Mechanical behaviour and morphology of toughened aliphatic polyamides

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The mechanical properties of a series of polyamide materials (nylon 6,12, nylon 11, nylon 12 and nylon 12,12) were investigated using triblock copolymers of the type styrene–(ethylene-*co*-butene)–styrene (SEBS) and a maleic anhydride (MA) functionalized version (SEBS-*g*-MA). The results have been compared with the behaviour reported for nylon 6 and nylon 6,6 in our previous work. A much lower concentration of the maleated elastomer (SEBS-*g*-MA) was required for effective toughening of these polyamides with higher CH_2/NHCO ratios. This is believed to be a consequence of the inherently more ductile polyamide matrix as the methylene content of the nylon material increases. Further evidence of this was given by the trends in the ductile–brittle transition temperature of the various binary blends of these polyamides with SEBS-*g*-MA. Transmission electron microscopy techniques used for investigating the morphology generated in these blends showed that ternary blends of nylon *x* (monofunctional) materials (nylon 11 and nylon 12) yielded regular, spherical rubber particles with a continuously varying particle size as the proportion of SEBS and SEBS-*g*-MA in the rubber phase was changed, while ternary blends of nylon *x,y* (difunctional) materials (nylon 6,12 and nylon 12,12) yielded rubber particles with varying levels of complexity and size depending on the SEBS-*g*-MA/SEBS ratio. Such differences in the blend morphology between nylon *x* and nylon *x,y* materials have been attributed to differences in the basic chemical structure of these polyamides. The existence of critical limits on the rubber particle size for toughening these blends was also explored.

(Keywords: polyamides; block copolymers; rubber toughening)

INTRODUCTION

The accompanying paper[1] reported the morphology of aliphatic polyamide blends with maleic anhydride functionalized styrene–(ethylene-*co*-butene)–styrene (SEBS-*g*-MA) triblock copolymers. Polyamide functionality was identified as an important factor in the morphology generation for these blends. In general, the polyamides which have only one amine end per chain (monofunctional) yield rubber particles that are relatively simple in shape and significantly smaller in size than the polyamides which have two amine ends on some of the nylon chains (difunctional). Also, among the difunctional polyamides, the steady decrease in rubber particle size with increasing methylene content in the polyamide repeat unit (CH_2/NHCO ratio) was attributed to its influence on the nature of the rubber–polyamide interface.

Recent publications[2–5] have shown that nylon 6,6 can be toughened by SEBS-*g*-MA alone, while a combination of SEBS-*g*-MA and SEBS is needed to achieve maximum toughening of nylon 6. Binary blends of SEBS-*g*-MA with nylon 6 yield rubber particles about 0.05 μm in diameter (apparently too small for toughening), while replacing some of this maleated rubber with SEBS increases particle size into an optimum range for toughening. On the other hand, binary blends of SEBS-*g*-MA with nylon 6,6 are tough at room temperature and have much larger rubber particles (0.5–1.0 μm).

Most of the previous work on toughening of polyamides has dealt mainly with nylon 6 and nylon 6,6 materials. This study examines the rubber toughening of selected monofunctional and difunctional polyamides with higher methylene content (CH_2/NHCO ratio) in the repeat unit, namely nylon 6,12, nylon 11, nylon 12 and nylon 12,12. This study also explores the effects of adding a combination of the maleated (SEBS-*g*-MA) and unmaleated (SEBS) elastomers on the morphology and mechanical properties of these materials. Polyamides with a high CH_2/NHCO ratio are potentially more ductile, which no doubt will influence the final properties of the blends. According to Wu, the rubber particle size (or interparticle distance) required for optimum toughening depends on the inherent ductility of the matrix[6,7].

MATERIALS AND METHODS

Table 1 summarizes pertinent information about the materials used in this study. All materials containing polyamides were dried for at least 12 h in a vacuum oven at 85°C prior to melt processing steps. All blends were prepared in a Killion single-screw extruder ($L/D = 30$, 2.54 cm screw diameter) by the simultaneous extrusion of all components based on the apparent insensitivity of the properties of such blends to the mixing protocol as revealed in an initial screening. The extruded material was then injection-moulded into standard tensile (ASTM

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Table 1 Polymers used in this study

Polymer	Description	$\bar{M}_n$ ($\times$1000)	Endgroup content (μeq g^{-1}) NH$_2$	COOH	Relative melt viscosity[a]	Source
Nylon 11	BMNO TL	25	46.4	43.4	2.4	Atochem Inc.
Nylon 12	AESNO TL	16	44.7	50.5	0.5	Atochem Inc.
Nylon 12,12	Zytel 40-401	NA	46.0	47.6	1.5	E.I. du Pont Co.
Nylon 6,12	Zytel 158 L	NA	43.8	41.7	1.8	E.I. du Pont Co.
Nylon 6,10	Nylon 6,10	25	32.6	NA	0.7[b]	Aldrich Chemical Co.
Nylon 6,9	Nylon 6,9	28	33.9	NA	1.2[b]	Aldrich Chemical Co.
(SEBS)	Kraton G 1652	Styrene block = 7 EB block = 37.5	–	–	0.6	Shell Chemical Co.
(SEBS-*g*-MA) 1.84% maleic anhydride	Kraton 1901X	NA	–	–	1.0	Shell Chemical Co.

[a,b] Brabender torque of SEBS-*g*-MA relative to the polyamide, all measured after 10 min at 60 rev min^{-1} at [a]240°C or [b]280°C
NA, not available

D638 type I) and Izod (ASTM D256) bars (0.318 cm thick) using an Arburg Allrounder screw injection-moulding machine. Injection-moulded test samples were visually inspected for air bubbles, and those with defects were discarded. The samples were tested 'dry as-moulded'. Tensile testing was performed on an Instron apparatus in accordance with ASTM D638 using a crosshead speed of 5.08 cm min^{-1}. An extensometer strain gauge with a 5.08 cm gap was used to obtain the modulus and yield strain values. Izod testing was conducted using ASTM D256 method A with specimens 0.318 cm thick. For some specimens, a new razor blade was pressed into the standard machine-made notch to give a sharp notch.

Most of the work reported here on mechanical properties involved two monofunctional (nylon 11 and nylon 12) and two difunctional (nylon 6,12 and nylon 12,12) polyamides. However, to understand the implications of changing the inherent ductility of the matrix and varying particle size via the CH_2/NHCO ratio[1], the ductile–brittle transition behaviour of blends of some other polyamides (see *Table 1*) was also examined.

Transmission electron microscopy (TEM) techniques similar to those described in the previous paper[1] were used to determine the morphology of blends stained by RuO_4.

MECHANICAL PROPERTIES

Table 2 compares the room temperature mechanical properties of the various polyamides of interest here. In general, stiffness and strength decrease while notched Izod and elongation at break increase as the hydrocarbon content (or CH_2/NHCO ratio) in the polyamide repeat unit increases. Nylon 12 and nylon 12,12 have substantially higher impact strength than all the other polyamides. Of course, nylon 6 and nylon 6,6 have quite low Izod values, which is one of the reasons for the intense interest in rubber toughening of these materials. Previous studies[2,3] have shown how the Izod impact strength of these materials responds to blending with SEBS-*g*-MA.

