Trilobal	0.83	0.52	0.54	0.34	1.54	1.53
Tetrakelion	0.92	0.50	0.56	0.30	1.64	1.67

(Concluded)

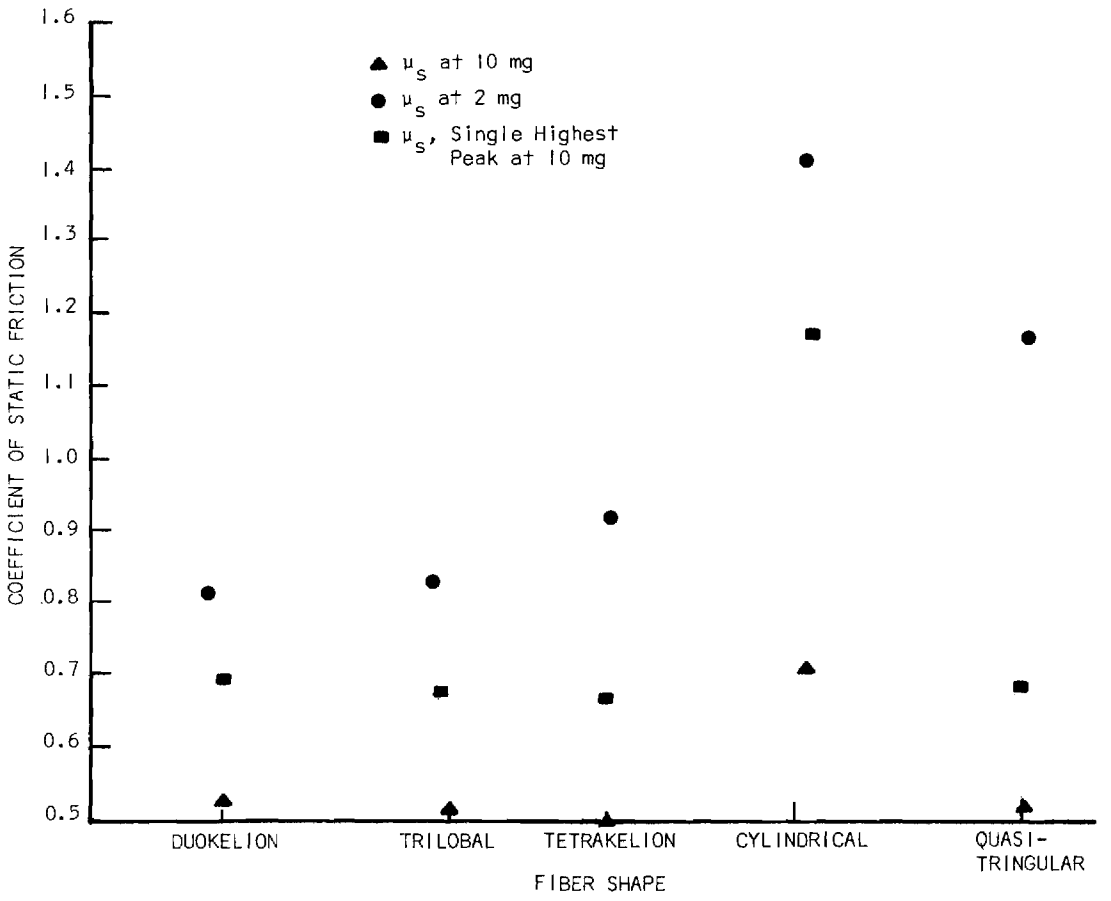


Figure 43. Coefficient of Static Friction Versus Fiber Cross Sectional Shape for 15 Denier Nylon 6.

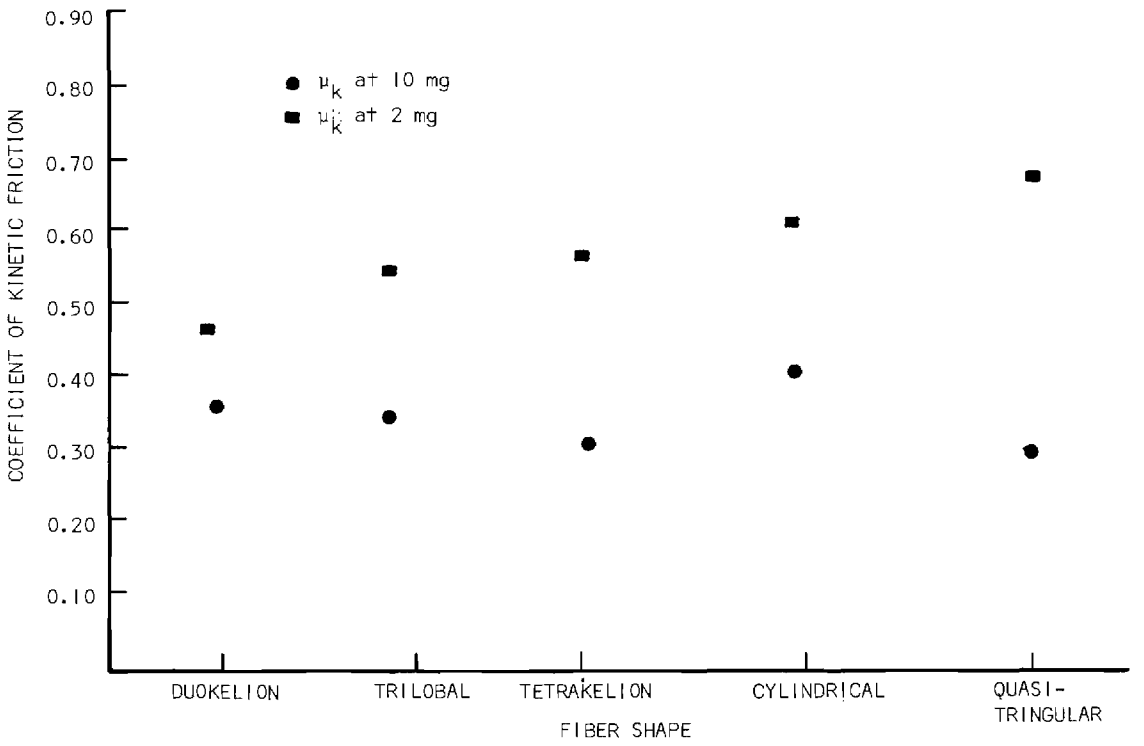


Figure 44. Coefficient of Kinetic Friction Versus Fiber Cross Sectional Shape for 15 Denier Nylon 6.

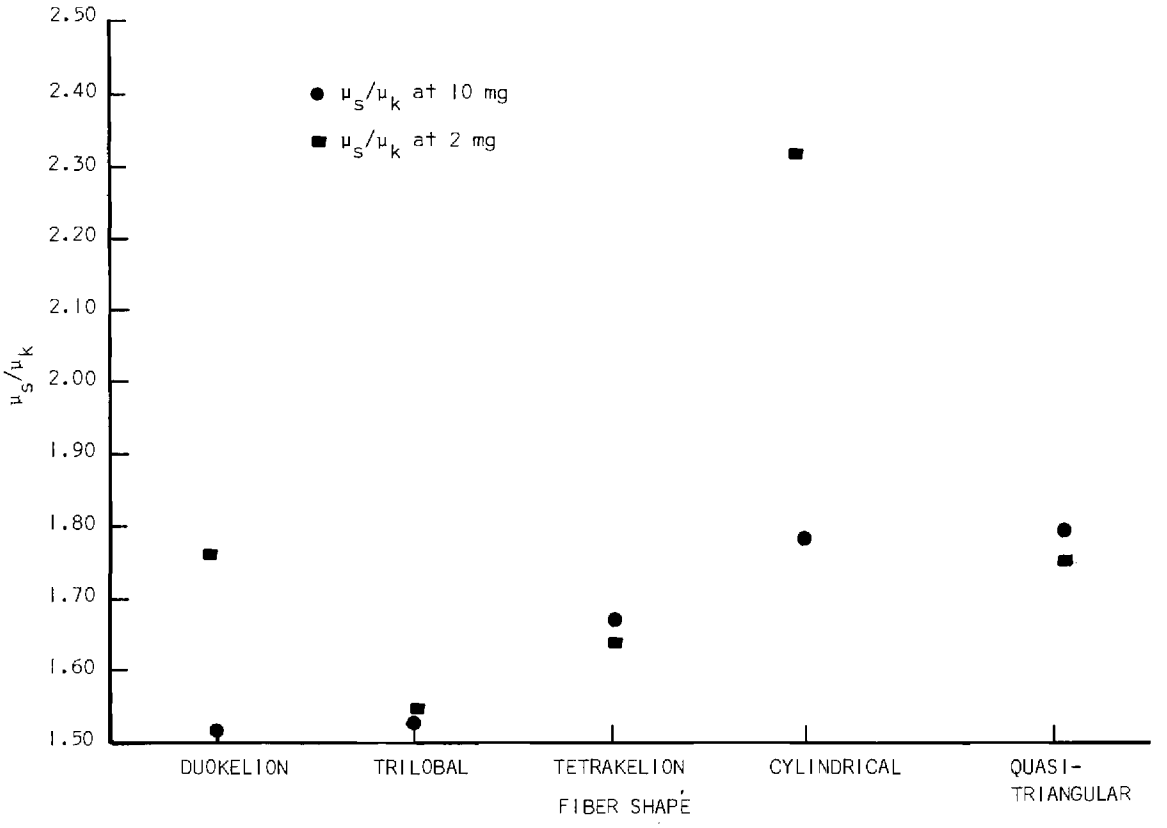


Figure 45. Values of μ_s/μ_k Versus Fiber Cross-Sectional Shape for 15 Denier Nylon 6.

force indicated much the same type of behavior. The cylindrical nylon exhibited the higher μ_s values than any other shape but the quasi-triangular fiber again exhibited behavior setting it somewhat apart from the others registering the highest μ_k value at 0.67 compared to 0.61 for the cylindrical fiber and 0.46, 0.54, and 0.56 for the duokelion, trilobal, and tetrakelion fibers respectively. The values of all the frictional parameters were higher for normal forces of 2 mg than at 10 mg and somewhat more erratic. A portion of the latter behavior can be ascribed to the fewer measurements made which were really insufficient to establish more than the general trend in behavior at the lower normal force. It is interesting to note, however, that the duokelion fiber, which most resembles cotton in cross section, departed to the greater degree from the behavior of the cylindrical fiber.*

It is worthwhile to note that the values reported for cylindrical nylon by Gunther and Huff respectively using the same instrument but modified by Huff at 2 mg normal force compared as follows:

	Gunther (2 mg)	Huff (2 mg)	Gunther (1 mg)
μ_s	1.19	1.42	1.63
μ_k	0.48	0.61	0.65
μ_s/μ_k	2.48	2.32	2.51

Gunther made more measurements than Huff and at 1 mg normal force his were slightly higher than the values cited by Huff for 2 mg normal force. Huff

*An analysis that should be done but which has not been completed is the average energy of the 10 high sticks. These may be highly important to the mode of fiber travel. Each fiber type should also be measured against one of its own shape.

had a somewhat improved instrument and a better integrating device. Since the data plot of coefficient of friction versus normal force has a steep slope in this normal force region fractional adjustment errors will result in large measurement differences. However, the trend of the coefficients here is clearly upward as the normal force is reduced and downward as the shape veers away from the circular section.

A factor not examined was twist in individual fibers which may have had some significance. The known high convolution concentration of the cotton, 3-plus per mm, undoubtedly reduces the μ_k value even lower than for the duokelion nylon fiber. The effect on its frictional properties of twisting the latter fiber should be examined. An analysis of the integrated energy in the higher stick peaks would undoubtedly reveal additional information of value in understanding the mode of fiber travel.

C. EFFECT OF FIBER MATERIAL

1. General

In the preceding section we discussed the effect of the fiber shape upon its friction. In this section we will discuss the friction intrinsic to various materials. Since many natural materials come in a shape specific to the material, the shape will interfere with a true determination of the friction of the material in the form of a cylindrical fiber. Secondly, there is a size effect as reported by Pascoe and Tabor.[12] One approach to a study of this complexity is to measure the fiber friction for the shape existing and to interpolate to the projected friction of the cylindrical form using a suitable correction factor. However, at this stage of our investigation insufficient data are available for an accurate projection. We will therefore present the data as obtained for

various materials in the shape examined and will then analyze to the degree feasible the resulting data with respect to its intrinsic fiber shape.

2. Examination of a Series of Man-Made Fibers

Gunther[22] in his thesis (January 1968) compared friction measurements of Acrilan, Dynel, Orlon, Viscose, Dacron, and Nylon 6. Three successive friction measurements were made for three fiber pairs each. The fiber sections were generally as follows:

Fiber	Shape of Cross Section and Description
Acrilan (Acrylic)	Circular, rough surface
Dynel (Acrylic or Modacrylic)	Dogbone, rough surface
Orlon (Acrylic)	Circular and some dogbone, rough surface
Viscose (Cellulose)	Very irregular, ridged, but roughly circular
Dacron (Ester)	Circular, smooth
Nylon (Polyamide)	Circular
Cotton (Cellulose)	Ribbon, irregular, and rough surface

The measurements were conducted at a normal force of 10 mg, a fiber tension of 425 mg, and a traversing rate of 0.114 mm/second. The specimens were obtained from supplies available in the A. French Textile School of the Georgia Institute of Technology, but the history of each specific specimen, except the cotton, has not been established. The nylon was semigloss Nylon 6 of 15 denier.

Frictional data for typical specimens are exhibited in Figures 46 through 48. The average values for the various materials are detailed in Table 6. These data are compared graphically in Figures 49 and 50.

If one reexamines the data in Table 6 including the columns on cross

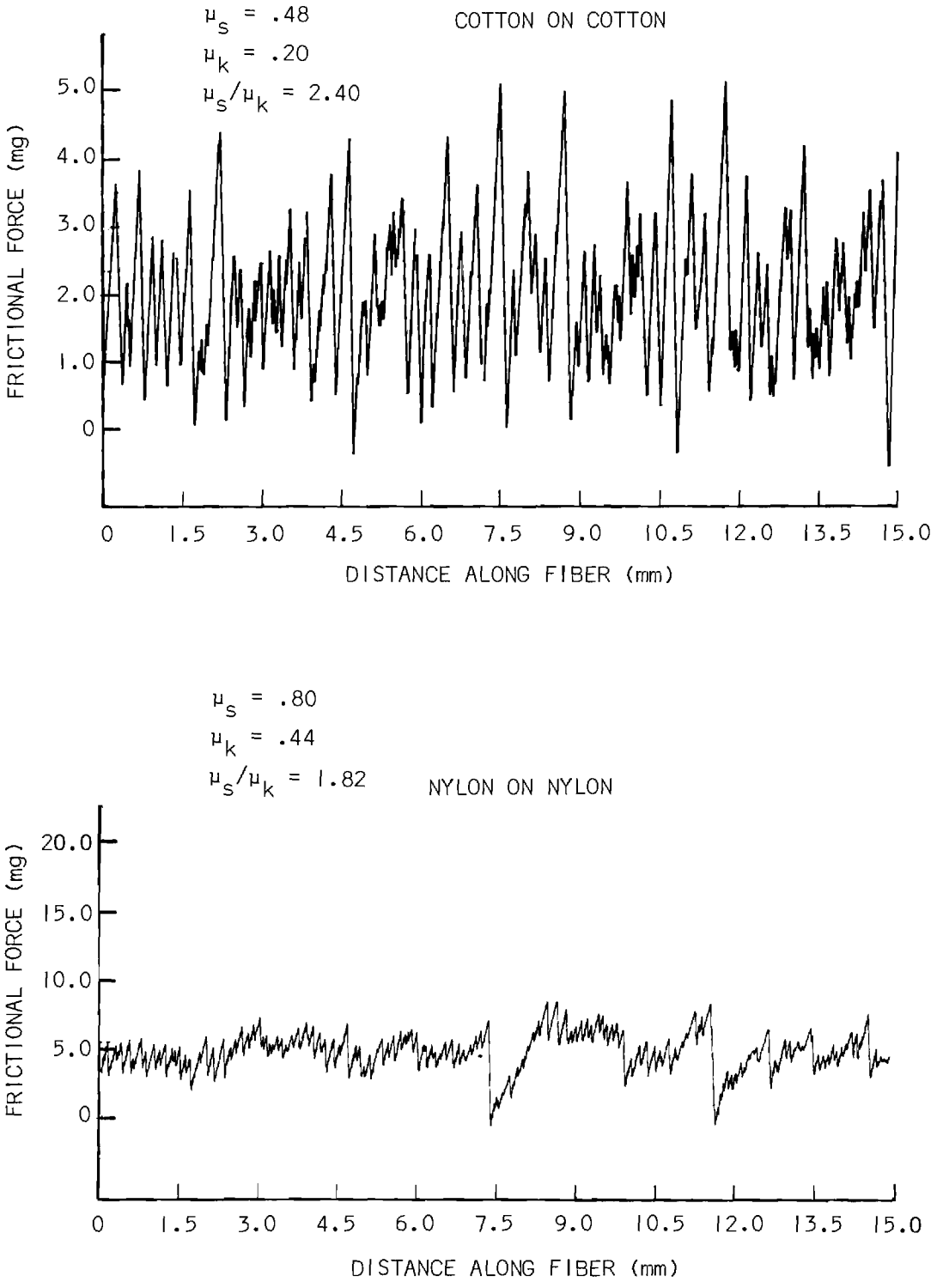
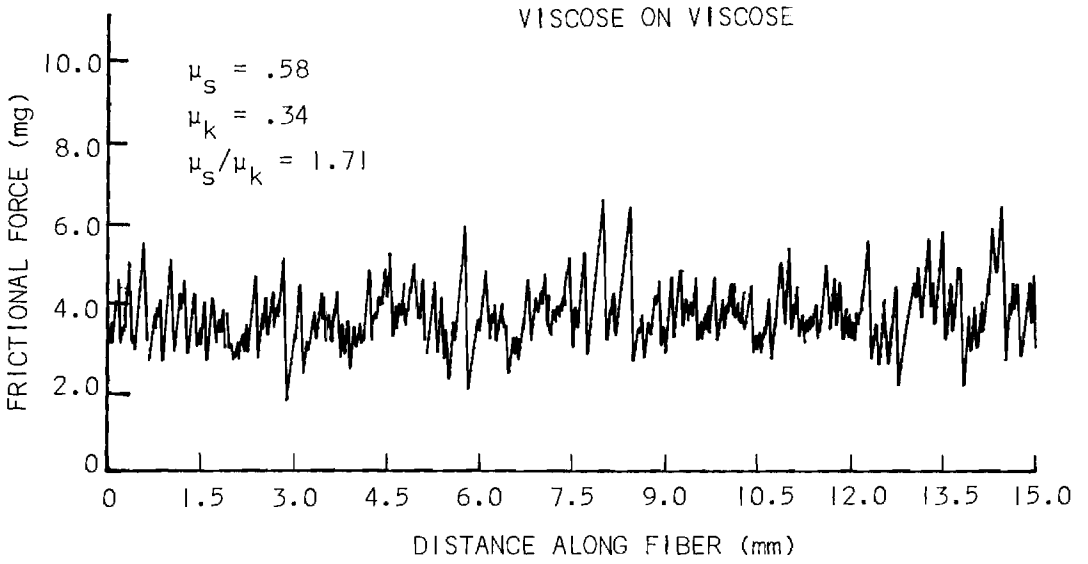


Figure 46. Typical Friction Data Plots of Fiber Pairs of Empire WR Cotton and Nylon 6 at 10 mg NF and .270 in/min Relative Velocity.



