Morphology of nylon 6/ABS blends compatibilized by a styrene/maleic anhydride copolymer

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Transmission electron microscopy (TEM) was used to examine the morphologies of nylon 6/ABS blends compatibilized with a styrene/maleic anhydride (SMA) copolymer containing 25% maleic anhydride (SMA 25). Several staining techniques were employed for identifying the various phases. The morphologies of a nylon 6/ABS blend compatibilized with an imidized acrylic polymer and the commercially available Triax™ material were also examined by these TEM techniques. While increasing concentration of the SMA 25 copolymer clearly leads to more efficient dispersion of the ABS phase, there is an optimum level of SMA 25 to achieve maximum toughness. Various factors that might contribute to the subsequent loss in toughness with higher SMA 25 levels are discussed. It is concluded that the limitations of the SMA 25 copolymer as a compatibilizer stem mainly from its high level of reactive functionality.

(Keywords: nylon 6; ABS; morphology)

INTRODUCTION

Compatibilization of multiphase polymer blends is frequently necessary in order to achieve optimum properties and a stable morphology. An attractive approach is to form block or graft copolymers at the domain interface during processing by *in situ* reaction of functional groups, i.e. reactive compatibilization[1–5]. One strategy is to incorporate into the blend functional polymers that are miscible with one phase and that will react with the other phase[6,7]. Blends of polyamides with ABS materials are of commercial interest[8–18] and are a useful model system for exploring this concept owing to the inherent reactivity of the polyamide and the abundant options for designing functional polymers that would be miscible with the sytrene/acrylonitrile copolymer (SAN) matrix of ABS. In an earlier paper[6], this idea was explored using styrene/maleic anhydride copolymers (SMA) and an imidized acrylic polymer as the additives. Both types can be miscible with the SAN[19–22] and have functionalities that readily react with the polyamide amine end-groups.

Some success was attained in generating tough nylon 6/ABS blends using an SMA copolymer containing 25% maleic anhydride (SMA 25)[18]. It was found that the impact strength of these compatibilized blends is extremely sensitive to the amount of SMA 25 copolymer added. Moreover, at the composition where the average Izod impact strength was a maximum, there existed a wide distribution of impact strength values among the samples tested at ambient temperature. Fracture behaviour at low temperatures was not examined.

Some attempts were made to understand the morphology of these compatibilized nylon 6/ABS blends using scanning electron microscopy (SEM); however, it was concluded that transmission electron microscopy (TEM) would be required for unambiguous characterization of the phase morphology. In this paper, we re-examine these blends in a variety of ways, but with a main focus on their morphologies as determined by TEM using several different techniques for selective enhancement of phase contrast.

This paper also briefly examines the blends compatibilized by the imidized acrylic polymer[22,23] reported by Triacca *et al.*[18]. The materials described by Triacca *et al.*[18] were not very tough; however, further work, to be reported in subsequent papers, has shown that the imidized acrylic polymers can be very effective compatibilizers for nylon 6/ABS blends, resulting in materials with excellent toughness. Examination of these previous blends here will be important in understanding their evolution to the very tough materials to be described in subsequent papers.

MATERIALS AND PROCEDURES

Table 1 summarizes the sources of and some pertinent information about the materials used in this study. The nylon 6 is a commercially available material. The ABS material, BL-65, consists of an SAN copolymer grafted to a butadiene-based latex rubber which has a broad distribution of particle sizes. The SAN contains 24% acrylonitrile (AN) by weight and only 40% of it is chemically grafted to the rubber. This material was designated as SAN-g in our previous work[18] and shall be referred to here simply as ABS. Most of the blends investigated in this work were compatibilized with a

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Table 1 Polymers used in this study

Polymer	Material/description	Composition	Molecular weight	Relative melt viscosity[a]	Source
Nylon 6	Capron 8207 F	End-group content: $NH_2=47.9\ \mu eq\ g^{-1}$; $COOH=43.0\ \mu eq\ g^{-1}$	$\bar{M}_n=22000$	1.0	Allied-Signal
ABS	SAN-grafted emulsion rubber (BL-65)	50% rubber, 24% AN in SAN[b]	$\bar{M}_n=44000$[c], $\bar{M}_w=167000$	4.0	Sumitomo Naugatuck
SMA 25	Styrene/maleic anhydride copolymer[d,e]	25% MA	Viscosity = 4.73[f]	1.6	Monsanto
Imide	Imidized acrylic polymer[e]	57 wt% imide, 1.8 wt% free acid, 1.0 wt% anhydride	~90000	3.0	Rohm and Haas

[a] Brabender torque at 240°C and 60 rev min^{-1} after 10 min relative to nylon 6

[b] The AN content reported previously was 22.5%; however, further analyses indicate 24%

[c] From gel permeation chromatography using polystyrene standards. For ABS, the information shown is for the soluble SAN

[d] Contains a small amount of a third monomer

[e] Miscible with SAN 25

[f] Viscosity (mPa s) at 25°C of a 10% solution in methyl ethyl ketone

Table 2 Comparison of impact values with past results

Composition	SMA 25 in SAN-g (%)	Average Izod impact strength ($J\ m^{-1}$) (this study)	Average Izod impact strength ($J\ m^{-1}$) (ref. 1)
Nylon 6/(SAN-g/SMA 25) 60/40	4	750	550
Nylon 6/(SAN-g/SMA 25) 60/40	6	295	270

single material, a styrenic polymer containing 25% by weight of maleic anhydride, designated here as SMA 25. An imidized acrylic polymer was also selected for limited studies in our previous work (see *Table 1*). This polymer is miscible with the matrix phase of BL-65 and has some chemical functionality (free acid = 1.8%, anhydride = 1.0%) and 57% by weight of glutarimide units. Processing conditions identical to those described in detail by Triacca *et al.*[18] were used here. For mechanical testing, standard Izod (ASTM D256) bars (thickness = 0.3175 cm) were used.

