PHASE SEPARATION SPINNING OF POLYPROPYLENE FIBERS

by

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I. INTRODUCTION

The four most widely used processes for the manufacture of synthetic fibers are: wet spinning, melt spinning, dry spinning and gel-extrusion spinning. The manufacture of polyolefin fibers is done by melt spinning[7].

In 1967, a new process called phase-separation technique was explored. This process was meant to fit between gel spinning and wet spinning, and to be applied to polymers that cannot be melt spun without degradation[1]. A homogeneous solution of polymer and solvent is spun. But, as opposed to wet spinning, the solvent must have properties such that as the solution is spun into a fiber it will separate rapidly into a continuous phase consisting of pure polymer and a second phase consisting of pure solvent. This first study was exploratory in nature and, while many polymer-solvent combinations were successfully spun, there is little detailed information available about any specific polymer-solvent system.

The present investigation was limited to the polypropylene-naphthlene system and was conducted with two objectives. The first objective was to determine a range of polymer concentrations and solution temperatures where the phase separation technique was applicable. The second objective was to study the effect of the polymer concentration and spinning conditions on the structure and properties of the fibers.

II. LITERATURE REVIEW

Published information pertinent to the objectives of this investigation is summarized in this section.

Fiber Spinning Techniques

The four major processes for fiber spinning have been reviewed and described by Zwick[2].

The most economical of the spinning techniques is melt spinning. This process applies when a solid polymer can be liquified simply by heating it without suffering undue chemical decomposition, solidifies shortly after leaving the spinneret and the resulting fiber can be easily wound up. The winding speed varies from 800 to 1000 m/min or higher.

If the polymer does not melt without decomposition, it can be made by the addition of a suitable solvent. The polymer is ejected from the spinneret in thin streams into a coagulation bath where it reverts to solid state, this time in the shape of a fiber. Polymer concentrations usually vary from 5 to 25% and spinning speeds range from 50 to 100 m/min. Solvent losses and recovery add to the cost of the process. This method is called wet spinning.

In order to reduce the cost of solvent recovery and the amount of solvent needed, the dry spinning technique has been devised. This process, also called solvent-evaporation spinning, consists of forcing a polymer solution in the range of 20 to 30% through spinnerets into hot spinning shafts in which most of the solvent is

evaporated. Obviously, low boiling solvents are preferred. Temperature control problems are inherent to this method - rapid solvent recovery is desired, but overheating the fiber in the shaft leads to deformation of the cross section of the fiber.

If the boiling point of the solvent is such that dry spinning becomes difficult, the solids content of the solution can be increased. This leads to gel extrusion spinning for polymer concentration between 35 and 55%. At this solids content the solvent is better regarded as a plasticizer. The plasticized mass is extruded as in melt spinning and spinning speeds in excess of 500 m/min are possible.

Spinning of Fibers Undergoing Phase-Separation

This technique was meant to fit between gel spinning and wet spinning, to be applied to polymers that cannot be melt-spun without degradation, or to be used where gel spinning causes difficulties.

Theory. In an article published in 1967, M. M. Zwick introduced the idea of phase-separation. The idea was to start with a polymer solution in the wet spinning concentration range (10-25%) which can be made homogeneous at relatively low temperatures. The solvent or solvent mixture must be such, that in a temperature range below the polymer melting point, the solution would undergo phase-separation into a continuous polymer phase and a solvent or cosolvent phase. It can be

envisioned that a critical temperature exists below which the polymer separates from the solvent. When one solvent is used, this temperature lies between 50 and 160°C[3].

It must be noted that the resulting fiber, of high solids content, is a product of a phase separation process, rather than caused by solvent evaporation or precipitation[17].

Due probably to the wide variety of polymer-solvent systems studied, Zwick did not make a deep study of the effect of spinning conditions and of the process itself on the structure and properties of any one fiber.

In 1970-71, an independent study[18] was carried out by Williams and Fricke on the system polypropylene-naphthalene. Two polymer concentrations, 15 and 22%, were investigated at two different spinning temperatures, 145 and 160°C. Their conclusions as to the structure of the fiber were based on tensile strength data and on microscopic examination of the fiber surface. They deduced that the fibers made at those concentrations were probably porous. No quantitative measure of the void fraction was presented.

A brief review of the experimental procedures and results published follows.

Experimental

Precast rods. This procedure was reported by Zwick. In the beginning the polymer was dissolved in the molten solvent in a long glass tube and homogenized by stirring. Time was allowed for

trapped air to rise to the surface. The solution was allowed to cool slowly to room temperature and solidify. The glass tube was broken and the polymer-solvent stick was introduced into the spinning cylinder.

Direct Spinning. This procedure was used by both Zwick and Williams and Fricke[19]. In this case the solution was homogenized and charged into the spinning cylinder while in liquid state.

Spinning Temperature and Sample Collection. An agitated oil bath was provided to bring the solution to the desired spinning temperature. In both cases, a 30 - 45 min. period was allowed for the solution to reach thermal equilibrium inside the spinning cylinder before spinning was attempted. Once the spinnability of the solution had been satisfactorily proven, samples were collected in a winding unit located 1.5[4] to 2.5m[20] below the spinneret.

Spinning Operation Variables

For each spinning experiment the following variables must be fixed beforehand[5]

(a) Composition of spinning solution

(b) Number and size of holes of spinneret

The parameters to be fixed during the spinning operation are:

(c) Temperature of oil bath, reservoir or solution in spinning cylinder

(d) Velocity of the solution leaving the spinneret hole

(e) Take-up or winding speed

These additional variables were used by Zwick [6]:

(f) Composition of finishing agents

(g) Spinneret temperature

Fricke and Williams did not use finishing agents and the spinneret temperature was not controlled, only measured.

Some of the spinning conditions for experiments performed are presented in Table I.

Experimental Results and Properties of Commercial Polypropylene Fibers

Some of the published experimental results of the properties of polypropylene fibers made by the phase separation technique as well as those of fibers made by melt-spinning are presented in Table I. As can be seen the tenacities of the fibers reported by Williams and Fricke are very much lower than those reported by Zwick; however, while the latter optimized drawing conditions of the fibers, the former did not. The tenacities reported by Zwick compare favorably with those of commercial polypropylene fibers.

As an additional comment it must be said that no published information is available as to the stiffness or initial moduli of fibers made by the phase separation method. A range reported of 6 to 30 gr/denier compares favorably with the commercial fiber moduli which ranges between 5 and 20 gr/denier for melt spun polypropylene fibers.

Table I

Conditions for Polypropylene Fiber Production by Phase Separation Spinning and Melt Spinning, and Results of Some Property Tests on Fibers Produced

	Phase Separation Spinning				Melt Spinning
Polymer Concentration, %	17+	15+	22*	15*	100**
Solvents	Naphthalene	Naphthalene	Naphthalene	Naphthalene	--
	Paraffin wax	Paraffin Wax	--	--	--
Solvent mixture	50:50	50:50	--	--	--
No. Spinneret holes and diameter, mm	6 x 0.8	15 x 0.2	1 x 2.0	1 x 2.0	--
Reservoir temp., °C	175	175	160	145	> 360
Winding speed, m/min	400	300	490	530	800-1000
Velocity in spinneret holes, m/min	3.0	16.0	.56	.305	Gravity flow
Draw-down ratio	133	19	870	1740	--
Washing solvent	Petroleum ether	Pet. ether	Diethyl ether	Diethyl ether	--
Denier	6.4	2.5	6.9	2.2	6-35
Tenacity of Drawn Fibers gm/den	7++	6.4++	1.00	1.40	3-8
Draw	9/1	8/1	3/1	3/1	
Elongation at break, %	17	16	55	125	14-80

+Zwick, M.M.: Spinning of Fibers from Polymer Solutions Undergoing Phase Separation, Applied Polymer Symposia No. 6, p. 111 (1967).

*Williams, M.C.: Properties and Characteristics of Polypropylene Fibers Spun by the Phase Separation Technique, Master's of Science Thesis, VPI&SU Library, August 1972.

**Mark, Atlas and Cernia: "Man-Made Fibers, Science and Technology", Vol. 3, Guterscience Publishers, New York, N.Y., 1968.

Generally, it might be expected that a higher spinning temperature would yield a fiber with lower tenacity and higher elongation at break because of increased draw down before freezing. This expected result does not agree with the results obtained by Williams and Fricke[22] which show no effect of spinning temperature on tenacity. Fiber tenacity should increase and elongation at break decrease for increasing draw ratio. Published results do not agree with this expectation, showing that tenacity as well as elongation at break increase with increasing spinning draw ratio. There is no information published with respect to the effect of the spinning variables on the porosity of fibers spun by phase separation technique.

Some of the most interesting properties of polypropylene fibers spun by phase separation can be described only qualitatively. Immediately after spinning and before extraction, the fibers are weak and brittle with bright surfaces and little tendency to tangle or coil[21].

After the naphthalene is extracted, the fibers become dull, tough, possess a helical crimp in most cases, and tangle very easily. The crimp amounts to as much as 20 turns per inch for the low denier fibers.

The properties of the fibers after extraction of the separated solvent are very much different from those of polypropylene melt spun fibers. Melt spun fibers are straight, with very smooth surfaces and do not tangle with ease.

Advantages and Disadvantages of the Phase Separation As Opposed to the Melt Spinning Method for Polypropylene Fibers

One of the primary advantages of the phase-separation process is that spinning can be favorably carried out at temperatures below the melting point of polypropylene (167°C), while melt spinning requires extrusion temperature around 100°C higher than the melting point[8].

The addition of solvent to lower the spinning temperature involves at the same time the disadvantage that a washing step must be added to the process.

The denier for melt-spun fiber varies between 35 to 100 denier, with corresponding diameters of 70 to 380 microns[9]. Fibers made by phase separation are well below both ranges, exhibiting deniers between .9 and 8[24] after extraction and diameters around 25 microns. In addition, large spinneret holes can be used to obtain small denier and diameter fibers. The use of large spinnerets would help prevent spinneret clogging.

Fiber Testing

This section does not attempt to review all the procedures for fiber testing, but to describe a procedure similar to the one used in this investigation.

The tensile strength data reported for fibers made by phase separation technique has been obtained using a Tensilgraph[25].

In this investigation an Instron Tensile Tester was used. Thus the following review is concerned with information published for testing at constant crosshead speed, in which mode the Instron was operated.

Variables. In order to obtain maximum accuracy in determining stress-strain relationships, the following factors must be known (1) Length of fibers; (2) crosshead speed; (3) chart speed; (4) Full scale load; and (5) gauge length.

Item (1) is particularly important when single fibers are tested. It is an independent variable and is controlled largely by the particular sample to be tested. The longer the fiber, the less influence the clamp errors will have. However, the distribution of flaws along the fiber length introduces a greater probability of the sample length containing a large flaw. Hence, fiber tenacity decreases as gauge length is increased. General experience shows that for most commercial fibers, lengths of 3-7 in. do not show a significant tenacity-length relationship[11]. When a bundle of fibers or a number of fibers arrayed parallel to one another are tested the fiber length is not significant and most of the accuracy depends on how well item (5), gauge length, is measured.

The crosshead speed is a dependent variable selected to give the desired rate of extension. The standard of the textile industry is a rate of extension of 60%/min. This is a compromise between the limiting speed of the testing machine recorder control to detect

small changes in stress with time and the desirability of obtaining data in a short period of time[12].

An increase of straining rate is expected to increase both the tenacity and the moduli of the fibers tested. No quantitative measure or relationship between rate of extension and tenacity or moduli has been found for polypropylene fibers. However, some information is available for polyethylene fibers[13]. The reported results indicate that, for strain rates of 200%/min. or greater, all ductility is lost in the fiber while the tenacity improves very little.

The scale load and chart speed are factors which add to the ease of measure of the experimental results. A low chart speed will make moduli measurements extremely inaccurate. It is thus recommended[14] that the chart speed be such that the initial slope corresponds to a 45° angle. The full scale load should be adjusted so that the maximum stress is recorded at a point 50-85% of the value selected.

General Interpretation of Stress-Strain Behavior

In the low regions of strain, often referred to as the initial modulus region, the fiber exhibits high resistance to stretch due to the interference of molecular chains, as well as intermolecular secondary bonding. This resistance to deformation by the fiber relates to the hand, "crispness", or drape of fabrics made from this fiber, since these properties are observed at relatively minor deformations of the fibers[15]. At a higher level of stress, individual molecules acquire sufficient potential energy to slip by one another. This is called

"softening", and allows considerable fiber deformation for relatively small increases in stress. Depending on the crystallinity of the fiber, the process of translation of chain segments in respect to one another continues until the fibers break or, for lower crystallinity fibers, a reinforcement occurs at a higher level of extension[16].

III. EXPERIMENTAL

Plan of Experimentation

Solubility Studies. Tests were conducted to determine the minimum solubility temperature of polypropylene-naphthalene mixtures as a function of concentration. The range of polypropylene concentration studied was 5-80 wt. % polymer.

Equipment. The spinning apparatus designed by Williams and Fricke[26] and built in the Chemical Engineering shop of Virginia Polytechnic Institute and State University was used.

Solutions of various polymer compositions were spun at different temperatures to test the equipment and to determine feasible spinning conditions as well.

Fiber Spinning and Sample Collection. Tests were conducted to determine the feasible regions of temperature and concentration in which fiber were to be made.

A rotatable, split level, factorial experimental design was used to determine the points at which sample were collected. However, enough extra points were obtained such that a parametric analysis was also feasible. The statistical analysis of results was not performed due to the scattering of the data.

To collect a sample, the steel take-up drum was covered by sheets of Kraft cardboard in such a way that no slipping occurred between the cardboard sheet and the take-up drum. Once an appropriate amount of fiber was collected the cardboard drum was removed, another

was inserted in place and a new sample was obtained at different conditions.

Extraction Studies. Available data[21] showed that extraction of the naphthalene with diethyl ether was necessary. The midpoint in the range of polypropylene concentration was tested for the rate of extraction. All other samples were washed in diethyl ether for an appropriate length of time (greater than five minutes) and dried in air before testing.

Fiber Properties

Physical. Scanning electron microscope pictures were taken of the surface of the fiber samples. Fiber diameter and crossectional area (calculated) were determined by use of a filometer in an optical microscope. Denier of the individual fibers was obtained by measuring the length and weight, after extraction, of bundles of fibers spun at the same conditions, and counting the number of fibers in the bundle.

The void fraction of the fiber was calculated from the denier and diameter data already available, and a density value for pure polymer obtained from published literature.

Tensile. Measurements of fiber tenacity, initial modulus and percent elongation at break were obtained by means of an Instron Tensile Tester, with an attached recorder. The fibers were tested in bundles, with the fibers parallel to each other. Samples of all the fibers were tested after a 4 to 1 draw, at a constant initial rate of strain of 100%/min.

The drawing conditions of the sample corresponding to the mid-range of polymer concentration, spinning temperature, and draw ratio used in this investigation were varied. In doing so, the effects of draw and rate of strain on the tensile properties were investigated.

Procedures

Solution Preparation. Appropriate amounts of naphthalene and polypropylene were weighed. The naphthalene was introduced into a resin kettle which was covered by a heating mantle. Upon heating, the naphthalene liquified and the polymer was added slowly while the solution was stirred. As the polymer was added the temperature of the solution was increased by increasing the voltage to the heating mantle. After all the polymer had been added, the mixture was stirred until the polymer was in solution.

The temperature of the solution was increased prior to charging to prevent it from freezing inside the kettle.

Charging of Solution. The piston, as well as the inside of the spinning cylinder were thoroughly cleaned with tetralin. The solution, contained in the resin kettle, was charged into the cylinder so that it filled approximately three-quarters of the available volume.

This corresponds roughly to the level of the oil in the constant temperature bath surrounding the spinning cylinder. The piston assembly was then bolted to the spinning cylinder, connected

to the motor and forced downwards at maximum speed until it touched the solution. The point of contact was noticed by the high tension in the chain connecting the piston assembly to the motor.

Start-up Procedure. The heating assembly, consisting of three separate heaters, as well as the agitator assembly, was immersed in the oil and bolted to the frame of the bath.

The agitator was turned on and all heaters were turned to maximum output. A glass immersion thermometer was placed close to the spinning cylinder to determine the temperature of the oil bath. A heating tape was wrapped around the spinneret block.

At a temperature one degree lower than the desired temperature, the 1450 watt start-up heater was turned off while the remaining two heaters were kept at maximum output until the desired temperature was reached. At this point, the voltage output to both heaters was reduced to 100 volts and the temperature observed. The heating tape was turned on and the spinneret hole, .040 inches in diameter, was plugged with a piece of wire of approximately the same diameter.

Temperature Control. If the voltage applied to the heaters was not high enough it was increased by five volts on each heater. The start-up heater was used to bring the temperature to the desired point and then turned off. Again the temperature was observed and the same procedure was repeated if the temperature decreased. A five minute wait was necessary to determine fluctuations of temperature.

If the applied voltage was too high, all heaters were turned off and as soon as the temperature reached the right point, they were turned on with a voltage input five volts lower than the last setting. This trial and error procedure continued until the voltage settings of the two smaller heating units were such that the desired temperature was constant.

For temperature changes between runs, the same basic procedure was followed. If a lower temperature was desired all heaters were turned off, if a higher temperature was to be used, all heaters were turned on at maximum output. Upon reaching the right spinning temperature the procedure described above was used for control purposes.

Between 45 and 60 minutes were allowed for the solution to melt and reach thermal equilibrium for the first run.

When the spinning temperature was changed in subsequent runs, a 30 minute waiting period was used before spinning was attempted.

Spinning and Sample Collection

Once thermal equilibrium had been reached, the plugging wire was removed from the spinneret hole and the motor driven piston was turned on and allowed to travel downward at maximum speed. In all cases, the solution flowed smoothly through the spinneret. This procedure was repeated for every temperature change.

The motor speed was regulated until the velocity of the motor shaft was the one desired (in this experiment 64 sec/rev. was used).

This speed was checked before any sample was collected throughout the experiment.

The take-up drum was turned on and the speed was adjusted through a variable drive motor, then measured by a hand tachometer, until the desired speed was obtained. The spinneret temperature was recorded. A cardboard drum was inserted around the steel take-up drum, and the motor turned on again.

As the solution was forced through the spinneret, a fiber was formed which was picked up and wound rapidly by hand around the take-up drum until take-up began.

The fiber was guided by hand over the take-up drum to avoid build-up over any particular area of the drum. After enough fiber had been collected (about 500-1000 m.); the fiber was broken close to the spinneret hole and the take-up motor turned off. The cardboard drum was removed, another inserted in its place and the take-up speed changed. Again, the spinneret temperature was recorded and the fiber picked up and wound around the drum.

