

PROPERTIES AND CHARACTERISTICS OF
POLYPROPYLENE FIBERS SPUN BY THE
PHASE-SEPARATION TECHNIQUE

by

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I. INTRODUCTION

There are four major spinning processes which are used in the industrial production of synthetic fibers. These techniques are: melt spinning, dry spinning, wet spinning, and gel extrusion spinning. The nature of the polymer which is being spun and the type of product desired dictates, in general, which process should be used.

Another technique, which might be called phase separation spinning, has been developed. [8] A polymer is dissolved in a spinning solvent at polymer concentrations of 10-25 per cent and at temperatures well below the polymer's melt spinning temperature. The solvent is chosen such that the solution which is spun undergoes phase separation while cooling; that is, separation into a polymer phase and a solvent phase occurs. The solvent is subsequently removed from the fiber by extraction or washing.

Several polymer-solvent combinations have been successfully spun by this phase-separation method, but very little information is available on the production of any one type of fiber.

The first object of this investigation was to design, build, and test a suitable and reliable apparatus for spinning fibers by the phase-separation method. Secondly, the properties of polypropylene fibers spun in solution with naphthalene were to be determined.

II. LITERATURE REVIEW

Published information which pertains to this study is presented here.

Fiber Spinning Methods

Four types of spinning techniques are generally recognized. These methods are discussed by Zwick.

In melt spinning, [9] molten polymers are extruded through small orifices into a cool atmosphere where solidification occurs and fibers are formed. This is the most economical spinning method and it is used for production of polyamide, polyester, and polyolefin fibers at collection speeds of 800-1000 meters per minute.

Wet spinning [10] techniques are used in the production of polyacrylonitrile, poly (vinyl alcohol), and some cellulosic type fibers. These polymers do not melt without decomposition, but they can be made fluid by dissolving them in suitable solvents, sometimes after chemical modification. The solution is ejected into a coagulation bath where precipitation occurs, caused by either solvent dilution or by reaction, and a fiber is formed. Ordinarily, polymer concentrations of 5-25 per cent in solution and spinning speeds of 50-100 meters per minute are used. For economic reasons, solvents must be recovered and reused in this technique.

In dry spinning, [11] polymer concentrations on the order of 20-30 per cent are used; the polymer solutions are extruded into hot spinning

shafts where the solvent evaporates. Low-boiling solvents that can be condensed easily and recycled are usually employed. Cellulose acetate, poly (vinyl chloride), and polyacrylonitrile fibers are made by this method at spinning speeds of 300-500 meters per minute.

Gel extrusion spinning [12] proceeds similarly to melt spinning at spinning speeds at or above 500 meters per minute. In the polymer concentrations used, for example, 35-55 per cent, the polymer-solvent mixtures no longer are true solutions and the "solvent" might be more aptly regarded as a plasticizer. Polyacrylonitrile and poly (vinyl chloride) fibers have been produced in this manner and the solvents are removed by washing and/or drying. A major problem with this technique is inhomogeneities in the spinning gel.

Phase Separation Spinning

Yet another spinning technique was explored by Zwick in a paper published in 1967.

Theory. This technique [13] was meant for application to polymers which cannot be melt-spun conveniently without degradation or which cause difficulties in gel spinning.

The idea was to use a polymer-solvent combination which could be easily dissolved homogeneously in the wet-spinning concentration range at relatively low temperatures. A spinning solvent was to be chosen such that the polymer solution would undergo phase separation in the temperature range between the spinning temperature and room temperature. The solution would separate into a polymer phase, which

would be the final fiber, and a solvent phase, which would later be removed by washing.

Advantages [14] expected were: 1. solution preparation and spinning at relatively low temperatures, 2. high spinning speeds, 3. short spinning shafts, and 4. control of or influence on the fine structure of the fiber by the washing method.

An obvious disadvantage of the method is the need to recover and reuse solvents and washing fluids and the solvent losses involved with such an operation.

Several phase separation situations are listed by Zwick [15] involving solid and liquid solvents along with mixtures of solvents. In each case, the product fiber was expected to have a unique fine structure, as evidenced by the degree of interdispersion of phases, the molecular orientation, the degree and type of crystallinity, the homogeneity of cross-sections, and the surface texture.

Experimental. Zwick conducted experiments by two methods. [16] First, he used a 50 gram piston-ejection melt-spinning apparatus to melt and spin a pre-cast polymer-solvent stick. An advantage to this was the use of small quantities of solution. A great disadvantage was the need for cooling the solution to solidification in order to transfer it to the piston-ejection apparatus.

The second method of fiber production was spinning of the polymer solutions directly from the vessel in which they were prepared without any intermediate cooling. Zwick's spinning unit consisted essentially of a reservoir for preparation of the solution, a pump block, a

spinning head and a winding unit. Most of the work was conducted using this piece of equipment.

Before each experiment, [17] the following parameters were fixed:

1. Composition of spinning solution
2. Number and size of holes in spinning plate

The following parameters were adjustable during each experiment:

1. Temperatures (reservoir, block, head)
2. Output
3. Winding speed
4. Finishing agents

The primary objectives [18] of Zwick's work were to find polymer-solvent systems that would spin under phase separation conditions and to determine concentrations, temperatures, and spinneret hole sizes for optimum spinnability. The criterion for optimum spinnability was the highest possible winding speed. He also wished to examine various conditions of solvent removal and to determine how particular spinning and processing conditions influenced fiber properties and fine structure. For this reason, tensile strength and elongation measurements were conducted and selected microscopic examinations were made.

Some of the polymers from which fibers were successfully made were polypropylene, polyethylene, poly (vinyl alcohol), polyacrylonitrile, and poly (vinyl chloride).

Commercial Polypropylene Fibers

Polypropylene fibers [4] are characterized by high strength and

light weight. By proper control of processing variables, the best properties for different applications can be obtained.

Manufacture. The manufacture [7] of ordinary polypropylene fibers is done by melt-spinning. Because of its very high melt viscosities, polypropylene must be spun at temperatures about 100°C higher than the melting point of 165°C.

The spun filaments are stretched mechanically under various conditions of temperature and stretching to create axial orientation which provides the required tensile strength. Monofilaments of 35-1000 denier are produced for use in bristles or ribbons. For fine fibers of 1 1/2 - 15 denier, very high winding speeds of 305-915 meters per minute must be used. Multifilament yarns or staple fiber may be produced after heat-stretching and crimping. Ordinary stretch ratios vary from 1.3-1.8 and crimping is usually mechanical. Self-crimp may be induced by selection of proper drawing conditions.

A common problem [1] in spinning polypropylene is a periodic variation in filament diameter that reduces strength and causes processing difficulties. The fibers appear to be formed from a series of droplets. One method suggested to remedy this is the use of a higher spinning temperature; however, polypropylene's tendency to degrade limits the spinning temperature.

Properties. Table I shows the range of properties typical of commercially produced polypropylene fibers as compared to other commercial fibers.

In general, polypropylene fibers [2] exhibit good processing

Table I
Some Comparative Mechanical Properties of Commercial Fibers

Fiber	Specific Gravity	Tenacity gm/den	Elongation, %		Stiffness gm/den	Toughness gm/den
			Dry	Wet		
Cotton	1.50	3-6	3-7	-	60-70	0.15
Wool	1.32	1-2	25-35	25-50	4.5	0.35
Nylon 6,6/6	1.14	4.5-9	20-40	20-40	15-45	0.5-0.7
Polyester	1.38	4.5-5	14-25	19-25	18-36	0.35-0.55
Polypropylene	0.91	4-7	15-35	15-35	25-40	0.5-0.6

Mark, H. F. et al: "Man-Made Fibers, Science and Technology," Vol. 3, p. 379. Interscience Publishers, New York, N. Y., 1968.

behavior, good wrinkle resistance, excellent abrasion resistance, and complete resistance to microorganisms and insects. At present, they are typically spun dyed and show very good color fastness. Piece and stock dyeing using acid, chrome, and metallized dyestuffs have also been fairly successful.

Applications. The main applications [5] for monofilaments of polypropylene are for rope, cordage, fish nets, seat covers, outdoor furniture webbing, and industrial filters. The most important use for polypropylene fiber is in indoor and outdoor carpeting; however, it is also used in knitwear, mostly blended with wool or cotton.

Polypropylene Fibers Spun by Phase Separation

Zwick found that a number of readily available solvents [19] could be used in the phase separation spinning of polypropylene fibers. According to his studies, the order of solvation ability of the solvents with their corresponding polymer concentrations is given as follows:

Naphthalene	<	p-dichlorobenzene	≤	stearic acid	<	paraffin wax
11.5%		15-17%		15-17%		16.5-21%

He also observed that the better the solvent, the greater the amount of polymer that could be used in the spinning solution.

Zwick found that a fiber could be drawn for several hundred meters from a beaker of 15 per cent polymer solution at 150-160°C when a 50:50 mixture of naphthalene and paraffin wax was used as the spinning solvent. Because of this self-spinning property, [20] fiber was produced

under the force of its own weight at wind-up speeds of 2000 meters per minute.

Using the direct spinning [21] unit, Zwick found that he could easily employ spinneret hole sizes of up to two millimeters and spinning speeds of up to 1000 meters per minute. He also found that it was possible to heat-stretch the fiber with the solvent still in it.

Table II shows the results of some of Zwick's tests with polypropylene fibers. It should be emphasized that his was merely an exploratory study and that no attempts were made to optimize spinning conditions and fiber properties. Zwick primarily endeavored to find polymer-solvent combinations from which fibers could be produced using phase-separation spinning.

Polypropylene fibers [22] spun from three different solvents, paraffin wax, p-dichlorobenzene, and naphthalene, were examined under a microscope before and after solvent extraction with petroleum ether. The paraffin wax spun fibers appeared round and smooth; the p-dichlorobenzene spun fibers were kinked, indented, and crenulated; and the naphthalene spun fibers were round with a striated surface. It was theorized by Zwick that, for a given cooling rate, the lower the freezing point of the solvent, the more time the fiber has to lose its solvent while still liquid and thereby close internal voids. This might tend to produce a crimp such as was displayed in the fiber spun from a p-dichlorobenzene solution after extraction and heat-stretching.

Finally, Zwick [23] observed that fibers could be prepared by this technique from polymers of very high molecular weight (10^6)

Table II

Conditions for Polypropylene Fiber Production by Phase-Separation Spinning and Results of Some Property Tests on Fibers Produced (Washing Solvent - Petroleum Ether)

Polymer concentration % Solvents	17 Naphthalene Paraffin wax	15 Naphthalene Paraffin wax	16 p-dichlorobenzene Paraffin wax
Solvent mixture	50:50	50:50	88:10:2
No. spinneret holes and diameter, mm.	6x0.8	15x0.2	6x0.6
Reservoir temp., °C	175	175	170
Winding speed, m/min	400	300	200
Velocity in spinneret holes, m/min	3.0	16.0	5.4
Draw-down ratio	133	19	37
Single fiber den/den- sity (calcd.)	5.75	2.25	10.8
Heat-stretching temp., °C	80/125	80/125	125
Heat-stretching factor	9.0	8.0	8.0
Denier after heat- stretch (calcd.)	0.58	0.26	1.22
Tensile strength, gm/den	7.0	6.4	7.4
Break elongation, %	17	16	20
No. of filaments	56	255	24
Actual denier	0.72	0.33	2.14

Zwick, M. M.: Spinning of Fibers from Polymer Solutions Undergoing Phase Separation, Applied Polymer Symposium No. 6, 132-3 (1967).

and he also produced fibers with high filler content and large filler particle sizes.

III. EXPERIMENTAL

This section contains a plan of experimentation, a description of procedures used, and the results obtained for this investigation.

Plan of Experimentation

This study was undertaken using the following plan of experimentation.

Equipment Design, Construction, and Testing. A spinning apparatus was designed and built in the Chemical Engineering Shop of Virginia Polytechnic Institute and State University. The polymer solution was contained in a vertical, heated cylinder. Solution was to be forced out of the cylinder through a spinneret by a piston driven by a variable speed drive. The resulting fiber was to be taken up on a rotating steel drum, also equipped with a variable speed drive.

Several different polymer solution compositions and spinning temperatures were used in testing the spinning apparatus.

Fiber Spinning and Sample Collection. Four tests were conducted to obtain fiber samples for property testing. Combinations of two different polymer solution compositions and two different spinning temperatures were used.

At the beginning of each test, feasible ranges for spinning rate and take-up speed were determined. Four values of take-up speed and four values of spinning rate were selected, giving a total of 16 variable combinations, or points, for each combination of solution composition and temperature. At each point, two samples of fiber were collected

from the take-up drum, one for extraction studies and one for property testing.

Extraction Studies. Tests were conducted to determine whether or not chemical extraction of the naphthalene with diethyl ether was necessary and, if it was, the rate at which this extraction would proceed.

Fiber Testing. Scanning electron microscope pictures were taken of four selected fiber samples. Fourteen of the total of 64 fiber samples made were selected for further testing. Denier, tenacity, per cent elongation at break and secant modulus were determined for each of the 14 samples. Four fiber samples were photographed for visual comparisons.

