

Dynamic Mechanical Properties of Some Nylons and Their Blends*

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Synopsis

The dynamic mechanical properties of nylons 6, 11, 12, 66, 610, 612, and 666 are compared. The spectra are very similar with peaks in the loss curves at about -120 , -40 , and 85°C . The similarity suggests that attempts to determine whether the nylons could in fact be incompatible when blended might not be successful. Calculations based in turn on calculated cohesive energy densities and interaction parameters also suggest that only nylon, 6 and 66 would be compatible. By using the nature of the major loss peak at the glassy transition which is high and narrow for nylons 11 and 12 and broader and shorter for nylons 6, 66, and 666, it is possible to deduce that nylons 6 and 12 are somewhat incompatible but that the other combinations are most likely dynamically compatible.

INTRODUCTION

Nylons form a large series of polymers prepared from diamines and diacids or from ω -amino acids. Those studied were some of the linear aliphatic nylons in which α,ω -disubstituted monomers were used in the synthesis. The products form a family of engineering resins and fiber-forming plastics. The nylons have been reviewed.^{1,2}

The dynamic mechanical properties of some of the more common nylons have been recorded,^{3,4} but a more complete study of the aliphatic series does not seem to have been done.

There is considerable interest in blends of polymers, polymer alloys, particularly to determine compatibility. Dynamic mechanical analysis enables one to detect whether two or more polymers form a single new phase or partially mixed phases or whether there remain significant regions of the pure homopolymers. It is of particular interest to try to detect the limits of miscibility in homologous series of polymers, such as the nylons.

Nylon has been blended with other polymers to a limited extent but the literature on blending of nylons themselves is very limited.^{1,5} A recent extensive review of compatibility or incompatibility of blends may be found in the book by Solč.⁶ Although several properties may be used to judge whether a blend is compatible, that used here is the anelastic spectrum and the glassy and other transitions and relaxations noted thereby. The polymers tend to be opaque so that transparency alone is not a criterion.

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EXPERIMENTAL

Materials

The nylons are designated in the usual way. Nylons 6, 11, and 12 are based on the ω -amino acids with the same number of carbon atoms. Nylons 66, 610, and 612 are based on hexamethylene diamine and the diacids with 6, 10, and 12 carbon atoms. Nylon 666 is described as a copolymer, 9:1 ratio, of nylon 66 and 6. The details of the polymers are in Table I.

The samples were dried for 24 h at 50°C and kept dry for use. The nylons were received as pellets. Films were pressed on a laboratory press between plattens covered with release-agent-coated aluminum foil at about 10°C above the melting point of the nylon, the higher melting point when blends were used. The pellets were spread on the lower platten, the press was closed lightly until the pellets melted, and a 5000 psi load was applied for 45 s followed by 15 s at no load. The sample was rotated 90° and the process repeated, followed by a third pressing after a second 90° rotation in the same direction. Films were about 0.018 ± 0.003 cm thick. For dynamic tests, strips 0.4×2.5 cm were cut from the best portions of the film in the radial direction, and the thickness and width measured accurately.

Blends were made from the appropriate quantities of pellets by fluxing in a twin roll mixing chamber on the Brabender Plasticorder. The charge was 54 g of premixed pellets and fluxing was for 15 min at 10°C above the higher melting temperature, and at 40 rpm for the first 5 min and 100 rpm thereafter. The blends were removed immediately and allowed to cool since discoloration in air above 200°C was noticeable. The cooled blends were pelletized in a Brabender pelletizer to an average size of about 3 mm and stored dry.

The stress-strain and stress-relaxation data for many of the polymers were published by Bradley and Williams.⁷

Procedures

The dynamic mechanical properties were measured with a Rheovibron DDV-II Direct Reading Viscoelastometer. The usual corrections were applied to the data. The loss and storage moduli were calculated by computer, and the $\tan \delta$ and loss and storage moduli were plotted vs temperature using a Gould Plotter. The temperature range was usually -120-160°C and the heating rate was $1.5 \pm 0.5^\circ\text{C}/\text{min}$. The sample chamber was purged with dry nitrogen to avoid condensation.

The thermomechanical properties were measured using the differential scanning calorimeter attachment for the DuPont 990 Thermal Analyzer. Both the glassy transition temperature and the melting point were estimated by this approach (Table I). Both decreased with increasing methylene to amide ratio.

Percent crystallinity was calculated (Table I). The values were low but could be increased by annealing. The anelastic spectrum is altered by changes in crystallinity. It was assumed that changes in molecular weight and crystallinity would be negligible on blending carefully and that any small decreases would not affect the results.

TABLE I
Properties of the Nylons

Nylon	Sources	Methylene/amide	T_m (°C)	T_m^a (°C)	T_g^b (°C)		Calculated ^a δ (J/cm ³) ^{1/2}	Crystallinity ^d (%)
					Rheo	DSC		
6	Algemene Kunstzijde Polysciences	5	225	220	50	52	28.01	8-11
11	Rilsan BDH Chem. Canada Ltd.	10	182	186	60	47	23.66	7-10
12	Rilsan BDH Chem. Canada Ltd.	11	171	178	62	36	23.16	9-12
66	Du Pont Canada Ltd.	5	257	260	57	46	28.01	6-9
610	BDH Chem. Canada Ltd. Polysciences	7	214	222	65	51	—	5-8
612	Du Pont Canada Ltd. Polysciences	8	212	210	50	50	24.93	4-9
666	Du Pont Canada Ltd.	5	240	250	40	55	28.01	4-8

^a By Ref. 10.