The same procedure was repeated until all desired take-up speeds at that temperature had been obtained.

Shut-down Procedure

Once all the desired samples were collected, the heating tape was turned off and the spinneret block was unbolted from the spinning cylinder. The piston drive was turned on at maximum speed and the remainder of the solution was forced out of the spinning cylinder.

The piston drive was reversed and the piston was allowed to travel upwards at maximum speed until it reached its maximum height. The heaters and agitator were turned off, disconnected, and removed from the oil bath. The piston assembly was disengaged from the motor, unbolted and removed from the spinning cylinder.

The spinneret as well as the piston were cleaned with tetralin and then left to soak overnight in the same solvent.

Results

The results of the solubility studies, shown in Figure 1, confirm the expected result, that the addition of solvent lowers the melting point of the solution.

The results of the extraction studies, presented in Figure 2 and Table II, show that all the naphthalene present can be removed from the sample fibers in less than five minutes and that more than 90% of it is removed in less than 15 seconds.

The initial denier, diameter and void fraction of the fibers are given in Tables III & IV, Figures 3 and 6 show denier and fiber diameter as functions of draw ratio.

The tensile properties of the fibers are given in Tables V-VI, Figures 8, 9 and 11-14 show tenacity, percent elongation at break and modulus as functions of draw ratio.

The effect of testing conditions on tensile properties is shown in Table VII, Figures 15 and 16 present the same properties as functions of draw and rate of extension, respectively.

IV. DISCUSSION

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

Procedures and Equipment

For concentrations of polypropylene below 35 wt. %, the preparation of the spinning solutions was easily accomplished. Solutions of higher polymer contents, however, were very difficult to prepare. As the polymer concentration was increased the solutions gained rapidly in viscosity, thus the electric stirrer used to mix the less concentrated solutions was useless. It was therefore necessary to use manual agitation and higher temperatures to obtain a solution.

The main difficulty encountered in this part of the operation, however, came in charging the concentrated solutions in the spinning cylinder. Due to the high viscosities, the solutions flowed slowly from the resin kettle into the spinning cylinder. If the temperature of the solution was relatively low (about 20°C above the solubility temperature), most of the solution solidified inside the kettle and all of the procedure had to be repeated. It was found that for solutions of higher polymer concentrations (above 40 wt. %) the temperature of the solution before charging should be between 30 and 40°C above the minimum solubility temperature.

The use of a heating tape solved a difficulty mentioned by Williams[28] that when the hot solution was poured into the cool cylinder

some phase separation occurred at the bottom of the cylinder. Thus, as the solution was heated the naphthalene first melted, leaving an amount of highly concentrated polymer solution, which could not be easily forced through the spinneret hole by the piston.

By means of the heating tape, the concentrated polymer phase at the bottom of the cylinder, was kept molten and could be forced through the spinneret easily by the piston.

The spinning apparatus worked well throughout this study. However, if higher spinning speeds were desired, the present limit on take-up speed of 600 m/min would be insufficient.

The use of cardboard drums to pick up the fibers was very valuable in two ways. First, it saved time during the collection of the fibers and, second, it allowed the fiber samples to be stored under constant tension until their properties were measured. The main inconvenience experienced during the collection of the fiber samples was that of having to guide the fiber manually over the take-up drum.

Extraction Studies

This study, presented in Figure 2 and Table II confirms the published results[27] that for the polypropylene-naphthalene system, the spinning solvent can be easily removed by diethyl ether. As an average, over 90 percent of the naphthalene present in the fiber was removed after 10 seconds of extraction time in diethyl ether, and almost all of the naphthalene in less than five minutes.

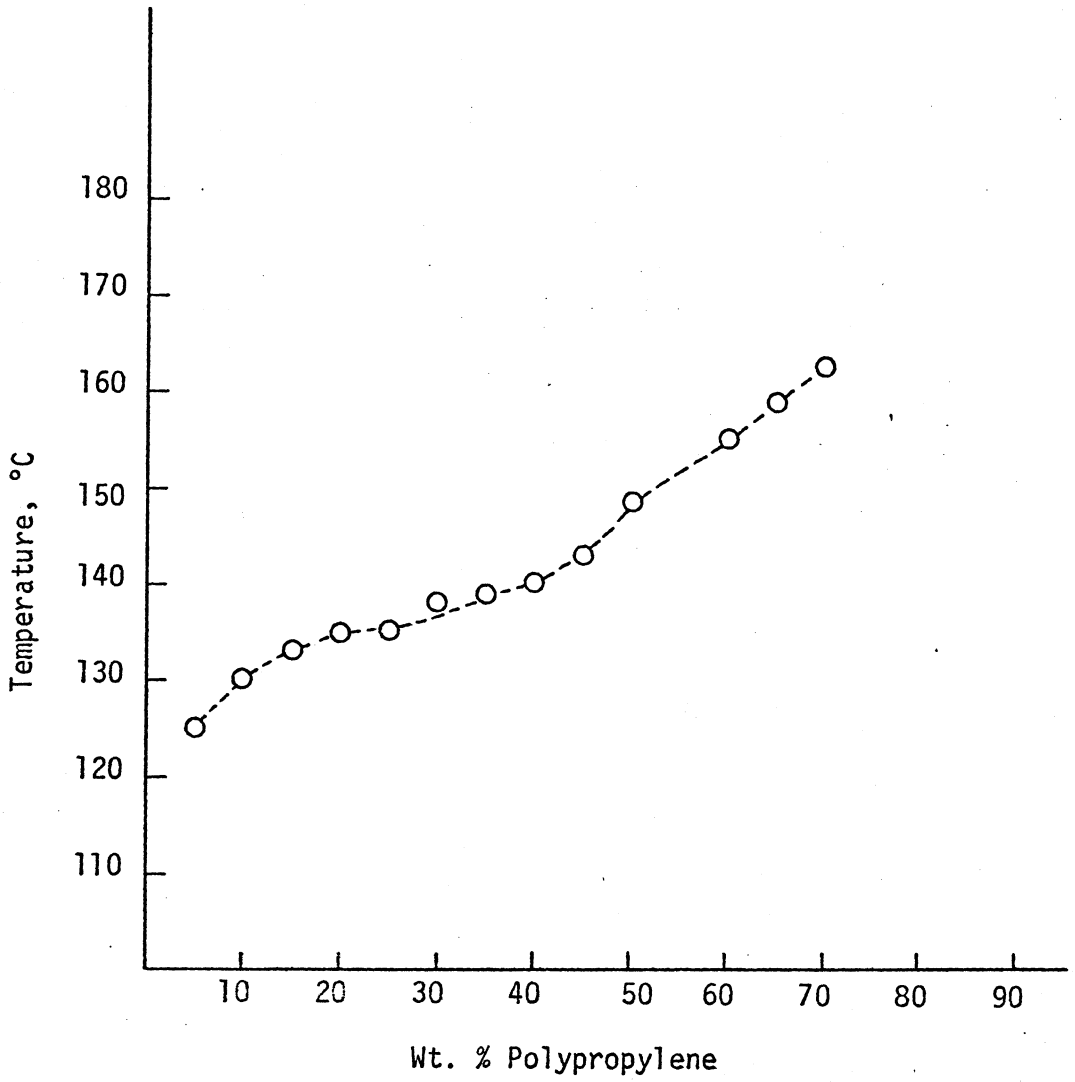

Figure 1 Solubility Curve for the System Polypropylene-Naphthalene

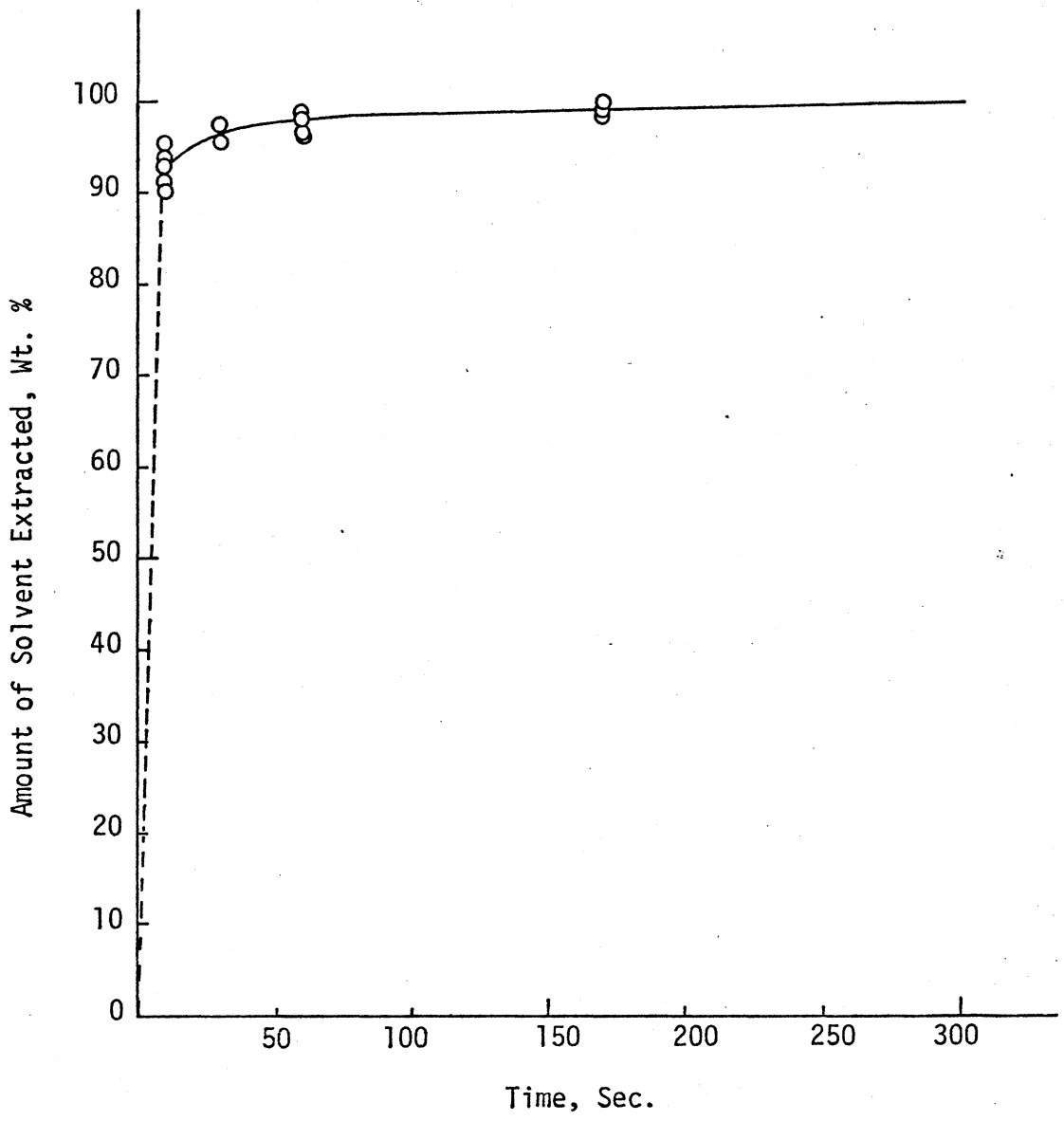

Figure 2 Extraction Rate of Naphthalene by Diethyl Ether, From Polypropylene Fibers

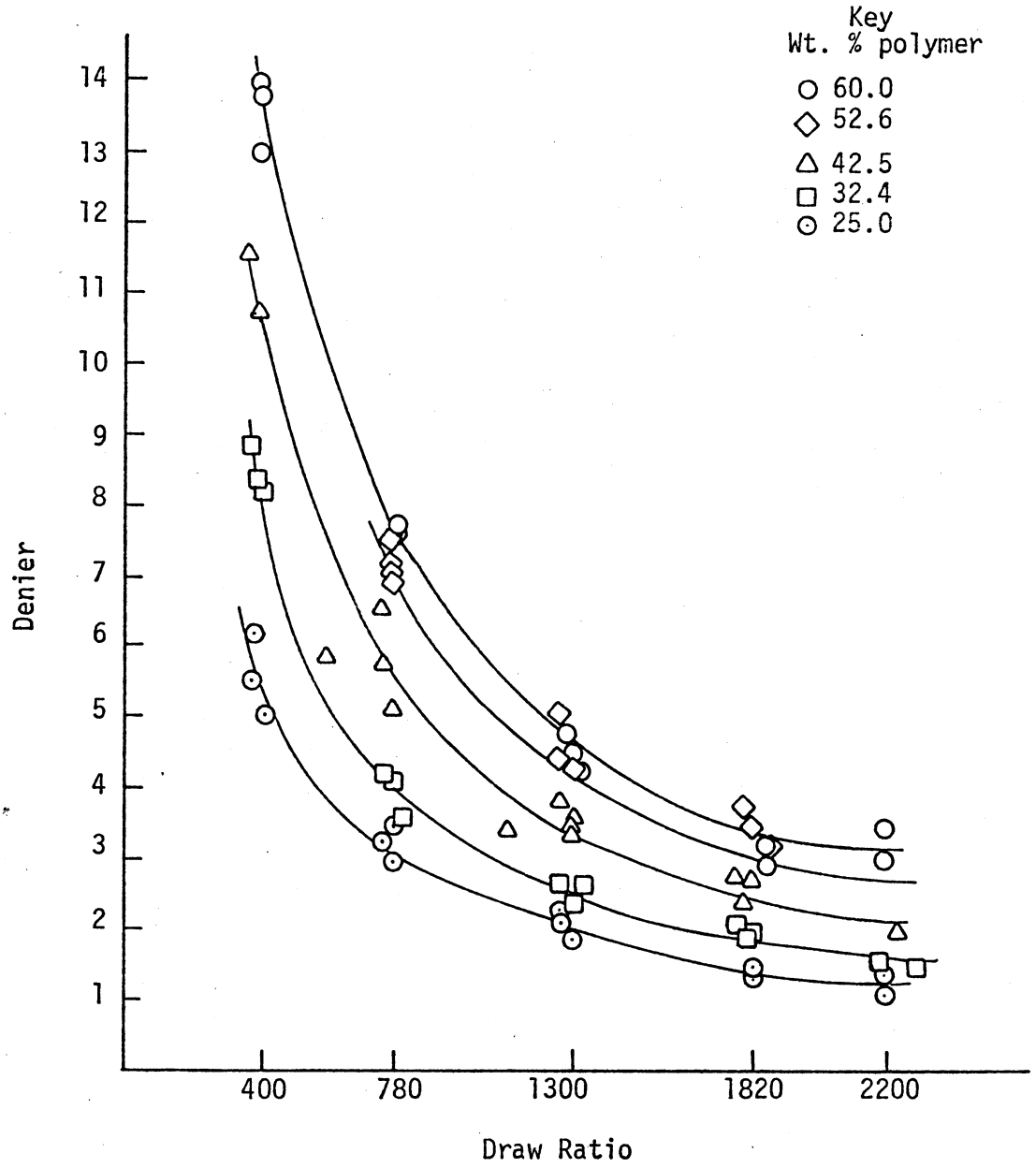

Figure 3 Effect of Spinning Draw Ratio on Initial Denier of Fiber

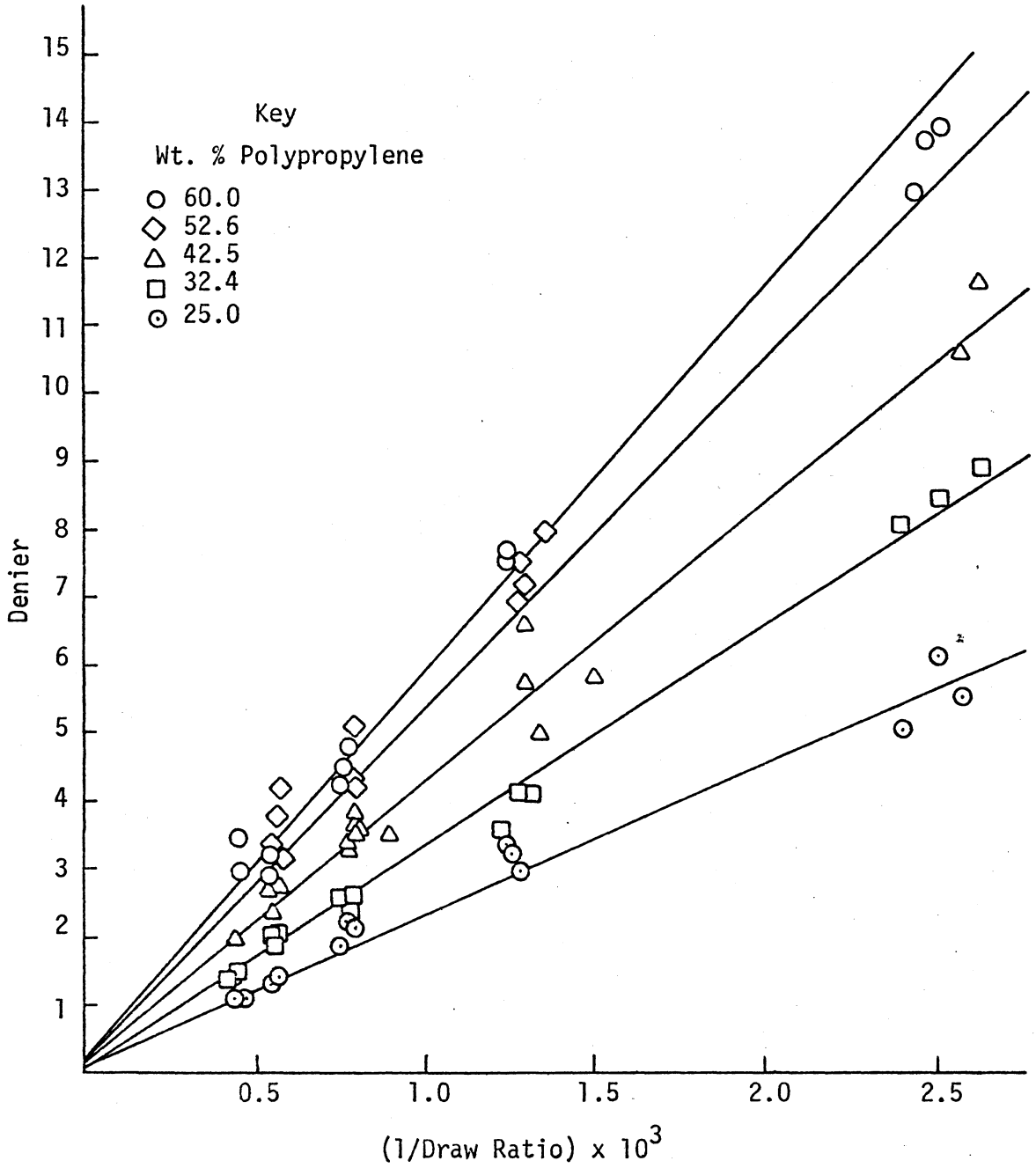

Figure 4 Graphical Test of the Relationship Between Denier of Fiber and Inverse Spinning Draw Ratio