Procedures

The following procedures were used during the course of this investigation. A complete description of the equipment used is in the Appendix.

Solution Preparation. Before each fiber spinning test, the polymer solution to be spun was prepared in a closed, agitated, resin kettle equipped with an electrical heating mantle. First, the appropriate amounts of polypropylene and naphthalene were weighed out. The naphthalene was poured into the kettle through a funnel and heated.

When the naphthalene had melted at about 80°C, the agitator was started, and the polymer powder was added to form a slurry. The temperature of the slurry was slowly increased until the polymer dissolved;

dissolution was assumed complete when the liquid in the kettle appeared clear and homogeneous. Slow agitation was maintained throughout the dissolution step. The solution was then heated to an upper limit of 170°C or to a high enough temperature to facilitate loading of the spinning cylinder.

Spinning Apparatus Start-Up. While the solution was being prepared, the spinneret assembly was mounted to the bottom of the spinning cylinder. At the same time, all three heaters in the spinning temperature control bath were turned to maximum output and the oil bath was heated to about 80°C. The heaters were then turned off.

After the solution had been prepared, it was poured into the cylinder through a funnel to a height of about four inches from the top of the cylinder. The piston was next inserted in the cylinder and the piston assembly was bolted to the cylinder. Care was taken to avoid contact with hot oil. Finally, the oil bath cover with agitator was fastened in place. The drive chain for the piston was connected and the bracket to hold the piston assembly and cylinder in place was fastened to the frame.

The heaters in the oil bath were turned on maximum output and the agitator in the bath was started. The oil bath temperature was measured with a thermometer. When the desired spinning temperature was reached, the 1450 watt start-up heater was turned off and the power to the other heaters was reduced. The thermostat was set so that the indicator light and controlled heater were off. Voltages to the two heaters in use were kept equal at a value where the temperature did not drift over one

degree Centigrade. Seventy volts was found to be a good setting for this purpose.

After about a 30 minute wait to make certain that the spinning cylinder was at constant temperature, the temperature of the spinneret face was measured with a surface pyrometer, and spinning was attempted. The piston drive motor was turned on; the piston was driven downward at maximum speed until it reached the molten polymer solution. Then, to start spinning, the piston was advanced at a setting of about 1.0 on the variable speed motor controller. If solution did not start to flow from the spinneret, then a heating cord was tied around the spinneret and it was heated until solution could be forced from it by the piston. Then the heating cord was removed and the spinneret was allowed to cool to its normal operating temperature before spinning was continued.

The piston drive was turned on; when a fiber started forming as the solution exited from the spinneret hole, the drum drive was switched on and the fiber was wound around the drum by hand until take-up began. The apparatus was then ready for testing and fiber sample collection.

Temperature Changes. When it was necessary to change the spinning temperature, all three oil bath heaters were switched to maximum output. When the desired temperature was reached, the start-up heater was cut off, power to the other heaters was again reduced, and the thermostat was reset. To insure constant temperature in the system, a fifteen minute wait was allowed before spinning was continued.

Preliminary Testing. Spinning was attempted for a series of

temperatures. This gave an indication of the conditions of composition and temperature at which fiber could be produced.

Selection of Variable Ranges. Immediately after start-up and temperature equilibration, the spinnability ranges at the test composition and temperature were determined. Maximum drum speed was determined as the speed above which the fiber broke at the minimum spinning speed control setting of 0.5 or spinning rate of 0.125 meters per minute. A maximum spinning rate was chosen to give a reasonable range for the drum speed. The minimum drum speed was defined as the drum speed below which the fiber was taken up so slowly that it did not flow smoothly from the spinneret, but rather flowed in droplets.

Sample Collection. After the variable combinations to be used were selected, sample collection was begun. The piston drive motor was turned on and the control was set for the desired spinning rate. As the fiber began to form, the take-up drum was started and adjusted to the first drum speed; the drum speed was measured using a hand tachometer. After fiber formation seemed to be stable (about 10 seconds), a bundle was allowed to form on the take-up drum. When the desired amount of fiber (15-30 seconds running) had been taken up, the piston and drum drive were switched off. The sample was removed from the take-up drum by cutting it parallel to the axis of the drum. The 32-inch sample was transferred to a paper-lined drawer, but about six inches of it were cut off and placed in a capped sample vial.

The next sample was taken at another drum speed in the same manner. The procedure was continued until fiber samples had been collected at

four drum speeds. The spinning speed was set to a new value and samples were taken again at the same four drum speeds. The test was completed when all four drum speeds were used at each of the four spinning speeds. A total time of about 90 minutes was required for the collection of the 16 samples.

Spinning Apparatus Shut-Down. When a test was complete, shut-down was begun by turning off all switches except the oil bath agitator, which was left on to assist in cooling the apparatus. Then the piston was returned to the top of the cylinder.

After the apparatus was cooled, the piston assembly was unbolted and removed along with the spinneret assembly. The piston was detached from the piston assembly, and the piston and spinneret were thoroughly cleaned with tetralin.

Extraction Studies. A number of fiber samples, which had been kept in air-tight vials, were tested to determine the rate at which naphthalene could be extracted from the fibers with ether. The fibers were weighed carefully, placed at room temperature in about 50 milliliters of ether for a time, then allowed to dry in air until constant weight was reached. A control fiber sample was weighed, exposed to air for a time equal to the drying time, and then reweighed.

Before the tensile strength and related tests were run, random samples of the fiber that were to be used were tested for naphthalene content as above.

Denier and Tensilgraph Studies. Fifty individual fibers were chosen at random from each of the fiber bundles to be tested. The

length of each of the selected fibers was measured to the nearest millimeter. Care was taken to extend each fiber to its full length without any stretching. Each group of 50 fibers was bundled together for the tensile testing.

The Scott Tensilgraph was next prepared for operation. The sample length was set at three inches and a clean piece of chart paper was inserted for each test. The recording pen was set to zero on the stress and strain axes of the chart. When the fiber bundles were fastened between the clamps, care was taken to include all fibers and to extend them to their full lengths without stretching. The 250-gram capacity carriage was used.

The Tensilgraph was started and allowed to operate until the fibers were extended to the full capacity of the machine, i.e., six inches, for a total length of nine inches. Then, while this extension was maintained, the carriage clamp was loosened, and the carriage was moved back to its initial position. The fibers were tightened in the carriage clamp again and the Tensilgraph was restarted. When the fibers broke, the amount of extension was noted. The stress-strain chart and the fibers were removed and saved.

After all tensile strength measurements were completed, the deniers of the fiber samples were determined. The 50-fiber bundle that had been used for tensile testing was carefully weighed and the original fiber denier was calculated.

Results

The results of the extraction studies, shown in Table III, indicate that all the naphthalene can be removed from the sample fibers in less than one minute by extraction by ether.

The initial denier and tensile properties of the fibers tested are given in Tables IV and V, respectively. Figures 1-4 show denier, tenacity, per cent elongation at break, and secant modulus, respectively, as functions of draw ratio. Table VI gives the second-degree regression coefficients for the curves of Figures 1-4.

Photographs of four fiber samples are shown in Figure 5.

Table III
Study of Extraction of Naphthalene from Fibers by Ether^a

Test No.	Extraction Time min	% Weight Retained
1	0	94.2
2	1	27.0
3	2	26.8
4	4	26.8
5	8	27.3
6	16	27.6

^aThe fibers for each test were dried in air at room temperature for one hour after extraction.

Table IV
Draw Ratios and Deniers of Fiber Samples

Sample No.	Solution Composition %	Spinning Temperature °C	Draw Ratio	Initial Denier	Denier After 3/1 Draw
I-4-A	15	160	1099	3.47	1.16
-C	15	160	1319	3.03	1.01
-D	15	160	1462	2.61	0.87
II-4-A	15	145	1319	3.40	1.14
-B	15	145	1615	2.81	0.95
-C	15	145	1912	2.40	0.81
-D	15	145	2198	2.56	0.85
III-4-A	22	145	1202	4.59	1.55
-B	22	145	1392	4.40	1.48
-C	22	145	1582	4.07	1.36
-D	22	145	1772	3.79	1.26
IV-4-A	22	160	759	7.97	2.68
-B	22	160	1139	5.34	1.80
-C	22	160	1646	3.78	1.27

Table V

Tensile Properties of Fiber Samples After 3/1 Draw

Sample No.	Solution Composition %	Spinning Temperature °C	Draw Ratio	% Elongation at Break	Tenacity gm/den	Secant Modulus gm/den
I-4-A	15	160	1099	50.0	1.11	23.2
-C	15	160	1319	76.7	1.23	24.2
-D	15	160	1462	80.0	1.33	26.8
II-4-A	15	145	1319	116.7	1.18	22.6
-B	15	145	1615	93.3	1.27	25.6
-C	15	145	1912	120.0	1.71	26.1
-D	15	145	2198	136.7	1.36	26.9
III-4-A	22	145	1202	80.0	1.15	21.8
-B	22	145	1392	100.0	1.27	23.9
-C	22	145	1582	163.3	1.38	24.6
-D	22	145	1772	130.0	1.42	29.6
IV-4-A	22	160	759	50.0	0.89	17.1
-B	22	160	1139	73.3	1.13	25.0
-C	22	160	1646	106.7	1.38	25.2