^b Earlier data from Amity M. C. Lam: Rheo = Rheovibron, and DSC = differential scanning calorimeter.

^c Intrinsic viscosity.

^d As received and after annealing, calculated¹⁰ by Mary W.-Y. Yeung from DSC data.

^e *m*-Cresol solvent; otherwise formic acid.

RESULTS AND DISCUSSION

Unblended Nylons

The dynamic mechanical properties of the pure, dried nylons are in Figures 1 and 2 for the loss factor and the dynamic and loss moduli respectively.

A loss peak attributable to the melting transition is not observed since it is above the range of the instrument in tension. Shear studies were not attempted. The presence of three loss peaks is obvious from Figure 1. The largest, at the highest temperature (85°C), is the glassy transition peak (T_g) and is attributed to the movement of large chain segments set free by the disappearance of hydrogen bonding with increasing temperature. The smaller peak at about -40°C ($T < T_g$) is explained by a crankshaft type motion involving an unbonded amide group and several methylene carbon groups. An incomplete peak probably centered at -120°C is typical of the γ -relaxations of chains of methylene groups, a T_γ .

Study of Figure 1 shows that the $\tan \delta$ peak for the glassy transition tends to be higher and narrower for the nylons with the largest methylene to amide ratio, nylons 11 and 12, and lowest and broadest for those nylons with the smallest methylene to amide ratios, nylons 6, 66, and 666. The effect may be related to the number of hydrogen-bonded amide groups in the same way as the melting point; freeing of the chains is not completed

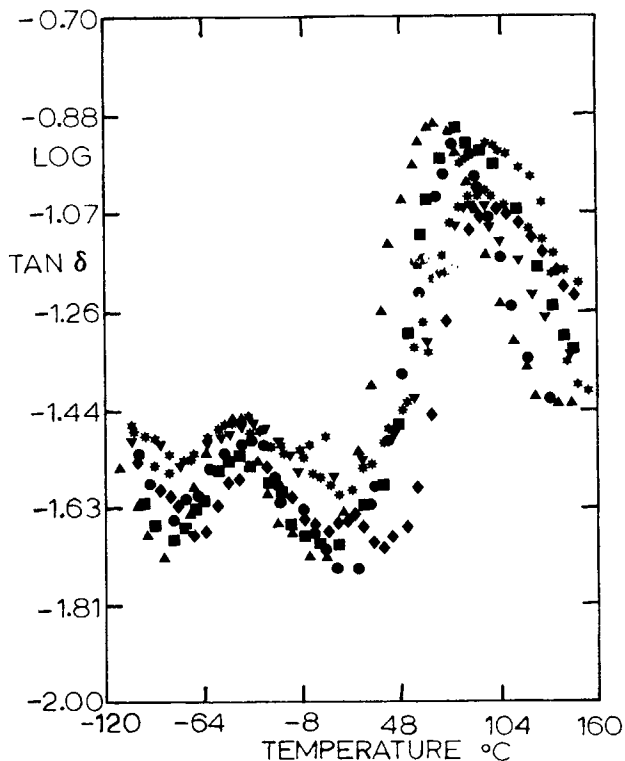


Fig. 1. Dynamic mechanical loss factors vs. temperature for the nylons: (■) nylon 6; (●) nylon 11; (▲) nylon 12; (◆) nylon 66; (●) nylon 610; (▼) nylon 612; (★) nylon 666. 110 Hz.

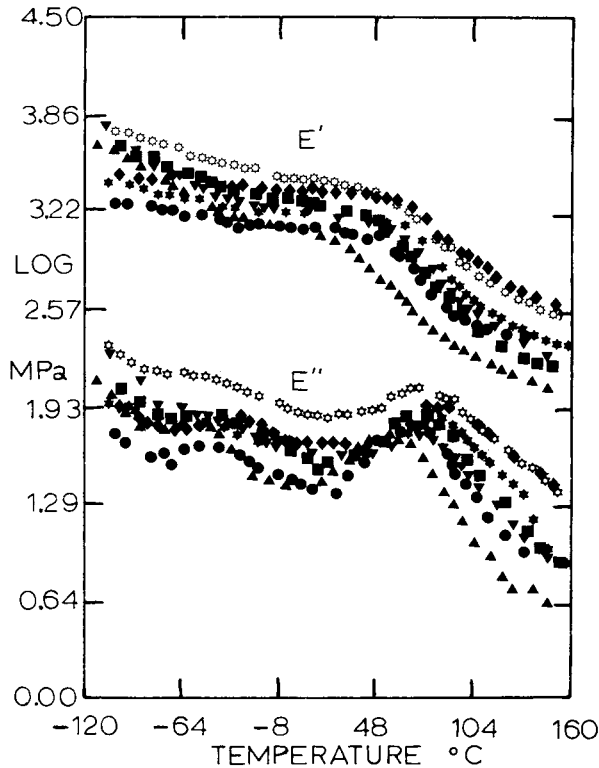


Fig. 2. Dynamic mechanical storage moduli (upper) and loss moduli (lower) for the nylons: (■) nylon 6; (●) nylon 11; (▲) nylon 12; (◆) nylon 66; (●☆) nylon 610; (▼) nylon 612; (★) nylon 666. 110 Hz.

until a higher temperature is reached, and the residual hydrogen-bonded amides raise the viscosity above the glassy transition to yield a higher value of the loss factor in that region, a value which decreases somewhat more slowly with increasing temperature.

