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MECHANICAL TREATMENT OF PULP FIBERS FOR PROPERTY DEVELOPMENT

A thesis submitted by

Richard R. Hartman

B.S. 1977 (Chemistry, Paper Science), Western Michigan University

M.S. 1979, Lawrence University

in partial fulfillment of the requirements
of The Institute of Paper Chemistry
for the degree of Doctor of Philosophy
from Lawrence University,
Appleton, Wisconsin

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ABSTRACT

The objective of this thesis was to gain a better understanding of the refining operation. Specifically, the goals were to produce internal fibrillation in pulp fibers, evaluate its influence on paper properties, and establish its importance relative to external fibrillation and fines in terms of paper property development.

An apparatus was constructed that subjected pulp fibers (in the form of a wet handsheet) to repeated compressive loading cycles. Pulp was also treated in an experimental apparatus designed to promote external fibrillation in a Valley beater and in a laboratory conical refiner. Fiber length, water retention, and Canadian Standard Freeness of the pulps were measured. The samples were also analyzed with scanning electron microscopy, transmission electron microscopy, and polarized light microscopy. TAPPI standard handsheets of the refined pulps were formed, and the following properties were measured: breaking length, density, specific light scattering coefficient, tear strength, and air resistance.

The results showed that internal fibrillation could be produced with the repeated compressive action of the apparatus. The effect of internal fibrillation on paper properties caused a threefold increase in breaking length, from 2 km to 6 km. This level was 75% of the highest breaking length achieved with the Valley beater, which was 8 km. The reason for the difference in breaking length between the samples is due to the (calculated) differences in fiber-fiber bond shear strength.

By adding external fibrillation to internally fibrillated fibers and forming the sheet, no change in breaking length was achieved. However, adding fines to a suspension of internally fibrillated fibers increased the sheet breaking length, almost to the level produced by Valley beaten fibers.

In conclusion, it was found that internal fibrillation of pulp fibers is the most important refining effect in improving sheet breaking length. It was also shown that fines are needed to improve interfiber bonding and to increase the breaking length to levels achieved in the Valley beater. Internal fibrillation and fines can be produced in separate steps, indicating an additive approach to refining is possible. The effect of external fibrillation has no apparent influence on improving breaking length, but its importance may lie elsewhere in the sheet forming process.

INTRODUCTION

Wood pulp fibers produced by the pulp mill are generally unsuitable for direct usage by the paper machine. A sheet of paper made from unbeaten fibers is characterized by its low tensile strength; its bulkiness; and open, irregular surface. For most commercial papers, these characteristics are undesirable, but they can, to a large extent, be suitably altered by mechanical processing.

Beating is a general term which means either the batch treatment of a pulp slurry in a beater or passing the pulp continuously through a refiner of some kind before the stock goes to the paper machine. In either case, the structures of most of the fibers are disrupted in the presence of water. This is accomplished by mechanical treatment between the edges and closely spaced faces of rapidly moving bars, with simultaneous mechanical interaction between the fibers themselves. (The terms beating and refining are used as synonyms in this thesis.)

The changes in sheet characteristics brought about by refining are shown in Fig. 1 (1). The improvements are not necessarily proportional to the duration of refining or the amount of work done on the stock. For example, as refining increases, tear resistance passes through a maximum, and the opacity of the sheet decreases steadily. In some cases, the changes in paper properties that are desirable for some grades are undesirable for others.

The main results of refining on fiber properties are threefold. The fibers are shortened; they are crushed and fibrillated internally; and they are abraded and fibrillated on their exterior surface, part of which causes a buildup of small particle size material (called fines) (2). Some of the disadvantages of

using conventional refiners to study the effects of refining are that all three of these effects are occurring in the fibers during the operation.

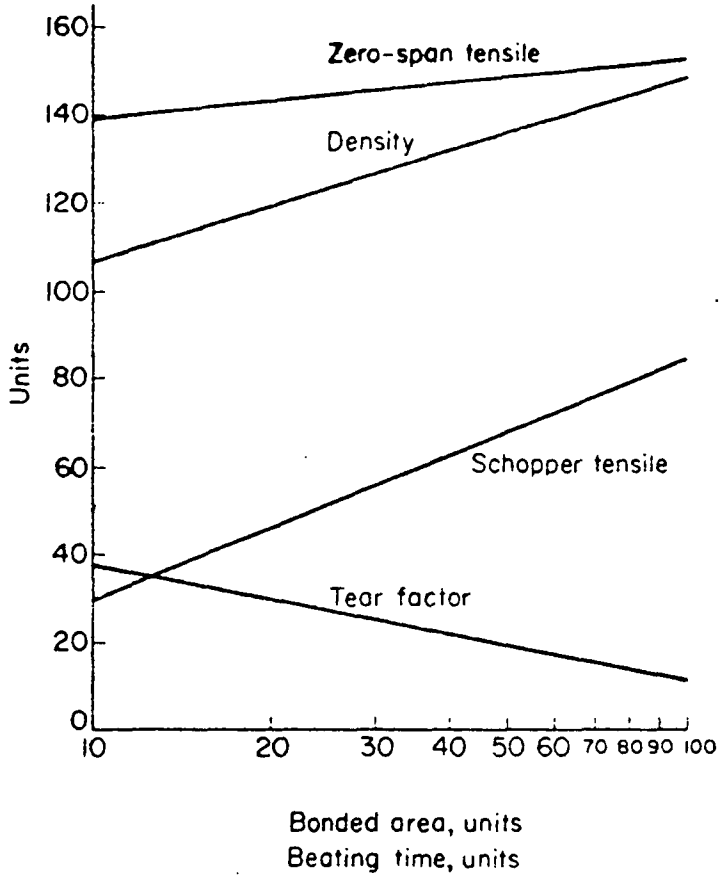


Figure 1. Principal relationship of strength properties and interfiber bonding or refining.

Considerable quantities of power are used in refining. Published estimates of refining energy consumption suggest that the process, when viewed as a mechanical operation, is inefficient (3-6). The major developments in the field of refining over the past 50 years have been almost entirely directed at keeping pace with the increasing production rates of the paper machine. Increases in refiner size, changes in the mode of operation of the refiner, and changes in the refiner configuration have been achieved, yet the basic mechanism of transforming the raw fibers to usable material is still unknown.

Regardless of these improvements over the years, there remain unwanted effects associated with the process. If these undesirable components could be identified and somehow eliminated while still reaching the desired goals of refining, then this would certainly be a step in the right direction toward improving the overall process of refining.

A question of long-standing importance to the process is, what is the goal of refining? In this thesis, refining will be carried out to improve the strength properties of paper (particularly tensile strength). To do so, certain nonproductive actions of conventional refining will be eliminated or minimized. By designing an apparatus which will apply mechanical action to the pulp fibers in a straightforward and controlled manner, the importance of the isolated action can be examined. The effect of this isolated action on fiber and paper properties can also be evaluated with respect to other refining actions (produced by other methods) to establish its relative importance.

This thesis explores the field of refining by specifically addressing three questions:

1. What mechanical treatment is required to produce internal fibrillation in pulp fibers?
2. How do internally fibrillated fibers influence sheet properties, particularly tensile strength?
3. What is the relative importance of internal fibrillation compared to external fibrillation and fines in terms of developing sheet strength?

BACKGROUND

EFFECT OF REFINING ON FIBER PROPERTIES

The structure of a tracheid fiber is shown in Fig. 2 (7). The fiber is protected by a thin membrane called the primary wall (P). Inside the primary wall is a secondary wall which surrounds the lumen. It consists of a number of concentric layers numbered from the outside to the lumen as S_1, S_2, and S_3. These layers are made up of spirals of fibrils which lie at various angles to the major axis of the fiber. The important points to note are: 1) the primary wall, P, and the S_1 secondary wall have very high fibril angles with respect to the fiber axis, and 2) the secondary wall is the predominant layer in the cell wall, which has a very low fibril angle. Therefore, the major tensile stress bearing layer in the axial direction is the S_2 layer. The major restraints against swelling are the S_1 and P layers.

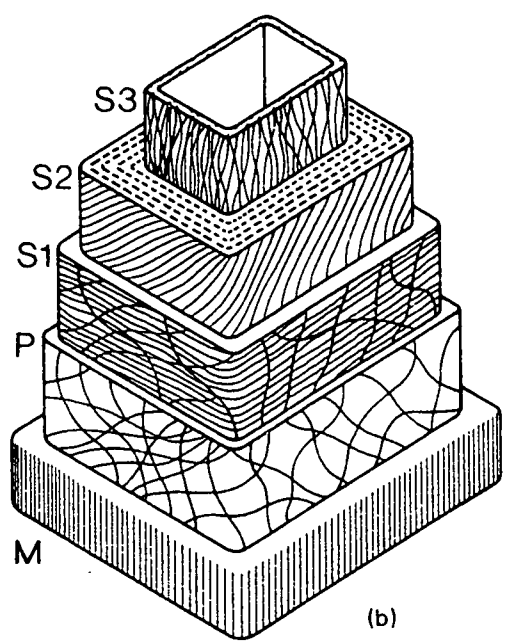


Figure 2. Cell-wall architecture: M = middle lamella, P = primary wall, S_1 = transition lamella, S_2 = main layer of secondary wall, S_3 = tertiary wall, or tertiary lamella of secondary wall.

The most important point about the structure of the fibers is that none of these walls are solid blocks with radially directed fibrils, microfibrils, or elementary fibrils (8). They consist of layers joined together by interlamellar bonds, water, lignin, and hemicelluloses. Under mechanical stress, the layers in the fiber wall can delaminate. When this delamination occurs in the presence of water, the fibers imbibe the water in the separated regions and swell. The water acts as a lubricant between the lamellae, allowing the previously bonded surfaces to slide past one another. The result is an increase in fiber flexibility (decrease in bending stiffness), giving the fiber properties desirable for papermaking (9).

Conventional refining processes have many effects on fibers. In this review, Ebeling's (10) summary is used to categorize the effects as follows:

- Internal structural changes - internal fibrillation; caused by breaking intrafiber hydrogen bonds and replacing the bonds with water molecules.
- External structural changes - external fibrillation, which is defined as pulling fibrils out of the outer walls of the fiber and primary wall removal; fines formation, due to the removal of parts of the outer walls and breaking off of the fibrils.
- Fiber shortening or cutting.

INTERNAL STRUCTURAL CHANGES

The forces of any applied action, whether classified as stressing, pressing, bending, flexing, curling, bruising, kneading, rubbing, twisting, crushing, etc., may be absorbed by the fiber and cause breakage of internal bonds. The bonds in question are between cellulosic fibrils, between fibrils and hemicellulose, between cellulose and lignin, and between hemicellulose and lignin. Mechanical action on the

fiber of such intensity that bonds will be broken will usually result in swelling. Swelling of the fiber takes place mainly in the amorphous hydrophilic hemicellulosic interfibrillar material (11). However, partial crystallization and hydrogen bonds will limit swelling, as will the presence of lignin (8). The primary wall and the S_1 layer of the secondary wall will also restrain the swelling during the earliest stages of refining due to their hydrophobic nature and the high fibril angle of the S_1 layer (8,11).

MacIntosh (12), Page and De Grace (13), Kibblewhite (14-16), and Stone and Scallan (17) add to the theory of bond cleavage by observing delamination of the wet fiber walls into many lamella upon refining. As mentioned previously, desirable papermaking properties result from these internal structural changes.

EXTERNAL STRUCTURAL CHANGES

Removal of the outer parts of the cell wall will expose the S_2 layer. By removing these regions of the fiber, which act as swelling restraints, the fiber becomes more flexible. Steenberg (18) stated that fiber flexibility, both wet and dry, is the second most important item (after fiber length) in strength development. Giertz (19) reported that, at least very early in the beating process, the fraction of fiber surface free from the primary wall can be directly correlated with tensile strength. Therefore, as more hydrophilic hemicelluloses in the S_2 layer are brought to the surface, they replace the greater number of hydrophobic lignin molecules in the P and S_1 layers, and promote interfiber bonding.

Another reason for causing external surface changes is the amount of surface area that is generated for fiber bonding. Throughout his book, Clark (20) emphasizes the importance of external fibrils as the main entity in interfiber bonding. There has never been much denial that the larger fibrils developed from a well-beaten pulp

will enhance the cohesiveness of the fibers in a wet sheet (21). Many researchers, however, (22-24) do not put as much importance on external fibrillation as Clark.

As a consequence of most refining actions, fines are generated. In this thesis, fines are defined by the method by which they are made. They are generated by cutting fibers, detachment of large parts of the lamellar structure of the fiber, and breaking off of external fibrils. Many of the reports center around the influence of fines on flow characteristics of the fiber suspensions (25-26), and the influence of fines on paper properties (27-30). The presence of fines is detrimental to drainage characteristics of the pulp because of the additional surface area introduced with the suspension. The high water holding capacity of the fines also impedes drainage. Some disagreement exists among authors as to the influence of fines on sheet properties; therefore, this topic is treated in detail in the Results and Discussion section.

FIBER SHORTENING

If the strain on a fiber is great enough, it will break or be bent and deformed. The mechanism of fiber shortening has been studied, with Cottral (31) stating that two of the mechanical effects of refining cause transverse subdivision of the fibers. Accordingly, fibers may be cut by direct shearing forces of the passing refiner bars, or they may fail when pulled from a network of other fibers. Steenberg's theories on cooperative process (32) tend to favor the idea of stress failure due to network forces rather than by direct shear of the passing bars.

Regardless of the mechanism, current refining processes do shorten fibers. For this thesis, it is assumed that reduction in the length of the fibers is not desired. It seems counterproductive to reduce the length of fibers in the refiner

when a shorter material could be substituted, or a small length fraction could be blended in to the papermaking stock to obtain the desirable qualities.

EFFECT OF REFINING ON PAPER PROPERTIES

As stated in the Introduction, the goal of refining in this study is to increase the strength of the sheet, particularly tensile strength. The tensile strength of paper has been studied extensively (33-35) and Page's (36) theory was chosen to describe the present system. Page gives a theoretical consideration to the mechanism of tensile failure of paper in terms of fiber strength and bond strength. His analysis led to an equation for the tensile strength of paper in terms of seven basic fiber and paper properties:

$$\frac{1}{T} = \frac{9}{8Z} + \frac{12\ A\rho g}{bPL\ (RBA)} \qquad (1)$$

where T = the finite-span tensile strength of paper (expressed as breaking length)

Z = the zero-span tensile strength expressed as breaking length (a measure of fiber strength)

A = the mean fiber cross sectional area

ρ = the density of the fibrous material

g = the gravitational constant

b = the shear strength per unit area of the fiber-fiber bonds

P = the perimeter of the average fiber cross section

L = the mean fiber length

RBA = the fraction of fiber surface that is bonded in the sheet.

The equation was tested experimentally, and this verified the linear relationship between tensile and fiber length and tensile and RBA (36).

The significance of Page's theory to this thesis may be stated as follows: To increase tensile strength through refining, the refining actions should 1) preserve fiber length, 2) preserve zero-span breaking length, and 3) provide either a high bond shear strength per unit area or a large RBA. The other parameters in the equation (which are directly related to fiber dimensions), A, ρ, P, are assumed to have a smaller influence on the tensile strength. This is because, as refining progresses, A, ρ, P should not change drastically if the same pulp is used from experiment to experiment.

Choosing refining conditions to preserve fiber length and fiber strength are not as difficult as producing high bond shear strength or high RBA. In fact, using many conventional refiners at mild operating conditions could probably satisfy the first two criteria. Bond strength and RBA involve additional consideration. To reiterate a point made earlier about the three effects of refining, fiber shortening is considered an undesirable action and in developing sheet strength should be avoided.

In discussing the bond shear strength and how to influence it, Van den Akker (37) states that the architecture of the fiber-fiber bond is important. There are basically three classes of fiber-fiber bonds in a sheet of paper; $S_1 - S_1$, $S_1 - S_2$, and $S_2 - S_2$. It follows that there will at least be three levels of what he calls "intrinsic bonding strength". Therefore, a method to vary the bond shear strength in terms of the tensile equation would be to produce fibers with uniform amounts of S_1 and S_2 present on the fiber surfaces, and then form them into sheets. A drawback of using bond shear strength as a key parameter is that there is not a scientifically valid method for measuring the property. On the other hand, the reliability of methods for determining RBA is better than that of bonding strength.

The RBA of a sheet of paper has been considered in principle as important to all the mechanical characteristics of paper (38). The optical technique for measuring

RBA originated in the work of Parsons (39). It is favored by a number of investigators because it is directly correlated to unbonded fiber area (40), and the test may be performed quickly without destroying the sheet.

In a simplified sense, paper consists of two components: pulp fibers (approximately 1-3 mm in length and 5-10 by 20-50 μm in thickness), and air. Visible light which strikes the sheet will reflect and refract light at all fiber-air interfaces. Interfaces are considered both external and internal to the fiber wall and include those of the lumen (if the fiber is not totally collapsed). Interfaces between bonded fibers are not included since these are optically continuous. Light scattering measurements are incapable of detecting surface separations much less than half the wave length of visible light; therefore, fiber surfaces which are separated by less than 200-300 nm are then registered as being in contact (40). This explains why, as the sheet density increases with refining, more surfaces come into closer contact with each other, and the light scattering coefficient decreases.

RBA is calculated from the light scattering coefficient of the unbonded sheet, S_u, less the scattering coefficient of the bonded sheet at some defined strength level, S_b.

$$RBA_b = \frac{S_u - S_b}{S_u}, = 1 - S_b/S_u \qquad (2)$$

A linear relationship is often found experimentally between scattering coefficient and breaking length (39). This observation is utilized to characterize the fibers in the unbonded state, S_u, by extrapolation of experimental data to zero breaking length (40).

Another objective of refining is to increase the RBA between fibers by making the fibers more flexible. The increased fiber flexibility will allow the fibers to deform under the surface tension forces during drying, and create a dense, well-bonded sheet.

The aim of refining fibers to increase tensile strength is, therefore, to isolate a mechanical action that will preserve the fiber length and fiber strength of the pulp. Also, the action should either increase the flexibility of the fibers to provide increased RBA, or the action should improve the intrinsic bond strength.

REFINING EQUIPMENT

The literature review for this section focused on identifying an apparatus or method whereby the desirable effects of refining (preservation of fiber length and fiber strength, increase in RBA, or controlled removal of fiber wall P and S_1 to improve bond strength) could be achieved. The method by which raw pulp is transformed into refined stock is essentially one of attrition. Refiners provide the opportunity for fibers, in a water slurry, to be interposed between two edged bars in close proximity. One bar moves with respect to the other such that the fibers are under the influence of both bars simultaneously. As a result, the physical form of the fiber is altered.

The character and extent of the alteration depend on many factors, such as bar (or blade) material, sharpness of bar edge, roughness of bar surface, width of bar face, clearance between bar faces, normal force exerted by the bars on each other (separated by a film of fibers), angular orientation of bars, space between bars, relative velocity of bars, and consistency of stock (41).

Besides the fact that there are many variables associated with the operation of one refiner, Clark (20) has made an extensive review of the various machines used to refine pulp, both in the lab and in the mill. The end result is that none of the refiners that were reviewed isolate the mode of operation to achieve the refining effects desired for this thesis. Therefore, the decision was made to design and build such an apparatus (see Appendix I).

In his review of the current literature, Ebeling (10) proposed a series of events which would most likely refine fibers in a manner more efficiently than conventional refiners. These events (summarized below) provide a useful framework for explaining the design of the apparatus for this thesis.

First of all, in order to control the refining action, the refiner probably needs several types of refining zones. Each one would be tailored to produce one or two of the primary refining effects. Secondly, to increase efficiency, the refining zone should be designed so that all fibers entering it have a high probability of treatment. Thirdly, the treatment zone should be decreased in volume. This would reduce losses due to turbulence and transporting fibers around inside the housing.

As mentioned earlier, fiber shortening was not one of the desirable refining effects for improving sheet tensile strength, leaving consideration of external fibrillation and internal fibrillation. External fibrillation may be caused not only by the direct mechanical action in a refiner, but also by other equipment, such as simple agitation or ultrasonic radiation (42). The ultrasonic techniques are very energy intensive, and to keep energy consumption to a minimum, this method was disregarded. Higgins (43) completed a study evaluating the effect of abrasive wear on fibers. His findings showed that large amounts of external fibrillations could be achieved by treating pulp fibers between two rotating parallel disks. By varying

the surface roughness of the disks, different levels of external fibrillation could be obtained. However, the external changes in the fiber were achieved at the expense of reductions in fiber length. This information, coupled with the findings that external fibrillation is accompanied by losses in fiber strength (44), caused the attention of the equipment design to focus on internal fibrillation. Higgins' apparatus will be used to promote external fibrillation, and is described in detail in Appendix II. Therefore, the primary refining effect which will be focused on in this thesis is internal fibrillation. Actual development of the apparatus is found in the Refiner Model section of this thesis.

EXPERIMENTAL PROGRAM

OBJECTIVES

The general objective of this thesis is to understand the refining process and its influence on fiber and paper properties. The approach was divided into three specific objectives:

1. Isolate a refining action that would produce internal fibrillation in pulp fibers.
2. Determine how internal fibrillation influences paper properties.
3. Establish the relative importance of internal fibrillation, external fibrillation, and fines.

REFINER MODEL

A model of the process was the first step in designing the refiner for this thesis. Physical components of the system (the fibers, the working surfaces, and water) were the only elements included in the model. Within this model there are generally two ways in which fibers can absorb energy in quantities large enough to make appreciable changes in their physical structure. The first is from their direct contact with the active surfaces of the machine (refiner tackle, refiner walls) and the second is from direct contact with other fibers. Nordman et al. (45) experimentally show that hydraulic forces, alone, have negligible refining effects on pulp fibers.

To simplify the refining model, consideration was given to a single fiber as it travels through a refining machine. Basically, the fiber is suspended in some medium (which can be a fiber network or water) and is subjected to a transfer of

energy from an external source. One possible sequence of events may be: moving fiber, immobilized fiber, energy transfer, moving fiber. By viewing the operation on the fiber scale, a simple refining system would be a) to immobilize the fiber, b) to apply the action necessary to cause internal fibrillation, and c) to release the fiber, allowing it to move on to the next treatment. This fiber-by-fiber procedure is impractical when large numbers of fibers must be treated to provide a single handsheet. The basic concept of the idea could be scaled up, however, to allow treatment of a sufficient number of fibers for handsheet testing purposes.

Variables that have the greatest effect on the fiber transformation were expected to be the intensity of the individual treatment (impulse) and the number of times the fibers are treated (46). Control of these two variables should be straightforward because they are functions of mechanical load and time.

Steenberg (47) found that at high speeds of operation, immobilizing the fibers may not be necessary in order to achieve energy transfer, which would eliminate one step in the model. Banks (48), on the other hand, found that power consumption was related to the second power of the speed. Since dynamic interactions are difficult to control (i.e., in conventional refiners) and since one of the objectives of designing the apparatus was to gain control of the fiber treatment, dynamic forces were avoided as much as possible by performing the experiments at relatively low speeds.

The size of the apparatus was also an issue. Scaling up from a single fiber treatment to treatment of a fiber network provides enough fibers to evaluate in testing, but it decreases the homogeneous nature of the treatment. Banks (48) found that the power consumption was also directly related to the fifth power of the size of the refiner, indicating the smaller design is better. Therefore, the dimensions

of the apparatus were such that the working action was on a fiber size scale. Also, a large enough sample size was treated for pulp and paper testing.

One of the major energy losses associated with conventional refining is in pumping the large amount of water associated with the pulp at typical refining consistencies (2-6%) (49). Eliminating enough water to reduce pumping losses but providing enough to allow swelling of the fiber wall should prove to make energy transfer more effective. Therefore, processing of fibers was carried out at higher consistencies, in the 25-30% range.

The initial experimentation with the model evaluated methods to immobilize fibers for transport to the refining zone. Based on the reports of Smith (50), Stephansen (51), and Imset (52), the phenomenon of fiber stapling was evaluated as a method for fiber immobilization and transport. An initial series of flow stapling experiments (spraying fibers over a bar edge) showed that there were many complex interactions that were tangential to the goal of the apparatus design. Nonuniformity of the stapled fibers, difficulty in fiber handling, and low consistencies were the major drawbacks. A second method, fibers in the form of a thin wet handsheet, was selected for use in the design. The wet sheets provided an easily controlled method for immobilizing and uniformly transporting the fibers to the refining zone.

It has been shown that abrading the fibers in a shear field produces external fibrillation and shortening (43). The other main stress on the fiber inside a conventional refiner is compression between the working surfaces. Therefore, compression cycles on the fibers with a minimum of shear were chosen as the action needed to achieve internal fibrillation.

A simple method to put compression work into a wet fiber network is to press it between two platens. Continuously repeating the cycle would not guarantee that a

uniform refining effect would be applied to the fibers. Due to nonuniformities in the thickness of the sheet, localized stress buildup would occur, with some fibers receiving harsh treatment and others receiving none at all. This is analogous to the problems encountered in calendering paper (53). High spots in the sheet receive more work, and the fibers can actually be crushed, while low spots absorb very little work. However, by using the idea of rolling contact and machining grooves into the surface of one of the rolls, a more uniform stress distribution should result. By making the land areas of the roll approximately the dimensions of a fiber, more effective treatment should result.

A system containing the previously described components was evaluated. It consisted of a stainless steel platen with a polished surface and a brass cylinder with grooves machined into its circumference. A lightweight handsheet (unpressed, undried) was placed on the platen, and the grooved roll was loaded and rolled over the fibers a specified number of times. The gross amount of energy used in refining the fibers could also be measured using this system, and the friction losses associated with the operation could also be obtained.

In summary, a prototype "refiner" that treated pulp in the desired manner was designed. The basic action was compression-decompression of a wet fiber mat. The action of the prototype apparatus did produce fibers with internal fibrillation. The apparatus was scaled up to process larger fiber samples. Details of the components and the operational ranges of the refiner are presented in Appendix I and Appendix III, respectively.

OUTLINE OF EXPERIMENTS

Bleached spruce sulfite pulp was obtained from Mr. Jan-Erik Levlin of Keskuslaboratorio Centrallaboratorium (KCL) in Helsinki, Finland. This particular pulp was chosen because a sample was available from the same pulp used in their refining studies with the Escher-Wyss conical refiner. Complete refining data was supplied with the pulp. Of special interest were the net refining energy input measurements that were supplied with the pulp. Therefore, by using this pulp, the variable of pulp source was eliminated when comparisons between refiners were made.

The pulp was received in dry lap form. Prior to use, it was soaked overnight and defibered for 50 counts in a British disintegrator. The pulp was formed into handsheets on a Formette Dynamique handsheet machine (54,55). The operating conditions of the Formette were adjusted to obtain a 30 g/sq m (dry basis) handsheet with approximately 5 to 1 MD to CD fiber orientation. The nozzle used was H 1/8 U2504, and the wire speed was 1100 m/min. The wire, obtained from Appleton Wire, Appleton, Wisconsin, was manufactured for use in a lightweight tissue application (84 x 68 mesh). Once formed, the laboratory sheet was couched onto a wet blotter (at 30% consistency), the wire was removed, and another wet blotter was added to make a wet blotter/wet sheet/wet blotter sandwich.

A schematic of the roll refiner is shown in Fig. 3. The basic components of the apparatus are the vertical support columns, A; the horizontal support beam, B; the refining roll (60 mm diameter), C; the support roll (160 mm diameter), D; the oscillator motor and eccentric, E; the loading mechanism, F; the support roll bearing, G; and the drive mechanism, H.

The refining roll has grooves machined into its surface to break up the line contact between the two rolls into discrete segments. Subdividing the refining roll

and oscillating it along its axis approximately the distance of one groove width (1.2 mm) allows new configurations of fibers to be refined with each revolution of the support roll. The amount of slippage between the two rolls with fibers in the nip was less than 0.1%. With this small amount of macroscopic shear in the nip, the major stress applied to the fibers is a repeated compression-decompression loading cycle.

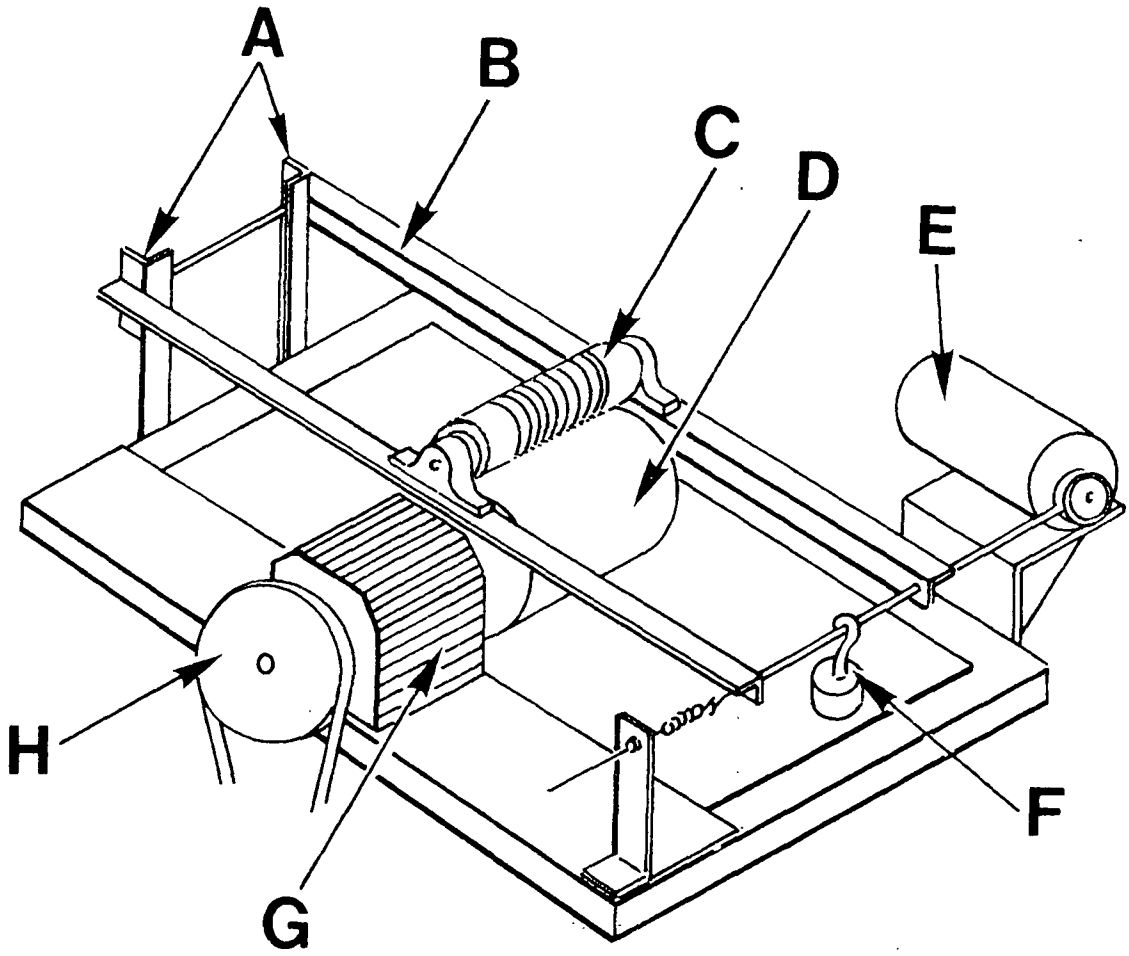


Figure 3. Schematic of roll refiner.

The full size of a Formette sheet is 21 x 91 cm. To fit the roll refiner format, the sheets were trimmed to a size of 10 x 50 cm. The blotters were removed after trimming, and the sheets were placed in the roll refiner in two layers. This configuration gave a final basis weight of 60 g/sq m around the complete circumference of the support roll. The wet handsheets were refined at 28-30% consistency

to various energy input levels, governed by the number of compression loading cycles (125-4000 cycles). The normal load at the contact nip was 25 kg.

In separate experiments, pulp was abraded between rotating parallel disks to promote external fibrillation. A schematic of this abrasion refiner developed by Higgins is shown in Fig. 4 (43). The stainless steel rotor was driven by a 1/2-hp variable speed motor. Rotor speeds of up to 1100 rpm were possible. The stator, made from Plexiglas to aid flow visualization, was threaded into a steel housing which was loaded against the rotor through a Teflon seal. The internal diameter of the chamber so formed was 102 mm. The threads in the housing allowed the clearance between the rotor and stator to be adjusted to any setting from 10 mm to apparent zero. The roughness of the disk surfaces could be altered by attaching silicon carbide sandpaper with various grit sizes. All results reported here used a 120 grit surface roughness.

