GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF RESEARCH ADMINISTRATION

RESEARCH PROJECT INITIATION

Date: March 20, 1975

Project Title: Advanced Technology Applications in Garment Processing

Gr. Co

Project No: E-27-627

Principal Investigator Dr. W. D. Freeston

Sponsor: National Science Foundation

12-31-78

Agreement Period: From 1/1/75 Until 6/30/76#

#12 months budget period plus 6 months for submission of required reports
etc.

Type Agreement: Grant No. APR74-02326

Amount:

	NSF		GIT	
AE:	E-16-662	$10,895	E-16-353	#
ChE:	E-19-634	10,556	E-19-328	#
TE:	E-27-627	77,149	E-27-313	#
		$98,600		$ #

*To be determined;
tentatively, $25,000
total in 3rd year.

Reports Required:
Annual Letter
Technical, Final Report

Sponsor Contact Person (s):
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thru ORA
Mr. Gaylord L. Ellis
Grants Officer
National Science Foundation
Washington, D. C. 20550
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RA—3 (6—71)

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SPONSORED PROJECT TERMINATION

Date: _November 28, 1979_

Project Title: _Advanced Technology Applications in Garment Processing_

Project No: _E-27-627 (Sub-projects are E-16-662/Bangert/AE and E-19-634/Muzzy/ChE)·_

Project Director: _Dr. W. D. Freeston_

Sponsor: _National Science Foundation_

Effective Termination Date: _12/31/78_

Clearance of Accounting Charges: _12/31/78_

Grant/Contract Closeout Actions Remaining:

- _ Final Invoice and Closing Documents
- X Final Fiscal ~~Report~~ _Accounting_ (FCTR)
- _ Final Report of Inventions
- _ Govt. Property Inventory & Related Certificate
- ¬ Classified Material Certificate
- _ Other _____

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E-27-627

Advanced Technology Applications
in Garment Processing

by

Louis H. Bangert
John L. Lundberg
John D. Muzzy
W. Denney Freeston

Georgia Institute of Technology
Atlanta, Georgia 30332
July 1975

First Semi-annual Technical Progress Report

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in Garment Processing

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July 1975

First Semi-annual Technical Progress Report

Abstract

   New, direct, automatic, fast and inexpensive methods are being
developed for converting polymer chips and/or staple fiber into fabrics
without carding, spinning and weaving or knitting, and garments without
cutting and sewing. Other objectives are to reduce material, labor, capital,
and energy costs in textile and garment manufacture. The successful
development of one or more of these processes will contribute to improving
the United States' competitive position in textiles and clothing in the
U.S. and world markets.

   An appropriately qualified post-doctoral fellow and support personnel
have been hired. Specifications for long lead-time equipment have been
developed, and purchase orders for this equipment and selected chemical
supplies written. Equipment for planned experiments has been assembled
and is being checked out. Portions of the literature surveys being
conducted are contained herein.

   Results to date indicate that: 1) linear polypropylene is a suitable
polymer for fibrillation; 2) specific additives appear to improve the
uniformity of fibrillation. 3) A combination of additives is necessary to
produce a uniformly fibrous and porous sheet.

Table of Contents

I. Executive Summary

The various phases of the studies and the work schedule are given in Table 1. The principal areas of research during the first year are:

1. Gas-polymer solutions

2. Effects of additives, crystallization and deformation conditions on film fibrillation

3. Use of tubular biaxial stretching for fabric formation

4. Single fiber response to pressure and shear forces from a fluid stream

As can be seen from the work plan, the program proposed is inter-disciplinary in approach. The research requires the talents of the polymer chemist, the mechanical engineer, and the textile engineer. Georgia Tech offers the unique capability of focusing the attention of specialists from all these disciplines on such a problem. Drs. Freeston and Lundberg are members of the School of Textile Engineering, Dr. Muzzy, the School of Chemical Engineering and Dr. Bangert, Aerospace Engineering.

The portion of the activity focusing on direct polymer to garment tech-nology is being directed by Dr. John Lundberg. Certain phases of the in-vestigations are being carried out by Dr. Lundberg and others by Dr. John Muzzy.

The portion of the activity focusing on fiber to garment technology is being directed by Dr. Freeston with the early phases of the investigations carried out by Dr. Louis Bangert.

Dr. Freeston is responsible for the overall planning, coordination and supervision of the work. The program management plan is shown in Figure 1. To promote a continual interchange of ideas among the personnel involved in the reseach, reviews are scheduled monthly.

Table 1 Work Plan

	Months from Start		
0	**12**	**24**	**36**

A. Polymer-to-Garment

   a. Gas-polymer solutions   X---------X

   b. Effects of additives on fibrillation   X---------X

   c. Effects of crystallization and deformation conditions on fibrillation   X---------X

   d. Continuous yarn formation from inhomogeneous polymer mixtures and from gas-polymer solutions   X---------------------X

   e. Use of tubular biaxial stretching for fabric formation   X--------------------X

   f. Development of laboratory scale yarn or fabric formation process and extension of yarn or fabric formation process to garment making   X---------X

B. Fiber-to-Garment

   a. Single fiber response   X---------X

   b. Multiple fiber suspension   X----------X

   C. Fiber Weaving and entanglement   X--------------X

C. Utilization Plan;

	0	12	24	36
Progress Reports	X	X	X	X
Research Reviews		X	X	X

Presentation to:

  State Textile Industrial Associations   X---------X

  AAMA Research Committee   X---------------------------X

Presentations to:

  National Textile & Apparel Associations   X----X

Technical Conference & Demonstration   X

Summary Technical Report   X

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                        W. D. Freeston, Jr.
                         Project Director


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    │                              │                              │



 J. L. Lundberg                 J. D. Muzzy                  L. H. Bangert


                                                          ┌──────────┴──────────┐
                                                          │                     │


Postdoctoral Fellow          Postdoctoral Fellow       Graduate        Technician
    ½ time                       ½ time                Student          Support


    │                             │
    │                             │
 Technician                  Technician
 Support                     Support
```
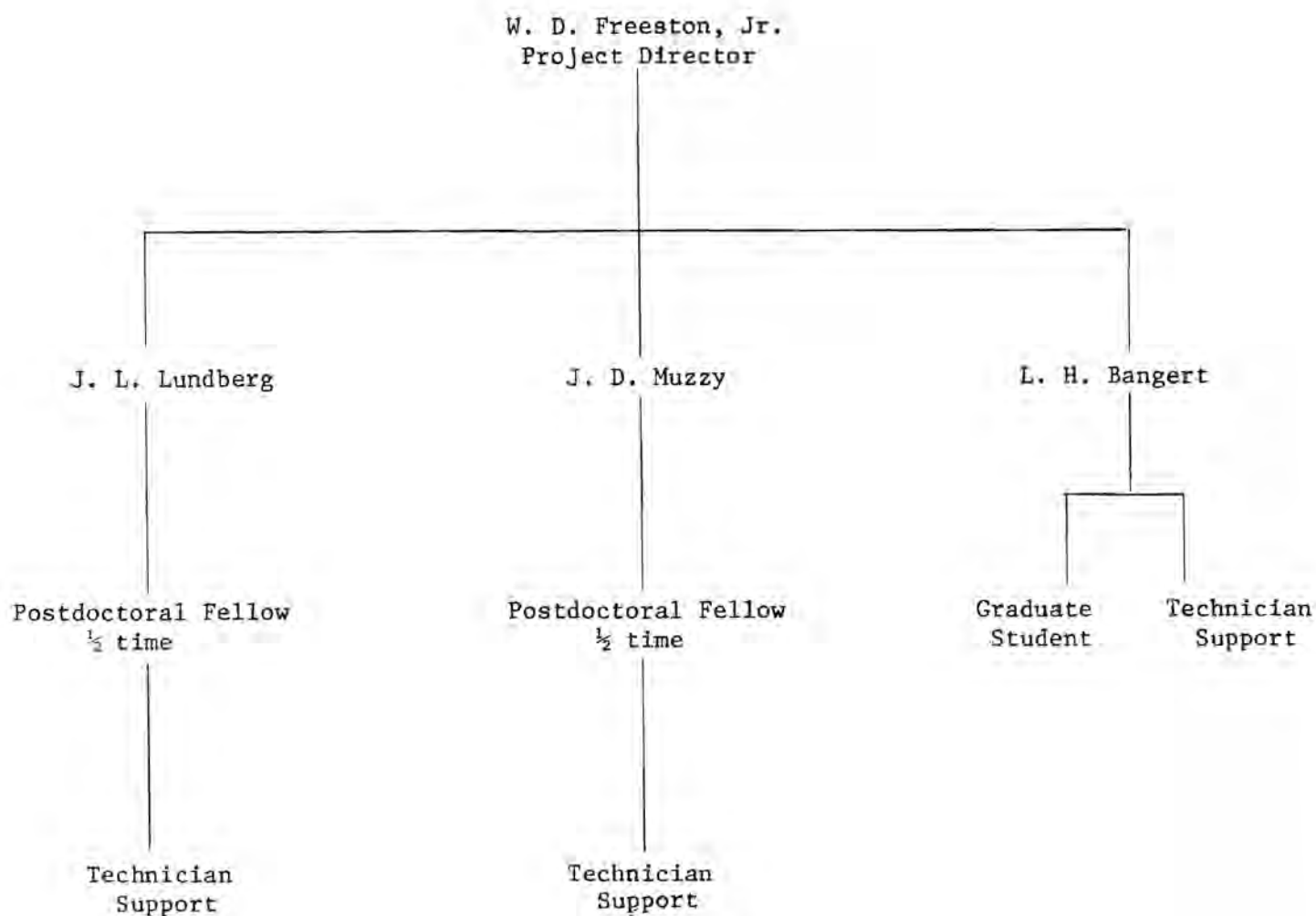
Figure 1.   Program Management Plan

Technology transfer will be by presentations and demonstrations at the regular meetings of the various state Textile Manufacturers Associations and the annual meetings of the American Textile Manufacturers Institute and American Apparel Manufacturers Association. At least one Conference on the program accomplishments will be held at Georgia Tech. A detailed final report will be distributed thoughout the textile and apparel industries. The Utilization plan schedule is given in Table 1.

Detailed schedules of the activities planned for the first year are given in Tables 2-4.

Notification of the Contract award was received in February, part way into the Winter Academic quarter. Since work assignments for each quarter are established at the beginning of the quarter, work on this program could not be initiated until the end of the quarter, i.e., late March. Consequently, although June 30 is six months from the initiation date on the contract, this report is only on approximately three months of effort.

The major activity during the first three months has been to resolve items with long lead times. The position for a postdoctoral fellow was advertised in Chemical and Engineering News, at the May meeting of the Society of Plastics Engineers, and by private communication with a large number of industrial and academic engineers and chemists. Mr. Alvin Levy, an able and experienced polymer and fiber chemist and engineer, was selected and will begin work on Spetember first. He has completed all requirements for his Ph.D degree in chemistry from Georgia Tech except for writing his thesis. His research involves the adsorption of gases on solids under the direction of Professor R. A. Pierotti. Mr. Levy also has several years of

1. Methods for Converting Polymers to Garments   Table 2   Work Schedule for the First Year

A. Gas Solubilities – J. L. Lundberg	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
Hire post doctoral fellow	X			X								
Literature search	X			X								
Set-up high pressure apparatus	X		X									
Solubility Studies												
(1) polyproylene in propane and butane				X			X					
(2) polybutene 1 – butane				X			X					
(3) nylon in CO_2, NH_3 & butane			X						X			
(4) polyester in CO_2										X		X
(5) polyacrylonitrile in NH_3 & CO_2										X		X
B. Additives for Fibrillation – J. D. Muzzy												
1. Obtain polymer	X		X									
2. Obtain fillers	X			X								
3. Obtain liq./gases	X		X									
4. Set-up for sample preparation	X			X								
5. Prepare samples	X								X			
6. Fibrillate samples		X								X		
7. Examine by SEM				X						X		
8. Meas. Phys. Properties				X						X		
9. Relate properties				X								X
10. Liq. feed system	X			X								

C.   Parameters for Fibrillation – J. D. Muzzy

Table 3   Work Schedule for the First Year

	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
1. Uniaxial sample set-up	X————	————	————X									
2. Biaxial sample set-up				X————	————							
3. Uniaxial sample preparation		X————	————	————								
4. Build biaxial test jig				X————	————							
5. Uniaxial sample testing		X————	————	————								
6. Biaxial sample preparation												
7. Biaxial sample testing												
8. Examine by SEM			X————	————	————							
9. Meas. Phys. Properties				X————	————							
10. Relate properties					X————	————						
11. Thermal control of take-off	X————	————	————	————X								

H.   Tubular Fabric Forming   J. D. Muzzy

	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
1. Obtain Blown Film (System BFS)	X————	————	————X									
2. Design modification of BFS	X————	————	————									
3. Test BFS				X————	————							
4. Process modifications					X————	————						
5. Retrospective literature	X————	————	————	————X								

2. Methods for Converting Fibers to Garments

Table 4   Work Schedule for the First Year

A. Fiber Alignment Study – L. H. Bangert	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March

1. Air Supply

   (1) Plumbing by Physical Plant
   (2) Pressure Regulator

2. Wind Tunnel Design & Fabrication

   (1) Design
   (2) Fabrication & Installation
   (3) Probe Holder & Actuator
   (4) System Checkout

3. Counterflow Jet System

   (1) Design
   (2) Fabrication & Installation

4. Fiber Injector

   (1) Design
   (2) Fabrication & Installation

5. Hycam Camera

   (1) Operational Technique
   (2) Checkout with WT system

6. Analysis of Fiber Motion

   (1) Add eqs. of motion of a single
       fiber to existing program
   (2) Predictions of fiber motion for
       various initial conditions

7. Experiments on Single Fiber Motion

   (1) Variation of Flow Parameters
       $\dot{m}_j/\dot{m}_a$, θj, Re_h
   (2) Evaluation of films of fiber motion

industrial experience with duPont in the film extrusion area. A research engineer, Mr. John Harper, was hired in April to work with Dr. Bangert. A pre-baccalaureate student in chemical engineering, Carl Day, began working on the program with Dr. Muzzy May 1st.

Specifications for a blown film extrusion die and take-off system were developed and the equipment ordered. The unit was installed June 17th. A considerable number of polymer grades and additives have also been obtained.

During the past three months, the related recent literature has been reviewed, the sequence of investigation in the various areas established, experimental procedures resolved, and equipment assembled and checked out. The technical activities are reported in Section III.

Results to date indicate that: 1) linear polypropylene is a suitable polymer for fibrillation; 2) specific additives appear to improve the uniformity of fibrillation. 3) A combination of additives is necessary to produce a uniformly fibrous and porous sheet.

II. Introduction

Man's need for textiles is second in importance only to his need for food among the three essentials, food, clothing and shelter. Consequently, the United States must maintain a vigorous, inventive, viable, productive, responsive textile industry.

Although the textile industrial complex, America's largest manufacturing industry, is generally strong, the primary textile and apparel segments have weaknesses at present and show symptoms of serious future difficulties. Profits are too low, and investment in new plant and equipment is inadequate to insure their ability to complete in the world market place during the next decade. This situation is further aggravated by tax subsidy policies of foreign governments, such as Japan, that encourage their textile companies to scrap older plants and build modern facilities. In the recent past, as a consequence of these policies, the trade deficit in textiles has been as large as $2.5 billions per year.

In the primary textile and garment industries, research and development has been indeed small. In most organizations the attitude has been to let the fiber producers, chemical suppliers, and equipment manufacturers do the R & D.

Although the U.S. has never been the leader in textile machinery development, until recently our position was respectable and strong. Now, however, most new developments occur overseas. Consequently, over half the new machinery purchased by the primary textile industry comes from abroad.

The great danger to the future of the American textile industry is the competition of foreign technology (1). From a dominant technological position in textiles and fibers in 1946, we have slipped to a position considerably

inferior to our combined foreign competitors in texiles and to equality in fibers. The U. S. cannot compete with lower labor cost producers with inferior or equal technology. We must make every effort to regain a technological advantage.

Because of fragmentation of the industries, R & D for the primary textile and garment industries require stimulus from outside the industries, probably from the government. The RANN program of the National Science Foundation appears to be an ideal mechanism for promoting the necessary work.

The Georgia Tech, School of Textile Engineering sponsored a conference on Textile Research, September 19-20, 1972 and hosted a User/Developer Conference June 26-28, 1973. A combined total of around three hundred representatives of the primary textile and apparel industries, and textile and apparel machinery manufacturers from throughout the U.S. participated in these conferences. Most companies were represented by their directors of research and/or by corporate officers. Two of the major conclusions of the conferences were the need for:

1) new processes and machinery for producing fiber assemblies that increase industry productivity, decrease energy consumption and waste, and meet OSHA noise and dust requirements and

2) new methods for garment formation.

Over half the fabric produced is used in garments. Thus, the ultimate in garment making would be to go directly from chemical raw materials to finished garments in a rapid, closed, automated system. Such a process would eliminate much of the labor cost not only in textile making but also

in the labor - intensive garment industry as well. This latter twenty-five to thirty billion dollar per year (wholesale value) portion of the textile industrial complex must have decreased cost in labor and fabric waste (currently as much as 30%) if it is to survive foreign competition. This research program focuses on the direct raw material to garment goal.

There are two major thrusts to the program, direct polymer-to-garment systems and direct fiber-to-garment systems. Several possible approaches are being investigated in each of these areas.

The objectives of the polymer-to-garment research are:

(1) to develop direct, automated, fast methods for forming flexible bulky yarns suitable for use in garments without conventional yarn spinning, (2) to extend these methods to fabric formation without weaving or knitting and to garment formation without cutting and sewing, (3) to reduce the cost of spun-like yarns and fabrics by eliminating solvents or solvent recovery in extrusion.

From ten to twenty processing steps are necessary to convert staple fiber to woven fabric; at least six steps are needed to make knitted fabric from continuous filament. Several of these steps, particularly those leading to woven fabrics, have relatively high labor costs. The most costly group of processes in the "textile chain" (from raw material for fiber to finished garment) is yarn formation from staple fiber; fabric formation by weaving is next in cost. Therefore, to be significant, advances must be in yarn and fabric formation, but not at the expense of the comfort characteristics of conventional fabrics woven or knitted from bulky yarns.

We propose to investigate alternate routes for yarn, fabric, and garment

formation eliminating groups of processing steps. These investigations require some rather fundamental physico-chemical information in order to begin to study alternate processes. Three necessary, fundamental investigations are studies of:

      a. Gas-polymer solutions,

      b. Effects of additives on fibrillation,

      c. Effects of crystallization and deformation conditions on fibrillation.

The specific process alternatives to be investigated are:

      d. Continuous yarn formation from inhomogeneous polymer mixtures and from gas-polymer solutions,

      e. Use of tubular biaxial stretching for fabric formation,

      f. Development of a laboratory scale yarn or fabric formation process based on one or more of the above and extension of the yarn or fabric formation process to garment making.

The background information pertinent to the fundamental studies and the related alternate processes for yarn, fabric and garment formation are discussed in turn.

      a. Gas-polymer solutions

One of the proposed process studies requires polymer-gas solubility information at high pressures and high temperatures. Relatively little is known about gas-polymer solubility properties under these conditions (2), especially in the gas phase (3).

      b. Effects of additives on fibrillation

Fundamental studies of the effects of structure and morphology of polymers on fibrillation have been under way for some time at Georgia Tech (4). These studies indicate that additives acting as heterogeneous

nucleation sites may promote fibrillation or control fineness of fibrillation. Further, volatile or gaseous additives expanding in bubbles in the polymer can provide internal means of biaxially stretching film to form and maintain a microfibrillar structure.

c. Effects of crystallization and deformation conditions on fibrillation

Crystallization and deformation parameters can influence the stability of fibrillation by stretching and the sizes of fibrils produced. The parameters include flow orientation effects on nucleation of crystallization, crystallization temperature, preorientation of film, stretching temperature, direction of stretching, and amount of stretching.

d. Continuous yarn formation from inhomogeneous polymer mixtures and from gas-polymer solutions

Direct extrusion of yarn or fabric can reduce production costs by decreasing raw material, labor, and energy requirements as well as capital investment. These savings can be realized for the following reasons:

1) Reducing the number of processing steps can reduce the overall scrap rate and number of operators.

2) Smaller fibrils compared to staple fiber can be produced by directly extruding yarn, as illustrated by the DuPont flash spinning process (5,6); hence, fabrics of lower basis weight can be produced without sacrificing cover.

3) In comparing yarn or fabric to single filaments, more material is processed per linear foot of product, significantly reducing the investment in materials handling and storage facilities.

4) By processing yarn or fabric, product with considerably greater lateral dimensions compared to fibers, and fewer, but larger and

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more efficient drive systems can be used thus reducing utility and maintenance costs.

Direct extrusion of yarn is practiced commercially for the production of low cost fabrics such as carpet backing, wall covering and industrial cleaning cloths. This direct spinning of yarn reduces the fabric production cost by about one-third and the required investment by about one-half compared to conventional fiber to fabric systems (7).

Commercial technques for producing yarn directly from polymer can be classified either as flash spinning or film fibrillation. In flash spinning a solvent or carrier rapidly evaporates as the polymer solution or blend leaves the spinneret. The liquid to vapor expansion biaxially strains and orients the polymer and splits the extrudate into a web of fibrils. This bundle of fibrils is quite similar to a yarn. The flash spun yarn has more surface area than an equivalent weight of conventional yarn. Thus, the fabric basis weight can be reduced without sacrificing fabric opacity. Normally, the flash spun yarn is laid down "randomly" and melt bonded to give a rather inflexible and coarse "spun-bonded" fabric or synthetic paper (6). Woven spun yarn can be as flexible as woven cotton (8). Because of the high cost of weaving, the woven fabric is considerably more expensive than the melt bonded fabric.

In film fibrillation an extruded and hot stretched film is slit and mechanically fibrillated to produce yarn. Also, fabric can be produced by excluding the slitting step and laterally stretching the oriented film. Unlike flash spun yarn, a significant number of large fibrils are obtained in film fibrillation; these increase the opacity and impart excessive rigidity

to the resulting fabric (7). Therefore, fabrics produced from fibrillated films also are not suitable for use in garments.

Flash spun yarns do have suitable properties for garment use. However, the use of mixed solvents in flash spinning require expensive solvent recovery systems. This makes imperative the use of the low cost random laydown, melt bonding technique in making fabrics. Melt bonding of fibers produces relatively hard nodules in the nonwoven fabrics, these make the fabrics quite stiff (9).

Basic studies of the mechanism of fibrillation during polymer drawing suggest that virtually all drawn polymers are microfibrous (10, 11, 12). However, the microfibrous texture of drawn polymers is not readily apparent because the microfibrils can coalesce through shear heating or high temperature annealing (11). For example, drawing at higher temperatures produces larger fibrils due to microfibrils coalescing. In flash spinning, the expansion of the solvent prevents the fibril from coalescing. However, it is not necessary to use a costly vaporizing solvent to obtain biaxial drawing.

The objective of this proposed research is to produce by direct extrusion yarns and fabrics which will be sufficiently flexible for garment applications. The existing technology suggests that fabrics comprised exclusively of microfibrils would meet the desired objective. Also, basic studies indicate that polymers are inherently capable of forming the desired microfibrillar web without the use of volatile solvents. The flash spun yarn process provides the desired texture but a a relatively high cost. The film fibrillation process is economically more attrative but does not provide the desired texture. Therefore, development of processes which fill the considerable gap between the flash spun yarn and fibrillated film processes are sought. The following novel modifications of the flash spinning and film fibrillation

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processes are suggested:

1) Flash spinning from slit or circular dies followed by biaxial stretching in order to eliminate the random laydown and melt binding steps in conventional flash spinning which results in a rigid fabric.

2) Substitution of inert gases, inorganic fillers and incompatible polymers for solvents to promote concurrent fibrillation and biaxial expansion at lower processing costs.

The suggested modifications, in total or in part, should meet the desired objective of producing soft, bulky yarns. If successful, these will be novel processes for making novel products. Furthermore, the proposed innovations should meet the objective of reducing costs principally by minimizing raw material losses, specifically, by eliminating the use of organic solvents to produce microfibrils.

In flash extrusion (spinning) followed by fibrillation, the extrusion of gaseous polymer solutions from the gas-liquid critical region should also be most attractive. In this region we may expect rather high solubility of polymer in gas and that the solubility may be varied over rather wide limits (3). The rapid heat and mass transfer characteristics (13) of matter in the gas-liquid critical region should permit high extrusion rates, rapid precipitation and, perhaps, crystallization of polymer in films, and rapid onset of fibrillation as gas escapes. Particularly attractive is the possibility that a gas such as carbon dioxide might be used; this would eliminate need for gas or solvent recovery sustems.

    e. Use of tubular biaxial stretching for fabric formation

        In the discussion of study d, it was indicated that microfibrils can readily coalesce during uniaxial film fibrillation due to shear heating

and proximity. It is anticipated that biaxial stretching to induce fibrilla-tion should also minimize coalescense of the microfibrils. Also lower crystallization and stretching temperatures can reduce the tendency of microfibrils to coalesce. As indicated previously, fabric composed of smaller fibrils are expected to have better drape, comfort and hand.

Since tubular films can be extruded readily, tubular biaxial stretching to form fibrillated fabric can be considered. Tubular biaxial stretching is particularly desireable because:

1) it is mechanically simpler than flat film biaxial stretching;

2) biaxial stretching is done concurrently rather than sequentially, thereby reducing the tencency of microfibrils to coalesce; and,

3) the tubular form of the fabric produced is advantageous for garment formation. Since the mass of material obtained from a circular die is con-siderably greater per linear foot of product compared to fiber extrusion, it is anticipated that lower stretching temperatures can be implemented with-out increasing the tendency to catastrophically fracture the product. Thus, an additional means of reducing fibril size is accessible through tubular extrusion and stretching.

f. Development of laboratory scale yarn or fabric formation process and garment making

In the latter stages of this study, the most promising process or combination of processes for yarn or fabric formation will be set up in the laboratory and examined for feasibility.

If formation of tubular fabric by biaxial stretching and fibrillation of tubular films is successful, the shrink forming technique of garment manu-facture will be used with this fabric (14). This method for making garments

is low in cost; its labor costs and material waste are considerably below those in cutting and sewing.

A probable system is given in Figure 2. This envisions use of a gas-polymer system or polymer melt, extrusion through a circular die, biaxial stretching and fibrillation to form a tubular fabric. Process modifications necessary to use nylons and polyesters will be studied.

The objectives of the fiber-to-garment research are: (1) to develop the technology required to produce fiber-woven garments with qualities similar to those tailored from yarn-woven fabrics; (2) to replace the processes of spinning staple fibers into yarn, of weaving yarn into fabric and of tailoring garments from fabrics, by the process of fiber weaving in order to increase productivity and reduce material waste and energy consumption.

The process of fiber weaving consists of (1) separation of fiber tufts into individual fibers, (2) straightening individual fibers, (3) orienting fibers in prescribed directions, (4) transporting the oriented individual fibers, (5) depositing individual fibers in parallel arrays and densification of the web and, (6) intertwining the fibers.

The techniques to be developed in the proposed research are aerodynamic or hydrodynamic fiber transport and orientation. Electrostatic fields may be used to augment orienting forces in aerodynamic systems.

Pneumatic systems have been used in textiles for processing picker laps, cleaning lint from fibers, conveying fiber tufts, air-vortex spinning, to insert filling in airjet looms. The movement of fibrous materials in air currents has been investigated by Potapov (15), Morozov and Shal'kin (16) and Ermolaev (17). The effects of electrostatic and aerodynamic forces on carding
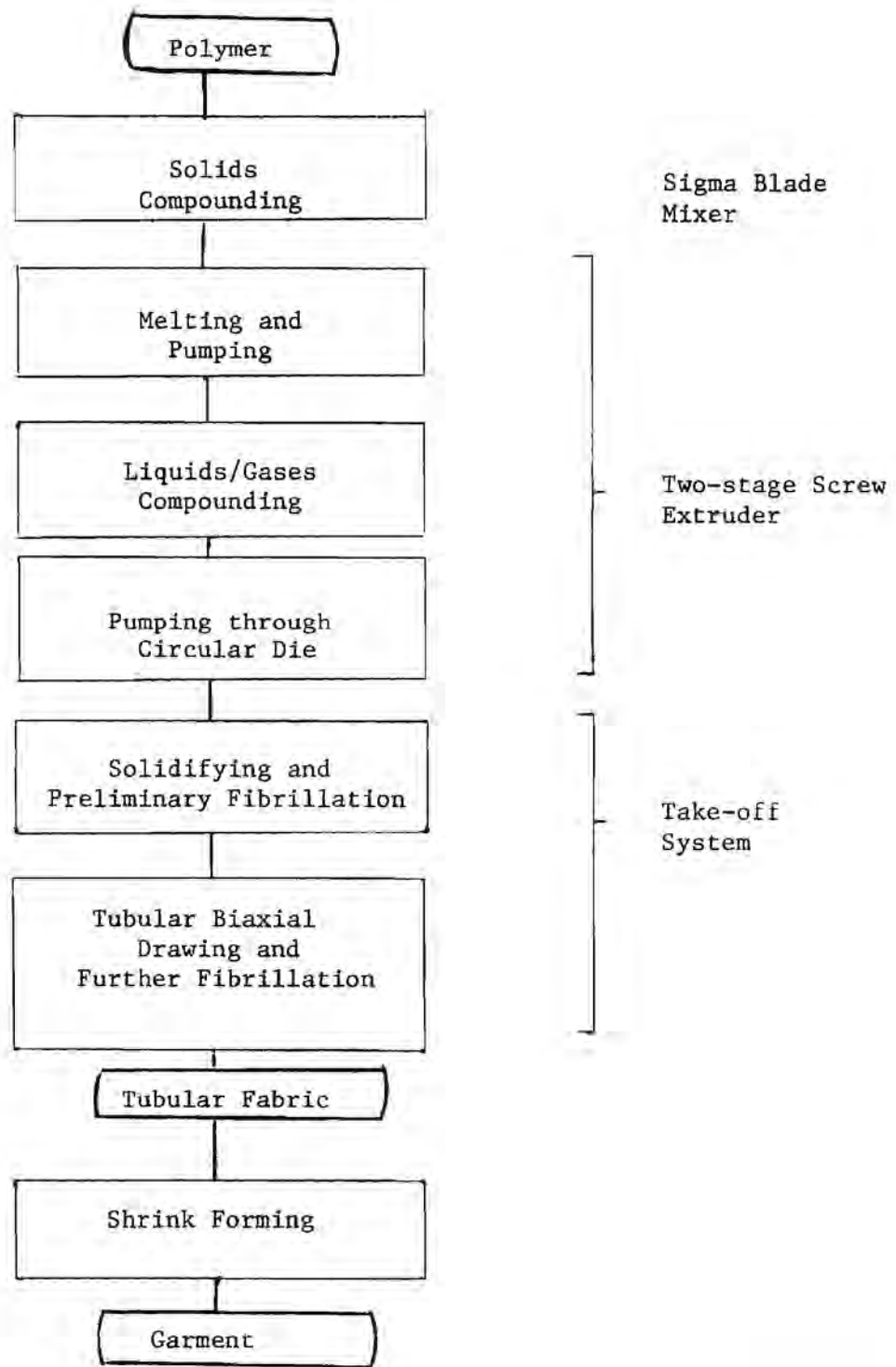
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                    ┌──────────────┐
                    │   Polymer    │
                    └──────────────┘
                           │
        ┌──────────────────────────────────┐
        │            Solids                 │        Sigma Blade
        │         Compounding               │        Mixer
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        │          Melting and              │  │
        │           Pumping                 │  │
        └──────────────────────────────────┘  │
                           │                   │
        ┌──────────────────────────────────┐  │  Two-stage Screw
        │         Liquids/Gases             │  ├  Extruder
        │         Compounding               │  │
        └──────────────────────────────────┘  │
                           │                   │
        ┌──────────────────────────────────┐  │
        │        Pumping through            │  │
        │         Circular Die              │  │
        └──────────────────────────────────┘  ┘
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        ┌──────────────────────────────────┐  ┐
        │        Solidifying and            │  │
        │    Preliminary Fibrillation       │  │
        └──────────────────────────────────┘  │  Take-off
                           │                   ├  System
        ┌──────────────────────────────────┐  │
        │        Tubular Biaxial            │  │
        │          Drawing and              │  │
        │        Further Fibrillation       │  │
        └──────────────────────────────────┘  ┘
                           │
        ┌──────────────────────────────────┐
        │        Tubular Fabric             │
        └──────────────────────────────────┘
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        ┌──────────────────────────────────┐
        │         Shrink Forming            │
        └──────────────────────────────────┘
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        ┌──────────────────────────────────┐
        │           Garment                 │
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Figure 2 Potential Direct Fabric Extrusion and Biaxial Drawing System

of cotton was described qualitatively by Miller and colleagues (18). These investigators dealt with the interaction of air streams and fiber agglomerates, such as tufts and clumps in which the fibers are bent and tangled. The response of single fibers to sudden changes of the air flow field was investigated theoretically in an over-simplified manner by L. Feldman (19) and in a combined experimental and analytical study by Edberg and Stork (20). The experimental work by Edberg and Stork included observations of fiber behavior in air tanks, in both laminar and turbulent flows in tubes, and on the forces required to straighten cotton fibers. Their observations on separation, orientation and straightening of fibers conclude that paralleling of staple fibers in laminar flow of air is possible but not effective enough to be practical. Turbulent convergent air flows with axial velocity gradients of approximately 10^3 per second were found to make parallel about 85 percent of all fibers in the investigated stream.

Air streams have been used extensively in yarn and fabric production processes. Several U. S. patents have been granted (21) for interlacing filaments into yarn by lateral water jet impingement, for drawing and straightening continuous fibers and for intertangling fibers as well as for applying external twist to fibers around an inner, twist-free core.

A patent granted to Evans (22) describes a process to consolidate any web, mat, batt or the like of loose fibers, supported on an apertured surface such as a perforated plate or a woven wire screen, by the use of high-energy (200 psig) liquid jet streams energing from a row of small orifices and impinging perpendicularly to the mat while passing over it. This process produces stable, strong nonwoven fabrics which resemble textile

fabrics prepared by conventional spinning and weaving.

Analytical and experimental studies of the microrheology of suspended particles in creeping flow by Jeffrey (23) describe the motion of a small ellipsoidal particle in a uniformly sheared viscous fluid at low Reynolds numbers. His work was extended to the study of rigid discs and rods (24) and deformable spheres, rods and discs (25). Interest in pulp and paper led to the measurement of the deformation of elastomer filaments with length to diameter ratios of approximately 800. Measurements were performed with moving microscopes to observe the periodic rotation of rigid particles and the snake-like coil formation of filaments. Rigid rods appear to migrate toward the center of a channel in Poiseuille flow. Predicted rotational speeds have been verified experimentally; the research has been extended to the interactions among suspended particles in dispersions.

The motion of rigid spheres (26) can be described in terms of three nondimensional groups, the Reynolds numbers of slip, shear and rotation (27). Lateral magnus forces and drift velocities in creeping flows were found to vary proportionally to the one-half power of the shear rate, causing the particles to move toward the tube center.

These studies will be helpful in the formulation of the equations of motion and in the selection of effective free-stream velocity fields.

Several patents for the use of electrostatic fields for spinning have been granted. In these individual and parallel staple fibers are supplied to a tubular, high voltage electrode; twist is imparted by a coaxial, rotating counter electrode, thereby producing spun yarn. No quantitative descriptions of the response of filaments to electrostatic fields have been found in the literature.

Staple fibers must be arranged in orderly patterns to produce fiber-woven fabrics of the quality achieved in conventional woven fabrics. The fibers in the final fabric should be oriented principally in two perpendicular directions, so that the fibers retain the bending and sliding characteristics of warp and fill yarns in conventional woven fabric.

The processes of separating, paralleling, and depositing the fibers are the central problems in producing fiber-woven garments. These processes must not only be achieved individually, but also they should be combined into one single continuous process, see Figure 3.

Techniques are available to mechanically and aerodynamically separate and parallel fibers but the techniques result in terminal fiber velocities which are too high to be compatible with the requirement for depositing fibers, that the fibers emerge from the applicator nozzle at vanishing tangential velocity relative to the surface supporting the fiber array. Deceleration would return the fibers to more random orientation.

More efficient techniques to separate, orient and deposit stable fibers at low fiber and fluid velocities are required.

```
            ┌─────────────────────┐
            │     Bulk fiber      │
            └──────────┬──────────┘
                       │
            ┌──────────┴──────────┐
            │  Individualization  │
            └──────────┬──────────┘
                       │
            ┌──────────┴──────────┐
            │     Orientation     │
            └──────────┬──────────┘
                       │
            ┌──────────┴──────────┐
            │      Conveying      │
            └──────────┬──────────┘
                       │
            ┌──────────┴──────────┐
            │     Deposition      │
            └──────────┬──────────┘
                       │
            ┌──────────┴──────────┐
            │   Fiber Weaving     │
            │         &           │
            │   Entanglement      │
            └──────────┬──────────┘
              ┌────────┴────────┐
         ┌────┴────┐       ┌────┴─────┐
         │ fabric  │       │ garment  │
         └─────────┘       └──────────┘
```
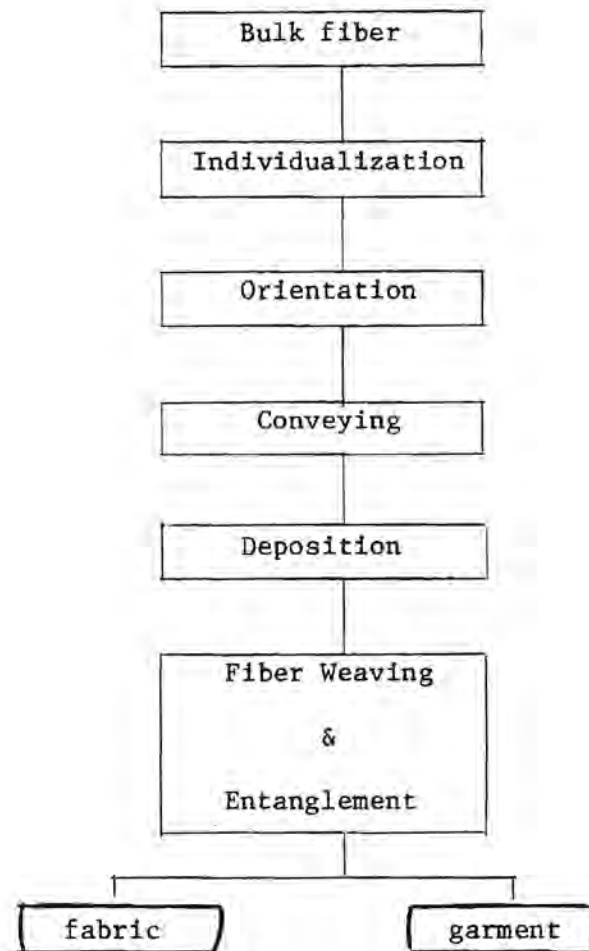
Fiber 3   Fiber-to-Garment

III. Technical Activities

The progress to date in the various research areas is reported below.

A. Polymer-to-Garment

Common features in the process alternatives described in the Introduction are the use of polymer mixtures and solutions, the use of these mixtures or solutions to promote fibrillation, and the use of biaxial stretching to promote fibrillation. To provide rational guidelines for developing the proposed process alternatives, three fundamental studies pertaining to common features of these processes are being investigated in the first year. The development and demonstration of the proposed processes is planned for the second and third years.

1. Gas-polymer solutions

The solubility behavior of a few promising gas-polymer systems are being measured with sufficient care to eliminate those in which polymers are not sufficiently soluble at reasonable pressures and temperatures to be good candidates for extrusion and fibrillation. Among the first candidates to be screened are poly(vinyl alcohol)-ammonia, poly(vinyl chloride)-methylene chloride, nylons in carbon dioxide and ammonia, polyesters in methylene chloride, polypropylene in hydrocarbons, etc. Methods for gas phase studies are similar to those used by Ehrlich in his studies of polyethylene-hydrocarbon systems (3) with the addition of light scattering to detect critical immiscibility of gases (28). Gas solubilities in polymers in condensed phase are being made in apparatus and using methods described in nitrogen-polyethylene, and methane-polyethylene, polystyrene, and polyisobutylene studies (2) and methanepolypropylene work carried out previously at Georgia Tech (29).

A search of the literature for information on gaseous solutions of polymers is continuing. Few papers exist.

An existing pressure barricade was rebuilt so that it can accommodate a pressure vessel with windows and operate at temperatures up to 250°C. Heaters, insulting brick, a rocker assembly, and a laser beam sighting system have been installed.

A rather old pressure vessel has been modified to begin these studies. Because methods to estimate liquid volumes below critical miscibility points in the pressure vessel are not known, a pressure vessel cannot be designed for the experiment before experiments are performed. Fortunately a 100 cc pressure vessel with one inch diameter windows is on hand. This vessel is designed for 45,000 psi at room temperature; it has been operated at 30,000 psi. Later in the studies, when more is known as to the kind of pressure vessel needed, a suitable vessel will be constructed.

The pressure assembly will be assembled and tested before the end of July. Solubility studies with polypropylene in propane and butane and poly-butene-1 in butane will be initiated. These should be miscible over wide ranges of concentration at pressures below 600 atm and temperatures below 160°C.

2. Effects of Additives on Fibrillation

The number of factors affecting fibrillation are extremely large, including variations in polymer characteristics, additive characteristics, extrusion parameters, take-off parameters, and post-extrusion processing parameters. Rather than determine the influence of each primary factor on fibrillation, the approach followed consists of making an educated selection of the most desireable material characteristics and processing parameters

and then experimentally confirming or refuting these selections. The sequence begins with a preferential selection of polymers and ends with an experimental verification of suitable post-extrusion processing conditions. The first material under investigation is polypropylene because it is easier to fibrillate. Then nylon and polyethylene terephthalate will be studied.

At this time the experimental procedures have been partially developed and are outlined in Table 5. The details for performing the steps I through IV have been established and step V, the SEM examination, is currently being developed. Step VI will be initiated once reasonable structures are produced. Suitable testing equipment is on hand for Step VI. Steps VII and VIII have already begun.

A significant amount of new information has been published since this program was proposed. A review of this literature is underway and will be presented in the next report.

A high molecular weight, linear polypropylene, Hercules Profax* 6823, was chosen as a polymer base. This polymer is readily drawn a room temperature, facilitating the experimental work. Flat film was extruded, cut into tensile bars, and drawn at 20% per minute using an Instron tensile tester. Necking and stress whitening occured, indicative of microfibrillation. Extensions between 400 and 500% were acheived. Since only uniaxial drawing was performed, a porous structure was not achieved.

Additives of various types, particle sizes, and concentrations have been compounded with the Profax 6823. Silicon dioxide, calcium carbonate,

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* Trademark, Hercules Chemical Corp.

Table 5

Experimental Procedures

I.   Prepare polymer/additive mixture

II.   Extrude compounded mixture

III.   Produce tubular film

IV.   Draw tubular film

V.   Examine fibrous structure by Scanning Electron
       Microscopy (SEM)

VI.   Measure Fabric Properties

VII.   Modify materials or processing conditions

VIII. Repeat the above

and polypropylene with a chemical blowing agent have been the principle additives utilized to date. Small particle size additives have been emphasized. The inorganic additives appear to be active nucleating agents, based on changes in differential scanning calorimetry crystallization traces, and also appear to improve the uniformity of fibrillation. Samples containing as much as 10% calcium carbonate by weight were readily drawn. Partially foamed samples have been produced but have not been drawn at this time. The foamed samples should be more porous, assuming they can be drawn.

Since the blown film equipment has arrived, the study of additives will be switched to this system. The onstream biaxial orientation which can be obtained with this equipment should provide a more critical test for the additives compared to the flat film equipment.

3. Effects of crystallization and deformation conditions on fibrillation.

In order to study the effects of orientation of crystallites and temperature of crystallization, preorientation of film, and temperature, direction, and amount of stretching on fibrillation, uniaxial and biaxial stretching tests will be performed under controlled conditions on polyethylenes varying these parameters.

This aspect of the research program has not been emphasized to date. Process conditions favoring rapid crystallization have been used since rapid crystallization should improve fibrillation stability. In uniaxial drawing, low temperatures (room temperature) and slow extension rates have been used to promote fibrillation and test fibrillation stability respectively.

Biaxial drawing has not been evaluated, except for preliminary tests

using the blown film equipment, due to the difficulty in setting up an off-stream test. Instead, some sequential drawing tests in two directions have been performed. The Profax 6823 can withstand this severe test and some degree of porosity is achieved. It is expected that inorganic additives and/or foaming agents should assist the development of porosity through this sequential drawing process. Also, it is expected that blown film will provide a better initial structure for this sequential drawing process.

4. Use of tubular biaxial stretching for fabric formation

The blown film equipment has been obtained and installed. It is currently being evaluated using low density polyethylene. Blown film which is laterally uniform but longitudinatly non-uniform has been produced. The cause of the non-uniformity has been identified and is being corrected. Once this difficulty is removed and some familarity with processing parameters is obtained, polypropylene systems will be investigated.

As indicated previously, the blown film equipment will be utilized in the above studies. It will also form the basis for tubular biaxial stretching to form fabric, which will require considerable equipment modification. Before changing the design of the equipment, off-stream tests will be performed to ascertain whether the proposed design changes are warranted. The first modification to be considered will be a combined blown film-tenter frame drawing system since this modification would be downstream from the blown film tower. In operation the formation of a continuous blown film is envisioned followed by the introduction of porosity by the tenter frame drawing system.

B. Fiber-to-Garment

The problem is being attacked in three consecutive parts each requiring

about one year. These are (1) determining the interaction between a single fiber and a distributed-force field (fluid flow, electrostatic), (2) establishing the response of a group of fibers in close proximity to each other to such fields, and (3) design, construction, and operation of prototype equipment to produce flat samples of fiberwoven fabrics.

1. Single Fiber Response

The objective of this study is to predict the response of a single fiber to pressure and shear forces from a fluid flow. This is being done by a combination of experiment and analysis. Systems already exist for satisfactorily separating fibers. This program concentrates on developing a system that aligns the fibers with the main stream and provides controlled deposition of the fibers.

A. Design of Experiments

1) Wind Tunnel and Counterflow Jets

The overall objective of these experiments is to develop a system whereby fibers are aligned with a mainstream air flow, allowing a controlled and orderly deposition of the fibers. In general, fibers injected into a fluid stream will have a random orientation. If the fluid has uniform velocity, so that the relative velocity between the fiber and the fluid is the same all along the fiber, there will be no change in fiber orientation. Velocity gradients in the fluid are necessary to produce forces and moments on the fiber that will bring it into alignment with the mainstream.

It is very desirable to be able to produce a wide variety of velocity gradients experimentally to evaluate the influence of fluid motion on fiber alignment. A flexible method for doing this is by the use of counterflow

jets (Figure 4) (30). The velocity profiles can be easily changed by changing the jet velocity and/or the jet angle. No time-consuming and expensive changes in hardware geometry are required.

In the first year of this program, the experiments are directed toward the study of the motion of single fibers in an air stream. These air flows will be turbulent.

A sketch of the wind tunnel and counterflow jet system for studying fiber motion is shown in Figure 5. This figure shows the dimensions, materials, and other system components. Detailed drawings have also been prepared for shop use. Although not shown in the sketch, one side of the test section is hinged, so that it can be opened for easy access.

The nominal test section conditions are $U = 10$ ft/sec. and $\dot{m} = 0.763$ lb_m/sec. The expected upper limit on the mainstream air flow is $U = 30$ ft/sec and $\dot{m} = 2.29$ lb_m/sec. The counterflow jet can be directed from 0^o (directly upstream) to 180^o (directly downstream). The design of the counterflow jet system has been based on that of Morkovin, et al, (30). The maximum air flow rate through each injection tube will be about 0.01 lb_m/sec. Each tube has an outer diameter of 11/16 inch, with 19 equally-spaced orifices of 3/32 inch diameter. The maximum velocity through each of these orifices will be about 140 ft/sec.

A proble holder and traverse mechanism will be provided to permit flow field measurements in the test section. This traverse mechanism will be manually operated. A lead screw will provide closely-controlled vertical positioning. A longitudinal slot in the upper wall of the test section will allow traverses in the mainstream direction. The proble holder will be mounted on two rails, so that it can be moved to any logitudinal position.
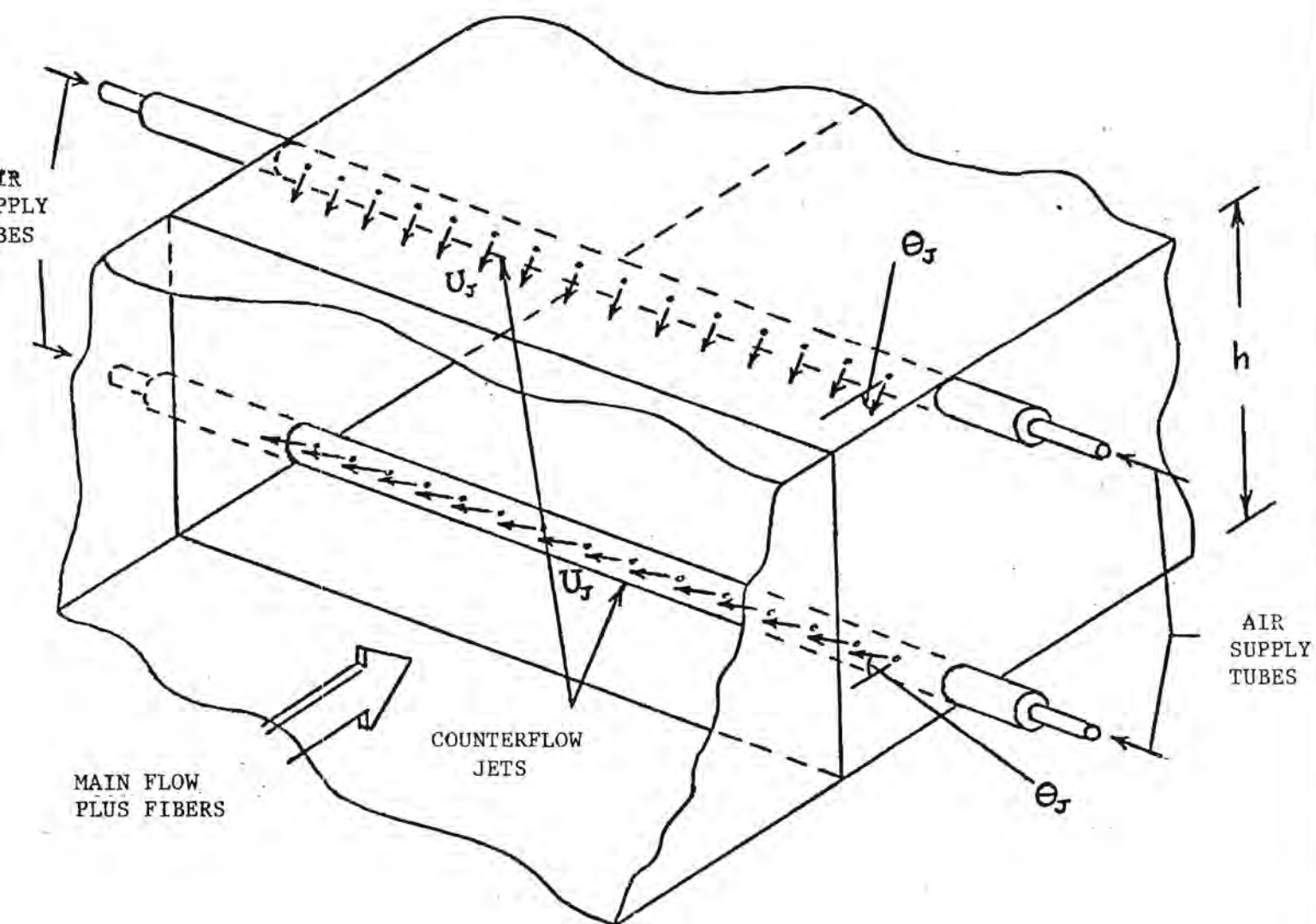
FIGURE 4. COUNTERFLOW JET SYSTEM FOR ADJUSTING VELOCITY GRADIENTS

EXHAUST TO ROOF

CONTRACTION CONE

COUNTER FLOW AIR INJECTION TUBES

72   24   27   72

16.25

7°

TRANSITION   DIFFUSOR

51

PLENUM WITH DAMPING SCREENS

12 x 12 TEST SECTION

SECONDARY AIR

4" PVC SUPPLY

6" PVC

PRESSURE REGULATOR - WITH REDUCING COUPLING

4" HEADER

FLOOR LINE

PRIMARY AIR
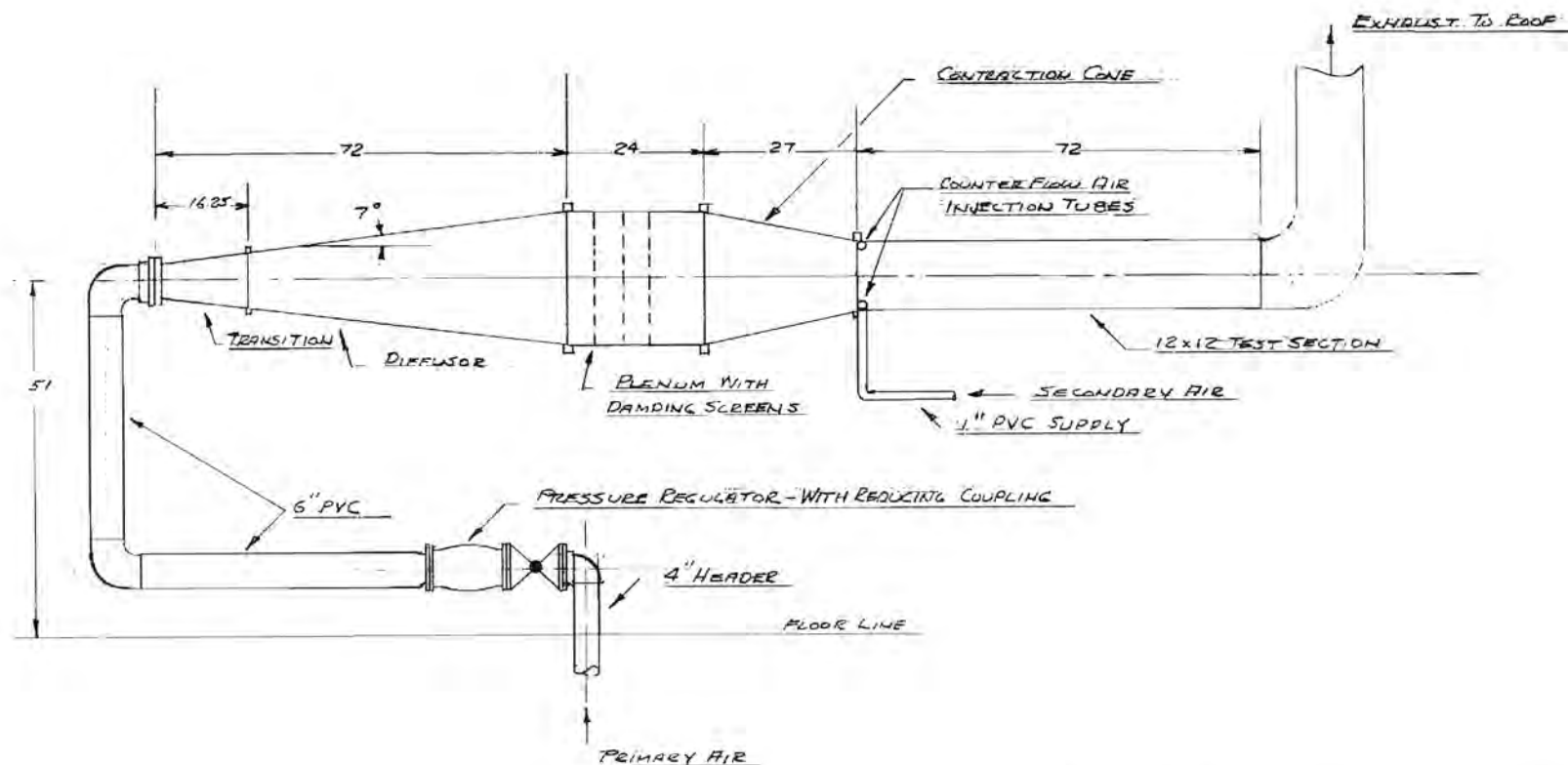
PLENUM IS 1.97' x 1.97'

CONTRACTION RATIO : 3.88

CONSTRUCTION MATERIAL

TRANSITION = FIBER GLASS
DIFFUSOR —   3/8 PLYWOOD
PLENUM   —   3/4 PLYWOOD
CONTRACTION — 3/8 PLYWOOD
TEST SECTION — 3/8 PLEXIGLAS
EXHAUST DUCT 26 GA. GALV.

Velocity profiles in the test section will be measured by traverses of a pitot-static tube, for each combination of mainstream and counterflow conditions. A knowledge of the air flow conditions will be important in evaluating the effects of the air flow on the fiber motion.

2) Air Supply

The air supply for both the mainstream and the counterflow is from a 1000 ft^3 storage tank at ground level. (The laboratory is on the second floor). This tank is charged by a compressor to 100 psig.

The mainstream air passes through a pressure regulator (control valve). Adjustment of the pressure regulator outlet pressure provides the desired mainstream flow conditions. The specifications for this pressure regulator have been determined, bids obtained, and a purchase made. The pressure regulator is a 2-inch globe control valve, pilot controlled, by Fisher. The outlet controlled pressure range is 0.1 to 2.0 psig, with a maximum upstream pressure of 100 psig.

The counterflow jet air supply will use existing supply lines and controls, plus some minor additions.

The complete air supply system has been designed and the materials purchased. This also applies for the wind tunnel exhaust through the roof. Installation will begin as soon as all materials are delivered.

3) Fiber Injector

The purpose of the fiber injector is to provide a fixed and known orientation of the fiber relative to the mainstream, before release of the fiber. The initial conditions of the fiber motion must be known, in order to properly evaluate the effect of the air flow on the subsequent fiber motion. For example, the experiments are expected to establish the

influence of initial fiber angle on the required velocity gradient and the required flow distance to achieve parallelization.

Some design criteria for the fiber injector are: (1) produce minimal distrubance to the air flow; (2) provide a range of initial fiber angles from 0^o to 90^o, relative to the mainstream; (3) provide smooth release of the fiber.

A sketch of the design that has been tentatively chosen is shown in Figure 6.

The fiber is held at the clamp points, each fiber end between the disc and the end of the tube. The tubes are stationary, but each disc is attached to a wire that runs through the tube and is attached to a small plate, as shown. These small plates are pushed leftward (closed position) by the action of the springs, and rightward (open position) by the action of the solenoid.

Thus, the fiber is held in place by the springs until the solenoid is activated. The discs then move to the open position, and the fiber is carried away by the airstream.

Note that only the tubes protrude into the airstream. The solenoid, springs, etc., are outside the tunnel. The mounting slots in the tunnel side wall allow the injector to be rotated to provide any desired initial fiber angle.

4) Photographs of Fiber Motion

Photographs of the fiber motion will compose the primary data on the effects of the air flow on fiber alignment. The original proposal considered the use of a Hycam high-speed motion picture camera to record the fiber motion. Reconsideration since then has suggested that analysis and handling

SPRING

FIBER CLAMP POINTS
(OPEN POSITION)

SOLENOID

PLATE MOUNTED ON
TEST-SECTION SIDE WALL
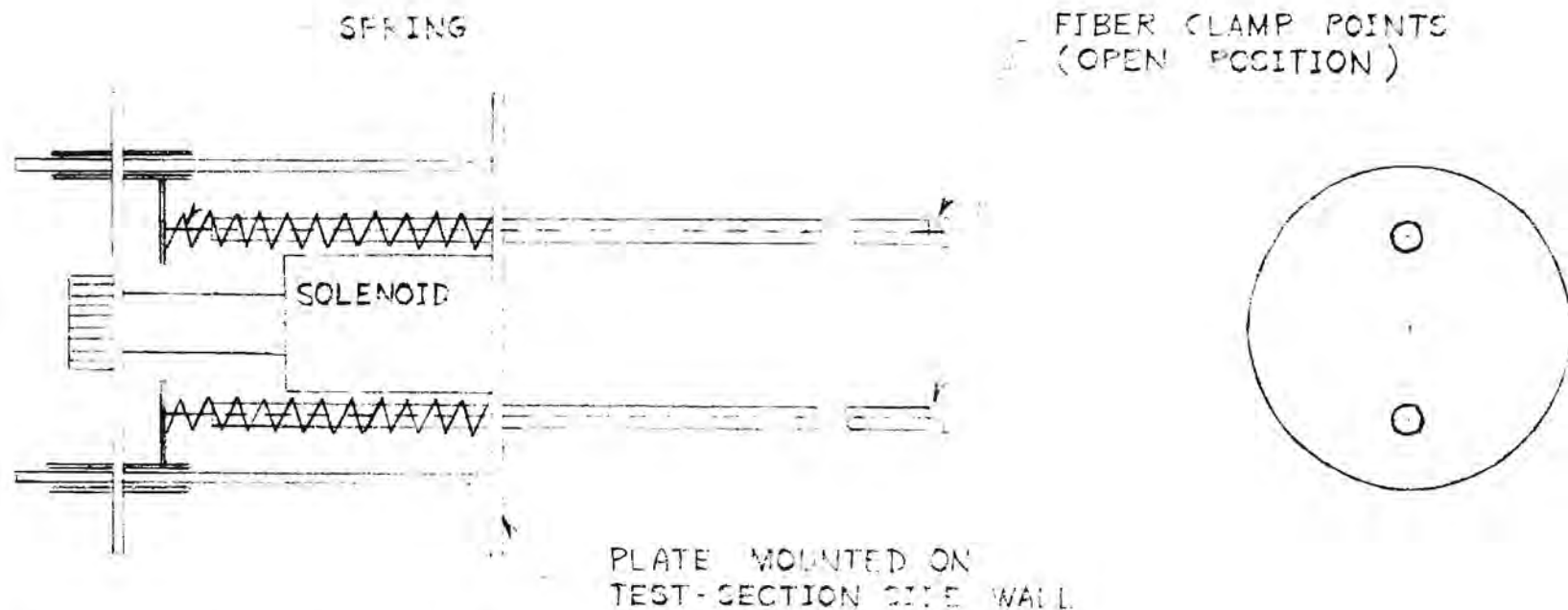
SOLENOID PULLS TO THE RIGHT TO RELEASE
FIBER. SPRING RETURNS CLAMPS TO CLOSED POSITION.

MOUNTING SLOTS

TEST-SECTION SIDE WALL

FIGURE 6. SKETCH OF SINGLE-FIBER INJECTOR

of the photographs of fiber motion can be simplified by using a different technique. The concept is based on Bourot's experiments as described by Cady (31).

With the room darkened, the test section will be illuminated by stroboscopic light at frequencies up to about 100 Hz (or, 6000 flashes per minute). By adjusting the camera shutter, approximately ten flashes are recorded. The fiber will appear in successive positions across the photograph. Hopefully this technique will allow convenient measurement of fiber velocity, inclination, and angular velocity.

A photographic system based on the above principles is now being assembled.

B. Analysis of Fiber Motion

The analysis of the motion of a single fiber in an airstream is being performed with the aid of an existing computer program for the calculation of turbulent flows. A subroutine for the fiber dynamics has been added to this program. This subroutine will be described below.

The main program for the flow field analysis uses the two-equation turbulence model described by Launder and Spalding (32). An advanced turbulent flow method of this type is necessary to compute, with reasonable accuracy, turbulent flows with rapid changes in velocity profile. Flows of this type are of primary interest here.

In analyzing the fiber motion, it is assumed that the presence of the fiber does not affect the airflow. This assumption is based on the fiber's small mass and small size relative to the flow field.

Initial calculations have been made for a rigid fiber of length 27 mm, diameter 18 μm, and specific gravity 1.55. These latter properties are appropriate for cotton fibers. Figure 7 shows the coordinate system used.
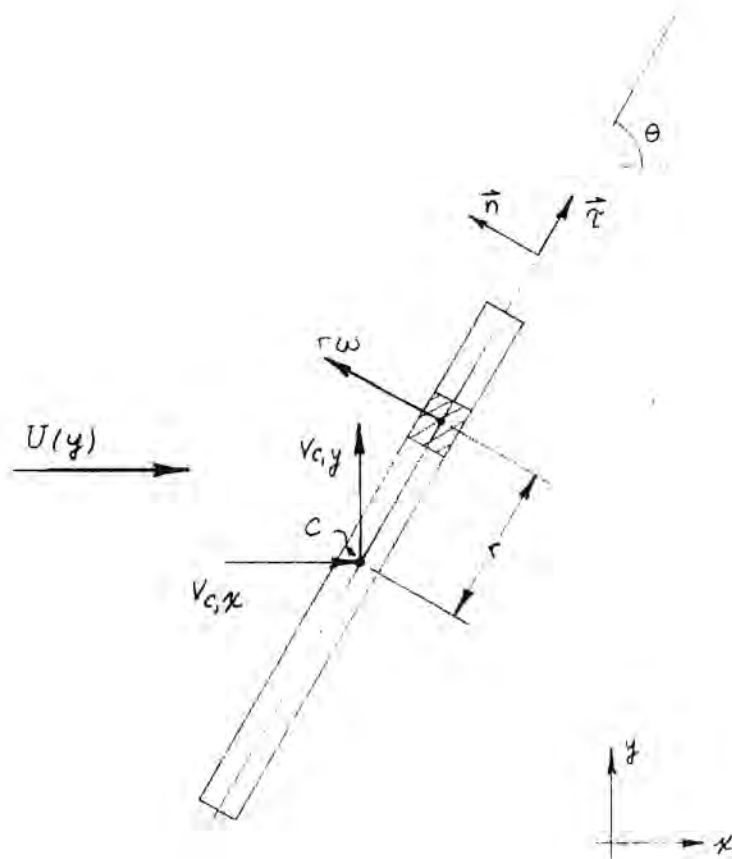
Figure 7. Coordinate System & Velocity Components

The flow and fiber motion are assumed to be two-dimensional and in the x-y plane.

The equations of motion are:

$$F_x = m \, d^2x_c/dt^2 \tag{1}$$

$$F_y = m \, d^2y_c/dt^2 \tag{2}$$

$$M = I \, d\omega/dt \tag{3}$$

In the above, F_x and F_y are the forces in the x and y directions by the air on the fiber, m is the fiber mass, M is the moment of the fiber about its center of mass, ω is the fiber angular velocity, and I is the moment of inertia of the fiber about its center of mass.

F_x, F_y, and M result from the relative velocity between the air and the fiber. At any point along the fiber, the relative velocity is caused by $U(y)$, V_{cx}, V_{cy} and ω. Thus, the force per unit length on the fiber varies continuously along its length. In the analysis, this behavior is approximated by dividing the fiber into a number of segments, each having a constant relative velocity. The component of relative velocity normal to the fiber axis is:

$$W_n = (U - V_{cx}) \sin \theta + r\omega + V_{cy} \cos \theta \tag{4}$$

(See Figure 7). The analysis of Choo and Casarella (33) shows that both the normal and tangential forces on each segment of the fiber can be determined in terms of a Reynolds number $\rho W_n d/\mu$, where d is the fiber diameter, and ρ and μ are the fluid density and viscosity, respectively.

Initial conditions for the fiber motion are the values of V_{cx}, V_{cy}, and θ, and ω. These, plus $U(y)$ from the flow-field solution, allow the computation of the force on each segment of the fiber, and the total moment M. The calculation then marches forward by Δx. During this interval (which is small) the forces on the fiber are assumed to be constant. Then,

the values of V_{cx}, V_{cy}, ω, x_c, y_c, and Θ at the end of the interval are computed by integrating equations (1) - (3). In evaluating the forces on the fiber, the semi-empirical relationships given by Choo and Casarella have been used.

Figure 8 shows some preliminary results that have been obtained. In the first case, the initial air velocity profile was assumed to be logarithmic, which is characteristic of fully-developed channel flow. The initial fiber position was one-fourth the channel width from one wall, with $\Theta = 90^o$ and $V_{cx} = V_{cy} = \omega = 0$. The calculation shows the gradual fiber alignment that results.

For comparison, a linear velocity profile was assumed next. This is a simplification of a profile that may result from strong upstream blowing by the counterflow jets. As expected, the resulting change in Θ is much more rapid than for the logarithmic profile. After proceeding a short distance, however, the calculation failed because the attempted forward step size was too large.

This is an illustration of the principal difficulty that has been experienced so far in the program development. It is related to the small fiber mass and the consequent large fiber accelerations that occur at the start of the motion. A suitable step-size criterion is still being develop-ed, to allow the step size to increase as the relative velocity decreases, and so permit enconomical computation times.
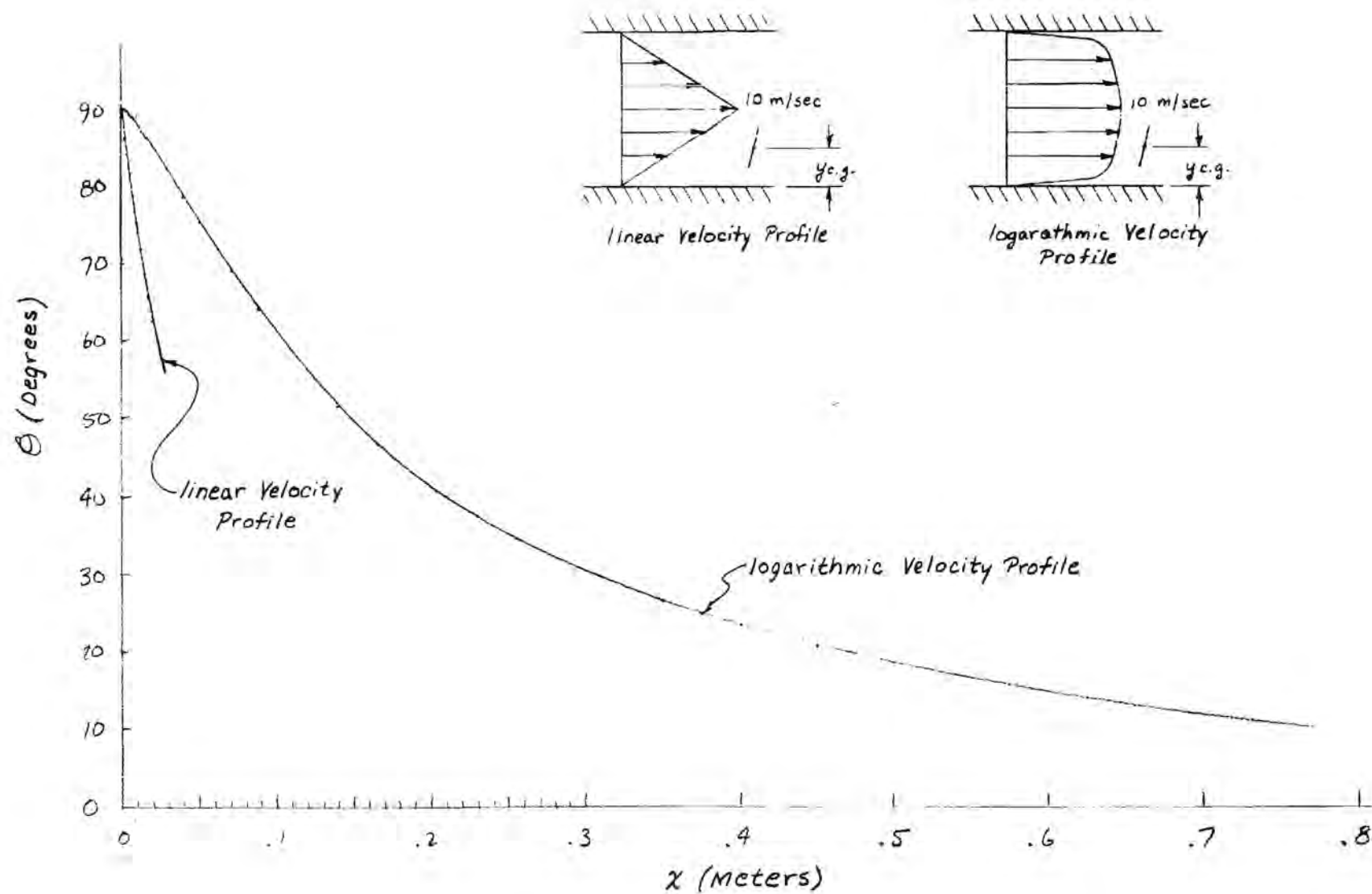
Figure 8. Fiber Inclination θ vs. Distance X

IV. Utilization Plan

Georgia Tech is located in a geographical area which is the center of the U. S. primary textile industry and the second largest concentration of garment manufacturing. This enhances the possibility for effective, rapid transfer of the technology developed under the program to those who will benefit from its development.

The procedures and time schedule that are planned to ensure that the technology developed under the proposed program is transferred into the textile and apparel industries are outlined in Table 6.

The complete research program will be reviewed semiannually. The Georgia Tech research team and representatives of the National Science Foundation will examine the scientific and engineering progress of each phase of the work. Written semiannual progress reports will also be prepared detailing the accomplishments and next steps to be undertaken.

Presentations will also be made at the semiannual meetings of the Georgia Textile Operating Executives and the annual meeting of the Textile Education Foundation Division of the Georgia Textile Manufacturers Association. These presentations will keep local industry apprised of the direction of the research and the accomplishments. Industry comments and suggestions will be solicited.

Offers will also be made to make presentations at meetings of the Alabama, South Carolina and North Carolina counterparts of the GTMA; and to the Research Committee of the American Apparel Manufacturers Association.

During the latter part of the second year of research, efforts will be made to secure an opportunity to present a report on the research at the annual

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Table 6 Utilization Plan

	0	12	24	36		
Progress Reports	X	X	X	X	X	X
Research Reviews		X		X		X
Presentations to:						
State Textile Industrial Associations			X------X			
AAMA Research Committee			X------X			
Technical Conference & Demonstrations				X		
Summary Technical Report				X		

meetings of the ATMI and AAMA. A half - to - full day conference on the research would be held early in the third year as well as at the conclusion of the program. Representatives from primary textile and apparel producers, and textile and apparel machinery manufacturers would be encouraged to attend.

A final report detailing the technology developed under the research program will be prepared and distributed throughout the textile and apparel industries. Every effort will be made to draft the report in a form that will facilitate industry utilization of the new technology through the use of flow charts, sketches and detailed drawings.

On January 27, 1975 the National Science Foundation distributed a press release on this program. As a consequence, we have received many telephone calls and letters requesting more detailed information on the program. The names and business affiliations of the U.S. residents that have contacted us are listed in Section VI. As part of our effort to transfer the technology developed and obtain input from knowledgeable, interested persons in industry, we plan to send a copy of this report and all future reports to these persons.

Two additional opportunities for technology transfer are being pursued. The Society of Plastics Engineers (SPE) is interested in sponsoring a Regional Technical Conference (RETEC) in Atlanta during the Fall of 1976. The Southern Section of SPE supports the theme of "Fibrillation: Processes and Products". This subject must be submitted to the Executive Committee of SPE for approval. A favorable response is expected.

Georgia Tech is hosting a Computer Assisted Apparel Production/
Distribution Conference on October 28 & 29, 1975. The Conference is
sponsored by the Apparel Research Committee of the American Apparel
Manufacturers Association (AAMA). The Apparel Research Committee will
meet the day after the Conference, October 30. At this meeting, we
will make a presentation on this research program. Activities planned
and accomplishments to date will be discussed.

V. Conclusions

Since little work could be initiated until April, it is not possible to report many conclusions at this time. Several preliminary conclusions are as follows:

1. High molecular weight, linear polypropylene is a suitable polymer for fibrillation.

2. Uniaxial drawing of this polypropylene at room temperature induces fibrillation but does not create any porosity.

3. Sequential drawing of this polypropylene in two directions produces a fibrous, slightly porous sheet.

4. Specific additives appear to improve the uniformity of fibrillation in this polypropylene.

5. A combination of additives is necessary to produce a uniformly fibrous and porous sheet.

6. Blown film is preferable to flat film as an initial structure in fabric formation.

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VII. Report Distribution

The First Semi annual Technical Progress Report will be distributed to the names listed below. These persons have specifically written requesting copies of the report. It is planned that future reports containing more significant findings will be given substantially wider distribution.

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E-27-627

Advanced Technology Applications
in Garment Processing

by

Louis H. Bangert
John L. Lundberg
John D. Muzzy
Alvin C. Levy
W. Denney Freeston

Georgia Institute of Technology
Atlanta, Georgia 30332
March 1976

First Annual Technical Progress Report

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Abstract

New, direct, automatic, fast and inexpensive methods are being developed for converting polymer chips and/or staple fiber into fabrics without carding, spinning and weaving or knitting, and garments without cutting and sewing. Other objectives are to reduce material, labor, capital, and energy costs in textile and garment manufacture. The successful development of one or more of these processes will contribute to improving the United States' competitive position in textiles and clothing in the U.S. and world markets.

Polypropylene has been shown to be soluble in n-butane at relatively low pressures ($\sim$ 180 atm). Preliminary results for other polymer-gas systems are encouraging. A pressure system for batchwise extrusion of polymer from the gas phase has been designed.

Studies using polypropylene indicate that the blown film extrusion process is a promising approach for the production of fabric and garments directly from polymer. Additional research is required to achieve fibrillated webs with sufficient strength, weight and opacity to be suitable for most garment applications.

The counterflow jet system aligns fibers parallel to the air stream with flow velocities of less than 20 ft/sec.

Table of Contents

I. Executive Summary

The various phases of the studies and the work schedule are given in Table 1. The principal areas of research during the first year were:

1. Gas-polymer solutions

2. Effects of additives, crystallization and deformation conditions on film fibrillation

3. Use of tubular biaxial stretching for fabric formation

4. Single fiber response to pressure and shear forces from a fluid stream

The program management plan is shown in Figure 1. Dr. Freeston is responsible for overall planning, coordination and supervision of activities. To promote a continual interchange of ideas among the personnel involved in the research, program reviews are held regularly. The members of the review committee are:

W. D. Freeston, School of Textile Engineering, mechanics of fiber assemblies

J. L. Lundberg, School of Textile Engineering, polymer physics

W. C. Tincher, School of Textile Engineering, polymer physics

A. Tayebi, School of Textile Engineering, mechanics of fiber assemblies

J. D. Muzzy, School of Chemical Engineering, polymer engineering

G. A. Fowles, School of Chemical Engineering, polymer engineering

L. H. Bangert, School of Aeronautical Engineering, fluid mechanics

G. M. Rentzepis, School of Engineering Science and Mechanics, engineering mechanics

Table 1 Work Plan

	Months from Start			
	0	12	24	36

A. Polymer-to-Garment

  a. Gas-polymer solutions   X-------------X

  b. Effects of additives on fibrilla-tion   X---------------------X

  c. Effects of crystallization and deformation conditions on fibrillation   X---------------------X

  d. Continuous yarn formation from inhomogeneous polymer mixtures and from gas-polymer solutions   X---------------------------X

  e. Use of tubular biaxial stretching for fabric formation   X---------------------X

  f. Development of laboratory scale yarn or fabric formation process and extension of yarn or fabric formation process to garment making   X---------X

B. Fiber-to-Garment

  a. Single fiber response   X-------------X

  b. Multiple fiber suspension   X-----------X

  c. Fiber weaving and entanglement   X---------X

C. Utilization Plan;

	0	12	24	36
Progress Reports	X	X	X	X
Research Reviews		X	X	X

Presentation to:

  State Textile Industrial Associations   X---------------------X

  AAMA Research Committee   X-----------------------------------X

Presentations to:

  National Textile & Apparel Assocs.   X-------------------------------------X

	0	12	24	36
Technical Conference & Demonstration				X
Summary Technical Report				X

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W. D. Freeston, Jr.
Project Director

J. L. Lundberg	J. D. Muzzy	L. H. Bangert
Postdoctoral Fellow ½ time Mr. Alvin Levy	Postdoctoral Fellow ½ time Mr. Alvin Levy	Research Engineer Mr. John Harper
Technician Support Graduate Students	Technician Support	Technician Support

Figure 1.   Program Management Plan

The research on polymer solutions is being directed by Dr. John Lundberg. He is being assisted by Mr. Alvin Levy, a post doctoral fellow and Ken Ko, a graduate student.

The research on polymer fibrillation is under the direction of Dr. Muzzy. Mr. Levy and Carl Day, a part-time student assistant, have provided assistance.

Dr. Lou Bangert is directing the research on fiber aerodynamics. A research engineer, Mr. John Harper, is assisting him.

Technology transfer is being accomplished by presentations at textile and apparel trade association meetings, professional society meetings, seminars presented at companies, private discussions with company representatives, and seminars at Georgia Institute of Technology. Progress reports are being distributed to interested textile and apparel companies. The Utilization Plan Schedule is given in Table 1.

The schedules for the studies planned during the second year are given in Table 2.

Results to date demonstrate that polypropylene is soluble in n-butane at 180 atmospheres. This finding and preliminary results with other polymer-gas systems indicate that it may be feasible to extrude a polymer foam from the gas phase. An attractive feature of this approach is the possibility of using gases such as carbon dioxide, thereby eliminating the need for gas or solvent recovery systems.

Fibrous webs potentially suitable for some apparel applications have been produced by the blown film extrusion process. Further research will be necessary to increase web tensile strength, increase web weight, and decrease

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A. Polymer-to-Garment
1. Gas-polymer solutions

<div align="center">Table 2 Work Plan</div>

Task	April 76	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
1. Solubility Studies												
(1) polypropylene in propane and butane	completed											
(2) polybutene 1 - butane	───	───										
(3) nylon in CO_2, NH_3 & butane			───									
(4) polyester in CO_2				───								
(5) polyacrylonitrile in NH_3 & CO_2					───							
2. Assemble extrusion system	───	───	───	───	───	───	───	───	───			
3. Extrude polypropylene-propane						───	───	───				
4. Extrude polybutene 1 - propane or butane								───	───	───	───	
5. Extrude polyester - CO_2								───	───	───	───	
6. Extrude nylon - CO_2								───	───	───	───	
7. Extrude other gas - polymer systems											───	───
8. Extrude film to produce fabric											───	───

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2. Polymer fibrillation

Task _____

	April 76	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March

1. Process Design Changes

2. Process Parameter Evaluation

3. Polyester Fabric Formation

4. Polyester Fabric Evaluation

5. Nylon Fabric Formation

6. Nylon Fabric Evaluation

B. Fiber-to-Garment

Task

	April 76	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March

1. Single Fiber Response

    Experiments on Single Fiber Motion

2. Multiple-Fiber Flows

    (1) Multiple - Fiber Injector Design,
         Fabrication, & Checkout.

    (2) Multiple - Fiber Orientation &
         Transportation Experiments.

    (3) Multiple - Fiber Deposition
         Experiments

         (1) Design & Evaluation of Fiber
              Collectors
         (2) Variation of Flow Parameters

web air permeability.

It has been demonstrated that the counterflow jet system provides a method for aligning fibers parallel to the air stream with an air stream velocity of less than 20 ft/sec. Fiber alignment occurs in a very short distance.

II. Introduction

Although the textile industrial complex is generally strong, the primary textile and apparel segments have weaknesses at present and show symptoms of serious future difficulties. Profits are too low, and investment in new plant and equipment is inadequate to insure their ability to complete in the world market place during the next decade.

In the primary textile and garment industries, research and development has also been indeed small. Thus the great danger to the future of the American textile industry is the competition of foreign technology.

Because of fragmentation of the industries, R & D for the primary textile and garment industries require stimulus from outside the industries, probably from the government. The RANN program of the National Science Foundation hopefully will provide this stimulus.

There are two major thrusts to the Georgia Tech NSF RANN program - investigation of (1) direct polymer-to-garment systems, and (2) direct fiber-to-garment systems. Several possible approaches are being investigated in each of these areas.

The objectives of the research are:

(1) to develop direct, automated, fast methods for forming flexible bulky yarns suitable for use in garments with conventional yarn spinning, (2) to extend these methods to fabric formation without weaving or knitting and to garment formation without cutting and sewing, (3) to reduce the cost of spun-like yarns and fabrics by eliminating solvents or solvent recovery in extrusion.

From ten to twenty processing steps are necessary to convert staple fiber to woven fabric; at least six steps are needed to make knitted fabric from continuous filament. Several of these steps, particularly those leading to woven fabrics, have relatively high labor costs. The most costly group of processes is yarn formation from staple fiber; fabric formation by weaving is next in cost. Therefore, to be significant, advances must be in yarn and fabric formation, but not at the expense of the comfort characteristics of conventional fabrics woven or knitted from bulky yarns.

Alternate routes for yarn, fabric, and garment formation, eliminating groups of processing steps are being investigated under the Georgia Tech NSF RANN program. However some rather fundamental physio-chemical information is required before alternate fabric and garment manufacturing processes can be developed. Three necessary, fundamental investigations are studies of:

     a.   Gas-polymer solutions,

     b.   Effects of additives on fibrillation,

     c.   Effects of crystallization and deformation conditions on fibrillation

These studies have been pursued during the past year. The data obtained are encouraging.

The specific process alternatives to be investigated that will utilize the results of these fundamentals studies are:

     d.   Continuous yarn formation from inhomogeneous polymer mixtures and from gas-polymer solutions,

     e.   Use of tubular biaxial stretching for fabric formation,

f. Development of a laboratory scale yarn or fabric
    formation process based on one or more of the above
    and extension of the yarn or fabric formation process
    to garment making.

Direct extrusion of yarn is practiced commercially for the production
of low cost fabrics such as carpet backing, wall covering and industrial
cleaning cloths. This direct spinning of yarn reduces the fabric production
cost by about one-third and the required investment by about one-half com-
pared to conventional fiber to fabric systems[1].

Commercial techniques for producing yarn directly from polymer can be
classified either as flash spinning or film fibrillation. In flash spinning
a solvent or carrier rapidly evaporates as the polymer solution or blend
leaves the spinneret. The liquid to vapor expansion biaxially strains and
orients the polymer and splits the extrudate into a web of fibrils. This
bundle of fibrils is quite similar to a yarn. The flash spun yarn has more
surface area than an equivalent weight of conventional yarn. Thus, the
fabric basis weight can be reduced without sacrificing fabric opacity.
Normally, the flash spun yarn is laid down "randomly" and melt bonded to
give a rather inflexible and coarse "spun-bonded" fabric or synthetic paper.
Woven spun yarn can be as flexible as woven cotton[2]. Because of the high
cost of weaving, the woven fabric is considerably more expensive than the
melt bonded fabric.

In film fibrillation an extruded and hot stretched film is slit and
mechanically fibrillated to produce yarn. Also, fabric can be produced by
excluding the slitting step and laterally stretching the oriented film. Un-
like flash spun yarn, a significant number of large fibrils are obtained in

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film fibrillation; these increase the opacity and impart excessive rigidity to the resulting fabric[1]. Therefore, fabrics produced from fibrillated films also are not suitable for use in garments.

Flash spun yarns do have suitable properties for garment use. However, the use of mixed solvents in flash spinning requires expensive solvent recovery systems. This makes imperative the use of the low cost random laydown, melt bonding technique in making fabrics. Melt bonding of fibers restricts the fiber freedom of motion during deformations thereby making the fabrics quite stiff[3].

Basic studies of the mechanism of fibrillation during polymer drawing suggest that virtually all drawn polymers are microfibrous[4,5,6]. However, the microfibrous texture of drawn polymers is not readily apparent because the microfibrils can coalesce through shear heating or high temperature annealing[5]. For example, drawing at higher temperatures produces larger fibrils due to microfibrils coalescing. In flash spinning, the expansion of the solvent prevents the fibril from coalescing. However, it is not necessary to use a costly vaporizing solvent to obtain biaxial drawing.

The objective of the Georgia Tech research is to produce by direct extrusion yarns and fabrics which will be sufficiently flexible for garment applications. The existing technology suggests that fabrics comprised exclusively of microfibrils would meet the desired objective. Also, basic studies indicate that polymers are inherently capable of forming the desired microfibrillar web without the use of volatile solvents.

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The flash spun yarn process provides the desired texture at a relatively high cost. The film fibrillation process is economically more attractive but does not provide the desired texture. Therefore, development of processes which fill the considerable gap between the flash spun yarn and fibrillated film processes are sought. The following novel modifications of the flash spinning and film fibrillation processes are suggested.

1) Flash spinning from slit or circular dies followed by biaxial stretching in order to eliminate the random laydown and melt bonding steps in conventional flash spinning which results in a rigid fabric.

2) Substitution of inert bases, inorganic fillers and incompatible polymers for solvents to promote concurrent fibrillation and biaxial expansion at lower processing costs.

These suggested modifications, in total or in part, should meet the desired objective of producing soft, bulky yarns. If successful, these will be novel processes for making novel products. Furthermore, the proposed innovations should meet the objective of reducing costs principally by minimizing raw material losses, specifically, by eliminating the use of organic solvents to produce microfibrils.

In flash extrusion (spinning) followed by fibrillation, the extrusion of gaseous polymer solutions from the gas-liquid critical region should also be most attractive. In this region we may expect rather high solubility of polymer in gas and that the solubility may be varied over rather wide limits[13]. The rapid heat and mass transfer characteristics of matter in the gas-liquid critical region should permit high extrusion rates, rapid precipitation

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and, perhaps, crystallization of polymer in films, and rapid onset of fibrillation as gas escapes. Particularly attractive is the possibility that a gas such as carbon dioxide might be used; this would eliminate the need for gas or solvent recovery systems.

Microfibrils can readily coalesce during uniaxial film fibrillation due to shear heating and proximity. However, the use of biaxial stretching to induce fibrillation should also minimize coalescense of the microfibrils. Also, lower crystallization and stretching temperatures can reduce the tendency of microfibrils to coalesce. Fabric composed of smaller fibrils are expected to have better drape, comfort and hand.

Since tubular films can be extruded readily, tubular biaxial stretching to form fibrillated fabric is being investigated. Tubular biaxial stretching is particularly desireable because:

1)   it is mechanically simpler than flat film biaxial stretching;

2)   biaxial stretching is done concurrently rather than sequentially, thereby reducing the tendency of microfibrils to coalesce; and,

3)   the tubular form of the fabric produced is advantageous for garment formation. Since the mass of material obtained from a circular die is considerably greater per linear foot of product compared to fiber extrusion, it is anticipated that lower stretching temperatures can be implemented without increasing the tendency to catastrophically fracture the product. Thus, an additional means of reducing fibril size is accessible through tubular extrusion and stretching.

In the latter stages of the program, the most promising process or combination of processes for yarn or fabric formation will be set up in the

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laboratory and examined for feasibilty.

If formation of tubular fabric by biaxial stretching and fibrillation of tubular films is successful, the molding or shrink forming techniques of garment manufacture will be used with this fabric[7]. This method for making garments is low in cost; labor costs and material waste are considerably below those in cutting and sewing.

A probable system is given in Figure 2. This envisions use of a gas-polymer system or polymer melt, extrusion through a circular die, biaxial stretching and fibrillation to form a tubular fabric.

The objectives of the fiber-to-garment research are: (1) to develop the technology required to produce fiber-woven garments with qualities similar to those tailored from yarn-woven fabrics; (2) to replace the processes of spinning staple fibers into yarn, of weaving yarn into fabric and of tailoring garments from fabrics, by the process of fiber weaving in order to increase productivity and reduce material waste and energy consumption.

Staple fibers must be arranged in orderly patterns to produce fiber-woven fabrics with aesthetic and performance characteristics comparable to conventional woven fabrics. The fibers in the final fabric should be oriented principally in two perpendicular directions so that the fibers retain bending and sliding characteristics similar to those of warp and fill yarns in conventional woven fabric.

The processes of separating, paralleling, and depositing the fibers are the central problems in producing fiber-woven garments. These processes

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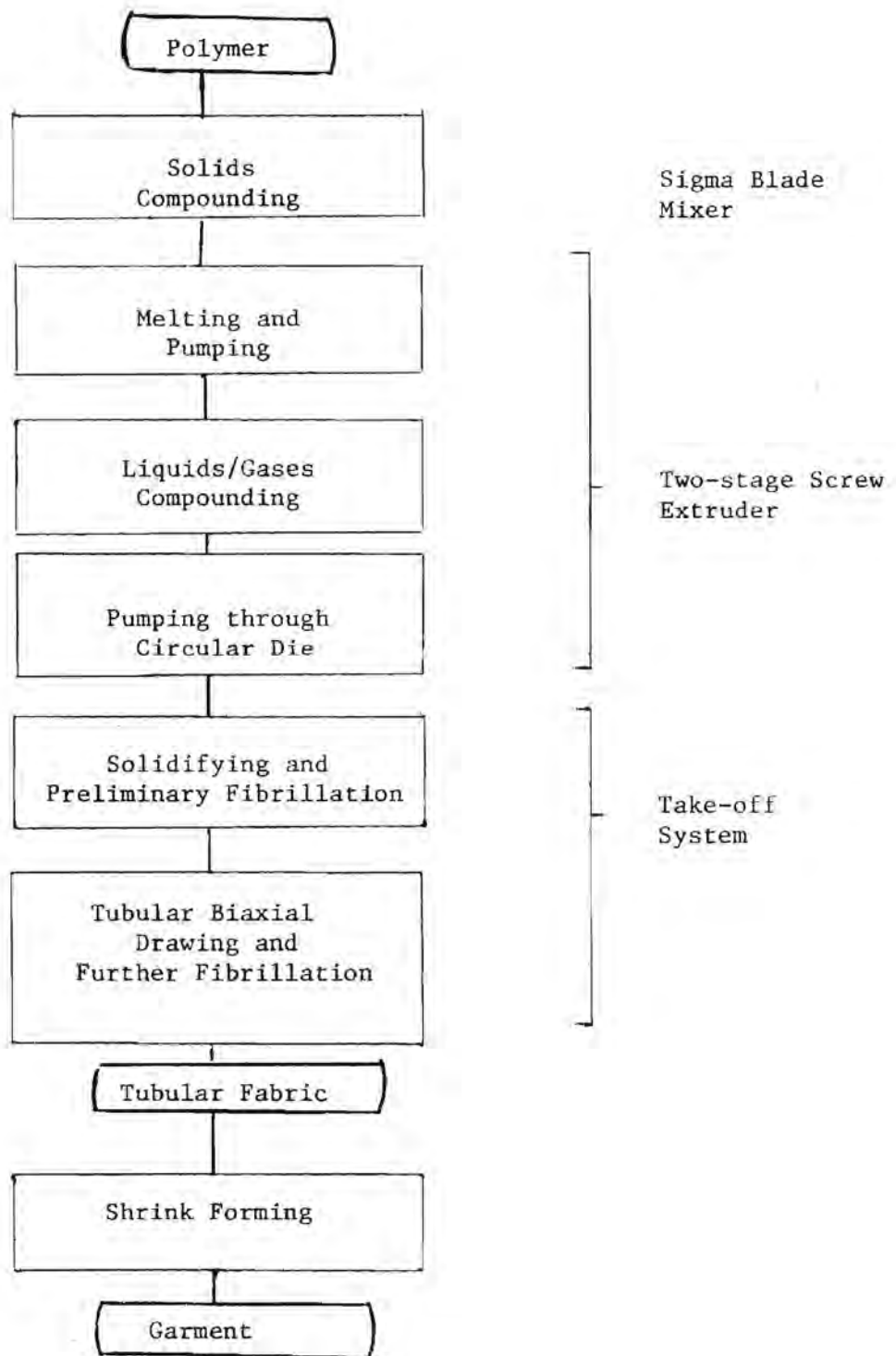
Figure 2 Potential Direct Fabric Extrusion and Biaxial Drawing System

must not only be achieved individually, but also they should be combined into one single, continuous, process, see Figure 3.

The use of air flows and aerodynamic forces for paralleling, conveying and depositing fibers is the current focus of the Georgia Tech research. Techniques are available to aerodynamically separate and parallel fibers but they result in terminal fiber velocities which are too high to be compatible with the requirement for depositing fibers, i.e. that the fibers emerge from the duct at low tangential velocity relative to the surface supporting the fiber array. Deceleration returns the fibers to more random orientation. However, it is anticipated that the use of counterflow jets in an air stream will orient fibers at low flow velocities[8].

Research will be undertaken to develop methods for simultaneously depositing fibers oriented in two perpendicular directions. The resulting fiber web will be densified and the fibers interwoven by high energy fluid streams, and garments fabricated by fabric molding techniques[7].