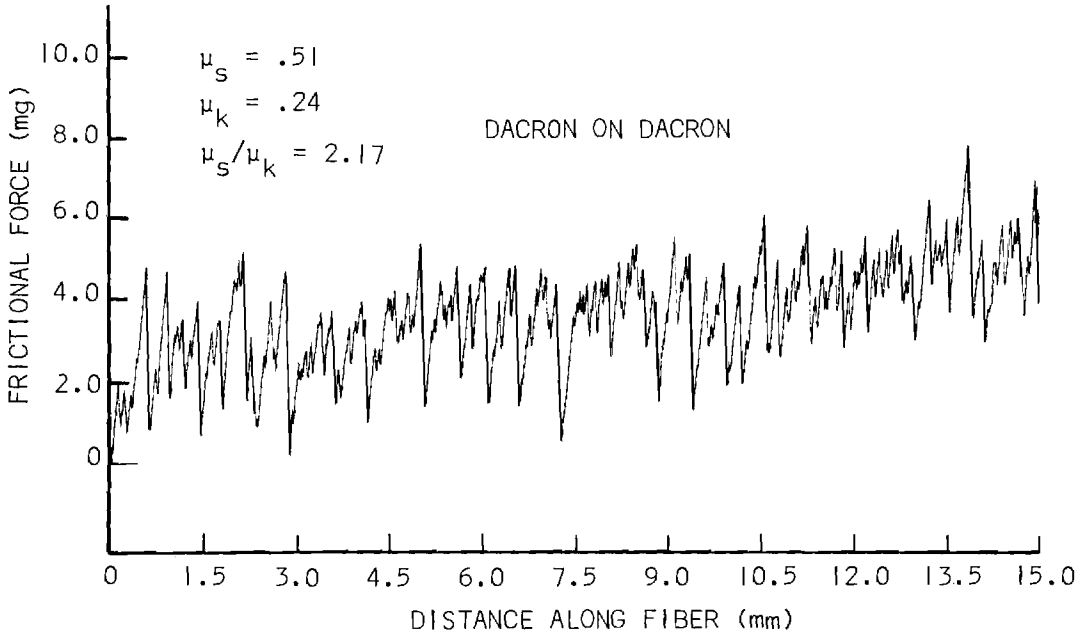


Figure 47. Typical Friction Data Plots of Fiber Pairs of Viscose and Dacron at 10 mg NF and .270 in/min Relative Velocity.

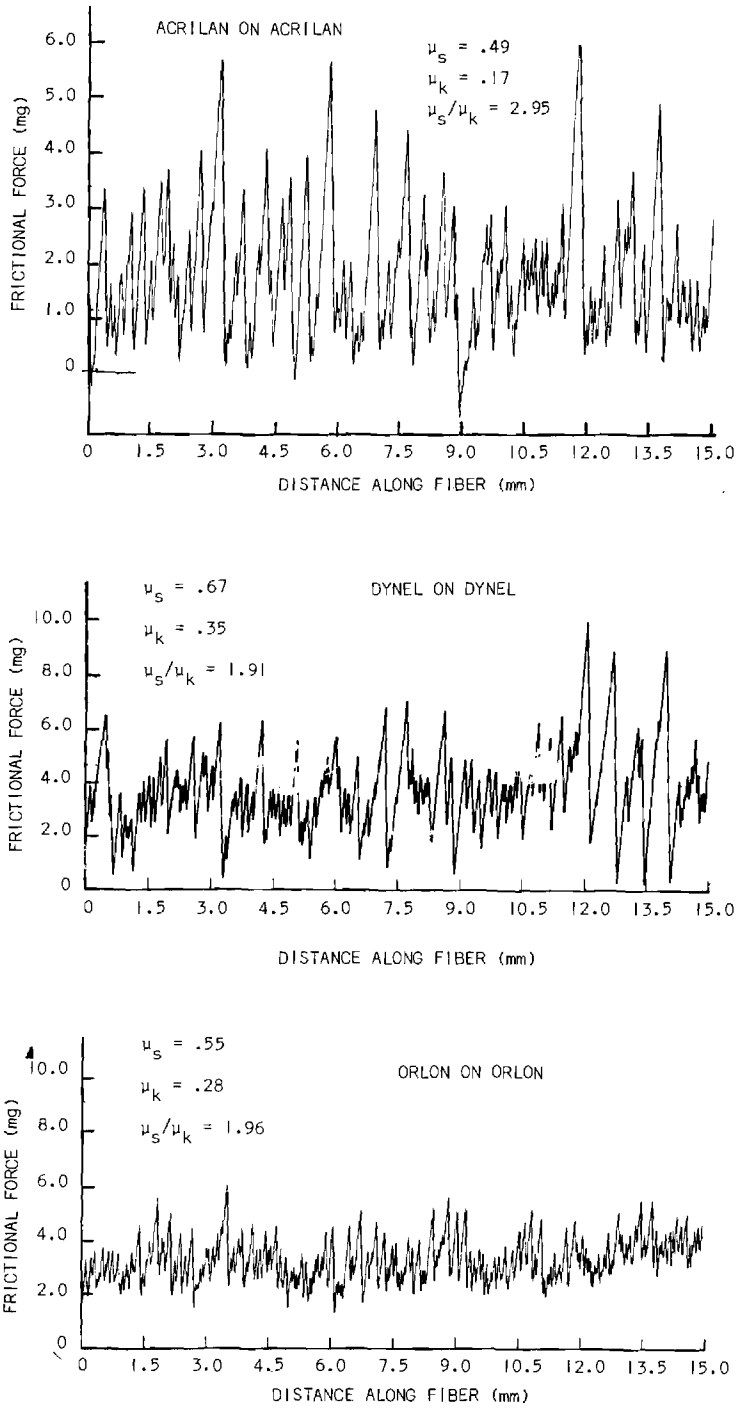


Figure 48. Typical Friction Data Plots of Fiber Pairs of Acrilan, Dynel, and Orlon at 10 mg NF and .270 in/min Relative Velocity.

Table 6. Frictional Parameters for Various Fibers*

Material	Ratios of Areas of Fiber Cross Sections	(Data of Column One)$^{\frac{1}{4}}$**	μ_k	μ_s	μ_s/μ_k	Description
Cotton	1.00	1.00	0.24	0.62	2.58	Ribbon, rough, twisted
Dacron	1.06	1.01	0.24	0.51	2.17	Circular, smooth
Acrilan	1.35	1.08	0.17	0.49	2.95	Circular, rough
Orlon	1.35	1.08	0.27	0.53	1.97	Circular, rough
Viscose	1.60	1.11	0.34	0.55	1.72	Irregular circle, rough
Dynel	2.00	1.19	0.36	0.67	1.86	Dogbone, rough
Nylon	15.00	1.94	0.45	0.80	1.78	Circular

*Normal Force 10 mg; tension 425 mg; traversing velocity = 0.270"/min = 6.86 mm/min = 0.114 mm/sec.

**Using area of cross section of cotton as denominator in accordance with the expression provided by Pascoe and Tabor, $\mu = C_1 \, S \, W^{-0.26} \, D^{0.52}$ for crossed nylon cylinders where C_1 is a constant of about 1.4, S is the shear strength of nylon set at 1.5 kg/mm^2, W is the normal force, and D is the fiber diameter. Hence, the frictional force and coefficient should vary as $D^{\frac{1}{2}}$ or Area$^{\frac{1}{4}}$. We can then compare the fourth roots of column 1 against actual increase in frictional coefficients with some degree of confidence that they will give reasonable agreements with values in columns 3 or 4. Pascoe and Tabor were discussing only cylindrical fibers and μ_s values where all sticks were counted.

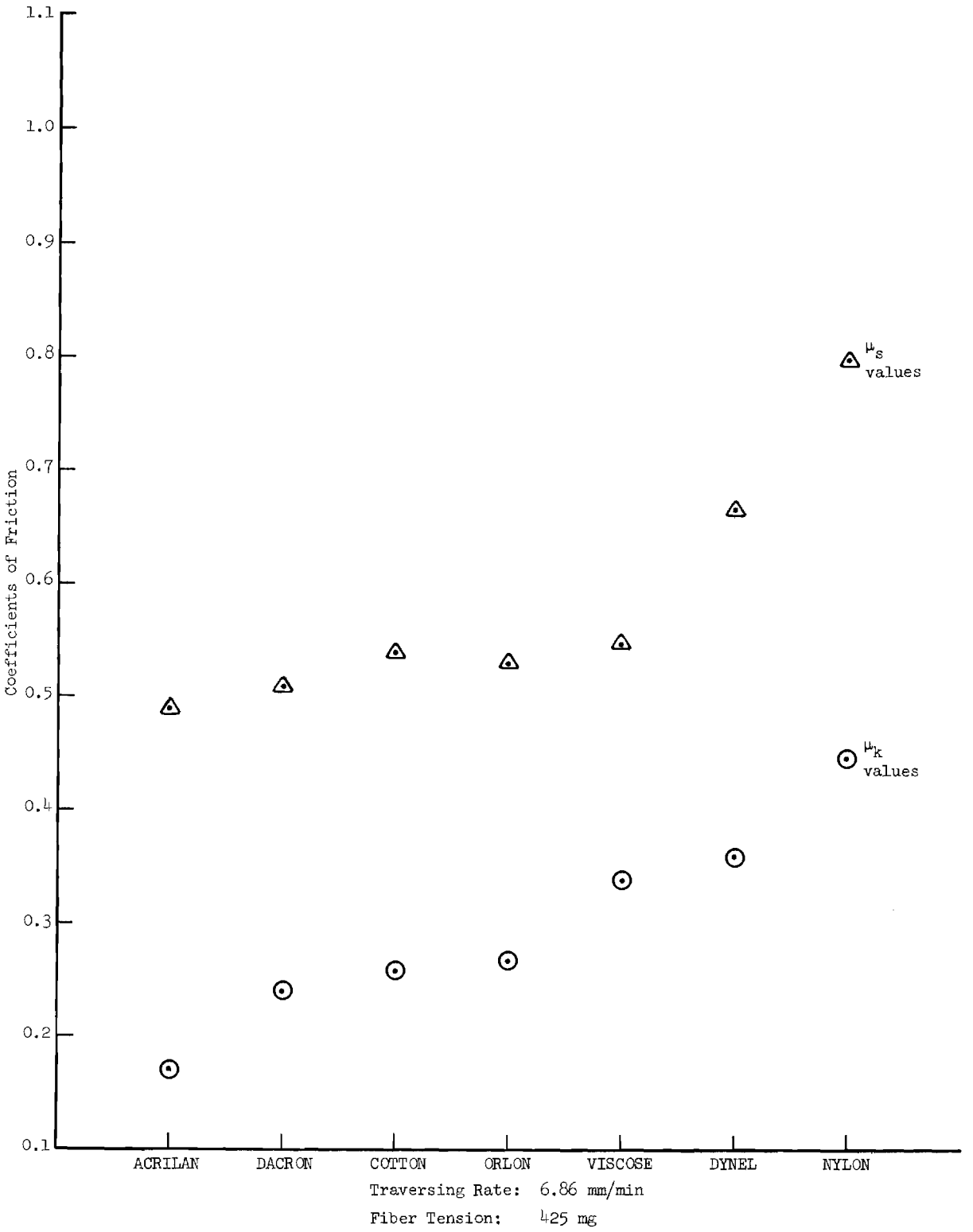


Figure 49. Coefficients of Friction of Various Fibers at 10 mg Normal Force.

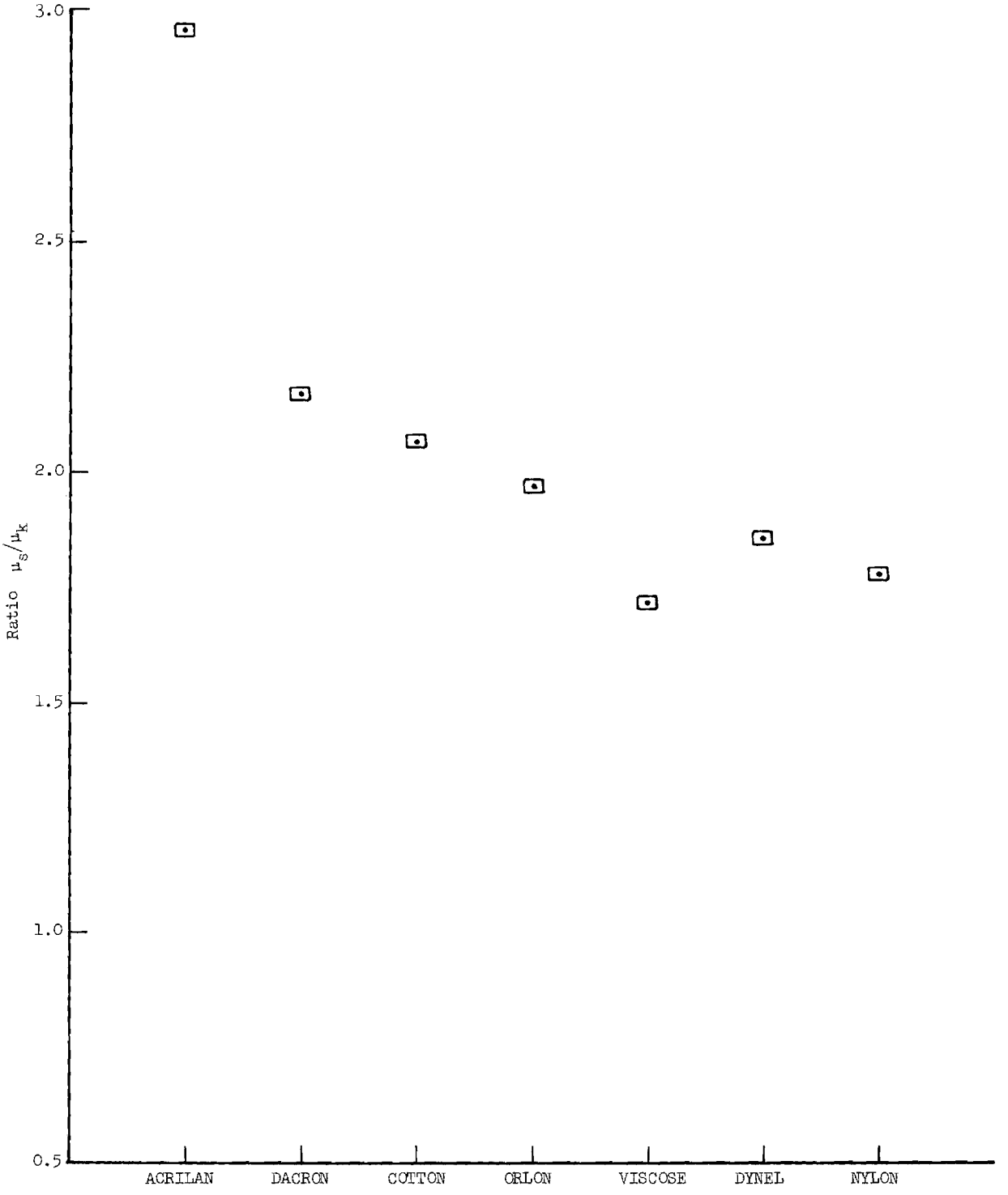


Figure 50. Ratio μ_s/μ_k for Various Fibers Plotted in the Same Order as Figure 49.

sectional shape, area ratios, and (area ratios)$^{\frac{1}{4}}$, we can rearrange the fibers according to the ratio of the area of the fiber cross section to that of cotton. It is observed now that with the exception of Acrilan, the kinetic coefficients of friction are in ascending order. The static coefficients of friction are roughly 0.52 for the circular section smooth fibers of similar cross section area. Cotton, very irregular and twisted, gives high μ_s as does the dogbone dynel and the large circular nylon.

It would appear from this analysis that the shape of the cross sectional area, the fiber diameter or area of section, its twist, and roughness are all important in determining the frictional parameters of the fiber.

Returning now to the discussion of the preceding Section it will be noted that shaped fibers of nylon also exhibited a μ_s value near 0.52 and a μ_k value of about 0.32.

The material differences of friction thus existing in these types of fibers appears therefore to be small, with a very high shape, fiber size and twist factor (for cotton) superimposed on it. Of the fibers, Acrilan alone appears to be markedly out of line with respect to its kinetic coefficient of friction.