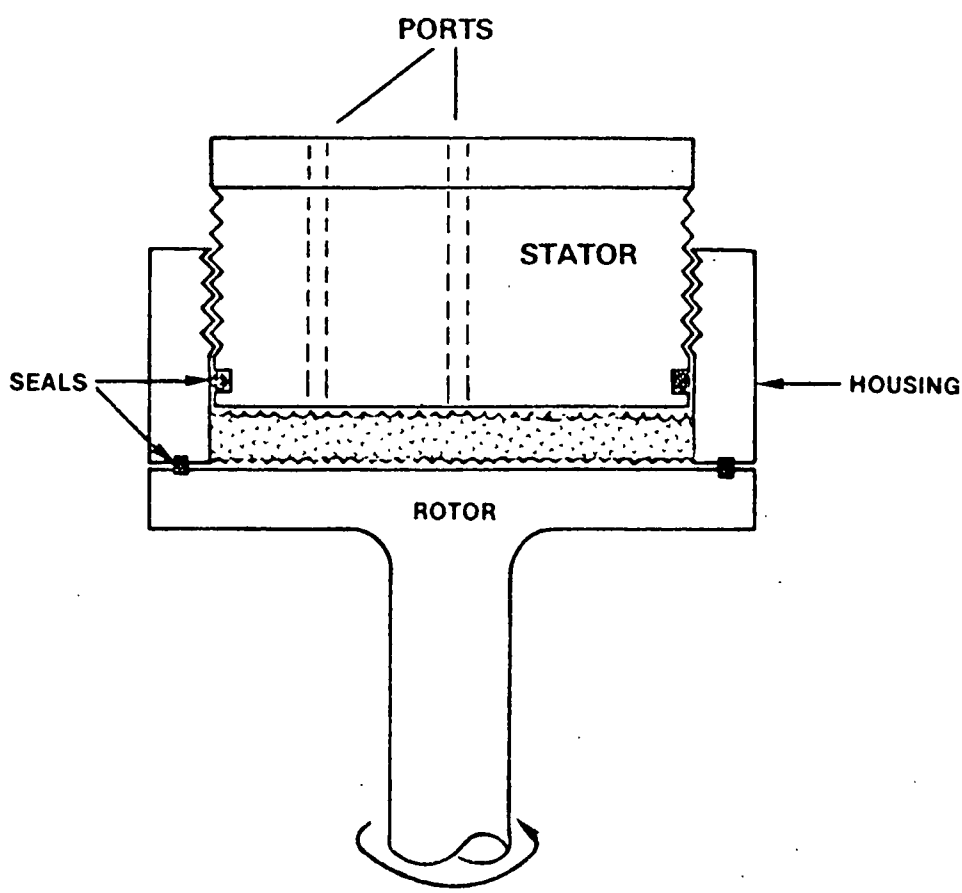


Figure 4. Schematic of the abrasion refiner.

One gram samples of unrefined bleached spruce sulfite pulp were treated at 1000 rpm for five minutes in the abrasion refiner at a gap of 4 mm. A second set of samples was first refined for 500 cycles in the roll refiner and then treated in the abrasion refiner at the same operating condition as above. The consistency during abrasion treatment was approximately 3%.

Fine material was generated by refining the bleached spruce sulfite pulp for 21 hours in a Valley beater. The quality of the fines was evaluated with a light microscope to ensure that no fiber fragments were present. Freeness of the final product was 720 mL (all material passed through the screen of the tester). The fines generated in this manner were added to an unrefined pulp sample and to a sample that had been roll refined for 500 cycles. As a basis of comparison, the sulfite pulp was treated in the Valley beater, following TAPPI Standard T 200.

After each refining experiment, the fibers were formed into standard handsheets using TAPPI standard conditions. Small samples of wet fibers were collected and the following tests were made: fiber length distribution, scanning and transmission electron microscopy (the SEM samples were critical point dried to minimize fiber collapse), polarization microscopy, Canadian Standard Freeness, and a centrifugal water retention test.

All handsheet testing was performed in a controlled environment of 23°C and 50% relative humidity. The following paper properties were measured: sheet density, specific light scattering coefficient, Gurley air resistance, breaking length, tear resistance, and zero-span breaking length.

Full details of the experiments and conditions employed are presented in Appendixes IV and V.

RESULTS AND DISCUSSION

The results and discussion section of the experimental work in this thesis is divided into three sections. The first section establishes the effects of the roll refiner on fiber properties, with an emphasis on internal fibrillation. The second section discusses the effects of the roll refiner action on paper properties. The results are compared to results obtained with an Escher-Wyss refiner and a Valley beater. The third section compares the relative importance (in terms of paper properties) of three refining effects: internal fibrillation, external fibrillation, and fines generation.

EFFECT OF ROLL REFINER ON FIBER PROPERTIES

One objective of this section was to establish that internal fibrillation was the primary effect caused by the compressive action of the roll refiner. Initially, the degree of fiber shortening and external fibrillation (both undesirable in the context of this section) was determined.

The effect of roll refining on weighted average fiber length and the fiber length distribution of the pulp samples is shown in Fig. 5 and 6. In Fig. 5, the samples have been treated for 0, 125, 250, and 500 cycles in the roll refiner. As the level of treatment increased, the weighted average fiber length decreased from 2.3 mm to 2.2 mm, and only small changes in the fiber length distribution were observed. The effect of higher levels of roll refining is shown in Fig. 6. The fiber length distributions shift toward a greater amount of short length material, with a decrease in the weighted average fiber length of seventeen percent.

Figure 7 compares the effect of roll refining and Valley beating on fiber length measurements. The two graphs on the left and the two graphs on the right represent

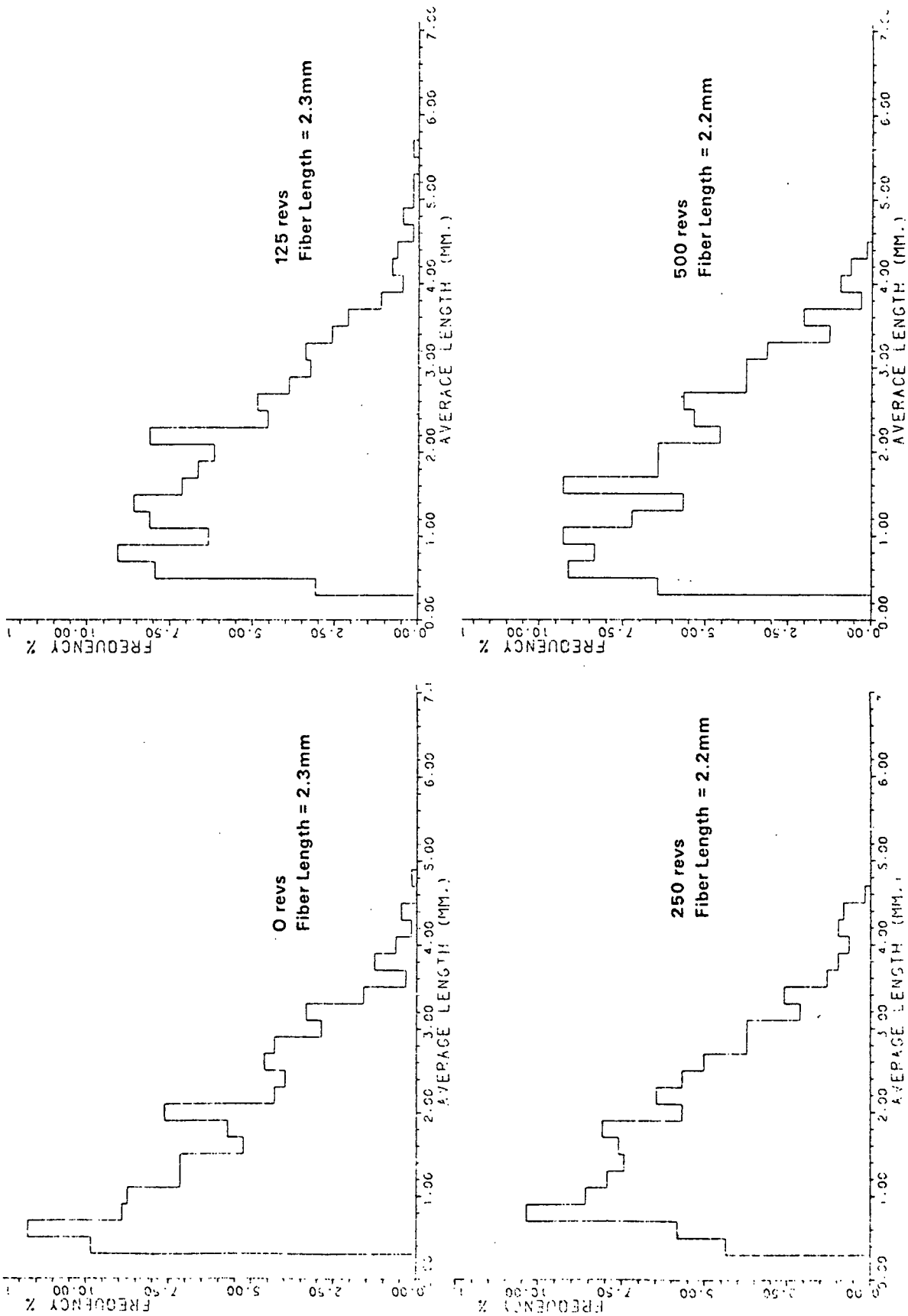


Figure 5. Fiber length distributions for roll refined pulps: fiber length distributions and weighted average fiber lengths for samples refined up to 500 revolutions.

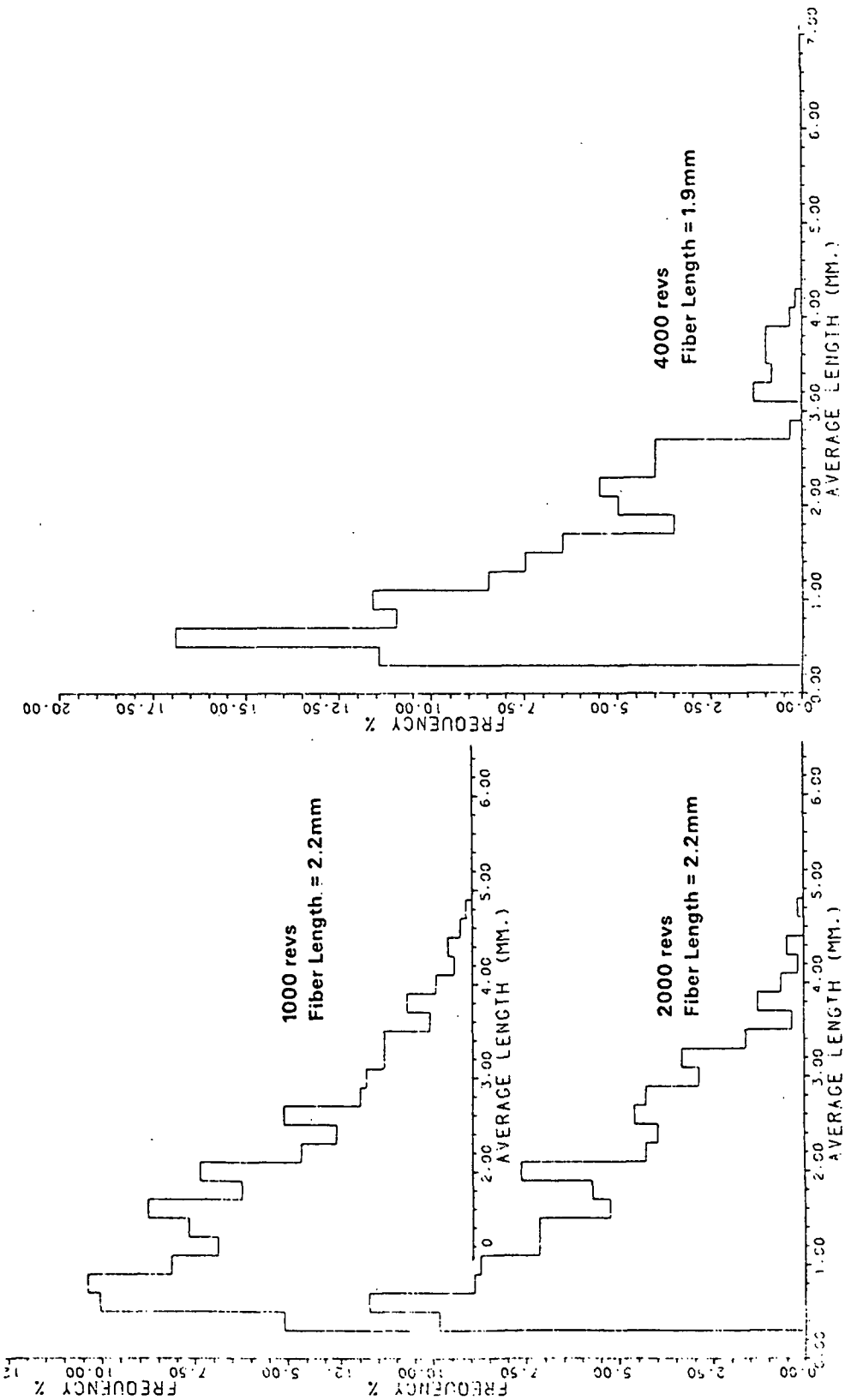


Figure 6. Fiber length distributions for roll refined pulps: fiber length distributions and weighted average fiber lengths for samples refined for 1000, 2000, and 4000 revolutions.

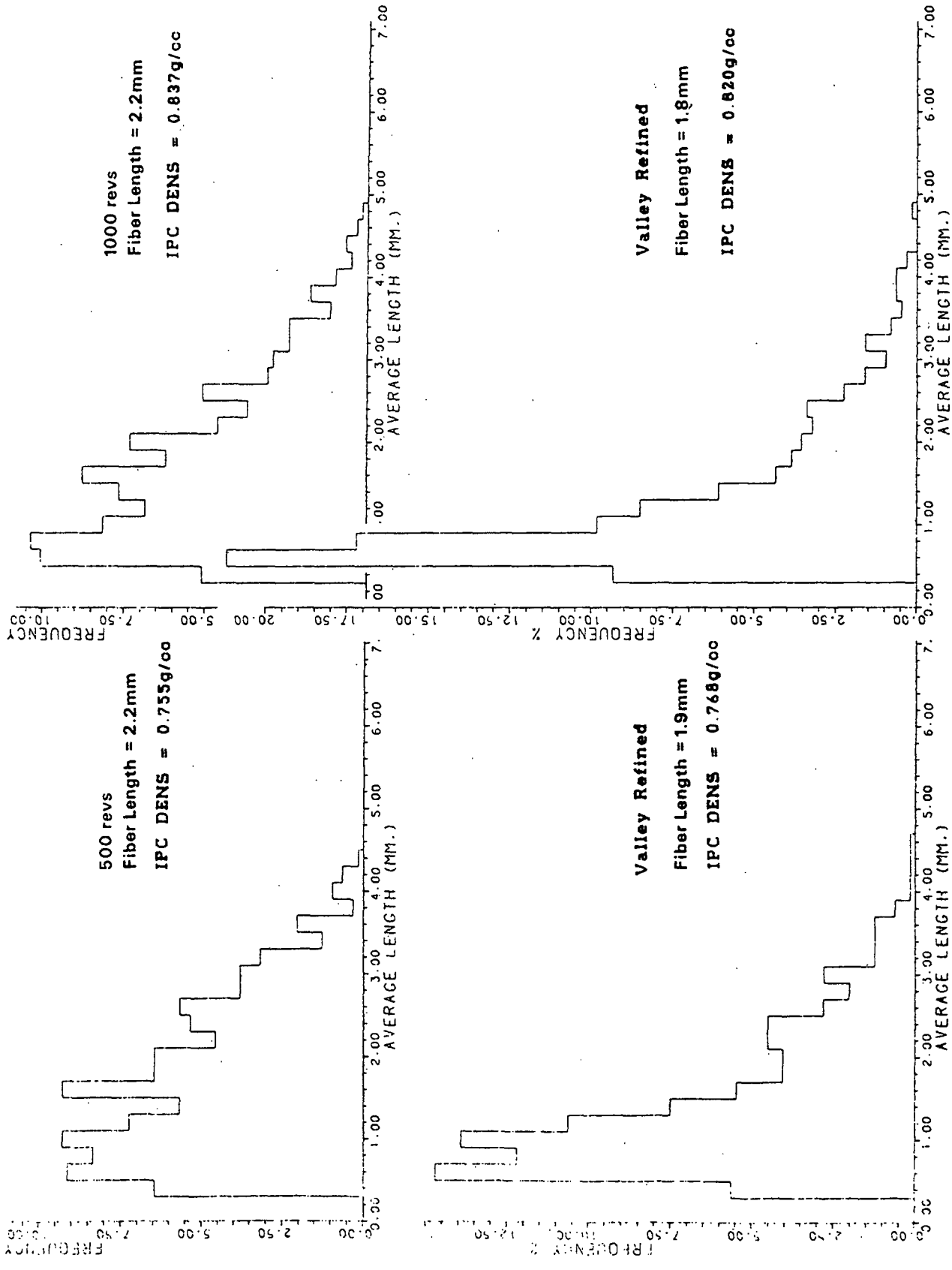


Figure 7. Fiber length distributions for roll and Valley refined pulps. Upper row: roll refined pulps. Lower row: Valley refined pulps.

fiber samples that give comparable sheet densities at a constant wet pressing level. Roll refined sample comparisons (top two graphs) show there is little change in either fiber length distribution or weighted average fiber length as refining increases in the range considered. For the Valley beaten pulp, the weighted average fiber length decreases and the fiber length distribution shifts to shorter lengths with increased refining time. This trend is expected for Valley beaten pulp due to the low consistency and, hence, the general shortening action of the beater.

The action of the roll refiner, therefore, does not shorten the fibers. As a result, sheets from roll refined pulp contain longer fibers than sheets from Valley beaten pulp at the same sheet density level. Moreover, to better preserve fiber length, refining experiments should be performed for less than 1000 cycles in the roll refiner.

The amount of external surface area developed in the pulp samples was monitored with scanning electron microscopy. Scanning electron micrographs (SEM's) of roll refined and Valley beaten fibers are found in Fig. 8. The SEM samples were prepared using critical point drying, which minimizes fiber and fibril collapse. The arrangement of the micrographs in Fig. 8 is the same as in Fig. 7. The micrographs show that, for a constant sheet density level, more external fibrillation is being generated in the Valley beater. Conversely, the roll refined fibers (top two micrographs) show only minor changes in the amount of external surface with refining. These observations are expected, because the simple compressive action of the roll refiner is different from the complex action occurring between the bars in the Valley beater.

Figure 9 is a plot of Canadian Standard Freeness (CSF) vs. refining time, which illustrates one of the major differences between roll refined and Valley beaten

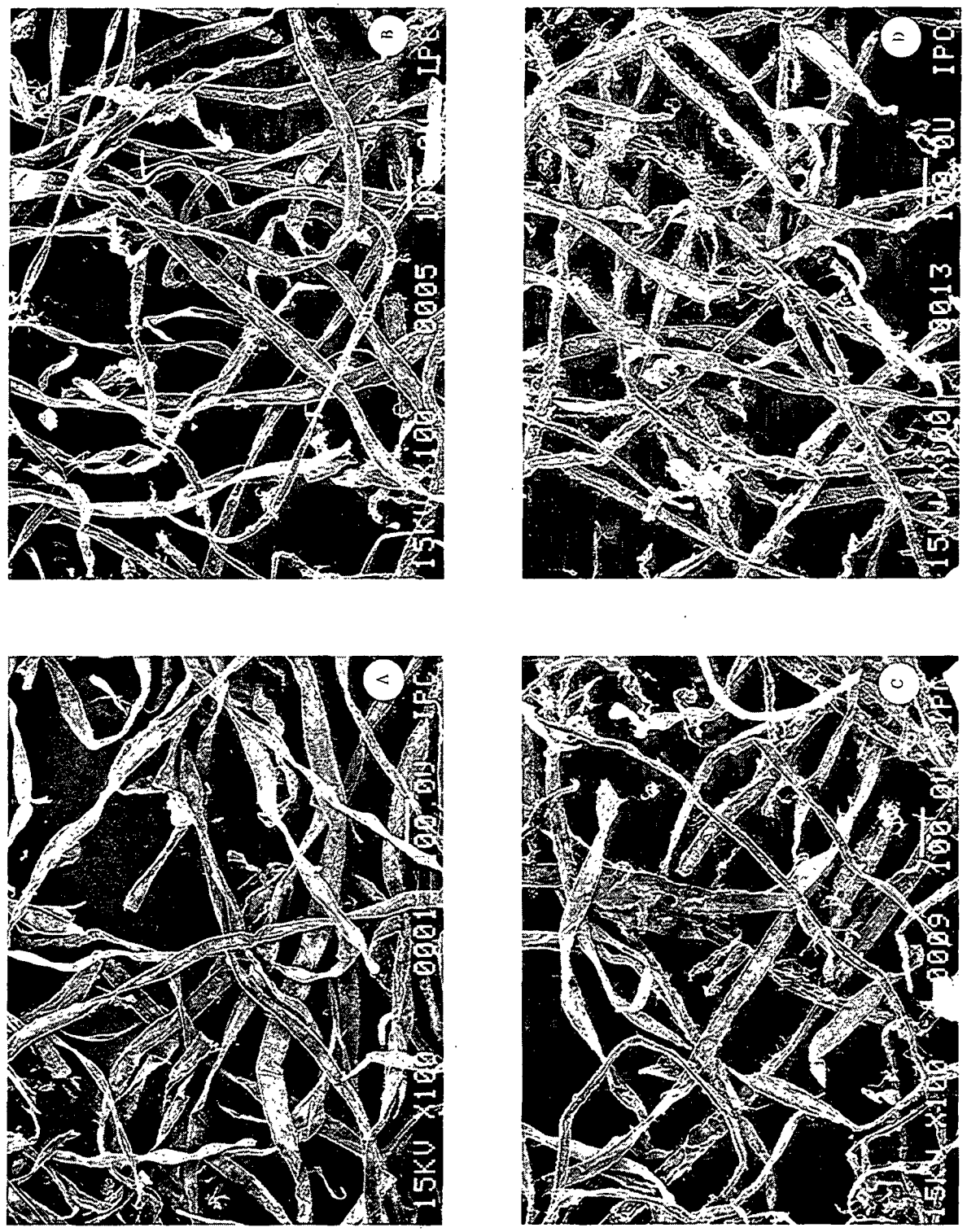


Figure 8. Scanning electron micrographs of the samples described in the previous figure.

samples. Although there remains speculation as to what the CSF test measures, CSF of the pulp does not change with roll refining. In other words, there is no change in drainage resistance. The Valley beaten pulp, however, exhibits a large change in drainage resistance (lower CSF) with refining because the Valley beater produces external fibrillation and fines. With negligible reduction in fiber length, and little external fibrillation caused by the roll refiner, it follows that the energy consumed in refining with the roll refiner is spent on internal structural changes.

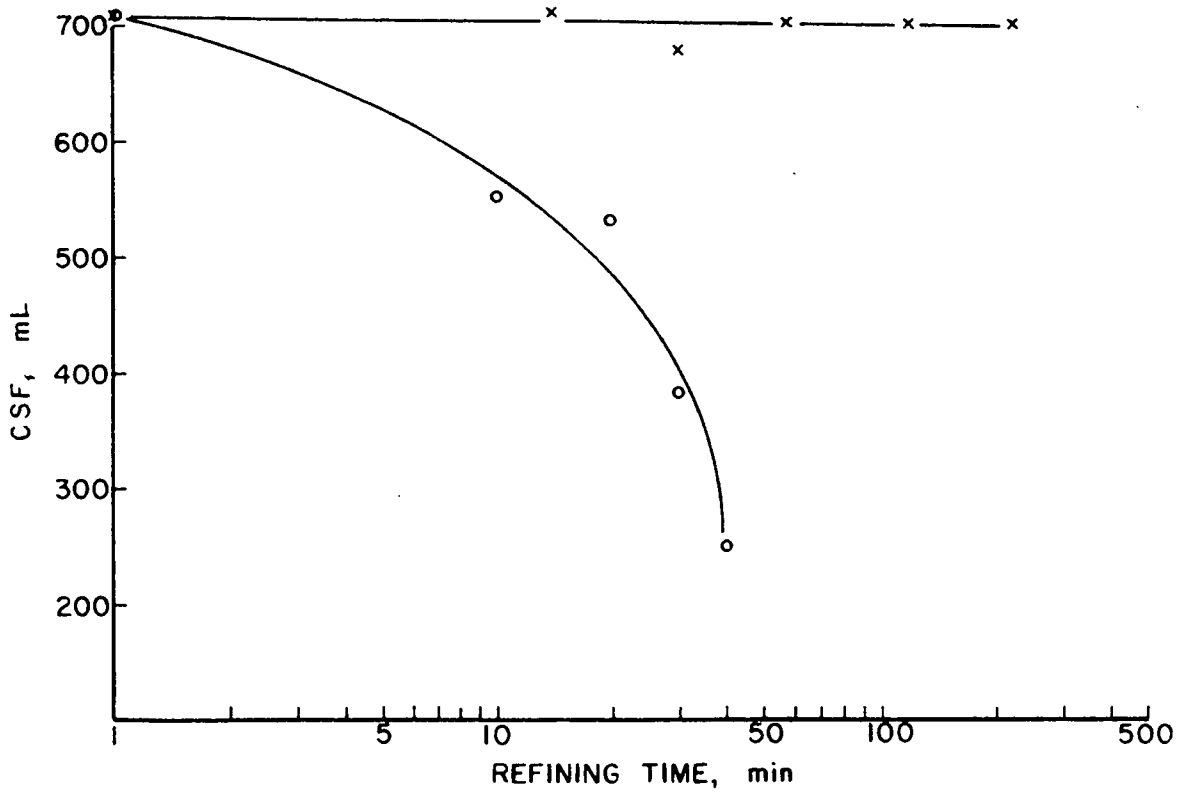
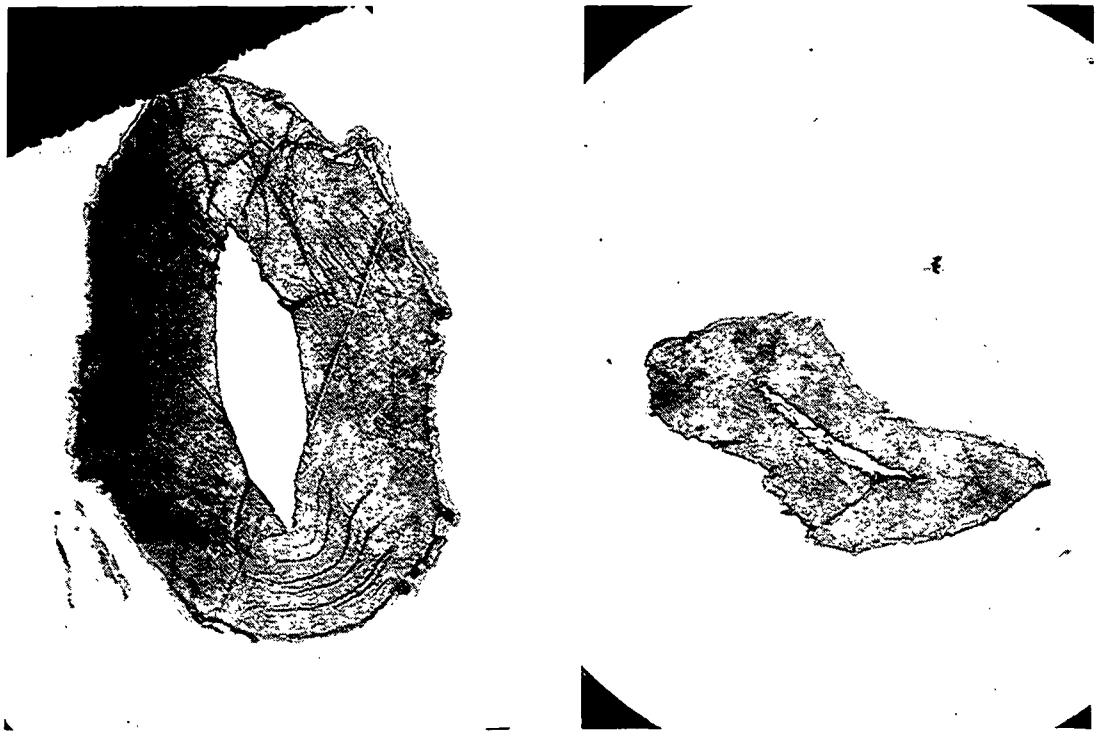


Figure 9. Canadian Standard Freeness vs. refining time.
x = roll refiner
o = Valley beater

Transmission electron micrographs (TEM's) of fiber cross sections show direct evidence that internal fibrillation results in the roll refiner. TEM's were made of an unrefined sample (Fig. 10), a roll refined sample (Fig. 11), and a Valley beaten sample (Fig. 12). All the samples were once-dried; consequently the unrefined



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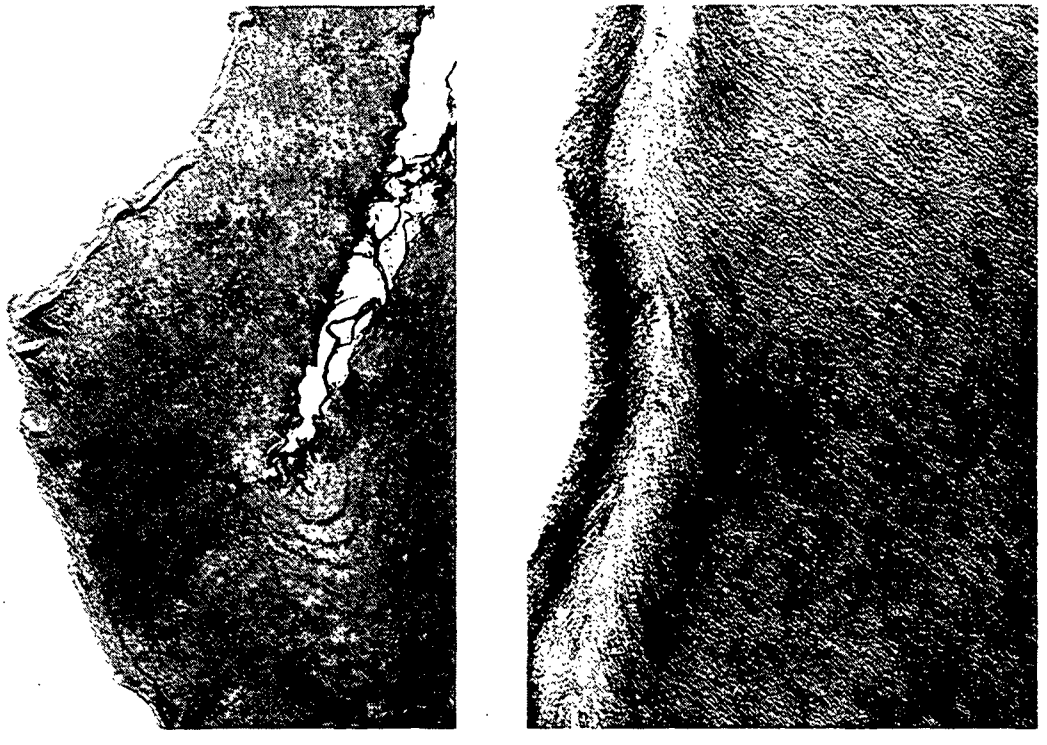

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Figure 10. TEM micrographs of fiber cross sections, bleached spruce sulfite pulp (unrefined).

