A Study on Blends of Nylon-6 and Nylon-66

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Synopsis

Blending of nylon-6 and nylon-66 was carried out by coextrusion in the whole range of compositions with particular emphasis on small amounts of one polymer in the other. Most significant improvement in properties is achieved at low blend compositions in which the minor component remains amorphous while severely affecting the crystallization behavior of the major component. The nylon-6-rich blends crystallize predominantly in the γ-crystalline form, whereas the nylon-66-rich blends exhibit low crystallinity. Because of this property, the blends are more easily drawable, giving rise to a more highly oriented structure with improved tensile properties and uniformity. High tenacity and modulus are observed in drawn blends containing 70% nylon-6 and 30% nylon-66.

INTRODUCTION

There are many ways of formulating a polymeric material with unique properties. Of late, polymer blending has generated considerable interest because this is an easy and economic method of tailoring a polymer to suit specific end uses. The polymers constituting a blend are usually not completely miscible. Phase segregation at some level is desirable in most cases because it may give rise to synergistic improvements in properties.

This study deals with malt blends of nylon-6 and nylon-66. Preliminary investigations[1] showed that less than 5% of one nylon in the other severely affects the crystallization of the major component without itself crystallizing. Little work has been done on nylon blends, and almost no study has dealt with small amounts of one nylon in the other. In this investigation the whole range of composition has been studied, with special emphasis on 0–10% of one component in the other.

EXPERIMENTAL

Commercial fiber-grade nylon-6 and nylon-66 chips were used. Characterization by gel permeation chromatography in hexafluoroisopropanol showed that the $\overline{M}_n$ and polydispersity values for nylon-6 and nylon-66 were, 14,600 and 2.7 and 14,900 and 2.2, respectively.[2] Blended monofilaments were obtained by coextrusion of the nylons in a specially designed melt-spinning unit incorporating a static mixing block. Fifteen blends were prepared containing 0, 2, 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 95, 98, and 100% nylon-6 by weight, respectively, the rest of the fraction being nylon-66. These were designated as B0, B2, B5, B10, etc. The as-spun filaments

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were cold-drawn to the maximum draw ratio (MDR) on a laboratory-model drawing unit. It was found that the blend filaments containing comparable amounts of the two nylons were less uniform. The sample B30 and B40 were uneven to preclude any drawing at all.

The melting and crystallization behavior of the as-spun and drawn blends was evaluated on the Du pont 990 Differential Scanning Calorimeter. A dry nitrogen atmosphere was used. The melting endotherm was obtained in the heating cycle at 10°C/min. The sample was heated up to 270°C, held for 1 min to erase the thermomechanical history, and then cooled at 10°C/min to obtain the crystallization exotherm.

Wide-angle x-ray diffractograms of powdered as-apun samples were obtained on the Philps x-ray generator using CuKα x-ray radiation of wavelength 1.54 Å. From the diffractograms, the fraction of γ-crystalline form was obtained by the method of Kyotani and Mitsuhashi.[3] The percent crystallinity (X_c) of the sample was calculated from the method suggested by Alexander.[4]

X-ray diffraction photographs of oriented bundles of drawn filaments were obtained with an exposure time of 3 h and a sample-to-film distance of 3.5 cm. The crystalline orientation function, F_c, was calculated by the method of Farrow and Bogley.[5] From the F_c and birefringence, the value of the amorphous orientation function was obtained by the method of Stein and Norris.[6]

Birefringence (Δ_n) of the drawn samples was obtained using Vicker's Polarising Microscope with a Leitz Wetzlar tilting-plate-type compensator.

The density of the as-spun and drawn samples was obtained on a Davenport density gradient column, using a density range from 1.10 to 1.25 g/cm.[3] The weight fraction density crystallinity (X_w) was calculated as

$$X_w = \frac{\rho_c(\rho - \rho_a)}{\rho(\rho_c - \rho_a)}$$

where ρ = density of the sample
ρ_a = density of amorphous nylon a (1.09 for both nylon-6 and nylon-66)
ρ_c = density of crystalline region = $1.23\alpha_c + 1.16\gamma_c$, where 1.23 and 1.16 are the densities of the amorphous and crystalline forms, and α_c and γ_c are the fractional contents, respectively. In blends samples containing more than 50% nylon-66, the γ-content was taken as zero.