3. Friction of Common and Spider Silks Against Nylon

Rushing[30] investigated the friction of common and Arachnid silks using the electrically actuated normal force friction measuring instrument. The normal force maintained was 10 mg and the traverse rate 0.11 mm/sec. Fiber mounting tension was approximately 425 mg.

Using the method described in Section V.C, the frictional properties of silk against nylon were obtained for three specimens of spider cocoon

silk and five specimens of silk worm silk. Three measurements were made for each specimen of spider cocoon silk and two for each specimen of silk worm silk. A few measurements were made of cocoon support filament, spider web, and spider drag-line silk. The bottom fiber, on the galvanometer, was 15 denier nylon. Data obtained are shown in Table 7. A typical friction data pattern is shown in Figure 51.

The data exhibit the fact that the kinetic and static coefficients of silk against nylon are in the same general range as the previously reported data for nylon on nylon. However, a physical size effect on friction is indicated here because of the small denier of the silk. This is especially apparent in the spider cocoon and drag line silk. We unfortunately did not obtain a diameter for the spider web silk.

The data indicate rather clearly that a true comparison of the friction of fibers is difficult without some real measure of the effect of the size of the fiber on the friction. This effect, compounded with the shape effect discussed in Section C.2 preceding, intrudes variables that have not been properly evaluated. However, the fact that they exist is clearly established. The contact area between the two fibers is one of the more important parameters. This is obviously a function of the fiber radius and of its shape. The size and shape of the sensor fiber must also be considered since they affect the frictional contact area. In addition, the contact area between two fibers is affected by the normal force between them and by the tension as has already been shown. The area is also affected by the yield strength of the fiber and its viscoelastic properties. To actually compare the true friction of a fiber material as a fiber we must compare the fibers where all these factors are

Table 7. Frictional Parameters of Common Silk and Spider Silk

Material	Diameter ($mm \times 10^3$)	Diameter$^{\frac{1}{2}}$** ($mm^{1/2} \times 10^3$)	Denier (Calculated)	μ_k	μ_s	μ_s/μ_k	Description (Section, Surface)
Cocoon Support Silk (A Diademetus)	15	3.88	2.0	0.31	0.77	2.48	Circular, smooth
Cocoon Silk (A Diademetus)	7.9	2.82	0.7	0.25	0.46	1.84	Circular, smooth
Web Silk (A Benjaminus)	-	-	No data	0.19	0.28	1.47	No data
Dragline Silk (A Benjaminus)	(very small)		0.07*	0.23	0.43	1.87	Circular, smooth
Common Silk (Bombyx Mori)	11	3.32	1.00	0.31	0.63	2.03	Trapezoidal, rough

*Reported for A Diadematus by Lucas.

**$D^{\frac{1}{2}}$ column shows size relation in accordance with data of Pascoe and Tabor.[12]

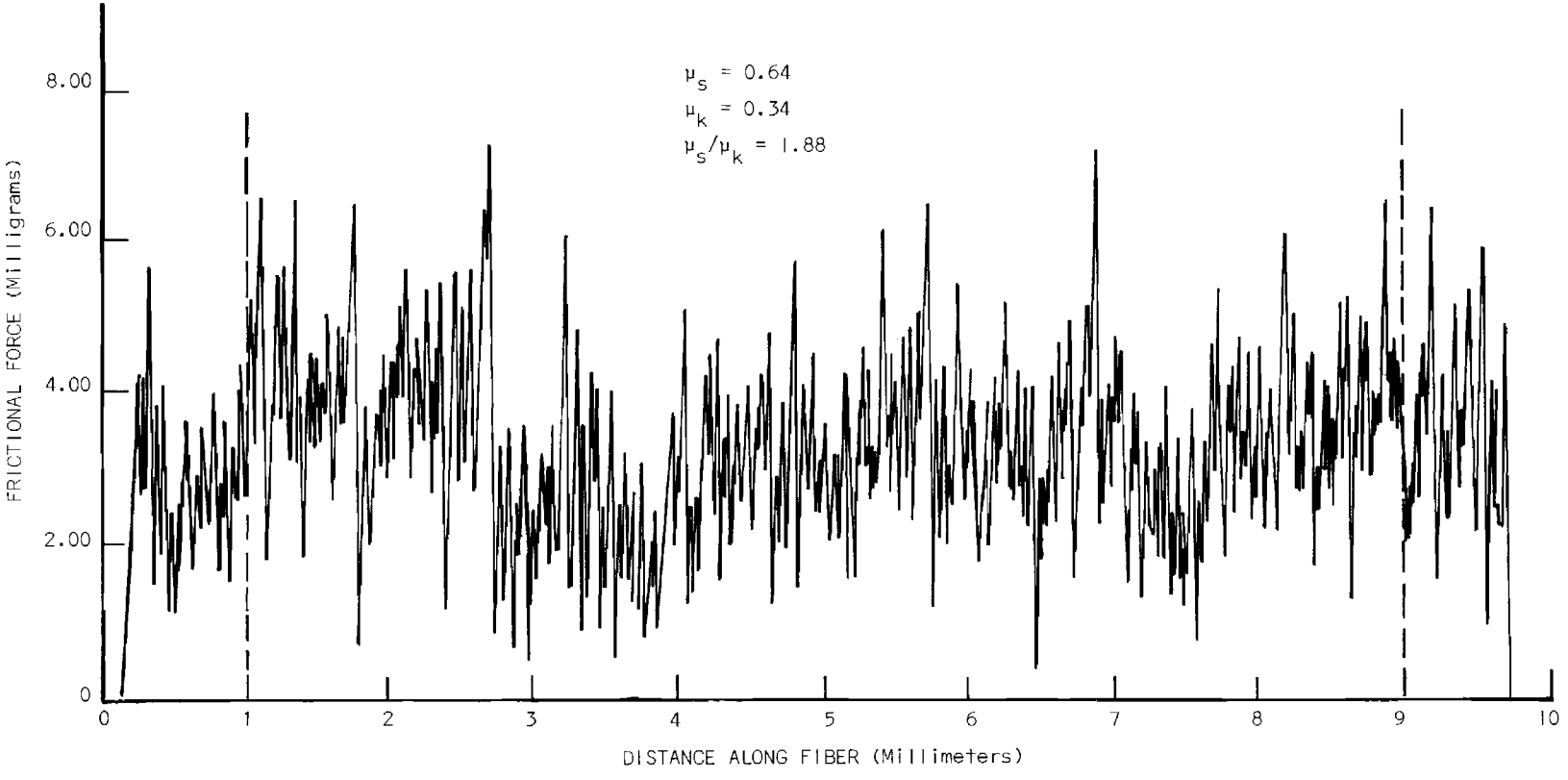


Figure 51. Typical Friction Data Plot for Common Silk (Bombyx Mori) at 10 mg Normal Force.

controlled to rather precise limits.

The values of friction indicated for the silks against nylon are thus considerably affected by the size effect. The common silk is further affected by a shape effect superimposed upon the size effect. Indications are that the coefficients of friction of most natural and synthetic textile fibers are going to be less affected by the material of the fiber than by the intrinsic fiber size and shape.

4. Friction Measurements of Metallic Fibers

In preliminary experiments with the low normal force apparatus fine metal wires were employed. Frictional data were obtained for the metals gold, aluminum, and tungsten at normal forces of 2, 4, 8, or 16 milligrams. Wires were cleaned with acetone before measurements. A typical example of frictional data is shown in Figure 52 for the metal tungsten. In these data also a new method of obtaining the coefficient of static friction was utilized. A statistical plot of all stick maxima was made as shown in Figure 53. The median was then determined from the plot. Data for the various metals are shown in Table 8, and ratios of μ_s for the ten maxima to those obtained by the statistical plot are shown. Calculations for μ_k values were not made.

It will be noted that there are large differences between the values μ_s obtained for the several metals with gold exhibiting much the higher frictional coefficient. The oxidized surface of the aluminum, the rougher surface of tungsten, and the hardness of the latter undoubtedly contributed to the lower value found for these metals.

Subsequently, some additional data were obtained on the gravity loaded frictional instrument using a 20 mg normal force. One mil wires

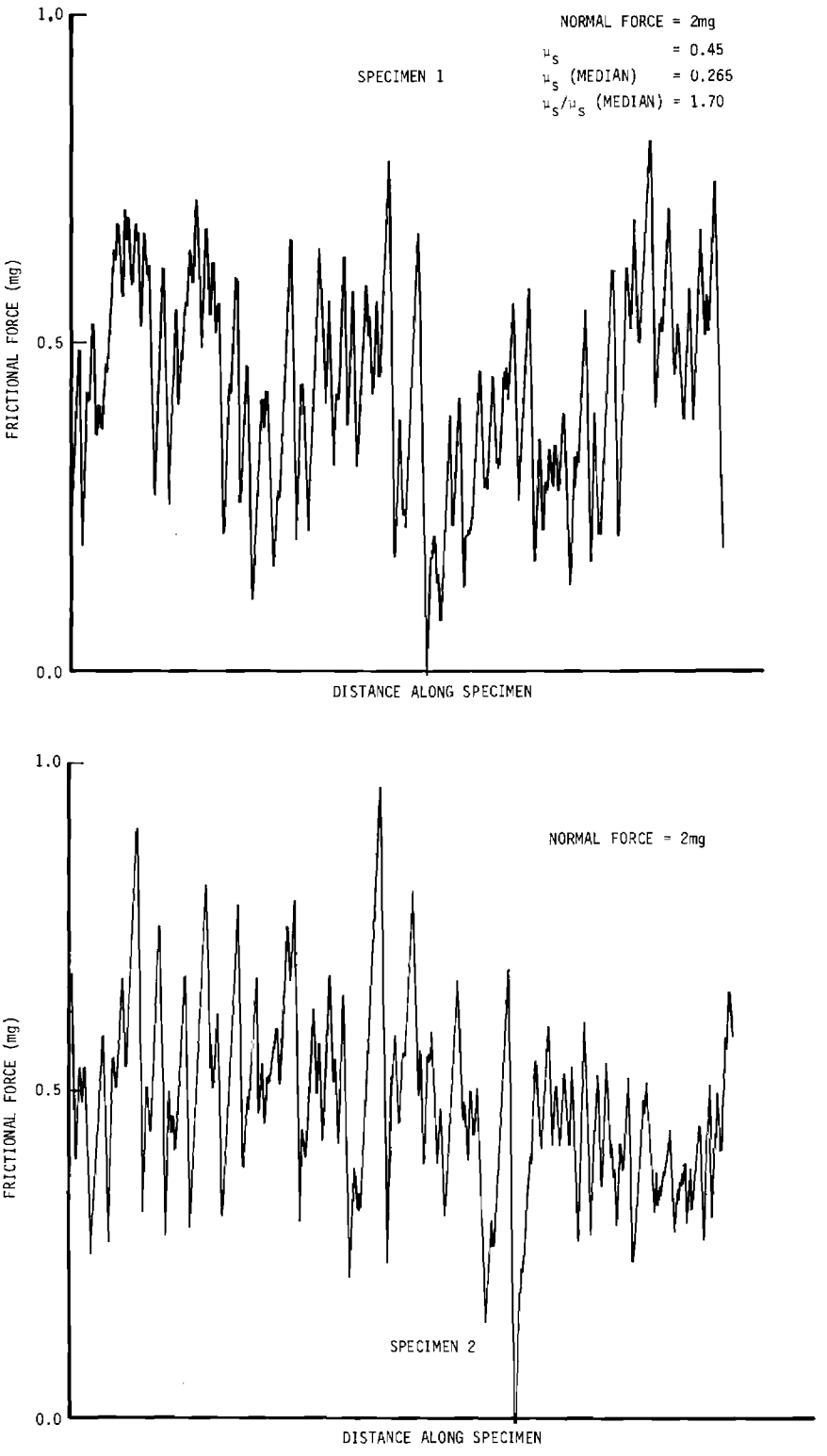


Figure 52. Frictional Data for a Tungsten Wire Against a Second Tungsten Wire (.0005" diameter).

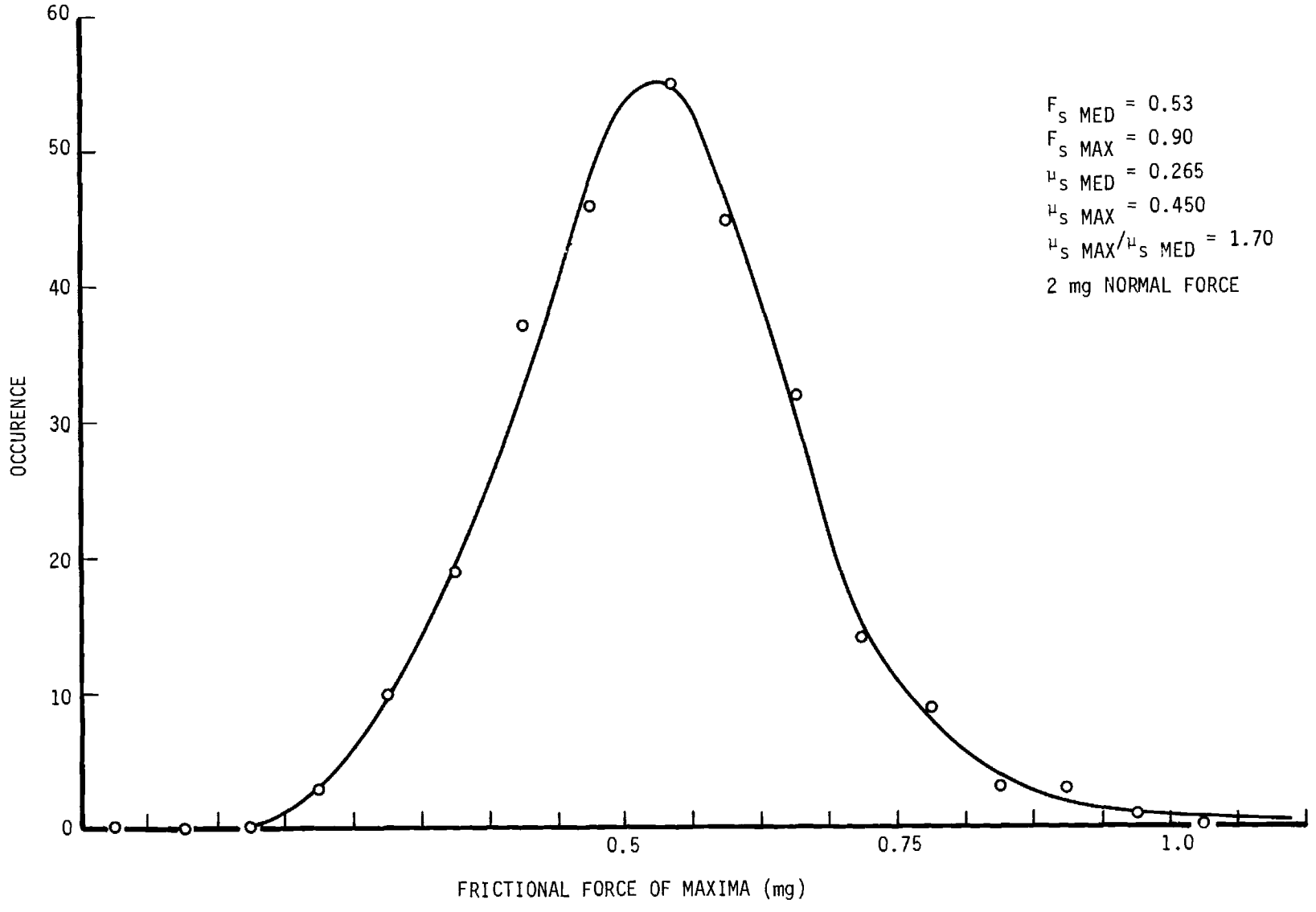


Figure 53. Plot of Frequency Versus Static Peak Frictional Force for a Pair of 0.0005" Diameter Tungsten Wires at 2 mg Normal Force.

Table 8. Coefficients of Friction of Gold, Aluminum, and Tungsten at Low Normal Forces

Substance	Normal Force mg	Static Friction* Median Value	Static Friction Greater Ten Values	Ratio $\frac{\text{Greater Ten}}{\text{Median}}$	Remarks**
Au on Au	2	0.470	1.050	2.23	0.002" wire
Au on Au	4	0.420	0.950	2.27	
Au on Au	8	0.425	0.890	2.09	
Al on Al	2	0.115	0.250	2.21	0.002" wire
Al on Al	2	0.137	0.285	2.08	
Al on Al	4	0.165	0.234	1.42	
Al on Al	8	0.122	0.230	1.88	
W on W	2	0.265	0.450	1.70	0.0005" wire
W on W	8	0.243	0.435	1.79	
W on W	16	0.280	0.355	1.27	

Data from First Apparatus at 15 to 25 mg NF (10 mil wires)***

Substance	μ_k	μ_s	μ_s/μ_k
Au on Au	0.368	1.150	3.14
Ag on Ag	0.337	0.526	1.56
Al on Al	0.287	0.620	2.16

*The median value of the coefficient of static friction is higher than μ_k by a small percentage.

**It must be noted here that in accordance with earlier discussions concerning the area of contact being dependent on the bulk compression strength of the metal and the radius of the wire that the friction of very hard metals would be expected to be less than the softer ones. In addition friction of cross cylinders of the same material has also been shown to vary approximately as $D^{\frac{1}{2}}$ (diameter). Hence for good comparisons all wires should be of the same diameter. The data are not good enough to identify the exponent n in $F = kW^n$ and $\mu \sim W^{n-1}$ where the value n-1 may vary from 0 to -0.33 roughly as discussed in Chapter III.A.

***This work conducted on original apparatus at higher normal forces.

were employed. The wires were uncleaned initially but successive measurements of the same wire pair were made resulting in cleaning by the traversing action alone. Selected data are shown in Table 9.