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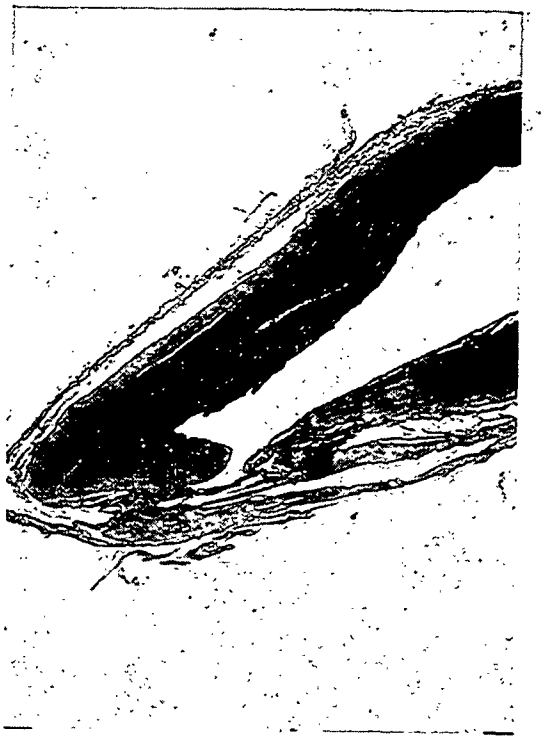

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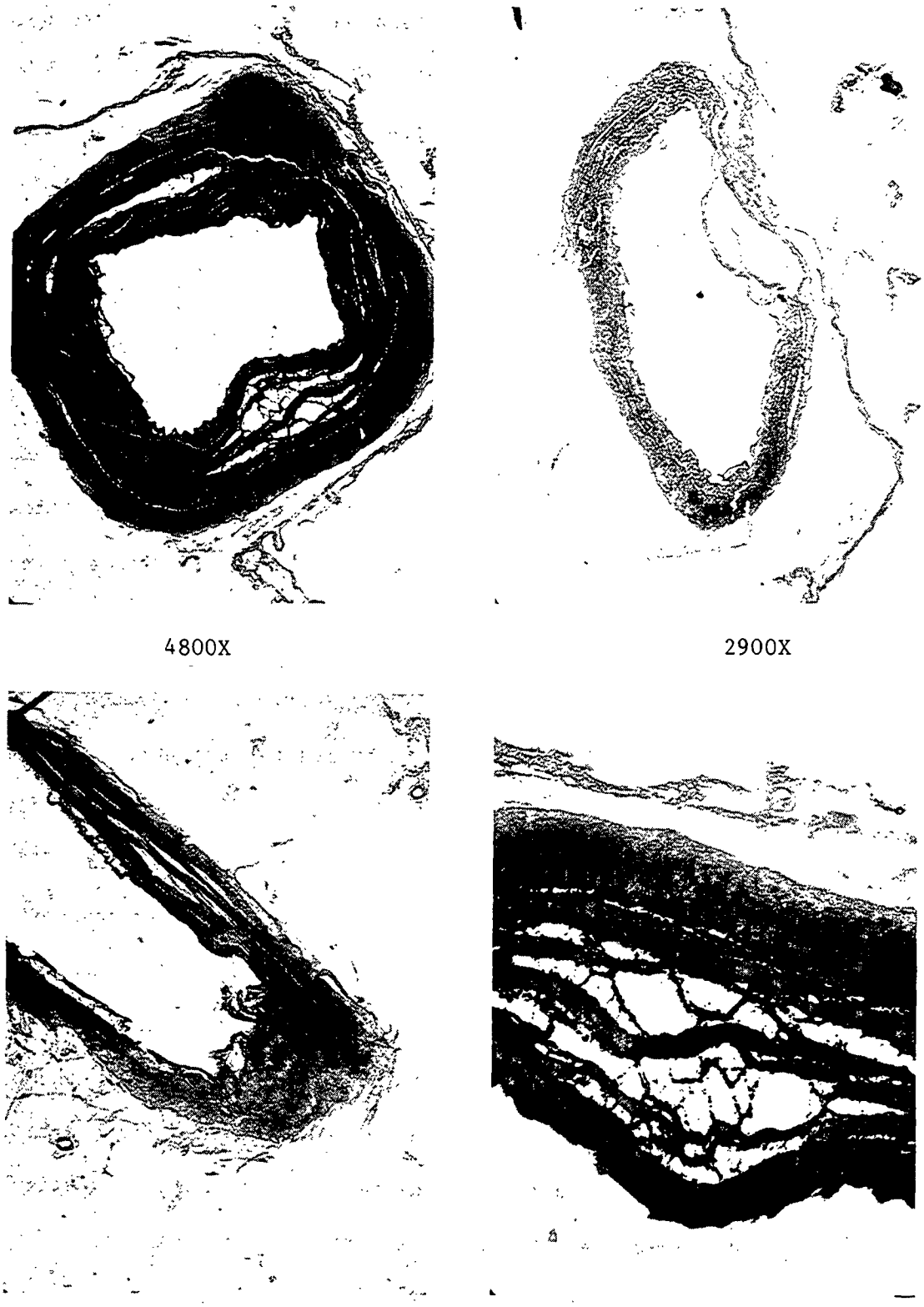
Figure 11. TEM micrographs of fiber cross sections, bleached spruce sulfite pulp, roll refined 1000 revolutions.



4800X 2900X

7200X 14,000X

Figure 12. TEM micrographs of fiber cross sections, bleached spruce sulfite pulp, Valley beaten 30 minutes.

sample does exhibit some regions of internal delamination. However, the outer layers are distinct and still intact. The roll refined fibers (Fig. 11) have more delaminations in the cell wall than the unrefined sample. The outer layers of the cell wall are detached, but their continuity has not been disrupted. The Valley beaten fibers (Fig. 12) show more internal and external disruptions in the cell wall than either the unrefined or roll refined fibers. The delaminations in the cell wall of both the roll refined fibers and the Valley beaten fibers are the result of the compressive action they receive in the working zone of each refiner. However, the Valley beaten fibers are subjected to various other complex actions which disrupt both the internal and external regions of the cell wall. Often these actions are severe enough to cut the fibers, illustrated by the decrease in fiber length shown earlier.

Polarization microscopy was also used to observe internal structural changes in the fiber cell wall. The method is based on observing the components of the fiber cell wall (particularly the crystalline cellulosic regions) in plane polarized light. An approximate model of the fiber cell wall is that the cellulosic microfibrils are embedded in a matrix of noncellulosic polysaccharides and other substances possessing voids of submicroscopic dimensions (56). In a material that is anisotropic, the velocity of propagation of plane polarized light varies as it passes through the material (57). The effect of this mixture of substances is to retard the passage of light which results in a rotation of the plane of polarization. Such a material is called birefringent because it presents two different indices of refraction (corresponding to the respective different velocities of transmission).

In the polarizing microscope (PM), when the polarizer and the analyzer are crossed, plane polarized light is not transmitted and the field appears black. Under this condition, if a birefringent specimen is placed on the stage, the plane

of polarization will deviate according to the retardation introduced by the specimen, and the specimen will become visible against the dark background.

Figure 13 contains PM micrographs of samples that were viewed in the crossed condition. From top to bottom, the rows of micrographs are of an unrefined sample, a Valley beaten sample, and a roll refined sample. The micrographs in the right column are of fibers that were placed on a slide, dried, and then rewet before the photo was made. The micrographs in the left-hand column were not dried following refining. Each of the samples (when viewed from top to bottom) show different degrees of birefringence. Ranked in order of increasing birefringence, the unrefined sample is the lowest, Valley beaten is in the middle, and the roll refined sample is the highest.

The significance of birefringence of a fiber cell wall depends on the heterogeneity of the lamellae (56). The position of the specimen in the PM relative to the polarizing elements measures the degree of orientation (or order) of microfibrils in the cell wall. For the unrefined sample, we see there is a preferential orientation of the microfibrils because they birefringe at the prescribed PM setting; only a few fibers on a similar angle appear white. The fibers on other angles appear dark.

Following refining in the Valley beater, and even more so in the roll refiner, a change in the orientation of long axis of the molecules has occurred. The preferential orientation of the microfibrils found in unrefined fibers has been removed. This can occur by either a change in the crystallinity of the cellulose or a change in the orientation of the fibrils in the lamellae with respect to each other (56). X-ray analysis showed no significant difference in the degree of crystallinity between roll refined and Valley beaten fibers. Apparently, the major changes due to

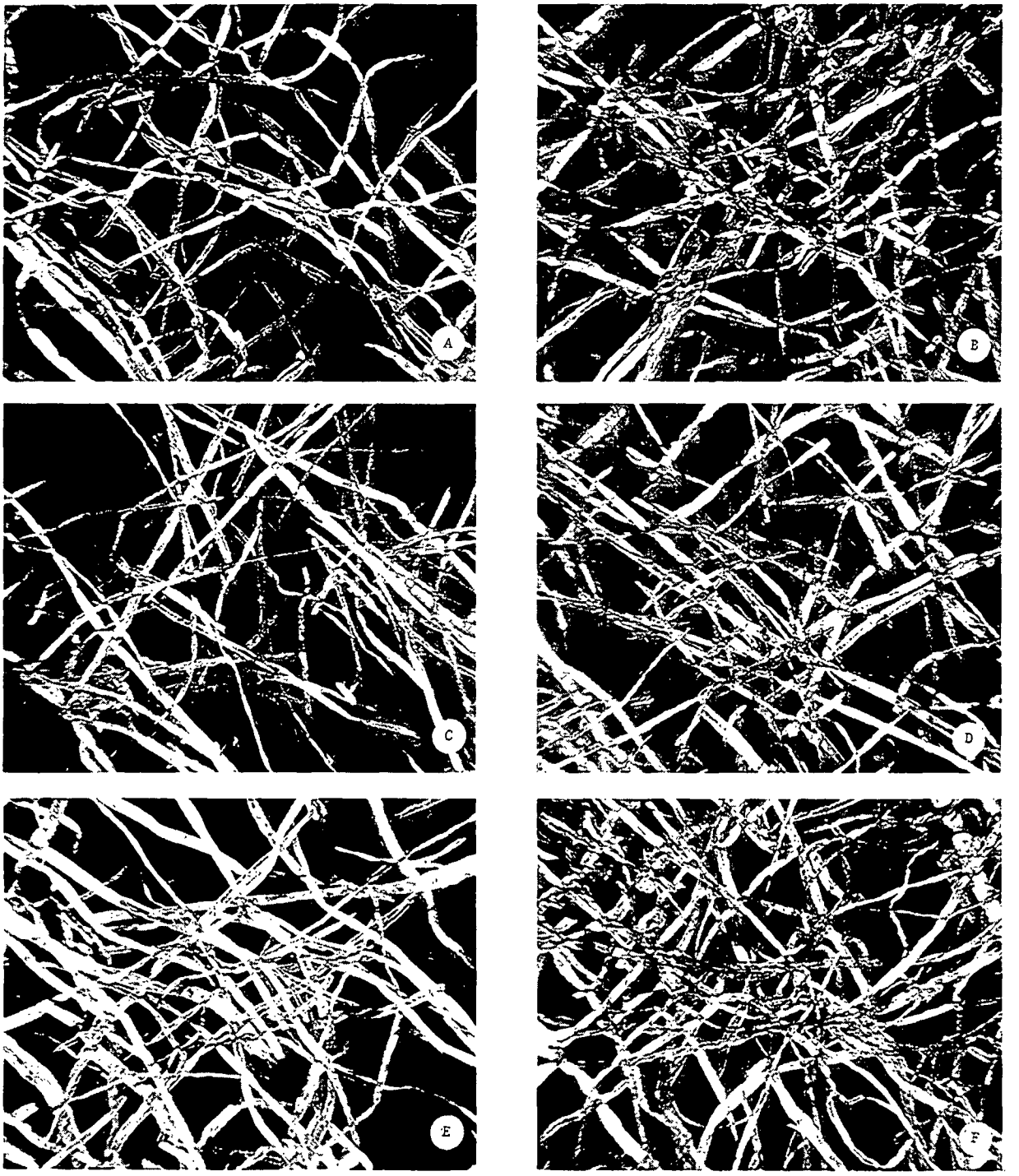


Figure 13. Polarized light micrographs (magnification 40X). A, unrefined fibers (wet); B, (dry); C, Valley beaten fibers, 40 minutes (wet); D, (dry); E, roll refined, 4000 revolutions (wet); F, (dry).

refining are occurring between lamella, reinforcing the observations from fiber cross section analysis.

Another interesting point is the effect of drying on birefringence (Fig. 13). Drying by any method which permits shrinkage has at least two undesirable effects on the birefringence (56). First, the act of shrinking is liable to change the orientation of the microfibrils, and therefore the birefringence. Secondly, the change in volume and increase in refractive index of the noncrystalline component (by withdrawal of water) changes the birefringence. In Fig. 13, the differences in birefringence due to drying (viewed from left to right) are not easily predicted. However, the relative amount of birefringence due to refining is the same for the three samples.

The evidence from the two microscopic techniques suggests that after refining, there are new voids within the fiber cell wall which can fill up with water. This was investigated using the water retention value method (WRV) (58-60), in which a pulp is centrifuged under standard conditions. The test measures the affinity of a pulp for water.

There are three categories whereby water is held in a cellulose mat: a) water outside the cell wall; b) water inside the cell wall held by capillary forces such as absorbed water in the lumen; and c) water inside the fibers chemically bonded to the walls (particularly hydrogen bonded). The latter is often termed bound water (61-63). Bound water cannot be removed by mechanical pressing (63), which leaves only the water in the other two categories amenable to removal by centrifugation.

An indication of the size of the changes occurring in the cell wall is a calculation of the pore size of the fibers. By assuming the pores created by refining

are cylindrical, and that the contact angle of water on the pore surface is 0, Young's Eq. (64), which measures the pressure needed to force water out of a capillary, is

$$\Delta P = \frac{2\gamma}{r} \qquad (3)$$

where ΔP = pressure difference

γ = surface tension of water, 72.5 dynes/cm, and

r = pore radius.

The level of centrifugal force used in these experiments was 1185 x g. According to the literature, this should be a high enough pressure drop to expel capillary water from the fiber (63). The radius of smallest pore from which capillary water can be centrifuged is then,

$$r = \frac{2 \times 72.5 \text{ dynes/cm}}{1185 \times 9.8 \times 10^2 \text{ cm/sec}^2 \times 1 \text{ g/cm}^2} = 1.25 \ \mu \qquad (4)$$

The water remaining in the fiber is therefore in pores with a diameter less than 3 μ or is bound to exposed or accessible cellulosic hydroxyl groups in the cell wall.

If the refining action increases the internal or external surface area of a pulp fiber, an increase in the WRV over the unrefined value should be expected. For the unrefined sample in this case, the WRV was 179 ± 31 grams water/100 grams pulp, and after roll refining for 500 cycles, the values increased to 240 ± 24 grams water/100 grams pulp. Since no external fibrillation or change in CSF was observed, the change in WRV due to roll refining is presumed to be due to an increase in the internal surface area of the fiber cell wall.

To summarize this section, the major effect of the roll refiner is to cause internal fibrillation of the fiber cell wall. The type of internal fibrillation is delamination of the lamellae within the cell wall. This is supported by the TEM's of fiber cross sections and by polarization microscopy of the cell wall. Furthermore, an increase in the water retention value indicates the presence of water in these newly formed internal surface regions. The action of the roll refiner does not appreciably shorten the fibers, or cause external fibrillation.

EFFECT OF ROLL REFINER ON PAPER PROPERTIES

The second objective of this thesis was to determine the influence different levels of roll refining had on paper property development.

BREAKING LENGTH

Breaking length is plotted against density in Fig. 14 for pulps treated in the roll refiner and Valley beater. Compared at a constant density level, sheets made from roll refined pulp have lower breaking lengths than sheets made from Valley beaten pulp. For roll refined pulps, the highest breaking length achieved is 5.8 km. This represents nearly a threefold increase in breaking length over the unrefined pulp. For the Valley beaten pulp, breaking length increases from 2 to 8 km. Reasons for the differences in breaking length between the two methods are explained below.

In his theory for the tensile strength of paper, Page (36) states that tensile strength is a function of seven variables which may be combined into two groups. The first group of variables is related to the strength of individual fibers, and the second is related to the nature of bonds holding the fibers in the network. These relationships can be expressed by the following equation:

$$\frac{1}{T} = \frac{1}{F} + \frac{1}{B} \tag{5}$$

where T = the tensile strength of the sheet

F = an index that describes only the resistance of the fibers to breakage, and

B = an index that describes only the resistance of bonds to breakage.

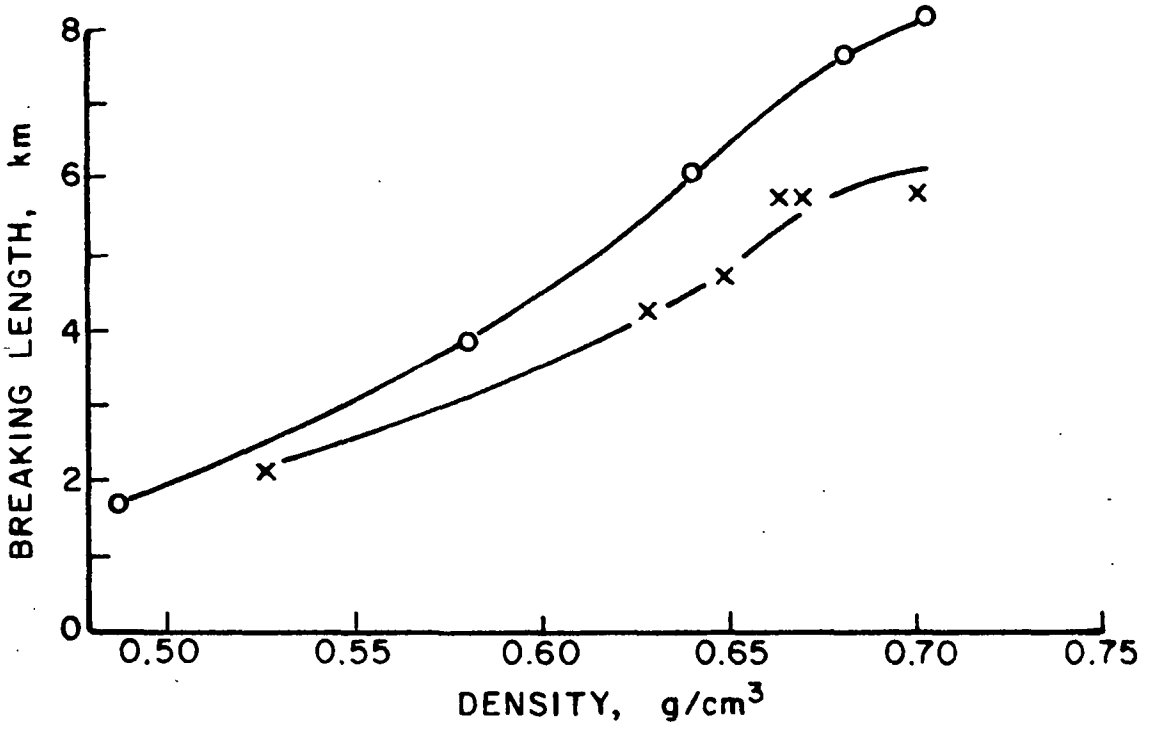


Figure 14. Breaking length vs. density.
x = roll refined
o = Valley beaten

If formation of the sheet of paper is uniform, so that the number of fibers crossing the rupture line is the same as the number crossing any other line in the strip, then the zero span breaking length test can be used to obtain F. A plot of zero span breaking length, Z, vs. sheet density for roll refined and Valley beaten pulps is presented in Fig. 15. The data illustrate, first of all, that the zero-span breaking length increases from 12 km to about 16 km as refining levels increase.

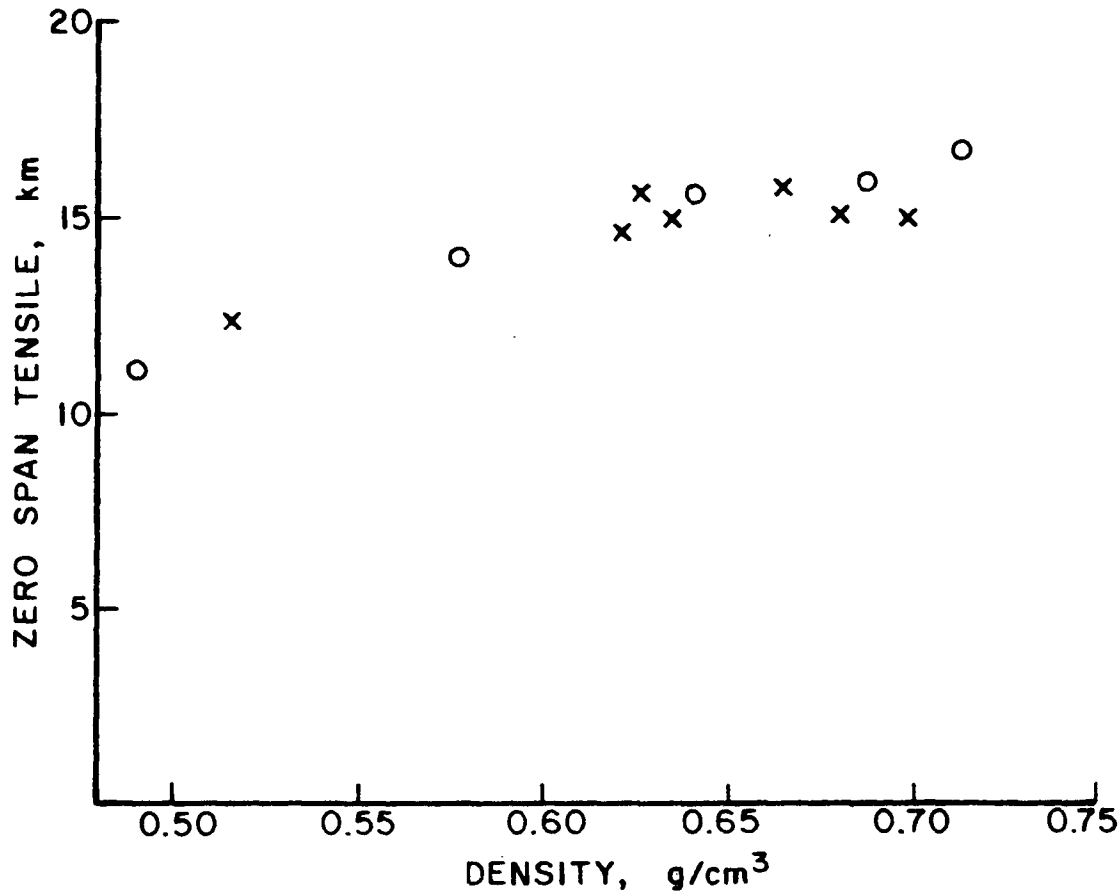


Figure 15. Zero span tensile vs. density.
x = roll refined
o = Valley beaten

Strengthening of the fibers during the early stages of refining has been reported (20,65) and is probably due to removal of kinks and microcompressions in the cell wall during straining (66). The second, and more important, point to make about the results in Fig. 15 is the zero span breaking length development is the same for each refining treatment. This means that, over the range of refining levels considered, neither refining action is damaging the fibers in terms of fiber strength. F is also a function of the density of the fiber wall, ρ, the gravitational constant, g, and the average fiber cross sectional area, A. Assuming 1) any difference in fiber cross-sectional area between the two refining methods is not large enough to entirely account for any difference in breaking length that may be observed in the sheets,

and, 2) because ρ, g, and Z are the same for each refining method sample, then any differences in breaking length between the two refined samples are most likely related to variables influencing interfiber bonding.

The variables that affect the resistance of bonds to breakage, B, are given by:

$$B = f\ [b,\ P,\ L/4,\ RBA] \tag{6}$$

where b = bond shear strength per unit bonded area

P = perimeter of the fiber cross section

L = fiber length

RBA = relative bonded area.

The relative bonded area of pulp fibers in a sheet has been studied (34,37,67, 68) and a method for determining RBA was given by Swanson and Steber (40). By plotting breaking length versus specific scattering coefficient for pulp treated in each refiner (Fig. 16), and extrapolating the regression lines to zero breaking length, a value for the specific free surface area for an unbonded sheet is found. The scattering coefficient at a finite breaking length was determined and both values were used in Eq. (2) to obtain the RBA at the chosen breaking length. The RBA values for both samples over a range of breaking lengths are presented in Table I. It is clear that roll refined fibers have higher RBA's than Valley beaten fibers at a constant breaking length.

In order for the roll refined sheets to have almost twice the RBA as the Valley beaten sheets, one of the other factors that influence bond strength must be correspondingly lower. Rearranging Eq. (1) to find the bond shear strength per unit bonded area, we obtain:

$$b = \frac{96\ A\ \rho\ g\ Z\ T}{(8Z-9T)\ P\ L\ (RBA)} \quad . \tag{7}$$

Values for Z, T, RBA and L were measured in this study. To complete the calculation of bond shear strength, the following values of A, ρ, and P for a spruce sulfite pulp were used (69):

$$A = 2.4 \times 10\text{-}6\ cm^2$$

$$P = 9.0 \times 10^{-3}\ cm$$

$$\rho = 1.56\ g/cm^3$$

	Roll Refined	Valley Beaten
T	6.0 km	6.0 km
Z	15.5 km	15.5 km
L	0.22 cm	0.17 cm
RBA	0.535	0.356
b	4.417×10^7 dynes/cm^2	8.591×10^7 dynes/cm^2

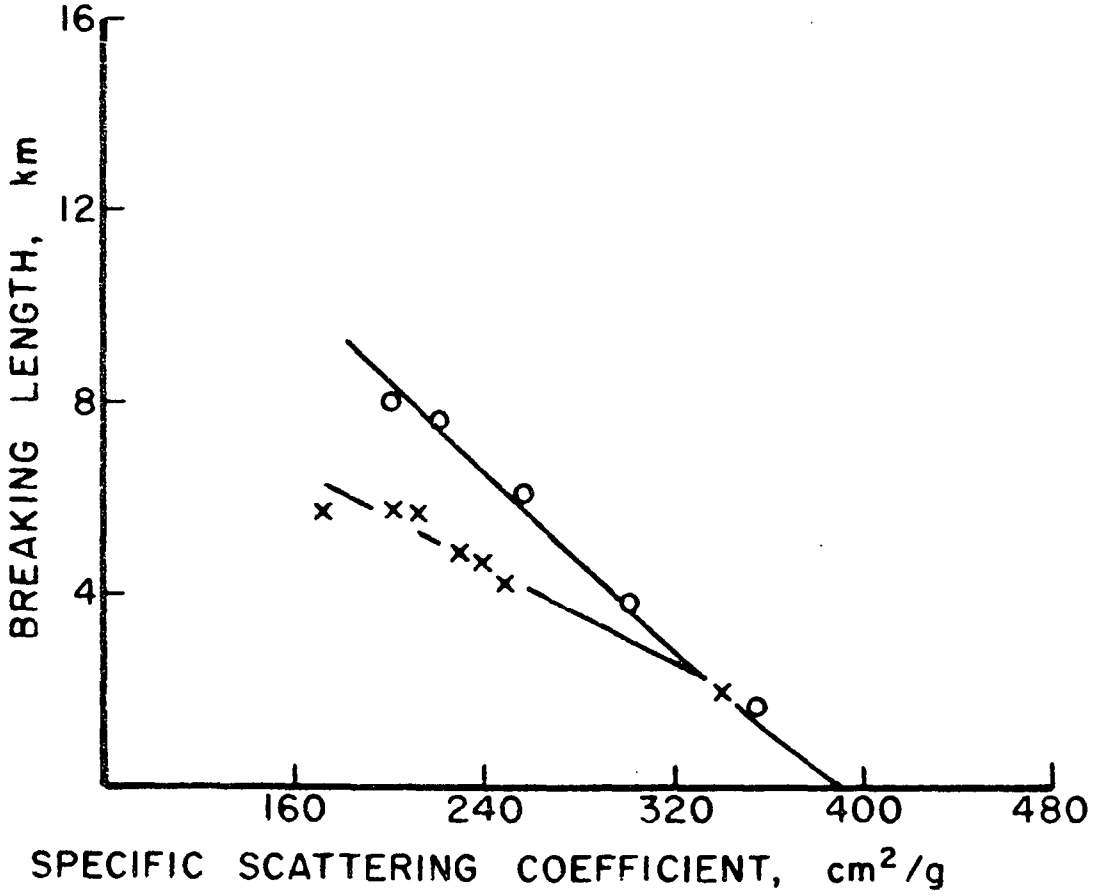


Figure 16. Breaking length vs. specific scattering coefficient.
x = roll refined
o = Valley beaten

TABLE I

RELATIVE BONDED AREA FOR ROLL REFINED AND VALLEY BEATEN PULP

Breaking Length, km	0	3	4	5	6
Spec. scattering co., cm^2/g					
Roll refined	390	304	263	221	181
Valley refined	390	316	294	272	251
RBA, %					
Roll refined	0	22.0	32.6	43.3	53.5
Valley refined	0	19.0	24.6	30.2	35.6

Assuming A and P are the same for each type of refined fiber, then the difference in b is almost a factor of two, which is substantial. If the roll refined fibers are assumed to be flattened out after refining and the Valley beaten fibers remain constant in cross section, then the difference in the calculated bond shear strength between the two samples becomes even greater. The perimeter of the flattened fibers has a stronger influence on the A/P ratio than cross-sectional area. As the fibers become flatter, P increases faster than A, and the resulting value for b decreases.

Regardless, the results suggest that the difference in tensile development between roll refined fibers and Valley beaten fibers is due to differences in interfiber bonding characteristics. Specifically, roll refined fibers require a larger relative bonded area to attain a given breaking length because of lower bond shear strength per unit bonded area. The effect of changing the interfiber bond characteristics of roll refined fibers is discussed in the Importance of Refining Effects section.

TEAR FACTOR

A plot of tear factor vs. breaking length for several different roll refining conditions is shown in Fig. 17. The combination of tear factor and breaking length provides a balanced measure of the success of refining, because as one property increases the other usually decreases. The results show that as breaking length increases, tear factor goes through a maximum. Figure 18 is a plot of tear factor versus density for various levels of roll refined pulp and Valley beaten pulp. Again, tear factor decreases at high refining levels for both apparatuses.

The resistance of a sheet to tear is directly proportional to the ability of the sheet to absorb energy before it fractures. The work involved in tearing consists of two components: the work to pull fibers out of a network and the work to rupture the fibers. The tearing resistance of paper from unbeaten pulp is almost entirely due to work in pulling fibers out of the network (65). Since fiber to fiber contact is low, frictional resistance is small, and tear resistance is low. After slight beating, fiber to fiber bonding increases, and tearing resistance increases because the frictional resistance of pulling the fibers out of the network has also increased. However, as seen in Fig. 18, the maximum level of tear resistance reached by roll refined pulp is less than the Valley beaten pulp, further suggesting a difference in interbonding between the two pulps. Finally, as beating increases to its highest levels, the fibers in the sheet no longer slip past each other, increasing the number of fibers that rupture across the tearing zone. The tearing action becomes more of a shearing action than pulling out. Since the work to rupture is less than the work to pull out (65), the energy required to tear decreases.

The preceding mechanism for tear resistance is acceptable for explaining the results of the roll refining data because it doesn't include the variable of fiber

length. Another explanation for the decreasing portion of the tear curve is due to decreases in fiber length found in conventional refining. The roll refiner, however, does not appreciably reduce the fiber length. Therefore, the influence of fiber length on tear resistance is strongest in the unrefined state because the added length can increase the frictional drag when pullout work is important.

