# Toughening of Nylon 6 with Core-Shell Impact Modifiers: Effect of Matrix Molecular Weight

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#### **SYNOPSIS**

Rubber particle size is an important issue in toughening of engineering thermoplastics. Use of core-shell impact modifiers offers the advantage of a predetermined particle size; however, these particles must be appropriately dispersed in the matrix polymer to be effective for toughening. Recent work has shown that core-shell modifiers having a poly(methyl methacrylate) (PMMA) shell can be dispersed in nylon 6 with the aid of certain styrene/maleic anhydride (SMA) copolymers. These materials are miscible with PMMA and can also react with polyamides during melt processing. Enhanced interaction between the rubber and matrix phases as a result of the formation of *in situ* graft copolymers at the interface was suggested to contribute to the improved dispersion. However, rheological issues also influence the dispersion of core-shell modifier particles in the matrix. This article examines the influence of the matrix melt viscosity on the dispersion of the core-shell particles in the nylon 6 matrix and the resulting mechanical properties of the blends using four nylon 6 materials of different molecular weights. © 1996 John Wiley & Sons, Inc.

#### INTRODUCTION

Polyamides like nylon 6 are readily toughened by blending with rubber provided that an appropriate rubber particle size, or interparticle distance, is established during mixing and there is adequate adhesion between the polyamide and rubber phases.<sup>1-9</sup> Factors that influence adhesion, e.g., physical interactions or chemical reactions between the two phases, also generally have a strong effect on blend morphology; thus, it is usually not possible to vary interphase adhesion and particle size in a totally independent way. Furthermore, the absolute and relative rheological characteristics of the matrix and rubber phases are important factors during mixing that determine the final rubber particle size in the blend.<sup>10-14</sup>

Rather than establishing the rubber particle size during the blending operation, it is possible to do this independently using core-shell impact modi-

fiers.<sup>15</sup> Such impact modifiers are usually produced by a multistage emulsion polymerization. Typically, the core is an acrylic or butadiene-based rubber over which a hard shell, often of poly(methyl methacrylate) (PMMA), is formed to aid isolation of the particles among other reasons. The size of such particles can be precisely controlled during emulsion polymerization, so that, in principle, the exact optimum particle size, for a given matrix, can be formed without regard to details of the subsequent mixing process. This, of course, assumes that it is possible to mix the impact modifier into the matrix in such a way that all agglomerates of these particles are broken up and that the particles are individually dispersed in the matrix. Good physical interaction, or even miscibility, of the chains forming the shell with those of the matrix often permit this ideal to be achieved; for example, a PMMA shell permits such dispersion into poly(vinyl chloride)<sup>15</sup> and polycarbonate<sup>16</sup> matrices. However, the interaction of PMMA with polyamides, and some other engineering thermoplastics, do not so readily permit adequate dispersion of core-shell impact modifiers in these materials. Our recent work<sup>17</sup> has shown that

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core-shell impact modifiers can be dispersed in nylon 6 with the aid of a polymer additive that is miscible with the chains of the shell layer and that chemically reacts with the polyamide. For PMMA shells, certain styrene/maleic anhydride (SMA) copolymers satisfy both requirements and are useful in the production of supertough nylon 6 compositions containing core-shell modifiers of optimum particle size.

The degree to which core-shell modifier particles can be dispersed individually into a polymer matrix depends on certain aspects of the mixing process and the rheological properties of the matrix. A recent article<sup>18</sup> described the dispersion of a core-shell impact modifier in nylon 6 using a twin-screw extruder at high shear conditions. Similarly, the more viscous the matrix, the larger the stress that can be applied to aggregates of emulsion particles for breaking them up.<sup>10-14</sup> Thus, to some extent, the matrix viscosity is expected to play a role in the dispersion of coreshell particles into nylon 6 assisted by SMA copolymers.<sup>12,14</sup> This article explores this issue and some of its ramifications.