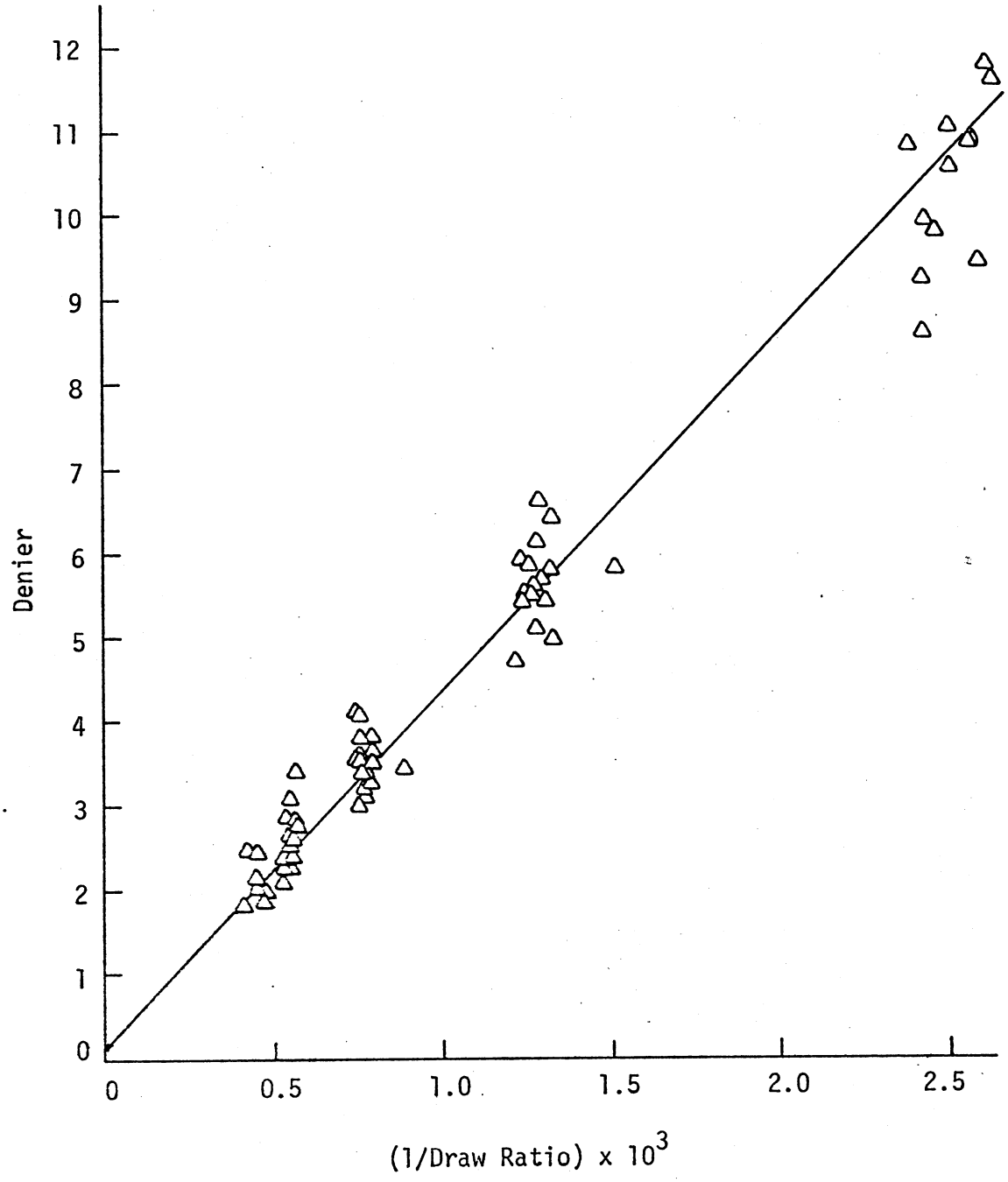

Figure 5 Graphical Test of the Relationship Between Concentration of the Spinning Solution and Denier

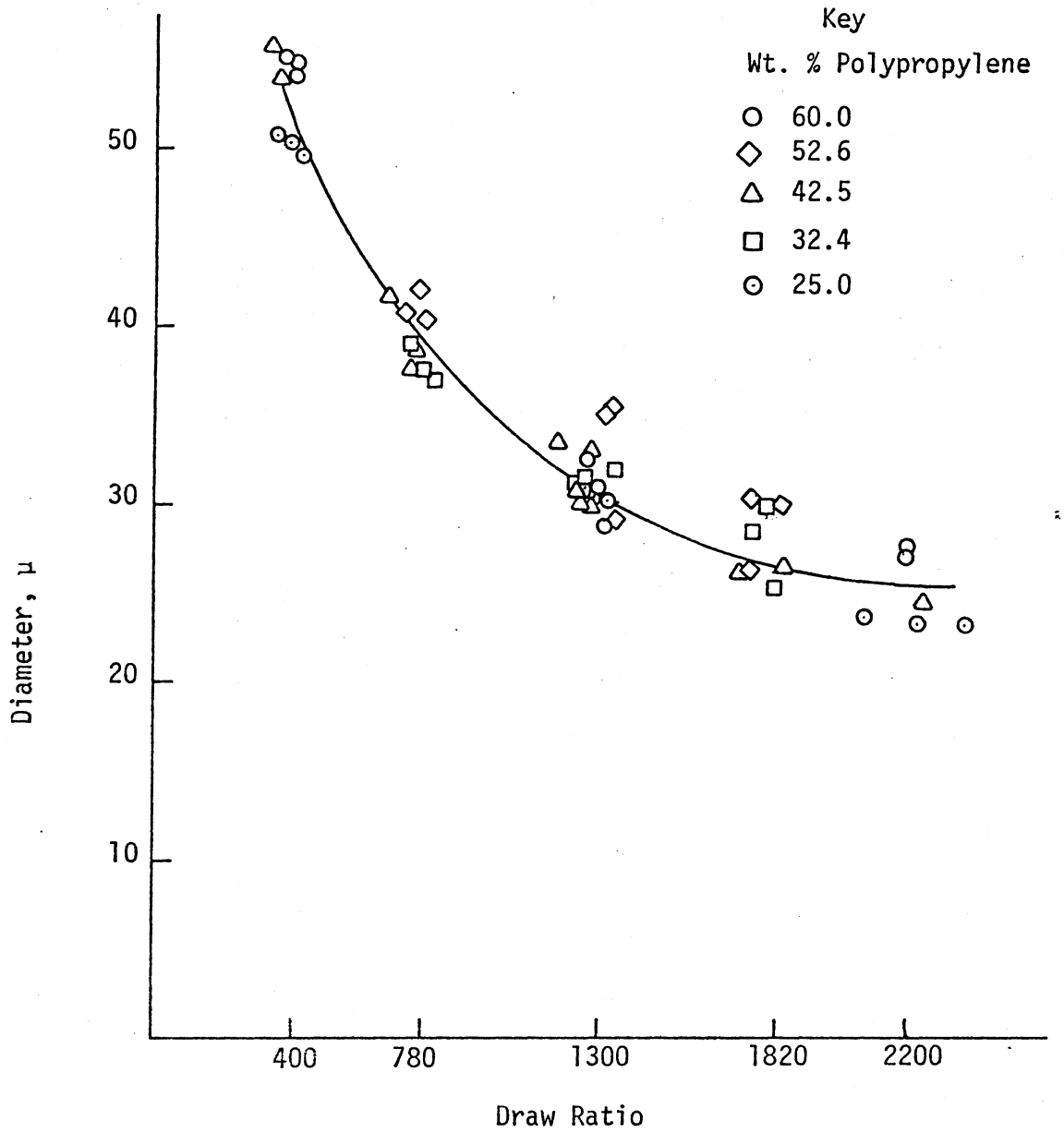

Figure 6 Effect of Spinning Draw Ratio on Fiber Diameter

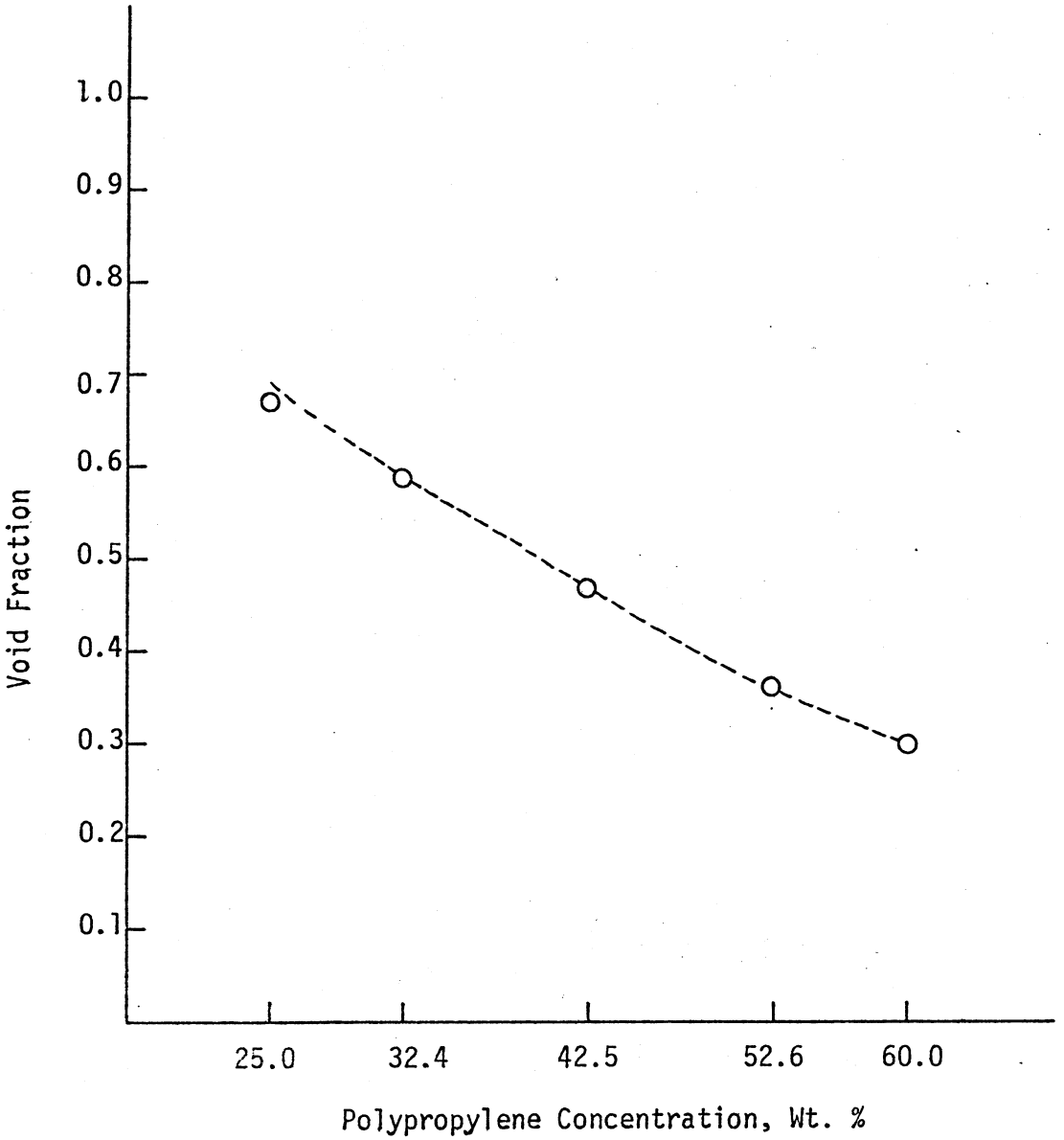

Figure 7 Effect of Concentration on Fiber Void Fraction
Fibers Spun 10°C Above Minimum Solubility Temperature
at Draw Ratio ≃ 1300. This curve is representative
of the trend, not of the exact relationship.

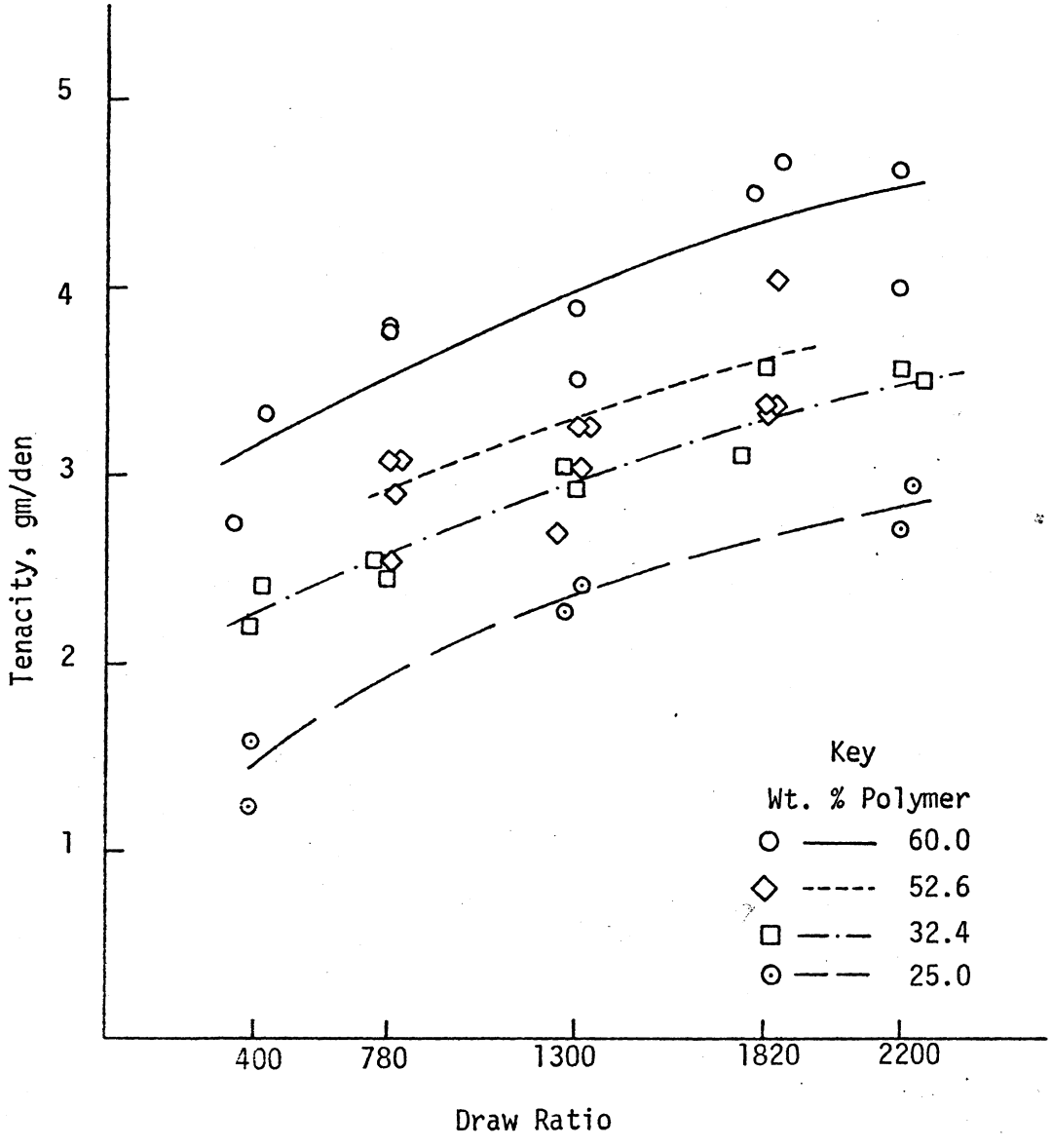

Figure 8 Effect of Spinning Draw Ratio on Fiber Tenacity (Fibers Cold Drawn 4/1. Initial Rate of Extension, 100%/min.)

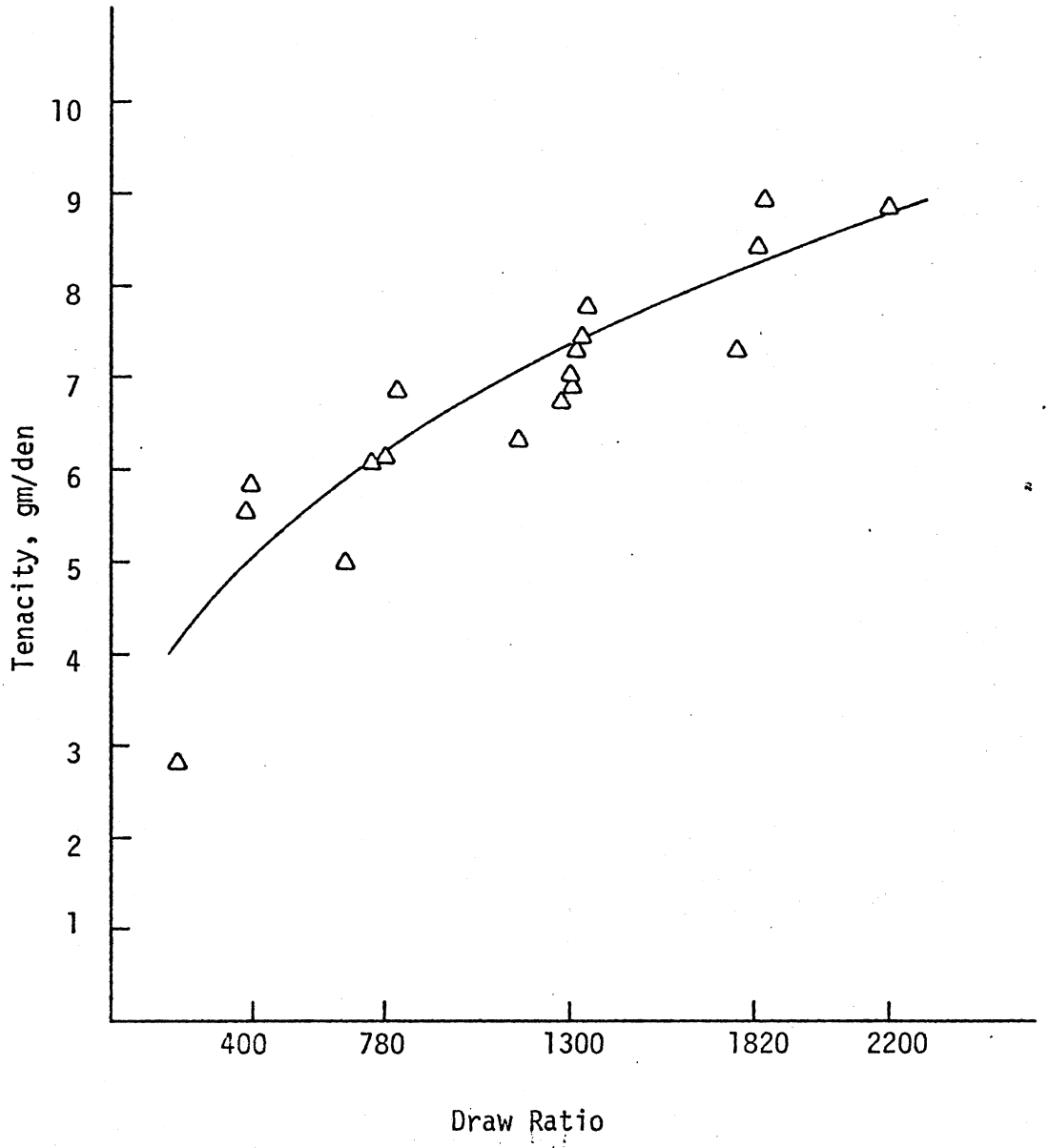

Figure 9 Effect of Spinning Draw Ratio on Tenacity of Fibers Spun from a 42.5 wt. % Polymer Solution. (Fibers Cold Drawn 4/1, Initial Rate of Extension 100%/min.)