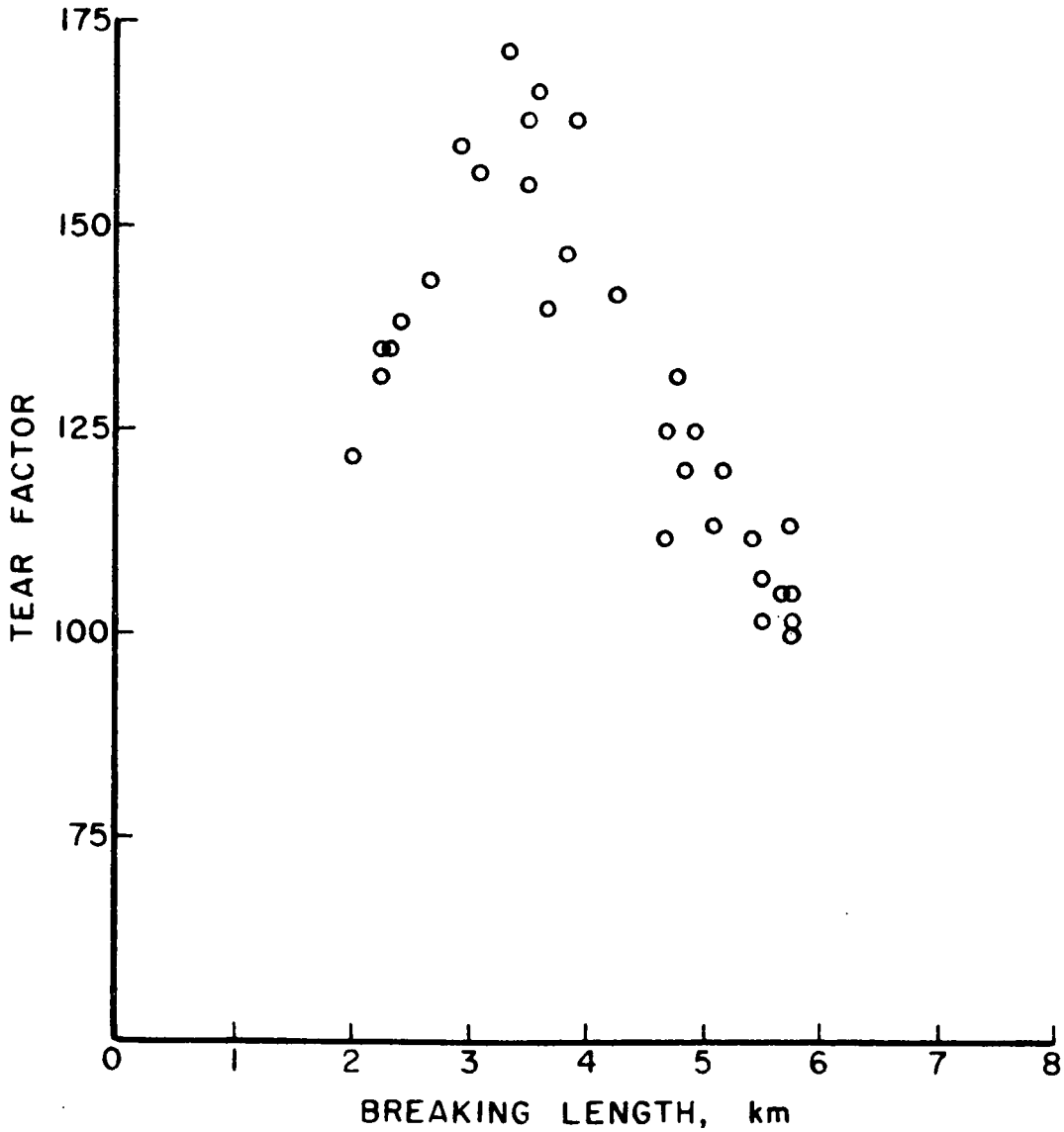


Figure 17. Tear factor vs. breaking length.
o = roll refined

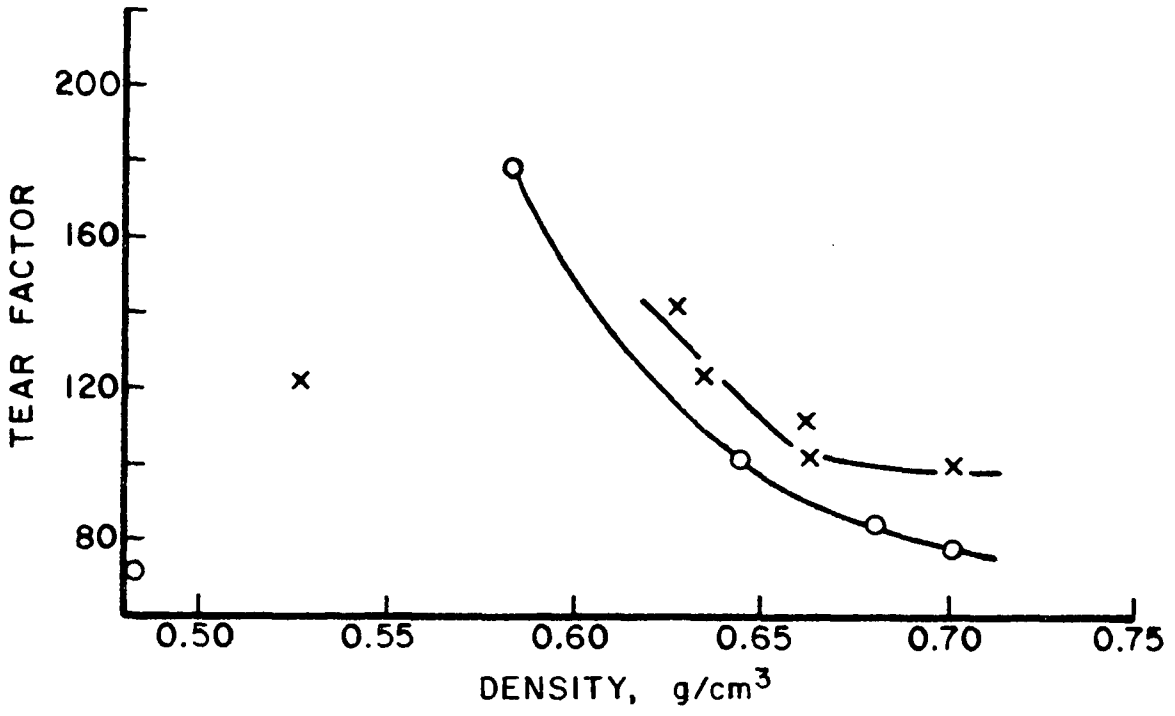


Figure 18. Tear factor vs. density.
x = roll refining
o = Valley beaten

AIR RESISTANCE

A plot of Gurley air resistance versus sheet density is shown in Fig. 19. The pores in paper form a complicated system of interlocking, crooked and criss-crossing channels which range in size from fairly large diameters to capillary dimensions (65). We see from the figure that sheets from roll refined pulps have lower air resistance values than sheets from Valley beaten pulp. Comparing the samples at a constant sheet density, the reason for the differences in air resistance between the refining methods is due to the differences in external surface area and fines of the fibers. The external fibrillation and fines generated by the Valley beater fill in the voids in the sheet and reduce the size of the pores for air passage. At higher sheet densities (higher refining levels), the pores begin to close up in the roll refined sheet due to better bonding. This raises the air resistance.

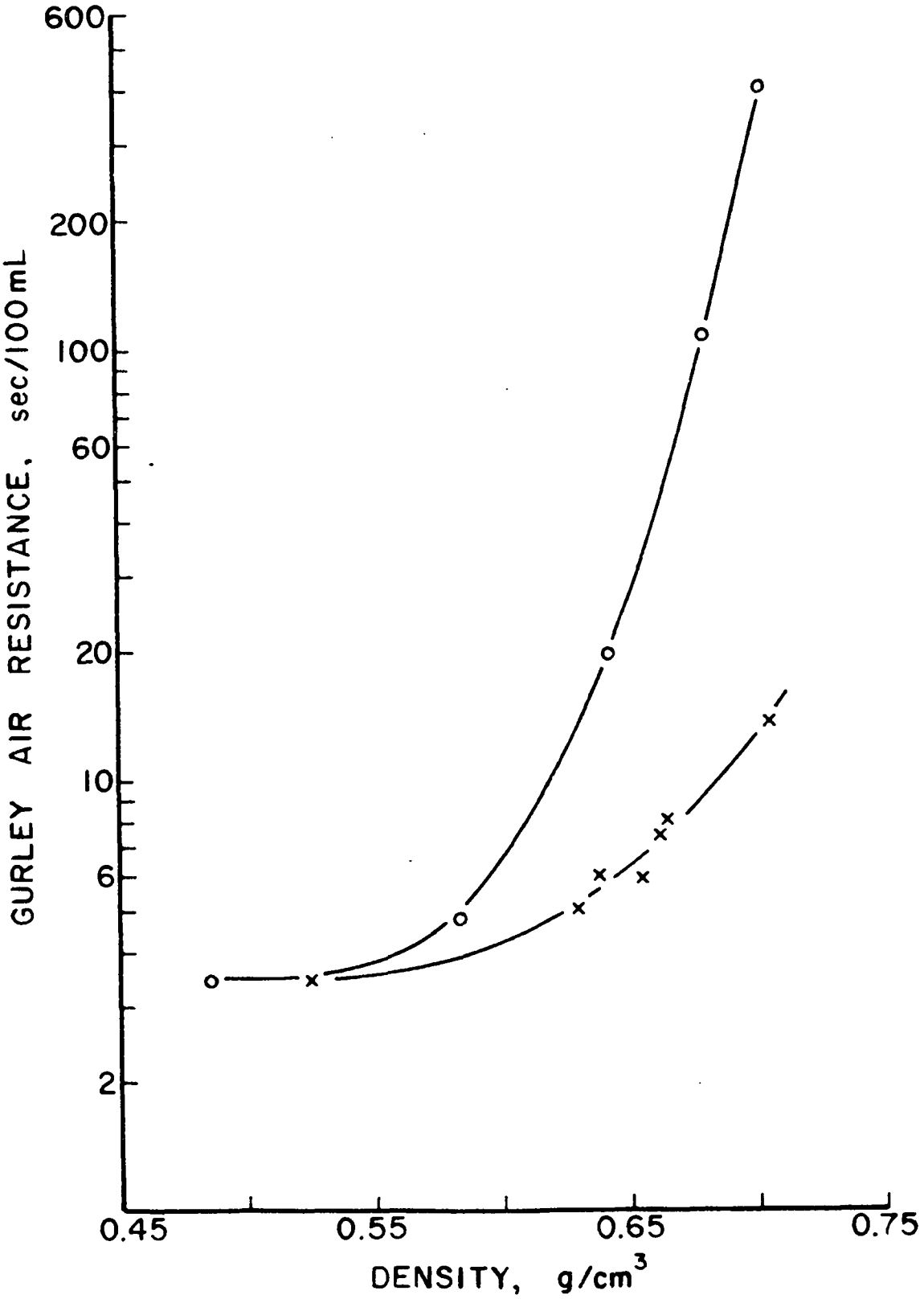


Figure 19. Gurley air resistance vs. density.
x = roll refined
o = Valley beaten

The difference in air resistance between the samples increases with density. This is most likely due to more fines being generated during refining in the Valley beater. These fines are also retained in the network, filling in more pores and increasing air resistance.

To summarize, paper properties of roll refined pulps follow the same trends as do the properties for Valley beaten pulps. For example, a threefold increase in breaking length, an increase in sheet density, a decrease in specific light scattering coefficient, and a maximum in tear strength were observed. The magnitude of these changes, however, was generally less than in the case of the Valley beaten pulp. The differences in breaking length and tear resistance between the two refining methods is believed to result from a difference in interfiber bond characteristics, specifically bond shear strength per unit area. Air resistance of sheets from roll refined pulp increased with increasing sheet density, yet the levels were much lower than those obtained with the Valley beater. The main difference in the air resistance results is the greater amount of external fibrillation and fines generated by the Valley beater and their ability to fill in the pores in a sheet.

PAPER PROPERTIES VS. ENERGY INPUT

One of the objectives in developing the apparatus was to provide measurements of the energy used to process the fibers in the roll refiner. The method of measuring energy consumption in the roll refiner is described in Appendix I. Attempts to compare property development vs. net energy input to what happens in a Valley beater were hampered by difficulties in obtaining a good estimate of the net energy input to the Valley beater. Therefore, the roll refiner comparisons were made with the Escher Wyss conical refiner. The net specific energy consumed in the Escher-Wyss refiner was calculated by subtracting as many of the operating losses as could be measured (49).

Plots of four sheet properties as a function of net specific energy input are shown in Fig. 20-22. Figure 20 shows breaking length development is more rapid for the roll refined pulps in the initial stages of refining than for either intensity of Escher-Wyss refining. The strength level of 5.7 km is reached at one-third to one-sixth of the refining energy required of the other two methods. But no further increases in breaking length are obtained with increased roll refining. Figure 21 shows density development is greater for roll refined pulps at low refining energy consumptions. Density also reaches a plateau at approximately 50 kWh/t of energy consumption. The specific scattering coefficient (Fig. 22) for the roll refined samples drops off very quickly during the initial stages of refining. Compared with Escher-Wyss refined samples at equal refining energy inputs, sheets from roll refined pulp have consistently lower specific scattering coefficients.

The roll refiner will develop paper with acceptable strength and optical properties. If, for example, the end product in question had specifications of 5.8 km breaking length and a TAPPI density of 0.66 g/cu.cm, then these property levels could be obtained with the roll refining action. Moreover, these properties can be reached with the roll refiner at net energy inputs of 50 kWh/t, about 80% less than the net energy consumed in the Escher Wyss to reach the same property level. The potential to reduce the overall energy demand in refining should be possible by isolating the desirable components of refining from the energy dissipative, less useful components.

As mentioned in the section on Breaking Length, an upper limit on the breaking length was obtained with the roll refiner at approximately 5.8 km. The previous discussion held that the difference in breaking length between roll refined fibers and Valley beaten fibers was due to the respective differences in interfiber bonding

characteristics. A related reason for the limit in breaking length may be due to the method used to refine the fibers. The amount of useful work in roll refining may be very high in the beginning of a particular experiment, but past a certain number of cycles, additional units of work are being ineffectively applied. This is illustrated in Fig. 20, where the highest breaking lengths are achieved during the early stages of roll refining. The same trend was also observed with density and light scattering coefficient (Fig. 21, 22).

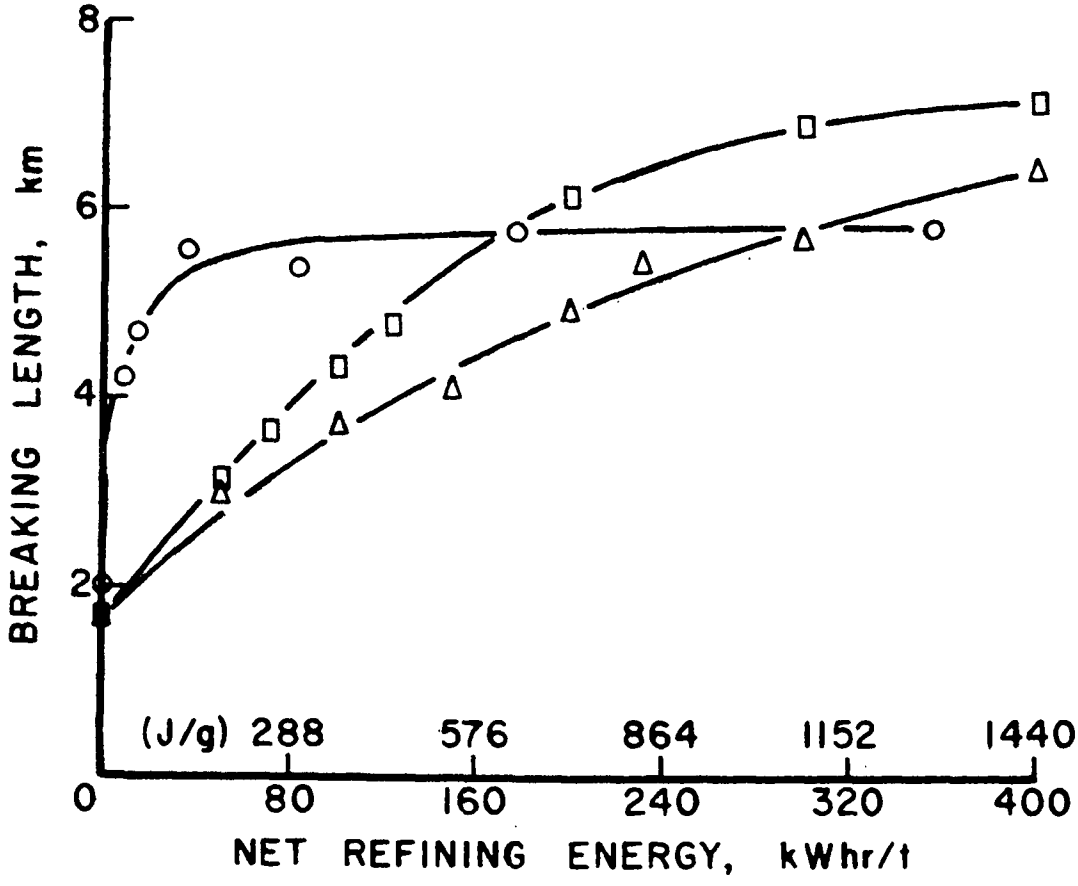


Figure 20. Breaking length vs. net refining energy.
x = roll refined
Δ = Escher-Wyss refined - low intensity
□ = Escher-Wyss refined - high intensity

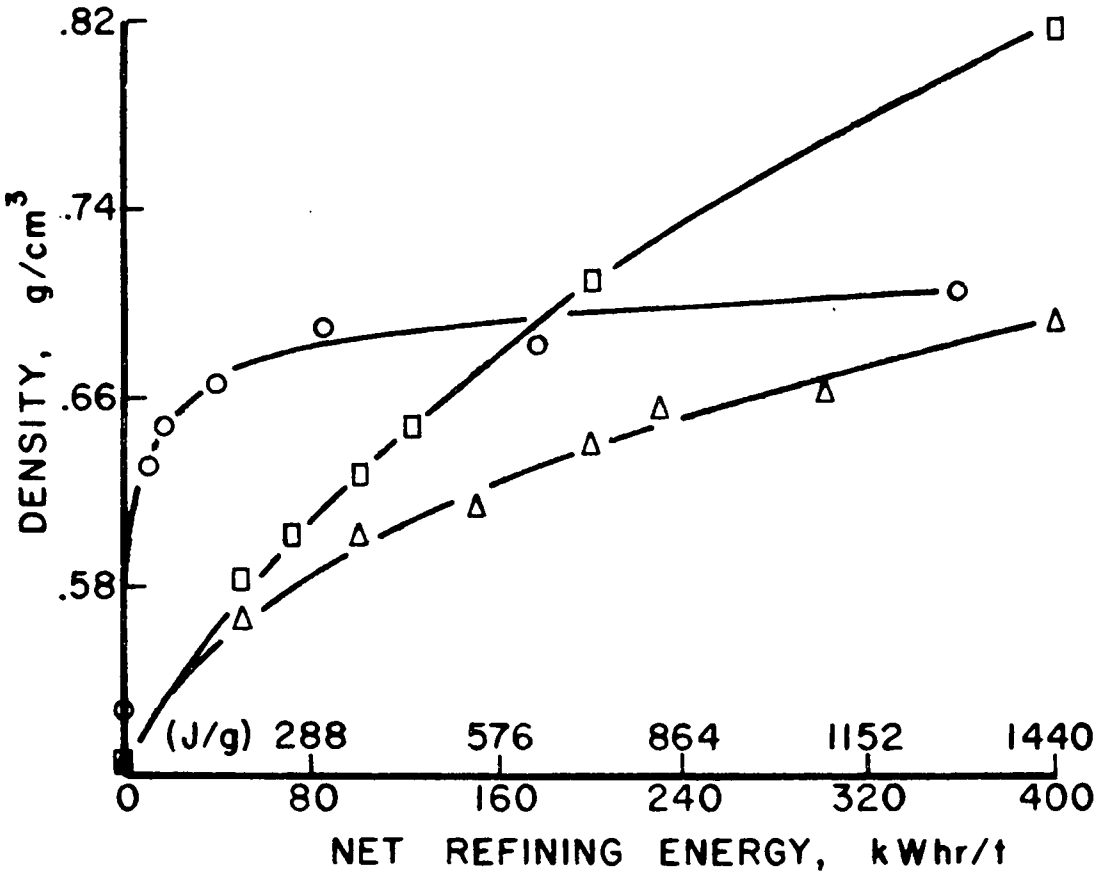


Figure 21. Density vs. net refining energy.
x = roll refined
Δ = Escher-Wyss refined - low intensity
□ = Escher-Wyss refined - high intensity

These observations may be explained by the possibility that past the threshold in property development, there no longer exists the potential for fibers in the network to deform under the simple compressive action. It may be necessary to rearrange the fibers on the support roll at this stage in the process to facilitate further effective action.

It would be very interesting to investigate whether the roll refining process could be made to be effective up to high refining levels by allowing rearrangement of the fibers on the roll. This could be done simply (but at great expense of time) by periodically removing the fibers from the roll, reslushing them, forming a new sheet and resuming the refining operation.

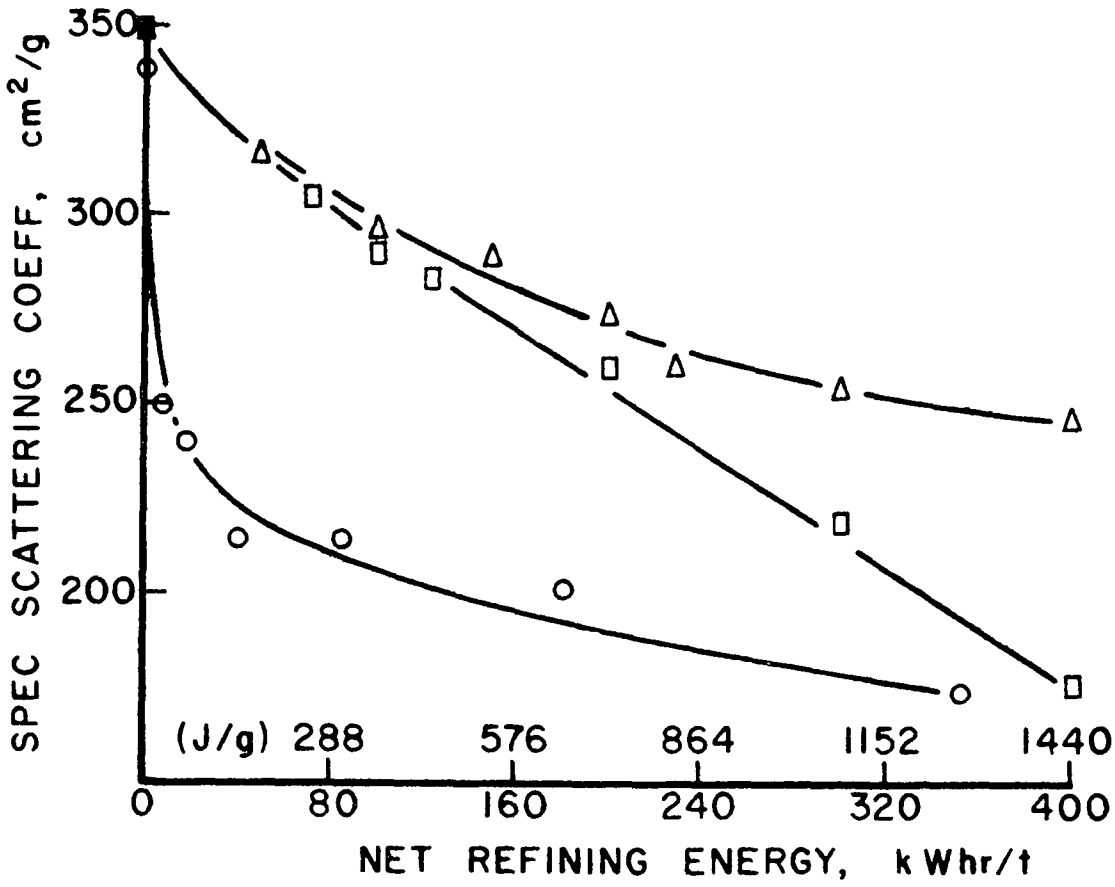


Figure 22. Specific scattering coefficient vs. net refining energy.
x = roll refined
Δ = Escher-Wyss refined - low intensity
□ = Escher-Wyss refined - high intensity

IMPORTANCE OF REFINING EFFECTS

The final objective of this thesis was to establish the relative importance of internal fibrillation, external fibrillation, and fines. The breaking length of sheets from internally fibrillated fibers was found to be lower than the values developed by the Valley beater. The probable reason for this difference was because of low bond shear strength. The obvious question then is, what can be done to improve the bond shear strength of roll refined fibers?

The refining effects not found in roll refined fibers are external fibrillation and fine material. It seems reasonable that by roll refining pulp fibers to a

moderate level and then adding fines or a treatment to promote external fibrillation (being careful not to alter fiber strength or length), the bonding characteristics would change. The addition of a refining effect to internally fibrillated fibers may improve bond strength enough to increase the breaking length to levels achieved in the Valley beater. Moreover, by treating unrefined fibers to promote external fibrillation and by adding fines to the unrefined fibers, the relative importance of the three refining effects can be obtained.

ABRASION REFINING - EFFECT ON FIBER PROPERTIES

The operating conditions of the abrasion refiner were chosen to provide mainly external fibrillation without decreasing fiber length or zero span breaking length. This was done using fine grit surfaces at large plate spacings. Qualitative proof that the abrasion refiner does cause external structural changes in pulp samples at this set of conditions is shown in Fig. 23. The micrographs show the relative amounts of external fibrillation in samples of A) unrefined, B) abrasion refined, C) roll refined, and D) a combination of roll and abrasion refined pulps. Comparing A and C with B, there is a definite increase in the amount of external fibrillation due to the action of the abrasion refiner.

The effect of abrasion refining on fiber length and zero span breaking length is shown in Fig. 24. Small changes in the shape of the histograms and no significant change in the weighted average fiber length are the result of roll refining, abrasion refining, and their combination. Zero span breaking length data indicate the various mechanical treatments did not degrade fiber strength. The abrasion apparatus, therefore, produced fibers that differed from each other mainly in the relative amount of external fibrillation.

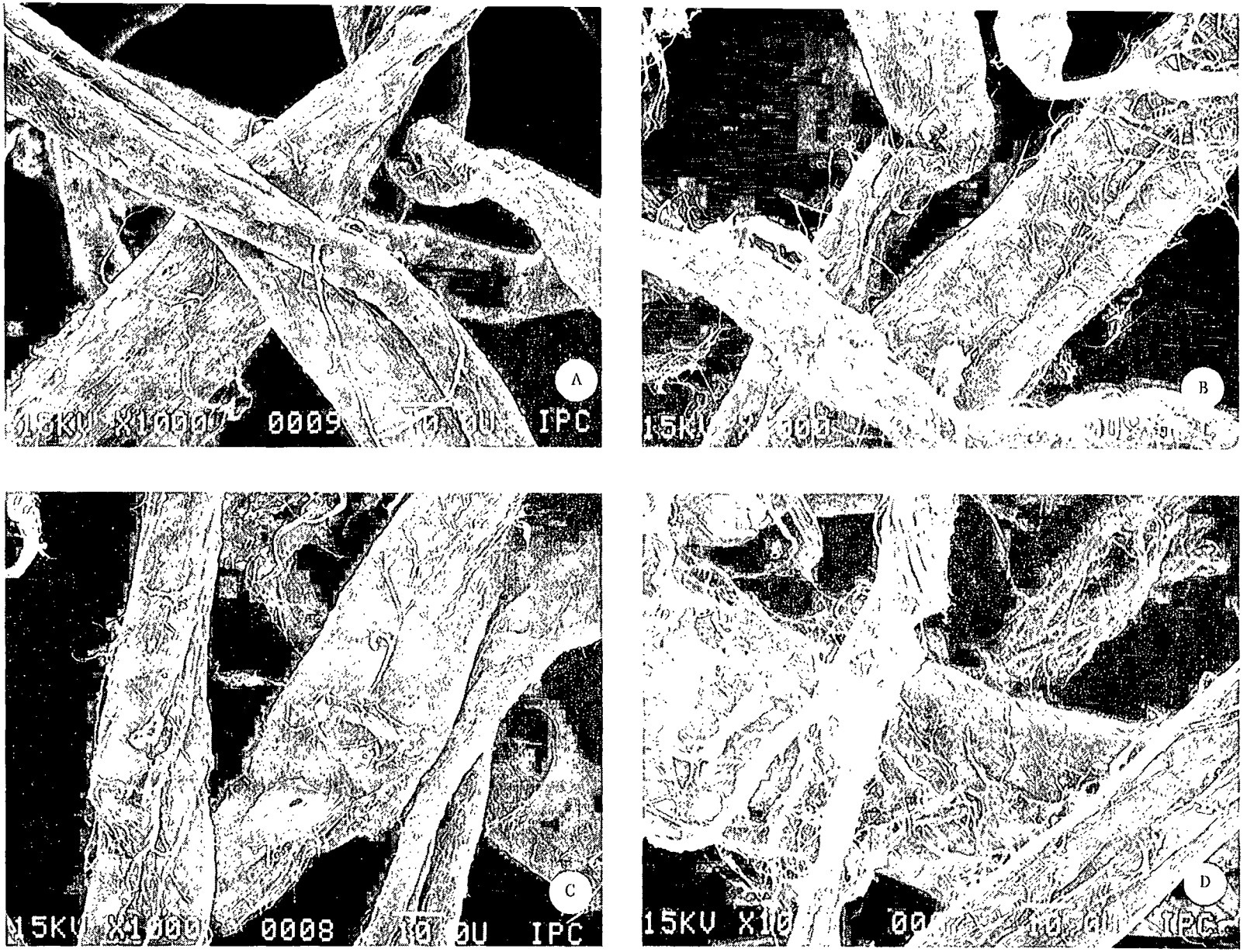


Figure 23. SEM micrographs of bleached spruce sulfite pulp (1000X) showing the amount of external fibrillation. A, unrefined; B, abrasion refined, 1000 rpm for 5 min, 4 mm gap; C, roll refined, 500 revolutions; D, combination 500 revolutions roll refiner, 1000 rpm for 5 min in abrasion refiner.

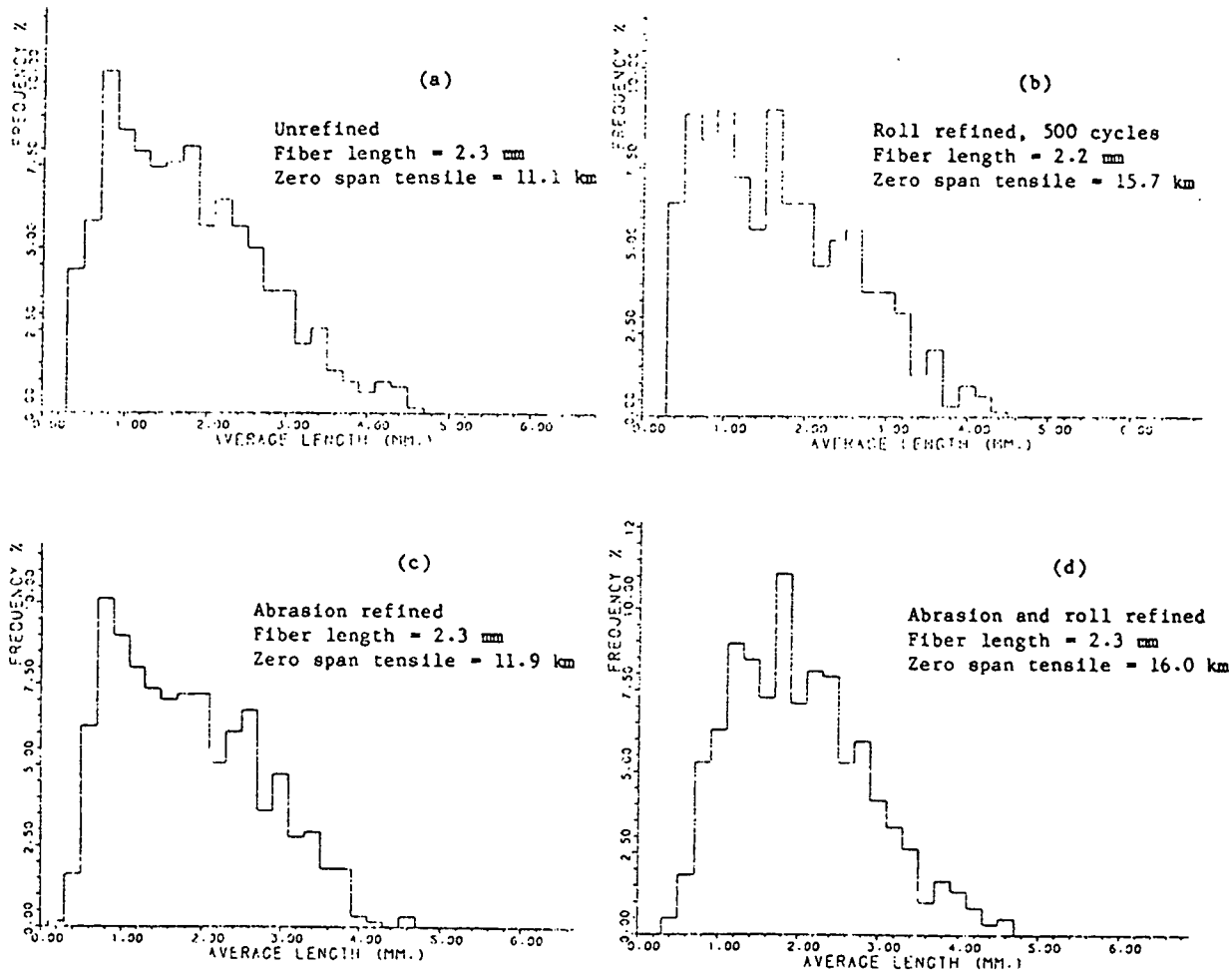


Figure 24. Fiber length distributions for different refining treatments.

EFFECT OF EXTERNAL FIBRILLATION ON PAPER PROPERTIES

Figures 25-28 show the effect of adding external fibrillation to pulp samples that were unrefined and previously roll refined for 500 cycles. By adding external fibrillation to these pulps, the breaking length and density relationships (Fig. 25) are altered only in density. The density of the handsheets increases approximately seven percent without an increase in tensile. This change was accomplished without decreasing fiber length or fiber strength. The influence of external fibrillation on interfiber bonding is therefore at the center of the question.