```
            ┌─────────────────────┐
            │     Bulk fiber      │
            └─────────────────────┘
                       │
            ┌─────────────────────┐
            │  Individualization  │
            └─────────────────────┘
                       │
            ┌─────────────────────┐
            │     Orientation     │
            └─────────────────────┘
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            ┌─────────────────────┐
            │     Conveying       │
            └─────────────────────┘
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            ┌─────────────────────┐
            │     Deposition      │
            └─────────────────────┘
                       │
            ┌─────────────────────┐
            │   Fiber Weaving     │
            │         &           │
            │   Entanglement      │
            └─────────────────────┘
                 │           │
         ┌───────────┐   ┌───────────┐
         │  fabric   │   │  garment  │
         └───────────┘   └───────────┘
```

Fiber 3   Fiber-to-Garment

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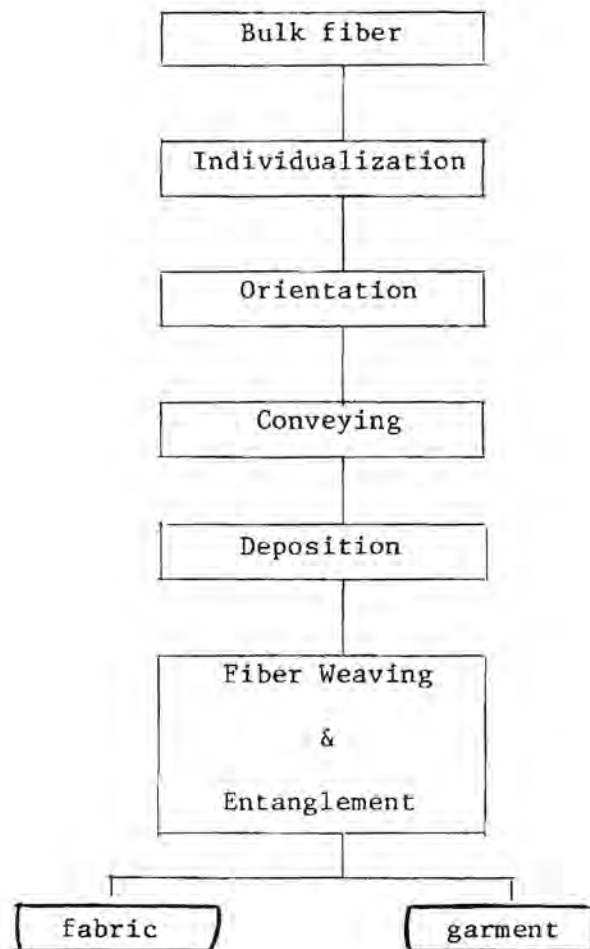
III Technical Activities

   A. Polymer-to-Garment

1.   Gas-Polymer Solutions

      J. L. Lundberg, A. Levy, & K. Ko

a.   Introduction

   Low viscosity and rapid heat and mass transfer are desirable and
even necessary to transport and form polymers into end use products.
Extrusion of polymer melts is costly in energy and equipment and con-
tributes to degradation of polymers. Further, extrusion is limited to
relatively low melt viscosities. Melt viscosities increase with about
the 3.4 power of molecular weight[9]. Many properties such as fiber
strength are increasing functions of molecular weight at least to some
limiting molecular weight[10]. Therefore, in many applications polymers
with molecular weights greater than those which can be melt extruded
would be desirable. A low viscosity dispersion of polymer with rapid
heat and mass transfer would be valuable as a transport and fabricating
medium. Gas phase solutions of polymers have these desirable properties.
Further, gaseous solutions should be excellent media for the flash
extrusion of fibers, foamed sheets, etc. For these reasons the solubilities
of common, fiber forming polymers in gases are being studied.

   The phase separation regions in gaseous solutions of polymer can be
seen easily because of the extreme scattering of light in the gas-liquid
critical region. Phase separation occurs in gaseous polymer solutions
over relatively small ranges of pressure. Therefore the solubility of
polymer in gas can be estimated quite accurately if the volume of the
pressure system is known.

b.   Experimental

   The pressure system is shown schematically in Figure 2. Essential
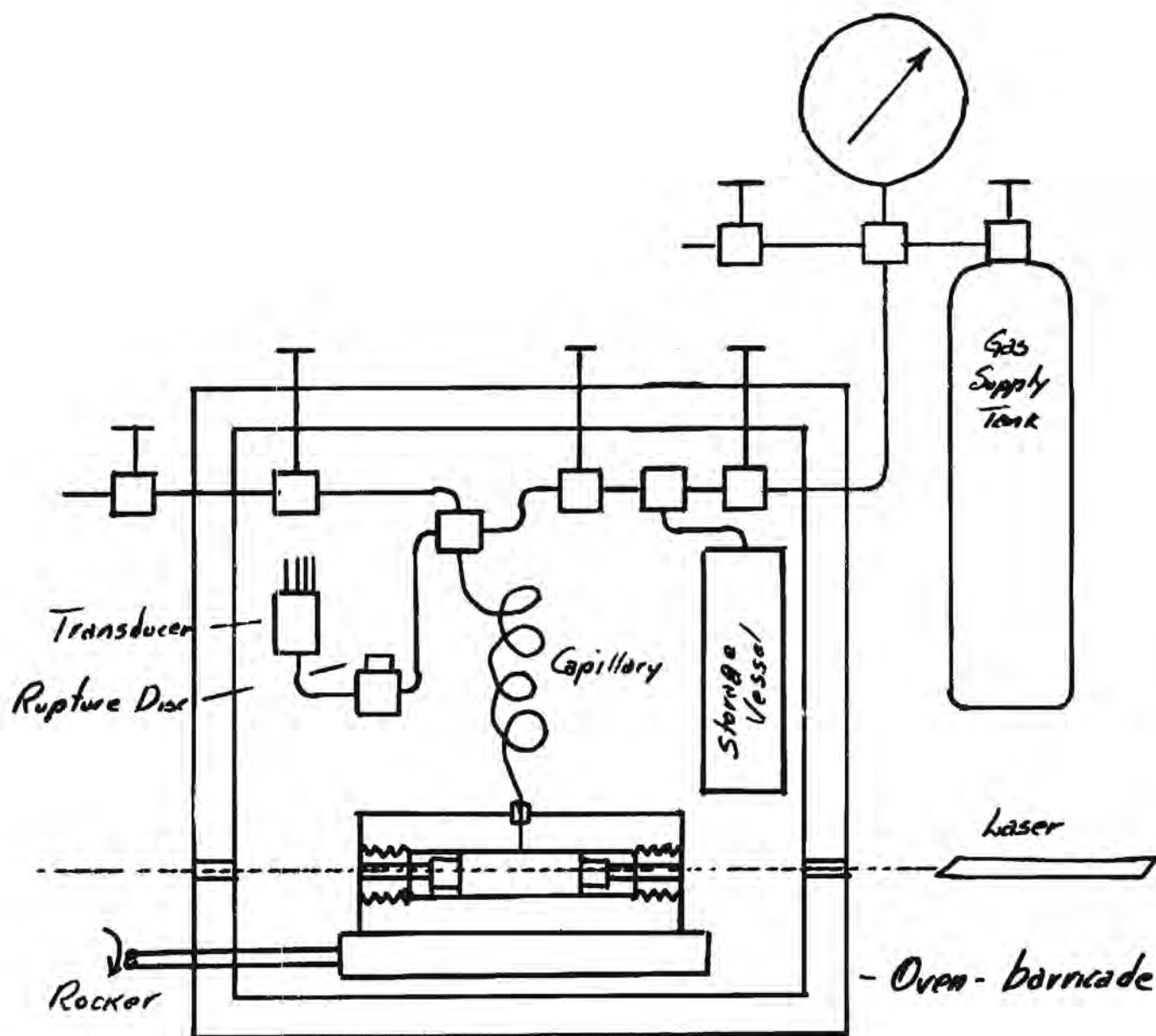
Figure 2 Schematic Diagram of Polymer in Gas Solubility
         Apparatus

components are as follows:

1) Cylindrical pressure vessel, 30,000 psi working pressure, 116 ml volume, 3.81 cm (1.5 in) I.D., with windows along axis (Figure 3)

2) Diaphragm type unbonded strain gage transducer with recording Wheatstone bridge read-out

3) Bourdon tube gage

4) Stainless steel capillary, 0.043 cm (0.017 in) I.D., connecting pressure vessel to system

5) Rocker assembly

6) Oven-barricade

7) Laser

The pressure transducer is calibrated using free piston gages.

In a typical measurement of solubility a weighed amount of polymer is placed in the pressure vessel along with a few steel balls to provide stirring when the vessel is rocked. Gas is condensed in the pressure vessel by cooling below the critical temperature of the gas or the temperature of the supply cylinder. The pressure system containing condensed gas is isolated and heated. The pressure vessel is rocked by rotating through about $\pm$ 60° about its cylindrical axis. As the temperature rises above the critical temperature of the gas, the gas pressure increases. If sufficient gas is isolated in the system, polymer will dissolve in the gas at a temperature at or a few degrees below its melting point. When the desired temperature is reached, the system is held at constant temperature and the pressure vessel is rocked for a reasonable time. Uniform transmission of the laser beam through the pressure vessel shows homogeneity of the gas-polymer system. The system is bled slowly reducing the pressure until the
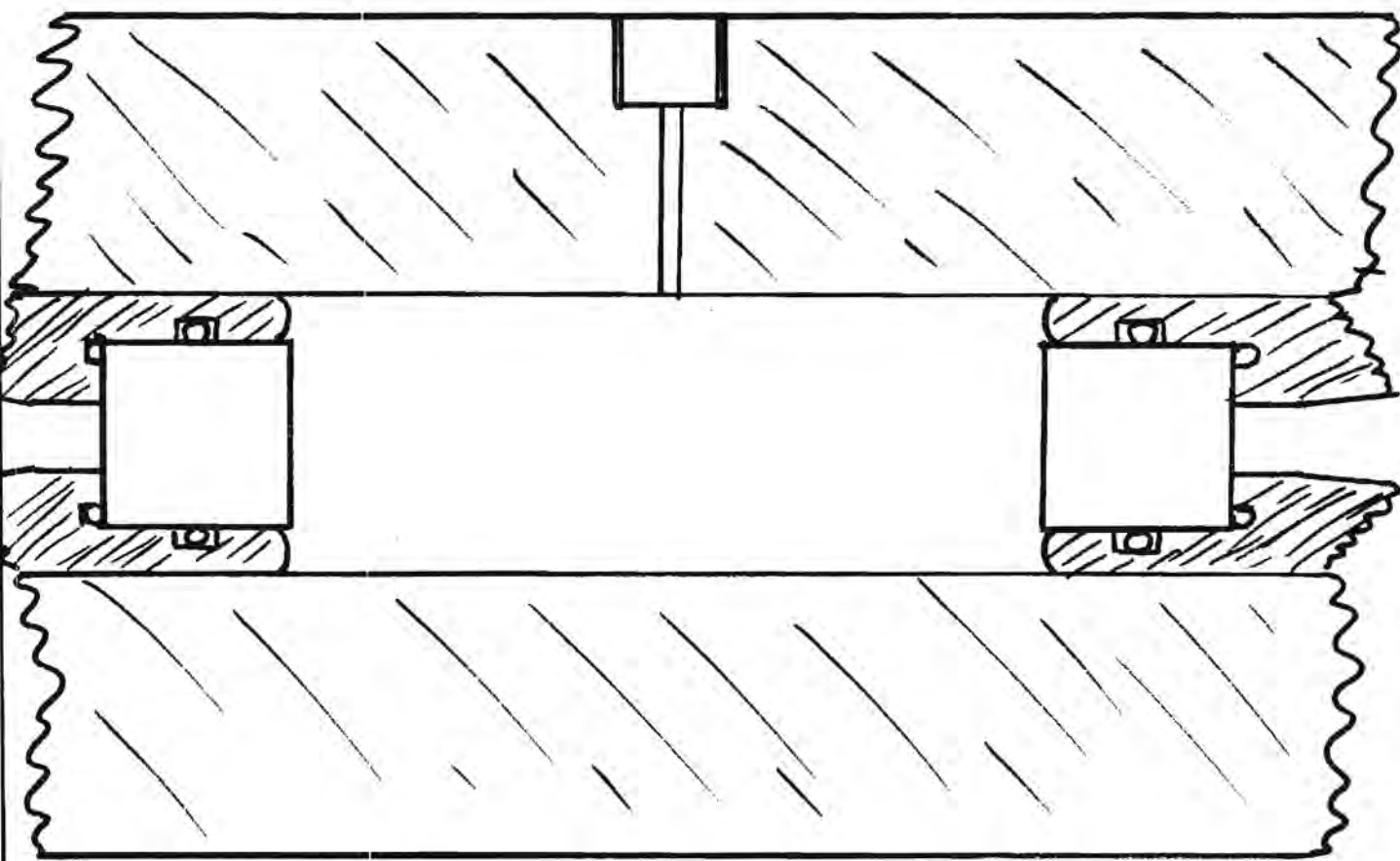
Figure 3 - Cross Section View of Pressure Vessel with
Windows on Cylindrical Axis

scattering of light obliterates the exit beam of the laser. The pressure measured in this period of intense scattering is taken as a measure of the "dew point", "cloud point", or incipient phase separation. With practice, the pressure of the onset of phase separation can be estimated within a few atmospheres and measured within one to two atmospheres. The fine bore, 0.043 cm, of the capillary connecting the pressure vessel to the system causes distillation of gas into the pressure vessel to be slow, but it effectively contains the gaseous polymer solution in the pressure vessel.

Materials used to date are as follows:

Polymers

Polypropylene, Hercules Company Profax®6823, melt indices 0.4 and 12

Polybutene 1, Witco Chemical, Witron®0200, nominal melt index 2.0.

Nylon 6, American Enka Company, fiber grade

Gases

n-Butane, Matheson Gas Products, "C.P." grade, 99.0 percent minimum purity

Carbon dioxide, Matheson Gas Products, "Coleman Instrument" grade, 99.99 percent minimum purity

c.   Data Analyses

Polymer weight is known from initial loading of the pressure vessel. Gas weight w_g at phase separation is calculated from the pressure measured at the onset of intense scattering using the equation for a real gas

$$w_g = \frac{M\,p\,V}{z\,R\,T}$$

where M = molecular weight of gas,

p = pressure measured at onset of phase formation,

V = volume of pressure vessel minus volume of polymer and volume of steel balls,

z = compressibility factor of gas at p and T,

R = gas constant,

T = temperature $^{\circ}$K.

The polymer volume is calculated using the approximate density given in
Table 3. The volume of steel balls is simply that of the number of spheres
used times the volume calculated from measurements of diameters. Because
little if any polymer can find its way through the 0.043 cm I.D. capillary,
only the volume of the pressure vessel need be considered in calculating
gas concentration. The compressibility data are taken from values listed
in the "American Petroleum Institute Tables"[11].

Estimates of the precision of pressure measurements at the "dew point",
"cloud point", or onset of extreme scattering of light are ± 1.5 atm for the
system butane-polypropylene. The accuracy of pressure measurements is
approximately ± 2 atm based upon free piston calibration measurements of
the diaphragm, strain gage transducers.

Densities of gaseous polymer solutions are estimated assuming polymer
contributes negligibly to the measured pressure.

d.   Results

At temperatures above the critical point of butane ($T_c \sim 152^{\circ}$C,
425°K; $p_c \sim 37$ atm)[11], polypropylene is quite readily soluble, 5.5 to
21.1 percent by weight, at relatively low pressures, 122 to 170 atm, at
temperatures from 167° to 193°C. Solubility data are given in Table 3,
Solubility isotherms interpolated from these data are plotted in Figure 4.
Densities of solutions are estimated to be from 0.44 to 0.53 g/cc in the
5.6 to 20.4 weight percent polypropylene range.

One attempt to dissolve nylon 6 in carbon dioxide at $\sim 190 ^{\circ}$C and ~ 550
atm was not successful. Temperatures near or above the melting point of
nylon 6, $\sim 215 ^{\circ}$C [12], should be necessary to dissolve nylon in carbon

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Table 3

Solubility of Polypropylene in n-Butane

Pressure atm	Temperature °C	Solubility Weight Percent Polypropylene	Solution Density g/cc	Melt Index of Polypropylene	Compressibility Factor of Butane z
30.0	166.6	5.5	0.45_2	12	0.492
40.3	176.0	5.6	0.44_5	12	0.527
59.6	193.2	5.7	0.43_7	12	0.625
26.7	168.2	10.8	0.47_4	12	0.481
40.3	176.9	10.9	0.46_8	12	0.528
53.4	186.4	11.0	0.46_3	12	0.574
22.0	167.6	20.4	0.52_8	12	0.467
39.5	181.4	20.9	0.51_6	12	0.532
30.8	176.2	20.7	0.51_9	0.4	0.501
47.3	190.2	21.1	0.51_1	0.4	0.559

Assumed density of polypropylene: 0.92_0 g/cc[17]

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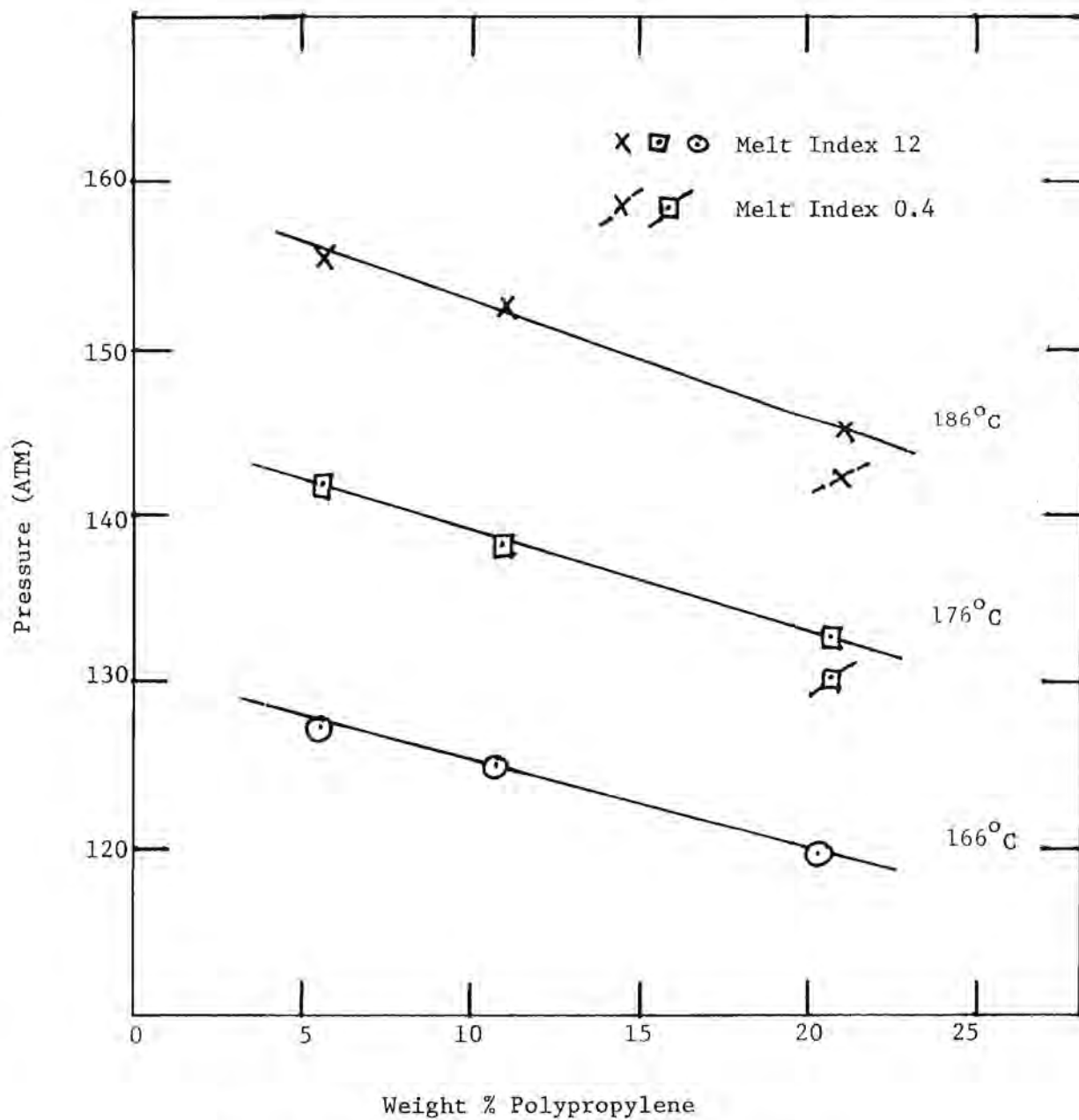
Figure 4   Solubility of Polyproplene in n-Butane

dioxide. New, high temperature valves must be installed in the pressure system before nylon-carbon dioxide studies are continued.

Polybutene-1-butane studies are underway as this report is written.

e. Discussion

The pressure - temperature - concentration behavior of the polypropylene - n-butane system is quite similar to that reported for polyethylene - n-butane by Ehrlich and Kurpen[13]. Polypropylene is soluble in butane at lower pressures, 122 to 170 atm, than is polyethylene, 204 to 255 atm. Because of its lower melting point, polyethylene is soluble in n-butane at lower temperatures than is polypropylene.

The pressure - concentration isotherms (Figure 4) slope downward as they must if pressure approaches zero as the weight fraction of polymer approaches one. These isotherms are somewhat similar to those for the polyethylene and gaseous alkane systems[13,14]. The solubility of polypropylene in n-butane increases somewhat with increasing molecular weight based upon measurements using polypropylenes with melt indices of 0.4 and 12, which differ in molecular weight by a factor of about three. Solubility of polyethylene in ethane decreases with increasing molecular weight[13,14]. The solubility of poly-propylene in n-butane increases slightly with increasing temperature as does the solubility of polyethylene in n-butane[13]. The solubility of polyethylene in ethane decreases somewhat with increasing temperature[13,14]

The approximate densities of the solutions about 0.44 g/cc at $\sim$ 5.5 weight percent polypropylene, 0.46 g/cc at $\sim$ 10.9 weight percent and 0.52 g/cc at $\sim$ 20.6 weight percent appear to be reasonable when compared to the density of pure butane, d $\sim$ 0.42 g/cc, in the p-T region of this investigation.

These estimates are based upon the assumption that the compressibility factors of butane in the polymer solution are the same as in the pure gas; solubility estimates also require this assumption. This assumption probably is not exactly true because of the vast difference in sizes among polymer molecules and butane molecules. Further, volume additivity assumed in making the solubility estimates probably is not correct. Supercritical solutions of hydrocarbons have anomolous volumetric behavior[15]; supercritical solutions of polymers in gases may behave similarly[16]. Never-the-less, these departures from volume additivity probably are not great enough to affect seriously the density estimates given here.

f. Conclusions

Polypropylene is soluble in n-butane at relatively low pressures (p ~ 180 atm, ~ 2700 psi). Solution densities are relatively low, from 0.44 to 0.53 g/cc. The system polypropylene-n-butane should present no pressure - temperature problems when used as a polypropylene transporting and forming system.

Polybutene-1 should dissolve in n-butane at temperatures not higher than its melting point, $125^{\circ}C$ [12], and at pressures comparable to those found for polypropylene-n-butane.

The systems, nylon - CO_2, polyester - CO_2, and polyacrylonitrile - CO_2 will require higher temperatures and higher pressures to form gaseous solutions.

2. Effects of Additives on Fibrillation
       J. Muzzy

a. Introduction

The extrusion of polymer through slit dies produces impermeable film which certainly is unsuitable for garment applications. Microscopic examination of the film often reveals a microfibrous morphology. By incorporating blowing agents and nucleating agents it is possible to produce open webs rather than films and influence the microfibrous morphology. Such structures may be suitable for garment applications. In this section, the influence of additives and polymer grades on fibrillation, as indicated by permeability, tensile properties, and light micrographs, is reported.

Fibrillation is also influenced by process design and operating conditions which are discussed in sections 3 and 4. Due to these additional influences only the trends in properties, and not the absolute values, are meaningful in considering the effects of additives.

b. Testing

Samples for evaluation were prepared by continuous extrusion and web formation using a laboratory scale system. The system is described in detail in section 4. All mixtures for extrusion were prepared by manually mixing the various solids. Since all the components were solids, reasonable blends were obtained. Gross non-uniformities in the samples due to inadequate blending of the powders were not observed. The processing conditions are discussed in section 3.

Basis weights were obtained by weighing four samples 8" long in the machine direction (MD) by 1½" wide in the traverse direction (TD).

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These samples were used to obtain MD tenacities. The TD tenacities were determined from different samples but the same basis weights were used. An Instron Universal Testing Machine was used to determine breaking strengths at an extension rate of 100%/minute with a 2 inch sample gauge length. The tenacities reported are calculated from the breaking strengths and basis weights in the units of (lb/in)/(oz/sq./yd.) in order to minimize the direct influence of basis weight on tenacity.

Air permeability measurements were performed and reported in accordance with ASTM D737. Air permeability provides an approximate assessment of the degree of fibrillation and porosity of the webs. While pore and fibril sizes can be measured directly by quantitative analysis of photomicrographs, this procedure is time consuming, and hence, not justified at this time. Some photomicrographs have been obtained for illustration.

These tests are adequate for evaluating the effects of additives. Additional property evaluations would place an excessive burden on the testing program.

c.   Polymer selection

Polypropylene was selected for study because of its availability in powder or flake form, its ease of handling and processing, and its similarity, as a crystalline thermoplastic, to synthetic textile fibers. Since polypropylene is hydrophobic it is not necessarily a good choice for garment applications. It is anticipated that the property trends observed in stuyding polypropylene would be obtained in nylons and polyesters.

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The first web structures formed were obtained from an experimental grade of polypropylene supplied by Hercules, Inc. This grade contains approximately 0.5% blowing agent which is probably azodicarbonamide. Typical processing conditions and web properties for this grade of polypropylene are reported in Table 4. Photomicrographs of this web are shown in Figure 5. This web exhibits the following features:

1. The basis weight is low.

2. The MD tenacity is greater than the TD tenacity (tenacity imbalance).

3. The average tenacity is low.

4. The permeability is high.

5. The pores are large and nonuniform.

All these features need improvement. In the remainder of this section the impact of materials changes on these features will be reported. The impact of processing and design changes on these features is reported in section 3.

The effect of changing the polymer grade, principally the melt flow or molecular weight of the polymers, is reported in Table 5. The results for Profax 6823 suggest that it is virtually the same grade as 4907, the experimental grade. Changing the melt flow from 0.4 to 12.0, which is equivalent to decreasing the molecular weight, does not change the physical properties substantially. The slightly lower physical properties for Profax 6623 have been observed at other processing conditions, and hence, apparently is not experimental error. Visually the webs are different. The 4907 and 6823 grades have film-

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TABLE 4

BASE CASE

Materials

Linear Polypropylene (Hercules Profax EX4907-42-1)	99.5%
Fumed Silica (Cab-O-sil M-5, Cabot Corp.)	0.5%

Processing Conditions

Zone 1	200°C
Zone 2	230°C
Zone 3	230°C
Zone 4	260°C
Screw Speed	60 RPM
Drive Roll Speed	36 FPM
Mandrel/Die Diameter Ratio (D_M/D_D)	$2\frac{1}{2}$
Cooling Ring Air Pressure	20 psig

Processing Results

Outputs	3.4 lbs/hr
Die Pressure	1200 psig

Web Properties

Basis Weight	0.38 oz/sq.yd.
MD Tenacity	2.3 (lb/in)/(oz/sq.yd)
TD Tenacity	0.55(lb/in)/(oz/sq.yd)
Average Tenacity	1.5 (lb/in)/(oz/sq.yd)
Air Permeability	off scale[a]

---

a.   Exceeds 926 cu. ft/sq.ft/min.

1X

|—— 1.0 inch ——|

4X
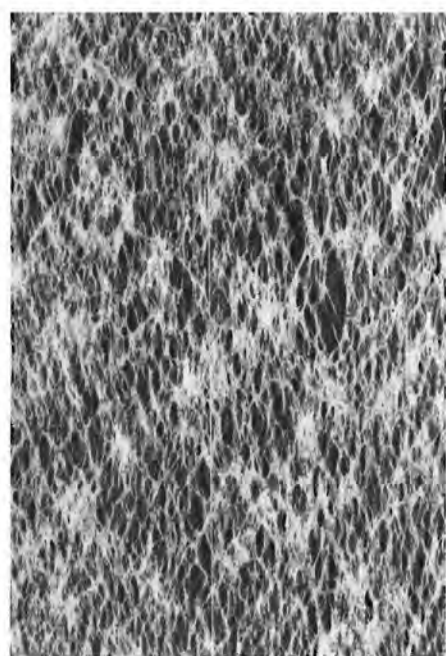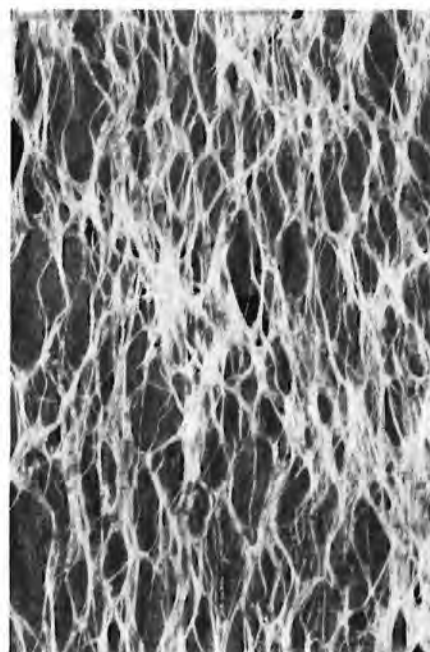
|—— 0.25 inch ——|

Figure 5

BASE CASE SAMPLE

like remnants whereas Profax 6323 does not. Also, the 6323 grade can be processed with larger diameter forming mandrels than the other grades. Thus, the high melt flow (low molecular weight) grades of polypropylene are preferred for web formation.

d.   Blowing Agents

Without the use of blowing agents polypropylene film is produced. By using a blowing agent either a foam or porous web can be produced. Since the die gap is small (0.040") web formation is promoted. Blowing agents can be inert gases, volatile liquids,or solids which decompose and liberate gases at the processing temperatures. Solid blowing agents have been studied because of the relative ease of processing powder blends of blowing agent and polymer of known concentration.

Azodicarbonamide is a common solid blowing agent suitable for polypropylene. The commercial polypropylene grades reported in Table 5 were blended with Kempore 125, an azodicarbonamide powder from Stepan Chemical Co. Further studies with azodicarbonamide are reported in Table 6. The data in Table 6 was obtained from webs produced at a higher screw speed than in Table 5 (75 versus 60 RPM), and hence, the properties are better. Switching from Kempore 125 to 200, which represents a decrease in particle size, did not improve the properties. However, using Kempore 200MC, which is a nucleated, fine particle size version of azodicarbonamide, does increase the average tenacity and decrease the permeability significantly. Increasing the weight percent blowing agent to 1% led to a decline in properties due to overblowing.

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Table 5

EFFECT OF POLYMER GRADE ON WEB PROPERTIES[a]

Polymer Grade, Hercules Profax No.	4907[b]	6823[c]	6623[c]	6323[c]
Melt Flow, g/10 min. at 230°C[d]	unknown	0.4	2.0	12.0
Die Pressure, psig	1200	1300	750	200
Output, lbs/hr	3.4	3.4	3.8	3.9
Basis Weight, oz/sq.yd.	0.38	0.41	0.47	0.43
MD Tenacity, (lb/inc)/(oz./sq.yd)	2.3	2.1	1.5	2.1
TD Tenacity, (lb/in)/(oz./sq.yd)	0.55	0.41	0.33	0.36
Ave.Tenacity,(lb/in)/(oz/sq.yd)	1.5	1.3	0.9	1.2
Air Permeability,(cu ft/sq ft/min)		off scale		

[a] Processed as in Table I

[b] Formulated as in Table I

[c] Formulated as 99 wt% polymer, 0.5% azodicarbonamide, and 0.5% fumed silica

[d] Taken from manufacturer's literature

TABLE 6

Effect of Additives

Polymer: Hercules Profax 6323

Conditions: 200°, 230°, 230°, 230°, 75 RPM, 36 FPM

Additives	Basic Weight oz/sq. yd	Average Tenacity (lb/in)/(oz/sq yd)	Permeability (cu.ft/sq.ft./min)
Kempore* 125 (0.5 wt %)	0.53	2.0	331
Kempore 200 (0.5 wt %)	0.54	1.4	386
Kempore 200MC (0.5 wt %)	0.52	2.6	145
Kempore 200MC (1.0 wt %)	0.49	1.4	530
Kempore 200MC plus Cab-o-Sil** M5 (each 0.5 wt%)	0.47	2.5	162

---

*   Stepan Chemical Co. (azodicarbonamide)

**   Cabot Corp. (fumed silica)

Adding fumed silica (Cab-O-Sil M5) with the Kempore 200 MC did not improve the properties. However, using fumed silica with Kempore 125 does improve the properties moderately. Apparently the fumed silica acts like a nucleating agent for bubble formation; but, it is not as effective as the nucelating agent incorporated in Kempore 200 MC.

Significant improvements in web properties have been achieved by selecting a nucleated, fine particle size, solid blowing agent. It remains to be seen whether further improvements can be achieved by utilizing other blowing agents and nucleating agents separately or in combination.

e.   Other Additives

A number of additives were tested to discern whether further improvements in the uniformity of fibrillation could be achieved. These additives were selected on the basis of their potential to nucleate bubble formation or fibrillation. These additives will be discussed in turn.

Fumed silica (Cab-O-Sil M5) was discussed in section 2-d. The small particle size and incompatibility of this material could nucleate more uniform fibrillation. Some benefit was achieved in combination with coarse blowing agents, but not with the fine particle, nucleated blowing agent. Since the fumed silica increases the viscosity of the mixture, the output at a given extruder speed was decreased. Fumed silica is not an essential additive at this time.

Zinc stearate is partially compatible with the polymer and could

improve bubble nucleation. However, process tests indicate that zinc stearate causes pre-blowing of the blowing agent, producing a web with a film-like appearance. It does not appear to be a desireable additive.

Sodium salicylate can nucleate polymer cystallization, and hence, it may nucleate fibrillation. In processing, no beneficial effect was observed. It did result in a web with a light brown tint and a relatively high sheen.

Linear polyethylene flake up to 10% by weight was processed with polypropylene and blowing agent with no benefits achieved. By processing semi-compatible polymers some enhancement in fibrillation was expected. These preliminary trials do not support this expectation.

f.   Conclusions

The primary conclusions from this study of the effect of the feed mixture on the uniformity of fibrillation are: (1) lower molecular weight polypropylene is easier to process and produces a less film-like web, and (2) nucleated, fine particle size, solid blowing agent improves the uniformity of fibrillation. At this time no other components improve the uniformity of fibrillation substantially. Further studies of additives appear warranted, but due to a overwhelming variety of additives which could be evaluated, a carefully considered, fundamental approach to this problem will be followed.

3.   Effects of Crystallization and Deformation Conditions on Fibrillation

a.   Introduction

It is expected that the crystallization and deformation conditions will influence the web properties. These conditions include the

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material temperature, the stretching rate, and the degree of MD and TD stretch. To facilitate process development, these conditions have been studied in terms of processing variables. The pertinent process variables are: (1) extruder temperature profile (material temperature), (2) cooling air flow, temperature, and direction (material temperature), (3) wind-up rate less extrusion rate divided by mandrel height (stretching rate), (4) wind-up rate divided by extrusion rate (MD stretch), and (5) mandrel diameter divided by die diameter (TD stretch). To simplify the presentation, only the direct influence of particular processing parameters on web properties will be reported.

b.   Extruder Temperature Profile

The extruder temperature profile was determined primarily by the decomposition characteristics of the azodicarbonamide blowing agent. Referring to Table 4, the temperature in zone 1, next to the feed hopper, was selected to avoid melting in the hopper and premature blowing agent decomposition while providing early melting in the extruder barrel. The zone 2 temperature was selected to closely match the temperature in zone 3 in order to provide a relatively uniform melt temperature.

The temperature in zone 3, the final extruder temperature control zone, was limited to 210-250°C. Below 210°C insufficient blowing occurred. Above 250°C excessive pre-blowing occurred. Normally, the temperature in zone 3 was set at 230°C.

Similar restrictions apply to zone 4, the circular die. In this zone the acceptable temperature range is 230°C - 260°C. Within this range, the influence of die temperature on web properties, as indicated in Table 7,

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TABLE 7

Effect of Die Temperature on Web Properties[a]

Die Temperature, °C	230	240	260	270
Basis Weight, oz/sq.yd.	b	0.34	0.39	c
MD Tenacity, (lb/in.)/(oz/sq. yd)		2.7	2.5	
TD Tenacity, (lb/in.)/(oz/sq. yd)		0.54	0.49	
Ave Tenacity, (lb/in.)/(oz/sq. yd)		1.6	1.5	

a. Materials and processing as in Table I except for die temperature

b. Inadequate blowing, processing unstable

c. Excessive pre-blowing

is not great. There is a moderate tendency towards improved properties at lower die temperatures. Also, by using lower molecular weight polypropylene (Profax 6323 instead of Profax 4907), processing at 230°C can be performed and is preferable to higher die temperatures.

Clearly, the selection of the extruder temperature profile is controlled by solid blowing agent. There appears to be some incentive for processing at lower temperatures, but it would be necessary to catalyze the decomposition of the blowing agent or use different blowing agents. These modifications have not been explored at this time.

c.   Wind-up Rate

At constant output, increasing the wind-up rate (drive roll speed) increases the extent of MD stretching. As Table 8 indicates, increasing drive roll speed lowers the basis weight, increases the MD tenacity, decreases the TD tenacity and does not change the average tenacity.

Thus, in order to produce a balanced web, a lower drive roll speed is preferred. Unfortunately, it is not feasible with the present design to decrease the drive roll speed below 25.7 feet per minute (FPM). Below this speed the web piles up on the mandrel. Consequently, further design changes are necessary to permit processing at lower drive roll speeds or higher extrusion speeds in order to promote balanced web formation.

d.   Extruder Screw Speed

Increasing the extruder screw speed while other conditions remain constant increases the output and therefore the web basis weight -- see Figure 6. The same relatively linear trend is observed for forming mandrels with different diamters (D_M = 2.50" and D_M = 3.75"). The basis weights at

TABLE 8

Effect of Drive Roll Speed on Web Properties[a]

Drive Roll Speed, FPM	25.7	36.0	45.2
Basis Weight, oz/sq.yd	0.52	0.39	0.34
MD Tenacity, (lb/in)/(oz/sq.yd)	2.1	2.5	2.6
TD Tenacity, (lb/in)/(oz/sq.yd)	0.78	0.49	0.36
Ave Tenacity, (lb/in)/(oz/sq.yd)	1.5	1.5	1.5

[a] Materials and Processing as in Table 3 except for drive roll speed
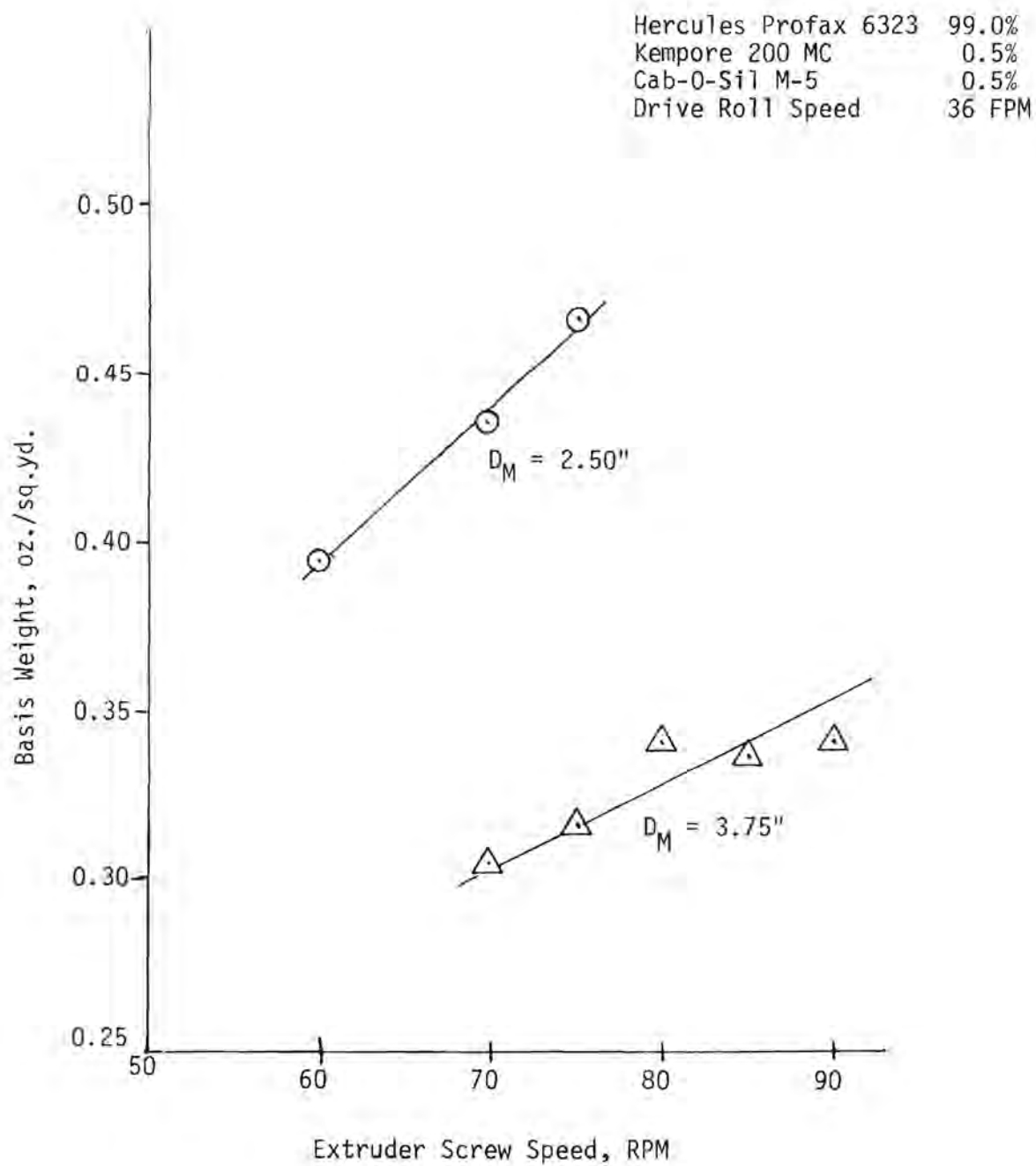
Figure 6

EFFECT OF PROCESS AND DESIGN CONDITIONS

the same processing conditions are lower for the larger diameter mandrel due to the additional TD stretching introduced by the larger mandrel.

Since higher basis weights are desired, it is apparent from Figure 6 that higher screw speeds are preferred. Unfortunately, at higher speeds than the indicated data the web piles up on the mandrel -- as observed with lower drive roll speeds. As discussed in section 3-d, design changes are necessary.

The effect of extruder screw speed on specific tenacity is shown in Figure 7. Specific tenacity, the tenacity divided by the basis weight, is shown in order to remove the direct impact of basis weight changes on tenacity. Despite this adjustment, all the specific tenacities increase with screw speed. It was expected that the MD tenacity would decrease, the TD tenacity would increase and the average tenacity would remain constant as screw speed increased. It appears that all the tenacities increase because the higher basis weight webs exhibit less dependence on individual flaws as well as a direct dependence on basis weight. This additional dependence on basis weight can be seen in Figure 8 in which average specific tenacity is plotted against a basis weight. Without this dependence on flaws the average specific tenacity should be independent of basis weight.

Further evidence of the dependence of the mechanical properties on flaws can be ascertained from the reported tensile strength and density of unoriented Profax 6323 of 5,300 psi and 0.903 g./cm.^{3}. Converting this data to specific tenacity, a value of 7.85 (lb/in)/(oz/sq. yd.) is obtained, significantly greater than the average specific tenacities obtained.
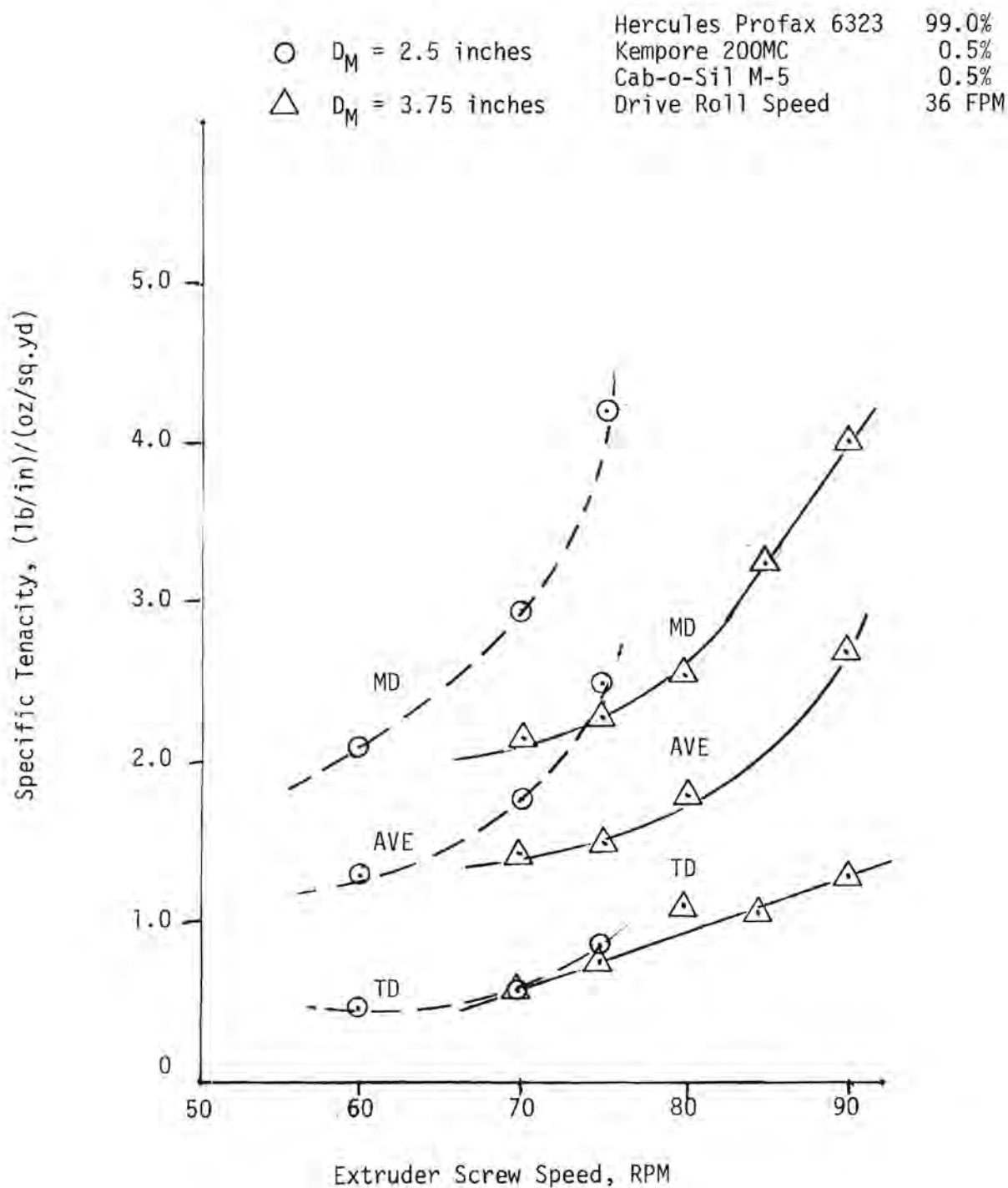
Figure 7

EFFECT OF PROCESS AND DESIGN CONDITIONS

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Hercules Profax 6323   99.0%
Kempore 200MC   0.5%
Cab-o-Sil M-5   0.5%
Drive Roll Speed   36 FPM

$D_M = 3.75$

$D_M = 2.50$

Average Specific Tenacity, (lb/in)/(oz/sq. yd.)

Basis weight, oz./sq. yd.
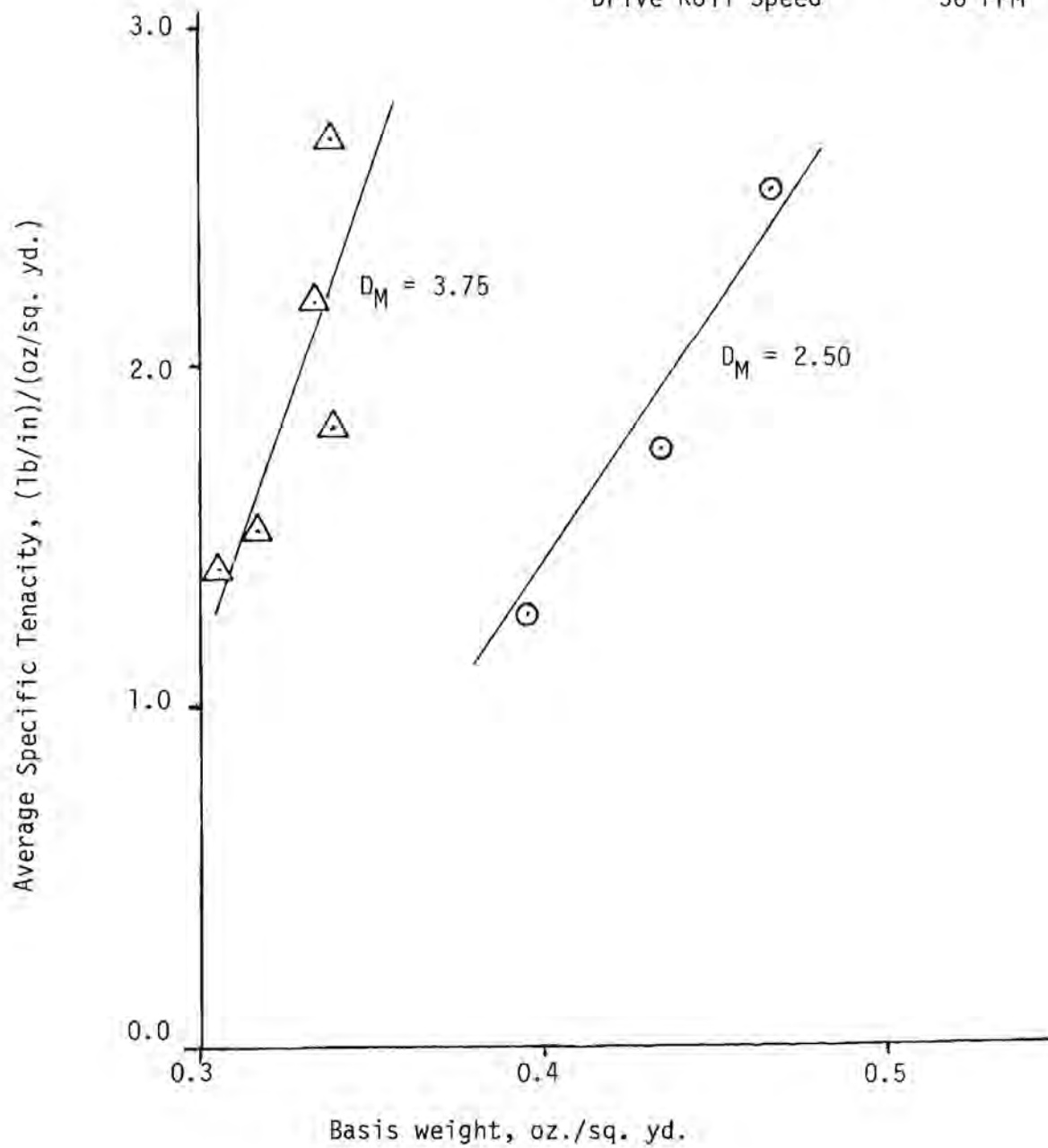
Figure 8

EFFECT OF BASIS WEIGHT ON TENSILE PROPERTIES

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It is noteworthy in examining Figure 8 that average specific tenacity is greater with the larger diameter mandrel for comparable basis weights. Also the tenacity imbalance is much less for the larger diameter mandrel. These desirable features imply that a study of mandrels larger than 3.75" in diameter would be beneficial. Further work in this direction is planned.

The effect of extruder screw speed on air permeability is shown in Figure 9. The permeability decreases as screw speed increases because the basis weight increases and finer fibrillation is obtained. Since the permeability should decrease as the basis weight increases a normalized value of permeability can be obtained by multiplying the permeability by the basis weight. When this calculation is made the normalized permeability still decreases as the screw speed increases, suggesting that finer fibrillation is obtained at higher screw speeds. Also, for comparable basis weights, the normalized permeability is lower when the larger diameter mandrel is used. Once again, higher screw speeds and larger diameter mandrels are preferred.

Photomicrographs which indicate the effect of process and design conditions on web structure are shown in Figure 10. Comparing the micrograph on the extremes, the sample on the right has a lower basis weight and also a lower permeability. Thus, more fibrillation has been induced by processing with the larger diameter mandrel at higher rates.

e.   Other parameters

Air blowing is used to cool the extrudate. In most of the process experiments room temperature air and external blowing in a fixed, inward direction has been used. The quantity of air flow has been adjusted by
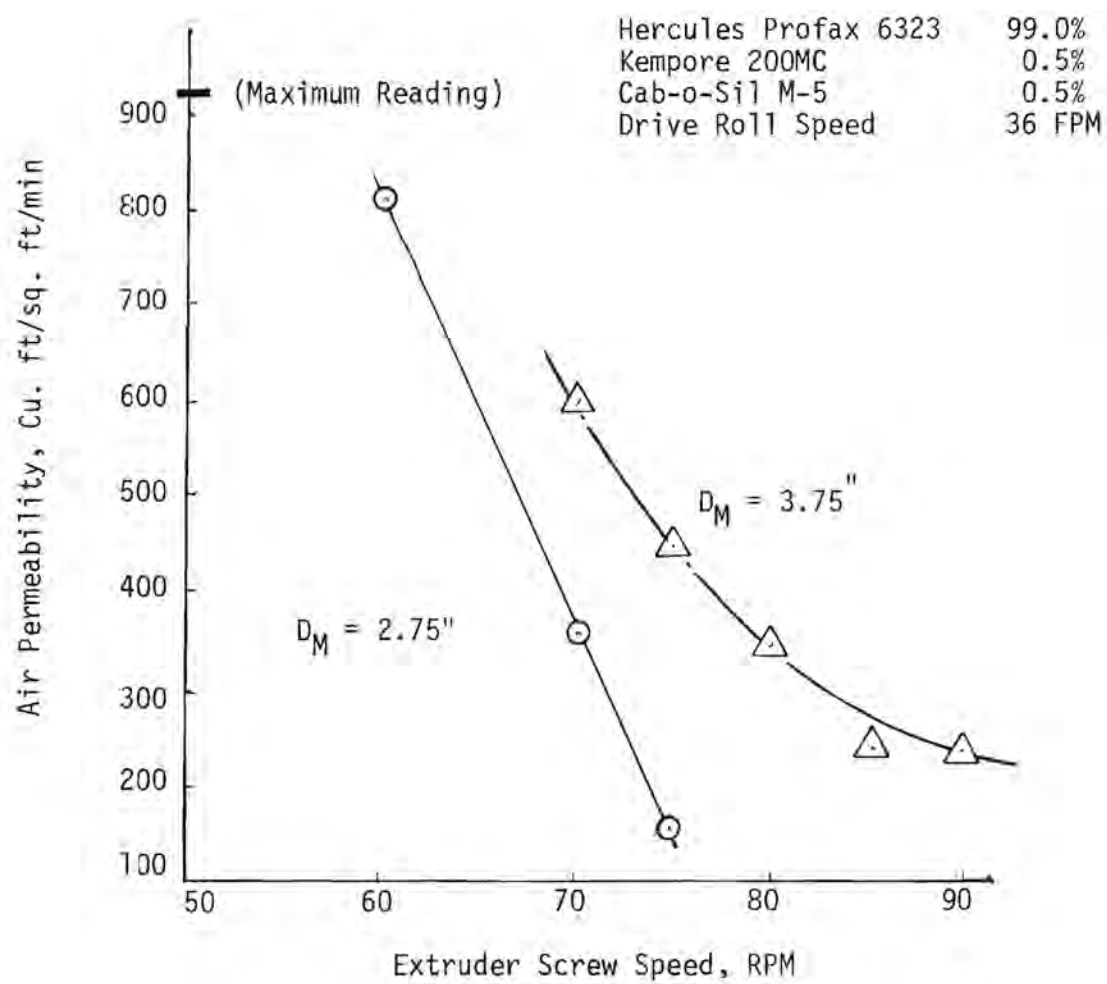
Figure 9

EFFECT OF PROCESS AND DESIGN CONDITIONS

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$D_M = 2.5"$
70 RPM

0.25 inch

$D_M = 3.75"$
70 RPM

4X Magnification

0.25 inch

$D_M = 3.75"$
85 RPM

0.25 inch
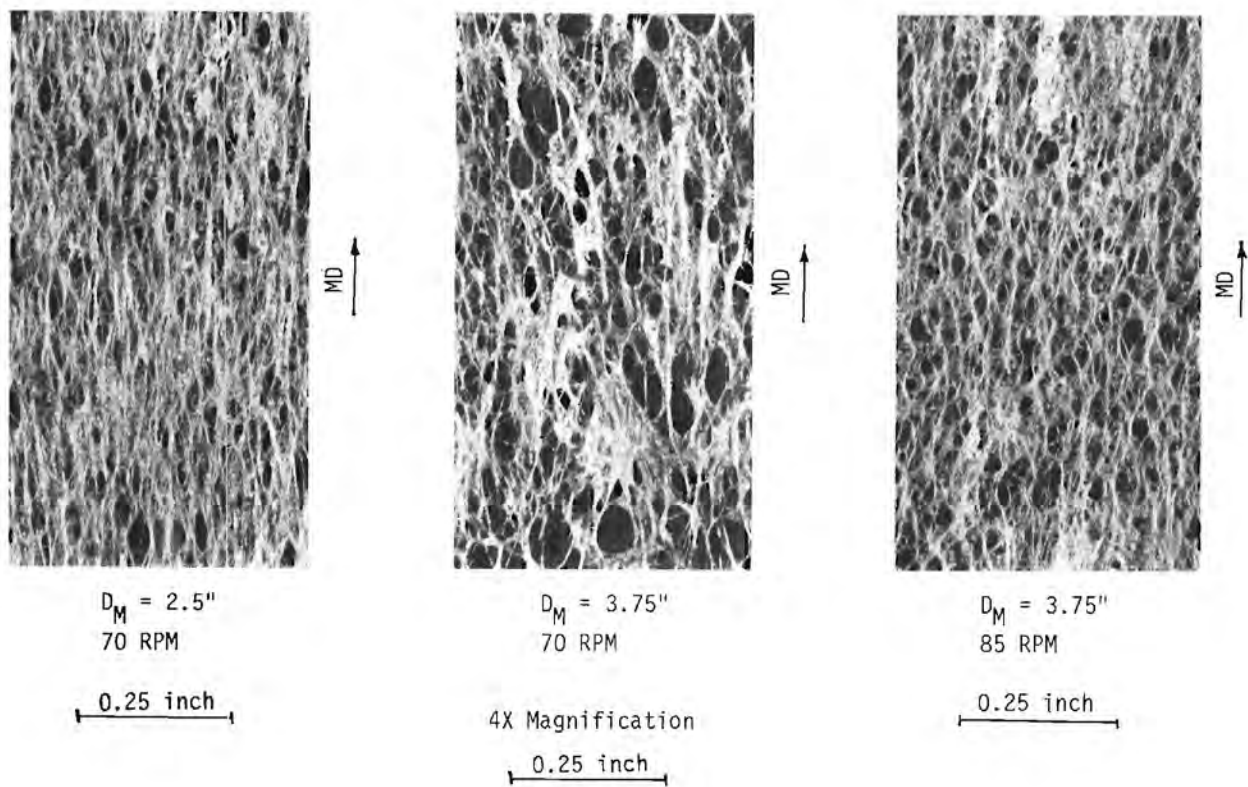
Figure 10

EFFECT OF PROCESS AND DESIGN CONDITIONS

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regulating the air pressure, but the quantity of air flow was not measured. Generally an air pressure near 20 psig was necessary to obtain stable web formation. Also it was necessary to increase the air pressure moderately as the screw speed was increased. Excessive air flow would cause too much cooling and it would not be possible to stretch the web over the mandrel. Inadequate air flow would cause excessive thinning and then failure of the web. Because the air flow influenced the ability to operate the process, it was not possible to evaluate the effect of air flow on web properties. It is expected that air temperature and flow direction can influence web properties, but the necessary equipment modifications to evaluate these factors have not been made at this time.

Preliminary studies of heat treatment and further stretching downstream from the mandrel have been started. It is anticipated that heat treatment and further stretching would improve the strength of the fibril junctions, permit further molecular orientation and reduce the tenacity imbalance. Since an on-stream, post-forming treatment is difficult to construct, an off-stream system was built. This system consisted of a heated cylinder and hand stretching.

Stretching tests were run at 140 and 145°C. Stretching was performed in four ways: (1) MD direction, (2) TD direction, (3) MD and then TD direction and (4) TD and then MD direction. The degree of stretch could not be precisely controlled and the samples were free to contract laterally. Some test results for stretching at 145°C are reported in Table 9. By stretching in one direction significant improvements in tenacity can be achieved without causing a major drop in basis weights. When TD stretching is performed, the

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TABLE 9

Post Extrusion Stretching

145°C Stretch Temperature

Stretch Direction	Elongation Ratio[1] MD	TD	Tenacity (lb/in)/(oz/sq.yd.) MD	TD	Basis Weight (oz/sq.yd.)
None	1.0	1.0	2.8	1.1	0.36
1. MD only	1.3	0.9	3.9	-[2]	0.36
2. TD only	0.7	2.0	-	2.4	0.32
3. MD and then TD	1.1	1.5	3.1	-	0.23
	1.0	1.9	3.5	-	0.19
	1.0	1.5	-	1.3	0.29
	1.0	1.6	-	1.8	0.23
4. TD and then MD	1.1	1.3	2.5	-	0.27
	1.3	1.3	2.0	-	0.30
	1.2	1.5	-	1.2	0.28
	1.6	1.3	-	1.4	0.19

1. Final length/original length

2. Sample too small to test

MD tenacity drops and a web with balanced properties can be obtained. By sequential stretching it is possible to improve the tenacity in both directions provided stretching is done in the MD direction first. Through sequential stretching a signifcant drop in the basis weights occurs.

To date, the improvements in properties due to post extrusion stretching are not as great as expected. Also, it appears desirable to construct an on-stream post extrusion stretching device in order to obtain more consistent results. An on-stream system is being designed.

f. Conclusions

Considerable variations in web properties can be achieved by modifying process parameters. It appears particularly desirable to increase the extruder speed and/or decrease the wind-up rate in order to achieve higher basis weights which, in turn, increases the specific tenacity. Also, operating with larger diameter mandrels increases the degree of fibrillation and lowers the tenacity imbalance. Heat treatment and post extrusion stretching provide further improvements. At the present time, design modifications must be made in order to exploit these processing trends.

4. Use of Tubular Biaxial Stretching for Fabric Formation

a. Introduction

A fabric of relatively high tenacity in two directions is desired. Expansion of tubular structures, as in blown film extrusion, is a convenient means for introducing orientation and strength in two dimensions. The task in considering tubular expansion of fabric can be reduced to modifying a blown film extrusion system to permit biaxial stretching of a permeable web.

b.   Process Description

The process is shown in Figure 11. The extruder, obtained from Haake, Inc., has a 3/4" barrel diameter and a 25/1 barrel length to diameter. There are three barrel temperature control zones and a fourth die temperature control zone. A constant taper screw with a 3/1 compression ratio has been used for the bulk of these studies.

The circular die, air cooling ring and blown film take-off system were all obtained from Haake, Inc. for this research. The blown film die is 1" O.D. with a 40 mil gap. It is equipped for air blowing through the center of the die. The drive roll speed can be continuously varied. The forming mandrel was developed for this work and is discussed below.

c.   Process Development

It was not possible to biaxially stretch the permeable web by air blowing. Various attachments were designed to direct the air passing through the center of the die outward against the web. Some web stretching was achieved but operation of the process was unstable. Consequently, a mechanical forming mandrel was developed.

The two principal forming mandrels used in this work are shown in Figure 12. As Figure 12 suggests, the web only touches the mandrel at the top. This feature, by minimzing the dynamic friction between the web and the mandrel, significantly contributes to stable web formation.

d.   Conclusions

A process has been developed for extruding and biaxially stretching tubular webs. However, the web formed is not of adequate quality for fabric use. As shown in Table 10 the web produced compares favorably with web

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Polymer Flake
and Additives

Tubular Fabric
Forming Mandrel

Drive Rolls

Air Cooling Ring

Drive

Extruder

Circular
Die
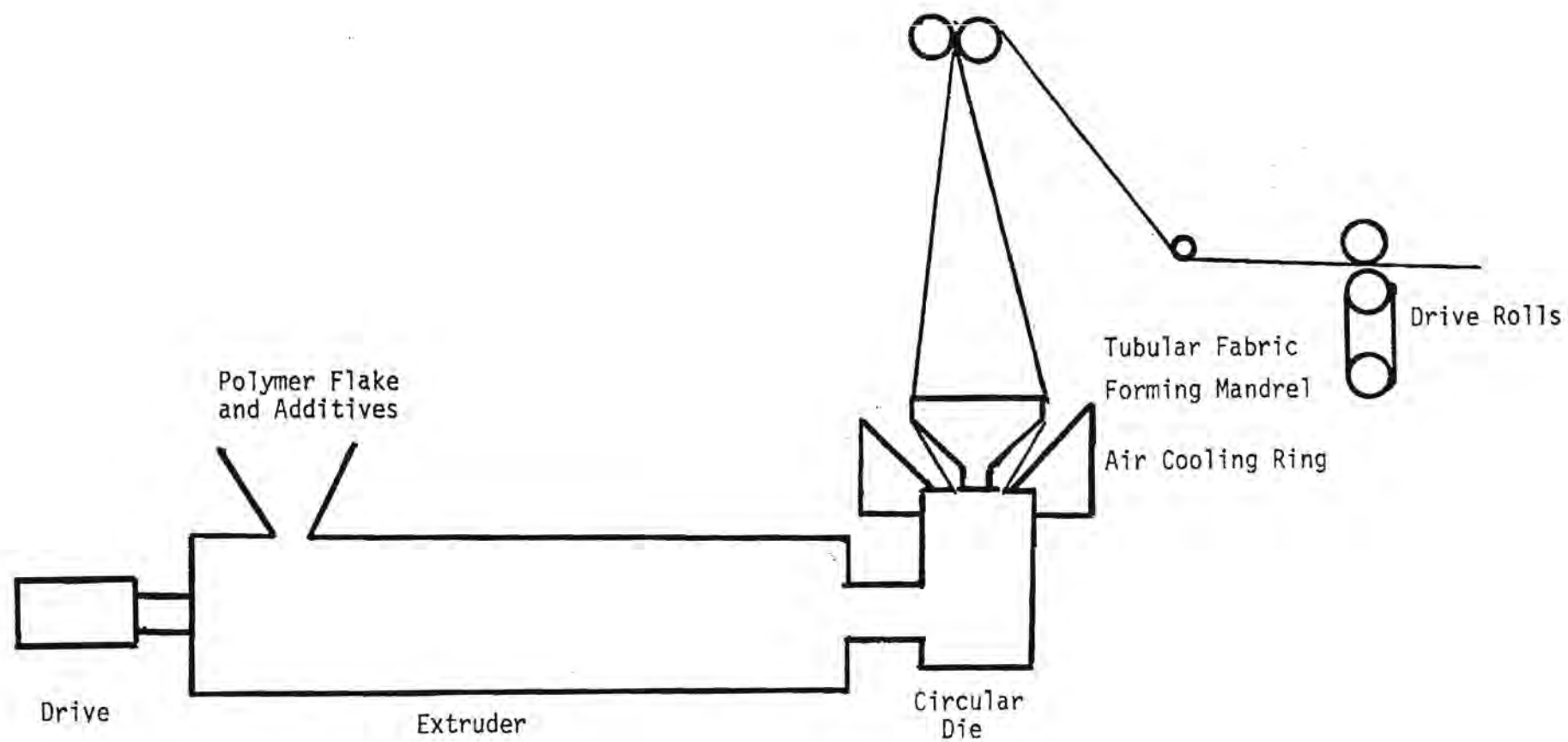
Figure 11

TUBULAR FIBRILLATION PROCESS

Figure 12

TUBULAR FIBRILLATION PROCESS

FORMING MANDREL DESIGN
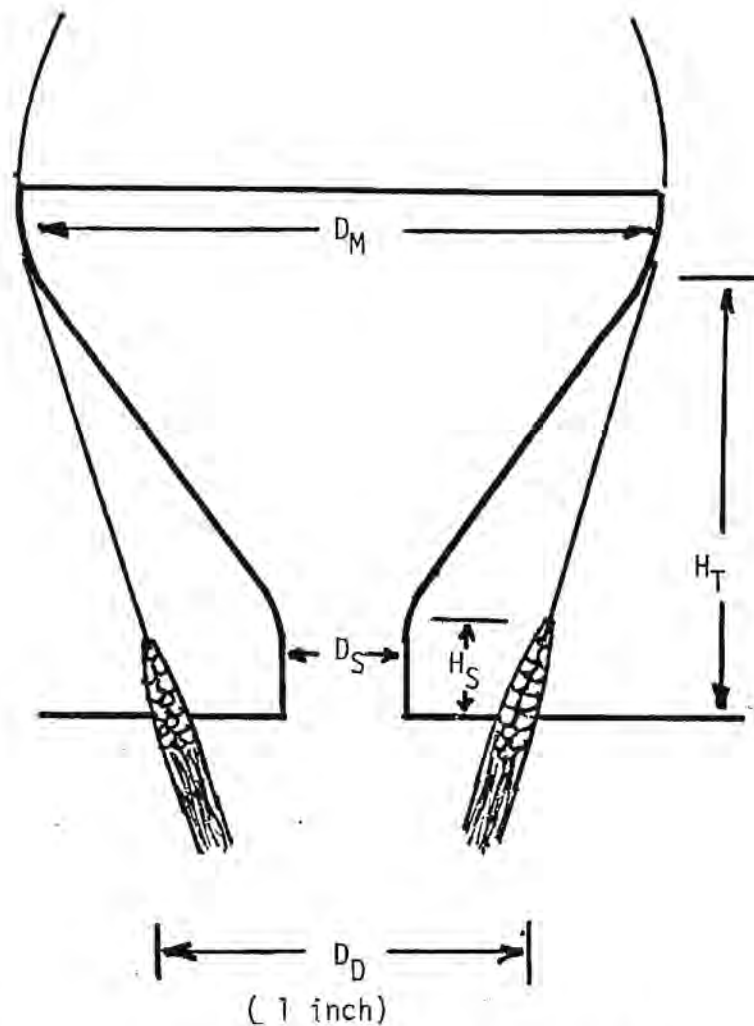
Mandrel A

D_M = 2.5 inches

H_T = 1.75 inches

Mandrel B

D_M = 3.75 inches

H_T = 3.0 inches

55

TABLE 10

Comparison of Polypropylene Web Properties

from Different Sources

Source	Basis Weight (oz/sq.yd.)	Specific Tenacity (lb/in)/(oz/sq.yd.)		Air Permeability (cu ft/sq ft/min)
		MD	TD	
Current Work	0.34	4.0	1.3	220
PNC	0.18	5.9	0.2	>926
W. R. Grace	0.19	1.2	0.5	>926

produced by similar means at W. R. Grace and PNC, Inc.

As indicated in the prior sections a number of additional modifications are necessary to extend the processing range of the system in order to improve the properties of the web. These modifications are primarily changes in die and mandrel design to permit formation of higher basis weight webs with less tenacity imbalance. Also further heat treatment and stretching downstream from the mandrel will be introduced to upgrade the mechanical properties of the webs.

B. Fiber-to-Garment

   L. Bangert

a. Introduction

   The objectives of the fiber-to-garment research are: (1) to develop the
technology required to produce fiber-woven garments; and (2) to replace the
processes of spinning staple fibers into yarn, of weaving yarn into fabric,
and of tailoring garments from fabric, by the process of fiber weaving.
Accomplishment of these objectives could increase productivity and reduce
material consumption.

   Fiber weaving consists of:

   (1) separation of tufts into single fibers, or strands with a small
number of fibers;

   (2) straightening fibers;

   (3) orienting fibers or strands of fibers in prescribed directions;

   (4) transporting the oriented fibers;

   (5) depositing individual fibers in parallel arrays;

   (6) intertwining the fibers.

In the present research, the effort is concentrated on orienting, trans-
porting, and depositing the fibers using air flows and aerodynamic forces.

   There have been some studies of the movement of fibers and other solid
matter by air flows for textile-industry related applications[18-23]. Only
the work of Edberg[18] is related to the problems of straightening and orienting,
as well as transporting fibers. In his studies, air flows with fibers were
observed in straight ducts which had different degrees of convergence. He

found that large percentages of the fibers could be made parallel. To do so, however, required high air speeds (30 to 100 m/sec), which is undesirable for ordered deposition of the fibers.

There have also been a number of studies of the fluid mechanics of fiber suspensions[24-29]. These studies are related to such problems as the resistance to motion of these fiber suspensions, and so to pumping requirements, etc. Some of these basic results may prove to be of value to the present study, however, when the transport and orientation of multiple fibers is studied.

As stated above, the present research is concerned with orienting, transporting and depositing the fibers. Once the fibers have been deposited in an ordered way, they must be intertwined. A technique that is potentially applicable is that due to Evans[30]. Evans' process consolidates any web, mat, or batt of loose fibers, supported on an apertured surface such as a perforated plate or a woven wire screen, by the use of high-pressure (200 psig) liquid jet streams impinging perpendicularly to the mat. This process produces stable, strong nonwoven fabrics which resemble textile fabrics prepared by conventional spinning and weaving.

b. Experimental Apparatus

The objective of this study is to develop a system which aligns fibers parallel to the mainstream air flow, and which provides an orderly deposition of the fibers. In general, fibers injected into an air stream have a random orientation. If the air stream has uniform velocity in the transverse direction, the relative velocity between the fluid and the fiber is the same all along the fiber. Then, no torque will be applied to the fiber, and so there

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will be no change in fiber orientation. Thus, velocity gradients in the air stream are required to produce changes in fiber orientation. These velocity gradients must be of special types, however, so that the fibers are turned parallel and then remain so for a sufficiently large distance.

At the beginning of this study, it was considered very important to have a system which could produce a wide variety of velocity gradients in the air flow. This system needed to be flexible, so that changes in the flow conditions could be made rapidly and easily. These considerations led to the use of a small wind tunnel with counterflow jets. Sketches of the counterflow jets and of the system are shown in Figures 13 and 14. The main air stream moves through the wind tunnel. The velocity profiles of the main air stream can be easily changed by changing the jet flow rate and/or the jet angle. The jets can be directed from 0° to 180°. Each tube has an 11/16 inch O.D., with 37 equally-space orifices of 3/32 inch diameter.

A probe holder and traverse mechanism has been provided to allow flow field measurements in the test section. A longitudinal slot in the upper wall of the test section allows traverses in the main flow direction. Velocity profiles in the test section have been measured by traverses of a pitot-static tube, for each combination of main-stream and jet conditions.

A fiber injector was designed to provide a fixed and known orientation of the fiber relative to the mainstream, before release of the fiber. The objective is to relate the initial fiber angle to the final fiber angle, as a function of the other flow parameters. The injector holds the fiber at its ends. The clamps are held closed by springs. The fiber is released by the

Figure 13. Counterflow Jet System for Adjusting Velocity Gradients

EXHAUST TO ROOF

CONTRACTION CONE

72   24   27   72

16.25

7°

COUNTER FLOW AIR
INJECTION TUBES

TRANSITION   DIFFUSOR

12×12 TEST SECTION

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PLENUM WITH
DAMPING SCREENS

SECONDARY AIR

1" PVC SUPPLY

6" PVC

PRESSURE REGULATOR — WITH REDUCING COUPLING

4" HEADER

FLOOR LINE

PRIMARY AIR

PLENUM IS 1.97' × 1.97'
CONTRACTION RATIO : 3.88

CONSTRUCTION MATERIAL

TRANSITION - FIBER GLASS
DIFFUSOR - 3/8 PLYWOOD
PLENUM - 3/4 PLYWOOD
CONTRACTION - 3/8 PLYWOOD
TEST SECTION - 3/8 PLEXIGLAS
EXHAUST DUCT 26 GA. GALV.

SHOWN		J.J.H		J.J.H	E-16-662
MATERIAL	NO. REQ.	DRAWN BY	APPROVED BY	3 O QUANER	PROJECT
SCALE 1"=20'		FIG. 5 - FIBER MOTION			SK-1
DATE 6-24-75		WIND TUNNEL			

SCHOOL OF AEROSPACE ENGINEERING
(DANIEL GUGGENHEIM SCHOOL OF AERONAUTICS)
GEORGIA INSTITUTE OF TECHNOLOGY

Figure 14. Wind Tunnel for Studies of Fiber Motion

action of a solenoid, which pushes the clamps open. This injector has proved to have one deficiency, in that when it is in the open position, the fiber must be carried away by the force of the air stream on the fiber. There are some difficulties with this, which will be discussed in the section on Results.

The principal data on the fiber motion are multiflash photographs of the fiber trajectory. Each photograph shows the fiber at several points along its path. The photographs are made using a stroboscopic light source, with the room darkened. The camera being used is a Nikon F 35 mm, with a 50 mm f/1.4 lens. The light source is a General Radio Type 1538-A Strobotac electronic stroboscope. Most of the photographs have been made with Kodak Tri-X (ASA 320) film. The distance from the camera to the moving fiber is approximately 3 ft. Apertures f/2 and f/2.8 have been used primarily.

The present lighting arrangement has the lamp mounted on the wind tunnel centerline, about 4 ft downstream of the jets. With this setup, the fibers travel close to the center of the light beam, and receive maximum illumination relative to their surroundings. This lamp position does not interfere with the flow in the region of interest.

c. Results

The basic concept of fiber orientation using counterflow jets was first demonstrated in experiments using a water table. Figure 15 shows a sketch of the flow configuration. The successive positions of the fiber are drawn from photographic records.

Upstream of the jets, the fiber is in a uniform flow, and its orientation does not change. The jets produce an effective nozzle wall for the mainstream

COUNTERFLOW JET

MAIN FLOW

FIBER

COUNTERFLOW JET
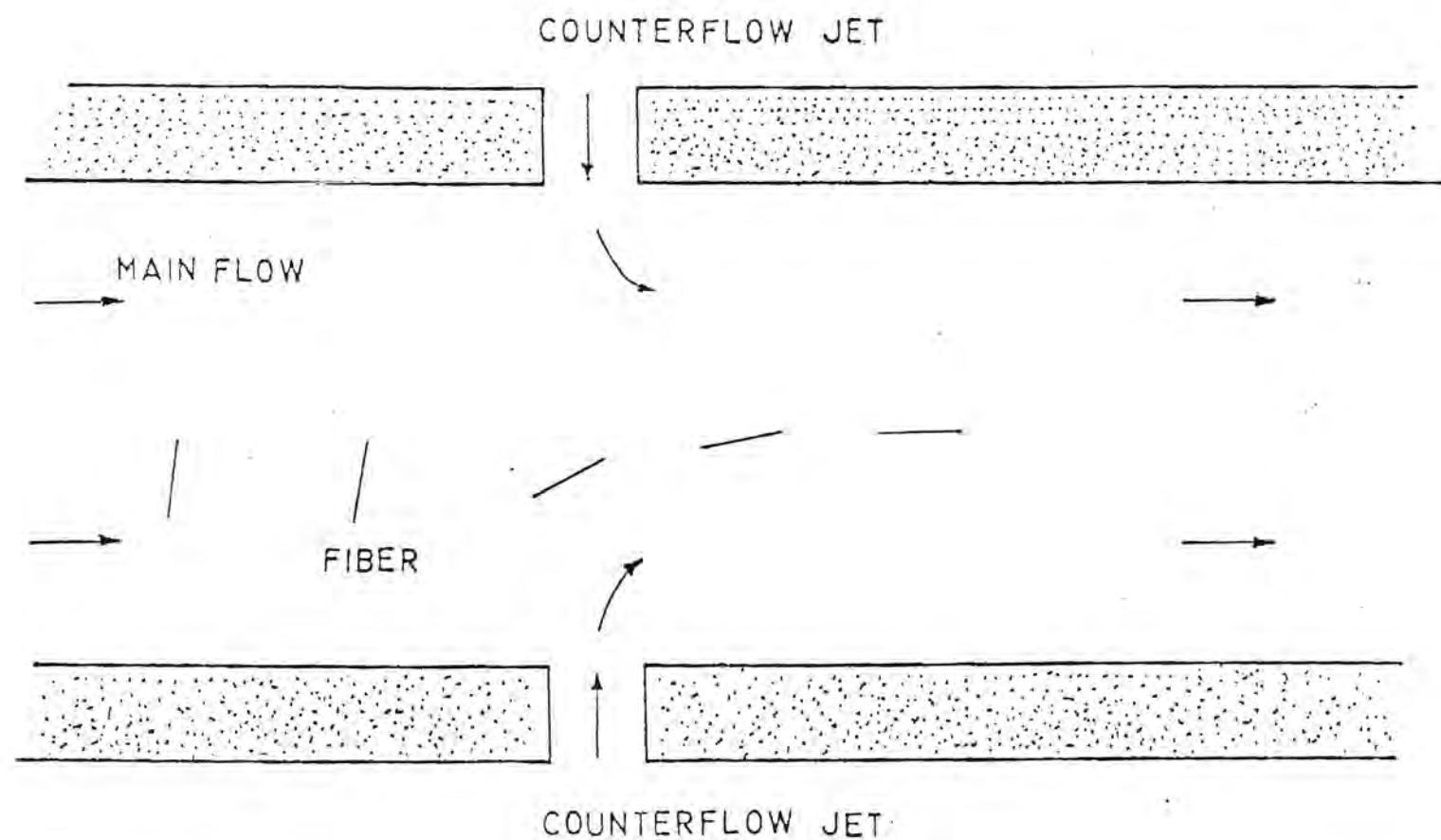
Figure 15. Sequence of Fiber Positions as Observed on the Water Table

so that the streamlines converge toward the center of the duct and the flow accelerates. Downstream of the jets, these main flow streamlines become essentially parallel to the duct centerline.

A fiber that is parallel to the streamlines upstream of the jets remains so throughout, and continues parallel to the duct centerline downstream of the jets. A fiber that is initially perpendicular to the mainflow streamlines is rotated to a parallel orientation by the streamline convergence near the jets. The streamlines nearer the duct wall are accelerated toward the centerline, so that a resultant moment is applied to a fiber that lies across the streamlines. This moment goes to zero when the fiber becomes parallel to the streamlines, so the fibers retain their parallel orientation downstream of the jets.

The same parallel orientation has also been observed in the wind tunnel experiments. This behavior is shown in Figures 16 - 18. These photographs are but a small sample of many that have been taken. Parallel fiber orientation has not been achieved in some cases, however. Possible reasons for this will be discussed below.

The nondimensional parameters that govern the fiber motion in the wind tunnel are as follows:

(1) y_c/h, the position of the fiber release above the lower wall of the test section. Here, h is the height of the test section.

(2) $\dot{m}_\infty/\dot{m}_J$, the ratio of mainstream flow rate to jet flow rate.

(3) Θ_J, the jet angle

(4) Φ_o, the initial fiber inclination relative to the mainstream
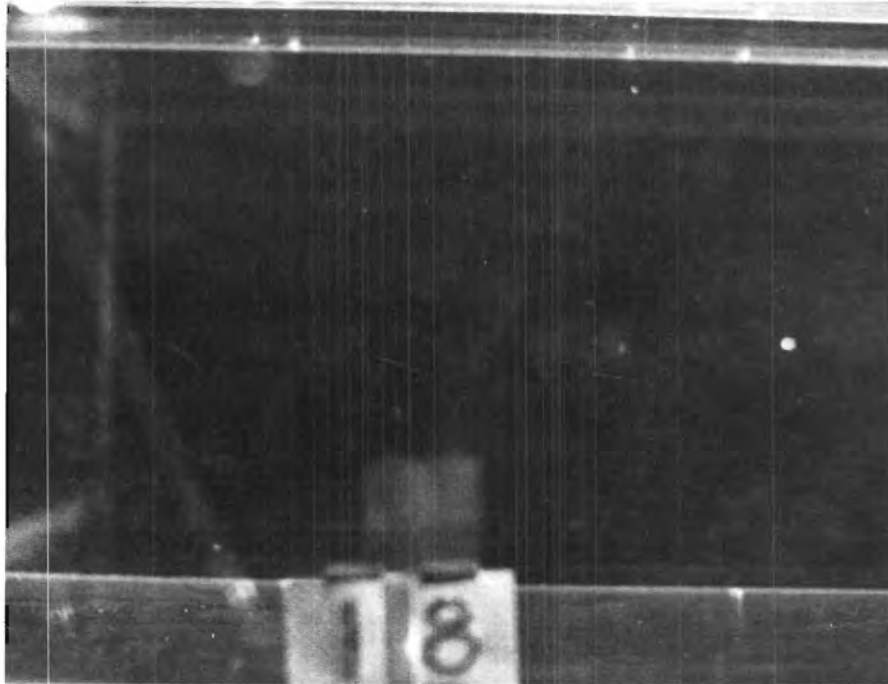
Figure 16. Fiber Parallelization by Aerodynamic Forces.
$\dot{m}_\infty/\dot{m}_J = 74$, $\theta_J = 90°$, $\phi_o = 90°$
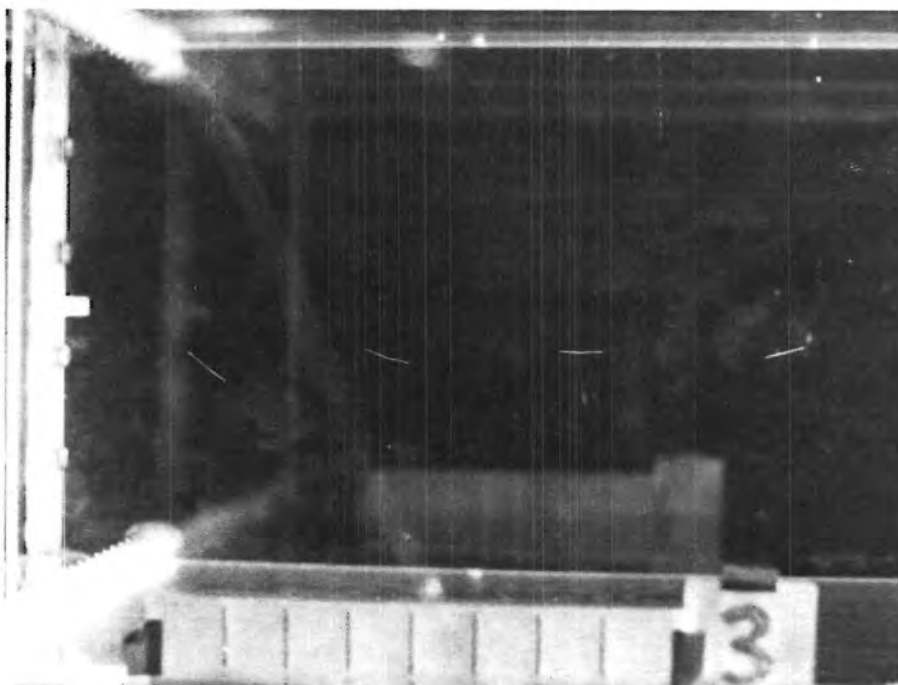
Figure 17. Fiber Parallelization by Aerodynamic Forces.
$\dot{m}_\infty/\dot{m}_J = 90$, $\theta_J = 90°$, $\phi \cong -50°$
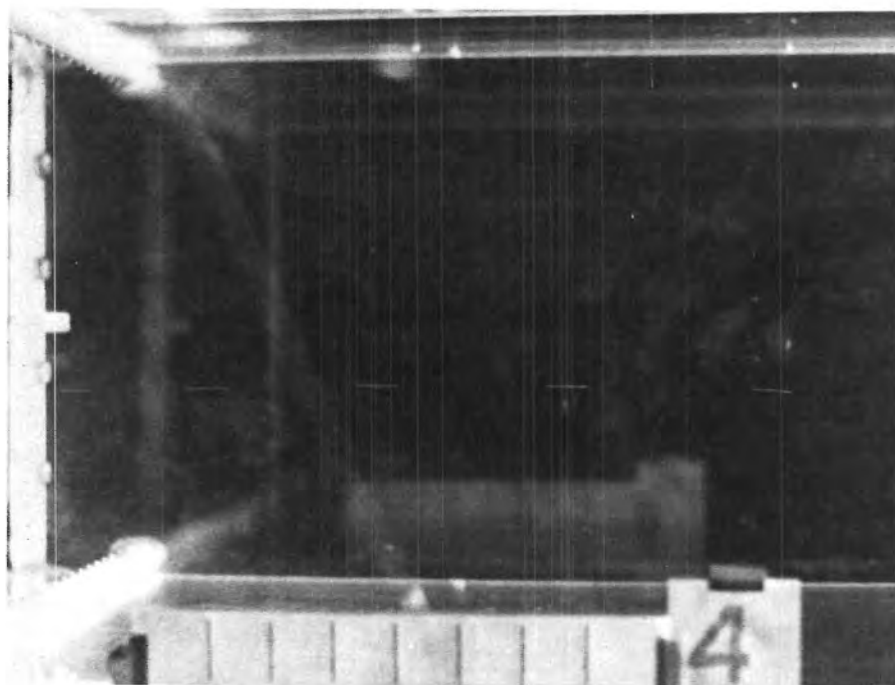
Figure 18. Fiber Parallelization by Aerodynamic Forces.
$m_\infty/m_J = 90$, $\theta_J = 90°$, $\phi_o \cong 0°$

(5) ℓ_F/d_F, the ratio of fiber length to diameter.

(6) ρ_F/ρ_∞, the ratio of fiber density to mainstream air density

(7) ℓ_F/h, the ratio of fiber length to test section height.

(8) $\rho_\infty u_\infty^2/\rho_F d_F g$, the ratio of the aerodynamic lift force on the fiber to the gravitational force. This parameter is a measure of how rapidly the fiber drops toward the lower wall of the test section.

(9) $\rho_\infty u_\infty d_F/\mu_\infty$, the Reynolds number.

In the experiments performed to date, the main parameters that have been varied are $\dot{m}_\infty/\dot{m}_J$ and θ_J. Some variations in Φ_o and in $\rho_\infty u_\infty^2/\rho_F d_F g$ have also been examined.

For almost every case tested, u_∞ = 20 ft / sec. The fiber was cotton, 1 inch long, and was one of three strands that compose No. 50 white cotton mercerized thread. This yields a d_F of approximately 62 μm. The values of the nondimensional parameters used thus far in the experiments are given below:

$y_c/h = 0.4$

$\dot{m}_\infty/\dot{m}_J = 26$ to 130

$\theta_J = 90°$ (normal to mainstream), 135°, and 180° (directly upstream)

$\Phi_o = 90°$ and $45°$

$\ell_F/d_F = 360$ to 620

$\rho_F/\rho_\infty = 1300$

$\ell_F/h = 0.083$

$\rho_\infty u_\infty^2/\rho_F d_F g = 48$

$\rho_\infty u_\infty d_F/\mu_\infty = 25$

Air properties at standard conditions have been used in the above, as these are close to the actual mainstream conditions. Possibly a more meaningful Reynolds number would be one based on the relative velocity between the fiber and the air. This relative velocity is much less than u_∞, so that the corresponding Reynolds number would be markedly less than 25.

The principal experimental difficulties that have been experienced thus far are related to the behavior of the fiber injector. Evaluation of many photographs has revealed that the fiber injector often does not provide the controlled release that was intended. The apparent reason for this is that the fiber ends stick to the injector jaws for a very brief interval after the jaws are opened. When this happens, one fiber end often releases before the other, and the fiber is rotated to a different initial angle before complete release occurs. In several cases, this uneven release provided an initially erratic motion that caused the fiber to descend into the region of rotational flow near the wall and downstream of the jets.

Figure 16 has $\dot{m}_\infty/\dot{m}_J$ = 74, Θ_J = 90°, and Φ_o = 90°. The flow is from left to right. The jet tubes can be seen at the far left at the top and bottom of the test section. This is an example of the fiber orientation system working properly. The flash rate of the stroboscopic light is 3600 flashes/min. The fiber release is at 0.4 h, so that the fiber experiences a nonsymmetrical flow field as it enters the influence of the jets. The streamlines converging toward the center produce a counter-clockwise moment on the fiber and rotate it parallel to the streamlines. The parallelization occurs in less than 1 ft. distance, and the fiber remains at low speed.