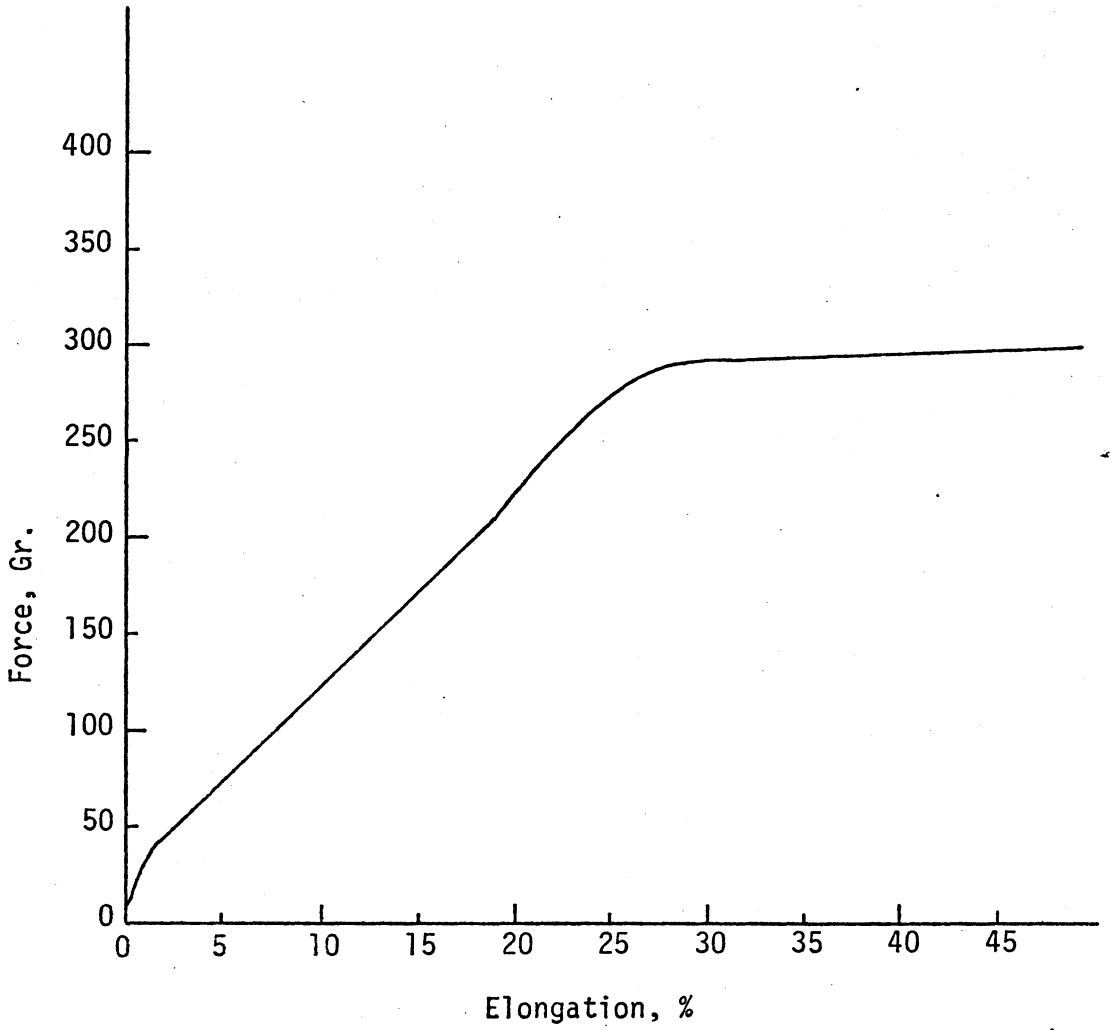

Figure 10 Typical Curve Used to Analyze Stress-Strain Behavior. (Fibers Cold Drawn 4/1, Initial Rate of Extension 100%/min.). This Curve is Representative of the Behavior of All the Fiber Samples Analyzed.

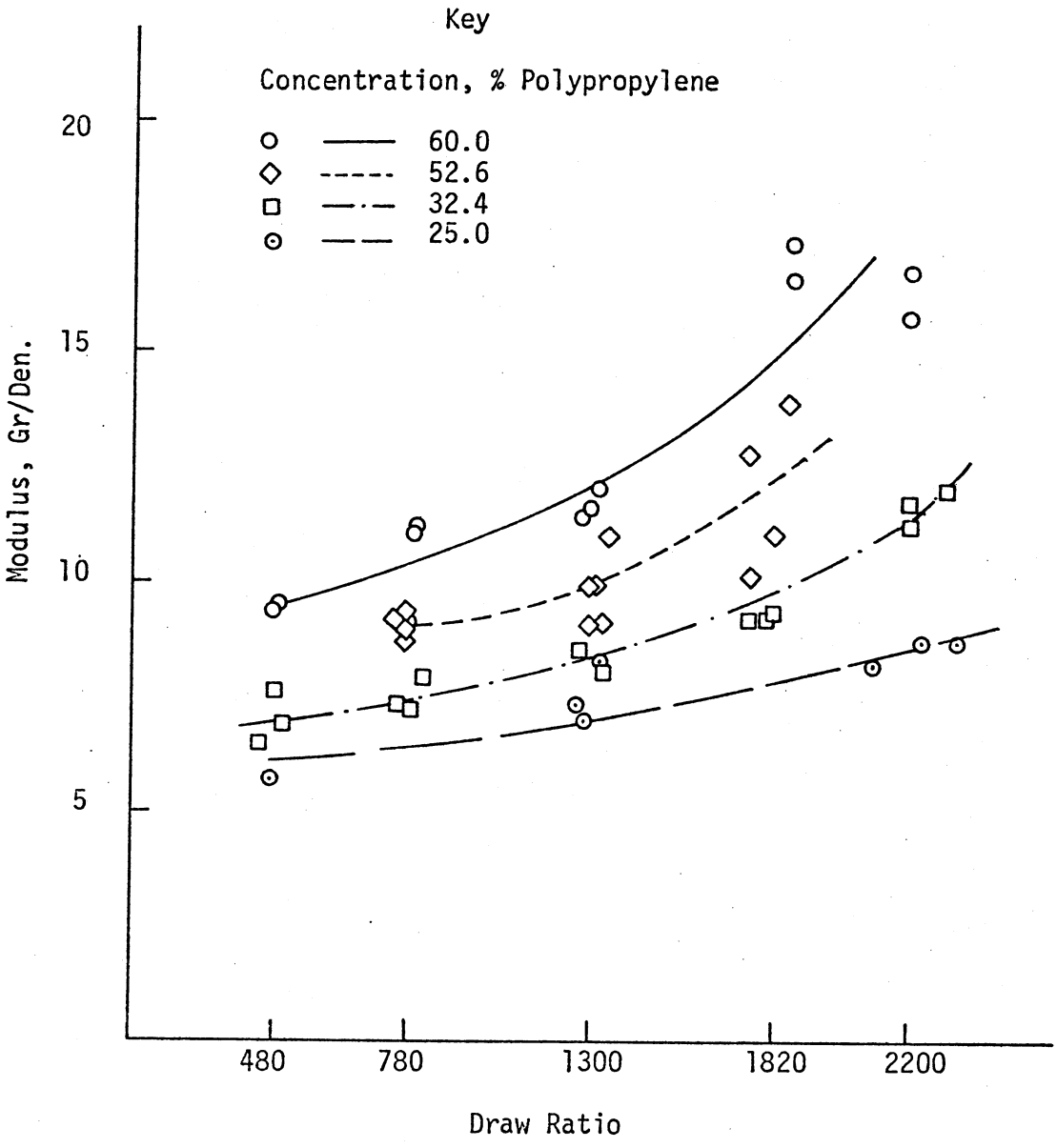

Figure 11 Effect of Spinning Draw Ratio on the Initial Modulus of Fibers (Fibers Cold Drawn 4/1, at Initial Rate of Extension 100%/min.)

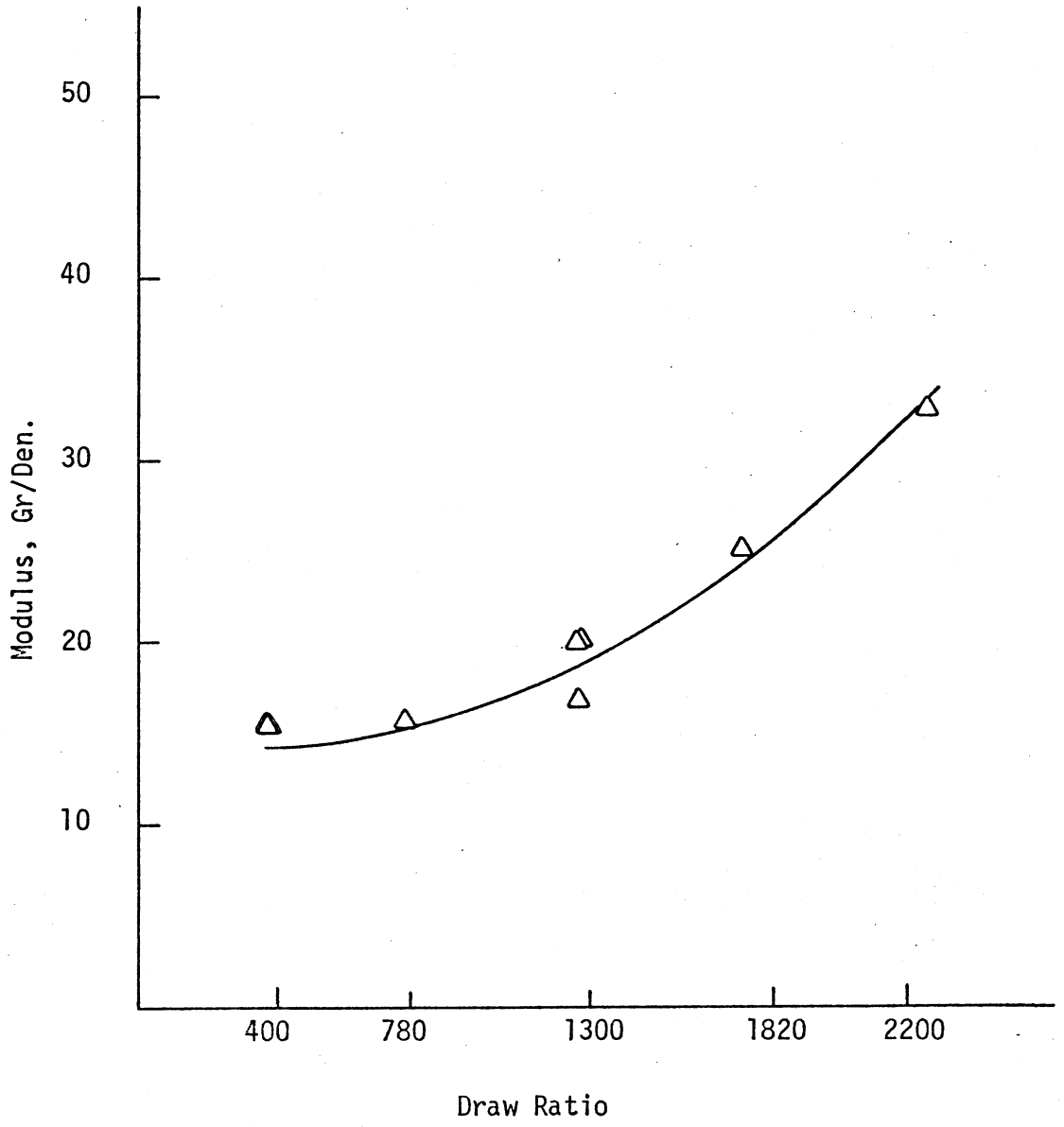

Figure 12 Effect of Spinning Draw Ratio on Initial Modulus of Fibers Spun from a 42.5 Weight Percent Polypropylene Solution, 10°C above the Melting Point of the Solution (Fibers Cold Drawn 4/1 at Constant Rate of Extension 100%/min). This Curve is Representative of the Behavior of Fibers Spun from the Same Solution

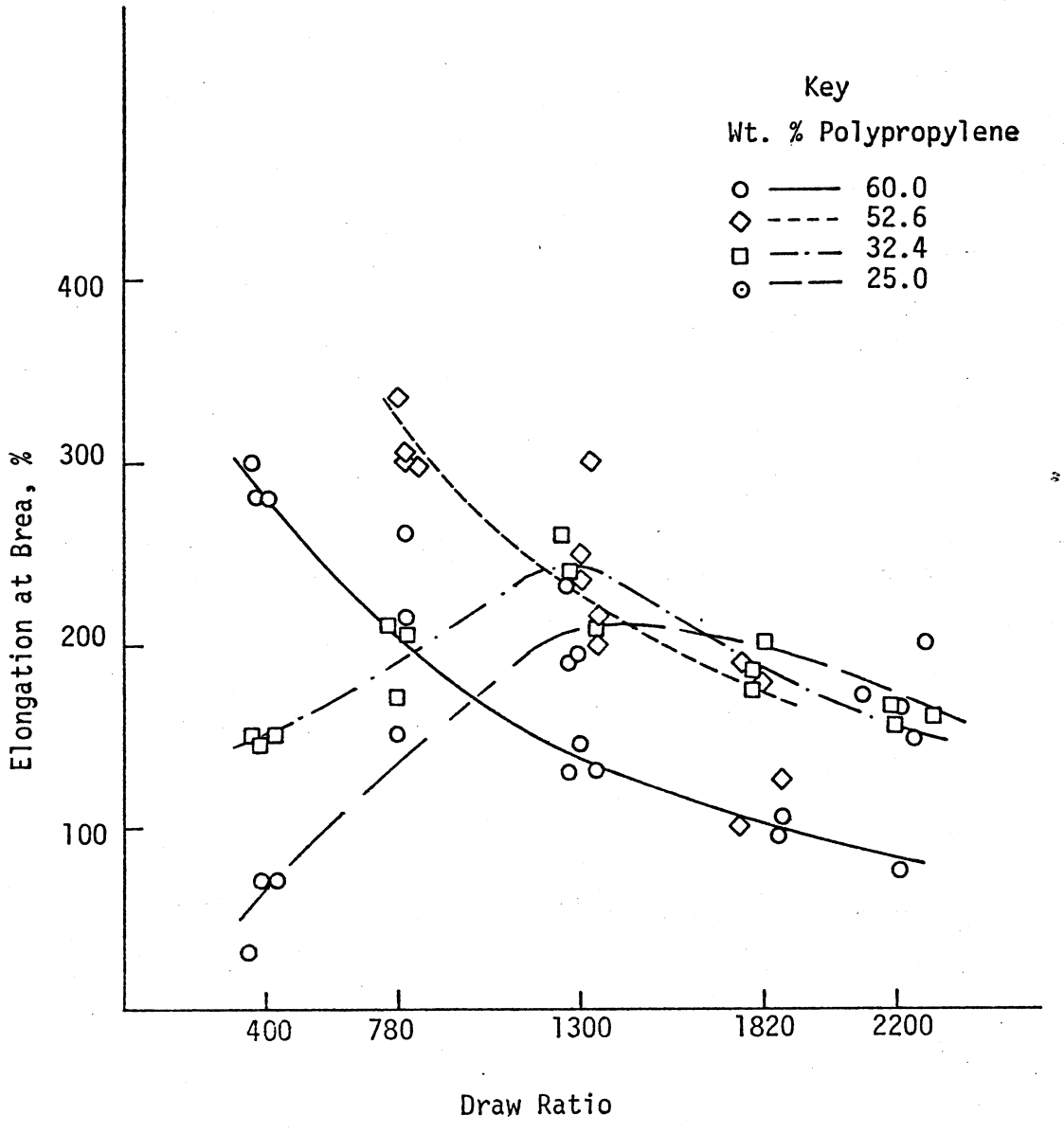

Figure 13 Effect of Spinning Draw Ratio on Elongation at Break of the Fiber (Fibers Cold Drawn 4/1 at Initial Rate of Extension 100%/min.)