The tensile properties of the as-spun and drawn blends were evaluated on the Instron model 1112 tensile tester, using a gauge length of 5 cm and a jaw speed of 10 cm/min. Sonic modulus was obtained on the PPM-5 sonic modulus tester at a tension of 5 g. Dynamic mechanical properties of the drawn filaments were measured on the Rheovibron model DDV II direct-reading viscoelastometer. Measurements were carried out for storage modulus and loss tangent (tan δ) at 30 lb, 110 Hz, from room temperature to 175°C at 3°C/min.

RESULTS AND DISCUSSION

Fusion and Crystallization

The melting and crystallization data for blend samples, as obtained by DSC, are given in Table I. In blends of nylon-6 containing up to 10% nylon-66, i.e., B98, B95, and B90, only a single melting (T_m) and crystallization (T_c) peak are observed at positions corresponding to those in B100. This implies that the minor component, nylon-66, does not crystallize but remains in the amorphous state. Therefore, it must reside in the amorphous regions of the blend or between crystalline lamellae within the spherulites of nylon-6. Such a morphology has been described by Stein et al.[7] for blends of polyethylene terephthalate–polybutylene terephthalate and atactic and isotactic polystyrene blends. Mitomo and Tonami[8] have observed that nylon-6 and nylon-66 molecules are miscible in the amorphous regions of the blend.

Nylon-66 remains in the amorphous form possible because it is present in small quantities, and therefore, during melt spinning, where crystallization occurs very rapidly, the minor component does not have time to diffuse and come together in order to crystallize. In blends with higher proportions of nylon-66, i.e., B80, B70, B60, B50, B40, and B30, nylon-6 and nylon-66 crystallize independently, as seen from Table I, were two distinct peaks are observed during melting. In blends with nylon-66 as the major component and up to 20% nylon-6, i.e., B2, B5, B10, and B20, again, only

TABLE I
Melting and Crystallization Behavior of Blends

Sample	T_m (°C)		T_c (°C)	
	Undrawn	Drawn	Undrawn	Drawn
B 100	225.0	224.0	188.5	190.0
B 98	225.0	227.0	188.0	188.0
B 95	224.0	227.0	186.5	189.0
B 90	223.0	226.0	187.5	186.5
B 80	223.5	230.0	190.0	193.0
	259.0		228.5	225.5
B 70	234.0	226.5	194.0	187.0
	262.5		232.5	
B 60	223.0	228.0	188.0	188.0
B 50	227.0	222.0	234.0	231.0
	262.0	259.0		
B 40	226.0	—	235.0	—
	262.5			
B 30	225.0	—	192.0	—
	261.5		234.5	
B 20	260.0	260.0	229.0	231.0
B 10	263.0	260.5	235.0	233.0
B 5	262.5	260.6	234.5	232.0
B 2	262.5	263.0	235.0	234.5
B 0	263.5	262.0	238.0	234.0

a single T_m and a single T_c are obtained, corresponding to those of pure nylon-66. In these blends, too, the minor component, nylon-6, does not crystallize. This is probably because of the much higher crystallization rate of nylon-66, which prevents diffusion and crystallization of nylon-6. Nylon-66 has a higher crystallization rate because it is a symmetric molecule, and at any crystallization temperature its undercooling is higher than that of nylon-6.

A slight depression in T_m and T_c is observed in the blends containing up to 20% of the minor component. A similar small depression has also been observed by Mitomo and Tomani,[8] Kitao et al.,[9] and Ke.[10] The factors involved in the depression of T_m in a compatible blend are complex. Sufficiently strong polymer–polymer interactions exist at the crystal/amorphous phase interface and should be relfected by a drop in T_m.[11] However, several other factors can also lead to a melting point depressioin. The lamellar thickness can change in blends as compared to the pure polymer prepared under identical conditions. Crystalline perfection may also be altered when crystallization occurs in the presence of a second compatible polymer. Studies on model blend systems[12,13] have shown that the physical nature of the amorphous phase surrounding the crystalline component is also an important factor, determining the experimentally observed T_m. At higher blend compositions individual melting peaks are observed for each component, with a slight depression of melting temperature only in the higher melting component. Such an effect has also been observed by Inoue.[14]

There is no clear-cut relationship trend between the T_m and T_c values of the drawn, melt-blend samples as compared to those of the undrawn ones. On the whole, the slight increase in temperature in the case of the former may be attributed to the greater order present. A superheating phenomenon in drawn filaments, due to their more compact structure and more stable crystallites, has been reported by several authors.[15-17]

Crystallinity And Density

Table II lists the percent crystallinity of the as-spun blend samples, calculated by two methods. The general trend shows that there is a significant fall in the crystalline content when nylon-6 is blended into nylon-66, but not vice versa. This may be because at the T_c of nylon-66, nylon-6 is still molten and hence restricts the crystallization of the former. However, at T_c of nylon-6, nylon-66 is far below its T_m and may even be acting as a nucleating agent[18] for nylon-6. A fall in crystallinity due to blending of nylon-6 and nylon-66 was also observed by Mitomo and Tomani.[8] They have, however, observed a linear decrease with composition, whereas in our case the decrease is maximum at small percentages of the second component.