Table 2 Room temperature mechanical properties of polyamides

Polyamide	Notched Izod impact (J m^{-1})	Modulus (GPa)	Yield strength (MPa)	Elongation at break (%)
Nylon 6	39	2.86	80.9	272
Nylon 6,6	45	2.65	80.1	210
Nylon 6,12	81	1.86	52.4	240
Nylon 11	72	1.22	41.4	250
Nylon 12	203	1.34	40.8	300
Nylon 12,12	220	1.58	44.8	400

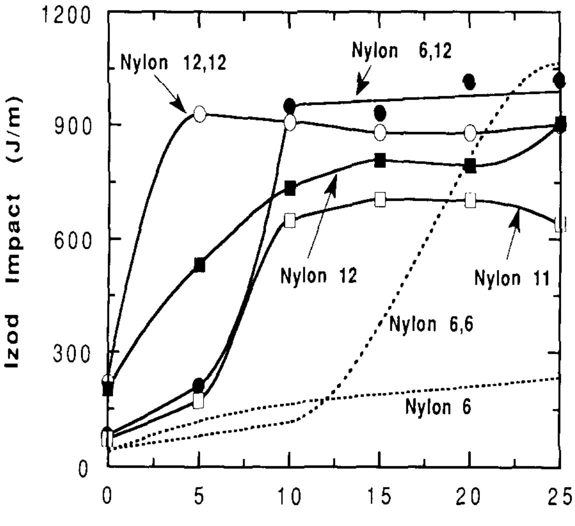


Figure 1 Notched Izod impact strength of polyamides with different concentrations of SEBS-*g*-MA

Figure 1 shows how the impact strength of the higher polyamides shown in *Table 2* responds to addition of SEBS-*g*-MA. It is clear that significant toughening occurs at fairly low levels of SEBS-*g*-MA (5–10%) for these polyamides. Increasing the SEBS-*g*-MA content beyond this level does not lead to any significant enhancement in toughness. These results differ considerably from those for nylon 6 and 6,6[2,3] shown by the broken lines in *Figure 1*. In the case of nylon 6, supertough blends were obtained only through specific combinations of the maleated (SEBS-*g*-MA) and the unmaleated (SEBS) elastomers, while in the case of nylon 6,6, tough blends could be obtained with SEBS-*g*-MA alone[3]. However, for both materials nearly 20% rubber was required to achieve supertoughness.

To explore these differences further, ternary blends of the higher polyamides were prepared in which the

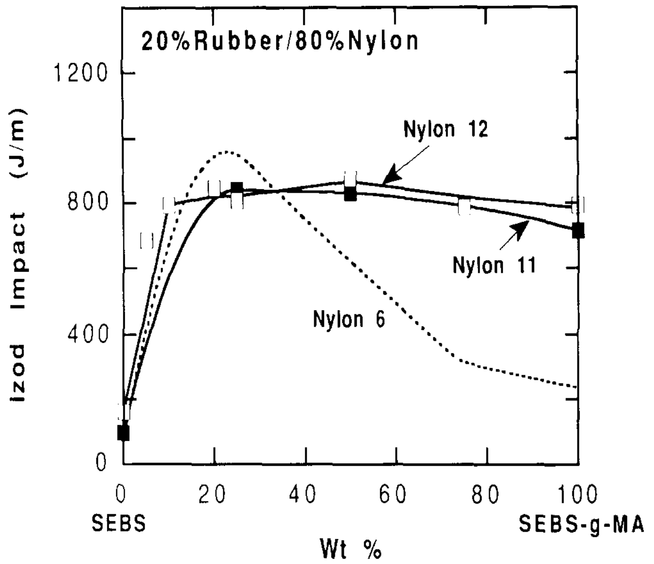


Figure 2 Notched Izod impact strength for 20% rubber/80% nylon (monofunctional) blends for various ratios of SEBS/SEBS-g-MA

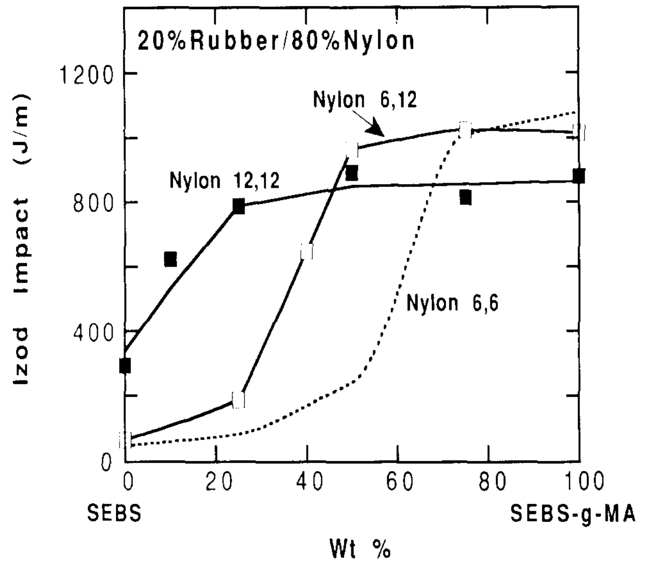


Figure 3 Notched Izod impact strength for 20% rubber/80% nylon (difunctional) blends for various ratios of SEBS/SEBS-g-MA

proportion of SEBS and SEBS-g-MA was varied while holding the total amount of rubber constant at 20%. *Figures 2* and *3* show the impact properties of the monofunctional and difunctional polyamides, respectively, as a function of the SEBS and SEBS-g-MA proportion. The data for nylon 6 and nylon 6,6 were taken from the work of Oshinski *et al.*[2,3]. For all the polyamides shown, addition of SEBS alone leads to very small increases in impact strength. For nylon 11 and nylon 12, supertough blends are generated with SEBS-g-MA alone (*Figure 2*) which is contrary to the observations for nylon 6. As in the case of nylon 6,6, addition of SEBS-g-MA alone also fully toughens nylon 6,12 and nylon 12,12. Apparently, the nylons with higher CH_2/NHCO ratio require smaller amounts of the grafted rubber (SEBS-g-MA) for effective toughening. This no doubt reflects the greater inherent ductility of the polyamide matrix and the improved interfacial coupling between the two phases as the CH_2/NHCO ratio increases. The differences in the inherent ductility of the various nylons are apparent from the data shown in *Table 2*.

Table 3 compares the Izod impact strength of various blends of the four higher polyamides under different conditions. All blends are brittle at −40°C, while some retain toughness at −20°C. A detailed analysis of the ductile–brittle transition temperatures for selected blends will be discussed later. The impact strength for most polymers is a function of the notch radius[8–10]. The standard notch, with radius of 0.01 ± 0.002 in., can be sharpened with a razor-blade to increase the stress concentration. Although the absolute impact values of all these blends were reduced by the sharp notch, this sharper notch did not lead to dramatically increased brittle failure.

