

Processing and Properties of Poly(*p*-Phenylene Benzobisthiazole) / Nylon Fibers

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Synopsis

Composite fibers of poly (*p*-phenylene benzobisthiazole) (PBT) with nylons were spun from dilute acid solutions. The effects of wet-stretching, heat treatment time, tension, and temperature on the tensile properties are reported. Nylon 6,6 and nylon 6 at several molecular weights were studied. Moduli of 40 GPa and tensile strengths of 375 MPa were achieved for 30/70 PBT/nylon composites. Heat treatment of the nylon/PBT fibers at 160–225°C for 12–19 h increased the tensile modulus by 20–50% and the tensile strength by a smaller amount. At the same time, the intrinsic viscosity of the nylons increased as much as 100%, indicating the solid-state polymerization of the nylon. The largest tensile modulus attained is less than half the theoretical value predicted by a linear “rule of mixtures” as might be expected for an oriented molecular composite. Although differential scanning calorimetry shows a melting transition at temperatures 5–10°C higher than the pure nylons, the composite does not flow at temperatures above this transition. Sulfuric acid dissolves most of the nylon, but does not destroy the mechanical integrity of the fibers; differential scanning calorimetry indicates that the remaining fiber contains little or no nylon. The results are consistent with a microstructure consisting of a microfibrillar network of PBT, surrounded by a separate nylon phase.

INTRODUCTION

The tensile properties of a polymeric material improve with the addition of reinforcing fibers; increasing the aspect ratio of the reinforcing fibers at constant concentration also improves tensile properties.^{1–4} The influence of reinforcing fibers on the matrix also depends on the strength of the interfacial bond between the fiber and the matrix. At a critical aspect ratio, the load on the composite will be shared equally between the fiber and the matrix, and the force required to pull the fiber out of the matrix will be greater than the strength of the reinforcing fiber. Below this critical aspect ratio, the fiber contributes little to the composite strength and failure occurs when the fiber is pulled from the matrix.³ Finally, long fibers reduce the stress concentration, and increase the probability of deflecting crack growth.⁵ A highly desirable reinforcing fiber, therefore, would seem to be a single, extended, rodlike polymer molecule.⁶ This is the basis of the “molecular composite” concept.

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Rodlike, extended-chain molecules that could be used for these molecular composites have been synthesized and studied.⁷⁻⁹ One such material is poly(*p*-phenylene benzobisthiazole) (PBT), a wholly aromatic, heterocyclic polymer. PBT degrades before melting, has a high thermal and oxidative stability (weight retention of 50% after 200 h at 371°C in circulating air⁶), and is soluble only in strong acid solvents.¹⁰⁻¹² A blend of PBT with a "matrix" polymer is the desired material for a "molecular composite," but the lack of a melting point and a limited solubility dictates the use of solution processing in strong acids. Furthermore, the existence of a nematic liquid crystalline phase transition requires that the solutions be dilute to avoid large scale phase separation.

The theory of Halpin and Tsai^{13,14} has been used to estimate the composite tensile modulus from the properties of the components. For large aspect ratios, the composite modulus in a highly oriented sample is predicted to follow a linear "rule of mixtures," i.e., the composite modulus is a linear combination of the rod and the matrix properties weighted by volume fraction. Such reinforcement has been reported for composites of PBT in aromatic, heterocyclic matrix polymers,¹⁵⁻¹⁷ and there has been a recent effort to achieve this with thermoplastic matrix materials.^{5,18} Of course, such reinforcement is a necessary but not sufficient test for the existence of a composite on a molecular level.

We report here on the tensile properties and processing of composite fibers made from nylons and PBT. In the next section, we describe the particular materials used for this study, the processing conditions and the techniques used for post-treatment and mechanical property measurements. The following section summarizes typical tensile properties and the effects of wet stretching and heat-treating the composite fibers after spinning. Following this, we report some of the variations of properties with the matrix molecular weight and the effects of more extensive heat treatment. We interpret the results in a final section, point out the possibility of and evidence for a structure other than the molecular composite in the PBT/nylon materials.