The results of successive measurements of the same wire are clearly evident in the data. Cleaning with chromic acid and Tesla coil discharge (separately) were also employed in some instances.

If one analyzes the data exhibited, it is found that the uncleaned softer metals exhibit an increase in friction with successive runs of the same fiber. We may assume that the last coefficient of friction on the third run obtained for gold is approximately correct. The values are 0.253 and 0.256 for two different specimens of 1 mil wire at 20 mg normal force. A specimen cleaned with chromic acid gave 0.257.

Likewise for aluminum wire we observe a large jump to 0.361 for μ_k for Measurement No. 4. The μ_k values for the harder metal tungsten and the alloy stainless steel did not increase with successive runs but that of nickel did slightly.

The effect of the Tesla discharge on the μ_k of stainless steel is large, nearly tripling its value. The surface of the metal appeared to be oxidized and pitted by the discharge.

Lubrication with light oils reduced kinetic friction considerably when the friction was a maximum such as after successive runs or exposure to a Tesla discharge. Viscous oils or greases increased the measured friction at these normal forces.

The coefficient of static friction was less predictable as was the ratio μ_s/μ_k. There is evidently much more data required to analyze the effect of the lubricant.

Table 9. Effects of Successive Measurements, Cleaning, and Lubrication on Friction of Metal Wires

	Specimen	μ_k	μ_s	μ_s/μ_k
	1 Mil Gold Wire (Specimen #1)			
1	347-A uncleaned, run #1	0.185	0.392	2.12
2	347-B uncleaned, run #2	0.189	0.419	2.22
3	347-C uncleaned, run #3	0.256	0.416	1.62
4	Specimen cleaned with chromic acid	0.257	0.641	2.50
5	347-D with whale oil lubricant (see 347-C)	0.202	0.343	1.70
	1 Mil Gold Wire (Specimen #2)			
6	Uncleaned, run #1	0.191	0.344	1.80
7	Uncleaned, run #2	0.219	0.409	2.02
8	Uncleaned, run #3	0.253	0.494	1.95
9	Luberex on upper wire of #8	0.146	0.280	1.92
	1 Mil Aluminum Wire			
10	Uncleaned, run #1	0.168	0.256	1.52
11	Uncleaned, run #2	0.158	0.259	1.64
12	Uncleaned, run #3	0.302	0.478	1.58
13	Uncleaned, run #4	0.361	0.660	1.83
	1 Mil Tungsten Wire			
14	Uncleaned, high tension, run #1	0.344	0.612	1.78
15	Uncleaned, high tension, run #2	0.297	0.597	2.00
16	Uncleaned, high tension, run #3	0.259	0.532	2.05
17	Silicone grease added	0.453	0.677	1.49
	1 Mil Stainless Steel			
18	352-A uncleaned, run #1	0.221	0.433	1.96
19	352-B uncleaned, run #2	0.195	0.364	1.87
20	352-C uncleaned, run #3	0.192	0.394	2.03
21	352-D plus weak Tesla discharge	0.540	1.170	2.18
22	352-E lubricated surface	0.317	0.632	2.00
23	352-F succeeding run	0.244	0.633	2.57
	1 Mil Nickel Wire			
24	343-A uncleaned, run #1	0.578	1.180	2.05
25	343-B uncleaned, run #2	0.638	1.100	1.72
26	343-C uncleaned, run #3	0.596	0.906	1.52
27	343-D upper wire lubricated with oil	0.519	0.985	1.90
28	343-E subsequent measurement	0.524	0.965	1.84

A comparison of the various values found for the metals at various times as related to wire size and normal force is pertinent to the discussion. The normal force effect can be seen in Table 8. In general, coefficients of friction of metals and the ratio μ_s/μ_k decrease as the normal force increases as for polymeric materials. If we examine Table 8 for gold at 2 mg and 4 mg respectively we find that μ has reduced from 0.47 to 0.42. This is to a value 89 per cent of the original value. In this case if we apply the criteria, $\mu = kW^{n-1}$, where $n = 5/6$, we determine that μ varies as $W^{-1/6}$. Where the weight was doubled the value of μ becomes $\frac{1}{\sqrt[6]{2}} = \frac{1}{1.123} = 0.89$ of its original value. However, there are insufficient data to belabor the point here except that the general trend of the data is correct. A more extensive study of metal wires would undoubtedly verify the correct value for the several metals. This value also checks closely with some data provided by Rabinowicz.[35] These matters have been discussed in some detail in Chapter III.A. In this we observe that theoretically $F = kW$ and $\mu = kW^{n-1}$, where n is a value between 2/3 and 1.

Data exhibited in Table 10 compare the effects of filament size on the coefficient of friction obtained. The values for gold and tungsten suggest an increase in friction as the fiber diameter increases and this is agreement with the work of Pascoe and Tabor on nylon that the frictional coefficient is proportional to $D^{0.52}$ for cross cylinders of small diameter. For aluminum, the effect of the initial oxide surface gives very low friction. Once the oxide is fractured or damaged, the friction increases rapidly. However, no suitable comparison of size effect is available because the 8 mg data had not apparently gone through the transition stage from low to high friction. Insufficient data on silver, stainless steel

Table 10. Selected Frictional Data Comparing Measurements According to Wire Size

Gold	1 Mil	2 Mil*	10 Mil
μ_k (cleaned)	0.260 (20 mg)	0.425 (8 mg)	0.368 (20 mg)
μ_s	0.641	0.890	1.150
μ_s/μ_k	2.500	2.090	3.140
Aluminum			
μ_k	0.361	0.122 (8 mg)	0.287
μ_s	0.660	0.230	0.620
μ_s/μ_k	1.830	1.880	2.160
Tungsten Wire	0.5 Mil Diameter	Value at 0.5 Mil x $\sqrt{2}$	1 Mil Diameter
μ_k	0.243 (8 mg NF)	0.344	0.344
μ_s	0.435	0.615	0.612
μ_s/μ_k	1.790		1.780

*In the above data we may observe the principal that friction of crossed cylinders varies as approximately $D^{0.52}$, or for practical purposes $D^{\frac{1}{2}}$. Unfortunately normal force values were varied in the various experiments, some of which were done on different instruments.

and nickel were made for a comparison of the effect of size on their respective coefficients of friction.

The very high friction of nickel wires is rather remarkable and consistent. It has not been satisfactorily explained at this time. It may possibly be a result of the well known work hardening and galling properties of nickel. More subtle factors are also undoubtedly involved. A similar action might be expected in titanium, and possibly in copper.

5. Comments

The kinetic coefficients of most polymer fiber materials measured at 10 mg normal force have fallen within the range 0.20 to 0.40 with static coefficients, based on the ten highest peaks, of approximately twice the respective value.

Superimposed upon the measured values have been effects of fiber size, fiber shape, fiber tension, normal force, and relative humidity. In the case of cotton, size and shape could not be controlled so as to be compared properly with other materials. Nor in the measurements were a gradual series of fiber sizes examined. Direct comparisons of the various materials as a generic substance are not thus possible at this time. The evidence available indicates that the basic polymer fiber materials will have a μ_k in the range of 0.25 to 0.40, at normal forces of about 10 mg and that the friction intrinsic to a specific fiber is established principally by its shape, size, and the very low normal forces present in the fiber assembly in early processing stages.

Metal wires fall generally within the same behavior pattern as the polymers with a somewhat greater spread of values. Hard oxide coating, hardness, stiffness, finish and other intrinsic parameters affect the

measurements to some degree and account for the principal differences observed. Among the metals gold, silver, aluminum, stainless steel, tungsten, and nickel, only nickel displayed a large excursion from the expected behavior as was discussed in the preceding section, C.4.

D. EFFECTS OF FIBER SIZE

The friction measured for a given fiber pair at a designated normal force and at other fixed experimental conditions is a function of the area of contact between the two fibers as outlined in Chapter III.A and in Section VIII.C preceding this one. This area is dependent on the radii and shapes of the respective contiguous fibers and upon the normal force between them. For a viscoelastic material, it is also a function of the time of contact. In addition, the tension of the respective fibers affects the contact area.

This work has touched on each of these matters but effects of fiber size have only been determined by inference as indicated in Table 6 for polymers, Table 10 for metals, and as reported for silk in Section C.3 and Table 7. In addition, Levy[36] obtained a μ_k value for carded Empire WR cotton of 0.289 at a micronaire value of 4.37 micrograms/inch as compared to 0.225 recorded for Pima Menoufi by Whitworth[27] at 3.7 micrograms per inch for comber lap. For comber noil, Whitworth's value of μ_k dropped to 0.173 for a micronaire of 2.8 micrograms per inch. Hence, the evidence is conclusive and its consideration in data evaluation has clarified a number of otherwise anomalous data. The size of the fiber mounted on the recording galvanometer must also be known and considered. A few cases such as in cotton against nylon and silk against nylon, the fibers mounted on the sensor were of 15 denier which was a large size in

comparison with the traversing fiber of about 1 denier.

The smallest fiber examined was spider dragline silk at 0.07 denier (approximately) against the 15 denier nylon. The value of 0.23 for μ_k was obtained. The larger cocoon silk at 2.0 denier gave a value of only 0.31 and common silk of smaller radius and stated to be 1 denier gave the same value, 0.31. It would appear from these data that the friction coefficient of common silk might be higher than for spider cocoon silk of the same denier and that the dragline might be the highest of all if corrected to the same denier.

The tungsten wire at 1/2 mil diameter gave a friction coefficient of 0.243 (at 8 mg normal force) compared to 0.344 for 1 mil wire at 20 mg normal force. Multiplying 0.243 times $\sqrt{2}$ gave 0.344 which checks with the theory of Pascoe and Tabor if we assume normal force had no effect on tungsten because of its high compressibility strength. Successive measurements of the same tungsten wire, however, reduced the value to 0.259 which suggests that the initial high values were due to surface roughness of the tungsten which might be expected from its sintered fibrous structure rather than the dimension change. In this case, the hardness of the tungsten and its surface condition may have reduced the expected difference as a result of size and load effects by a marked amount for the fibers of the two dimensions.

Additional measurements of the size and load effects in fiber friction will require careful measurements of the frictional force between cylindrical fibers of the same material and a series of graded diameters. Fibers of several different substances should be employed. Additional graduate theses covering these areas appear desirable. The magnitude of

the effects have already been established by Pascoe and Tabor and their co-workers, but an increase of existing data may be expected to furnish improved knowledge of the intrinsic coefficients of friction for the various materials examined in the past and facilitate the application of this knowledge to yarn and processing improvement.

E. EFFECTS OF COATING FIBERS ON FIBER FRICTION

Effects of coating fibers on fiber friction measurements have been measured in a few instances during the course of this work. Effects measured frequently had superimposed upon them inadvertently effects of other variables among those discussed in the preceding paragraphs.

The earliest endeavor of this nature performed here was that of McBride[21] in which he examined the friction of Empire WR cotton fibers before and after extracting the natural wax coating with cold chloroform. These measurements were performed for Empire WR cotton of 1 inch staple length at 12 mg normal force. The respective kinetic coefficients of friction for natural and dewaxed cotton were 0.43 and 0.41 respectively and the μ_s values were 0.88 and 0.72, respectively. However, the static peaks fluctuated much more widely in value for the dewaxed fiber than for the natural fiber. The data for the dewaxed cotton indicated many occasional very high stick peaks. In retrospect, the lower average μ_k value appears to have been the result of an instrumental problem at the low normal force of 12 mg, which was below the accuracy capability of the instrument at the time.

Subsequently, Wakelyn[32] examined the effects of quaternary ammonium salts as antistatic agents on polyacrylonitrile fibers. In this work he examined frictional changes of the fibers resulting from the various

coatings. These revealed a drop in μ_k and μ_s for Zefkrome, unfinished, from 0.29 and 0.61 respectively to 0.19 and 0.37 for the best coating (C_{16} Me Br). Application of any of the coatings to the fiber resulted in appreciable decreases in both friction coefficients.

The experiments with metal fibers as shown in the preceding Table 9 resulted in similar drops in frictional coefficients of the fibers when these were lubricated with a light oil such as porpoise or whale oil. In addition, an increase in friction occurred when the uncleaned gold or aluminum fibers were successively measured. The traversing wear removed the outer surface coating, adsorbed material, or oxide, baring the clean surface and increasing the measured coefficient of friction.

Ample evidence exists to indicate that light lubricants reduce frictional effects between fibers at low normal force levels. One of the principal mechanisms appears to be reduction of the amplitude and time of each stick thus reducing the total energy of the major sticks and the energy per unit length of traverse of one fiber across a second.

F. EFFECTS OF FIBER PROCESSING ON FIBER FRICTION

1. General

In Sections VII.B, VII.C, VII.D, and VII.E, preceding, the effects of cross sectional shape, fiber material, fiber size, and coatings on fiber friction have been discussed. Previously in Chapters VI and VII, effects of longitudinal shape, asperities on the surface, and fiber shape abnormalities and damage have been outlined. Effects of controllable experimental parameters such as temperature, humidity, fiber tension, fiber traverse velocity, and normal force have been delineated.

The consideration of these varied data point to three principal

intrinsic factors affecting interfiber friction at the very low normal forces under which fibers usually travel through processing. These are fiber material, area of contact between contiguous fibers, and excursions of the fiber from a straight line axis. This latter category, the longitudinal shape, encompasses for cotton, crimp, convolutions, reversals, and any abnormality resulting in an abnormal growth or asperity. In man-made fibers one can add the delusterant filler materials such as titania.

In fiber processing the fibers are stretched, ironed, bruised, broken, and successively selected to rid the fiber stock of damage detritus or undersized fibers. These various processes straighten the fibers, reduce asperities, expel a high percentage of damaged, broken, and short fibers, and add to the total of bruised and damaged fibers present. These actions appear to be more significant in processing a fiber of a non-circular cross-sectional area, of an irregular longitudinal shape, and of a varying size distribution. Cotton is thus an outstanding example for which one may observe effects of processing on friction.

2. Experimental Data

In the preceding sections, VII.B and VII.C, it has been shown that three successive frictional traverses of the same cotton fiber result in approximately a 5 per cent reduction in friction for the second and third passes respectively. Some viscoelastic flow is also revealed as discussed in Section VI.B. Changes in surface condition may occur as outlined in Section VIII.C.4 for metal wires where absorbed and oxide coatings were removed.

For the cotton fibers at very low normal forces, 2 mg, the friction

changes on successive passes are small in comparison with those at 20 mg normal force. Cylindrical fibers appear to be less affected than other shapes. Thus, a principal friction reducing vector appears to be the longitudinal shape change effected by the ironing or stretching of a fiber accompanied by some viscoelastic flow. The surface changes of metal wires (at 20 mg) indicate a cleaning effect in some instances and a smoothing effect in others (such as tungsten). Similar surface cleaning effects appear to occur concurrently with other effects in polymer fibers but little direct evidence has been obtained.

Levy[36] and Cromer,[37] in theses prepared as a part of this research, measured fiber damage as a result of processing cotton from the bale to the yarn. Per cent damaged fibers determined by the Congo Red method increased from 18 per cent before opening to 68 per cent after spinning as shown in Figure 54.

Changes in coefficients of kinetic and static friction were also measured. Observed changes are plotted in Figures 55 and 56, along with the data of Whitworth,[27] who measured changes in a Pima-Menoufi blend of cotton as a result of combing, drawing, roving, and spinning.

It will be noted that Levy[36] observed an increase in the coefficients of friction of cotton fibers up through carding and that Cromer and Whitworth both recorded reductions in the coefficients as processing progressed beyond carding. Examination of Levy's original data indicate that he had measured friction of numerous damaged fibers as indicated by very high static frictional peaks. The fiber damage undoubtedly contributed to the increasing trend in fiber friction up to the carding stage. Beyond this stage, however, the frictional coefficient values are reduced by the processing. Some fiber selection on the part of the experimenters

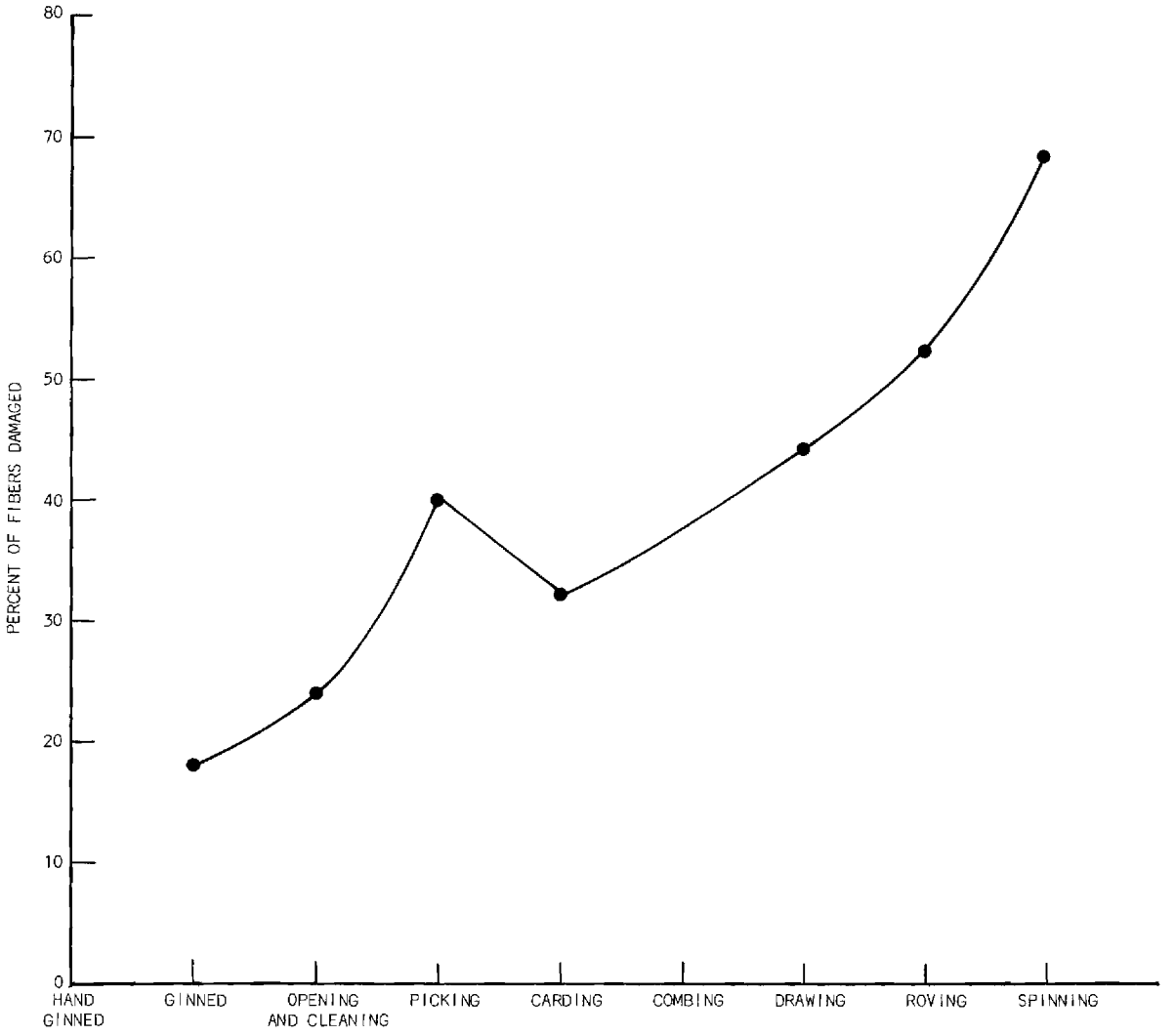


Figure 54. Per Cent Fiber Damage as a Result of Processing Empire WR Cotton from the Bale to the Yarn Determined by the Congo Red Method.