Figures 4 and *5* compare the tensile properties of the different ternary blends of these higher polyamides with nylon 6 and 6,6. As expected, both the modulus and tensile strength of the ternary blends of these polyamides are lower than those of nylon 6 and 6,6. As expected,

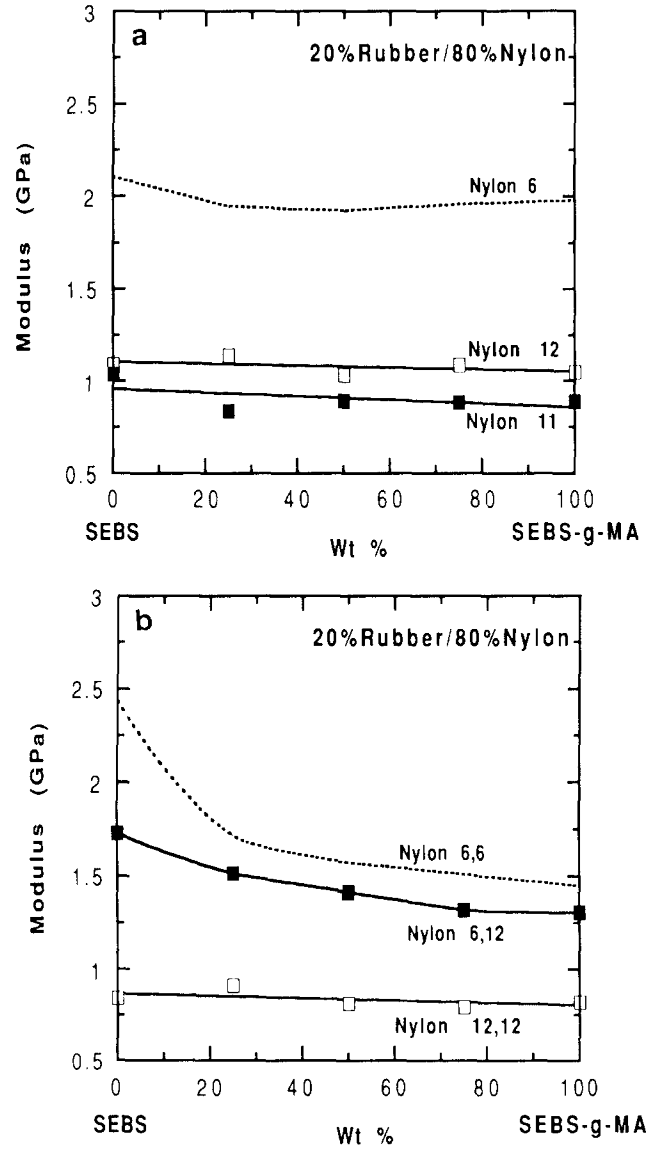


Figure 4 Tensile modulus for 20% rubber and 80% (a) monofunctional and (b) difunctional polyamide blends for various ratios of SEBS/SEBS-g-MA

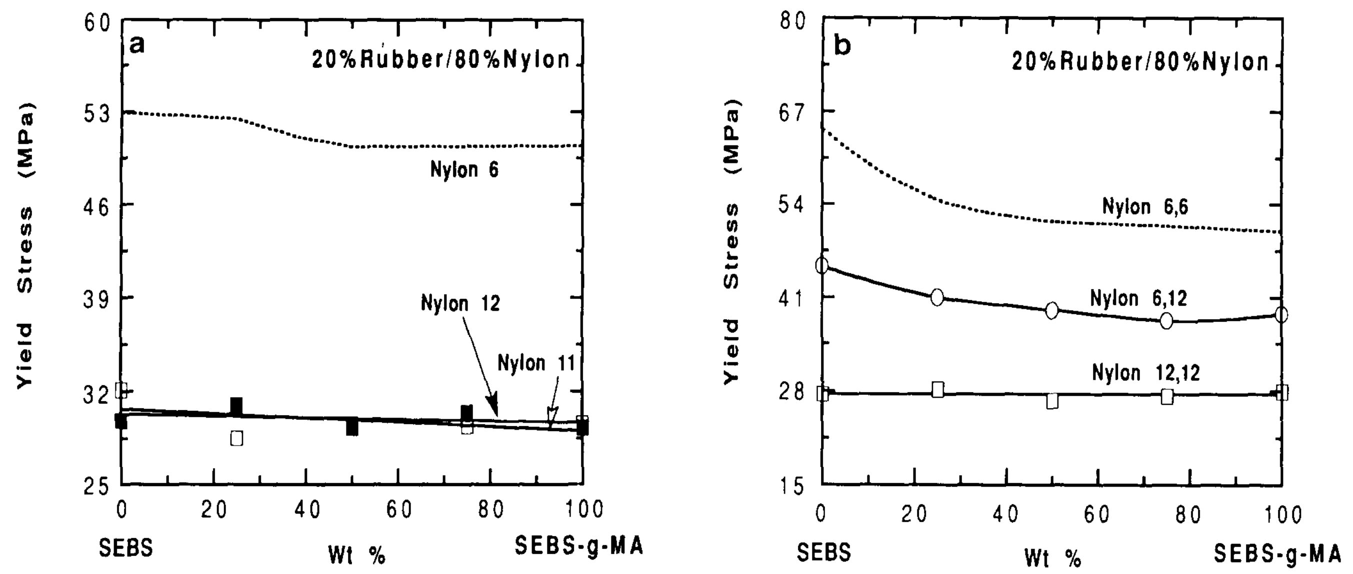


Figure 5 Tensile yield stress for 20% rubber and 80% (a) monofunctional and (b) difunctional polyamide blends for various ratios of SEBS/ SEBS-*g*-MA

Table 3 Mechanical properties of blends containing 80% nylon and 20% rubber

	Izod impact strength (J m^{-1})						
Wt% SEBS-*g*-MA in rubber	Standard notch (−40°C)	Standard notch (−20°C)	Standard notch (25°C)	Sharp notch (25°C)	Modulus (GPa)	Yield stress (MPa)	Elongation at break (%)
Nylon 6,12							
0	NT	70	68	60	1.73	45.3	43
25	NT	73	188	167	1.51	40.9	71
50	NT	148	964	810	1.41	39.2	200
75	NT	194	1024	841	1.32	37.7	265
100	NT	223	1015	767	1.30	38.7	244
Nylon 11							
0	NT	60	115	103	1.03	32.1	237
25	NT	106	730	562	0.84	28.5	350
50	NT	198	788	613	0.89	29.6	231
75	NT	155	754	617	0.89	29.4	245
100	NT	497	690	506	0.89	29.6	339
Nylon 12							
0	100	94	161	143	1.08	29.8	28
25	117	425	807	674	1.13	31.0	329
50	128	727	875	669	1.03	29.3	280
75	145	773	788	632	1.09	30.4	192
100	146	847	794	601	1.05	29.3	251
Nylon 12,12							
0	292	133	292	224	0.84	27.5	35
25	787	294	787	609	0.91	28.2	388
50	891	946	891	764	0.81	26.7	374
75	815	950	815	681	0.79	27.2	291
100	880	964	880	683	0.82	27.9	290

NT, not tested

among the ternary blends of these higher polyamides, nylon 6,12 shows a higher modulus and tensile strength across the entire spectrum of SEBS-*g*-MA/SEBS ratio owing to its least aliphatic character (lowest CH_2/NHCO ratio). Also, as the amount of SEBS-*g*-MA is increased in some of these polyamide blends (nylon 6,12, nylon 6 and nylon 6,6) keeping the total amount of rubber constant, a clear reduction in the magnitude of the tensile modulus and the yield strength is observed. For the other polyamides used in this work this decline is less apparent. Such trends may be related to changes in the crystalline structure of the matrix as the SEBS-*g*-MA/SEBS ratio (hence, extent of grafting) is varied. For the higher polyamides, such trends are less apparent. It is beyond the scope of this present work, however, to investigate these differences further. All four of these polyamides have very high elongation-at-break values when the rubbery phase is SEBS-*g*-MA (*Table 3*).