TABLE I
The Effect of Wet Stretching on the Tensile Properties of PBT/Nylon Fibers

	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at break (%)
<i>Nylon 6,6 (13,700) / PBT</i>			
As Spun	5.3	102.3	30.8
Wet Stretched, 40% draw	7.0	156.0	21.4
<i>Nylon 6,6 (17,500) / PBT</i>			
As Spun	6.1	113.5	23.3
Wet Stretched, 10% draw	8.4	150.2	20.2
<i>Nylon 6 (14,500) / PBT</i>			
As Spun	3.2	64.5	2.6
Wet Stretched, 10% draw	5.2	127.5	37.9
<i>Nylon 6 (20,000) / PBT</i>			
As Spun	2.9	63.5	2.9
Wet Stretched, 100% draw	5.9	141.2	17.0

EXPERIMENTAL

Poly(*p*-phenylene benzobisthiazole) (PBT) was prepared by SRI International as described elsewhere.¹⁹ The polymer had an intrinsic viscosity of 25 dL/g in dilute methane sulfonic acid solution, corresponding to a weight average molecular weight of 36,400 according to a correlation developed by Berry et al.²⁰ The PBT was received as an anisotropic solution in polyphosphoric acid and was extracted in water, washed with methanol, and thoroughly dried. Nylon 6 and 6,6 were obtained from Polysciences and DuPont. Two samples of nylon 6 with number average molecular weights of 14,500 and 20,000 and of nylon 6,6 with number average molecular weights of 13,700 and 17,500 were used. Except for drying, the nylons were used as received.

The proportions of polymer were 30 wt % PBT and 70 wt % nylon in the work reported here. The concentration of polymer in the methane sulfonic acid (98%, Aldrich Chemical Co.) solution was 3.5 wt % for nylon 6 and 3.7 wt % for nylon 6,6. These concentrations were close to but slightly less than the critical values for the isotropic-nematic transition, as determined by optical microscopy.

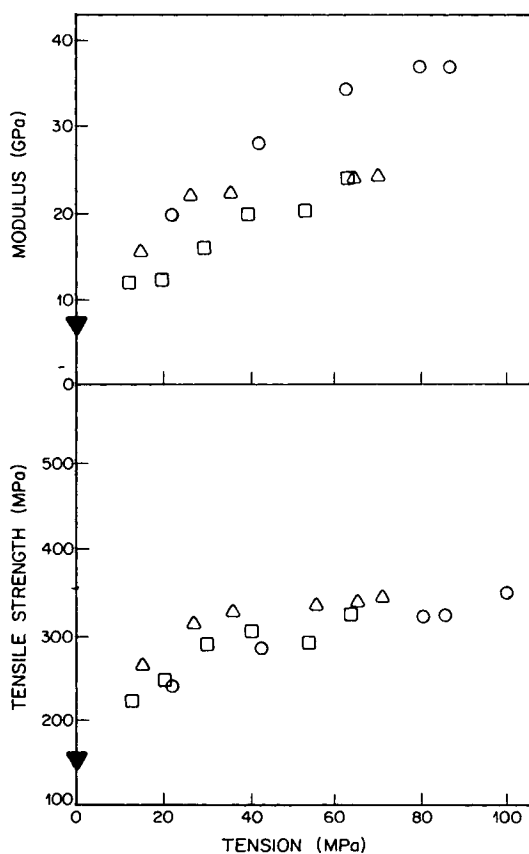


Fig. 1. Tensile properties as a function of the heat treatment time and tension for nylon 6,6 (13,700)/PBT fibers heat treated at 225°C: (○) 0.5 min; (△) 1.0 min; (□) 2.0 min; (▼) wet-stretched.

Fibers were spun through a die with a single $330\ \mu\text{m}$ diameter hole into a water bath at approximately 20°C . All solutions were filtered during the spinning process with a 7X Dynalloy micromesh filter. A constant extrusion rate of $2.8\ \text{cm/h}$ was used, corresponding to a fiber velocity of $13\ \text{cm/min}$. No tension was applied during take-up, but after coagulation the fibers were wet-stretched between corotating rollers at draw ratios of 1.1–2.0. Heat treatment was done under dry nitrogen at 215, 255, or 295°C for residence times of 0.5, 1, and 2 min. Tensions in the range of 10–100 MPa were applied during heat treating with the highest value corresponding to fiber breakage.