A very interesting observation has been made regarding the crystallization behavior of blends containing nylon-6 as the major component. Although nylon-66 remains amorphous up to a 10% content in the blend, even a 2% content changes the crystalline form in which nylon-6 crystallizes. Wide-angle x-ray diffraction shows that the blend crystallizes predominantly in the γ form with a single diffraction peak at $2\theta = 21.7°$ as compared to the double peak pattern of 100% nylon-6 at $2\theta = 20°$ and $24°$.

TABLE II
Percent Crystallinity of As-spun Samples

Sample	X-ray crystallinity (%)	Weight of density crystallinity
B100	46	39
B 98	45	38
B 95	44	38
B 90	46	38
B 80	40	37
B 70	43	44
B 60	41	44
B 50	44	33
B 40	51	30
B 30	46	28
B 20	30	30
B 10	26	31
B 5	40	28
B 2	68	38
B 0	61	38

The fraction in the γ form is tabulated in Table III. Uchida et al.[19] has shown that crystallization of nylon-6 at 100°C gives a 78% γ structure, i.e., incorporation of nylon-66 has the same effect as a reduction in the crystallization temperature; in other words, the presence of nylon-66 in nylon-6 restricts the mobility of the latter.

As the content of nylon-66 increases, the fraction of the γ form falls to values closer to those in 100% nylon-6. This is due to phase segregation at higher compositions, as supported by melting data.

Dynamic Relaxation Behavior

The variation of the storage modulus, E', and that of the loss tangent, tan δ, as a function of temperature are shown in Figures 1 and 2, respectively, for selected samples. It is well known that nylon exists in a large number of crystalline orders,[20] which would lead to different levels of order in the amorphous region. This accounts for the broad relaxation observed for the nylon-6 sample (B100). A similar relaxation spectrum for nylon-6 has also been observed by Willet.[21]

TABLE III
Fraction of γ-Crystalline From in As-spun Blends

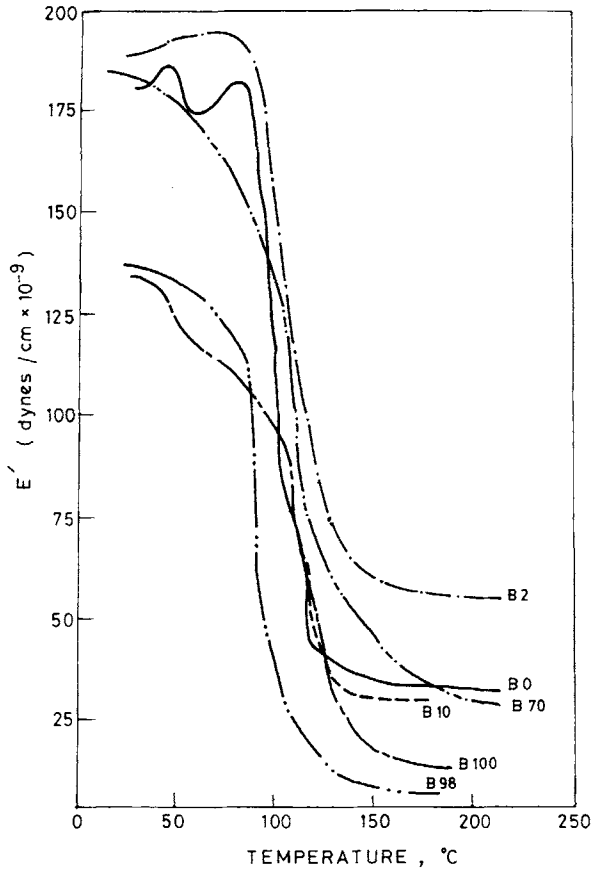| Sample	Percent γ-form
B100	48
B 98	74
B 95	71
B 90	50
B 80	54
B 70	59
B 60	61
B 50	45