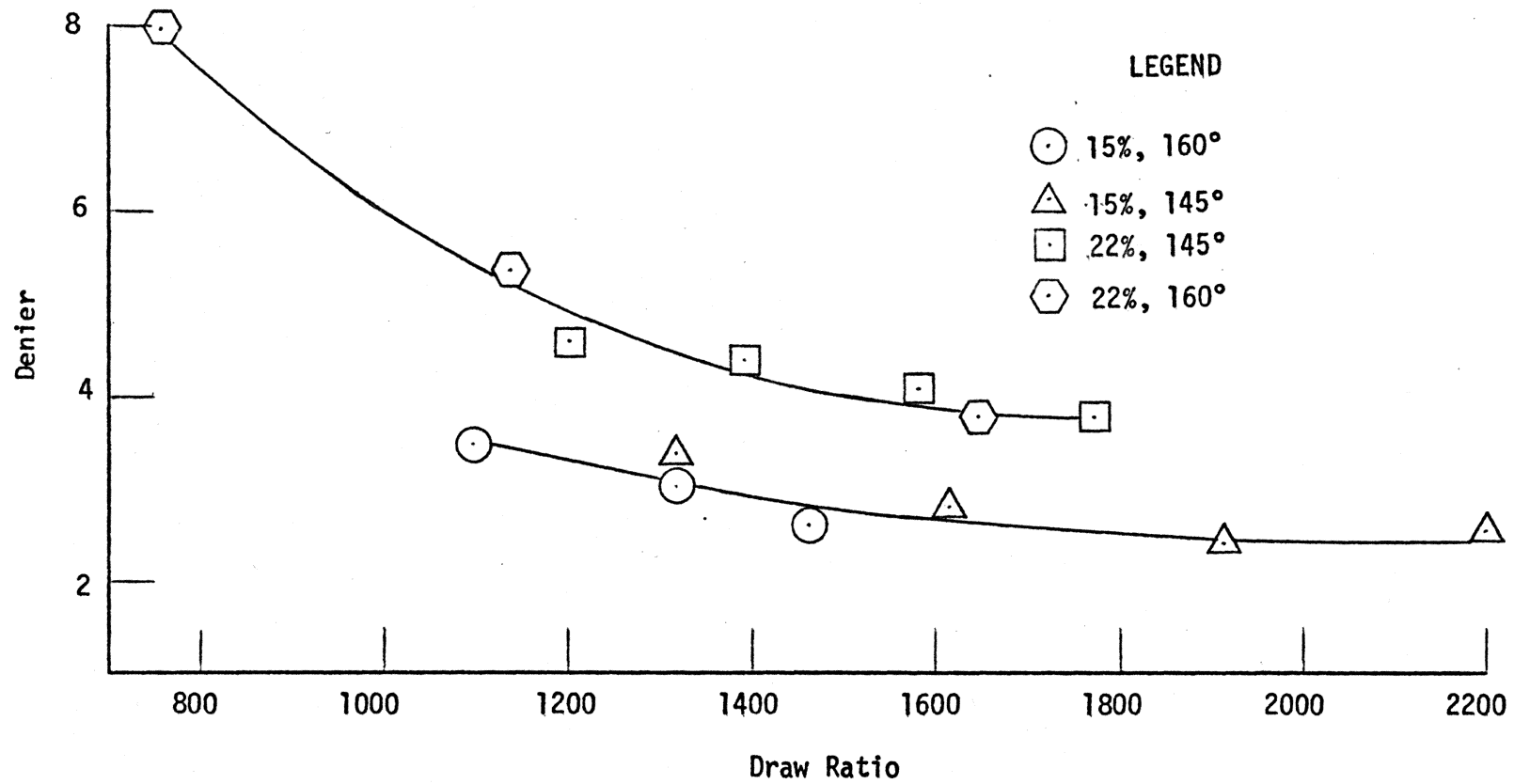


Figure 1. Initial Denier vs. Draw Ratio for Fibers

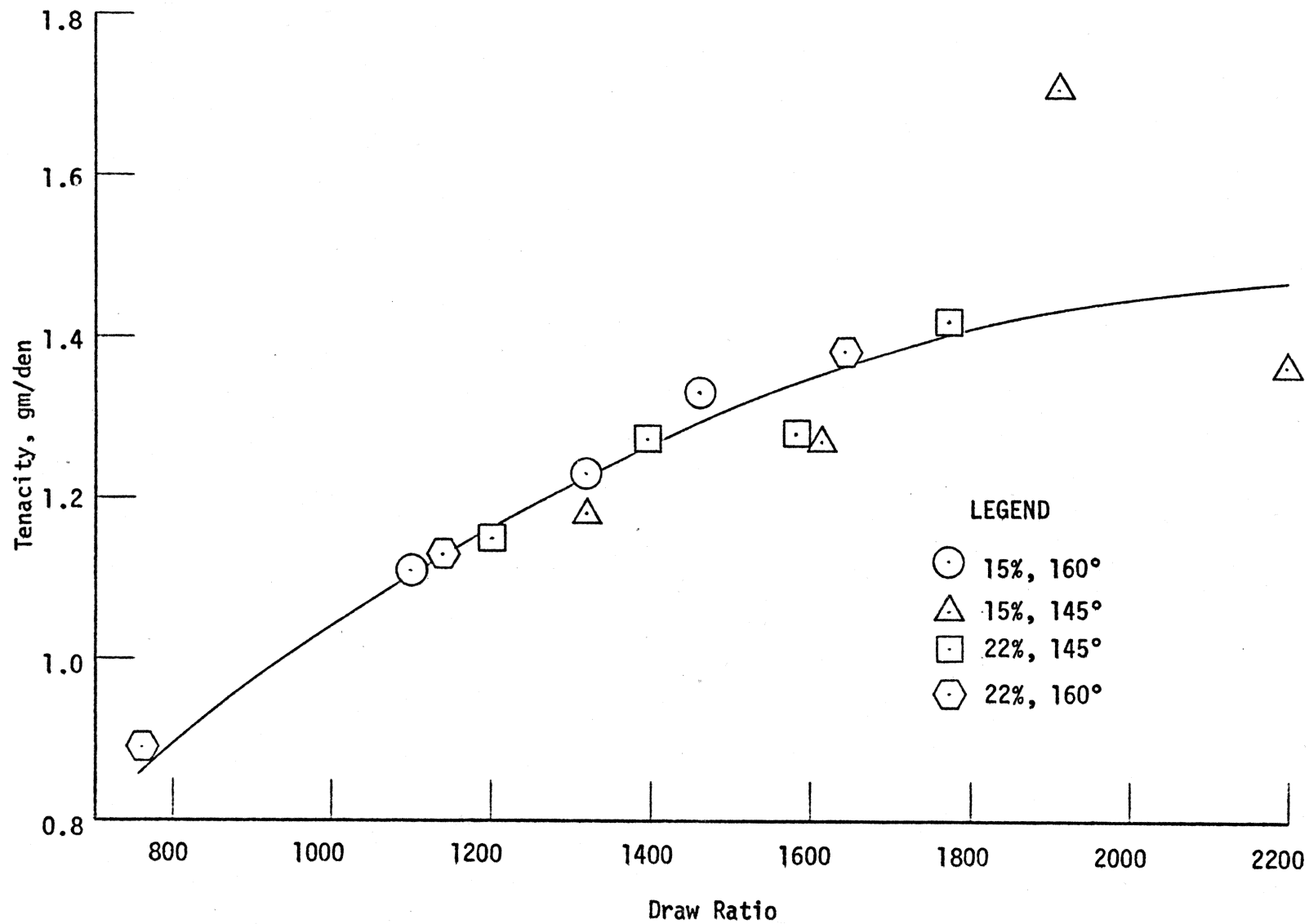


Figure 2. Tenacity versus Draw Ratio for Fibers Drawn 3/1

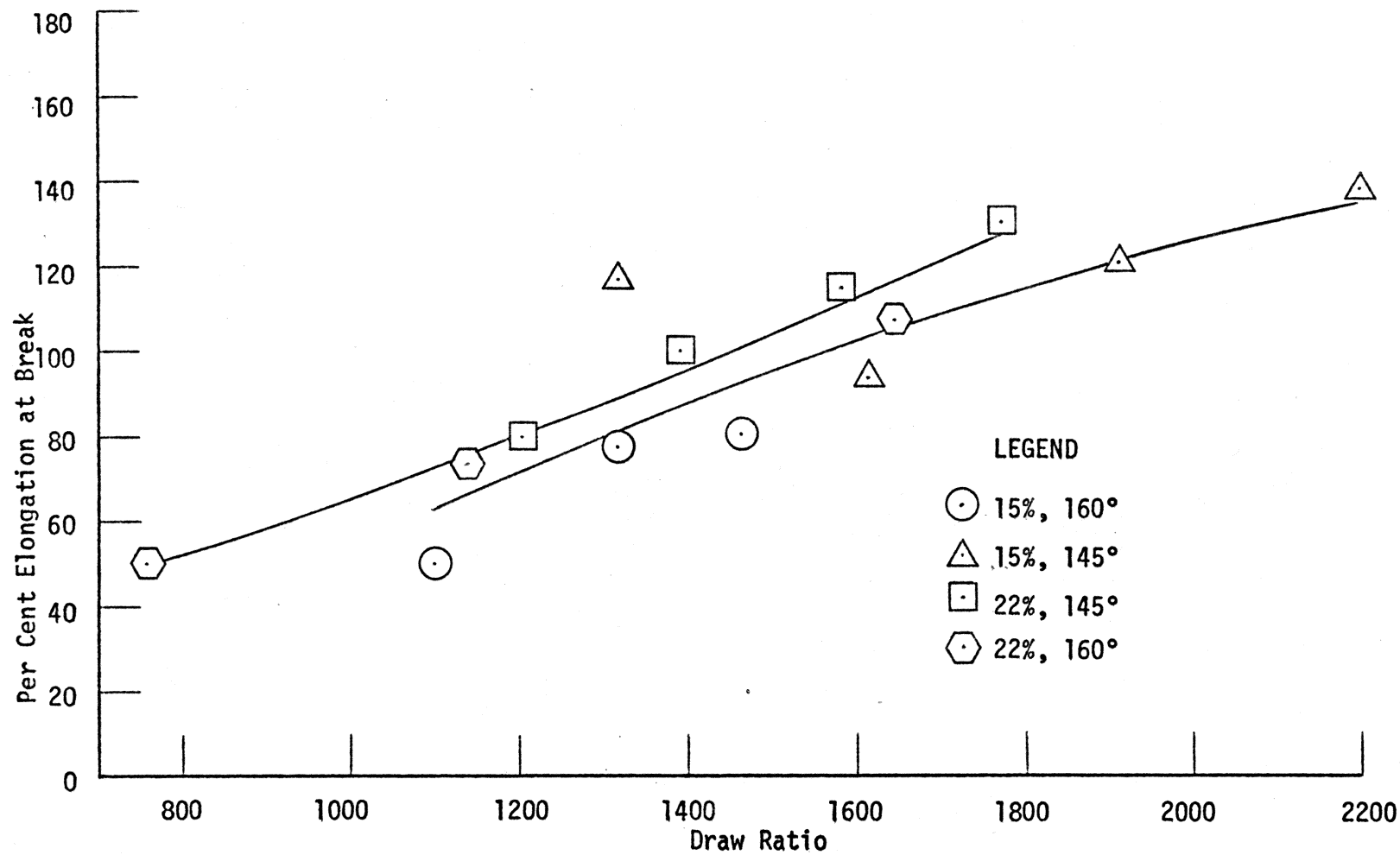


Figure 3. Per Cent Elongation at Break versus Draw Ratio for Fibers Drawn 3/1

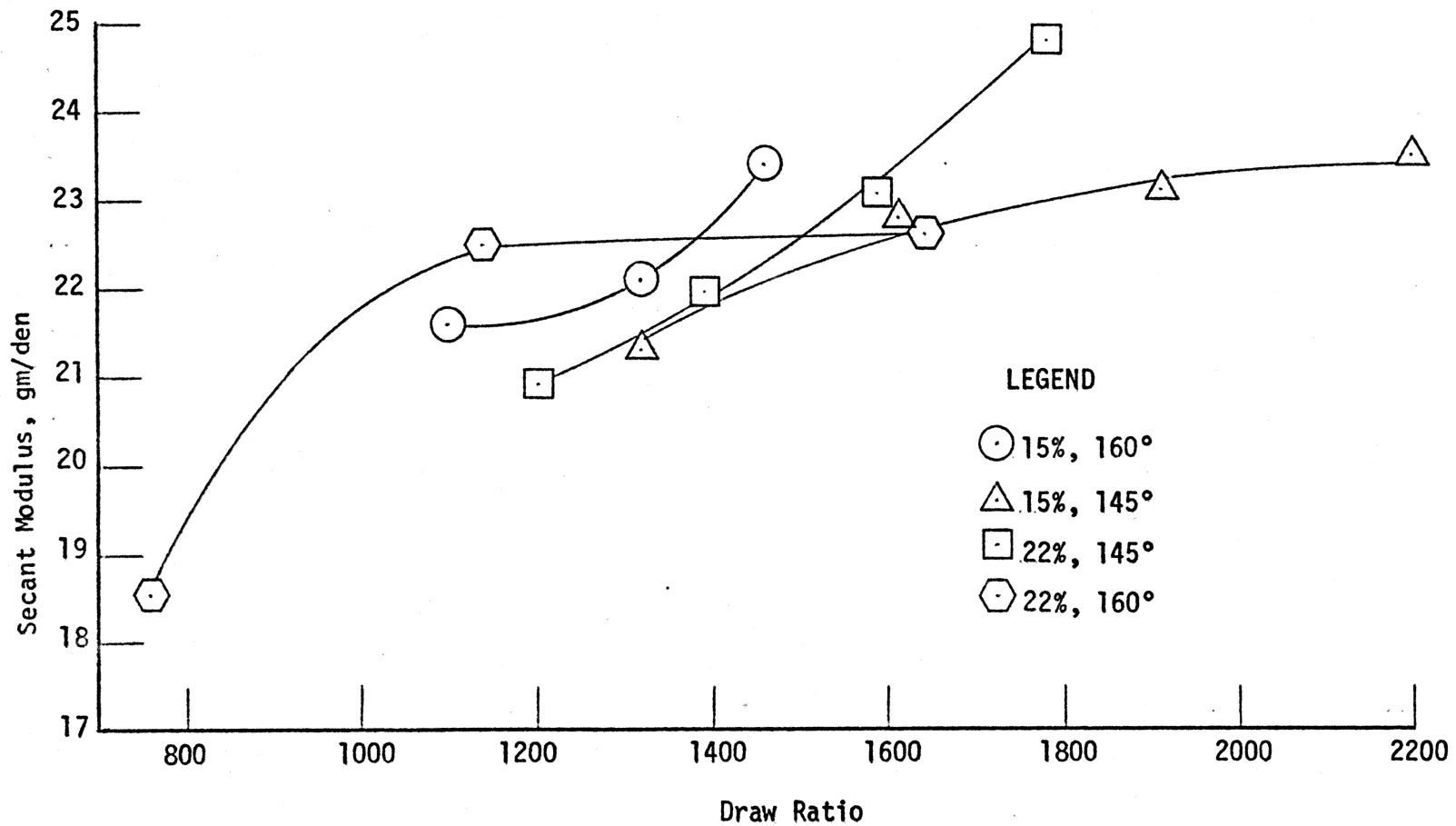


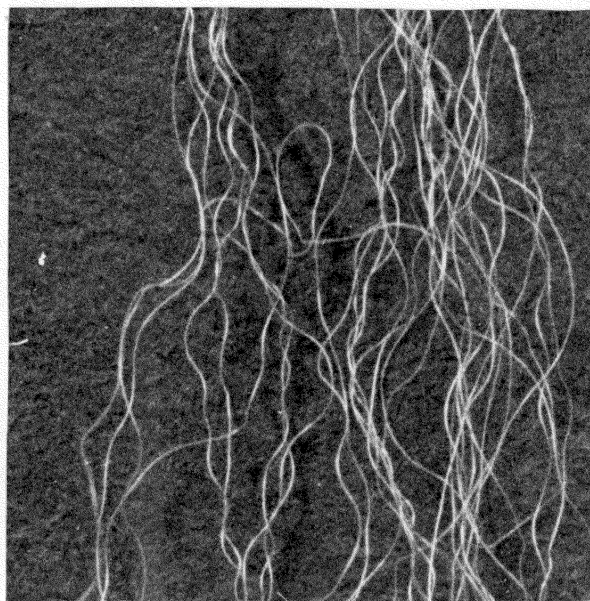
Figure 4. Secant Modulus vs. Draw Ratio for Fibers Drawn 3/1