For a similar reason the glassy transition temperature deduced from the loss factor peaks increases as the ratio of methylene to amide groups decreases (Table I).

The $T < T_g$ relaxation yields a peak at about -40°C , and the size and shape changes in much the same way as does the glassy transition peak.

The dynamic and loss moduli are shown in Figure 2. In both cases nylons 11 and 12 have the lowest values and nylons 66 and 610 the highest. The width of the glassy transition peak increases with decreasing methylene to amide ratio primarily from higher values of the loss modulus above the glassy transition temperature. The $T < T_g$ peak is very small in the loss modulus curve, and there is little indication of that relaxation in the dynamic modulus curve. Both sets of curves indicate high values of the moduli at the γ -relaxation at about -120°C , increasing from those values for nylon 11 to the highest values for nylon 610.

It is generally considered that the degree of compatibility can be estimated if the glassy transition temperature of the components are at least 20°C apart. Figure 1 suggests that this difference is unlikely to be found. How-

ever, it was considered that including consideration of the size and shape of the loss peaks and the magnitude of the moduli along with the glassy transition data might offer sufficient evidence.

Earlier Rong and Williams⁸ observed that moisture had a pronounced effect on the loss factor of nylon 6 in the glassy transition region. To avoid this problem, all nylons were blended dry and tested that way. It was assumed that moisture would not alter the compatibility but would, of course, alter the numerical data.

Blends of Nylons 11 and 12

Blends of nylons 11 and 12 showed no unusual features except that the yellowish color intensified with increasing nylon 12. The loss factor data are plotted in Figure 3 and the storage and loss moduli in Figure 4.

Obviously, the loss curves (Fig. 3) are very similar for the polymers and their blends. At the glassy transition region the loss factor of the blends lies between those of the component polymers on the low temperature side of the peak and above on the high temperature side. The loss factors for the blends lie below those of the components in the $T < T_g$ region and above on the high temperature side of the γ -relaxation peak. There is no obvious explanation for these observations. The differences are small, but more particularly the data indicate that the two polymers are indeed com-

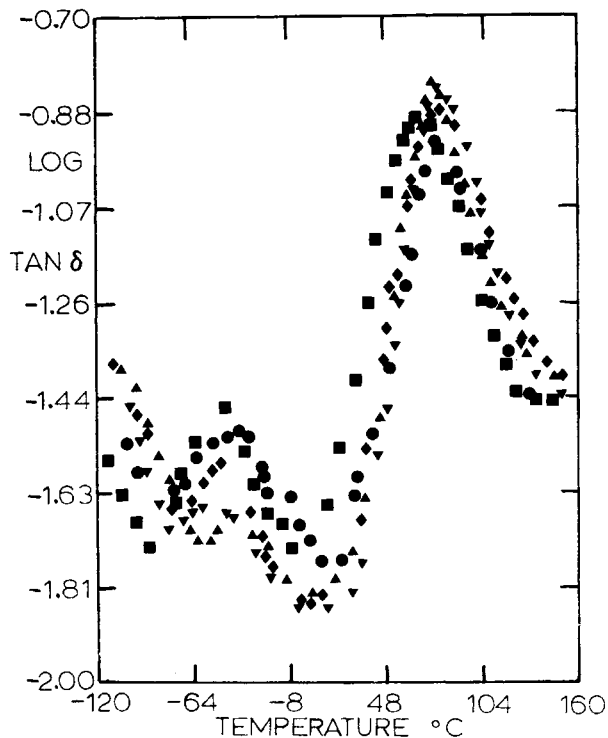


Fig. 3. Loss factors at 110 Hz vs. temperature for nylons 11 and 12 and blends: (■) nylon 12; (▲) 75/25 nylon 12/nylon 11; (◆) 50/50 nylon 12/nylon 11; (▼) 25/75 nylon 12/nylon 11; (●) nylon 11.

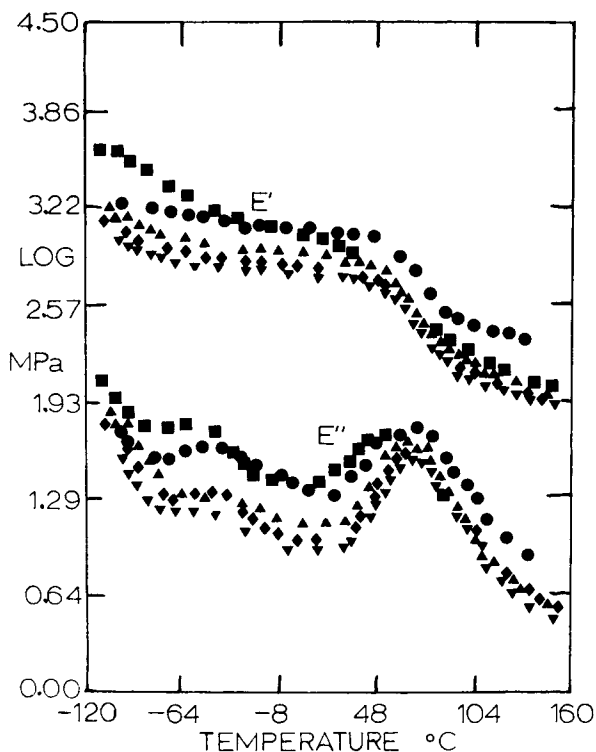


Fig. 4. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for nylons 11 and 12 and blends: (■) nylon 12; (▲) 75/25 nylon 12/nylon 11; (◆) 50/50 nylon 12/nylon 11; (▼) 25/75 nylon 12/nylon 11; (●) nylon 11.

patible. The data for the blends tend to lie closer to those for nylon 11 than to those for nylon 12.