#### EXPERIMENTAL

Table I summarizes pertinent information about the various nylon 6 materials used here. The numberaverage molecular weights of these nylon 6 materials were determined by end-group titration techniques.<sup>19</sup> The nylon 6 used in our previous work, Capron 8207F, has a number-average molecular weight of about 22,000. The core-shell impact modifiers, Paraloid EXL 3300 (EXL 3300) and Paraloid EXL 3607 (EXL 3607) from Rohm and Haas Co., are the same as those used in the previous work.<sup>17</sup> EXL 3300 has an *n*-butyl acrylate core (80% of mass) and a grafted PMMA shell (20% of mass) and a particle diameter of about 0.3  $\mu$ m. EXL 3607 has a butadiene-based

rubber core (80% of mass) grafted with a PMMA shell and a particle diameter of 0.18  $\mu$ m. A random SMA copolymer containing 8% maleic anhydride by weight (SMA8) from Arco Chemical Co. with  $\bar{M}_n$ = 100,000 and  $\overline{M}_w$  = 200,000 was used as the dispersant.

Rheological information was obtained using a Brabender Plasticorder, with a 60 mL mixing head and standard rotors, operated at 60 rpm and 240°C. Blends were prepared in a Killion single-screw extruder (L/D = 30, 2.54 cm screw diameter) at 40 rpm. The temperature of extrusion was 240°C. The pelletized extrudates were dried before injection molding in an Arburg Allrounder screw injectionmolding machine. All molded test specimens were placed in sealed plastic bags and stored in a desiccator. They were tested dry as molded. An Instron was used for tensile testing in accordance with ASTM D638 at a crosshead speed of 5.08 cm/min. An extensometer strain gauge with a 5.08 cm gap was used to obtain the modulus and yield stress values. Notched Izod impact tests were conducted according to ASTM D256. All test specimens were 0.318 cm thick.

The morphology of selected blends was examined by transmission electron microscopy (TEM) using a JEOL JEM 200CX. Ultrathin samples were obtained by microtoming molded specimens using a Riechert-Jung ultramicrotome at -80°C and stained with a 2% phosphotungstic acid aqueous solution for a period of 30 min or with  $RuO_4$  (vapor) for a maximum of 15 min.

### RHEOLOGY

Brabender torque measurements provide an approximate characterization of the melt viscosity at the conditions that exist inside the extruder. Figure 1 shows the Brabender torque of binary blends of

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Table I Physical Properties of Nylon 6 Materials of Different Molecular Weight

	$ar{M}_n^{\ st}$	Brabender Torque <sup>b</sup> (m-g)	Yield Strength (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Heat of Fusion (J/g)	Source
Capron 8202	16,700	500	68.4	2.45	90	50	63.2	Allied signal
Capron 8207F	22,000	800	72.0	2.61	145	50	60.4	Allied signal
Capron 8209F	31,450	1440	69.5	2.67	170	75	57.2	Allied signal
B-5	35,600	3040	68.1	2.47	240	100	48.8	BASF

<sup>a</sup> From end-group analysis by A. J Oshinski.

<sup>b</sup> Measured at 240°C and 60 rpm after 5 min.



Figure 1 Brabender torque response for blends of nylon 6 of different moleclar weights with SMA8 after 5 min at 240°C and 60 rpm. Dotted lines show torque values for the neat impact modifiers used here.

SMA8 with four nylon 6 materials having different molecular weights. The dashed lines represent the torque measured for the neat EXL 3300 and EXL 3607 under the same conditions, respectively. EXL 3607 has a higher torque value than that of EXL 3300. This is possibly due to the smaller particle size of EXL 3607 or other factors related to the nature of these particles. The torque values of pure Capron 8202 and Capron 8207F are much lower, while the values for Capron 8209F and B-5 are closer to that of EXL 3300. On the other hand, the torque values of all the nylon 6 materials are lower than that of EXL 3607.