Table VI
Second-Degree Regression Coefficients for Curves of Figures 1-4

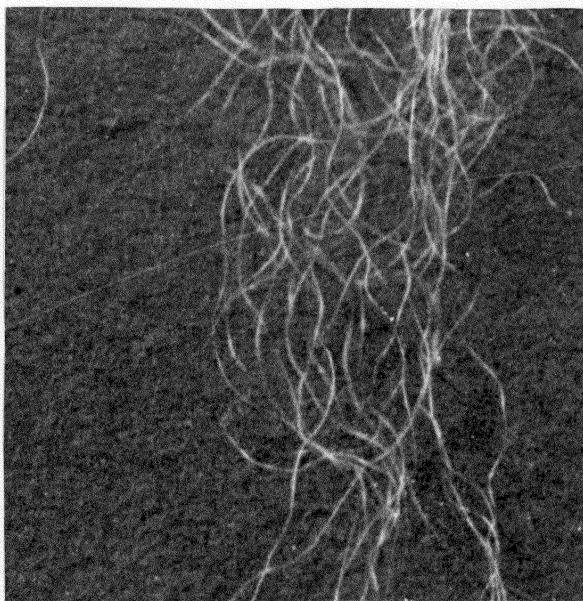
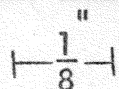
$$Y = A + BX + CX^2$$

X = Draw Ratio

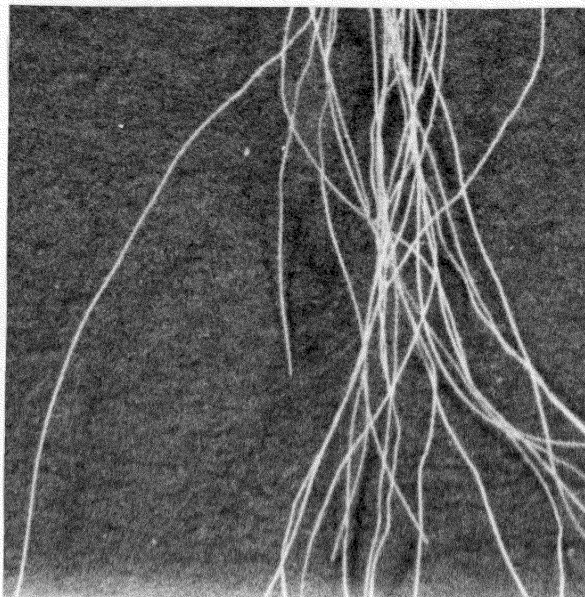
Y	A	Bx10 ³	Cx10 ⁶	Std. Devi- ation Bx10 ³	Std. Devi- ation Cx10 ⁶
Denier (15%) gm/den	7.70	-5.20	1.30	0.497	0.086
Denier (22%) gm/den	17.49	-16.3	4.86	0.464	0.092
Tenacity gm/den	0.105	1.20	-0.26	0.216	0.039
Elongation (15%) %	-62.44	137.4	-21.8	5.48	0.83
Elongation (22%) %	6.43	48.3	11.2	2.40	0.473
Modulus (15%, 145°C) gm/den	-1.67	26.9	-6.34	1.14	0.191
Modulus (22%, 145°C) gm/den	24.67	-13.2	9.00	0.91	0.167



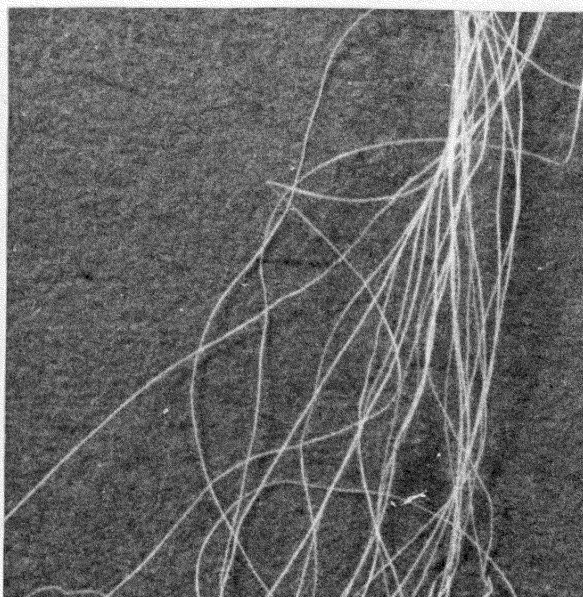
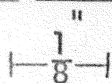
Sample No. III-4-D



Sample No. II-2-D



Sample No. IV-4-A



Sample No. I-4-B

Figure 5. Photographs of Polypropylene Fibers Spun by the Phase-Separation Technique

IV. DISCUSSION

This section is a discussion of the procedures and equipment used in this investigation and of the results obtained. A list of recommendations for further study and equipment modification is included.

Choice of System

The system of polypropylene and naphthalene was selected for this investigation for several reasons. Important were the relatively low melting points and the availability of both components. It was observed that polyolefins are not the most widely used polymers for fiber production although they are among the cheapest and lightest of polymers. It is felt that polypropylene fiber might be made useful for more applications by effecting certain improvements in its characteristics and properties. The phase-separation spinning technique was used in an attempt to accomplish some of these improvements.

Diethyl ether was chosen as the extraction solvent because it is a non-solvent [3] for polypropylene below 80°C, but a solvent for naphthalene. Important also to the choice of diethyl ether is its high volatility which affords fast drying of the fibers after extraction.

Procedures and Equipment

Preparation of the spinning solutions was accomplished, in all cases, without difficulty. The components were easily mixed and dissolved in 30 minutes at concentrations as high as 25 per cent polymer

at solution temperatures of 125°C or less.

Difficulties were encountered when the polymer solution was transferred from the resin kettle to the spinning cylinder. When the hot (160°C) solution was poured into the comparatively cool (80°C) cylinder, partial solidification and some phase separation occurred at the bottom of the cylinder. As the cylinder was heated to its operating temperature, the naphthalene which had precipitated first melted, leaving an amount of highly concentrated polymer solution. The piston could not easily force this concentrated mass through the small spinneret hole. A heating cord placed around the spinneret was used to melt this polymer plug until flow would begin. The mass of concentrated polymer was forced from the spinning cylinder before sample collection was begun.

The spinning apparatus worked well; the chain driven piston mechanism delivered a smooth flow of solution through the spinneret when the take-up drum was operating at proper speed. However, the range of spinning rates that could be used was limited by the maximum drum speed. At low drum speeds, variations were observed in the fiber diameter with droplets rather than a smooth stream of polymer solution flowing from the spinneret. Higher spinning rates could be used if take-up speeds higher than the present limit of 670 meters per minute could be attained.

Collection of fiber samples by cutting them from the take-up drum was sufficient for the purposes of this investigation. However, some problems were encountered in separating individual fibers from the

bundles because of tangling that occurred during handling of the fiber bundles. The accuracy of the denier measurements was probably adversely affected by the use of short (20-30 centimeters) fibers.

Extraction Studies

The results of the extraction studies show that, for the polypropylene-naphthalene system, the spinning solvent could be easily removed by a continuous washing step although continuous washing was not done by the investigator. This step might be incorporated as a part of an industrial spinning line for polypropylene fiber production.

The very low extraction time might indicate that most of the naphthalene migrates to the fiber's surface or that a very porous fiber structure is formed during phase separation.

In any case, use of the phase-separation technique in industry would be influenced by the choice of spinning solvent and washing solvent. For economic reasons, both must necessarily be recovered. At about 90°C, the vapor pressures [6] of naphthalene and diethyl ether are about 15 and 3800 millimeters of mercury, respectively. This high relative volatility would facilitate separation of the solvents for reuse. The use of a much cheaper, but less volatile, washing solvent, such as petroleum ether, would also be possible as evidenced by the work of Zwick. [24]

The washing method used in practice might have considerable control over the characteristics and properties of the fibers. Slower or faster extractions might allow more or less time for the fiber structure

to relax after the solvent is completely removed and before the fibers are cold-drawn or processed further.

Denier Measurements

Figure 1 shows that the initial denier of the polypropylene fibers varied inversely with the draw ratio used in spinning, as would be expected. The fibers made from 15 per cent polymer solution at a spinning rate of 0.277 meters per minute had deniers which were about 25 per cent lower than those of the 22 per cent polypropylene fibers spun at 0.241 meters per minute. Spinning temperatures had little, if any, effect on denier, which is somewhat surprising. Normally, a decrease in denier with increased spinning temperature would be expected. Higher temperatures would result in a more fluid polymer solution which would undergo increased draw-down under its own weight.

Polypropylene fibers spun at comparable spinning rates and draw ratios by the melt-spinning technique might have deniers in the 15-20 range. Therefore, one of the obvious major advantages of the phase-separation technique is the fact that large quantities of fibers with smaller deniers can be produced from large spinnerets by phase-separation spinning. The use of spinnerets with large holes is very advantageous because they are easier to manufacture than spinnerets with small holes, are not as easily plugged, and result in less pressure drop. Also, high draw ratio in any spinning process increases the rate of fiber production.

The deniers obtained in this work, i.e., 2.5-8.0, are certainly

in a very useful range for staple or yarn, but other properties of the fiber might also be of great interest for practical applications.

Tensile Properties

The following is a discussion of the results obtained from measurements made using the Tensilgraph.

Tenacity. It can be seen from Figure 2 that the tenacities of the fibers that were cold-drawn 3:1 increased almost linearly with draw ratio in a range from 0.9 to 1.6 grams per denier. This is the behavior that would be expected since an increase in draw tends to increase the amount of molecular orientation along the fiber axes. An increase in orientation would, in turn, result in improved fiber strength.

It is difficult to establish if any variations in tenacity were caused by differences in the spinning solution concentrations or temperatures. However, a doubling of the draw ratio caused about a 50 per cent increase in the tenacity.

Although the tenacities of the fibers spun by phase-separation spinning are too low for most applications, it should not be difficult to greatly improve those values. This improvement might be accomplished for the same spinning rates and temperatures by the use of different draw ratios and more drawing prior to testing since no attempt was made in this investigation to optimize the conditions. The technique of heat-stretching would also be very useful for the improvement of tenacities by increasing fiber orientation. The high per cent elongation at break values attained indicate that much more drawing than

has been performed would be possible. Again, the type of washing step that is used on the fiber might have some effect on the tensile properties.

Per Cent Elongation at Break. The plot of per cent elongation at break, Figure 3, reveals that if the draw ratio were doubled, the elongation of the fibers which were cold-drawn 3:1 would about double. The values of elongation ranged from about 50 to 150 per cent. Fibers spun from 15 percent polymer solution had about 20 per cent lower elongations at break than did the fibers spun from the 22 per cent solution. The spinning temperatures used had little effect on the elongation values, which is again surprising. Fibers spun from the 15 per cent solutions achieved less elongation at break than those from 22 per cent solutions because they contained less polymer in a given volume or cross-section to hold the fibers together. Higher spinning temperatures should result in more draw-down while the fibers are still fluid, thereby reducing the amount of molecular orientation which can be attained. This would be expected to cause a decrease in per cent elongation to break.

The values of per cent elongation at break that were obtained are too high for most applications. However, just as in the case of the tenacities, this problem can be corrected by several means. Proper combinations of spinning draw ratio and cold-draw ratio or the use of heat-stretching could decrease the elongation values while increasing the tenacities. Again, the washing conditions might also have some control over the elongation.

Secant Modulus. It can be seen from Figure 4 that the values of secant modulus for the fibers which were cold-drawn 3:1 increased with the spinning draw ratios in a range from 17 to 30 grams per denier. Such behavior is expected due to the increase in molecular chain orientation with increasing draw ratio. It is difficult to observe any general trends caused by differences in spinning solution compositions and temperatures, however, especially when the accuracy of the measurements is considered. Small errors in reading the Tensilgraph charts resulted in very large errors in the calculated values due to the constantly changing slopes of the stress-strain curves.

The values of secant moduli calculated are in a useful range for most fiber applications.. It should be noted that, as in the case of tenacities and per cent elongations at break, the moduli of fibers can be improved by the use of optimum spinning conditions and draw ratio, coupled with more cold-drawing or heat-stretching to increase molecular orientation along the fiber axes.

Physical Characteristics of Fibers

In the fibers industry, many of the properties of fibers are judged in almost purely qualitative manners. Very important in determining the usefulness of fibers for most applications are such qualities as crimp, tangling, bulk, and hand.

The fibers that were made by phase-separation spinning were very weak and brittle prior to removal of the naphthalene. Their surfaces were relatively bright and the odor of naphthalene was strong. The

brittleness probably indicates that the naphthalene was present throughout the fine structure of the fibers after phase separation. The surfaces of the fibers were probably covered with naphthalene, also.

After the naphthalene had been removed, the fibers acquired very different properties. As can be seen in the photographs, Figure 5, varying amounts of crimp were present in the fibers. Fibers of lower denier had as many as 15-20 helical crimps per inch while the heavier fibers had little or no crimp at all. This self-crimp was probably caused by stresses that were developed at the surfaces of the fibers as extraction of naphthalene occurred, and such a phenomenon would have more effect on fibers of small cross-section.

Even when the fibers were cold-drawn, some of the crimp was retained. Crimp is very important in many fiber applications and self-crimping is the most desirable type of crimp process. The amount of crimp and the retention of crimp in the fibers can probably be varied by changes in the spinning conditions, the extraction methods, and the drawing steps.