Fig. 1. Storage modulus, E' as a function of temperature for a few blends.
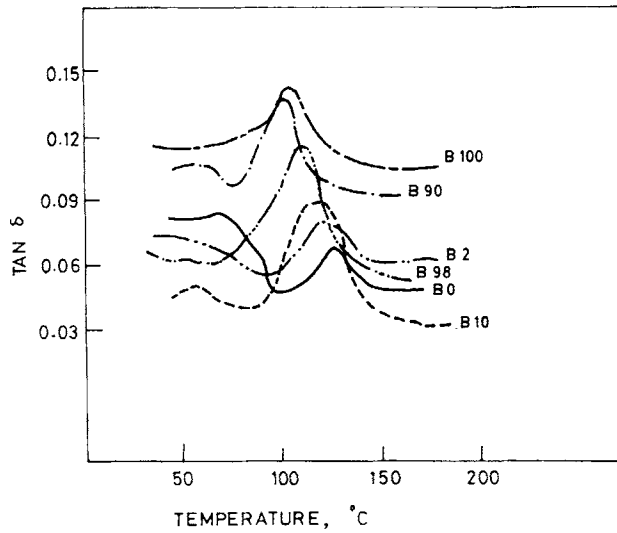

Fig. 2. Tan δ as a function of temperature for a few blends.

The tan δ peak appears at 100°C for nylon-6 and at 110°C nylon-66. This is in agreement with the data of Prevorsek and Beringer.[22] The blends show a single tan δ peak in all cases, which gradually shifts from 100° to 110°C with an increase in nylon-66 content. The presence of a single peak and the gradual shift suggest compatibility of nylon-6 and nylon-66 in the amorphous phase. By addition of nylon-66, the nylon-6 spectrum narrows (Fig. 2), probably because of a reduction in the levels of order in nylon 6. At the nylon-66 end, a broadening is observed, which is probably due to the strong interactions between the two nylons.

Orientation and Drawability

Table IV shows the elongation behavior of the blends. The natural draw ratio (NDR) and maximum draw ratio (MDR) were computed from the stress–strain graphs, as obtained from the Instron tensile tester. The MDR obtained by slow stretching during testing on the Instron was generally higher than the actual MDR obtained during the drawing process. This is expected because drawing at higher speeds generates greater stress, leading to fracture at lower draw ratios.

Table IV shows that the breaking elongation of undrawn blends is almost 80–100% higher than that of the homopolymers, at low compositions. The increase in breaking elongation is less in blends containing comparable amounts of the two nylons, i.e., B30, B40, and B50. The MDR and NDR also increase significantly at low blend compositions and fall in the blends B60, B50, B40, B30, and B20.

Table V shows the yield stress values for the various undrawn blends, calculated from the stress–strain graphs, and shows that there is a significant drop in blends of low compositions, i.e., the stress required to initiate

TABLE IV
Elongation Behavior of Blends

	Elongation at break (%)				MDR (drawing) (m/cm)
Sample	As-spun sample	Drawn Sample	NDR	MDR (Instron)	
B100	240	50	1.4	5.4	3.8
B 98	478	32	2.2	5.8	4.1
B 95	526	30	2.6	6.3	4.8
B 90	378	29	1.2	5.0	6.6
B 80	450	31	1.4	5.5	4.7
B 70	440	25	1.3	5.4	4.4
B 60	468	52	1.3	5.7	3.8
B 50	402	32	1.1	5.4	2.1
B 40	360	—	1.1	4.6	—
B 30	331	—	1.2	4.4	—
B 20	448	38	1.3	5.0	2.0
B 10	476	34	1.9	5.8	4.2
B 5	524	34	1.7	6.2	6.0
B 2	380	44	1.5	4.8	3.8
B 0	354	47	1.2	5.0	2.8

TABLE V
Yield Stress and Tenacity of Blends

| Sample | Yield stress (g/d) | | Tenacity (g/d) | |
	As-spun	Drawn	Drawn	50% Elongation at break
B100	0.36	3.2	4.1	6.1
B 98	0.32	3.8	4.9	6.5
B 95	0.28	3.5	5.3	7.1
B 90	0.33	3.7	5.0	6.0
B 80	0.26	5.1	6.0	7.6
B 70	0.37	1.2	6.1	9.7
B 60	0.27	3.4	4.9	7.2
B 50	0.26	2.3	3.3	4.5
B 40	0.38	—	—	—
B 30	0.17	—	—	—
B 20	0.33	2.8	3.8	5.2
B 10	0.30	4.6	4.9	6.6
B 5	0.34	6.3	6.6	8.6
B 2	0.62	2.2	4.5	6.7
B 0	0.81	1.8	4.2	6.6

and propagate plastic deformation in blends is lower. This accounts for the greater drawability and elongation in these blends. This supports the data of Table II, which shows that the undrawn blends B98, B95, and B90 crystallize predominantly in the γ-crystalline form. This is known to be the metastable form of nylon 6, where hydrogen bonds exist between twisted nylon molecules.[8] The γ form is known to convert easily to the more stable α form during drawing.[23] This partly explains the drop in yield stress of these blends.