DUCTILE–BRITTLE TRANSITION TEMPERATURE

An abrupt transition from ductile to brittle behaviour is often observed as rubber content, blend morphology, testing rate, notch radius, temperature, etc. are varied. Many of these ductile-to-brittle transitions have been studied in varying degrees of detail for blends of polyamides with elastomers grafted with maleic anhydride[2–7,11–14]. For both maleated EPDM (ethylene/propylene/diene monomer rubber) and SEBS, upper and lower critical limits on rubber particle size for toughening have been established. Within this size range, the lowest ductile–brittle transition temperatures are obtained[11,14].

The preceding paper has shown that for binary blends of nylon *x,y* materials with SEBS-*g*-MA prepared in a single-screw extruder, there is a steady decrease in the particle size with increasing CH_2/NHCO ratio. For the nylon *x* series, the particle size does not vary significantly over the same range. *Figure 6* shows the variation of the ductile–brittle transition temperature over a wide range of CH_2/NHCO ratios for various monofunctional and difunctional polyamides. Interpretation of the trends shown in *Figure 6* is complicated by the fact that both rubber size and inherent ductility of the matrix are changing simultaneously in these blends. The latter may be the dominant issue since a single curve seems to describe most of the results even though there is a difference in particle size between the monofunctional and difunctional polyamide blends. Nylon 6 clearly emerges as an exception, but it must be recalled that the rubber particle size in this blend is below the lower critical limit for toughening[2]. Unfortunately, other monofunctional polyamides are not available to probe possible differences that may exist in the gap between nylon 6 and nylon 11.

MORPHOLOGY

The preceding paper described the morphology generated in binary blends of different polyamides and SEBS-*g*-MA. Here, we extend this to include ternary blends of nylon 6,12, nylon 11, nylon 12 and nylon 12,12 with SEBS and SEBS-*g*-MA since this will be useful in understanding the relationship between mechanical properties and morphology in these systems. *Figures 7–10* show TEM photomicrographs for several blend series. For binary blends of these polyamides with SEBS, large particles (more than 4 μm in diameter) similar to those observed in blends with nylon 6 and nylon 6,6[2,3] are generated (*Figures 7e* and *9e*) in all cases. For binary blends of these polyamides with SEBS-*g*-MA, different morphological features emerge depending on the functionality of the polyamide with respect to its reaction with maleic anhydride, as pointed out in the preceding paper[1]. For the monofunctional polyamides (nylon 11 and nylon 12), small, nearly spherical rubber particles are generated (*Figures 8a* and *9a*), while in the case of difunctional polyamides, significantly more complex, larger particles are formed (*Figures 7a* and *10a*). For ternary blends with nylon 11 and nylon 12 containing both SEBS and SEBS-*g*-MA, rubber particles which are essentially spherical in shape with different particle size distributions are generated across the entire range of concentrations investigated here (*Figures 8b–d*, *9b–d*). For ternary blends based on nylon 6,12 or nylon 12,12, the rubber particles exhibit varying levels of complexity and sizes depending on the SEBS-*g*-MA/SEBS ratio as shown by *Figures 7b–d* and *10b–d*. A combination of spheroidal and complex stringy particles is observed in these ternary blends. As the blend includes more SEBS-*g*-MA, the proportion of spheroidal particles decreases while that of the more complex ones increases. This is similar to what has been observed previously for nylon 6,6[3].

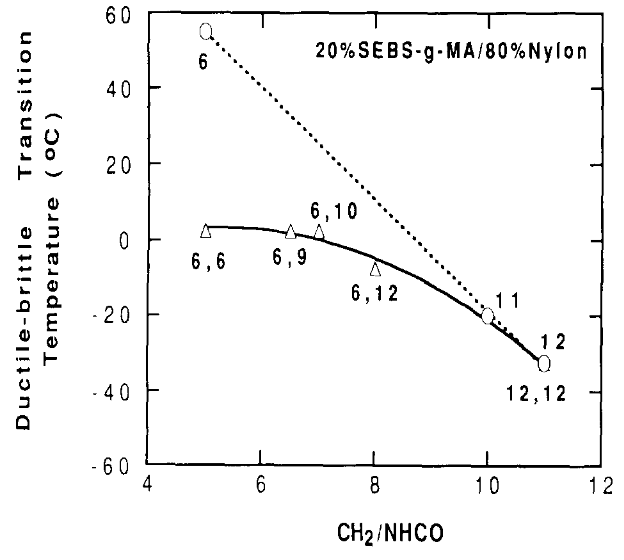
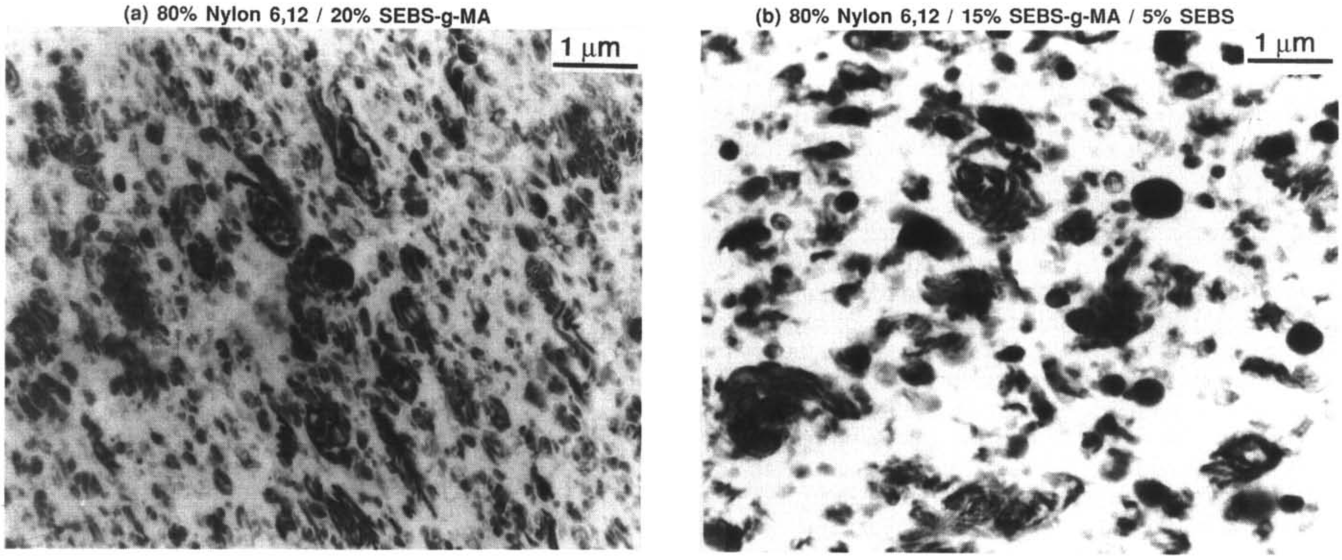