Composite fibers of nylon 6/PBT (nylon molecular weight 14,500) and nylon 6,6/PBT (nylon molecular weight 13,700) and solution spun fibers of the corresponding pure nylons were used for extended heat treatment studies. The composite fibers were solution-spun, wet-stretched, and heat-treated under tension before being exposed to extended heat treatment. The nylon fibers were tested without additional stretching or heat treatment. Nylon 6 and 6,6 pellets that had not been dissolved in acid were also subjected to the extended heat treatment. Temperatures of 170 and 220°C were used for the nylon 6,6 samples; nylon 6 was treated at 160 and 225°C . The nylon 6,6 and

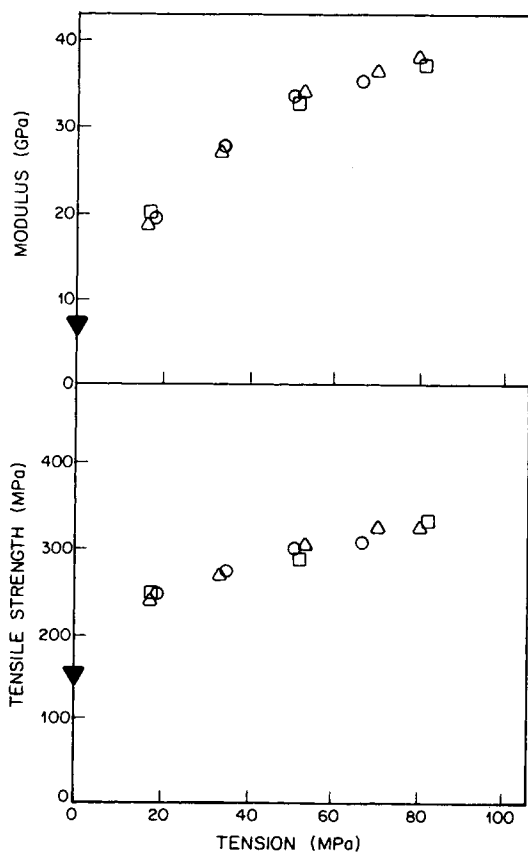


Fig. 2. Tensile properties as a function of the heat treatment time and tension for nylon 6,6 (17,500)/PBT fibers heat treated at 295°C : (○) 0.5 min; (△) 1.0 min; (□) 2.0 min; (▼) wet-stretched.

composite fibers were placed in an oven under vacuum for 12 h while the nylon 6 and composite fibers were treated for 17–19 h. Intrinsic viscosities in formic acid were measured after the extended heat treatment.

Fibers of nylon 6 (20,000)/PBT that were heat treated for 1 min were soaked in sulfuric acid. The two samples had been heat-treated at tensions of 86 and 55 MPa and the sulfuric acid was 98% (Fisher Chemical Co.), a solvent for nylons but not for PBT.²¹ The composite fiber (heat treated at 86 MPa) was placed loosely in a beaker of sulfuric acid, and allowed to remain for 12 days. The fiber was then held at constant length, washed in water for 2 days, and then dried in air. The fiber which had been heat treated at 55 MPa was held under tension during extraction for 7 days, in contrast to the first sample. The fiber was then washed without tension, held at constant length, and dried in air.

Fiber samples with a gauge length of 3.0 cm were tested on an Instron Universal Testing Machine at a crosshead speed of 0.1 cm/min, under ambient conditions. Samples were mounted on paper tabs with Devcon 5-min epoxy and then cured overnight. Diameters were measured with an optical microscope under 200 \times magnification. The mechanical properties reported are the average of seven samples.