Examination of the fiber surfaces under an optical microscope at a magnification of 400x revealed that the fibers appeared round with rough surface characteristics. The roughness was caused by pockets or pores from which the naphthalene had been removed.

One of the most interesting characteristics was the hand of the fibers. A waxy hand or feel is normally associated with polyolefin fibers and is a disadvantage for the use of such fibers in apparel applications. However, the polypropylene fibers produced by phase-

separation spinning exhibited a much softer, textured hand such as might be expected in cotton. This fact was due primarily to the already mentioned rough surface character of the fibers.

The fibers tended to be very bulky when bundled and tangling occurred very easily. High bulk and tangling are extremely important for fibers in apparel applications because they increase the insulational qualities of yarn.

Fine Structure of Fibers

Many of the properties and characteristics of the fibers produced by phase-separation spinning suggest that the fibers have a fine porous structure. The high solvent extraction rates, the rough surface characteristics, and the low denier all tend to substantiate this statement.

The scanning electron microscope pictures were intended to allow investigation of the fine structure of the fibers more thoroughly.

The porosity of the fibers is of interest when related to fiber applications. High porosity is the major reason for the fact that the fibers are so lightweight and exhibit such a high degree of bulk. A porous structure would also enable fibers to be produced with very high filler content by the mixing of solid filler particles into the spinning solution itself.

Another reason that ordinary polypropylene fibers are not widely used in apparel applications is that they have such low water absorbency or moisture retention. A porous fine structure would substantially

increase the amount of capillary action possible and aid in alleviation of the moisture retention problem.

Recommendations

The following is a list of recommendations for equipment modification and further study.

Equipment and Procedure Modifications. One of the major problems encountered in producing fibers by phase-separation spinning was the necessity of loading the spinning cylinder while it was still relatively cold. This resulted in formation of a plug in the spinneret, making spinning difficult. This problem could be remedied by installing an injection cylinder as part of the apparatus or by preparation of the spinning solution in the spinning cylinder itself. In the former case, the solution could be loaded into the already heated spinning cylinder. In the latter case, it would not be necessary to transfer the solution at all.

The Leesona variable tension fiber take-up unit should be included as part of the spinning apparatus. Samples that are collected on a cylindrical package using this device could be much more easily handled during extraction and actual testing.

The take-up drum drive should be modified to reduce vibration and to increase the range of speeds available. A drive motor with higher speeds or a different pulley arrangement would make the latter modification possible.

Further Studies. The most immediate further study that would be recommended is complete testing of all of the 64 fiber samples that were made and collected in this investigation. A much more thorough analysis of the results would be obtained.

Secondly, many more fiber samples should be made using different combinations of spinning conditions. A series of statistically designed experiments would have much to offer.

Experiments involving different means of solvent extraction and varying degrees of cold- and hot-stretching should be conducted in an attempt to improve the tensile properties of the fibers which are made.

V. CONCLUSIONS

A phase-separation technique for fiber production was studied in this investigation. The system of polypropylene and naphthalene was spun from a 2.00 millimeter spinneret hole at polymer solution concentrations of 15 and 22 per cent and temperatures of 145 and 160°C. The results of the investigation led to the following conclusions:

1. Polypropylene fibers could be spun by the phase-separation technique using the system and apparatus studied.

2. Feasible spinning rates and take-up speeds varied with spinning solution composition and temperature in ranges from 0.125 to 0.277 and from 183 to 610 meters per minute, respectively.

3. Deniers of tested fibers ranged from 2.5 to 8.0 and varied inversely with draw ratio.

4. Tenacities, per cent elongations at break, and initial moduli of fibers drawn 3:1 increased with draw ratio. Values ranged from 0.9 to 1.6 grams per denier, from 50 to 150 per cent, and from 17 to 30 grams per denier, respectively.

5. The fibers exhibited good hand, bulk, crimp, and other physical characteristics which are very important properties in industrial applications.

VI. SUMMARY

The first purpose of this investigation was to design, build and test an apparatus for making polypropylene fiber by the phase-separation technique. The second purpose was to produce fibers using the apparatus and to determine the properties of the fibers.

Solutions of polypropylene in naphthalene were spun at solution compositions of 15 and 22 per cent polymer and temperatures of 145 and 160°C.

Extraction of naphthalene from the fibers by diethyl ether occurred almost instantaneously; however, this extraction was not necessary prior to properties testing because all of the naphthalene was lost into the air.

Deniers of 14 samples selected varied inversely with draw ratio and directly with solution composition. After the samples were cold-drawn 3:1, tensile testing revealed that tenacities and initial moduli varied directly with draw ratio and that per cent elongation at break varied directly with draw ratio and solution composition.

The fibers exhibited good hand, bulk, and self-crimping characteristics.

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20. Ibid., p. 145-6.
21. Ibid., p. 146.
22. Ibid., pp. 129, 134-5.
23. Ibid., p. 148.
24. Ibid., p. 130.

VIII. APPENDIX

The appendix contains information which would be required in order to reproduce the results of this investigation. A complete listing and description of all materials and apparatus used is also included.

Data Tables

This section contains the data obtained during the investigation. Tables VII-X give the spinning conditions for each test and list the samples that were collected. Tables XI-XIII are data from the extraction studies, the denier measurements, and the tensile properties measurements, respectively.

Calibration of Piston Drive Motor Control Settings

Figure 7 is a calibration curve for obtaining the linear velocity of the melt solution in the spinneret from the piston drive motor control setting. The time for one revolution of the motor's drive shaft was determined at three different control settings. At settings of 0.5, 0.75, and 1.0, the revolution times were 35, 20.5, and 13.7 seconds, respectively. These times corresponded to 1.72, 2.93, and 4.38 revolutions per minute.

The two sprockets in the chain drive had nine and 25 teeth; the threaded rod had 20 threads per inch, so the rod and piston travelled $1/20$ or 0.05 inches per revolution of the large sprocket. Thus, for the 0.5 control setting, the piston travel rate was calculated as follows:

Table VII

Conditions and Samples Collected for Test I

Composition of Spinning Solution = 15.0% Polypropylene
 Bath Temperature = 160°C
 Spinneret Temperature = 94°C
 Spinning Rate Range = 0.5 to 0.9 (control setting)
 Drum Speed Range = 304 to 405 m/min

Sample No.	Spinning Rate (control setting)	Drum Speed (m/min)
I-1-A	0.5	305
I-1-B	0.5	335
I-1-C	0.5	366
I-1-D	0.5	405
I-2-A	0.65	305
-B	0.65	335
-C	0.65	366
-D	0.65	405
I-3-A	0.75	305
-B	0.75	335
-C	0.75	366
-D	0.75	405
I-4-A	0.9	305
-B	0.9	335
-C	0.9	366
-D	0.9	405

Table VIII

Conditions and Samples Collected for Test II

Composition of Spinning Solution = 15.0% Polypropylene
 Bath temperature = 145°C
 Spinneret Temperature = 90°C
 Spinning Rate Range = 0.5 to 0.9 (control setting)
 Drum Speed Range = 366 to 610 m/min

Sample No.	Spinning Rate (control setting)	Drum Speed (m/min)
II-1-A	0.5	366
-B	0.5	448
-C	0.5	530
-D	0.5	610
II-2-A	0.65	366
-B	0.65	448
-C	0.65	530
-D	0.65	610
II-3-A	0.75	366
-B	0.75	448
-C	0.75	530
-D	0.75	610
II-4-A	0.9	366
-B	0.9	448
-C	0.9	530
-D	0.9	610

Table IX

Conditions and Samples Collected for Test III

Composition of Spinning Solution = 22.0% Polypropylene
 Bath Temperature = 145°C
 Spinneret Temperature = 90°C
 Spinning Rate Range = 0.5 to 0.8 (control setting)
 Drum Speed Range = 290 to 427 m/min

Sample No.	Spinning Rate (control setting)	Drum Speed (m/min)
III-1-A	0.5	290
-B	0.5	335
-C	0.5	381
-D	0.5	427
III-2-A	0.6	290
-B	0.6	335
-C	0.6	381
-D	0.6	427
III-3-A	0.7	290
-B	0.7	335
-C	0.7	381
-D	0.7	427
III-4-A	0.8	290
-B	0.8	335
-C	0.8	381
-D	0.8	427

Table X
Conditions and Samples Collected for Test IV

Composition of Spinning Solution = 22.0% Polypropylene
Bath Temperature = 160°C
Spinneret Temperature = 94°C
Spinning Rate Range = 0.5 to 0.8 (control setting)
Drum Speed Range = 183 to 488 m/min

Sample No.	Spinning Rate (control setting)	Drum Speed (m/min)
IV-1-A	0.5	183
-B	0.5	274
-C	0.5	396
-D	0.5	488
IV-2-A	0.6	183
-B	0.6	274
-C	0.6	396
-D	0.6	488
IV-3-A	0.7	183
-B	0.7	274
-C	0.7	396
-D	0.7	488
IV-4-A	0.8	183
-B	0.8	274
-C	0.8	396
-D	0.8	488

Table XI
Data from Extraction Studies^a

Test No.	Initial Weight of Fibers (gm)	Extraction Time (min)	Final Weight (gm)
1	0.0870	0	0.0819
2	0.0953	1	0.0257
3	0.1356	2	0.0364
4	0.1085	4	0.0291
5	0.1115	8	0.0304
6	0.1030	16	0.0284

^aThe fibers for each test were dried in air at room temperature for one hour after extraction.

Table XII
Data from Denier Measurements

Sample Number	Total Length of Fiber (cm)	Weight of Fiber (gm)
I-4-A	1476.7	0.0057
I-4-C	1543.6	0.0052
I-4-D ^c	1308.6	0.0038
II-4-A	1269.9	0.0048
-B	1315.7	0.0041
-C	1428.1	0.0038
-D ^b	809.9	0.0023
III-4-A	1489.3	0.0076
-B ^a	397.7	0.0019
-C	1371.7	0.0062
-D	1043.9	0.0044
IV-4-A	1377.2	0.0122
-B	1196.0	0.0071
-C	1334.2	0.0056

^aOnly 15 fibers used in this measurement.

^bOnly 40 fibers used in this measurement.

^cOnly 46 fibers used in this measurement.

Table XIII

Data from Tensile Property Tests for 3/1 Drawn Fibers

Sample Number	Number of Fibers	Actual Stretch (in)	Breaking Strength (gm)	Force at 0.1 in. Stretch (gm)
I-4-A	49	1.5	63	44
-C	49	2.3	61	40
-D	45	2.4	52	35
II-4-A	50	3.5	67	43
-B	48	2.8	61	39
-C	49	3.6	67	35
-D	38	4.1	44	29
III-4-A	49	2.4	86	55
-B	50	3.0	93	59
-C	44	3.5	77	52
-D	49	3.9	88	61
IV-4-A	48	1.5	113	73
-B	48	2.2	97	72
-C	49	3.2	85	52

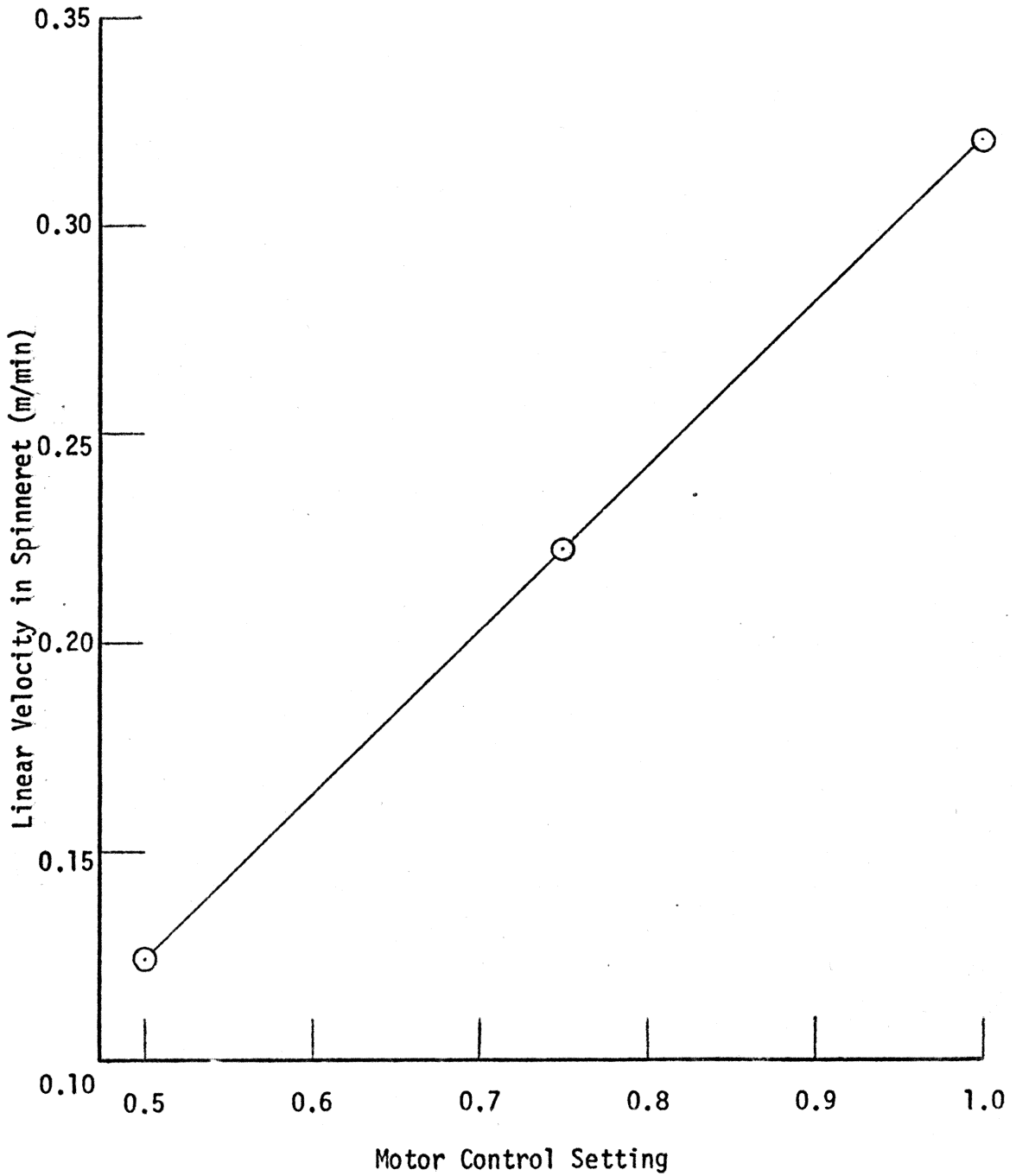


Figure 7. Calibration for Piston Drive Motor Control

$$\text{Piston travel rate} = 1.72\left(\frac{9}{25}\right)(0.05) = 0.0309 \text{ in/min}$$

Similar calculations were made for the other control settings.