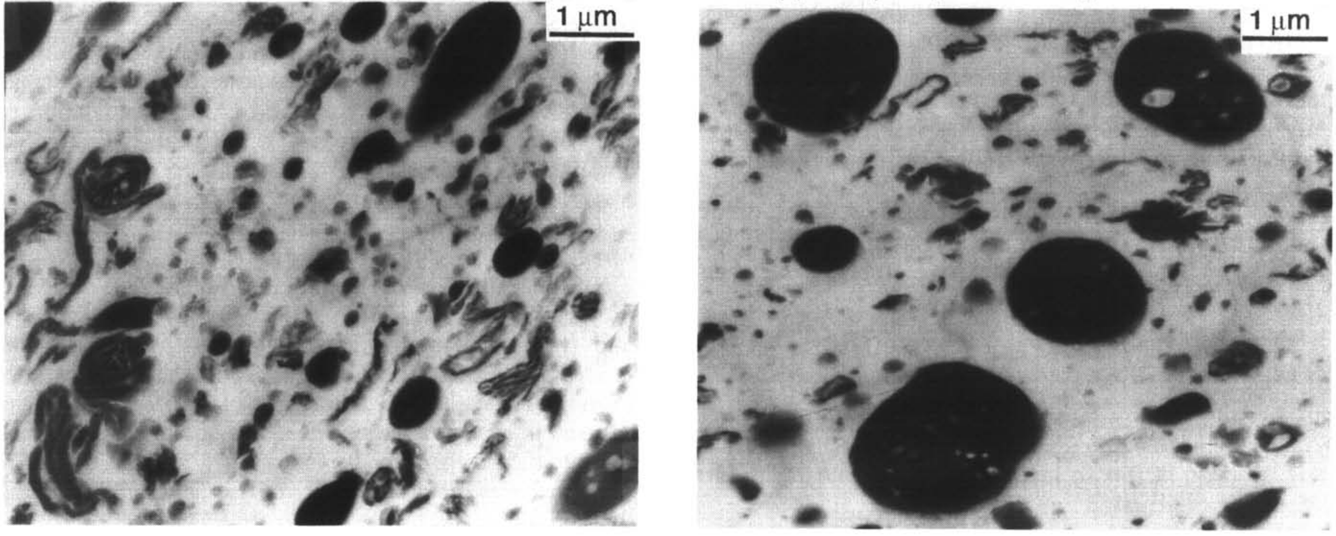
Figure 6 Ductile/brittle transition temperature as a function of polyamide CH_2/NHCO ratio for blends containing 20% rubber/80% nylon

The weight-average particle diameter, $\bar{d}_w$, is plotted *versus* the composition of the rubber (sum of SEBS and SEBS-*g*-MA is 20% of the blend) in *Figure 11* for various polyamide matrices. The particle size data for nylon 6 and nylon 6,6 are from the earlier work of Oshinski *et al.*[2,3]. For each polyamide, there is a steady decrease in the particle size as the fraction of SEBS-*g*-MA in the rubber phase increases. The lines for nylon 6,6 and nylon 6 blends form the upper and lower limits of a zone that includes all the other polyamides. Among the difunctional polyamides shown in *Figure 11*, nylon 6,6 has the lowest CH_2/NHCO ratio, which, according to the arguments advanced in the previous paper[1], leads to the generation of larger particles than other polyamides of this type having higher CH_2/NHCO ratio. Nylon 12,12, with the highest CH_2/NHCO ratio, leads to the smallest particles among the difunctional polyamides. Among the monofunctional polyamides, the particles in nylon 6 are the smallest while those with nylon 11 are the largest. Comments about this trend have been offered in ref. 1.

Recently, it has become apparent that there is a lower limit on particle size ($\bar{d}_{min}$) for room temperature toughening of nylon 6 using functionalized rubber[2,11] in addition to the better known upper limit ($\bar{d}_{max}$) for nylon 6,6 and nylon 6. There appears to be no information about such limits for polyamides other than nylon 6 and nylon 6,6; however, it is quite likely that these limits would depend on the repeat unit structure of the polyamide, e.g. the CH_2/NHCO ratio. *Figure 12* organizes all the current data and that from previous papers[1–3] in a manner that addresses this issue. The particle sizes generated to not encompass a broad enough range to define upper and lower critical limits in most