In Figures 17 and 18 $\dot{m}_\infty/\dot{m}_J = 90$, $\Theta_J = 90°$, and $\Phi_o = 90°$. The scale at the bottom of the photograph shows distance in 1 inch intervals. In Figure 17, the actual Φ_o is less than 90°, because of improper release by the injector. The fiber is rotated counterclockwise to a parallel position in a distance of about 1 ft. At the far right of the photograph, the fiber is at about 20°. This is possibly caused by interaction with the shear layer produced by the lower jet. This serves to illustrate an important point regarding this method of fiber orientation and deposition. This is that the fiber deposition will occur a short distance downstream of the jets (about 1 foot), as no greater distance is required for orientation.

Figure 18 shows a case in which the fiber was actually released parallel to the mainstream. It then continued to move parallel to the streamlines, as expected.

Another result of these experiments is that, at the lower jet mass flow rates ($\dot{m}_\infty/\dot{m}_J = 120$ and 130), there was no effect of the jets on the fiber motion. This was because, at this injector location, the streamlines were not deflected enough to produce the necessary moments on the fiber.

From the results that have been obtained thus far, it may be concluded that the basic principal of operation of the counterflow jets to produce parallel fiber orientation has been demonstrated. There are a large number of parameters yet to be examined, however, as is apparent from the above list of nondimensional parameters. First, a more complete evaluation of the effects of $\dot{m}_\infty/\dot{m}_J$ and Θ_J for the present geometry and fiber properties will be performed. Then, the effects of changes in geometry, such as a different

injector location relative to the jets, will be considered.

At this time it also seems necessary to modify the fiber injector so that greater control over the initial fiber angle Φ_o can be obtained. The new injector design has not yet been completed. Its basic feature, however, will be a provision for positive release, instead of relying on aerodynamic drag to remove the fiber.

An analysis of single-fiber motion in a two-dimensional shear was also developed. This analysis is a combination of a computer code for the calculation of turbulent shear flows with a code for the fiber dynamics. This new combined code is completely operational.

Figure 19 shows the results of calculations for which the initial air velocity profile was: (1) logarithmic, and (2) linear. The logarithmic profile is characteristic of fully-developed channel flow. The linear profile is a simplification of a profile that would result from strong upstream blowing by the counterflow jets. The larger transverse velocity gradient for the linear profile produces a much more rapid change in fiber angle, so that the fiber is essentially parallel within two duct widths of the point or release.
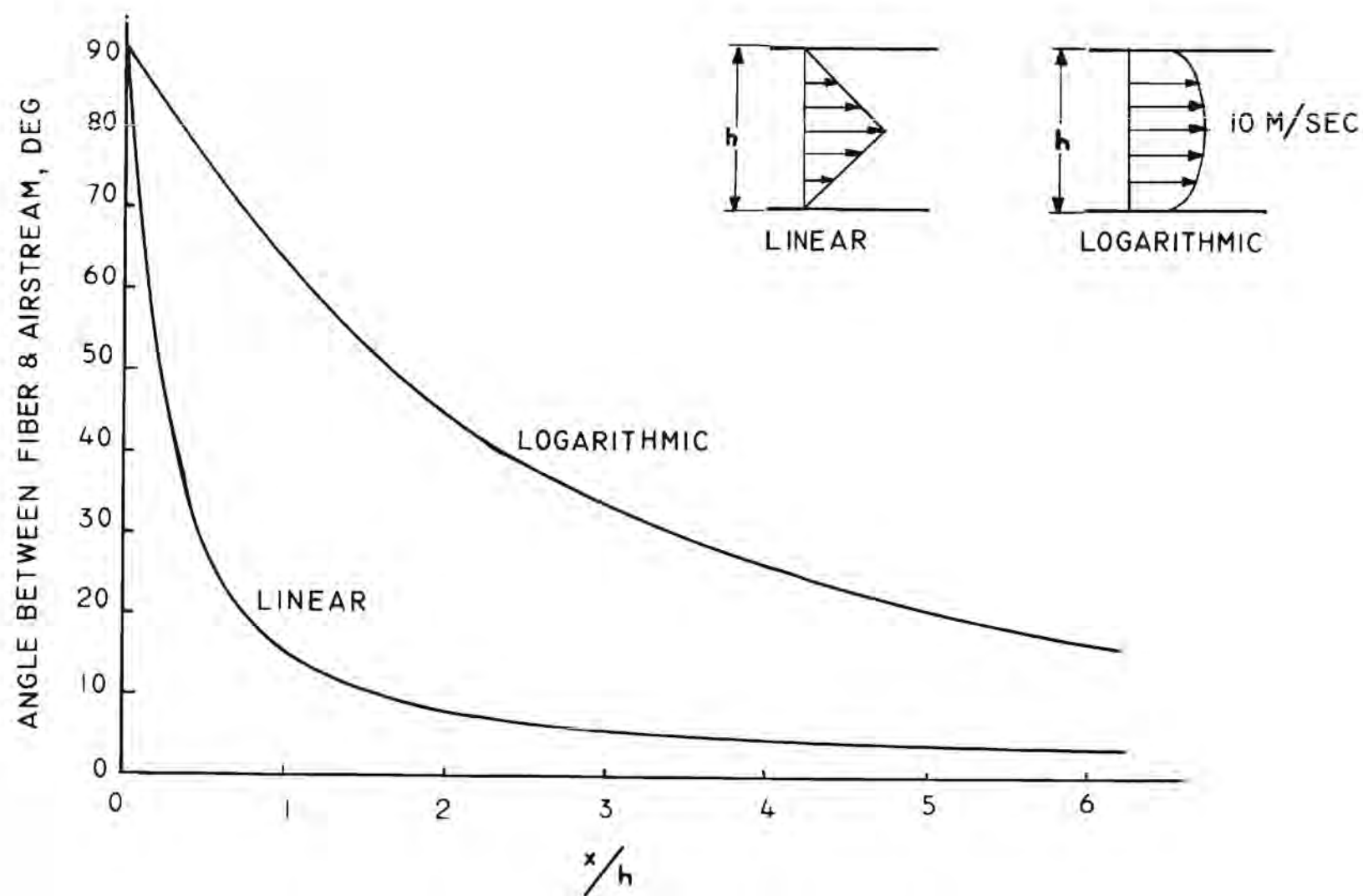
Figure 19. Fiber Orientation

d. Conclusions

Experiments have been performed to study the orientation and transportation of a single fiber, using the counterflow jet system to affect fiber orientation. Preliminary experiments were conducted using a water table, while the bulk of the experiments have been performed using a small wind tunnel. The following conclusions may be drawn from these results:

(1) For the range of experimental parameters considered, the counterflow jet system provides a method for aligning the fibers parallel to the air stream.

(2) The fiber alignment occurs in a very short distance (less than one foot), while maintaining a low air stream velocity (20 ft/sec or less).

(3) The intended flexibility of the counterflow jet system has been realized, in that easy and rapid changes in flow conditions can be achieved.

Present efforts are directed toward further defining the ranges of the experimental parameters for which parallel fiber orientation can be achieved. These experiments include consideration of fiber material other than cotton (and so alter ρ_F/ρ_∞ and $\rho_\infty u_\infty^2/\rho_F d_F g$).

e. Future Work

Future work will be concentrated on studies of multiple-fiber
flows, as this is the case of interest for practical applications.
Again, these studies will be directed toward orienting the fibers
parallel to the main stream, transporting them a short distance, and
depositing them in an ordered manner. The techniques developed
in the single-fiber studies will be applied to the multiple-fiber
flows, and modifications introduced if necessary.

The same basic experimental facilities will be used, as they
were designed for this purpose. A multiple-fiber injector will be
designed. It will not have to provide controlled initial orientation,
however.

The major additional parameters being introduced in these experiments
are $\dot{m}_F/\dot{m}_\infty$, the ratio of fiber mass flow rate to main stream air mass
flow rate; and, the characteristics of the fiber deposition devices.
Fiber deposition will be an important part of the future work.
For example, a possible arrangement is to mount a screen in the test
section downstream of the jets, so that the screen makes a small
angle with the flow. The pattern of fiber deposition can then be
evaluated from the arrangement of the fibers collected on the screen.
The desired results of these experiments are measures of the degree of
satisfactory fiber orientation and deposition (e.g., per cent
fibers in desired direction) as a function of $\dot{m}_F/\dot{m}_\infty$, $\dot{m}_\infty/\dot{m}_J$, and θ_J.

IV.   Utilization Plan

The procedures and time schedule that are planned to ensure that the technology developed under the proposed program is transferred into the textile and apparel industries are outlined in Table 11.

On January 27, 1975 the National Science Foundation distributed a press release on this program. As a consequence, we received approximately fifty telephone calls and letters requesting more detailed information. As part of our effort to transfer the technology developed and obtain input from knowledgeable, interested persons in industry, a copy of the first semi-annual progress report was sent to these persons.

Georgia Tech hosted a Computer Assisted Apparel Production/Distribution Conference on October 28 & 29, 1975. The Conference was sponsored by the Apparel Research Committee of the American Apparel Manufacturers Association (AAMA). The Apparel Research Committee met the day after the Conference, October 30. At this meeting Drs. Freeston, Muzzy and Bangert made presentations on Advanced Technology Applications in Garment Processing. Activities planned and accomplishments to that point in time under Georgia Tech's RANN program were discussed.

On December 18, 1975, Dr. Muzzy presented a seminar on his activities at Deering Milliken Research Corp. in Spartanburg, S.C.

On March 2, 1976, Dr. Muzzy presented a paper at the International Nonwoven and Disposables (INDA) Conference in Atlanta. Drs. Freeston, Muzzy and Lundberg meet with the principals of PNC Company on March 7. PNC has been conducting research on fibrillated film for a number of years. A representative

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Table 11 Utilization Plan

	0	12	24	36

	0		12		24		36
Progress Reports	X	X	X	X	X	X
Research Reviews		X		X		X

Presentations to:

	0	36
State Textile Industrial Associations	X———————————————X
AAMA Research Committee	X ——————————————X

	36
Technical Conference & Demonstrations	X
Summary Technical Report	X

of W.R. Grace & Co. who was attending the INDA Conference met with Dr. Muzzy on March 4 to discuss the NSF RANN program. W. R. Grace has patents on fiber fibrillation techniques

Hercules, Inc. has made polypropylene polymer available for this program. As a consequence, Dr. Muzzy has kept them informed on the program progress.

On April 8, 1976 Dr. Freeston made a presentation on the program at the American Apparel Manufacturers Association (AAMA) Technical Forum II in Washington, D.C. The theme of the session was Showcase for Apparel Progress.

A review of the program is planned for April 12 at Georgia Institute of Technology. About sixty representatives from industry and Government have been invited. Their names are given in Section VIII.

Dr. Freeston will present a seminar on the program at Scott Paper Company, in Philadelphia on May 20. An invitation to speak at INDA's Idea 76 Conference in Chicago on October 20 has also been accepted. All expenses associated with these presentations will be reimbursed by the requesting organization.

Dr. Muzzy, in cooperation with the Society of Plastics Engineers (SPE), is organizing a conference on "Fibrillation Processes and Products."

Presentations will be made at the 1976/1977 annual meetings of the Georgia Textile Operating Executives and the Textile Education Foundation Division of the Georgia Textile Manufacturers Association. These presentations will keep local industry apprised of the direction of the research and the accomplishments. Industry comments and suggestions will be solicited.

Offers will also be made to make presentations at meetings of the Alabama, South Carolina and North Carolina counterparts of the GTMA; and again to the Research Committee of the American Apparel Manufacturers Association.

During the latter part of the second year of research, efforts will be made to secure an opportunity to present a report on the research at the annual meeting of the America Textile Manufacturers Institute (ATMI). A half-to-full day conference on the research will be held early in the third year as well as at the conclusion of the program. Representatives from primary textile and apparel producers, and textile and apparel machinery manufacturers would be encouraged to attend.

V.   Conclusions

A.   Polymer-to-Garment
 1.   Gas-polymer solutions

Polypropylene is soluble in n-butane at relatively low pressures (p ∼180 atm, ∼2700 psi). Solution densities are relatively low, from 0.44 to 0.53 g/cc. The system polypropylene-n-butane should present no pressure – temperature problems when used as a polypropylene transporting and forming system.

This result and preliminary results with other polymer-gas systems are sufficiently encouraging to justify fabrication of a pressure system capable of batchwise extrusion of polymer from the gas phase during the coming year. A particularly attractive aspect of this approach is the possibility of using gases such as carbon dioxide thereby eliminating the need for gas or solvent recovery systems.

 2.   Polymer fibrillation

The blown film extrusion process has been shown to represent a feasible approach to the production on nonwoven fabric for garment application. Certain formulation parameters can be varied to give improved product properties. These include:

1)   use of low molecular weight versus high molecular weight
     polymer for ease of processing;

2)   formulation with small particle size, self-nucleating
     blowing agents versus large particle, non-nucleating
     agents;

3)   minimization of the level of blowing agent ($\leq 0.5\%$) to pre-
     vent over-blowing and proper adjustment of the extruder

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temperature profile to prevent pre-blowing; and

4) formulation with 0.5% fumed silica (Cab-O-Sil M5) for

slightly improved properties.

Process parameter studies indicated that:

1) increasing extruder RPM at a fixed wind-up rate gives

improved properties;

2) decreasing wind-up speed at a fixed extruder RPM gives

improved properties;

3) design modifications are needed to operate at higher

RPM or lower wind-up speeds;

4) use of larger OD stretching funnels gives finer fibrillation

more balanced tensile properties and a higher level of

tensile properties; and

5) the operable temperature range is limited by the de-

composition properties of the blowing agent

The following ingredients when added to the formulation were found to be

negative in effect:

1) zinc stearate - it caused preblowing;

2) linear polyethylene - it resulted in decreased tensile properties:

3) sodium salicylate - off-color fabric with no property improvement

resulted.

Post processing treatments such as heat-stretching offer potential routes to

improve tensile properties and balance.

    In order to obtain fabric acceptable for garment application it will be

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necessary to

1) increase tensile properties

2) decrease non-uniformity and air permeability and

3) increase basis weight.

B. Fiber-to-Garment

It has been demonstrated that the counterflow jet system aligns fibers parallel to the air stream with an air stream velocity of less than 20 ft/ sec. Fiber alignment occurs in a very short distance.

Additional experiments are underway to define the ranges of the experimental parameters for which fiber parallelization can be accomplished.

VI. Future Plans

   A.   Polymer-to-Garment
      1.   Gas-polymer solutions

   A pressure system capable of batchwise extrusion of polymer from the gas
phase will be built early during the second year. A 250 ml capacity system
should be adequate to demonstrate the method. Operating conditions will be in
the 200 to 2000 atmosphere pressure range at temperatures of 100° to 250°C.

   The system will consist of a piston-less pump section contained in an oven
and barricade. In this "pump" gas is condensed in a pressure vessel, isolated,
warmed through the critical region, and heated to the temperature desired in
forming the polymer solution. Connected to the "pump", is another pressure
vessel in an oven and barricade; in this part of the system, polymer solutions
are formed. The pressure vessel can be tapped to extrude polymer solution in
either gas or polymer phase. The exit can be provided with a spinneret to
produce yarn-like structure or dies to form flat or tubular films.

   A line diagram of the extrusion system is given in Figure 20.

   Effects of pressure and temperature of extrusion and quenching on the
physical form of the extrudate will be studied. Particular attention will be
given to the difference between precipitating polymer inside the pressure
vessel followed by extrusion and extruding gaseous polymer solution which
should lead to precipitation in the spinneret holes and outside the spinneret.

   The effects of pressure and temperature on expansion and biaxial straining
of polymer (to draw it) will be examined. First emphasis will be on determining
the degree of expansion necessary to cause enough drawing to increase signifi-
cantly the strength and stiffness of polymer and to fibrillate the extrudate.
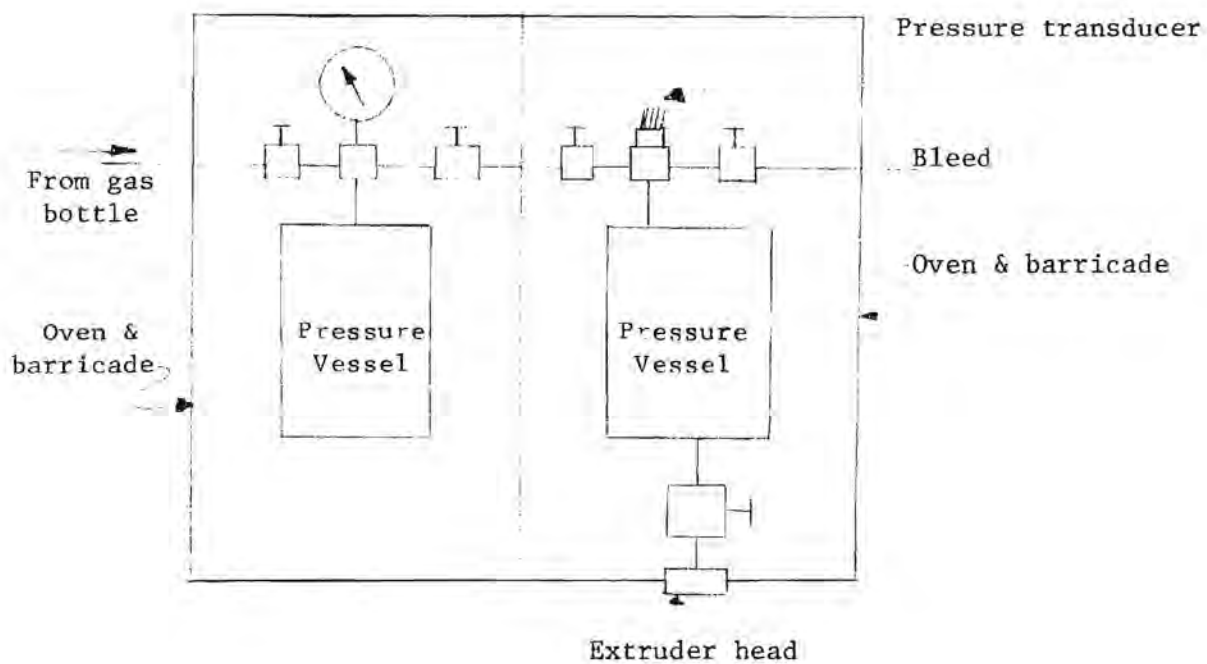
Figure 20 – Line Diagram of Extrusion System for Gas-Polymer
         Solutions

Extrusion from a ring slit in a die to produce a tubular film will be tried. This should work quite well using polymer phase containing dissolved gas. Subsequent expansion of the tube with compressed air and of the polymer in the tube walls by bubble formation from dissolved gas should produce quite highly drawn, fibrillated, fabric-like structure.

The following gas-polymer systems will be studied:

polypropylene-propane

polybutene-1-propane or butane

polyester - carbon dioxide

nylon-carbon dioxide

Other systems will be added to this group depending on results of solubility studies.

Extrudate will be tested for density (degree of expansion) and orientation by scanning electron microscopy and X-ray diffraction. Tensile strengths and stress-strain behavior of fibrous structures will be measured. Estimates of abrasion resistance and qualitative assessments of bulkiness and "hand" will be made for any fabric-like structures produced.

2.   Polymer fibrillation

During the next year the extrusion and take-up equipment will be modified in order to achieve: (1) higher fibrillated web strengths; (2) better balance of properties between the machine and transverse direction; (3) higher web basis weight. An in-line post stretching and heat treatment process will be designed and evaluated. A means for using pressurized gas as a blowing agent will be investigated. Continuous yarn formation from polymer fibrillated by the

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blown film process will also be evaluated.

B.   Fiber-to-Garment

Research during the second year will be concentrated on studies of multi-ple-fiber flows. These studies will be directed toward orienting the fibers parallel to the main stream, transporting them a short distance, and depositing them in an ordered manner. The techniques developed in the single-fiber studies will be applied to the multiple-fiber flows, and modifications intro-duced if necessary.

The same basic experimental facilities will be used, as they were designed for this purpose. A multiple-fiber injector will be designed. It will not have to provide controlled initial orientation, however.

Efforts also will be made to develop procedures for simultaneously de-positing fibers oriented in two perpendicular directions. As discussed in the Introduction, this is considered necessary to accomplish fiber weaving.

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VIII. Report Distribution

The First Annual Technical Progress Report is being distributed to the names listed below. These are persons who have requested copies of the report or persons we have identified as appropriate to invite to the April 12, 1976 seminar on the program.

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APPENDIX

Patent Search

Following is a compilation of patents searched in connection with
the adaptation of the blown film extrusion process to the production of
nonwoven fabrics. The patents are grouped into five categories:

(1) Non-woven fabrics-blown film process
(2) Non-woven fabrics-flash and spray spinning processes
(3) Mechanical fibrillation and/or formation of non-woven
     fabrics by post treatments;
(4) Microfiber and/or net formation through conventional spinning and
(5) Micro-porous film formation.

Although some patents may be applicable to more than one area, each has been
grouped under the most appropriate heading. The patents in the first catagory
are briefly summerized as this area is most closely related to the project.
The patents in the other catagories are simply listed. A review of non-
woven textile patents may be found in Chemical Technology Review No. 43., Non-
woven Textiles, Noyes Data Corporation, Park Ridge, New Jersey, 1975.

1. Non-woven fabrics - blow film process

        3,403,203 - filed March 13, 1964; patented Sept. 24, 1968
        3,539,666 - filed June 18, 1968; patented Nov. 10, 1970
        3,717,541 - filed June 18, 1968; patented Feb. 20, 1973

        W. R. Grace & Company
        H. G. Schirmer
        Methods for preparing a non-woven fabric-like member

These patents describe a process of making a non-woven fabric by extruding
a foamed polymer and orienting the film by a blown-film process. This causes a
rupture of cellular structure giving a porous fabric-like member. The claims
cover methods of either blowing or stretching a foamed extrudate over a mandrel
in order to rupture the cell structure. These three patents are closely related
to phase 1 of NSF Rann Sub-Topic - Use of Foaming Agents to Form Web Structures
in Polypropylene by simulating the W. R. Grace methods so described.

2. Non-woven fabrics - flash and spray spinning process

        3,546,063 - filed Oct. 29, 1954; patented Dec. 8, 1970

        E. I. duPont de Nemours
        Alvin L. Breen
        Microfibers and Shaped Structures Containing Microfibers

        3,549,453 - filed April 6, 1967; patented Dec. 22, 1970

        E. I. duPont de Nemours
        J.G. Smith
        Flash Spinning Apparatus for Nonwoven Fibrous Sheet Making.

        3,615,998 - filed July 10, 1968; patented Oct. 26, 1971

        Celanese Corporation
        Charles J. Kolb
        Method of Biaxially Orienting Nonwoven Tubular Material

3,772,417 - filed Oct. 28, 1970; patented Nov. 13, 1973

Clifford M. Vogt
Method for Improving Physical Properties of Spray-Spun
Fibrous Sheet Materials

3. Mechanical fibrillation and/or formation of non-woven fabrics by
post treatments

3,235,644 - filed Aug. 8, 1962; patented Feb. 14, 1966

Continuous Lateral Stretching of Orientable Sheet Material
Phillips Petroleum Company

3,548,048 - filed May 20, 1968; patented Dec. 15, 1970

Phillips Petroleum Company
James K. Hughes, Jake E. Williams
Method of Producing Polymeric Articles Resistant to Fibrillation

3,607,500 - filed June 4, 1969; patented Sept. 21, 1971

E. I. duPont de Nemours and Company
Nathan D. Field
A Molding Fibrous Webs

3,619,339 - filed July 8, 1969; patented Nov. 9, 1971

E.I. duPont de Nemours and Company
William Lee Garrett
Porous Nonwoven Film-Fibril Sheet and Process for Producing
Said Sheet.

3,851,034 - filed Nov. 17, 1966; patented Nov. 26, 1974

Johnson & Johnson
Carlyle Harmon
Methods of Making Sheet Material

3,876,014 - filed April 30, 1973; patented April 15, 1975

Beloit Corporation
James J. Melead
Process for Matting Melt Blown Fibers

4. Microfiber and/or net formation through conventional spinning

        3,549,734 - filed June 27, 1967; patented Dec. 22, 1970

        Takeshi Yasuda Etal
        Method of Forming Microfibers

        3,600,751 - filed June 24, 1969; patented Aug. 24, 1971

        FMC Corporation
        Theodore H. Fairbanks
        Apparatus for Extrusion of Interlaced Webs

        3,862,878 - filed April 13, 1973; patented Jan. 28, 1975

        Kimikazu Azuma
        Methods of Manufacturing Foamed Plastic Tubular Nets and Apparatus

        3,874,834 - filed March 16, 1973; patented April 1, 1975

        Ignacio Aurrecoeches Arechavaleta
        Continous Extrusion Machine for Manufacturing Plastic Nets.

5. Film forming technology

        3,231,642 - filed July 9, 1964; patented Jan. 25, 1966

        E. I. duPont de Nemours and Company
        Extrusion and Stretching of Thermoplastic Film

        3,231,643 - filed July 9, 1964; patented Jan. 25, 1966

        E. I. duPont de Nemours and Company
        Heater Concentricity in the Extrusion of Thermoplastic to Make
          Tubular Film

        3,231,652 - filed July 9, 1964; patented Jan. 25, 1966

        E. I. duPont de Nemours and Company
        Extrusion Process for Making Thermoplastic Tubular Film

        See Patent abstracts for 6,231,642 and 6,231,643

        3,231,653 - filed July 9, 1964; patented Jan. 25, 1966

        E. I. duPont de Nemours and Company
        Pressure Isolation in the Manufacture of Thermoplastic Tubular
          Film by Extrusion.

5. Film forming technology (con't)

3,887,673 – filed July 13, 1973; patented June 3, 1975.

Wasuke Sato
Asamu Uemura
Tokoyo, Japan
Apparatus and Method for Manufacturing Tubular Film of
   Thermoplastic Resin

3,888,609 – filed May 11, 1973; patented June 10, 1975

Leco Industries Limited
Daniel R. Saint Eve
Apparatus for Producing Films in Accordance with Blown
   Tube Process

3,890,421 – filed Aug. 1, 1972; patented June 17, 1975

La Cellophane
Fortune Habozeit
Methods for Biaxially Drawing Plastic Films

3,231,651 – filed Jan. 3, 1961; patented Jan. 25, 1966

Dow Chemical Company
Plastic Film Windup Apparatus

6. Micro-porous film formation

3,839,240 – filed May 21, 1973; patented Oct. 1, 1974.

Celenese Corporation
Daniel Zimmerman
High Melt Index Microporous Films

3,849,528 – filed Jan. 8, 1973; patented Nov. 19, 1974

Polysar Limited
Albert Ernest Smith
Microporous Polymer Sheets

3,870,593 – filed April 29, 1974; patented Mar. 11, 1975

Minnesota Mining and Manufacturing Co.
Robert L. Elton
John F. VanderLouw
Stretch Oriented Porous Films and Preparation and Use Thereof

E-27-627

Advanced Technology Applications
in Garment Processing

by

Louis H. Bangert
John L. Lundberg
John D. Muzzy
W. Denney Freeston, Jr.

Georgia Institute of Technology
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December 1976

Eighteen Month Technical Progress Report

Advanced Technology Applications
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Louis H. Bangert
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December 1976

Eighteen Month Technical Progress Report

Abstract

New, direct, automatic, fast and inexpensive methods are being developed for converting polymer chips and/or staple fiber into fabrics without carding, spinning and weaving or knitting, and garments without cutting and sewing. Other objectives are to reduce material, labor, capital, and energy costs in textile and garment manufacture. The successful development of one or more of these processes will contribute to improving the United States' competitive position in textiles and clothing in the U.S. and world markets.

No additional data on the solubilities of polymers in dense gases have been obtained during the past six months due to equipment problems.

The feasibility of forming yarns suitable for selected textile applications by foam fibrillation has been demonstrated. Polypropylene fibrillated yarn has been produced with mechanical properties similar to commercial staple fiber yarn.

Foam fibrillated polypropylene webs with basis weights greater than 1 oz/sq.yd. have been produced. Efforts to produce strong fibrillated webs from polybutylene, nylon 66 and polyesters are continuing.

A method utilizing air flows and aerodynamic forces for orienting staple fiber and depositing the fibers in parallel arrays has been developed.

Table of Contents

I.   Executive Summary

The various phases of the studies and the work schedule are given in Table 1.

1.   The principal areas of research during the past six months have been:

   1.   Measurements of solubilities of polymers in dense gases

   2.   Continuous yarn formation from fibrillated polymer mixtures

   3.   Use of tubular biaxial stretching for fabric formation

   4.   Orientation of staple fiber and deposition of fibers in parallel arrays using air flows and aerodynamic forces.

The program management plan is shown in Figure 1. Dr. Freeston is responsible for overall planning, coordinating and supervision of activities. To promote a continual interchange of ideas among the personnel involved in the research, program reviews are held regularly. The members of the review committe are:

W. D. Freeston, School of Textile Engineering,
   mechanics of fiber assemblies

J. L. Lundberg, School of Textile Engineering,
   polymer physics

W. C. Tincher, School of Textile Engineering,
   polymer physics

A. Tayebi, School of Textile Engineering,
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G. A. Fowles, School of Chemical Engineering,
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L. H. Bangert, School of Aeronautical Engineering,
   fluid mechanics

G.M. Rentzepis, School of Engineering Science and Mechanics,
   engineering mechanics

Table 1

Program Schedule

	0	12	24	36

A. Polymer-to-Garment

   a. Gas-polymer solutions   X---------------------X

   b. Effects of additives on fibrillation   X---------------------X

   c. Effects of crystallization and deformation conditions on fibrillation   X---------------------X

   d. Continuous yarn formation from inhomogeneous polymer mixtures   X---------------------------X

   e. Use of tubular biaxial stretching for fabric formation   X---------------------X

   f. Development of laboratory scale yarn or fabric formation process and extension of yarn or fabric formation process to garment making   X----------X

B. Fiber-to-Garment

   a. Single fiber response   X----------X

   b. Multiple fiber suspension   X----------X

   c. Fiber weaving and entanglement   X----------X

C. Utilization Plan;

	0	12	24	36
Progress Reports	X	X	X	X
Research Reviews		X	X	X

Presentation

   Textile Trade Assocs.   X----------------------------X

   Professional Societies   X--X

Presentations to

   National Textile & Apparel Assocs. X--X

	0	12	24	36
Technical Conference & Demonstration				X
Summary Technical Report				X

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Support   Student   Support   Students   Support   Student
              K. Ko   I. Moradina   P. Sagdeo
                                          Y.H. Park

Figure 1. Program Management Plan

Table 2
Work Plan

A. Polymer-to-Garment
    1. Gas-polymer solutions

	Jan. '77	Feb.	Mar.	April	May	June
1. Solubility Studies						
(1) Polypropylene in butane	Completed					
(2) Nylon 6 in CO_2				————————		————
(3) Polypropylene in CO_2	————————		————			
(4) Other gas-polymer systems					————	————
2. Assemble extrusion system	————————		————			
3. Extrude nylon $6 - CO_2$				————————		————
4. Extrude polypropylene CO_2				————————	————	

    2. Polymer fibrillation

1. Process design Changes	————	————				
2. Process Parameter Evaluation	————	————				
3. Polyester Yarn Formation & Evaluation			————	————————		————
4. Polyester Fabric Formation & Evaluation				————————		————
5. Nylon Yarn Formation & Evaluation				————————		————
6. Nylon Fabric Formation & Evaluation				————————		————

B. Fiber-to-Garment

Design of continuous fiber-feed system			————	————————	————	
Alignment in uniform shear flow						
Feasibility investigation			————	————		

-4-

The research on polymer solutions is being directed by Dr. John Lundberg. He is being assisted by Mr. Robert Newson, research engineer, and Ken Ko, a graduate student.

The research on polymer fibrillation is under the direction of Dr. John Muzzy. He is being assisted by Mr. G. H. Hoyos, a research engineer, and graduate students I. Mordina and Y. H. Park.

Dr. Lou Bangert is directing the research on fiber aerodynamics. Mr. John Harper, a research engineer, and Mr. P. Sagdeo, a graduate student, are assisting him.

Technology transfer is being accomplished by presentations at textile and apparel trade association meetings, professional society meetings, seminars presented at companies, private discussions with company representatives, and seminars at Georgia Institute of Technology. Progress reports are being distributed to interested textile and apparel companies. The Utilization Plan Schedule is given in Table 1.

The schedules for the studies planned during the remainder of the second year are given in Table 2.

No additional data on the solubilities of polymers in dense gases have been obtained during the past six months due to the difficulty in achieveing glass to metal seals around windows in the 210 - 230°C temperature region and the 800 to 1400 atmosphere pressure range. A new pressure vessel with windows capable of holding gases at temperatures to 300°C and pressures to 1700 - 2000 atmopsheres will be designed and fabricated during the next several months. Such a vessel should permit studies of solubilities of nylon 6 in carbon dioxide without too

much difficulty. Further, within this pressure range, the higher melting nylon 66 and poly(ethylene terephthalate) should be soluble in carbon dioxide. Studies of these solubilities should be possible (but not easy) with this apparatus.

While the new pressure vessel is being constructed, studies of solubilities of polypropylene in carbon dioxide will be completed. Apparatus for batch extrusion of gas-polymer solutions will be assembled while these studies are underway. Investigation of the extrusion of polypropylene in carbon dioxide should begin before June 1977.

The feasibility of forming yarns suitable for textile applications by fibrillation of polymer mixtures has been demonstrated. Polypropylene yarn has been produced with mechanical properties similar to commercial staple fiber yarn.

Investigations of the feasibility of producing strong fibrillated webs from polybutylene, nylon 66 and polyesters are continuing. It is believed that this can be accomplished by making improvements in the formulation of the feed mixture, in process design and in process control.

A method utilizing air flows and aerodynamic forces for orienting staple fiber and depositing the fibers in parallel arrays has been developed. It is believed that this will permit the formation of webs with fibers arranged in a manner that will result in a superior nonwoven fabric after it is strengthened by an entangling process, such as passing the web under a series of high energy fluid streams.

II. Introduction

The primary objective of the program is to develop new, direct, automatic, fast and inexpensive methods for converting polymer chips and/or staple fiber into fabrics without carding, spinning and weaving or knitting, and garments without cutting and sewing. Other objectives are to reduce material, labor, capital, and energy costs in textile and garment manufacture. The successful development of such processes will contribute to improving the United States' competitive position in textiles and clothing in the U.S. and world markets.

The current studies have three major thrusts:

1) the extrusion of gas-polymer solutions

2) the formation of yarn and fabric from fibrillated polymer mixtures.

3) The orienting of staple fiber and deposition of fibers in parallel arrays using air flows and aerodynamic forces.

The results of the investigations during the past six months are presented in the next section.

III. Technical Activities

A. Gas-polymer solution studies

Measurements of solubilities of polymers in dense gases are necessary if gaseous solutions of polymers are to be used in the transport and forming of polymers, especially in extrusion of polymers from the gas phase. Solubilities of polypropylene in n-butane at concentrations of 5.5 to 21.1 percent polypropylene by weight in the 167° to 193°C temperature range and the 120 to 156 atmosphere pressure (of n-butane) region were reported previously.

Studies of the solubilities of polybutene-1 and other polymers in butane and propane were deferred in favor of studies of solubilities of nylon-6 in carbon dioxide. Carbon dioxide is quite inert compared to butane and propane which are extremely flammable and explosive. Extrusion of polymers in carbon dioxide should be reasonably safe. Much higher gas pressures should be necessary to reach gas densities at which polymers will dissolve in carbon dioxide (critical temperature 31°C) than in n-butane (critical temperature 153°C). Higher pressures and higher temperatures will be required to dissolve nylon-6 (m.p. 216° - 220°C) in carbon dioxide than polypropylene (m.p. 160° - 176°C). In spite of the higher pressures and temperatures necessary, solubility studies on nylon-6 in carbon dioxide were undertaken at this point because of the better fiber characteristics of nylon-6 as compared to polypropylene and the non flammability of carbon dioxide.

Measurements of solubilities in the 210° - 230°C temperature region and the 800 to 1400 atmosphere pressure range strain the glass to metal seals around windows beyond the capabilities of ordinary gasket materials. The inadequacy of seals around windows and the short-comings of commercial gaskets were

underestimated in planning this work. Designing and constructing a new pressure vessel with windows which hold gases at high pressures without leaking is expensive and time consuming. Therefore, attempts to develop seals which don't leak at gas pressures of 1000 atmospheres and temperatures up to 220°C for the existing pressure vessel continue.

An apparently successful seal using a fluorocarbon polymer O ring and a split-ring, steel back-up ring has been developed for the circumferential seal around the windows of the pressure vessel. A combination fluorocarbon polymer O ring and epoxy resin cement seal is being tested for the ends of the windows. Several combinations of materials, geometries, and sizes have been tried in developing suitable O ring seals.

Solubility studies are almost a year behind schedule. Half of this is due to a late start on the program and our inability to engage a competent chemist-engineer to make the measurements until September 1975. Dr. Alvin Levy developed the pressure system, measured the solubilities of polypropylene in butane, and improved the measuring system and techniques by the end of March, 1976, when he took a permanent position in a private organization. No experienced replacement for Dr. Levy could be found. Therefore, Mr. Robert Newsom, a research technician experienced in mechanical and electrical measurements but not in high pressure techniques, was hired to make these studies. Mr. Newsom began to modify the apparatus and to learn high pressure techniques in August, 1976.

In spite of slow progress and frustrations in attempting to develop glass to metal seals to hold carbon dioxide at high pressures and temperatures,

efforts should continue. Carbon dioxide should be a good solvent for many materials including several polymers. Indeed, its value as a solvent is at least proportional to the difficulty in containing it with gaskets and seals. Based upon our experience in trying to develop suitable polymeric hydrocarbon and halocarbon seals, carbon dioxide appears to be a good solvent in the 200°C and 1000 atmosphere region.

B.   Foam Fibrillation

1.   Continuous Yarn Formation From Fibrillated Polymer Mixtures

a.   Introduction

The tubular fibrillation process reported previously (1) and in section

B.2 can be altered to produce yarn. The primary objective of the present

study is to ascertain the potential of this process for producing commercially

useful yarn.

In the process investigated, polymer melt containing a blowing agent is

extruded through a circular annulus and biaxially stretched over a circular

mandrel resulting in a fibrous web. This web is drawn over a heated roll,

twisted and heat set to form a yarn. Since the yarn produced in this manner

has connected fibrils, it differs from conventional continuous filament or

staple fiber yarns. The yarn is similar to mechanically fibrillated film

yarns in appearance, but significant differences in structure and properties

are anticipated. Since this process route should be inexpensive compared to

conventional yarn production, the range of properties which can be achieved

are being explored.

b.   Process Description

The primary focus of these preliminary studies of yarn formation has been

on the drawing and twisting operations. Consequently, the materials and ex-

trusion conditions have been fixed. The feed mixture consists of 99.0 wt%

polypropylene (Profax 6323, Hercules, Inc.), 0.5 wt% azodicarbonamide (Kempore

200 MC, Stepan Chemical Co.) and 0.5 wt% fumed silica (Cab-o-sil M5, Cabot

Corp.). This mixture enters a 3/4" barrel diameter, 25 to 1 length to diameter

extruder (Rheomex 254, Haake, Inc.). The rear, middle, front and die tempera-

tures are controlled at 200, 230, 230 and 230°C respectively. The extruder's
screw speed is set at 30 RPM. A circular die with a 1" outside diameter and
0.92" inside diameter annulus is used. Protruding from the center of the die
is a biaxial stretching mandrel. In most cases a 3 1/4" diameter mandrel is
used. The extrudate, which emerges from the die as a foam, is stretched over
the mandrel, which splits the foam into a web. As the extrudate is stretched
it is cooled by compressed air at 20 psig, corresponding to a flow rate of 60
cubic feet per minute. The web is wound up at 82 feet per minute.

The as-extruded web has a denier of approximately 5,000. The tenacity
is 0.14 gr/den without a mandrel and increases to 0.20 and 0.19 gr/den. when
the 2 1/2" and 3 3/4" mandrels are utilized (see Table 3). The elongation
of the web is approximately 75%.

Significant increases in tenacity are achieved by twisting and drawing
by hand over a heated cylinder at 136°C (see Table 3). It is difficult to
twist the yarn more than 2 1/2 TPI[*] because of the large denier. Also, drawing
beyond three times the original length is hindered by fracture. Since both
twisting and drawing improve tenacity, a sequential drawing and twisting cycle
was devised to achieve higher draw ratios and twist densities.

Figure 2   illustrates the effect of draw temperature on sequentially
drawn and twisted yarn. This yarn has a total draw ratio of 4, excluding ex-
trusion draw down, and a final twist of 2 TPI. The denier is 1,385. A tenacity
exceeding 3 gr/den with an elongation of 25% can be achieved. Further increases
in tenacity can be obtained with additional drawing and twisting. Somewhat
better properties have been obtained with slit webs processed into yarns with
lower denier. As Table 4 illustrates the yarn produced by this process route
is comparable to staple fiber yarn produced commercially.

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[*] turns per inch

TABLE 3

POLYPROPYLENE YARN

MATERIALS:	HERCULES PROFAX 6323	99.5%
	KEMPORE 200MC	0.5%

PROCESS CONDITIONS:	SCREW SPEED	30 RPM
	DRIVE ROLL SPEED	82 FPM

TENACITY (GR/DEN.)

TWISTS PER INCH (TPI)	0	0	2½	2½
DRAWING AT 136°C	NONE	2x	2x	3x
MANDREL DIAMETER (INCHES)				
NONE	0.14	0.35	0.78	1.12
2.5	0.20	0.71	1.22	1.76
3.75	0.19	0.60	1.04	1.75

TREATMENT: DRAWN 2X, TWISTED 4TPI AND DRAWN 2X AGAIN
DENIER: 1,385
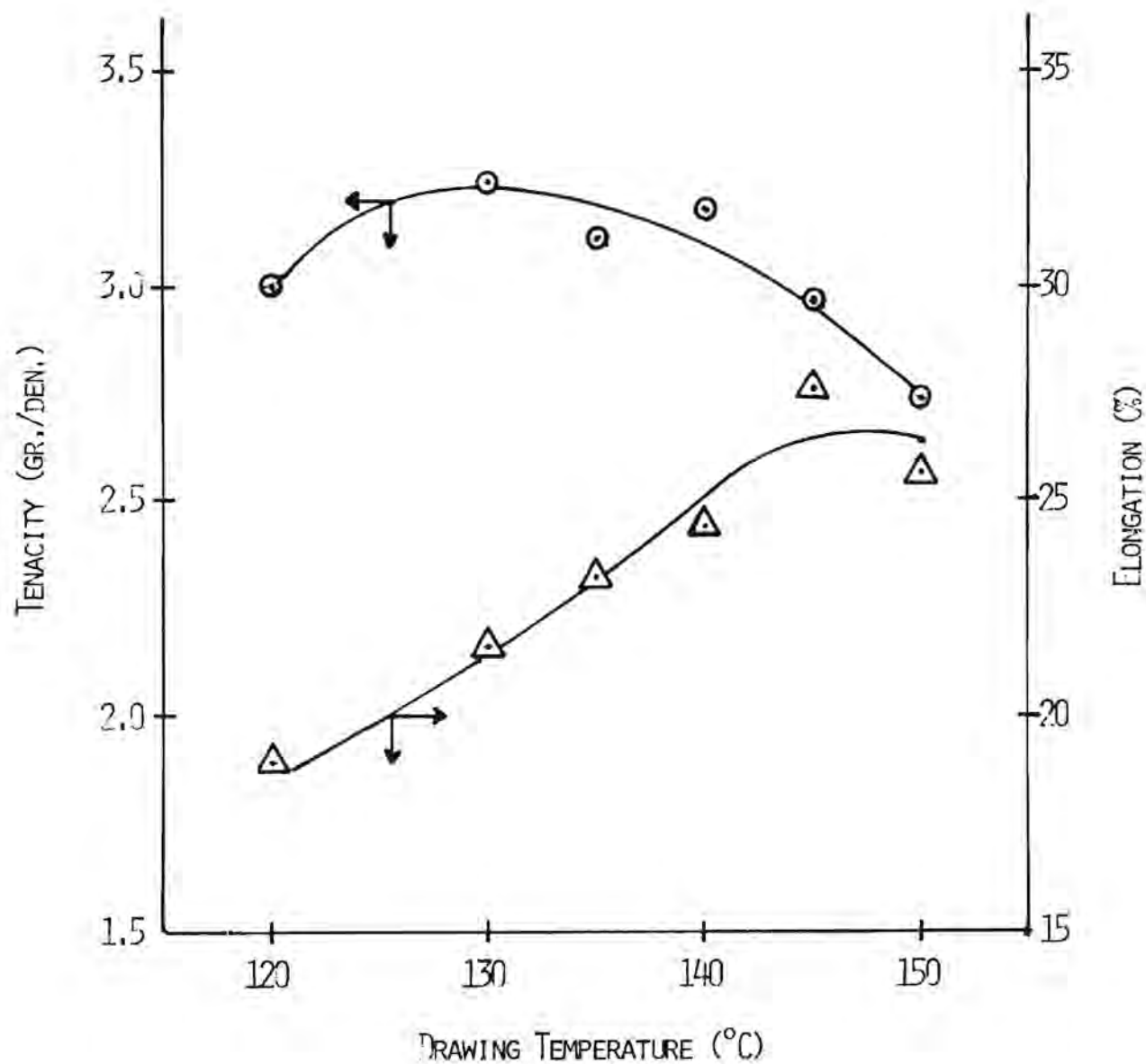
FIG. 2   EFFECT OF DRAW TEMPERATURE ON YARN PROPERTIES

TABLE 4

COMPARISON OF EXPERIMENTAL YARN
WITH COMMERCIAL POLYPROPYLENE
STAPLE FIBER YARN

PROPERTY	COMMERCIAL YARN	EXPERIMENTAL YARN
DENIER	1800	1385
TWISTS/INCH	3	2
TENACITY (GR/DEN.)	2.5	3.2
ELONGATION (%)	36	22

The current level of properties achieved are not considered the best possible. The effect of extrusion conditions and material composition have not been explored. Also, improvements are expected in converting from hand drawing to continuous, high speed drawing. Also, the direct extrusion of twisted yarns using a rotating die may improve the physical properties.

Contrary to the work schedule for the second year, the evaluation of nylon and polyester yarns has not started. This delay is due to difficulties encountered in forming nylon and polyester webs. These problems are discussed in Section B2.

c.   Conclusions

The feasibility of forming yarns suitable for textile applications has been demonstrated. Substantial increases in tenacity are achieved by hot drawing and twisting. Also, increased biaxial stretching of the foam extrudate improves the ultimate yarn tenacity. Experimental polypropylene yarn has been produced with mechanical properties similar to commercial staple fiber yarn.

In future work a continuous drawing system will be developed to achieve better control of drawing conditions and to facilitate increased yarn production for product evaluation. The effect of extrusion conditions and material composition will be evaluated - particularly higher molecular weight grades of polypropylene. Also, nylon and polyester yarns will be evaluated as soon as reasonably uniform webs are produced.

2. Use of Tubular Biaxial Stretching for Fabric Formation

a. Introduction

In the previous progress report (1) considerable information was presented on the formation of polypropylene webs by biaxially stretching a tubular foam extrudate. In that report a number of web deficiencies were noted: 1) low basis weight, 2) tenacity imbalance, 3) low tenacity, 4) high permeability and 5) large nonuniform pores. While a number of these features were improved during the first year, additional improvements were necessary.

The primary objective of the present study is to make further improvements in web properties. The principal approach to improving web properties is through process design changes. An additional objective of the present work is to evaluate nylon and polyester web formation.

b. Process design changes

One dozen design changes are listed in Table 5. These will be discussed in turn.

1. A liquid blowing agent feed system was constructed consisting of a liquid reservoir pressurized by nitrogen followed by a long capillary tube to the extruder hopper. The long tube provides a large pressure drop with a low flow rate. Compared to solid blowing agent, the liquid blowing agent should be more uniformly dispersed in the polymer melt before decomposing; hence, more uniform cell formation is expected. This system is ready for evaluation.

2. A gas feed system was attached at the vent port of the extruder in order to mix gas with polymer melt. Only the gas pressure is controlled and

TABLE 5
Tubular Fibrillation Process
Design Changes

Change	Principal Benefit Expected
1. Liquid blowing agent feed	Improve web uniformity
2. Gas feed system	Improve web uniformity
3. Static mixer	Improve web uniformity
4. Die pressure sensor	Better process control
5. Wide angle die	Minimize extrudate impingement
6. Rotating core die	Increase traverse direction orientation
7. Collapsing mandrel	Facilitate start-up
8. Stagewise mandrel	Increase traverse direction orientation
9. Modified cooling ring	Facilitate start-up
10. Hot air quench	Vary quench conditions
11. Downward extrusion	Facilitate start-up
12. Cryovac system	Evaluate spiral mandrel

not the gas flow rate. A flow-controlled system was designed but rejected because it would be expensive considering the variable downstream pressure. The gas feed system has been evaluated using nitrogen and polypropylene. Relatively uniform webs can be produced provided Cab-o-sil and silica flour are incorporated in the polymer feed. These webs have lower tenacities compared to samples prepared with solid blowing agent (Kempore 200MC). However, the reticulation (removal of cell walls) is better using nitrogen with silica than using solid blowing agents. Further studies of fibrillation using gaseous blowing agents will be performed because 1) extrusion conditions are not dependent on the decomposition characteristics of chemical blowing agents and 2) gases with different solubility characteristics can be selected.

3. A static mixer (a six element, 1/2 inch i.d., Kenics mixer) was added between the extruder and the die to improve the uniformity of the foam extrudate by improving the dispersion of foaming agent in the melt. After incorporating the mixer inferior product was obtained in most cases. This decline in quality is attributed to the inability of this mixer to disperse gas in the polymer melt. If a significant quantity of chemical blowing agent decomposes in the extruder, the mixer cannot prevent the evolved gases from coalescing. However, the mixer should improve the dispersion of solids and soluble gases in the polymer melt. Thus, the mixer is helpful in some cases, but it is harmful when chemical blowing agents are used.

4. A die pressure sensor was added to assess the consistency of processing conditions. This sensor has not been used extensively to date.

5. A wide angle die was contructed in an attempt to direct the extrudate outward towards the transverse direction. By directing the extrudate outward in the direction of biaxial stretching, less chance of extrudate impingement on the stretching mandrel should occur. In practice, little difference in performance was observed.

6. The die was modified to provide for rotating the core and the stretching mandrel attached to the core. This modification should increase orientation in the transverse direction which currently exhibits the lowest strength. Since incorporating the rotating core it has been difficult to maintain a consistent die gap due to slack in the shaft. Consequently, the uniformity of the extrudate is reduced. In pursuing high rotational speed trials the webs collected have remained twisted. This feature is undesirable in producing webs, but may be beneficial in forming yarns. The system needs to be improved before an adequate assessment of the utility of the rotating core can be made.

7. A ring mandrel which could be tilted on a shaft was constructed in order to facilitate startup. By starting with a ring initially tilted towards the machine direction, a lower degree of biaxial stretch is incurred which can gradually be increased by tilting the ring away from the machine direction. This modification has been tested and found to be inadequate because the mandrel wobbles too easily.

8. Stagewise biaxial stretching has been briefly evaluated by following a small diameter conical mandrel with a larger diameter ring mandrel. This approach can be performed and improved tenacities are obtained. Further testing is needed to ascertain the feasibility of this modification.

9.   The cooling ring was completely redesigned in order to facilitate startup by improving access to the die face. The new cooling ring has been beneficial, permitting significantly higher extrusion rates without disrupting the process. Consequently, higher basis weights can be obtained.

10. The air quench system has been modified to permit extrudate cooling with air up to 80°C. The quench temperature can significantly alter the deformation characteristics of the extrudate, introducing an additional processing parameter. This system has not been utilized in processing studies to date since it has been difficult to operate the air heater for long periods of time without heating coils failing.

11. The direction of extrudate take-up was switched from upward to downward to facilitate formation of nylon and polyester webs. Initial processing studies with nylons and polyesters were hindered by polymer sagging and then sticking to the die face. By extruding downward, the extrudate tends to sag due to gravity away from the die. Despite implementing this modification, the formation of nylon and polyester webs still suffers from extrudate sticking to the die face or the mandrel. These surfaces will be treated with release agents in the future to reduce this problem.

12. Recently, the pilot system developed by the Cryovac Division of W. R. Grace for forming webs was loaned to Georgia Tech. After installation of this system is completed in January, its performance will be compared with the existing system which differs signficantly in design.

c.   Process parameter evaluation

Parametric effects discussed in the previous report (1) will not be
covered here. Two subjects will be discussed: improving the fibrous structure
of the webs and a preliminary evaluation of web formation using nitrogen as a
blowing agent. Polypropylene (Hercules Profax 6323) is the base polymer in
these studies.

After modifying the system to include the static mixer, the rotating
core die, the modified cooling ring and downward extrusion (see Table 5),
the webs formed using axodicarbonamide (Kempore 200MC) blowing agent were not
as uniform as previous samples. This decrease in uniformity is attributed
to the longer flow path for the melt, permitting premature blowing, and in-