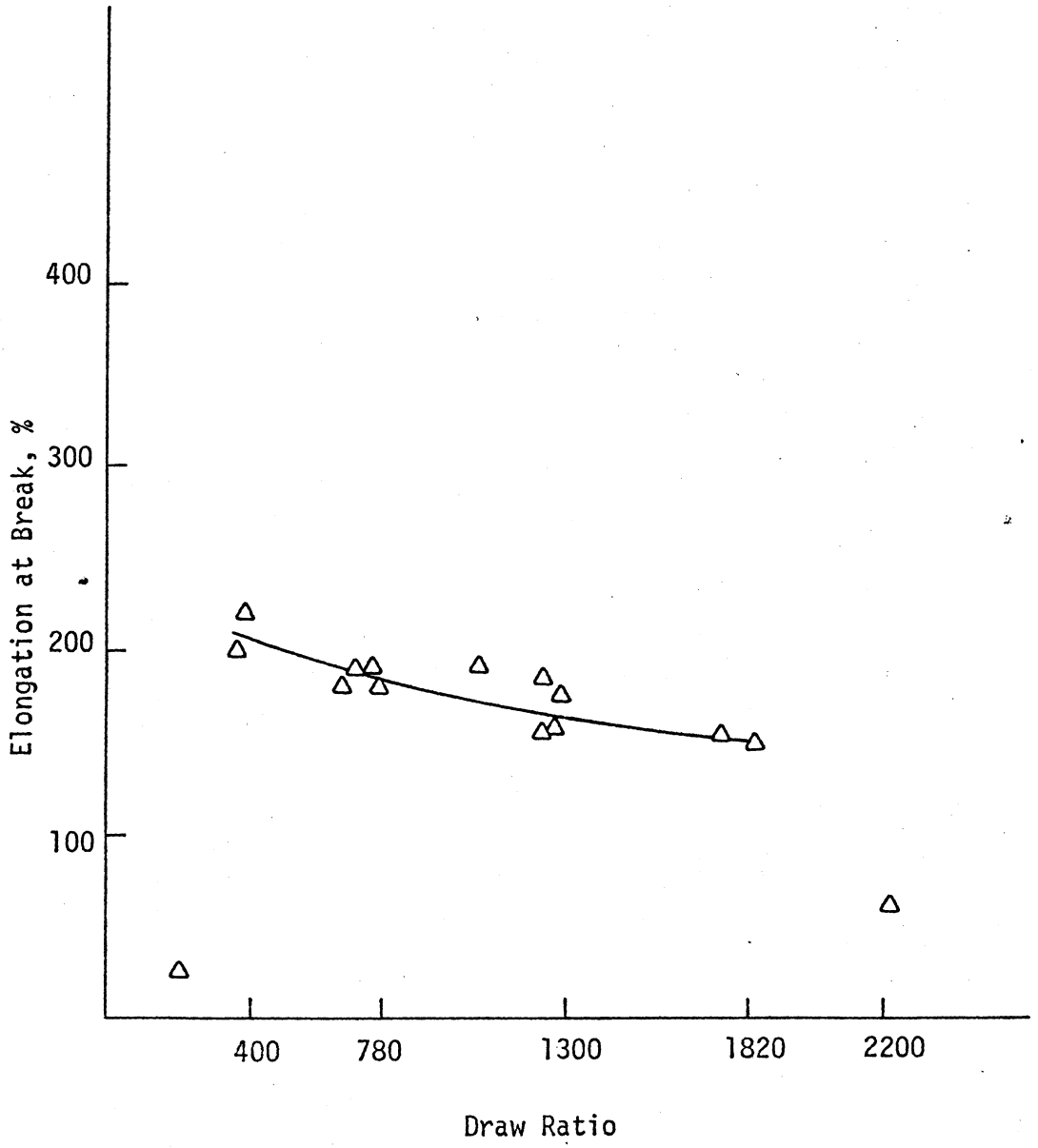

Figure 14 Effect of Spinning Draw Ratio on the Elongation at Break of Fibers Spun from a 42.5 Weight Percent Polypropylene Solution. (Fibers Cold Drawn 4/1 at Initial Rate of Extension 100%/min.)

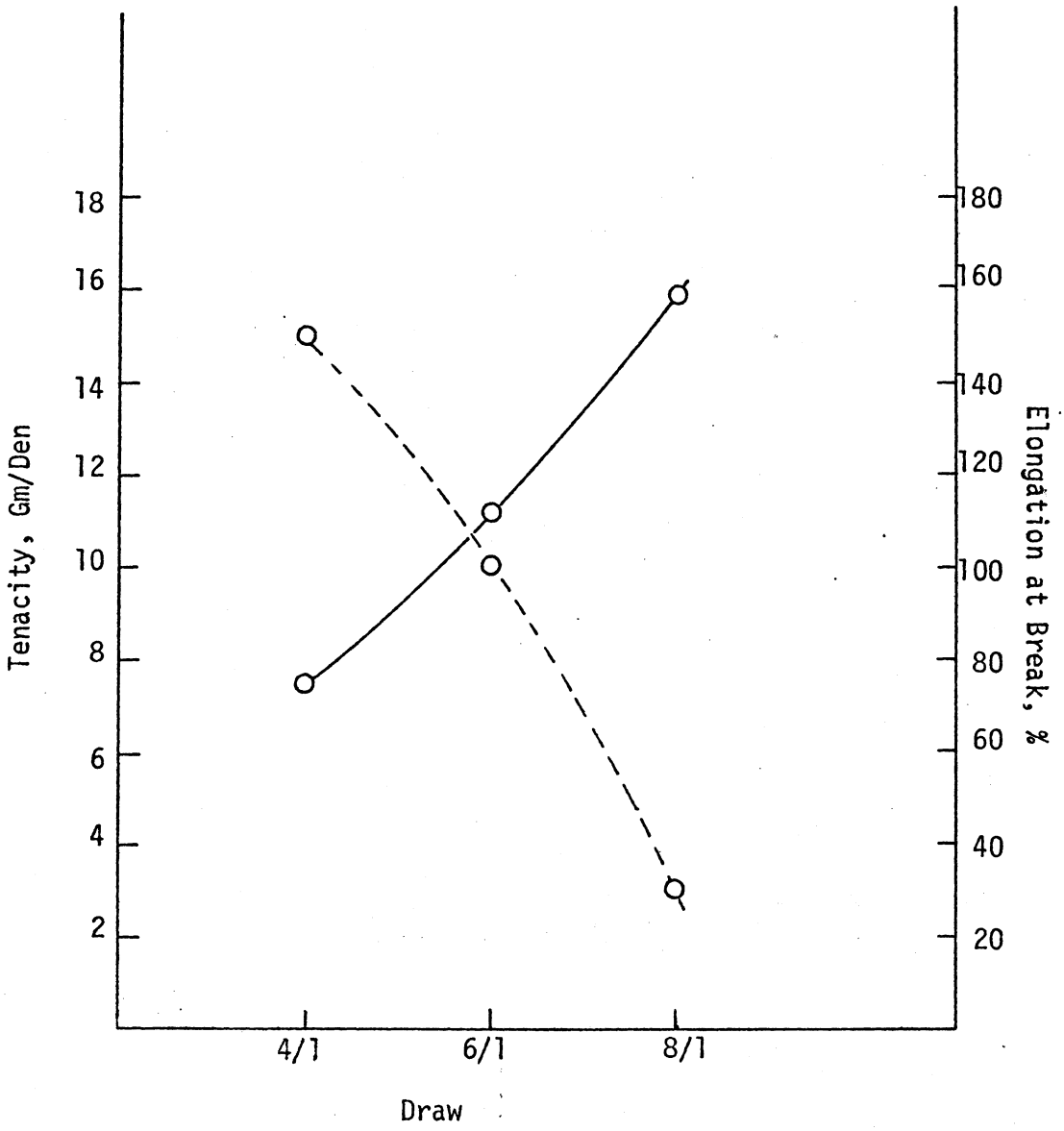

Figure 15 Effect of Draw on Fiber Tenacity of Sample No. AB, 0, 0, 0. Fibers Cold Drawn at Initial Rate of Extension of 100%/min.

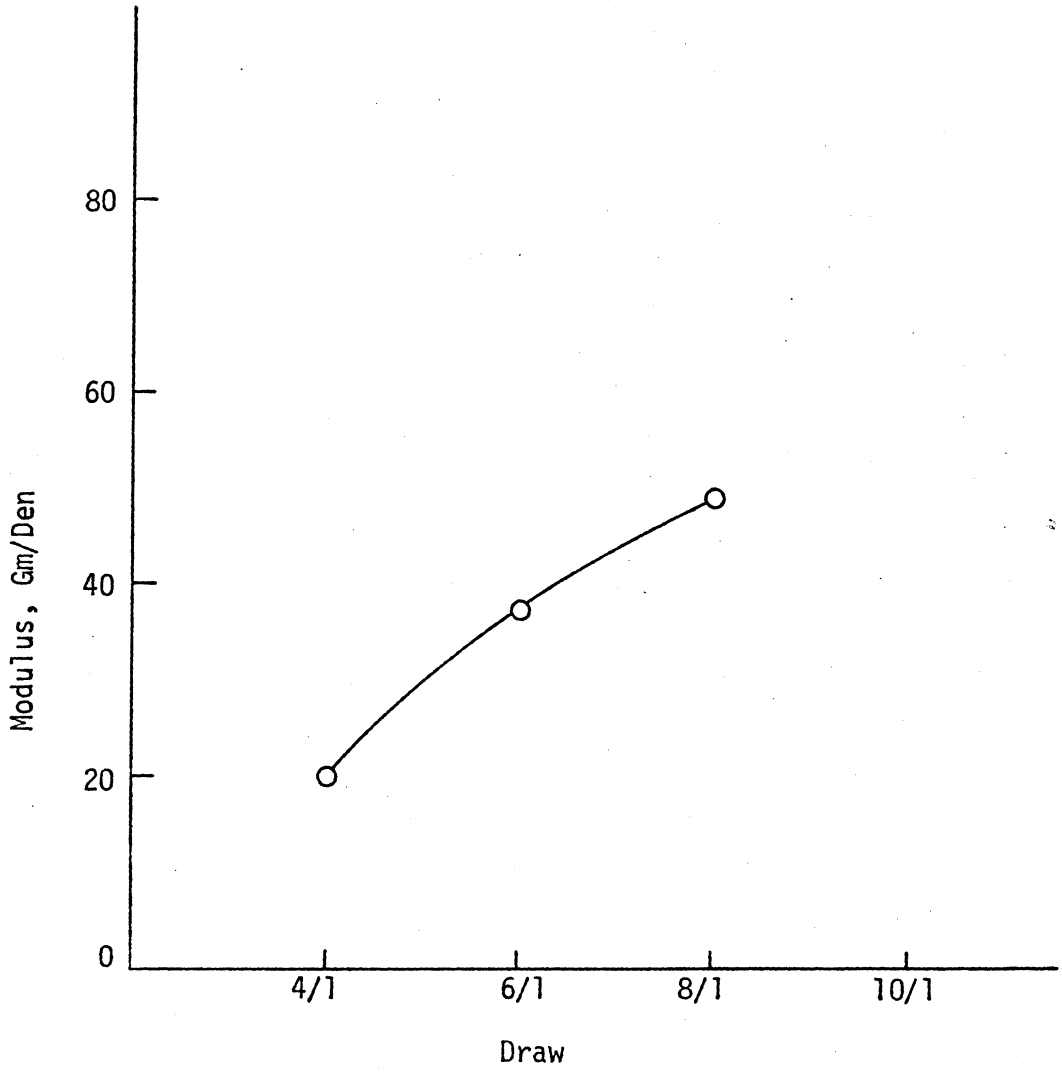

Figure 16 Effect of Draw on Initial Modulus of Fiber Sample No. AB, 0, 0, 0. Fibers Cold Drawn at Initial Rate of Extension of 100%/min.

Table II

Data on Extraction of Naphthalene by Diethyl Ether From Polypropylene Fibers

Sample No.	Extraction Time (sec)	Weight of Naphthalene After Initial Extr. (grams)	Total Weight of Naphthalene Extr. (grams)	% Extr.
A*,0,$\sqrt{3}$,0	10	0.07495	0.07955	94.22
	30	0.09250	0.09550	96.86
	60	0.07065	0.07200	98.13
	180	0.06665	0.06790	98.16
A*,0$\sqrt{3}$,$\sqrt{3}$	10	0.03200	0.03310	96.68
	30	0.06670	0.06780	98.38
	60	0.05135	0.05240	98.0
	180	0.07375	0.08075	91.33
A*,0,$\sqrt{3}$,-$\sqrt{3}$	10	0.08660	0.09165	94.49
	30	0.15335	0.15795	97.09
	60	0.10725	0.10795	99.35
	180	0.12610	0.12790	98.59
B,0,1,0	10	0.01975	0.02125	92.94
	30	0.02040	0.02650	76.98
	60	0.02730	0.02830	96.47
	180	0.04910	0.05015	97.91
B,0,0,0	10	0.02130	0.02230	95.52
	30	0.05186	0.05401	96.02
	60	0.04235	0.04285	98.83
	180	0.03035	0.02550	100.02
B,0,-1,0	10	0.00535	0.00595	89.92
	30	0.02340	0.02395	97.7
	60	0.03850	0.03975	96.86
	180	0.03110	0.03150	98.73
A,0,-$\sqrt{3}$,0	10	0.07490	0.08240	90.90
	30	0.03265	0.03325	98.20
	60	0.02270	0.02295	98.91
	180	0.03900	0.03950	98.73

Table III

Regression Coefficients for Least Squares Fit of Denier Results
Equation: Denier = A + B/Draw Ratio

Wt. % Polypropylene in Spinning Solution	A	B x 10^{-3}
25.0	0.15	2.4
32.4	0.00	3.3
42.5	0.16	4.2
52.6	0.20	5.6
60.0	0.18	5.9
42.5**	0.15	4.1

**All Denier Values Shifted According to Equation Denier. C = A' + B'/Draw Ratio, where C = Shifting Factor (concentration ratio).

Table IV

Spinning Draw Ratios, Deniers, Diameters (Means) and Void Fractions of Fiber Samples

Sample No.	Spinning Draw Ratio	Denier	Diameter μ	Void Fractions Corresponding to Pure Polymer Densities of: .91 gr/cm^3	.89 gr/cm^3
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	2200	3.48	27.8	.30	.28
A,$\sqrt{3}$,0,$\sqrt{3}$	2200	3.01	27.2	.37	.35
A,$\sqrt{3}$,$\sqrt{3}$,0	1320	4.26	28.8	.20	.18
A,$\sqrt{3}$,0,0	1280	4.81	32.7	.30	.28
A,$\sqrt{3}$,-$\sqrt{3}$,0	1300	4.57	31.0	.26	.25
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	412	13.00	54.3	.31	.30
A,$\sqrt{3}$,0,-$\sqrt{3}$	406	13.78	55.3	.30	.28
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	412	13.98	55.0	.28	.26
B,1,1,1	1848	3.58	30.4	.40	.38
B,1,0,1	1763	4.24	30.5	.29	.27
B,1,-1,1	1763	3.29	26.2	.25	.24
B,1,1,0	1340	5.13	35.8	.38	.36
B,1,0,0	1320	5.10	35.2	.36	.34
B,1,-1,0	1340	4.38	29.2	.20	.18
B,1,1,-1	792	7.24	40.5	.31	.30
B,1,0,-1	780	7.60	42.1	.33	.32
B,1,-1,-1	755	7.93	40.8	.26	.24

Table IV (continued)

Sample No.	Spinning Draw Ratio	Denier	Diameter μ	Void Fractions Corresponding to Pure Polymer Densities of: .91 gr/cm^3	.89 gr/cm^3
B,-1,1,1	1765	2.13	28.6	.60	.58
B,-1,0,1	1820	2.06	25.4	.50	.49
B,-1,-1,1	1790	1.96	30.0	.66	.65
B,-1,1,0	1270	2.37	31.5	.63	.62
B,-1,0,0	1340	2.69	32.1	.59	.58
B,-1,-1,0	1260	2.69	31.2	.59	.53
B,-1,1,-1	760	4.13	38.9	.57	.56
B,-1,0,-1	785	4.20	37.1	.53	.51
B,-1,-1,-1	815	3.60	36.0	.52	.51
A,-$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	2097	1.13	23.8	.69	.68
A,-$\sqrt{3}$,0,$\sqrt{3}$	2234	1.14	23.7	.68	.68
A,-$\sqrt{3}$,-$\sqrt{3}$,$\sqrt{3}$	2303	1.46	23.3	.58	.57
A,-$\sqrt{3}$,$\sqrt{3}$,0	1260	2.17	31.0	.65	.64
A,-$\sqrt{3}$,0,0	1320	1.96	30.1	.67	.67
A,-$\sqrt{3}$,-$\sqrt{3}$,0	1280	2.25	30.9	.63	.62
A,-$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	387	5.55	50.6	.66	.65
A,-$\sqrt{3}$,0,-$\sqrt{3}$	400	6.19	50.2	.62	.61
A,-$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	412	5.04	49.5	.68	.67

Table IV (continued)

Sample No.	Spinning Draw Ratio	Denier	Diameter μ	Void Fractions Corresponding to Pure Polymer Densities of: .91 gr/cm^3	.89 gr/cm^3
A,0,0,+$\sqrt{3}$	2244	2.00	24.4	.48	.46
B,0,1,1	1830	2.73	26.5	.39	.38
B,0,0,1	1745	2.78	26.2	.37	.35
B,0,1,0	1296	3.42	30.1	.41	.40
A,0,0,0	1266	3.30	31.1	.47	.45
AB,0,0,0	1276	3.56	30.0	.38	.37
B,0,-1,0	1286	3.87	33.2	.45	.44
A,0,-$\sqrt{3}$,0	1296	3.85	30.8	.37	.35
B,0,1,-1	746	5.00	37.5	.45	.43
B,0,0,-1	776	6.67	44.8	.48	.47
B,0,-1,-1	770	5.76	38.5	.39	.38
A,0,0,-$\sqrt{3}$	383	11.68	55.9	.42	.40
A0,-$\sqrt{3}$,-$\sqrt{3}$	390	10.82	54.0	.42	.40
A,0,$\sqrt{3}$,-.4	1122	3.43	33.6	.52	.50
A,0,$\sqrt{3}$,-1.2	663	5.81	41.6	.48	.47
A,0,$\sqrt{3}$,-2.1	204	20.51	76.6	.45	.44

Table V

Additional Spinning Draw Ratios and Deniers of Fiber Samples

Sample No.	Spinning Draw Ratio	Denier
AB,$\sqrt{3}$,$\sqrt{3}$,1	1877	2.97
AB,$\sqrt{3}$,0,1	1877	3.23
AB,$\sqrt{3}$,$\sqrt{3}$,-1	804	7.72
AB,$\sqrt{3}$,0,-1	804	7.68
AB,$\sqrt{3}$,-$\sqrt{3}$,-1	780	11.00
AB, 1,$\sqrt{3}$,1	1820	3.84
AB, 1,$\sqrt{3}$,0	1300	4.54
AB,1,-$\sqrt{3}$,0	1300	4.38
AB,1,$\sqrt{3}$,-1	780	6.98
AB,1,-$\sqrt{3}$,-1	780	7.21
AB,-1,1,$\sqrt{3}$	2370	1.41
AB,-1,0,$\sqrt{3}$	2180	1.54
AB,-1,-1,$\sqrt{3}$	2180	1.55
AB,-1,1,-$\sqrt{3}$	420	8.23
AB,-1,0,-$\sqrt{3}$	400	8.40
AB,-1,-1,-$\sqrt{3}$	380	8.84

Table V (continued)

Sample No.	Spinning Draw Ratio	Denier
AB,-$\sqrt{3}$,$\sqrt{3}$,1	1790	1.35
AB,-$\sqrt{3}$,0,1	1834	1.42
AB,-$\sqrt{3}$,-$\sqrt{3}$,1	1848	1.40
AB,-$\sqrt{3}$,$\sqrt{3}$,-1	780	3.00
AB,-$\sqrt{3}$,0,-1	792	3.26
AB,-$\sqrt{3}$,-$\sqrt{3}$,-1	804	3.49

Table VI

Tensile Properties of Fiber Samples After 4/1 Draw

Initial Rate of Extension: 100%/min.