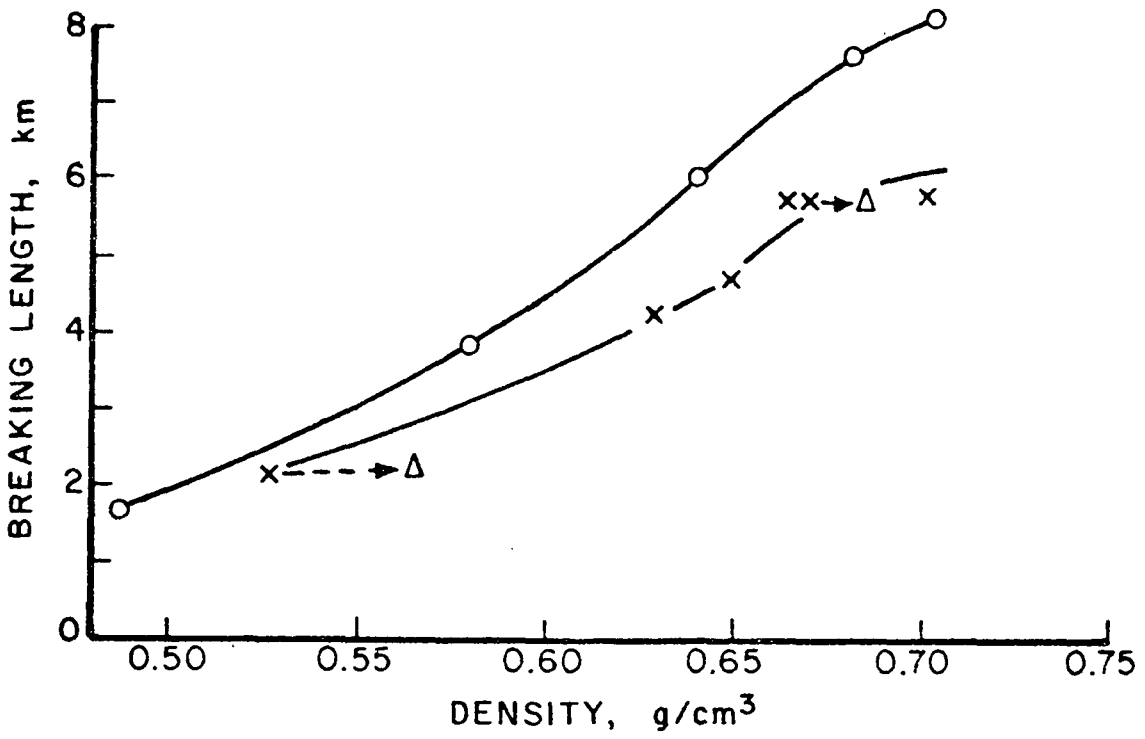


Figure 25. Breaking length vs. density.
x = roll refined
o = Valley beaten
Δ = effect of abrasion

The relationships between breaking length and light scattering coefficient after abrasion refining are presented in Fig. 26. The results indicate the amount of free surface in the sheets of both samples increases following abrasion; however, no increase in breaking length was observed. External fibrillation developed in conventional refining represents a large amount of surface area while the fibers are in suspension (Fig. 23 B and D). On drying, however, the large amount of external fibrils are expected to aid in bonding (20), thus lowering the scattering coefficient and increasing the breaking length. This was not observed in the present study. The abrasion refiner, therefore, might produce a different kind of fibrillation, one that keeps the fibers from coming into optical contact with each other. Or, it might be that the influence of external fibrillation in interfiber bonding is

not as important as authors like Clark have reported (20). The importance of external fibrillation may, however, lie in the sheet forming operation (21), or in the idea that the existence of external fibrillation is important only because it is a precursor to fines (70).

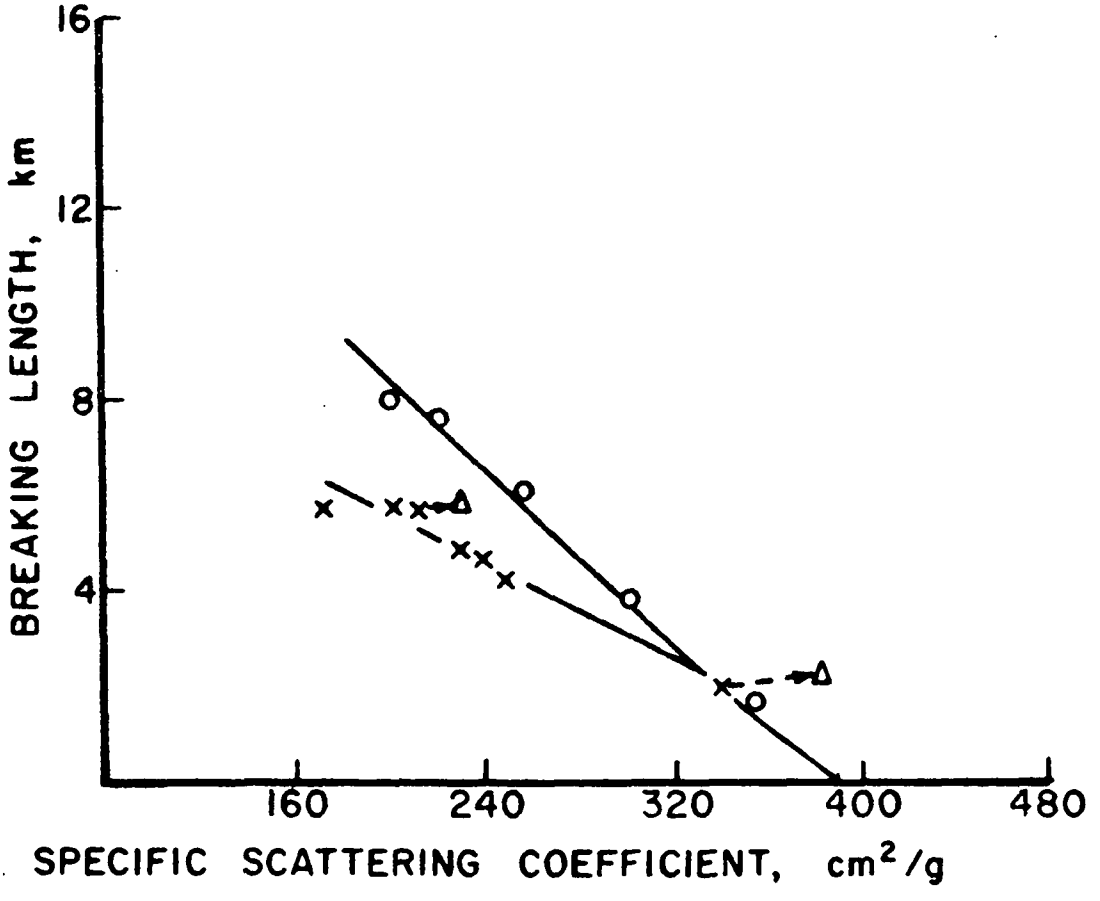


Figure 26. Breaking length vs. specific scattering coefficient.
o = roll refined
x = Valley beaten
Δ = effect of abrasion

Tear factor was unaffected by adding external fibrillation (Fig. 27). The results of adding external fibrillation to an unrefined sample may be an explanation to reinforce the theory for tear strength given on page 45. As mentioned there, the initial rise in tear factor to a maximum is due to the increase in bonding, hence, an increase in the work to pull fibers out of the network. By adding mainly external surface, which apparently does not bond well, without decreasing the length of

the fibers, no real gain in tear factor is achieved. These results may indicate that tear factor is affected mainly by interfiber bonding and the changes bonding goes through during refining.

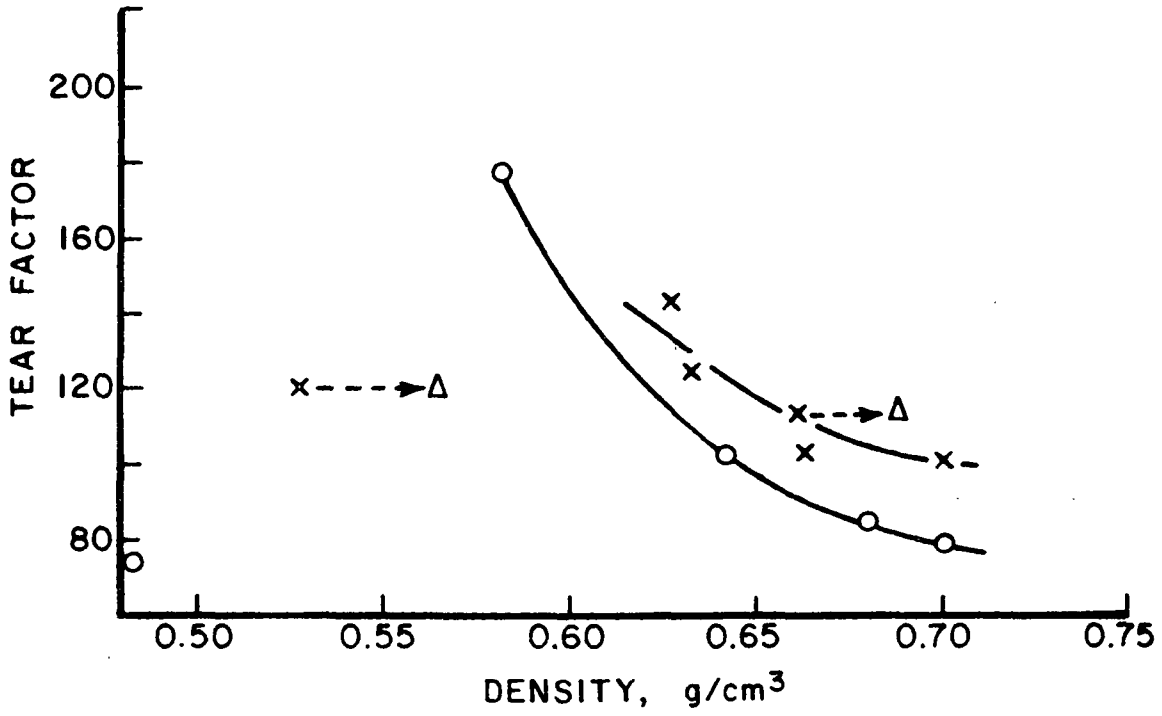


Figure 27. Tear factor vs. density.
x = roll refined
o = Valley beaten
Δ = effect of abrasion

Perhaps the largest change in the paper properties due to external fibrillation is in air resistance (Fig. 28). The additional external surfaces caused by abrasion refining act to reduce the size of, and even seal off, pores that existed in the sheets without external fibrillation.

Abrasion of an unrefined sample yields a higher air resistance than abrasion of a previously roll refined sample. The explanation for this observation may be due to the difference in bending stiffness of each sample. Rigid, unrefined fibers are

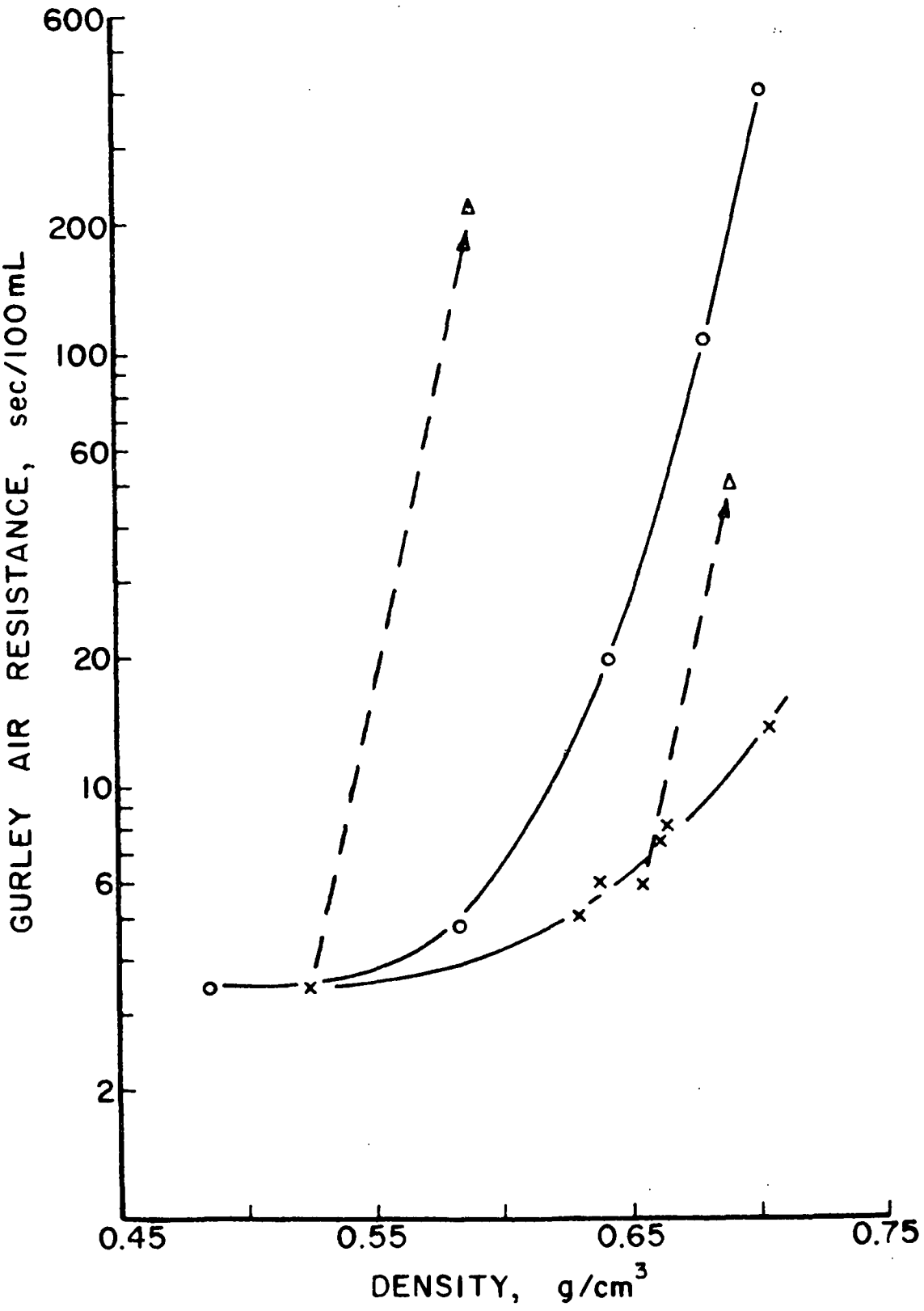


Figure 28. Gurley air resistance vs. density.
x = roll refined
o = Valley beaten
Δ = effect of abrasion

not able to avoid the stresses inside the abrasion refiner and, therefore, received more treatment. The roll refined samples are probably more flexible and can align themselves easily with the flow patterns inside the refiner, thus avoiding mechanical action.

There is apparently no advantage to externally fibrillating the bleached spruce fibers in the abrasion refiner in terms of breaking length or tear factor. The results suggest that the external fibrillation produced by the abrasion refiner may be of an unusual type, producing poorly bonding debris. This limited information may cast new light on the effect of external fibrillation and its lack of importance on sheet strength. Additional work with the abrasion refiner should be done.

EFFECT OF FINES ON PAPER PROPERTIES

Figures 29-32 illustrate the influence of adding fines to an unrefined pulp suspension and a suspension that was roll refined for 500 cycles. The amount of fine material in the final sheet was 16% on an o.d. basis. Figure 29 shows that immediate increases in both breaking length and density occur as a result of fines addition. The new paper property levels obtained by adding fines were close to the levels developed in the Valley beater.

A plot of breaking length *vs.* specific light scattering coefficient (Fig. 30) shows the influence of fines: 1) they increase both light scattering and breaking length when they are added to unrefined fibers and 2) they increase the breaking length to the level of the Valley beaten pulp when they are added to the roll refined pulp.

Tear strength development as a function of fines addition is shown in Fig. 31. By adding fines to the unrefined sample, tear strength and density increase as expected. The presence of the fines densifies the sheet, which increases the work to

pull fibers out of the network. Hence, tearing resistance increases. When fines were added to the roll refined sample, the density of the sheet increased and tear strength decreased to a level produced by a higher degree of roll refining. Qualitatively, this is the same effect produced by the Valley beater.

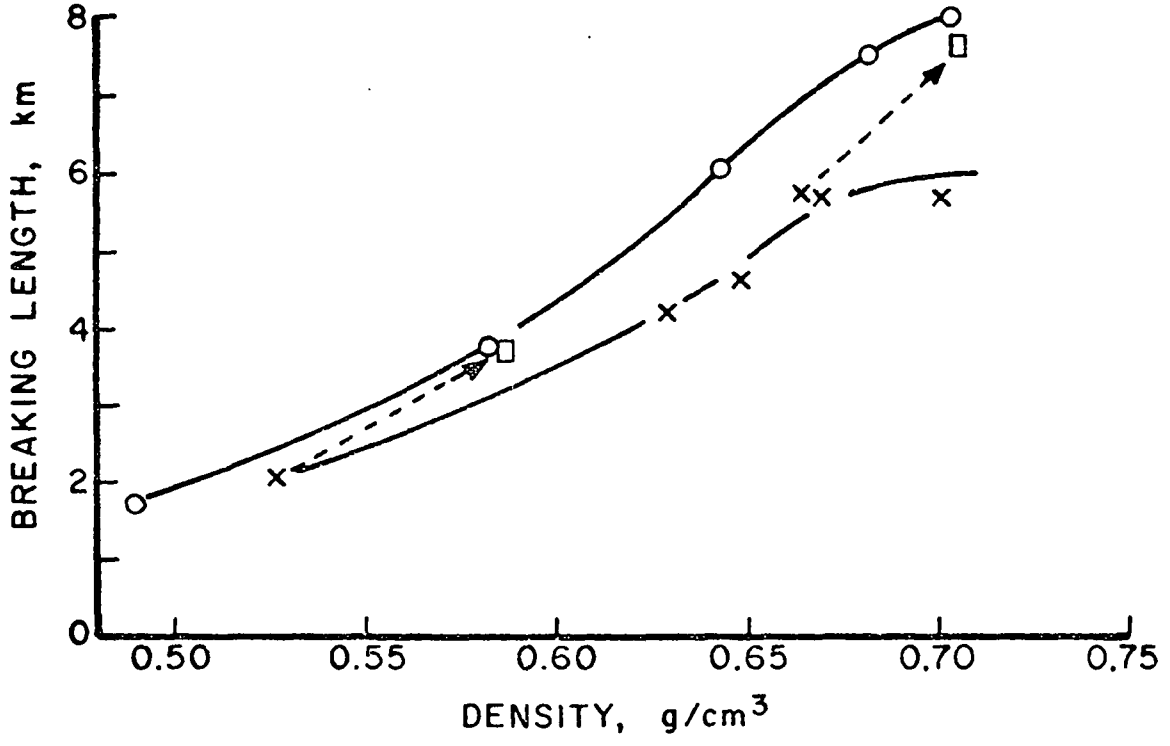


Figure 29. Breaking length vs. density.
x = roll refined
o = Valley beaten
□ = fines addition

The air resistance of the sheets increased dramatically following fines addition (Fig. 32). The increase in specific surface area of the sample following fines addition over their original state shows up in a "tighter" sheet. The air resistance for the combination roll refined/fines addition sample reaches the level higher than the Valley beaten sample.

After reviewing several papers on the role of fines in suspension and in sheets, comparisons were drawn with the results for this section of experiments.

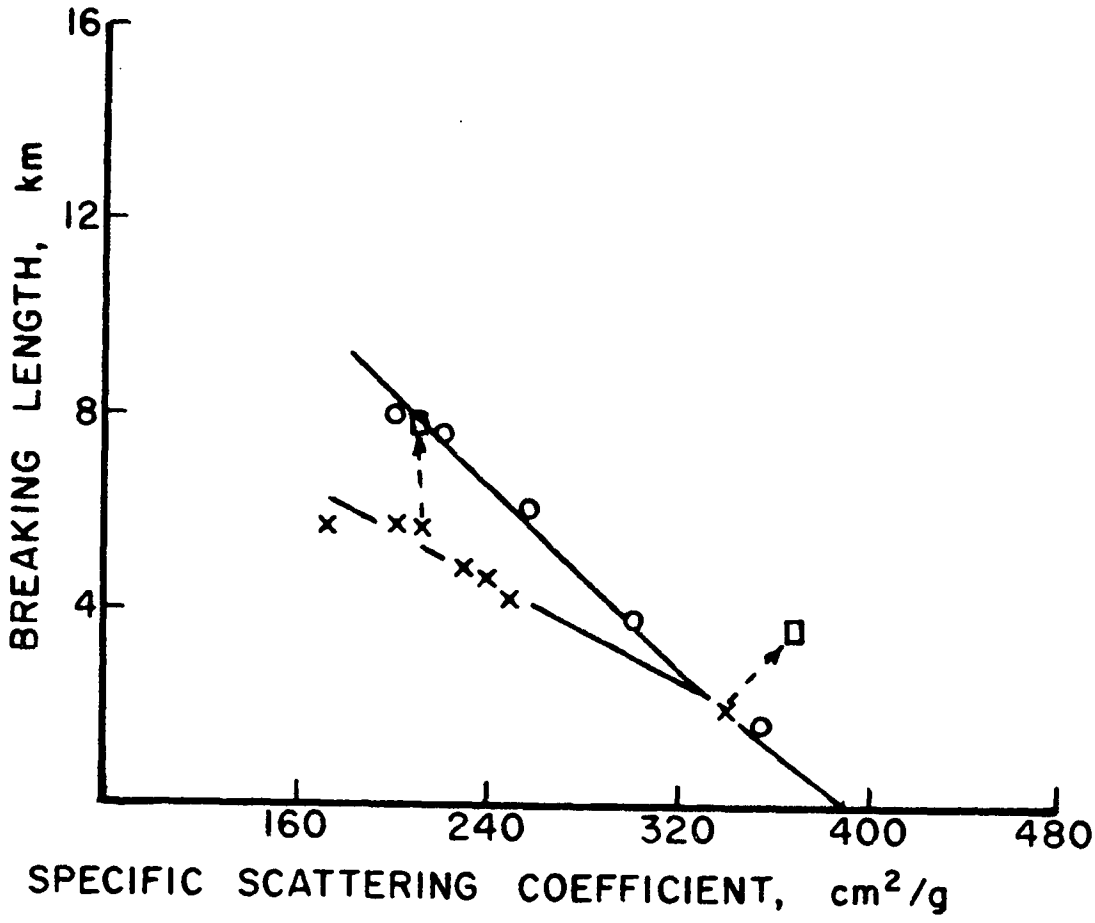


Figure 30. Breaking length vs. specific scattering coefficient.
x = roll refined
o = Valley beaten
□ = fines addition

The fines used in this experimental work were produced by highly refining pulp fibers. They were not screened or classified, so they include both primary and secondary fines fractions (27,28). Primary fines are free particles associated with the unbeaten pulp and secondary fines are the free particles generated in refining. Although there is belief that the chemical nature of the fines is no different from the longer fibers (71), evidence suggests that, for properties of concern in papermaking, the chemical reactivity of fines is quite different from the fibers (28). In the present case, there are no differences in the chemical nature *per se* because the same fibers that were ground up were used in sheet forming. However, the fact

that the fibers were ground up to a material resembling a gel suggests that different chemical entities (hemicelluloses in particular), formerly inside the fiber, became more accessible. The fines' ability to influence bonding, therefore, increases greatly.

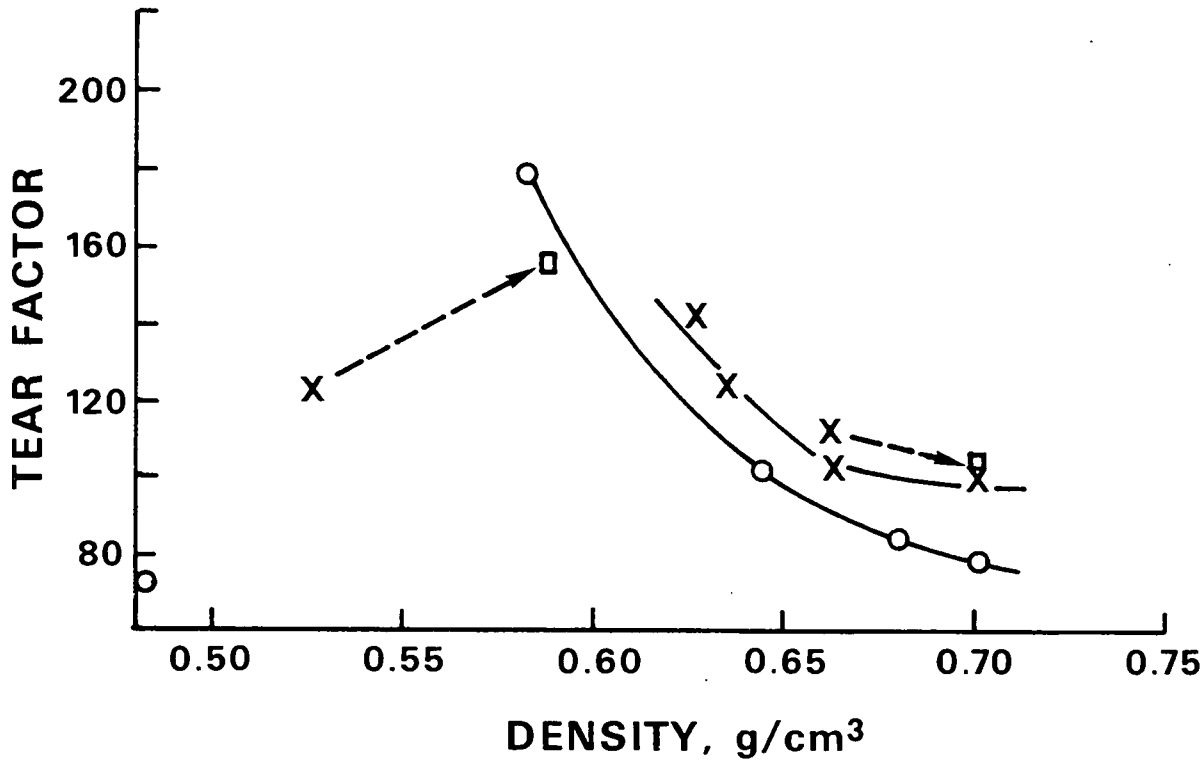


Figure 31. Tear factor vs. density.
x = roll refined
o = Valley beaten
□ = fines addition

A second point to consider is how fines influence the sheet forming process. Figure 33 is a simplified model of two idealized fiber crossings in dry paper in three alternate cases (27). In Fig. 33, the sheet has been made from unbeaten fibers without fines present. The fibers are stiff and only partly collapsed. The number of bonds is low and the bonded area per bond is small. As a consequence, the sheet has low strength.

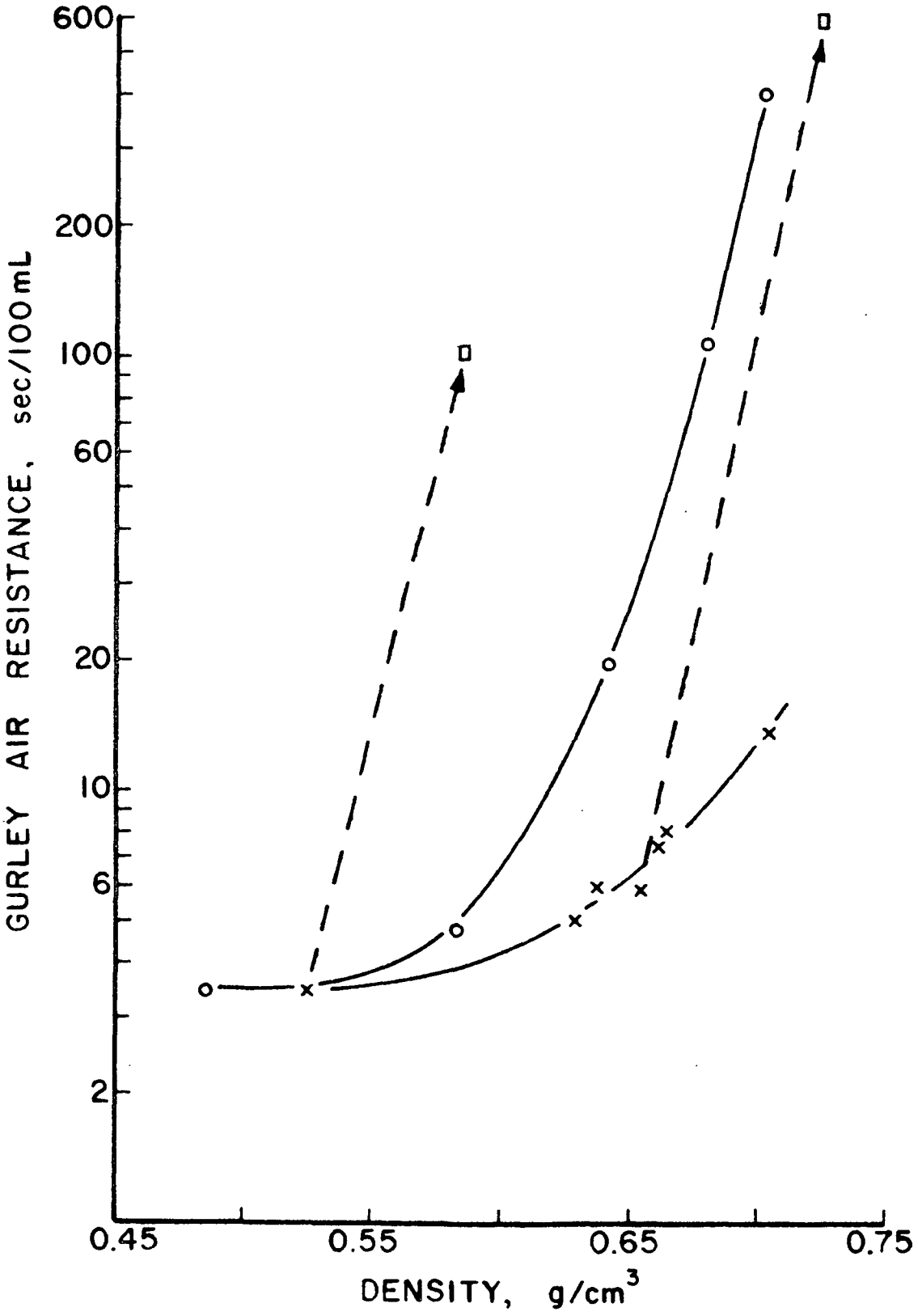


Figure 32. Air resistance vs. density.
x = roll refined
o = Valley beaten
□ = fines addition

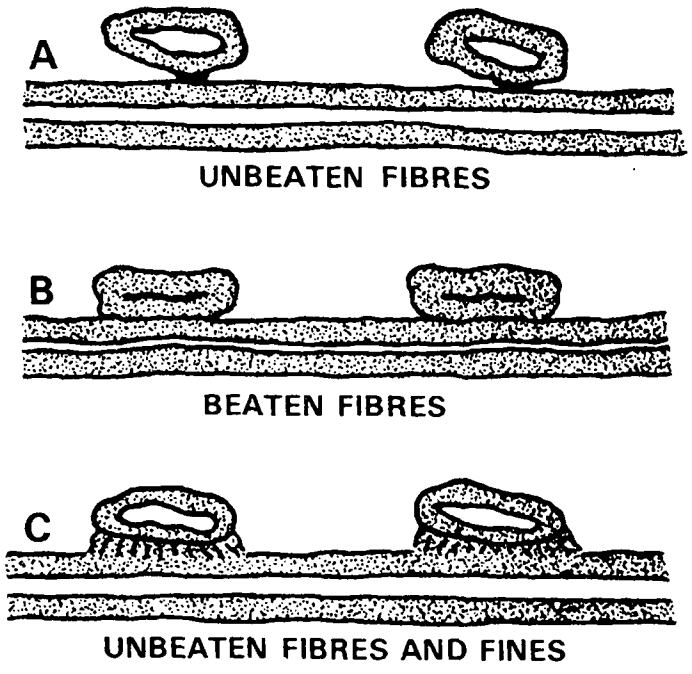


Figure 33. Models of interfiber bonding (27).

Figure 33(B) represents the corresponding situation in a sheet of roll refined fibers without fines present. As the RBA values indicated (see Effect of Roll Refiner on Paper Properties), interfiber bonding increased with refining, which in turn increased the breaking length and density.

Figure 33(C) depicts the influence of fines addition to unbeaten fibers which effectively increases the fiber to fiber contact area. The characteristics of the fine material in this situation are important for two reasons; their influence in sheet consolidation and their influence in a dry sheet under tensile stress. Their size, form, and their large specific surface are important during consolidation of the fibers into a sheet. It has been observed that the last free water in a drying web exists as menisci held in fiber crossings (72). Some degree of fines concentration may thus occur in the fiber crossings if the fines have some mobility to follow the water phase (73). On this basis, it is assumed that the fines increase

the effective contact area of the bonded fiber crossings and, in the case of unrefined fibers, possibly also help establish contacts between fibers which would have been unbonded if fines were not present. This assumption is supported by the increase in density which occurs when fines were added to unbeaten fibers (Fig. 29-32).

In a dry sheet, fines reduce local stress concentrations in the bonded fiber crossings (71). This allows for a more uniform stress distribution when a load is applied to the sheet. To explain the low tensile strength obtained with only internally fibrillated fibers followed by the improved tensile strength by adding fines, consideration was given to the work of Button (74). He has shown that when two strips of cellophane are bonded, the resulting structure is still weak. The explanation given was that as the ends of the strips are loaded, stresses build up at the edges of the bond. The stress concentration leads to a peeling off of one strip from the other, and the bond behaves as if it were brittle. This may be analogous to the case of using only roll refined fibers to form a sheet. The pulp develops substantial breaking length. The fibers in the sheet appear to be bonded well, as indicated by decrease in light scattering coefficient (Fig. 30). However, the bonds still behave in a brittle fashion for large strain levels. When fines are added, they will be present at the fiber-fiber interfaces (72) and impart a different quality to the bonds. Instead of bonding two S_1-S_1 layer surfaces together, a pseudo- S_2-S_2 architecture may result because of the fines. This S_2-S_2 architecture provides a means for allowing large local strains (at the microscopic level) to occur, thus distributing the stress over the whole surface of the bond. Thus, fines in the bonded region help increase the tensile strength of a sheet and, based on such an interpretation, this should be the major effect for the behavior of the results observed in this section of the thesis.