ability to maintain a uniform die gap with the modified die. After signifi-
cantly modifying the processing conditions a web with improved physical
properties could be formed (see sample 1 in Table 6). As the low air per-
meability of this sample suggests, this web is too filmy. Consequently a
program was initiated to improve the fibrous texture of the webs.

The remaining samples in Table 6 indicate methods for improving the
fibrous texture of the webs. By adding fumed silica to the extrusion mixture,
compare samples 1 and 2, fibrillation is enhanced. Either fumed silica is
nucleating fibrillation directly or it improves the dispersion of the evolved
gases by stabilizing small scale bubbles. Unfortunately, reducing the filmy
texture of the web also reduces the mechanical properties of the web.

A switch to nitrogen as a blowing agent was made in order to evaluate
web formation independent of the decomposition characteristics of a chemical
blowing agent. Without the addition of fumed silica and silica flour, filmy

Table 6

Improving Fibrous Structure

Sample	1	2	3	4
Materials, wt%				
1 Polypropylene (Profax 6323)	99.5	99.0	99.0	98.0
2 Azodicarbonamide (Kempore 200MC)	0.5	0.5		
3 Fumed silica (Cab-o-Sil M-5)		0.5	0.5	1.0
4 Silica flour			0.5	1.0
Processing Conditions				
Screw type, No. of stages	1	1	2	2
Zone 1, °C	170	170	170	170
Zone 2, °C	180	180	180	180
Zone 3, °C	190	190	190	190
Mixer, °C	175	195	195	195
Die, °C	230	220	220	220
Vent Port feed, yes/no (Nitrogen)	NO	NO	YES	YES
Vent port, psi	N.M.	N.M.	500	400
Zone 3, psi	N.M.	N.M.	N.M.	N.M.
Die, psi	N.M.	N.M.	N.M.	N.M.
Mandrel diameter, inches	3.75	3.75	2.5	2.5
Quench air, °C	25	25	25	25
Quench air, psig	25	25	25	25
Screw speed, RPM	90	90	90	100
Wind-up speed, FPM	68	48	48	48
Web Properties				
Basis Weight, oz/sq. yd.	0.30	0.39	0.45	0.53
MD Tenacity, (lb/in)/(oz./sq.yd.)	6.40	3.25	3.07	2.75
TD Tenacity, (lb/in/(oz./sq.yd.)	0.44	0.27	0.33	0.25
MD Elongation %	200	89	85	71
TD Elongation, %	110	81	100	110
Air Permeability, cu. ft./sq.ft./min.	27	200	680	752
Structural quality	filmy			fibrous

N.M. = not measured

webs and coarse bubble formation occured. By using moderate amounts of silica, webs similar to chemical blowing with silica present could be formed—compare samples 2 and 3 in Table 6. A further increase in the amount of silica present improved the fibrous texture with a moderate decrease in mechanical properties - compare samples 3 and 4 in Table 6.

Since webs produced by nitrogen blowing can be formed with properties comparable to webs produced by chemical blowing agents, a further study of nitrogen blowing was performed—see Table 7. These studies have been limited to conditions where a reasonably fibrous texture results. The processing conditions for these studies closely approximate the conditions reported for samples 3 and 4 of Table 6 except as noted in Table 7.

It is noteworthy that basis weights exceeding 1 oz/sq.yd. can be achieved whereas previously (1) extruded basis weights could not exceed 0.5 oz/sq.yd. These higher basis weights were achieved primarily by design modifications to the mandel and cooling ring, permitting higher extrusion rates (screw speed) and lower wind-up speeds. As-extruded basis weights considerably greater than 1 oz/sq.yd. will require further design modifications, including a larger extruder.

There are moderate declines in mechanical properties - tenacity and elongation - as the concentration of silica flour is increased. These declines are more apparent in the transverse direction (TD). Unfortunately, the trends with increasing amount of silica flour are not consistent due to scatter in the results.

The amount of silica flour does not substantially alter the normalized air permeability. Apparently, increasing the amount of silica flour above

Table 7

Web Formation Using Nitrogen Gas Blowing Agent
Polypropylene: Hercules Profax 6323
Fixed additive: 1% Cab-o-Sil M-5

Property	Screw Speed RPM	Wind-up Speed FPM	Silica Flour %		
			0.5	1.0	1.5
Basis Weight	100	26.6	0.89	0.76	0.79
		47.6	0.46	0.53	0.48
Oz/sq yd	120	26.6	1.08	0.84	0.93
		47.6	0.60	0.64	0.58
M D Tenacity	100	26.6	2.76	2.50	2.44
		47.6	2.49	2.75	2.77
(lb/in)/(oz/sq yd)	120	26.6	4.07	2.59	2.10
		47.6	2.59	2.58	2.71
T D Tenacity	100	26.6	0.58	0.44	0.39
		47.6	0.23	0.25	0.22
(lb/in)/(oz/sq yd)	120	26.6	0.70	0.45	0.40
		47.6	0.30	0.25	0.21
M D Elongation	100	26.6	61	61	59
		47.6	65	71	88
%	120	26.6	53	54	30
		47.6	59	93	69
T D Elongation	100	26.6	70	64	66
		47.6	145	108	83
%	120	26.6	70	55	66
		47.6	89	91	94
Air Permeability (normalized)	100	26.6	400	330	423
		47.6	286	398	356
	120	26.6	386	445	415
(ft/min)x(oz/sq yd)		47.6	308	396	354

0.5 wt% does not substantially improve fibrillation. Based on these obser-
vations, only a low concentration of silica flour (below 1%) is necessary.
More silica flour can be detrimental to the mechanical properties.

Increasing screw speed increases the web basis weight but does not change
tenacity, elongation and permeability significantly. Increasing wind-up speed
decreases basis weight, TD tenacity and air permeability, but increases MD and
TD elongation. The impact of wind-up speed on MD tenacity is not consistent.
These trends, whether increasing, decreasing or not changing, require further
verification due to the general scatter of the results. Based on an overall
evaluation of these twelve samples it would appear to be preferable to operate
at a screw speed of 120 RPM with a wind-up speed of 26.6 FPM using 0.5%
silica flour.

d.   Evaluation of other polymers

Two polyesters, nylon 66 and polybutylene have been studied in addition
to polypropylene. None of these polymers have been processed as successfully
as polypropylene. The difficulties in processing these polymers will be
discussed in turn.

Poly (tetramethylene terephthalate), i.e., PTMT (Tenite 6 PRO A, Eastman
Chemical Products, Inc.), could not be processed without incorporating a minor
percentage of polypropylene. At the time the equipment was set up to extrude
upward and the polyester extrudate readily sagged or broke due to inadequate
melt strength. Samples of PTMT blended with polypropylene are reported in
Table 8.   Better properties are obtained when the percentage of polypropylene
is low and the screw speed is high – see Sample 3. The basis weight of sample

3 is 1.04 oz/sq.yd., which is encouraging; but, all of the samples have a coarse pore structure compared to polypropylene samples. The coarse structure is probably due to the high processing temperatures which would cause the blowing agent to decompose prematurely. In future studies of PTMT dissolved gases will be evaluated as blowing agents in order to avoid pre-blowing.

Processing filament grade poly (ethylene terephthalate) i.e., PET, obtained from Eastman Chemical Products, Inc., has not been successful to date. The polymer tends to stick to the die face or mandrel too easily and the melt strength is low. Blending polypropylene with PET has not facilitated web formation. Further modifications of the feed mixture and processing conditions are planned in order to obtain webs of PET.

Only one unsuccessful attempt to process nylon 66 (Zytel 101, Dupont Co.) has been made. Severe degradation of the polymer occurred which was attributed to inadequate drying. Also, sticking and low melt strength, as incurred in processing PET, was observed. Once suitable conditions for processing PET have been resolved, this technology will be applied to nylon 66 processing.

Polybutylene (Witron 0100, Witco Chemical Corp.) has been briefly evaluated. Well fibrillated webs formed but the webs tended to break easily while stretching over the mandrel. At the time of the test the die gap was out of alignment which may explain why the web broke easily. A sample was not collected for testing. Further evaluations of polybutylene will be performed when time is available.

All of the polymers tested, except for polypropylene, were received in pellet form. With the small scale extruder powdered resins are preferred

Table 8

PTMT/Polypropylene Webs

Sample	1	2	3
Materials, wt. %			
1 Tenite 6 PRO (PTMT)	60	75	85
2 Profax 6323 (polypropylene)	39	24	14
3 Kempore 200MC	1	1	1
4			
Processing Conditions			
Screw type, No. of stages	1	1	1
Zone 1, °C	220	220	200
Zone 2, °C	260	285	260
Zone 3, °C	280	285	280
Mixer, °C (not present)			
Die, °C	280	285	280
Vent Port feed, yes/no	NO	NO	NO
Vent Port, psi	NM	NM	NM
Zone 3, psi	NM	NM	600
Die, psi	NM	NM	NM
Mandrel diameter, inches	2.5	2.5	2.5
Quench air, °C	25	25	25
Quench air, psig	30	30	30
Screw Speed, RPM	100	80	110
Wind-up speed, FPM	36	36	36
Web Properties			
Basis Weight, oz/sq. yd.	0.68	0.71	1.04
MD Tenacity, (lb/in)/(oz/sq. yd.)	1.81	1.45	2.03
TD Tenacity, (lb/in)/(oz/sq. yd.)	0.47	0.46	0.58
MD Elongation, %	NM	NM	NM
TD Elongation, %	NM	NM	NM
Air Permeability, cu. ft./sq. ft./min.	722	828	522
Structural quality			

NM = not measured

for dry blending with the various additives. Mixing pellets with powders or pelletized concentrates did not produce as uniform a product as powdered blends. Grinding the pellets improved the uniformity but in some cases, particularly polybutylene, the polymer was difficult to grind. Improved grinding equipment is being sought.

e.   Conclusions

A number of design modifications have been made which extend the effective range of processing conditions. It is now possible to extrude polypropylene webs with basis weights greater than 1 oz/sq.yd. Also, it is possible to form webs using nitrogen as a blowing agent. Further improvements are needed to improve process control, and hence web uniformity, which has deteriorated somewhat in implementing a number of the design changes.

It has become apparent in conducting process studies that conditions which lead to high web tenacities and low permeabilities tend to correspond to poor fibrillation. By adding fumed silica and silica flour both web uniformity and fibrillation improve. However, lower tenacities and higher permeabilities are incurred. These fine particle additives appear to be essential components for forming webs using nitrogen as a blowing agent. With these additives, webs formed using nitrogen compare favorably with webs formed using chemical blowing agents.

The capability to form webs from polybutylene, nylon 66 and polyesters has not been adequately demonstrated. Improvements in the formulation of the feed mixture, in process design and in process control are needed in order to process nylon 66 and PET. Polybutylene and PTMT can be processed, but more work must be performed to form webs which equal or exceed the polypropylene webs in quality.

C. Fiber Aerodynamics

1.0 Introduction

The process of fiber weaving offers increased productivity and reduced material waste and energy consumption, compared with conventional processes of spinning staple fibers into yarns and of weaving yarn into fabric. Staple fibers must be arranged in orderly patterns to produce fiber-woven fabrics with aesthetic and performance characteristics comparable to conventionally woven fabrics. The fibers in the final fabric should be oriented principally in two perpendicular directions so that the fibers retain bending and sliding characteristics similar to those of warp and fill yarns in conventional woven fabric.

The basic steps in fiber weaving are:

(1) separation of tufts into individual fibers

(2) orienting fibers in a prescribed direction;

(3) depositing fibers in parallel arrays;

(4) intertwining the fibers

The present research program is concerned with discovering methods for orienting and depositing fibers in a desired manner by using air flows and aerodynamic forces. It is believed that successful development of such methods will make fiber weaving feasible, as methods are already available for accomplishing the other steps.

In a previous report(1), a new method for alignment of individual fibers parallel to an air stream was described. Section C.2 of the present report describes studies directed toward extending this concept to provide a

method for continuously aligning and depositing fibers. Further studies on the motion of individual fibers are described in section C.3, which serve to clarify the process of fiber alignment parallel to the free stream flow.

2.0 Multiple-Fiber Alignment and Deposition

   2.1 Fiber Injector

      In order to study the alignment and deposition of a number of fibers it was necessary to provide a device (i.e., a fiber injector) on which fibers could be mounted prior to interaction with the air flow, and which would allow fiber release so that the desired alignment could occur. Figure 3 is a photograph of the injector, and Figure 4 shows the injector mounted in the wind tunnel.

      The principles of the injector operation are as follows. Fibers are mounted on the outside of the injector screen, being held in place by radially inward air flow produced by a large industrial vacuum cleaner. The axis of the injector is a tube, perforated over its length within the injector, which is connected by flexible plastic tubing to the vacuum. Before each test run, fibers are mounted on the portion of the injector which is outside the tunnel. The injector is then rotated so that the fibers are inside the tunnel, but on the upstream part of the injector. An airtight shroud is then placed around the portion of the injector that extends outside of the tunnel. The purpose of the shroud is to prevent outflow of air from the tunnel through the injector after the tunnel flow is started. With the fibers thus in place, and the shroud in position, the main tunnel air flow and the counterflow jets are started, and the vacuum is turned off. The injector is then rotated in the streamwise direction, and fibers are blown off the injector screen when they
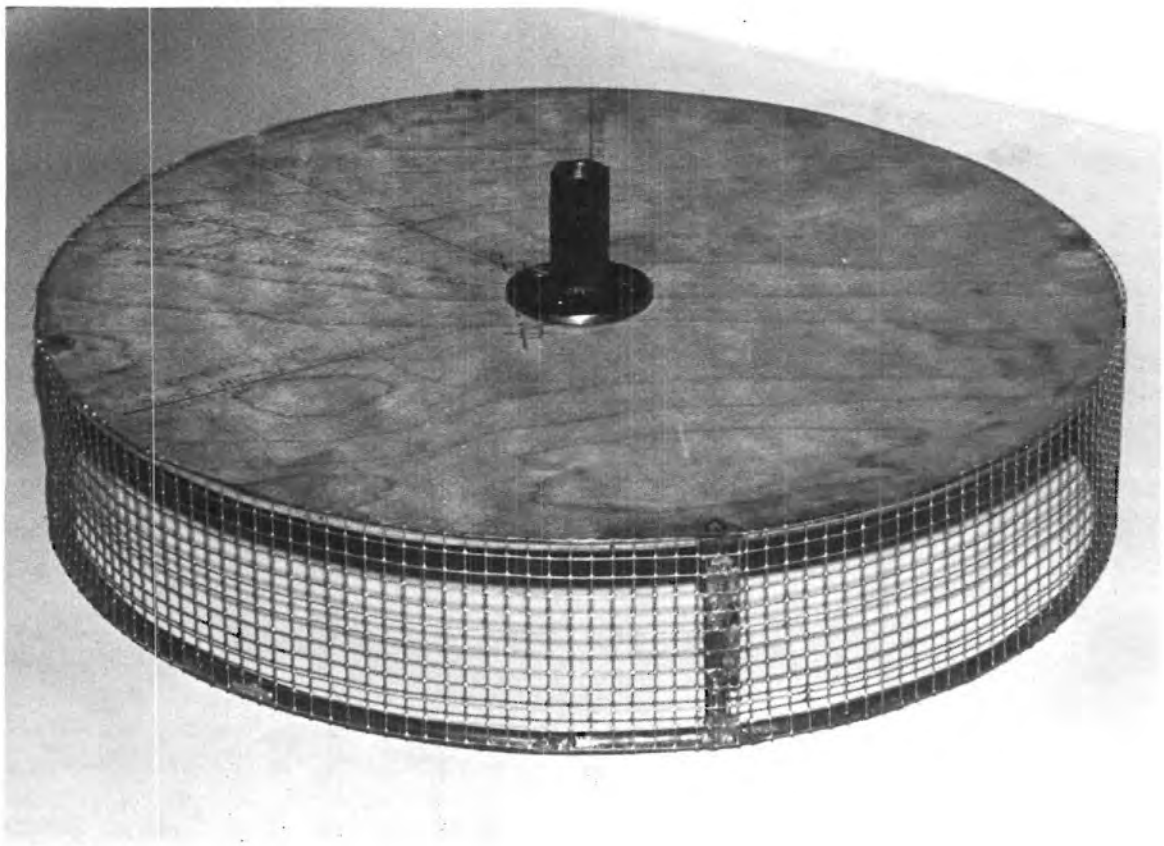
Figure 3.   Fiber Injector
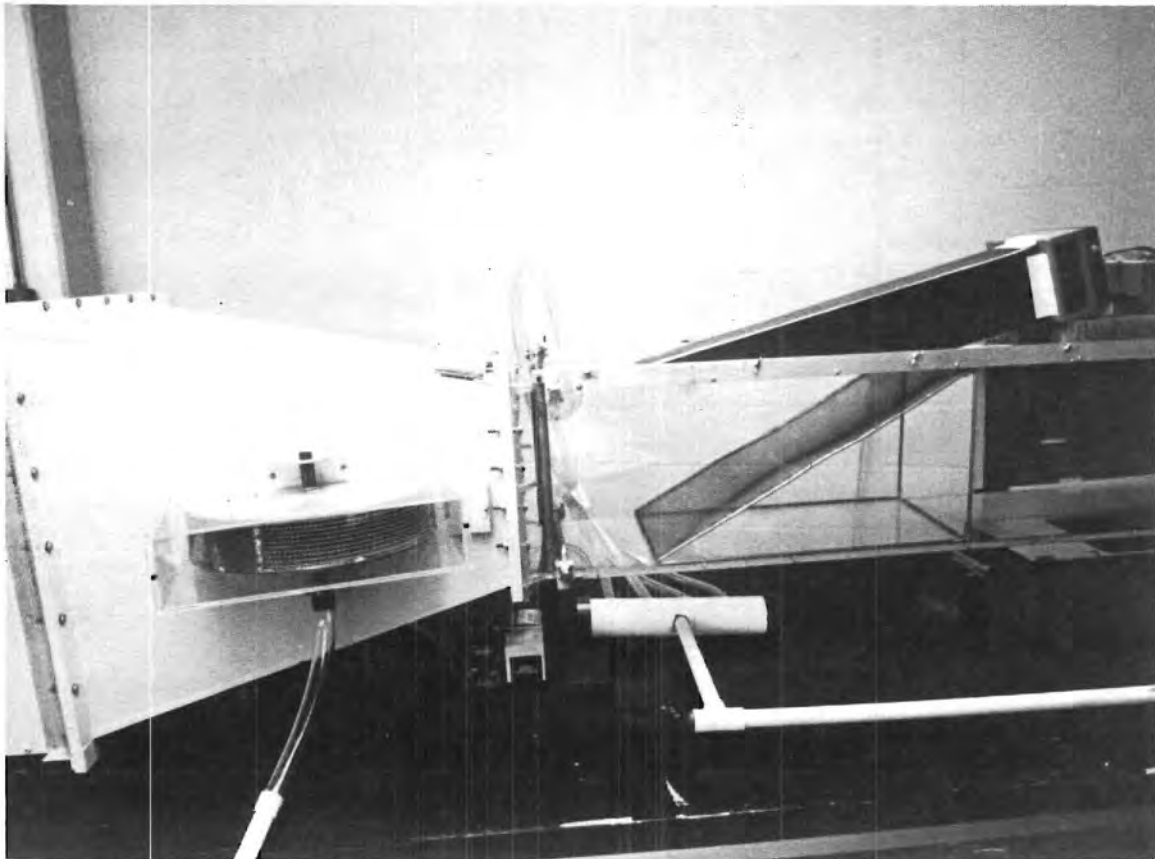
Figure 4.   Experimental arrangement. Wind tunnel with fiber injector (left),
jet tubes (center), and fiber deposition screen (right).
Stroboscopic light source is on top of the test section.

reach the point where the free stream velocity vector is approximately tangent to the injector screen.

Before describing some of the results obtained in these experiments, it seems worthwhile to mention that considerable difficulties were encountered and overcome in arriving at a system which allowed controlled handling of the fibers. Some of these difficulties were as follows: (1) During fiber loading of the injector, random puffs of air from the atmosphere at the tunnel exhaust duct outlet produced disturbances that blew the fibers off the injector. The exhaust duct outlet was modified to eliminate this problem. (2) The transparent plexiglas shroud tended to build up a static charge because of air flow over its inner surface. This also made fiber loading very difficult until the injector was redesigned. (3) The shroud must be relatively airtight, as a significant outflow of tunnel air past the injector causes the fibers to be held against the injector screen. These difficulties were mainly related to keeping the fibers on the injector screen during loading, and then achieving satisfactory release from the injector screen when desired. The influence of random puffs of air and of static electricity had to be overcome during loading. Tunnel outflow at the injector and injector screen blockage had to be minimized to achieve the desired fiber release. It is expected that improvements can still be made. The present fiber injector does provide a working system for releasing fibers into the stream in a reasonably controlled manner, however.

2.2 Results of Recent Fiber Alignment and Deposition Experiments

In the most recent configuration, two jet tubes are used whose axes are vertical (i.e., parallel to the z-axis). These emit a flow which

deflects the main stream so that forces on each fiber act to rotate it into the xz – plane. Alignment within the xz – plane is not expected to be important. This is because the fibers are intercepted by the deposition screen, whose plane is inclined at 30° from the horizontal (xy) plane. Thus, in the absence of a cross-flow at the screen surface, a fiber which is in the xz – plane will remain so when it is brought to rest on the screen.

Experiments with the apparatus described above are in the early stages. Nevertheless, some very promising results have been obtained. The experimental procedure is as follows. Approximately 10 fibers are loaded onto the injector screen. This relatively small number of fibers is being used at this stage of the study to avoid possible entangling before release. (A production system could readily be designed to feed fibers onto the screen in such a way as to avoid entanglement). After the fibers have been deposited on the collecting screen, a photograph of the result is made immediately. Figures 5 – 8 are examples of such photographs, taken from a series of runs for which U_∞ = 20 ft/sec, $\dot{m}_\infty/\dot{m}_j$ = 28.7, and U_j/U_∞ = 10. The fibers are not actually individual fibers, as the diameter of these is too small (a few microns) to be easily visible on the photographs. Instead, one of the three strands comprising No. 50 white cotton mercerized thread has been used. These have a diameter of approximately 60 microns (0.0024 inch). The fiber length in these experiments is one inch.

In order to obtain a quantitative evaluation of the fiber deposition, the following system is being used at present. Fibers whose long axis is within 20° of the free stream direction (x-axis) are considered parallel. Those between 20° and 70° are labeled as 45°. Those between 70° and 90° are labeled as 90°. Finally, when a deposited fiber has a curved, or hook, shape it is labeled "hooked".
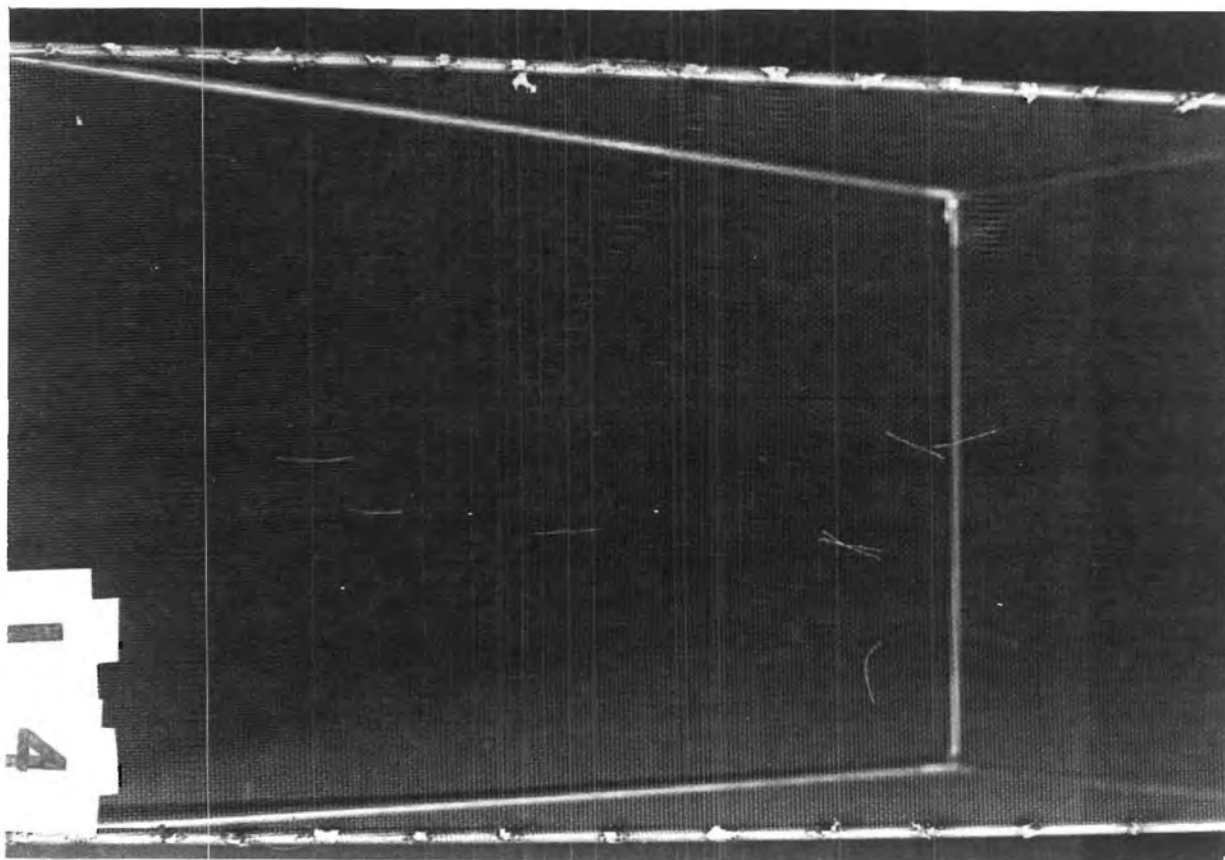
Figure 5.   Fibers collected on deposition screen.
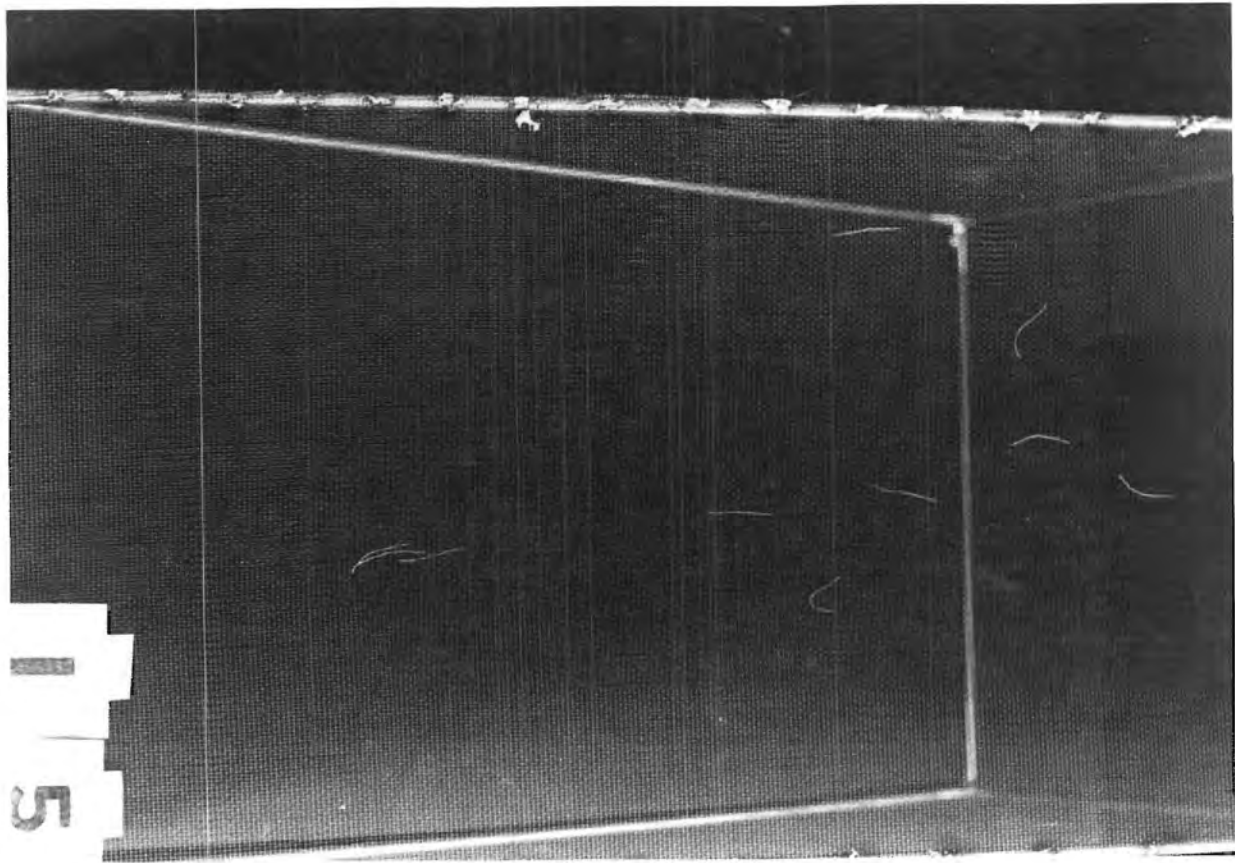
Figure 6.   Fibers collected on deposition screen.
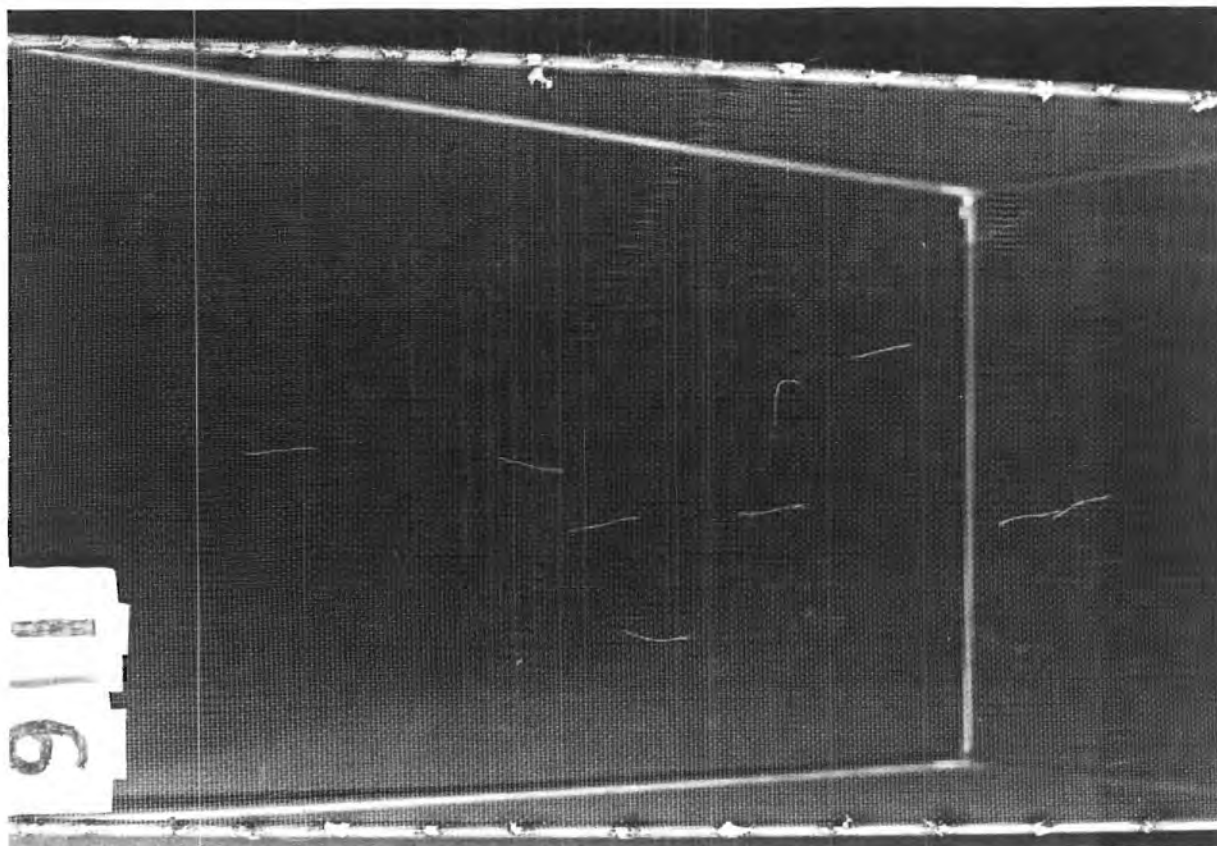
Figure 7.   Fibers collected on deposition screen.
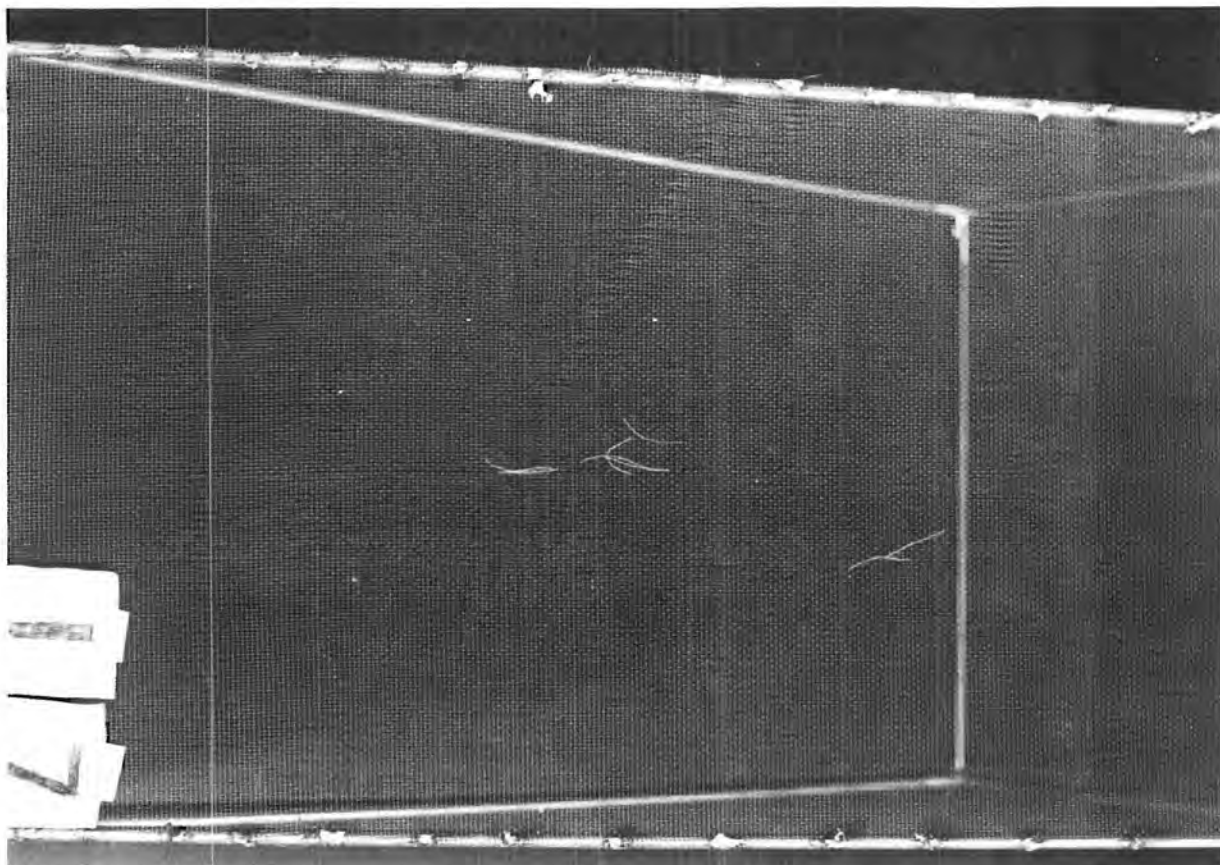
Figure 8.   Fibers collected on deposition screen.

For the tests identified above, of which figures 5-8 are a part, this evaluation system gave the following results:

Parallel	76%
45°	2%
90°	2%
Hooked	20%

These results are very encouraging, as the percentage of parallel fibers is large. Also, there is a reasonable expectation that refinements in the system can produce increases in the percentage of parallel fibers. This expectation is especially reinforced by certain observations made during the tests. It was observed that many of the "hooked" fibers were initially straight and parallel when they landed on the screen. Before the end of the run, however, one end of the fiber moved so as to form a hook shape. The exact cause of this behavior is not known at present, although it may be caused by a weak recirculation produced by the jets. Alleviation of this behavior is an obvious area for future efforts.

3.0   Single-Fiber Alignment Studies

Since the work reported in Reference 1, additional studies of the align-
ment of individual fibers have been made to clarify the mechanisms involved.
The following describes the principal results of this work. In section 3.1
an analysis of the dynamics of a single fiber is presented. A number of sim-
plifications are introduced, especially regarding the interaction of the counter-
flow jets and the free stream flow. Because of these simplifications, pro-
bably only qualitative comparisons can be made between the numerical pre-
dictions of fiber behavior and the actual photographed behavior. These quali-
tative predictions have proved to be very valuable, however, in providing in-
sight on the basic mechanisms that produce fiber alignment. Section 3.2 pro-
vides a brief comparison of numerical predictions with photographic results.

3.1   Dynamics of Single-Fiber Motion

3.1.1 Equations of Motion

In the analysis, the fiber was represented by a rigid cylinder.
Figure 9 shows the fiber nomenclature. The motion of the fiber was assumed to
be two-dimensional, i.e., parallel to the xy-plane. The equations of motion
of the fiber are then as follows:

Conservation of x-momentum: $F_x = m_f \cdot du_G/dt$ (1)

where, $u_G = dx_G/dt$ (2)

Conservation of y-momentum: $F_y = m_f \cdot dv_G/dt$ (3)

where, $v_G = dy_G/dt$ (4)
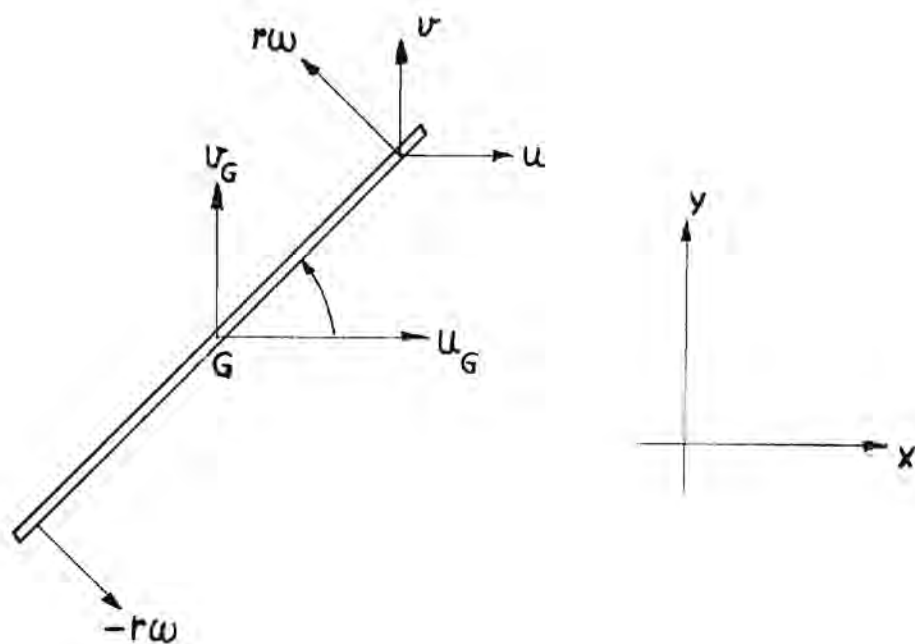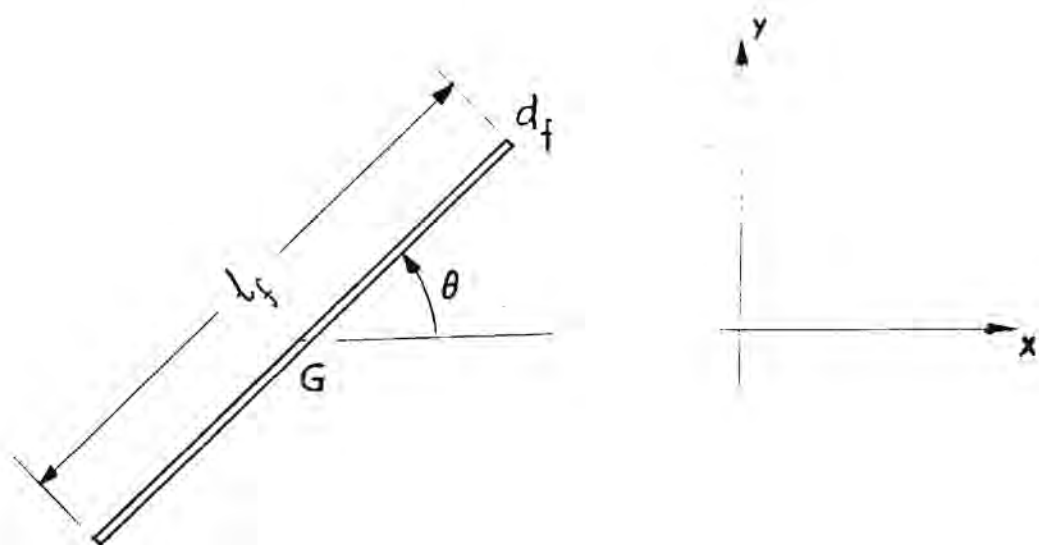
Figure 9.   Fiber Nomenclature

Conservation of z-angular momentum about the fiber center of mass G:

$$M_G = I_G \ d\omega/dt \tag{5}$$

where, $\omega = d\theta/dt$ (6)

All quantitites in the preceding equations were made nondimensional by the
following reference conditions: free stream density, ρ_∞ ; free stream velocity,
u_∞; and duct height, h. F_x and F_y are the components of the resultant force
on the fiber, and M_G is the resultant moment about G (directed parallel
to the z-axis). u_G and v_G are the velocity components of the fiber center
of mass. The fiber mass is $m_f = (\pi/4)d_f^2 \ \ell_f \ \rho_f$, where ρ_f is the fiber
density. x_G and y_G are the spatial coordinates of the fiber center of mass,
and t is time. The fiber moment of inertia about G is $I_G = m_f \ell_f^2 /12$.

The resultant forces and moments on the fiber are caused by the
relative velocity between the fiber and the surrounding air flow. Let W_n
be the nondimensional relative velocity normal to the fiber at a point on
the fiber. Then,

$$W_n = (u - u_G) \sin \ \theta \ - (v - v_G) \cos \theta \pm r\omega \tag{7}$$

where u and v are the velocity components of the air flow at the point
on the fiber, and r is the distance from G to the point on the fiber.
The $(\pm)$ preceding $r\omega$ denotes that one half of the fiber rotates into the
air stream, while the other half rotates away from it. Also, let W_τ be the
relative velocity tangent to the fiber at a point on the fiber. Then,

$$W_\tau = (u - u_G) \cos \theta + (v - v_G) \sin \theta \tag{8}$$

It should be emphasized that W_n and W_τ vary from point to point along the fiber, because of the spatial variation of u and v, and because of the variation of r. Equations (7) and (8) are valid for $0 \le \theta \le 180°$. For $180° < \theta < 360°$, they must be modified to the following:

$$W_n = - (u - u_G) \sin \theta + (v - v_G) \cos \theta \pm r\omega \qquad (7a)$$

$$W_\tau = - (u - u_G) \cos \theta - (v - v_G) \sin \theta \qquad (7b)$$

Let F_n' be the nondimensional normal force per unit length of fiber acting at a point on the fiber. This can be written in terms of a drag coefficient C_d as:

$$F_n' = (1/2) \ W_n^2 \ d_f \ C_d \qquad (9)$$

C_d is a function of R_n, the Reynolds number based on W_n, d_f, and ν, where ν is the kinematic viscosity of the air. Thus, $R_n = R_\infty \ W_n \ d_f$, where $R_\infty = u_\infty h / \nu$. Accurate values of C_d have been found by Choo and Casarella (2) for a wide range of Reynolds numbers. Because of the low relative velocities that occur for the present application, only the lower range of R_n is of interest. Then (2),

$$C_d = (8\pi / R_n S) \ (1 - 0.87 S^{-2}) \ (0 < R_n \le 1) \qquad (10)$$

where, $\quad S = - 0.077215665 + \ln (8/R_n)$

and, $C_d = 1.45 + 8.55 \ R_n^{-0.9} \ (1 < R_n < 30) \qquad (11)$

Next, let F_τ' be the tangential force per unit length of fiber acting at a point on the fiber. Then (2)

$$F_\tau' = (\pi W_\tau / R_\infty) \, (0.55 \, R_n^{1/2} + 0.084 \, R_n^{2/3}) \tag{12}$$

To evaluate the resultant force and moment acting on the fiber, it is divided into $(n + 1)$ segments (n is even) of length $\Delta\ell = \ell_f/(n+1)$, and the values of F_n'' and F_τ' at the center of each segment are assumed to apply over the whole segment. Thus,

$$F_n = \sum_{i=1}^{n+1} F_{n,i}' \cdot \Delta\ell \tag{13}$$

$$F_\tau = \sum_{i=1}^{n+1} F_{\tau,i}' \cdot \Delta\ell \tag{14}$$

$$M_G = -\sum_{i=1}^{n/2} r_i \, F_{n,i}' \cdot \Delta\ell + \sum_{i=n/2+1}^{n} r_i F_{n,i}' \cdot \Delta\ell \tag{15}$$

This method of evaluating the forces on the fiber assumes that each segment experiences the same force per unit length as on an infinite cylinder immersed in a uniform stream having velocity components u, v.

Here the convention is followed that counterclockwise moments are positive. F_x and F_y are then obtained from,

$$F_x = F_n \sin \theta + F_\tau \cos \theta \tag{16}$$

$$F_y = F_n \cos \theta + F_\tau \sin \theta \tag{17}$$

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for $0^\circ \leq \theta \leq 180^\circ$, while for $180^\circ < \theta < 360^\circ$, the corresponding formulae are,

$$F_x = - F_n \sin \theta - F_\tau \cos \theta \qquad (16a)$$

$$F_y = F_n \cos \theta - F_\tau \sin \theta \qquad (17a)$$

3.1.2 Specification of Air Velocity Field

In a general calculation of the fiber motion, the air velocity field $u, v = fn (x,y)$ is also unknown, because the presence of the fiber alters u and v from their undisturbed values. An essential simplification intro-duced here is that $u, v = fn (x,y)$ are known functions. This velocity field is chosen to approximate that produced by the interaction of a uniform free stream with air jets blowing perpendicular to the free stream. The presence of the fiber is assumed to have a negligible effect on the air velocity field.

The present experimental configuration has a uniform flow between plane parallel walls, with air jets blowing inward from the upper and lower walls. It is well known from studies of jets in cross flows (e.g., Abramovich(3), Taylor(4)) that the jet flow is deflected until it is nearly parallel to the cross flow direction. To estimate the effect of the jets on the main stream, a technique similar to that of Taylor (4) was used. The similarity is that the jets were assumed to have the same effect on the main flow as some combination of sources and sinks parallel to the x-axis. The difference from Taylor is that the present boundary conditions require $v = 0$ at the upper and lower

walls, and also along the central plane of the duct, by symmetry. The flow field between adjacent sources (or, sinks) of an infinite row of sources parallel to the y-axis satisfies these boundary conditions (e.g. Streeter)(5). A combination of a uniform velocity in the direction of the positive x-axis, one row of sources, and two rows of sinks was used to approximate the flow field. The resulting formulae for the air velocity components are:

$$u = 1 + C_1 \, \pi \, \sinh x_1'/(\cosh x_1' - \cos y')$$

$$- C_2 \, \pi \, \sinh x_2'/(\cosh x_2' - \cos y')$$

$$- C_3 \, \pi \, \sinh x_3'/(\cosh x_3' - \cos y') \tag{18}$$

$$v = \pi \, \sin y' \left[C_1/(\cosh x_1' - \cos y') - C_2/(\cosh x_2' - \cos y') \right.$$

$$\left. - C_3/(\cosh x_3' - \cos y') \right] \tag{19}$$

The stream function is given by:

$$\Psi = - y - C_1 \, \tan^{-1} \left[\tan (y'/2)/ \tanh (x_1'/2) \right] \tag{20}$$

$$+ C_2 \, \tan^{-1} \left[\tan (y'/2)/ \tanh (x_2'/2) \right]$$

$$+ C_3 \, \tan^{-1} \left[\tan (y'/2)/ \tanh (x_3'/2) \right]$$

where $x_i' = 2\pi(x - x_1)$, $x_2' = 2\pi(x - x_2)$, $x_3' = 2\pi(x - x_3)$ and $y' = 2\pi y$. C_1 is the strength of the sources located at $x = x_1$, $y = 0$, h. C_2 and C_3 are the strengths of the sinks located at $x = x_2$, $y = 0$, h and $x = x_3$, $y = 0$, h, respectively.

It is desirable to relate the characteristics of the effective body produced by the sources and sinks to the characteristics of the actual jets. In particular, it is desired to relate C_1, C_2, and C_3 to the jet mass flow rate, $\dot{m}_j$ and the jet velocity v_j. Then it is possible to relate the results of a theoretical calculation to a particular experimental situation.

Taylor's analysis[4] is most accurate in the region close to the jet orifice. One of his main results was that

$$c_o = - 2 \, v_j^2 \, b \tag{21}$$

where c_o is the radius of curvature at the upstream stagnation point of the effective body produced by a single two-dimensional jet, and b is the width of the jet slot. The corresponding mass-flow rate through a single jet is $\dot{m}_{j,1}$, and:

$$\dot{m}_{j,1} / \dot{m}_\infty = v_j \, b \tag{22}$$

With jets at $x = 0$, $y = 0$ and $x = o$, $y = h$ the total jet flow rate is $\dot{m}_{j,2} = 2\dot{m}_{j,1}$, so that

$$c_o = - v_j \cdot \dot{m}_{j,2} / \dot{m}_\infty \tag{23}$$

A corresponding expression for c_o can be derived for the flow field and effective body described by equations (18) – (20). This is:

$$c_o = - (3/2) \, C_1 / (1 + \pi \, C_2') \tag{24}$$

where $C_2' = C_2 + C_3$.

In deriving (24), it was assumed that the sinks were located relatively far downstream, $x_2 - x_b > 1$ and $x_3 - x_b > 1$, so that $\tanh (x_2'/2) \equiv$

- 48 -

tanh $(x_3'/2) \equiv -1$. Equating (23) and (24) gives:

$$(3/2)C_1 / (1 + \pi C_2') = v_j \, \dot{m}_{j,2} / \dot{m}_\infty \tag{25}$$

If it is required that the main flow have the same uniform velocity at $x = \pm \infty$, then $C_1 = C_2'$. With this requirement, C_1 can be related to the jet parameters by equation (25).

The mixing between the jets and the main flow results in a growing shear layer between them as x increases. As a result, the preceding theoretical flow description becomes progressively less accurate in the downstream direction. In the present application to fiber alignment, interest is concentrated in the region near the jet injection, $x < 0.5$, however. Thus, it is expected that the preceding analysis will at least give the correct qualitative behavior of the fiber.

3.1.3 Numerical Results

A number of calculations have been made for the following particular case: $C_1 = 0.2$, $C_2 = C_3 = 0.1$, $x_1 = 0$, $x_2 = 2$, $x_3 = 4$. The upstream part of the effective body shape produced by the sources and sinks for this case is shown in Figure 10. It is apparent that this combination causes the mainstream flow to converge rapidly and then diverge slowly. This is qualitatively the same behavior as produced by the counterflow jets. This type of convergence and divergence of the main stream, asymmetric with respect to the yz - plane, is essential for producing fiber alignment nearly parallel to the x-axis.

EFFECTIVE JET SHAPE
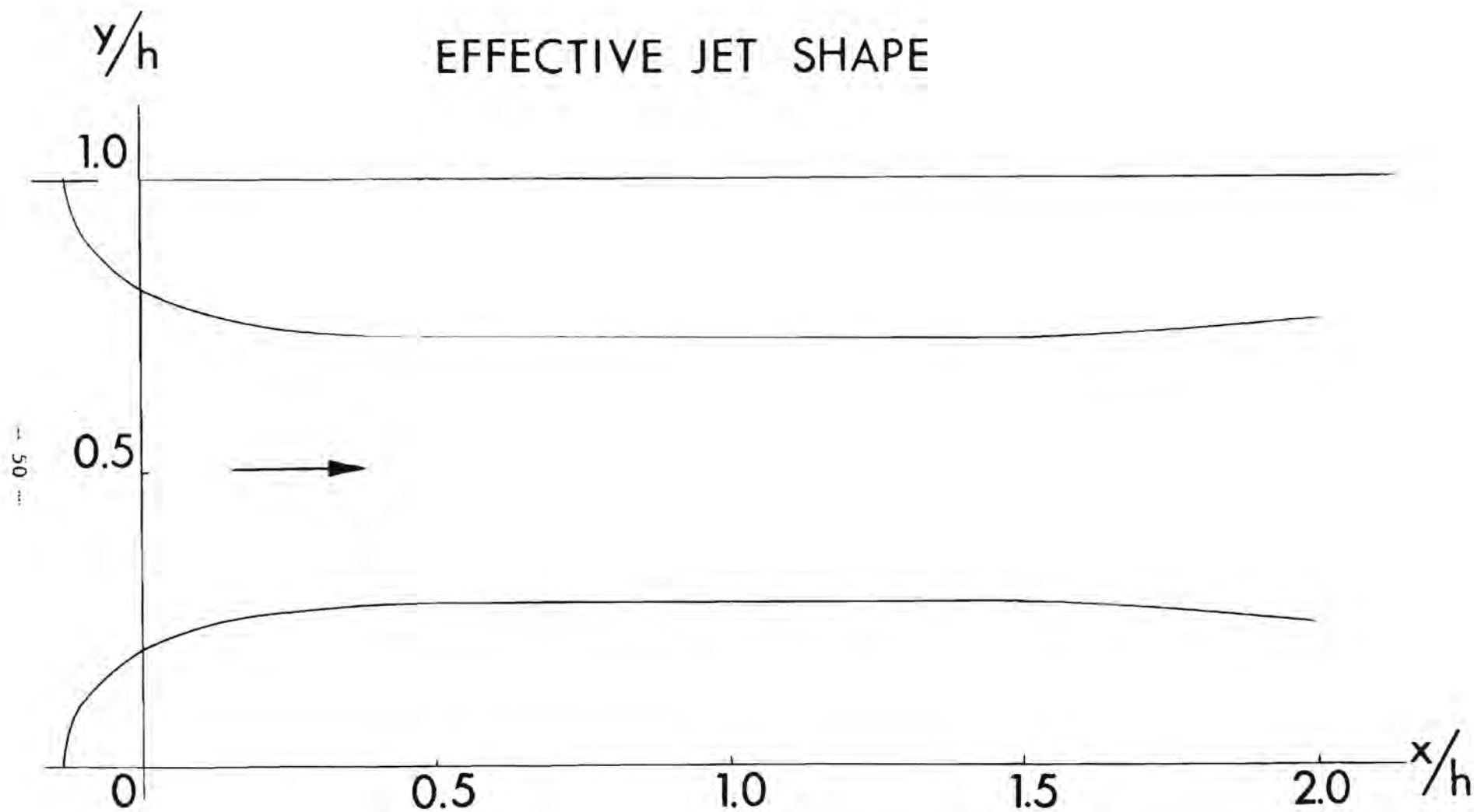
Figure 10.   Effective body shape produced by sources and sinks, approximating the effects of the counterflow jets.

Figures 11 and 12 illustrate the predicted influence of the initial fiber inclination angle, θ_0, on the fiber inclination angle $\theta_{0.5}$ at $x = 0.5$. The calculated results of Figure 11 are based on the assumption that gravity acts in the z-direction, and so does not affect the fiber dynamics. The release point was $x_{Go} = -0.5$ (i.e., one-half duct height upstream of the jets). Curves are shown for $y_{Go} = 0.3$, 0.4, and 0.5. Also, it was assumed that $\omega_o = 0$, $v_{Go} = 0$, and $u_{Go} = 0.1 \, (u)_{x = -0.5}$. The nonzero value for u_{Go} was chosen to avoid numerical difficulties in computing the very large fiber acceleration from rest. By choosing a small, but nonzero, value for u_{Go} the essential physical behavior was retained. The fiber parameters are $\rho_f = 1290$ (cotton), $\ell_f = 0.0833$, and $d_f = 2.0 \times 10^{-4}$. For a duct height $h = 1 \, ft$, as used in the experiments, this corresponds to a fiber of 1 inch length and 60 μm (0.0024 inch) diameter.

The calculated results of Figure 11 show that for θ_o from 0° to nearly 90°, the fibers are rotated clockwise by the action of the jets, so that they are aligned nearly parallel to the x-axis (i.e., within 20° of the x-axis). There is an intermediate range of values of θ_o for which the fibers do not become parallel to the main stream. Within this interval the resultant direction of rotation changes from clockwise to counterclockwise. Finally, for values of θ_o greater than about 120°, the fibers are rotated counterclockwise so that they are essentially parallel to the x-axis.

The variation of θ with x is shown in Figure 12 for several values of θ_o and $y_{Go} = 0.3$. Again, it is apparent that alignment occurs within a short distance, and that most of the change in θ has been completed upstream of $x = 0.5$.
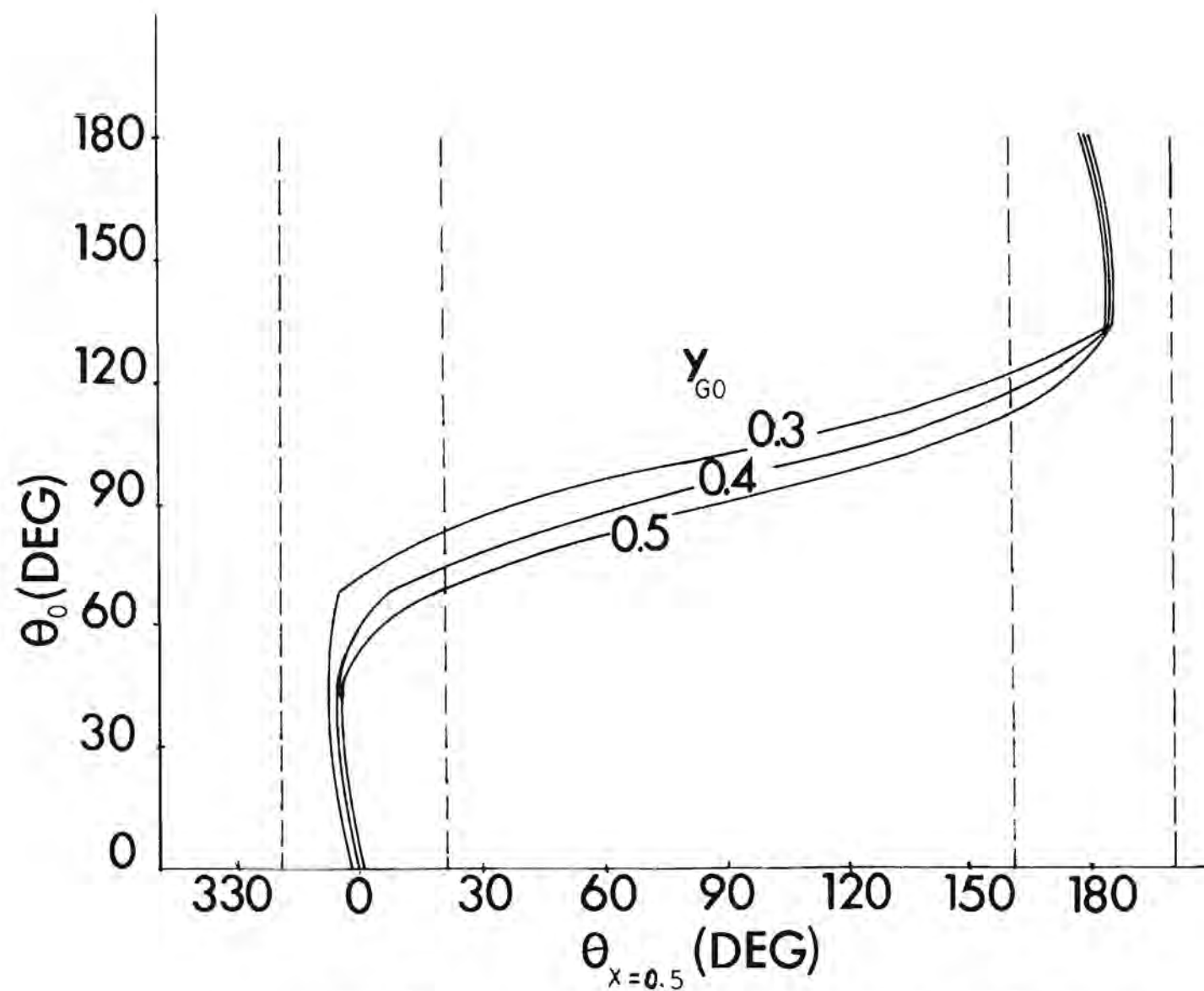
Figure 11. Effect of initial fiber inclination angle, θ_o,
on inclination angle at x = 0.5.
No gravitational effect.
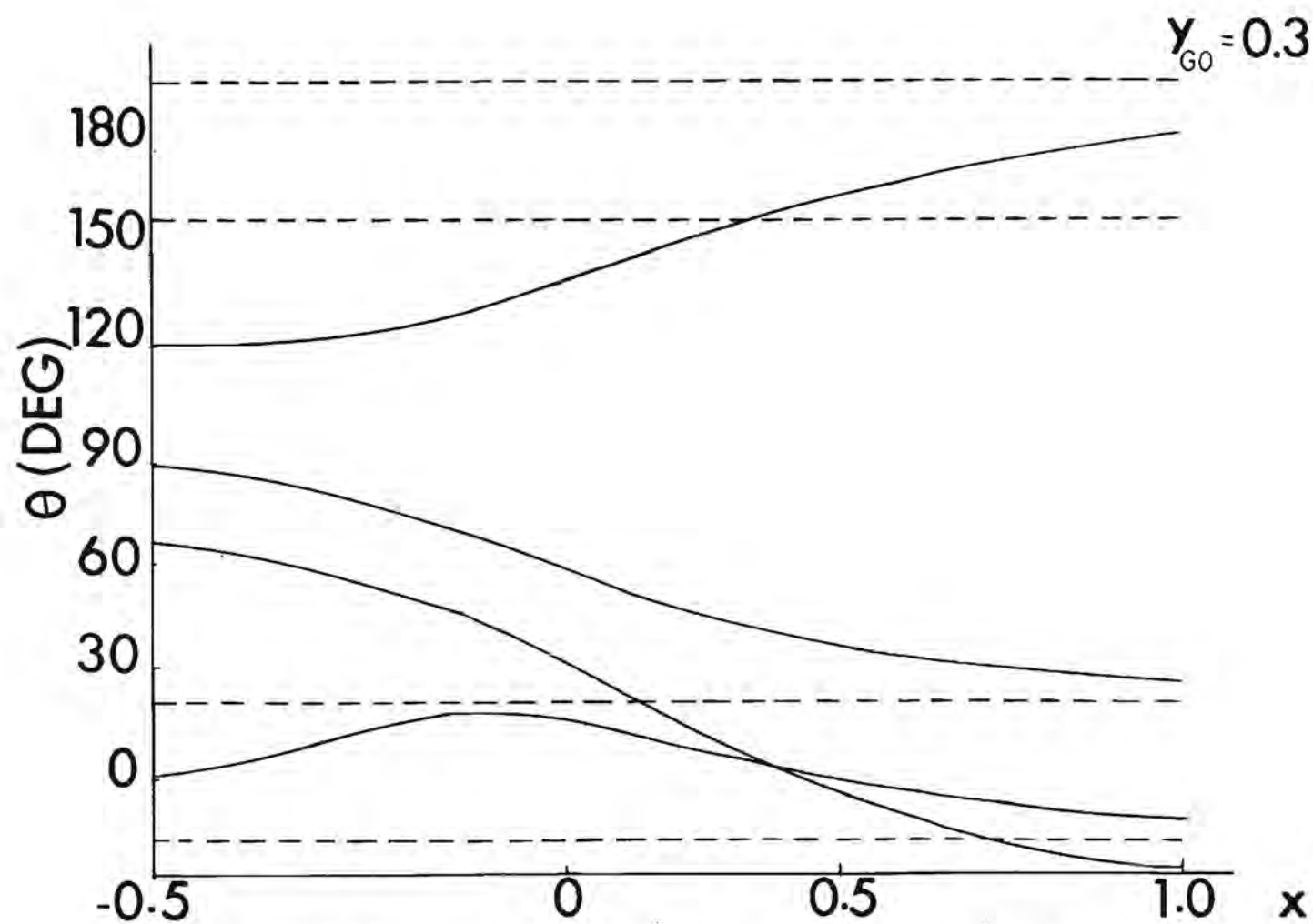
Figure 12.   Variation of fiber inclination angle with axial
distance. y_{Go} = 0.3. No gravitational effect.

The application of this system to fiber weaving requires that the fibers become nearly parallel to the x-direction. The preceding analysis indicates that θ_o must be less than about 90° for this to occur. (Calculations have not been made for a wide range of fiber and jet parameters, but it seems clear that there will always be a small intermediate range of θ_o for which the direction of rotation is reversed. Within this small range, the final alignment will not be parallel to the x-axis). This suggests that a fiber-feed system should be used which restricts θ_o to less than 90°. The simple fiber injector described in section 2 satisfies this requirement completely. This is because the fibers are blown off the injector screen close to the point where the screen is tangent to the main stream velocity vector. As a result, the inclination of the fiber for the xz-plane is less than 90°. The photographs of Figures 5 - 8 provide some confirmation. Thus, this system of fiber alignment and deposition seems to be feasible for application to the fiber weaving process.

3.2 Comparison of Calculations with Photographs of Fiber Motion

A brief comparison is given here of the calculated results described in section 3.1, and multiflash photographs of the fiber motion. In these experiments, the fibers were released at $x_{Go} = -0.5$ and $y_{Go} = 0.6$. Before release, the fibers were held fixed relative to the tunnel by the fiber holder visible at the far left in Figures 13 - 15. The fibers were released when a solenoid acted to open the jaws of the fiber holder. Simultaneously, the camera shutter opened. The force exerted by the main stream on the fiber then carried it downstream. The camera shutter stayed open for one

second, and the stroboscopic light source flashed at 3600 flashes per minute. Cotton fibers with diameter 60 μm and length one inch were used. Corresponding free stream velocity was 20 ft/sec and the duct cross-section is 1 ft by 1 ft. The jet velocity was 150 ft/sec and the jet mass flow rate (both jets) was 2.4 lb_m/min.

Because of the arrangement of the experimental apparatus, gravity was acting in the negative y-direction. That is, in the photographs of Figures 13 - 15, gravity acted downward. For this reason, computations were made in which gravity acted in the negative y-direction, and the results are shown in Figure 16. The computed behavior is very similar to that shown in Figure 11. Because of the influence of gravity, values of y_{Go} above the center line were chosen, so that the fiber would not fall into the shear layer during its trajectory.

It is only possible to make qualitative comparisons between the calculated motion of Figure 16 and that observed in Figures 13-15. This is because of the various approximations in the analysis, discussed previously. With this restriction, the calculated and observed results appear to be consistent. In the photograph of Figure 13, $\theta_o = 90°$. A small counterclockwise rotation is observed, consistent with the prediction of Figure 16 for $y_{Go} = 0.6$. In Figure 14, $\theta_o = 60°$, and the fiber quickly rotates clockwise to a parallel alignment as anticipated from Figure 16. Further downstream, it is caught up in the rotational flow of the shear layer between the jet and the main stream. This illustrates the desirability of depositing the fibers a short distance downstream of the jets, as was done in Figures 5-8. Lastly, in Figure 15, $\theta_o = 120°$. The fiber rotates counterclockwise until parallel to the free stream. Again, this is consistent with the predictions of figure 16.
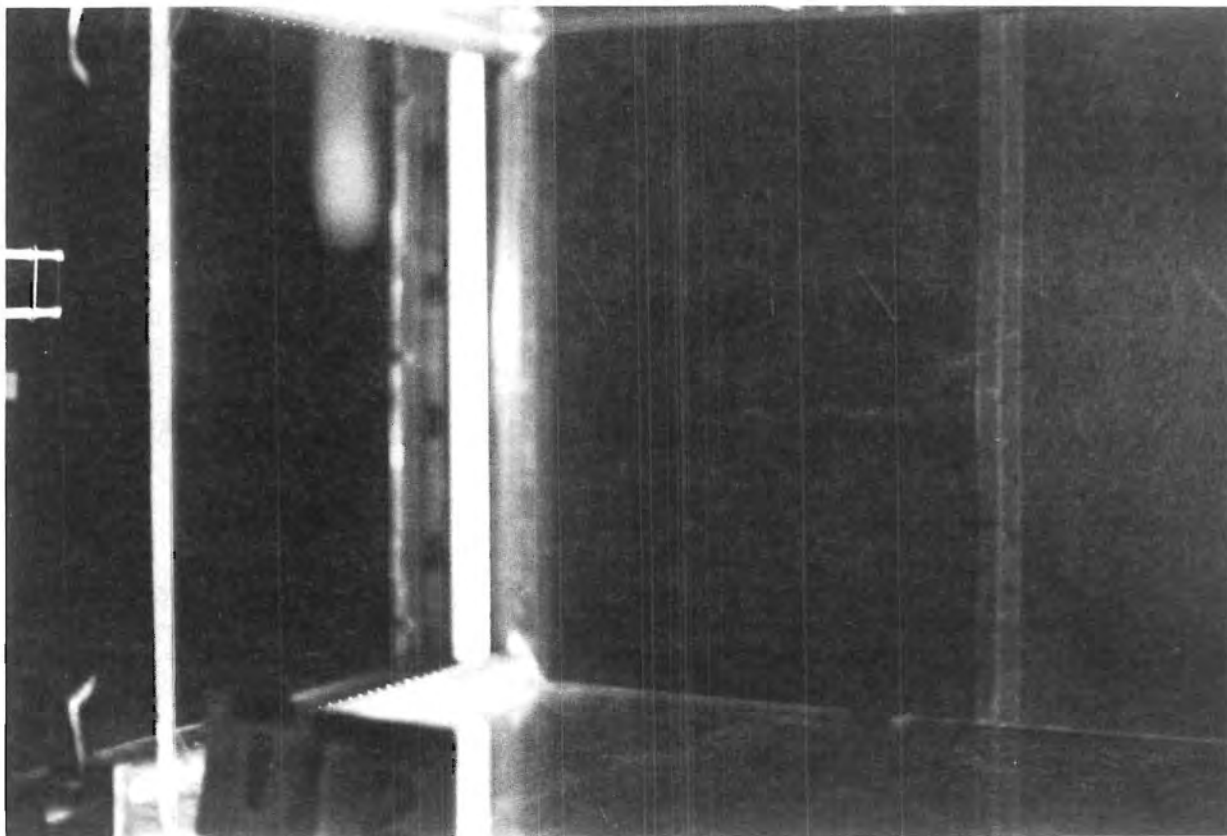
Figure 13.   Multiflash photograph of single-fiber motion.
$\dot{m}_{j,2}/\dot{m}_\infty = 0.026$; $v_j/u_\infty = 7.4$; $\theta_o = 90°$.
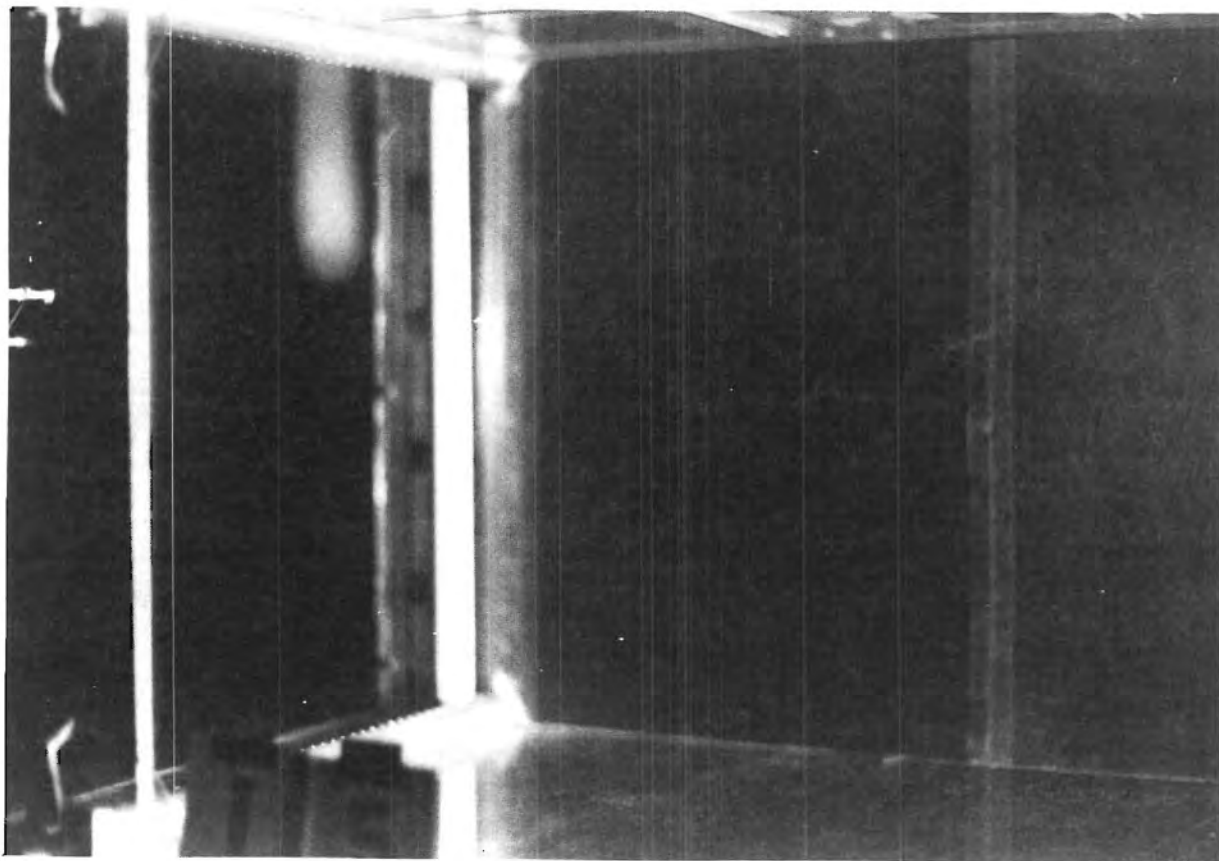
Figure 14.   Multiflash photograph of single-fiber motion.
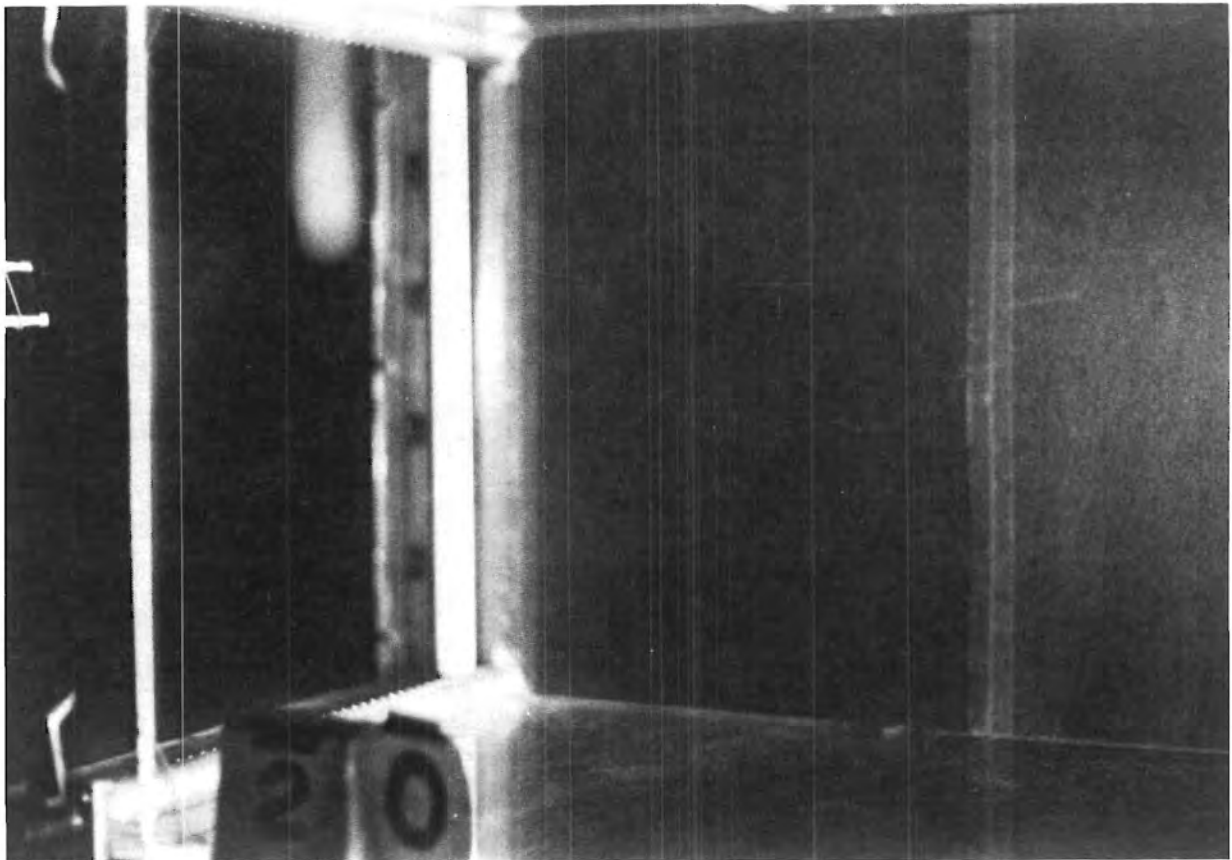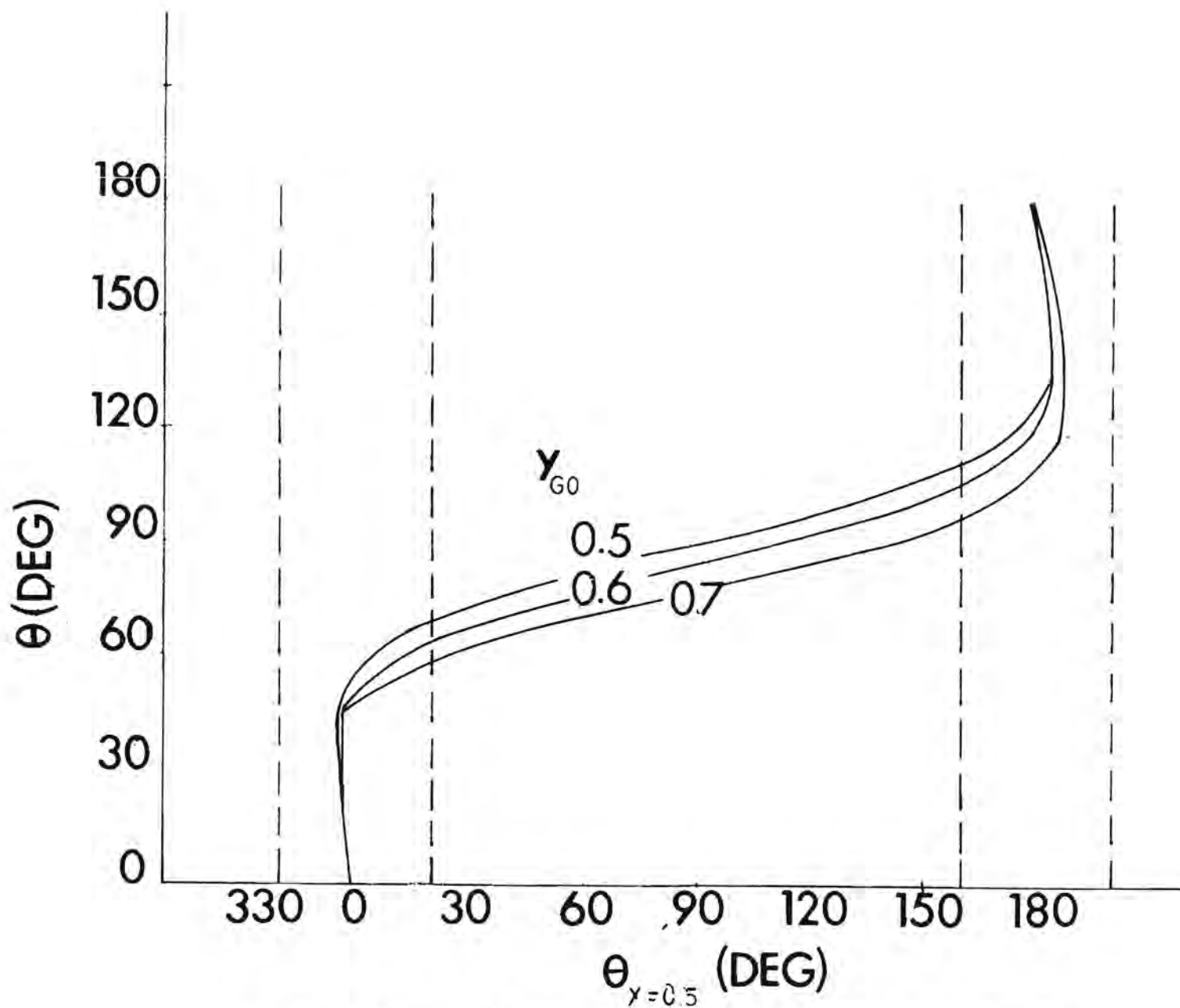$\dot{m}_{j,2}/\dot{m}_{\infty} = 0.026$; $v_j/u_{\infty} = 7.4$; $\theta_o = 60°$.

Figure 15.   Multiflash photograph of single-fiber motion.
$\dot{m}_{j,2}/\dot{m}_\infty = 0.026$; $v_j/u_\infty = 7.4$; $\theta_o = 120°$.

Figure 16.   Effect of initial fiber inclination angle, θ_o, on inclination angle at $x = 0.5$. Gravity acts in negative y-direction.

4.0 Summary

This research is directed toward the development of processes that will make fiber weaving technically feasible and economically attractive. In particular, this work has concentrated on discovering methods whereby fibers can be oriented and deposited in parallel arrays by using air flows and aerodynamic forces.

Recent experiments on fiber alignment and deposition are described. The results are very encouraging, as a very high percentage of fibers were deposited in the desired parallel direction for certain test conditions. As these are early results, and the system has not been optimized, there is a reasonable expectation that refinements can result in higher percentages of parallel fibers.

The preceding developments were based on a method of single-fiber alignment discovered earlier in this research program. A more detailed analysis of this method has been performed and is presented herein. This analysis has been of great value in clarifying the basic mechanisms that lead to fiber alignment, and in the design of the experiments on multiple-fiber alignment.

5.0 Future work

The basic experiments on multiple-fiber alignment and deposition.
described in section 2.0 will continue. The objective is to increase the
percentage of fibers deposited in parallel arrays. The effects of variations
in system parameters will be examined, as well as possible changes in the
design of the deposition screen and the fiber injector.

Because of the very encouraging results achieved to date on multiple-
fiber alignment and deposition, the next major step is to provide for a con-
tinuous supply of fibers to the system. One approach now being considered
is to have a device, such as an open-end spinning head, deposit individual
fibers onto the injector screen. The latter would then carry the fibers
into the main steam, as described previously. Once this system is designed
and built, it will be possible to perform alignment and deposition experiments
involving large numbers of fibers. These experiments will lead directly to
experiments on web formation, with the objective of depositing large numbers
of fibers in two perpendicular directions. The fibers would then be inter-
twined by some existing method. The system design and fabrication will begin
during the remainder of this (second) year of the program. The bulk of this
work will be done in the third year of the program.

Another, very different, technique for fiber alignment is also being
investigated. This technique is based on certain characteristics of the
interaction between solid cylinders and a surrounding fluid which is in a
state of nearly uniform shear. Under certain conditions these cylinders take
up fixed orientation with respect to the fluid. The next step is to determine

whether these conditions are suitable for application to a fiber weaving system. The results of this preliminary investigation will be given in the annual progress report for this year.

IV.   Utilization Plan

The procedures and time schedule that are planned to ensure that the technology developed under the proposed program is transferred into the textile and apparel industries are outlined in Table 9.

Since the First Annual Technical Progress Report, the following presentations have been made and papers published.

1.   "Polypropylene Web Structures by Blown Film Extrusion", Proc. of the 4th Tech. Symp. of INDA, 71, J.D. Muzzy, C. Day and A.C. Levy, March 1976.

2.   "Web and Yarn Formation by Biaxially Stretching Foam", J. Appl. Polymer Symp., in preparation (1977). Presented at the ACS Meeting on "Fiber Structure and Properties", Knoxville, Tenn., Nov. 16, 1976, J.D. Muzzy.

3.   Seminar on New Nonwoven Processes presented by W.D. Freeston at Scott Paper Company, Philadelphia, PA, May 20, 1976.

4.   "Nonwovens" presented by W.D. Freeston at the Conference on New Directions in Textile Technology, sponsored by the Engineering Foundation at Rindge, N.H., Aug. 25, 1976.

5.   "Apparel Research-Polymer-to-Garment" presented by W.D. Freeston at the International Nonwovens and Disposables Assocation, IDEA 76 Conference, Chicago, Illinois, October 20, 1976.

The conference on "Fibrillation Processes and Products to be sponsored by the Society of Plastics Engineers and organized by Dr. Muzzy has been cancelled due to an inadequate number of abstracts being submitted. The response was sufficient for a one day conference and Dr. Muzzy is considering this possibility.

As soon as Dr. Lundberg has solubility data for the nylon-CO_2 system and some preliminary extrusion results, he plans to report on solubilities of polymers in gases and extrusion of gaseous solutions. The presentations will be at meetings of appropriate professional societies and trade associations.

Table 9. Utilization Plan

	0	12	24	36
Progress Reports	X X X X X X
Research Reviews	X X X
Presentations to:				
Trade Associations	X-----------------------X
Professional Societies	X-----------------------X
Technical Conference & Demonstrations				X
Summary Technical Report				X

Dr. Muzzy is preparing a paper entitled "Foam Extrusion of Yarns and Webs". It will be co-authored by G.H. Hoyos and Y.H. Park, and will appear in SPE Tech Papers 23. Dr. Muzzy will present the paper at the SPE ANTEC, Montreal, Canada, April 25 - 28, 1977.

Dr. Bangert has prepared a Record of Invention titled, "Method for Fiber Alignment Using Fluid Dynamic Forces", dated 15 September 1976. A copy was sent to the National Science Foundation on 7 October 1976. This material has been reviewed by Georgia Tech's patent attorneys, and they believe that a patent may be obtainable. Accordingly, a formal patent application is being prepared.

The results of the studies on single fiber alignment, including the material contained in this report, are being written up as a paper. This will be submitted for publication in the near future.

V. Conclusions

No additional data on the solubilities of polymers in dense gases have been obtained during the past six months. A change in support personnel caused some delays. However, the major problem has been the difficulty in achieving glass to metal seals around windows in the 210-230°C temperature region and the 800 to 1400 atmosphere pressure range. This difficulty indicates that carbon dioxide is a good solvent for hydrocarbon and halocarbon polymers at elevated temperatures and pressures.

The feasibility of forming yarns suitable for textile applications by foam fibrillation has been demonstrated. Substantial increases in tenacity are achieved by hot drawing and twisting. Also, increased biaxial stretching of the foam extrudate improves the ultimate yarn tenacity. Experimental polypropylene yarn has been produced with mechanical properties similar to commercial staple fiber yarn.

Foam fibrillated polypropylene webs with basis weights greater than 1 oz/ sq.yd. have been extruded. The capability to form webs from polybutylene, nylon 66 and polyesters has not been adequately demonstrated. Improvements in the formulation of the feed mixture, in process design and in process control are needed in order to process nylon 66 and PET. Polybutylene and PTMT can be processed, but more work must be performed to form webs which equal or exceed the polypropylene webs in quality.

It has become apparent in conducting process studies that conditions which lead to high web tenacities and low permeabilities tend to correspond to poor fibrillation. By adding fumed silica and silica flour both web uniformity and fibrillation improve. However, lower tenacities and higher per-

meabilities are incured. These fine particle additives appear to be essential components for forming webs using nitrogen as a blowing agent. With the additives, webs formed using nitrogen compare favorably with webs formed using chemical blowing agents.

Continuing investigations of methods for orienting staple fiber and depositing fibers in parallel arrays using air flows and aerodynamic forces are encouraging. A system has been developed by which a very high percentage of fibers are deposited in the desired parallel direction. It is anticipated that optimization of the system will yield higher levels of fiber alignment.

VI.   Future Plans

Carbon dioxide appears to be quite a good solvent for gaskets made of flurocarbon polymers at or above 200°C. Therefore, making of gas-tight glass to metal and metal to metal seals with organic "O" rings in the 225°C and 1000 plus atmosphere region necessary to dissolve nylon 6 in carbon dioxide is difficult and wasteful of time. For this reason, a new pressure vessel with windows capable of holding gases at 300°F and pressures up to 1700 or 2000 atmospheres will be designed in cooperation with and obtained from a pressure instruments maker. Such a vessel should permit studies of solubilities of nylon 6 (mp. 212° - 225°C) in carbon dioxide without too much difficulty. Further, within this pressure range the higher melting nylon 66 (m.p. 250° - 265°C) and poly (ethylene terephthalate) (mp. 248° - 293°C) should be soluble in carbon dioxide. Studies of these solubilities should be possible (but not easy) with this apparatus.

While the new pressure vessel is being constructed, studies of solubilities of polypropylene (mp 162° - 176°C) in carbon dioxide will be completed. The batch extrusion apparatus (for which most essential parts are on hand) will be assembled. Studies of the extrusion of polypropylene in carbon dioxide will begin within the next six months.

A continuous system for drawing foam fibrillated yarn will be developed to achieve better control of drawing conditions and to facilitate increased yarn production for product evaluation. The effect of extrusion conditions and material composition will be evaluated - particularly higher molecular weight grades of polypropylene. Also, nylon and polyester yarns will be evaluated as soon as reasonably uniform webs are produced.

Efforts to produce strong fibrillated webs from polybutylene, nylon 66 and polyesters will be continued. It should be possible to accomplish this by making improvements in the formulation of the feed mixture, process design and process control.

Experiments on multiple-fiber alignment and deposition will continue. The objective will be to increase the percentage of fibers deposited in parallel arrays. The effects of variations in system parameters will be examined, as well as possible changes in the design of the deposition screen and the fiber injector.

VI. References

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Advanced Technology Applications
in Garment Processing

by

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Second Annual Technical Progress Report

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Abstract

New, direct, automatic, fast and inexpensive methods are being developed
for converting polymer chips and/or staple fiber into fabrics without carding,
spinning and weaving or knitting, and garments without cutting and sewing.
Other objectives are to reduce material, labor, capital, and energy costs in
textile and garment manufacture. The successful development of one or more
of these processes will contribute to improving the United States' competitive
position in textiles and clothing in the U.S. and world markets.

Polybutene-1 and polypropylene have been shown to be readily soluble in
n-butane and carbon dioxide at concentrations which should be useful in
transporting and fabricating these polymers. Nylon-6 is dissolved easily in
carbon dioxide at suprisingly low pressures.

Preliminary trials show that fibrous polymers can be produced by extrud-
ing gaseous solutions of polymers into the atmosphere.

Polypropylene yarn suitable for some industrial applications and com-
parable to commercial textile yarn has been produced by foam extrusion. Some
success has been achieved using mixtures of poly(butylene terephthalate) (PBT)
and polypropylene and also pure PBT.

A fiber injection system has been designed and constructed to continuously
supply individual fibers to an aerodynamic alignment system. Experiments
underway on web formation are encouraging.

Table of Contents

I. Executive Summary

The various phases of the studies and the work schedule are given in Table 1.

1. The principal areas of research during the past six months have been:

1. Measurements of solubilities of polymers in dense gases

2. Continuous yarn formation from fibrillated polymer mixtures

3. Orientation of staple fiber and deposition of fibers in parallel arrays using air flows and aerodynamic forces.

The program management plan is shown in Figure 1. Dr. Freeston is responsible for overall planning, coordinating and supervision of activities. To promote a continual interchange of ideas among the personnel involved in the research, program reviews are held monthly.

The research on polymer solutions is being directed by Dr. John Lundberg. He is being assisted by research engineers G.H. Hoyos and A.R. Enuga, and Ken Ko, a graduate student.

The research on polymer fibrillation is under the direction of Dr. John Muzzy. He is being assisted by research engineers G.H. Hoyos and A.R. Enuga, and graduate students I. Moradinia and Y.H. Park.

Dr. Lou Bangert is directing the research on fiber aerodynamics. Mr. John Harper, a research engineer, and Mr. P. Sagdeo, a graduate student, are assisting him.

Technology transfer is being accomplished by presentations at textile and apparel trade association meetings, professional society meetings, seminars presented at companies, private discussions with company representatives, and seminars at Georgia Institute of Technology. Progress reports are being distributed to interested textile and apparel companies. The Utilization Plan Schedule is given in Table 1.
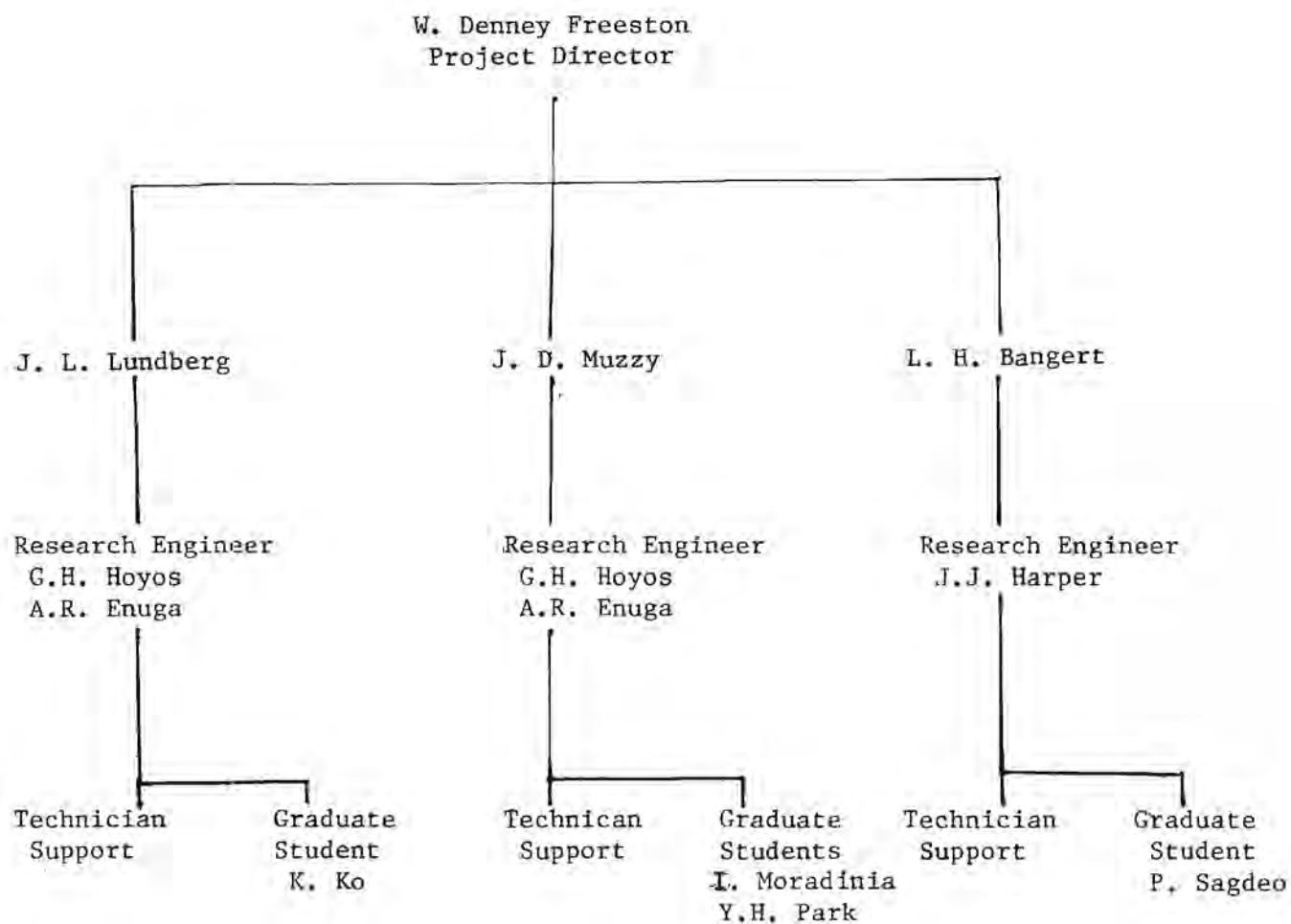
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Figure 1.   Program Management Plan

Table 1

Program Schedule