(a) 80% Nylon 6,12 / 20% SEBS-g-MA

(b) 80% Nylon 6,12 / 15% SEBS-g-MA / 5% SEBS


(c) 80% Nylon 6,12 / 10% SEBS-g-MA / 10% SEBS

(d) 80% Nylon 6,12 / 5% SEBS-g-MA / 15% SEBS

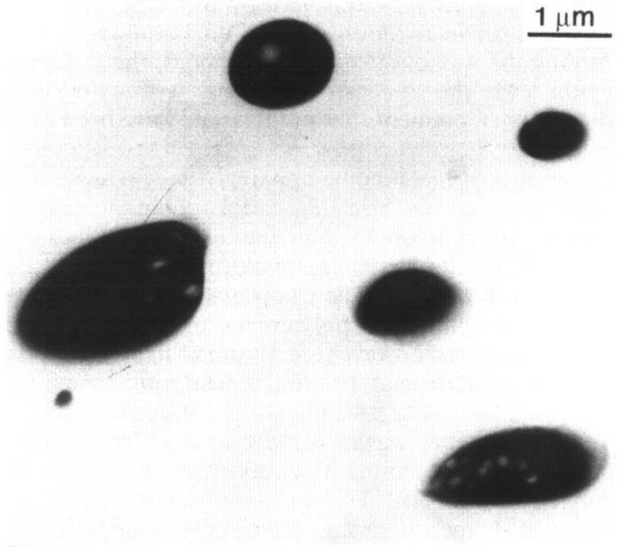
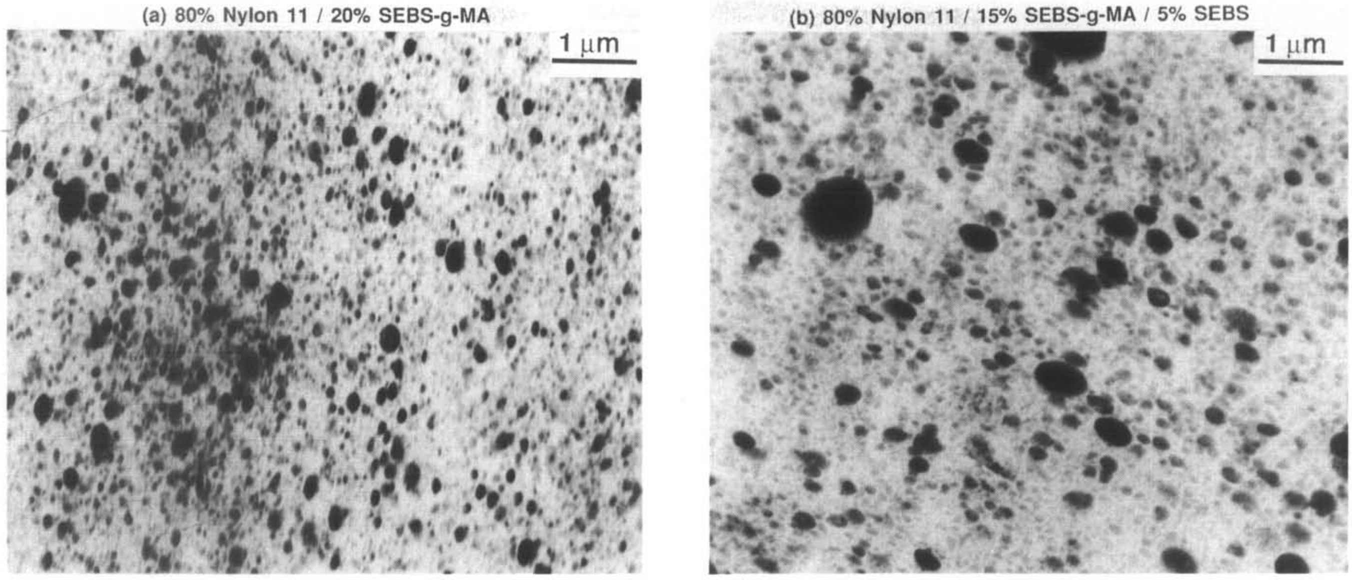
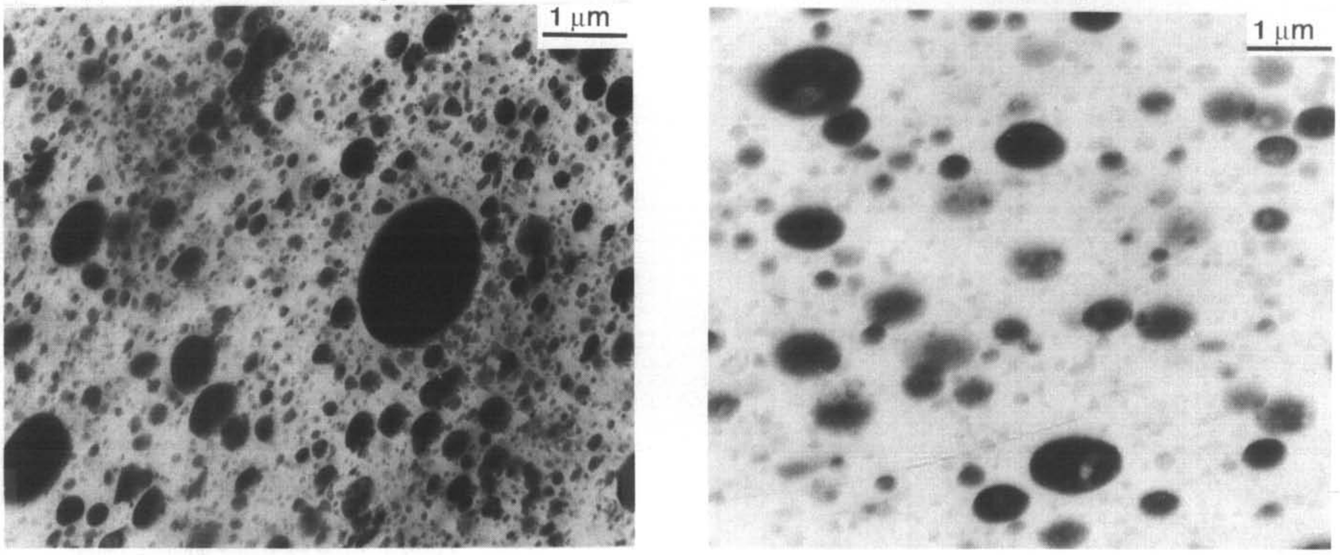

(e) 80% Nylon 6,12 / 20% SEBS

Figure 7 TEM photomicrographs of 20% rubber/80% nylon 6,12 blends at various ratios of SEBS/SEBS-*g*-MA. Samples were cryogenically microtomed from injection-moulded bars and stained with RuO_4



(a) 80% Nylon 11 / 20% SEBS-g-MA

(b) 80% Nylon 11 / 15% SEBS-g-MA / 5% SEBS



(c) 80% Nylon 11 / 10% SEBS-g-MA / 10% SEBS

(d) 80% Nylon 11 / 5% SEBS-g-MA / 15% SEBS

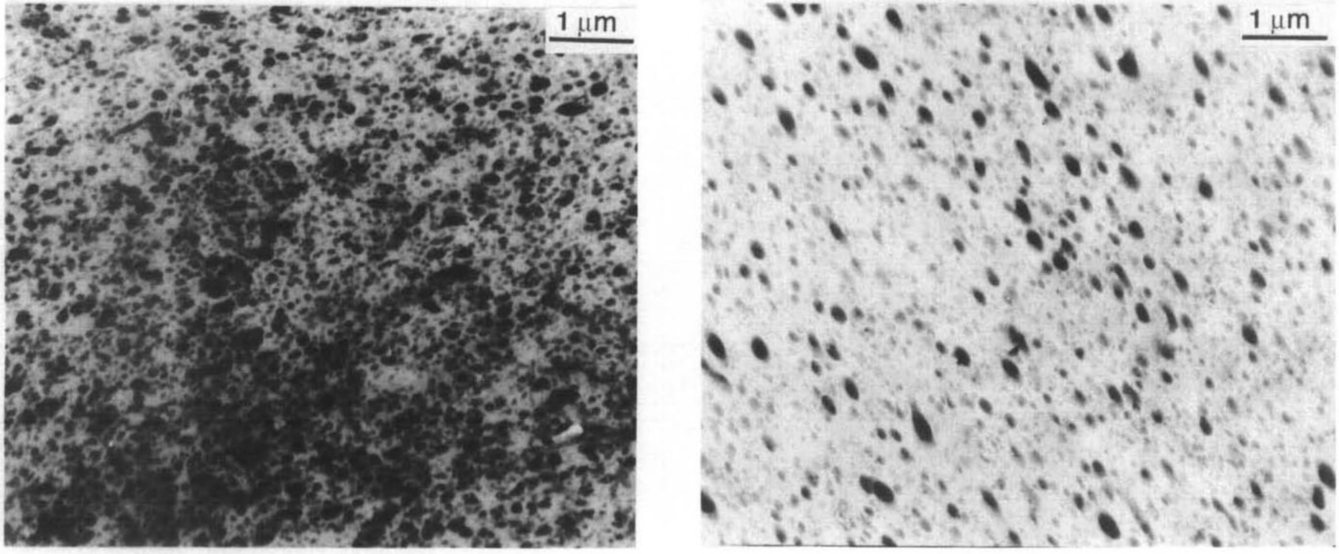
Figure 8 TEM photomicrographs of 20% rubber/80% nylon 11 blends at various ratios of SEBS/SEBS-*g*-MA. Samples were cryogenically microtomed from injection-moulded bars and stained with RuO_4

cases. However, it can be seen that the room temperature upper limit, $\bar{d}_{max}$, for nylon 6,12 (CH_2/NHCO ratio = 8) is about the same as that for nylon 6 and nylon 6,6 while the room temperature lower limit, $\bar{d}_{min}$, for one particular molecular weight grade of nylon 11 (CH_2/NHCO = 10) investigated in the preceding paper is apparently substantially lower than that for nylon 6.