The crystallinity, density, and yield stress data show that the structure of the as-spun blends is less compact than that of the parent polymers. This seems to be especially true in those blends where the minor component does not crystallize, i.e., in the blends B98, B95, B20, B10, and B2.

A point to be noted is that after drawing, the drawn blends have a higher yield stress (Table V) and lower elongation at break (Table IV) as compared to the homopolymers. This indicates that the drawn blends are more compact than the drawn nylon-6 and nylon-66. This is probably an outcome of the greater alignment produced, due to the greater extent of draw possible in the blends. The yield stress of the drawn blends B30, B50, and B60 is low. The low maximum draw ratio of these blends is associated with extensive phase segregation, due to independent crystallization.

Table VI shows the orientation characteristics of the drawn blends. The results are consistent with the drawability data. The blends containing up to 10% of nylon-6 in nylon-66, and vice versa, exhibit high drawability and also show much higher birefringence values.

It can be seen from Table VI that the increase in birefringence of the blends B98, B95, B10, B5, and B2 is largely due to an increase in the amorphous orientation, as shown by the large increase in F_a. In these blends, the minor component remains amorphous; therefore, we may infer

TABLE VI
Orientation Characteristics of Drawn Blends

Sample	Δ_n	F_c	F_a
B100	0.0379	0.957	0.284
B 98	0.0659	0.945	0.947
B 95	0.0490	0.953	0.779
B 90	0.0470	0.960	0.530
B 80	0.0485	0.951	0.510
B 70	0.0400	0.941	0.343
B 60	0.0369	0.955	0.207
B 50	0.0331	0.954	0.208
B 20	0.0383	0.046	0.266
B 10	0.0512	0.944	0.565
B 5	0.0531	0.939	0.615
B 2	0.0382	0.935	0.260
B 0	0.0341	0.948	0.161

that the improved orientation is largely due to interference between nylon-6 and nylon-66 molecules.

The crystalline orientation function is more or less unaffected by blending. In the blends containing comparable amounts of the two nylons—particularly B70, B60, B50, and B20—the F_a value is low because in these samples, where phase separation is almost complete, drawing proceeds by slippage at phase boundaries, which does not add to molecular orientation.

Tensile Properties

Table V shows the tenacity values of the drawn filaments. The tenacity increases significantly in blends as compared to the 100% nylon-6 and nylon-66, except for the blends B20, B30, B40, and B50, where the values are low. This trend is same as that for draw ratio and orientation. This is to be expected because tenacity reflects the amount of alignment, compactness, and uniformity in the structure.

An extremely high tenacity was observed for the sample B70. As the two nylons crystallize independently, a possible explanation for the high strength may be the highly interconnected structure of fine fibrils along with a high density of tie molecules and fewer defects in terms of microfibril ends. High tenacity has also been observed at some compositions in the case of PE–PP blends.[24]

Comparison of Tables V and VI shows that in some cases, drawn samples with lower orientation exhibit higher tenacity. This indicates that some residual draw is still present in the drawn filaments. To have a more meaningful comparison of breaking stress, interpolation of graphs of draw ratio versus breaking stress and those of draw ratio versus breaking elongation was carried out.[2] Values of breaking stress were computed for samples drawn to such an extent that all of them had an equal breaking elongation of 50%. These values are given in Table VII. Here, the increase in breaking stress in blends containing small amounts of the minor component is more apparent.

TABLE VII
Breaking Stress of Samples Having Elongation at Break of 50%

Sample	Breaking stress (g/d)
B100	6.1
B 98	6.5
B 95	7.1
B 90	6.0
B 80	7.6
B 70	9.7
B 60	7.2
B 50	4.5
B 20	5.2
B 10	6.6
B 5	8.8
B 2	6.7
B 0	6.6

Initial and sonic modulus values of the drawn and undrawn samples are shown in Table VIII, which demonstrates that both initial and sonic modulus are higher for nylon-66 than for nylon-6. This is consistent with the fact that nylon-66 has a higher crystaline content. The undrawn blend samples have lower modulus value at all blend compositions. This is in agreement with the low-yield stress values observed in Table V and the greater drawability possible. The drawn blend samples have a greater degree of order and compactness than the homopolymers, as shown by higher value of moduli for the drawn filaments, shown in Table VIII. The increase is most pronounced in the blends containing up to 10% of the minor component. The high modulus of B70 may be explained in a similar way as high strength.