Figure 33(B) helps to illustrate why the fines may contribute less to the paper properties when added to conventionally beaten long fibers than when added to unbeaten fibers. In several studies (27,29,71,75) the relative importance of the fines, both in consolidation and in the dry sheet under load, was reduced because of the modifications in the properties of the fibers due to beating. They reasoned a smaller fraction of the fines in the sheet is active in the bonded regions because of fiber collapse. Also, the mobility of the fines may be lower in the wet sheet of collapsed, swollen, and externally fibrillated fibers compared to the more open structure in a wet sheet of unbeaten fibers.

However, the results of the fines addition experiments for this thesis show the same effect whether the fines are combined with unrefined or roll refined pulps. The reasoning here is that the external surfaces of the fiber cell wall of the roll refined fibers are very similar to unbeaten fibers - both in physical components and chemical makeup. Therefore, the magnitude of the effect of fines addition should be the same on unrefined and roll refined samples, which it is.

To provide a qualitative comparison of the sheets under discussion in the results section, a series of photomicrographs of the following treatments is presented in Fig. 34; A, unrefined; B, abrasion refined; C, roll refined; D, roll refined with 16% fines added; E, combination roll refined and abrasion refined; and F, Valley refined for 40 minutes.

A summary of the results of the different refining actions on fiber and sheet properties is given in Table II.

To conclude the section on the relative importance of refining actions on paper properties (especially paper strength), the following statements are given:

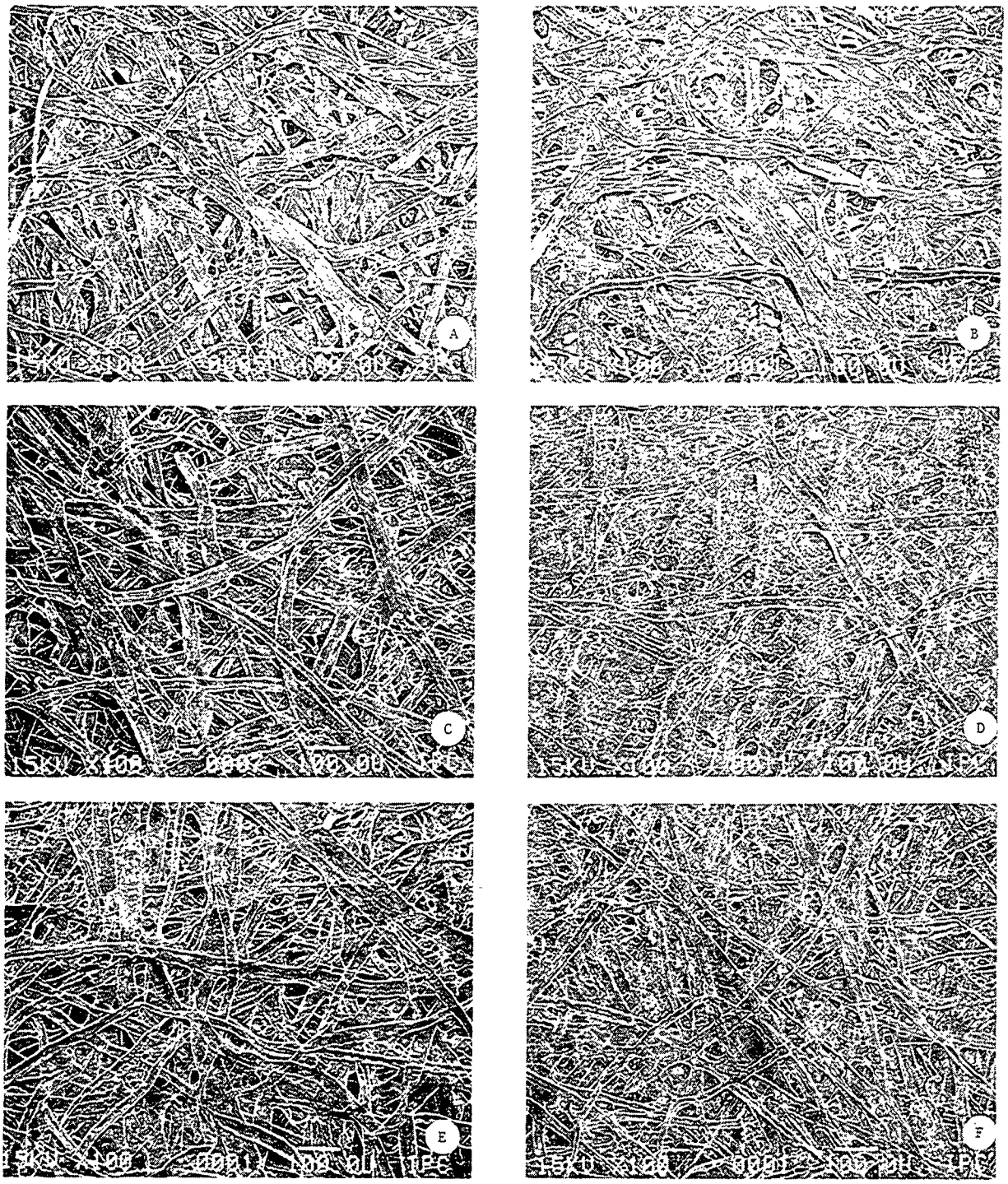


Figure 34. Scanning electron micrographs of the sheet surfaces: A, unrefined; B, abrasion refined, 5 min., 1000 rpm; C, roll refined, 500 revolutions; D, roll refined, 500 revolutions plus fines addition; E, roll refined, 500 revolutions, abrasion refined, 5 minutes, 1000 rpm; F, Valley refined, 40 minutes.

- Internal fibrillation is the single most important refining effect for making dense, strong sheets.

- Fines are a required supplement to internally fibrillated fibers in order to achieve Valley beaten strength levels. They act to improve bonding and increase the breaking length in the dry sheet.

- The effect of external fibrillation was of no advantage in improving the breaking length or tear resistance of a dry sheet.

TABLE II

RESULTS OF REFINING EXPERIMENTS ON PRIMARY PROPERTIES

	Refining Treatment						
Property	Un-refined	Roll Refined	Abrasion	Roll and Abrasion	Roll and Fines	Valley	Escher-Wyss Low Intensity
Internal fibrillation	--	Y	--	Y	Y	Y	Y
External fibrillation	--	N	Y	Y	N	Y	Y
Fines	--	--	--	--	Y	Y	Y
Shortening	--	N	N	N	N	Y	Y
Zero-span breaking length, km	12.3	15.7	11.9	16.0	13.8	16.6	--
Breaking $length_{max}$, km	2.01	5.77	2.28	5.75	7.62	8.02	6.45
TAPPI $density_{max}$, g/cu cm	0.527	0.702	0.556	0.685	0.728	0.702	0.693
Spec. scattering Co_{max}, cm^2/g	339	171	375	232	189	199	246

CONCLUSIONS

The following conclusions may be drawn from the results of this thesis:

1. The primary effect on pulp fibers of the repeated compressive action of the roll refiner is internal fibrillation.
2. Internal fibrillation appears to be the most influential refining effect on paper property development. Major paper strength development (70% of the breaking length achieved in the Valley beater) is possible with internally fibrillated fibers. Properties are developed at low net refining energy consumptions.
3. It is possible to impart the refining effects in pulp fibers in isolated steps. Combining internal fibrillation with fines produces a sheet that is equivalent in paper properties to a sheet from Valley beaten pulp. External fibrillation in addition to internal fibrillation does not increase the strength properties of a dry sheet.
4. Energy savings and better utilization of materials will probably be the result of separating refining into selective actions.

FUTURE WORK

A number of interesting questions have arisen from the results of this thesis which may be used as guidelines for future work.

Fiber and sheet properties can be developed by the action of the roll refiner. But why do these properties reach an upper limit and remain constant at higher levels of roll refining? Is it because the method of energy input has reached a maximum at approximately 50 kWh/t (i.e., should the fibers be slurried and reformed to make a new network)? Or, is it because the upper limit on property development for internally fibrillated fibers has been reached and other refining effects are required to produce the desired end result?

When the other effects of refining (fines, external fibrillation) were added to the fibers, some interesting paper property results were observed. Additional questions arise: Is the difference between the results due entirely to interfiber bonding? What interfiber bonding mechanism is causing the difference?

Now that the equipment exists to impart widely different effects in pulp fibers (roll refiner, abrasion refiner), thought could be given to in which order to perform the treatments or how much of each treatment should be used.

ACKNOWLEDGMENTS

This thesis is a culmination of the efforts of many people. For their direct involvement, my sincere appreciation is extended to my Thesis Advisory Committee; Dr. Douglas Wahren (Chairman), Dr. Earl Malcolm, and Dr. Gary Baum. I thank you for your advice, guidance, and timely encouragement.

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APPENDIX I

DESCRIPTION OF ROLL REFINER

Figure 35 is a schematic of the roll refiner with dimensions. The basic components of the apparatus are the vertical support columns, A; the horizontal support beam, B; the refining roll, C; the support roll, D; the oscillator motor and eccentric, E; loading mechanism, F; the support roll bearing, G; and the drive mechanism.

Prior to roll refining, the fibers are formed into a sheet 10 x 50 cm, which is placed on the polished chrome surface of the support roll. The refining roll has grooves machined into its surface to break up the line contact between the two rolls into discrete segments. Subdividing the roll surface and oscillating it along its axis allows new configurations of fibers to be refined each time they enter the nip. The refining roll is oscillated approximately the distance of one groove width (1.2 mm) by means of an eccentric fitted on the shaft of a gear-reduced motor. The oscillation amplitude insures complete coverage of all areas of the sheet on the support roll. Evidence to support full coverage of the fiber mat was obtained by gluing NCR paper on the support roll and operating the refiner and oscillator. The dye capsules were ruptured throughout the areas contacting the refining roll, indicating complete coverage.

The refining roll is mounted in high quality ball bearings which allow the roll to rotate freely as it rides on the sheet. Measurements showed that the amount of slippage between the two rolls was less than 0.1%. The bearing housings for the refining roll are mounted on the horizontal beams, which are hinged to the vertical support columns at one end and connected via a rod at the loading end.

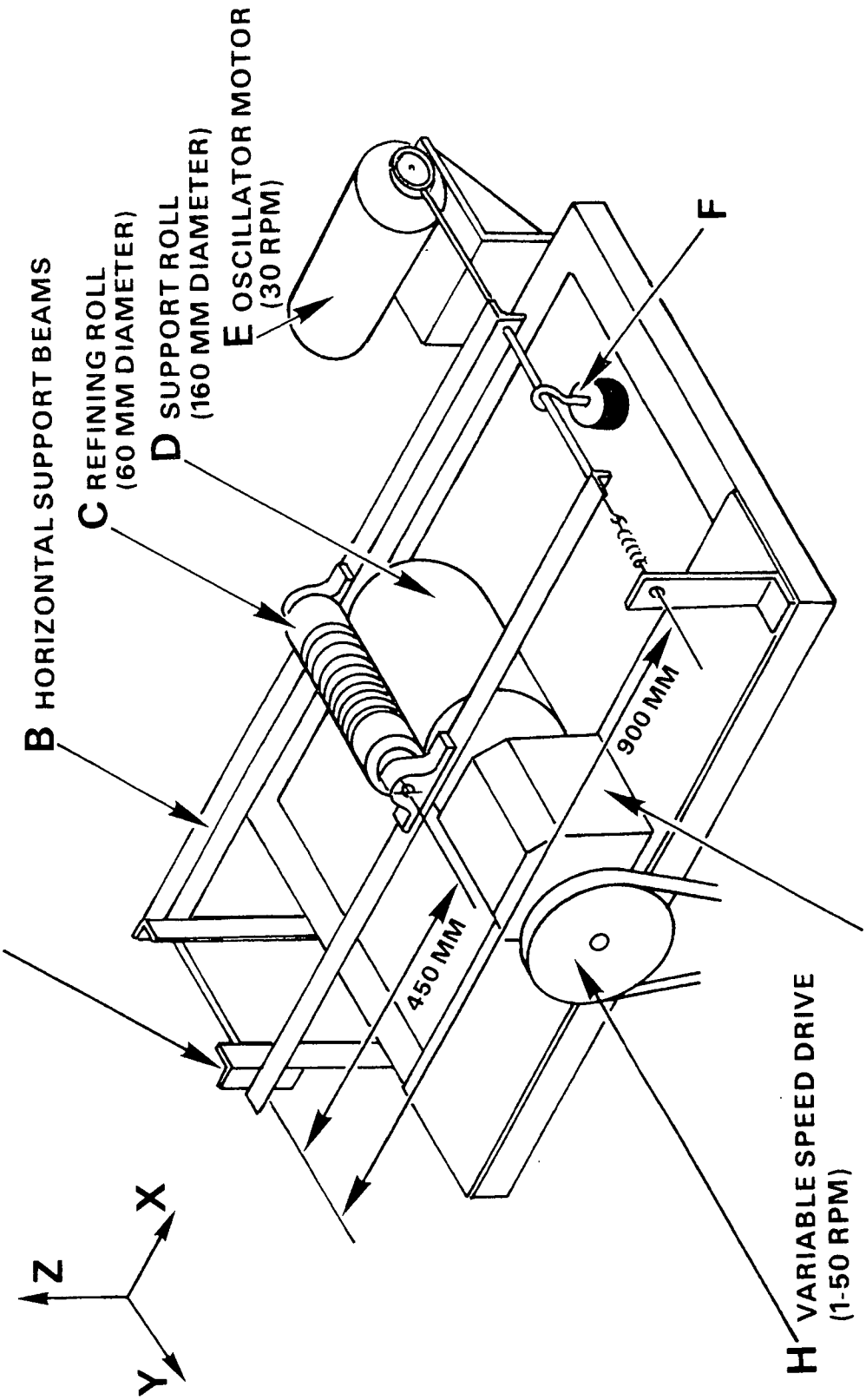

Figure 35. Schematic of the roll refiner.

Alignment of the refining roll location to top dead center of the support roll was possible with a screw-type adjustment of the support roll bearing. The adjustment was made by adding weights to the mass hangar and monitoring the deflection of the vertical support columns. If the roll centers were not aligned, the top roll would be pushed (or pulled) out of the "at rest" position with the addition of the weights. Corrections in the position of the support roll were made until deflections in the support columns were minimal. After the corrections, roll centers remained aligned with the addition of weights and at any speed of operation.

The radii of the rolls that form the nip (30 mm and 80 mm) are large relative to the thickness of the sheet being refined (0.15 mm). As mentioned above, the slippage differential between the rolling surfaces was negligible. Hence, deformation of the sheet was mainly by compression, and the shear deformation was small - at least on a macroscopic scale.

The vertical support columns were machined to be relatively flexible, thus allowing for some deflection in the direction of motion of the support roll at the line of contact with the top roll. Note: The principal directions x, y and z are shown in Fig. 35. The slight deflection in the columns was measured with a Linear Variable Displacement Transducer (LVDT), and the measurements were utilized to get a reading of the tangential force between the rolls. The units of displacement were calibrated against a known force which was applied by pulling on the ends of the horizontal beams in the direction tangential to rolls. Figure 36 is the calibration curve for deflection of the support columns as a function of applied force.

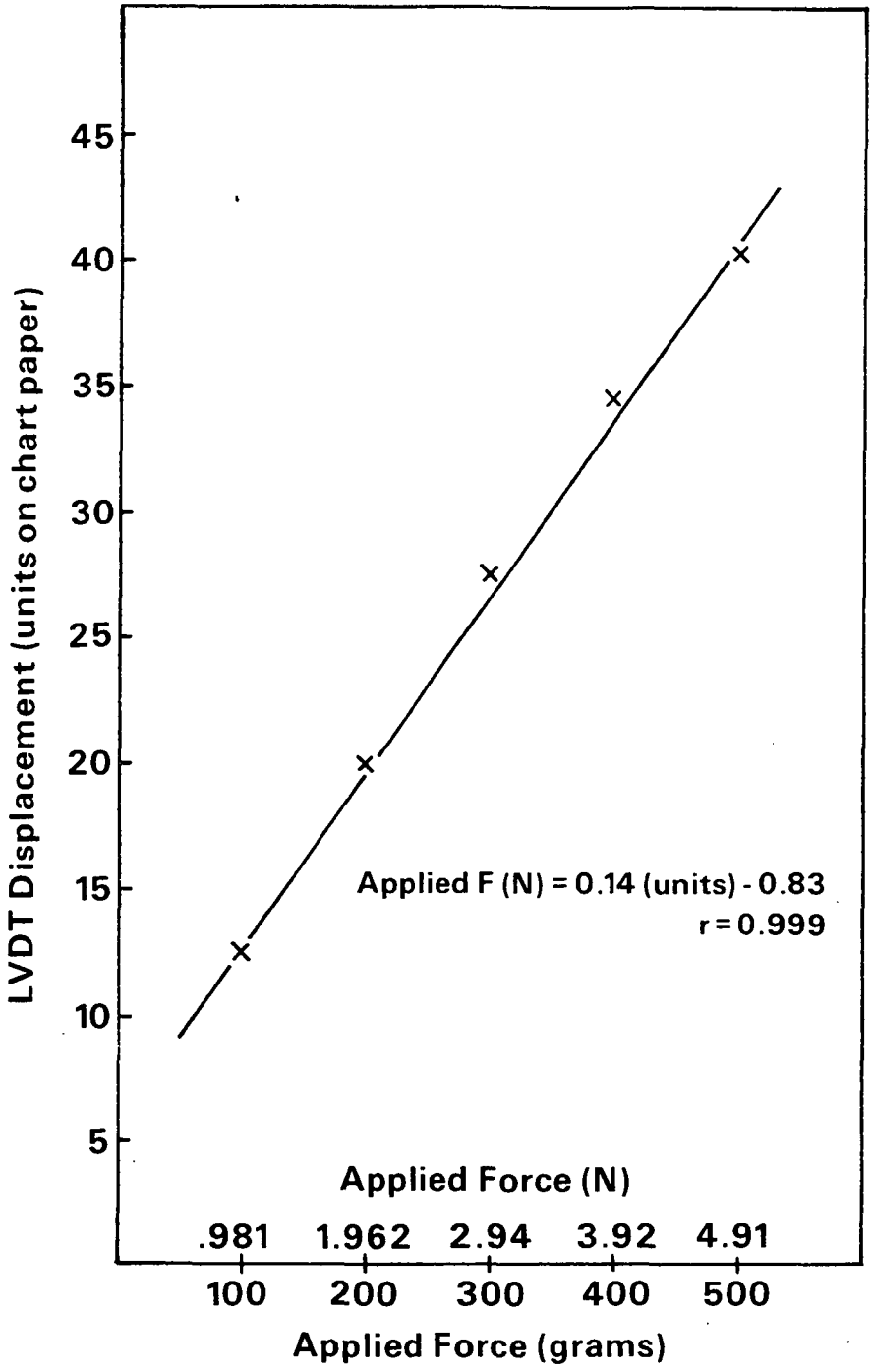

Figure 36. Calibration curve x-direction force.

ENERGY MEASUREMENT

X-DIRECTION COMPONENT

The energy consumed in a particular experiment could be measured. In vector notation, Work, W, is equal to

$$W = \vec{F} \cdot \vec{r} \tag{8}$$

where $\vec{F}$ is a force vector and $\vec{r}$ is a distance vector. The dot product of these vectors is:

$$\vec{F} \cdot \vec{r} = F \cdot r \cos \theta \tag{9}$$

where F is the magnitude of $\vec{F}$, r is the magnitude of the distance vector, and θ is the angle between the two vectors. Since the force is measured tangential to the surface of the two rolls, it is also parallel to the direction of motion. The angle between $\vec{F}$ and $\vec{r}$ is zero, and the work consumed in a particular experiment is equal to:

$$W = F \cdot r \tag{10}$$

$$\text{Since} \quad \theta = 0 \text{ and } \cos 0 = 1$$

F was measured as described above and the distance, r, is a product of the support roll circumference and the number of revolutions of the support roll.

An actual recorded trace of the force using a small sheet, which covered only part of the support roll circumference, is shown in Fig. 37. The numbers in the figure represent the difference in tangential force, F_T, with and without a sheet between the rolls. This difference is directly attributable to the presence of fibers in the nip. The relationship used to incorporate the change in force and the total displacement during a particular run is

$$F_T \cdot (9.81 \text{ m/sec}^2) \cdot (0.16\pi \text{ m/rev}) = 4.93 \, F_T \text{ J/rev} \quad . \tag{11}$$

Y-DIRECTION COMPONENT

Work done on the fibers due to oscillation of the refining roll at right angles to the main motion was determined according to the same force through a distance

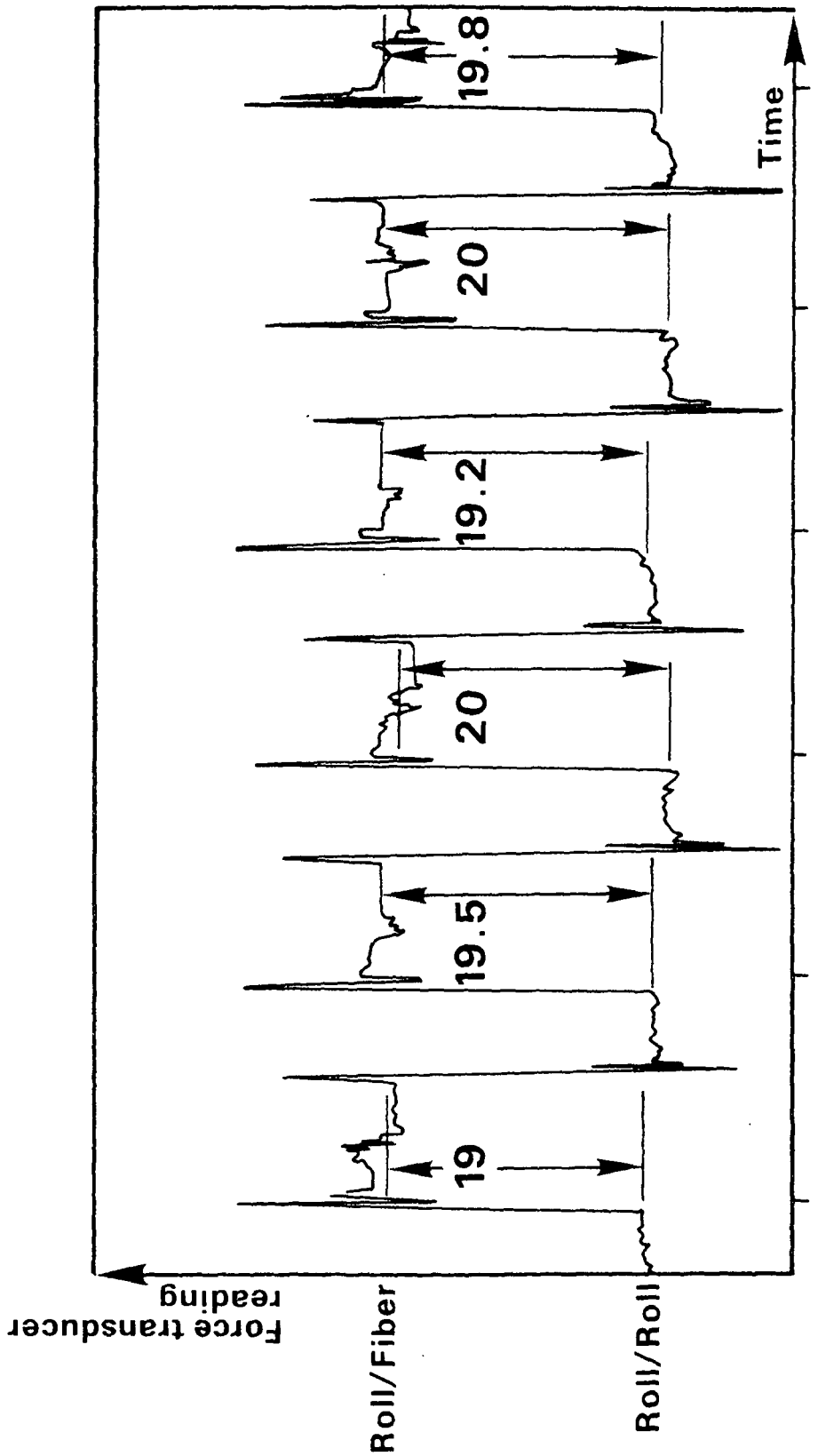

Figure 37. Recorder printout for x-direction force measurement.

principle described in the previous section. However, the measuring arrangement was different. Two pieces of spring steel were used to attach the refining roll shaft to the horizontal support beam. Figure 38 is an end view of the arrangement, looking directly into the nip. The armature of the LVDT was threaded into one of the pieces of spring steel, and the LVDT was positioned to measure relative movement between the horizontal beam and the shaft of the roll refiner. When the refiner was in operation, the transducer traced out a signal of the deflection of the spring steel as a function of time. The deflection of the spring steel support was calibrated against known applied forces (see Fig. 39). The range used in the actual refining experiments went up to about 30N. Within this range, the calibration curve is linear with a correlation coefficient of $r = 0.998$.

The force needed to move the refining roll across the fibers in the y-direction was not constant. Both the force and the displacement varied approximately sinusoidally with time (Fig. 40). For approximately sinusoidal variations, a diagram like Fig. 41 would result. The enclosed area would be equal to the work done (or consumed) per cycle.

The measurement of force as a function of displacement could not be made, however, without some expense. But it was observed that the force and the displacement were very nearly in phase. Hence, the area representing work in the diagram would be a very small fraction of the product of force and displacement amplitudes, exemplified in Fig. 42. This latter product could easily be calculated. From the recorder trace and calibration curve, the peak-to-peak force amplitude was 32.7N and the peak-to-peak displacement was 1.168×10^{-3} m. The upper bound on energy consumed in the y-direction is

$$32.7\text{N} \cdot 1.168 \times 10^{-3}\ \text{m} = 0.04\ \text{J}. \qquad (12)$$

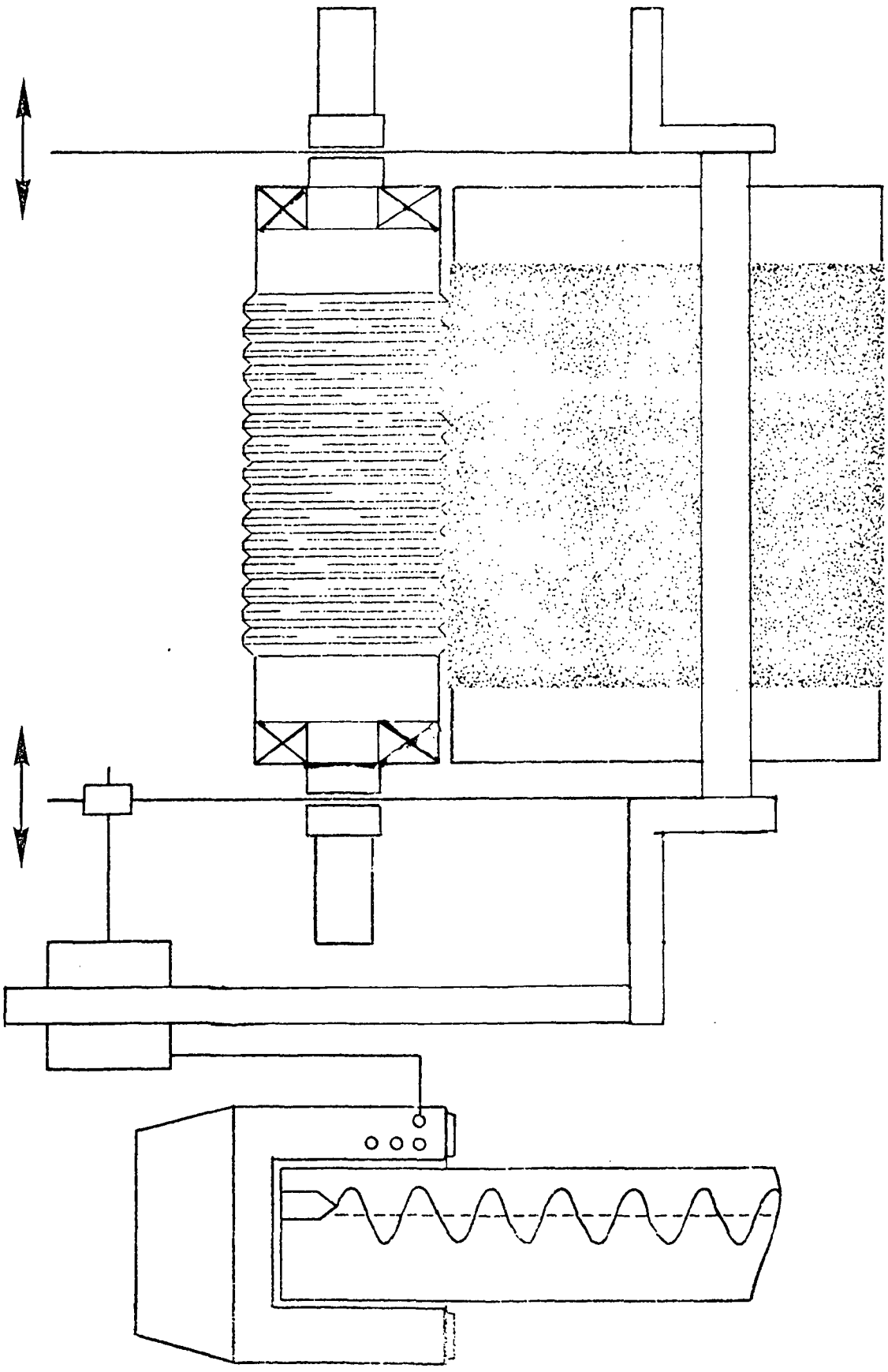

Figure 38. Schematic for force measurement in the y-direction.

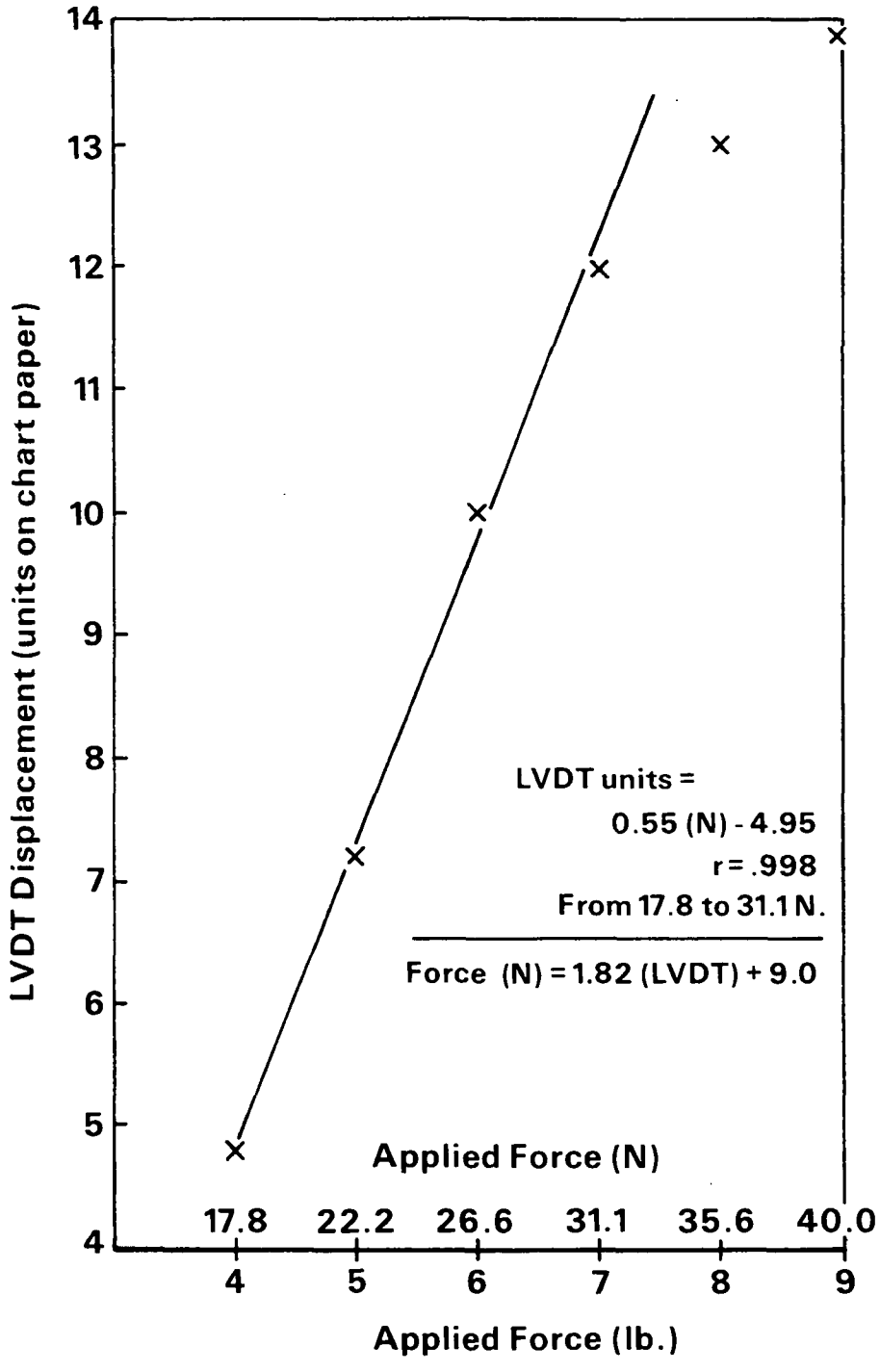

Figure 39. Calibration curve y-direction force.