Sample No.	Initial Modulus gm/den	Tenacity gm/den	Elongation at Break %
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	16.70	4.63	165
A,$\sqrt{3}$, 0,$\sqrt{3}$	15.70	3.99	75
AB,$\sqrt{3}$,$\sqrt{3}$, 1	17.40	4.69	95
AB,$\sqrt{3}$, 0, 1	16.60	4.50	105
A,$\sqrt{3}$,$\sqrt{3}$, 0	12.00	3.90	130
A,$\sqrt{3}$, 0, 0	11.50	3.51	130
A,$\sqrt{3}$,-$\sqrt{3}$, 0	11.55	4.04	145
AB,$\sqrt{3}$,$\sqrt{3}$,-1	11.10	3.84	260
AB,$\sqrt{3}$, 0,-1	11.20	3.83	215
AB,$\sqrt{3}$,-$\sqrt{3}$,-1	9.10	2.83	145
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	7.30	3.35	280
A,$\sqrt{3}$, 0,-$\sqrt{3}$	9.40	2.73	280
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	9,50	2.92	300
AB,1,$\sqrt{3}$, 1	11.00	3.33	180
B,1,1,1	13.90	4.05	125
B,1,0,1	10.10	3.35	190
B,1,-1,1	12.80	3.35	100
AB,1,$\sqrt{3}$, 0	9.95	3.03	235

Table VI (continued)

Sample No.	Initial Modulus gm/den	Tenacity gm/den	Elongation at Break %
B,1,1,0	11.00	3.26	215
B,1,0,0	9.95	3.25	305
B,1,-1,0	9.13	2.68	200
AB,1,$\sqrt{3}$,0	9.13	3.00	250
AB,1,$\sqrt{3}$,-1	9.35	3.11	300
B,1,1,-1	8.70	2.93	300
B,1,0,-1	8.73	2.55	335
B,1,-1,-1	9.18	3.10	300
AB,1,-$\sqrt{3}$,-1	8.95	2.78	300
AB,-1,1,$\sqrt{3}$	12.00	3.50	160
AB,-1,0,$\sqrt{3}$	11.70	3.57	165
AB,-1,-1,$\sqrt{3}$	11.15	3.58	155
B,-1,1,1	9.20	3.13	185
B,-1,0,1	9.30	3.59	200
B,-1,-1,1	9.30	3.35	175
B,-1,1,0	8.50	3.06	240
B,-1,0,0	8.00	2.94	210
B,-1,-1,0	6.20	2.36	260
B,-1,1,-1	7.30	2.56	210
B,-1,0,-1	7.20	2.46	150
B,-1,-1,-1	7.90	2.92	205

Table VI (continued)

Sample No.	Initial Modulus gm/den	Tenacity gm/den	Elongation at Break %
AB,-1,1,-$\sqrt{3}$	6.85	2.42	150
AB,-1,0,-$\sqrt{3}$	7.60	2.21	145
AB,-1,-1,-$\sqrt{3}$	6.50	1.96	150
A,-$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	8.10	2.71	170
A,-$\sqrt{3}$, 0,$\sqrt{3}$	8.60	2.95	150
A,-$\sqrt{3}$,-$\sqrt{3}$,$\sqrt{3}$	8.60	3.19	200
A,-$\sqrt{3}$,$\sqrt{3}$, 0	7.30	2.27	235
A,-$\sqrt{3}$,0,0	8.30	2.44	195
A,-$\sqrt{3}$,-$\sqrt{3}$,0	6.90	2.41	190
A,-$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	7.70	1.60	30
A,-$\sqrt{3}$,0,-$\sqrt{3}$	5.70	1.24	70
A,-$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	8.20	1.85	70

Table VII

Tensile Properties of Fiber Samples Spun From 42.5 wt% Polypropylene Solution After 4/1 Draw Initial Rate of Extension: 100%/min.

Sample No.	Modulus gm/den	Tenacity gm/den	Elongation at Break %
A,0,$\sqrt{3}$,-.4	16.50	6.26	190
A,0,0,$\sqrt{3}$	32.75	8.85	60
B,0,1,1	20.60	7.28	150
B,0,0,1	25.25	8.45	145
B,0,-1,1	30.30	8.92	90
A,0,$\sqrt{3}$,-1.2	14.20	5.00	180
B,0,1,0	18.00	6.94	175
A,0,0,0	20.10	6.72	150
B,0,0,0	18.75	7.00	185
AB,0,0,0	20.40	7.94	150
B,0,-1,0	22.65	7.30	95
A,0,-$\sqrt{3}$,0	26.60	7.79	100
B,0,1,-1	18.05	6.88	190
B,0,0,-1	15.70	6.02	180
B,0,-1,-1	15.00	6.10	190
A,0,$\sqrt{3}$,-2.1	11.70	2.81	25
A,0,0,-$\sqrt{3}$	15.60	5.86	200
A,0,-$\sqrt{3}$,-$\sqrt{3}$	15.80	5.50	220

Table VIII

Tensile Properties of Fiber Samples
Rate of Extension 100%/min.

Sample No. Code	Draw	Moduli gr/den	Tenacity gr/den	Elongation at break %
A,0,0,0	4/1	23.50	7.02	120
A,0,0,0	6/1	47.70	11.25	65
B,0,0,0	6/1	37.20	10.42	55
AB,0,0,0	6/1	37.40	11.17	100
A,B,0,0,0	8/1	49.10	15.84	30

Table IX

Tensile Properties of Fiber Samples
Rate of Extension 200%/min.

Sample No. Code	Draw	Moduli gr/den	Tenacity gr/den	Elongation at Break %
A,0,0,0	4/1	21.20	7.57	130
B,0,0,0	4/1	19.20	6.91	120
AB,0,0,0	4/1	20.00	7.43	160
A,0,0,0	6/1	44.60	11.36	45
B,0,0,0	6/1	37.60	10.82	50
AB,0,0,0	6/1	40.10	12.00	60

This low extraction time and the results presented in the section dealing with void fraction measurements indicate that most of the naphthalene migrates to the fiber's surface or that a very porous structure is formed during phase separation. This was first proposed by Williams[28]. It must be noted, however, that petroleum ether has also been used successfully as a washing solvent by Zwick[63].

It was observed that after washing the fiber ceased to be brittle and relatively free of tangling. Also, as opposed to having a bright surface, the surface of the fibers became dull. These observations are similar to those presented in the literature[18].

Denier Measurements

Effect of Spinning Draw Ratio on Initial Fiber Denier. Figures 3 and 4 show that the denier of the polypropylene fiber was inversely proportional to the spinning draw ratio. This result was expected.

Figure 4 is a plot of the equation:

$$\text{Denier} = A + B/\text{Draw Ratio} \qquad (1)$$

In theory, A should equal zero, since at an infinitely high draw ratio the denier of the spun fiber should be zero. The coefficients of equation (1) are shown in Table III. The coefficient A is very small as expected. The effect of the polymer concentration on the fiber denier is directly related to the slope of the curves presented in Figure 4 and thus, to the coefficient B.

As expected, the spinning temperature does not have any effect on the fiber denier.

For small deviations in spinning draw ratios (<5%), some rather large deviations (up to 12%) can be observed in denier. These deviations, which occur mostly at high polymer concentrations, are strong functions of the difficulty in obtaining a solution, as was discussed earlier.

Effect of Polymer Concentration on Denier. The denier was very dependent on the composition of the solution being spun. For constant draw down ratio the effect of the solution composition can be manifest in two ways: 1) an increase in fiber diameter with increasing polymer content in the solution and 2) an increase in denier. Obviously, since denier is a linear density measurement it does not include the effect of different diameter sizes of the fibers. This situation can be complicated further by having a porous fiber. However, if the diameter of the fibers does not vary much as the solution concentrations are changed, it can be easily envisioned that the effect of concentration on denier will be linear. Thus, by choosing a proper shifting factor, namely a polymer concentration ratio, a plot of the deniers of fiber samples spun from solutions of different polymer contents versus the inverse of the draw ratio should be a straight line. The graphical test of this proposed relationship is shown in Figure 5.

The deniers of fiber samples spun from solution concentrations ranging from 25 to 60 wt. % polypropylene are shown after being shifted about the midrange polymer concentration used, 42.5 wt. %

polypropylene. The shifting factor (c) for this curve is defined as the ratio of the concentration about which the deniers are being shifted (in this case 42.5%) over the actual polymer concentration of the solution from which the sample was spun. Therefore, for all fiber samples, equation (1) becomes:

$$\text{Denier} \times C = A' + B'/\text{Draw Ratio}$$

Coefficients A' and B' are shown in Table III. It can be seen that since the coefficient B corresponding to a least squares fit of equation (1) for deniers of samples spun from a solution concentration of 42.5 wt. % polymer and B' differ by less than 5% the proposed relationship fits the experimental data well.

Deniers ranging from 1.1 to 6.1 for 25, and from 3.0 to 14.0 for 60 wt. % polypropylene were obtained as the draw ratio varied from 2200 to 400.

Fiber Diameter and Void Fraction

It can be seen from Figure 6 that the diameter of the fiber samples was inversely proportional to spinning draw ratio. This was a generally expected result. Concentration, however, had only a minor effect on fiber diameter. As can be observed in Table IV fibers spun from solutions of high polymer content had larger diameters than those fibers spun from 25 and 32.4 wt. % polypropylene solutions. But, the maximum deviation observed was only about 15%, between fibers made from 25 and 60 wt. % polypropylene solution, at

the highest draw ratio used for comparison (2200). This variation is small relative to the error introduced in measuring the fiber diameter, which is about 9%.

The diameters ranged from 23 to 55 microns as the draw ratio varied from 2200 to 400.

Temperature also had a minor influence on the diameter. The diameters of fibers spun at or slightly above (4°C) the minimum solubility temperatures of the spinning solutions, were consistently lower than those of the fibers spun at higher temperatures (from 10 to 20°C above the melting point of the solution). This may indicate that as the melting point of the solution is approached the naphthalene migrates close to the surface of the fiber. While no published references have been found to confirm this behavior, a phase separation and migration of the solvent to the surface seems to occur.

The void fraction of the fiber is a calculated result, obtained from the denier and diameter measurements (See equation (2), Appendix). These results, presented in Table IV and Figure 7 show the effect of the concentration of polymer in the spinning solution on the void fraction. While the trend of the curve is reasonable in that for a high polymer concentration the void fraction of the fibers is smaller, there is a high degree of uncertainty as to the value of the void fraction at concentrations higher or lower than those studied, since a nonlinear relationship is expected then.

The calculated values of the void fractions range from .20 to .69 as the concentration decreases from 60 to 25 wt. % polypropylene in

the spinning solution.

There was generally no observed effect of draw ratio on void fraction. This result is expected, since in the calculation of the void fraction, denier is divided by the diameter squared and both quantities are inversely proportional to the spinning draw ratio, thus their effects cancel.

Temperature, however, had an important effect on the fiber void fraction. Fibers spun at temperatures close to the melting point of the solution had a lower void fraction than those fibers from the same solution spun at higher temperatures. This result is easily explained because, as discussed earlier, denier is independent of temperature while the fiber diameter decreases at temperatures close to the melting point of the solution. Since the fraction of the fiber volume occupied by pure polypropylene is directly proportional to the denier and inversely proportional to the diameter of the fiber squared, as the spinning temperature approaches the melting point the fraction of the volume occupied by the polymer increases and, as a result, the void fraction decreases.

Due to lack of accurate information at this point with respect to the actual value of the density of pure polypropylene, two values (.91 and .89 gr/cm^3) have been used to calculate the void fractions. This range of densities is common for pure polypropylene[30].

In view of the fact that the necessary equipment is not available, several questions remain unanswered such as, the pore size

distribution, the average pore diameter and the permanence of the voids inside the fiber. Even so, it is the opinion of the author that the findings with respect to void fraction open new possibilities to the phase separation technique to be applied not only to fibers but to films as well.

A careful study of the spinning conditions and finishing treatments on the fibers should lead to correlations with respect to the amount of porosity, pore size, etc. which could be useful in different areas. In the case of polypropylene, for example, its water absorbency could be greatly improved.

Tensile Properties

Tenacity. The effect of spinning draw ratio on tenacity is shown in Figure 8. As the draw ratio increased from 400 to 2200 tenacities increased by a factor of about 1.7, for all solutions studied except that of 42.5 wt. % polymer. This behavior is expected since increased draw ratio should increase the orientation of the molecules and chains of polymer making up the fiber. As the axial orientation increases, higher tenacities are expected.

Also, as the polymer content of the spinning solution was increased higher tenacities were obtained. This is also an expected result, since for a constant draw ratio, the highly concentrated solutions have greater viscosity, thus, as a fiber is drawn, it is under the effect of a greater stress than fibers spun from lower viscosity solutions. This results in higher orientation and thus

higher tenacity. According to this reasoning, however, fibers spun from solutions of different polymer contents under the same stress-strain conditions should have the same tenacity. This, in fact, is not usually the case. The maximum draw ratio of the fiber spun from a solution of low polymer contents will be reached before the fiber can reach the same level of tenacity of the fiber spun from a more concentrated solution. In summary, fiber tenacities should not be compared on the basis of constant draw ratio for different polymer concentrations, but rather on the basis of constant percent of maximum feasible draw ratio. This comparison, however, is beyond the scope of the present investigation.

Fiber tenacities obtained after a 4/1 cold draw range from 1.5 to 2.75 g/den for 25 wt. % and from 3.2 to 4.6 g/den for 60 wt. % polypropylene spinning solution compositions, as the draw ratio increased from 400 to 2200.

In addition to the observations made in the preceding paragraphs, it must be noted that for a given fiber sample there is usually a specific set of conditions that will optimize the tensile properties of the fibers. The effect of draw and rate of extension will be examined later, but additional variables such as stretching temperature and heat stretching factor can improve tensile properties enormously. Thus, the values obtained in this study are not considered the best possible.

As it is shown in Table VI the effect of spinning temperature on tenacity was not consistent. The expected result was a decrease

in tenacity with increasing temperature, due to an increase in draw down before freezing[17].

Tenacities presented in this study, for 25.0, 32.4, 52.6 and 60.0 wt. % polymer spinning solutions, are in the low region of tenacities found in commercial textile polypropylene fibers (3-8 gr/den)[30] but an optimization type of study on the tensile properties of the fiber samples presented in this investigation was beyond the scope of the project. However, it is felt that the tenacities could be improved substantially by proper drawing.

Tenacities of Fibers Spun from 42.5 Weight Percent Polypropylene Solution. These tenacity measurements are discussed separately, since they had singular characteristics, different from those measurements discussed above. The effect of draw ratio on tenacity was the same for these fibers as for the other fiber samples analyzed, that is, an increase of tenacity was observed as the spinning draw ratio increased, as shown in Figure 9. The actual tenacity values, however, were an average of 160% higher than those for fibers spun from the 60% polypropylene solution under the same conditions. These results are highly unexpected and a satisfactory explanation has not been found. A brief description of the peculiarities of the fibers spun from this 42.5 wt. % polypropylene solution will be attempted as follows:

A. Spinning Difficulty: This solution was by far the most difficult one to spin. Spinning was attempted four

times before the fiber samples were collected. The main problem was fiber breakage at relatively low draw ratios of about 1300, even at temperatures 20°C above the minimum solubility temperature. Higher and lower solution concentrations were spun easily.

B. Unusual Coiling: As opposed to the other fiber samples, which developed coiling only at high draw ratios (above 1820); the fibers spun from the 42.5% solution developed good coiling even at such low draw ratios as 780. Upon cold drawing these fibers showed a decrease in coils per inch accompanied by an increase of the diameter of the coils.

Any explanation based on human error in making the spinning solution can be discarded since the denier, diameter and void fraction data correlate well with the results obtained from other fiber samples.

An explanation based on fiber structure parameters, such as pore diameter and distribution, would require information beyond the scope of this investigation.

Modulus. Figure 10 shows a typical force versus elongation curve from which the tensile properties of the fiber were obtained. This curve was obtained using Sample No. AB,0,0,0. As can be seen the initial slope decreases after an elongation of approximately 1% and remains constant until an elongation of 18% is reached. The slope of the curve between 1% and 18% elongation was measured and

interpreted as the elastic modulus of the fiber. Published results on creep and relaxation behavior are not available for polypropylene, but they are available for polyethylene films[31]. These results show that the slope of the curve below 1% elongation is not significant and that if the samples tested are maintained under constant stress for an appropriate length of time in the region below which hardening occurs and then relaxed, an elastic type of recovery will occur. Also, it can be expected that as the rate of extension of the fibers increases a uniform slope can be obtained. This would prove that the fibers made during this investigation have a good degree of elasticity.

The moduli of the fiber samples tested, corresponding to spinning solutions of 25, 32.4, 52.6 and 60 wt. % polypropylene are listed in Table VI. Figure 11 shows the effect of spinning draw ratio on the initial modulus of these fibers, after the fibers had been cold drawn 4/1 at an initial rate of extension of 100%/min.

The modulus of the fibers was very dependent on both the concentration of the spinning solution and the draw ratio. Also, the effect of draw ratio was larger on fibers spun from highly concentrated solution than on fiber made from more dilute solutions. The same explanation offered to analyze the effect of solution concentration and draw ratio on fiber tenacity applies.

The values of the moduli of the fiber samples varied from 5.8 to 8.3 gr/den for 25 wt. % and from 9.4 to 16.5 gr/den for 60 wt. %

polypropylene in the spinning solution. These values are in the range of the moduli of commercial textile polypropylene fibers.

Some moduli values of fibers spun from the 42.5% polypropylene solution are shown in Figure 12. The moduli of these fibers range from 15 to 32 gr/denier, almost twice as high as the moduli of fibers spun from a 60.0 wt. % polypropylene solution. The peculiarities of the 42.5% fibers were discussed earlier.

Fibers spun at temperatures close to the melting point of the spinning solution had lower moduli than those of the fibers spun at higher temperatures. As it can be easily recalled these fibers also possessed lower void fractions, but more information with regard to the distribution of the voids inside these fibers would be needed to explain these results.