CONCLUSIONS

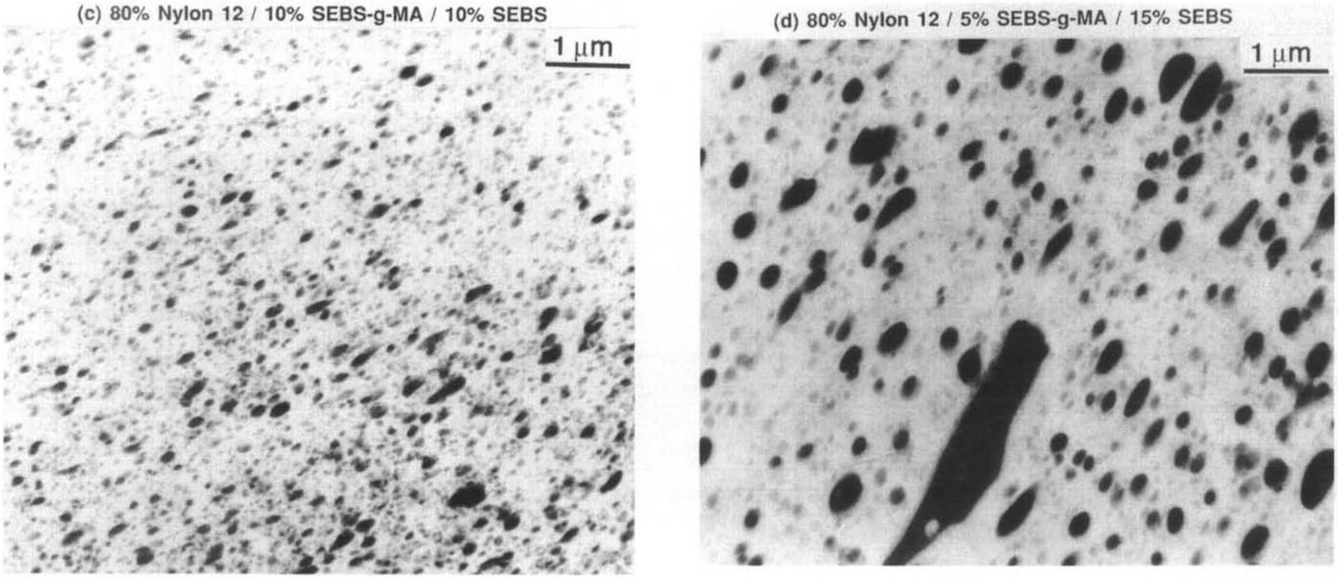
Compared to nylon 6 and nylon 6,6, it is easier to toughen the polyamides used in this work that have a higher CH_2/NHCO ratio. Relatively small concentrations of SEBS-*g*-MA can toughen nylon 6,12, nylon 11, nylon 12 and nylon 12,12. For ternary blends of these polyamides with SEBS and SEBS-*g*-MA, toughening is produced with much lower concentrations of SEBS-*g*-MA in the rubber phase. It is believed that these differences stem mainly from the inherently more ductile nature of the matrix polyamide as the CH_2/NHCO ratio is increased (see *Table 2*). Further evidence of this is shown by the trends in ductile–brittle transition temperatures of the binary blends of various polyamides with SEBS-*g*-MA (*Figure 6*).

TEM analysis of the ternary polyamide/SEBS/SEBS-*g*-MA blends revealed that the morphology generated in these blends depends on the functional character of the polyamide in a manner similar to that observed previously for nylon 6 and nylon 6,6[2,3]. The monofunctional polyamides exhibit nearly spherical particles across the entire range of SEBS/SEBS-*g*-MA ratios used in the rubber phase, while in the case of the difunctional polyamides, complex stringy particles were produced when SEBS-*g*-MA was present in the rubber phase. As explained previously[2,3], the polyamides with only one amine end per molecule (monofunctional) can only endlink with maleated rubber particles, which leads to efficient dispersion of the rubber in the polyamide matrix, while those polyamides containing molecules with two



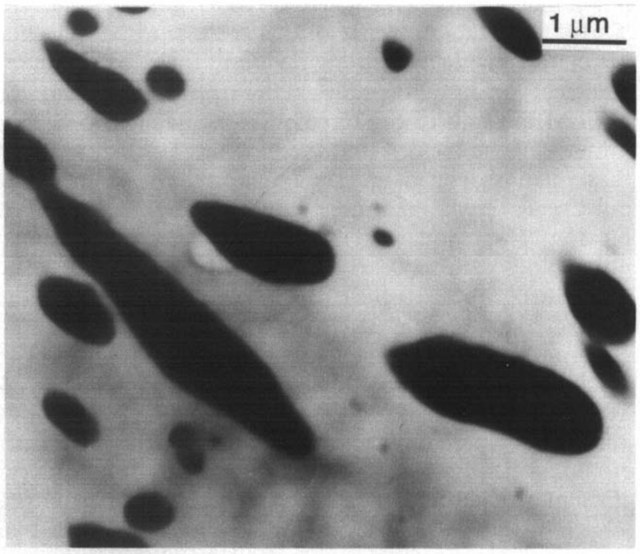
(a) 80% Nylon 12 / 20% SEBS-g-MA

(b) 80% Nylon 12 / 15% SEBS-g-MA / 5% SEBS



(c) 80% Nylon 12 / 10% SEBS-g-MA / 10% SEBS

(d) 80% Nylon 12 / 5% SEBS-g-MA / 15% SEBS

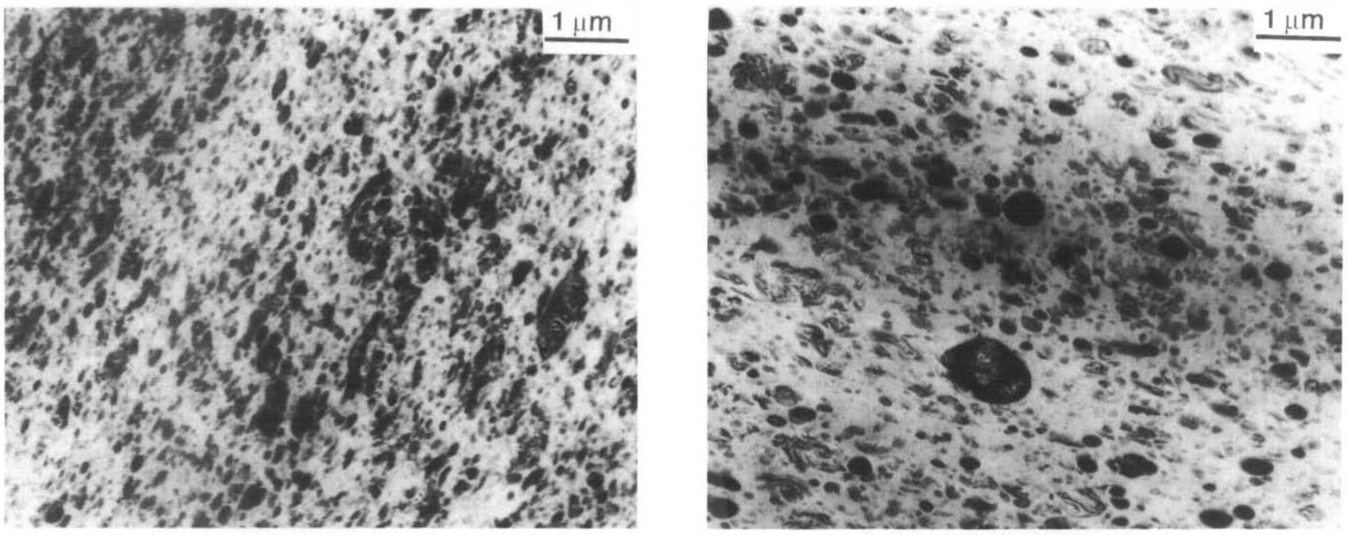


(e) 80% Nylon 12 / 20% SEBS

Figure 9 TEM photomicrographs of 20% rubber/80% nylon 12 blends at various ratios of SEBS/SEBS-*g*-MA. Samples were cryogenically microtomed from injection-moulded bars and stained with RuO_4