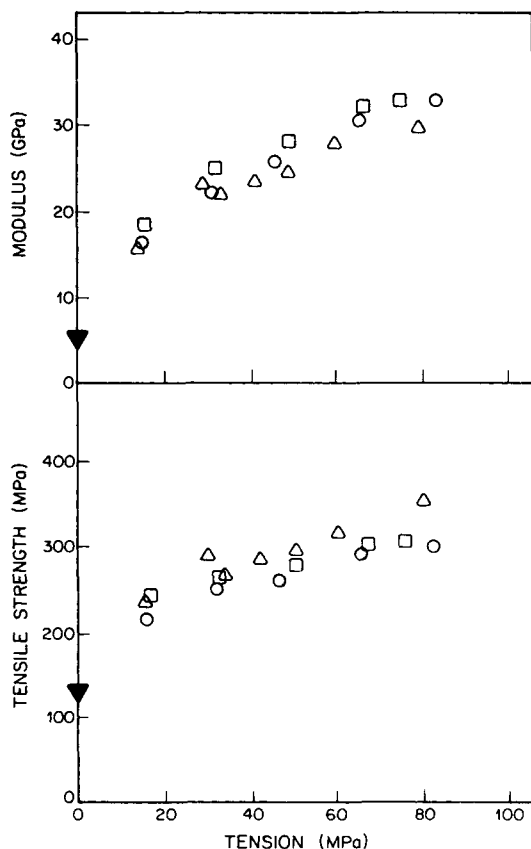


Fig. 3. Tensile properties as a function of the heat treatment time and tension for nylon 6 (14,500)/PBT fibers heat treated at 215°C: (○) 0.5 min; (△) 1.0 min; (□) 2.0 min; (▼) wet-stretched.

RESULTS

The influence of wet stretching, heat treatment time, heat treatment tension, and temperature on the mechanical properties were investigated. The effect of matrix molecular weight on the tensile properties was also studied.

The solutions lacked sufficient viscosity to allow stable drawing during the spinning process, and no stretching was applied during the wet-spinning process. Wet-spun fibers generally exhibit low mechanical properties compared with dry-jet wet-spun fibers or melt-spun fibers,^{22,23} and this would be expected in the PBT/nylon system due primarily to a lack of orientation. However, wet stretching after coagulation provides an orienting step in these composites.

Table I shows substantial increases in the tensile modulus and strength as a result of wet stretching. The nylon 6,6/PBT fibers were subjected to a low degree of wet stretching, and yet increases of 32–38% are seen in the moduli and 32–53% in the tensile strengths. The elongation at break drops by 10–30% as a result of wet stretching, indicating a greater orientation in the fibers. Nylon 6/PBT fibers were subjected to a higher degree of wet stretching (as measured by a decrease in the cross sectional area to one half the original).

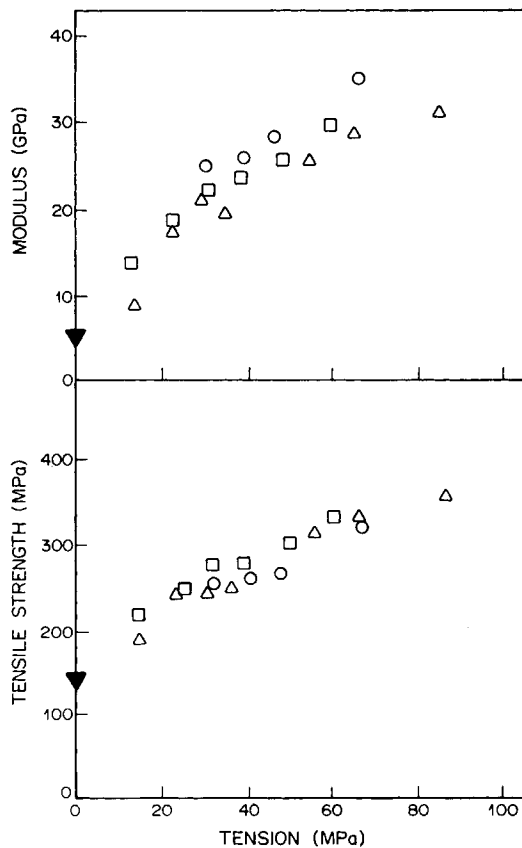


Fig. 4. Tensile properties as a function of the heat treatment time and tension for nylon 6 (20,000)/PBT fibers heat treated at 255°C: (○) 0.5 min; (Δ) 1.0 min; (□) 2.0 min; (▼) wet stretched.

Increases of 63–123% in the moduli and tensile strengths were found. The as-spun nylon 6/PBT fibers are brittle compared to the nylon 6,6/PBT fibers, but the strength and strain at break increased dramatically by wet-stretching.