The inner diameters of the spinning cylinder and spinneret hole were 0.998 and 0.079 inches, respectively. Cross-sectional areas of each are:

$$\text{Area} = \frac{\pi}{4} (\text{Diameter})^2$$

$$\text{Area (cylinder)} = \frac{\pi}{4} (.998)^2 = 0.784 \text{ in}^2$$

$$\text{Area (spinneret)} = \frac{\pi}{4} (0.079)^2 = 0.0049 \text{ in}^2$$

Thus, the linear velocity in the spinneret hole could be determined for each of the three control settings. For the 0.5 setting:

$$\text{Linear velocity} = \frac{0.784}{0.0049} (0.0309) = 4.94 \text{ in/min} = 0.125 \text{ m/min}$$

For the two higher settings, the values were 0.222 and 0.320 meters per minute. The calibration curve, Figure 7, was drawn using these three calibration points.

Sample Calculations

The following are examples of calculations made during this investigation.

Draw Ratio. Draw ratio is the tangential drum speed divided by the spinning rate or linear velocity in the spinneret hole. For a spinning rate control setting of 0.9, the corresponding linear velocity in the

spinneret from Figure 7 is 0.277 meters per minute. For Sample No. II-4-C, the drum speed was 530 meters per minute from Table VIII. The draw ratio was calculated as follows:

$$\text{Draw Ratio} = \frac{530 \text{ m/min}}{0.277 \text{ m/min}} = 1912$$

Denier. Denier is defined as the weight in grams of 9000 meters of a fiber or:

$$\text{Denier} = 900,000 \frac{(\text{weight of fiber, gm})}{(\text{total length of fiber, cm})}$$

Substituting data from Table VIII, for Sample No. II-4-C:

$$\text{Denier} = 900,000 \frac{(0.0038)}{1428.1} = 2.395$$

Tenacity. Tenacity at break for the drawn fiber is defined as follows:

$$\text{Tenacity} = \frac{\text{Breaking strength per fiber, gm}}{\text{Denier of drawn fiber, den}}$$

Figure 8 is the tensilgraph chart for Sample No. IV-4-B. The breaking strength was recorded as 97 grams. Since there were 48 fibers intact in this case, the breaking strength per fiber was:

$$\text{Breaking Strength per fiber} = \frac{97}{48} = 2.02 \text{ gm}$$

From Table IV, the initial denier was 5.34. Since the fiber was drawn 200 per cent, the denier of the drawn fiber was one-third of this or 1.80. Finally, the tenacity was calculated as follows:

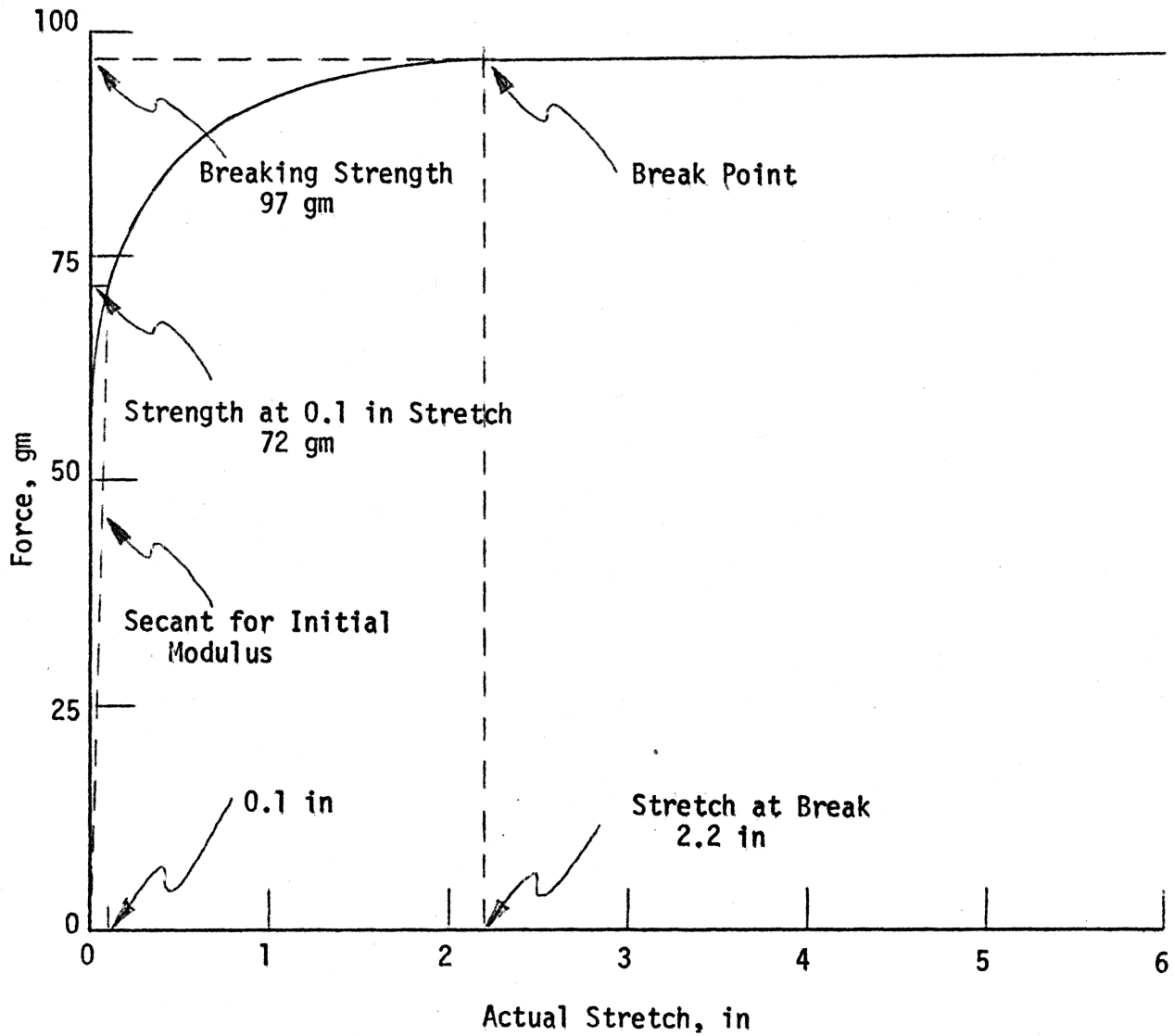


Figure 8. Replica of Stress-Strain Chart for Sample No. IV-4-B

$$\text{Tenacity} = \frac{2.02}{1.80} = 1.134 \text{ gm/den}$$

Elongation at Break. Per cent elongation at break is expressed as follows:

$$\% \text{ Elongation} = \frac{\text{Change in length at break, in}}{\text{Original length, in}} \times 100$$

Again referring to Figure 8, the change in length at break for the drawn fiber of Sample No. IV-4-B can be read as 2.2 inches. The original length of all fibers was 3.0 inches. Then per cent elongation of the drawn fiber was calculated as follows:

$$\% \text{ Elongation} = \frac{2.2}{3.0} \times 100 = 73.3\%$$

Secant Modulus. The initial modulus of a fiber is the slope of the stress-strain curve at a strain infinitesimally greater than zero. For these tests, the secant approximation was used. A secant to each curve was drawn between the origin and a point on the curve at 0.1 inches of stretch.

The slope of this straight line was taken as the secant modulus, an approximation of the initial modulus. Fiber moduli are ordinarily expressed in units of grams per denier, so the following equation for the secant modulus was used:

$$\text{Secant Modulus} = \frac{\text{Force per fiber at 0.1 inches stretch, gm}}{\text{Denier of drawn fiber} \times \text{strain}}$$

The strain, of course, was constant for a 0.1 inch stretch as:

$$\text{Strain} = \frac{\text{Change in length, in}}{\text{Original length, in}} = \frac{0.1}{3.0} = 0.0333$$

The force at 0.1 inch stretch can be read from Figure 8, as 72 grams for Sample No. IV-4-B. Since 48 fibers were intact:

$$\text{Force per fiber} = \frac{72}{48} = 1.50 \text{ gm}$$

From Table IV, the denier of the drawn fiber was 1.80. Then, the secant modulus for Sample No. IV-4-B was calculated as follows:

$$\text{Secant Modulus} = \frac{1.50}{1.80(.0333)} = 25.0 \text{ gm/den}$$

Materials

The materials used in this project are described in this section.

Diethyl Ether. Anhydrous. Reagent grade. Purchased from Fisher Scientific Co., Fairlawn, N. J. Used to extract naphthalene from fibers.

Hydroquinone. Crystals, purified. Purchased from Fisher Scientific Co., Fairlawn, N. J. Used to inhibit diethyl ether.

Naphthalene. Crystals, certified. Lot 704164. Residue after ignition 0.001%. Purchased from Fisher Scientific Co., Fairlawn, N. J. Used as solvent in polymer solution.

Polypropylene. Dry powder, uninhibited. Lot No. 95-02000-2943. Purchased from Enco by Preston Durill. Used as polymer in fiber production.

Stopcock Grease. Sisco 300. Manufactured by Swedish Iron and Steel Corp., Westfield, N. J. Used to seal resin kettle.

Tetrahydronaphthalene (Tetralin). Purified. Purchased from Fisher Scientific Co., Fairlawn, N. J. Used as cleaning solvent for glassware and spinning apparatus.

Apparatus

The apparatus used in this project are described in this section.

Agitator. With chuck and stirrer. 115 volts. Purchased from Fisher Scientific Co., Chicago, Ill. Used to stir solution in resin kettle.

Balance. Type H15. Capacity 160 grams. No. 263600. Manufactured by Mettler Instrument Corp., Hightstown, N. J. Purchased from Scientific Products, Evanston, Ill. Used to weigh fibers for denier measurements.

Balance. Single-beam type. Capacity 1100 grams. Manufactured by Eimer and Amend, New York, N. Y. Purchased from Fisher Scientific Co., Pittsburgh, Pa. Used to weigh components of polymer solution.

Chart Paper. Chart No. SCT10(C19). Ten-x six-inch axes. Manufactured by Graphic Controls Corp., Buffalo, N. Y. Used to record stress-strain curves for tensilegraph tests.

Heater. Autemp. Type H12. 115 volts. Purchased from Fisher Scientific Co., Chicago, Ill. Used to heat tetralin for cleaning equipment.

Heating Mantle. 115 volts. No. 5709. Manufactured by Glas-Col Apparatus Co., Terre Haute, Ind. Used for heating resin kettle when melting polymer solution.

Heating Cord. Cal-cord 400. Flexible. Limit 400°C. Rating 120

watts, 115 volts. Three feet long. Manufactured by Glas-Col. Purchased from Fisher Scientific Co., Chicago, Ill. Used to heat spinneret during startup.

Pyrometer. Portable. Type 4200, No. 155418. With Type 4220 extension arm and Type 4090 thermocouple. Five °F accuracy. Range 0-600°F. Manufactured by Alnor Instrument Co Division, Illinois Testing Laboratories, Inc., Chicago, Ill. Used to measure temperature of spinneret face.

Resin Kettle. Two piece type. Pyrex glass. Top has one 14/35, two 24/40, and one 19/42 fittings. With metal kettle clamps. Manufactured by Ace Glass, Inc., Vineland, N. J. Used as vessel in which polymer solution was made.