The dynamic and loss moduli curves (Fig. 4) follow the same general patterns. The loss and dynamic moduli usually lie below those of the nylon 11 or nylon 12 except that, at and above the glassy transition temperature, the blend data are closer to those of nylon 12.

The location of the relaxation peaks were found with care and yielded the data in Table II.

Blends of Nylons 12 and 612, Nylons 6 and 612, and Nylons 6 and 12

Blends of nylons 12 and 612 appeared similar to the nylons 11 and 12 blends with the yellowish color increasing with the nylon 612 content. The loss factor data are in Figure 5 and the dynamic and loss moduli data in Figure 6.

Since the differences in the parent polymers are much greater, the effects of blending are easier to note. The loss factor (Fig. 5) indicates that the blends are intermediate between the two components at the glassy transition region but much closer to the data for nylon 612. Again at the $T < T_g$ relaxation region the data for the blends are all below those for the components, quite markedly so. The data suggest that at the γ -relaxation

TABLE II
Summary of Temperatures for Transitions and Relaxations

Composition by weight	$T < T_g$ (°C)	T_g (°C)		
		By $\tan \delta$	By E'	By E''
Nylon 11/nylon 12		$\chi = 9.6 \times 10^{-3} \text{ a}$		
100:0	-37	80	78	68
75:25	-52	80	74	69
50:50	-44	79	78	67
25:75	-38	78	72	65
0:100	-45	68	61	56
Nylon 12/nylon 612		$\chi = 0.126 \text{ a}$		
100:0	-45	68	61	56
75:25	-47	80	71	63
50:50	-42	81	75	61
25:25	-40	82	76	73
0:100	-37	89	82	72
Nylon 6/nylon 612		$\chi = 0.375 \text{ a}$		
100:0	-40	83	84	69
75:25	-46	95	76	71
50:50	-43	92	80	74
25:75	-37	88	84	66
0:100	-37	89	82	72
Nylon 6/nylon 12		$\chi = 0.936 \text{ a}$		
100:0	-40	83	84	69
75:25	-40	94	80	69
50:50	-40	87	66	66
25:75	-46	80	65	57
0:100	-45	68	61	56
Nylon 612 in a nylon 6/nylon 12 blend (50/50)				
100:00	-37	89	82	72
75:25	-47	88	74	66
50:50	-42	84	76	64
25:75	-41	83	64	59
0:100	-40	87	66	66
Nylon 666 and a blend of nylon 66/nylon 6 (90/10)				
100 copolymer	-41	99	77	80
90:100 blend	-50	99	85	79
Nylon 666 in a blend of nylon 66 and 6 (50/50)				
25:75	-35	98	83	75
50:50	-36	97	79	75
75:25	-40	96	74	74
100:0	-41	99	77	79

(continued)

TABLE II
(Continued from previous page)

Composition by weight	$T < T_g$ (°C)	T_g (°C)		
		By $\tan \delta$	By E'	By E''
Unblended				
Nylon 66	-30	105	81	87
Nylon 610	-45	95	88	80

^a Critical interaction parameters $\chi_{AB}(Cr) = 0.004$.

the blend data would lie between and above those for the components, i.e., closer to those for nylon 612. Again there is no evidence for incompatibility.

The dynamic and loss moduli data (Fig. 6) show that these are between or below the data for the components but not greatly so. In these cases the blend lie closer to those for nylon 12 but the effect is small. The numerical data for the location of the temperature peaks for the relaxations and transitions are in Table II.

The data for the nylon 6 and nylon 612 blends are in Figures 7 and 8. The brownish yellow blends did not show other changes.

The loss factor data are in Figure 7. The results for the blends lie between those for the components. However, the blend richer in one component lay

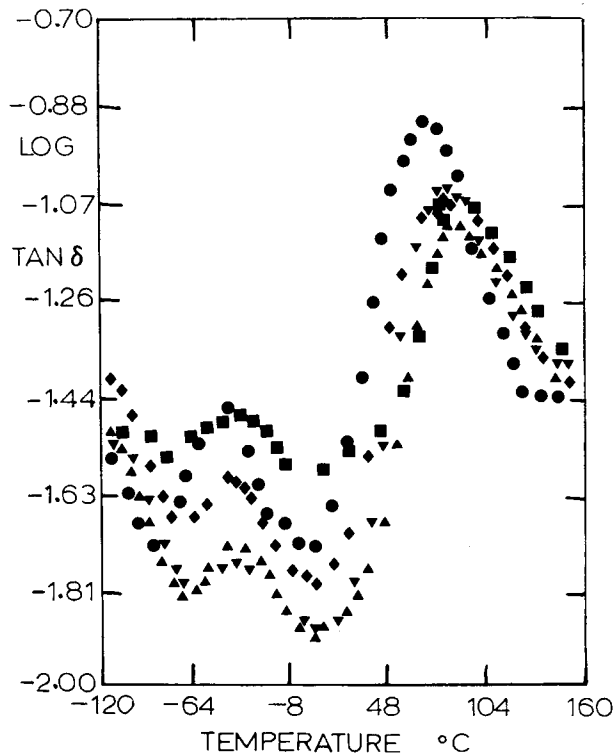


Fig. 5. Loss factor at 110 Hz vs. temperature for nylons 12 and 612 and blends: (■) nylon 612; (▲) nylon 75/25 nylon 612/nylon 12; (◆) 50/50 nylon 612/nylon 12; (▼) 25/75 nylon 612/nylon 12; (●) nylon 12.