Nylon/PBT composite fibers were heat treated under tension at 215, 255, and 295°C for 0.5, 1.0, and 2.0 min. Tension was applied at increments increasing from 10 up to at most 100 MPa, until the limiting value for fiber breakage was reached. The standard deviation of the data was approximately 6%.

The tensile properties as a function of heat treatment time and tension for nylon 6,6/PBT are shown in Figures 1 and 2. The fibers containing lower molecular weight nylon (13,700) and heat treated at 255°C show some sensitivity to the residence time as seen in Figure 1. Heat treatment for 0.5 min results in a 25–30% improvement in modulus over heat treatment for 1 or 2 min. However, the longer times result in 5–10% higher tensile strength values. The tensile properties for the fibers containing a higher molecular weight nylon (17,500) and heat-treated at 295°C are shown in Figure 2. The higher molecular weight nylon results in a diminished sensitivity to oven residence time. In fact, the moduli and tensile strengths are identical within the experimental error at the three residence times studied.

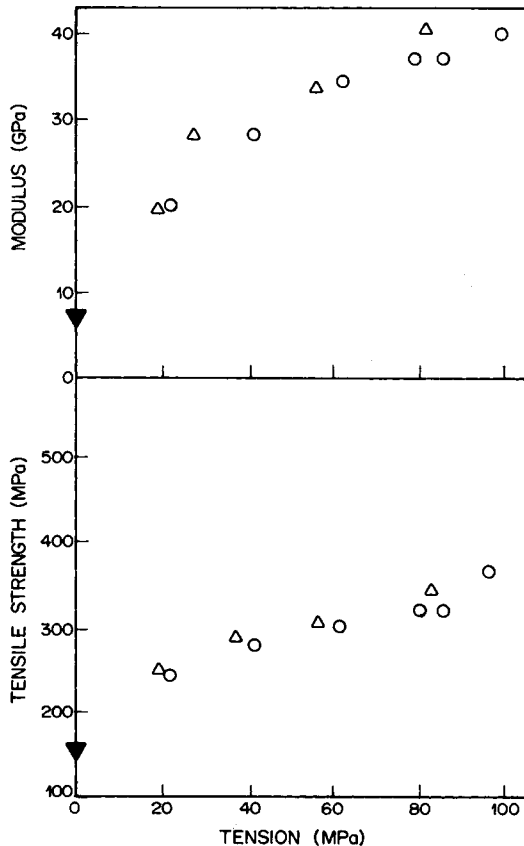


Fig. 5. Tensile properties as a function of the heat treatment temperature and tension for nylon 6,6 (13,700)/PBT fibers heat treated for 0.5 min: (○) 255°C; (Δ) 295°C; (▼) wet-stretched.

The tensile properties of nylon 6/PBT fibers are shown as a function of heat treatment tension and temperature in Figures 3 and 4. The lower molecular weight nylon (14,500) and heat treatment at 215°C gives a 5–10% higher modulus at the 2-min residence time and a 5–10% higher tensile strength at the 1-min time, indicating some sensitivity to residence time. The high molecular weight nylon (20,000) yields composite fibers that are slightly less sensitive to oven residence time. From Figure 4, the modulus is 5–15% higher at the 0.5-min time, while the tensile strengths are 5–15% higher at the 1- and 2-min times.

Tension applied during heat treatment drastically improves the modulus and, less dramatically, the tensile strength. Also, the rate of increase of modulus and tensile strength diminishes with increasing heat treatment tension and appears to reach an upper limiting value. The tensile properties are not as strong a function of residence time for the high molecular weight nylons. However, the highest tensile strengths are achieved with the higher tensions during heat treatment that can be applied at the lower residence times.

Temperatures of 255 and 295°C were chosen for heat treatment of fibers containing nylon 6,6; for nylon 6, 215 and 255°C were used.