For most of the nylon 6 materials, the torque increases almost linearly on the addition of SMA8. The increase in torque on the addition of SMA8 to each nylon 6 material is the result of the grafting reaction between these components.<sup>7,8,20-22</sup> It is noted from Figure 1 that torque values comparable to EXL 3300 are reached when 13% SMA8 is added to Capron 8202. The addition of only 5% SMA8 to Capron 8207F brings the torque to this level, while for Capron 8209F, only 2% SMA8 is needed. On the other hand, the nylon 6 having the highest molecular weight, i.e., B-5, has a higher torque value than that of EXL 3300 and adding more than 2.5% SMA8 actually tends to cause a reduction in torque, probably because of a change in the flow mechanism of such an elastic material. Similarly, it takes 10% of SMA8 for Capron 8207F, 4% SMA8 for Capron 8209F, and 2% SMA8 for B-5, and even higher levels of SMA8 than studied here for Capron 8202 to reach the torque level of EXL 3607 (see Fig. 1). The influence of the rheological properties of these nylon 6 materials with or without adding SMA8 on the dispersion of the core-shell rubber particles is discussed below.

#### MECHANICAL PROPERTIES

Table I shows that the nylon 6 materials used in this work all have comparable yield strength, modulus, and break elongation. While each material has a high elongation at break in a slow-speed tensile test, all of them fail in a brittle manner in the notched Izod impact test. However, Capron 8209F and B-5 have slightly higher values of impact strength, which may be related, at least in part, to their lower crystallinity as indicated by their lower heats of fusion.

As reported earlier,<sup>17</sup> the Capron 8207F material can be toughened by addition of a core-shell impact modifier dispersed with the aid of small amounts of SMA8. Table II shows the mechanical properties of

Composition							Ductile-Brittle	
% Nylon 6	% SMA8	% EXL 3300	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Transition Temperature (°C)	
80	0	20	51.2	2.11	20	80		
79	1	20	56.0	2.11	20	150	55	
78	2	20	57.3	2.19	90	600	20	
76	4	20	59.7	2.27	160	1150	-5	
75	5	20	60.0	2.33	160	1230	-10	
72.5	7.5	20	60.6	2.36	160	1340	-12.5	
70	10	20	61.3	2.37	120	1280	10	

Table II Mechanical Properties of (80 - x)% Capron 8207F + x% SMA8/20% EXL 3300 Blends



Figure 2 Effect of SMA8 content on room-temperature notched Izod impact strength and the ductile-brittle transition temperature for blends based on Capron 8207F containing 20% EXL 3300.

blends based on this nylon 6, containing 20% EXL 3300 and 0-10% SMA8, prepared by first mixing SMA8 with EXL 3300 and then incorporating the nylon 6 in a second extrusion. This mixing protocol should give the greatest opportunity for SMA8 to play its proposed function as an interfacially acting dispersant.<sup>17</sup> The binary blend of Capron 8207F with EXL 3300 is brittle due to poor dispersion of the core-shell particles.<sup>17</sup> The addition of SMA8 improves room-temperature impact strength and reduces the ductile-brittle transition temperature as shown in Figure 2. The best toughness is achieved when the SMA8 content is in the range of 4-8%; these compositions have been shown to have a good dispersion of the modifier particles in the polyamide matrix.<sup>17</sup> Binary mixtures of Capron 8207F and SMA8 in this composition range have comparable Brabender torque (melt viscosity) as the neat EXL 3300 modifier (see Fig. 1). Beyond this range of

75% Capron 8202 + 5% SMA8/20% EXL3300



**Figure 3** TEM photomicrograph of 75% Capron 8202 + 5% SMA8/20% EXL 3300 blend (+ sign indicates second extrusion step). The sample was cryogenically microtomed from a injection-molded bar and stained by RuO<sub>4</sub>.

SMA8 content, the nylon 6/SMA8 mixtures have a much larger melt viscosity than that of EXL 3300.

Table III shows the mechanical properties of blends based on Capron 8202 containing 20% EXL 3300 and 0-10% SMA8. Surprisingly, all these blends are brittle at room temperature; even at 10% SMA8, impact strength is only marginally improved. Figure 3 reveals that the core-shell particles are poorly dispersed in the matrix when 5% SMA8 is used as compatibilizer: large aggregates of particles are clearly evident. Capron 8202 has the lowest molecular weight among the four nylon 6 materials used here, and the melt viscosity of this material is below that of EXL 3300 even when more than 12% SMA8 is added. It appears that the low matrix viscosity is the cause for poor dispersion of the core-shell particles in this material and the low toughness of the blends, in spite of the proposed ability of SMA8 to act as a dispersing agent.