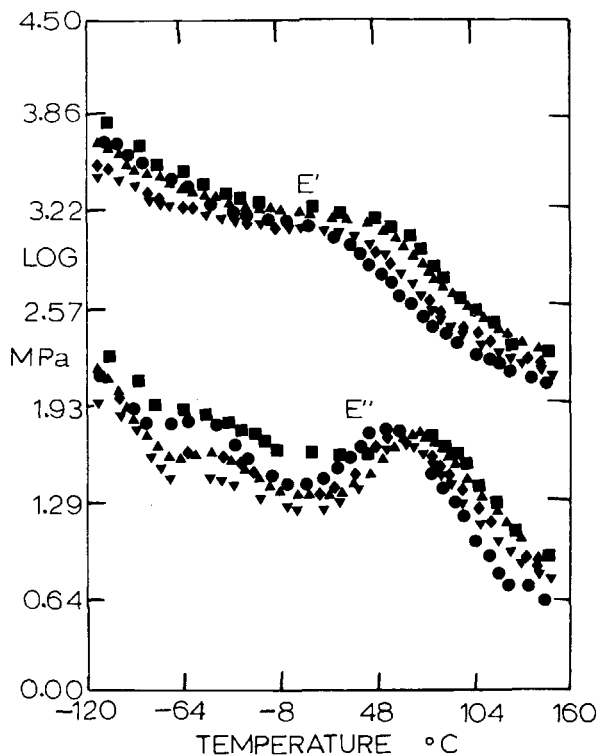


Fig. 6. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for nylon 12 and 612 and blends: (■) nylon 612; (▲) 75/25 nylon 612/nylon 12; (◆) 50/50 nylon 612/nylon 12; (▼) 25/75 nylon 612/nylon 12; (●) nylon 12.

closer to the curve for that component. The 50:50 blend did not clearly show evidence of incompatibility.

As in the previous cases, the loss factor for the blends lay well below those of the pure components in the $T < T_g$ relaxation region and appeared to be rising to between or even above the values for the components in the γ -relaxation region.

Unlike the previous results the dynamic and loss moduli (Fig. 8) follow very closely those of the components with some preference for higher values. The data for the peak locations by various techniques are in Table II.

The blends of nylons 6 and 12 showed no signs of degradation and were milky yellow in color. The loss factors are plotted in Figure 9 and the dynamic and loss moduli in Figure 10.

The loss factor curves (Fig. 9) show that the blends are composite and relate to the pure polymers. The peaks for the blends are lower at the glassy transition and the "high" nylon 12 peak is followed by a shoulder representing the contribution of the nylon 6.

The $T < T_g$ peaks are between or below those of the parent polymers. The beginnings of the γ -peak are inadequate for detailed analysis.

The dynamic and loss moduli curves (Fig. 10) suggest the same conclusions. The dynamic moduli data mostly lie between those of the pure polymers. However, the 50/50 blend points suggest two glassy transitions. The

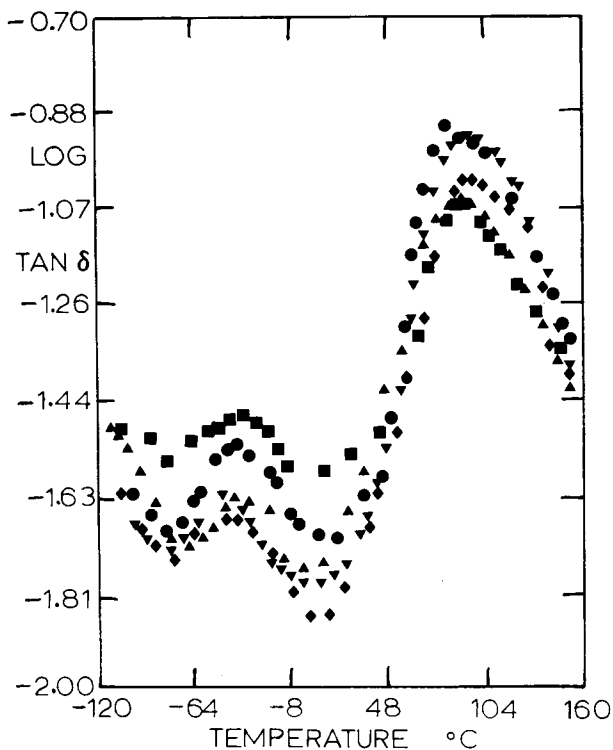


Fig. 7. Loss factor at 110 Hz vs. temperature for nylons 6 and 612 and blends: (■) nylon 612; (▲) 75/25 nylon 612/nylon 6; (◆) 50/50 nylon 612/nylon 6; (▼) 25/75 nylon 612/nylon 6; (●) nylon 6.

loss moduli peaks are broader and lower, also suggesting two overlapping peaks.

At lower temperatures the storage and loss moduli tend to be lower than for the pure polymers, the lowest values being for the 50/50 blend, again suggesting incompatibility but not complete separation. The peak locations are summarized in Table II. They tend to separate into values near nylon 6 and near nylon 12, but, being ambiguous for the 50/50 blend depending on which peak is emphasized, the nylon 6 peak in the E'' curve and the nylon 12 peak in the E' curve.