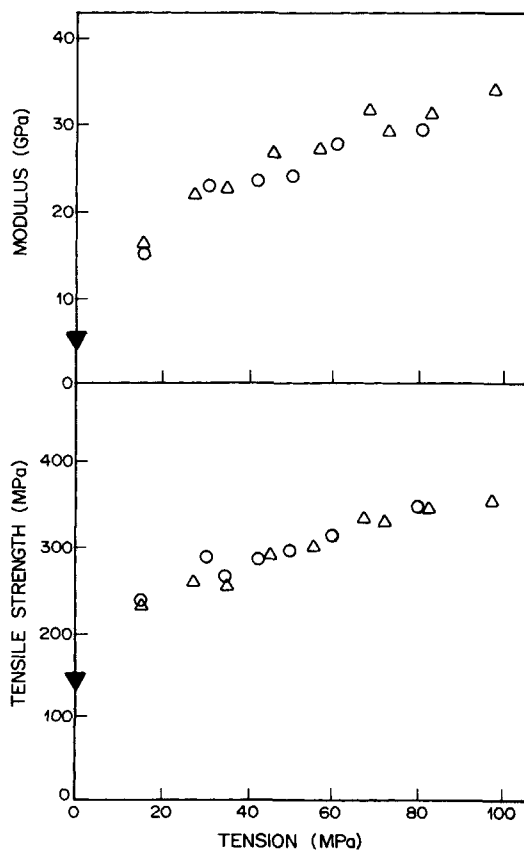


Fig. 6. Tensile properties as a function of the heat treatment temperature and tension for nylon 6 (14,500)/PBT fibers heat treated for 1.0 min: (○) 215°C; (△) 255°C; (▼) wet-stretched.

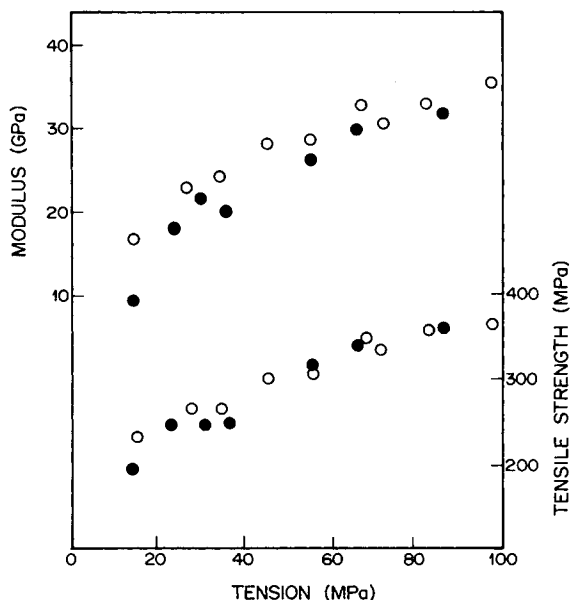


Fig. 7. The mechanical properties of nylon 6/PBT fibers as a function of the nylon molecular weight and the applied tension. The fibers were heat-treated at 255°C for 2 min: (○) 14,500 g/mol; (●) 20,000 g/mol.

Typical tensile property results are shown in Figures 5 and 6. The composite mechanical properties are not a strong function of heat treatment temperature for the residence times and the oven temperatures studied, although the properties are somewhat higher at the higher temperatures. This is perhaps unexpected because these temperatures are approximately 40°C higher than the melting point of the matrix polymers.

The tensile strength and modulus of nylon 6/PBT and nylon 6,6/PBT fibers are shown as a function of matrix molecular weight and heat treatment tension in Figures 7 and 8. The results are surprising because the properties of the fibers containing lower molecular weight nylon are equivalent or slightly higher than for the high molecular weight nylon at any tension. However, the lower molecular weight nylon fibers can be treated at higher tensions and thereby achieve the highest properties. The rate of increase of the modulus and tensile strength diminishes with increasing applied tension.

Stress-strain curves for nylon 6,6/PBT fibers before and after extended heat treatment are shown in Figure 9. Extended heat treatment results in a 23–49% higher modulus and a tensile strength increase of 1–29% without significant changes in the elongation at break. The lower temperature of 170°C results in higher tensile properties as compared to the fibers treated at 220°C or for those not heat treated for the extended period.