Ring Stand. Purchased from Fisher Scientific Co., Chicago, Ill. Used to support resin kettle and agitator.

Spinning Apparatus. Assembled in the Chemical Engineering Shop, Virginia Polytechnic Institute and State University, Blacksburg, Virginia. Figures 9 and 10 are, respectively, a schematic of the apparatus and a wiring diagram for the same.

The spinning apparatus consists primarily of a framework, a vertical spinning cylinder surrounded by an oil bath, a piston with a variable speed drive assembly, a single-hole spinneret, and a fiber take-up drum with variable speed drive.

The framework, which was constructed in the shop of one-inch angle iron, is three feet wide by two feet deep and has two levels. A lower level, which is enclosed by plywood on the sides and the back, is seven

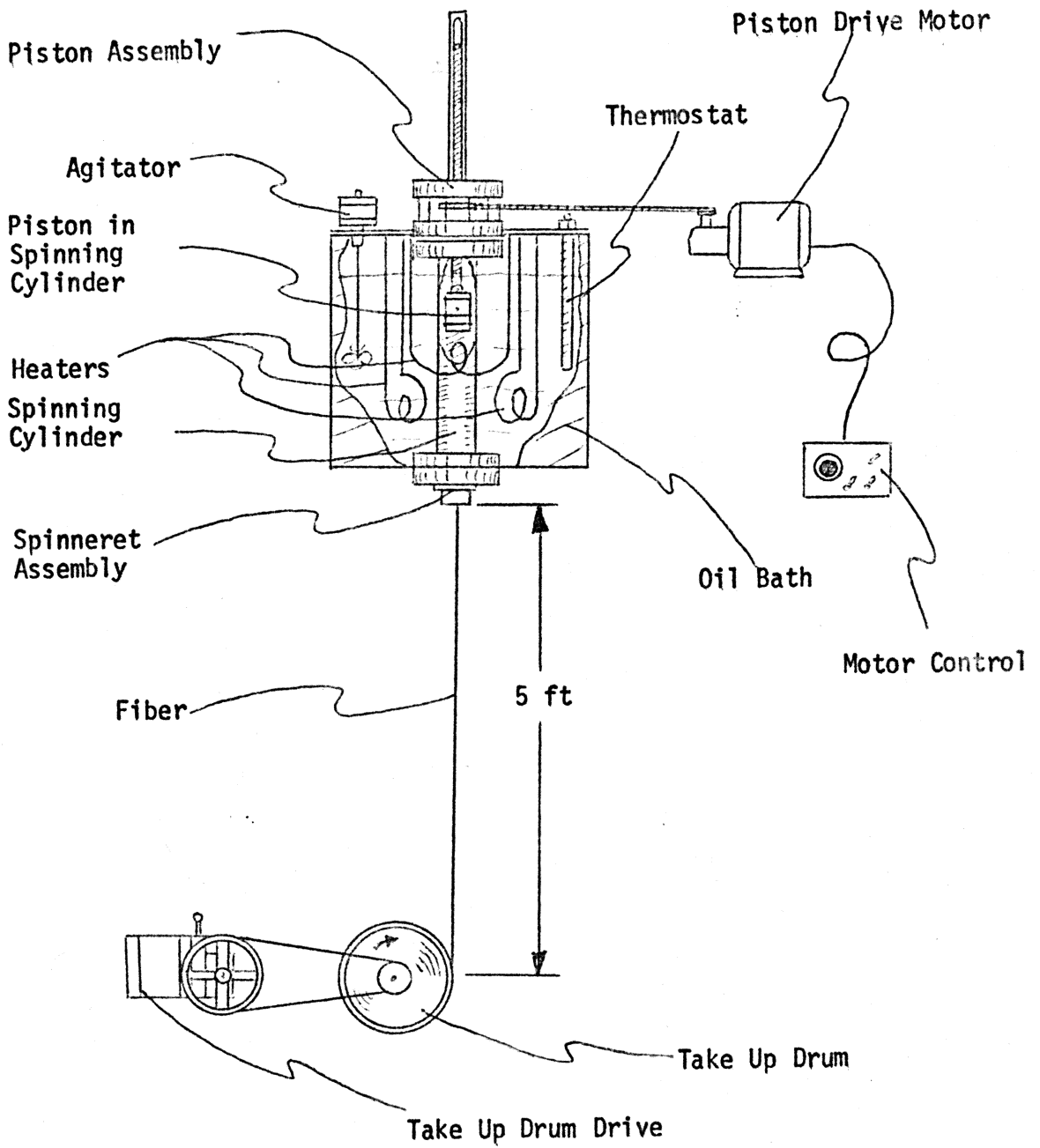


Figure 9. Schematic Diagram of Fiber Spinning Rig

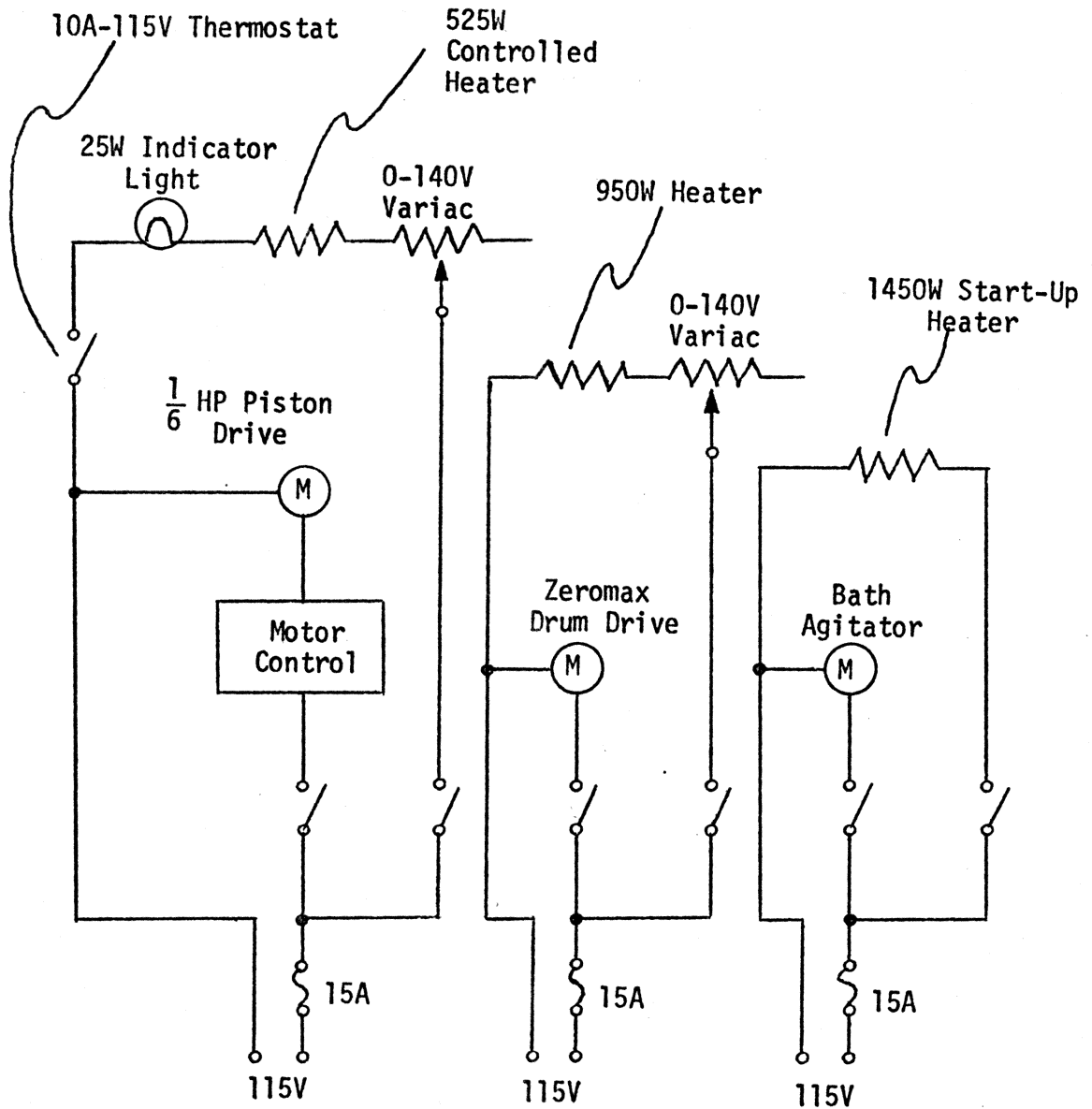


Figure 10. Spinning Apparatus Electrical Diagram

feet high. A control panel is located on the right side of this level. The upper level, which is 18 inches high, serves to support the spinning cylinder, oil bath, piston assembly, and piston drive. A 1 1/2-inch wide, 1/4-inch thick steel bar with a half circle slot cut in one side can be bolted to the sides at the top of the frame to hold the spinning cylinder in place during spinning.

The oil bath was constructed of sheet metal and is one foot square by 14 inches high. Four 1/2-inch bolt holes and one two-inch hole in which the spinning cylinder fits were cut in the bottom of the oil bath. A valve was installed near the bottom for draining oil and one-inch insulation was applied to the sides and bottom. Teresso 65 oil was used as a heating medium.

Two rectangular, 1/4-inch thick, 12- x six-inch sheets of bakelite served as a cover for the oil bath and as mounts for an agitator, three tubular heaters, and a thermostat. Each sheet is fastened to a rim around the oil bath by three metal screws.

One heater is used only for start-up; another is operated during start-up and during each test. The third is controlled by the thermostat and is used during start-up and the tests. All heaters are controlled by switches and the latter two by variacs. A light indicates when the controlled heater is in operation.

Figure 11 is an exploded view of the piston assembly, spinning cylinder, and spinneret assembly. The following should describe the various parts shown in Figure 11 and their uses.

The piston itself was machined from an aluminum block. It is two

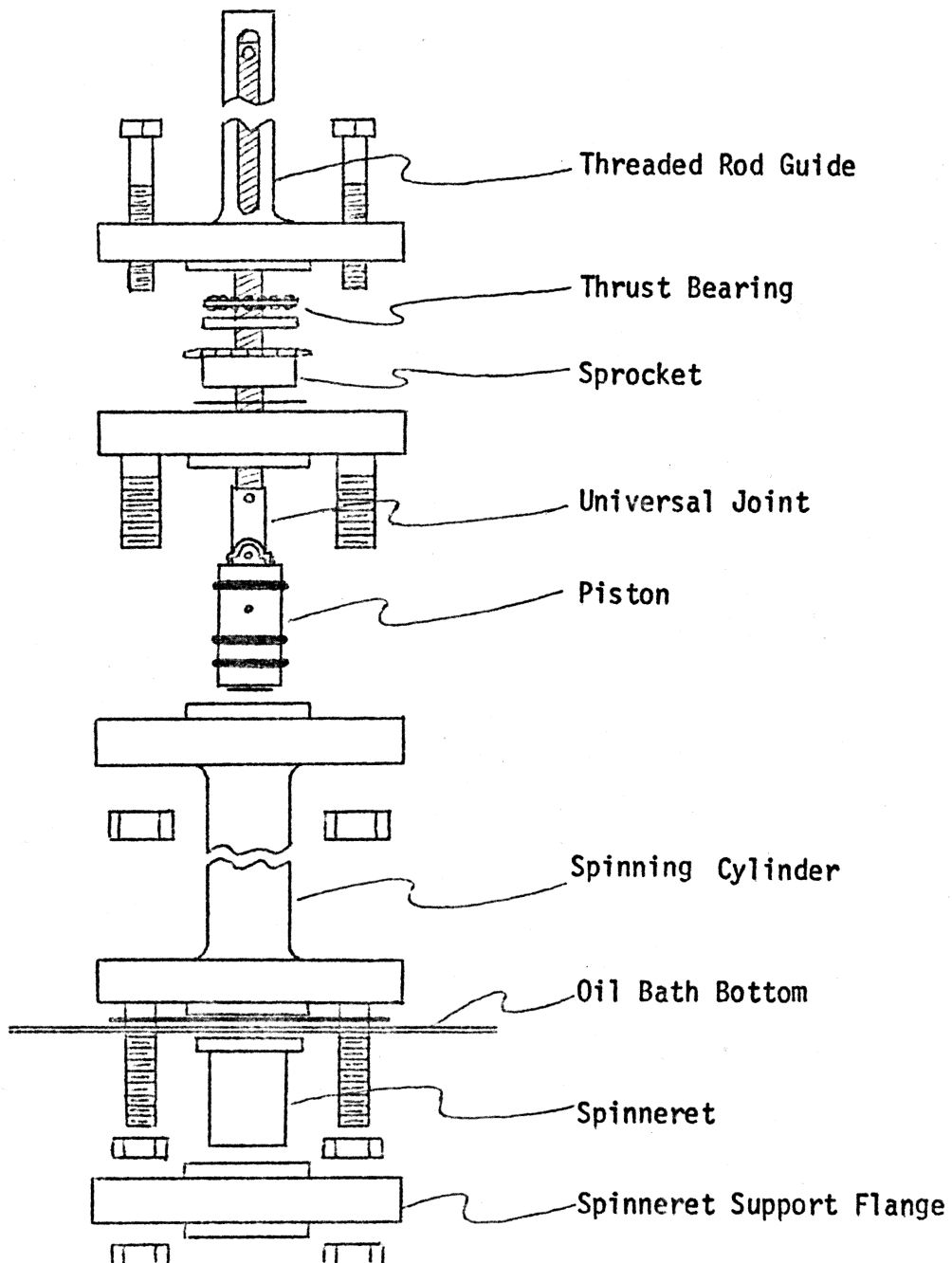


Figure 11. Assembly View of Piston Assembly, Spinning Cylinder, and Spinneret Assembly

inches long and 0.995 inches in diameter. Grooves for Parker No. 2-210 O-rings were cut $\frac{3}{8}$ inch from each end and about $\frac{3}{4}$ inch from the bottom end. The piston is hollow and fits on a $2 \frac{1}{4}$ inch long universal joint, held in place by two $\frac{1}{8}$ -inch set screws. A valve was placed in the bottom of the piston to enable air to escape from the cylinder when the piston is guided into it. The valve is brass and consists of a $\frac{1}{32}$ -inch thick, $\frac{3}{4}$ -inch diameter disc which is welded to a $\frac{3}{16}$ -inch diameter, triangular cross-section rod. This rod slips easily into a hole drilled through the piston bottom. The end of the rod, which is threaded, extends about $\frac{3}{16}$ inch into a hollow space between the end of the universal joint and the inside bottom of the hollow piston. The valve is held in place by a nut which allows about $\frac{1}{16}$ inch of displacement for valve operation.