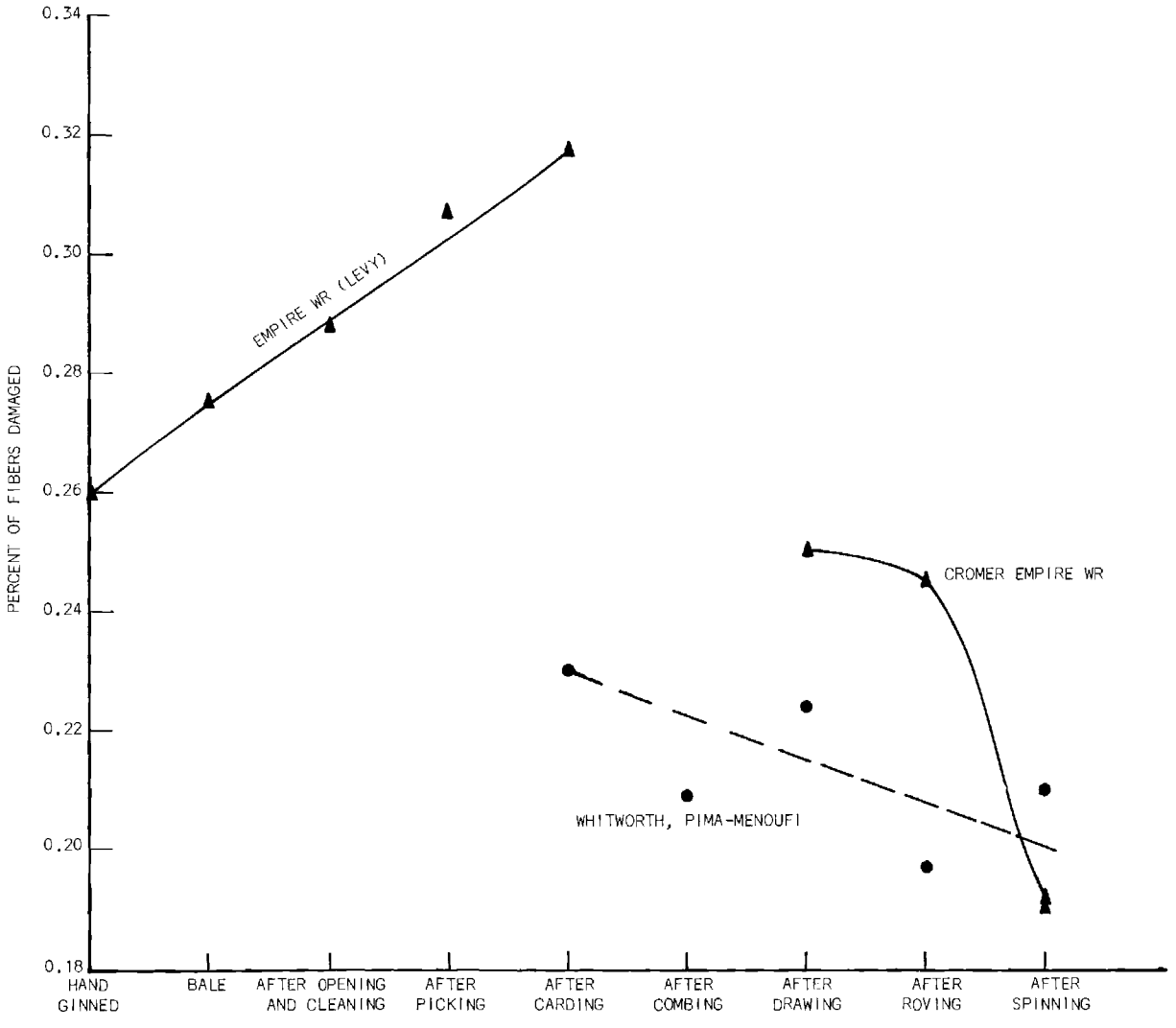


Figure 55. Changes in the Kinetic Coefficients of Friction of Empire WR Cotton Fibers After Successive Processing Stages from Boll to Yarn and of a Pima-Menoufi Blend After Carding.

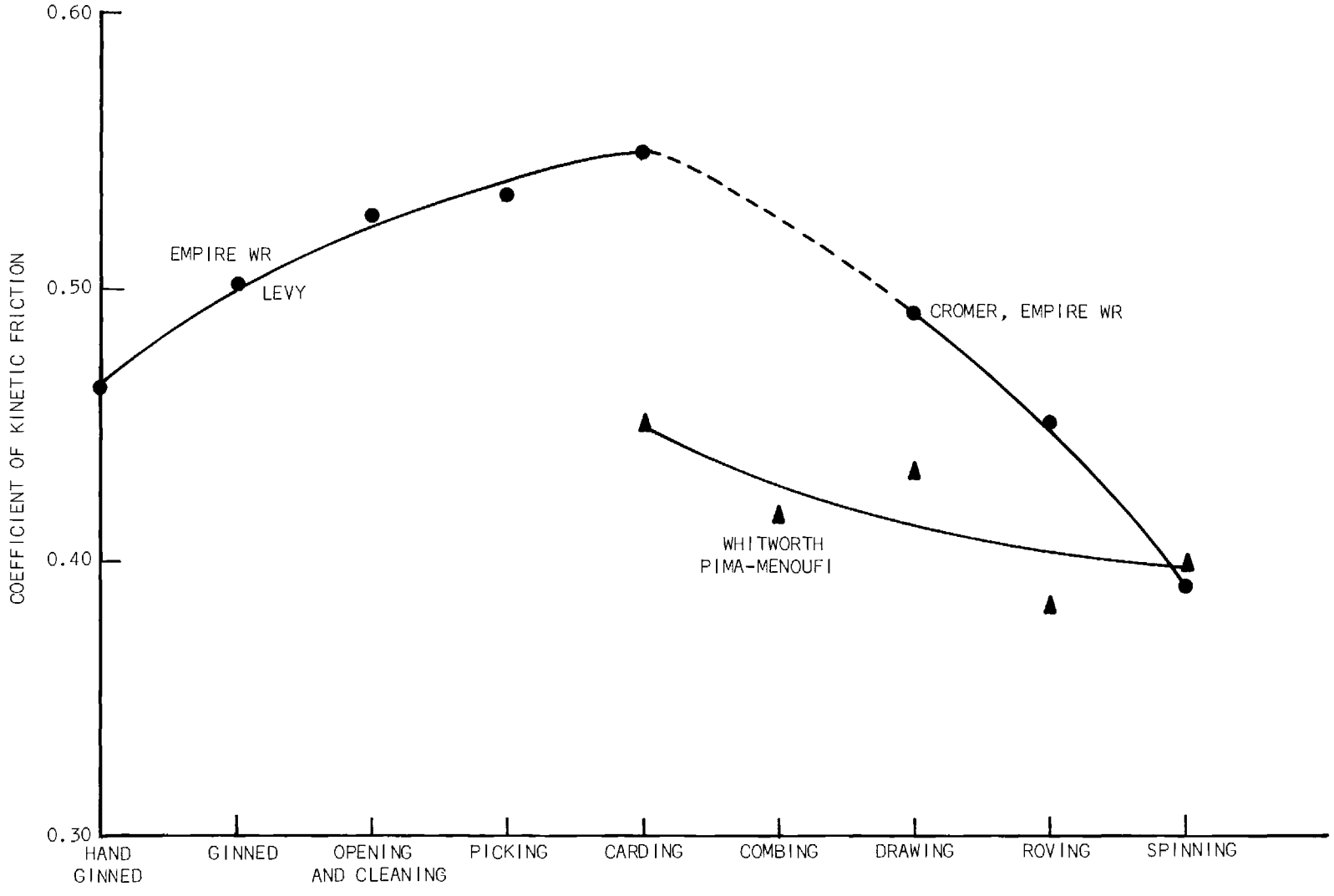


Figure 56. Changes in the Static Coefficient of Friction of Empire WR Cotton Fibers After Successive Processing Stages from Boll to Yarn and of a Pima-Menoufi Blend After Carding.

also may have excluded damaged fibers from the measurement since severely damaged fibers may break during mounting. Likewise, the processing itself eliminates broken and severely damaged fibers thus reducing total fiber yield. Whitworth's data starting with a different cotton of 3.7 micrograms per inch, compared to 4.0 micrograms per inch reported by Levy for Empire WR, exhibited a lower friction value after carding than the Empire WR cotton but continued the downward trend through spinning, ending up near the same final value in the 0.19 to 0.20 range for μ_k and 0.39 to 0.40 for μ_s.

Cromer[37] in his thesis exhibits two measurement distribution charts for frictional measurements of Empire WR cotton fibers after drawing, roving, and spinning. These are shown as Figures 57 and 58. The μ_k measurements are generally well grouped but exhibit a consistent downward trend of the median value. The μ_s values are much less well grouped but the median value trend is still downward. Values after spinning are the most uniformly grouped in each case. These charts clearly indicate the trend of the friction coefficients as a result of fiber processing. They indicate further the selectivity going on; the trend is to move the energy level of the high sticks down and to group them more closely about the median, indicating closer average energy levels of the high peaks.

Fiber selection and length distribution are important in processing. The fiber damage changes fiber lengths and the processing tends to exclude the shorter fibers after they are formed. Cromer[37] showed fiber mean length of Empire WR cotton reduced successively from 0.92" to 0.88" to 0.74" through the stages drawing, roving, and spinning. The upper half mean lengths were 1.09", 1.04", and 1.01" respectively as measured on the

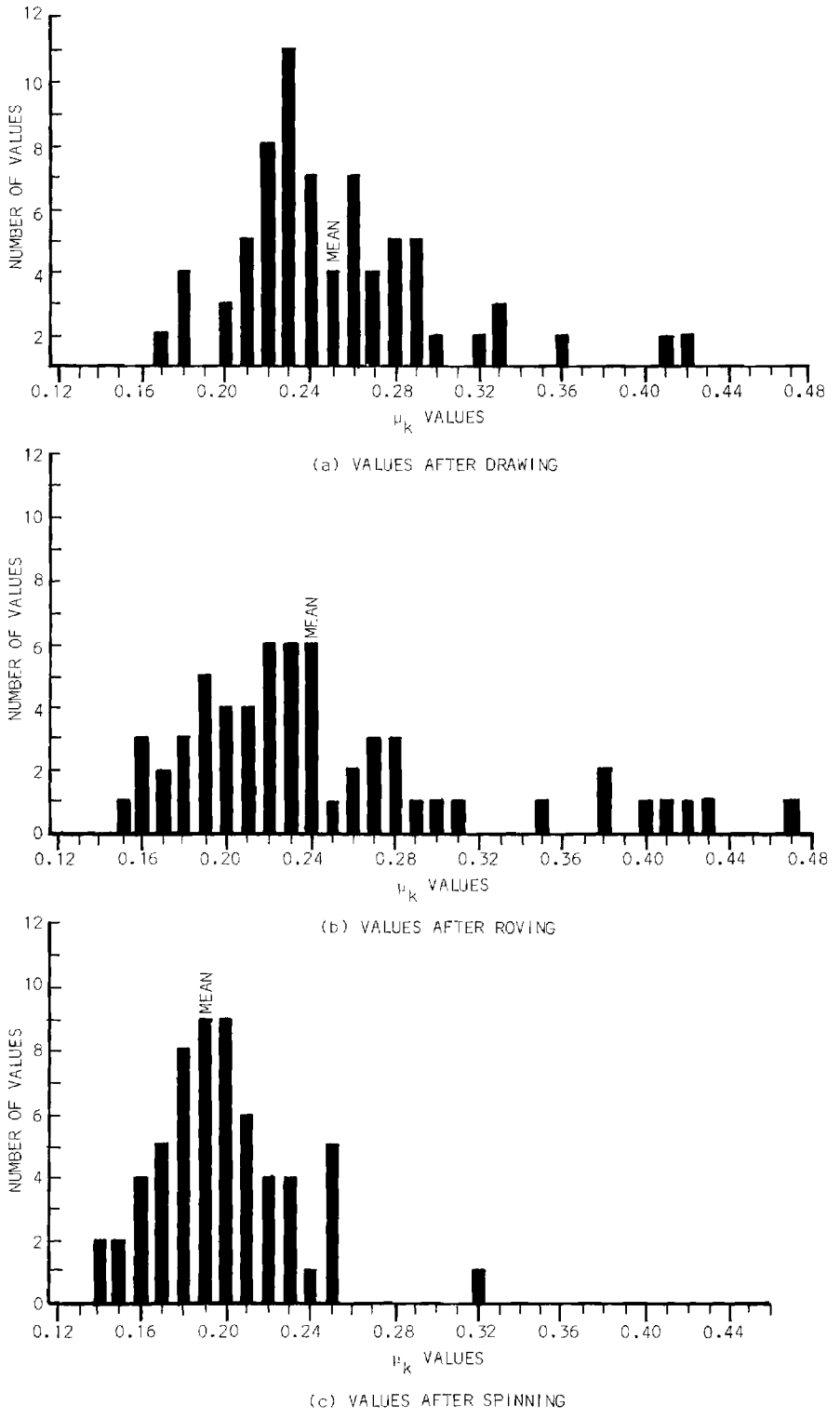


Figure 57. Frequency Distribution of the Coefficients of Kinetic Friction of Empire WR Cotton Specimens Selected After Drawing, Roving, and Spinning.

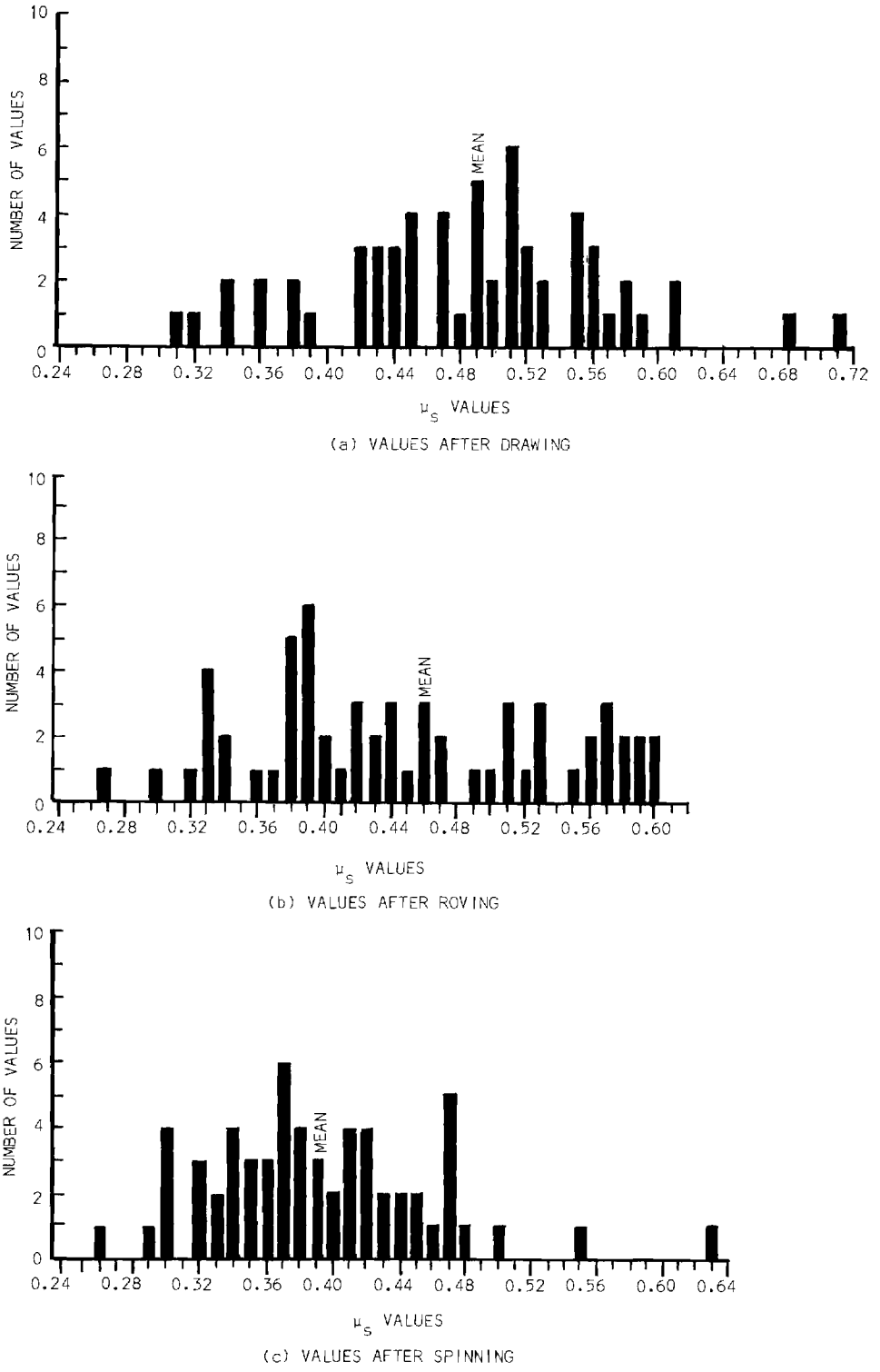


Figure 58. Frequency Distribution of the Coefficients of Static Friction of Empire WR Cotton Specimens Selected After Drawing, Roving, and Spinning.

fibrograph. The fineness remained at approximately 3.76 micrograms/inch,* during these stages whereas Levy[36] had registered an increase from 4.00 micrograms per inch for the bale to 4.37 micrograms per inch for the carded fiber.