```
                                        0        12       24       36       42
                                        _____

A.  Gas-Polymer Solutions              X -------------------------------------X

B.  Gas-Polymer Solutions
      Extrusion Studies                                 X--------------X

C.  Foam Fibrillation

    a.  Effects of additives on        X----------------X
          fibrillation
    b.  Effects of crystallization
          and deformation conditions   X----------------X
          on fibrillation
    c.  Continuous yarn formation from
          inhomogeneous polymer mixtures        X----------------------------X
    d.  Use of tubular biaxial stretch-
          ing for fabric formation     X------------------------------------X
    e.  Development of laboratory scale
          yarn or fabric formation
          process                               X----------------------X

D.  Aerodynamic Fiber Alignment

    a.  Single fiber response          X------------X
    b.  Multiple fiber suspension          X--------X
    c.  Fiber weaving and entanglement          X----------------X

E.  Electrostatic Fiber Alignment                       X--------------X

F.  Utilization Plan

    Progress Reports                   X--------X--------X----------------X
    Research Reviews                       X--------X--------------X
    Presentations to
          Professional Societies       X----------------------------------------X
          Textile & Apparel Assocs.    X----------------------------------------X
    Technical Conference & Demonstrations                                  X
    Summary Technical Report                                               X
```

The schedules for the studies planned during the third year are given in Table 2.

Polybutene-1 and polypropylene are readily soluble in n-butane and carbon dioxide at concentrations which should be useful in transporting and fabricating these polymers. Nylon-6 is dissolved easily in carbon dioxide at surprisingly low pressures. Solubility measurements on polyethylene terephthalate, polyacrylonitrile and polyparaphenylene terephthalamide will be attempted during the next several months.

Preliminary trials show that fibrous polymers can be produced by extruding gaseous solutions of polymers into the atmosphere. The first system being investigated is nylon 6 dissolved in carbon dioxide.

It has not been possible to produce foam fibrillated polymer webs suitable for textile applications. Webs produced by foam extrusion of polypropylene under optimum conditions are not uniform, have low tensile properties in both the machine and the transverse directions, and show large directional imbalances of these tensile properties.

Polypropylene yarn suitable for some industrial applications and comparable to commercial textile yarn has been produced from fibrillated polymer mixtures. Pilot-scale quantities of yarn will be produced for evaluation in various fabric constructions during the next year. Economic evaluations will also be made for producing yarn in commercial quantities.

Initial attempts to produce an oriented fiber web using an aerodynamic alignment system are encouraging. This study will be continued during the next year. The feasibility of producing an aligned web by conveying fibers through an electrostatic field will also be investigated.

TABLE 2

Work Plan

	July 1977	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	Apr.	May	June 1978
A. Gas Polymer Solutions												
1. Nylon 6 in CO_2												
2. Other gas-polymer systems												
B. Extrusion of Polymers from Gaseous Solutions												
1. Assemble extrusion system												
2. Extrude nylon 6 - CO_2												
3. Extrude polypropylene CO_2												
4. Extrude other polymer-gas systems												
C. Foam-Fibrillation												
1. Web Formation												
2. Yarn Formation												
a. Process Design												
b. Process Parameter Evaluation												
c. Polypropylene Yarns												
d. Nylon and Polyester Yarns												
e. Fabrics from Yarns												
f. Economic Evaluation												

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Task	July 1977	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	Apr.	May	June 1978
D. Aerodynamic Fiber Alignment												
1. Fiber Supply System												
2. Fiber deposition screen design												
3. Experiments on web formation												
4. Evaluation of fiber alignment in web												
5. Fiber entanglement and/or bonding to form fabric												
6. Evaluation of fabric												
E. Electrostatic-Fiber Alignment												
1. Web formation												
2. Evaluation of web												

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II. Introduction

The primary objective of the program is to develop new, direct, automatic, fast and inexpensive methods for converting polymer chips and/or staple fiber into fabrics without carding, spinning and weaving or knitting, and garments without cutting and sewing. Other objectives are to reduce material, labor, capital, and energy costs in textile and garment manufacture. The successful development of such processes will contribute to improving the United States' competitive position in textiles and clothing in the U.S. and world markets.

The current studies have five major thrusts:

1) Determination of the solubility of several polymers in selected gases.

2) Extrusion of polymers from gaseous solutions.

3) Continuous yarn formation from fibrillated polymer mixtures.

4) Production of oriented fiber web using air flows and aerodynamic forces.

5) Production of oriented fiber web using air flows and electrostatic forces.

The results of the investigations during the past six months are presented in the next section.

III. Technical Activities
  A. Gas-Polymer Solutions
    1. Solubility Studies
      a. Method

Solubilities of gases in polymers are measured by observing 'cloud points' as pressure is decreased causing phase separation to occur. In practice, two pressures are recorded, one at which light scattering in the polymer solution becomes sufficient to distort the laser beam used as a probe and one at which extinction or near extinction is observed.

The method consists of placing a weighed amount of polymer and a few steel ball bearings in a pressure vessel with windows, closing the vessel, installing the vessel in a pressure system, condensing gas (such as carbon dioxide whose critical temperature is $31°C$) in the pressure vessel containing polymer and in the dosing vessel (Figure 2), heating the pressure system to about the melting point of the polymer, gently rocking the pressure vessel to stir the polymer-gas solution by the gentle rolling of the steel ball bearings, slowly bleeding gas to decrease pressure to cause phase separation, observing the pressure at which the strong scattering of light in the near critical region distorts the laser beam probe and the pressure at which scattering is sufficient to effectively extinguish or drastically distort the beam, changing the temperature, adding gas if necessary, rocking the vessel to stir the solution, and repeating the bleeding process. Pressures are measured using a diaphragm strain gage which is calibrated from time to time with free piston gages. A precise bourdon tube gage provides occasional checks, during solubility experiments on pressures recorded by the strain gage transducer.

The weight of gas in solution with the polymer can be estimated using a real gas equation and the unoccupied volume of the pressure vessel. A
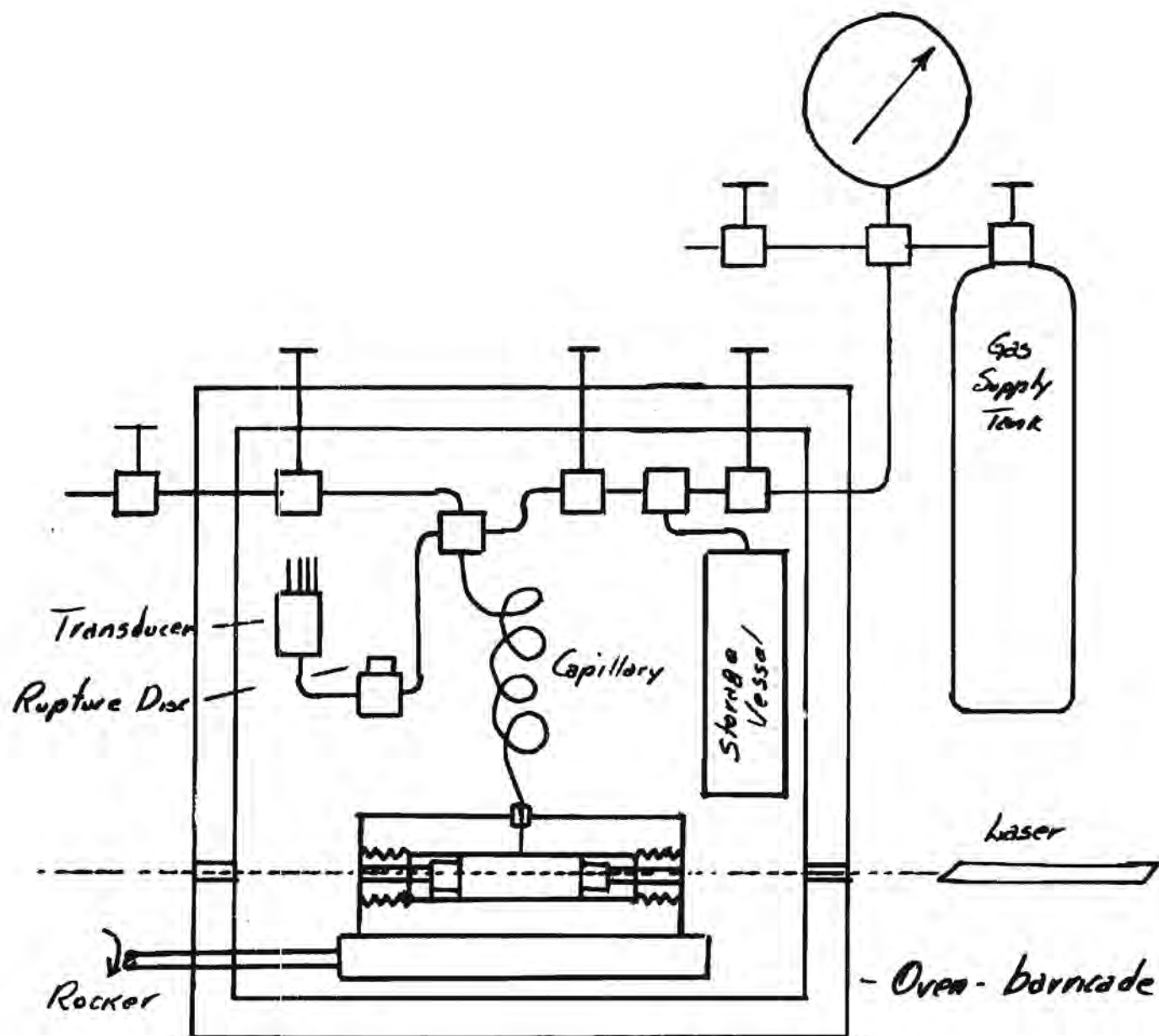
Figure 2   Schematic Diagram of Polymer in Gas Solubility
Apparatus

capillary (0.007 in , 0.178 mm ID) connects the pressure vessel to the rest
of the system. If care is exercised in bleeding the system slowly, little
or no polymer is extruded from the pressure vessel into other parts of the
pressure system. Thus, the capillary serves as a connector to the vessel
and as a barrier to prevent polymer migration. The volume of gas in solution
is the volume of the pressure vessel minus the volume of polymer and the
volume of the steel balls. Therefore the weight of gas, in solution is
given by:

$$W_{gas} = \frac{M \cdot P (V_{vessel} - V_{polymer} - V_{bearings})}{z \cdot R \cdot T} \qquad (1)$$

where

M = molecular weight of gas

p = pressure

V_{vessel} = measured volume of vessel

$V_{polymer}$ = volume of polymer

$V_{bearings}$= volume of steel ball bearings

z = compressibility factor of gas

R = gas contact

T = absolute temperature

Volume of polymer is taken as the crystal volume of the polymer; the weight
of polymer is divided by an average of the better measurements of crystal
density from the literature(1). Steel balls of volume 0.248_3^{cc} were used.
Compressibility values for gases are estimated from the good measurements
of p-V-T behavior published(2). Weight fractions of polymer, $X_{polymer}$, are
calculated using the weights of gases from equation 1 and the known weights
of polymer added to the pressure vessel, thus

$$X_{polymer} = \frac{W_{polymer}}{W_{gas} + W_{polymer}} \qquad (2)$$

where $W_{polymer}$ = weight of polymer

W_{gas} = weight of gas from equation 1.

Densities of polymer solutions, d_{soln}, are calculated by dividing the sums of weights of polymer and gas by the volume of the pressure vessel minus the volumes of the steel ball bearings

$$d_{soln} = \frac{W_{polymer} + W_{gas}}{V_{vessel} - V_{bearings}} \tag{3}$$

For approximate measurements of solubilities, the simple method used probably is adequate. Careful measurement of gas added to the system is difficult at best. In any case, estimates of gas concentrations are dependent on p-V-T data. In the p-T region of these studies z is a strong function of p and T ranging from about 0.88 to 1.25; solubility estimates depend quite strongly on the compressibility factor assumed. In the analysis using equation 1, the contribution of polymer to the observed pressure is assumed to be negligible; the effect of polymer on the compressibility factor of the gas is ignored, and the volume occupied by polymer molecules is taken as the crystal volume of the polymer. Of these assumptions, the first probably is the best; the partial pressure of polymer should be very small compared to that of the gas. The third assumption probably is incorrect but not by more than a few percent. Based upon our knowledge of volumes of small molecules estimated from crystal structures and from gas viscosities or the b in the V-b term in van der Waal's equation, crystal volumes overestimate molecular volumes in gases (3). However, $V_{polymer}$ in equation 1 is proportional to the partial volume of the gaseous polymer in solution; this should be greater than the molecular volume even

for a component with very small partial pressure. The effect of the polymer molecules upon the compressibility of the gas is not known; we expect that the effect should be to decrease z. Large molecules can dissipate collision energies in a multitude of vibrational, bending, and twisting modes. This coupled with the relatively large polarizability of the polymer molecules and molecular segments giving rise to greater attractive forces should cause compressibilities of the mixtures and the gaseous component to be lower than the compressibilities of pure gas. For the foregoing reasons, estimates of solubilities made in these studies should be taken as approximate estimates, not refined measurements.

b. Results

Polybutene-1 is dissolved quite easily in carbon dioxide at moderate pressures and temperatures; namely 0.063 to 0.38 weight fraction polybutene-1 is dissolved in CO_2 in the 300 to 900 atm and the 130° to 150°C pressure-temperature region. Pressure decreases with increasing temperature at constant concentration; consequently, solubility increases with increasing temperature at constant pressure (Figure 3, Table III). Pressure increases with increasing concentration of polymer at constant temperature. These dependencies of solubility and pressure on temperature and of pressure on concentration are the opposite of those reported for the polybutene-1 in n-butane system (4) Table III. The critical miscibility regions over which strong scattering of light is observed are rather broad, from 10 to ~40 atm changes in pressure, for the polybutene-1-carbon dioxide system (Figure 3).

Polypropylene dissolves in carbon dioxide from 0.067 to 0.38 weight fraction polypropylene in the 450 to 950 atm and 163° to 208°C pressure-

TABLE III

	Melting Range of Polymer	Critical Point Properties of Gas			Weight Fraction Polymer	Solution Range			Changes with p,T & Concentration		
		Temp. °C	Pressure atm	Density g/cc		Pressure atm	Temp. °C	Density g/cc	p with T	solubility with T	p with conc.
Polybutene-1-nbutane	~126°	152°	37.5	.228	.055-.21	120-170	167°-190°	.44-.53	+	−	−
Polybutene-1 − CO_2	~126°	31°	72.9	.468	.063-.38	300-900	131°-150°	.53-.91	−	+	+
Polypropylene − CO_2	162°-176°	31°	72.9	.468	.067-.38	450-950	163°-208°	.60-.87	−	+	+
Nylon 6 − CO_2	212°-225°	31°	72.9	.468	.138-.165	400-510	233°-241°	.49-.59	−	+	?
Polybutene-1-nbutane	~126°	152°	37.5	.228	~.20	135-150	171°-182°		+	−	?
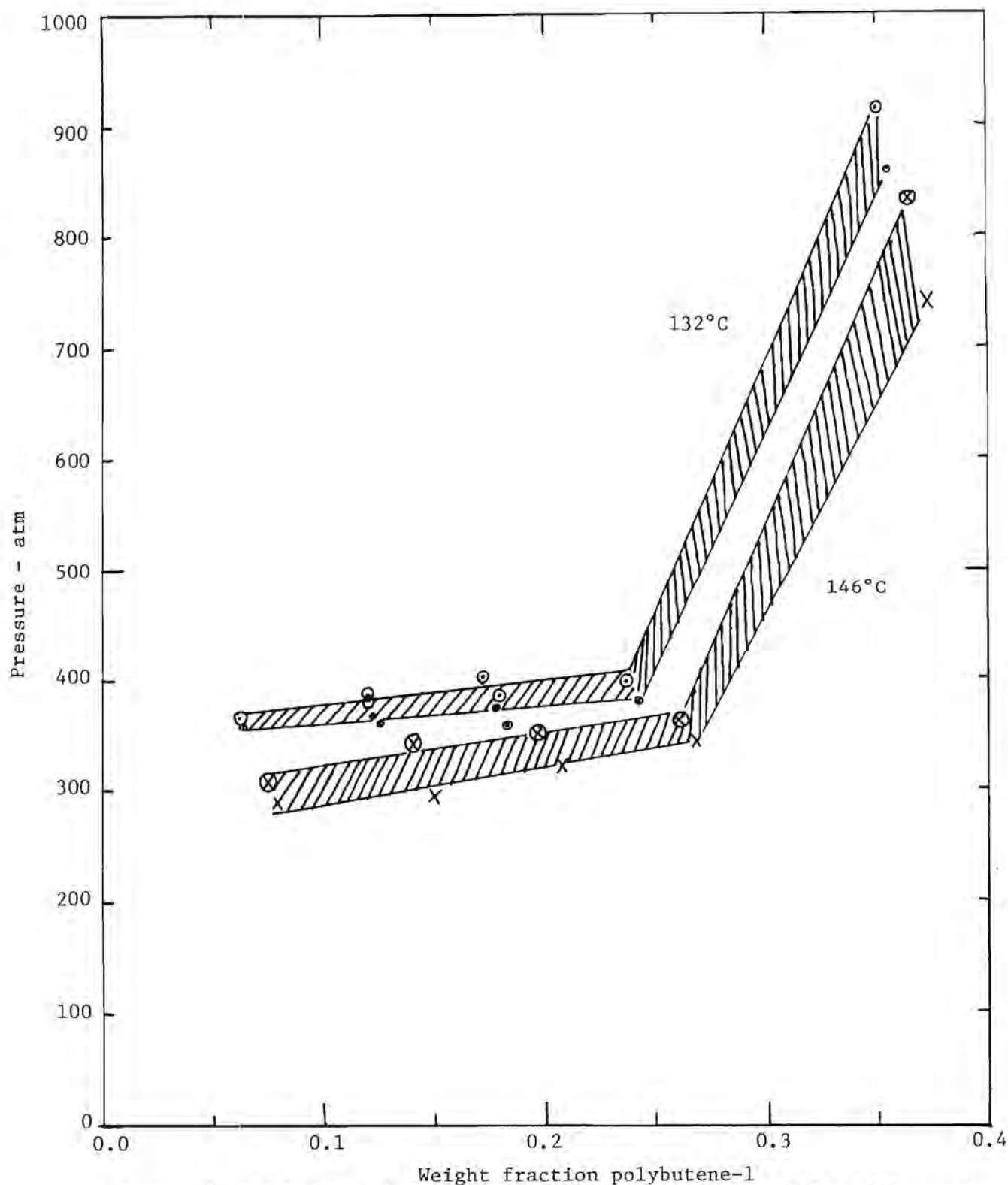
- 13 -
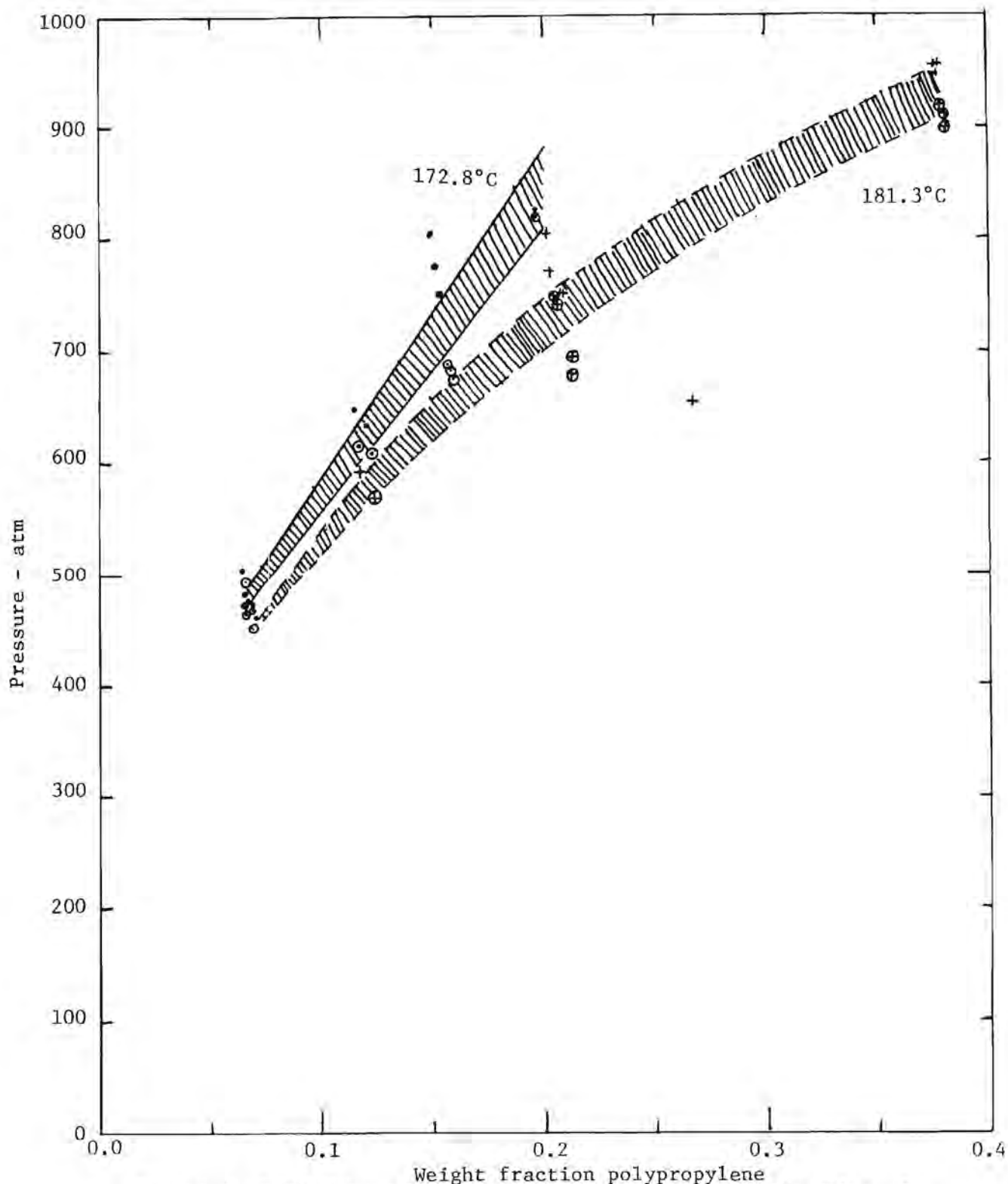
Figure 3   Solubility of Polybutene-1 in Carbon Dioxide: Plot of Pressure
Versus Weight Fraction Polybutene at 132° and 146°C. (Upper
edges of shaded areas indicates pressures at onset of strong
scattering of light characteristic of critical miscibility
regions.   Lower edges are at pressures at which the laser beam
probe was severely distorted or extinguished.)

temperature region (Table IV). As is the case for polybutene-1 dissolved in carbon dioxide, the pressure of carbon dioxide in solution with polypropylene decreases with increasing temperature at constant concentration; therefore, the solubility of polypropylene in carbon dioxide increases with increasing temperature at constant pressure (Figure 4, Table III). Pressures increase with increasing concentration of polymer. These dependences of solubility and pressure on temperature and of pressure on concentration are similar to those for the polybutene-1-carbon dioxide system and opposite to those reported for polybutene-1 in n-butane solutions (4) (Table III). The critical misability regions over which strong scattering of light is observed are about a 10 or 20 atm pressure range at $X_{polymer}$ = .067 increasing to about 70 atm pressure range at $X_{polymer}$ $\sim$ .2 to .38 (Figure 4).

Pressure-temperature dependence of the lower miscibility pressures for the system carbon dioxide - polypropylene at six different, approximately constant concentrations are given in Figure 5. These are raw data; no inter- polations for concentration difference have been applied. The $X_{polymer}$ $\sim$.07 data are from 0.068 to .071 weight fraction polypropylene from two different runs made separated in time by 15 days and two solubility runs at other concen- trations. Data are consistent from the two runs. The data at $X_{polymer}$ $\sim$ 0.13 (actually from 0.11 to 0.15 weight fraction polypropylene) are from two runs made 38 days and 6 solubility runs apart. Data are quite consistent. The $X_{polymer}$ $\sim$ 0.17 (0.16 to 0.18 weight fraction polypropylene) are from a single run. The $X_{polymer}$ $\sim$ 0.21 (0.20 to 0.22 weight fraction polypropylene) are from two runs made six days apart; fair reproducibility is evident. Measure- ment made at $X_{polymer}$ $\sim$ 0.27 are not consistent with the other data for the system. Data at $X_{polymer}$ $\sim$ 0.38 are from a single run over a narrow temperature range, 181.8° to 183.8°C. In all cases pressure decreases with increasing

TABLE IV

Approximate Energies & Entropies of Dilution

System	Weight Fraction Polymer	Pressure atm	Temperature °C	Partial molal energy-$\Delta\bar{E}$, kcal/mol	Partial molal entropy-$\Delta\bar{S}$, $\frac{cal}{mol-°K}$
n-Butane-polypropylene	.05 -.20	130-190	$166°$-$186°$	$-4._0$	$-18._6$
CO_2 - polypropylene	.067-.20	450-870	$172°$-$181°$	$+3._0 \longrightarrow +6._2{}^{*}$ $+2._6 \longrightarrow +7._6{}^{+}$	$-5._5 \longrightarrow .4_2{}^{*}$ $-6._5 \longrightarrow 3._6{}^{+}$
CO_2 - polybutene -1	.06 - .26	280-410	$132°$-$146°$	$+4._5 \longrightarrow +2._6{}^{*}$ $+6._1 \longrightarrow +3._2{}^{+}$	$-.6_7 \longrightarrow -5._4{}^{*}$ $3._5 \longrightarrow -4._0{}^{+}$
CO_2-nylon-6	$.13_8$-$.16_5$	400-510	$233°$-$241°$	$+5._0{}^{*}$ $+8._5{}^{+}$	$-2._3{}^{*}$ $+4._7{}^{+}$

*From higher pressures where onset of critical scattering is observed.

+From lower pressures where extreme scattering or extinction of laser beam is observed.

Figure 4   Solubility of Polypropylene in Carbon Dioxide: Plot of
            Pressure Versus Weight Fraction Polypropylene at 172.8°
            and 181.3°C. (Upper edges of shaded areas indicates pres-
            sures at onset of strong scattering of light characteristic
            of critical miscibility regions.   Lower edges are at pressures
            at which the laser beam probe was severely distorted or
            extinguished.)
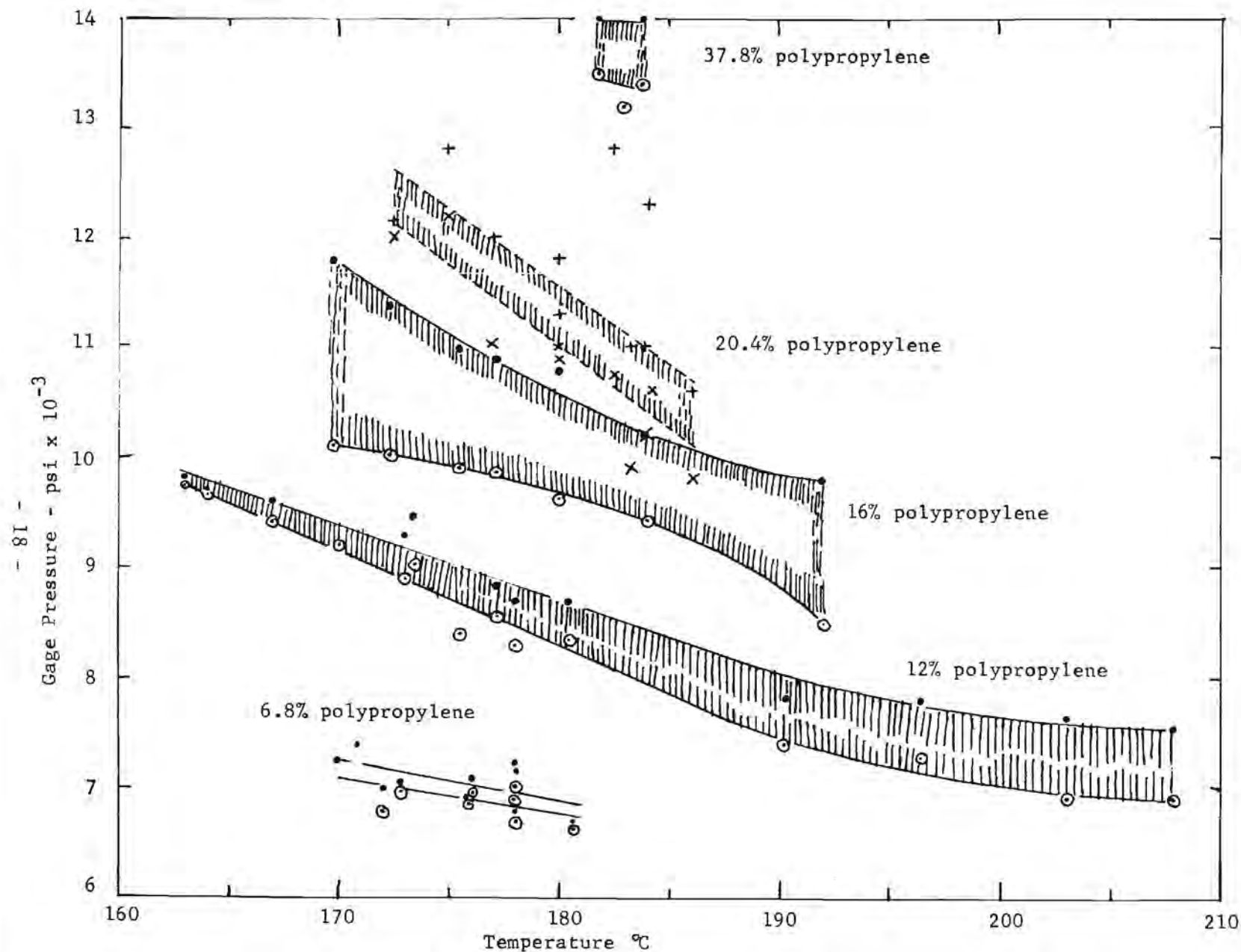
Figure 5. Approximate Pressure - Temperature Dependence at
Nearly Constant Compositions of Polypropylene
Dissolved in Carbon Dioxide

temperature in spite of slight increases in concentration of polymer with temperature.

One run of four observations of the solubility of nylon-6 in carbon dioxide at 233° to 241°C indicates that nylon is readily soluble in carbon dioxide at about 400 to 510 atm pressure, and $X_{polymer} \sim 0.138$ to 0.165. The solubility of nylon increases with temperature at constant pressure as shown by the decrease in pressure with increasing temperature at approximately constant composition. Considering the relatively high softening temperature of nylon-6 ($\sim 212^{\circ}$ to 225°C) and the relatively low critical temperature of carbon dioxide ($T_c = 31^{\circ}$C), solubility at reasonable concentrations at this relatively low pressure is unexpected and gratifying. These data indicate that carbon dioxide dissolves nylon more readily than polypropylene or poly-butene-1. If this be borne out by further experiment, carbon dioxide with no dipole but strong quadrapoles (strong bond dipoles) may be a good solvent for relatively polar, hydrogen bonded and, perhaps, other dipole bonded polymers.

Gentle stirring is necessary to dissolve polypropylene in carbon dioxide. Gentle rocking of the pressure vessel through 120° about the cylindrical axis at about 0.5 cycles per second for two minutes is quite sufficient to produce what appears to be a homogeneous solution as judged from the laser beam's passing through the pressure cell without distortion. If the gentle stirring is omitted, the laser is distorted at gas pressures well above the critical miscibility region.

Some minimum densities of the gaseous solutions may be necessary to dissolve polymers in the gas phase. Plots of densities versus weight fraction polymer are given in Figure 6. Polypropylene in butane solutions have densities from 0.44 to 0.53 g/cc in the concentration range of $X_{polymer} = .055$ to 0.21. These densities are about twice the critical density of n-butane, 0.216 g/cc (5). The densities of solutions of polybutene-1 in carbon dioxide are

Density – g/cc

1.0

0.9 •⦿ 132.0°
     +⊕⊕ 146.0°
     ⊕

CO$_2$ – Polypropylene

0.8 ⊗ 172.8°C
     ⊞ 181.3°C
     ※

0.7

                                    CO$_2$ – Polybutene – 1

0.6

                    167.5°C
                  + 176.5°C
0.5   × 186.4°C

              Butane – polypropylene

0.4

0.3

0.2

0.1

0.0
  0.0   0.1   0.2   0.3   0.4

Weight fraction polymer
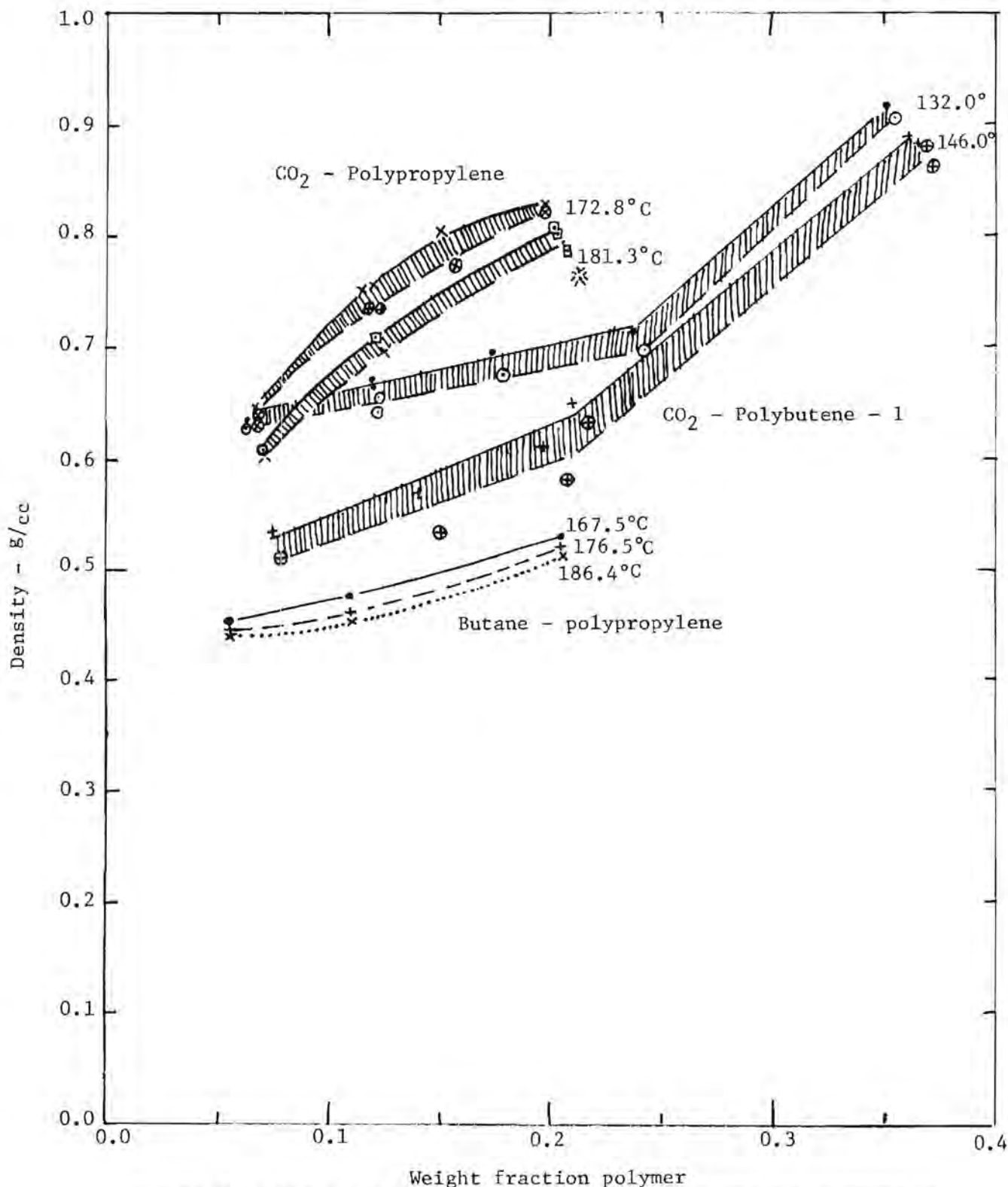
Figure 6   Plots of Densities Versus Weight Fractions of Polymers at
           Various Temperatures for Polypropylene Dissolved in Butane
           and in Carbon Dioxide and for Polybutene-1 in Carbon Dioxide.
           (Upper edges of shaded areas indicate densities at pressures
           at onset of strong scattering of light characteristic of critical
           miscibility and gas–liquid critical regions. Lower edges are
           for densities at pressures at which the laser beam probe was
           severely distorted or extinguished.)

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from 0.53 to 0.91 g/cc in the 0.063 to 0.37 weight fraction polybutene-1 range (Figure 6). The densities of polypropylene in carbon dioxide solutions range from 0.62 to 0.87 g/cc in the 0.069 to 0.38 weight fraction interval (Figure 6). The densities of the nylon-6 in carbon dioxide solution studied at four temperatures and pressures are from 0.49 to 0.59 g/cc in the 0.13_8 to 0.16_5 weight fraction polymer range. These densities are greater than but less than twice the critical density of carbon dioxide, 0.468 g/cc (6). Densities of the three solutions studied over sufficiently wide ranges of concentration, polybutene-1 in carbon dioxide and in n-butane and polypropylene in carbon dioxide, increase with increasing polymer concentrations as must be expected. Densities of polypropylene in CO_2, polybutene-1 in CO_2, and nylon in CO_2 decrease with increasing temperature. This is consistent with the solubilities of these polymers in CO_2 increasing with temperature at constant concentrations (Figures 3 and 4, Table III). Densities of solutions of polypropylene in n-butane increase with increasing temperature (Figure 6) in keeping with the decrease in solubility of polypropylene in n-butane with increasing temperature and the increase in pressure with temperature at constant concentration (4). The critical pressure and density of carbon dioxide are higher than those of most gases, by about a factor of two. Thus, to find polymers soluble in carbon dioxide in the density range of 1.05 times to twice the critical density of carbon dioxide at temperatures substantially above the critical temperature of the gas (from 1.332 T_c to 1.684 T_c) is quite consistent with polypropylene dissolving in n-butane at densities about twice to 2.5 times the critical density of n-butane at temperatures not far above the critical temperature of the gas (from 1.028 T_c to 1.075 T_c).

3. Approximate Thermodynamics of Mixing

To a very good approximation, the solubility apparatus used is a constant volume system. Therefore, thermodynamic activity or fugacity of the gaseous solvent should be defined in terms of the partial molal free energy at constant volume, the Helmholtz free energy. Thus, for activity a_1 and fugacity f_1 of gas

$$\Delta \bar{A}_1 = \Delta \bar{E}_1 - T\Delta \bar{S}_1 = -RT \ln a_1 = RT \ln f_1/f_1^{\,0} \tag{4}$$

where $\Delta \bar{A}_1 \equiv$ change in partial molal free energy at constant volume

$\Delta \bar{E}_1 \equiv$ change in partial molal energy

$\Delta \bar{S}_1 \equiv$ change in partial molal entropy

$a_1 \equiv$ thermodynamic activity of gas

$f_1 \equiv$ fugacity of gas in solution

$f_1^{\,0} \equiv$ fugacity of pure gas at a specified standard state

The p-V-T behavior of any gas may be represented by an equation of the form [8]

$$V = \frac{RT}{p} - \alpha\,(V,T). \tag{5}$$

Remembering that the pressure dependence of the change in Helmholtz free energy is given by [9]

$$\left(\frac{\delta A}{\delta V}\right)_T = p \tag{6}$$

the fugacity at constant volume may be calculated using the equation (at constant temperature)

$$RTd\ln f = pdV \tag{7}$$

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by substituting the derivative of equation 5 (at constant temperature)

$$dV = - \frac{RT}{p^2} \ dp \ - \ \left[\frac{\delta \alpha (V,T)}{\delta V} \right]_T dV \qquad (8)$$

in equation 7 and integrating. Thus for the fugacity

$$RT \int_{V*}^{V} d\ln f = RT \int_{p*}^{p} \frac{dp}{p} \ - \ \int_{V*}^{V} \left[\frac{\delta \alpha V,T}{\delta V} \right]_T dV \qquad (9)$$

where V* is the volume as p approaches zero; then

$$RT \ \ln f \ - \ RT \ \ln f* \ = \ RT \ \ln p \ - \ RT \ \ln p* \ - \ \int_{V*}^{V} \left[\frac{\delta \alpha (V,T)}{\delta V} \right]_T dV. \qquad (10)$$

In the limit of infinite volume or zero pressure, all gases behave ideally so that the second term of equation 5, (V,T), approaches zero and the fugacity approaches the pressure. Thus the fugacity may be calculated from

$$RT \ \ln f \ = \ RT \ \ln p \ - \ \int_{0}^{p} \left[\frac{\delta \alpha (V,T)}{\delta V} \right]_T dV \qquad (11)$$

As a standard state, the hypothetical ideal gas state at one atmosphere, which differs insignificantly from the real gas at one atmosphere for the gases used in these studies, is convenient.

In order to estimate the partial molal energies of solution of gas in the solutions, the temperature dependence of the partial molal Helmholtz free energy is calculated using the equation

$$\left(\frac{\delta A}{\delta T} \right)_V = - \ S \qquad (12)$$

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which leads to

$$\left[\frac{\delta\,(\Delta\bar{A}_1/T)}{\delta(1/T)}\right] = \Delta\bar{E}_1 = R\left[\frac{\delta\,\ln f_1}{\delta(1/T)}\right]_V = R\left[\frac{\delta\,\ln p_1}{\delta(\frac{1}{T})}\right]_V - \int_{p=o}^{p}\frac{\alpha\,(V,T)}{dV}\,dV - \frac{1}{T}\frac{\delta}{\delta\left(\frac{1}{T}\right)}\int_{p=o}^{p}\left[\frac{\delta\alpha(V,T)}{\delta\,(V)}\right]_T dV \quad (13)$$

The partial molal entropy is given by

$$\Delta\bar{S}_1 = \frac{\Delta\bar{E}_1}{T} - \frac{\Delta\bar{A}_1}{T} = \frac{R}{T}\left[\frac{d\,\ln\,p_1}{d(1/T)}\right]_V - \frac{1}{T}\frac{\delta}{\delta(1/T)}\int_{p=o}^{p}\left[\frac{\delta\,\alpha\,(V_1T)}{\delta V}\right]_T dV - R\,\ln p_1. \quad (14)$$

The partial molal energies of solution of gas in solution (often called partial molal energies of dilution) were estimated by interpolating pressures and compositions at constant temperatures for specified concentrations, $X_{polymer} = .06, .10$, etc, and then determining the dependence upon reciprocal temperature of $\ln p$. Linear least squares fits were used for interpolation and determining slopes of $\ln p$ with $1/T$. Ignoring the nonideality terms, the $\alpha(V,T)$ terms, in equations 13 and 14 probably is not too serious because these do not change much with temperature and pressure over the small ranges of temperature and pressure used in these studies.

The smoothed pressures of the onset of obvious critical opalesesence and of extreme scattering together with partial molal energies and entropies of dilution (adding gas) are shown as functions of weight fraction polymer for gaseous solutions of polybutene-1 and polypropylene in carbon dioxide in Figures 7 and 8. In both cases partial molal energies of dilution are positive which is to say solubilities increase with increasing pressure. Partial molal energies and entropies of dilution decrease with increasing concentration of polybutene-1 (Figure 7); these increase with increasing concentration of polypropylene (Figure 8). Since entropy and energy vary so
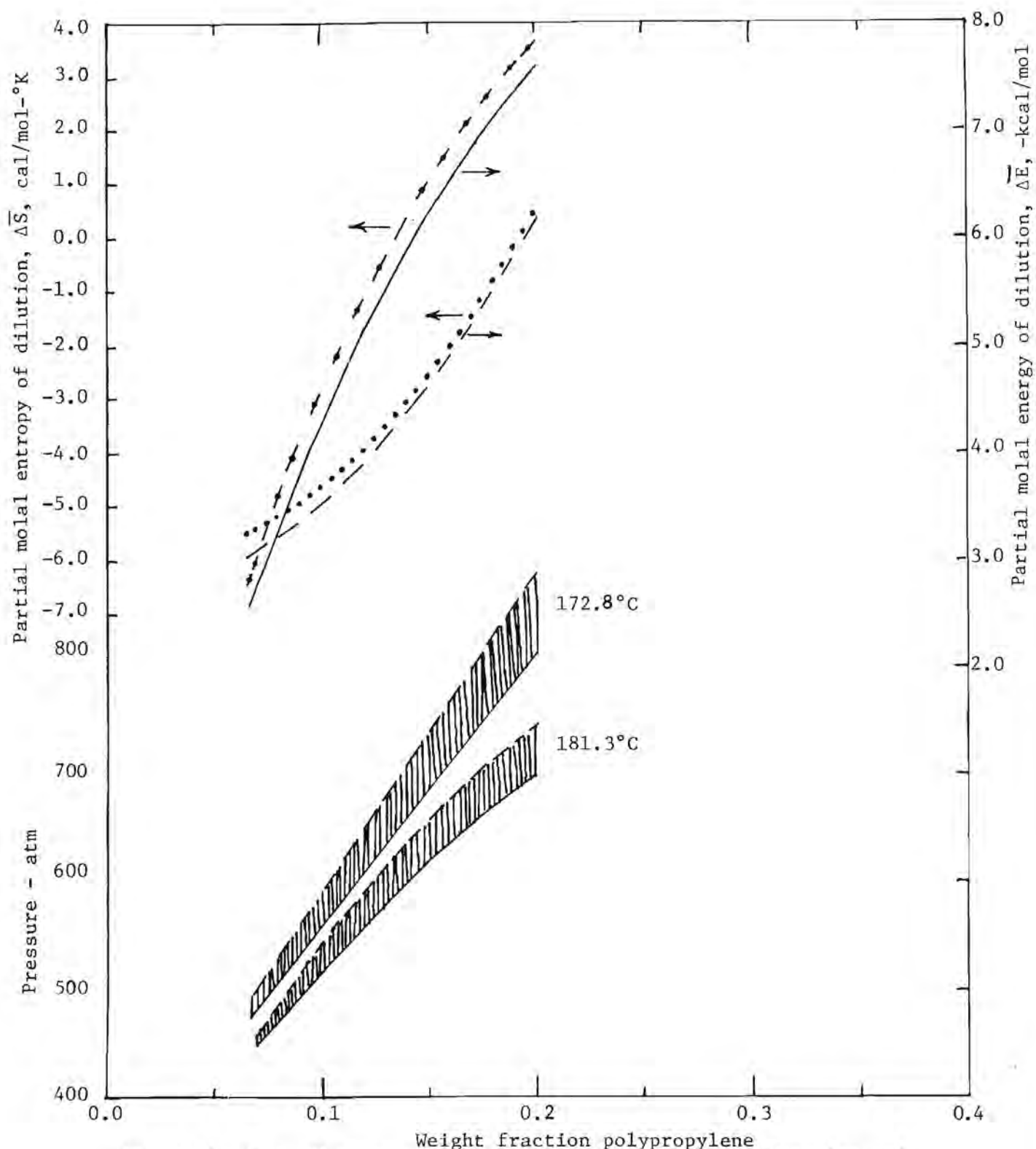
- 24 -
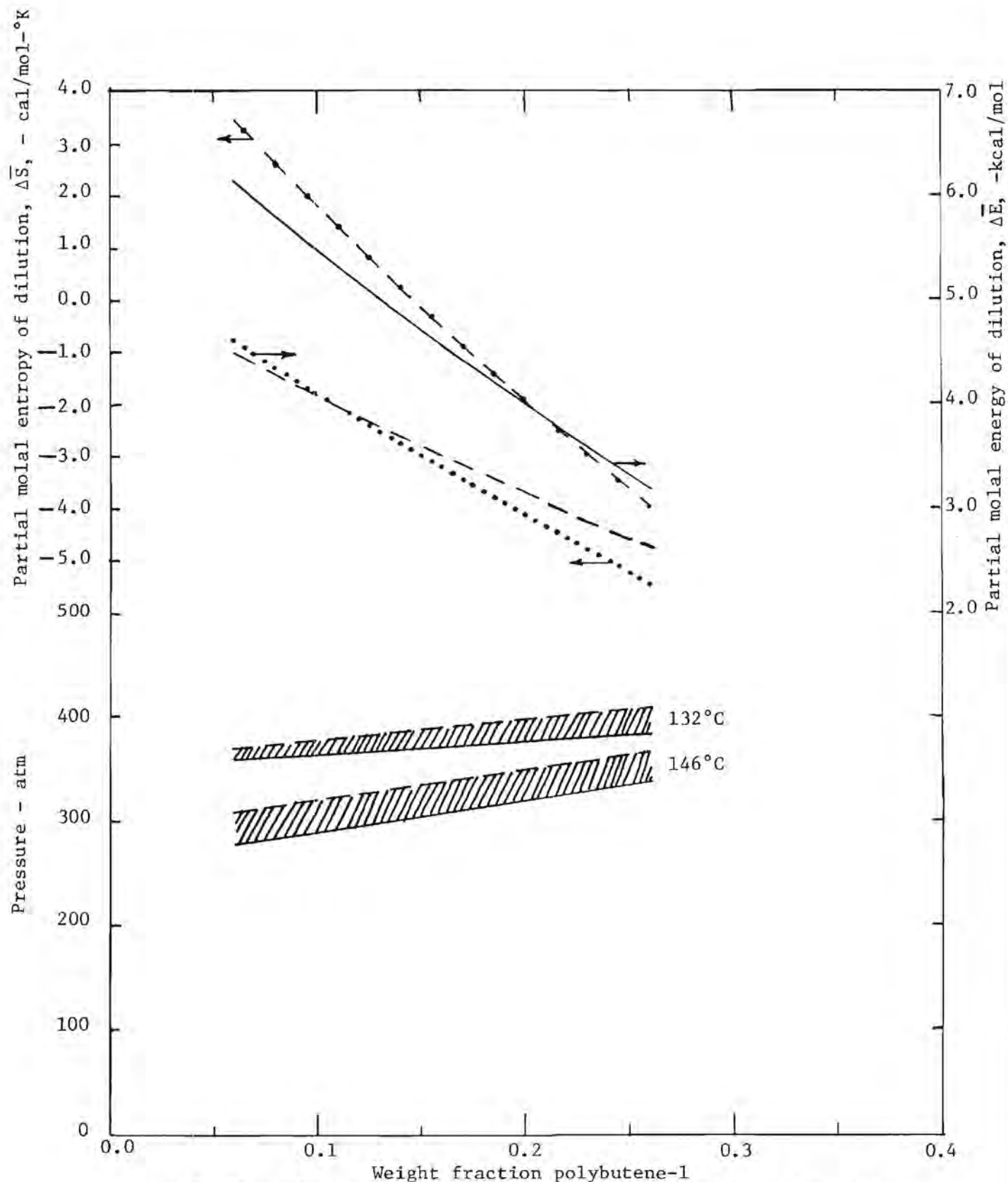
Figure 7   Plot of Approximate Changes in Partial Molal Energies and
Entropies of Dilution (Addition of Carbon Dioxide) and
Pressure Versus Weight Fraction Polybutene-1 for Solutions
of Polybutene-1 Carbon Dioxide. (Dashed and dotted lines
refer to pressures at onset of strong scattering of light
characteristic of critical miscibility and gas-liquid
critical regions. Solid lines and —•— —•— line refer to pres-
sures at which the laser beam probe was severely distorted
or extinguished.)

Figure 8   Plot of Approximate Changes in Partial Molal Energies and
Entropies of Dilution (Addition of Carbon Dioxide) and
Pressure Versus Weight Fraction Polypropylene for Solutions
of Polypropylene in Carbon Dioxide. (Dashed and dotted lines
refer to pressures at onset of strong scattering of light
characteristic of critical miscibility and gas-liquid critical
regions.   Solid lines and ─•─ ─•─ line refer to pressures at
which the laser beam probe was severely distorted or extinguished.)

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as to minimize the departure of the free energy from zero, the partial molal entropies of dilution behave as do the energies of dilution. The partial molal energies of dilution, $\Delta\bar{E}_1 \sim 3$ to 6 kcal/mol, and the partial molal entropies of dilution, $\Delta\bar{S}_1 \sim -6$ to $+4$ cal/mol-^{o}K, are quite comparable in magnitude for both systems; they differ only in their concentration dependence (Figures 7 and 8, Table IV). An approximate estimate of the lower bounds on the partial molal energies and entropies of dilution at about 0.15 weight fraction nylon in carbon dioxide can be made from the solubility observations at four temperatures in the single run reported. These are for the upper pressure curve indicating the onset of critical region scattering $\Delta\bar{E}_1 \sim 5$ kcal/mol and $\Delta\bar{S}_1 \sim -2._3$ cal/mol $- ^{o}$K and for the lower pressure curve indicating severe scattering $\Delta\bar{E}_1 \sim 8._5$ kcal/mol and $\Delta\bar{S}_1 \sim + 4._7$ cal/mol $- ^{o}$K (Table IV). When data are available so that interpolation in concentration can be made, these values probably will be revised downward. The partial molal energy of dilution of n-butane in solutions of polypropylene is negative, $\Delta\bar{E}_1 \sim - 4._0$ kcal/mol, and appears to be independent of concentration in the 0.06 to 0.26 weight fraction polypropylene range (Table IV). As is to be expected with a negative partial molal energy of solution, a strongly negative partial molal entropy of dilution, $\Delta\bar{S}_1 \sim - 18._6$ cal/mol $- ^{o}$K, is observed. Since thermodynamic data are estimated from temperature derivatives of smoothed experimental data (interpolated for small differences in concentration), the apparent precision of these data is illusory. The thermodynamic data given are approximate only because of scatter of experimental data, assumptions about polymer volumes and compressibilities of gases in calculating concentrations, and neglecting departures from ideality in estimating temperature derivitives of the logarithms of fugacities.

B. Extrusion of Polymers from Gaseous Solutions

   1. Preliminary trials

   Polypropylene in n-butane solution was extruded into the atmosphere.
Since the polymer and carbon dioxide solution passed through about two
feet of 0.007 inch I.D. capillary and thence through 0.087 inch I.D. pres-
sure tubing, one tee, and two valves (Figure 2), the location and manner
of precipitation of polymer could not be determined. Very fine fibers
of polymer were produced in this extrusion to atmospheric pressure. Fiber
diameters were of the order of a few microns in diameter judging from
settling rates in air. So little of this material was produced that char-
acterization was impossible.

   Nylon-6 in carbon dioxide was extruded from the solubility apparatus
(Figure 2). Pressure in the system was about 950 atm ($\sim$ 14000 psi) at
$230^{\circ}C$; initial nylon-6 concentration was about 0.2 weight fraction polymer.
Fibrous nylon containing what appear to be small solidified droplets was
formed as shown in Figure 9, a scanning electron micrograph at 200X
magnification. Fiber diameters range from about 2 to 10 microns with some
larger fibers up to 25 microns or larger in diameter also present (Figure
9 and 10).

   The pressure in the system was sufficient that the nylon probably
was dissolved in the carbon dioxide. No evidence of phase separation was
observed before throttling the solution to the atmosphere. Flows in the
system were subsonic. The pressure difference between the solution in
the pressure vessel and the atmosphere was many times that necessary to
produce flow in a properly designed nozzle. Therefore, choking flow must
have occurred, probably in the capillary leading from the pressure vessel.
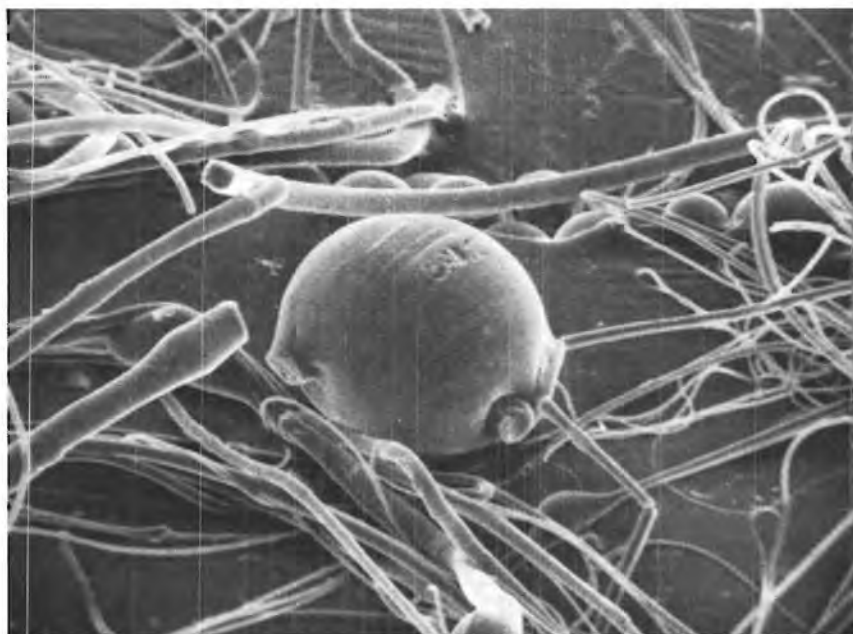
Figure 9   Fibrous Nylon Produced by Extruding Nylon from
           Gaseous Solution in Carbon Dioxide at Approximately
           950 Atmospheres and 230°C. Magnification: 200 X.
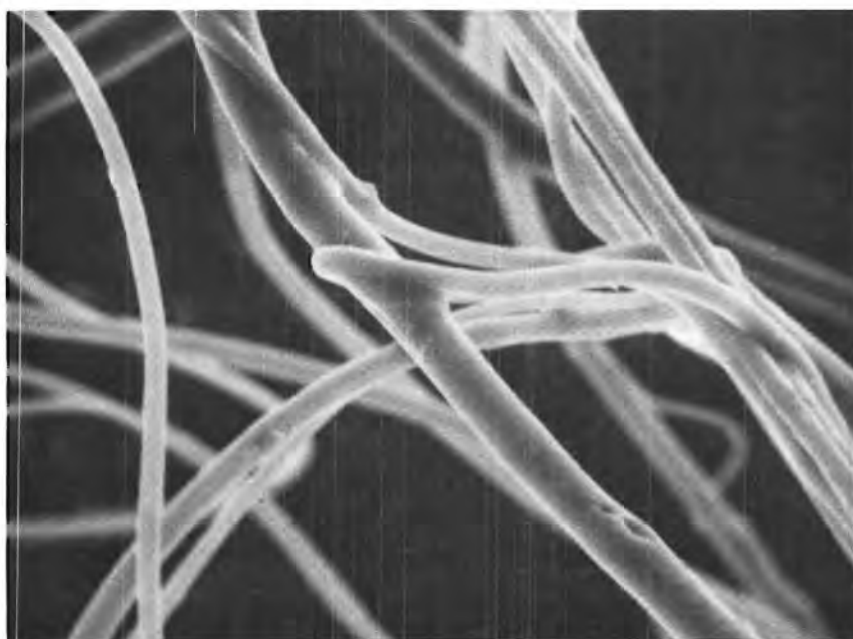
Figure 10 Fibrous Nylon Produced by Extruding Nylon from
           Gaseous Solution in Carbon Dioxide at Approximately
           950 Atmospheres and 230°C. Magnification: 1000 X.

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Pressure differences were great enough that flow velocities approached a substantial fraction of Mach one.

Preliminary trials show that fibrous polymers can be produced by extruding gaseous solutions of polymers into the atmosphere.

2. Current and future work

A barricade and heated pressure vessel for extrusion of polymers from gaseous solutions is almost complete. Testing of this system should begin shortly. The first system to be investigated will be nylon 6 dissolved in carbon dioxide. The system has been designed so that the smallest constriction limiting flow will be the orifice through which the solution will be extruded into the atmosphere. Only with such a system can effects of flow in the capillary and through other constrictions be minimized.

C. Foam Fibrillation

1.0 Introduction

Extrusion of foam followed by biaxial orientation represents a greatly simplified approach to forming webs and yarns compared to conventional textile formation processes. The objective of this research is to evaluate and enhance the quality of the webs and yarns obtained by foam fibrillation with respect to garment applications.

In previous reports a number of deficiencies in the webs and yarns had been identified. The as-extruded webs have low basis weights, low tensile strengths, less strength in the transverse direction and relatively large, non-uniform pores. The as-extruded yarns have high filament and yarn deniers and low tenacities. In addition, web and yarn formation has been limited almost exclusively to polypropylene. In response to these deficiencies, during the period since the last report (10) the primary tasks have been: 1) implementing process design changes to improve web and yarn structure, 2) optimizing feed composition and operating conditions for polypropylene, 3) processing nylons and polyesters, 4) improving yarn properties and 5) designing a continuous drawing system. The progress achieved in these areas is reported in the ensuing sections. Yarn formation has received the most attention since this structure appears to have the greatest probability for successful commercial utilization.

2.0 Design Changes

A number of design changes were discussed in the last report which had not been fully implemented or evaluated. The status of these modifications is discussed below.

2.1 Liquid blowing agent feed system

The purpose of this system is to reduce the size of the pores in webs.

Since the size of the pores is reduced as the particle size of solid chemical blowing agents is reduced, then the ultimate in small pore size should be obtained by using a liquid blowing agent, provided adequate mixing can be achieved. In order to use liquid blowing agents a low flow rate, constant volume pump is necessary.

A liquid resevoir with a constant pressure head and a long capillary tube outlet was constructed to provide the necessary flow rates. However, consistent flow rates were not obtained. Blending the liquid blowing agent with the polymer prior to extruding the mixture resulted in poor and insufficient mixing. Consequently, further work on this system was terminated.

2.2 Gas Feed System

A gas feed system was attached to the vent port of the extruder in order to evaluate the use of nitrogen and carbon dioxide gases as physical blowing agents. By using these gases greater freedom in processing nylons and polyesters should be obtained.

Even though the gas flow rate could be carefully controlled by regulating the gas pressure at the point of injection (the extruder's vent port), the webs produced were non-uniform. Some improvement in web uniformity was obtained by incorporating the static mixer between the extruder and the die. The static mixer provides enough residence time for diffusion of the gas into the polymer. Since the webs tended to alternate between filmy and fibrous regions with time, consistent axial mixing was not achieved. The use of a sparging ring in place of the vent port was investigated as a means of improving axial mixing, but the extruder could not be modified in this manner. No further improvements in the gas feed system have been made.

2.3 Die pressure sensor

A die pressure sensor was incorporated to improve manual process control.

Unfortunately, when the rotating die is used, a constant die gap cannot be maintained. Consequently it is difficult to reproduce die pressure readings under apparently identical processing conditions. It is apparent that the quality of the web is sensitive to die pressure as high die pressures tend to produce filmy webs.

2.4 Rotating core die

A rotating die was installed in order to increase transverse direction orientation in the webs. At low rotational speeds no increase in transverse direction orientation is evident. However, at these low rotational speeds the uniformity of the webs is improved. At high speeds the webs produced remain twisted.

Further study of the rotating die will be performed with regard to yarn formation. In this case twisting can facilitate the ensuing drawing steps.

2.5 Downward extrusion

The take-up system was modified to permit downward extrusion in order to facilitate start-up. With this modification it is possible to extrude nylon and polyester without coating the die face with a release agent. With poly (ethylene terephthalate) (PET) it may still be necessary to coat the mandrel with a release agent.

2.6 Cryovac system

The pilot extrusion system developed by the Cryovac Division of W. R. Grace for forming webs has been installed and operated. Several webs exhibiting a high degree of uniformity but relatively large pores have been produced from mixtures of nylon and polyethylene. In addition, polypropylene webs were formed from Hercules Profax 6323 in order to compare the product formed by the Cryovac and Georgia Tech systems. Webs of similar quality were

produced. Since a significant improvement in web quality was not obtained in using the Cryovac system, a quantitative evaluation of web properties was not pursued.

2.7 Auxiliary equipment

A dryer and grinder from Polymer Machinery Corp. have been purchased and installed. This equipment facilitates processing of nylons and polyesters. Drying is necessary to minimize polymer degradation during extrusion. The nylon and polyester pellets are ground into powder form to improve dry blending with the solid blowing agents prior to extrusion.

3.0 Web Formation

3.1 Polypropylene

Since the last report (10) the primary task in studying polypropylene web formation has been optimizing feed composition and processing conditions. Since the quality of the webs obtained with the static mixer is inferior to those obtained without the mixer when chemical blowing agents are used, the mixer was removed and a small heater was placed between the extruder and the die to eliminate temperature variations at the neck.

The conditions for processing polypropylene into webs by foam fibrillation were optimized by assuming that all parameters are independent and do not affect the optimum values of other parameters. The criteria used for determining the optimum values were the appearance and the uniformity of the webs rather than their mechanical properties. This basis was chosen because filmy webs show artificially high tensile properties and artificially low permeabilities. Also, any improvement in mechanical properties is meaningless unless it is an improvement over webs with the same degree of uniformity and filminess. Unfortunately a scale for measuring filminess and uniformity is not available, and the optimization procedure was purely subjective.

The temperature profile was optimized by testing webs extruded using different temperature profiles while keeping all other conditions fixed as shown in Table 5. The amount of Kempore 200MC was fixed at 0.5% since larger percentages cause excessive overblowing and preblowing. The amount of silica flour, Min-u-sil 5, was fixed at 1.0%, which was shown to be the upper limit for silica flour content in the last report (10). As shown in Table 5, temperatures lower than 230°C at the second and third zones of the extruder yield filmy extrudates whose degree of filminess decreases as the temperatures are raised. Only samples 7 and 8 do not show filmy sections. The temperature profile corresponding to sample 8 was chosen to be optimum inspite of the lower tensile properties and larger permeability due to the slight improvement in uniformity. Temperatures higher than 240°C at the die result in webs with poor strength and no further improvement in uniformity.

Next, the amount of fumed silica was optimized by using the best temperature profile and different concentrations of Cab-o-sil M5. As shown in Table 6, tensile properties are fairly independent of fumed silica content; however, the degree of filminess at constant screw speed decreases as silica content increases. The optimum fumed silica concentration was chosen to be 2% since it is possible to obtain non-filmy webs at screw speeds as high as 100 RPM with fairly good uniformity and without a decrease in mechanical properties. Concentrations higher than 2% do not yield any significant improvement.

Using the optimum conditions, stretching around two different size mandrels was compared to a stepwise biaxial stretching combination consisting of a 2.5" mandrel followed by a 2.5" ring. Results are shown in Table 7. The main advantage of using the 3.75" mandrel or the mandrel-ring combination is that uniformity is improved. Also, the traverse direction tenacity increases slightly, but not enough to justify the rather large increase in permeability.

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TABLE 5

Optimization of Temperature Profile for Polypropylene Extrusion

Sample	1	2	3	4
Materials				
1 PROFAX 6323, %	97.5	97.5	97.5	97.5
2 KEMPORE 200 MC, %	.5	.5	.5	.5
3 CAB-O-SIL M5, %	1.0	1.0	1.0	1.0
4 MIN-U-SIL 5, %	1.0	1.0	1.0	1.0
Processing Conditions				
Screw type	2 stage	2 stage	2 stage	2 stage
Zone 1, °C	200	200	200	200
Zone 2, °C	200	200	200	200
Zone 3, °C	210	210	220	220
Neck, °C	210	210	220	220
Die, °C	230	240	230	240
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	–	–	–	–
Zone 3, psi	350	400	300	300
Die, psi	240	240	240	240
Mandrel diameter, inches	2.5	2.5	2.5	2.5
Quench air, °C	room	room	room	room
Quench air, psig	25	25	25	25
Screw speed, RPM	80	80	80	80
Wind-up speed, FPM	35	35	35	35
Die, RPM	0	0	0	0
Web Properties				
Basis Weight, oz/sq. yd.	0.661	0.945	0.756	0.774
MD Tenacity, (lb/in)/(oz/sq. yd.)	3.19	2.83	2.02	3.23
TD Tenacity, (lb/in/(oz./sq.yd.)	0.96	0.25	0.95	0.58
MD Elongation, %	46.0	74.0	56.0	56.0
TD Elongation, %	44.0	110.0	68.0	81.0
Air Permeability, cu.ft./sq.ft./min	173.3	425.5	376.3	82.10
Structural quality	filmy	filmy	filmy	filmy

TABLE 5 (continued)

Optimization of Temperature Profile for Polypropylene Extrusion

Sample	5	6	7	8
Materials				
1 PROFAX 6323, %	97.5	97.5	97.5	97.5
2 KEMPORE 200 MC, %	.5	.5	.5	.5
3 CAB-O-SIL M5, %	1.0	1.0	1.0	1.0
4 MIN-U-SIL 5, %	1.0	1.0	1.0	1.0
Processing Conditions				
Screw type	2 stage	2 stage	2 stage	2 stage
Zone 1, °C	200	200	200	200
Zone 2, °C	200	200	230	230
Zone 3, °C	230	230	230	230
Neck, °C	230	230	230	230
Die, °C	230	240	230	240
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	–	–	–	–
Zone 3, psi	330	330	350	400
Die, psi	240	240	240	240
Mandrel diameter, inches	2.5	2.5	2.5	2.5
Quench air, °C	room	room	room	room
Quench air, psig	25	25	25	25
Screw speed, RPM	80	80	80	80
Wind-up speed, FPM	35	35	35	35
Die, RPM	0	0	0	0
Web Properties				
Basis Weight, oz/sq. yd.	0.926	0.699	0.708	0.898
MD Tenacity, (lb/in)/(oz/sq. yd.)	3.20	2.78	3.06	2.67
TD Tenacity, (lb/in/(oz./sq.yd.)	0.28	0.73	0.76	0.22
MD Elongation, %	55.0	45.0	46.0	70.0
TD Elongation, %	79.0	59.0	54.0	112.0
Air Permeability, cu.ft./sq.ft./min	406.5	688.0	556.3	1010.0
Structural quality	filmy	filmy	web	web

TABLE 6

Optimization of Fumed Silica Content for Polypropylene Extrusion

Sample	1	2	3	4
Materials				
1 PROFAX 6323, %	98.0	98.0	97.5	97.5
2 KEMPORE 200 MC, %	.5	.5	.5	.5
3 CAB-O-SIL M5, %	.5	.5	1.0	1.0
4 MIN-U-SIL 5, %	1.0	1.0	1.0	1.0
Processing Conditions				
Screw type	2 stage	2 stage	2 stage	2 stage
Zone 1, °C	200	200	200	200
Zone 2, °C	230	230	230	230
Zone 3, °C	230	230	230	230
Neck, °C	230	230	230	230
Die, °C	240	240	240	240
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	-	-	-	-
Zone 3, psi	n.m.	n.m.	n.m.	n.m.
Die, psi	n.m.	n.m.	n.m.	n.m.
Mandrel diameter, inches	2.5	2.5	2.5	2.5
Quench air, °C	room	room	room	room
Quench air, psig	25	25	25	25
Screw speed, RPM	60	80	60	80
Wind-up speed, FPM	35	35	35	35
Die, RPM	10	10	10	10
Web Properties				
Basis Weight, oz/sq. yd.	0.50	0.51	0.44	0.51
MD Tenacity, (lb/in)/(oz/sq. yd.)	2.13	2.08	1.91	2.25
TD Tenacity, (lb/in/(oz./sq.yd.)	0.38	0.66	0.32	0.85
MD Elongation, %	74.0	60.0	73.0	70.0
TD Elongation, %	97.0	62.0	77.0	84.0
Air Permeability, cu.ft./sq.ft./min	565.0	509.0	533.0	644.0
Structural quality	web	web	web	web

n.m. = not measured

TABLE 6 (continued)

Optimization of Fumed Silica Content for Polypropylene Extrusion

Sample	5	6	7	8
Materials				
1 PROFAX 6323, %	97.0	97.0	96.5	96.5
2 KEMPORE 200 MC, %	.5	.5	.5	.5
3 CAB-O-SIL M5, %	1.5	1.5	2.0	2.0
4 MIN-U-SIL 5, %	1.0	1.0	1.0	1.0
Processing Conditions				
Screw type	2 stage	2 stage	2 stage	2 stage
Zone 1, °C	200	200	200	200
Zone 2, °C	230	230	230	230
Zone 3, °C	230	230	230	230
Neck, °C	230	230	230	230
Die, °C	240	240	240	240
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	–	–	–	–
Zone 3, psi	n.m.	n.m.	n.m.	n.m.
Die, psi	n.m.	n.m.	n.m.	n.m.
Mandrel diameter, inches	2.5	2.5	2.5	2.5
Quench air, °C	room	room	room	room
Quench air, psig	25	25	25	25
Screw speed, RPM	60	80	60	80
Wind-up speed, FPM	35	35	35	35
Die, RPM	10	10	10	10
Web Properties				
Basis Weight, oz/sq. yd.	0.42	0.56	0.45	0.52
MD Tenacity, (lb/in)/(oz/sq. yd.)	1.82	1.77	1.86	2.25
TD Tenacity, (lb/in/(oz./sq.yd.)	0.28	0.85	0.30	0.85
MD Elongation, %	79.0	89.0	81.0	76.0
TD Elongation, %	86.0	64.0	107.0	80.0
Air Permeability, cu.ft./sq.ft./min	727.0	569.0	560.0	529.0
Structural quality	web	web	web	web

n.m. = not measured

TABLE 7

Optimization of Mandrel for Polypropylene Extrusion

Sample	1	2	3	4
Materials				
1 PROFAX 6323, %	96.5	96.5	96.5	96.5
2 KEMPORE 200 MC, %	.5	.5	.5	.5
3 CAB-O-SIL M5, %	2.0	2.0	2.0	2.0
4 MIN-U-SIL 5, %	1.0	1.0	1.0	1.0
Processing Conditions				
Screw type	2 stage	2 stage	2 stage	2 stage
Zone 1, °C	200	200	200	200
Zone 2, °C	230	230	230	230
Zone 3, °C	230	230	230	230
Neck, °C	230	230	230	230
Die, °C	240	240	240	240
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	–	–	–	–
Zone 3, psi	n.m.	n.m.	n.m.	n.m.
Die, psi	n.m.	n.m.	n.m.	n.m.
Mandrel diameter, inches	2.5	2.5	3.75	3.75
Quench air, °C	room	room	room	room
Quench air, psig	25	25	20	20
Screw speed, RPM	60	80	60	80
Wind-up speed, FPM	35	35	35	35
Die, RPM	10	10	10	10
Web Properties				
Basis Weight, oz/sq. yd.	0.56	0.61	0.34	0.47
MD Tenacity, (lb/in)/(oz/sq. yd.)	2.09	1.78	1.26	2.00
TD Tenacity, (lb/in/(oz./sq.yd.)	0.13	0.31	0.15	0.33
MD Elongation, %	74.0	59.0	95.0	57.0
TD Elongation, %	105.0	94.0	125.0	71.0
Air Permeability, cu.ft./sq.ft./min	899.0	546.0	1526.0	765.0
Structural quality	web	web	web	web

n.m. = not measured

TABLE 7 (continued)

Optimization of Mandrel for Polypropylene Extrusion

Sample	5	6		
Materials				
1 PROFAX 6323, %	96.5	96.5		
2 KEMPORE 200 MC, %	.5	.5		
3 CAB-O-SIL M5, %	2.0	2.0		
4 MIN-U-SIL 5, %	1.0	1.0		
Processing Conditions				
Screw type	2 stage	2 stage		
Zone 1, °C	200	200		
Zone 2, °C	230	230		
Zone 3, °C	230	230		
Neck, °C	230	230		
Die, °C	240	240		
Vent Port feed, yes/no	no	no		
Vent port, psi	–	–		
Zone 3, psi	n.m.	n.m.		
Die, psi	n.m.	n.m.		
Mandrel diameter, inches	2.5" mandrel + 2.5" ring			
Quench air, °C	room	room		
Quench air, psig	20	20		
Screw speed, RPM	60	80		
Wind-up speed, FPM	35	35		
Die, RPM	10	10		
Web Properties				
Basis Weight, oz/sq. yd.	0.48	0.51		
MD Tenacity, (lb/in)/(oz/sq. yd.)	2.00	1.70		
TD Tenacity, (lb/in/(oz./sq.yd.)	0.22	0.27		
MD Elongation, %	76.0	71.0		
TD Elongation, %	80.0	92.0		
Air Permeability, cu.ft./sq.ft./min	1053.0	1008.0		
Structural quality	web	web		

n.m. = not measured

Attempts were made to pull the extrudate around a 2.5" mandrel followed by a 3.75" ring, but the resulting webs had very large and randomly distributed pores.

Some trials were made to extrude polypropylene without the static mixer using physical blowing agents, such as N_2 and CO_2, but the extrudate was always extremely coarse and non-uniform regardless of extrusion conditions. The mixer is essential in the present system when using physical blowing agents; however, when the mixer was put back on the extruder and CO_2 was injected at the vent port to a mixture of polypropylene, fumed silica and silica flour, the webs were inferior to those obtained when using N_2 under the same conditions. The properties of webs formed using N_2 have been reported previously (10).

3.2 Poly (ethylene terephthalate) (PET) and nylon 66

The primary emphasis since the last report (10) has been to demonstrate the feasibility of forming PET and nylon 66 webs. Previously both these polymers exhibited excessive degradation and would stick to the die. The switch to downward extrusion and purchase of drying equipment were made to eliminate these sticking and degradation problems respectively.

It has been possible to extrude PET film by removing the mixer from the experimental set up. Prior to extrusion PET must be recrystallized at 160°C and dried for about three hours. Also, it must be ground using the 46 SP grinder purchased from Polymer Machinery Corporation with a 1/8" screen in order to reduce the pellet size and to improve mixing with the additives. With the temperature profile set at 220-260-260-280 (neck)-280°C, no degradation was observed; feeding and extrusion proceeded smoothly and continuously.

Kemtec 500, a new blowing agent manufactured by Sherwin Williams Co., Chem. Div. and which decomposes into CO_2, was compounded with PET, but blowing

did not take place regardless of the temperature profiles and additive concentrations used. Extrusion with 0.5% Kempore 200MC did produce some blowing without degradation; however, both the melt strength and the viscosity of the extrudate were too low and extrusion could not be forced to proceed continuously. Compounding with polypropylene – Hercules Profax 6323 or 6823 – in order to increase the viscosity will be tried in future runs. New additives should be considered in order to increase melt strength.

As in the case of PET, good nylon 66 film was obtained without the mixer when the temperature profile was set at 210-250-250-290 (neck)-290°C. Drying for long periods of time even at low temperatures (80-100°C) caused slight degradation during extrusion. Drying for 5 hrs at 140°C also resulted in some degradation. Nylon should be dried for 3 hrs or less at temperatures at least as high as 160°C.

Mixing with 0.5% Kempore 200MC proved to be unsatisfactory when extruding with the profile shown above. By switching to 0.5% Expandex 5PT and by compounding with 20% polypropylene (Profax 6323) it was possible to pull the extrudate around the mandrel but only at screw speeds as low as 20 RPM. Even at this speed the extrudate broke easily and the structure was coarse and non-uniform.

3.3 Poly (butylene terephthalate) (PBT)

Webs of PBT/polypropylene blends were discussed in the last report (10). In continuing work, methods for improving web quality and reducing the percentage of polypropylene present have been explored.

For PBT extrusion, the two stage screw was replaced by the single stage screw and the rotating die was used rather than the fixed die as was the case in nylon 66 and PET extrusion. No predrying of PBT is required as there are no signs of degradation. PBT pellets must be ground prior to mixing with

any additive in powder form.

With the temperature profile set at 220-260-260-260- (neck)-260°C it has been possible to produce webs from mixtures of PBT and polypropylene (Profax 6323) containing 50% and 75% PBT, and also from pure PBT, by adding 0.5% Kempore 200MC. The extrudate can be easily stretched around the 2.5" mandrel. The process is continuous at screw speeds as high as 50 RPM and with cooling air pressures of 10 psig. At higher screw speeds the degree of filminess increases significantly. Below 50 RPM, webs present the same deficiencies as do polypropylene webs. Adding 1% Cab-o-sil M5 does not improve uniformity but it does produce smoother webs.

It had not been possible to produce pure PBT webs previously in our experimental apparatus. The results obtained are encouraging although there is significant room for improvement in web quality.

4.0 Yarn Formation

The formation of polypropylene yarns has been reported previously (10) and recently Y. H. Park completed his thesis on this subject (11). The results of Park's study will be summarized here as well as additional work on reducing yarn denier and evaluating PBT yarns.

In Park's study the materials and extrusion conditions were fixed. The feed mixture consists of 99.0% polypropylene (Profax 6323), 0.5% Kempore 200MC and 0.5% Cab-o-sil M5. The temperature profile along the extruder barrel was controlled at 200-230-230-230 (neck)-230°C. The screw speed was 30 RPM, the winding speed was 82 fpm and the cooling air pressure was 20 psig. The extruded web has a denier of about 5,000 and a tenacity of 0.14 g/denier.

With this yarn Park studied the effect of drawing and twisting conditions on the structure and properties of the yarn.

The following conclusions are developed in Park's thesis (11):

1. When the extrudate is drawn twice, given 4 twists per inch (TPI) and drawn twice again, the highest tenacity, 3.0 g/denier, is obtained when the drawing temperature is 130°C.

2. Sequential drawing and twisting cycles with roughly the same twists in the first and second twisting steps at a given total twist improves tenacity. Twisting introduces mutual cohesion between the fibrils but also some fibril obliquity; both effects combine to yield an optimum twist below and above which tenacity decreases.

3. Studies of the dependence of birefringence on tenacity show that there is a correspondence between tenacity and orientation in the polymer. The higher the orientation, the higher the tenacity.

4. The density of yarn samples stretched to a draw ratio of 4 has been measured by the flotation method in solutions of water and methanol using a hydrometer. Results show that density increases as drawing temperature increases; this may be due to a decrease in bubbles in the fibrils.

5. The effect of draw ratio on yarn properties was studied by drawing at 135°C and twisting 2 TPI. Results showed that tenacity, birefringence and density increase and the elongation at break decreases with increasing draw ratio.

6. Fibrils produced by blown-film extrusion do not have uniform diameters. The resulting distribution becomes sharper as draw ratios increase, but the most probable fibril diameter remains constant at 75 microns.

Further experiments have been performed using polypropylene compounds with Kempore 200MC in order to reduce the denier while maintaining a high tenacity. The "as-extruded" denier of a mixture of 99% polymer and 1.0% Kempore 200MC was 3,930, lower than that analyzed by Park (11). All other extrusion conditions

were kept the same in this experiment as in Park's experiments. The denier of the original web was reduced by splitting the web to 1/2, 1/4 and 1/8th its original width as shown in Table 8. All samples were then drawn 3 times and twisted 2 TPI. The denier and the tenacity of the resulting yarns is shown in Table 8. As can be seen, tenacities close to 2.00 gr/denier are obtained while the denier is reduced to 163. The un-split yarn has a similar tenacity to that obtained by Park for the same draw ratio and twist (3X, 2TPI).

Several unsuccessful attempts have been made to extrude continuously nylon 66 and PET yarns. The difficulties encountered are the same as those for web extrusion. However, it has been possible to produce yarn using mixtures containing PBT and polypropylene in a 1 to 1 ratio and in a 3 to 1 ratio by weight. Also, pure PBT yarn has been produced. In all cases 1.0% Kempore 200MC has been used as a blowing agent. All other processing conditions are shown in Table 9. The appearance of samples 1 and 4 is better than samples 2 and 3, which were coarse. The temperature of the die was 20°C higher than the extrusion temperature for samples 2 and 3, which may explain the poorer quality of these samples. Adding 1.0% fumed silica to the feed mixture did not reduce the coarse texture of the yarns.