These data agree with the observation by Kyotani that nylon 6 and nylon 12 separately crystallized from stirred, mixed solution.⁵

Blend of Nylon 612 in a 50/50 Blend of Nylon 6 and Nylon 12

Blends of these polymers showed a brownish yellow color but no obvious degradation. The loss factors are plotted in Figure 11 and the dynamic and loss moduli in Figure 12.

It was surmised that nylon 612 would be a compatibilizing agent for the nylon 6 and nylon 12. The loss factor (Fig. 11) suggests that this is true. The loss factor above the glassy transition temperature differs from that

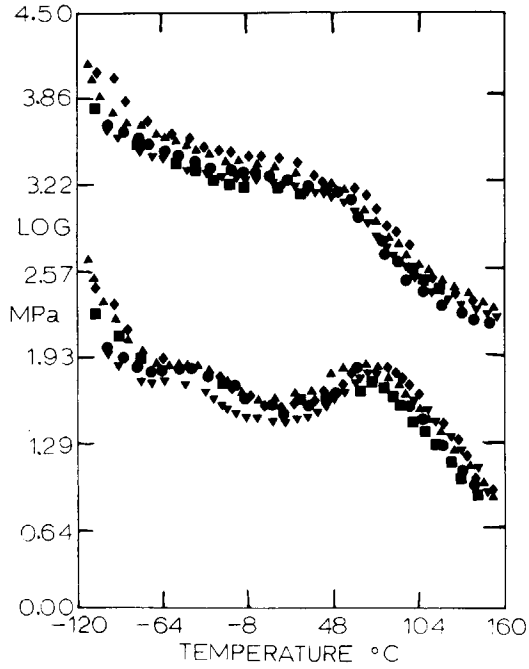


Fig. 8. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for nylons 6 and 612 and blends: (■) nylon 612; (▲) nylon 75/75 nylon 612/nylon 6; (◆) 50/50 nylon 612/nylon 6; (▼) 25/75 nylon 612/nylon 6; (●) nylon 6.

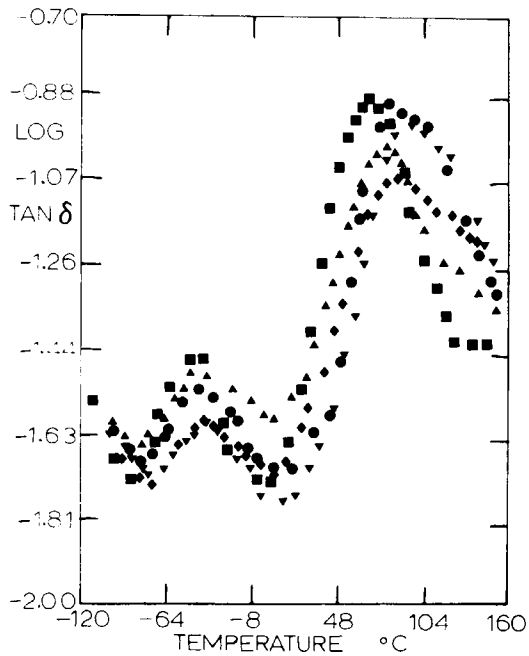


Fig. 9. Loss factor at 110 Hz vs. temperature for nylons 6 and 12 and blends: (■) nylon 12; (▲) 75/25 nylon 12/nylon 6; (◆) 50/50 nylon 12/nylon 6; (▼) 25/75 nylon 12/nylon 6; (●) nylon 6.

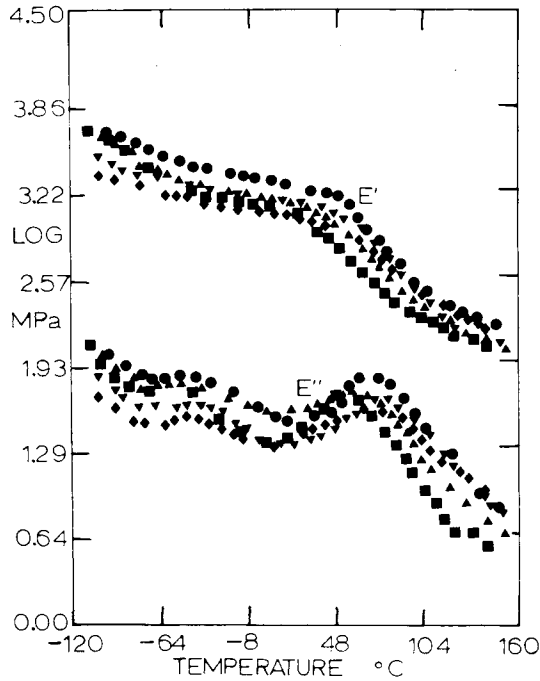


Fig. 10. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for nylons 6 and 12 and blends: (■) nylon 12; (▲) 75/25 nylon 12/nylon 6; (◆) 50/50 nylon 12/nylon 6; (▼) 25/75 nylon 12/nylon 6; (●) nylon 6.