TABLE VIII
Modulus of Blends

Sample	Initial modulus (g/d)		Sonic modulus (g/d)	
	Undrawn	Drawn	Undrawn	Drawn
B100	4.2	22.8	22.3	51.8
B 98	2.6	37.1	22.0	57.1
B 95	3.7	38.6	21.9	71.1
B 90	4.2	46.9	28.1	58.4
B 80	3.1	44.3	20.5	52.9
B 70	3.5	58.5	24.0	66.8
B 60	2.3	26.8	21.6	66.8
B 50	2.5	29.2	24.1	50.7
B 40	4.1	—	26.0	—
B 30	3.8	—	23.4	—
B 20	5.9	39.6	22.1	56.8
B 10	6.3	43.4	19.0	55.1
B 5	6.1	43.7	23.0	56.6
B 2	5.3	25.1	28.6	58.6
B 0	7.8	16.1	36.0	58.6

The tensile properties discussed above are well depicted in the charac-teristic stress–strain diagrams for the undrawn and drawn samples in Fig-ures 3 through 6. The graphs show that as-spun blends are more easily deformable than the homopolymers, but after drawing, the blends show superior tenacity and modulus with comparable breaking elongations.

Uniformity of Blend Filaments

Table IX shows the coefficient of variation (C.V.%) of cross section and breaking load calculated for the as-spun filaments. The data indicate that blend filaments containing up to 10% of the minor component have vastly improved uniformity, as compared to the homopolymers. In blends with comparable amounts of both nylons, the filaments are highly nonuniform. In fact, the samples B30 and B40 were so uneven that it was not possible to draw them at all.

The uniformity data once again lead to the inference that nylon-6/nylon-66 blends containing up to about 10% of one component and 90% of the other are uniformly heterogeneous, with the points of heterogeneity well dispersed in the matrix. Therfore, even during spin drawing and take-up,
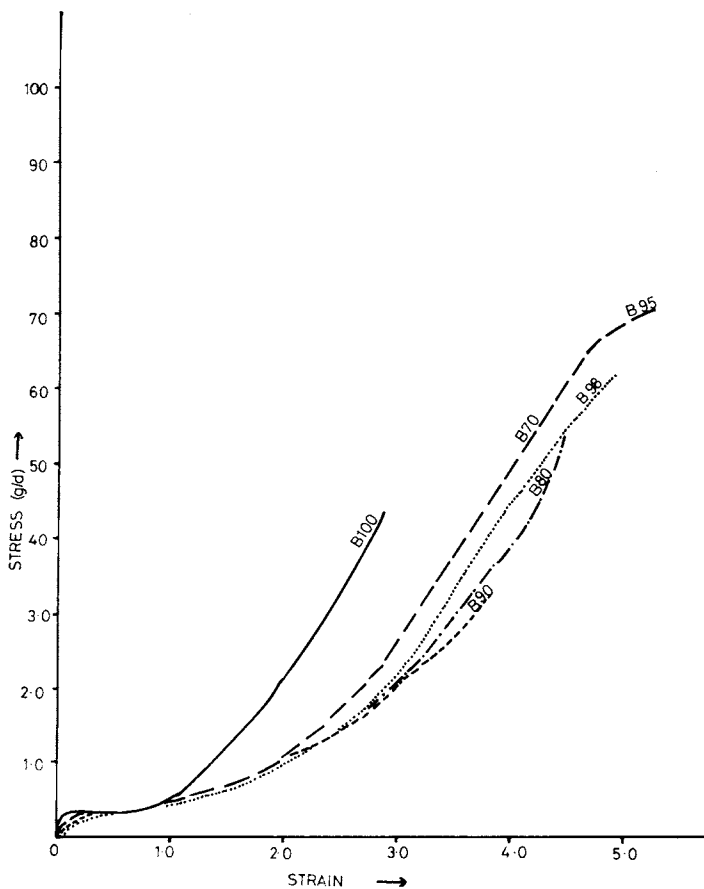

Fig. 3.   Stress–strain graph of selected as-spun blends.

the reduction in diameter by necking is uniform since the ease with which molecules can slip past each other is similar throughout the polymer blend sturcture.