Figure 1 shows that neat Capron 8209F has a Brabender torque close to that of EXL 3300. Figure

Composition							Ductile-Brittle
% Nylon 6	% SMA8	% EXL 3300	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Transition Temperature (°C)
80	0	20	52.7	1.67	7	50	
78	2	20	54.1	1.90	10	100	
75	5	20	58.3	2.04	8	120	50
70	10	20	60.1	2.15	11	338	25

Table III Mechanical Properties of (80 - x)% Capron 8202 + x% SMA8/20% EXL 3300 Blends

4(a) shows that even without SMA8 the core-shell particles can be well dispersed in the Capron 8209F nylon 6 matrix. This is no doubt due to the high matrix viscosity. With 2% SMA8, there does not seem to be any substantial change in the dispersion of EXL 3300 particles, while at 5 and 10% SMA8,

80% Capron 8209 F + 20% EXL3300



Figure 4 TEM photomicrographs of Capron 8209F blends containing 20% EXL 3300 with various amounts of SMA8: (a) 80% Capron 8209F/20% EXL 3300; (b) 78% Capron 8209F + 2% SMA8/20% EXL 3300; (c) 75% Capron 8209F + 5% SMA8/20% EXL 3300; (d) 70% Capron 8209F + 10% SMA8/20% EXL 3300. All samples were stained by 2% phosphotungstic acid solution.



75% Capron 8209 F + 5% SMA8/20% EXL3300

70% Capron 8209 F + 10% SMA8/20% EXL3300



Figure 4 (Continued from the previous page)

the dispersion actually becomes poorer [see Fig. 4(b)-(d)]. This appears to be due to the large mismatch in melt viscosities shown in Figure 1 as more than 2% SMA8 is added. It is clear from Figure 5 that Capron 8209F nylon 6 can be toughened by addition of this core-shell impact modifier in the absence of SMA8. It is supertough at room temperature and has a ductile-brittle transition temperature of 0°C. However, addition of small amounts of SMA8 slightly improves the room-temperature impact strength and lowers the ductile-brittle transition temperature to  $-10^{\circ}$ C. This improvement in low-temperature toughness caused by SMA8 may be attributed to a strengthened interface or perhaps



**Figure 5** Effect of SMA8 content on room-temperature notched Izod impact strength and the ductile-brittle transition temperature for blends based on Capron 8209F containing 20% EXL 3300.

some improvement in the degree of dispersion not evident in TEM photomicrographs. As more than 3% SMA8 is added, however, the impact strength is reduced and the ductile-brittle transition temperature increases as shown in Figure 5, apparently because of the poorer dispersion seen in Figure 4. Other mechanical properties of these blends are summarized in Table IV.

The nylon 6 material designated as B-5 has a slightly higher torque than that of EXL 3300 (see Fig. 1). Similarly, the core-shell particles can be dispersed in this nylon 6 matrix without the addition of SMA8 as shown in Figure 6(a). The addition of 2% SMA8 does not seem to change the degree of dispersion significantly [see Fig. 6(b)], while adding 5% SMA8 causes what appears to be linear aggregates of some core-shell particles, as seen in Figure 6(c). Again, the reason for this observation appears to be the high matrix viscosity caused by adding 5%

SMA8. At 10% SMA8, the blend becomes so viscous that it is impossible to extrude. The mechanical properties of blends based on B-5 nylon 6 are summarized in Table V. Because of the good dispersion seen in Figure 6(a), the binary blend of B-5 type nylon 6 with the core-shell impact modifier is tough at room temperature; however, the ductile-brittle transition temperature is only 10°C. The addition of 2% SMA8 did not change the dispersion significantly as seen in Figure 6(b), but the low-temperature toughness is improved. Addition of 5% SMA8 leads to aggregation of impact-modifier particles and, consequently, to brittle blends.