Whitworth[27] noted an increase in micronaire values as a result of processing Pima-Menoufi cotton, from 3.7 micrograms per inch before combing to 4.2 after combing with only a small change in fineness thereafter. The mean length increased from 0.747 inches before combing to 0.850 inches after roving and the upper half mean length from 1.40 inches to 1.45 inches.

3. Comments and Conclusions

The evidence submitted does not cover all possible effects influencing the friction of the fibers. However, it is evident that a factor tending to increase friction is fiber damage; a factor diminishing it is fiber straightening. Fiber selection works to exclude damaged fibers and to increase fiber average diameter. More subtle influences such as surface smoothing, surface chafing, and changes in physical properties may also affect frictional properties but were not specifically evaluated in these data.

Areas of contact would tend to be increased by the processing as would total time of contact during a fiber traverse unless fiber damage is overly severe. In addition, selection of larger fibers would increase the areas of the contact zones. Damaged fibers would tend to increase

*These values are offset from the last reported by Levy, 4.37 micrograms/inch after carding.

stick peak heights. However, the energy of these sticks appears to lessen. The principal factors working for friction reduction are fiber straightening and the energy reduction of the sticks which appear to be overriding factors causing the overall frictional decreases. Krowicki[38] has suggested the polishing of the cotton wax as a factor in friction reduction on successive measurements he made. Nylon fibers of various cross sectional shapes also exhibited reduction in friction with successive traverses according to Huff,[23] whereas the cylindrical fiber exhibited little change.

The evidence points to the change in the longitudinal shape as the principal factor active in reducing the interfiber friction as a result of processing cotton fibers.

Effects of processing on the friction of cylindrical fibers have been little examined by us, and sufficient evidence is not yet available to indicate other than that smaller changes would be expected than for cotton; these may be positive or negative depending on the per cent delusterant incorporated and its effect on the fiber surface roughness, interlock, and related matters. Scardino and Lyons,[39, 40, 41] in a recent series of papers, have discussed cohesion changes in fibers resulting from various percentages of delusterant and as a result of some processing stages. These have indicated, generally, increases in cohesion for the smoother fibers, hence, his cohesion data agree essentially with the data we are reporting principally for cotton fibers. They have also indicated a decrease in the number of asperities but not in the asperity height as a result of processing.

G. OTHER EXPERIMENTS

In the early stages of this research, infrared spectroscopy* was employed to examine cotton wax with the expectation that changes in the wax as a result of heating, aging, weathering, or other influences might appreciably affect the fiber friction of cotton.

X-ray diffraction was similarly employed to examine changes in crystallinity of the fiber with the expectation of possible correlation between crystallinity, effects of processing fibers, and fiber friction. However, other variables in the friction measurements were too large and our understanding of them at the time too small to effectively utilize the potential of either method in the work. Some related areas were investigated as outlined in Semiannual Reports No. 2[34] and No. 5[42] and in theses by Kirkland[43] and Hicks.[44] These data, since they are not closely related to the friction data comprising the principal topic of this report, are covered in the Appendix.

*Under the direction of Dr. James A. Knight, Jr., Research Professor of Chemistry and Head of the Radioisotopes Laboratory, Engineering Experiment Station, Georgia Institute of Technology.

IX. DISCUSSION

A. GENERAL

Bowden and Tabor in their books on "The Friction and Lubrication of Solids" have presented the best information and explanation of the friction of solids currently available. Pascoe, under their tutelage and in collaboration with Tabor, has presented the most fundamental information available concerning the friction of crossed cylindrical fibers of man made fibers. These data are also outlined by Tabor in the book by Howell, Mieszkis, and Tabor.[5]

In particular, it was shown by Pascoe and Tabor[12] for cylindrical nylon fibers that the coefficient of friction, $\mu = c_1 \, s \, W^{-0.26} \, D^{0.52}$ where c_1 is a constant of about 1.4, s is an adjusted shear value of nylon considered to be about 1.5 kg/mm^2, W is the normal force, and D is the diameter of the fibers used (both of the same diameter).

Calculated values of μ, using this expression, fit the curve obtained by experiments using fibers of a series of different diameters and a normal force range of 10^{-6} to 10^{4} grams. Similar agreement for fibers of other materials than nylon were also obtained using the appropriate values of shear strength.

These data indicated clearly that the value of μ was increased as the normal force was reduced and that μ decreased as the diameter of the fiber was decreased.

Tabor[5] has discussed the area of contact further and in a load range of 1 to 50 mg for crossed fibers of 42 microns in diameter, the circle of contact of a diameter 4 microns was the value obtained at the highest load (50 mg).

Rabinowicz[35] has discussed diameters of point-to-point contacts between spheres and between spheres and plane surfaces for both metals and polymers. He points out that such point contacts possess diameters in the range 10^{-3} to 10^{-4} cm theoretically (10 to 1 micron) based on the expression $A_r \geq L/p$ where A_r is the area of real contact, L is the normal force and p is the penetration hardness (the largest compressive strength the material can withstand without yielding, approximately three times the yield strength.) Measurements of the contact zones of metals obtained by measuring diameters of the microweld zones gave values of 5 microns to 31 microns for the diameters using loads in the range 100 to 1000 grams.

From the data provided by Tabor and by Rabinowicz, it is evident that contact areas are of the magnitude of a few microns in diameter. Tabor reported data by Howell and Mazur[9] indicating a shift in the magnitude of the frictional force as the fiber size and normal force increased. He suggested that in the expression, Frictional Force = $k W^n$, W being load, n = 0.74 for very low loads and n = 0.9 for higher loads. This behavior implied a transition from single point contact to multi-point contact behavior.

The friction measurements of Pascoe and Tabor[12] were taken by a method giving only the static coefficient of friction. However, for a cylindrical fiber the coefficient of static friction, according to our data, may be only slightly greater than the coefficient of kinetic friction if all stick positions are recorded and the frequency of slips is large. Under the conditions data were taken the trends of the static and kinetic coefficients of friction would be expected to be the same although the exact values of the kinetic coefficients would be somewhat less than the numbers cited by Pascoe and Tabor.

A system of the type used by Pascoe and Tabor is not applicable readily to the examination of the friction force of a cotton fiber against a second cotton fiber although the friction of a cotton fiber against a nylon or other cylindrical fiber could be examined. Secondly, the method is handicapped by the many visual observations required. However, the data presented by them without question have delineated the fundamental elements of fiber frictional behavior and have emphasized the importance of contact area and normal force in establishing the frictional force and the coefficients of friction determined therefrom when the contact area is essentially a single point as is normally the case with fine textile fibers.

B. THE SERVO-CONTROLLED FRICTION INSTRUMENT

The servo-controlled friction instrument in its present form, as described by us, readily records friction of fibers of any length greater than 0.5" and could be modified to examine even shorter fibers. It will perform this task at forces as low as 1 mg or less, or with a force established by the weight of the fiber only. It faithfully gives a specific fingerprint of a fiber to the degree that for cotton fibers the individual fiber is recognizable from its trace if it is not demounted and remounted. The instrument allows concurrent integration of the total area under the frictional curve for immediate determination of the average frictional force and kinetic coefficient of friction; and the trace in the reverse direction may be obtained inverted and almost as a mirror image of the first along with its resultant integral.

The static frictional forces and the static coefficients of friction are readily obtainable from the data sheet. The character of the curves

and the ratios of μ_s/μ_k have been shown to have definite value in interpretation of frictional properties of a material. The relative speed of measurement is also quite excellent. The most time consuming task is the mounting of the individual fibers. Duplicate fiber mounting frames can expedite greatly this act.

C. COMPARISON OF THE FRICTIONAL PROPERTIES OF VARIOUS FIBERS

In spite of the excellent features of the friction measuring instrument the very nature of friction makes comparison of the frictional properties of various fibers most difficult. Especially this is true when one wishes to compare frictional values obtained in the literature to those obtained in these experiments. We have already shown that the measured fiber friction is dependent on the ambient humidity, temperature history, traversing velocity, and area of contiguous fiber to fiber contact. The latter in turn is affected by fiber tension, the normal force, cross-sectional shape, diameter, and longitudinal shape. In few instances in the literature have all of these been reported, known, or even recognized as variables in the measurements reported.

In our own data we have varied some of these conditions perforce or before recognizing the importance of them. In addition, our established objective of examining cotton lead us far afield before the importance of the size and shape factors was impressed upon us. In retrospect, however, the anomalies of a coefficient of friction value for cotton against nylon being greater than for cotton against cotton resolves itself into a size effect caused by the use of 15 denier nylon as the lower fiber. Similarly, for the low denier spider silk dragline, we have a low frictional

coefficient which may prove high on correction for the much smaller radius. Again the value for 15 denier nylon against 15 denier nylon is higher than for cotton against cotton; but correction to the same denier would equalize or reverse the order of the values.

The area of real contact becomes the single most important variable in measuring fiber friction since it is affected by the other factors of tension, normal force, diameter, fiber cross-sectional shape, and longitudinal shape.

Thus, the friction of fiber materials can only be compared by utilizing materials of the same size and under the same experimental conditions. Where similar sizes cannot be obtained, calculations and interpolations can be made to arrive at an estimated comparative value. Otherwise the values of fiber friction obtained by various investigators cannot feasibly be compared.

D. EFFECT OF FIBER SHAPE

We have observed in the case of cotton and for nylon of five cross-sectional shapes some effects of fiber shape. The nylon results were discussed in some detail in Chapter VIII.B. In general, it was found that application of a diverse cross-sectional value to these fibers reduced the friction against a cylindrical fiber as compared to the friction between two cylindrical fibers. This action again appears to be the result principally of a reduction of the area of contiguous contact. A more proper knowledge would have been obtained of the subject if each type of fiber, duokelion, quasi-triangular, trilobal, and tetrakelion, had been run against itself as well as against each of the other fibers. Allotted time did not allow an investigation of these various arrangements.

In addition, measurements of the energy of major sticks appear important but have not been done because of objective and time limitations.

Referring now to the work described in sections VII.B and VII.F and as discussed in more detail in the thesis by Gunther, we observe in Figures 31 and 32 the atypical coefficient of friction versus normal force behavior of cotton fibers. The maximum in friction coefficients at about 7 mg and the subsequent reduction of these at lower normal forces is an effect of shape. It requires a much larger energy to override a stick in cotton than any other fiber we have examined. The relatively high μ_s/μ_k ratio is an effect of shape, and of this high energy value perstick. It needs to be pointed out here that the natural shape of cotton is quite different from the nylon fibers of variously extruded cross sections. These lack convolutions, crimp, reversals, and growth irregularities that constitute a part of the natural diversity of cotton.

The much higher μ_s/μ_k ratios for cotton at low normal forces brings to our attention again that the static coefficient or sticks of very high energy rather than the kinetic coefficient of friction must be the principal agency responsible for fiber mass travel during processing into yarn. Furthermore, shaped nylon fibers of the type examined did not replicate cotton in frictional behavior in the limited measurements made. However, some information, principally with respect to the general decrease in frictional coefficients as a result of area of contact changes, was observed. The sticks did not display very high energy values. Additional examination of fibers of specific cross section design and added twist or crimp as well as an analysis of stick energy should give better information concerning desirable shapes for improved processing and better data concerning the shape effect on fiber friction.

E. PARALLEL DEVELOPMENT IN FRICTION MEASUREMENT

1. General

Frictional measurements related to the data discussed in the previous chapters have been reported by Hertel and Lawson[42] and by Scardino and Lyons[39, 40, 41] in papers published recently. These data are pertinent to a clearer understanding of interfiber friction.

2. Oscillating Shear Method of Friction Measurement

Hertel has developed an oscillating shear method of measuring interfiber friction based on the damping of a pendulum driving a moving plate, parallel to a second which is stationary; between the two plates is mounted a fiber assembly of the cotton specimen to be measured. The latter is formed by carding 50 grams of cotton to form a web 8 inches wide which is wound on a drum to a depth of about 30 layers. The lap is pulled in half and folded twice to form four layers of 8" x 18" area and 120 webs thick. A load of 124 grams is applied between the plates. After the pendulum is set in oscillation, amplitude measurements of the pendulum after successive swings can be inserted into an equation shown as

$$S = K \frac{\Delta^2 T}{\Sigma} \quad ,$$

where K is a constant of the instrument,

Δ is the difference between successive amplitudes,

T is the thickness of the cotton lap, and

Σ is $(A^n + A^{n+1})$ where A = amplitude.

For any number of pendulum swings the equation is

$$S = \frac{K(A_o - A_n)^2 T}{n^2(A_o + A_n)} \quad .$$

The value S is dimensionless and has been named shear friction by Hertel and Lawson. Since the value K has been corrected for load or normal force and area, the value S is proportional to the kinetic coefficient of friction. The factor which is missing to convert S to μ_k is the actual number and real area of the fiber to fiber contacts and the real fiber to fiber normal force values. The energy expended in bending fibers or compressing them, in pulling the pendulum to the release position, is assumed to be largely restored to the pendulum on the return swing. Some losses due to internal friction in the fibers as well as fiber friction contribute to the total energy loss but are believed to be a minor proportion of the total in most instances.

Using this equipment Hertel and Lawson have investigated the shear friction of different varieties of cotton, cotton after various stages of processing, mercerized cotton, and viscose. For Eastern cotton varieties an average value of S of 1.787×10^{-4} was reported.

The value of S could feasibly be converted to essentially the value μ_k by use of a multiplier incorporating the number of fiber to fiber contacts involved and the normal force between the fibers at the points of contact. However, the paper cited did not report the value of such a multiplier. The data obtained do appear to provide excellent and reproducible information concerning fiber frictional properties in a dimensionless number proportional to μ_k. Because of the large number of contacts an excellent average value of friction is measured in a single measurement.

Conclusions arrived at by Hertel were that,

(1) The shear friction method was capable of revealing small differences in frictional properties of fibers;

(2) Friction of cotton varies between bales, varieties, and regions;

(3) Shear friction was reduced when cotton was heated above the melting point of the wax and increased if the temperature was raised sufficiently to cause wax deterioration;

(4) Shear friction is reduced by processing;

(5) Shear friction varies slightly with humidity from 30 to 65% RH and sharply between 65 and 80% RH;

(6) Spinning wastes are higher in friction than the roving being spun; and

(7) The variation of the shear friction of the cotton within a bale is small.

These conclusions are generally in agreement with our measurements in single fiber friction studies within the limits of our own investigation which did not look extensively at items (2), (3), and (7). Item (6) was not examined. The differences between cotton species, bales and cotton from various regions would be expected from differences in fiber fineness, convolutions, crimp and other factors contributing to real area of contact and time of contact during slip.

In addition, Hertel and Lawson reported higher values of the friction of viscose and of mercerized cotton than for raw or mechanically processed cotton. The smoother fibers give a larger number of contacts per fiber and the probably larger rayon fiber gives a larger area for each contact. Likewise, the total time of contact per fiber during a single swing cycle increases for the smooth fibers.

In general, we feel that our frictional measurements support the

data reported by Hertel and Lawson and aid in a fuller understanding of the data obtained by them.

3. Fiber Cohesion Measurements of Scardino and Lyons

Scheier and Lyons[46, 47] have reported in a series of papers methods of measuring fiber roughness and fiber friction with instruments developed at the Textile Research Institute. Scardino and Lyons more recently have applied these, the ASTM static cohesion measuring method,[39,40,41] and a drafting analyzer for measurement of the dynamic cohesion of the fibers to investigations of surface and cohesive properties of fibers of Dacron polyester fiber specimens containing 0.1 and 2.0 per cent, respectively, of TiO_2 delusterant.

The fibers were processed through worsted, cotton, and woolen systems and examined for changes in cohesion, surface asperity count and height, friction, and other physical properties. In general, it was found that the cohesion of the smooth fibers was greater than for the rough fibers at all stages of processing. Cohesion decreased with processing.

Friction data were reported in only a few cases and appeared to be inconclusive possibly because the instrument was unable to measure friction accurately at the lowest normal force of 4.8 mg. (We were unable to obtain good measurements for cotton below 5 to 7 mg of normal force with our best instrument.) At a normal force of 15.3 mg, the smooth fibers displayed a slightly greater frictional force than the rough ones.

Surface asperity count was reduced by processing causing differences between the rough and smooth fiber cohesion behavior to decrease. Inserted crimp was also reduced. Yarn uniformity was better for the rough fiber

than the smooth.