On the other hand, at equal proportions of the constituent polymers, phase segregation occurs, and hence the regions of heterogeneity are more discrete. Therefore, during spinning and take-up, the points at which molecules slip are also not uniformly dispersed. In such circumstances, under a given take-up tension, different areas stretch differently along the spin line, resulting in a nonuniform filament.

Interchange Reaction

The effect of holding the blend melt at its T_m for 30 min was studied to determine the extent of chemical changes that take place between the two components, as has been observed in the case of blends and copolymers of polyester.[18] Table X shows the properties of the filaments obtained from the melt held at T_m for 30 min and of filaments obtained under identical conditions but without holdup time. The blend studied was B10. Examination under a microscope showed that the heat-treated filament was slightly wavy as compared to the smooth, control filament. There seemed to be signs of melt fracture in the heat-treated filament.

Increase in melt viscosity leading to melt fracture has been observed for pure nylons held in the molten state for some time.[25] This has been attributed to an increase in molecular weight due to delayed polymerization. This phenomenon may account for the slight increase in stress at break for the
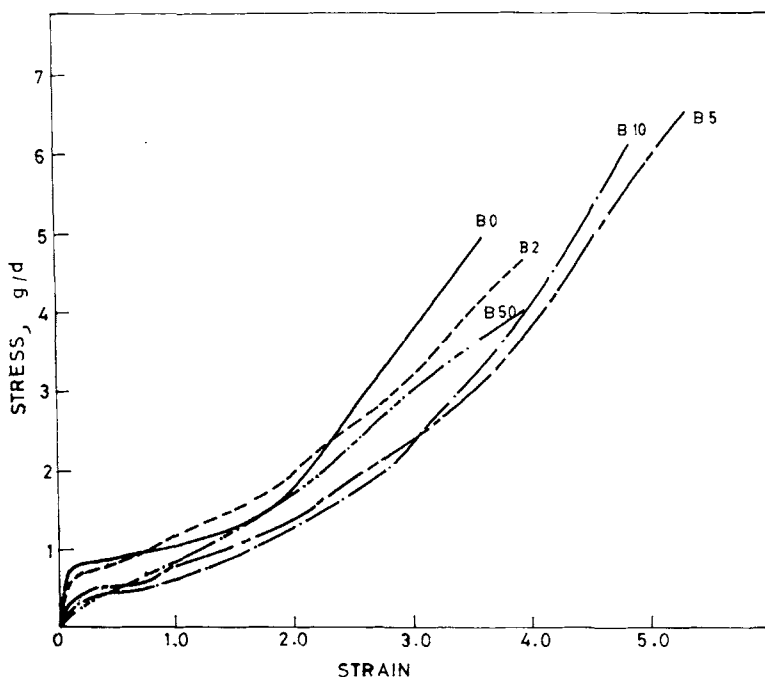

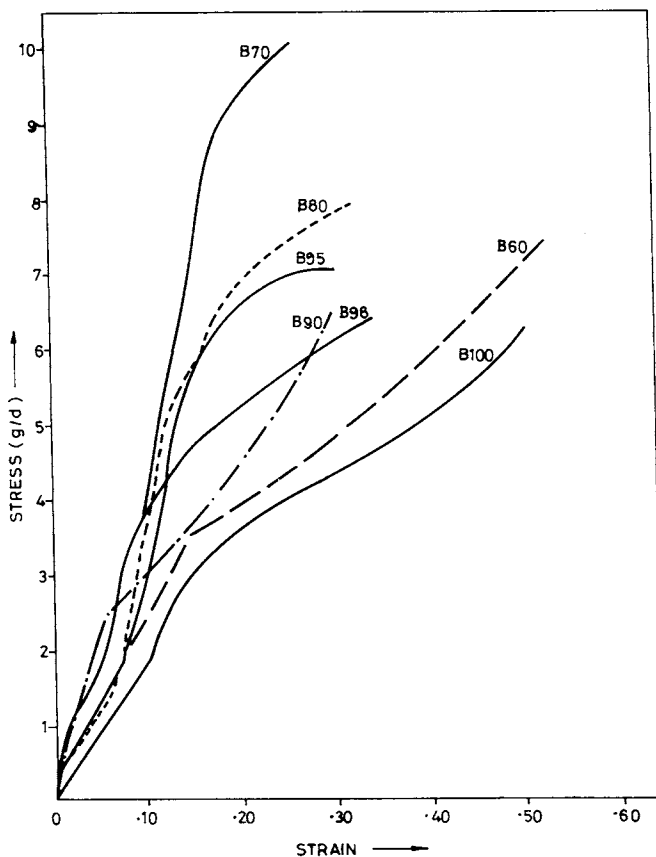Fig. 4. Stress–strain graph of selected as-spun blends.