Increased crystallinity or solid state polymerization of the matrix could be important factors in the extended heat treatment, and a sample of nylon pellets was heat treated along with the fibers for comparison. The intrinsic viscosity of the untreated nylon pellets (measured in 88% formic acid, Fisher Chemical Co.) was compared to the value for the pellets which had undergone

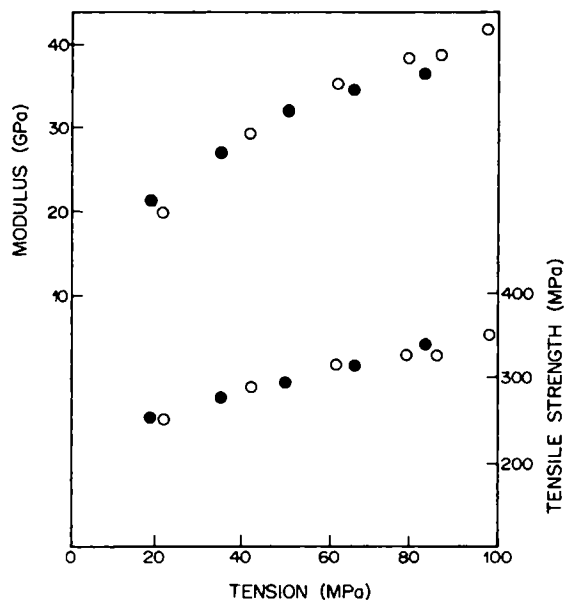


Fig. 8. The mechanical properties of nylon 6,6/PBT fibers as a function of the nylon molecular weight and the applied tension. The fibers were heated treated at 255°C for 0.5 min: (○) 13,700 g/mol; (●) 17,500 g/mol.

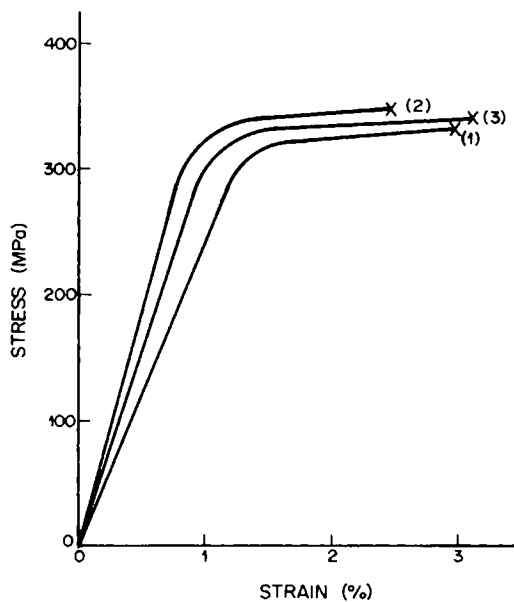


Fig. 9. Typical stress-strain curves for nylon 6,6 (13,700)/PBT fibers after heat treatment: (1) 255°C for 1.0 min at 70.9 MPa applied tension; (2) 170°C for 12 h with no tension; (3) 220°C for 12 h with no tension.

TABLE II
The Effect of Extended Heat Treatment

	Intrinsic viscosity at 25°C in 88% formic acid (dL/g)	
	Before	After
<i>Nylon 6, treated for 16 h</i>		
at 160°C	1.10	1.32
at 220°C	1.14	2.07
<i>Nylon 6,6, treated for 12 h</i>		
at 170°C	0.99	1.11
at 220°C	0.95	1.96

extended heat treatment, with the results shown in Table II. The nylon treated at 220°C shows an intrinsic viscosity that is nearly double that of the untreated material, while the pellets treated at 170°C show a smaller increase in viscosity. We interpret this as evidence for a solid state polymerization of the nylon. These results are also consistent with the finding that the lower molecular weight matrix polymer has the better tensile properties. Apparently, the higher temperature results in a higher matrix molecular weight, although these fibers exhibit lower moduli and tensile strengths than those treated at 170°C.

The nylon 6/PBT fibers, when soaked for extended periods in sulfuric acid, did not disintegrate as might be expected for a molecular composite of PBT in nylon. The fibers remained intact, presenting evidence for the existence of a network of PBT within the nylon matrix. A yellow color was noted in the sulfuric acid bath which can be attributed to the dissolution of nylon, as can the reduction of 30–40% in fiber diameter that occurred as a result of extraction of the nylon.