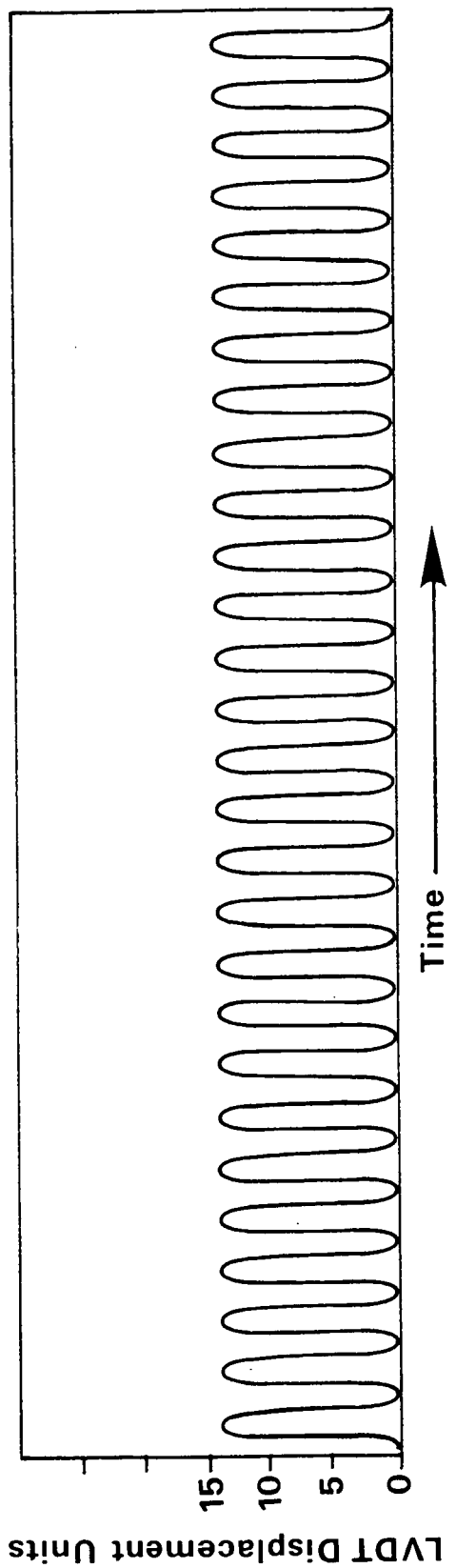

Figure 40. Recorder printout for y-direction force measurement.

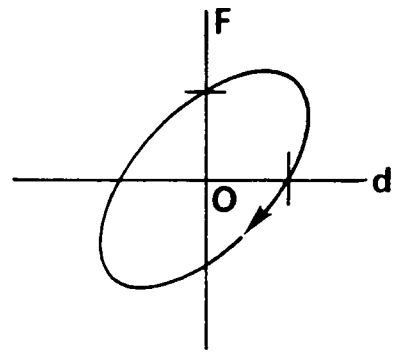

Figure 41. Force-displacement diagram for an oscillating motion which is out of phase.

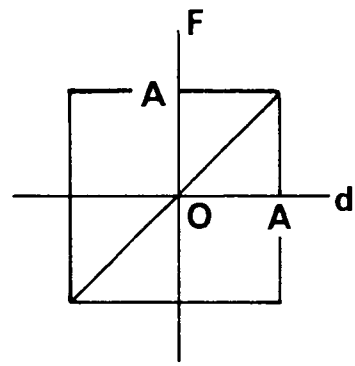

Figure 42. Product of force and displacement amplitudes for an oscillating motion.

Since the rotational speed of the y-direction oscillator was 30 rpm and that of the support roll was 8.5 rpm, the upper bound on energy consumed in the y-direction per revolution of the support roll was

$$0.04 \text{ J} \times \frac{30 \text{ rpm}}{8.5 \text{ rpm}} = 0.135 \text{ J}. \qquad (13)$$

This value represents approximately 14% of the energy expended in the x-direction at a normal load of 245N. Increasing the load on rolls against the fibers did not change the apparent force amplitude more than approximately 7% over the full range of loads. This is an indication that the main part of the recorded force amplitude

represents elastic displacement of the spring steel connected to the transducer. The work consumed in one-half of a cycle is almost completely recovered each cycle. Hence, the work input in the y-direction is estimated to be one-tenth of the upper bound calculated in Eq. (13). Over the full range of normal loads, the fraction of work input in the y-direction ranges from less than 1 to a maximum of 5% of the work input in the x-direction. Included in the total work input column in Table III is a y-component of input of 0.014 J/revolution.

Z-DIRECTION COMPONENT

The energy input in the z-direction is insignificant. The theoretical maximum would be represented by the vertical force, i.e., the load, times a vertical displacement equal to the sheet thickness. With maximum load, 245N, and the maximum sheet thickness, 0.14 mm, the total work done during an entire experiment would be 0.03 J. This is an insignificant contribution, which has been disregarded.

TABLE III

ENERGY USAGE CALCULATIONS

Revs.	N Force	T Force	Mass	Energy, J/g	Energy, kWh/t
50	15	0.035	1.03	9.05	2.52
50	33	0.26	0.775	83.6	23.2
100	12	0.03	1.03	15.7	4.36
100	23	0.185	1.03	89.9	25
100	33	0.26	0.775	167.2	46.4
100	25	0.19	3.1	30.67	8.52
100	12	0.03	3.1	5.2	1.45
125	25	0.19	3.1	38.3	10.64
200	54	0.43	3.1	137.7	38.24
200	15	0.035	1.55	24	6.69
250	25	0.19	3.1	76.7	21.3
500	12	0.03	1.03	78.6	21.8
500	25	0.19	3.1	153.4	42.5
925	12	0.03	3.1	48.3	13.4
1000	25	0.19	3.1	306.7	85
1200	12	0.03	6.2	31.3	8.7
2000	12	0.03	3.1	104.5	29
2000	25	0.19	3.1	613.3	170.4
2500	25	0.19	3.1	766.6	212.9
4000	25	0.19	3.1	1226.7	340.7

Revs. = number of revolutions of the support roll.
N Force = normal force on the refining roll, in kg.
T Force = tangential force, in kg.
Mass = mass of fibers on the support roll.
Energy, J/g = energy input to the fibers, in J/g.
= Revs. x (0.014 J + 4.93 T Force)/mass.
Energy, kWh/t = energy input to the fibers, in kWh/t.
= (energy, J/g)/3.6.

APPENDIX II

ABRASION REFINER

Abrasive wear occurs when a rough, hard surface slides against a softer surface and plows out fragments from it (43). For abrasive wear to occur in pulp suspension flows, the fibers must make contact with the abrading surface and move relative to it when contact is made. A schematic of the experimental apparatus used to achieve the abrasive effect in pulp fibers is shown in Fig. 43.

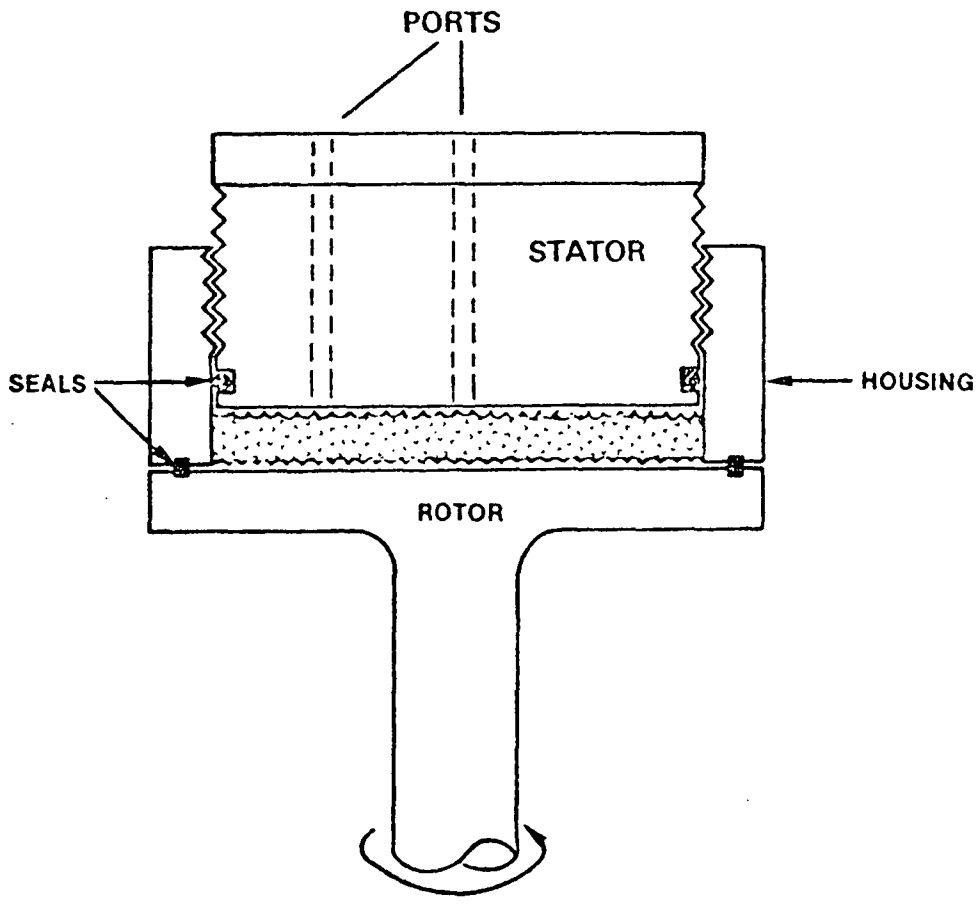

Figure 43. Schematic of the apparatus; stator diameter = 102 mm.

The rotor was made from stainless steel and was driven by a 1/2 hp variable speed motor. Rotor speeds of up to 1100 rpm were possible. The stator, made from Plexiglas to aid flow visualization, was threaded into a steel housing which was

loaded against the rotor through a Teflon seal. The internal diameter of the chamber so formed was 102 mm. The threads in the housing allowed the clearance between the rotor and stator to be adjusted to any setting.

To aid the removal of any air bubbles that were trapped in the chamber after the addition of pulp, two ports were located in the stator, one of which was at the center. When the suspension was sheared the trapped air bubbles migrated toward the chamber center where they were removed through the center port by displacement with water introduced at the outer port.

To promote abrasive wear the roughness of the rotor/stator surfaces was altered by attaching silicon carbide sandpaper with various grit sizes. The sandpaper used ranged from a 400 grit, which had a grit size of approximately 38 μm or less, to 60 grit, which had a grit size of 240 μm or less. Two pulp abrasion experiments were performed on the bleached spruce sulfite pulp. A previously unrefined sample was treated in the abrasion refiner for five minutes at 1000 rpm. The grit size in use was 120 and the clearance was 4 mm. One gram samples were treated each time.

The remaining sample received a combination of treatments. One gram samples of a sample that had been previously roll refined for 500 revolutions were abraded for 5 minutes at 1000 rpm, a clearance of 4 mm and a 120 grit size.

APPENDIX III

ESTABLISHING OPERATING CONDITIONS OF THE ROLL REFINER

A list of the main variables in this study is presented in Table IV. The goal of this section was to establish the operating conditions necessary to achieve roll refined pulp fibers. The first level of experiments were carried out to establish useful ranges of the following variables: speed of the rolls, sheet moisture during refining and applied load.

TABLE IV

MAIN VARIABLES

A. Primary independent variables

- rpm
- Moisture during refining
- Normal load
- Basis weight of laboratory sheet
- Fiber orientation
- Roll type
- No. of revolutions
- pH

B. Primary dependent variables

1. Refiner variables
   - Energy variables
   - Refining gap
2. Paper variables
   - Density
   - Breaking length
   - Zero span breaking length
   - Air resistance
   - Modulus
   - Tear factor
   - Light scattering coefficient
3. Other Characteristics
   - Canadian standard freeness
   - Fiber length
   - Scanning Electron Micrography
   - Scanning Transmission Electron Micrography
   - Polarized light micrography

REFINER SPEED

The roll refiner was equipped with a variable speed controller. The range of roll surface speeds was 0.8 to 42 cm/sec, which corresponded to 1 and 50 rpm, respectively. To minimize dynamic losses, a low speed was desired. However, production of ample amounts of fibers for testing was also desired. One other constraint imposed on the roll speed was the effect of the y-direction oscillator. The frequency of the oscillator was fixed at 30 cycles per minute. To prevent a fixed phase relationship from developing between the x- and y-direction motions (which would cause standing wave patterns in the sheet) the support roll frequency was chosen to be 8.5 rpm (7 cm/sec). This frequency combination proved to be acceptable because not one experiment was aborted due to excessive speed or standing waves in the sheet.

TEMPERATURE AND MOISTURE

Sheet moisture during refining was controlled with a cold (21 ± 1°) room humidifier. The humidifier was fitted with a nozzle to distribute the mist uniformly across the width of the sheet. The output of the humidifier was controlled with a Variac. During refining, the sheet on the roll was very sensitive to moisture ratios (MR's) outside the range 2.33 to 2.6, which correspond to consistencies of 30% and 28%, respectively. At high MR's, the sheet was very weak and could be easily disrupted. At MR's below 2.3, the moisture level could not be controlled with this simple equipment arrangement and the sheet became dry. Hence, the sheet moisture was controlled within narrow limits, 2.3 - 2.6, for all experiments. After equilibration for approximately 5 minutes the sheet temperature was the same as the mist temperature.

NORMAL LOAD AND PRESSURE

Initial experimentation was carried out to find the range of loads and pressures that could be applied to the fibers. The loads were developed by increasing the weight on the lever arm of the apparatus. Pressures in the nip were varied by altering the contact area of the refining roll and the sheet. Three rolls with different contact areas were tested. A numerical value for ranking the effectiveness of each roll was determined from the formula:

$$P = \frac{1}{R_1} + \frac{1}{R_2} \tag{14}$$

where P = ranking factor

R_1 = major roll radius

R_2 = radius of curvature of the working zone.

Each of the rolls had major radii of 30 mm, but the radii of the contact surface varied in width from 0.05 mm to ∞. The resulting rating factors are 1, 25, and 600 which reflect the relative "sharpness" of the working surface of the roll.

The refining effect of roll numbers 600 and 1 was monitored by forming standard handsheets and placing them on a smooth platen. The refining rolls were rolled over the sheets at the same normal load. The fibers were collected, slurried, and were formed and tested. The results in Table V show the yield stress of the fibers must have been reached with the 600 roll because the breaking length has decreased when compared with the unrefined sample. The density of the sheets did not increase with the sharp roll, indicating the fibers were probably being cut. Further experimentation was done using rolls with lower ranking factors.

The normal load on the refining roll was taken as high as 500N (52 kg). However, the number of loading cycles on the sheet became the constraint against using this

high level of loading. The handsheets would degrade rapidly at high loads, preventing uniform treatment of all the fibers. To get the benefit of long refining runs, loads were kept below 30 kg. More elaborate experiments with the working surfaces and the optimum loading conditions are described later in this appendix.

TABLE V

EFFECT OF WORKING SURFACE GEOMETRY ON SHEET PROPERTIES

	Unrefined	Roll 1	Roll 600
Density, g/cu cm	0.499	0.640	0.463
Breaking length, km	2.34	4.79	1.97

EFFECT OF pH

Experiments were completed to determine if the roll refiner should be operated at a pH level other than neutral. Formette sheets were made at tap water conditions (pH 7.5). The wet blotters were saturated with buffer solutions (pH 4.0, 7.0, 10,0), the handsheets were placed between the blotters and were stored for 24 hours at these conditions. (The pH of the handsheet at each level was checked prior to refining by squeezing a small volume of solution out of the sheet.) During refining, the water normally used in the humidifier was replaced with the buffer solutions. The sheets were refined for 500 revolutions and the results from the experiments are shown in Table VI.

The results show the changes in paper properties are insignificant as a function of pH. In fact, the weakest sheet is found at the pH 10 level. With no apparent gain in sheet properties by refining at nonneutral pH's, the choice was made to carry out all experiments at tap water conditions. Machine cleanliness was also a problem when using the buffer solutions.

TABLE VI

EFFECT OF pH ON ROLL REFINED PULP

	pH		
	4.0	7.0	10.0
Basis weight, g sq m	60.4	60.6	62.8
Breaking length, km	5.77	5.92	4.94
TAPPI density, g/cc	0.657	0.658	0.648
IPC density, g/cc	0.840	0.847	0.824
Zero span, km	14.4	14.4	14.5
Specific scattering co., cm^2/g	220	219	230
Tear factor	101	107	126
Modulus, GPa	9.67	9.93	8.89

OPTIMIZATION EXPERIMENTS

The second block of experiments involved determining the specific operating conditions necessary to achieve a significant degree of roll refining. The variables investigated were the refining nip geometry, refining load, the number of loading cycles, and the orientation of the fibers in the network placed on the refining roll. The sheet density, breaking length, elastic modulus, zero-span breaking length and light scattering coefficient were properties chosen to monitor the effect of varying the operating parameters.

A half-factorial experiment was carried out using the parameters shown in Table VII. The two levels of each parameter were used. By taking a high and a low value for each parameter, there are 2^{4-1} or 8 possible combinations of 4 variables. Each trial was run in duplicate and the combinations are summarized in Table VIII.

TABLE VII

OPERATING PARAMETERS USED IN FACTORIAL EXPERIMENTS

Parameter	"-" Level	"+" Level
Working surface geometry[a]	25	1
Normal Load, kg	12	25
Revolutions	100	500
Fiber Orientation[b]	MD	CD

[a]Working surface geometry is equivalent to ranking factor of the rolls.

[b]Fiber orientation of the handsheets was measured ultrasonically and the ratio V^2_{MD}/V^2_{CD} is used to designate orientation.
For a MD sheet, orientation is 5.0.
For a CD sheet, the same sheet was placed on roll 90° different to MD, to give orientation of 0.2.

TABLE VIII

EXPERIMENTAL ARRAY

	A	B	C	D
Trial No.	Working Surface Geometry	Normal Load	No. of Revs.	Fiber Orientation
I	-	-	-	-
II	+	-	-	+
III	-	+	-	+
IV	+	+	-	-
V	-	-	+	+
VI	+	-	+	-
VII	-	+	+	-
VIII	+	+	+	+

After each experiment was run at the prescribed settings, analysis of variance in the measured responses was analyzed using two methods; a statistical computer program (BMD02V)(76) and a hand computation using Yates algorithm. Details are given in Davies (77) and Box, Hunter, and Hunter (78).

The results of the computer ANOVA were compared to F-test values at two levels of confidence to determine which main effects and interactions were significant. The effects of the operating parameters on the measured responses are found in Tables IX-XIII. A criticism of the computer analysis was that it did not indicate which level of a given parameter was desirable; it only gave which parameter was significant. The Yates method did provide such information. Analysis of variance using Yates algorithm was computed and the comparative results are printed in Table XIV.

TABLE IX

EFFECT OF FACTORIAL EXPERIMENT[a] ON IPC DENSITY

Grand mean = 0.7124 g/cu cm

F_{table} at $0.95_{1,8}$ confidence level = 5.32

Source of Variation	d.f.	S.S.	M.S.	F_{calc}[b]	Significance 0.95
A	1	0.00636	0.00636	11.2	Yes
B	1	0.00551	0.00551	9.6	Yes
C	1	0.03177	0.03177	55.7	Yes
D	1	0.00139	0.00139	2.4	No
AB	1	0.00032	0.00032	--	No
AC	1	0.00059	0.00059	--	No
BC	1	0.00074	0.00074	--	No
Within replicates	8	0.00456	0.00057	--	No
Total	15	0.05124			

F_{table} at $0.99_{1,8}$ confidence level = 11.3

Significant factors at this level = C, the number of revolutions

---

[a]Variables
A = Working surface geometry.
B = Normal load.
C = Revolutions.
D = Fiber orientation.

[b]$F_{calc} = \frac{\text{M.S. effect}}{\text{M.S. estimate}}$.

TABLE X

EFFECT OF FACTORIAL EXPERIMENT[a] BREAKING LENGTH

Grand mean = 2.975 km

F_{table} at $0.95_{1,8}$ confidence level = 5.32

Source of Variation	d.f.	S.S.	M.S.	F_{calc}[b]	Significance 0.95
A	1	0.66522	0.66422	10.0	Yes
B	1	0.64000	0.64000	9.7	Yes
C	1	3.29422	3.29422	49.9	Yes
D	1	0.84640	0.84640	12.8	Yes
AB	1	0.06302	0.06302	--	No
AC	1	0.60040	0.60040	--	No
BC	1	0.04203	0.04203	--	No
Within replicates	8	0.52710	0.06589	--	
Total	15	6.01740			

F_{table} at $0.99_{1,8}$ confidence level = 11.3

Significant factors at 0.99 = C: revolutions
= D: fiber orientation

See Table IX for footnotes.

TABLE XI

EFFECT OF FACTORIAL EXPERIMENT[a] ON ELASTIC MODULUS

Grand mean = 6.512 GPa

F_{table} at $0.95_{1,8}$ confidence level = 5.32

Source of Variation	d.f.	S.S.	M.S.	F_{calc}[b]	Significance 0.95
A	1	2.74731	2.74731	10.6	Yes
B	1	2.73076	2.73076	10.6	Yes
C	1	17.91406	17.91406	69.8	Yes
D	1	1.77556	1.77556	6.8	Yes
B	1	0.20026	0.20026	--	No
AC	1	0.06376	0.06376	--	No
BC	1	0.04951	0.04951	--	No
Within replicates	8	2.05345	0.25667		
Total	15	27.53464			

F_{table} at $0.99_{1,8}$ confidence level = 11.3

Significant factors at 0.99 = C: revolutions

See Table IX for footnotes.

TABLE XII

EFFECT OF FACTORIAL EXPERIMENT[a] ON ZERO SPAN BREAKING LENGTH

Grand mean = 14.338 km

F_{table} at $0.95_{1,8}$ confidence level = 5.32

Source of Variation	d.f.	S.S.	M.S.	F_{calc}[b]	Significance 0.95
A	1	0.0400	0.0400	--	No
B	1	0.1225	0.1225	--	No
C	1	1.5625	1.5625	3.75	No
D	1	1.2100	1.2100	3.02	No
AB	1	0.0900	0.0900	--	No
AC	1	0.0900	0.0900	--	No
BC	1	0.2025	0.20251	--	No
Within replicates	8	3.2000	0.4000		
Total	15	6.5175			

F_{table} at $0.99_{1,8}$ confidence level = 11.3

No significant factors

See Table IX for footnotes.

TABLE XIII

EFFECT OF FACTORIAL EXPERIMENT[a] ON SPECIFIC LIGHT SCATTERING COEFFICIENT

Grand mean = 263.06 cm^2/g

F_{table} at $0.95_{1,8}$ confidence level = 5.32

Source of Variation	d.f.	S.S.	M.S.	F_{calc}[b]	Significance 0.95
A	1	500.6	500.6	--	No
B	1	9550.2	9550.2	--	No
C	1	23186.2	23816.2	6.669	Yes
D	1	3953.3	3953.3		No
AB	1	2654.8	2654.8		No
AC	1	2249.1	2249.1		No
BC	1	2263.4	2263.4		No
Within replicates	8	28565.3	3570.7		
Total	15	73552.9			

F_{table} at $0.99_{1,8}$ confidence level = 11.3

No significant factors

See Table IX for footnotes.

TABLE XIV

ANALYSIS OF VARIANCE - COMPARISON OF CALCULATION METHODS

	Variable[a] with Significant Effect on Response from each ANOVA Method		
	BMD02V		
Measured Response	95% confidence	99% confidence	Yates Method
IPC density	A, B, C	C	+A, +B, +C
Breaking length	A, B, C, D	A, D	+A, B, +C, -D
Elastic modulus	A, B, C, D	C	+A, B, +C, -D
Zero span breaking length	None	None	None
Specific light scattering coefficient	C	None	+A, +B, +C

[a]Variables
A = Working surface geometry.
B = Normal load.
C = Revolutions.
D = Fiber orientation.

SUMMARY

The following operating conditions were used in the refining experiments. The support roll was driven at 8.5 rpm (7 cm/sec). A normal load level of 25 kg was applied to the fibers at the nip of the two rolls. The refining roll chosen was #25, with a 1.2 mm radius of curvature at the working surface. The fibers were oriented in the machine direction at a 5.0 MD/CD velocity ratio. The pH of the system was 7.5. The number of revolutions was used as the primary independent variable.

APPENDIX IV

PROCEDURES FOR SHEETMAKING

This procedure, Institute Method 411, deals with a method of forming sheets from pulps before or after beating (TAPPI Standard 205).

Normally, the beating procedure will specify the disintegration, if any. Without further treatment, sufficient slush stock to give the desired number of sheets is diluted to 0.15% consistency and sheets are formed.

DISINTEGRATION

Make the mixture up to 2000 mL with cold water (1.2% consistency) and disintegrate for 75,000 revolutions in the standard disintegrator with the propeller running at 3000 rpm in the stock. After disintegration, dilute the stock to 16 liters with cold water (0.15% consistency).

SHEETMAKING

Partially fill the sheet machine container with water, taking care that the needle valve is closed. Then, let out the water until its level is just above the wire, so as to ensure driving out all the air from beneath the wire. Accurately measure out and pour into the container 800 mL of the well-mixed dilute stock at 0.15% consistency, the water supply being simultaneously turned on until the depth of the dilute suspension is 35 cm above the wire surface. Note: The ultimate requirements are sheets each weighing between 1.14 and 1.26 grams (dry), corresponding to a tolerance of 5%. In removing the stock for each sheet, it is preferable to agitate the suspension with a small Lightnin' stirrer unless the pulp has a low freeness. The first sheet of each set shall be couched, dried, and weighed in the

heated balance. Calculate the consistency of the diluted stock and the amount required to give a sheet weight of 1.20 grams (oven dry).

Insert the perforated stirrer and in six seconds move it steadily down and up six times, keeping the disk beneath the level of the liquid. Repeat this double movement very slowly, once in 10 seconds, and gently withdraw the stirrer. After a pause of 10 seconds, during which time the surface of the liquid should have become almost motionless, fully open the drain cock of the machine with a rapid movement and let all the water drain from the sheet under suction.

Immediately after the water has drained from the sheet, tilt the container and couch the sheet off the wire. Unless the machine is of the new pattern with an air-inlet groove at the side of the grid plate, open the needle valve before couching.

COUCHING

Lay two pieces of standard blotting paper centrally on the pulp sheet on the wire. Now lay the flat brass couch plate centrally on the blotters and place the brass couch roll gently on the middle of the plate. Move the roll backward, no other pressure being applied to within less than 1/4 inch of the edge of the plate, which is maintained horizontal by placing the fingers on the opposite edge; then forward to within less than 1/4 inch of the front edge; then back and forth four times; and finally to the middle, then lift off the roll. The time taken to perform the five complete rolls shall be about 20 seconds.

Remove the pulp sheet and blotters and covering brass plate from the wire in a manner similar to that of opening the cover of a book, when the sheet will be found adhering to the blotter next to it (the couch blotter). Immediately detach the couch blotter and sheet from the other blotter (couch filler) and plate and, by

means of the press template, lay it centrally, test sheet uppermost, on one dry blotter (the press filler) which has been previously placed in the press. Place a mirror-polished plate on the test sheet and cover with another press filler, ready to receive the next couch blotter and sheet.

Make the next test sheet in precisely the same way. Make a minimum of five sheets in all so that the stack in the press, when complete, will consist of press filler, couch blotter, sheet and plate, repeated five times. In most instances, only five sheets need be prepared for the complete physical testing of the pulp at any degree of beating. The maximum number of sheets that may be pressed at one time shall be 15. Lay a single blotter on the uppermost plate to avoid possible damage to the press cover.

FIRST PRESSING

Put on the cover of the press and screw the wing nuts down hand tight. Raise the pressure, as indicated by the gage, to 50 pounds per square inch in 1/2 minute from the time the needle begins to move, and maintain it there for five minutes. At the end of that time, release the pressure and remove the press cover.

SECOND PRESSING

After the first pressing, remove the polished plates with test sheets attached from the stack of moist blotters. Using the press template, lay the plate that was on top of the stack during the first pressing centrally on a dry blotter, test sheet uppermost, and cover the sheet with one new blotter. This blotter also serves as a base for the next plate and test sheet. The stack so built on a foundation blotter will comprise plate, sheet, and press filler, repeated for each sheet. The order of the sheets is thus reversed in the second pressing. Place the cover in position as before and raise the pressure rapidly to 50 pounds per square inch as indicated by

the gage and keep it there for two minutes. (There is no necessity to fix a time period for attaining the stipulated pressure in the second pressing, as the tendency for the pressure to fall off is very much less than before.) During couching and pressing, the felt sides of the blotters and not the wire sides shall be in contact with the sheets; and, for precise work, it is desirable that those in contact with the sheet be new blotters.

For the wet pressing study, identical times and sequences were followed as stated above, except the wet pressing pressures were increased to 100 to 250 psi.

DRYING

Remove the stack from the press and fit the plates with the attached test sheets into a set of drying rings, each test sheet uppermost and in contact with the rubber of the next ring, and place a heavy weight on top of the pile. Do not allow the sheets to dry below the normal moisture content reached by drying in an atmosphere at 50% relative humidity and 23°C. It is advisable to place the pile of rings in the constant humidity room and dry and condition them concurrently. The sheets shall be fully dried in this position before removal.

If there is a shortage of drying rings when making sheets from relatively free stock, it is possible to clamp two sheets, with their plates back to back, between each pair of drying rings.

CONDITIONING

The sheets shall be kept in an atmosphere of 50 ± 2% relative humidity and 73 ± 3°F until they attain equilibrium as to moisture content, which shall be determined by accurate weighings at not less than half-hour intervals. With a suitable arrangement for circulating conditioned air over the sheets, they may be dried and conditioned in six hours.

APPENDIX V

PROCEDURES FOR SHEET TESTING

The sheets were tested according to the Institute paper testing methods unless otherwise specified. The IPC methods are adapted from TAPPI 220.

WEIGHT PER UNIT AREA

The sheets were individually weighed and the average weight, W, of five sheets was computed. After later determining the moisture content, M, of the sheets, the weight per unit area (oven dry basis) was determined from the following formula.

$$\text{Basis Weight, gsm} = \frac{50\ W\ (100 - M)}{100} \qquad (15)$$

THICKNESS (CALIPER)

The thickness of the standard handsheets was determined using TAPPI method (Institute method 508) and the IPC soft platen method. The IPC soft platen method involves placing the test specimen between two soft rubber sheets. The sheets were each 1/32 inch thick and made of soft Neoprene rubber described as having a 5-10 durometer test result. The thickness of this rubber-paper-rubber sandwich was measured using an instrument manufactured at The Institute of Paper Chemistry. This instrument, referred to here as the "IPC thickness gage", has been described in an unpublished report by Hardacker (79). The rubber-paper-rubber sandwich was placed between the platens of the IPC thickness gage, and weight was applied to adjust the clamping pressure. The thickness of the sandwich was determined at a clamping pressure of 50 Kpa.

Calibration of the instrument was completed before each test by placing a flat metal plate of known thickness between the two pieces of rubber and measuring the

sandwich thickness. The thickness of the paper sample was determined by assuming the rubber contributed equally to the measured thickness in each test.

SHEET DENSITY

Density values computed by the TAPPI method are termed TAPPI densities, and values found using the IPC soft platen method are called IPC densities. From measurements of the thickness (both methods) and the weight of each specimen, the density of each set of samples can be calculated as follows:

$$\text{Density} = \frac{\text{Oven Dry Basis Weight, g/sq m}}{\text{Thickness (microns)}} = \text{g/cu cm} \qquad (16)$$

The thickness of the paper samples determined by the IPC method always results in a lower value than by the TAPPI method. This observation is the result of the fact that the rubber tends to conform to the uneven surface of the sheet whereas the hard metal platens record only the thickness of the higher areas of the surface. This results in the measured IPC density always being greater than the pressured TAPPI density.