Fig. 5. Stress–strain graph of selected drawn blends.

heat-treated filament. However, there is also a decrease in melting and crystallization temperature, density, and heats of fusion and crystallization, which may be due to the lesser crystallinity in the heat-treated melt. Inoue[18] has observed a drop in T_c, H_c, and crystallization rate of nylons with an increase in temperature of the melt. It is likely that the effect due to increasing residence time of the melt is similar. The effect may be related to increased kinetic energy of the molecules. The drop in modulus after heat treatment is probably due to the changes in molecular weight distribution. During the short holdup times in the process of melt spinning, no chemical changes of any significance may take place between nylon-6 and nylon-66.

CONCLUSIONS

Improvement in uniformity, tenacity, modulus, and amorphous orientation can be obtained by blending small amounts of nylon-66 in nylon-6 and vice versa. In such blends the minor component remains amorphous. In the case of nylon-6-rich blends, the presence of up to 10% of nylon-6
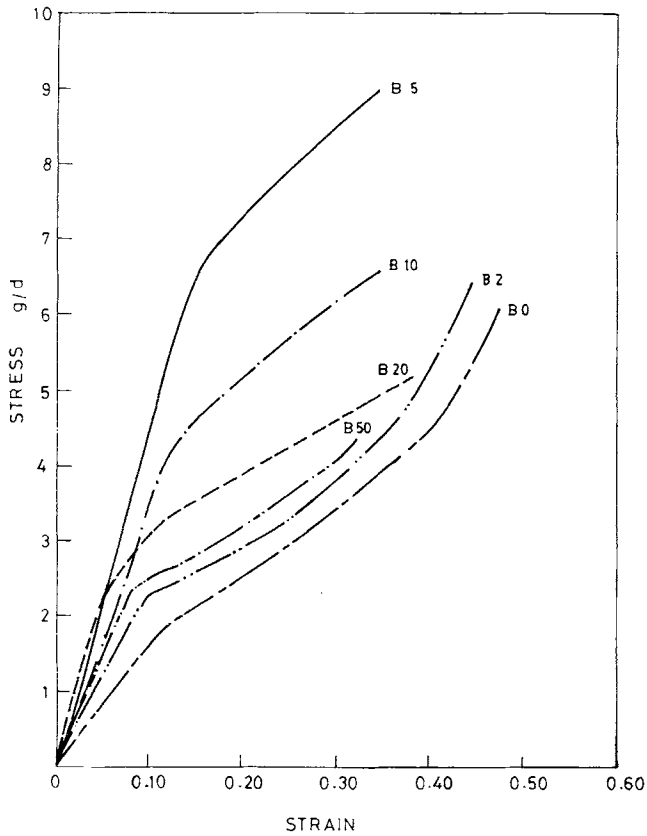
Fig. 6.   Stress–strain graph of selected drawn blends.

TABLE IX
Uniformity of Blends

| Sample | C.V.% (coefficient of variation %) of: | |
	Cross section	Breaking load
B100	11	16
B 98	3	13
B 95	8	6
B 90	9	26
B 80	10	22
B 70	8	15
B 60	24	19
B 50	48	26
B 40	52	41
B 30	56	23
B 20	40	22
B 10	6	11
B 5	4	12
B 2	9	21
B 0	9	20

TABLE X
Effect on Properties of Holding Melt at T_m for 30 min

Property	Filament from heat-treated melt	Normally spun filament
Stress at break (g/d)	6.8	6.2
Elongation at break (%)	528	524
Uniformity of diameter (C.V.%)	13.4	11.8
Initial modulus (g/d)	2.7	6.3
Melting temp. (°C)	259	263
Crystallization temp. (°C)	230.5	235
H_f (cal/g)	8.8	9.9
H_c (cal/g)	9.2	11.2
Density (g/cm³)	1.127	1.134
Density crystallinity (%)	21	27

causes the former to crystallize predominantly in the metastable, γ form during spinning.

In these blends the as-spun filaments are more easily drawable in terms of a lower yield stress and modulus, and after drawing produce filaments having higher modulus and tenacity as compared to the homopolymers.

In blends containing comparable amounts of the two nylons, each component crystallizes independently, and because of the phase segregation the filaments are uneven and of inferior tensile properties.

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