From the above results, it is not clear whether it is the absolute melt viscosity of the matrix or its value relative to that of the impact modifier that influences the dispersion of the core-shell particles. This question is briefly examined here by using a core-shell material that has a higher melt viscosity, i.e., EXL 3607, due in part to its smaller particle size. Figure 1 indicates that EXL 3607 has a higher torque value than that of EXL 3300 and also of all the nylon 6 materials used here. As reported earlier,<sup>17</sup> binary blends of Capron 8207F with EXL 3607 are brittle due to poor dispersion and at least 5% SMA8 is needed to ensure good dispersion of this modifier and to render the blends tough as shown in Figure 7. On the other hand, EXL 3300 can be dispersed in the Capron 8209F matrix without the addition of SMA8 and their binary blend is tough at room temperature because Capron 8209F has a melt viscosity close to that of EXL 3300. However, Figure 8 shows that the binary blend of Capron 8209F and EXL 3607 is brittle at room temperature and 2-5% SMA8 is needed to make the blends tough at room temperature and lower. Figure 1 indicates that the binary mixtures of Capron 8209F and SMA8 in this composition range have a comparable Brabender torque as the neat EXL 3607

Composition							Ductile-Brittle	
% Nylon 6	% SMA8	% EXL 3300	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Transition Temperature (°C)	
80	0	20	52.7	1.92	160	1250	0	
79	1	20	57.4	2.12	160	1310	0	
78	2	20	57.9	2.22	150	1420	-10	
77	3	20	59.8	2.27	145	1490	-10	
75	5	20	64.8	2.74	30	1350	10	
70	10	20	60.1	2.46	20	270	30	

Table IV Mechanical Properties of (80 - x)% Capron 8209F + x% SMA8/20% EXL 3300 Blends



B5 + EXL3300 (80/20)

**Figure 6** TEM photomicrographs of blends based on B-5 nylon 6 containing 20% EXL 3300 with various amounts of SMA8: (a) 80% B-5/20% EXL 3300; (b) 78% B-5 + 2% SMA8/20% EXL 3300; (c) 75% B-5 + 5% SMA8/20% EXL 3300. All samples were stained by 2% phosphotungstic acid solution.

modifier. Adding 10% SMA8, however, leads to a brittle blend (see Fig. 8), probably due to poor dispersion caused by the large mismatch in melt

viscosity between the rubber and the matrix phases. This evidence suggests that the melt viscosity of the matrix phase relative to that of



Figure 4 (Continued from the previous page)

the impact modifier is important in the dispersal of core-shell impact modifiers into nylon 6 matrices.

## EFFECT OF MIXING PROTOCOL

As mentioned earlier, all the ternary blends were made by first mixing the core-shell impact modifier with SMA8 followed by blending with nylon 6 in a second extrusion step. This mixing protocol was proposed<sup>17</sup> to give the greatest opportunity for SMA8 to locate at the interface region and to play its role as a dispersant. Here, three mixing protocols were examined with two nylon 6 materials, Capron 8207F and Capron 8209F: (1) simultaneous blending of all three components using a single extrusion, (2)a first extrusion to blend nylon 6 and SMA8 followed by addition of EXL 3300 in a second extrusion, and (3) a first extrusion to blend SMA8 and EXL 3300 followed by addition of nylon 6 in a second extrusion.

Table VI shows that when the level of EXL 3300 is held at 20% and the SMA8 content is 5% all three mixing protocols lead to high-strength and tough blends of Capron 8207F at room temperature. However, protocol 3 gives the best low-temperature toughness. The effect of mixing order on the mechanical properties of Capron 8209F blends containing 3% SMA8 and 20% EXL 3300 is shown in Table VII. Again, all three mixing protocols lead to

Composition							Ductile–Brittle	
% Nylon 6	% SMA8	% EXL 3300	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Transition Temperature (°C)	
80	0	20	54.8	2.05	200	1210	15	
79	1	20	59.0	2.46	170	1430	0	
78	2	20	58.5	2.32	150	1380	-5	
77	3	20	60.1	2.49	120	1480	0	
75	5	20	55.2	2.57	10	70	50	
75	10	20		To	oo viscous to pro	cess		

Table V Mechanical Properties of (80 - x)% B-5 + x% SMA8/20% EXL 3300 Blends



Figure 7 Effect of SMA8 content on room-temperature notched Izod impact strength and the ductile-brittle transition temperature for blends based on Capron 8207F containing 20% EXL 3607.

high-strength and tough blends but protocol 2 gives the highest ductile-brittle transition temperature of 10°C, which is 20°C higher than that of the other two protocols. This might be expected since this protocol gives the least opportunity for SMA8 to locate at the polyamide-modifier particle interface.