The universal joint is connected to a $16 \frac{3}{4}$ inch long, $\frac{1}{2}$ -inch diameter, 20 threads per inch, steel, threaded rod. The rod passes vertically upward through a standard, 600-pound, one-inch blind flange which has been drilled to allow passage of the rod.

A steel, $1 \frac{1}{2}$ -inch diameter, $\frac{1}{4}$ -inch pitch sprocket gear with 25 teeth is fitted on the rod. The inner bore of this sprocket was tapped to fit the threaded rod. The sprocket is a No. 25B25, manufactured by Browning Manufacturing Division, Emerson Electric Co., Maysville, Kentucky and purchased from Power Transmission Corp., Roanoke, Virginia. This sprocket, when turned, causes the piston to move vertically.

Directly above the sprocket in the piston assembly is a thrust bearing and back-up plate. The diameter of the bearing is $1 \frac{1}{4}$ inches

and it contains thirteen 3/16-inch balls. The thrust bearing, in turn, fits into a depression which was cut in the bottom of another 600-pound blind flange. This flange was also drilled to allow free passage of the threaded rod.

Welded to the top of the upper flange is a vertical 15-inch length of 1/2-inch black-iron pipe. This pipe serves as a guide and support for the threaded rod as it moves. A 1/4-inch wide, vertical slot was cut along the length of the pipe, and a pin attached to the end of the threaded rod fits through this slot. The slot and pin serve to prevent the rod from turning as the sprocket turns.

The sprocket, bearing, and a thin brass washer are held together between the two flanges by four 2 1/2 inch long, 3/8-inch bolts. These bolts pass through holes in the top flange and screw into threaded holes in the bottom flange. These holes are located halfway between the four standard boltholes in the flanges. Proper spacing between the flanges is maintained by brass sleeves which fit over each bolt. Four two inch long, 5/8-inch bolts were welded in the boltholes of the bottom flange.

The spinning cylinder was made by boring and honing a 14-inch length of one-inch, schedule 80, black-iron pipe to an inside diameter of 0.998 inches. A standard, 600-pound, slip-on flange was welded to each end of this pipe. Four two inch long, 1/2-inch bolts were welded in the boltholes of one flange. The cylinder is mounted in the bottom of the oil bath with these bolts. A Garlock gasket serves to prevent oil leakage.

The piston assembly is mounted on the top flange of the cylinder by the bolts in the bottom flange of the piston assembly.

Figure 12 is a drawing of the spinneret and its supporting flange. The spinneret was made from aluminum and the flange is a modified, one-inch, 600-pound, blind flange. This assembly is mounted to the bottom of the spinning cylinder using the bolts which extend through the bottom of the oil bath.

When in operation, the piston assembly is held in place by a steel bar which extends across the top of the frame. The sprocket on the piston assembly is connected by a chain and another sprocket to a variable speed motor. The motor was mounted on the right side of the top level of the frame. Vertical displacement speed for the piston can be varied from zero to one inch per minute.

The fiber take-up drum and drive are mounted on the back frame of the spinning unit beneath the spinneret, two feet from the floor. This leaves a vertical distance of about five feet for the fiber to travel between the spinneret and drum. The drum is connected by a rubber belt and pulleys to a variable speed drive. Drum speed can be varied from zero to 670 meters per minute.

The following list describes some pieces of equipment that were used in building the spinning apparatus:

1. Agitator - with double blade rod. 115 volts. Purchased from Fisher Scientific Co., Chicago, Ill. Used to agitate oil bath.
2. Controller - Model SA12. Purchased from B&B Motor Control Corp., New York, N. Y. Used to control motor for piston drive.

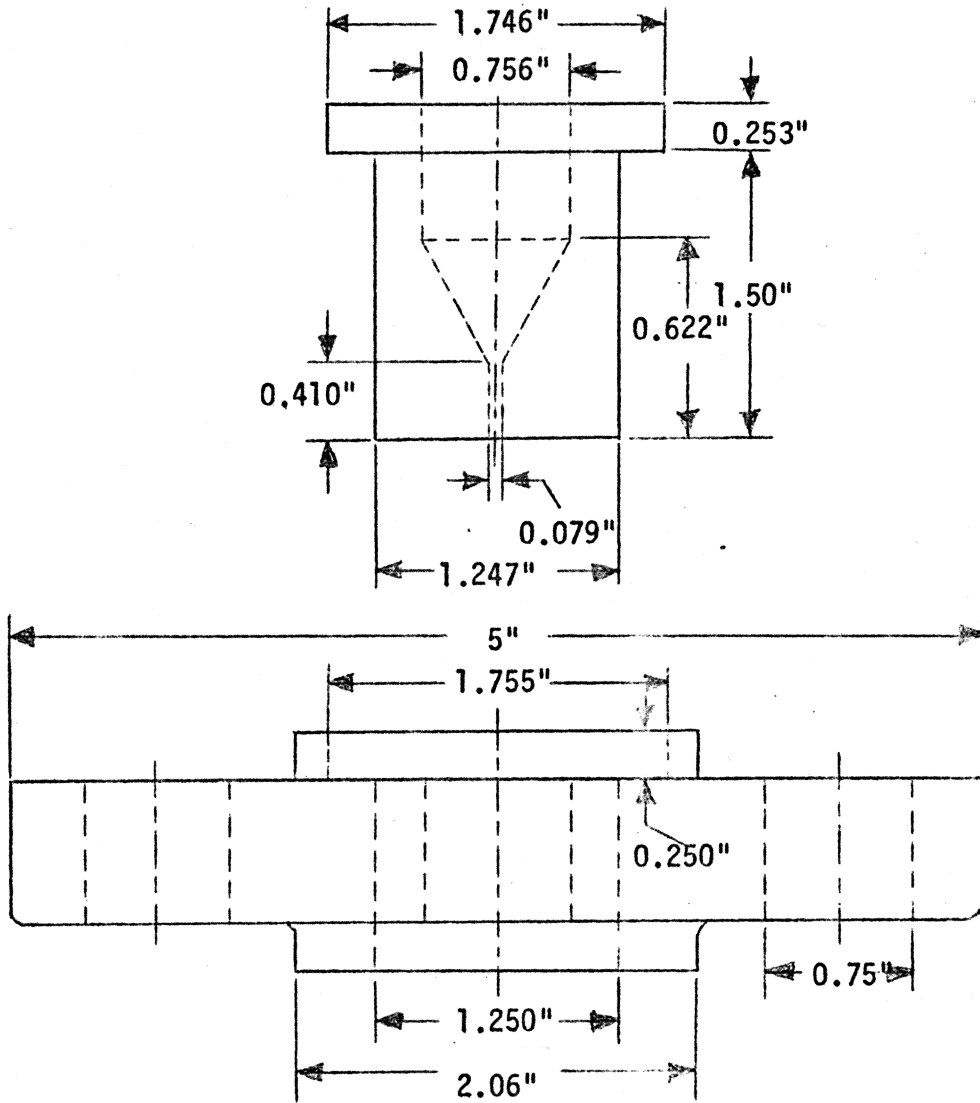


Figure 12. Actual Size Drawings of Spinneret and Assembly Flange

3. Drive - Zeromax Model M14R, Type A. Ten inch-pounds torque, 0-400 rpm. Manufactured by Zero-Max Co., Minneapolis, Minn. Used to drive take-up drum.

4. Drum-Steel. Ten inch diameter, six inch face, 7/8-inch bore. Cut down from 12-inch face pulley in shop. With shaft, two type SC ball bearings. Pulley and bearings purchased from Parker-Nimmo Supply Co., Inc., Salem, Virginia. Used to take up fiber.

5. Heaters - (3) Chromalox tubular heating elements. Round cross-section, steel sheath, type TRS, 0.315-inch diameter. All 120 volts. No. 3248-525 watts; No. 5248-950 watts; No. 7648-1450 watts. Purchased from Virginia Technical Associates, Richmond, Virginia. Used to heat oil bath.

6. Motor - Model B-2420C-40L. One-sixth horsepower, 73 inch-pounds torque, 2.6-52 rpm. Manufactured by and purchased from B&B Motor Control Corp., New York, N. Y. Used to drive piston in cylinder.

7. Pulleys - (2) Steel. One eight-inch and one three-inch diameter. Eight-inch pulley No. 800A, manufactured by Central Die Casting and Manufacturing Co., Chicago, Ill. Source of three-inch pulley unknown. Used to connect drum drive with drum.

8. Sprocket and chain - 1/4-inch pitch, steel. Sprocket No. 25B9, 9 teeth, 7/16-inch diameter. Chain No. 25, riveted. Manufactured by Browning Manufacturing Division, Emerson Electric Co., Maysville, Kentucky. Purchased from Power Transmission Corp., Roanoke, Virginia. Used in piston drive assembly.

9. Thermostat - Thermoswitch. Rating 10 amps - 115 volts. Range

-100 to 600°F. Contacts open on temperature rise. Controls to within one C°. Manufactured by Fenwal Inc., Ashland, Mass. Used to control temperature in oil bath.

Tachometer, Hand. Type 25A, No. T679. With Type 61A one-foot circumference disc. Range 0-10,000 fpm. Manufactured by Metron Instrument Co., Denver, Colorado. Used to measure speed of take-up drum.

Tensigraph. Model IP-2, No. C1703. Manufactured by Scott Testers, Inc., Chicago, Ill. Obtained from Allied Chemical Corp., New York, N. Y. Used to cold draw fibers and to measure tenacity, elongation, and secant modulus of fibers.

Thermometers. (2) Immersion type. Range 0-250°C. One C° divisions. Manufactured by Ace Glass Inc., Vineland, N. J. Used to measure temperatures of solution in resin kettle and of oil bath.

Variacs. (2) Powerstat voltage regulators, Type 3PN116, 0-140 volts output. Manufactured by Superior Electric Co., Inc., Bristol, Conn. Purchased from Fisher Scientific Co., Chicago, Ill. Used to control voltage to resin kettle heating mantle and spinneret heating cord.

Versatile Stirrer. 0-115 volts output. Purchased from Fisher Scientific Co., Chicago, Ill. Used to control speed of kettle agitator.

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PROPERTIES AND CHARACTERISTICS OF
POLYPROPYLENE FIBERS SPUN BY THE
PHASE-SEPARATION TECHNIQUE

by

Matthew Carl Williams

(ABSTRACT)

A spinning apparatus was built for the investigation of the phase-separation technique as applied to the production of polypropylene fibers from naphthalene solutions. The solutions were spun through a single 2.00 millimeter spinneret at compositions of 15 and 22 per cent polymer and at temperatures of 145 and 160°C. Linear velocities in the spinneret and draw ratios ranged from 0.125 to 0.277 meters per minute and from 759 to 2198, respectively.

It was found that naphthalene could be extracted from the fibers by diethyl ether in less than one minute; however, this extraction was not necessary prior to properties testing because all of the naphthalene in the fibers exposed to air was lost by sublimation.

Deniers of 14 selected fiber samples varied inversely with draw ratio and directly with solution composition in a range from 2.5 to 8.0. After the samples were cold-drawn 3:1, tests with a Scott Tensigraph showed that tenacities, per cent elongations at break, and secant moduli varied directly with draw ratio in ranges from 0.9 to 1.6 grams per denier, from 50 to 150 per cent, and from 17 to 30 grams per denier, respectively.

Per cent elongations at break also varied directly with solution composition.

The fibers, in general, exhibited good hand, bulk, tangling, and self-crimping characteristics. Photographs of four of the fiber samples were made to show the tangling and crimp qualities.