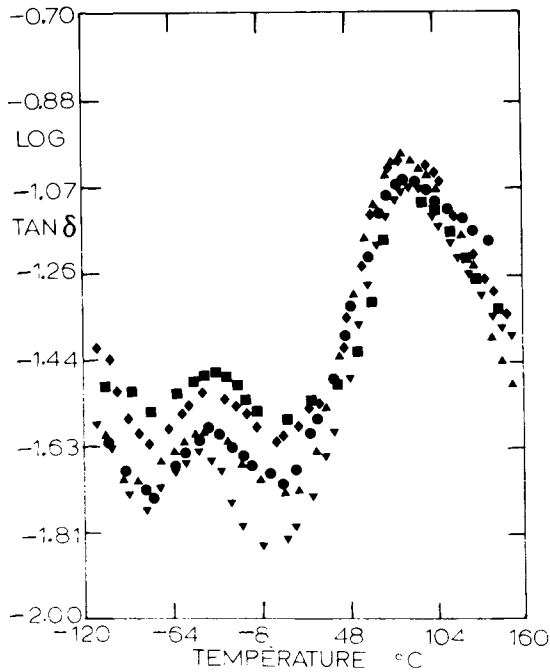


Fig. 11. Loss factor at 110 Hz vs. temperature for a 50/50 blend of nylons 6 and 12, nylon 612, and blends; (■) nylons 6-12 blend; (▲) 25/75 nylon 612/nylons 6-12 blend; (◆) 50/50 nylon 612/nylons 6-12 blend; (▼) 75/25 nylon 612/nylons 6-12 blend; (●) nylon 612.

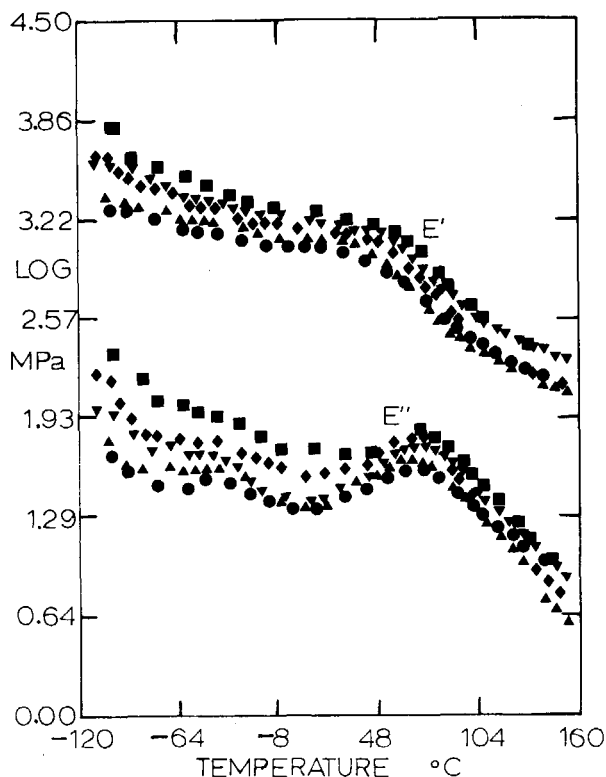


Fig. 12. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for a 50/50 blend of nylon 6 and 12, nylon 612, and blends: (■) nylons 6-12 blend; (▲) 25/75 nylon 612/nylons 6-12 blend; (◆) 50/50 nylon 612/nylons 6-12 blends; (▼) 75/25 nylon 612/nylons 6-12 blend; (●) nylon 612.

of the 50/50 blend and approximates that of the nylon 612. The loss peak is somewhat narrower and sharper also.

The $T < T_g$ data are in keeping with these conclusions. The 50/50 blend of nylon 612 with 50/50 nylon 6 and nylon 12 blend approximates nylon 612 alone whereas the other blends approximate the 50/50 nylon 6 and nylon 12 blend. The start of the γ -loss relaxation peak is typical. The blend data are intermediate and are similarly placed to the $T < T_g$ peaks.

The dynamic and loss moduli curves (Fig. 12) suggest compatibility at and above T_g and a tendency of the blends to resemble nylon 612 at lower temperatures. The temperatures at the peaks were estimated and are recorded in Table II.

Nylon 666 and Blends of Nylons 66 and 6

Nylon 666 is described as a copolymer of nylons 66 and 6 in the ratio of 9:1 by weight. Attempts were made to prepare a comparable polymer by blending nylons 66 and 6. The product was brown in color and brittle, and mixing in the blending chamber relatively poor. Nevertheless, useable samples were prepared by blending at 263°C for a shorter time of 10 min.

The data for the loss factor are in Figure 13. Particularly noticeable is the similarity between the glassy transition peaks of the mechanical blend

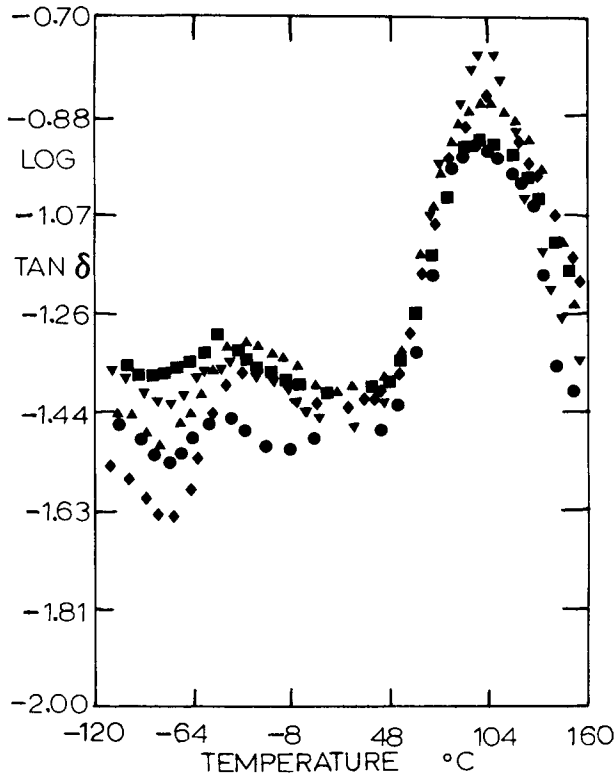


Fig. 13. Loss factor at 110 Hz versus temperature for nylon 666, blends of nylon 66 and 6, and blends of nylon 666 with 50/50 blend of nylons 66 and 6: (●) nylon 666; (■) mechanical blend of nylons 66 and 6 (90/10); (▲) 25/75 nylon 666/nylons 66-6 blend (50/50); (◆) 50/50 nylon 666/nylons 66-6 blend (50/50); (▼) 75/25 nylon 666/nylons 66-6 blend (50/50).

and the copolymer. The most obvious difference is the lower value for the loss factor of the copolymer at lower temperatures, although the loss curve has the same form with narrower peaks.