All of the yarn samples except number 1 are very weak and break very easily during stretching over a hot cylinder. Twisting these yarns before hot stretching does not reduce this tendency to break. On the other hand, yarn sample 1, which has the highest % polypropylene, can be easily hot stretched at 160°C.

The effects of drawing, twisting and slicing yarn sample 1 in order to reduce the denier and increase the tenacity is shown in Table 10. Sequence number 1 is the as-extruded yarn with 2 TPI. It is weak and highly extensible. Sequence 2 illustrates the effect of hot stretching this yarn 2X at 160°C

TABLE 8

Properties of Polypropylene Yarns Formed from Split Webs

Sequence No.	No. of Web Slices	Draw-Twist Sequence	Denier	Tenacity (g/denier)	Elongation %
1	1	3X →2TPI	1310	2.05	27.5
2	2	3X →2TPI	655	1.70	34.0
3	4	3X →2TPI	327	1.64	26.0
4	8	3X →2TPI	163	1.98	30.0

TABLE 9

Extrusion of PBT/Polypropylene Yarns

Sample	1	2	3	4
Materials				
1　PBT, %	49.5	74.25	74.25	99.0
2　PP 6323, %	49.5	24.75	24.75	0.0
3　KEMPORE 200 MC, %	1.0	1.0	1.0	1.0
4				
Processing Conditions				
Screw type	1 stage	1 stage	1 stage	1 stage
Zone 1, °C	220	220	220	220
Zone 2, °C	260	260	260	260
Zone 3, °C	260	260	260	260
Neck, °C	260	280	280	260
Die, °C	260	280	280	260
Vent Port feed, yes/no	no	no	no	no
Vent port, psi	–	–	–	–
Zone 3, psi	n.m.	n.m.	n.m.	n.m.
Die, psi	n.m.	n.m.	n.m.	n.m.
Mandrel diameter, inches	2.5	2.5	2.5	2.5
Quench air, °C	room	room	room	room
Quench air, psig	10	10	10	10
Screw speed, RPM	25	40	40	40
Wind-up speed, FPM	86	86	110	110
Die, RPM	10	10	10	10
Web Properties				
Basis Weight, oz/sq. yd.				
MD Tenacity, (lb/in)/(oz/sq. yd.)				
TD Tenacity, (lb/in/(oz./sq.yd.)				
MD Elongation, %				
TD Elongation, %				
Air Permeability, cu.ft./sq.ft./min				
Structural quality				

n.m. = not measured

TABLE 10

Effect of Drawing, Twisting and Slicing a 1/1 PBT/Polypropylene Yarn

Sequence No.	No. of Web Slices	Draw Twist Sequence	Denier	Tenacity (g/denier)	Elongation %
1	1	2TPI	6032	0.21	145.0
2	1	2X → 2TPI	3016	0.49	32.3
3	2	2X → 2TPI	1508	0.42	27.5
4	4	2X → 2TPI	754	0.38	20.6
5	8	2TPI → 2X	377	0.42	16.3

before imparting 2 TPI. The tenacity did not increase as much as expected, but the elongation decreased more than expected in comparison to similar studies on polypropylene (10). In sequences 3 and 4 the extruded yarn was sliced into lower denier yarn before drawing and twisting as in sequence number 2. When the extruded yarn was divided 8 times, sequence number 5, it was necessary to twist the yarn prior to drawing. The resulting lower denier yarns exhibit approximately the same tenacity but progressively lower elongations compared to sequence number 2. In general, the yarns consisting of 50/50 mixtures of PBT/polypropylene have poorer mechanical properties than 100% polypropylene yarns.

5.0 Continuous Drawing System

A continuous drawing system has been designed and is being constructed by Killion Extruders, Inc.. Operation of this system will permit pilot production of drawn yarns for product evaluation and an investigation of drawing temperatures, rates and extension ratios on yarn properties. A flexible system has been designed to permit off-line drawing of yarn from a pay-off roll as well as on-line drawing of the extrudate. The latter case is illustrated in Figure 11.

The off-line system is primarily intended for drawing yarn after it has been twisted. It consists of a pay-off roll, a hot silicone oil bath, two pull roll assemblies and a winder. The first pull roll assembly is immersed in the silicone oil bath and its speed can be varied between 0 and 100 fpm. The second pull roll assembly is located outside the bath and has a speed range of 0-600 fpm to provide for a wide range of draw ratios. The tank for the silicone oil bath has 4 idler rolls. Idler rolls I1 and I4 are removable and idler rolls I2 and I3 are adjustable height rolls, as is the upper roll of the tank pull roll assembly. There will be heaters placed at the bottom
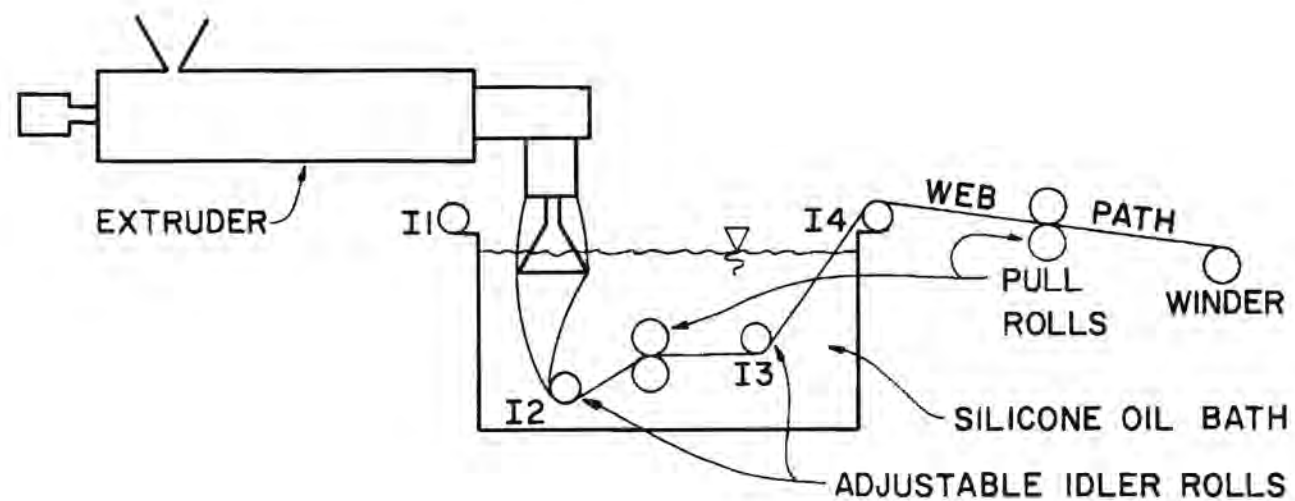
Figure 11. Schematic of Continuous Drawing System shown
in line with Extruder.

of the tank with enough power to raise the fluid temperature up to 250°C. The tank itself is placed on threaded adjustable rods which will allow lowering and raising of the tank and its contents.

The drawing system is split into three independent sections, each placed on four casters. The first section contains the pay-off assembly, the second section consists of the hot oil bath and the last section consists of the second pull roll assembly and the winder. By separating these sections it will be possible to adjust the distance between the bath and the second set of drive rolls in order to allow for cooling of the yarn before it reaches the pull roll assembly. In addition, the pay-off assembly can be removed and the oil bath placed underneath the extrusion die to control the quenching and initial drawing of the extrudate as shown in Figure 11.

For start up of the drawing operation all adjustable rolls in the bath will be in the "up" position. The silicone oil will be preheated to the desired temperature and yarn will be fed from the pay-off assembly above roll I1, below roll I2, below the upper roll of the tank pull roll assembly, below I3, above roll I4 and finally pulled by the second pull roll assembly. Once continuous and stable flow is achieved, all adjustable rolls will be lowered into the bath, the motor driving the tank pull roll assembly will be started, and drawn yarn will be collected in the winder.

Alternatively, it will be possible to feed yarn directly from the extruder, as shown in Figure 11. In this case, roll I1 will be removed, all adjustable rolls will be in the "up" position, and the tank itself will be in the "down" position. Yarn will be fed from the extruder below roll I2, below the upper roll of the tank pull roll assembly, below roll I3, above roll I4 and finally pulled by the second set of drive rolls. Once stable operation is achieved under these conditions, the bath will be raised to

cover the mandrel, all adjustable rolls will be lowered into the bath, the
first set of drive rolls will be started and drawn yarn will again be col-
lected in the winder.

D.   Fiber Weaving by Aerodynamic Forces

1.0   Introduction

       The process of fiber weaving is important because of its poten-
tial for increased productivity and reduced material waste and energy
consumption, compared with conventional processes of spinning fibers
into yarns and of weaving yarn into fabric. The fibers must be ar-
ranged in orderly patterns to produce fiber-woven fabrics whose per-
formance characteristics are comparable to those of conventionally
woven fabrics. In particular, the fibers in the fabric should be
aligned principally in two perpendicular directions. This portion of
the present research program is concerned with discovering methods for
aligning and depositing fibers in this manner by using air flows and
aerodynamic forces.

       An article, "On Fiber Alignment Using Fluid Dynamic Forces",
by L. H. Bangert and P. M. Sagdeo, has been accepted for publication
by the Textile Research Journal. This is a description of earlier
work under this grant on the alignment of individual fibers moving
in an air stream. Also, a patent application has been filed by L. H.
Bangert (assigned to Georgia Institute of Technology) for, "Method
for Fiber Alignment Using Fluid - Dynamic Forces". This method uses
counterflow air jets to create a desired streamline behavior in a main
air stream. This desired behavior causes the fibers to rotate so that
they are parallel to the main stream.

       The following describes an apparatus in which a continuous
supply of fibers is provided for experiments on fabric formation.

Also described are some preliminary results of experiments directed toward a method of measuring the degree of alignment of a collection of fibers.

2.0 Multiple – Fiber Alignment and Deposition

A fiber injection system has been designed and constructed to provide a continuous supply of fibers to the alignment system. This fiber injection system has the following basic functions: (1) Receive a sliver of fibers and produce individual fibers; (2) Inject the individual fibers into the tunnel air stream. After the fibers enter the tunnel air stream, they are aligned by the action of the transverse air jets. This procedure was described in the Eighteen Month Technical Progress Report, December 1976, and in the journal article and patent application named previously.

The operation of the fiber injection system can be described with the aid of Figures 12 and 13, which are top and side views, respectively. A sliver is fed in with the aid of a feed roll. A toothed combing roll separates individual fibers from the sliver. The individual fibers are transported away, as indicated in Figure 12, by the motion imparted by the combing roll and by the air flow induced by a vacuum pump. The fibers land on the rotating injector screen. They are held there by the radially-inward, vacuum-induced air flow. Note that the axis of the rotating screen is a tube, perforated over its length within the injector. This axis is connected by flexible tubing to the vacuum pump. The latter is a Stokes Microvac Model 148-H, with a displacement of 40 cfm.

PLENUM

CONTRACTION CONE

TEST SECTION

SCREEN DRIVE MOTOR

ROTATING SCREEN

PLASTIC SHROUD

FIBER PATH

FIBER FEED DRIVE MOTOR

TO VACUUM PUMP

COMBING ROLL

COMBING ROLL DRIVE MOTOR

SLIVER INLET
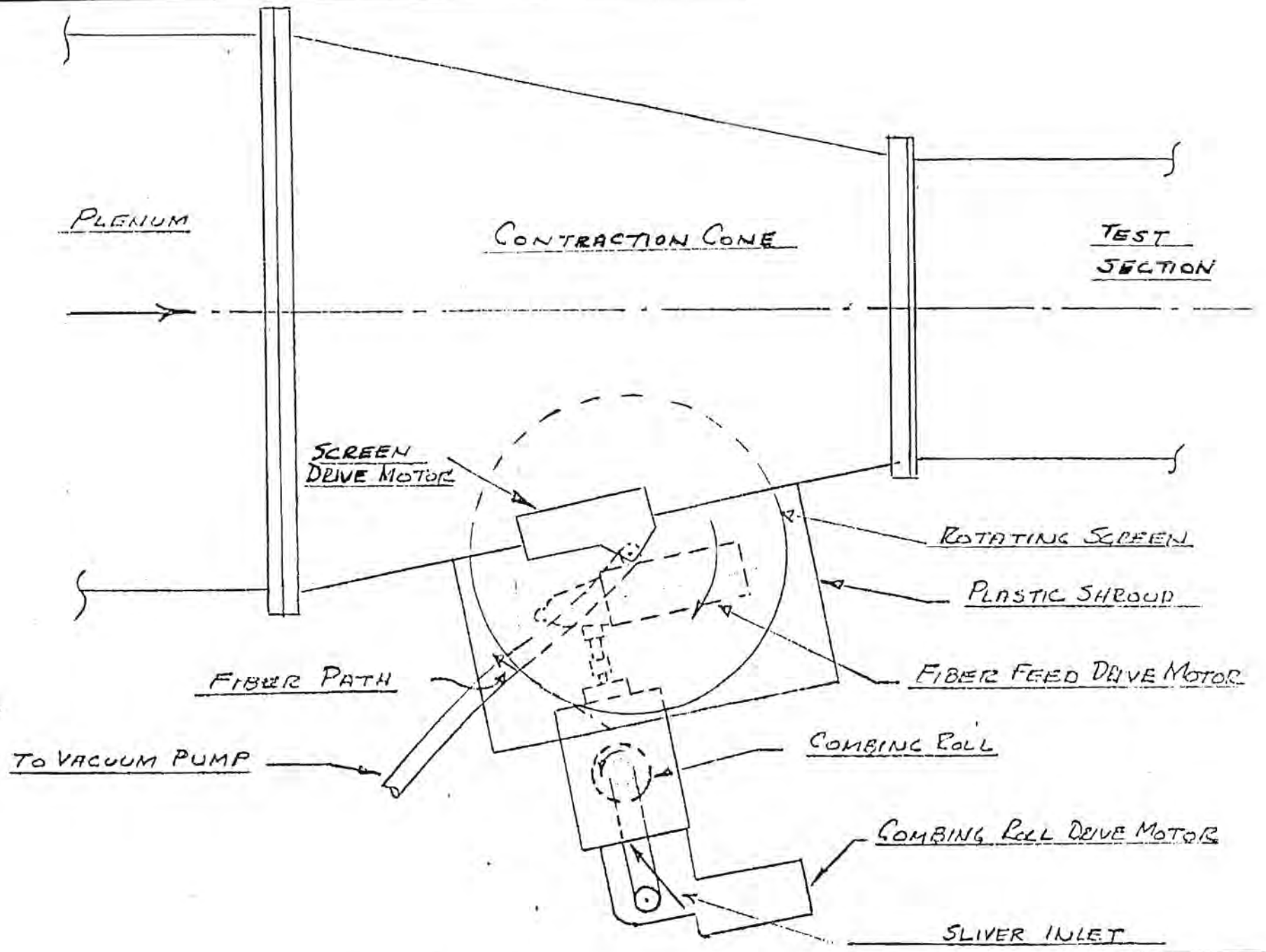
Figure 12. Top view of fiber injection system.

SCREEN DRIVE MOTOR

WIND TUNNEL CONTRACTION CONE

PLASTIC SHROUD

PLENUM

TEST SECTION

ROTATING SCREEN

COMBING ROLL DRIVE

TO VACUUM PUMP

FIBER FEED DRIVE

ENLARGED SIDE VIEW OF FIBER INJECTION SYSTEM

- 57 -
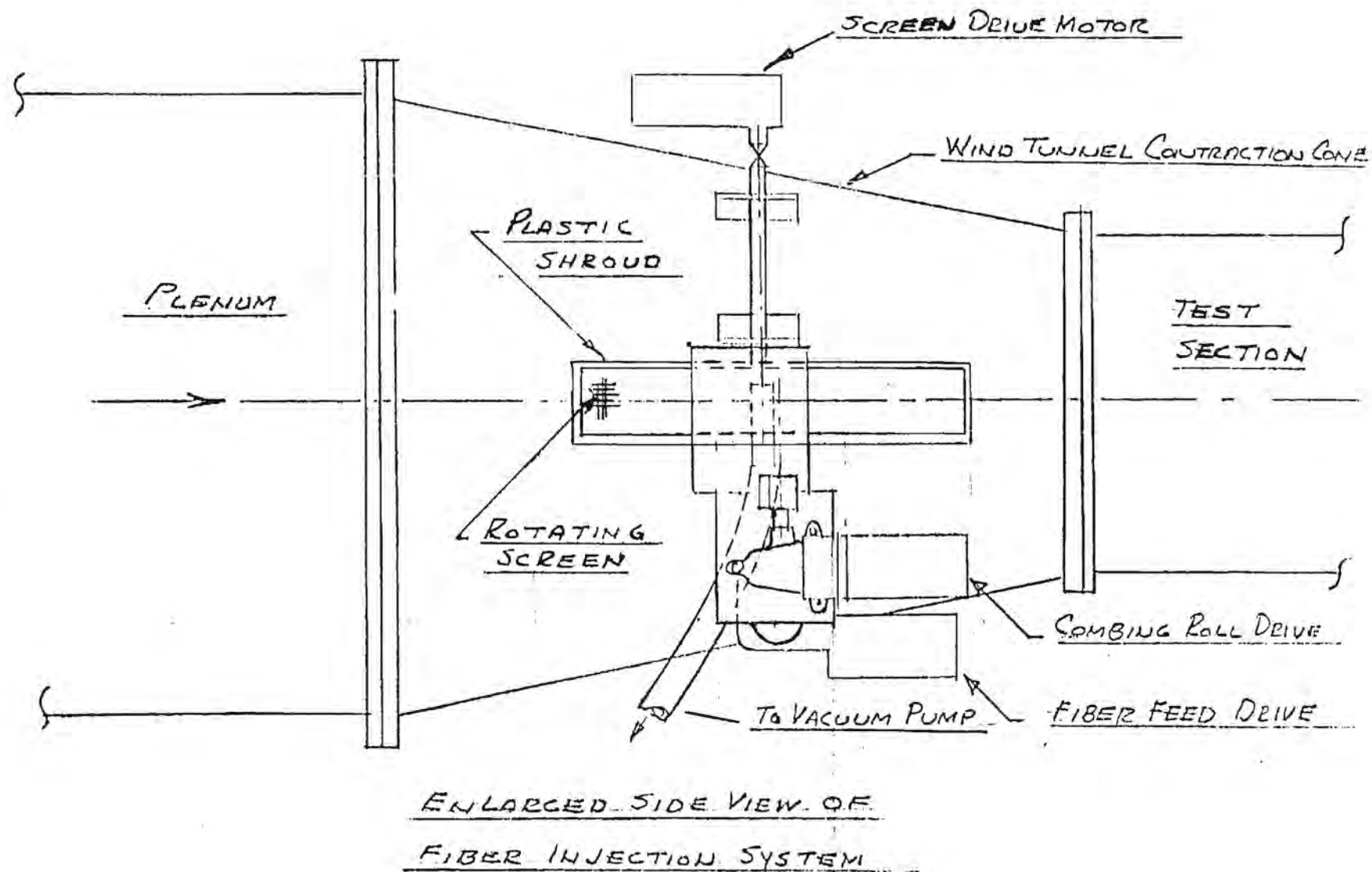
Figure 13.   Side view of fiber injection system.

The rotating injector screen carries the fibers inside the tunnel. The vacuum is ineffective at this stage because of the intervening tunnel side wall. The fibers are initially on the upstream side of the injector, however, so they are held in place by the tunnel air flow. As the screen rotates, the fibers are blown off when they reach the point where the tunnel flow velocity vector is approximately tangent to the injector screen. The transverse jets are located where the contraction cone joins the test section. The fibers are aligned within a distance of one test section height downstream of the jets and then deposited on a collecting screen.

A simpler version of the fiber injection system was tested earlier, in which fibers were placed on the injector screen by hand. For this reason, only small numbers of fibers were used. Some of these results were included in the Eighteen Month Technical Progress Report.

The installation of the fiber injection system is nearly complete. Experiments are planned to begin in early October 1977.

3.0   Measurement of Fiber Alignment

Knowledge of the degree of alignment of a collection of fibers is important in a number of applications related to textiles. For example, in the present application a large number of fibers are deposited on a screen. The object is that all the fibers be parallel to each other. (Then successive laydown processes 90° apart will produce the desired mutually perpendicular fiber arrangement.) There is a need to measure the degree of alignment that is actually achieved. The ultimate test is an evaluation of the properties of the fabric that is formed, as these will depend on the degree of fiber alignment. It is anticipated that there will be a need for an intermediate measurement of alignment, however.

An investigation has been started of the feasibility of measuring average fiber alignment in a group of fibers by an optical method. The particular optical method being studied makes use of certain properties of polarized light. When plane polarized light is directed toward a polarizer whose plane is 90° from that of the incident light, nearly all of the incident light is absorbed. If an optically anisotropic fiber is placed between the two crossed polarizers, the light incident on the fiber will be converted to elliptically polarized light. The receiving polarizer then transmits the portion of the light that is elliptically polarized.

This principle was utilized in two related patents. The first (J. R. Roehrig, "Apparatus for Grading Anisotropic Fibers," U.S. Patent 2,824,487, 25 Feb. 1958) is a method for measuring the diameter of individual fibers. The second (S. W. Bridges and J. R. Roehrig, "Apparatus for Grading Fibers," U.S. Patent 2,824,488, 25 Feb. 1958) is a method for sensing the type of fiber. Again, each measurement is for a single fiber.

If the plane of polarization of the incident light is parallel or perpendicular to the longitudinal axis of the fiber, then none of the incident light will be elliptically polarized. The maximum amount of elliptically polarized light is expected when the longitudinal axis of the fiber is at 45° relative to the plane of polarization of the incident light. The present investigation is directed toward using these principles to measure the average alignment of a group of fibers.

A simple experiment was performed to determine whether the intensity of light elliptically polarized by a group of parallel fibers was indeed dependent upon their alignment relative to the plane of polarization. The basic parameters in this experiment were: (1) fiber type; (2) incident light intensity; (3) fiber area density (number/area); (4) fiber orientation (with all parallel); and (5) photomultiplier tube voltage. Initial results were obtained using Dupont Lycra Type 125 fibers, 40 denier, 2 inches long. The incident light intensity was also held constant. The fiber area density, fiber orientation, and photomultiplier tube voltage were each varied independently.

Figure 14 is a schematic of the experimental apparatus. Figure 15 is a photograph of the apparatus, including the associated instrumentation. The overall height is about 3 feet and width about 1.5 feet. The diverging light beam from the source is collimated by the upper lens, and then passed through a plane polarizer, the fiber sample and the supporting glass plate, another plane polarizer whose plane is perpendicular to the first, and finally through the lower focusing lens onto a photomultiplier. The latter senses the intensity of the light incident upon it.

The light source was a tungsten filament lamp, rated at 6 candlepower at 6.5 volts and 1.02 amperes. The results of this experiment are sensitive to
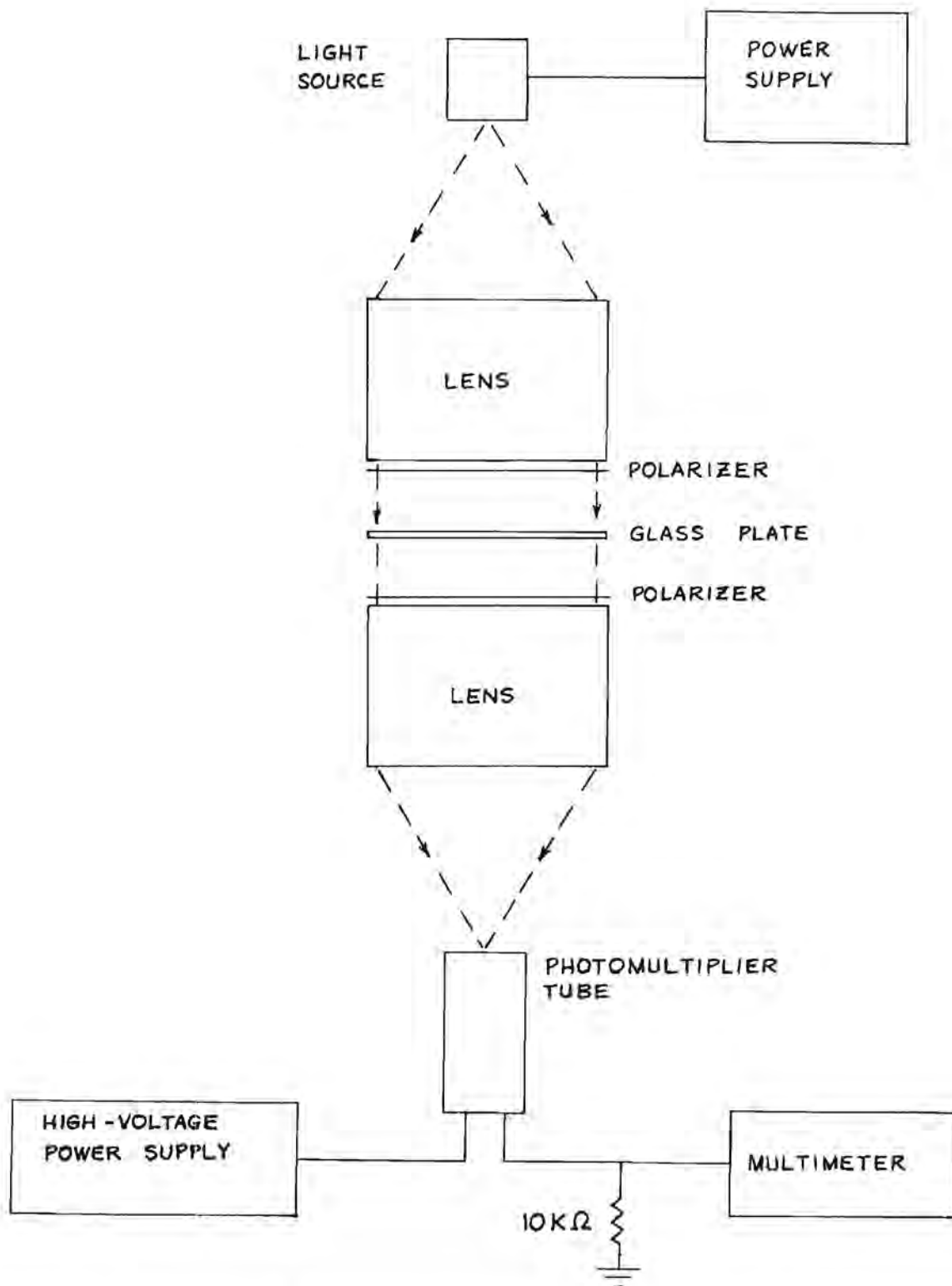
Figure 14. Schematic of apparatus for optical measurement of fiber alignment.

Figure 15. Photograph of apparatus for optical measurement of
fiber alignment.

fluctuations in line voltage. For this reason the lamp was connected to a highly-controlled power supply (Hewlett-Packard 62288 DC Power Supply) and operated at 6 volts and 1.1 amperes. The lenses were Kodak AERO-EKTAR lenses originally used in aerial photography. They have the advantage of large size coupled with short focal length (f/2.5 with 12 inch focal length). The upper polarizer was Polaroid Corporation HN32, while the lower was HN22. These are both sheet polarizers. They were aligned with no fibers present, so that the light transmitted to the photomultiplier tube was a minimum.

The photomultiplier tube was an EMI Gencom 9656B. The power supply to the PM tube was a Fluke 412B High Voltage Power Supply. The output voltage was sensed by a Fluke Multimeter. The two power supplies and the multimeter are shown on top of the enclosing cabinet in Figure 15. The measurements were made with the apparatus completely enclosed, so that only light from the controlled source would reach the PM tube. (In Figure 15 the access panel has been removed).

Experiments were made with 60 and 120 fibers arranged in parallel on the glass plate. Figure 16 is a photograph of the arrangement with 60 fibers. The light-transmitting surface had a 3 inch diameter. PM tube voltages of 850 and 930 were used. Measurements of intensity were made for fiber angles of - 90° to + 90° at 10° intervals.

It was discovered that the glass plate being used was anisotropic. There were variations of up to 6% in the transmitted intensity, depending on the orientation of the plate relative to the plane of polarization. This defect will be corrected in future experiments.

Figure 17 shows the percentage increase in intensity relative to the zero fiber case, versus the angle of the fibers. This ordinate was chosen to partially compensate for the small anisotropy of the glass plate. These
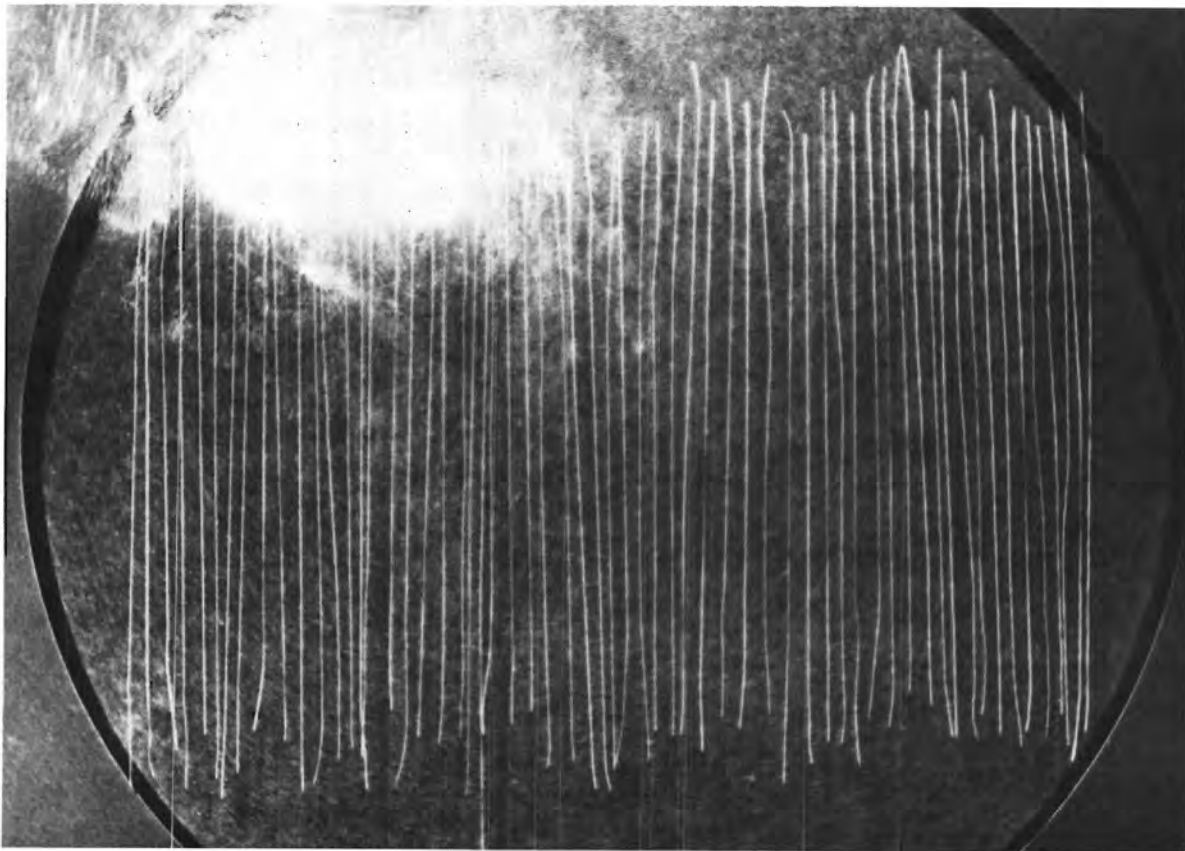
Figure 16. Photograph of 60 fibers mounted on the glass plate.

$(I - L)/I_o \sim \%$

○ 60 FIBERS , 850 V
☐ " " 930 "
△ 120 " 850 "
◇ " " 930 "

25

20

15

10

5

0

−90°   −60°   −30°   0°   30°   60°   90°

FIBER ANGLE RELATIVE TO PLANE OF POLARIZATION
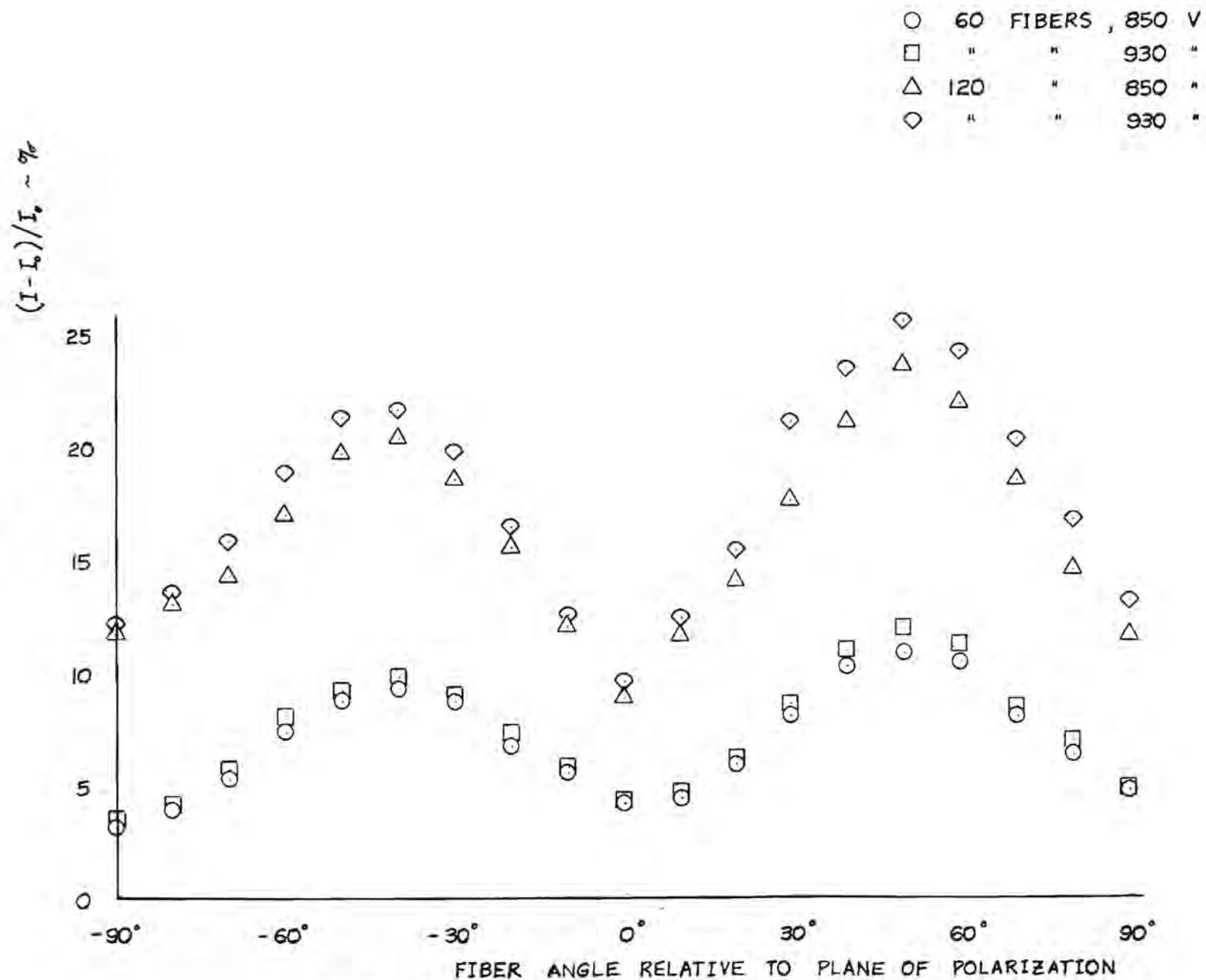
Figure 16.   Variation of transmitted intensity, relative to
plane glass, with fiber angle.

data confirm the expectation that the transmitted intensity is a function of the fiber orientation relative to the plane of polarization. It was also confirmed that the maximum transmitted intensity occurs when the fibers are aligned 45° from the plane of polarization. The variation of voltage level into the PM tube had a small effect, for the values considered. The amount of light elliptically polarized was very strongly dependent on the fiber area density. The transmitted light intensity was more than two times greater for 120 fibers than for 60 fibers. These results seem to be consistent with the theory outlined previously.

The next experiments will consider cases where the fibers are not all parallel. The objective is to be able to relate transmitted intensity to a measure of average fiber angle. Thus these data will represent a further calibration of the system.

E. Aligned Fiber Web Formation Using an Electrostatic Field

The purpose of this portion of the work has been to construct apparatus for conveying fibers into an electrostatic field for fiber alignment and deposition of those fibers while aligned to form a web. Insulated wall ductwork has been assembled using polymethylmethacrylate plastic sheet for convection of fibers through the alignment zone. Alignment occurs in a section of the ductwork in which two opposite faces are brass, connected to a variable high voltage power supply. This set-up is illustrated in Figure 18.

Air is drawn through the duct by a centrifugal fan. Air velocity is controlled by a vent located downstream from the alignment and collection regions of the duct. Using a pitot tube located three diameters downstream from the air entry opening, and taking into account duct expansion to the alignment region, air velocities ranging from 4 to 12 feet per second have been obtained with this apparatus.

The applied voltage in the alignment section is variable from 1 to 20 Kv. Electrostatic field strength is essentially constant between planar surfaces so long as the size of the planes is much larger than their separation distance. There is approximately one order of magnitude difference between the planar dimension and separation distance in the vertical alignment section, and same order of magnitude for the horizontal alignment section. After initial tests are completed, the horizontal alignment section will be modified to enlarge the charged surfaces and reduce their separation to improve electrostatic field strength uniformity.

BRASS EXTENDS ± 6 in. (BOTH SIDES)

PITOT

EXHAUST TO FAN

BRASS TOP AND BOTTOM

2" x 14" ALIGNMENT SECTION x 24" LENGTH

L
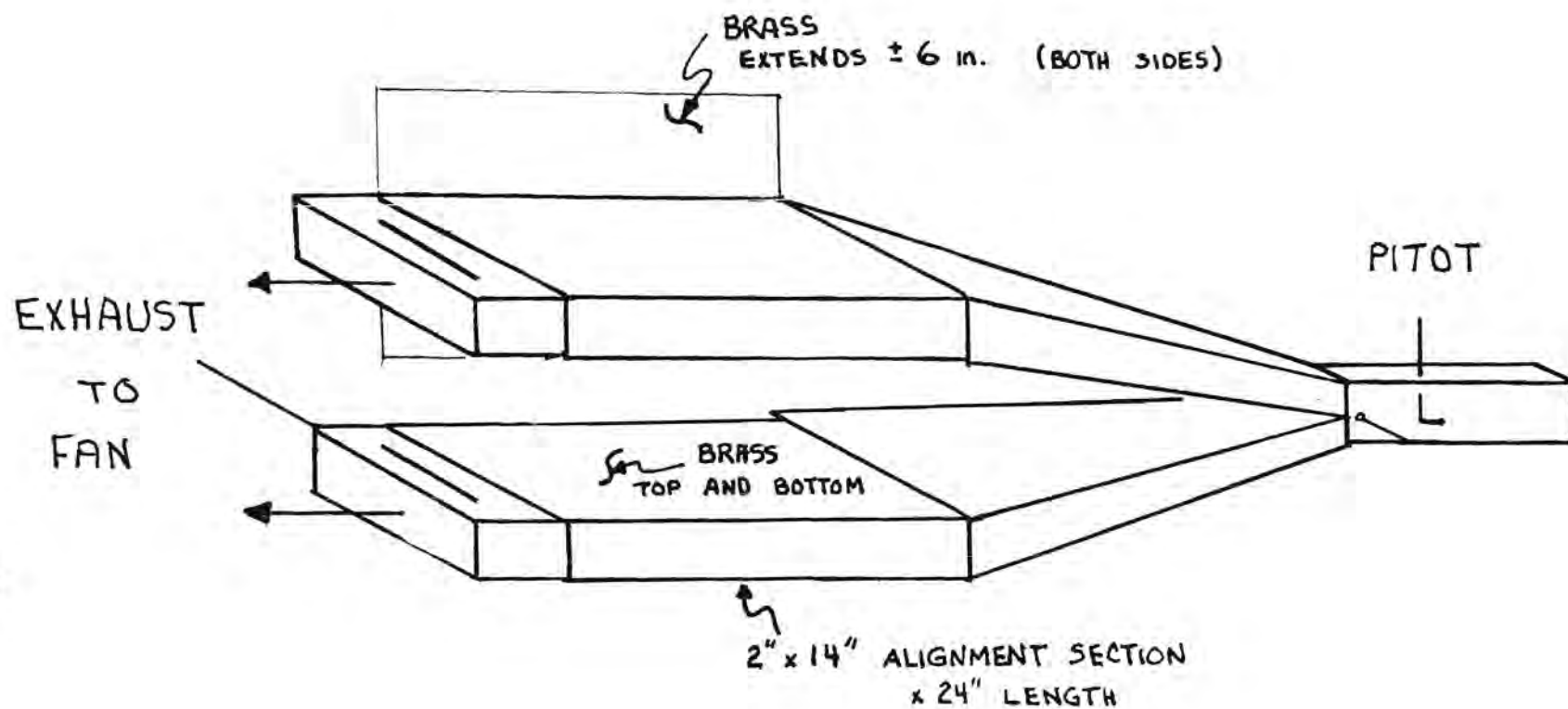
FIGURE 18. DUCT ARRANGEMENT

Initial attempts to produce a web by drawing a screen through slits in the duct near the alignment region met with some difficulty. The slits allow entry of an air stream which in turn causes turbulence. The first web sample produced with this system contained approximately 40% aligned fibers with the remainder being randomly oriented. These fibers were hand fed and generally were not well separated.

Upcoming work will include adding a carding system and fiber pick-up nozzle to the front end of the duct system to improve fiber separation and feed uniformity. Several options are possible to improve fiber deposition. An attempt will be made to deposit fibers in a batch process rather than a continuous process as is currently being tried. A deposition screen made of insulating material will be placed in the alignment section to acquire sample webs whose quality and degree of alignment may be assessed in order to determine the best direction for further development of this process.

IV.   Utilization Plan

The procedures and time schedule that are planned to ensure that the technology developed under the proposed program is transferred into the textile and apparel industries are outlined in Table 11.

Presentations:

"Gaseous Polymer Solutions", J.L. Lundberg, 29th Southeast Regional Meeting, American Chemical Society, Tampa, Florida, November 2, 1977.

"Foam Extrusion of Yarns and Webs", J.D. Muzzy, A.I.Ch.E. Section Meeting, Greenville, S.C., April 1977.

"Foam Extrusion of Yarns and Webs", J.D. Muzzy, Soc. Plastics Engineers ANTEC, Montreal, Canada, April 1977.

Publications:

"Foam Extrusion of Yarns and Webs", Soc. of Plastics Engineers Tech. Papers, 23, 44 (1977).

"On Fiber Alignment Using Fluid Dynamic Forces", L.H. Bangert and P.M. Sagdeo, Textile Research Journal, December 1977.

Patent

"Method for Fiber Alignment Using Fluid Dynamic Forces", L.H. Bangert, Patent Application No. 828893, filed Aug. 29, 1977, assigned to Georgia Institute of Technology.

Presentations of research results will continue to be made during the coming year at meetings of appropriate professional societies and trade associations.

Around mid 1978 a technical conference with demonstrations of the technologies developed under the program will be held at Georgia Tech. The purpose of the Conference will be to supplement the final summary technical report by providing industry with an opportunity to view first-hand the processing equipment and products developed.

Table 11. Utilization Plan

	0	12	24	36
Progress Reports	X X	X X	X X	
Research Reviews		X	X	X
Presentations to:				
Trade Associations		X---------------------X
Professional Societies		X---------------------X
Technical Conference & Demonstrations				X
Summary Technical Report				X

V. Conclusions

Polybutene-1 and polypropylene are readily soluble in n-butane and
carbon dioxide at concentrations which should be useful in transporting and
fabricating these polymers. Nylon-6 is dissolved easily in carbon dioxide
at surprisingly low pressures.

No correlations of pressure-temperature-concentration behavior are
obvious from solution data collected to date.

Preliminary trials show that fibrous polymers can be produced by
extruding gaseous solutions of polymers into the atmosphere. Fiber diameters
ranging from about 2 to 10 microns with some larger fibers up to 25 microns
or larger were obtained.

It has not been possible to produce foam fibrillated polymer webs
suitable for textile applications. Webs produced by foam extrusion of poly-
propylene under optimum conditions are not uniform, have low tensile
properties in both the machine and the transverse directions, and show
large directional imbalances of these tensile properties.

Polypropylene yarn suitable for some industrial applications and compar-
able to commercial textile yarn has been produced by foam extrusion. Nylon 66
and PET yarns have not been produced, but some success has been achieved
using mixtures of PBT and polypropylene and also by foam extrusion of pure
PBT. PBT yarn is very weak and cannot be drawn unless it contains large
amounts of polypropylene.

A fiber injection system has been designed and constructed to continuously
supply individual fibers to an aerodynamic alignment system. Experiments under-
way on web formation are encouraging.

Preliminary experiments made on an optical system for measuring the
average alignment of a group of fibers resting on a flat transparent surface

confirm the anticipated dependence of transmitted light intensity on fiber orientation and fiber area density. Future experiments will attempt to establish relationships between transmitted intensity and average fiber orientation for nonparallel fibers.

Initial attempts to produce an aligned web by conveying fibers through an electrostatic field met with some difficulty. The first web sample produced contained approximately 40% aligned fibers with the remainder being randomly oriented. It is anticipated that greater orientation will be achieved with improvements in fiber separation and feed uniformity, modifications to the electric field, and a redesign of the fiber deposition system.

VI. Future Plans

Studies of the solubility of nylon 6 in carbon dioxide will continue. Upon completion of these experiments, solubility measurements on polyethylene terephthalate will be attempted. If carbon dioxide can be contained at the higher temperatures required for this study, measurements of solubilities of polyacrylonitrile and polyparaphenylene terephthalamide will also be attempted.

Experiments on the extrusion of polymers from gaseous solutions will be initiated in the near future. The first system to be investigated will be nylon 6 dissolved in carbon dioxide.

Under the foam fibrillation task emphasis will be placed on yarn development. A continuous drawing system will be installed by January, 1978. After studying processing variables with this system, pilot-scale quantities of yarn will be produced for evaluation in various fabric constructions. Economic evaluations will be made for producing yarn in commercial quantities.

Polypropylene will be utilized in most of the trials to demonstrate the technology of forming yarns by foam fibrillation because the greatest success has been achieved with this polymer. Efforts to improve the quality of yarns made from other polymers, particularly polyesters and nylons, will continue.

Experiments on aerodynamic web formation will be initiated. The object is to have an air laydown process in which the fibers are mainly aligned in two mutually perpendicular directions. The first step will be to determine a combination of speeds for the feed roll, the combing roll, and the injector

screen that avoid clumps of fibers being injected into the air stream. The second step will involve variation of aerodynamic parameters to achieve maximum alignment of the deposited fibers. Included will be tests to evaluate fiber alignment in the web. These may be mechanical (e.g. multi-direction tensile tests), or possibly optical.

Efforts will also be continued toward the development of an optical system for evaluating average fiber alignment of a large number of fibers. The next experiments will attempt to relate transmitted light intensity to average fiber orientation for nonparallel fibers.

Experiments on the production of aligned fiber webs using a combination of aerodynamic and electrostatic forces will continue.

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3. See, for example, W.J. Moore, Physical Chemistry, 3rd Ed. (Prentice-Hall, Englewood Cliffs, N.J., 1962), p. 229.

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    b. J.G. Kirkwood and I. Oppenheim, Chemical Thermodynamics (McGraw-Hill Book Co., Inc., New York, Toronto and London, 1961), pp. 53, 86, 90, 104.

8. Reference 7a, p. 197.

9. Reference 7b, p. 50

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Advanced Technology Applications
in Garment Processing

by

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Final Summary Technical Report

FOREWORD

This program was funded by the National Science Foundation, Advanced Productivity Research and Technology Division.

The program had three principal thrusts: (1) gas-polymer solutions; (2) polymer melt foam fibrillation; (3) fiber orientation in air streams. The principal investigators in each of the areas were Drs. John L. Lundberg, John D. Muzzy and Louis H. Bangert respectively. Dr. W. Denney Freeston was the program manager. During the three and a half years of the program several full time research staff members and students contributed to the research. The following is a partial list: A. R. Enuga, John I. Harper, G. H. Hoyos, Ken Ko, Alvin C. Levy, I. Moradina, Robert Newson, Y. H. Park, P. Sagdeo.

Abstract

Several specific technologies have been explored which may form part of the knowledge base necessary for the successful conversion of polymer chips and/or staple fiber directly into garments without spinning, weaving or knitting, cutting and sewing.

It is shown that polybutene-1 and polypropylene are soluble in n-butane and carbon dioxide, and nylon-6 dissolves in carbon dioxide.

Light weight polypropylene, polybutylene and PBT fibrous webs were formed by melt foam fibrillation. Polypropylene foam fibrillated webs were converted into medium strength yarn.

Natural and staple fiber was oriented in low velocity air streams using counter-flow jets and deposited on a screen without loss of orientation.

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I. Executive Summary

The principal areas of research were:

1. Measurements of solubilities of polymers in dense gases;

2. Continuous sheet and yarn formation from fibrillated polymer mixtures;

3. Orientation of staple fiber and disposition of fibers in parallel arrays using air flows and aerodynamic forces.

The major accomplishments under the program are as follows:

Polybutene-1 and polypropylene are readily soluble in n-butane at relatively low pressures, about 120 atmospheres and upwards, at temperatures above the melting points of the polymers.

Polybutene-1 and polypropylene are soluble in carbon dioxide at higher pressures, from about 450 to 900 atmospheres, at temperatures above the melting points of the polymers.

Nylon-6 dissolves in carbon dioxide at 400 to 500 atmospheres and 233° to $241^{\circ}C$. Carbon dioxide appears to be a good solvent for this polar polymer.

Fine fibers of polypropylene and nylon-6 were produced during solution studies by bleeding solutions to the atmosphere. However, attempts to scale-up extrusions of gaseous polymer solutions to produce fibers were not successful.

Polypropylene fibrous webs were formed by melt foam fibrillation. They were characterized by:

1) basis weights below 1 oz./sq.yd.; 2) average tenacities below 2 lbs/in/ oz./sq.yd.; 3) tenacity imbalances in excess of 5 (MD/TD); 4) high air permeabilities; and 5) a tendency towards filmy junctions.

Polybutylene and polybutylene terephthalate were also successfully processed but with difficulty.

Polypropylene foam fibrillated webs were converted into yarn with properties which make it suitable for some textile applications.

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Natural and staple fiber was oriented in low velocity air streams using
counterflow jets, and deposited on a screen maintaining the orientation.

Papers on the research have been published in various technical journals.
A patent application has been submitted on one development.

II.   Introduction

The long range objective of the program was to undertake research that might form the basis for new, direct, automatic, fast and inexpensive methods for converting polymer chips and/or staple fiber into fabrics without carding, spinning and weaving or knitting, and garments without cutting and sewing. The successful development of such processes will contribute to improving the United States' competitive position in textiles and clothing in the U.S. and world markets.

The study had three major thrusts:

1) Determination of the solubility of several polymers in selected gases.

2) Continuous web and yarn formation from fibrillated polymer mixtures.

3) Production of oriented fiber web using air flows and aerodynamic forces.

Detailed discussions of the studies carried out in each of these areas, and the results, are presented in the following sections.

III. Technical Activities

  A. Gas-Polymer Solutions

INTRODUCTION

Polymer fabrication often is limited by the high viscosities and low rates of heat transfer of polymers. These limits on fabrication can and do limit applications of polymers. Low viscosity and rapid heat and mass transfer are desirable in transporting and forming polymers into end use products. Extrusion of polymer melts is costly in energy and equipment and contributes to degradation of polymers. Further, extrusion is limited to relatively low melt viscosities. Melt viscosities increase with about the 3.4 power of molecular weight (1). Many properties such as fiber strength are increasing functions of molecular weight at least to some limiting molecular weight (2). Therefore, in many applications polymers with molecular weights greater than those which can be melt extruded would be desirable. A low viscosity dispersion of polymer with rapid heat and mass transfer would be valuable as a transport and fabricating medium. Gas phase solutions of polymers have these desirable properties. Further, gaseous solutions may be suitable media for the flash extrusion of fibers, foamed sheets, etc. For these reasons the solubilities of common, fiber forming polymers in gases were studied.

Phase separations in gaseous solutions of polymers can be seen easily because of the extreme scattering of light in the gas-liquid critical regions. Phase separations usually occur over relatively small ranges of pressure. Therefore, the solubility of a polymer in a gas can be estimated if the volume of the pressure system is known.

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1. Method

Solubilities of gases in polymers were measured observing 'cloud points' as pressure was decreased causing phase separation to occur. In practice, two pressures were recorded, one at which light scattering in the polymer solution became sufficient to distort the laser beam used as a probe and one at which extinction or near extinction was observed.

The method consisted of placing a weighed amount of polymer and a few steel ball bearings in a pressure vessel with windows, closing the vessel, installing the vessel in a pressure system, condensing gas (such as carbon dioxide whose critical temperature is $31^\circ C$) in the pressure vessel containing polymer and in the dosing vessel, heating the pressure system to about the melting point of the polymer, gently rocking the pressure vessel to stir the polymer-gas solution by the gentle rolling of the steel ball bearings, slowly bleeding gas to decrease pressure to cause phase separation, observing the pressure at which the strong scattering of light in the near critical region distorts the laser beam probe and the pressure at which scattering is sufficient to effectively extinguish or drastically distort the beam, changing the temperature, adding gas if necessary, rocking the vessel to stir the solution, and repeating the bleeding process. Pressures were measured using a diaphragm strain gage which was calibrated from time to time with free piston gages. A precise bourdon tube provided occasional checks during solubility experiments on pressures recorded by the strain gage transducer.

The weight of gas in solution with the polymer was estimated using a real gas equation and the unoccupied volume of the pressure vessel. A

capillary (0.007 in, 0.178 mm ID) connected the pressure vessel to the rest of the system. If care was exercised in bleeding the system slowly, little or no polymer extruded from the pressure vessel into other parts of the pressure system. Thus, the capillary served as a connector to the vessel and as a barrier to minimize polymer migration. The volume of gas in solution was the volume of the pressure vessel minus the volume of polymer and the volume of the steel balls. Therefore the weight of gas, in solution, is given by:

$$W_{gas} = \underline{M \cdot P} \; \frac{V_{vessel} - V_{polymer} - V_{bearings}}{z \cdot R \cdot T}$$

where

M = molecular weight of gas

P = pressure

V_{vessel} = measured volume of vessel

$V_{polymer}$ = volume of polymer

$V_{bearings}$ = volume of steel ball bearings

z = compressibility factor of gas

R = gas constant

T = absolute temperature

Volume of polymer was taken as the crystal volume of the polymer; the weight of polymer is divided by an average of the better measurements of crystal density from the literature (3). Steel balls of volume 0.248_3cc were used. Compressibility values for gases were estimated from the good measurements of p-V-T behavior published (4). Weight factions of polymer, $X_{polymer}$, were calculated using the weights of gases from equation 1 and the known weights of polymer added to the pressure vessel, thus

$$X_{polymer} = \frac{W_{polymer}}{W_{gas} + W_{polymer}} \qquad (2)$$

where $W_{polymer}$ = weight of polymer

W_{gas} = weight of gas from equation 1.

Densities of polymer solutions, d_{soln}, were estimated by dividing the sums of weights of polymer and gas by the volume of the pressure vessel minus the volumes of the steel ball bearings

$$d_{soln} = \frac{W_{polymer} + W_{gas}}{V_{vessel} - V_{bearings}} \qquad (3)$$

For approximate measurements of solubilities, the simple method used probably is adequate. Careful measurement of gas added to the system is difficult at best. In any case, estimates of gas concentrations depend on p-V-T data. In the p-T region of these studies z is a strong function of p and T ranging from about 0.88 to 1.25; solubility estimates depend quite strongly on the compressibility factor assumed. In the analysis using equation 1, the contribution of polymer to the observed pressure was assumed to be negligible; the effect of polymer on the compressibility factor of the gas was ignored, and the volume occupied by polymer molecules was taken as the crystal volume of the polymer. Of these assumptions, the first probably is the best; the partial pressure of polymer should be very small compared to that of the gas. The third assumption probably is incorrect but not by more than a few percent. Based upon our knowledge of volumes of small molecules estimated from crystal structures and from gas viscosities or the b in the V-b term in van der Waal's equation, crystal volumes overestimate molecular volumes in gases (5). However, $V_{polymer}$ in equation 1 is proportional to the partial volume of the gaseous polymer in solution; this should be greater than the molecular volume even

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for a component with very small partial pressure. The effect of the polymer molecules upon the compressibility of the gas is not known; we expect that effect should be to decrease z. Large molecules can dissipate collision energies in a multitude of vibrational, bending, and twisting modes. This coupled with the relatively large polarizability of the polymer molecules and molecular segments giving rise to greater attractive forces should cause compressibilities of the mixtures and the gaseous component to be lower than the compressibilities of pure gas. For the foregoing reasons, estimates of solubilities made in these studies should be taken as approximate estimates, not refined measurements.

2. Results

Polycrystalline polymers should be soluble in gases of lower pressures the higher the critical temperatures of the gases and the lower the melting temperatures of the polymers. Solubilities of polymers in the five gas-polymer systems, polypropylene and polybutene-1 in n-butane and carbon dioxide and nylon-6 in carbon dioxide bear out this prediction except that nylon-6 is soluble in CO_2 at lower pressures than might be expected on the basis of melting temperatures of polymers, critical temperatures of gases, and solution behavior of the other systems. Solubility data are given in Table I and Figures 1, 2, and 3.

Polybutene-1 dissolves easily in n-butane at low pressures and temperatures above the melting point of polypropylene (Table I). For this system: pressure increases with increasing temperature at constant concentration, and pressure changes little, if at all, with increasing concentration at constant temperature. Higher pressures are necessary to dissolve polybutene-1 in carbon dioxide (Table I, Figure 1). The p-T-concentration behavior of polybutene-1 in CO_2 solutions is quite different from that in n-butane solutions.

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Table I - SOLUBILITIES OF POLYMERS IN GASES

System	Polymer C	Weight Fraction Polymer	Pressure atm	Temp. C	Density g/cc
Polybutene-1 n-butane	~ 126°	0.051 - 0.20	110 - 140	165° - 198°	0.40 - 0.46
Polypropylene n-butane	162° - 176°	0.055 - 0.21	120 - 170	167° - 190°	0.44 - 0.53
Polypropylene carbon dioxide	162° - 176°	0.067 - 0.38	450 - 950	163° - 208°	0.60 - 0.87
Polybutene-1 carbon dioxide	~ 126°	0.063 - 0.38	300 - 900	131° - 150°	0.53 - 0.91
Nylon-6 carbon dioxide	212° - 225°	0.138 - 0.165	400 - 510	233° - 241°	0.49 - 0.59

Critical properties of solvent gases

Gas	T_c ^{o}C	p_c atm	d_c g/$_{cc}$
Carbon dioxide	31°	72.9	0.468
n-Butane	152°	37.5	0.228
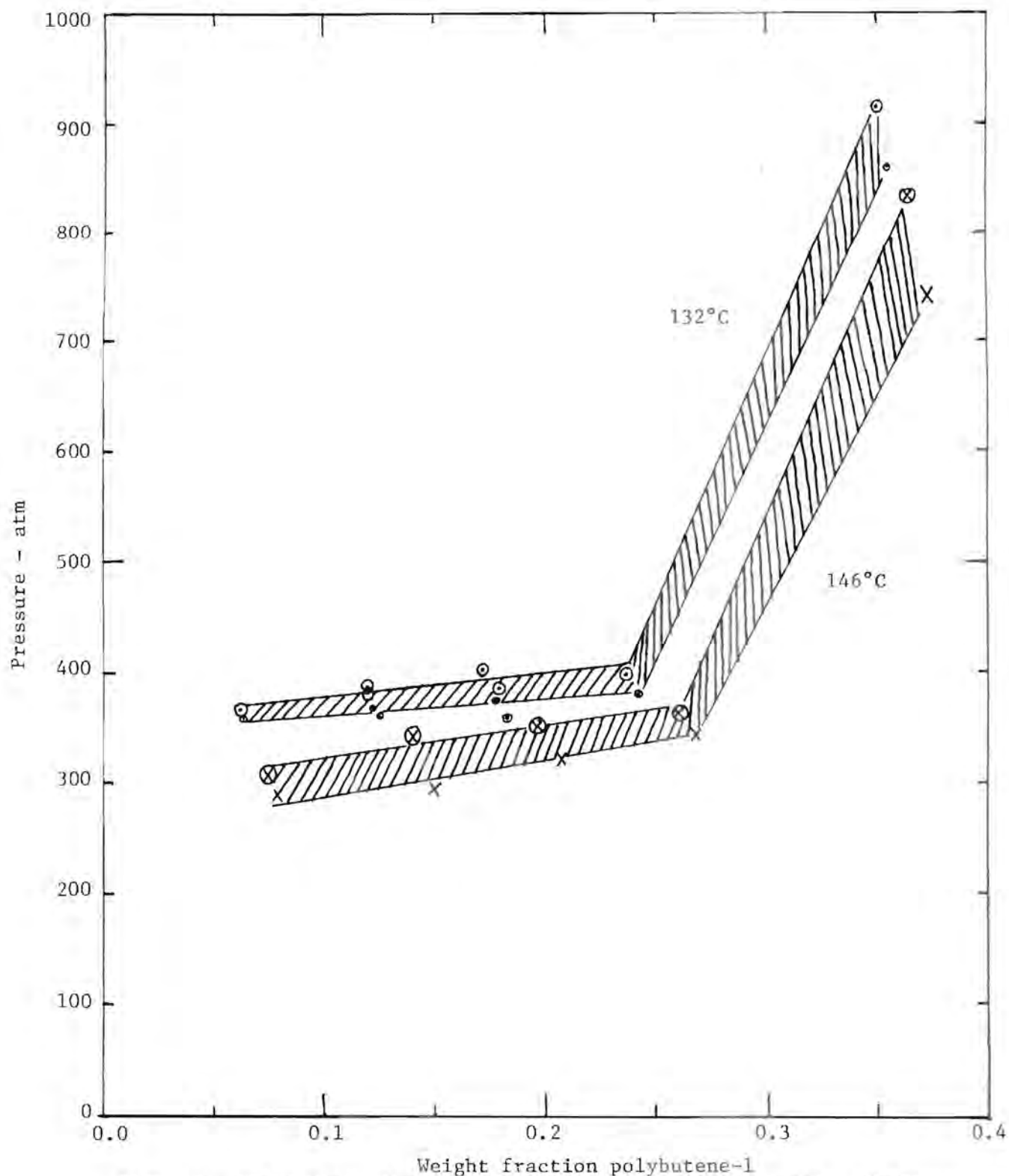
Figure 1   Solubility of Polybutene-1 in Carbon Dioxide: Plot of Pressure Versus Weight Fraction Polybutene at 132° and 146°C. (Upper edges of shaded areas indicates pressures at onset of strong scattering of light characteristic of critical miscibility regions. Lower edges are at pressures at which the laser beam probe was severely distorted or extinguished.)
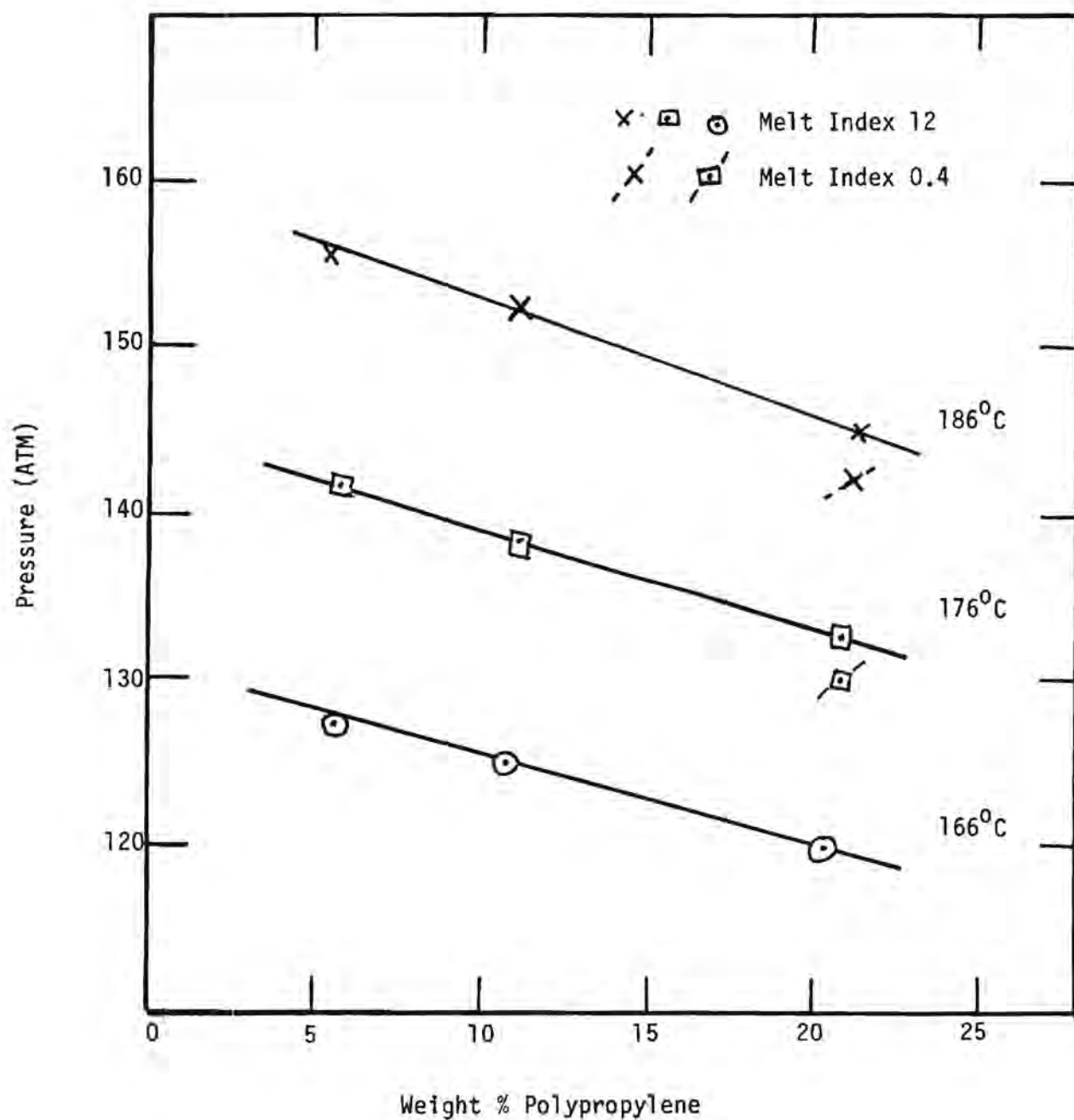
Figure 2 - Solubility of Polypropylene in n-Butane
Plot of Pressure Versus Weight Fraction
Polypropylene at 166°, 176°, and 186°C.
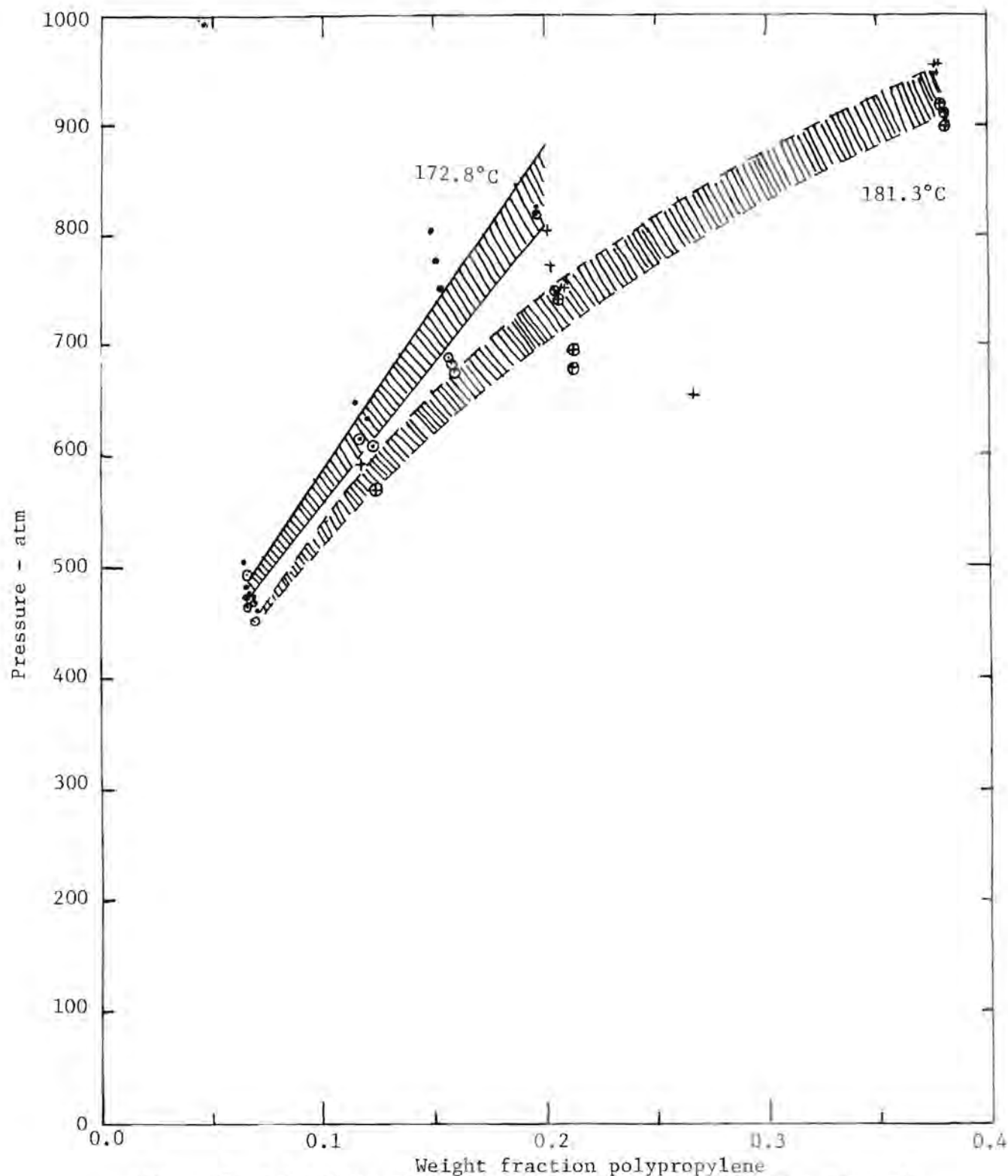
Figure 3 Solubility of Polypropylene in Carbon Dioxide: Plot of
Pressure Versus Weight Fraction Polypropylene at 172.8°
and 181.3°C. (Upper edges of shaded areas indicates pres-
sures at onset of strong scattering of light characteristic
of critical miscibility regions. Lower edges are at pressures
at which the laser beam probe was severely distorted or
extinguished.)

In polybutene-1 in CO_2 solutions, pressure decreases with increasing temperature at constant concentration; solubility increases with increasing temperature at constant pressure, and pressure increases with increasing concentration at constant temperature.

Polypropylene is soluble in n-butane at low pressures and temperatures above its melting point (Table I, Figure 2). Polypropylene and polybutene-1 have similar solution properties in n-butane. For the polypropylene-n-butane system pressure increases with increasing temperature at constant concentrat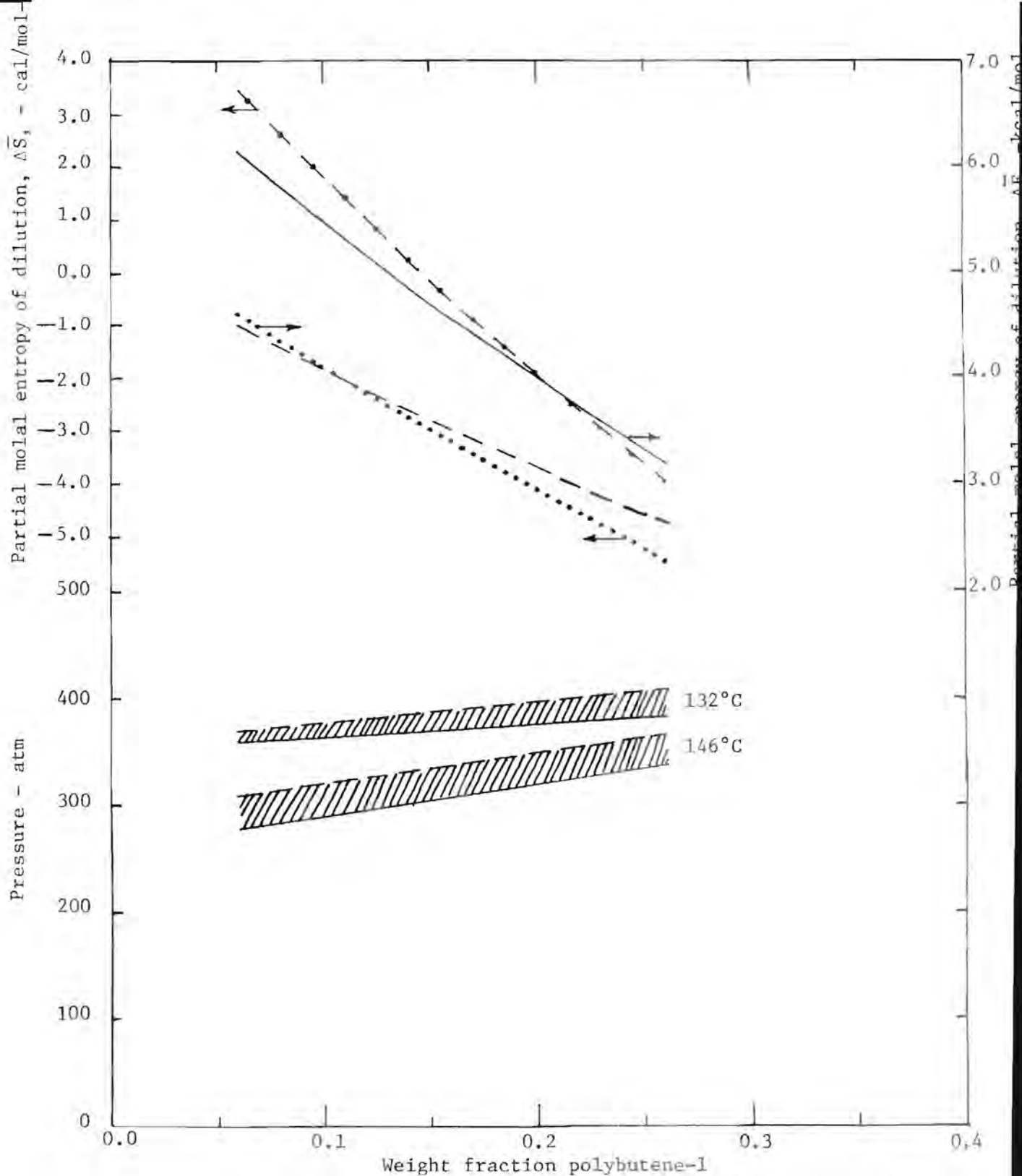ion; solubility of polypropylene decreases with increasing temperature at constant pressure, and pressure decreases with increasing concentration at constant temperature. Polypropylene dissolves in carbon dioxide at considerably higher pressures and at temperatures above the melting point of polypropylene (Table I, Figure 3). Behavior is like polybutene-1 in CO_2 and opposite to that of polypropylene in n-butane. For solutions of polypropylene in carbon dioxide, pressure decreases with increasing temperature at constant concentration; solubility increases with increasing temperature at constant pressure, and pressure increases with increasing concentration at constant temperature.

Nylon-6 is soluble in carbon dioxide at relatively low pressures considering its high melting point (Table I). Solution behavior is like the other polymers in CO_2. For nylon-6 in CO_2, pressure decreases with increasing temperature at constant concentration, and the solubility of nylon-6 increases with increasing temperature at constant pressure. Considering the relatively high softening temperature of nylon-6 ($\sim 212^{\circ}$ to $225^{\circ}C$) and the relatively low critical temperature of carbon dioxide ($T_C = 31^{\circ}C$), solubility at reasonable concentrations at this relatively low pressure is unexpected and gratifying. These data indicate that carbon dioxide dissolves nylon more readily than polypropylene or poly-butene-1.

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If this be borne out by further experiment, carbon dioxide with no dipole but strong quadrapoles (strong bond dipoles) may be a good solvent for relatively polar, hydrogen bonded and, perhaps, other dipole bonded polymers.

Gentle stirring is necessary to dissolve polypropylene in carbon dioxide. Gentle rocking of the pressure vessel through 120^0 about the cylindrical axis at about 0.5 cycles per second for two minutes was quite sufficient to produce what appeared to be homogeneous solutions as judged from the laser beam's passing through the pressure cell without distortion. If the gentle stirring was omitted, the laser was distored at gas pressures well above the critical miscibility region.

Some minimum densities of the gaseous solutions may be necessary to dissolve polymers in the gas phase. Plots of densities versus weight fraction polymer are given in Figure 4. Polypropylene in butane solutions have densities from 0.44 to 0.53 g/cc in the concentration range of $X_{polymer}$ = .055 to 0.21. These densities are about twice the critical density of n-butane, 0.216 g/cc (6). The densities of solutions of poly-butene-1 in carbon dioxide are from 0.53 to 0.91 g/cc in the 0.063 to 0.37 weight fraction polybutene-1 range (Figure 4). The densities of polypropylene in carbon dioxide solutions range from 0.62 to 0.82 g/cc in the 0.069 to 0.22 weight fraction interval (Figure 4). The densities of the nylon-6 in carbon dioxide solution studies at four temperatures and pressures are from 0.49 to 0.59 g/cc in the 0.13_8 to 0.16_5 weight fraction polymer range. These densities are greater than but less than twice the critical density of carbon dioxide, 0.468 g/cc (7). Densities of the three solutions studied over sufficiently wide ranges of concentrations, polybutene-1 in carbon dioxide and in n-butane and polypropylene in carbon dioxide, increase with increasing polymer concentrations as must be expected. Densities of polypropylene in CO_2, polybutene-1 in CO_2, and nylon in CO_2 decrease with increasing temperature. This is
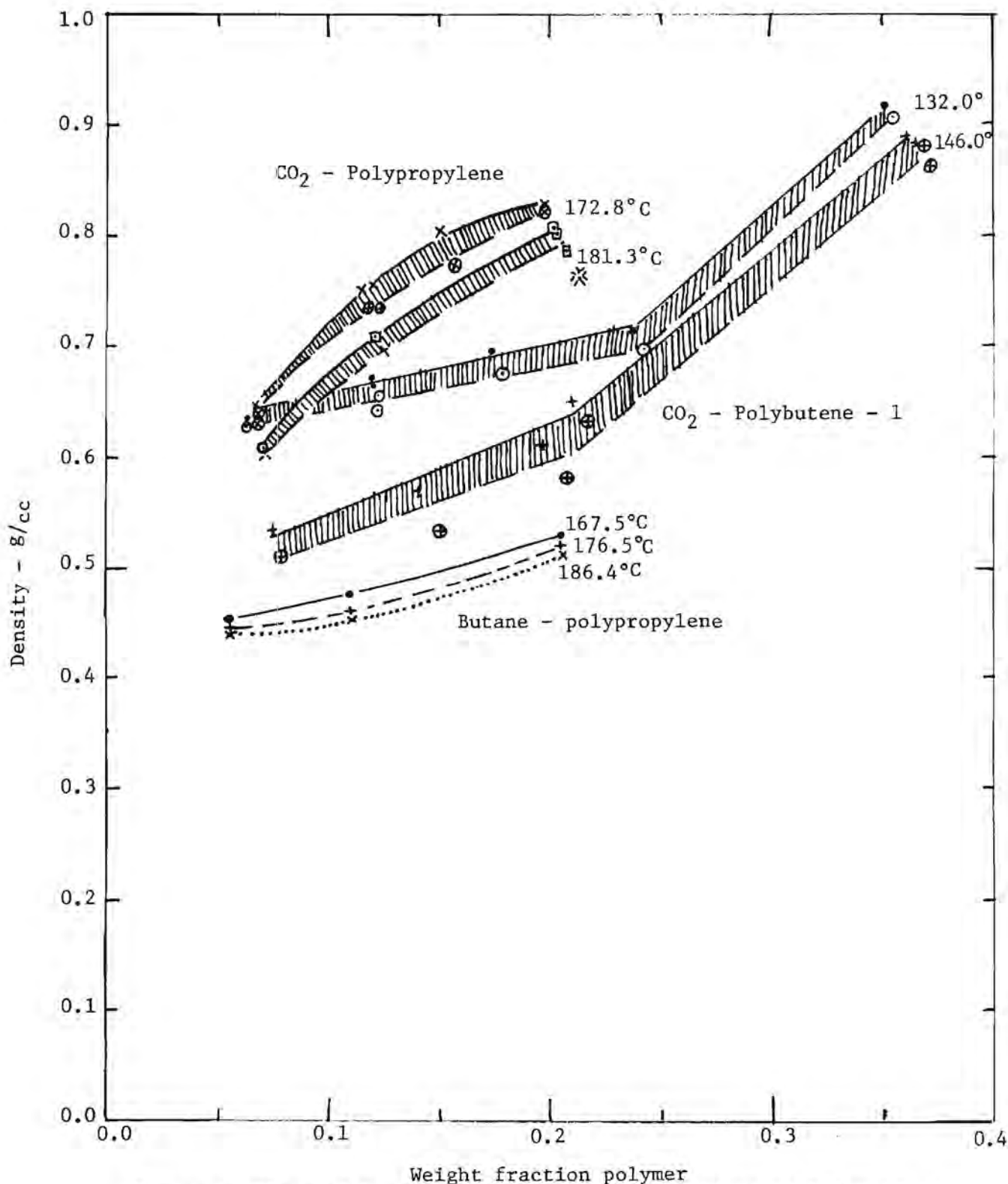
Figure 4   Plots of Densities Versus Weight Fractions of Polymers at
Various Temperatures for Polypropylene Dissolved in Butane
and in Carbon Dioxide and for Polybutene-1 in Carbon Dioxide.
(Upper edges of shaded areas indicate densities at pressures
at onset of strong scattering of light characteristic of critical
miscibility and gas-liquid critical regions. Lower edges are
for densities at pressures at which the laser beam probe was
severely distorted or extinguished.)

Partial molal entropy of dilution, $\Delta \bar{S}_1$ – cal/mol

Partial molal energy of dilution, $\Delta \bar{E}$ – kcal/mol

Pressure – atm

4.0
3.0
2.0
1.0
0.0
−1.0
−2.0
−3.0
−4.0
−5.0

7.0
6.0
5.0
4.0
3.0
2.0

500
400
300
200
100
0

132°C
146°C

0.0   0.1   0.2   0.3   0.4

Weight fraction polybutene-1

Figure 5   Plot of Approximate Changes in Partial Molal Energies and
Entropies of Dilution (Addition of Carbon Dioxide) and
Pressure Versus Weight Fraction Polybutene-1 for Solutions
of Polybutene-1 Carbon Dioxide. (Dashed and dotted lines
refer to pressures at onset of strong scattering of light
characteristic of critical miscibility and gas-liquid
critical regions. Solid lines and ▬●▬ ▬◆▬ line refer to pres-
sures at which the laser beam probe was severely distorted
or extinguished.)

Partial molal entropy of dilution, $\Delta \bar{S}$, cal/mol–°K

Partial molal energy of dilution, $\Delta \bar{E}$, -kcal/mol

Pressure – atm

4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0

8.0
7.0
6.0
5.0
4.0
3.0
2.0

800
700
600
500
400

172.8°C
181.3°C

0.0   0.1   0.2   0.3   0.4
Weight fraction polypropylene
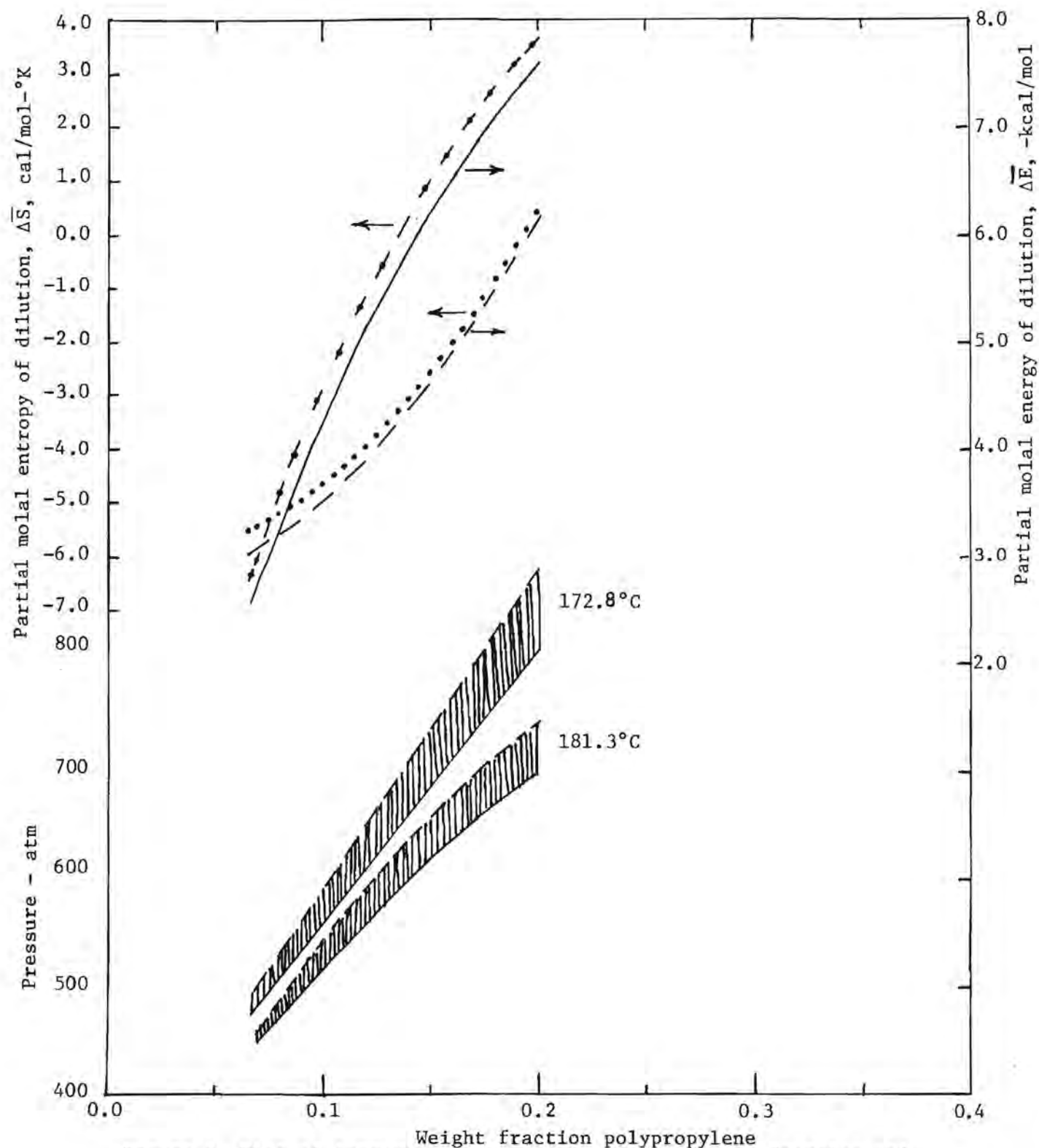
Figure 6   Plot of Approximate Changes in Partial Molal Energies and
Entropies of Dilution (Addition of Carbon Dioxide) and
Pressure Versus Weight Fraction Polypropylene for Solutions
of Polypropylene in Carbon Dioxide. (Dashed and dotted lines
refer to pressures at onset of strong scattering of light
characteristic of critical miscibility and gas-liquid critical
regions.   Solid lines and ⚊•⚊ ⚊•⚊ line refer to pressures at
which the laser beam probe was severely distorted or extinguished.)

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TABLE II

Approximate Energies & Entropies of Dilution

System	Weight Fraction Polymer	Pressure atm	Temperature °C	Partial molal	
				energy-$\Delta\bar{E}$, kcal/mol	entropy-$\Delta\bar{S}$, $\frac{cal}{mol-°K}$
n-Butane-polypropylene	.05 -.20	130-190	166°-186°	$-4._0$	$-18._6$
CO_2 - polypropylene	.067-.20	450-870	172°-181°	$+3._0 \longrightarrow +6._2{}^*$ $+2._6 \longrightarrow +7._6{}^+$	$-5._5 \longrightarrow .4_2{}^*$ $-6._5 \longrightarrow 3._6{}^+$
CO_2 - polybutene -1	.06 -.26	280-410	132°-146°	$+4._5 \longrightarrow +2._6{}^*$ $+6._1 \longrightarrow +3._2{}^+$	$-.6_7 \longrightarrow -5._4{}^*$ $3._5 \longrightarrow -4._0{}^+$
CO_2-nylon-6	$.13_8-.16_5$	400-510	233°-241°	$+5._0{}^*$ $+8._5{}^+$	$-2._3{}^*$ $+4._7{}^+$

*From higher pressures where onset of critical scattering is observed.

+From lower pressures where extreme scattering or extinction of laser beam
  is observed.

consistent with the solubilities of these polymers in CO_2 increasing with temperature at constant concentrations (Figure 3 and 1, Table I). Densities of solutions of polypropylene in n-butane increase with increasing temperature (Figure 4) in keeping with the decrease in solubility of polypropylene in n-butane with increasing temperature and the increase in pressure with temperature at constant concentration. The critical pressure and density of carbon dioxide are higher than those of most gases, by about a factor of two. Thus, to find polymers soluble in carbon dioxide in the density range of 1.05 times to twice the critical density of carbon dioxide at temperatures substantially above the critical temperature of the gas (from 1.332 T_c to 1.684 T_c) is quite consistent with polypropylene dissolving in n-butane at densities about twice to 2.5 times the critical density of n-butane at temperatures not far above the critical temperature of the gas (from 1.028 to 1.075 T_c).

3. Approximate Thermodynamics of Mixing

To a very good approximation, the solubility apparatus used is a constant volume system. Therefore, thermodynamic activity or fugacity of the gaseous solvent should be defined in terms of the partial molal free energy at constant volume, the Helmholtz free energy. Thus, for activity a_1 and fugacity f_1 of gas (8).

$$\Delta \overline{A}_1 = \Delta \overline{E}_1 - T\Delta \overline{S}_1 = - RT \ln a_1 = RT \ln f_1/f_1{}^o \tag{4}$$

where $\Delta \overline{A}_1 \equiv$ change in partial molal free energy at constant volume

$\Delta \overline{E}_1 \equiv$ change in partial molal energy

$\Delta \overline{S}_1 \equiv$ change in partial molal entropy

$a_1 \equiv$ thermodynamic activity of gas

$f_1 \equiv$ fugacity of gas in solution

$f_1{}^o \equiv$ fugacity of pure gas may be represented by an equation of the

from (9)

$$V = \frac{RT}{p} - \alpha(V,T). \tag{5}$$

Remembering that the pressure dependence of the chance in Helmholtz free energy is given by (10)

$$\left(\frac{\delta A}{\delta V}\right)_T = p \tag{6}$$

the fugacity at constant volume may be calculated using the equation (at constant temperature)

$$RTd\ln f = pdv \tag{7}$$

by substituting the derivative of equation 5 (at constant temperature)

$$dV = -\frac{RT}{p^2}\,dp - \left[\frac{\delta\,\alpha\,(V,T)}{\delta V}\right]_T dV \tag{8}$$

in equation 7 and integrating. Thus for the fugacity

$$RT\int_{V^*}^{V} d\ln f \quad RT\int_{p^*}^{P} \frac{dp}{p} - \int_{V^*}^{V}\left[\frac{\delta\alpha(V,T)}{\delta V}\right]_T dV \tag{9}$$

where V^* is the volume as p approaches zero; then

$$RT\ln f - RT\ln f^* = RT\ln p - RT\ln p^* - \int_{V^*}^{V}\left[\frac{\delta\alpha(V,T)}{\delta}\right]_T dV \tag{10}$$

In the limit of infinite volume or zero pressure, all gases behave ideally so that the second term of equation 5, $\alpha(V,T)$, approaches zero and the fugacity approaches the pressure. Thus the fugacity may be calculated from

$$RT\ln f = RT\ln p - \int_{0}^{p}\left[\frac{\delta\,\alpha\,(V,T)}{\delta}\right]_T dV \tag{11}$$

As a standard state, the hypothetical ideal gas state at one atmosphere, which differs insignificantly from the real gas at one atmosphere for the gases used in these studies, is convenient.

In order to estimate the partial molal energies of solution of gas in the solutions, the temperature dependence of the partial molol Helmholtz free energy is calculated using the equation (10).

$$\left(\frac{\delta \frac{A}{T}}{\delta T}\right)_V = -S \tag{12}$$

which leads to

$$\left[\frac{\delta(\bar{A}_1/T)}{\delta(1/T)}\right] = \Delta\bar{E}_1 = R\left[\frac{\delta \ln f_1}{\delta(1/T)}\right]_V = R\left[\frac{\delta \ln p_1}{\delta(\frac{1}{T})}\right]_V - \int_{p=0}^{P} \frac{\alpha(V,T)}{dV}\, dV - \frac{1}{T}\frac{\delta}{\delta(\frac{1}{T})}\int_{p=0}^{P}\left[\frac{\delta\alpha(V,T)}{\delta(V)}\right]_T dV \tag{13}$$

The partial molal entropy is given by

$$\Delta\bar{S}_1 = \frac{\Delta\bar{E}_1}{T} - \frac{\Delta\bar{A}_1}{T} = \frac{R}{T}\left[\frac{d\ln p_1}{d(1/T)}\right]_V - \frac{1}{T}\frac{\delta}{\delta(1/T)}\int_{p=0}^{P}\left[\frac{\delta\alpha(V_1 T)}{\delta V}\right]_T dV - R\ln p_1 \tag{14}$$

The partial molal energies of solution of gas in solution (often called partial molal energies of dilution) were estimated by interpolating pressures and compositions at constant temperatures for specified concentrations, $X_{polymer}$ = .06, .10, etc, and then determing the dependence upon reciprocal temperature of lnp. Linear least squares fits were used for interpolation and determining slopes of lnp with 1/T. Ignoring the nonideality terms, the $\alpha(V,T)$ terms, in equations 13 and 14 probably is not too serious because these do not change much with temperature and pressure over the small ranges of temperature and pressure used in these studies.

The smoothed pressures of the onset of obvious critical opalesesence and the extreme scattering together with partial molal energies and entropies

of dilution (adding gas) are shown as functions of weight fraction polymer for gaseous solutions of polybutene-1 and polypropylene in carbon dioxide in Figures 5 and 6. In both cases partial molal energies of dilution are positive which is to say solubilities increase with increasing temperature. Partial molal energies and entropies of dilution decrease with increasing concentration of polybutene-1 (Figure 5); these increase with increasing concentration of polypropylene (Figure 6). Since entropy and energy vary so as to minimize the departure of the free energy from zero, the partial molal entropies of dilution behave as do the energies of dilution. The partial molal energies of dilution, $\Delta \overline{E}_1 \sim$ 3 to 6 kcal/mol, and the partial molal entropies of dilution, $\Delta \overline{S}_1 \sim$ -6 to +4 cal/mol-^{0}K, are quite comparable in magnitude for both systems; they differ only in their concentration dependence (Figures 5 and 6, Table II).

An approximate estimate of the lower bounds on the partial molal energies and entropies of dilution at about 0.15 weight fraction nylon in carbon dioxide can be made from the solubility observations at four temperatures in the single run reported. These are for the upper pressure curve indicating the onset of critical region scattering $\Delta E_1 \sim$ 5 kcal/mol and $\Delta S_1 \sim$ -2.$_3$ cal/mol - ^{0}k and for the lower pressure curve indicating severe scattering $\Delta \overline{E}_1 \sim$ 8.$_5$ kcal/mol and $\Delta \overline{S}_1 \sim$ + 4.$_7$ cal/mol - ^{0}K (Table II). When data are available so that interpolation in concentration can be made, these values probably will be revised downward.

The partial molal energy of dilution of n-butane in solutions of polypropylene is negative, $\Delta \overline{E}_1 \sim$ - 4.$_0$ kcal/mol, and appears to be independent of concentration in the 0.06 to 0.26 weight fraction polypropylene range (Table II). As is to be expected with a negative partial molal energy of solution, a

strongly negative partial molal entropy of dilution, $\Delta \bar{S}_1 \sim -18._6$ cal/mol - ^{O}K, is observed.

The foregoing thermodynamic data are estimated from temperature derivatives of smoothed experimental data (interpolated for small differences in concentration); therefore, the apparent precision of these data is illusory. The thermodynamic data given are approximate only because of scatter of experimental data, assumptions about polymer volumes and compressibilities of gases in calculating concentrations, and neglecting departures from ideality in estimating temperature derivities of the logarithms of fugacities.