Elongation at Break. Figure 13 shows the effect of spinning draw ratio on the elongation at break of fibers spun from solutions of different polymer contents. For polymer concentrations of 60 and 52.6 wt. %, the percent elongation at break was inversely proportional to the spinning draw ratio and inversely proportional to concentration. This is an expected result, since for constant draw ratio the orientation of the crystalline planes inside the fiber is a function of the polymer concentration in the spinning solution. Thus, for a given crosshead speed, it takes less time for the chains of molecules forming the fiber to start slipping by one another and finally undergo breakage. Obviously, this results in a lower elongation at break. For spinning draw ratios greater than 1800 the

fibers spun from 25 and 32.4 wt. % polypropylene solutions behave in a similar manner.

In general, as can be seen in Table VI fibers spun at temperatures close to the melting point of the solution had low elongations at break. The distribution of the breakage, however, was usually wide, covering as much as 100% elongation before about two-thirds of the fibers making up the sample broke. This behavior might indicate a very wide distribution of pore sizes and locations. Again, measurements of the pore distribution would be needed to analyze these results accurately.

For draw ratios ranging from 400 to 1300, the elongation at break of fibers spun from solutions of 25 and 32.4% polypropylene contents increased as the draw ratio increased. Even though no explanation for this behavior has been proposed, published results show the same trend for solution compositions of 15 and 22% polypropylene. It is possible that at low draw ratios the low viscosity solutions are not under any strain, flow freely, and thus, only a minimum amount of orientation develops in the fiber. This crystalline disorder could easily be such that upon cold drawing the chains forming the fiber would not align themselves parallel to the fiber axis and develop internal stresses, which when further drawing is tried during the fiber testing result in breakage at short elongations.

It is interesting to note that all the tensile properties of fibers made weak polymer solutions (below 15% polymer), have been measured after the fibers have been heat stretched[3].

The elongations at break of fibers spun from a 42.5 wt. % polypropylene are shown in Figure 14. The expected trend is followed with the elongation, decreasing as the spinning draw ratio increases. However, the effect of draw ratio on the elongation of these fibers is not as marked as it is for fibers spun from more concentrated solutions.

Effect of Draw on Tensile Properties. As can be seen in Figure 15, an increase in draw of the fibers, previous to testing, brings about an increase in tenacity and a decrease in elongation at break. This is the expected behavior since, as previously discussed, an increase in draw results in better axial orientation of the chains of molecules forming the fibers which in turn causes higher tenacities and shorter elongations at break. As can be seen in Table VIII only one sample (No. AB,0,0,0) was tested at more than two different draws. Therefore, these results are by no means a proof that the relationship between draw and tenacity is linear; but rather show that the tensile properties of the fibers made during this investigation can be improved a lot further.

It can be observed in Figure 16 that the modulus of the sample tested also was directly proportional to the amount of draw undergone by the fibers before testing. This was also expected for the reasons previously discussed.

Examination of the results presented in Tables VIII and IX reveals that the initial rate of extension has only a minor effect on

tenacity and modulus. This, however, was expected, because only for very large increases in draw, of at least one order of magnitude, will rate of extension then show an important effect on tenacity.

The tenacities of the fibers tested after an 8/1 draw compare very favorably with the tenacities of commercial textile polypropylene fibers made by melt spinning and by phase spearation spinning.

Recommendations

The following is a list of recommendations designed to improve the present equipment at low cost, to alleviate any problems related to the procedure and for further studies.

Further Studies. The results obtained in this investigation merit future application of the phase separation method to the spinning of fibers which when spun by other methods exhibit low absorbency, low modulus and low tenacities. It should also be applied to systems where spinneret clogging is common. The results of this investigation, which confirm previous results, show that fibers of low denier and small diameter can be easily spun from large spinnerets.

The physical properties of the fibers spun by the phase separation technique, which showed ease to tangle, good crimp and hand, indicate that this spinning method could be applied advantageously to the production of fibers that when spun by other techniques have poor physical characteristics.

It is recommended that the tensile properties of the fibers spun during this investigation be optimized. For the fibers that showed an increase in tenacity jointly with a decrease in elongation, cold drawing should be tried first. For the fibers spun from low viscosity solutions which exhibited an increase in tenacity and elongation at break at the same time, the effect of hot drawing on these properties should be investigated.

The effect of these treatments, hot or cold drawing, on the void fraction of the fibers and on the pore site and size distributions of the fibers should be studied. The average pore diameter could be calculated easily by running absorption experiments to determine the total surface area of the fiber.

In all future studies of this type, the spinning draw ratio should not be measured as an independent variable, but as a function of temperature and concentration, in such a way that no comparisons should be made on the basis of absolute draw ratio; but rather on the fraction of the feasible spinning range that the absolute draw ratio represents.

Procedure. The precast rod method described by Zwick should be tried for the solution preparation and charging steps. This would eliminate any loss of solvent while the solution is charged into the spinning cylinder.

The use of cardboard drums for sample collection should be continued since it facilitates enormously the denier measurements and

the retention of samples for tensile testing. However, an automatic guide should be used to direct the fiber over the drum, thus freeing an operator from this tedious occupation.

Due to the amount of variables to be adjusted, controlled and measured, it is recommended that two operators be used during the spinning operation.

Equipment Modifications. The main drawbacks of the equipment used are related to the take-up units which limit the take-up speeds to about 600 meters per minute and vibrates at speeds higher than 650 revolutions per minute. The range of take-up speeds available could be easily expanded by increasing the size of the pulley fixed to the variable speed motor. The vibrations in the take-up drum, rather than being caused by a drum unbalance are caused by the shaft connecting the drum to the variable speed motor. Due to the weight of the drum the shaft presently used is slightly bent, thus it is recommended that the present shaft be replaced by one of larger diameter.

A high torque motor coupled with an anchor type agitator should be obtained to mix the more viscous spinning solutions.

V. CONCLUSIONS

The properties of fibers spun by phase separation spinning were studied in this investigation. The fibers were spun from polypropylene-naphthalene solutions using a one millimeter spinneret. The range of polymer concentrations used was 25-60 wt. % polypropylene. The spinning temperatures ranged from 0 to 20°C above the minimum solubility temperatures of the solutions used. The results of this study led to the following conclusions:

1. Low denier and small diameter polypropylene fibers could be easily spun from relatively large spinneret holes by the phase separation technique.

2. Deniers as well as diameters of the fibers made varied inversely with draw ratio.

3. Denier, much more than diameter, was directly dependent on the concentration of the spinning solution.

4. Deniers of fibers were independent of the spinning temperature, however, the diameters of fibers spun at temperatures close to the melting point of the spinning solution were smaller, indicating a migration of the solvent to the surface of the fiber.

5. The void fraction of the fibers tested was directly proportional to the polymer contents of the spinning solution, was relatively independent of the spinning draw ratio, and decreased at spinning temperatures close to the melting point of the solution.

6. Tenacities and initial modulus of fibers drawn 4/1 increased with spinning draw ratio. Except for the tenacities of fibers spun for a 42.5 wt. % polymer, tenacities directly related to the concentration of polymer in the spinning solution. The fibers spun from a 42.5 wt. % polypropylene solution showed higher tenacities than any other fibers. Tenacities ranged from 2.6 to 8 and modulus from 5 to 20 grams per denier.

7. Elongations at break of fibers spun from 42.5, 52.6 and 60.0 wt. % polypropylene solution were inversely proportional to the spinning draw ratio, ranging from 300 to 60 percent. Elongations at break of fibers spun from solutions of low polymer contents (25 and 32.4 wt. % polypropylene) increased with draw ratio, for draw ratios below 1500. For draw ratios above 1500 the elongations at break were inversely proportional to the spinning draw ratio.

8. An increase in the amount of cold draw undergone by the fibers before testing increased the tenacities and moduli of the fibers tested and reduced the elongation at break of the same fibers.

9. Polypropylene fibers spun by the phase separation technique exhibited physical characteristics such as good hand, bulk, crimp and ability to tangle. These characteristics are sought for in synthetic fibers commercially used.

VI. SUMMARY

The first objective of this investigation was to determine a range of polymer concentrations and solution temperatures where the phase separation spinning method could be used to produce polypropylene fibers. The solvent used was naphthalene.

Solutions of polypropylene in naphthalene ranging from 25 to 60 wt. % polymer were spun at temperatures ranging from 0 to 20°C above the melting point of the solution. Spinnability of solutions below 25 wt. % polymer had been shown in previous studies by Zwick[1] and by Fricke and Williams[17, 26]. Spinning of solutions with polymer contents above 60% was not attempted due to the difficulty in mixing a highly viscous solution with the present equipment.

The second objective was to investigate the effect of the polymer concentration and spinning conditions on the structure and properties of the fibers.

Deniers of all fiber samples varied inversely with spinning draw ratio and directly with solution composition. The fibers were porous. The void fraction of the fibers was independent of draw ratio and inversely proportional to the polymer content of the spinning solution. After the fiber samples were cold drawn 4:1, tensile testing showed that tenacities and initial moduli were directly proportional to draw ratio. The percent elongation at break of fibers spun from concentrated (or highly viscous) solutions varied inversely with draw ratio, while for solutions of low polymer contents it varied directly with draw ratio.

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

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VIII. APPENDIX

This section contains information which would be needed in order to reproduce the results of this investigation. A complete listing of all materials and apparatus used is included.

Data Tables

This section contains the data obtained during the investigation. Tables X-XIV contain information describing the spinning conditions under which fibers were spun from different polymer concentration solutions. Tables XV-XVI contain extraction, denier and diameter data. Tables XVII-XVIII contain tensile properties measurements.

Sample Calculations

These are examples of calculations made during this investigation.

Piston Travel Rate and Linear Velocity of the Spinning Solution in the Spinneret. From Table XII for Sample No. A,0,0,0, the motor shaft speed was 63 sec/rev. The two sprockets in the chain drive had 9 and 25 teeth; the threaded rod had 20 threads per inch, thus its speed was 0.05 inches per revolution of the large sprocket. The piston travel rate was calculated as follows:

$$\text{Piston Travel Rate} = \left(\frac{1}{63}\right)\left(\frac{9}{25}\right)(.05) = .000286 \text{ in/sec}$$

$$= .000435 \text{ m/min.}$$

The inner diameters of the spinning cylinder and spinneret hole were 0.998 and 0.040 inches, respectively. Thus, the linear velocity in

Table X

Spinning Conditions and Samples Collected for Test I

Composition of Spinning Solution: 25.0% Polypropylene

Cabinet Temperature: 25°C

Minimum Solubility Temperature (T_S): 136°C

Range of Bath Temperature (T)

Sample No.	$T-T_S$ (°C)	Spinneret Temp. (°C)	Motor Shaft Speed (sec/rev)	Take-up Speed (RPM)
A,$-\sqrt{3},\sqrt{3},0$	20	136	62.0	182
A,$-\sqrt{3},\sqrt{3},\sqrt{3}$	20	136	61.0	308
AB,$-\sqrt{3},\sqrt{3},-1$	20	136	64.0	109
AB,$-\sqrt{3},\sqrt{3},1$	20	136	63.0	255
A,$-\sqrt{3},\sqrt{3},-\sqrt{3}$	20	136	62.0	56
A,$-\sqrt{3},0,\sqrt{3}$	10	127	65.0	308
AB,$-\sqrt{3},0,-1$	10	127	65.0	109
A,$-\sqrt{3},0,0$	10	127	65.0	182
AB,$-\sqrt{3},0,1$	10	127	64.5	255
A,$-\sqrt{3},0,-\sqrt{3}$	10	127	64.0	56
A,$-\sqrt{3},-\sqrt{3},-\sqrt{3}$	0	119	66.0	56
A,$-\sqrt{3},-\sqrt{3},\sqrt{3}$	0	118	67.0	308
A,$-\sqrt{3},-\sqrt{3},0$	0	118	63.0	182
AB,$-\sqrt{3},-\sqrt{3},1$	0	118	65.0	255
AB,$-\sqrt{3},-\sqrt{3},-1$	0	118	66.0	109

Table XI

Spinning Conditions and Samples Collected for Test II

Composition of Spinning Solution: 32.4% Polypropylene

Cabinet Temperature: 25°C

Minimum Solubility Temperature (T_S) = 139°C

Range of Bath Temperatures (T) = 143 - 155°C

Sample No.	$T-T_S$ (°C)	Spinneret Temp. (°C)	Motor Shaft Speed (sec/rev)	Take-up Speed (rpm)
B,-1,1,0	16	132	62.5	182
AB,-1,1,$\sqrt{3}$	16	131	69.0	308
B,-1,1,1	16	130	62.0	255
AB,-1,1,-$\sqrt{3}$	16	130	67.0	56
B,-1,1,-1	16	130	62.5	109
B,-1,0,1	10	130	64.0	255
B,-1,0,0	10	128	66.0	182
AB,-1,0,$\sqrt{3}$	10	128	64.0	56
AB,-1,0,-$\sqrt{3}$	10	129	63.5	308
B,-1,0,-1	10	128	64.5	109
AB,-1,-1,$\sqrt{3}$	4	125	63.5	308
B,-1,-1,-1	4	125	67.0	109
B,-1,-1,0	4	125	62.0	182
B,-1,-1,1	4	125	63.0	255
AB,-1,-1,-$\sqrt{3}$	4	125	60.5	56

Table XII

Spinning Conditions and Samples Collected for Test III

Composition of Spinning Solution: 42.5% Polypropylene

Cabinet Temperature: 27°C

Minimum Solubility Temperature (T_s) = 141°C

Range of Bath Temperatures (T) = 141-161

Sample No.	$T - T_s$ (°C)	Spinneret Temp. (°C)	Motor Shaft Speed (sec/rev)	Take-up Speed (rpm)
A,0,$\sqrt{3}$	20	145	33.0	308
A,0,$\sqrt{3}$	20	147	33.0	182
A,0,$\sqrt{3}$	20	148	33.0	56
B,0,1,-1	16	143	62.0	109
B,0,1,0	16	143	64.5	182
B,0,1,1	16	143	65.0	255
A,0,0,0	10	147	63.0	182
B,0,0,-1	10	139	64.5	109
A,0,0,	10	140	66.0	308
B,0,0,0	10	137	63.0	182
B,0,0,1	10	135	62.0	255
A,0,0, $\sqrt{3}$	10	138	62.0	56
AB,0,0,0	10	138	63.5	182

Table XI (continued)

Sample No.	$T - T_s$ (°C)	Spinneret Temp. (°C)	Motor Shaft Speed (sec/rev)	Take-up Speed (rpm)
B,0,-1,-1	4	33	64.0	109
B,0,-1,0	4	130	64.0	182
B,0,-1,1	4	128	64.0	255
A,0,	0	122	63.0	56
A,0,	0	125	64.5	182

Spinning Conditions and Samples Collected for Test IV

Composition of Spinning Solution: 32.4% Polypropylene

Cabinet Temperature: 22°C

Minimum Solubility Temperature (T_S) = 149°C

Range of Bath Temperatures (T) = 149 - 169°C

Sample No.	T - T_S °C	Spinneret Temp. °C	Motor Shaft Speed (sec/rev)	Take-up Speed (rpm)
B,1,0,1	10	141	62.0	255
B,1,0,0	10	142	65.0	182
B,1,0,-1	10	142	64.0	109
B,1,1,-1	16	137	65.0	109
B,1,1,1	16	139	65.0	255
B,1,1,0	16	132	66.0	182
B,1,-1,0	4	130	66.0	182
B,1,-1,1	4	130	62.0	255
B,1,-1,-1	4	137	62.0	109
B,1,$-\sqrt{3}$,1	0	135	65.0	255
B,1,$-\sqrt{3}$,0	0	135	64.0	182
B,1,$-\sqrt{3}$,-1	0	136	64.0	109
B,1,$\sqrt{3}$,1	20	155	64.0	255
B,1,$\sqrt{3}$,-1	20	157	64.0	109
B,1,$\sqrt{3}$,0	20	155	64.0	182

Table XIV

Spinning Conditions and Samples Collected for Test V

Composition of Polymer Solution = 60.0% Polypropylene

Cabinet Temperature = 25°C

Minimum Solubility Temperature (T_s) = 155°C

Range of Bath Temperatures (T) = 155-175°C

Sample No.	$T-T_s$ (°C)	Spinneret Temp. (°C)	Motor Shaft Speed (sec/rev)	Take-up Speed (rpm)
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	20	159	64.0	308
A,$\sqrt{3}$,$\sqrt{3}$,0	20	159	65.0	182
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	20	152	66.0	56
AB,$\sqrt{3}$,$\sqrt{3}$, 1	20	159	66.0	255
AB,$\sqrt{3}$,$\sqrt{3}$,-1	20	159	66.0	109
A,$\sqrt{3}$, 0, 0	10	147	63.0	182
A,$\sqrt{3}$, 0,$\sqrt{3}$	10	146	64.0	308
A,$\sqrt{3}$, 0, -$\sqrt{3}$	10	147	65.0	56
AB,$\sqrt{3}$, 0, 1	10	147	66.0	255
AB,$\sqrt{3}$, 0, -1	10	147	66.0	109
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	0	140	66.0	56
A,$\sqrt{3}$,-$\sqrt{3}$, 0	0	141	64.0	182
AB,$\sqrt{3}$,-$\sqrt{3}$, -1	0	141	64.0	109

Table XV-A

Extraction Data

Sample No.	1st. Extr. Time (sec)	Wt. of Sample Before Extr. (grams)	Wt. of Sample After Extr. (grams)	Final Wt. of Sample (grams)
A*,0,$\sqrt{3}$,0	10	0.1609	0.086	0.0814
	30	0.1985	0.106	0.1030
	60	0.1485	0.077	0.0765
	180	0.1370	0.070	0.0691
A*,0,$\sqrt{3}$,$\sqrt{3}$	10	0.0795	0.047	0.0464
	30	0.1444	0.0777	0.0767
	60	0.1149	0.063	0.0625
	180	0.1627	0.088	0.0819
A*,0,$\sqrt{3}$,-$\sqrt{3}$	10	0.2086	0.122	0.1169
	30	0.3375	0.184	0.1796
	60	0.2476	0.140	0.1397
	180	0.3007	0.174	0.1728
B,0,1,0	10	0.0510	0.031	0.0298
	30	0.0560	0.035	0.0295
	60	0.0740	0.046	0.0457
	180	0.1246	0.075	0.0745

Table XV-A (continued)

Sample No.	1st. Extr. Time (sec)	Wt. of Sample Before Extr. (grams)	Wt. of Sample After Extr. (grams)	Final Wt. of Sample (grams)
B,0,0,0	10	0.0647	0.043	0.0424
	30	0.1429	0.091	0.0889
	60	0.1138	0.071	0.0710
	180	0.0729	0.042	0.0474
B,0,-1,0	10	0.0186	0.013	0.0127
	30	0.0663	0.042	0.0423
	60	0.1129	0.074	0.0732
	180	0.0907	0.0596	0.0592
A,0,-$\sqrt{3}$,0	10	0.2122	0.137	0.1298
	30	0.0905	0.057	0.0573
	60	0.0653	0.042	0.0424
	180	0.1069	0.067	0.0674

the spinneret was:

$$\text{Linear Velocity} = \frac{(0.998)^2}{(0.040)^2} \ (.000435) = .271 \text{ m/min}$$

Take-up Speed. This is the tangential speed at which the take-up drum moves. The mean value of the circumference of the cardboard drums was 0.805 m., the ratio of the actual drum speed to the speed measured in the hand tachometer was 2.342. For Sample No. A,0,0,0 the take-up speed was 182 revolutions per minute. The take-up speed was calculated as follows:

$$\text{Take-up speed} = (.805)(182)(2.342) = 343 \text{ m/min}$$

Draw Ratio. The draw ratio is defined as the take-up speed divided by the linear velocity in the spinneret hole. Again, for Sample No. A,0,0,0 it was calculated to be:

$$\text{Draw Ratio} = \frac{343 \text{ m/min}}{0.271 \text{ m/min}} = 1266$$

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

$$\text{Denier} = \frac{900{,}000 \text{ (Weight of fibers, gr)}}{\text{(Length of each fiber, cm)(No. of fibers)}}$$

Substituting data for Sample No. A,0,0,0, from Table XV

$$\text{Denier} = 900{,}000 \ \frac{(.0088)}{(39.4)(61)} = 3.30$$

Void Fraction. The void fraction was calculated from the following equation

$$\text{Denier} = d \times \frac{(\pi D^2)}{4}$$

where: d = Density of pure polymer, gr/cm^3. This value was assumed to lie between .91 and .89

x = Fraction of volume occupied by the polymer

D = Mean diameter of the fiber sample, cm

and denier is expressed as a linear density measure in gr/900,000 cm.