The remaining curves are for varying amounts of nylon 666 blended with a 50/50 blend of nylons 66 and 6. At the glassy transition the loss peak is very high but not wider than for the pure polymers. At lower temperatures the loss factors for the blends tend to deviate from those of the 9:1 blend and to approximate more the lower data for the copolymer.

The dynamic and loss moduli of the copolymer and blend (Fig. 14) are very similar, but the data for the 50/50 blend of nylons 66 and 6 with varying amounts of nylon 666 lie well above those for nylon 666 and its physically blended analogue; the more so the lower the temperature. At and above the glassy transition the differences are small.

The location of the peaks of the curves are indicated in Table II.

Comparison with Calculated Glassy Transition Temperatures and Calculation of Incompatibility

Calculation⁹ of the glassy transition temperatures for the blends by the Fox and the Kelley Bueche relationships failed to yield values comparable to the experimental results. It will be recalled that the glassy transition

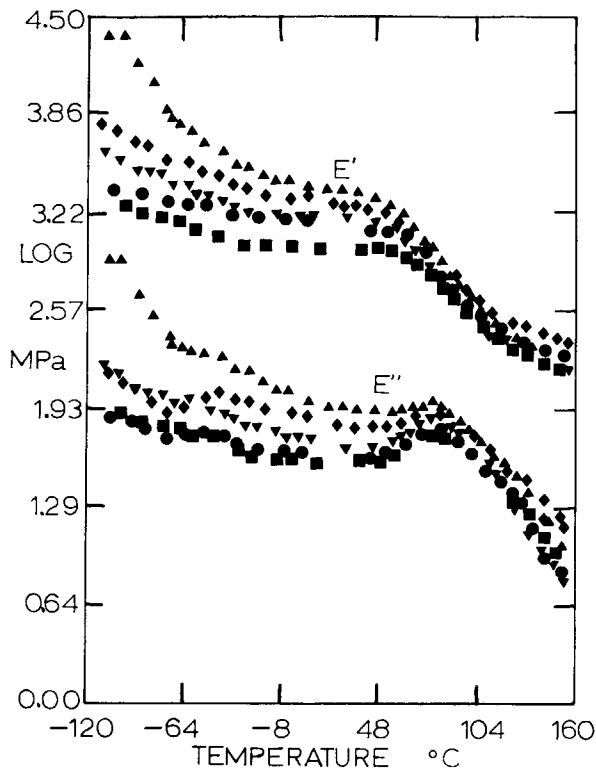


Fig. 14. Storage moduli (upper) and loss moduli (lower) at 110 Hz vs. temperature for nylon 666, 90/10 blend of nylons 66 and 6, and blends of nylon 666 with 50/50 blend of nylons 66 and 6: (●) nylon 666; (■) mechanical blend of nylons 66 and 6 (90/10); (▲) 25/75 nylon 666/nylons 66-6 blend (50/50); (◆) 50/50 nylon 666/nylons 66-6 blend (50/50); (▼) 75/25 nylon 666/nylons 66-6 blend (50/50).

temperatures as measured by the peak of the loss factor curves did not necessarily lie between those of the components. On the other hand calculations of the compatibility of the blends according to Paul and Seymour⁹ indicated that only the nylon 66 and nylon 6 pair should be compatible whereas dynamically only the nylon 6 and nylon 12 pair indicated incompatibility. The calculated interaction parameter¹⁰ for the nylon 11 and nylon 12 blend exceeded the critical value for compatibility,⁹ but was borderline. The other interaction parameters greatly exceeded the critical value.

SUMMARY

A study of the properties of nylons 6, 11, 12, 66, 610, 612, and 66 was made. It was found that as the methylene to amide ratio increased the melting point and the glassy transition point decreased, being lowest for nylons 11 and 12 and highest for nylons 6, 66, and 666. The storage or dynamic moduli also follow this trend.

The dynamic mechanical spectra of the nylons are very similar. Those for nylons 11 and 12 show a higher and narrower $\tan \delta$ peak at the glassy

transition whereas the nylons 6, 66, and 666 show broader and lower peaks skewed towards the high temperature side. The nylons would appear to be compatible, but by comparison of the size and shapes of the loss factor peak at the glassy transition temperature it was deduced that nylons 6 and 12 were at least partially, and perhaps mostly, incompatible when blended. The addition of nylon 612 to nylon 6 and 12 blends compatibilized the blend, and these blends appeared to be similar to nylon 612.

Nylon 666 which is a copolymer possesses higher moduli than a mechanical blend of the same composition but the anelastic spectra of the two products appear to be the same.

Schemes to test for compatibility by the measurement of the glassy transition temperatures and the interaction parameters did not predict the results since, whereas calculations showed that nylon 6 and 66 only would be compatible, all pairs tested were in fact compatible except nylon 6 and 12.

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