4. Corresponding States Correlation of Solubility Data

As a first approximation polymers may be expected to be soluble in gases at similar gas densities which is to say at similar reduced temperatures and pressures. This is a corresponding states argument. The gas pressure necessary to dissolve a polymer increases with temperature. As a first approximation this increase of pressure with temperature may be assumed to be linear; therefore, reduced pressure of phase separation may be proportional to reduced temperature. Solubility data in terms of reduced pressure P/P_c and reduced temperature T/T_c and the quotient of reduced pressure divided by reduced temperature are given in Table III.

The quotients of reduced pressure divided by reduced temperature at phase separation increase with concentration. The range of values is from 2.8 to 8.9 with the high values for polybutene-1 and polypropylene in carbon dioxide solutions at higher concentrations. At approximately the same concentration (~ 15 percent polymer by weight) at phase separations, the quotients of reduced pressure divided by reduced temperature range from

Table III.   Corresponding States Correlation of Solutibilities:

Reduced Pressures Divided by Reduced Temperatures of Phase Separation

Polymer	Gas	Concentration weight fraction	$\left(P/P_c\right)\Big/\left(T/T_c\right)$ at phase separation
Polybutene-1	n-butane	0.051 - 0.20	2.8 - 3.4
Polypropylene	n-butane	0.055 - 0.21	3.1 - 4.1
Polybutene-1	CO_2	0.063 - 0.38	3.1 - 8.9
Polypropylene	CO_2	0.067 - 0.38	4.3 - 8.2
Nylon-6	CO_2	$0.13_8 - 0.16_5$	3.3 - 4.1

Polymer	Gas	Concentration weight fraction	$\left(\dfrac{T}{T_c}\right)$	$\left(P/P_c\right)\Big/\left(T/T_c\right)$ at phase separation
Polybutene-1	n-butane	$\sim$ 0.15	1.042	3.0
			1.066	3.1
			1.089	3.3
Polypropylene	n-butane	$\sim$ 0.15	1.033	3.2
			1.056	3.4
			1.080	3.7
Polybutene-1	CO_2	$\sim$ 0.15	1.333	3.8 - 4.0
			1.379	3.0 - 3.4
Polypropylene	CO_2	$\sim$ 0.15	1.467	6.4 - 6.9
			1.495	5.7 - 6.0
Nylon	CO_2	$\sim$ 0.15	1.664	3.3
			1.691	4.1

3.0 to 4.1 except for polypropylene dissolved in carbon dioxide for which these quotients are from 5.7 to 6.9 (Table III). The implication from these data is that polypropylene has less tendency to dissolve in carbon dioxide than do nylon or polybutene-1 and polybutene-1 or polypropylene in n-butane.

No simple correlation of reduced pressures and reduced temperatures or the quotient of these at phase separations can be made on the basis of these few data. However, the low values of $(P/P_c)/(T/T_c)$ for solutions of poly-butene-1 and polypropylene are consistent with the observation that "like dissolves like" as regards to chemical structure. The carbon dioxide solutions do not show this simple relationship. Although carbon dioxide is symmetrical and hence nonpolar, it has strong bond dipoles; therefore, it may be more effective as a solvent for polar materials than are other non-polar gases. This can account for the relatively low reduced pressures at which nylon dissolves in carbon dioxide. The rather high reduced pressures necessary to dissolve polypropylene in carbon dioxide, as compared to polybutene-1 in carbon dioxide, cannot be explained by any simple observa-tions on solutility.

5. Measurements at High Temperatures

Solubilities of higher melting fiber forming polymers such as nylon-66 and poly(ethylene terephthalate) in gases have not been measured. Repeated attempts to make more measurements of the solubility of nylon-6 in carbon dioxide failed. At temperatures above about 190^oF, carbon dioxide appears to be a sufficiently good solvent for materials from which O-ring gaskets can be made that leaks around the windows of the pressure vessel were the rule in most experiments.

B. Pressure Vessel Development

In order to be reasonably certain that solutions are homogeneous and
to observe the onset of phase separation, visual observation is almost
essential. This requires windows with glass to metal seals which do not
leak at pressures and temperatures of the experiments. The pressure vessel
used in these solubility studies has windows sealed with polymeric O-rings.
These simple O-ring seals usually are gas tight, within the limits of
pressure versus time measurements in these studies, at temperatures below
about $190^{o}C$ using polychlorobutadiene rubber O-rings. Above about $200^{o}C$,
gas tight seals are rare even when using perfluorocarbon rubber (Kalrez)®
O-rings.

The only commerically available pressure vessel containing windows
designed for operation in the desired temperature and pressure range and
which could be obtained within any reasonable time did not hold gases.
The quartz to metal seals were ineffective. In this pressure vessel the
quartz to metal seals are made without gaskets as the quartz windows are
pushed against the covers of the vessel by the compressed fluid within
the vessel. Plating silver on the covers and evaporating and sputtering
silver onto the portions of the faces of the windows in contact with the
covers to provide a deformable metal gasket was not successful in producing
a gas tight seal.

Development of successful glass to metal seals which do not leak at
pressures up to 2000 atm and temperatures up to $300^{o}C$ are necessary in order
to continue the studies of solubilities of nylon-6 in carbon dioxide and to
extend the studies to nylon-66 and poly(ethylene terephthalate) in carbon
dioxide.

C. Extrusions from gaseous solutions

Attempts to make fine fibers by extruding polymers from the gas phase
were not successful. In most cases no fibers were produced; in a few cases
granular polymer plus a few, fine fibers were obtained.

Extrusions were made with polypropylene and carbon dioxide at temperatures
from 170° to 195° and pressures from 750 to 1330 atm. Orifice diameters of
0.018 and 0.036 cm and length to diameter ratios of 18, 150, 1400 and 14000
were used. Attempts with nylon and carbon dioxide were with orifice diameter
of 0.018 cm and length to diameter ratios 1400 and 14000. Temperatures
were 260° to 270^oC and pressures from 680 to 990 atm. Nominal concentrations
were in the ten and twenty percent by weight range.

Failures in these experiments were caused by blockages of orifices, no
or incomplete dissolution of polymers in gases, and perhaps by the tendency
of polymers to precipitate as granular aggregates rather than fibers. The
volume of the polymer solution cell in the extrusion apparatus is ten times
that of the optical cell in which studies are made. Dissolution of small
amounts of polymer in the small, optical cell by gentle rocking with bearings
for stiring is easy. Rather violent rocking was necessary with the larger cell.
The larger volume cell was used purposely so that some polymer solution could
be extruded without decreasing pressures sufficiently to cause phase separation
in the cell. With the larger cell agitation by rocking is inadequate to
dissolve the polymers. Use of a flow system with sufficient flow rates and
through-puts would permit use of flow agitation and permit extrusion without
decreasing pressures sufficiently to cause phase separation.

Fine fibers of polypropylene and nylon-6 were produced early in the
solution studies by bleeding solutions of polypropylene in n-butane and

nylon-6 in carbon dioxide to the atmosphere. Fiber diameters from about 2 to 10 microns with some as large as 25 microns were measured from electron micrographs for nylon extruded from CO_2 solution. Whether fibers were formed as polymer was precipitated or in subsequent steps is not known. The process remains an attractive possibility for fiber formation in spite of the failure of these studies to elucidate and scale-up the process.

Summary of Conclusion

1) Polymers are soluble in dense gases at lower pressures the lower the melting point of the polymer and the higher the critical temperature of the gas. Measurements of solubilities of polybutene-1 and polypropylene in n-butane and carbon dioxide and nylon-6 in carbon dioxide support this expectation.

2) Polybutene-1 and polypropylene are readily soluble in n-butane at relatively low pressures, about 120 atmospheres and upwards, at temperatures above the melting points of the polymers.

3) Polybutene-1 and polypropylene are soluble in carbon dioxide at higher pressures, from about 450 to 900 atmospheres, at temperatures above the melting points of the polymers.

4) Nylon-6 dissolves in carbon dioxide at 400 to 500 atmospheres and 233^0 to 241^0C Carbon dioxide appears to be a good solvent for this polar polymer.

5) Carbon dioxide is a solvent for polar and nonpolar polymers. This is consistent with its having no dipole but strong quadrapoles.

6) Partial molal energies of solution of CO_2 in these gaseous polymer solutions are positive, from about three to nine kilocalories. For propane-polypropylene solutions, $\Delta \bar{E} \sim -4$ kcal/mol, and the partial molal entropy of dilution is $\Delta \bar{S} \sim -19$ cal/mol-^{0}K. $\Delta \bar{S}$ is from ~ -7 to $\sim +5$ cal/mol-^{0}K for CO_2 in its solutions.

7) Comparing solutions at about 15 percent polymer by weight using a corresponding states argument indicates that polypropylene is less readily soluble in carbon dioxide than in n-butane and the other polymers in CO_2 or n-butane. For polypropylene in CO_2, $(P/Pc) / (T/Tc) \sim 5.7$ to 6.9; for the other four systems (polypropylene in n-butane, polybutene-1 in CO_2 and n-butane, and nylon-6 in CO_2), $(P/Pc)/(T/Tc) \sim 3.0$ to 4.1.

8) Attempts to measure solubilities of higher melting polymers in gases were not successful because of leaks in glass to metal seals. Gases, especially carbon dioxide, are good solvents for polymers; they cause failure in elastomaric O-ring seals.

9) An attempt to make a glass to metal seal by sputtering silver on windows and plating silver on metal was not successful. No organic O-rings make gas tight seals with any consistency at temperatures above about $200^{o}C$.

10) Attempts to scale up extrusions of gaseous polymer solutions to produce fibers were not successful. Fine fibers of polypropylene and nylon-6 were produced in solution studies by bleeding solutions to the atmosphere.

B. Foam Fibrillation

1. Introduction

Foam fibrillation provides a mechanism for directly converting polymer into webs or yarns. By comparison ten to twenty processing steps are necessary to convert staple fiber to woven fabric and at least six steps are needed to make knitted fabric from continuous filament. The most costly of these processing steps are yarn formation from staple fiber and fabric formation by weaving.

The purpose of this investigation is to explore the potential for foam fibrillation to directly form yarn or fabric suitable for garment applications. Direct formation would reduce the number of processing steps in converting polymer to garment. Consequently, foam fibrillation should provide savings in materials, energy and labor compared to conventional textile processing. In addition, foam fibrillation should reduce the capital investment and manufacturing cost in converting polymer to garment.

The ultimate goal is to convert polymer directly into garment. Thus, a considerable portion of this research is devoted to exploring the potential of foam fibrillation for directly forming fabric suitable for garment applications. It will be shown that the webs formed by foam fibrillation are not suitable for garment applications at their current state of development.

Since yarn formation from staple fibers represents an expensive sequence of processing steps, the potential for forming yarns by foam fibrillation was investigated. It will be shown that foam fibrillated yarn comparable to commerical staple fiber yarns can be formed inexpensively.

2. Background

Fibrillation is commercially practiced as an alternative to spinning staple

fiber or extruding continuous filament yarns. Two diverse fibrillation techniques for forming yarns are flash spinning and film fibrillation. In flash spinning a solvent or carrier rapidly evaporates as the polymer solution or blend leaves the spinneret. The liquid to vapor expansion biaxially strains and orients the polymer and splits the extrudate into a web of fibrils. This bundle of fibrils is quite similar to a yarn. The flash spun yarn has more surface area than an equivalent weight of conventional yarn. Thus, the fabric basis weight can be reduced without sacrificing fabric opacity. Normally, the flash spun web is laid down "randomly" and melt bonded to give a rather inflexible and coarse "spun-bonded" fabric or synthetic paper (11). Woven spun yarn can be as flexible as woven cotton (12). Because of the high cost of weaving, the woven fabric is considerably more expensive than the melt bonded fabric.

In film fibrillation an extruded and hot stretched film is slit and mechanically fibrillated to produce yarn. Also, fabric can be produced by excluding the slitting step and laterally stretching the oriented film. Unlike flash spun yarn, a significant number of large fibrils are obtained in film fibrillation; these increase the opacity and impart excessive rigidity to the resulting fabric (13). Therefore, fabrics produced from fibrillated films also are not suitable for use in garments.

Flash spun yarns do have suitable properties for garment use. However, the use of mixed solvents in flash spinning results in expensive solvent recovery systems. This makes imperative the use of the low cost random laydown, melt bonding technique in making fabrics. Melt bonding of fibers produces relatively hard nodules in the nonwoven fabrics, these make the fabric quite stiff (14).

Basic studies of the mechanism of fibrillation during polymer drawing suggest that virtually all drawn polymers are microfibrous (15, 16, 17). However,

the microfibrous texture of drawn polymers is not readily apparent because the microfibrils can coalesce through shear heating or high temperature annealing (16). For example, drawing at higher temperatures produces larger fibrils due to microfibrils coalescing. In flash spinning, the expansion of the solvent prevents the fibrils from coalescing. However, it is not necessary to use a costly vaporizing solvent to obtain biaxial drawing.

The objective of this research is to produce by direct extrusion yarns and fabrics which will be sufficiently flexible for garment applications. The existing technology suggests that fabrics comprised exclusively of micro-fibrils would meet the desired objective. Also, basic studies indicate that polymers are inherently capable of forming the desired microfibrillar web without the use of volatile solvents. The flash spun yarn process provides the desired texture but at a relatively high cost. The film fibrillation process is economically more attractive but does not provide the desired texture. Therefore, development of processes which fill the considerable gap between the flash spun yarn and fibrillated film processes are sought. The following modifications of the flash spinning and film fibrillation processes are suggested:

1) Extrusion from a circular die followed by biaxial stretching in order to eliminate the random laydown and melt bonding steps in conventional flash spinning which results in a rigid fabric.

2) Substitution of inert gases, inorganic fillers and incompatible polymers for solvents to promote concurrent fibrillation and biaxial expansion at lower processing costs.

The result of these suggestions is the extrusion of a polymer foam through a circular die followed by biaxial stretching of the extrudate over a mandrel. Both the foaming of the extrudate and biaxial stretching facilitate fibrillation,

leading to the direct formation of a web or yarn. The process is described in greater detail in the next section.

3. Process Description

The processing route adopted for forming webs by foam fibrillation consists of (a) blending polymer flake with a chemical blowing agent, (b) extruding the blend through a circular die and (c) biaxially expanding the foam extrudate over a conical mandrel. This processing route is analogous to blown film extrusion in which a conical mandrel is substituted for air blowing. A schematic diagram of the process is shown in Figure 7.

Specifications for the equipment utilized in studying this process are given in Table IV. The fibrillation mandrel shown in Figure 7 is shown in greater detail in Figure 8. A number of modifications to this mandrel were examined and are discussed in section B.6.e.

By altering the extrusion conditions a yarn rather than a web could be formed. The as-extruded yarn was subjected to additional twisting and drawing, both by hand and continuously. As illustrated in Figure 9, continuous twisting was accomplished by taking yarn off a bobbin rotating about a vertical axis onto a bobbin rotating about a horizontal axis. The pay-off bobbin was free to rotate about a horizontal axis at approximately the same periferal speed as the take-up bobbin. Continuous drawing was accomplished by passing yarn through a heated box under conditions such that the periferal speed of the take-up rolls exceeded the periferal speed of the feed rolls - see Figure 10.

4. Materials

The principal materials studied are polypropylene and azodicarbonamide blowing agent. The particular grade of polypropylene is Hercules Profax 6323. To facilitate blending the polymer was supplied in flake form. Hercules Profax 6323 has a melt index of 12 and is a linear, isotactic homopolymer.

POLYMER FLAKE
AND ADDITIVES

CIRCULAR
DIE

DRIVE

EXTRUDER

AIR-COOLING RING

FIBRILLATION MANDREL

DRIVE ROLLS
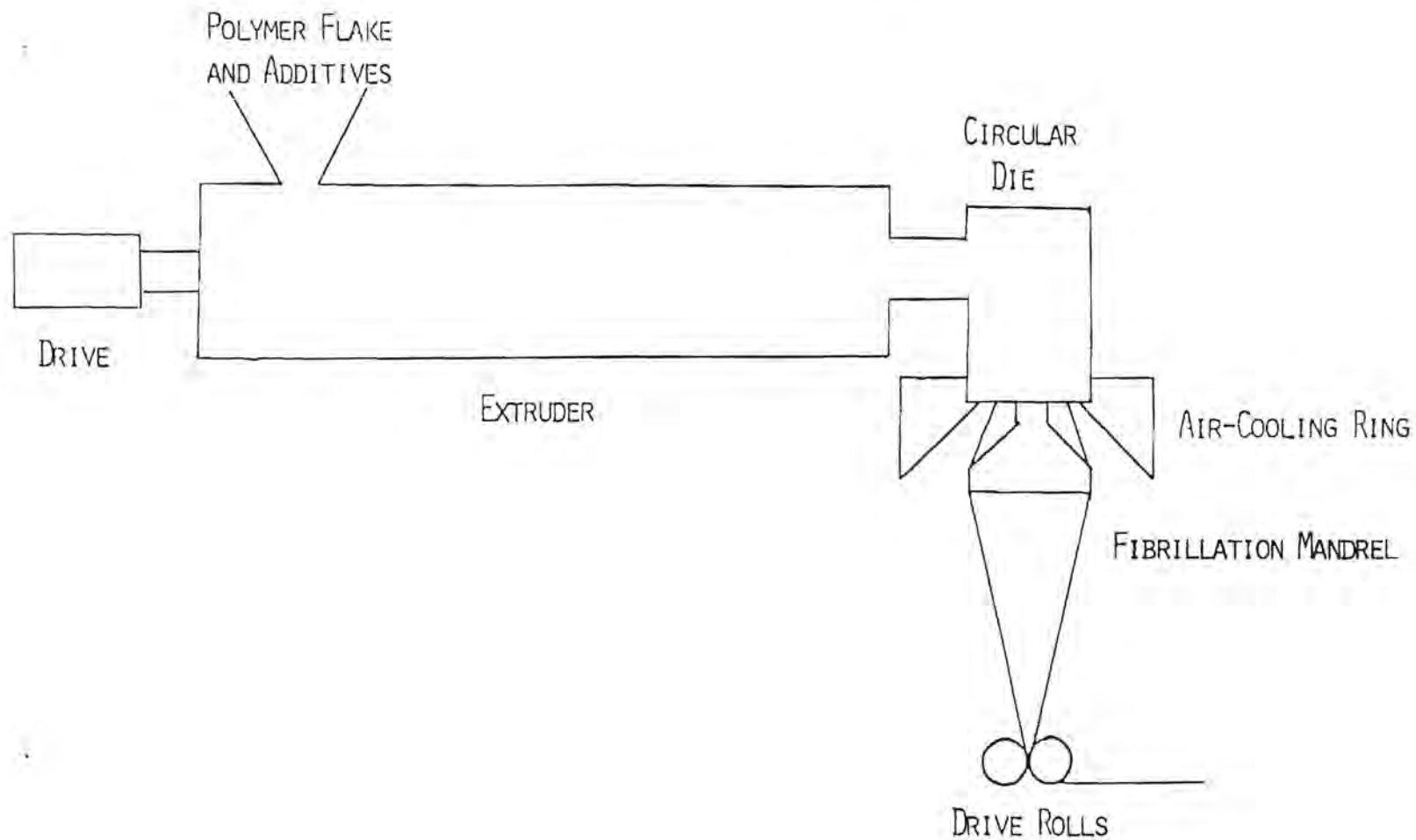
Figure 7. Extruder Unit with Mandrel
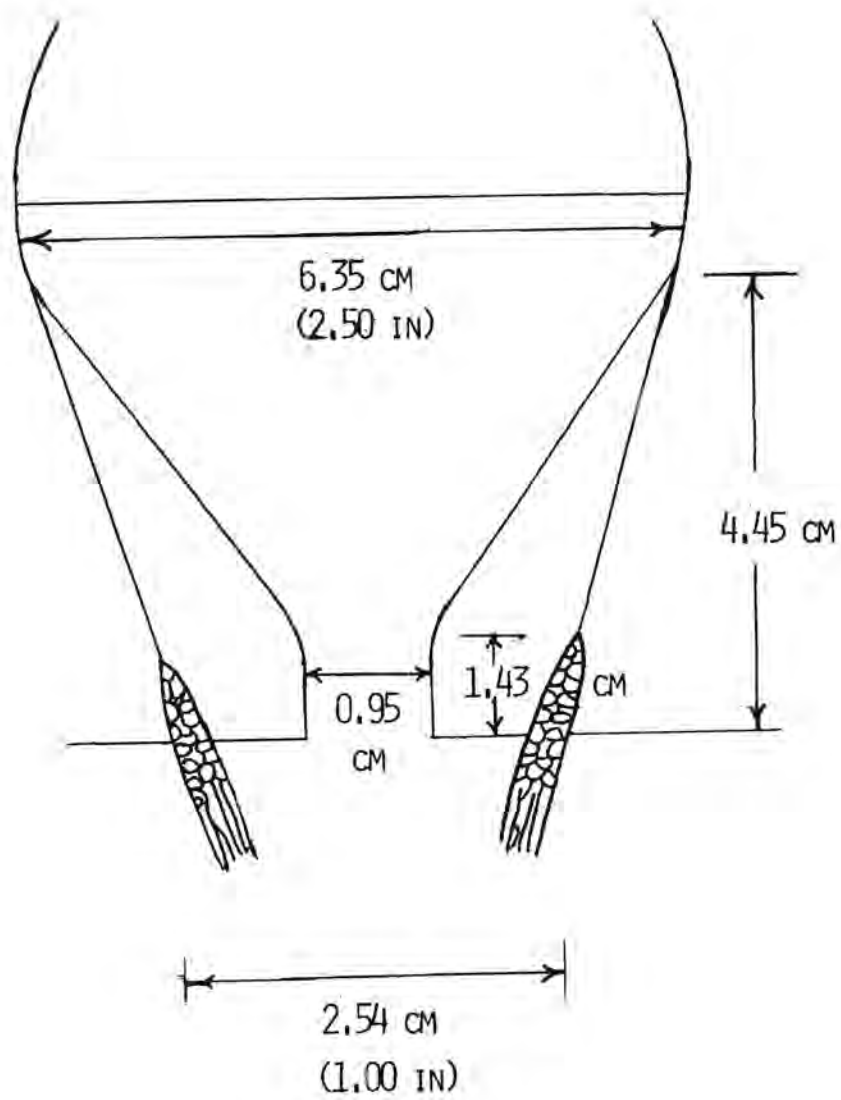
FIGURE 8. FIBRILLATION MANDREL

VARIABLE SPEED DRIVE

PAY-OFF BOBBIN

TAKE-UP
BOBBIN

VARIABLE SPEED DRIVE
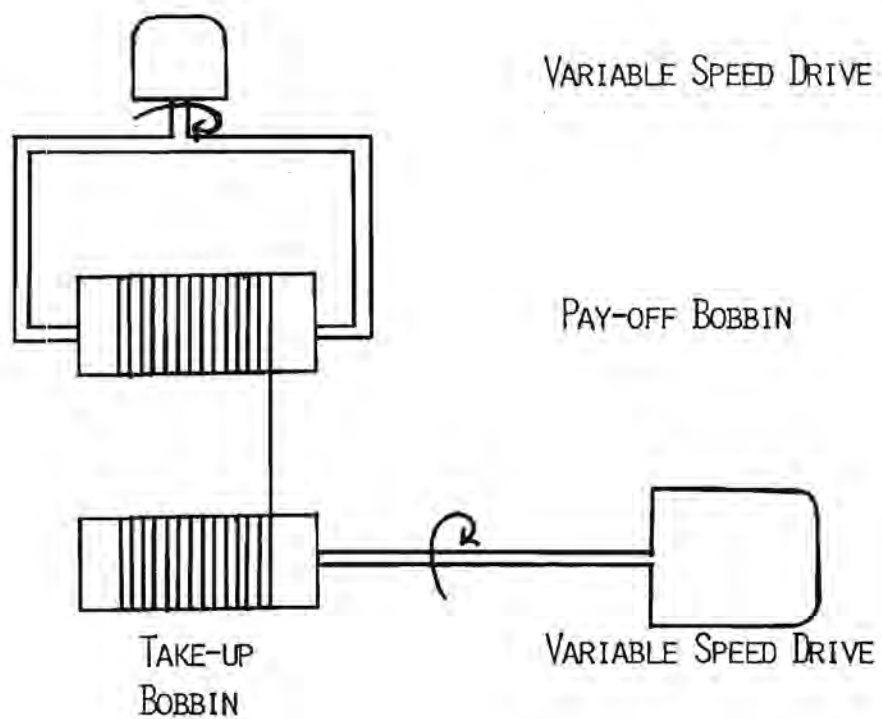
FIGURE 9. TWISTING APPARATUS
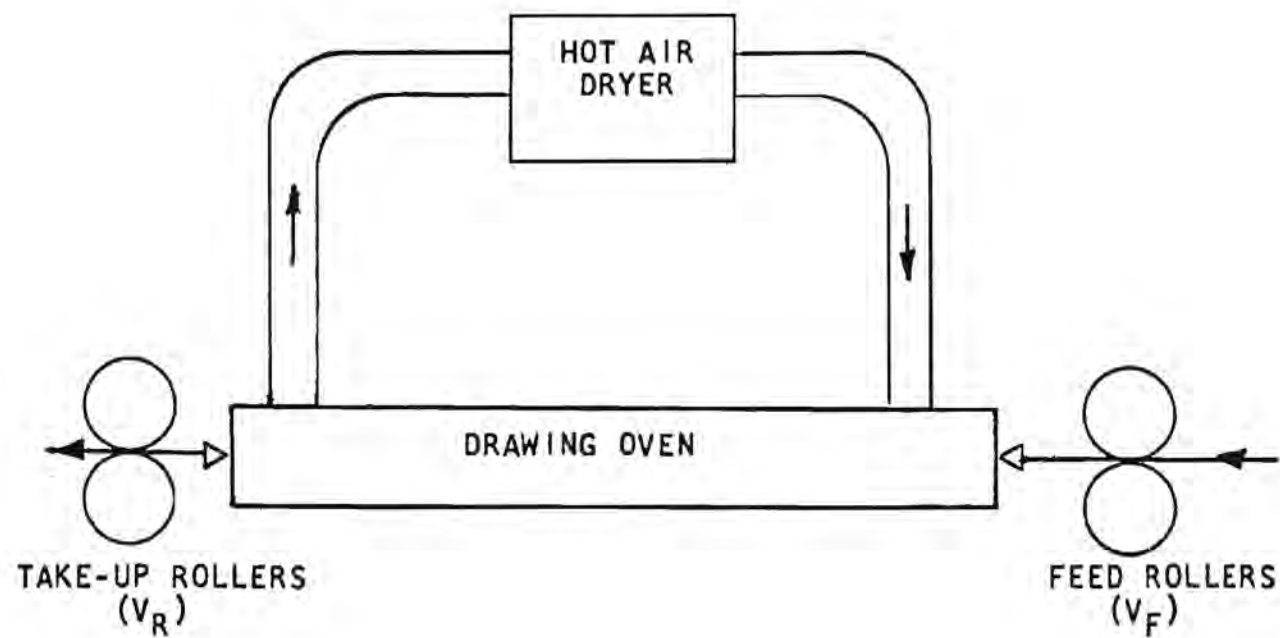
Figure 10. Continuous Drawing System

TABLE IV

EXTRUSION SYSTEM SPECIFICATIONS

· 3/4"D, 25 TO 1 L/D, 3 TEMPERATURE CONTROL ZONES

      (RHEOMIX 254, HAAKE, INC., CAT. No. 23-13-000)

· 3 TO 1 COMPRESSION RATIO, CONSTANT TAPER SCREW

      (HAAKE, INC., CAT. No. 23-00-033)

·1" OD, 0.92" ID DIE

      (HAAKE, INC., CAT. No. 23-00-55L)

· BLOWN FILM TAKE-UP SYSTEM

      (HAAKE, INC., CAT. No. 24-20-000)

Other polymers studied include various grades of polypropylene covering a range in melt index, polybutene-1, poly (tetramethylene terephthalate) and nylon 66.

The azodicarbonamide blowing agent most frequently used was Kempore 200MC supplied by Stepan Chemical Co. This blowing agent is nucleated, according to the manufacturer, and has an average particle size of 200 mesh. A variety of other blowing agents were studied, including different grades of Kempore azodicarbonamide, carbon dioxide and nitrogen. In addition, a number of additives for improving the uniformity of fibrillation were examined.

5. Testing

The primary quantitative method for evaluating webs and yarns is tensile testing to obtain tenacity and elongation. In order to minimize the direct influence of basis weights and linear densities on breaking strengths, the tenacities of the webs and yarns are reported in (lb/inch width)/(oz/sq. yd) and g/denier respectively. The breaking strengths and elongations were determined using an Instron Testing Machine, a 5" gage length and a 100%/ minute extension rate. For small webs the gage length was reduced to 2" but the extension rate was maintained at 100%/minute. The web samples were cut to a width of 1 1/2" for testing.

Basis weights were obtained by weighing four samples 8" long by 1 1/2" wide. Denier was determined by weighing samples two meters long.

Air permeability measurements were performed in accordance with ASTM D737. Flexural rigidities were determined using an FRL Cantilever Bending Tester in accordance with ASTM D 1388. Filament diameters were measured using an optical microscope equipped with a micrometer disc. Average filament deniers were determined by counting filaments in a yarn cross-section. Birefringence was determined by the oil immersion method (18) using a polarized light microscope.

Filament density was measured by the flotation method (19) in solutions of water and methanol using a hydrometer. In addition, selected yarns were studied by x-ray diffraction, scanning electron microscopy and light microscopy.

6. Web Formation

   a. Initial process development

Prior to obtaining the blown film tower and die described in Table IV, a slit die was used to extrude polypropylene containing fillers. The principal fillers used were silicon dioxide and calcium carbonate powders up to 10% by weight. Then these samples were drawn at room temperature. The fillers increased the degree of stress whitening in drawing, suggesting fibrillation occurred on a finer scale. Gross separation of the fibrils did not occur because only uniaxial drawing was performed.

Once the blown film equipment was in place, blown film extrusion of the filled polypropylene was attempted in order to facilitate fibril separation. Film formation was irratic because gross splits would develop. A fibrous web could not be formed.

At this stage the conical mandrel was substituted for air blowing in order to fibrillate the film extrudate. However, filled polypropylene could not be fibrillated in this manner. Finally, chemical blowing agents were substituted for inorganic fillers and it was discovered that foam extrudate could be fibrillated into a web by means of the conical mandrel attached to the die.

At this stage of development the processing system closely resembled the process patented by Schirmer (20, 21, 22, 23). In both systems foam is extruded and stretched over a conical mandrel to form a web. Since Schirmer did not explore the potential for using this process to form webs suitable for garment applications, research on this process was continued. Ultimately, W. R. Grace loaned the equipment Schirmer used to Georgia Tech in order to

facilitate this research effort.

    b. Base Case

Typical processing conditions and web properties are shown in Table V. Since the basis weight of the extrudate before stretching is estimated to be 27 oz/sq. yd., a considerable degree of stretching is induced at these conditions to obtain the final basis weight of 0.44 oz/sq. yd. Despite this degree of stretching, the tenacities obtained are below 7.85 (lb/in)/(oz/sq. yd.) (5,000 psi) for unoriented polypropylene film. Furthermore, a tenacity imbalance exists which is not desirable in a textile web. Examination of the webs (see Figure 11) reveals a large, nonuniform pore structure oriented in the machine direction. The low tenacities and tenacity imbalance are attributed to tthis pore structure.

The objectives of the ensuing studies are to 1) reduce the pore size and pore size distribution, 2) increase the basis weight, 3) increase tenacities, 4) reduce tenacity imbalance and 5) reduce air permeability. Improving the basis weight and pore structure should improve the mechanical and physical properties; hence, these objectives should not be conflicting.

    c. Material Modifications

Three grades of polypropylene ranging in melt index from 0.4 to 12.0 were evaluated. These grades exhibited similar mechanical properties but the high melt index grade (Profax 6323) was retained for further study because it is easier to fibrillate. The higher molecular weight grades of polypropylene contained too many filmy sections.

The amount and type of additives in the extrusion feed were studied and selected results are shown in Table VI. A significant improvement in web properties was achieved by switching from Kempore 125 to Kempore 200MC. The latter blowing agent is nucleated and finer in particle size compared to

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$D_M = 2.5"$
70 RPM

0.25 inch

(a)

$D_M = 3.75"$
70 RPM

4X Magnification

0.25 inch

(b)

$D_M = 3.75"$
85 RPM

0.25 inch

(c)
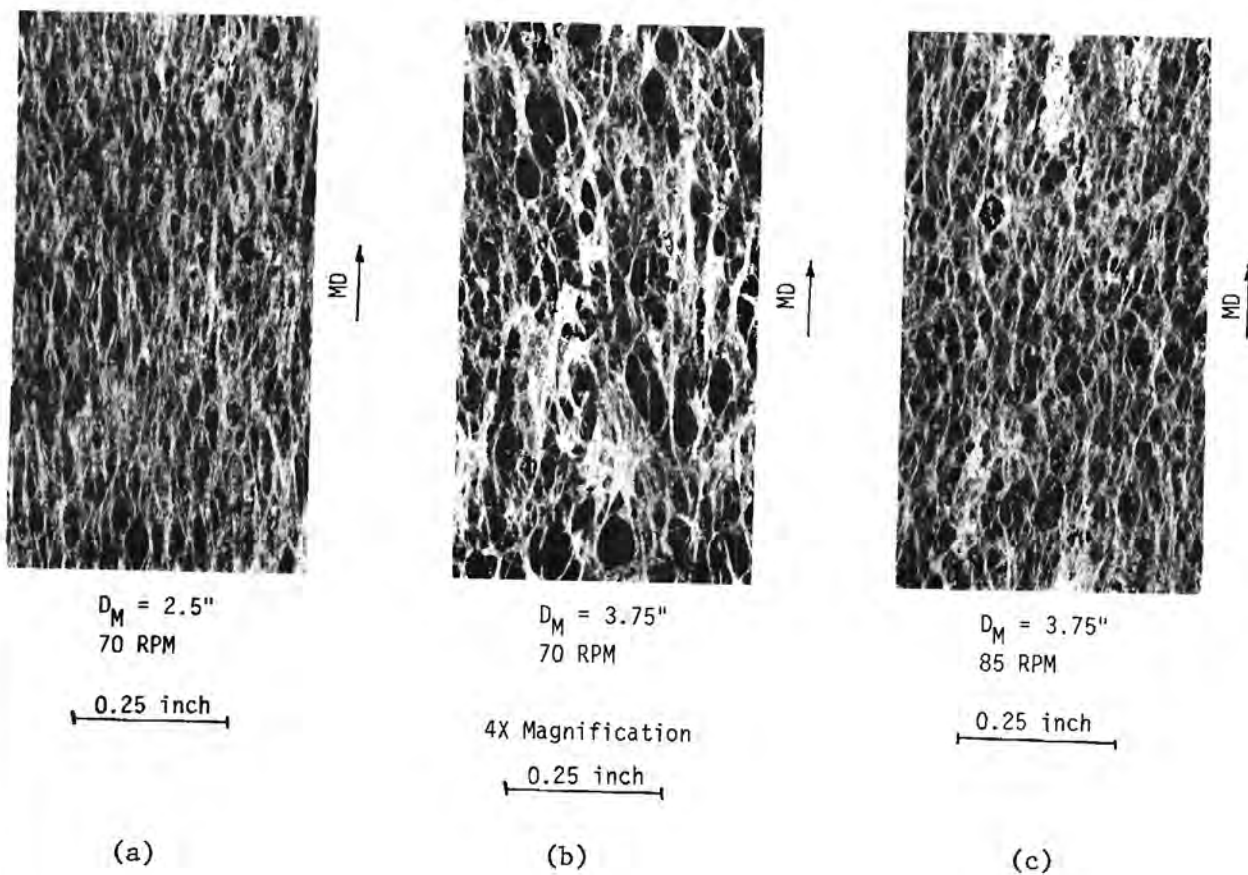
Figure 11.   Effect of Process and Design Conditions

TABLE V

TYPICAL PROCESS CONDITIONS AND RESULTS

MATERIALS

LINEAR POLYPROPYLENE (HERCULES PROFAX 6323)	99.0%
AZODICARBONAMIDE (KEMPORE 200MC, STEPHAN CHEM.)	0.5%
FUMED SILICA (CAB-O-SIL M-5, CABOT CORP.)	0.5%

PROCESSING CONDITIONS

ZONE 1	200°C
ZONE 2	230°C
ZONE 3	230°C
ZONE 4 (DIE)	230°C
SCREW SPEED	70 RPM
WIND UP RATE	36 FPM
MANDREL/DIE DIAMETER RATIO (D_M/D_D)	2 1/2
COOLING RING AIR PRESSURE	30 PSIG

WEB PROPERTIES

BASIS WEIGHT, OZ/SQ. YD.	0.44
MD TENACITY, (LB/IN)/(OZ/SQ. YD.)	2.7
TD TENACITY, (LB/IN)/(OZ/SQ. YD.)	0.57
AVERAGE TENACITY, (LB/IN)/(OZ/SQ. YD.)	1.7
AIR PERMEABILITY, (FT/MIN)·(OZ/SQ. YD.)	150

TABLE VI

EFFECT OF ADDITIVES

POLYMER: HERCULES PROFAX 6323

CONDITIONS: 200°, 230°, 230°, 230°, 75 RPM, 36 FPM

	Additives	Basis Weight oz/sq. yd	Average Tenacity (lb/in) (oz/sq yd)	Permeability (ft/min)·(oz/sqyd)
1.	KEMPORE * 125 (0.5 WT %)	0.53	2.0	175
2.	KEMPORE 200MC (0.5 WT %)	0.52	2.6	75
3.	KEMPORE 200MC (1.0 WT %)	0.49	1.4	260
4.	KEMPORE 200MC PLUS CAB-O-SIL **M5 (EACH 0.5 WT%)	0.47	2.5	75

---

*   STEPHEN CHEMICAL CO. (AZODICARBONAMIDE)

** CABOT CORP. (FUMED SILICA)

Kempore 125. The web in case 2 is more uniform and has smaller pores. As a result, the tenacity increased and the permeability decreased. Adding more Kempore 200MC, case 3, caused overblowing, hence the coarse web formed exhibits inferior properties. Adding fumed silica, Cab-O-Sil M5, doesn't alter the tenacity or permeability significantly. The formulation in case 4 is somewhat easier to fibrillate under different processing conditions than the mixture in case 2, hence case 4 is the formulation utilized in the ensuing processing studies.

Additional additives have been studied, but no benefits were realized. Zinc stearate was added to improve bubble nucleation, but it also caused pre-blowing, producing a filmy web. Sodium salicylate, a nucleating agent for polymer crystallization, had no apparent effect on web structure. Adding linear polyethylene, a semicompatible polymer, did not improve fibrillation; however, only 10% by weight was added.

The properties of the web are significantly effected by the selection of the polymer grade, blowing agent and other additives. Components which nucleate bubble formation and/or fibrillation are particularly desirable as the web properties improve as the pore size and pore size distribution are reduced.

d. Effect of Process Parameters

Using the feed composition cited in case 4 in Table VI, the effect of temperature, screw speed, wind-up rate and stretching mandrel diameter were studied in some detail. Additional parameters, such as cooling conditions, annealing and hot stretching, have only been studied briefly.

The extrusion temperature profile is limited in range by the chemical blowing agent. If the temperature is too low inadequate blowing occurs. If it is too high excessive pre-blowing occurs. The best performance was

obtained with a relatively flat profile of 200°C near the hopper and 230°C elsewhere, as indicated in Table V.

Increasing wind-up rate at constant output increases the extent of MD stretching. Consequently, the MD tenacity increases, but the basis weight and TD tenacity decrease. The average tenacity does not change. Thus, in order to minimize tenacity imbalance and increase basis weight, low wind-up rates are preferred. Unfortunately, the web tends to collect on the mandrel if the wind-up rate is too low.

Increasing the extruder screw speed while other conditions remain constant increases the output and therefore the web basis weight--see Figure 12. The same relatively linear trend is observed for forming mandrels with different diameters ($D_M = 2.50"$ and $D_M = 3.75"$). The basis weights at the same processing conditions are lower for the larger diameter mandrel due to the additional TD stretching introduced by the larger mandrel.

The effect of screw speed on tenacity is shown in Figure 13. Since the tenacity reported is normalized with respect to basis weight the improvements in tenacity with increasing screw speed are surprising. In addition to an increase in basis weight as screw speed increases, the average pore size decreases. Since there are fewer gross flaws per unit length, the tenacities are better.

The reduction in pore size can be seen in Figure 11--compare b and c. Also the normalized permeability decreases as screw speed increases (Figure 14) suggesting pore size decreases. Unfortunately, as pore size decreases the tendency for film bridging increases, hence not all of the decrease in permeability can be attributed to a decrease in pore size. The lower permeabilities using the 2.5" mandrel are attributed to a greater degree of filmy texture with this mandrel.

- 47 -
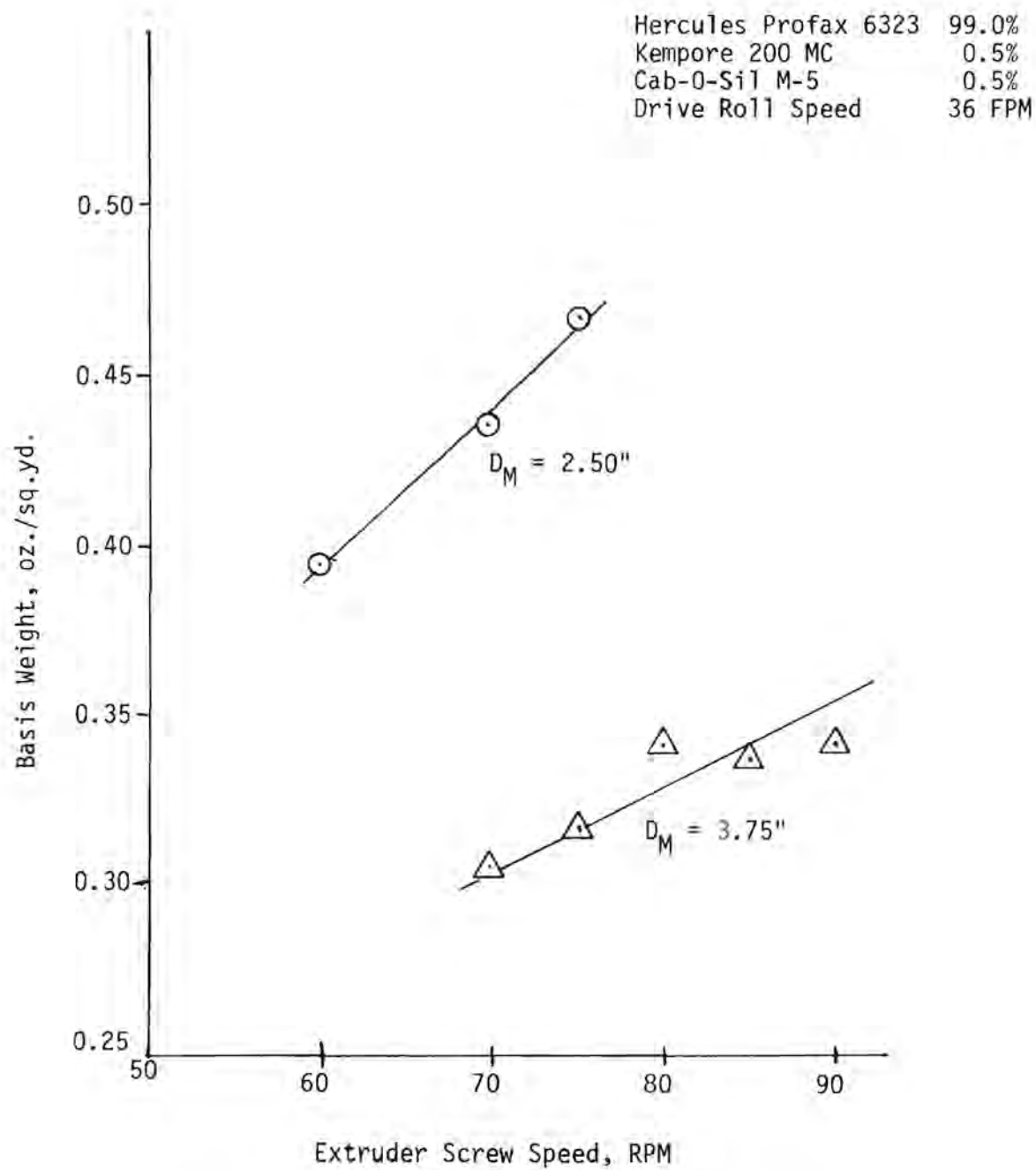
Figure 12. Effect of Screw Speed on Basis Weight
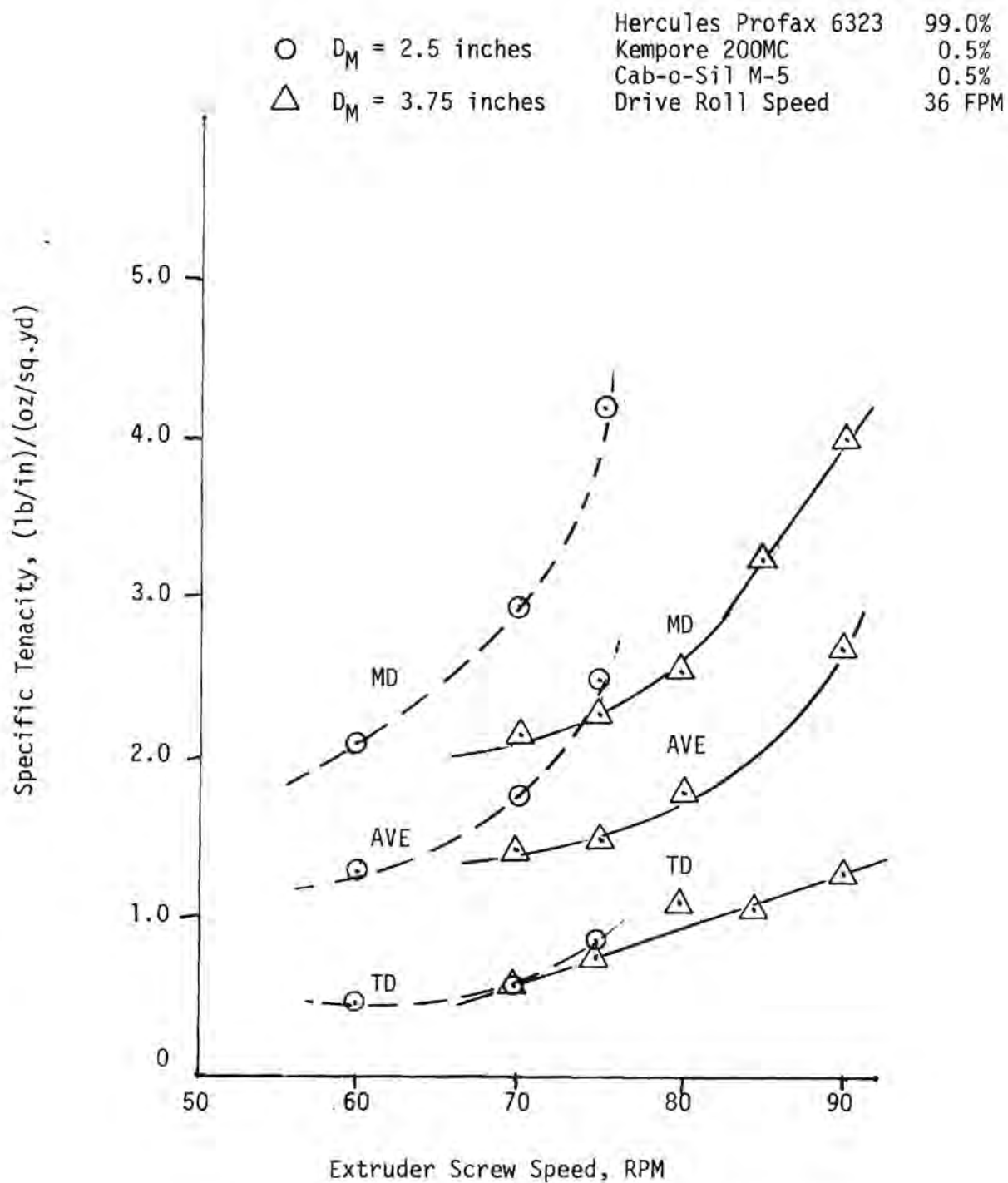
Figure 13.   Effect of Screw Speed on Tenacity.

HERCULES PROFAX 6323   99.0%
KEMPORE 200MC   0.5%
CAB-O-SIL M-5   0.5%
DRIVE ROLL SPEED   36 FPM
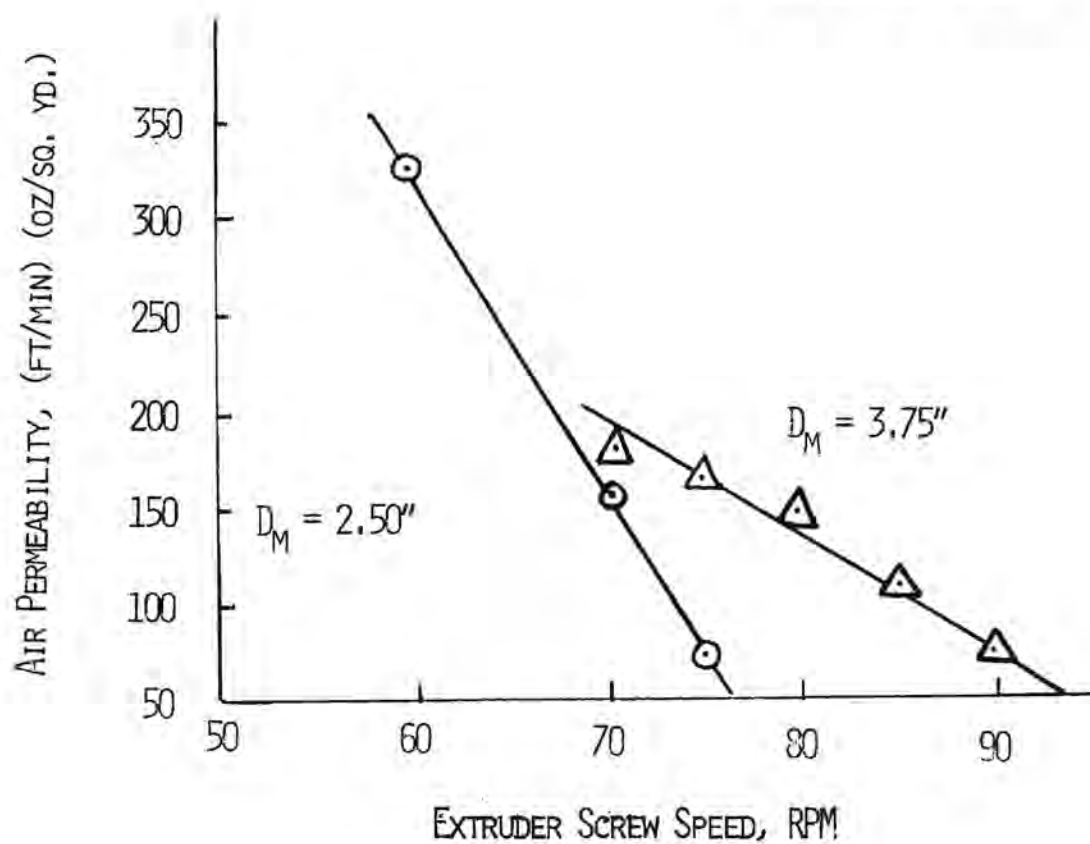
FIGURE 14.   EFFECT OF SCREW SPEED ON PERMEABILITY

Comparing the two mandrels, the 3.75" diameter mandrel appears preferable. While higher basis weights can be obtained with the 2.5" mandrel, the 3.75" mandrel provides comparable average tenacities, less tenacity imbalance and less filmy texture. All of the properties improve as screw speed increases except for the tendency to become filmy.

The range of web properties achieved by modifying processing conditions are not considered adequate for most textile applications. The TD tenacity is the weakest feature, making the web easy to tear. Through annealing and hot stretching in the transverse direction, these deficiencies can be improved substantially.

e. Equipment Modifications

In order to obtain further improvements in web properties a number of equipment design changes, listed in Table VII, were investigated. The impact of these changes will be discussed in turn.

Change 1. Liquid blowing agent feed

The purpose of this system is to reduce the size of the pores in webs. Since the size of the pores is reduced as the particle size of solid chemical blowing agents is reduced, then the ultimate in small pore size should be obtained by using a liquid blowing agent, provided adequate mixing can be achieved. In order to use liquid blowing agents a low flow rate, constant volume pump is necessary.

A liquid resevoir with a constant pressure head and a long capillary tube outlet was constructed to provide the necessary flow rates. However, consistent flow rates were not obtained. Blending the liquid blowing agent with the polymer prior to extruding the mixture resulted in poor and insufficient mixing. Consequently, further work on this system was terminated.

Change 2. Gas feed system

A gas feed system was attached to the vent port of the extruder in

TABLE VII
EQUIPMENT DESIGN CHANGES

	CHANGE	PRINCIPAL BENEFIT EXPECTED
1.	LIQUID BLOWING AGENT FEED	IMRPOVE WED UNIFORMITY
2.	GAS FEED SYSTEM	IMRPOVE WEB UNIFORMITY
3.	STATIC MIXER	IMPROVE WEB UNIFORMITY
4.	DIE PRESSURE SENSOR	BETTER PROCESS CONTROL
5.	WIDE ANGLE DIE	MINIMIZE EXTRUDATE IMPINGEMENT
6.	ROTATING CORE DIE	INCREASE TRAVERSE DIRECTION ORIENTATION
7.	COLLASPING MANDREL	FACILITATE START-UP
8.	STAGEWISE MANDREL	INCREASE TRAVERSE DIRECTION ORIENTATION
9.	MODIFIED COOLING RING	FACILITATE START-UP
10.	HOT AIR QUENCH	VARY QUENCH CONDITIONS
11.	DOWNWARD EXTRUSION	FACILITATE START-UP
12.	CRYOVAC SYSTEM	EVALUATE SPIRAL MANDREL

order to evaluate the use of nitrogen and carbon dioxide gases as physical blowing agents. By using these gases greater freedom in processing nylons and polyesters should be obtained.

Even though the gas flow rate could be carefully controlled by regulating the gas pressure at the point of injection (the extruder's vent port), the webs produced were non-uniform. Some improvement in web uniformity was obtained by incorporating the static mixer, change 3, between the extruder and the die. The static mixer provides enough residence time for diffusion of the gas into the polymer. Since the webs tended to alternate between filmy and fibrous regions with time, consistent axial mixing was not achieved. The use of a sparging ring in place of the vent port was investigated as a means of improving axial mixing, but the extruder could not be modified in this manner. No further improvements in the gas feed system have been made.

Change 3. Static mixer

A static mixer (a six element, 1/2 inch i.d., Kenics mixer) was added between the extruder and the die to improve the uniformity of the foam extrudate by improving the dispersion of foaming agent in the melt. After incorporating the mixer inferior product was obtained in most cases. This decline in quality is attributed to the inability of this mixer to disperse gas in the polymer melt. If a significant quantity of chemical blowing agent decomposes in the extruder, the mixer cannot prevent the evolved gases from coalescing. However, the mixer does improve the dispersion of solids and soluble gases in the polymer melt. Thus, the mixer is helpful in some cases, but it is harmful when chemical blowing agents are used.

Change 4. Die pressure sensor

A die pressure sensor was incorporated to improve manual process control.

Unfortunately, when the rotating die is used, a constant die gap cannot be maintained. Consequently it is difficult to reproduce die pressure readings under apparently identical processing conditions. It is apparent that the quality of the web is sensitive to die pressure as high die pressures tend to produce filmy webs.

Change 5. Wide angle die

A wide angle die was constructed in an attempt to direct the extrudate outward towards the transverse direction. By directing the extrudate outward in the direction of biaxial stretching, less chance of extrudate impingement on the stretching mandrel should occur. In practice, little difference in performance was observed.

Change 6. Rotating core die

A rotating die was installed in order to increase transverse direction orientation in the webs. At low rotational speeds no increase in transverse direction orientation is evident. However, at these low rotational speeds the uniformity of the webs is improved. At high speeds the webs produced remain twisted.

Change 7. Collapsing mandrel

A ring mandrel which could be tilted on a shaft was constructed in order to facilitate startup. By starting with a ring initially tilted towards the machine direction, a lower degree of biaxial stretch is incurred which can gradually be increased by tilting the ring away from the machine direction. This modification was tested and found to be inadequate because the mandrel wobbled too easily.

Change 8. Stagewise mandrel

Stagewise biaxial stretching has been briefly evaluated by following a small diameter conical mandrel with a larger diameter ring mandrel. This

approach can be performed and improved tenacities are obtained.

Change 9. Modified cooling ring

The cooling ring was completely redesigned in order to facilitate start-up
by improving access to the die face. The new cooling ring has been beneficial,
permitting significantly higher extrusion rates without disrupting the process.
Consequently, higher basis weights can be obtained.

Change 10. Hot air quench

The air quench system was modified to permit extrudate cooling with
air up to 80°C. The quench temperature can significantly alter the
deformation characteristics of the extrudate, introducing an additional
processing parameter. This system has been difficult to operate since
the air heater failed frequently.

Change 11. Downward extrusion

The direction of extrudate take-up was switched from upward to downward
to facilitate formation of nylon and polyester webs. Initial processing
studies with nylons and polyesters were hindered by polymer sagging and
then sticking to the die face. By extruding downward, the extrudate tends
to sag due to gravity away from the die. Despite implementing this modifi-
cation, the formation of nylon and polyester webs still suffer from
extrudate sticking to the die face or the mandrel.

Change 12. Cryovac system

The pilot extrusion system developed by the Cryovac Division of W. R.
Grace for forming webs has been installed and operated. Several webs
exhibiting a high degree of uniformity but relatively large pores have been
produced from mixtures of nylon and polyethylene. In addition, polypropylene
webs were formed from Hercules Profax 6323 in order to compare the product
formed by the Cryovac and Georgia Tech systems. Webs of similar quality were

produced. Since a significant improvement in web quality was not obtained
in using the Cryovac system, a quantitative evaluation of web properties
was not pursued.

　　f.　Optimization

Following these equipment modifications the feed composition and processing
conditions were carefully examined in order to optimize web properties. The
criteria used for determining the optimum values were the appearance and
uniformity of the webs rather than their mechanical properties. This basis
was chosen because filmy webs show deceptively high tensile properties and
deceptively low permeabilities. Also, any change in mechanical properties is
meaningless unless it is an improvement over webs with the same degree of
uniformity and filminess. Unfortunately a scale for measuring filminess and
uniformity was not available, hence the optimization procedure was subjective.

Changes in feed composition included substituting nitrogen and carbon
dioxide for the chemical blowing agent, varying the amount of fumed silica
(Cab-o-Sil M5, Cabot Corp.) between 0 and 2%, and varying the amount of
silica flour (Min-u-Sil 5, Cabot Corp.) between 0 and 1% by weight. Webs
comparable to Kempore 200MC blowing agent (azodicarbonamide) could be obtained
using nitrogen, provided fumed silica and silica flour were present. Webs
made using carbon dioxide as a blowing agent were inferior to nitrogen or
azodicarbonamide, the addition of fumed silica and/or silica flour did not
improve the quality of the webs significantly. In sum, little or no improve-
ment in web quality was obtained by these changes in feed composition. Specific
details are given in previous reports (24, 25).

Process parameters studied included the extruder-die temperature profile,
extrusion rate, wind-up rate and biaxial stretching rate. The temperature
profile was determined by the blowing agent with only a narrow temperature

span resulting in web equivalent to prior results. No substantial improvement in web quality was apparent.

Due to design changes to the mandrel and cooling ring it was possible to extrude webs with basis weights approaching 1 oz/sq. yd. whereas previously a basis weight near 0.5 oz/sq. yd. was the maximum. Higher basis weights could be obtained but the product was too filmy. Despite the increase in basis weights little increase in specific tenacity was obtained and the tenacity imbalance still exceeded 10.

No decrease in wind-up rate could be achieved and higher wind-up rates accentuated the tenacity imbalance. Increasing the degree of biaxial stretch by using a larger mandrel reduces the filminess of the web, thereby increasing the air permeability, but does not result in a significant decrease in tenacity imbalance. Since it is more difficult to operate with a large diameter mandrel, the use of the larger mandrel is not worthwhile. Specific details of these process parameter studies are given in previous reports (24, 25)

  g. Other polymers

Polypropylene has been studied in detail with the expectation that the processing characteristics observed would also apply to other polymers. For garment applications nylon and polyester webs would have more utility, but these polymers are harder to process. In fact webs comparable in quality to the polypropylene webs could not be formed using nylon 66 or polyethylene terephthalate (PET). Reasonably decent quality webs could be formed using polybutylene and polybutylene terephthalate (PBT).

The primary processing difficulties associated with PET are nylon 66 are sticking, material degradation and inadequate melt strength. Both polymers tended to stick to the die face and mandrel which would disrupt web formation. Both polymers required careful drying in order to avoid degradation. Also,

thermal degradation interfered with start-up. Both polymers exhibited low melt strengths which would result in extrudate tears and failures. In addition, the high temperature blowing agent utilized with these polymers, Kemtec 500, Sherwin Williams Co., did not result in a uniform foam extrudate. Thus, what little web that could be formed was coarse and nonuniform.

Polybutylene and PBT could both be formed into webs approaching the quality of the polypropylene webs. In general, it was easier to form webs from these polymers when they were blended with polypropylene. None of the webs formed from these other polymers exhibited mechanical properties better than the polypropylene webs. Further details on processing these other polymers are given in previous reports (24, 25).

H. Conclusions

The webs formed by foam fibrillation using the materials and processing conditions studied are not suitable for a broad range of garment applications. The primary deficiencies of these webs are: 1, basis weights below 1 oz./sq.yd.; 2, average tenacities below 2 lb/in/oz./sq.yd.; 3, tenacity imbalances in excess of 5 (MD/TD); 4, high air permeabilities; and 5, a tendency towards filmy junctions. The measured properties need to be improved by a factor of 5 to 10 for garment applications. After considerable equipment modifications and a parametric study of the feed composition and processing conditions, only minor improvements in properties were achieved.

The primary polymer studied was polypropylene. Other polymers, polybutylene and PBT, were successfully processed. However PET and nylon 66 were very difficult to process. Thus, the technology of foam fibrillation can be transferred to other thermoplastics, but not with equivalent processing ease.

Foam fibrillation does provide a simple process route for forming non-

woven webs which have considerable potential in non-garment applications. Such applications would include adhesive interlinings, curtains, wallcovering, soil stabilization, wind shields, disposable gowns and wiping cloths. These applications are characterized by a high surface-to-volume ratio and low fabrication cost.

6. Yarn Formation

While it is difficult to overcome the low basis weight and tenacity imbalance in forming webs by foam fibrillation, these features are desireable in forming yarns. Thus, foam fibrillation of polypropylene using the same extrusion and biaxial stretching equipment has been explored in order to determine the potential of this process route for forming yarn.

   a. Initial process development

As indicated in Table VIII the same grades of polypropylene and azodicarbona-mide blowing agent have been used to study yarn formation that were used to evaluate web formation. A low screw speed and high wind-up rate, 30 RPM and 82 FPM respectively, were used to accentuate yarn characteristics in the extrudate. The as-extruded web has a denier of approximately 5,000. The tenacity is 0.14 gr/den without a mandrel and increases to 0.20 and 0.19 gr/den. when the 2 1/2" and 3 3/4" mandrels are utilized (see Table VIII). The elongation of the web is approximately 75%.

   b. Enhancing tenacity

Significant increases in tenacity are achieved by twisting and drawing by hand over a heated cylinder at 136°C (see Table VIII). It is difficult to twist the yarn more than 2 1/2 TPI (turns per inch) because of the large denier. Also, drawing beyond three times the original length is hindered by fracture. Since both twisting and drawing improve tenacity, a sequential drawing and twisting cycle was devised to achieve higher draw ratios and twist densities.

TABLE VIII

INITIAL POLYPROPYLENE YARN

MATERIALS: HERCULES PROFAX 6323   99.5%
                            KEMPORE 200MC   0.5%

PROCESS CONDITIONS: SCREW SPEED   30 RPM
                     DRIVE ROLL SPEED   82 FPM

TENACITY (GR/DEN.)

TWISTS PER INCH (TPI)	0	0	2 1/2	2 1/2
DRAWING AT 136°C	NONE	2x	2x	3X
MANDREL DIAMETER (INCHES)				
NONE	0.14	0.35	0.78	1.12
2.5	0.20	0.71	1.22	1.76
3.75	0.19	0.60	1.04	1.75

Figure 15 illustrates the effect of draw temperature on sequentially drawn and twisted yarn. This yarn has a total draw ratio of 4, excluding extrusion draw down, and a final twist of 2 TPI. The denier is 1,385. A tenacity exceeding 3 gr/den with an elongation of 25% can be achieved. Further increases in tenacity can be obtained with additional drawing and twisting. As Table IX illustrates the yarn produced by this process route is comparable to staple fiber yarn produced commercially.

Based on these encouraging results more detailed studies were performed on: evaluating drawing, twisting and extrusion conditions, reducing yarn denier, converting to continuous drawing and twisting, forming fabric, and performing an economic analysis. The results of these studies follow.

C. Drawing and twisting conditions

In order to increase the tenacity of the as-extruded yarn, hand drawing of the yarn over a heated cylinder at 130°C and hand twisting was investigated (26). It was apparent that twisting the yarn permitted drawing to high draw ratios and improved the yarn's tenacity. The following draw-twist sequence was established: a) draw the yarn 2X, b) twist the yarn 2 TPI, c) draw the yarn again by 2X and d) twist the yarn 2 TPI. This sequence produces a yarn with a total draw ratio of 4 and total twist of 3 TPI. If more drawing than 2X is attempted in the first step, the yarn would frequently break. Additional twist in the second step was not attempted due to the high denier of the yarn. As Figure 16 illustrates, this draw-twist sequence produces yarn with a tenacity of 3.1 g/denier and an elongation at break of 25%.

The effect of total twist on hand-drawn yarn properties was investigated by varying the amount of twisting in steps b and d between 0 and 6 TPI. The results in Figure 16 indicate that the elongation at break increases with increasing twist within this experimental range. This is probably due to enhanced

TREATMENT: DRAWN 2X, TWISTED 4TPI AND DRAWN 2X AGAIN

DENIER: 1,335
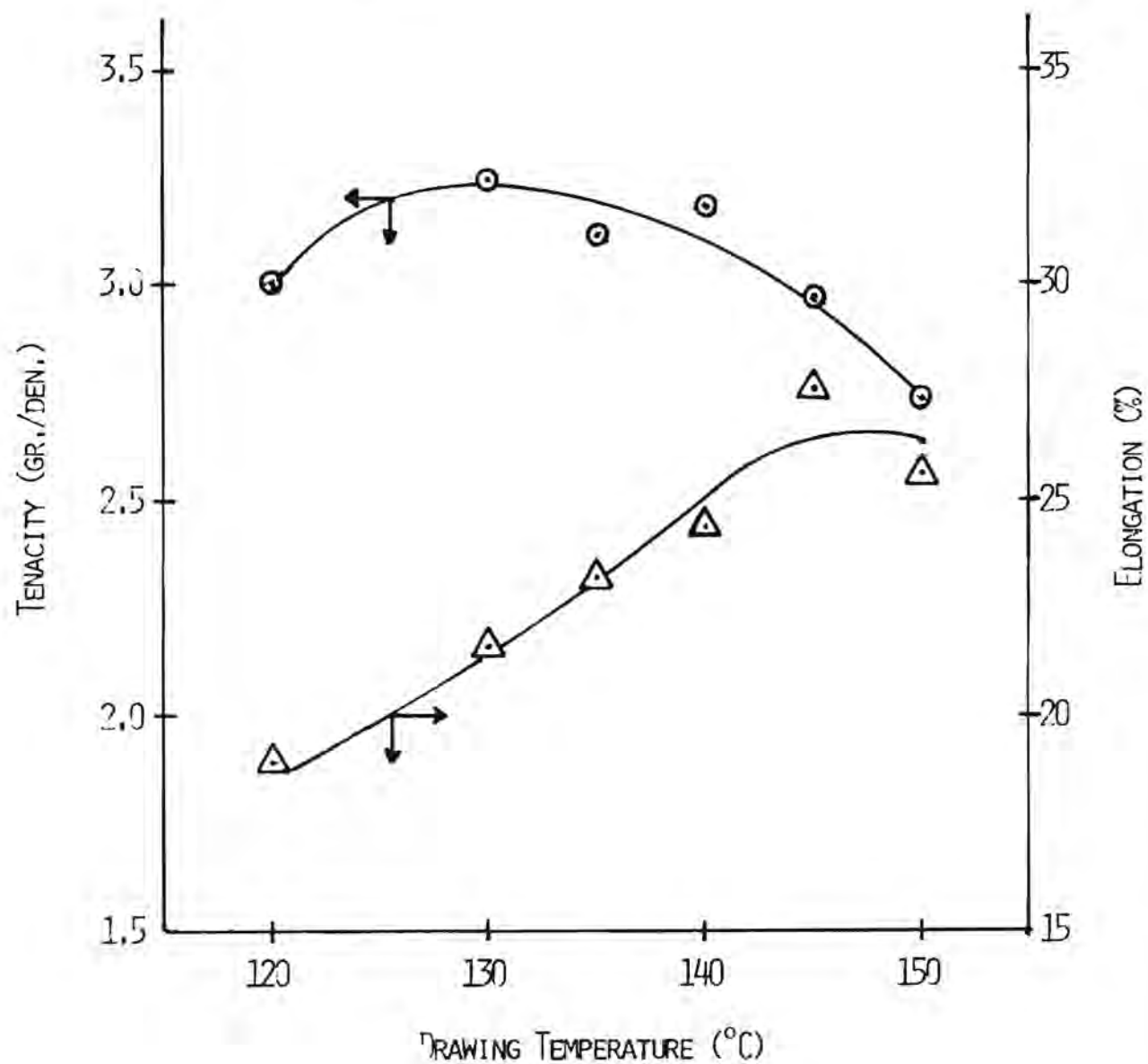
FIG. 15. EFFECT OF DRAW TEMPERATURE ON YARN PROPERTIES

TABLE IX

COMPARISON OF EXPERIMENTAL YARN
WITH COMMERCIAL POLYPROPYLENE
STAPLE FIBER YARN

PROPERTY	COMMERICAL YARN	EXPERIMENTAL YARN
DENIER	1800	1385
TWIST/INCH	3	2
TENACITY (GR/DEN.)	2.5	3.2
ELONGATION (%)	36	22
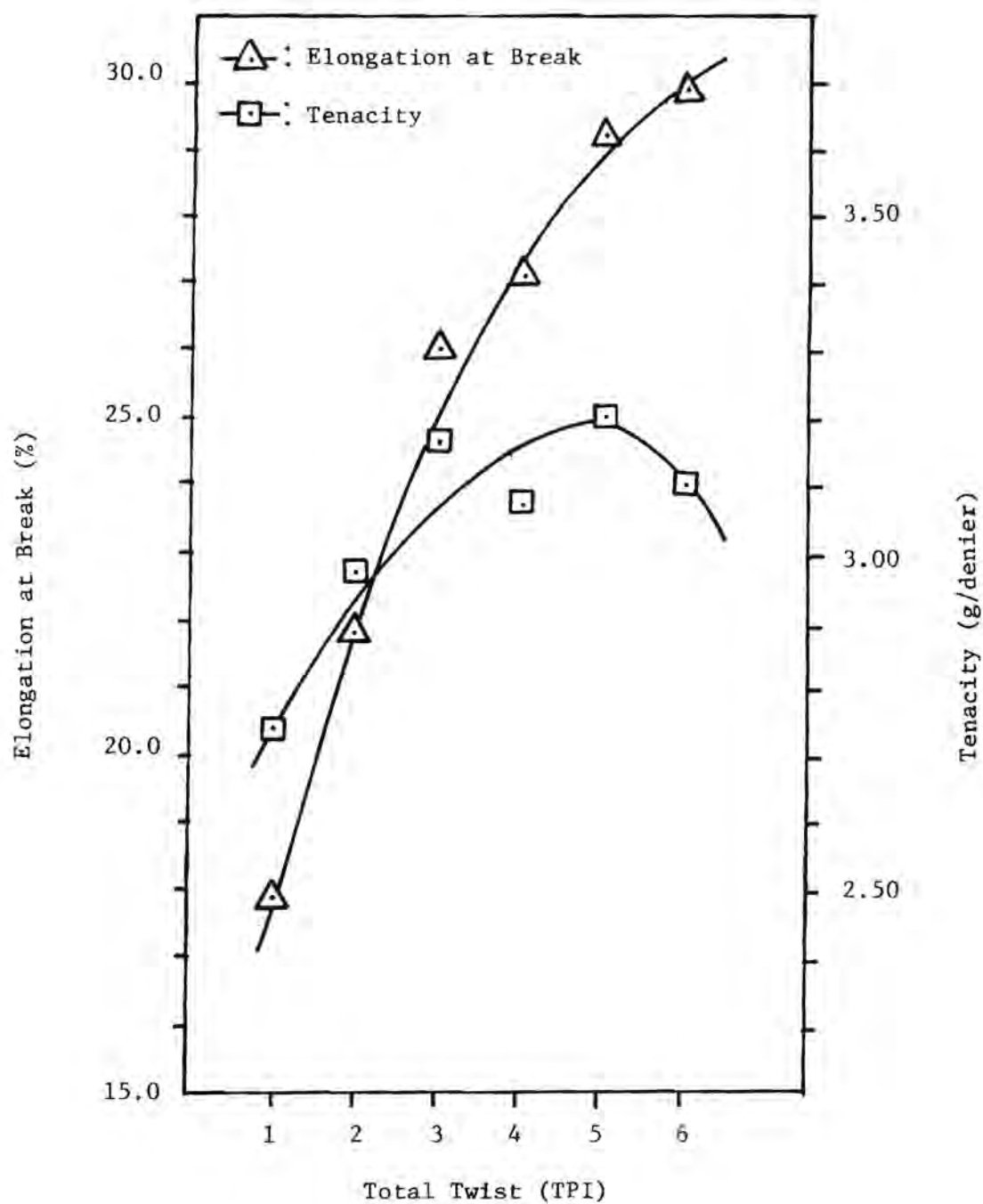
Figure 16. Elongation at Break and Tenacity-vs-
Total Twist with a Total Draw Ratio of 4 at
Drawing Temperature of 130°C

mutual support by the fibrils. Tenacity rises to a maximum value at a total twist of 5 TPI and then falls. The initial rise is probably due to the effects of twist which result in the support of weak places in fibrils by neighboring fibrils and the limitation of the initiation of breakage solely to fibrils near the most highly strained position in the yarn. The decrease in tenacity at high twists is attributed to a decrease in molecular orientation along the yarn axis.

The effect of drawing temperature on sequentially drawn and twisted yarn is shown in Figure 15. In this case the sequential drawing and twisting cycle is 2X, 4 TPI, and 2X. This corresponds to a total draw ratio of 4 and a total twist of 2 TPI. Figure 17 shows that the dependence of total orientation of polymer, or birefringence, on drawing temperature is similar. Comparing Figures 15 and 17, the dependence of tenacity and birefringence on temperature is similar. Therefore the first increase in tenacity is due to an increase in polymer orientation. As the drawing temperature exceeds $130^{\circ}C$, polymer is less oriented and a lower tenacity results. The increase in elongation at break is attributed to annealing which removes stress concentrations, potential fracture points, by healing junctions.

Draw Ratio

The effect of drawing without twisting on yarn properties is shown in Figures 18. The tenacity increases and the elongation at break decreases with increasing draw ratio. These trends indicate that a substantial increase in molecular orientation occurs with increasing draw ratio, which is confirmed by the increase in birefringence with draw ratio, Figure 19.

The increase in density observed with increasing draw ratio, Figure 19. is attributed to the rupture of bubbles in the fibrils as drawing increases. It is noteworthy that the densities of these yarn samples are significantly
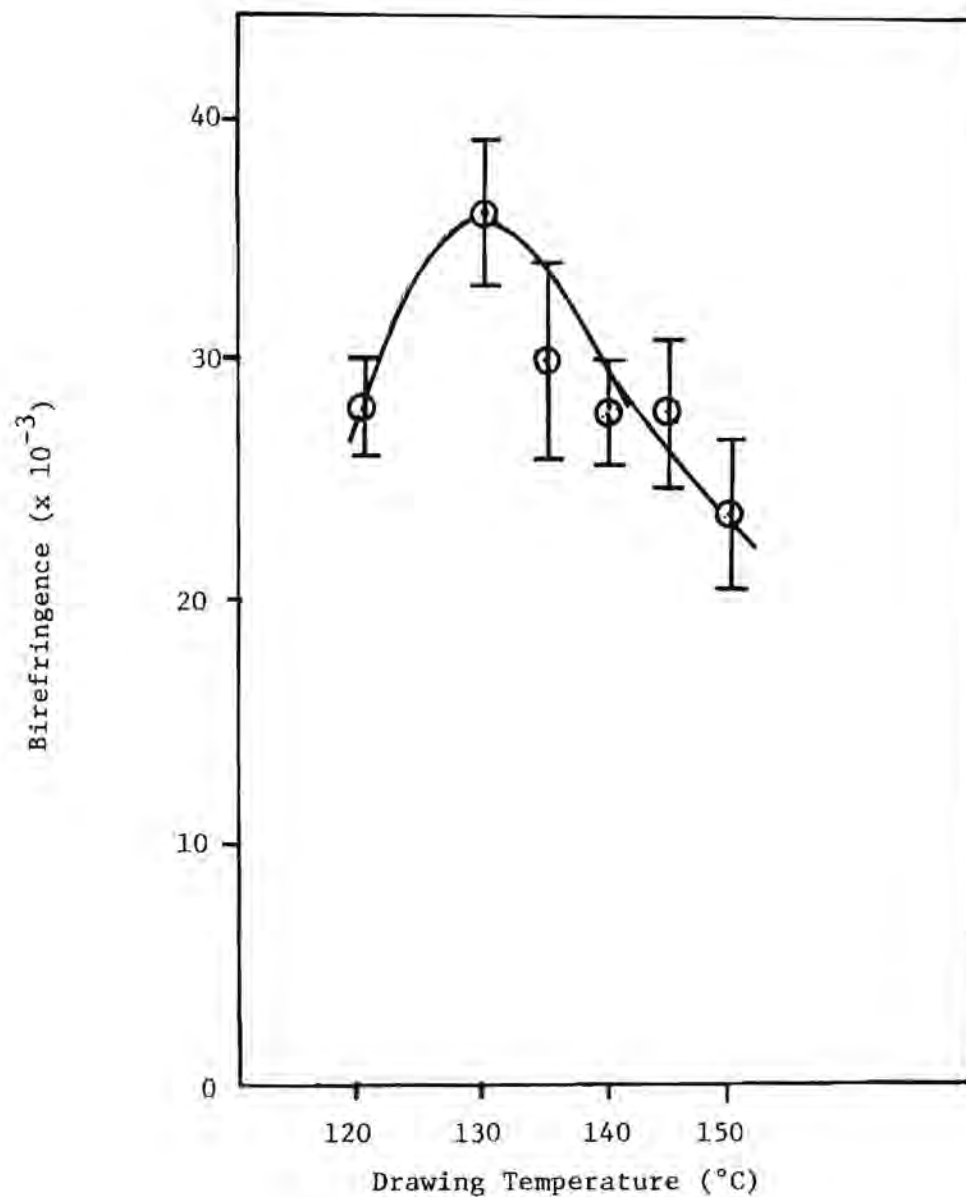
Figure 17. Birefringence vs Drawing Temperature with a Draw Ratio of 4

- 66 -
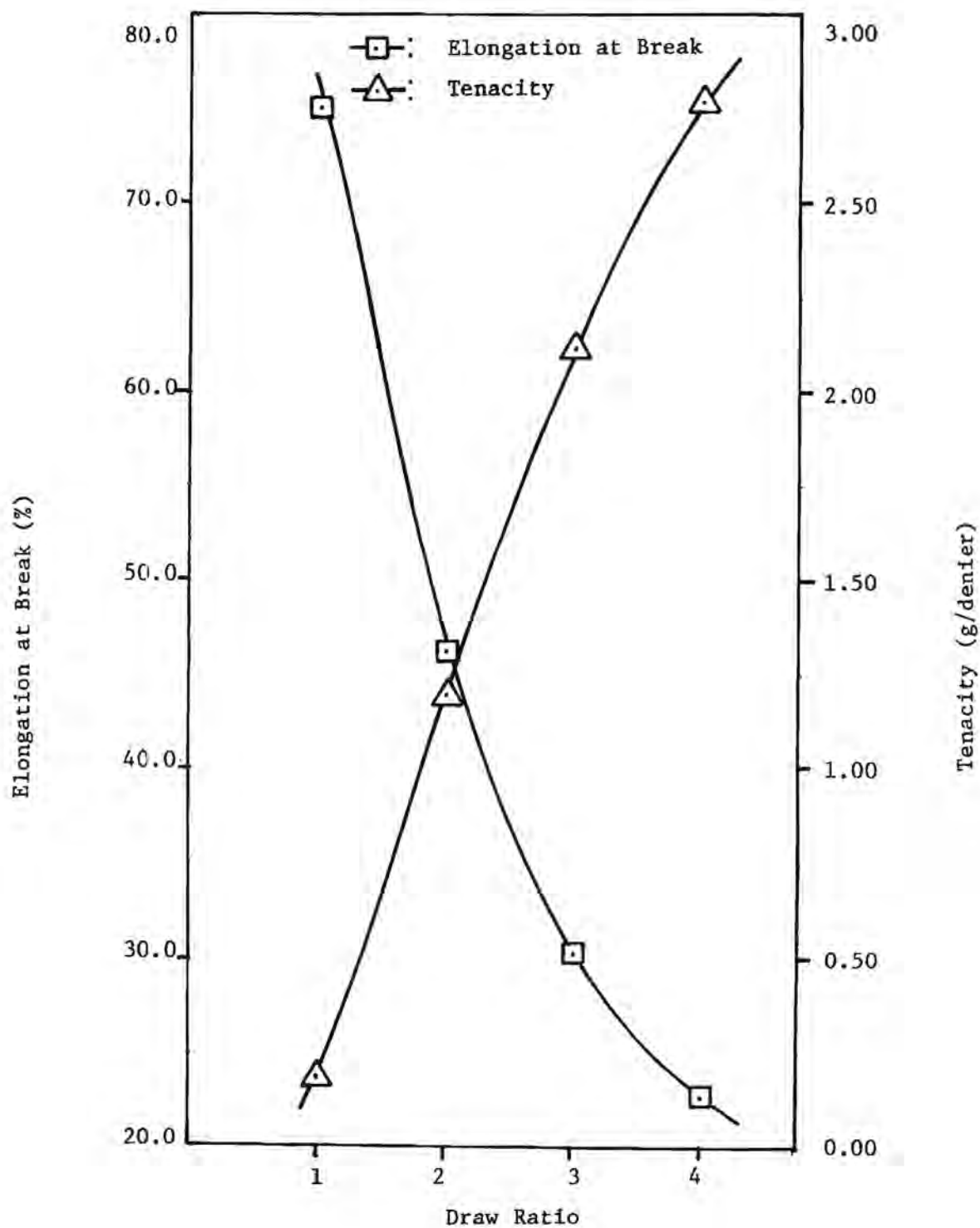
Figure 18. Elongation at Break and Tenacity-vs-
Draw Ratio at Drawing Temperature of 135°C
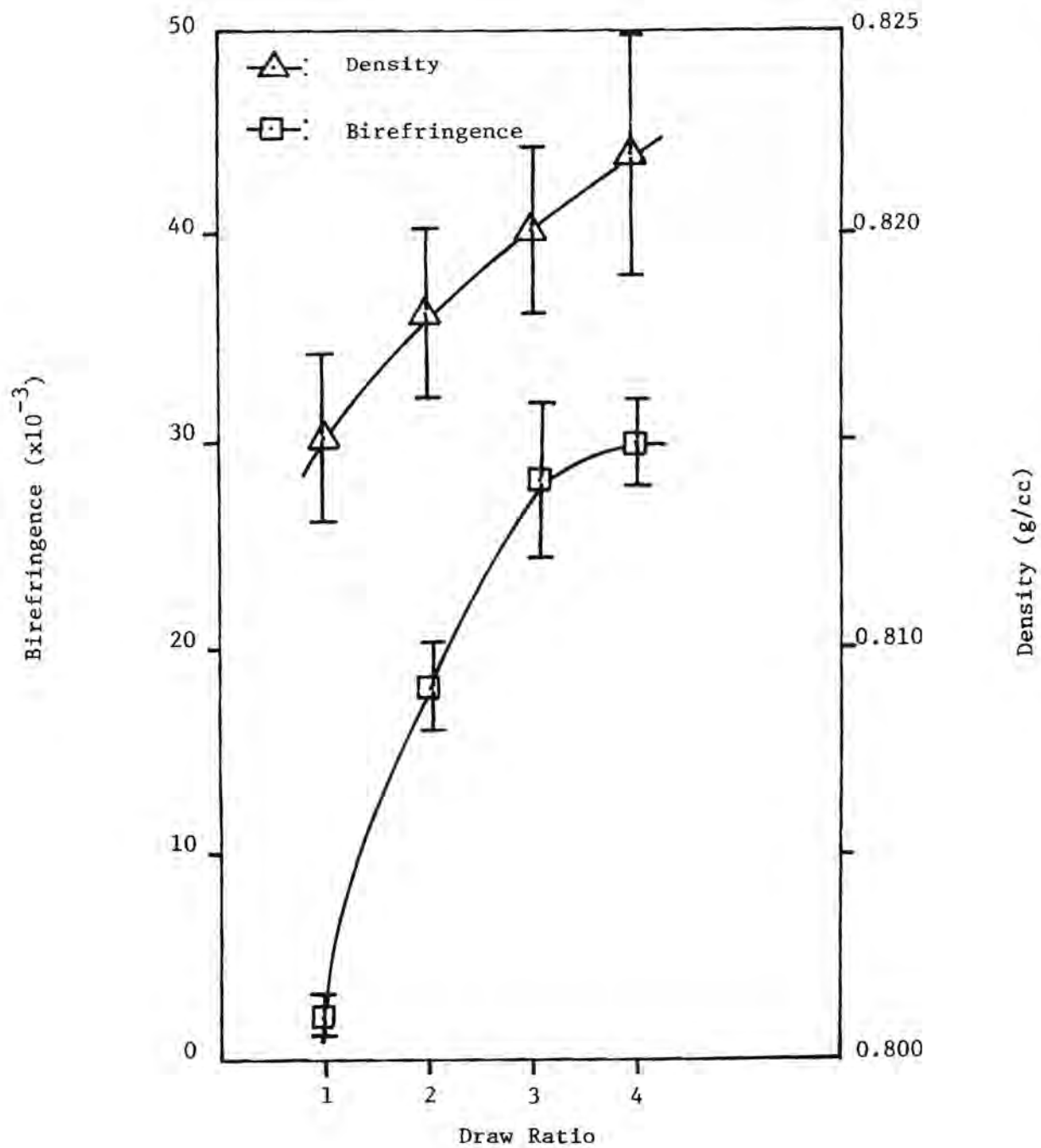
Figure 19. Birefringence and Density-vs-Draw
Ratio at Drawing Temperature of 135°C

below the typical density for polypropylene of 0.91 g/cm^3. This low density is attributed to the slightly cellular structure of the fibrils within the yarn.

The diameters of fibrils in a filament or staple fiber yarn are quite uniform because each fiber is produced through the same nominal size of orifices. The filaments produced by foam extrusion do not have uniform diameters and shapes. The filament size distributions of yarns with different draw ratios are given in Figure 20. As draw ratio increases, the filaments become finer and the filament size distribution becomes sharper. However, the most probable filament dimension remains constant within the range of 45 to 90 μm. Thus most of the yarn drawing is borne by the larger filaments. Hence, the filaments within the drawn yarn probably exhibit a considerable variation in draw ratio. To some extent the smaller filaments break while the larger filaments continue to draw.

    d. Extrusion conditions

Having established drawing and twisting conditions which result in yarn with attractive mechanical properties, an examination of extrusion parameters was performed in order to determine their impact on ultimate yarn properties. Particular attention has been given to reducing yarn and filament deniers and increasing yarn tenacity.

In order to study the effects of screw speed and wind-up rate, the draw-twist sequence was fixed as follows: a) draw 2X at 130°C, b) twist 2 TPI, c) draw 2X at 130°C and d) twist 2 TPI. Thus all yarns have a total draw ratio of 4 and total twist of 3 TPI. The results for varying the screw speed from 15 to 45 RPM and the wind-up rate from 66 to 124 ft/min are given in Table X.

As can be seen in Table X and Figure 21, the denier decreases with decreasing screw speed and increasing wind-up rate. Since the number of
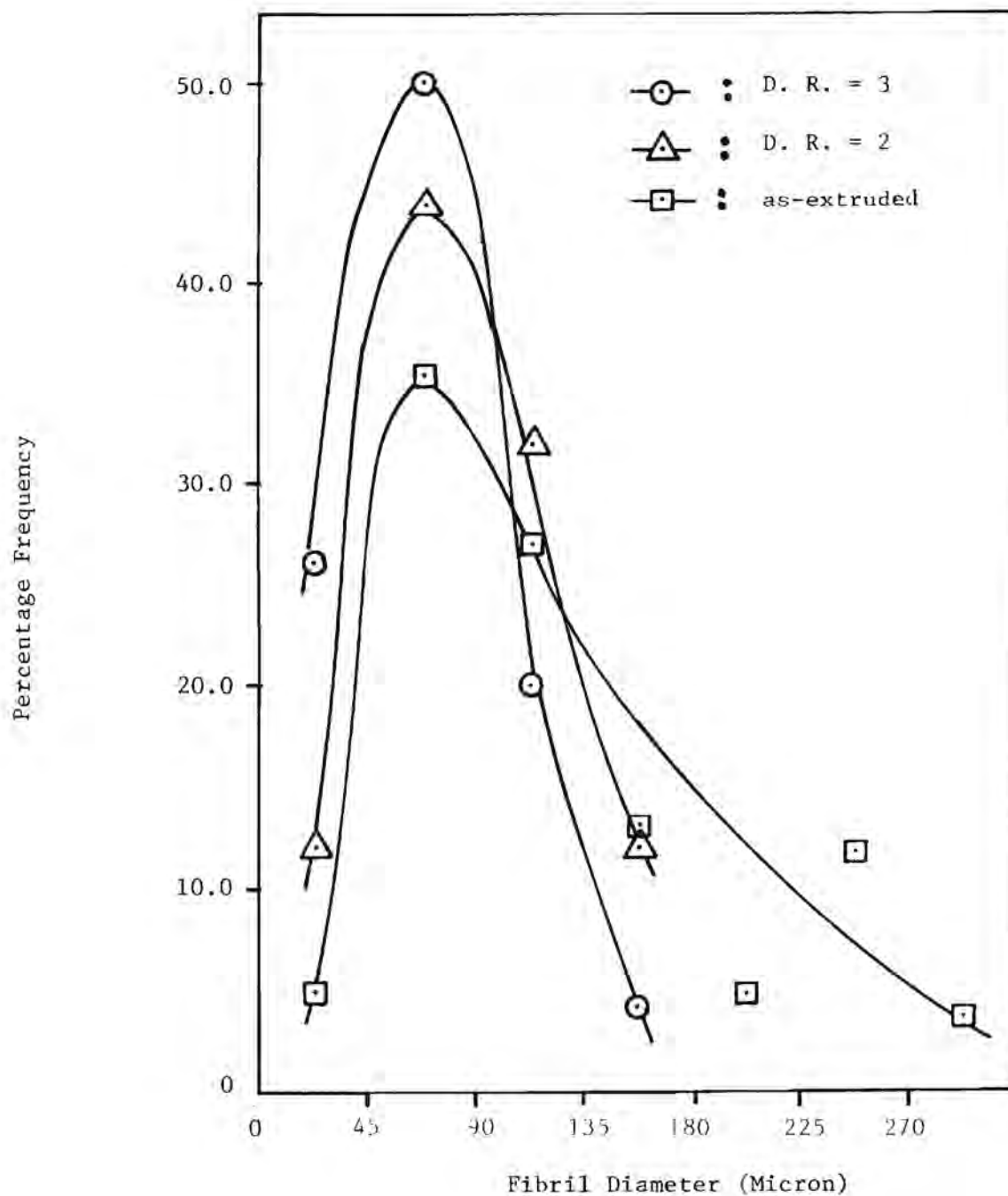
Figure 20. Percentage Frequency-vs-Diameter Range of
Fibrils Drawn at 130°C and with Different Draw Ratios

Table X.

Effect of Screw Speed and Wind-up Rate on Maximum
Yarn Tenacity and Minimum Yarn Linear Density

Process Conditions			Yarn Drawn 4X and Twisted 1.2 turns/cm		
Screw RPM	Wind-up Rate (cm/s)	Yarn linear density (dtex)	Tenacity (mN/dtex)	Elongation (%)	Average Filament linear density* (dtex)
15	33	1150	23.9	21	10.0
15	48	760	28.6	22	7.0
15	62	570	29.5	22	5.6
25	33	1870	17.1	20	N.M.
25	48	1240	21.5	20	N.M.
25	62	970	24.9	21	8.9
35	41	2020	19.9	19	N.M.
35	48	1680	19.6	20	N.M.
35	55	1500	21.5	22	N.M.
35	62	1310	21.9	21	11.1
45	41	2690	18.5	23	N.M.
45	48	2300	19.3	20	N.M.
45	55	1960	19.8	21	N.M.
45	62	1740	18.1	17	14.2

* N.M.

Converion Factors: 1 M/min = 3.28 FPM, 1 DTEX = 0.9 Denier and 1MN/DTEX = 0.11 G/DEN
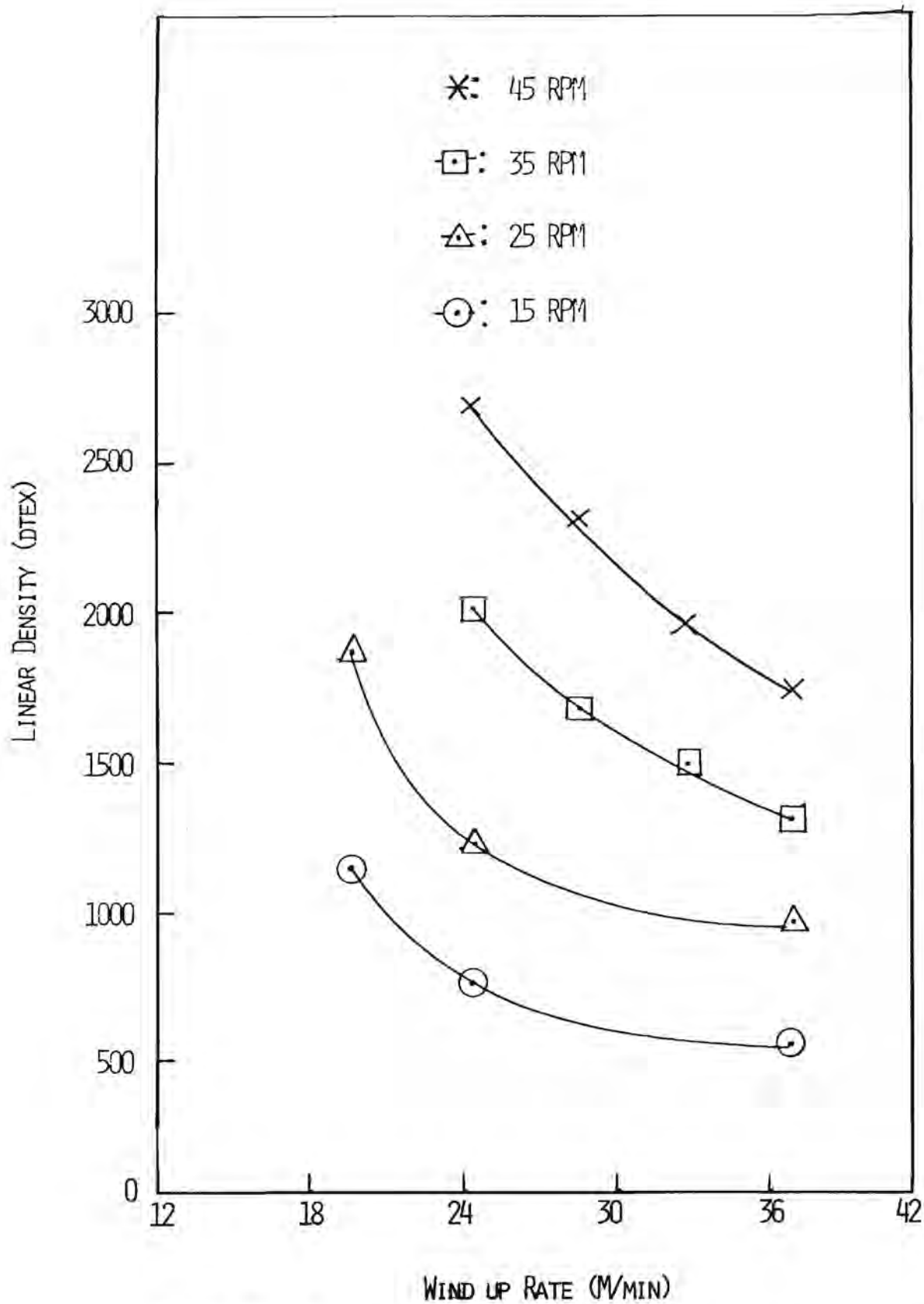
FIGURE 21. VARIATION OF YARN LINEAR DENSITY WITH WIND-UP RATE.

filaments counted in a yarn cross-section did not vary significantly with screw speed and wind-up rate, the filament denier correlates well with the yarn denier - see Figure 22.

As Figure 23 illustrates, the drawn yarn's tenacity increases with decreasing screw speed and increasing wind-up rate. These trends are consistent with the increasing extrusion drawing which occurs when the screw speed is lowered or the wind-up rate is increased.

With the current equipment it is not possible to produce yarn at a screw speed below 15 RPM or at a wind-up rate in excess of 124 ft/min, since the yarn breaks. Based on the property trends observed it is not desirable to produce yarn at very high screw speeds or very low wind-up rates. Under these conditions the extrudate tends to be filmy rather than fibrous. From the trends observed the best yarn properties, i.e. high tenacity and low denier, are obtained at low screw speeds and high wind-up rates.

The maximum draw ratio of several of the samples in Table X were determined by drawing the yarns at $130^{\circ}C$ to twice their original length, twisting 2 TPI and further drawing until break. It can be seen from Table XI that screw speeds do not significantly affect the maximum draw ratio whereas wind-up rates do. Therefore, the samples extruded at 15 RPM were evaluated further by drawing to twice their original length, twisting 2 TPI, drawing to 95% of their maximum draw ratio and finally twisting 2 TPI. The results in Table XI show that yarn produced at the highest wind-up rate still has the maximum tenacity despite the fact that it has the lowest post-extrusion draw ratio. It is noteable that additional drawing in the final drawing step results in a substantial increase in tenacity compared to drawing the yarn 2X in this final drawing stage.
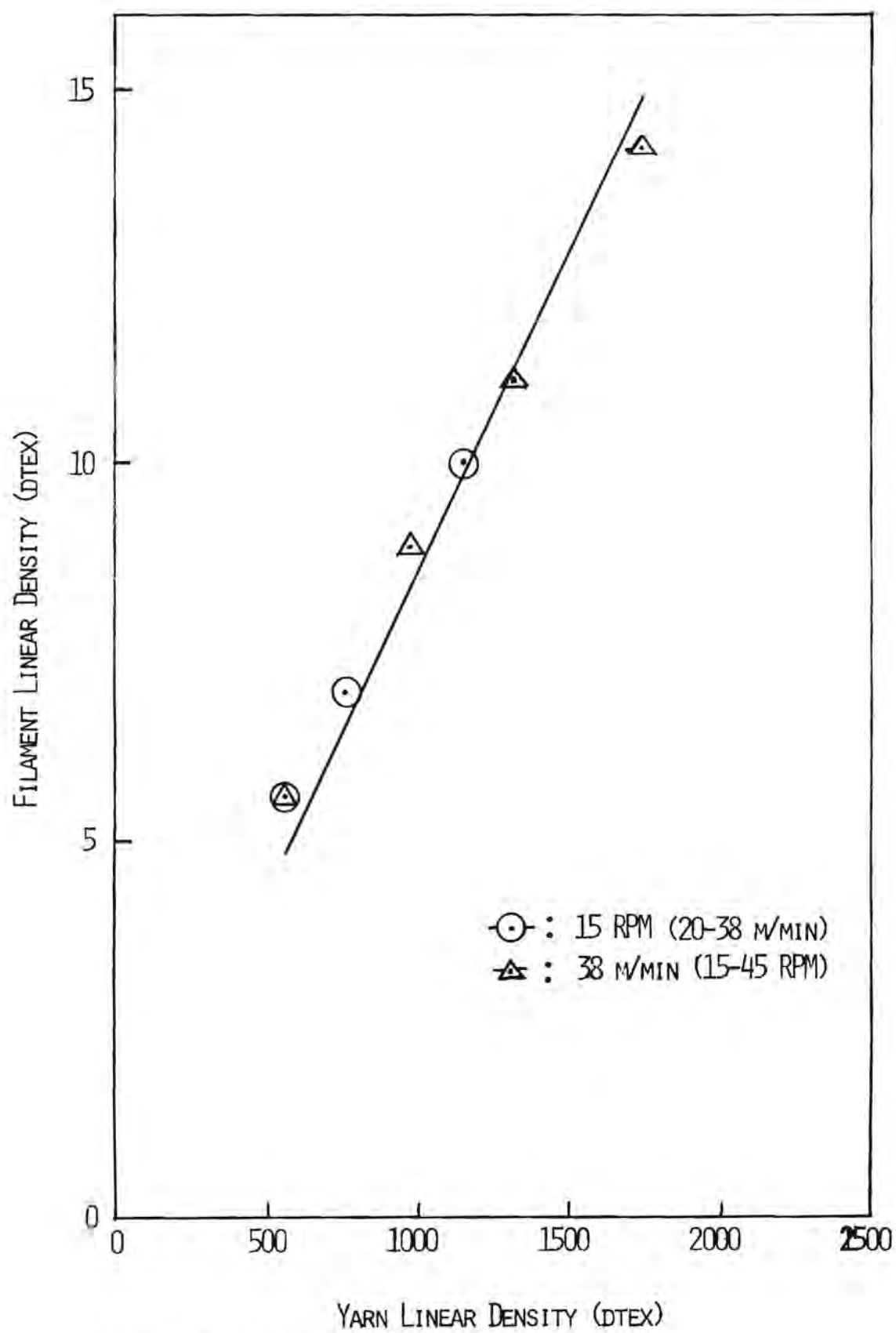
FIGURE 22. FILAMENT LINEAR DENSITY VS. YARN LINEAR DENSITY.
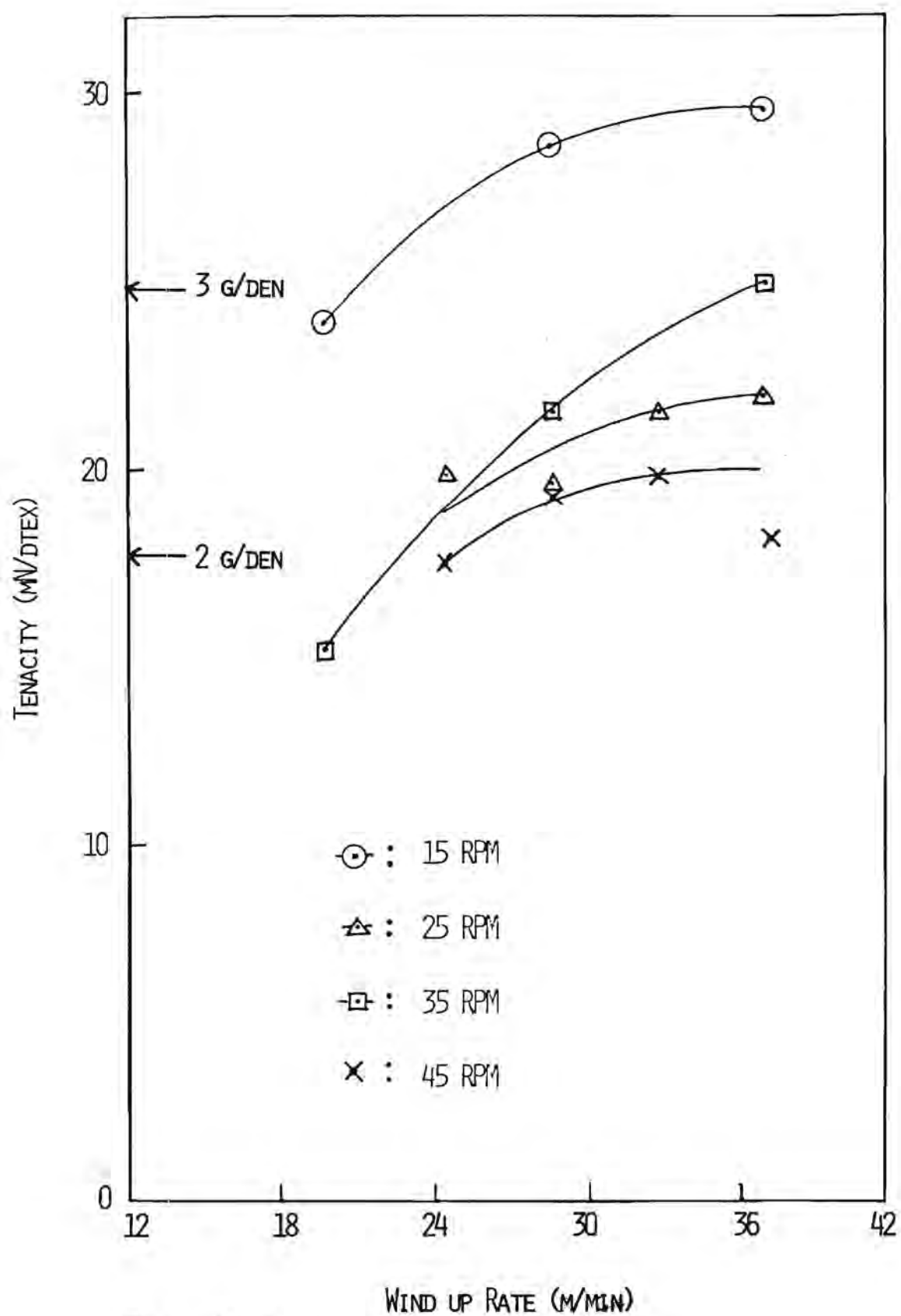
FIGURE 23. DRAWN YARN TENACITY AS A FUNCTION OF WIND-UP RATE AND SCREW SPEED. —75—

Table XI

Effect of Screw Speed and Wind-up Rate on Maximum
Yarn Tenacity and Minimum Yarn Linear Density

Process Conditions		Properties		
Screw RPM	Wind-up Rate (cm/s)	Max. Draw Ratio	Yarn linear density (dtex)	Tenacity (mN/dtex)
15	33	5.73	844	29.3
15	48	5.20	618	37.4
15	62	4.93	480	41.9
25	62	5.02	N.M.	N.M.
35	62	4.91	N.M.	N.M.
45	62	4.71	N.M.	N.M.

N.M. Not Measured

Conversion Factors: 1m/min = 3.28 FPM, 1 DTEX = 0.9 Denier and 1mn/DTEX =

0.11 G/DEN

e. Low denier yarns

In order to evaluate the properties of yarn less than 150 denier, as-extruded yarn was split axially before drawing and twisting. Yarn extruded at a screw speed of 15 RPM and a wind-up rate of 124 ft/min was split into two and then four axial sections. These samples were drawn 2X, twisted 2 TPI, drawn to 95% of their ultimate draw ratio and then twisted 2 TPI. As Figure 24 illustrates, the tenacity decreases as the denier is reduced by splitting while the ultimate elongation is virtually unchanged. The decline in tenacity is attributed to a reduction in the fraction of connected fibrils as the number of splits increases. Based on Figure 24, a 150 denier yarn would have a tenacity of 3.4 g/denier with an elongation of 20%.

f. Continuous drawing and twisting

Based on the promising results obtained by hand drawing and twisting, continuous processing was investigated. The design of a continuous drawing system was reported earlier (25). The hot silicone oil bath used in this drawing system caused the polypropylene yarn to fracture. Thus, this equipment could not be utilized.

Polypropylene yarn was successfully drawn using the system shown in Figure 10. The drawing oven is a 7' long wooden box which is 4" by 4" in cross section. The box is positioned between calibrated feed and take-up rollers. Hot air is supplied at specific temperatures by an electrically heated air dryer (Desikator [R], Polymer Machinery Corporation). The air is recycled back to the dryer. The desiccant bed in the dryer was bypassed, hence, no moisture was removed from the air.

Twisting was accomplished by rotations of a bobbin of yarn about a vertical axis and continuously collecting the yarn from the bobbin at a calibrated rate (see Figure 9).

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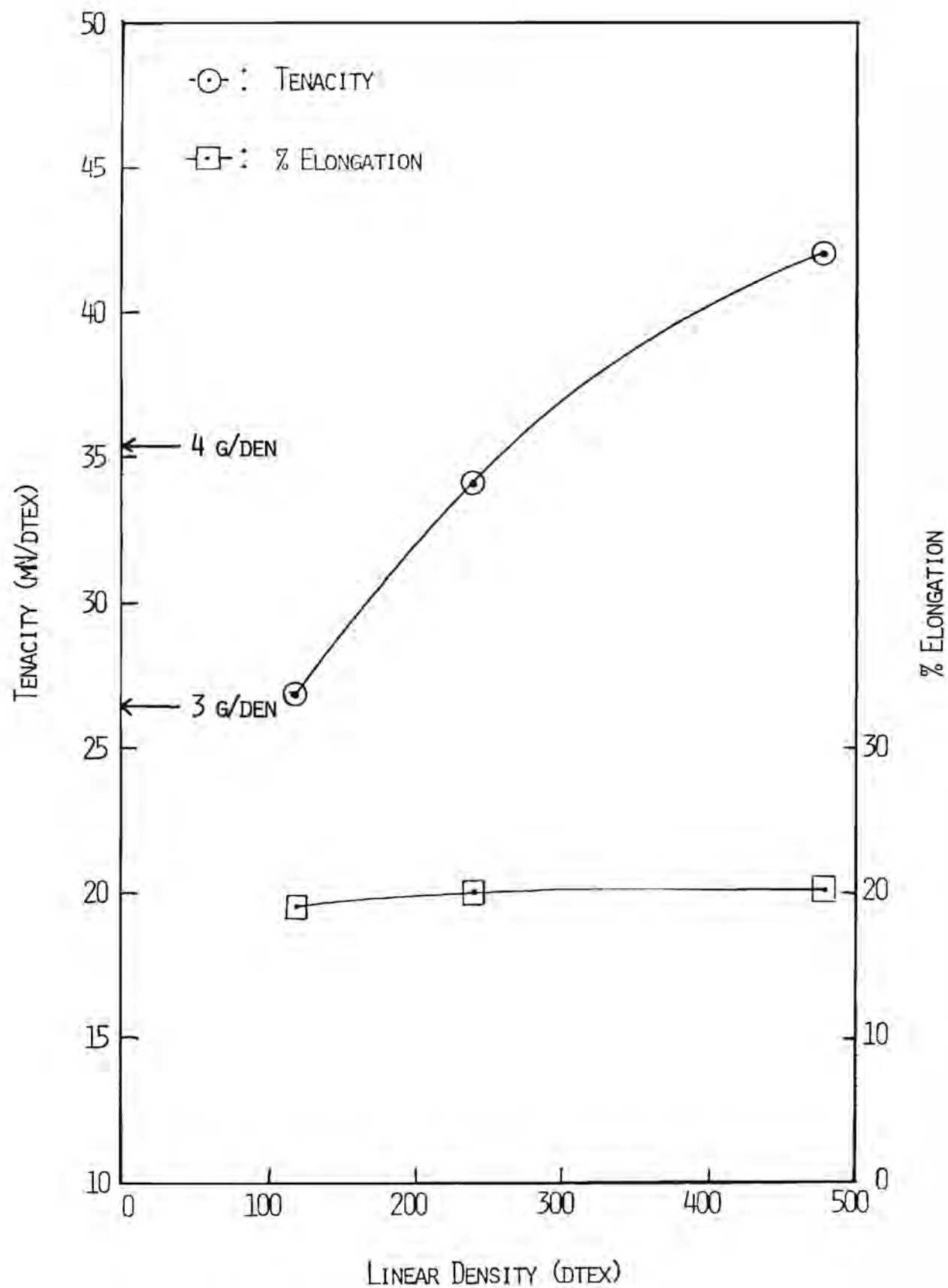
FIGURE 24. YARN TENACITY AND ELONGATION VS. YARN LINEAR DENSITY.

The as-extruded yarn used for studying continuous drawing and twisting was obtained from a feed mixture of 99 wt. % polypropylene (Hercules Profax 6323) and 1 wt. % azodicarbonamide (Kempore 200 MC). The screw speed and wind-up rate were set at 20 RPM and 65 ft/min respectively. These process conditions are below optimum but permit continuous operation in excess of one hour in order to obtain large samples. The as-extruded yarn had a denier of 4,500, a tenacity of 0.27 g/denier and an ultimate elongation of 168%.