### CONCLUSIONS

Good dispersion of the core-shell modifier particles is a prerequisite for effective toughening of nylon 6. From the results presented here, it is apparent that the nylon 6 molecular weight strongly influences the efficiency of dispersion of core-shell impact modifier particles in the matrix, through the effect on melt viscosity, in addition to any function of SMA8 as

75% Capron 8207F + 5% SMA8/20% EXL 3300°



Figure 8 Effect of SMA8 content on room-temperature notched Izod impact strength and the ductile-brittle transition temperature for blends based on Capron 8209F containing 20% EXL 3607.

an interfacially active dispersant. A high melt viscosity of the matrix translates into a high shear stress on aggregates of the core-shell modifier particles during processing which tends to break up particle clusters and to disperse the particles individually in the matrix. A large mismatch between the effective viscosities of rubber and matrix phases leads to poor dispersion of these core-shell particles even with the aid of a dispersant like SMA.

While matching the rheological properties of the polyamide, by adjusting its molecular weight or by adding a reactive additive such as SMA8, to that of the impact modifier is an important issue in achieving good dispersion and toughness, there is an interfacial role of such additives as suggested by the following: While EXL 3300 can be dispersed in Capron 8209F without the addition of SMA8 to pro-

160

2.33

1230

-10

Composition	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Ductile–Brittle Transition Temperature (°C)				
75% Capron 8207F/5% SMA8/20% EXL 3300 <sup>a</sup>	56.8	2.28	140	1400	0				
75% Capron 8207F/5% SMA8 + 20% EXL 3300 <sup>b</sup>	58.0	2.20	160	1260	0				

60.0

Table VIEffect of Mixing Order on Mechanical Properties of Capron 8207F Blends Containing 5%SMA8 and 20% EXL 3300

\* The three components were mixed simultaneously using a single extrusion step.

<sup>b</sup> Nylon 6 and SMA8 were extrusion-blended followed by addition of EXL 3300 in a second extrusion step.

° SMA8 and EXL 3300 were extrusion-blended followed by addition of nylon 6 in a second extrusion step.

Composition	Yield Stress (MPa)	Modulus (GPa)	Elongation at Break (%)	Izod Impact (J/m)	Ductile–Brittle Transition Temperature (°C)
77% Capron 8209F/3% SMA8/20% EXL 3300ª	58.5	2.37	150	1570	-10
77% Capron 8209F/3% SMA8 + 20% EXL 3300 <sup>b</sup>	58.7	2.18	170	1450	10
77% Capron 8209F + 3% SMA8/20% EXL 3300°	59.8	2.27	150	1490	-10

Table VIIEffect of Mixing Order on Mechanical Properties of Capron 8209F Blends Containing 3%SMA8 and 20% EXL 3300

<sup>a</sup> The three components were mixed simultaneously using a single extrusion step.

<sup>b</sup> Nylon 6 and SMA8 were extrusion-blended followed by addition of EXL 3300 in a second extrusion step.

° SMA8 and EXL 3300 were extrusion-blended followed by addition of nylon 6 in a second extrusion step.

duce tough blends, addition of 2% SMA8 improves low-temperature toughness. Mixing protocols that favor location of SMA8 in the shell of the impact modifier give the best low-temperature toughness in all cases. Finally, it was demonstrated earlier<sup>23</sup> that using a styrene/acrylic acid copolymer (SAA8), which is miscible with PMMA but reacts with nylon 6 more slowly than does SMA8 and causes only a very small increase in the matrix melt viscosity, can improve the dispersion of EXL 3300 in Capron 8207F and render tough blends.

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