ELASTIC MODULUS

The elastic modulus values for the standard handsheets were determined using an ultrasonic technique developed at the Institute. Because the method and apparatus have been described (80), only a brief description will be given here.

In general, the mechanical wave velocities are determined by measuring the time required for a pulse of sine waves to propagate through the sheet in a given direction. The time-of-flight was determined by measuring the time interval between the triggering of the wave pulse in a frequency generator and the detection of the pulse

after it has traveled a measured distance through the sheet. The electrical equipment used to measure these delay times has been described by Mann (81), the only change being the addition of an APPLE minicomputer for data acquisition and manipulation.

Usually the delay times were measured to the first peak in the received wave train. To determine the velocity, delay times were determined as a function of the distance the wave traveled through the sheet. For each velocity measurement, the delay time was determined at each of three distances ranging from 30 to 50 mm. At each transducer separation distance, the delay time was measured at 10 to 20 positions in the center of the sheet. This reduced any effects due to the edge of the sheet. The average of the delay times determined at each transducer separation was used to calculate the velocity.

Assuming paper to be two-dimensional, two velocities are required to determine the two Young's moduli (Ex and Ey) (82). These two values can be estimated from the longitudinal velocities in the x- and y-directions (Vx and Vy). The modulus values reported in the results section of this study are determined from the ultrasonic velocities as follows:

$$\frac{\rho_{IPC}\, V_x^2 + \rho_{IPC}\, V_y^2}{2} = \text{Elastic Modulus} \qquad (17)$$

SPECIFIC SCATTERING COEFFICIENT

The Technidyne TB-1 Brightness Color Opacity meter was used to measure the specific scattering coefficient of each of the paper samples. The single sheet reflectance, R_o, and the thick pad reflectance, R_∞, were found using illuminant A (Y) at a wavelength of 572 nm. The basis weight was obtained by punching out a

3.71 cm^2 disk of the sample area where the measurement was taken. Thus, knowing R_o, R_∞, and W, the following formula (in SCABA computer program) was used to find the specific scattering coefficient s.

$$sW = \frac{R_\infty}{1-R_\infty^2} \ln \left(\frac{1 - \Omega R_\infty^2}{1 - \Omega}\right) \text{ where } \Omega = R_o/R_\infty \qquad (18)$$

$$s = sW/W$$

AIR RESISTANCE

The air resistance of the sheets made from the beaten pulps was determined with the Gurley method. The results are reported as the length of time it takes to pass 100 mL of air through the sheet.

Following the nondestructive testing, the sheets were cut up to perform the remaining destructive testing. Figure 44 shows a method of dividing the test sheets.

TENSILE PROPERTIES (INSTITUTE METHOD 511)

The test span of the tensile tester was set at 5.0 inches and the testing speed used was 0.5 in/min. The chart speed on the recorder was 20 in/min. The specimens were clamped, one at a time, in the tester and the load-elongation relationship was obtained.

$$\text{Breaking Length} = (\text{km}) \frac{100 \times \text{tensile reading, kg/cm}}{\text{Basis weight g/m}^2} \qquad (19)$$

TEARING STRENGTH (INSTITUTE METHOD 512)

Clamp five sheets together in the tear tester, positioning the sheets to obtain four equally spaced tear tests across the sheets. Make two tests with the smooth side of the sheets toward the instrument and two with the opposite orientation.

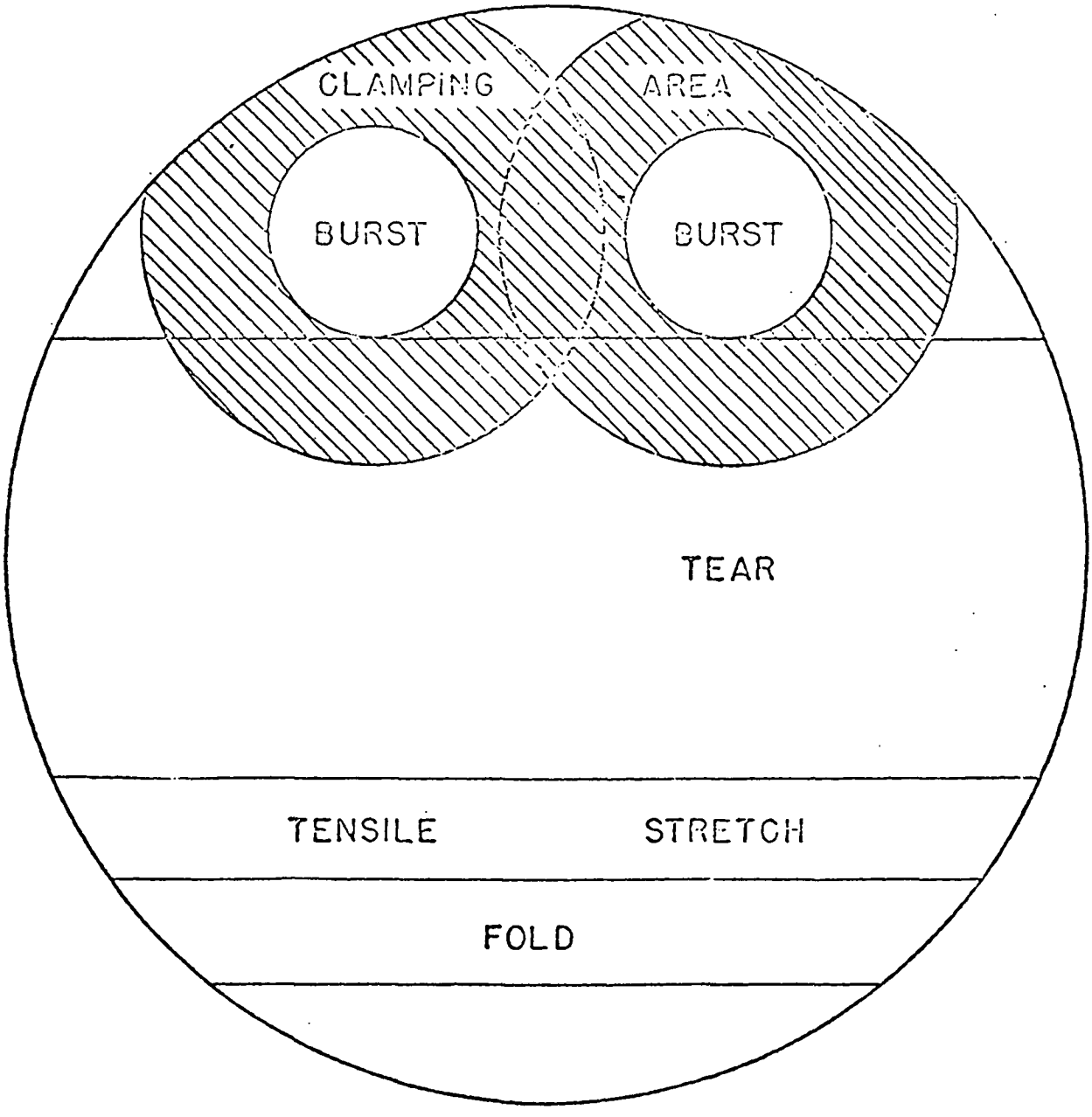

Figure 44. Division of sheet for testing.

Calculate the average tear, e, as g/sheet by multiplying the average instrumental reading by 3.2, and the tear factor from:

$$\text{Tear Factor} = \frac{100 \times \text{Tear (g/sheet)}}{\text{Basis Weight (g/m}^2\text{)}} \qquad (20)$$

ZERO SPAN BREAKING LENGTH

Using a 1.0 gram handsheet, sample disks are cut and weighed to insure accurate basis weight. The samples are clamped in the IPC zero span tensile tester and tested. The results are reported as km breaking length.

MOISTURE CONTENT (INSTITUTE METHOD 506)

Determine the moisture content from remnants of the test sheets (specimens from tear test are suitable). Weigh the moisture specimen in the 50% RH environment, place in weighing bottles with covers removed in a circulating oven at 105°C for 16 hours, cover the bottles and remove from oven, allow to cool 15 to 30 minutes, carefully loosen covers to equalize pressure, weigh, and finally determine tare weight of bottles. Calculate moisture content as a percentage of the air dry weight of the sample.

APPENDIX VI

TABULATED DATA

This appendix contains tabulated data from all of the refining experiments completed for this thesis. Tables XV-XVII contain the results of the roll refining, Valley beating, abrasion refining, and fines addition experiments discussed in the body of the thesis. The remaining tables contain data from preliminary experiments, optimization experiments and the summary experiments.

REFINER TYP = Type of refiner, listed in the table

BASIS WT = Basis weight of the sheets, in g/sq m

TAPPI T = TAPPI hard platen thickness, in microns

IPC T = IPC soft platen thickness, in microns

TAPPI DENS = TAPPI density, in g/cu cm.

IPC DENS = IPC density, in g/cu cm.

BREAK LENS = Sheet breaking length, in km

ZERO SPAN = Zero span breaking length, in km

SPEC SCT CO = Specific light scattering coefficient, in cm^2/g

TEAR FACT = Tear factor

POROSITY = Gurley air resistance, in sec/100 mL

MODULUS = Elastic modulus, in GPa

CSF = Canadian Standard Freeness, in mL

ENERGY = Specific refining energy input, in kWh/t

REF TIME = Refining time for Valley beater samples only, in min.

NET GSM = Network basis weight used in the roll refiner, in g/sq m

REVS = Revolutions in the roll refiner

LOAD = Normal load on the refining roll, in kg; or specific edge load in Escher Wyss refiner, in Ws/m

TABLE XV

VALLEY BEATEN PULP

Beating Time, minutes	0	10	20	30	40
Breaking length, km	1.02 ± 0.08	3.86 ± 0.19	6.02 ± 0.23	7.60 ± 0.2	8.02 ± 0.4
Tear factor	73 ± 3	137 ± 5	104 ± 3	85 ± 4	79 ± 3
Density, g/cu cm	484 ± 0.031	0.582 ± 0.020	0.643 ± 0.019	0.680 ± 0.019	0.702 ± 0.130
Gurley, sec/100 mL	3.4 ± 0.20	4.7 ± 0.23	19 ± 1.6	107 ± 5.2	408 ± 17
Specific scattering coefficient, cm^2/g	354 ± 6	301 ± 7	256 ± 4	220 ± 6	199 ± 5
Zero span breaking length, km	11.1 ± 0.6	13.9 ± 0.9	15.5 ± 0.7	15.8 ± 6	16.6 ± 0.9

TABLE XVI

ROLL REFINED PULP

Number of Loading Cycles	0	125	250	500	1000	2000	4000
BL	2.01 ± 0.09	4.21 ± 0.31	4.67 ± 0.18	5.72 ± 0.23	5.23 ± 0.34	5.76 ± 40	5.74 ± 0.52
Tear factor	122 ± 17	143 ± 0	125 ± 4	101 ± 3	120 ± 2	102 ± 3	106 ± 4
Density	0.527 ± 0.020	0.629 ± 0.012	0.637 ± 0.019	0.663 ± 0.018	0.654 ± 0.030	0.664 ± 0.015	0.702 ± 0.035
Gurley	3.4 ± 3	5.0 ± 1	6.0 ± 2	7.3 ± 2	5.94 ± 4	8.0 ± 4	13.3 ± 9
SSC	340 ± 4	249 ± 7	239 ± 8	214 ± 6	229 ± 3	201 ± 4	171 ± 37
ZST	12.3 ± 0.4	14.5 ± 8	14.9 ± 5	15.7 ± 6	14.2 ± 7	15.5 ± 1.1	14.9 ± 2.0

TABLE XVII

COMBINED TREATMENTS

	Unrefined	Roll Refined, 500 cycles	Abrasion Refined	Unrefined Fines Added	Roll Refined Abrasion Refined, 500 cycles	Roll Refined Fines Added, 500 cycles
BL	2.01 ± 0.09	5.72 ± 0.23	2.28 ± 0.12	3.88 ± 0.23	5.75 ± 0.61	7.61 ± 0.59
Tear factor	122 ± 17	101 ± 3	119 ± 6	197 ± 6	118 ± 7	99 ± 3
Density	0.527 ± 0.021	0.663 ± 0.018	0.556 ± 0.021	0.587 ± 0.019	0.685 ± 0.009	0.728 ± 0.012
Gurley	3.4 ± 0.1	7.3	196	102	39.8	624 ± 20
SSC	340 ± 10	214 ± 6	375 ± 10	365 ± 7	232 ± 7	189 ± 8
ZST	12.3 ± 1	15.7 ± 6	11.9 ± 0.5	12.3 ± 3	16.0 ± 4	13.8 ± 0.6

Set No. Variable	Refiner Type	Basis Weight	TAPPI T	IPC T	TAPPI Dens.	IPC Dens.
1	Roll 1	72.5	132.5	0	0.55	0
2	Roll 1	78.8	126	0	0.621	0
3	Roll 1	73.5	119	0	0.623	0
4	Roll 150	68.5	147	0	0.463	0
5	Roll 1	72	146.5	0	0.499	0
6	Valley	0	0	0	0.588	0
7		0	0	0	0.671	0
8		0	0	0	0.714	0
9		0	0	0	0.74	0
10	Escher Wyss	0	0	0	0.507	0
11	Low S.E.L.,	0	0	0	0.567	0
12	KCL	0	0	0	0.601	0
13		0	0	0	0.641	0
14		0	0	0	0.663	0
15		0	0	0	0.693	0
16		0	0	0	0.614	0
17		0	0	0	0.656	0
18	Escher Wyss	0	0	0	0.507	0
19	High S.E.L.	0	0	0	0.583	0
20		0	0	0	0.628	0
21		0	0	0	0.709	0
22		0	0	0	0.761	0
23		0	0	0	0.815	0
24		0	0	0	0.602	0
25		0	0	0	0.647	0
26		0	0	0	0.709	0
27		0	0	0	0.605	0
28	Valley, KCL	0	0	0	0.651	0
29		0	0	0	0.69	0
30		0	0	0	0.73	0
31		0	0	0	0.735	0
32		0	0	0	0.768	0
33		0	0	0	0.655	0
34		0	0	0	0.724	0
35		0	0	0	0.742	0
36	Roll 1	66.71	139.7	117.6	0.478	0.567
37	Roll 1	72.37	129.5	99.9	0.559	0.724
38	Roll 1	55.91	101.1	74	0.553	0.756
39	Roll 1	55.1	100.8	70.6	0.546	0.78
40	Roll 1	59.91	0	78.5	0	0.765

Set Numbers	Conditions
1-5	Roll refined. Preliminary experiments on Douglas-fir pulp.
6-9	Valley beater. Domtar 090 spruce kraft pulp supplier data.
10-17	EW refined, KCL. Low intensity (specific edge load = 1.00 Ws/m). Bleached spruce sulfite.
18-26	EW refined, KCL. High intensity (specific edge load = 3.0 Ws/m).
27-35	Valley beater, KCL. Bleached spruce sulfite.
36-42	Roll refined. Initial results at various operating conditions.

Set No. Variable	Breaking Length	Zero Span	Spec. SCT Co.	Tear Factor	Porosity	Modulus
1	3.19	0	309	0	0	0
2	4.79	0	278	0	0	0
3	5	0	257	0	0	0
4	1.97	0	325	0	0	0
5	2.34	0	365	0	0	0
6	3.9	0	0	240	0	0
7	10.6	0	0	118	0	0
8	11.5	0	0	105	0	0
9	12.1	0	0	100	0	0
10	1.68	0	349	92.3	1	0
11	2.96	0	316	147	3	0
12	3.73	0	297	130	7	0
13	4.92	0	273	117	20	0
14	5.7	0	254	104	49	0
15	6.45	0	246	93.3	150	0
16	4.12	0	289	125	11	0
17	5.43	0	260	108	39	0
18	1.68	0	349	92.2	1	0
19	3.11	0	315	121	4	0
20	4.34	0	289	105	18	0
21	6.13	0	259	81	290	0
22	6.89	0	218	64	0	0
23	7.12	0	175	54	0	0
24	3.64	0	304	115	10	0
25	4.76	0	282	100	81	0
26	6.13	0	259	81	290	0
27	3.15	0	0	120	0	0
28	5.01	0	0	113	0	0
29	5.96	0	0	113	0	0
30	6.64	0	0	96	0	0
31	7.59	0	0	88	0	0
32	8.21	0	0	86	0	0
33	5.12	0	0	112	0	0
34	6.54	0	0	97	0	0
35	7.7	0	0	87	0	
36	1.35	0	344	0	0	2.8
37	3.59	0	249	0	0	8.32
38	3.53	0	228	0	0	8.73
39	3.53	0	218	0	0	9.36
40	5.19	0	180	115.1	0	9.05

Set Numbers	Conditions
1-5	Roll refined. Preliminary experiments on Douglas-fir pulp.
6-9	Valley beater. Domtar 090 spruce kraft pulp supplier data.
10-17	EW refined, KCL. Low intensity (specific edge load = 1.00 Ws/m). Bleached spruce sulfite.
18-26	EW refined, KCL. High intensity (specific edge load = 3.0 Ws/m).
27-35	Valley beater, KCL. Bleached spruce sulfite.
36-42	Roll refined. Initial results at various operating conditions.

Set No. Variable	CSF	Energy In	Ref. Time	Net GSM	Revs.	Load
1	0	25	0	20	50	30
2	0	36	0	20	100	50
3	0	100	0	20	200	30
4	0	50	0	20	100	30
5	0	0	0	20	0	0
6	668	0	0	0	0	0
7	550	0	32	0	0	0
8	450	0	50	0	0	0
9	300	0	70	0	0	0
10	725	0	0	0	0	1
11	700	50	0	0	0	1
12	625	100	0	0	0	1
13	525	200	0	0	0	1
14	387	300	0	0	0	1
15	260	400	0	0	0	1
16	600	150	0	0	0	1
17	425	230	0	0	0	1
18	725	0	0	0	0	3
19	650	50	0	0	0	3
20	525	100	0	0	0	3
21	212	200	0	0	0	3
22	87	300	0	0	0	3
23	30	400	0	0	0	3
24	600	71	0	0	0	3
25	425	123	0	0	0	3
26	212	300	0	0	0	3
27	0	0	10	0	0	0
28	0	0	20	0	0	0
29	0	0	30	0	0	0
30	0	0	40	0	0	0
31	0	0	60	0	0	0
32	0	0	90	0	0	0
33	0	0	21	0	0	0
34	0	0	39	0	0	0
35	0	0	66	0	0	0
36	0	0	0	15	0	0
37	0	25	0	15	50	25
38	0	50	0	15	100	25
39	0	85	0	30	200	55
40	0	42	0	60	925	12

Set Numbers	Conditions
1-5	Roll refined. Preliminary experiments on Douglas-fir pulp.
6-9	Valley beater. Domtar 090 spruce kraft pulp supplier data.
10-17	EW refined, KCL. Low intensity (specific edge load = 1.00 Ws/m). Bleached spruce sulfite.
18-26	EW refined, KCL. High intensity (specific edge load = 3.0 Ws/m).
27-35	Valley beater, KCL. Bleached spruce sulfite.
36-42	Roll refined. Initial results at various operating conditions.

Set No. Variable	Refiner Type	Basis Weight	TAPPI T	IPC T	TAPPI Dens.	IPC Dens.
41	Roll 1	60.21	0	83.3	0	0.722
42	Roll 1	60.41	0	76.8	0	0.786
43	Valley	60.93	0	88.3	0.55	0.69
44	IPC	60.32	0	78.5	0.555	0.768
45	(Aane spruce)	61.22	0	74.7	0.57	0.82
46		60.8	0	70.2	0.6	0.866
47	Roll 1	55.1	0	78.8	0	0.699
48		52.9	0	74.2	0	0.713
49		59.96	0	96.1	0	0.624
50		59.11	0	91.8	0	0.644
51		63.3	0	93.4	0	0.678
52		64.4	0	92.9	0	0.672
53		62.16	0	79.7	0	0.78
54		63.38	0	86.3	0	0.734
55	Roll 25	50.98	0	64.6	0	0.789
56		58.04	0	74.9	0	0.774
57		60.36	0	93.1	0	0.648
58		57	0	86.9	0	0.656
59		58.77	0	77.7	0	0.756
60		68.11	0	80.26	0	0.825
61		62.12	0	90.4	0	0.687
62		61.57	0	83.4	0	0.738
63	Roll 25, 50 psi	62.6	0	83.2	0.69	0.757
64	100 psi	66.5	94.61	76.4	0.703	0.87
65	250 psi	65.79	91.31	72.1	0.72	0.912
66	Roll 25, 50 psi	63.2	91.31	75.5	0.692	0.837
67	100 psi	64.3	90.67	71.1	0.709	0.904
68	250 psi	63.42	87.02	67.5	0.729	0.94
69	Valley, 10 min 50 psi	60.93	0	88.3	0.55	0.69
70	100 psi	58.76	95	77.3	0.618	0.76
71	250 psi	62.38	94.61	73.2	0.659	0.852
72	Valley, 20 min 50 psi	60.32	0	78.5	0.55	0.768
73	100 psi	56.32	84.07	67	0.67	0.84
74	250 psi	60.18	84.71	65.4	0.71	0.92
75	Valley, 30 min 50 psi	61.23	0	74.7	0.57	0.82
76	100 psi	59.51	84.96	65.8	0.7	0.904
77	250 psi	59.68	82.55	63.1	0.723	0.946
78	Valley, 40 min 50 psi	60.8	0	70.2	0.6	0.866
79	100 psi	59.5	83.82	63.3	0.71	0.94
80	250 psi	61.79	83.19	61.2	0.743	1.01

Set Numbers	Conditions
43-46	Valley beater (IPC).
47-62	Roll refined. Half-factorial experiments - four variables at two levels.
63-65	Roll refined. 500 revs., wet pressed 50, 100, 250 psi.
66-68	Roll refined. 1000 revs., wet pressed 50, 100, 250 psi.
69-71	Valley beater. 10 min, wet pressed 50, 100, 250 psi.
72-74	Valley beater. 20 min, wet pressed 50, 100, 250 psi.
75-77	Valley beater. 30 min, wet pressed 50, 100, 250 psi.
78-80	Valley beater. 40 min, wet pressed 50, 100, 250 psi.

Set No. Variable	Breaking Length	Zero Span	Spec. SCT Co.	Tear Factor	Porosity	Modulus
41	0	0	256	142	0	6.9
42	0	0	227	149	0	9.1
43	3.93	0	283	154	0	6.6
44	5.95	0	245	106	0	8.9
45	7.25	0	230	95	0	9.5
46	7.63	0	200	86	0	10.4
47	2.66	13.2	279	144	0	6.02
48	3.07	15.2	265	157	0	6.56
49	2.27	13.8	315	133	0	4.81
50	2.34	14.3	320	135	0	4.88
51	2.25	13.6	299	135	0	5.28
52	2.39	14.5	297	139	0	5.42
53	3.89	15.2	240	164	0	8.46
54	3.3	14.5	260	172	0	7.33
55	3.65	14.2	224	140	0	8.32
56	3.47	14.6	237	164	0	7.6
57	2.23	13.7	311	133	0	5.05
58	2.24	13.5	311	133	0	5.18
59	3.86	14.8	249	147	0	7.73
60	3.53	15.5	250	155	0	8.54
61	2.92	14.2	296	160	0	5.92
62	3.53	14.6	264	155	0	7.07
63	3.6	14.8	250	168	0	7.89
64	5.19	13.5	211	121	7.9	8.07
65	5.06	14.1	207	114	9.2	8.88
66	5.38	14	216	113	7.1	8.82
67	5.64	13.3	198	105	10.9	10.58
68	5.47	14.7	191	103	12.7	10.31
69	3.63	13.9	295	153.6	4.7	6.6
70	3.73	12.9	283	143	5.2	7.02
71	4.08	13.8	264	133	7.4	7.25
72	5.65	15.4	249	106	19.4	6.1
73	5.78	15.2	245	100	13.56	8.9
74	5.77	14.4	223	98	18.2	9.87
75	7	15.8	230	95	107	9.5
76	7.05	15	230	89	67	10.21
77	7.44	15.4	206	82	64	10.93
78	7.63	16.6	212	86	408	10.4
79	8.06	14.6	200	78	264	11.28
80	8.03	15.3	191	74	376	10.79

Set Numbers	Conditions
43-46	Valley beater (IPC).
47-62	Roll refined. Half-factorial experiments - four variables at two levels.
63-65	Roll refined. 500 revs., wet pressed 50, 100, 250 psi.
66-68	Roll refined. 1000 revs., wet pressed 50, 100, 250 psi.
69-71	Valley beater. 10 min, wet pressed 50, 100, 250 psi.
72-74	Valley beater. 20 min, wet pressed 50, 100, 250 psi.
75-77	Valley beater. 30 min, wet pressed 50, 100, 250 psi.
78-80	Valley beater. 40 min, wet pressed 50, 100, 250 psi.

Set No. Variable	CSF	Energy In	Ref. Time	Net GSM	Revs.	Load
41	0	25	0	120	1200	55
42	0	75	0	60	2000	55
43	650	0	10	0	0	0
44	550	0	20	0	0	0
45	380	0	30	0	0	0
46	250	0	40	0	0	0
47	0	0	0	60	500	12
48	0	0	0	60	500	12
49	0	0	0	60	100	12
50	0	0	0	60	100	12
51	0	0	0	60	500	12
52	0	0	0	60	500	24
53	0	0	0	60	100	24
54	0	0	0	60	100	24
55	0	0	0	60	500	12
56	0	0	0	60	500	12
57	0	0	0	60	100	12
58	0	0	0	60	100	12
59	0	0	0	60	500	24
60	0	0	0	60	500	24
61	0	0	0	60	100	24
62	0	0	0	60	100	24
63	0	52.25	0	60	500	25
64	0	52.25	0	60	500	25
65	0	52.25	0	60	500	25
66	0	52.25	0	60	1000	25
67	0	52.25	0	60	1000	25
68	0	52.25	0	60	1000	25
69	650	0	10	0	0	0
70	650	0	10	0	0	0
71	650	0	10	0	0	0
72	550	0	20	0	0	0
73	550	0	20	0	0	0
74	550	0	20	0	0	0
75	380	0	30	0	0	0
76	380	0	30	0	0	0
77	380	0	30	0	0	0
78	250	0	40	0	0	0
79	250	0	40	0	0	0
80	250	0	40	0	0	0

Set Numbers	Conditions
43-46	Valley beater (IPC).
47-62	Roll refined. Half-factorial experiments - four variables at two levels.
63-65	Roll refined. 500 revs., wet pressed 50, 100, 250 psi.
66-68	Roll refined. 1000 revs., wet pressed 50, 100, 250 psi.
69-71	Valley beater. 10 min, wet pressed 50, 100, 250 psi.
72-74	Valley beater. 20 min, wet pressed 50, 100, 250 psi.
75-77	Valley beater. 30 min, wet pressed 50, 100, 250 psi.
78-80	Valley beater. 40 min, wet pressed 50, 100, 250 psi.

Set No. Variable	Refiner Type	Basis Weight	TAPPI T	IPC T	TAPPI Dens.	IPC Dens.
81	Roll 7	66.95	102.4	84.2	0.654	0.794
82		65.89	98.72	80.7	0.702	0.889
83		64.51	99.76	83	0.647	0.777
84		64.3	90.67	71.1	0.709	0.904
85	Valley Std.	56.02	96.3	78.07	0.582	0.718
86	Curve	62.46	129.01	106.87	0.484	0.584
87		59.98	93.3	73.47	0.643	0.816
88		60.18	88.56	67.9	0.68	0.886
89		59.31	84.49	64	0.702	0.926
90	Roll 7	60.27	114.3	96.6	0.527	0.624
91		60.45	96.18	76.87	0.629	0.786
92		61.38	96.35	76.2	0.637	0.806
93		58.68	88.5	68.93	0.663	0.851
94		61.53	92.62	70.27	0.664	0.875
95	Roll 7, pH 4.0	60.4	91.95	71.93	0.657	0.84
96	7.0	60.65	92.12	71.6	0.658	0.847
97	10.0	62.84	97.03	76.3	0.648	0.824
98	Fines 0%	63.03	98.8	77.87	0.638	0.809
99	Addition 5%	66.77	100.2	78	0.666	0.856
100	20%	76.09	104.6	77.4	0.728	0.983
101	500 revs., 60 grit	62.4	0	71.4	0.671	0.873
102	500 revs., 120 grit	61.8	0	72.4	0.685	0.853
103	Unrefined 120 g	63.3	0	95.2	0.556	0.665
104	Unrefined finer	69.9	0	97.8	0.587	0.714

Set Numbers	Conditions
81-84	Roll refined. High refining energy levels.
85-89	Valley beater. Standard curve experiment.
90-94	Roll refined. Low refining energy levels.
95-97	Effect of pH. Set 95 = pH 4.0. Set 96 = pH 7.0. Set 97 = pH 10.0.
98-100	Effect of fines addition. Set 98 = 0% addition. Set 99 = 5% addition. Set 100 = 20% addition.
101-104	Effect of abrasion. Set 101 = roll refined 500 revs., abrasion refined (60 grit). Set 102 = roll refined 500 revs., abrasion refined (120 grit). Set 103 = abrasion refined (120 grit). Set 104 = unrefined (20% fines added).

Set No. Variable	Breaking Length	Zero Span	Spec. SCT Co.	Tear Factor	Porosity	Modulus	Ref. pH
81	4.87	14.2	229	120	5.9	8.35	7.5
82	5.74	14.9	171	106	13.3	9.7	7.5
83	4.65	15	214	113	6.3	0	7.5
84	5.64	0	198	105	0	10.58	0
85	3.86	13.9	301	177	4.7	6.62	0
86	1.02	11.1	354	73	3.4	2.76	7
87	6.02	15.5	256	104	19.4	9	7
88	7.6	15.8	220	85	107.7	10.91	7
89	8.02	16.6	199	80	408	11.26	7
90	2.01	12.3	339	122	3.4	2.8	7.5
91	4.21	14.5	248	143	5	8.17	7.5
92	4.67	14.9	239	125	6	8.85	7.5
93	5.72	15.7	213	102	7.4	9.65	7.5
94	5.76	15.5	201	114	8	10.51	7.5
95	5.77	14.4	220	101	6.6	9.67	4
96	5.52	14.4	219	107	7.6	9.53	7
97	4.94	14.5	232	126	6.4	8.85	10
98	4.77	15.8	223	133	5.7	9.62	7.5
99	6.31	15.8	216	115	31.2	10.15	7.5
100	7.62	13.8	189	99	624	12.63	7.5
101	5.6	15.6	196	118	50.2	8.96	7
102	5.75	16	232	118	40	7.68	7
103	2.28	11.9	375.4	119	196	4.67	7
104	3.88	12.3	365	157	102	6.21	7

Set Numbers	Conditions
81-84	Roll refined. High refining energy levels.
85-89	Valley beater. Standard curve experiment.
90-94	Roll refined. Low refining energy levels.
95-97	Effect of pH. Set 95 = pH 4.0. Set 96 = pH 7.0. Set 97 = pH 10.0.
98-100	Effect of fines addition. Set 98 = 0% addition. Set 99 = 5% addition. Set 100 = 20% addition.
101-104	Effect of abrasion. Set 101 = roll refined 500 revs., abrasion refined (60 grit). Set 102 = roll refined 500 revs., abrasion refined (120 grit). Set 103 = abrasion refined (120 grit). Set 104 = unrefined (20% fines added).

Set No. Variable	CSF	Energy In	Ref. Time	Net GSM	Revs.	Load
81	0	104.5	0	60	1000	25
82	0	418.5	0	60	4000	25
83	700	104.5	0	60	1000	25
84	0	261.25	0	60	2500	25
85	550	0	10	0	0	0
86	707	0	0	0	0	0
87	550	0	20	0	0	0
88	380	0	30	0	0	0
89	250	0	40	0	0	0
90	0	0	0	0	0	0
91	707	13.06	0	60	125	25
92	667	26.12	0	60	250	25
93	706	52.24	0	60	500	25
94	700	209	0	60	2000	25
95	700	52.25	0	60	500	25
96	703	52.25	0	60	500	25
97	700	52.25	0	60	500	25
98	0	52.25	0	60	500	25
99	0	52.25	0	60	500	25
100	0	52.25	0	60	500	25
101	0	0	0	0	500	0
102	0	0	0	0	0	0
103	0	0	0	0	500	0
104	0	0	0	0	0	0

Set Numbers	Conditions
81-84	Roll refined. High refining energy levels.
85-89	Valley beater. Standard curve experiment.
90-94	Roll refined. Low refining energy levels.
95-97	Effect of pH. Set 95 = pH 4.0. Set 96 = pH 7.0. Set 97 = pH 10.0.
98-100	Effect of fines addition. Set 98 = 0% addition. Set 99 = 5% addition. Set 100 = 20% addition.
101-104	Effect of abrasion. Set 101 = roll refined 500 revs., abrasion refined (60 grit). Set 102 = roll refined 500 revs., abrasion refined (120 grit). Set 103 = abrasion refined (120 grit). Set 104 = unrefined (20% fines added).