A factorial experiment was designed to investigate the effect of oven temperature and feed speed on yarn tensile properties. The four average oven temperatures were 95, 112, 130 and 142°C, as measured by several thermometers along the wooden box. The oven feed speeds chosen were 10, 50 and 100 feet per minute. For each temperature and feed speed the drawing rolls were set at twice the speed of the feed rolls. After twisting the extruded yarn 2 TPI, the yarn is drawn. Next the yarn denier is measured and the twist adjusted to 2 TPI. Then the tenacity and elongation were measured.

It was found that due to slippage and relaxation the actual draw ratio was significantly less than the nominal draw ratio of 2. The actual draw ratio varied with feed speed, decreasing as feed speed increased. The oven temperature did not affect the actual draw ratio within the 95 to 142°C range studied.

For the experimental conditions studied the tenacity was found to be directly proportional to the actual draw ratio. The drawing temperature did not influence the tenacity and the feed speed only affected the tenacity by altering the actual draw ratio.

In order to test the dependence of tenacity on draw ratio a second set of experiments was run to extend the range of draw ratios. The feed speed was fixed at 50 feet per minute while the nominal draw ratio was varied from 2 to 4.

The oven temperature was varied over the same range as before and the final twist of the yarn was adjusted to 2 TPI. The results are shown in Figure 25, which confirms that tenacity is proportional to actual draw ratio.

Since the yarn exhibits a tendency to break during drawing when the nominal draw ratio is set above 2, a second cycle of twisting and drawing was attempted. After imposing a nominal draw ratio of 2 and retwisting the yarn to obtain 2 TPI, the yarn was redrawn with nominal draw ratios between 1.2 and 2.2 at a feed speed of 50 feet per minute and $130^{\circ}C$ oven temperature. After adjusting the yarn twist to 2 TPI the tenacity and elongation of the yarn was measured. The tenacity versus actual draw ratio has the same slope and intercept as in Figure 25. Thus, interrupting drawing in order to impose additional twist on the yarn does not alter the dependence of tenacity on actual draw ratio. However, by using a second twisting and drawing cycle it was possible to achieve relatively high draw ratios without frequently breaking the yarn during drawing.

As shown in Figure 26, there is no apparent difference in the mechanical properties of continuous and hand drawn yarn. Although the data in Figure 14 show that tenacity and elongation depend on drawing temperature, this temperature dependence is minor in comparison to the dependence on actual draw ratio.

Childs (27) performed a structural analysis of continuously drawn yarn. As seen in Figure 27 both birefringence and Hermans' crystalline orientation function increase with draw ratio. Thus, both molecular and crystalline orientation occurs during drawing. Sheehan, Wellman, and Cole (28) have correlated the tenacity of polypropylene monofilament with Hermans' orientation function. A comparison of their results with foam fibrillated yarn is given in Figure 28. The difference in crystalline orientation at the same tenacity is probably due to differences in techniques employed in determining the Hermans' orientation function. The general trend of an increase in tenacity with increasing orientation
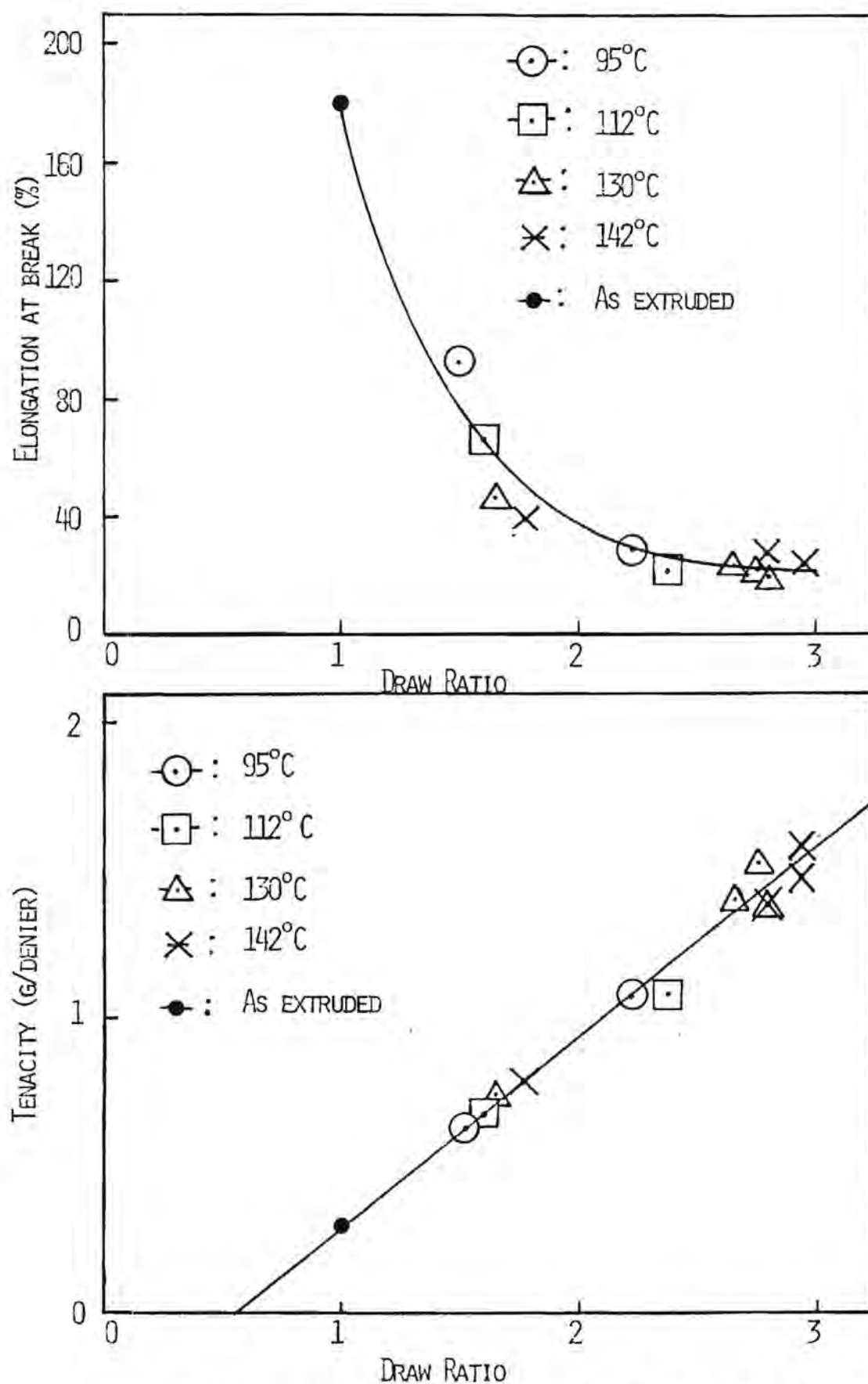
Figure 25. Effect of Oven Temperatures and Actual Draw Ratio on
Yarn Tenacity and Elongation.
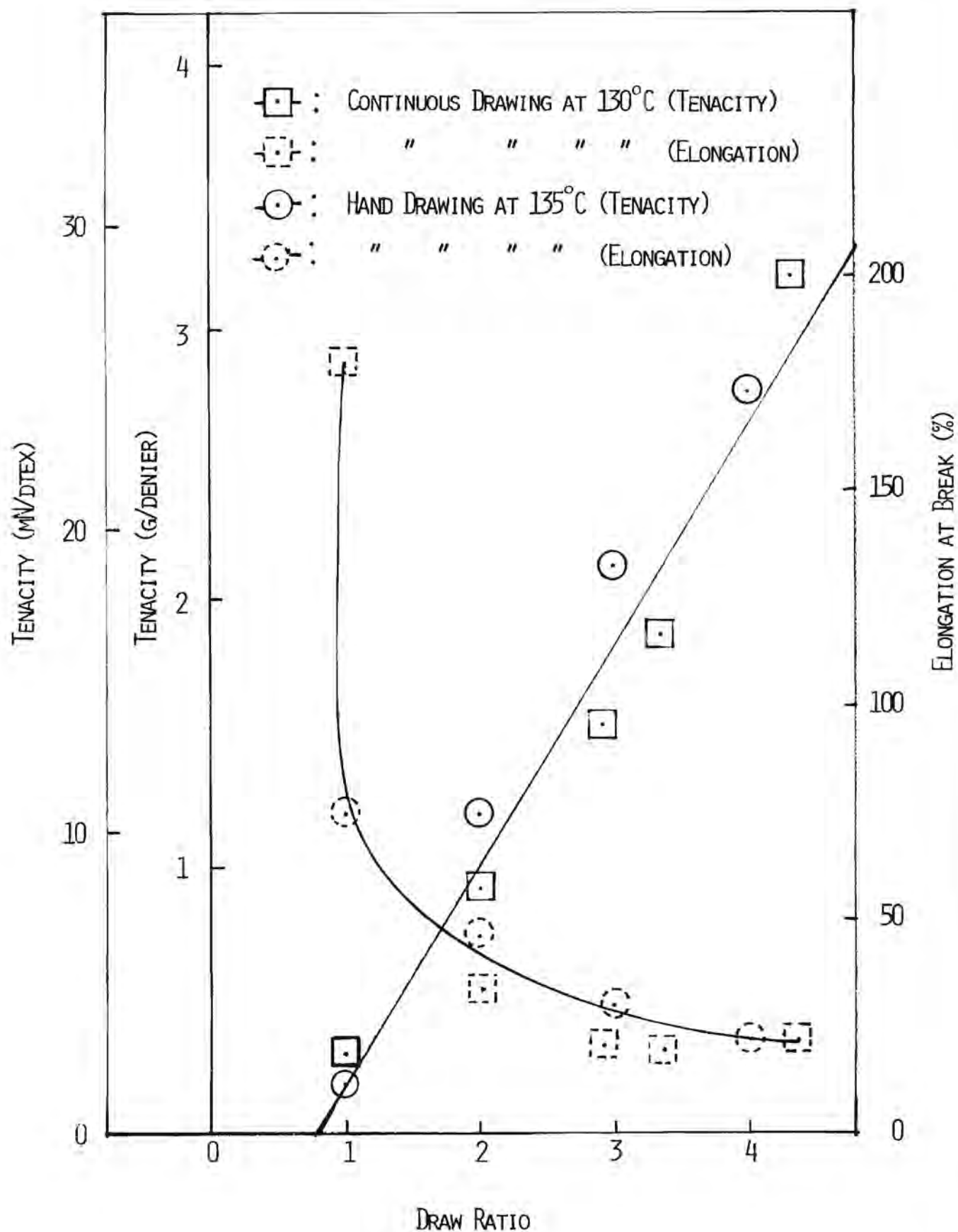
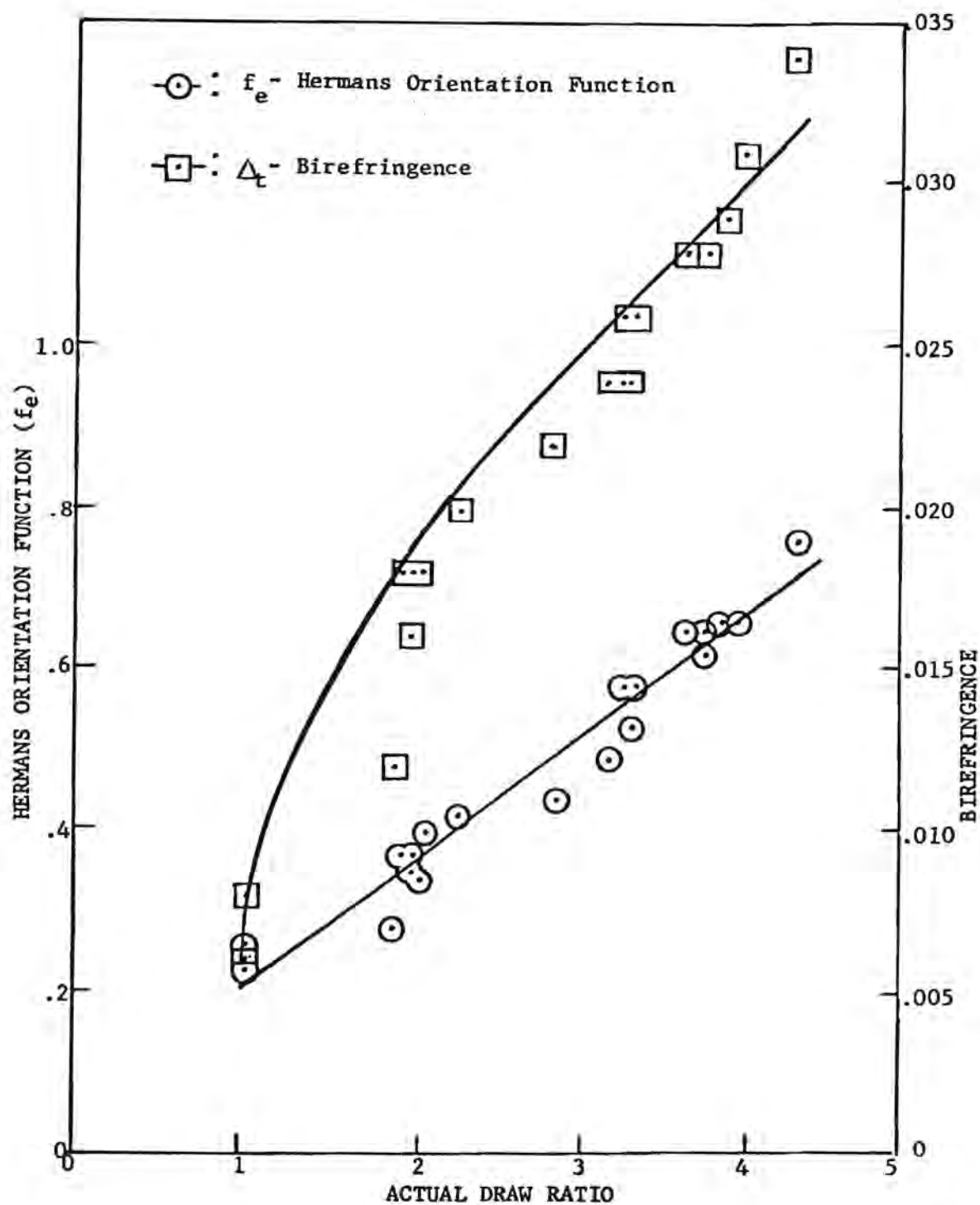Figure 26.   Comparison of Hand and Continuous Drawn Yarns.

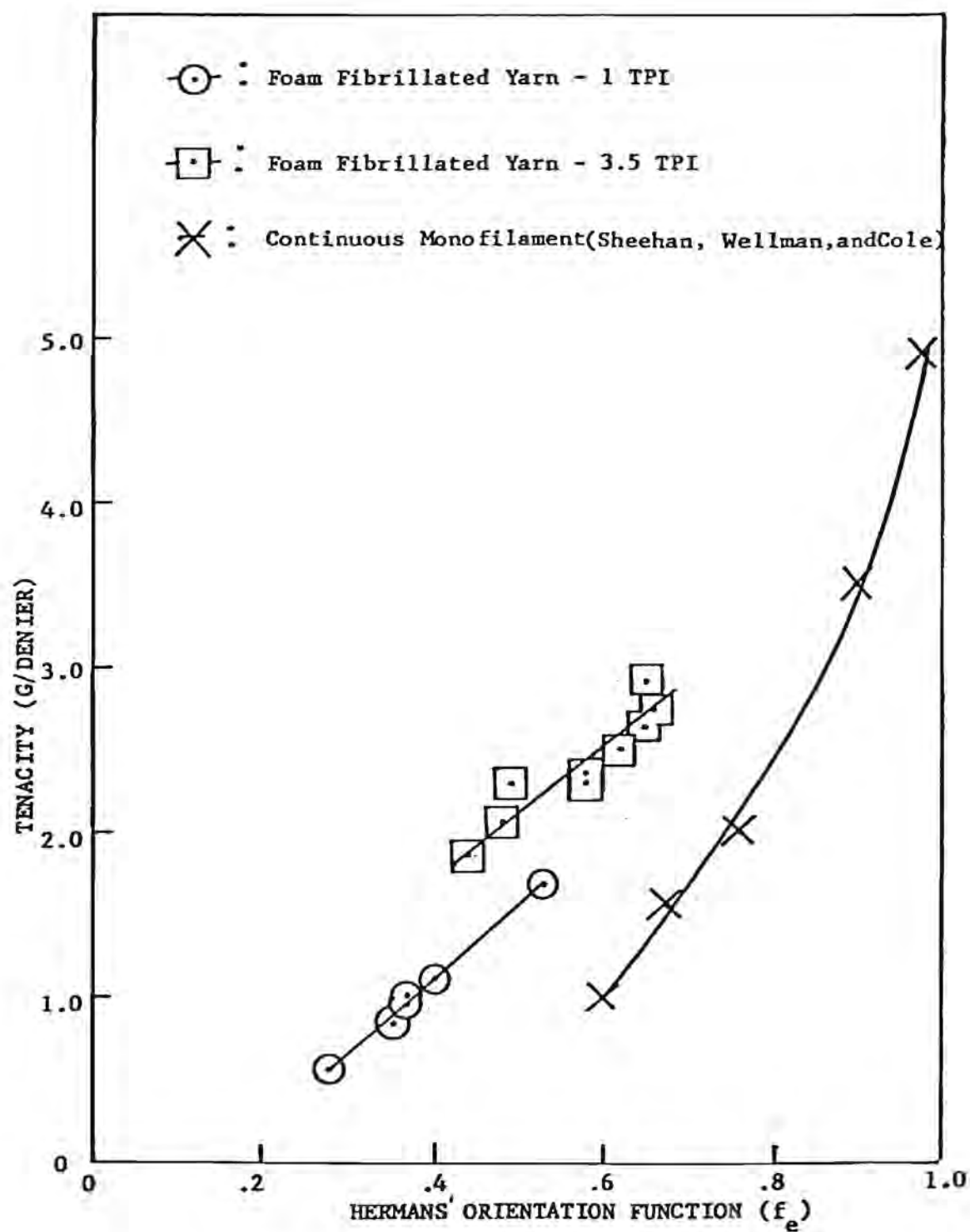Figure 27. The Hermans' Orientation Function and Birefringence as a Function of the Actual Draw Ratio

Figure 28. Comparison of Yarn Tenacity of Foam
Fibrillated Yarn and Monofilament as
a Function of the Hermans' Orientation
Function

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is valid for both yarns. Also, the fact that the foam fibrillated yarn is split into a number of connected filaments does not appear to harm the mechanical properties of the yarn. The controlling factor on tenacity is the degree of orientation achieved.

Childs (27) also studied the morphology of foam fibrillated yarn. Figure 29 reveals that the filaments have a ribbon shaped geometry. It was found that the number of filaments per cross section increases with increasing draw ratio. The number of filaments in one yarn sample increased from 122 for the undrawn yarn to 256 and 287 after drawing the yarn two times and four times, respectively.

The size distribution illustrates the number of filaments with a specific dimension for both the width and the thickness of the filament ribbon. Figures 30, 31 and 32 show the size distribution for yarn samples with undrawn conditions, a draw ratio of two, and a draw ratio of four, respectively. In these figures, the length of the lines indicate the relative number of filaments with the specified size (1 mm - 1 filament). The change in size distribution is noticeable as the ribbons become smaller and the distribution becomes sharper.

Figure 33 contrasts the undrawn yarn to a yarn with a draw ratio of two. The axial view points out the increase in fine texture accomplished by drawing.

g. Fabric formation

Seven thousand feet of yarn were extruded continuously and then drawn in two steps with intermediate twisting at an oven temperature of $130^{o}C$ and a feed speed of 50 feet per minute to obtain yarn having a denier of 1200. Two fabrics were woven from this yarn using an 18" wide hand loom. The first fabric was woven for display purposes; it has 20 filling yarns/inch and 11 warp yarns/inch. Non-destructive tests indicate an air permeability of 135 ft/min and a basis

Figure 29.   Foam Fibrillated Yarn
               Cross Section with
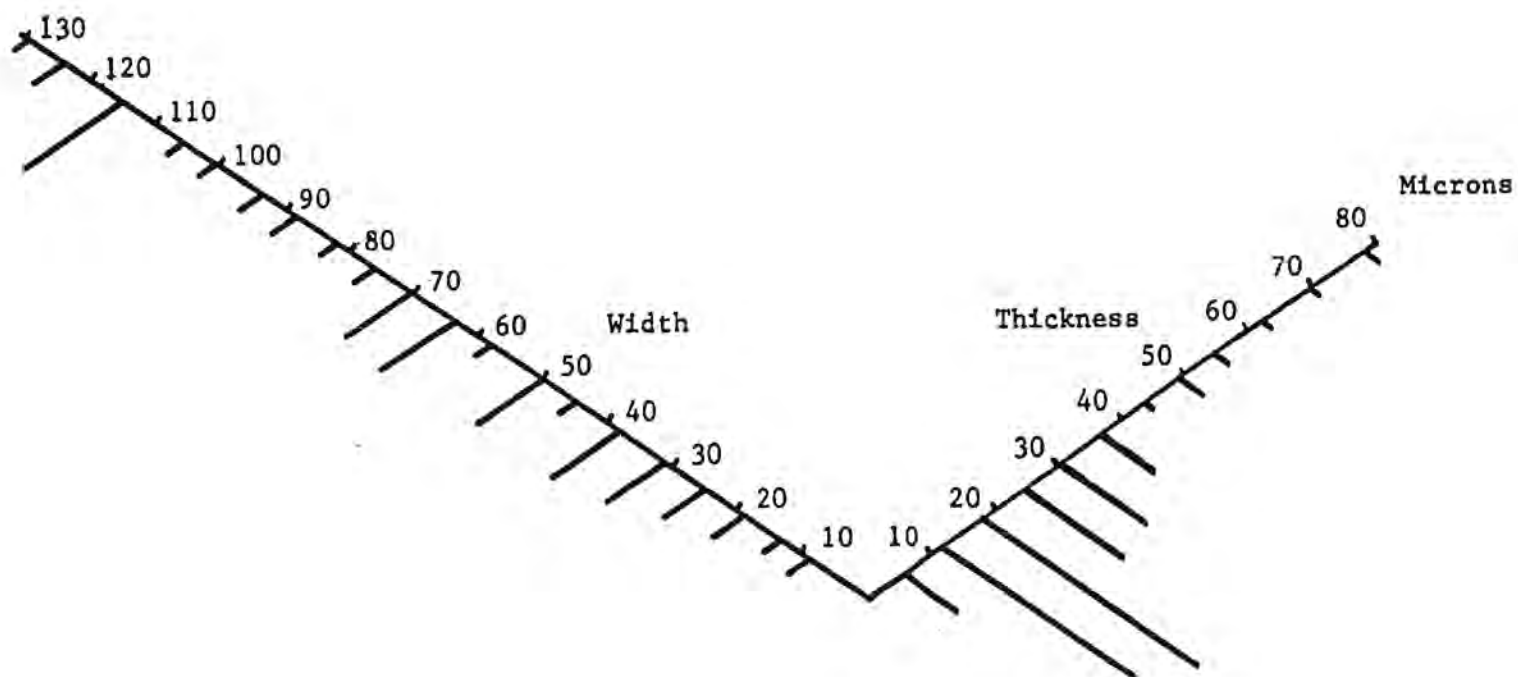   Actual Draw Ratio of 2

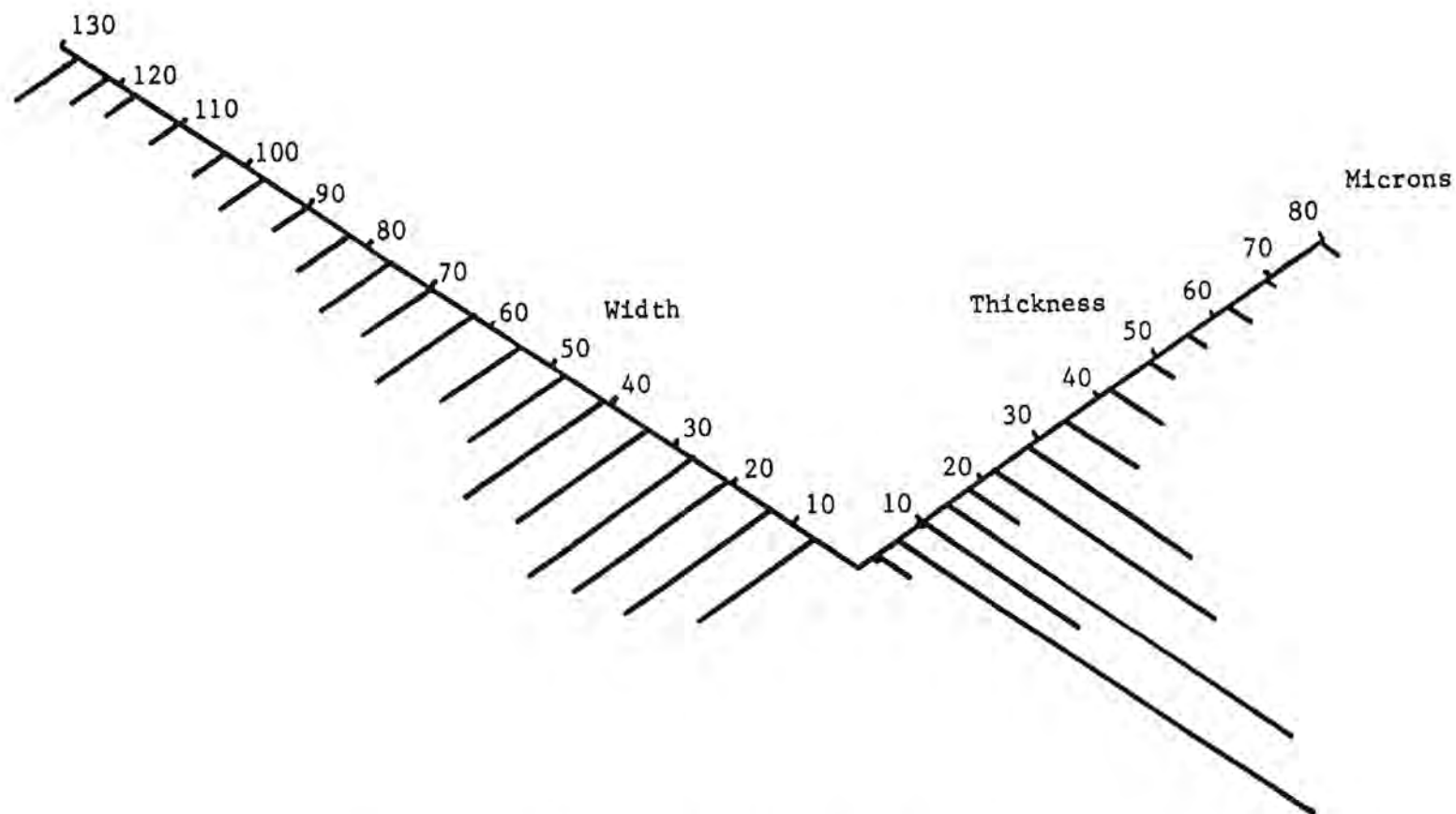Figure 30.   Filament Size Distribution of an Undrawn Yarn

Figure 31. Filament Size Distribution of a Yarn With Actual Draw Ratio of 2
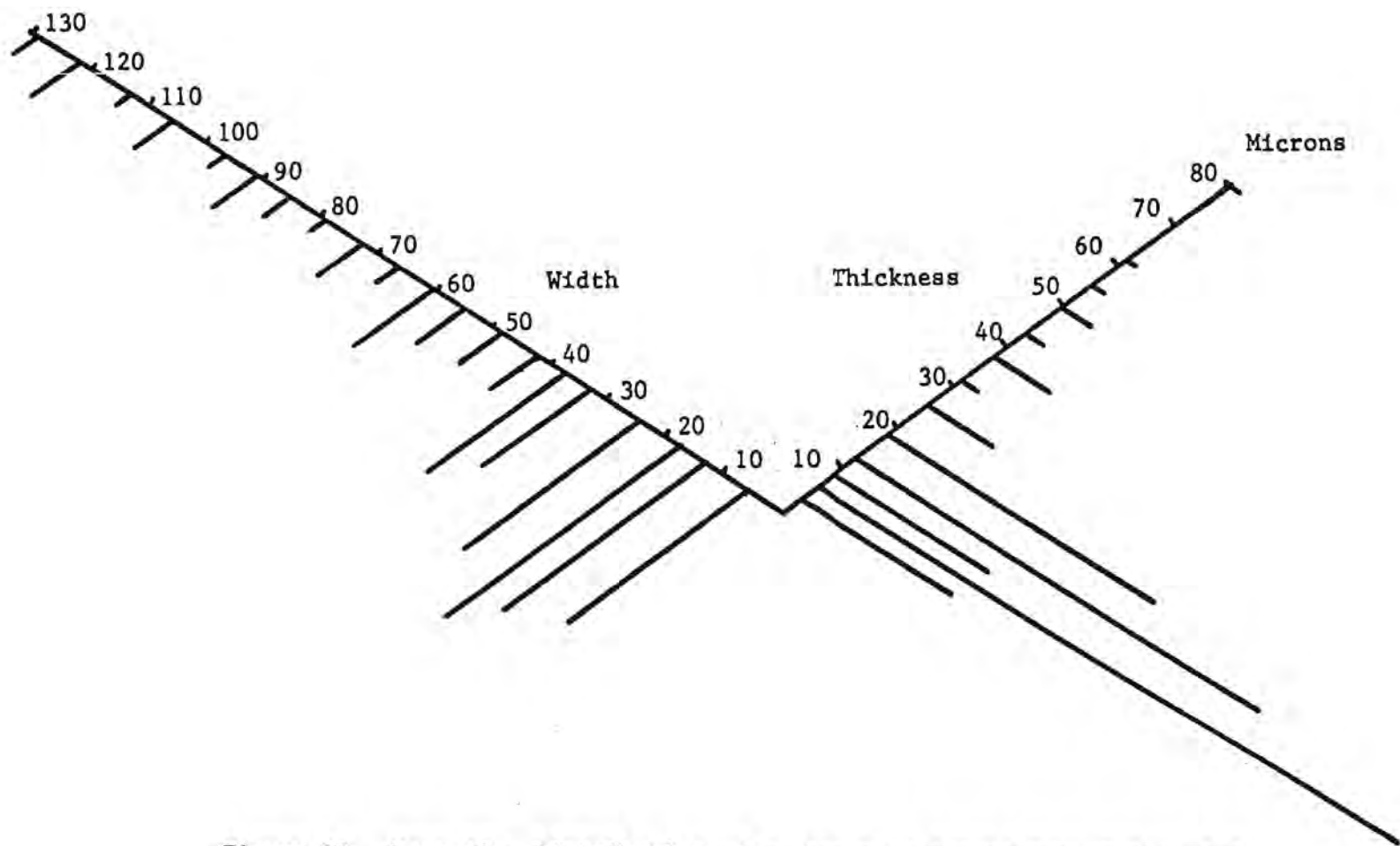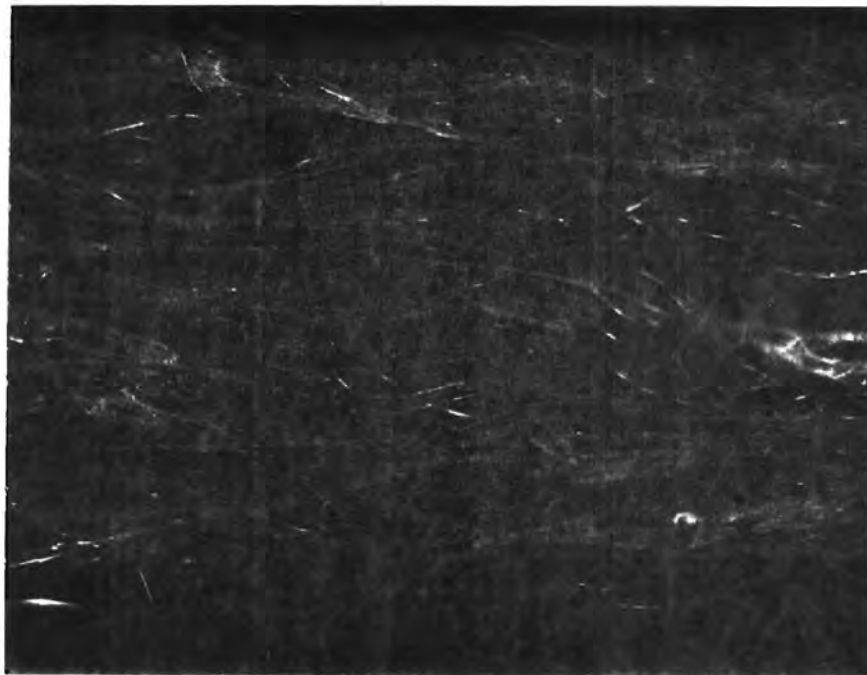
Figure 32.   Yarn Size Distribution of a Yarn with Actual Draw Ratio of 4

(a)

(b)

Figure 33.   Longitudinal View of Yarns
            a)   Undrawn Yarn
            b)   Drawn Yarn(Actual Draw Ratio of 2)

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weight of 5.75 oz/sq.yd. The second fabric was woven for testing purposes and it is described in some detail in Table XII together with several commercial fabrics which were obtained from remnants. Portions of all these fabrics were unraveled in order to test the individual yarns in both filling and warp directions. Foam fibrillated yarn properties are well in the range of commercial yarn properties. The tenacity of the experimental yarn can be increased by additional drawing and twisting. Table XIII is a comparison between the experimental and commercial yarns taken from these fabrics and it shows that foam fibrillated yarn may be used successfully in the manufacture of different types of fabrics. The most striking feature of the experimental fabric is its low air permeability. Fabric properties can be significantly altered by simply changing the number of yarns per inch in either filling or warp directions.

h. Economic analysis

Figure 34 is the proposed flowsheet for large scale production of foamed polypropylene yarn. The as-extruded web is slit into two halves and each half drawn 2X in a hot air oven set at 130°C. The webs are then slit into a number of ribbons which varies depending on the desired denier of the processed yarn. Each ribbon is given 2 TPI, collected in the twister spindles, drawn again 2X at 130°C and finally taken up on individual winders.

As the base case for evaluating the rate of return on investment in a foam fibrillated yarn plant it is assumed that 120 ribbons having a final denier of 900 are produced at a mass flow rate of 310 lb/hr. This is equivalent to having final line speeds of 640 ft/min. It is also assumed that the equipment is depreciated over a 10 year period and that 3% of the raw materials are wasted but can be recycled at a cost of $0.09/lb.

Table XIV is a list of the major pieces of equipment and approximate costs. An estimation of total capital investment was made following the

TABLE XII

COMPARISON OF PROPERTIES OF COMMERCIAL
FABRICS WITH EXPERIMENTAL FABRIC

		FABRIC TYPE			
	DIRECTION	EXPERIMENTAL	UPHOLSTERY	CARPET BACKING A	CARPET BACKING B
BASIS WEIGHT (OZ/SQ.YD.)		9.0	17.2	3.4	4.4
AIR PERMEABILITY ($FT^3/FT^2/MIN$)		40	137	748	525
TENACITY (LB/IN/OZ/SQ.YD)	FILLING WARP	18.6 4.6	2.7 12.6	22.4 18.4	6.4 9.2
ELONGATION (%)	FILLING WARP	31 38	50 45	53 40	25 33
FLEXURAL RIGIDITY (MG.CM)	FILLING WARP	5,400 2,900	1,100 7,300	500 800	1,500 800

TABLE XIII

COMPARISON OF YARNS FROM EXPERIMENTAL FABRIC WITH YARNS FROM COMMERCIAL FABRICS

	DIRECTION	FABRIC TYPE			
		EXPERI-MENTAL	UPHOLSTERY	CARPET BACKING A	CARPET BACKING B
TYPE YARN	FILLING	FOAM FIBRILLATED	MULTIFILAMENT (HERCULON)	MULTI-FILAMENT	MECHANICALLY FIBRILLATED
	WARP	FOAM FIBRILLATED	MULTIFILAMENT (HERCULON)	SLIT FILM	SLIT FILM
NO. OF YARNS PER INCH	FILLING	30	12	9	9
	WARP	11	16	9	12
YARN DENIER	FILLING	1,200	4,740	1,740	2,400
	WARP	1,200	1,720	930	1,030
YARN TENACITY (G/DEN)	FILLING	2.4	0.4	2.5	1.0
	WARP	2.0	4.1	3.7	2.5
YARN ELONGATION (%)	FILLING	25	50	31	12
	WARP	23	43	20	15
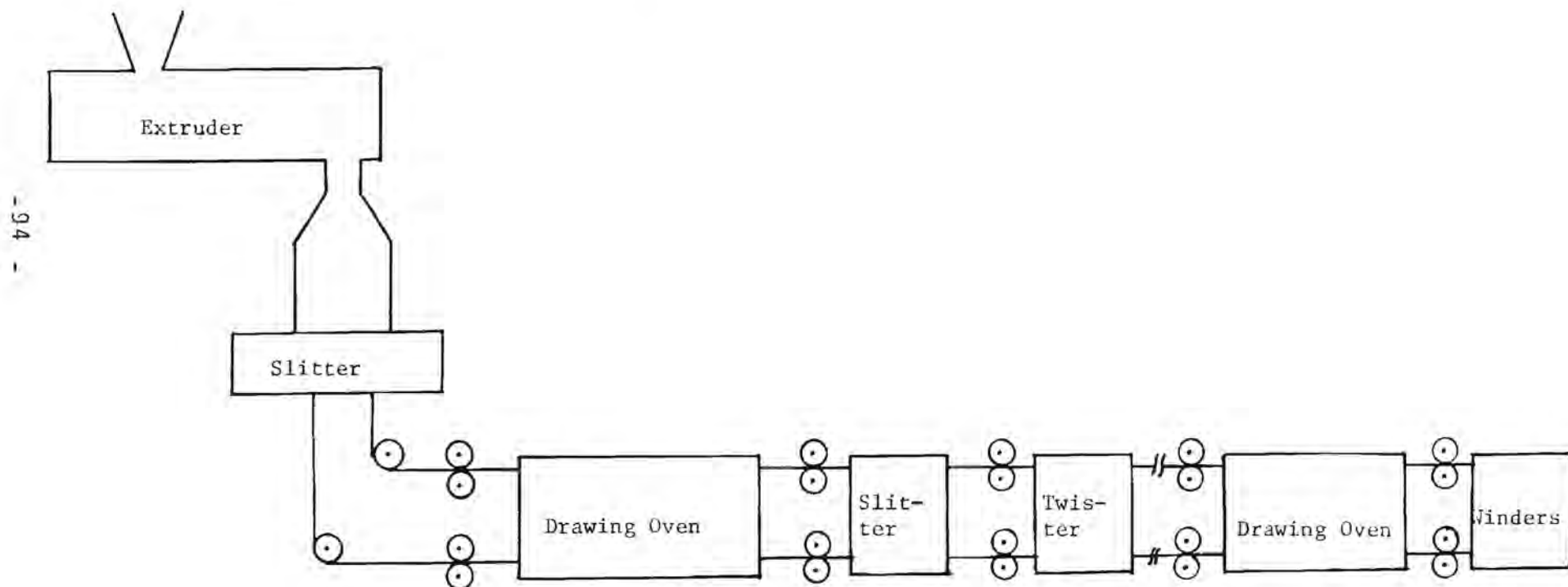
Figure 34. Flowsheet for the Production of Polypropylene Yarn from Foam.

guidelines in Peters and Timmerhaus (29). According to their procedure, the total capital investment is broken into its major components and each component is assigned an empirical cost factor which is some percentage of the purchased equipment cost. These cost factors were taken directly from Neil and Waechter (30) for a polymer processing plant and are generally lower than factors encountered in chemical plants. The total product cost was also estimated following the procedures outlined by Peters and Timmerhaus (29). Table XV is a summary of capital investment, product cost, and profitability figures for the base case assuming a selling price of $0.90 per pound of yarn.

Since the assumptions made for the base case cannot be fully confirmed until careful pilot plant runs are made, it is interesting to modify the base case in order to examine the sensitivity of the rate of return to some of the process variables. Three modifications are summarized in Table XVI. The three modifications are: (a) increasing output from 310 to 385 lb/hr (b) lowering the denier from 900 to 225 and (c) adding a texturing step following the final drawing operation. In all cases the rate of return is affected appreciably.

&. Conclusions

Polypropylene web, formed by extruding foam and biaxially stretching it over a circular mandrel, can be converted into yarn with properties which make it suitable for some textile applications. This is accomplished by twisting and drawing following extrusion.

Most of the filaments comprising the yarn are connected due to the original foam structure of the extrudate. They are not circular in cross-section and exhibit a distribution in denier. The yarn exhibits high tensile properties intermediate between staple fiber and split film yarn.

For drawing temperatures between 95 and 142°C and feed speeds from 10 to

TABLE XV

TOTAL PRODUCT COST AND PROFITABILITY ANALYSIS FOR THE
PRODUCTION OF POLYPROPYLENE YARN FROM FOAM (BASE CASE).

		ANNUAL COST ($)	UNIT COST ($/LB)
I.	MANUFACTURING COST		
	A. TOTAL DIRECT PRODUCTION COST	1,228,000	0.472
	B. FIXED CHARGES	103,500	0.040
	C. PLANT OVERHEAD	182,000	0.070
	TOTAL MANUFACTURING COST	1,513,500	0.582
II.	TOTAL GENERAL EXPENSES	251,000	0.096
III.	TOTAL PRODUCT COST	1,765,000	0.679
IV.	PROFITABILITY		
	GROSS ANNUAL SALES	2,340,000	0.90
	NET INCOME	299,000	
	TOTAL CAPITAL INVESTMENT	975,000	
	RATE OF RETURN = 31%		

TABLE XVI

Summary of Process Conditions and Economics for the Production of Polypropylene Yarn from Foam

Case:	Base	High Production Rate	Low Denier	Texturized Yarn
Extrusion rate, lb/hr	310	385	310•	310
No. of ribbons	120	120	480	120
Denier	900	900	225	900
Final line speed, ft/min.	640	800	640	640
Texturized	No	No	No	Yes
Investment, 10^3\$	975	975	2055	1125
Product cost, ¢/lb	68	62	79	69
ROR at 90¢/lb, %	31	48	7½	25

100 feet per minute, the tenacity obtained is linearly dependent on the
actual draw ratio achieved. A two cycle twist and draw sequence facilitates
drawing to higher draw ratios but does not alter the linear dependence of
tenacity on actual draw ratio.

Fabric samples exhibit properties similar to upholstery and carpet
backing samples. The air permeability of the experimental fabric is substan-
tially lower than the commercial fabrics tested.

Economic analysis of a proposed semi-continuous polypropylene yarn plant
which uses a slitting operation and a two step twist-draw sequence indicates
rates of return which are high but very dependent on production capacity and
final yarn denier. Pilot plant data is required to determine the properties
and structure of yarn produced from a large extruder since this would determine
the commercial utility of foam fibrillated polypropylene yarn.

C.   Fiber Orientation in Air Flows

1.   INTRODUCTION

The process of fiber weaving involves a direct step from individual fibers to fabrics. Intermediate steps of spinning staple fiber into yarn, and of weaving yarn into fabric, are omitted. If a fiber-weaving process could be developed, it would offer the possibility of increased productivity and of reduced material waste and possibly energy consumption. A major requirement for a fiber-weaving process is that the fibers must be arranged in orderly patterns. This is so that the fiber-woven fabrics will have aesthetic and performance characteristics comparable to those of convention-ally-woven fabrics. In particular, the fibers in the final fabric should be aligned principally in two perpendicular directions, so that the fibers retain bending and sliding characteristics similar to those of warp and fill yarns in conventionally-woven fabric.

The basic steps in fiber weaving are: (1) separation of material into individual fibers; (2) alignment of fiber in prescribed directions; (3) deposition of fibers in parallel arrays; and (4) entangling or bonding the fibers. The present study has been concerned with discovering methods for aligning and depositing fibers in a desired manner by using air flows and aerodynamic forces. There are existing techniques that are suitable for obtaining individual fibers and for entangling or bonding fiber webs.

In this research transverse air jets were used to control the main stream speed and direction. For certain conditions the fibers rotated so that they were parallel to the main stream. An important characteristic of this alignment process is that it can be performed with low-speed air flows. This is especially significant when it is desired to deposit the the fibers on a surface while maintaining their alignment.

The use of the transverse air jets is very advantageous. This is primarily because the effective flow geometry can be easily and rapidly altered to effect changes in various parameters.

2.   Dynamics of Single - Fiber Motion

There have been several studies of the movement of fibers and other solid matter by air flows for textile-industry related appliations (31-36). Only the work of Edberg is related to the problems of straightening and orienting, as well as transporting fibers. In his studies, air flows with fibers were observed in straight ducts which had different degrees of convergence. He found that large percentages of the fibers could be made parallel. To do so, however, required high air speeds (30 to 100 m/sec), which is undesirable for ordered deposition of the fibers.

There have also been a number of studies of the fluid mechanics of fiber suspensions (37-42). These studies are related to such problems as the resistance to motion of these fiber suspensions, and so to pumping requirements, etc.

As stated above, the present research is concerned with orienting, transporting and depositing the fibers. Once the fibers have been deposited in an ordered way, they must be intertwined. A technique that is potentially applicable is that due to Evans (43). Evans' process consolidates any web, mat, or batt of loose fibers, supported on an apertured surface such as a perforated plate or a woven wire screen, by the use of high-pressure above (200 psig) liquid jet streams impinging perpendicularly to the mat. This process produces stable, strong fabrics which resemble textile fabrics prepared by conventional spinning and weaving.

An analysis of the dynamics of a single fiber is presented below. A number of simplifications are introduced, especially regarding the interaction

of the counterflow jets and the free stream flow. Because of these simpli-
fications, only qualitative comparisons can be made between the numerical
predictions of fiber behavior and the actual photographed behavior. These
qualitative predictions have proved to be very valuable, however, in providing
insight on the basic mechanisms that produce fiber alignment. Section 2.2
provides a brief comparison of numberical predictions with photographic results.

2.1 Equations of Motion

In the analysis, the fiber was represented by a rigid cylinder. Figure
35 shows the fiber nomenclature. The motion of the fiber was assumed to be
two-dimensional, i.e., parallel to the xy-plane. The equations of motion of
the fiber are then as follows:

Conservation of x-momentum: $\quad F_y = m_f \cdot du_G/dt$ $\qquad\qquad\qquad$ (15)

where, $\quad u_G = dx_G/dt$ $\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad$ (16)

Conservation of y-momentum: $\quad F_y = m_f \cdot dv_G/dt$ $\qquad\qquad\qquad$ (17)

where, $\quad v_G = dy_G/dt$ $\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad$ (18)
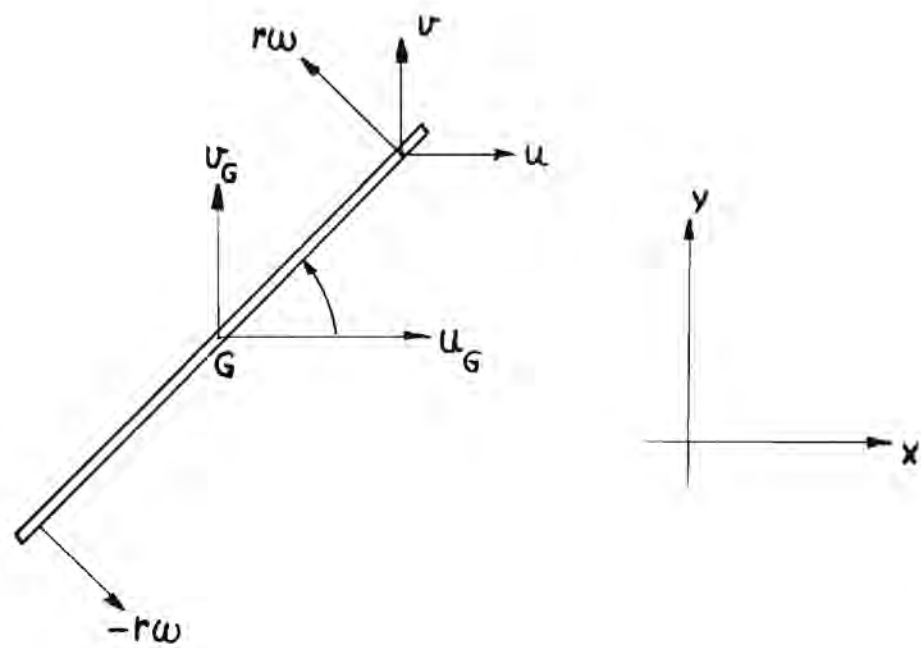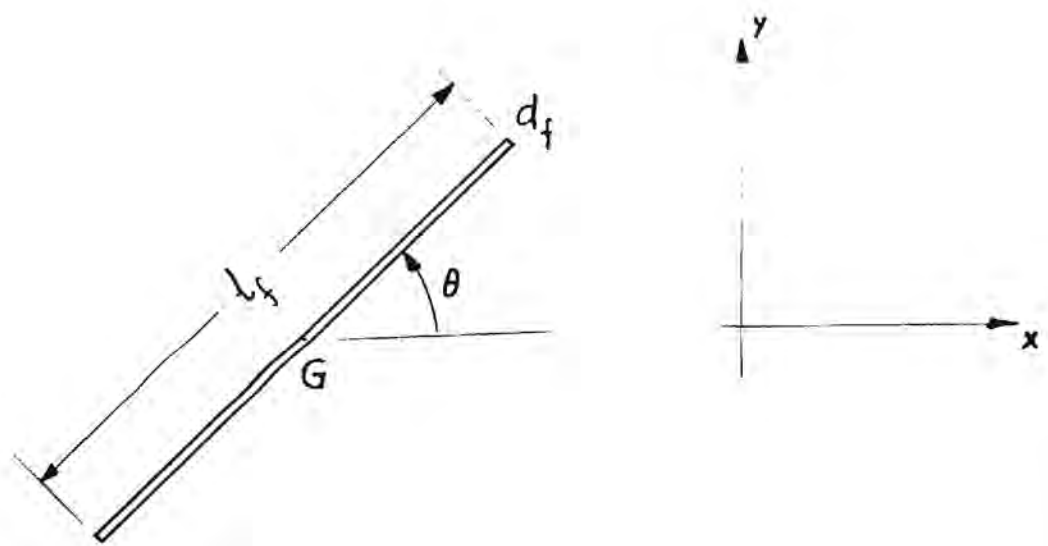
Figure 35.   Fiber Nomenclature

Conservation of z-angular momentum about the fiber center of mass G:

$$M_G = I_G \, d\omega/dt \tag{19}$$

where, $\qquad\qquad \omega = d\theta/dt \tag{20}$

All quantities in the preceding equations were made nondimensional by the following reference conditions: free stream density, ρ_∞ ; free stream velocity, u_∞; and duct height, h. F_x and F_y are the components of the resultant force on the fiber, and M_G is the resultant moment about G (directed parallel to the z-axis). u_G and v_G are the velocity components of the fiber center of mass. The fiber mass is $m_f = (\pi/4)d_f^2 \, \ell_f \, \rho_f$, where ρ_f is the fiber density. x_G y_G are the spatial coordinates of the fiber center of mass, and t is time. The fiber moment of inertia about G is $I_G = m_f \ell_f^2 /12$.

The resultant forces and moments on the fiber are caused by the relative velocity between the fiber and the surrounding air flow. Let W_n be the nondimensional relative velocity normal to the fiber at a point on the fiber. Then,

$$W_n = (u - u_G) \sin \theta - (v - v_G) \cos \theta \pm r\omega \tag{21}$$

where u and v are the velocity components of the air flow at the point on the fiber, and r is the distance from G to the point on the fiber. The (±) preceding $r\omega$ denotes that one half of the fiber rotates into the air stream, while the other half rotates away from it. Also, let W_τ be the relative velocity tangent to the fiber at a point on the fiber. Then,

$$W_\tau = (u - u_G) \cos \theta + (v - v_G) \sin \theta \tag{22}$$

It should be emphasized that W_n and W_τ vary from point to point along the fiber, because of the spatial variation of u and v, and because of the variation of r. Equations (21) and (22) are valid for $0 \leq \theta \leq 180^o$. For $180^o < \theta < 360^o$, they must be modified to the following:

$$W_n = - (u - u_G) \; \sin \theta + (v - v_G) \cos \theta \pm r\omega \tag{21a}$$

$$W_\tau = - (u - u_G) \; \cos \theta - (v - v_G) \sin \theta \tag{21b}$$

Let F_n' be the nondimensional normal force per unit length of fiber acting at a point on the fiber. This can be written in terms of a drag coefficient C_d as:

$$F_n' = (1/2) \; W_n^2 \; d_f \; C_d \tag{23}$$

C_d is a function of R_n, the Reynolds number based on W_n, d_f, and ν, where ν is the kinematic viscosity of the air. Thus, $R_n = R_\infty \, W_n \, d_f$, where $R = u_\infty h/\nu$. Accurate values of C_d have been found by Choo and Casarella (42) for a wide range of Reynolds numbers. Because of the low relative velocities that occur for the present application, only the lower range of R_n is of interest. Then (42),

$$C_d = (8\pi/R_n S) \; (1 - 0.87S^{-2}) \quad (0 < R_n \leq 1) \tag{24}$$

where, $S = - 0.077215665 + \ell n \; (8/R_n)$

and, $C_d = 1.45 + 8.55 \; R_n^{-0.9} \quad (1 < R_n < 30) \tag{25}$

Next, let F' be the tangential force per unit length of fiber acting at a point on the fiber. Then (42)

$$F'_\tau = (\pi W_\tau / R_\infty) \, (0.55 \, R_n^{1/2} + 0.084 \, R_n^{2/3}) \tag{26}$$

To evaluate the resultant force and moment acting on the fiber, it is divided into (n + 1) segments (n is even) of length $\Delta\ell = \ell_f / (n+1)$, and the values of F'_n and F'_τ at the center of each segment are assumed to apply over the whole segment. Thus,

$$F_n = \sum_{i=1}^{n+1} F'_{n,i} \cdot \Delta\ell \tag{27}$$

$$F_\tau = \sum_{i=1}^{n+1} F'_{\tau,i} \cdot \Delta\ell \tag{28}$$

$$M_G = - \sum_{i=1}^{n/2} r_i \, F'_{n,i} \cdot \Delta\ell + \sum_{i=n/2+1}^{n} r_i \, F'_{n,i} \cdot \Delta\ell \tag{29}$$

This method of evaluating the forces on the fiber assumes that each segment experiences the same force per unit length as on an infinite cylinder immersed in a uniform stream having velocity components u, v.

Here the convention is followed that counterclockwise moments are positive. F_x and F_y are then obtained from,

$$F_x = F_n \sin\theta + F_\tau \cos\theta \tag{30}$$

$$F_y = F_n \cos\theta + F_\tau \sin\theta \tag{31}$$

for $0^{\circ} \leq \theta \leq 180^{\circ}$, while for $180^{\circ} < \theta < 360^{\circ}$, the corresponding formulae are,

$$F_x = - F_n \sin \theta - F_\tau \cos \theta \qquad (30a)$$

$$F_y = F_n \cos \theta - F_\tau \sin \theta \qquad (31a)$$

2.2 Specification of Air Velocity Field

In a general calculation of the fiber motion, the air velocity field u, v = fn (x,y) is also unknown, because the presence of the fiber alters u and v from their undisturbed values. An essential simplification introduced here is that u, v = fn (x,y) are known functions. This velocity field is chosen to approximate that produced by the interaction of a uniform free stream with air jets blowing perpendicular to the free stream. The presence of the fiber is assumed to have a negligible effect on the air velocity field.

The present experimental configuration has a uniform flow between plane parallel walls, with air jets blowing inward from the upper and lower walls. It is well known from studies of jets in cross flows (e.g., Abramovich (45), Taylor (46)) that the jet flow is deflected until it is nearly parallel to the cross flow direction. To estimate the effect of the jets on the main stream, a technique similar to that of Taylor (46) was used. The similarity is that the jets were assumed to have the same effect on the main flow as some combination of sources and sinks parallel to the x-axis. The difference from Taylor is that the present boundary conditions require v = 0 at the upper and lower walls, and also along the central plane of the duct, by symmetry. The flow field between adjacent sources (or, sinks) of an infinite row of sources parallel to the y-axis satisfies these boundary conditions (e.g. Streeter) (47).

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A combination of a uniform velocity in the direction of the positive x-axis, one row of sources, and two rows of sinks was used to approximate the flow field. The resulting formulae for the air velocity components are:

$$u = 1 + C_1 \pi \sinh x_1' / (\cosh x_1' - \cos y')$$

$$- C_2 \pi \sinh x_2' / (\cosh x_2' - \cos y')$$

$$- C_3 \pi \sinh x_3' / (\cosh x_3' - \cos y') \tag{32}$$

$$v = \pi \sin y' \left[C_1 / (\cosh x_1' - \cos y') - C_2 / (\cosh x_2' - \cos y') \right.$$

$$\left. - C_3 / (\cosh x_3' - \cos y') \right] \tag{33}$$

The stream function is given by:

$$\psi = -y - C_1 \tan^{-1} \left[\tan (y'/2) / \tanh (x_1'/2) \right] \tag{34}$$

$$+ C_2 \tan^{-1} \left[\tan (y'/2) / \tanh (x_2'/2) \right]$$

$$+ C_3 \tan^{-1} \left[\tan (y'/2) / \tanh (x_3'/2) \right]$$

where $x_1' = 2\pi(x - x_1)$, $x_2' = 2\pi(x - x_2)$, $x_3' = 2\pi(x - x_3)$ and $y' = 2\pi y$. C_1 is the strength of the sources located at $x = x_1$, $y = 0$, h. C_2 and C_3 are the strengths of the sinks located at $x = x_2$, $y = 0$, h and $x = x_3$, $y = 0, h$, respectively.

It is desirable to relate the characteristics of the effective body produced by the sources and sinks to the characteristics of the actual jets. In particular, it is desired to relate C_1, C_2, and C_3 to the jet mass flow rate, m_j and the jet velocity v_j. Then it is possible to relate the results of a theoretical calculation to a particular experimental situation.

Taylor's analysis (46) is most accurate in the region close to the jet orifice. One of his main results was that

$$c_o = -2 v_j^2 b \tag{35}$$

where c_o is the radius of curvature at the upstream stagnation point of the effective body produced by a single two-dimensional jet, and b is the width of the jet slot. The corresponding mass-flow rate through a single jet is $m_{j,1}$, and:

$$m_{j,1}/\dot{m}_\infty = v_j b \tag{36}$$

With jets at $x = 0$, $y = 0$ and $x = 0$, $y = h$ the total jet flow rate is $\dot{m}_{j,2} = 2\dot{m}_{j,1}$, so that

$$c_o = -v_j \cdot \dot{m}_{j,2}/\dot{m}_\infty \tag{37}$$

A corresponding expression for c_o can be derived for the flow field and effective body described by equations (32) – (34) This is:

$$c_o = -(3/2) C_1 / (1 + \pi C_2') \tag{38}$$

where $C_2' = C_2 + C_3$.

In deriving (38), it was assumed that the sinks were located relatively far downstream, $x_2 - x_b > 1$ and $x_3 - x_b > 1$, so that $\tanh(x_2'/2) \cong \tanh(x_3'/2) \cong -1$. Equating (37) and (38) gives:

$$(3/2) C_1 / (1 + \pi C_2') = v_j \dot{m}_{j,2}/\dot{m}_\infty \tag{39}$$

If it is required that the main flow have the same uniform velocity at $x = \pm \infty$, then $C_1 = C_2'$. With this requirement, C_1 can be related to the jet parameters by equation (39).

The mixing between the jets and the main flow results in a growing shear layer between them as x increases. As a result, the preceding theoretical

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flow description becomes progressively less accurate in the downstream direction. In the present application to fiber alignment, interest is concentrated in the region near the jet injection, $x < 0.5$, however. Thus it is expected that the preceding analysis will at least give the correct qualitative behavior of the fiber.

2.3 Numerical Results

A number of calculations have been made for the following particular case: $C_1 = 0.2$, $C_2 = C_3 = 0.1$, $x_1 = 0$, $\chi_2 = 2$, $\chi_3 = 4$. The upstream part of the effective body shape produced by the sources and sinks for this case is shown in Figure 36. It is apparent that this combination causes the mainstream flow to converge rapidly and then diverge slowly. This is qualitatively the same behavior as produced by the counterflow jets. This type of convergence and divergence of the main stream, asymmetric with respect to the yz-plane, is essential for producing fiber alignment nearly parallel to the x-axis.
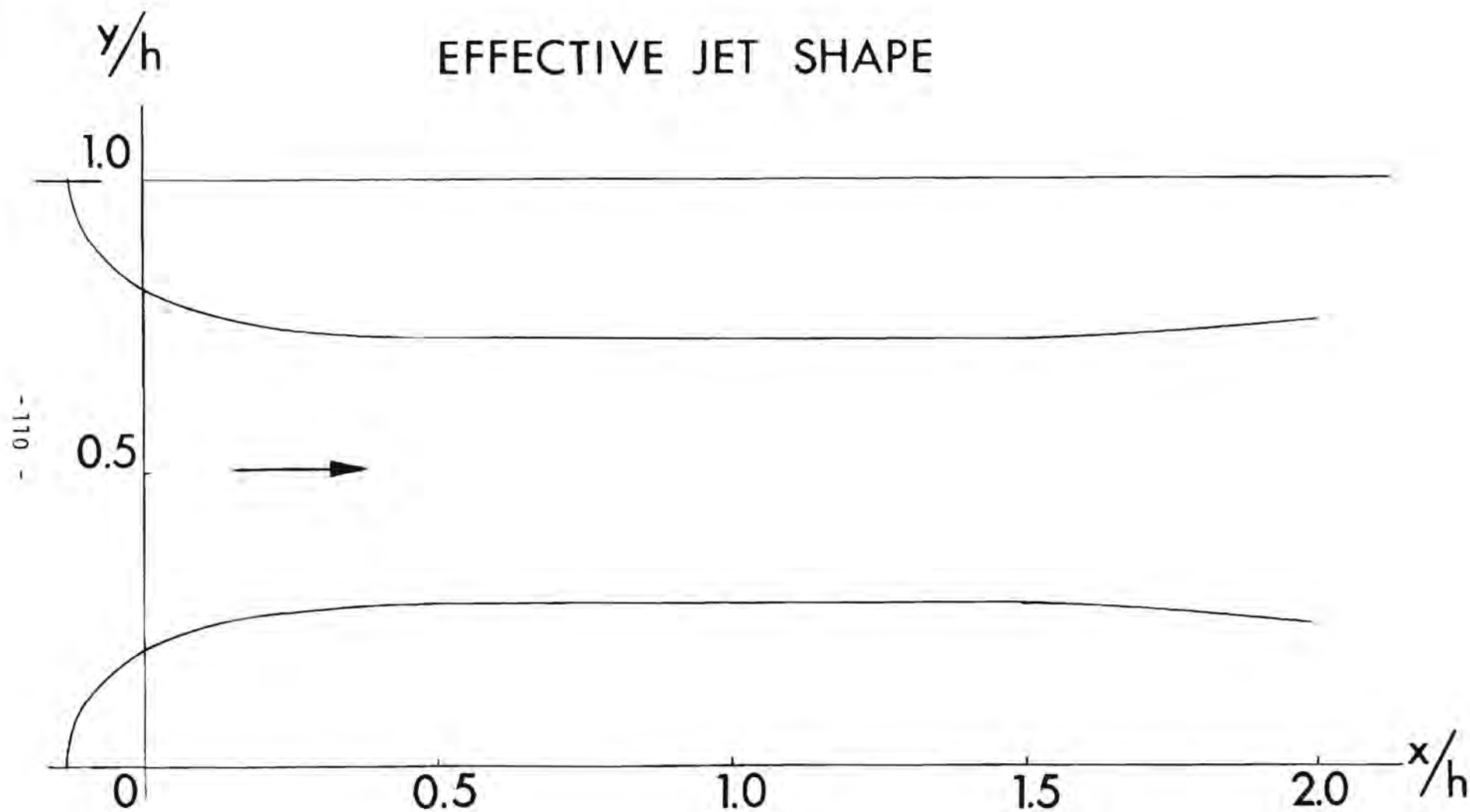
Figure 36.   Effective body shape produced by sources and sinks, approximating
the effects of the counterflow jets.

Figures 37 and 38 illustrate the predicted influence of the initial fiber inclination angle, θ_o, on the fiber inclination angle $\theta_{0.5}$ at x = 0.5. The calculated results of Figure 37 are based on the assumption that gravity acts in the z-direction, and so does not affect the fiber dynamics. The release point was x_{Go} = - 0.5 (i.e., one-half duct height upstream of the jets). Curves are shown for y_{Go} = 0.3, 0.4, and 0.5. Also, it was assumed that ω_o = 0, v_{Go} = 0, and u_{Go} = 0.1 $(u)_x$ = - 0.5. The nonzero value for u_{Go} was chosen to avoid numerical difficulties in computing the very large fiber acceleration from rest. By choosing a small, but nonzero, value for u_{Go} the essential physical behavior was retained. The fiber parameters are ρ_f = 1290 (cotton), ℓ_f = 0.0833, and d_f = 2.0 x 10^{-4}. For a duct height h = 1 ft, as used in the experiments, this corresponds to a fiber of 1 inch length and 60 μm (0.0024 inch) diameter.

The calculated results of Figure 37 show that for θ_o from 0° to nearly 90°, the fibers are rotated clockwise by the action of the jets, so that they are aligned nearly parallel to the x-axis (i.e., within 20° of the x-axis). There is an intermediate range of values of θ_o for which the fibers do not become parallel to the main stream. Within this interval the resultant direction of rotation changes from clockwise to counterclockwise. Finally, for values of θ_o greater than about 120°, the fibers are rotated counterclockwise so that they are essentially parallel to the x-axis.

The variation of θ with x is shown in Figure 38 for several values of θ_o and y_{Go} = 0.3. Again, it is apparent that alignment occurs within a short distance, and that most of the change in θ has been completed upstream of x = 0.5.
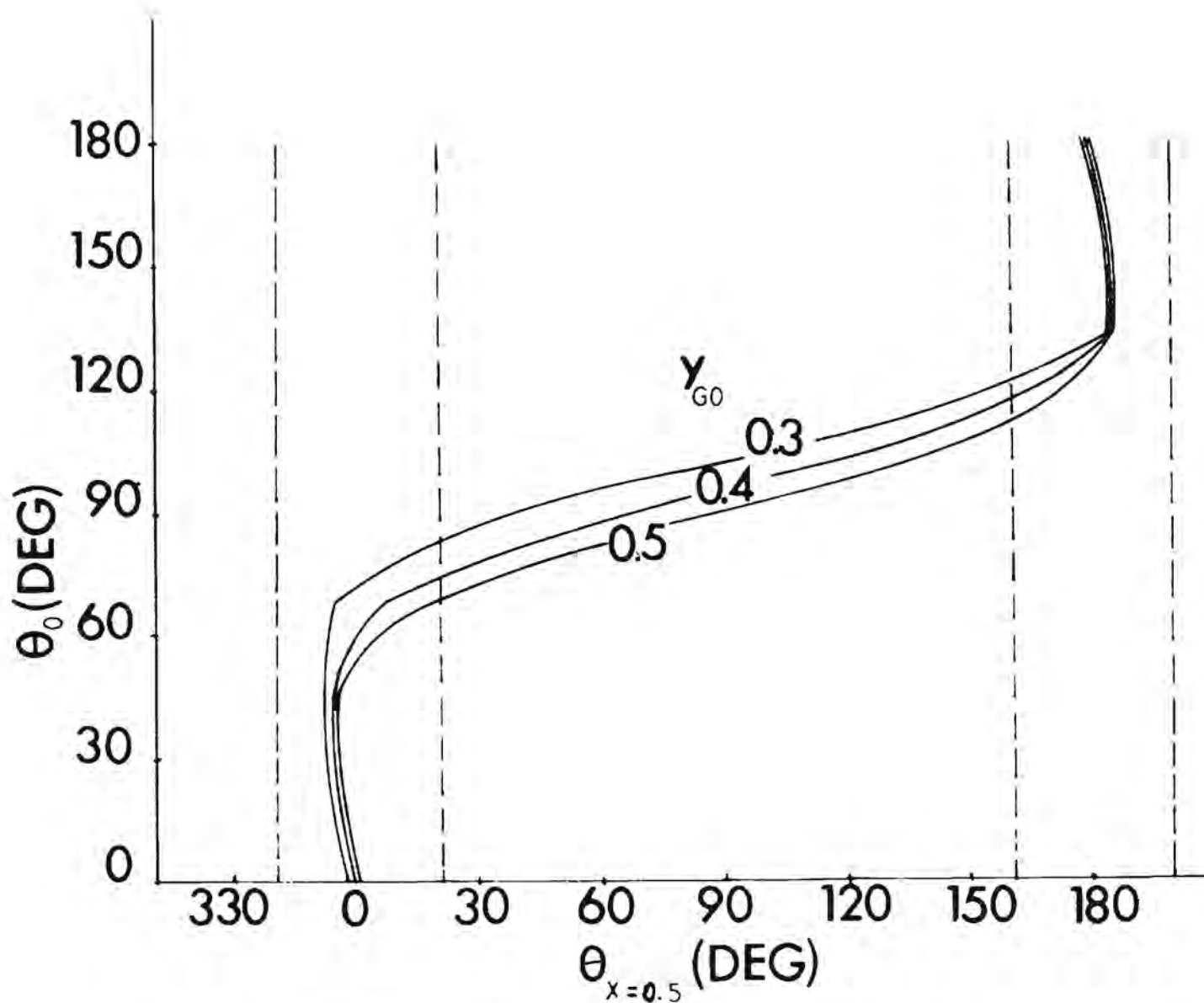
Figure 37. Effect of initial fiber inclination angle, θ_o, on inclination angle at X = 0.5. No gravitational effect.
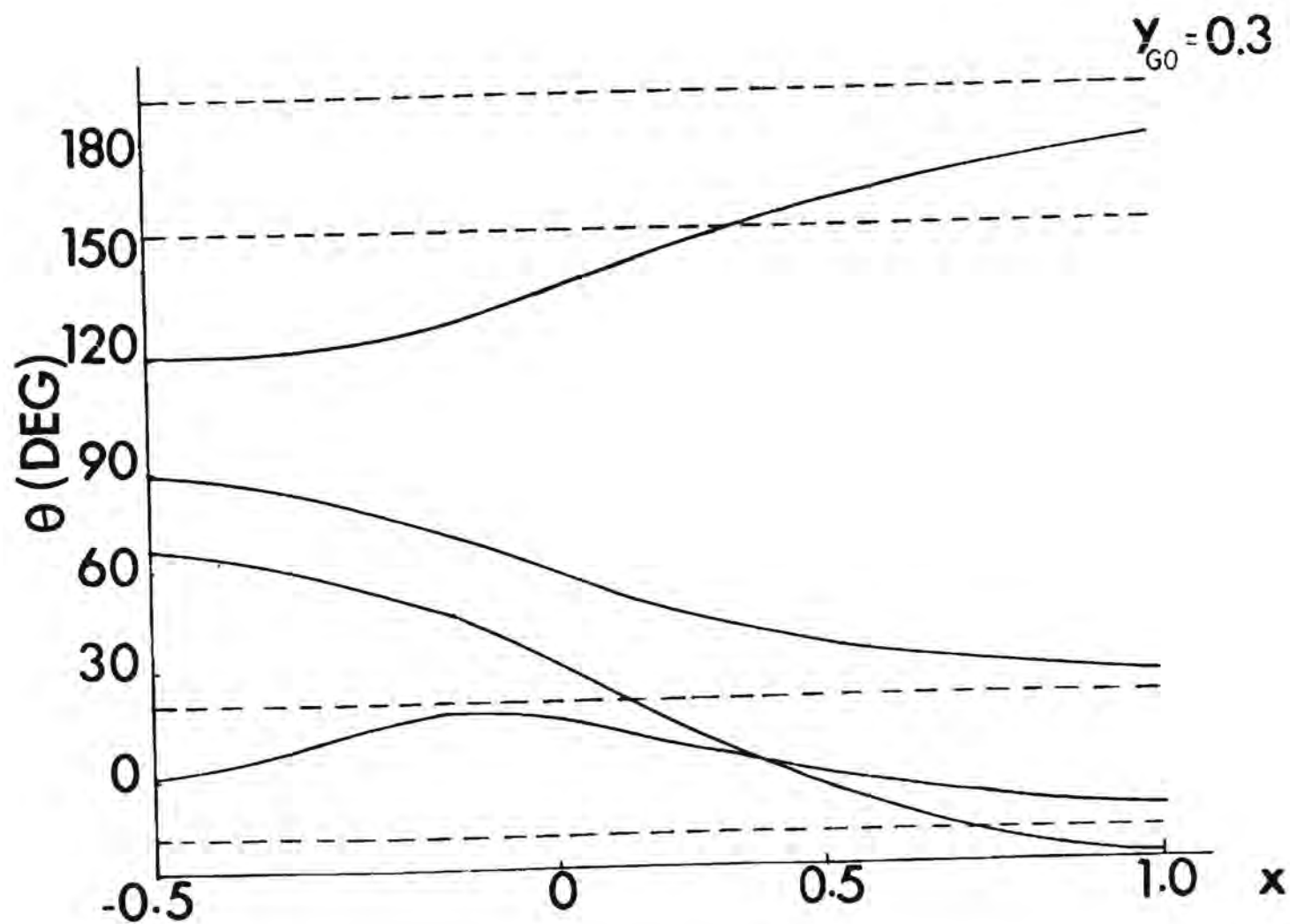
Figure 38. Variation of fiber inclination angle with axial distance.
y_{Go} = 0.3. No gravitational effect.

The preceding analysis indicates that θ_o must be less than about 90° for fibers to become nearly parallel to the x-direction. (Calculations have not been made for a wide range of fiber and jet parameters, but it seems clear that there will always be a small intermediate range of θ_o for which the direction of rotation is reversed. Within this small range, the final alignment will not be parallel to the x-axis). This suggests that a fiber-feed system should be used which restricts θ_o to less than 90°. The simple fiber injector described in section 3 satisfies this requirement completely. This is because the fibers are blown off the injector screen close to the point where the screen is tangent to the main stream velocity vector. As a result, the inclination of the fiber with the xz-plane is less than 90°.

2.2 Comparison of Calculations with Photographs of Fiber Motion

A brief comparison is given here of the calculated results described in section 2.1, and multiflash photographs of the fiber motion. In these experiments, the fibers were released at $x_{Go} = -0.5$ and $y_{Go} = 0.6$. Before release, the fibers were held fixed relative to the tunnel by the fiber holder. The fibers were released when a solenoid acted to open the jaws of the fiber holder. Simultaneously, the camera shutter opened. The force exerted by the main stream on the fiber then carried it downstream. The camera shutter stayed open for one second, and the stroboscopic light source flashed at 3600 flashes per minute. Cotton fibers with diameter 60 μm and length one inch were used. Corresponding free stream velocity was 20 ft/sec and the duct cross-section is 1 ft by 1 ft. The jet velocity was 150 ft/sec and the jet mass rate (both jets) was 2.4 lb_m/min.

Because of the arrangement of the experimental apparatus, gravity was acting in the negative y-direction. That is, gravity acted downward. For this

reason, computations were made in which gravity acted in the negative y-direction, and the results are shown in Figure 39. The computed behavior is very similar to that shown in Figure 37. Because of the influence of gravity, values of y_{Go} above the center line were chosen, so that the fiber would not fall into the shear layer during its trajectory.

It is only possible to make qualitative comparisons between the calculated motion of Figure 39 and that observed. This is because of the various approximations in the analysis, discussed previously. With this restriction, the calculated and observed results appear to be consistent. For $\theta_o = 90^o$, a small counterclockwise rotation is observed, consistent with the prediction for $y_{Go} = 0.6$. For $\theta_o = 60^o$ the fiber quickly rotates clockwise to a parallel alignment as anticipated from Figure 39. Further downstream, it is caught up in the rotational flow of the shear layer between the jet and the main stream. This illustrates the desirability of depositing the fibers a short distance downstream of the jets. Lastly, for $\theta_o = 120^o$ the fiber rotates counterclockwise until parallel to the free stream. Again, this is consistent with the predictions of Figure 39.
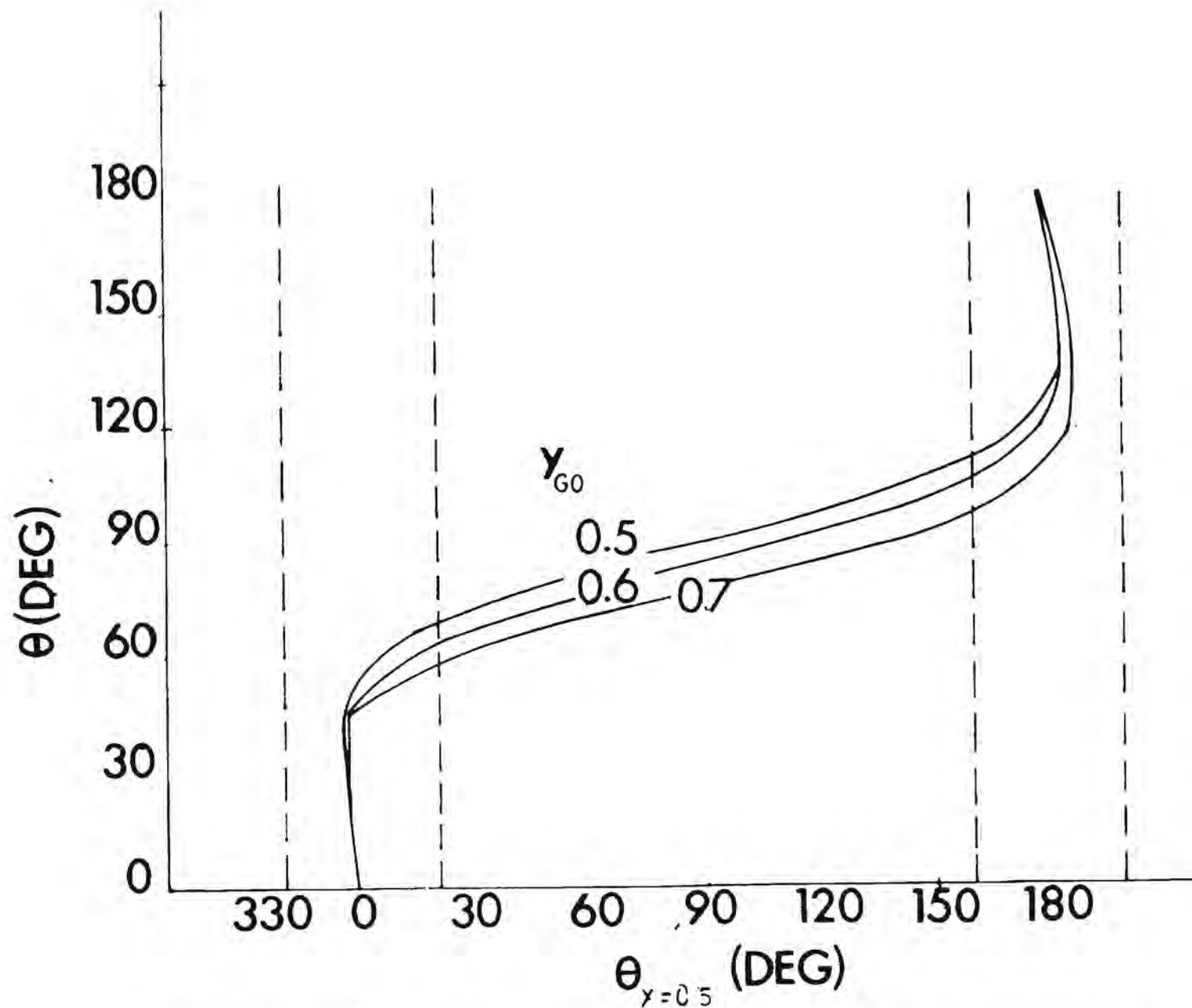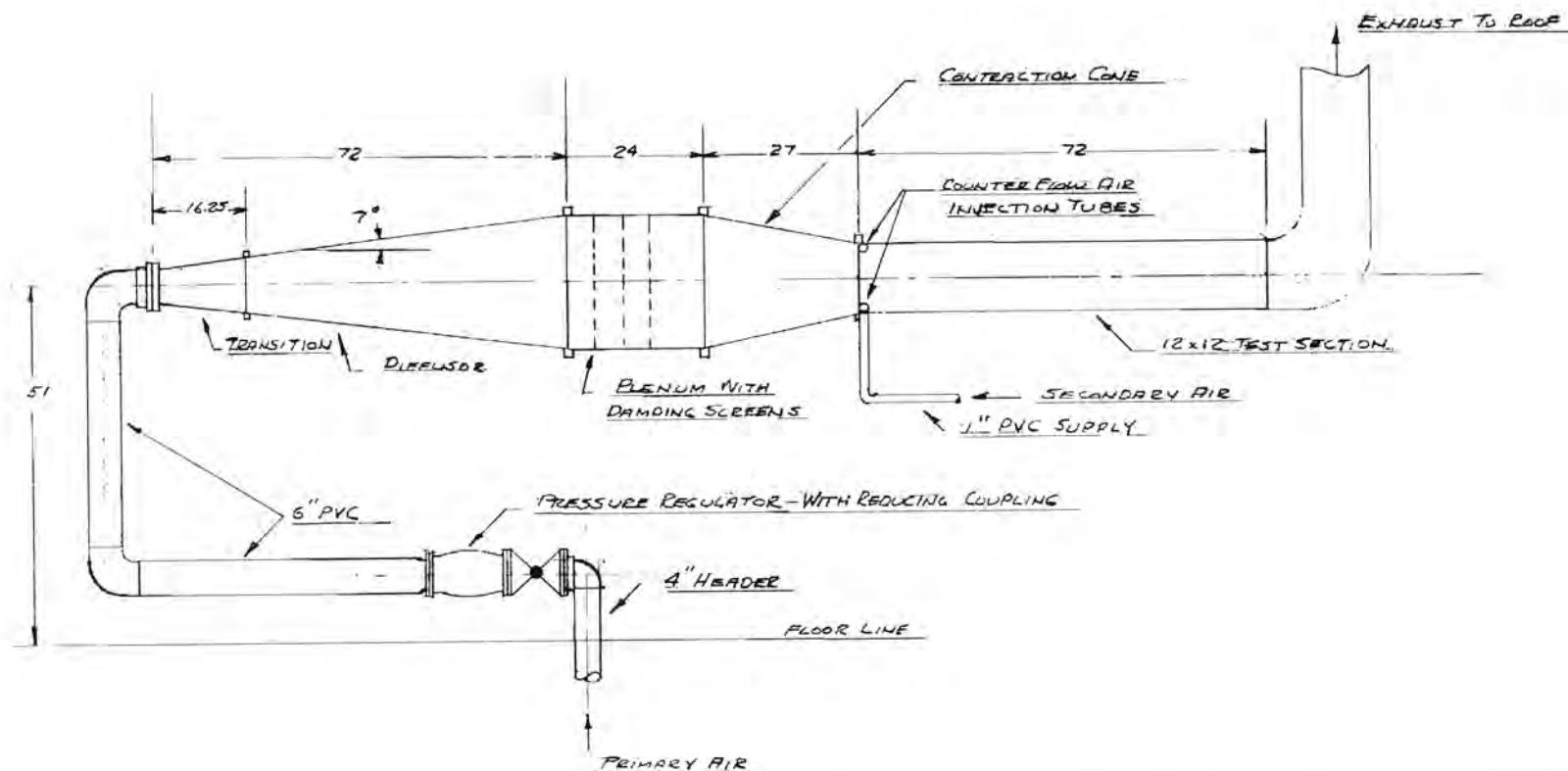
Figure 39.   Effect of initial fiber inclination angle, Θ_o, on inclination angle at X = 0.5.   Gravity acts in negative y - direction.

3.   APPARATUS

The air-flow system used to provide fiber alignment is shown in Figure 40. The principal system components and dimensions (in inches) are indicated. The main air stream moves through the wind tunnel. Fibers are released into it (as described later), and the main-stream velocity and the fiber orientation are altered by the action of the transverse jets. For the experiments reported here, the main stream velocity in the test section was 20 ft/sec, with the transverse jets off. This corresponds to an air mass flow rate of about 89 lb$_m$/-min. The test section has plexiglas walls to allow visual observation of the fiber motion. The transverse jet tubes have an outer diameter of 11/16 inch and extend across the test section at each side. Each tube has a row of 37 equally-spaced orifices of 3/32 inch diameter. These jet tubes can be easily rotated about their longitudinal axes so that the jet inclination angle can be continuously varied from $0°$ (directly downstream) to $180°$ (directly upstream). The transverse jet mass flow rate is also easily variable over a wide range, by means of a valve and a flowmeter in the secondary air supply line. By varying jet mass flow rate and/or inclination angle, the velocity gradients in the main stream are altered, and fiber orientation is changed.

This air flow system was designed at the beginning of the investigation. It was decided then to have a system that operated with pressures slightly greater than ambient, and to exhaust to ambient conditions. The only disadvantage to this is that it complicates fiber injection. That is, any aperture in the tunnel will have an outflow, which tends to impede fiber injection. The present arrangement, with the plexiglas shroud, overcomes this difficulty. It would be preferable in future applications to use an indraft wind tunnel, however, where the pressures are less than ambient. The method of fiber alignment and deposition is applicable independent of these conditions.

Figure 40. Wind Tunnel for Studies of Fiber Motion

A fiber injection system was designed and constructed to provide a continuous supply of individual fibers to the alignment system. This fiber injection system has the following basic functions: first, to receive a sliver of fibers and produce individual fibers; second, to inject the individual fibers into the tunnel main air stream. After the fibers enter the main air stream, they are aligned parallel to the main stream by the action of the transverse air jets.

The operation of the fiber injection system can be described with the aid of Figures 41 and 42, which are top and side views, respectively. Figure 43 is a photograph of the system. (The injection system was omitted from Figure 40 for clarity). A sliver is fed in with the aid of the feed roll. A toothed combing roll separates individual fibers from the sliver. The individual fibers are transported away by the motion imparted by the combing roll and by the air flow induced by a vacuum pump. The fibers land on the rotating injector screen, and are held in place by the radially-inward, vacuum-induced air flow. Note that the axis of the rotating screen is a tube, per-forated over its length within the injector. This axis is connected by flexi-ble tubing to a vacuum pump. The latter is a Stokes Microvac 148-H, with a displacement of 40 cfm.

The rotating injector screen (rotating clockwise as seen in Figure 41) carries the fibers inside the tunnel. This design was chosen as one method for getting the fibers into the main stream while providing low flow blockage and disturbance.

The fibers are initially on the upstream side of the injector so that they are held in place by the tunnel air flow. As the screen rotates, the fibers are blown off when they reach the point where the main stream velocity
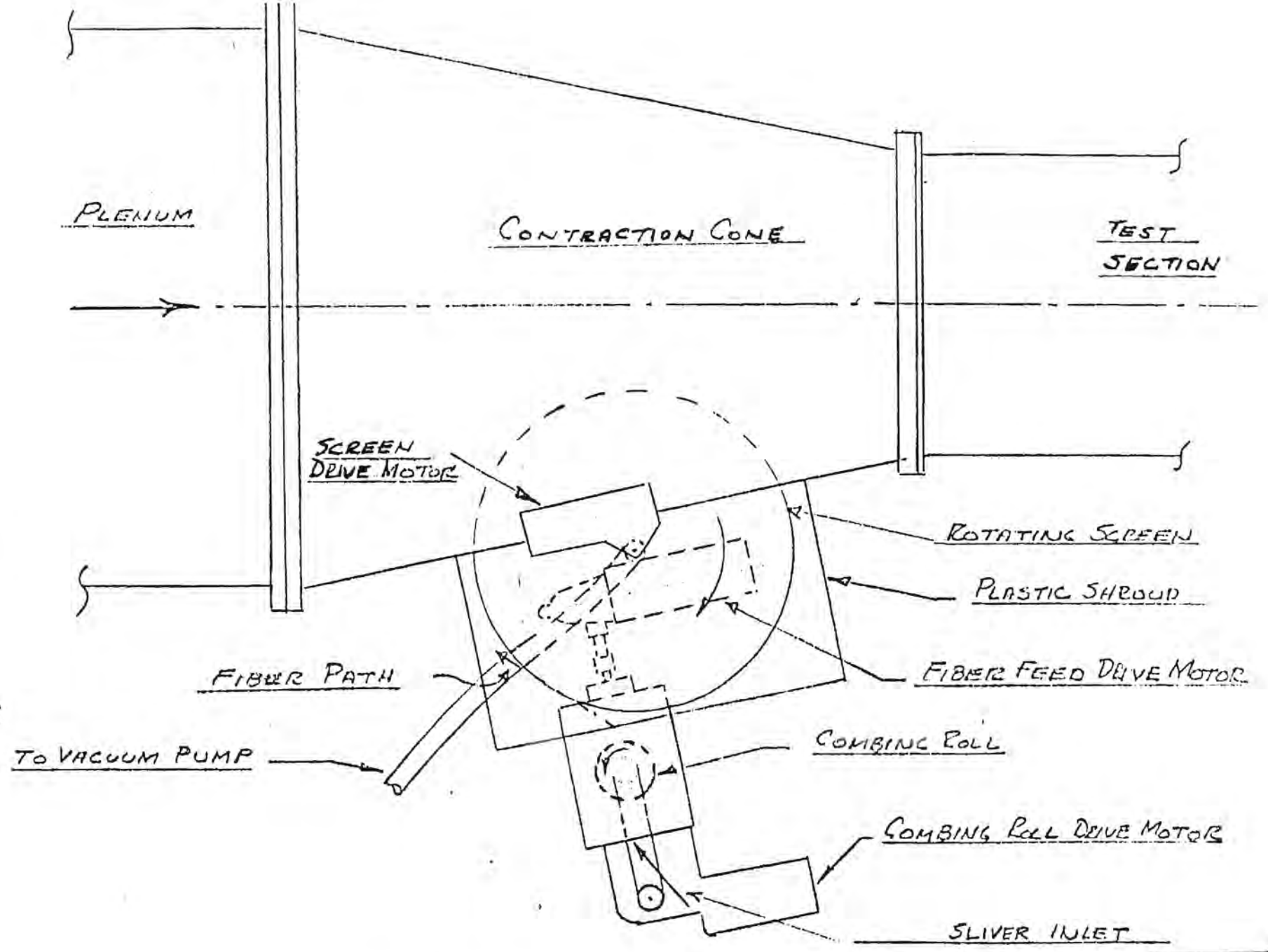
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PLENUM

CONTRACTION CONE

TEST SECTION

SCREEN DRIVE MOTOR

ROTATING SCREEN

PLASTIC SHROUD

FIBER FEED DRIVE MOTOR

FIBER PATH

COMBING ROLL

TO VACUUM PUMP

COMBING ROLL DRIVE MOTOR

SLIVER INLET

Figure 41. Top view of fiber injection system.

SCREEN DRIVE MOTOR

WIND TUNNEL CONTRACTION CONE

PLASTIC SHROUD

PLENUM

TEST SECTION

ROTATING SCREEN

COMBING ROLL DRIVE

TO VACUUM PUMP

FIBER FEED DRIVE

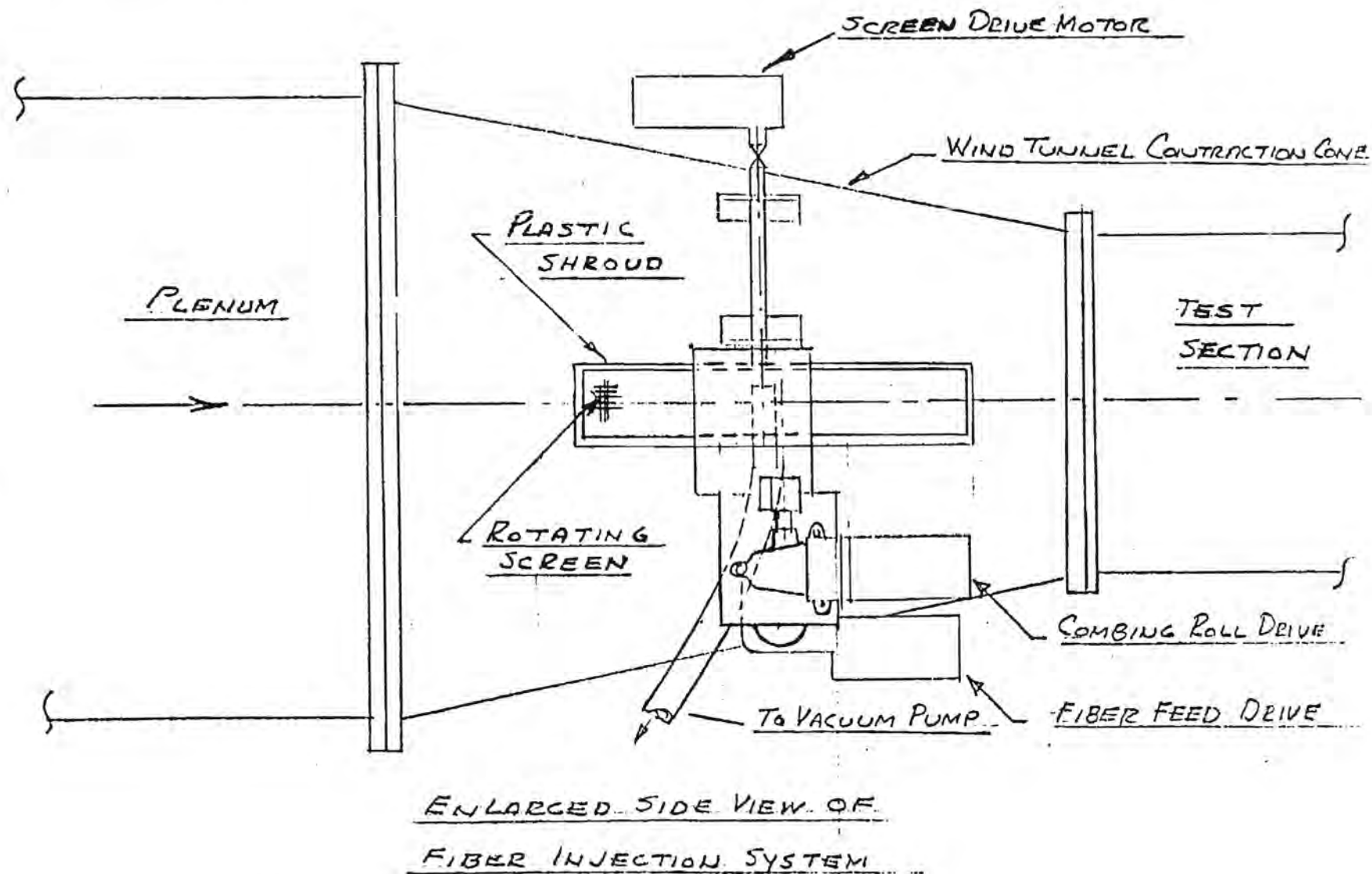ENLARGED SIDE VIEW OF FIBER INJECTION SYSTEM

- 121 -

Figure 42. Side view of fiber injection system.

is approximately tangent to the injector screen. The transverse jets are located a short distance downstream, where the contraction joins the test section. The fibers are aligned within a distance of one test section height down-stream of the jets and then deposited on a collecting screen.

The collecting screen and its associated apparatus is shown in Figure 43. The collecting screen is mounted so that it can be translated across the section. (The screen is shown outside of the test section in Figure 43.) This was done to provide a method for regulating fiber density across the screen surface. This collecting screen, which is 12 inches square, can also be easily remounted at 90° from its original orientation. This allows the fibers to be collected so that they are principally in two perpendicular directions on the collecting screen. A plexiglas shroud surrounds the collecting screen when it is outside of the tunnel. This is provided to prevent an outflow at this location.

In a commercial system fiber would be injected continuously into the air stream across the full width of the duct using a conventional lickerin roll. Orienting the ducts vertically would permit taking advantage of gravity and thereby the utilization of lower air flow velocities. Numerous air-flow systems could be used, each providing alternate 0° and 90° orientation. The collection screen could then be a continuous belt which collected fibers from first one air flow system and then the next.
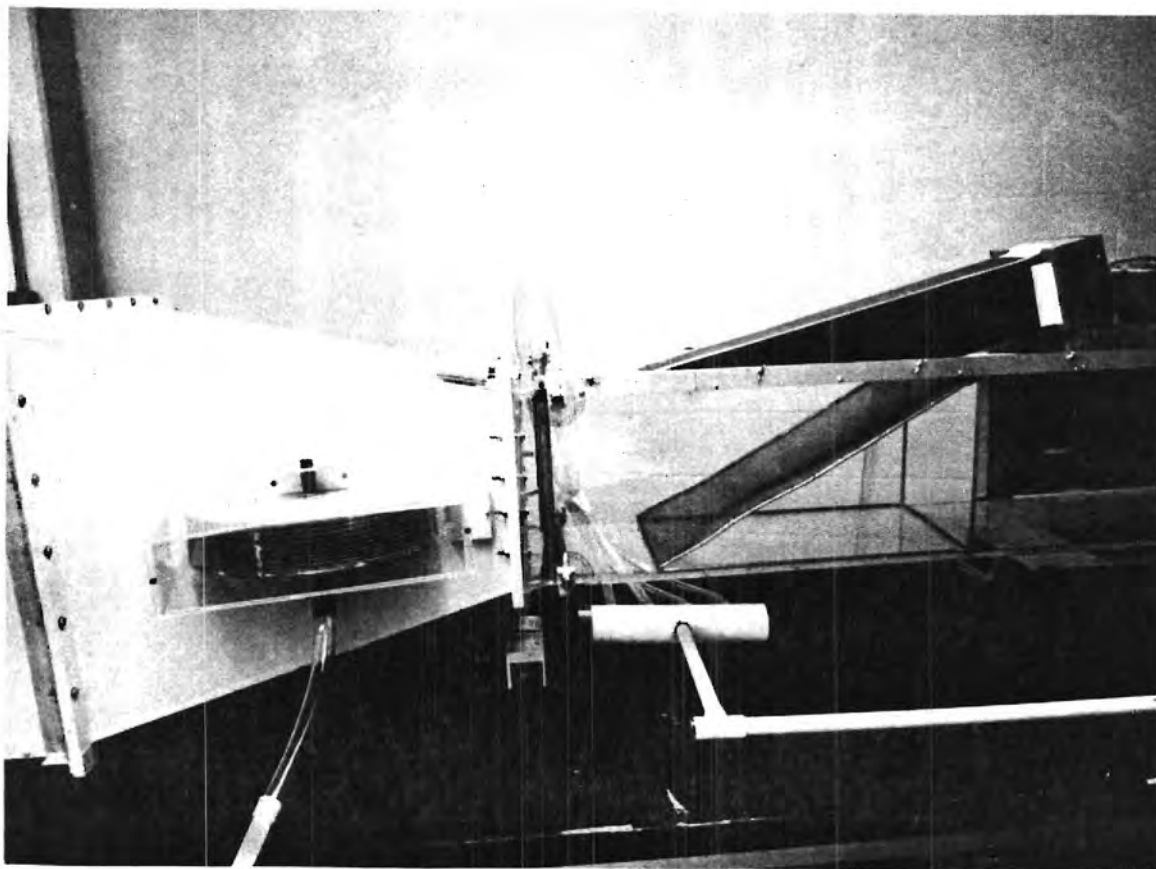
Figure 44. Fiber Injection System and Test Section.

4.   EXPERIMENTS

The experiments on web formation were performed using a main stream air speed of 20 ft/sec in the test section. For the test section area of 1 ft^2, this corresponds to a main stream flow rate of 89 lb$_m$/min. Total transverse jet flow rate was 3.2 lb$_m$/min, with a corresponding jet velocity of 180 ft/sec. The jets were directed at 90° relative to the main stream. These jet flow conditions were selected on the basis of some preliminary test results obtained using a manually-operated fiber injection system. No attempt has been made to explore the effects of a wide range of transverse jet or mainstream flow parameters.

A cotton sliver was fed into the fiber injection system. The feed roller was driven at about one-half rpm. The opening roller speed was set at 8000 rpm, while the rotating screen moved at 12 rpm. The latter speeds were measured with a stroboscope (General Radio Type 1538-A Strobotac). The diameter of the rotating screen is 15 inches. Thus, the perimeter of the rotating screen moved at about 0.8 ft/sec.

During early experiments, there was sometimes a tendency for parts of the web to lift off the collecting screen, resulting in disarray of the web and loss of fiber alignment. This tendency was greater after the direction of fiber deposition was changed by 90°. It also occurred when the web became relatively dense. This problem was completely solved by spraying the web at intervals with water from a simple, hand-held atomizer. The web, in turn, was rapidly dried by the air flow, which had previously been passed through a dryer.

No other significant problems were encountered in the web formation experiments.

The fiber deposition rate in these experiments was measured roughly at 0.003 lb$_m$/hr-ft^2. Although this number is very low, it should be pointed

out that the fiber injection system is far from optimum. In particular, it is believed that much higher fiber flow rates could be achieved before the fibers interfered with each other during the alignment process. On the other hand, even the maximum possible fiber flow rate with alignment may be significantly less than for systems where there is no alignment. This is because it is suspected that alignment cannot occur if there is substantial fiber interaction during the alignment process. However, as previously noted, numerous air flow systems could be mounted sequentially above a conveyor, thereby increasing the rate of fiber deposition.

5.   FIBER ORIENTATION ANALYSIS

Three fiber webs generated by fiber deposition onto a screen from a laminar air stream were selected for fiber orientation analysis. During deposition of fibers, the collection screen was rotated 90°. Approximately half the total web density was deposited in one continuous run of the laminar flow duct, followed by removal and reinsertion of the deposition screen at right angles for collection of the remaining fibers. Two of the webs were prepared for visual inspection and one was subjected to tensile testing for determination of strength, elongation, and modulus. Both visual inspection and tensile testing afford means for determining the relative numbers of fibers at various orientation angles.

The following discussion further explains the method of testing and the results obtained from the fiber webs.

Due to the low areal density of the webs, approximately 1.3 mg/cm^{2} (0.38 oz/yd^{2}), the webs were very fragile. To stabilize the webs during handling and for tensile testing, each was bonded with a 5% solution by weight of a commercial styrene-butadiene latex adhesive. The bonding technique involved saturation of the web with the latex solution, removal of excess latex, and curing at 93°C (200°F).

One web was bonded to a clear plastic sheet, while the other two were cured on a flat surface to which a mold release agent had been applied. The latex formed a clear, rubbery binder at fiber intersections, giving the web adequate integrity for evaluation.

Latex type KLM-3875-B, Southern Latex, Kensington, Ga.

Four specimens, each at angles of 0°, 30°, 60°, and 90° from the horizontal, referenced to the laminar flow duct, were cut from a bonded fiber web, using specimen dimensions of 2.5 x 10 cm. Each specimen was weighed on a precision balance and tested for breaking strength and elongation at break. Reported values of strength and modulus were normalized per unit areal density, termed specific strength or modulus, to account for web uniformity.

Web density was found to be 1.29 mg/cm^2 (0.38 oz/yd^2) $\pm$3% for averages of the four specimen densities in each angular group. Within group density variations ranged $\pm$9%.

The test results for each angular direction are given in Table XVII. Figure 44 illustrates the angular variation in specific modulus. The data for specific secant modulus indicates that fibers are oriented preferentially along the 0° and 90° directions. The 20% decrease in modulus at 30° and 60° indicates that relatively fewer fibers are directed along angles in the interval of 30° to 60°.

Due to the fragile nature of the unbonded webs, a web density determination before bonding could not be made. Thus, the binder content on the web is unknown, although an estimate based upon the bonding procedure which used a latex solution with 20% solids and removal of the excess is that solids pick-up should be less than 5%. Binder content at higher levels, due to increased binder stiffness, tends to mask the effect on tensile properties of fiber orientation.. Thus, the 20% decrease in modulus found in the 30° to 60° angular span should reflect a similar decrease in the number of fibers oriented at these angles.

TABLE XVII

Results of Tensile Tests

ANGULAR ORIENTATION	STRENGTH N (Lb)	SPECIFIC STRENGTH $N/mg/cm^2$	ELONGATION cm (%)	AREAL DENSITY, mg/cm^2 (oz/yd^2)		SPECIFIC SECANT MODULUS (SPECIFIC STRESS/STRAIN) $(N/mg/cm^2)/(cm/cm)$
0°	11.1 (2.5)	8.3	0.69 (9.1%)	1.31	(0.39)	91.2
30°	8.9 (2.0)	6.8	0.74 (9.8%)	1.28	(0.38)	73.0
60°	9.7 (2.2)	7.8	0.88 (11.5%)	1.25	(0.37)	69.4
90°	9.8 (2.2)	7.5	0.63 (9.1%)	1.30	(0.38)	94.7

Specific Modulus, $\frac{N/mg/cm^2}{cm/cm}$

(Horizontal)   Angular   (vertical)
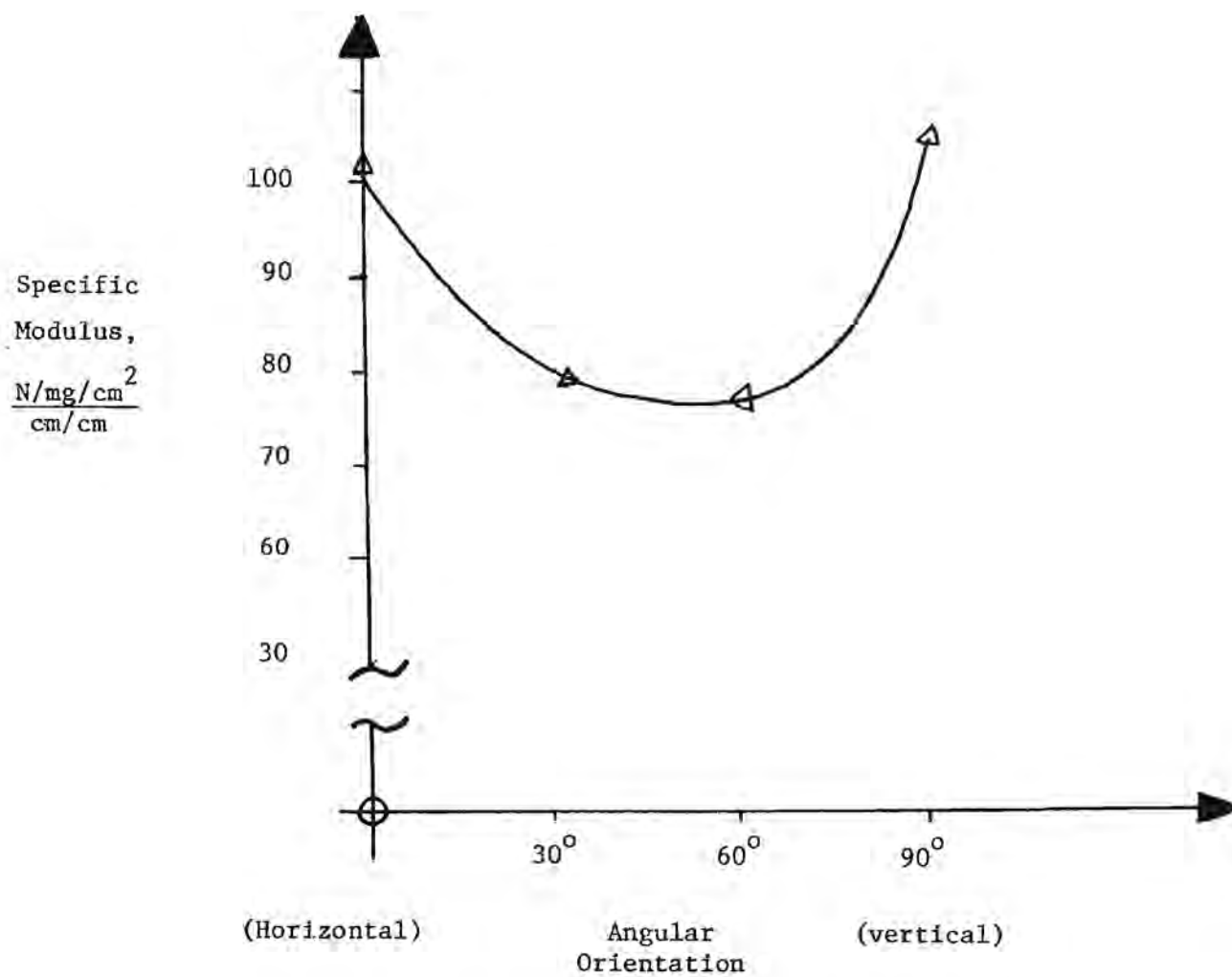   Orientation

Figure 44.   Specific Modulus vs. Orientation Angle

Attempts to measure numbers of fibers along angular intervals are difficult due to the fibers being crimped and curved rather than having well defined orientation directions, such as that of a straight rod. However, photographs indicate the fibers are mainly aligned in two perpendicular directions.

IV.   Technology Transfer

Presentations, publications and patent applications based on the program
are as follows:

Presentations by John Lundberg -

"Gaseous Polymer Solutions", 29th Southeast Regional Meeting,
American Chemical Society, Tampa, Florida, (November 2, 1977).

"American Physical Society, Division of High Polymer Physics,
Washington, D. C., (March 30, 1978).

"Gaseous Polymer Solutions: (paper read by James E. Mark, Univ.
of Cincinnati), (March 30, 1978).

Presentations by John Muzzy -

"Web and Yarn Formation by Biaxially Stretching Foam", University
of Tennessee - American Chemical Society Conference, Knoxville,
Tenn., (November, 1976).

"Foam Extrusion of Yarns and Webs"

1. Milliken Research Corp., Spartanburg, S. C. (December, 1975).

2. AIChe Section Meeting, Greenville, S.C., (April, 1977).

3. SPE ANTEC, Montreal, Canada, (April, 1977).

"Formation of Polypropylene Yarn from Foam"

1. Tennessee Eastman Co., Kingsport, Tenn. (March, 1978).

2. Textile Engineering Seminar, Georgia Tech (May, 1978).

3. Fiber Society, Knoxville, Tenn. (October, 1978).

"Process and Product Development of Foam Fibrillated Polypropylene
Yarn", SPE ANTEC, New Orleans, LA (May, 1979)

Presentations by Denney Freeston -

"Polymer to Garment"

1. American Apparel Manufacturers Association (AAMA) Research
Committee, Atlanta, GA (October, 1975).

2. AAMA Technical Forum II, Washington, D.C., (April, 1976).

3. Scott Paper Company, Philadelphia, PA, (May, 1976).

4. International Nonwovens and Disposables Association, IDEA 76 Conference, Chicago, Illinois, (October, 1976).

5. Phillips Fibers Corp., Greenville, S.C., (October, 1976).

Abstract: John Lundberg

"Gaseous Polymer Solutions", Bull Am. Phys. Soc., Ser. II, 23 (3), 404 (1978).

Publications —

"Foam Extrusion of Yarns and Webs", SPE Tech Papers, 23, 44, J. D. Muzzy, G. H. Hoyos and Y. H. Park, (1977).

"Formation of Polypropylene Yarn Foam", Textile Research Journal, submitted for publication (1978), J. D. Muzzy, G. H. Hoyos and A. R. Enuga.

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Patent

"Method for Fiber Alignment Using Fluid Dynamic Forces", L. H. Bangert, Patent Application No. 828893, filed August 29, 1977, assigned to Georgia Institute of Technology.

V.   Conclusion

Detailed conclusions are given at the end of each of the major sections of III, Technical Activities. These conclusions are summarized in I, Executive Summary.

It has been shown that common, fiber forming polymers are soluble in inexpensive, non hazardous gases at moderate temperatures and pressures. This type low viscosity dispersion of polymer will permit rapid heat and mass transfer and could be valuable as a low energy transport and fabricating medium. Gaseous solutions also may be suitable media for the flash extrusion of fibers and foamed sheets.

Research on polymer solutions is continuing under the sponsorship of the National Science Foundation.

At their current state of development both web and yarn formation by foam fibrillation have limited utility for garment applications. Even with further development it is unlikely that foam fibrillated webs would have substantial utility in garment applications. However, with further development foam fibrillated yarns could be readily utilized in selected types of apparel. The primary developments necessary would entail substituting synthetic polymers more suitable for garment applications for polypropylene and then tailoring. processing conditions to obtain yarn properties suitable for specific applications. With some difficulty it is possible to transfer foam fibrilla- tion process technology to other polymers, including nylon and polyester. The process route does provide sufficient flexibility to achieve a broad range of properties and structures.

There are numerous industrial nonwoven fabric applications for which the low basis weight, low cost and high surface area characteristics of foam

fibrillated webs are suitable. In addition it would be attractive to modify the foam fibrillation process in order to fabricate webs from liquid crystal thermoplastic polymers. Provided adequate web uniformity is obtained these webs should exhibit high strength as-extruded. Such webs could be manufactured at very low cost.

The preliminary studies of fabric formation from foam fibrillated yarn indicate that the yarn would be suitable for upholstery applications. Tailoring of processing conditions to meet specific end-product requirements would allow a better definition of product potential. Also, substituting different types of polymers would expand the range of utility of the foam fibrillation process. Considerable economic advantages could be obtained by eliminating or modifying the intermediate twisting step currently envisioned in the foam fibrillated yarn process. By drawing larger width webs or by using a false twist drawing process, drawing at least 4 times to achieve a tenacity above 3g/denier could be accomplished without introducing permanent twist. Without the need for permanent twist, processing costs would be substantially reduced.

Several industrial organizations have expressed interest in foam fibrillated yarn. However, to the best of our knowledge, to date none have decided to commercialize the process.

The counter flow jet technique for orienting natural and/or staple fiber in low velocity air streams offers several potential advantages when compared to current nonwoven processing techniques. It can be used to produce a broad range of web weights with fiber orientations from random to unidirectional. Fabric webs can be produced in which the fibers are oriented in two perpendicular directions uniformly throughout the web thickness. A patent is pending.

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PART I—PROJECT IDENTIFICATION INFORMATION

1. Institution and Address	2. NSF Program	3. NSF Award Number
School of Textile Engineering Georgia Institute of Technology Atlanta, Georgia 30332		APR74-02326 A03
	4. Award Period From 1/1/75 To 12/31/78	5. Cumulative Award Amount $297,300

6. Project Title

Advanced Technology Applications in Garment Processing

PART II—SUMMARY OF COMPLETED PROJECT *(FOR PUBLIC USE)*

The principal areas of research were: 1. Measurements of solubilities of polymers in dense gases; 2. Continuous sheet and yarn formation from fibrillated polymer mixtures; 3. Orientation of staple fiber and disposition of fibers in parallel arrays using air flows and aerodynamic forces.

The major accomplishments under the program are as follows:

Polybutene-1 and polypropylene are readily soluble in n-butane at relatively low pressures, about 120 atmospheres and upwards, at temperatures above the melting points of the polymers.

Polybutene-1 and polypropylene are soluble in carbon dioxide at higher pressures, from about 450 to 900 atmospheres, at temperatures above the melting points of the polymers.

Nylon-6 dissolves in carbon dioxide at 400 to 500 atmospheres and 233° to $241^{\circ}C$. Carbon dioxide appears to be a good solvent for this polar polymer.

Fine fibers of polypropylene and nylon-6 were produced during solution studies by bleeding solutions to the atmosphere. However, attempts to scale-up extrusions of gaseous polymer solutions to produce fibers were not successful.

Polypropylene fibrous webs were formed by melt foam fibrillation. They were characterized by: 1) basis weights below 1 oz./sq.yd.; 2) average tenacities below 2 lbs/in/oz./sq.yd.; 3) tenacity imbalances in excess of 5 (MD/TD); 4) high air permeabilities; and 5) a tendency towards filmy junctions.

Polypropylene foam fibrillated webs were converted into yarn with properties which make it suitable for some textile applications.

Natural and staple fiber was oriented in low velocity air streams using counterflow jets, and deposited on a screen maintaining the orientation.

Papers on the research have been published in various technical journals. A patent application has been submitted on one development.

PART III—TECHNICAL INFORMATION *(FOR PROGRAM MANAGEMENT USES)*

1. ITEM *(Check appropriate blocks)*	NONE	ATTACHED	PREVIOUSLY FURNISHED	TO BE FURNISHED SEPARATELY TO PROGRAM	
				Check (✓)	Approx. Date
a. Abstracts of Theses					
b. Publication Citations					
c. Data on Scientific Collaborators					
d. Information on Inventions					
e. Technical Description of Project and Results	▨				
f. Other *(specify)*	▨				

2. Principal Investigator/Project Director Name *(Typed)*	3. Principal Investigator/Project Director Signature		4. Date

INSTRUCTIONS FOR FINAL PROJECT REPORT
(NSF FORM 98A)

This report is due within 90 days after the expiration of the award. It should be submitted in two copies to:

National Science Foundation
Division of Grants and Contracts
Post-Award Projects Branch
1800 G Street, N.W.
Washington, D.C. 20550

INSTRUCTIONS FOR PART I

These identifying data items should be the same as on the award documents.

INSTRUCTIONS FOR PART II

The summary (about 200 words) must be self-contained and intelligible to a scientifically literate reader. Without restating the project title, it should begin with a topic sentence stating the project's major thesis. The summary should include, if pertinent to the project being described, the following items:

- The primary objectives and scope of the project.
- The techniques or approaches used only to the degree necessary for comprehension.
- The findings and implications stated as concisely and informatively as possible.

This summary will be published in an annual NSF report. Authors should also be aware that the summary may be used to answer inquiries by nonscientists as to the nature and significance of the research. Scientific jargon and abbreviations should be avoided.

INSTRUCTIONS FOR PART III

Items in Part III may, but need not, be submitted with this Final Project Report. Place a check mark in the appropriate block next to each item to indicate the status of your submission.

a. Self-explanatory.
b. For publications (published and planned) include title, journal or other reference, date, and authors. Provide two copies of any reprints as they become available.
c. Scientific Collaborators: provide a list of co-investigators, research assistants and others associated with the project. Include title or status, e.g. associate professor, graduate student, etc.
d. Briefly describe any inventions which resulted from the project and the status of pending patent applications, if any.
e. Provide a technical summary of the activities and results. The information supplied in proposals for further support, updated as necessary, may be used to fulfill this requirement.
f. Include any additional material, either specifically required in the award instrument (e.g. special technical reports or products such as films, books, studies) or which you consider would be useful to the Foundation.