(a) 80% Nylon 12,12 / 20% SEBS-g-MA

(b) 80% Nylon 12,12 / 15% SEBS-g-MA / 5% SEBS



(c) 80% Nylon 12,12 / 10% SEBS-g-MA / 10% SEBS

(d) 80% Nylon 12,12 / 5% SEBS-g-MA / 15% SEBS

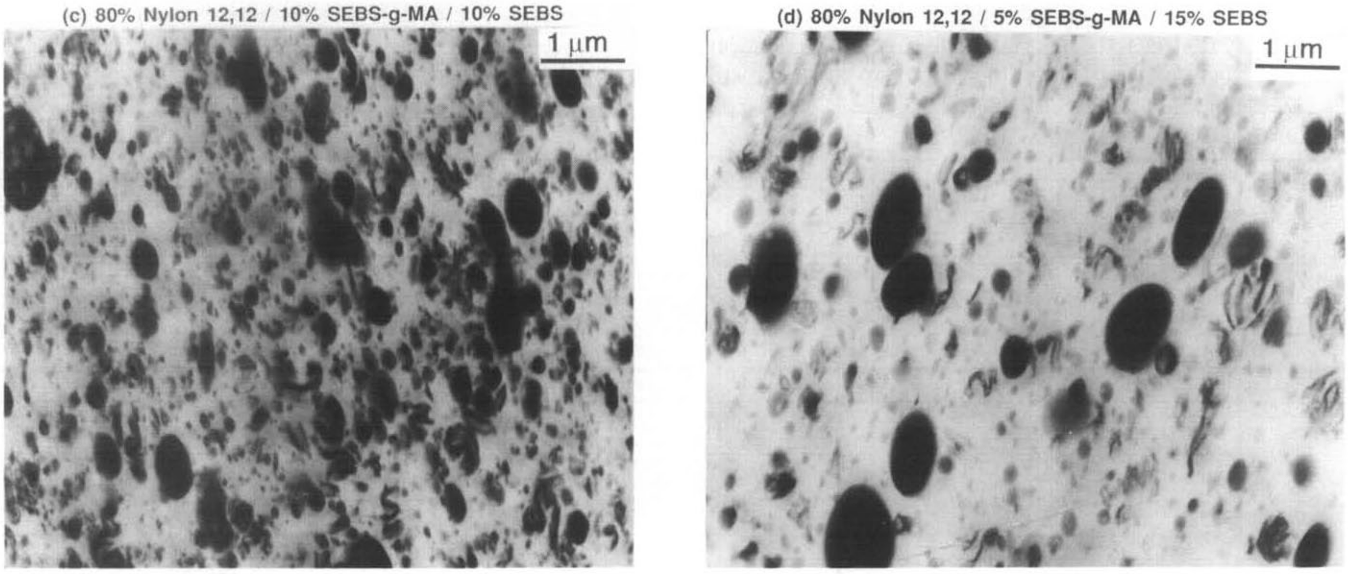


Figure 10 TEM photomicrographs of 20% rubber/80% nylon 12,12 blends at various ratios of SEBS/SEBS-*g*-MA. Samples were cryogenically microtomed from injection-moulded bars and stained with RuO_4

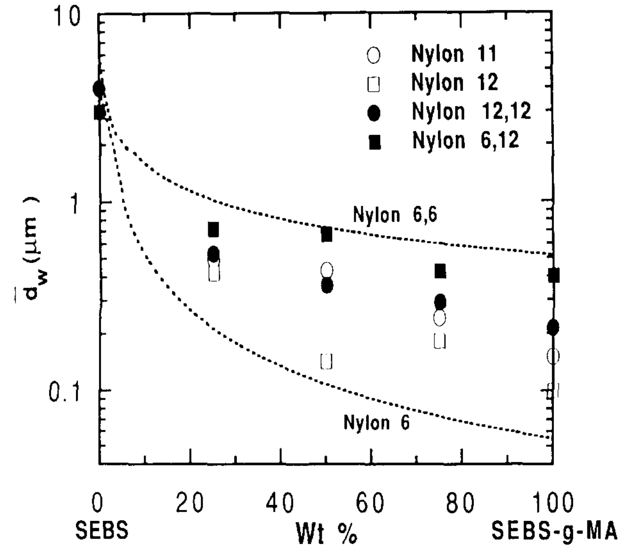


Figure 11 Variation in particle size with SEBS/SEBS-*g*-MA ratio for 20% rubber/80% nylon blends

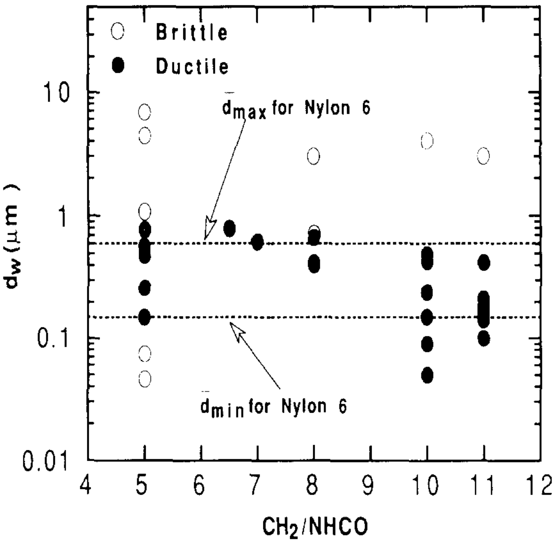


Figure 12 Effect of polyamide CH_2/NHCO ratio and rubber particle diameter (varied by the SEBS/SEBS-*g*-MA ratio) on the ductile–brittle behaviour at room temperature for blends containing 20% total rubber. Data for nylon 6 and nylon 6,6 are from previous studies[1–3]. The broken lines show the minimum and maximum rubber particle sizes between which nylon 6 blends are supertough at room temperature

amine ends undergo crosslinking-type reactions (loops on or bridges between rubber particles) that make dispersion more difficult, at least in a single-screw extruder.

From the particle size measurements made in this and the preceding paper[1], it is clear that at least the lower critical limit on the rubber particle size depends on the repeat unit structure of the polyamide (the CH_2/NHCO ratio). For polyamides with a greater aliphatic character than nylon 6 and nylon 6,6, the presence of a more ductile nylon matrix in the blend possibly leads to a shift in the critical limits for the rubber particle size required for effectively toughening these materials.

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