Each of these findings agree with our data on single fiber friction indicating that large smooth cylindrical fibers give large areas of contacts. Any element introduced to roughen the fibers such as a special cross-sectional shape, crimp, or delusterants tend to reduce the number of contacts in a fiber bundle, the area of individual contacts, and the time of contact during individual fiber traverse. This reduction in the overall interfiber frictional effects diminishes fiber bundle clumping and increases yarn uniformity.

4. Summary

Data obtained by us in single fiber frictional measurements of cotton and other fibers indicate, in agreement with data of Pascoe and Tabor,[12] that the area of individual fiber to fiber frictional contacts is one of the most important parameters determining interfiber friction of fiber assemblies at very low normal forces. When the load is very small or forces are only those of cohesion it is the overriding parameter except for large fiber surface irregularities giving stick peaks of high energy. Factors which reduce the area of contact between fibers, the number of individual contacts between the fibers, the energy of large friction stick peaks, and the integrated time of contact during a traverse of unit length of fiber reduce the interfiber friction. Fiber diameter, cross-sectional shape, longitudinal shape, and surface roughness affect the area of contact and the time of contact of contiguous areas during a fiber traverse or motion. Fiber tension also affects the area of contact.

Consideration of these various elements in the explanation of our own data, and those provided by Hertel and Lawson and by Scardino and Lyons,

clarify the data provided from each separate investigation. These data are found to mutually support each other by applying the interpretation that the frictional forces between fibers are principally dependent on real area of fiber to fiber contact, number of contacts, the energy of the major friction stick peaks, and integrated time of contact per unit of traverse length. The latter of course is affected by the energy of the individual stick peaks which appear to be larger in the case of cotton than of other fibers examined.

F. FRICTION OF COTTON

The various variables in the friction measurement of cotton, especially if the measurements were made only of cotton by the investigators, have concealed the true nature of its interfiber friction. Much data that was probably valid enough for some interpretation was ascribed to variability of the cotton itself.

If now we re-examine the data that we have available as shown in Table 11, it is seen that most of the μ_k values obtained for cotton lie within the range 0.25 ± 0.05. This limited range occurs in spite of known probably mismatch of conditions of humidity, tension, temperature, instrumentation, species, weathering, age, process stage, and normal force. Any of these variables may introduce a difference of up to approximately ± 10 per cent, or even more in the case of the instrument.

The μ_s values lie within the range 0.38 to 0.59. For unprocessed cotton, discounting the interpolated values of Morrow[48] and of Krowicki,[38] the range is only 0.49 to 0.59 or 0.54 ± 0.05. The μ_s/μ_k range is 1.73 to 2.46, the latter value being at a low normal force of 10 mg and possibly signifying bouncing of the instrument arm. The value 0.54 for μ_s

Table 11. Frictional Coefficients of Cotton Obtained by Various Investigators

Investigator	Method	Normal Force (mg)	Tension	μ_s	μ_k	μ_s/μ_k	Comments
Morrow	Fiber Pads			0.396*	0.220	1.80*	1931
Mercer and Makinson	Fiber to Fiber	170-180		0.570	0.316*	1.80*	1947
Krowicki	Rotating Incl. Plane			0.452*	0.249	1.80*	Deltapine 1960
McBride	Fiber to Fiber	26		0.540	0.300	1.80	Empire WR 1965
Viswanathan	Fringe			0.587	0.327	1.80*	1966
Bryant	Fiber to Fiber	20	425	0.523	0.290	1.80	Empire WR 1966
Levy	Fiber to Fiber	20	500	0.500	0.275	1.82	Empire WR Bale 1966
Levy	Fiber to Fiber	20	500	0.530	0.289	1.82	After Opening & Cleaning
Levy	Fiber to Fiber	20	500	0.540	0.307**	1.73	After Picking 1966
Levy	Fiber to Fiber	20	500	0.550	0.317**	1.73	After Carding 1966
Gunther	Fiber to Fiber	20	425	0.590	0.240	2.46	Empire WR 1966
Belser	Fiber to Fiber	20	425	0.520	0.271	1.91	High Draft 1967
Belser	Fiber to Fiber	20	425	0.496	0.243	2.04	Low Draft 1967
Cromer	Fiber to Fiber	20	425	0.490	0.250	1.94	Empire WR 1968
Cromer	Fiber to Fiber	20	425	0.450	0.245	1.82	Roving 1968
Cromer	Fiber to Fiber	20	425	0.390	0.190	2.03	Spinning 1968
Whitworth	Fiber to Fiber	20	425	0.453	0.225	2.02	Pima Menoufi Comber Lap 1967
Whitworth	Fiber to Fiber	20	425	0.417	0.209	1.98	Combed Sliver
Whitworth	Fiber to Fiber	20	425	0.413	0.213	1.94	Breaker Drawing
Whitworth	Fiber to Fiber	20	425	0.433	0.224	1.93	Finished Drawing
Whitworth	Fiber to Fiber	20	425	0.383	0.197	1.94	Roving
Whitworth	Fiber to Fiber	20	425	0.399	0.210	1.90	Spinning
Whitworth	Fiber to Fiber	20	425	0.386	0.173	2.23	Comber Noil

*Values were determined by calculation from only one value cited.

**These higher values apparently were run at a higher humidity than for the remaining measurements.

is probably high and the value μ_k of 0.25 is probably low. We obtain 0.275 for μ_k if we look only at values we have taken at 20 mg for unprocessed cotton, and 0.516 for μ_s giving a μ_s/μ_k ratio of 1.88 (using the highest ten peaks). These values appear to be reasonable in view of our now extensive experience.

If now the cotton varies markedly in fineness, convolutions, growth irregularities, reversals, or any other intrinsic feature which would decrease the surface area of contact or the time of contact during traverse, the measured friction values would decrease. As the normal force is decreased to the low values present in fiber processing, the roughness effect becomes more apparent in degrading the kinetic coefficient. However, individual fiber snagging, giving large stick peaks of high energy, seems to be the principal driving agency for fiber travel of rough fibers. This is evidenced in the study of high draft cotton performed for Mr. Grant as indicated by the large distribution range of static coefficients of friction.[28]

Rough features are also inserted by damage during processing but apparently are compensated for as to friction by the decrease in convolution heights, partial removal of crimp, fiber straightening effects resulting in lower energies for friction peak heights, and removal of short and damaged fibers at various process stages. The role of the cotton wax has not been properly determined. A few measurements made earlier indicated that wax removal increased the height of sticks and thus the ratio μ_s/μ_k. This is highly probable.

Krowicki[38] suggested that the cotton wax was smoothed out on successive runs on his instrument to give steadily decreasing values of μ_k.

We did not find this the case at a normal force of 20 mg between single fibers. Decreases observed in the initial two or three passes were ascribed to straightening of the fiber and viscous flow as shapes of sticks were changed and reduced in energy. Thirteen traverses by a single fiber revealed reductions in friction only during the first few passes. A pile up of wax at an asperity might result in a jump over the asperity with a lesser force and a lower energy for the stick. At very low normal forces the wax may play a larger part than we have yet observed. However, most evidence that we have does not particularly indicate this action as a real probability.

The part that the energy of the sticks plays in fiber travel at low normal forces is of great interest and should be further investigated. There is certainly some median desirable behavior which is not established by cylindrical uncrimped fibers. A degree of crimp, convolution, and surface roughness is apparently desirable for uniform processing. The crimp inserted into nylon was readily observable at no applied normal force as shown in Figure 22. The very variability of the fibers themselves in size and other features may be more desirable than not. The use of the scanning electron microscope in conjunction with friction examination of individual fibers appears to be a direction to go in order to relate more exactly the friction of an individual fiber to its shape and surface. Further studies along this line would fill in gaps in our current endeavor and allow one to predict more precisely a desired fiber friction and shape specification.

Although we have discussed only low normal force values for fiber processing it is also apparent that in the yarn high normal forces are

involved as a result of twist. Now that the low normal force is moderately well understood, studies at high normal forces may give improved knowledge of fiber behavior in yarns.

We have outlined in some detail the general behavior of cotton and other fibers in interfiber friction and believe that the principal items related to understanding frictional data in the literature or in practice have been delineated. The application of this information in the textile industries and in further experiments should lead to improved yarns and more definite knowledge concerning the few remaining points needing clarification.

X. CONCLUSIONS

Two servo-controlled fiber friction measuring instruments allowing graphical display of friction data and electronic integration of force-energy data have been constructed and applied to the measurement at low normal forces of both the static and the kinetic coefficients of cotton and other fibers arranged at 90 degrees to each other in a horizontal plane. Normal forces in the range 1 mg to 40 mg were feasibly utilized. By a special adaptation of the instrument, the friction of a single fiber in contact with another under its own weight alone may be measured. The data provided are sufficiently accurate to delineate a data plot character as a function of position along the traversing fiber specific to the fiber, and to repeatedly display major frictional sticks which may be identified with micrographic physical features of cotton fibers. The latter are generally convolutions, reversals, growth abnormalities, or fiber damage or shape changes inflicted by cotton processing equipment.

The fiber friction of cotton is affected by the ambient conditions of humidity and temperature and by the temperature history of the cotton. It is further affected by the experimental procedures involving mounting tension, fiber traverse rate, and normal force between the fibers. In general, any experimental or condition factor affecting the areas of contiguous zones of adjacent fibers was found to affect the friction since the frictional force apparently follows the same general principles as outlined by Pascoe and Tabor[12] for nylon on nylon.

It was shown by them that for crossed cylinders

$$F = C_1 \, s \, W^{2/m} \, D^{-2(2-m)/m}$$

where C_1 is a constant of about 1.4,

s is the shear strength of nylon used as 1.5 kg/mm^2 in this case,

W is the load or normal force, and

m is the slope of the line obtained by plotting log load versus the diameter of the impression made by a steel indenter in a nylon block.

For nylon, the value m = 2.7. Using this value in the preceding equation, $F = C_1 s W^{0.74} D^{0.52}$, and dividing by W^m, $\mu = C_1 s W^{-0.26} D^{0.52}$. This expression gave theoretical points matching experimental data for nylon and other fibers over a series of normal forces and diameters.

The friction of cotton is also affected by the normal force between fibers and the fiber diameter but does not lend itself to precise measurement of these effects. However, the trend in the data support a behavior of the type outlined by Pascoe and Tabor for nylon with differences caused by the generally rough and convoluted surface of cotton in contrast to the usually smooth cylindrical shape of the nylon.

In the case of rough fibers of non-cylindrical shape, similar to cotton which is a thin convoluted ribbon, the area of contact between contiguous fibers during traverse of one across another, or at some other angle approaching zero, continually changes in dimensions. Secondly, the rough and large features characteristic of the fiber tend to snag adjacent fibers and carry them along. The friction data measured at a 90 degree angle exhibit large stick peaks of high energy. When a slip occurs, the fibers are deflected apart and a time of no fiber to fiber contact exists. Roughness then contributes to snagging and to no contact time. The result, generally, is to reduce both kinetic and static coefficients of friction when compared to very smooth cylindrical fibers, which exhibit high contact

continuity and narrow and low energy stick peaks. High draft cotton displays a large number of high energy peaks and a large dispersion of energies as compared to low draft cotton.

Single fibers pulled from card lap or roving require relatively large forces, up to 100 mg, to withdraw them indicating a series of fibers moving at once or many points in contact since the highest force recorded for a single fiber in contact at a single point was about 0.75 mg. The force to pull a fiber from roving was two-thirds that for pulling the fiber from card lap indicating that processing reduced the snagging effect due to crimp, convolutions, reversals, or other growth features.

If the normal force between crossed cotton fibers is reduced below about 7 mg while measuring with the instrument designed here, the data plots indicate a reduction in coefficients of kinetic and static friction. On the other hand, for smooth nylon, the coefficients continue to increase to very high values even down to normal forces of 1 mg, the limit of the instrument. Although this behavior indicates an intrinsic fault of the instrument except at very low traverse velocities (well below 0.1 mm/sec) the behavior also exhibits an intrinsic behavior of the rough fiber. This is the relatively long time required to re-establish contact after each slip and the deflection of the fiber from any contact in the free state.

The high fiber velocities used in textile processing and the very low normal forces accentuate the behavior to the degree that the principal mode of travel of rough fibers appears to be due to snagging whereas for smooth fibers it is frictional forces of the large contiguous zones typical of these fibers.

Because of the high number of experimental and material variables in

measurements of the fiber friction of cotton and of other fibers it is infrequent that the measurements made by other investigators in the past can be validly compared. However, a value for the kinetic coefficient of cotton of about 0.25 ± 0.05 and for the static coefficient of 0.48 ± 0.10 encompass most of the measurements that have been published. For Empire WR cotton hand ginned, at 425 mg tension, 20 mg normal force, and 60 per cent relative humidity, we obtained 0.275 and 0.516 respectively.

Processing of cotton from the boll to the yarn increased the friction of many fibers due to damage but reduced the friction due to straightening of the fibers, reduction in the height of the topographic features, and changes in fiber length distribution due to damage and sorting in the processing. The overall result was friction reduction.

Specific conclusions are listed below:

(1) The friction coefficients of cotton fibers increase with relative humidity increase, with a sharp increase occurring above 65 per cent relative humidity;

(2) They increase when the cotton is temperature cycled above about 70°C (158°F);

(3) They decrease when fiber tension is increased;

(4) They increase when normal force is decreased;

(5) They vary with fiber size which affects the size of the areas of contact;

(6) The roughness of the fibers decreases over all frictional forces but contributes to snagging which appears to be the principal cause of cotton fiber travel in the processing stages;

(7) Processing reduces height of fiber features, inflicts fiber damage, and changes fiber length distribution which activities generally reduce the average kinetic or static coefficients of friction of the fiber;

(8) Removal of the cotton wax changed the character of the stick peaks and their magnitude, but did not greatly change average

friction coefficients (This supports the theory that high peaks and snagging are important in fiber travel and processing.);

(9) Measurements of cotton shear friction by a new method described by Hertel and Lawson, and of fiber cohesion effects in processing Dacron, conducted by Scardino and Lyons, have generally been in good agreement with data principles we have obtained and outlined herein; and

(10) The many variables injected into the friction measurements of cotton by others during the past make it difficult to compare data without a large margin of uncertainty or error. Comparative data of good quality will have to be produced under precise experimental controls of each of the listed items (1) through (5) above.

The friction measuring instruments as developed have provided a wealth of detailed frictional data on fibers which allow assignment of character to the individual fibers and allow new methods of analysis and interpretation of fiber friction. These measurements and analyses have contributed new understanding of the principles of interfiber friction and provide valuable information which should lead to better control of fiber frictional properties and their application to improved textile products.

APPENDIX

APPENDIX

A. INVESTIGATIONS OF STRUCTURES OF FIBERS BY TECHNIQUES OF INFRARED SPECTROSCOPY

Infrared spectra were made of cotton, cotton wax, and cotton fibers by standard methods outlined in detail in Semiannual Report No. 2 and a thesis by Kirkland. It was found that the wax percentage of a standard cotton specimen was too small for the wax on the cotton specimen alone to give a useful and interpretable spectra of the wax or changes in it. Solvent methods did not appear appropriate.

A fiber press was developed to use the fiber alone as the specimen. The press proved to be a useful method of preparing a specimen for obtaining a spectra but did not give sufficient additional amplitude or definition of the wax present to be of value for the intended purpose.

Cotton specimens were examined by use of a double beam, multiple internal reflectance attachment for the spectrometer,[34] expecting that the wax absorption spectra would be increased in amplitude of absorption but results did not warrant the study as a primary effort and it was not pursued further.

The crystallinities of cotton, ramie, and viscose were investigated by deuteration of fiber press specimens with the gaseous phase of D_2O, drying, and making spectra. Crystallinity was determined by a method outlined by Marrinan and Mann[49] employing the amplitude of the band at 6.15 microns.

Marrinan and Mann[49] deuterated viscose films, resubstituted the hydrogen using light water, and measured the refractive index of the resubstitution water to determine the amount of deuteration exchanged.

The percentage of amorphous material was calculated from the weight of the cellulose, the weight of the H_2O, and the amount of deuterium which exchanged.

If Beer's Law holds, the absorbance of a band at wavelength λ is related to the concentration of the sample by

$$\text{absorbance} = \log_{10} (I_o/I) = k_\lambda \, c \, \ell \quad ,$$

where I_o is the intensity of the radiation incident on the sample, I is intensity of the radiation transmitted, k_λ is the extinction coefficient per mole fraction, and ℓ is the pathlength of the radiation through the sample. Thus,

$$\frac{\log_{10} (I_o/I)_{OD}}{\log_{10} (I_o/I)_{OH}} = \frac{k_{OD} \, C_{OD}}{k_{OH} \, C_{OH}} \quad ,$$

where

$$C_{OD} + C_{OH} = 1 \quad .$$

From their measurements of absolute crystallinity, using the refractive index method, Marrinan and Mann[49] concluded the ratio $k_{OD}/k_{OH} = 1.11$ for cellulose. Therefore, the two equations in two unknowns can be solved for C_{OD}/C_{OH}.

Sepall and Mason,[50] in studying starches, assumed that the value of 1.11 found by Marrinan and Mann holds for the various forms of cellulose. There is no apparent reason why the value of this constant should not hold for fibrous cellulose.

Another possible method of obtaining crystallinity estimates employs only the 3μ band. Since the band is due to both crystalline and amorphous regions, the same band after deuteration is due to crystalline OH's only. Therefore,

$$\frac{A_{OH} \text{ after deuteration}}{A_{OH} \text{ before deuteration}} = \frac{A_{OH} \text{ crystalline}}{A_{OH} \text{ crystalline} + A_{OH} \text{ amorphous}} ,$$

where A is absorbance.

Using this method average crystallinity values for Empire WR cotton, ramie, and viscose were found as follows:

Cotton	76 Per Cent
Ramie	72 Per Cent
Viscose	42 Per Cent

These values compare favorably with those found in the literature by other methods as shown in Table 12.

Unfortunately, time did not permit the employment of this method in an examination to determine the crystallinity of cotton after various stages of processing. However, the development of the fiber press technique and its combination with the deuteration method of studying the crystallinity of cellulose fibers are considered to be useful contributions to fiber measurement methods.