Substituting data from Tables IV and XVI for Sample No. A,0,0,0:

$$\text{Void fraction} = 1 - X = 1 - \frac{(3.3)(4)}{(900{,}000)(d)(\pi)(.00311)^2}$$

$$= 1 - 0.482/d$$

For d = .91 Void fraction = 0.47

For d = .89 = 0.46

Tenacity. Tenacity at break for fibers is calculated as follows:

$$\text{Tenacity} = \frac{\text{Force at break per fiber, gm}}{\text{(Denier of drawn fiber, den)}}$$

From Table XVII for Sample No. A,0,0,0, the breaking strength was measured as 340 gms. Since 61 fibers were used, the breaking strength per fiber was:

$$\text{Breaking Strength per fiber} = \frac{340}{61} = 5.58 \text{ grms}$$

From Table IV the denier of the undrawn fiber was 3.30. Since the fiber was drawn 4 to 1 or 300%, the denier of the drawn fiber was one-fourth of this or .825. Thus, the tenacity was calculated as follows:

$$\text{Tenacity} = \frac{5.58}{.825} = 6.75 \text{ gr/den}$$

Table XV

Data From Denier Measurements

Sample No.	Length per fiber (cm)	No. of Fibers	Total Weight of Sample (gr)
A,$\sqrt{3}$,-0,$\sqrt{3}$	36.1	24	.0029
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	32.6	27	.0039
A,$\sqrt{3}$,-$\sqrt{3}$,0	31.3	27	.0043
A,$\sqrt{3}$,0,0	30.9	46	.0076
A,$\sqrt{3}$,$\sqrt{3}$,0	31.2	110	.0163
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	29.7	31	.0143
A,$\sqrt{3}$,0,-$\sqrt{3}$	38.5	34	.0200
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	33.7	26	.01265
AB,$\sqrt{3}$,0,1	60.9	16	.0035
AB,$\sqrt{3}$,$\sqrt{3}$,1	57.5	30	.0057
AB,$\sqrt{3}$,0,-1	57.6	37	.0182
AB,$\sqrt{3}$,$\sqrt{3}$,-1	56.5	33	.0160
AB,1,$\sqrt{3}$,1	28.7	93	.0114
B,1,1,1	24.7	31	.0030
B,1,0,1	24.7	187	.0218
B,1,-1,1	28.7	20	.0021
AB,1,$\sqrt{3}$,0	22.3	150	.0167
B,1,1,0	23.9	101	.0138
B,1,0,0	23.9	179	.0244
B,1,-1,0	23.5	68	.0068
AB,1,-$\sqrt{3}$,0	25.8	130	.0164
AB,1,$\sqrt{3}$,-1	29.3	103	.0242
B,1,1,-1	29.4	140	.0363
B,1,0,-1	25.1	100	.0212
B,1,-1,-1	27.7	91	.0203
AB,1,-$\sqrt{3}$,-1	29.1	96	.0217
A,0,$\sqrt{3}$,-.4	34.6	60	.0134
AB,0,0,0	38.9	52	.0080
A,0,-$\sqrt{3}$,0	38.7	16	.0026
B,0,1,0	39.5	32	.0048
A,0,0,0	39.4	61	.0088
B,0,0,0	37.9	58	.0030
B,0,-1,0	42.4	48	.0087
B,0,0,-1	44.6	39	.0129
B,0,-1,-1	41.1	31	.0081
B,0,1,-1	38.9	25	.0054

Table XV (continued)

Sample No.	Length per Fiber (cm)	No. of Fibers	Total Weight of Sample (gr)
B,0,0,1	37.1	41	.0047
B,0,-1,1	40.1	19	.0020
B,0,1,1	39.7	112	.0135
A,0,0,$-\sqrt{3}$	36.7	19	.0090
AB,0,$\sqrt{3}$,-2.1	37.6	22	.0188
A,0,$-\sqrt{3}$,$-\sqrt{3}$	38.8	24	.0112
A,0,0,$\sqrt{3}$	34.3	21	.0016
A,0,$\sqrt{3}$,-1.2	44.5	46	.0078
B,-1,1,1	33.5	95	.0080
B,-1,-1,1	34.1	96	.0072
B,-1,0,1	30.8	78	.0055
B,-1,1,0	34.6	63	.0056
B,-1,0,0	38.3	101	.0116
B,-1,-1,0	34.0	124	.0126
B,-1,1,-1	33.6	46	.0071
B,-1,0,-1	35.6	65	.0108
B,-1,-1,-1	32.2	58	.0074
AB,-1,1,$\sqrt{3}$	59.2	36	.0034
AB,-1,0,$\sqrt{3}$	57.7	77	.0076
AB,-1,-1,$\sqrt{3}$	59.0	67	.0068
AB,-1,1,$-\sqrt{3}$	59.2	17	.0092
AB,-1,0,$-\sqrt{3}$	59.1	23	.0127
AB,-1,-1,$-\sqrt{3}$	59.8	24	.0141
A,$-\sqrt{3}$,$-\sqrt{3}$,0	29.4	72	.0054
A,$-\sqrt{3}$,$-\sqrt{3}$,$-\sqrt{3}$	26.2	30	.0044
A,$-\sqrt{3}$,$-\sqrt{3}$,$\sqrt{3}$	32.1	68	.0035
A,$-\sqrt{3}$,0,$\sqrt{3}$	26.7	53	.0018
A,$-\sqrt{3}$,0,0	32.6	45	.0031
A,$-\sqrt{3}$,0,$-\sqrt{3}$	35.1	29	.007
A,$-\sqrt{3}$,$\sqrt{3}$,$-\sqrt{3}$	35.6	28	.0061
A,$-\sqrt{3}$,$\sqrt{3}$,0	34.6	30	.0025
A,$-\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	37.1	70	.0032
AB,$-\sqrt{3}$,$\sqrt{3}$,1	77.6	38	.0045
AB,$-\sqrt{3}$,0,1	77.7	40	.0049
AB,$-\sqrt{3}$,$-\sqrt{3}$,1	73.3	68	.0077
AB,$-\sqrt{3}$,$\sqrt{3}$,-1	77.8	20	.00520
AB,$-\sqrt{3}$,0,-1	73.7	17	.0046
AB,$-\sqrt{3}$,$-\sqrt{3}$,-1	70.9	44	.0121

Table XVI

Data on Fiber Diameter

Sample No.	Mean Diameter (μ)	Standard Deviation (μ)
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	27.8	0.8
A,$\sqrt{3}$,0,$\sqrt{3}$	27.2	1.5
A,$\sqrt{3}$,$\sqrt{3}$,0	28.8	1.4
A,$\sqrt{3}$,0,0	32.7	2.2
A,$\sqrt{3}$,-$\sqrt{3}$,0	31.0	1.2
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	54.3	2.2
A,$\sqrt{3}$,0,-$\sqrt{3}$	55.3	2.4
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	55.0	2.4
B,1,1,1	30.4	1.7
B,1,0,1	30.5	0.9
B,1,-1,1	26.2	0.8
B,1,1,0	35.8	2.3
B,1,0,0	35.2	1.7
B,1,-1,0	29.2	1.5
B,1,1,-1	40.5	1.8
B,1,0,-1	42.1	1.7
B,1,-1,-1	40.8	1.5
B,-1,1,1	28.6	1.0
B,-1,0,1	25.4	1.0
B,-1,-1,1	30.0	1.9
B,-1,1,0	31.5	1.5
B,-1,0,0	32.1	1.1
B,-1,-1,0	31.2	1.2
B,-1,1,-1	38.9	1.2
B,-1,0,-1	37.1	1.6
B,-1,-1,-1	36.0	1.4
A,-$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	23.8	1.5
A,-$\sqrt{3}$,0,$\sqrt{3}$	23.7	1.5
A,-$\sqrt{3}$,-$\sqrt{3}$,$\sqrt{3}$	23.3	1.1
A,-$\sqrt{3}$,$\sqrt{3}$,0	31.0	2.1
A,-$\sqrt{3}$,0,0	30.1	1.6
A,$\sqrt{3}$,-$\sqrt{3}$,0	30.9	1.3
A,-$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	50.6	2.9
A,-$\sqrt{3}$,0,-$\sqrt{3}$	50.2	2.4
A,-$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	49.5	2.2

Table XVI (continued)

Sample No.	Mean Diameter (μ)	Standard Deviation (μ)
A,0,0,$\sqrt{3}$	24.4	1.1
B,0,1,1	26.5	1.6
B,0,0,1	26.2	1.0
B,0,1,0	30.1	0.9
A,0,0,0	31.1	1.2
AB,0,0,0	30.0	1.6
B,0,-1,0	33.2	0.9
A,0,-$\sqrt{3}$,0	30.8	1.3
B,0,1,-1	37.5	0.8
B,0,0,-1	44.8	1.0
B,0,-1,-1	38.5	1.9
A,0,0,-$\sqrt{3}$	55.9	3.0
A,0,-$\sqrt{3}$,-$\sqrt{3}$	54.0	1.9
A,0,$\sqrt{3}$,-.4	33.6	1.5
A,0,$\sqrt{3}$,-1.2	41.6	1.1
A,0,$\sqrt{3}$,-2.1	76.6	2.3

Table XVII

Data on Tensile Measurements Fibers Drawn 4/1. Initial Rate of Extension During Draw and During Testing 100%/min

Sample No.	No. of Fibers	Initial Slope gr/min	Force at Break gr
AB,-1,-1,$\sqrt{3}$	67	313	93
AB,-1,0,$\sqrt{3}$	77	346	106
AB,-1,1,$\sqrt{3}$	36	152	44.5
B,-1,-1,0	123	514	197
B,-1,0,1	77	373	140
B,-1,1,1	93	464	154
B,-1,0,0	101	541	200
B,-1,1,1	94	436	151
B,-1,1,-1	46	348	119
AB,-1,1,-$\sqrt{3}$	16	239	75
AB,-1,0,-$\sqrt{3}$	23	366	103
AB,-1,-1,-$\sqrt{3}$	23	345	100
B,-1,1,0	63	318	115
B,-1,-1,-1	57	411	148
B,-1,0,-1	64	493	165
A,-$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	69	160	53
A,-$\sqrt{3}$,0,0	45	178	53
A,-$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	21	225	46
A,-$\sqrt{3}$,-$\sqrt{3}$,$\sqrt{3}$	68	214	79
A,-$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	30	309	70
A,-$\sqrt{3}$,-$\sqrt{3}$,0	71	280	97
A,-$\sqrt{3}$,$\sqrt{3}$,0	30	123	37
A,-$\sqrt{3}$,0,-$\sqrt{3}$	29	254	56
A,-$\sqrt{3}$,0,$\sqrt{3}$	52	130	45
AB,1,-$\sqrt{3}$,-1	96	1500	466
B,1,-1,-1	91	1510	510
B,1,1,-1	140	2420	815
AB,1,$\sqrt{3}$,-1	103	1735	578
AB,1,-$\sqrt{3}$,0	130	1300	426
B,1,-1,0	68	680	200
B,1,0,0	179	2270	741
B,1,1,0	101	1430	422
AB,1,$\sqrt{3}$,0	150	1695	509
B,1,-1,1	20	200	53
B,1,0,1	187	2010	660
B,1,1,1	31	385	113
AB,1,$\sqrt{3}$,1	93	984	297
B,1,0,-1	100	1660	484

Table XVII (continued)

Sample No.	No. of Fibers	Initial Slope gr/min	Force at Break gr
AB,$\sqrt{3}$,0,1	12	148	43
AB,$\sqrt{3}$,$\sqrt{3}$,1	30	349	94
A,$\sqrt{3}$,0,$\sqrt{3}$	22	983	72
A,$\sqrt{3}$,$\sqrt{3}$,$\sqrt{3}$	27	392	109
A,$\sqrt{3}$,-$\sqrt{3}$,-$\sqrt{3}$	31	1030	317
A,$\sqrt{3}$,0,-$\sqrt{3}$	34	1098	319
A,$\sqrt{3}$,$\sqrt{3}$,-$\sqrt{3}$	26	614	283
AB,$\sqrt{3}$,-$\sqrt{3}$,-1	9	223	70
AB,$\sqrt{3}$,0,-1	37	796	272
AB,$\sqrt{3}$,$\sqrt{3}$,-1	33	705	245
A,$\sqrt{3}$,-$\sqrt{3}$,0	27	356	125
A,$\sqrt{3}$,0,0	46	634	195
A,0,$\sqrt{3}$,-.4	19	650	260
A,0,0,$\sqrt{3}$	21	344	94
B,0,1,1	112	1575	560
B,0,0,1	41	720	240
B,0,-1,1	19	340	100
A,0,$\sqrt{3}$,-1.2	60	1240	435
A,0,0,0	61	1010	340
B,0,0,0	58	1000	370
AB,0,0,0	52	945	345
B,0,1,0	31	478	184
B,0,-1,0	48	1050	335
A,0,-$\sqrt{3}$,0	15	385	112
B,0,1,-1	25	565	215
B,0,0,-1	39	1020	390
B,0,-1,-1	31	670	280
A,0,$\sqrt{3}$,-2.1	13	780	185
A,0,0,-$\sqrt{3}$	19	865	315
A,0,-$\sqrt{3}$,-$\sqrt{3}$	24	1025	360

Table XVIII

Data on Tensile Measurements. Fibers Cold Drawn

Sample No.	No. of Fibers	Initial Rate of Extension %/min	Draw	Initial Slope (gr/min)	Force at Break (gr)
A,0,0,0	67	100	6/1	1770	396
B,0,0,0	115	100	6/1	2920	820
AB,0,0,0	131	100	6/1	3240	970
A,0,0,0	67	200	4/1	2240	400
B,0,0,0	115	200	4/1	4530	815
AB,0,0,0	131	200	4/1	5200	950
A,0,0,0	67	200	6/1	3140	400
B,0,0,0	115	200	6/1	5900	850
AB,0,0,0	131	200	6/1	6950	1040
AB,0,0,0	126	100	8/1	3040	995

Elongation at Break. The percent elongation at break was measured directly from the recording chart attached to the Instron Tensile B Tester. Thus, no sample calculation of this property is presented.

Initial Modulus. This property is measured as the slope of the stress-strain curve at a strain infinitesimally greater than zero. As discussed previously, a deviation occurred for elongation below 1%, thus the initial modulus was defined as:

$$\text{Initial Modulus} = \frac{\text{Slope of force vs. time curve, between 2\% elongation and the beginning of "hardening" region, gr/min (100)}}{(\text{Initial Rate of Extension, \%/min})(\text{Denier of Sample, den})}$$

For Sample No. A,0,0,0, from Table XVII the initial modulus was calculated as follows

$$\text{Initial Modulus} = \frac{1010}{(100)(\frac{61 \times 3.30}{4})}(100) = 20.0 \text{ gr/den}$$

Materials, After Williams[26]

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. Specification 6501. Donated by Hercules Co. 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 apparati 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 H20. Capacity 160 grams. No. A70269. 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.

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

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. 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 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 operating 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.

The piston itself was machined from an aluminum block. It is two inches long and 0.995 inches in diameter. Grooves for Parker No. 2-210 O-rings were cut 3/8 inch from each end and about 3/4 inch from the bottom end. The piston is hollow and fits on a 2 1/4 inch long universal joint, held in place by two 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 1/32 inch thick, 3/4 inch diameter disc which is welded to a 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 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 1/16 inch of displacement for valve operation.

The universal joint is connected to a 16 3/4 inch long, 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 1/2 inch diameter, 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 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.

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 600 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.

3. Drive - Zeromax Model M14R, Type A. Ten inch pounds torque, 0-400 rpm. Manufactured by Zero-Max Co., Minneapolis, Miss. 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-Nimo 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. Instron Tensile Tester - Manufactured by Instron Co. Property of Engineering Mechanics Department, Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

7. 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.

8. 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.

9. 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.

10. 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.

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

12. 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.

13. 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.

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PHASE SEPARATION SPINNING OF POLYPROPYLENE FIBERS

by

Carlos A. Andrade

(ABSTRACT)

Polypropylene fibers were spun from solutions of different polymer contents undergoing phase separation. The range of concentrations used was 25 to 60 wt. % polypropylene. The solvent used was naphthalene. After the naphthalene was extracted from the fibers using diethyl ether the fiber's properties were measured.

Deniers and diameters of the fibers varied inversely with the spinning draw ratio. The fibers had void fractions ranging from 0.20 to 0.70. The porosity of the fibers was inversely proportional to the polymer contents of the spinning solution.

Tenacities and moduli ranging from 1.5 to 8.5 and from 6 to 17 grams per denier, respectively, were measured after the fibers were cold drawn 4/1. These values varied directly with spinning draw ratio. Percent elongation at break of fibers spun from solutions of polymer contents greater than 35 wt. % polypropylene varied inversely with draw ratio. Fibers spun from solutions of less are 35 polymer contents showed elongations at break which varied directly with draw ratio for draw ratios of less than about 1500. For higher draw ratios and inverse relationship was observed.

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