The extracted fibers were also tensile tested; the results are compared in Table III. The modulus decreased for the fibers heat treated at 86 MPa tension, while the tensile strength increased. The lower modulus may be explained by the fact that the fiber was not held in tension during the

TABLE III
The Effect of Extraction with Sulfuric Acid

	Diameter modulus (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at break (%)
<i>Extracted without tension</i>				
<i>heat treatment at 86 MPa</i>				
Before extraction	62.9	31.2	354.9	1.2
After extraction	43.3	25.2	467.4	7.1
<i>Extracted under tension</i>				
<i>heat treatment at 55 MPa</i>				
Before extraction	67.2	26.0	314.2	1.4
After extraction	41.8	39.3	418.6	1.4

extraction and washing process, allowing the PBT to become less oriented as the nylon was removed. This is further supported by a much larger elongation at break after extraction, indicating a less oriented fiber. In contrast, the fiber treated at 55 MPa tension was held in tension during extraction, and both the modulus and the tensile strength were increased. The values reported in Table III support this interpretation.

DISCUSSION AND CONCLUSIONS

The tensile modulus and strength of nylon/PBT fibers can be increased by more than 100% by wet-stretching; the tensile modulus of wet-stretched fibers increases by a factor of 7 and the tensile strength increases by a factor of 3 as a result of heat treatment under tension. The tensile properties are not a strong function of heat treatment time in the range 0.5–2 min, although the modulus tends to be slightly higher at shorter residence times and the tensile strength higher at longer residence times. The highest tensile strengths are achieved with high tension which is possible at the low residence times. Similarly, the mechanical properties are not a strong function of heat treatment temperature (up to 295°C) or of the nylon molecular weight. However, a higher heat treatment temperature increases the tensile strength and modulus as does a lower molecular weight matrix material. The initial increase in strength with applied tension is more rapid than that of the modulus, but at higher tensions, this trend is reversed, e.g., Figure 1. This trend would be expected if the strain energy corresponding to tensile failure were approximately constant. For geometrically similar samples in the linearly elastic region, this energy is proportional to the ratio of strength squared to twice the tensile modulus—for the samples heat-treated for 2 min shown in Figure 1, this ratio varies from 1.3 to 1.6 Pa. Similar trends are seen in Figures 2, 3, 6, and 7.

Heat treatment for an extended period also improves the tensile properties, most notably the modulus. The modulus of fibers that were heat treated for an extended period increased by 20–50% and the tensile strength improved by 3–15%. This process also increased the melting point above the value for the matrix by 5–10°C. Bulk nylons subjected to extended heat treatment show an increase in solution viscosity, probably due to solid state polymerization. Fibers that were heat-treated for the same extended periods but at a lower temperature have a smaller increase in intrinsic viscosity, yet have better properties. This is consistent with the result that the lower molecular weight nylons also yield superior tensile properties.

However, it appears that a structure different than the “molecular composite” of PBT in nylon was present. For example, the rule of mixtures estimate for the modulus of the composite fibers is 81 GPa;[†] only about half of this value could be obtained in this study, in spite of the orienting effect of wet stretching, or the use of heat treatment. In addition, when the composite fibers are soaked in sulfuric acid, a solvent for nylon, the fibers remain intact

[†]Densities of 1.56 and 1.14 g/cc for PBT and nylon 6,6 were used to estimate the volume fractions as 24 and 26%, respectively. The tensile modulus for nylon 6,6 was estimated as 2.9 GPa and a value of 330 GPa was used for PBT. The latter was taken from Ref. 24.

and differential scanning calorimetry indicates that the remaining fiber contains little or no nylon.

The results of this study are consistent with the hypothesis that a network of PBT can be formed during solution spinning of fibers from PBT/nylon/MSA solutions. In fact, a microfibrillar structure in single-component PBT fibers has recently been reported.²⁵ In the composite materials, a lower molecular weight nylon, with a lower viscosity, may allow the network to form more completely than in the higher molecular weight matrix. Likewise, higher heat treatment temperatures give rise to a more perfect network. In addition, the fibers remain intact at heat treatment temperatures which are above the nylon melting point, and, although DSC studies show a melting transition for the material, the composites do not flow.

Further support for the presence of the network is found in the tensile property data. The modulus does not follow the "rule of mixtures," and both the modulus and the tensile strength appear to reach an upper limiting value as increasing tension is applied during heat treatment.

Although it may be that the molecular composite can be attained with other matrix materials or alternative processes, it appears that a network structure of PBT in a separate nylon phase is more stable and consistent with the results of this study.

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