Table 12. Comparison of Crystallinity Measurements of Cotton, Rayon, and Ramie as Determined by Different Investigators

Investigator	Per Cent Crystallinity			
	Cotton	Rayon	Ramie	Technique
Phillipp et al. (51)	85	68*	95*	Acid Hydrolysis
Roseveare (52)	57-77	40-57	--	NO_2 Oxidation
Magne et al. (53)	87	57	--	Calorimetric
Ward (54)	70	40	--	Heat of wetting
Heritage et al. (55)	--	43	--	X-ray
Ant-Wuorinen (56)	80	30	--	X-ray
Hermans (57)	70	39	70	X-ray
Segal et al. (58)	79	--	--	X-ray
Smith (59)	59	28	--	D_2O Exchange
Frilette (60)	75	44	--	D_2O Exchange
Mann (49)	69	26	--	D_2O Exchange
Nelson (61)	87	57	--	I.R. Band Ratios
Average	74.4	41.2		
Hicks	75.7	42	71.7	D_2O Exchange

*These values were not used in calculating the averages as they are obviously too high.

B. INVESTIGATIONS OF THE CRYSTALLINITY OF COTTON FIBERS BY X-RAY DIFFRACTION TECHNIQUES*

1. General

X-ray diffraction techniques have been used to examine the effects on cotton crystallinity of milling cotton fibers in a Wiley Mill to 20, 40, and 60 mesh, of ball milling them for a period of 15 hours, and for examining the crystallinity of cotton specimens before and after ginning.

2. Effect on Crystallinity of Milling Cotton in a Wiley Mill

a. Procedure

Samples of 1964 crop cotton, mechanically ginned, were chopped to pass a 20, 40, and 60 mesh screen in a Wiley Mill. X-ray powder diffraction patterns were run on each type. There were seven samples taken of each type and for each sample there was calculated a "crystallinity ratio;" C.R.:

$$C.R. = \frac{I_{002} - I_{am}}{I_{002}} ,$$

where I_{002} is the intensity of the 002 peak (at about 22.6°) and I_{am} is the intensity of the minimum at about 19° where there are no peaks, an intensity characteristic of amorphous scattering (hence, I_{am}).

The data are shown in Table 13, along with the mean values and the associated standard deviations, σ, defined as

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*These investigations were carried out under the direction of Dr. Robert A. Young, Research Professor of Physics and Head of the Diffraction Laboratories, Georgia Institute of Technology, principally by Harry Ellis, Graduate Assistant from the School of Physics.

Table 13. Comparison of Crystallinity Ratios of Cotton Milled to 20, 40, and 60 mesh in a Wiley Mill

Sample	20 Mesh	40 Mesh	60 Mesh
AA	0.784	0.802	0.807
A	0.792	0.795	0.797
B	0.765	0.797	0.803
C	0.781	0.789	0.808
D	0.769	0.782	0.804
E	0.783	0.799	0.792
F	0.786	0.794	0.797
Average Value of C.R.	0.780	0.794	0.801
Standard Deviation σ	0.010	0.007	0.006

$$\text{Crystallinity Ratio} = \frac{I_{002} - I_{am}}{I_{002}}$$

Differences:

$$\delta_{20,40} = 0.014 \pm 0.017 \qquad 1.8\% \pm 2.1\%$$

$$\delta_{40,60} = 0.007 \pm 0.013 \qquad 0.9\% \pm 1.6\%$$

$$\delta_{20,60} = 0.021 \pm 0.016 \qquad 2.6\% \pm 2.0\%$$

Note: By the analysis of variance there was a significance difference between the effects of the number of mesh at 95% confidence level. By Duncan's multiple range tests, there was no difference between the means of 40 mesh and 60 mesh at 95% significance level.

$$\sigma = \sqrt{\frac{\sum(\Delta x)^2}{n-1}} \quad ,$$

where Δx is the number $|x_o - x|$, x_o the mean value, and n, the number of samples (7 in this case).

The electronic settings were essentially the same throughout, with the PHA set each day. The same sample-holder was used, and patterns of this empty holder showed no scatter in the regions of interest.

The crystallinity ratio of the cotton subjected to ball milling for 15 hours was 0.18.

b. Conclusions

The data are insufficient to permit us to determine what, if any, is the difference in C.R. between the twenty and forty, and the forty and sixty mesh cotton. A larger number of samples would possibly allow determination of a more definite difference.

There does appear to be a slight increase in the crystallinity ratio of the sixty mesh as compared to the twenty, since we do get a $\delta_{20,60} > (\sigma_{20} + \sigma_{60})$. The difference is small, however, and a larger number of samples would be needed to establish a definite number for its magnitude.

3. Effects of Ginning on the Crystallinity of Seed Cotton

Crystallinity measurements were made on three types of cotton: (1) 1964 crop cotton which had been mechanically ginned; (2) 1965 crop cotton from the same soil which had also been mechanically ginned, and

*The term n-1 rather than n is used to give an additional allowance for the small number of measurements made.

(3) identical 1965 crop seed cotton which was hand-ginned. Ten samples were taken of each type, and all samples were chopped by a Wiley Mill to pass a 20 mesh screen. For each sample an x-ray diffraction pattern was run, and a crystallinity ratio (C.R.) was calculated:

$$C.R. = \frac{I_{002} - I_{am}}{I_{002}} \quad ,$$

where I_{002} is the intensity of the (002) diffraction peak (dependent on the density of crystalline material in the sample) and I_{am} is the intensity of the minimum at about 19° (dependent on the total mean density of the sample). A pattern made of the empty sample holder showed no scatter in the region of interest. The electronic settings were maintained constant throughout.

The results are shown in Table 14 along with the standard deviation σ:

$$\sigma = \sqrt{\frac{\sum(\Delta x)^2}{n-1}} \quad ,$$

where $\Delta x = (x_o - x)$; x_o is the mean value of the C.R. for a particular type, and n is the number of samples. The probable error shown is the sum of the standard deviation of the values involved.

The sum of the standard deviations is larger than the measured differences both between the mechanically ginned 1964 and 1965 cotton, and between the mechanically and hand-ginned 1965 cotton. There was inappreciable difference in crystallinity among the three types of cotton tested in this experiment.

Table 14. Comparison of Crystallinity Ratios of Hand and Mechanically Ginned Cotton

Sample	(α) 1966 Mech. Ginned	(β) 1965 Mech. Ginned	(γ) 1965 Hand Ginned
III	.778	.814	.789
IV	.807	.803	.800
V	.800	.800	.789
VI	.782	.784	.774
VII	.806	.817	.772
VIII	.790	.801	.803
IX	.803	.800	.803
X	.789	.800	.795
XI	.781	.799	.782
XII	.780	.823	.793
Mean	.792	.804	.790
Standard Deviation (σ)	.011	.011	.012

$\delta_{\alpha,\beta} = .012 \pm .022$ 1.5% ± 2.8%

$\delta_{\beta,\alpha} = .014 \pm .023$ 1.8% ± 2.9%

Note: A subsequent statistical analysis of these data showed there was a significant difference between means of these data at 95% significance level but not at the 99% level. By Duncan's multiple range tests, there was no difference between specimens α and γ at 95% level. But significant differences were observed between α and β, and γ and β at the same significance level.

Although very small changes in crystallinity may have occurred in the cotton fiber as a result of ginning, the friction changes as related to crystallinity changes did not appear to be of a paramount value to the investigation. In addition, lack of personnel available for this area of the research limited more extensive work. Correlation of crystallinity changes through all the processing stages of cotton with the frictional changes would be of interest in future work.

BIBLIOGRAPHY

LIST OF REFERENCES

1. da Vinci, Leonardo, "The Notebooks of L. da Vinci," (compiled by Edward MacCurdy), Reynal and Hitchcock, New York, 1939.

2. Amontons, G., "On the Resistance Originating in Machines," Mem. Acad. Roy. 206-222 (1699).

3. Coulomb, C. A., "The Theory of Simple Machines," Mem. de l'Acad. Roy. Sci. 10, 161-332 (1785).

4. Bowden, F. P. and D. Tabor, "The Friction and Lubrication of Solids," Part II, 214-241, Oxford Clarendon Press (1964).

5. Howell, H. G., K. W. Mieszkis, and D. Tabor, "Friction in Textiles," 28-53, Butterworths Scientific Publications, London (1959).

6. Howell, H. G., "The Laws of Static Friction," Textile Research Journal 23, 589-591 (1953).

7. Lincoln, B., "Frictional and Elastic Properties of High Polymeric Materials," Brit. J. of Appl. Phys. 3, 260-263 (1952).

8. Huntington, J. D., "Friction of Fiber Assemblies," Research London, 10, 163-164 (1957).

9. Howell, H. G. and J. Mazur, "Amonton's Law and Fibre Friction," J. Text. Inst. 44, T59-T69 (1953).

10. Lodge, A. S. and H. G. Howell, "Friction of an Elastic Solid," Proc. Phys. Soc. Lond. 67-B, 89-97 (1954).

11. Tabor, D., "The Hardness of Metals," Oxford Clarendon Press (1951).

12. Pascoe, M. W. and D. Tabor, "Friction of Nylon as a Function of Load and Surface Curvature," Research, London 8, S-15-S17 (1955).

13. Adderley, A., "The Clinging Power of Single Cotton Hairs," Textile Research Journal 13, 249-255 (1922).

14. B. G. Hood, "Frictional Properties of Textile Fibers," Textile Research Journal XXIII, 495-505 (1953).

15. Lindberg, J. and N. Gralen, "Friction Between Single Fibers: II Frictional Properties of Wool Fibers Measured by the Fiber Twist Method," Textile Research Journal 18, 287 (1948).

16. Mercer, E. H. and K. R. Makinson, "Textile Fibers: Frictional Properties," Journal Textile Institute XXXVIII, T227-T240 (1947).

17. Bowden, F. P. and L. Leben, "The Nature of Sliding and the Analysis of Friction," Proc. of the Royal Society CLXIX, 371-391 (1938).

18. Guthrie, J. C. and P. H. Oliver, "Interfiber Friction," Journal Textile Institute XLIII, T579-T595 (1952).

19. Penoyer, R. F., "Automatic Torque Balance for Magnetic Anisotropy Measurements," Rev. Sci. Instr. 30, 711-714 (1959).

20. Humphrey, F. B. and A. R. Johnson, "Sensitive Automatic Torque Balance for Thin Magnetic Films," Rev. Sci. Instr. 34, 348 (1963).

21. McBride, T. E., "Development of an Instrument to Measure Friction of Textile Fibers," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, Atlanta, Georgia (1965).

22. Gunther, D. H., Jr., "An Evaluation of Fiber Friction at Low Normal Forces," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, Atlanta, Georgia (1968).

23. Huff, J. O., "Measurement of the Relationship Between Textile Fiber Shapes and Their Frictional Characteristics," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1968).

24. Boys, T. R., "An Evaluation of Factors Affecting the Frictional Properties of a Selected Cotton Fiber Sample," Thesis for the M.S. Degree in Textile Engineering, Georgia Institute of Technology, Atlanta, Georgia (1964).

25. House, D. L., "Applications of Optical and Electron Microscopy to Studies of Textile Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1966).

26. Goldfarb, A. M., "Cotton Fiber Properties as Affected by Ginning," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1966).

27. Whitworth, L. B., "Alterations of Physical Properties of Long Staple Cotton by Combing, Drawing, and Roving," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1967).

28. Belser, R. B. and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 5, USDA (SURDD) Grant No. 12-14-100-7661(72), Georgia Institute of Technology, August 1967.

29. Bryant, J. P., "An Investigation of the Factors Which Influence the Frictional Properties of Textile Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1966).

30. Rushing, Eugene V., Jr., "An Investigation of Some of the Properties of Arachnid Silks," Special Problem Paper, A. French Textile School, Georgia Institute of Technology, March 1967.

31. Simmons, J. F., "Investigation of a Miniature Spinning System as a Screening Aid for Spin Finish Components on Polyacrylonitrile Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1967).

32. Wakelyn, P. J., "Quaternary Ammonium Salts as Antistatic Agents on Polyacrylonitrile Fibers," Thesis for the M.S. Degree in Textile Chemistry, Georgia Institute of Technology, Atlanta, Georgia (1967).

33. Belser, R. B. and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 3, USDA (SURDD) Grant No. 12-14-100-7661(72), Georgia Institute of Technology, August 1966.

34. Belser, R. B. and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 2, USDA (SURDD) Grant No. 12-14-100-7661(72), Georgia Institute of Technology, February 1966.

35. Rabinowicz, Ernest, "Friction and Wear of Materials," John Wiley and Sons, Inc., New York (1965), 52-108.

36. Levy, H. R., "The Effects of Opening, Cleaning, Picking, and Carding on the Physical Properties of Empire WR Cotton Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1966).

37. Cromer, E. D., "The Effects of Drawing, Roving, and Spinning on the Physical Properties of Empire WR Cotton Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1968).

38. Krowicki, R. S., "Some Frictional Properties of Cotton Fibers," Thesis for the M.S. Degree in Physics, University of Tennessee (1960).

39. Scardino, F. L. and W. J. Lyons, "Fiber Surface Properties in Relation to Linear Assemblies During Processing, Part I; General Considerations; Results on the Worsted System," Textile Research Journal 37, 874-880 (1967).
Scardino, F.L., "The Influence of Fiber Geometry on the Cohesion of Cotton Card Slivers," Textile Bulletin, April 1967, 1-5.

40. Scardino, F. L. and W. J. Lyons, "Fiber Surface Properties in Relation to Linear Assemblies During Processing, Part II: Results on the Cotton and Woolen Systems; Other Studies," Textile Research Journal 37, 982-988 (1967).

41. Scardino, F. L. and W. J. Lyons, "Fiber Surface Properties in Relation to Linear Assemblies During Processing, Part III: Effects of Processing on Surface and Geometric Properties of Fibers," Textile Research Journal 37, 1005-1008 (1967).

42. Belser, R. B. and J. L. Taylor, "Frictional Properties of Cotton Fibers," Semiannual Report No. 5, USDA (SURDD) Grant No. 12-14-100-7661(72), Georgia Institute of Technology, August 1967.

43. Kirkland, W. E., "An Investigation of Techniques to Determine Infrared Spectra of Textile Fibers," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1966).

44. Hicks, H. L., "A Crystallinity Study of Cellulosic Fibers Using Infrared and Deuteration Exchange Techniques," Thesis for the M.S. Degree in Textiles, Georgia Institute of Technology, Atlanta, Georgia (1968).

45. Hertel, K. L. and R. Lawson, "Shear Friction in Textile Processing," Textile Bulletin, 23-27 and 63 (May 1968).

46. Scheir, S. C. and W. J. Lyons, "Studies of the Surface Geometry of Fibers, Part II: Improved Instrumentation and Representative Results on Camel Hair and Dacron," Textile Research Journal XXXIV, 410-416 (1964).

47. Scheir, S. C. and W. J. Lyons, "Measurement of the Surface Friction of Fibers by an Electro-Mechanical Method," Textile Research Journal 35, 385-394 (1965).

48. Morrow, J. A., "The Frictional Properties of Cotton Materials," J. Tex. Inst. 22, T425-T440 (1931).

49. Mann, J. and H. J. Marrinan, "The Reaction Between Cellulose and Heavy Water. Part 1. A Qualitative Study by Infrared Spectroscopy. Part 2. Measurement of Absolute Accessibility and Crystallinity. Part 3. A Quantitative Study by Infrared Spectroscopy," Transactions of the Faraday Society 52, 481-497 (1956).

50. Sepall, O. and S. G. Mason, "Hydrogen Exchange Between Cellulose and Water," Canadian Journal of Chemistry 39, 1934 (1961).

51. Phillipp, H. J., M. L. Nelson, and H. M. Ziffle, "Crystallinity of Cellulose Fibers as Determined by Acid Hydrolysis," Textile Research Journal 17, No. 11, 585-593 (November, 1947).

52. Roseveare, W. E. and D. W. Spaulding, "Effect of Swelling and Supermolecular Structure on Reaction of Cellulose with Nitrogen Dioxide," Industrial and Engineering Chemistry 47, No. 10, 2172-2175 (October 1955).

53. Magne, F. C., H. J. Portas, and H. Wakeham, "A Calorimetric Investigation of Moisture in Textile Fibers," Journal of the American Chemical Society 69, 1896 (1947).

54. Ward, K., Jr., "Crystallinity of Cellulose and Its Significance for the Fiber Properties," Textile Research Journal 20, 366 (1950).

55. Heritage, K. J., J. Mann, and Roldan-Gonzales, "Crystallinity and Structure of Celluloses," Journal of Polymer Science Part A 1, 682 (1963).

56. Ant-Wuorinen, O. and A. Visapää, "X-ray Diffractometric Method for the Determination of the Crystallinity of Cellulose," Norelco Reporter 9, 52 (1962).

57. Hermans, P. H., "X-ray Investigations on the Crystallinity of Cellulose," Makromolekulare Chemie 6, 26 (1950).

58. Segal, L., J. J. Creely, A. E. Martin, and C. M. Conrad, "An Empirical Method for Estimating the Degree of Crystallinity of Native Cellulose Using the X-ray Diffractometer," Textile Research Journal 29, 786-794 (1959).

59. Smith, J. K., "Structural Study of Cellulosic Fibers," Journal of Polymer Science Part C No. 2, 406 (1963).

60. Frilette, V. J., J. Hanle, and H. Mark, "Rate of Exchange of Cellulose with Heavy Water," Journal of the American Chemical Society 70, 1107-1113 (March, 1948).

61. Nelson, M. L. and R. T. O'Connor, "Relation of Certain Infrared Bands to Cellulose Crystallinity and Crystal Lattice Type. Part 2. A New Infrared Ratio for Estimation of Crystallinity in Celluloses I and II," Journal of Applied Polymer Science 8, 1325-1344 (1964).