Ultrathin sections were cut from Izod bars perpendicular to the flow direction with a diamond knife using a Reichert–Jung Ultracut E microtome under cryogenic conditions (−50°C) inside the microtoming chamber. A cutting speed of 0.4 mm s^{-1} was maintained throughout the microtoming operation. The ultrathin (~50 nm) sections were then collected on a copper grid for subsequent staining operations to enhance the phase contrast for the microscopy observations. The various staining techniques utilized will be described in detail later. A JEOL 1200 EX transmission electron microscope operating at 120 kV, was used to examine the morphology of these blends.

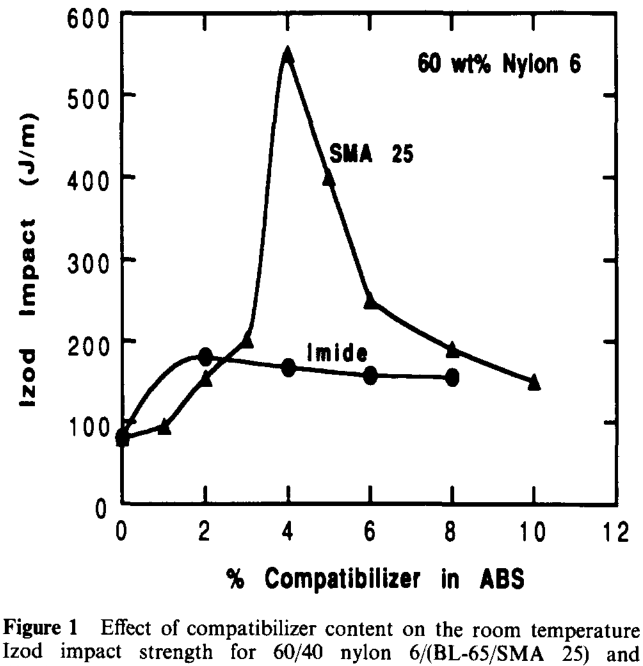

Figure 1 Effect of compatibilizer content on the room temperature Izod impact strength for 60/40 nylon 6/(BL-65/SMA 25) and 60/40 nylon 6/(BL-65/imide)

MECHANICAL PROPERTIES

Figure 1 shows how the notched Izod impact strength at room temperature for blends containing 60% nylon 6 changes with the content of the SMA 25 copolymer added to the ABS phase. A sharp maximum in impact strength is observed over a narrow range of SMA concentrations. At the composition where the maximum average impact strength was obtained, a wide variation in impact strength values from sample to sample was noted[18]. *Figure 1* also shows the impact strengths reported by Triacca *et al.*[18] using the imidized acrylic polymer as the compatibilizer. None of the latter blends approached the supertough range.

As part of this continuing investigation, the mechanical properties of selected compositions of nylon 6/ABS blends using SMA 25 as the compatibilizer were

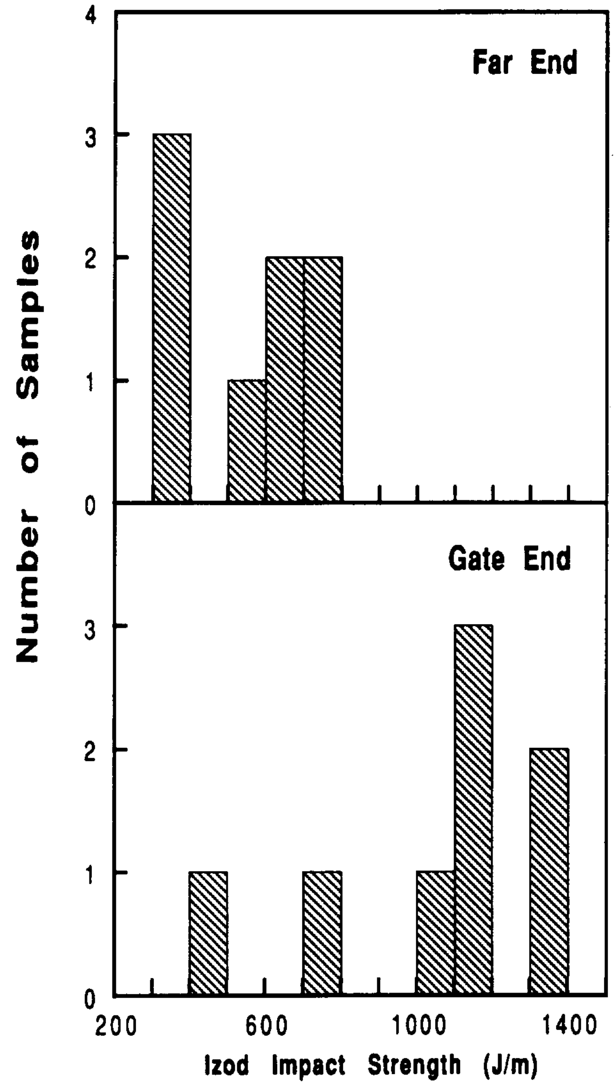

Figure 2 Sample-to-sample distributions of impact strength values for samples of the 60/40 nylon 6/(BL-65/SMA 25 96/4) blend taken from the far and gate ends of Izod bars

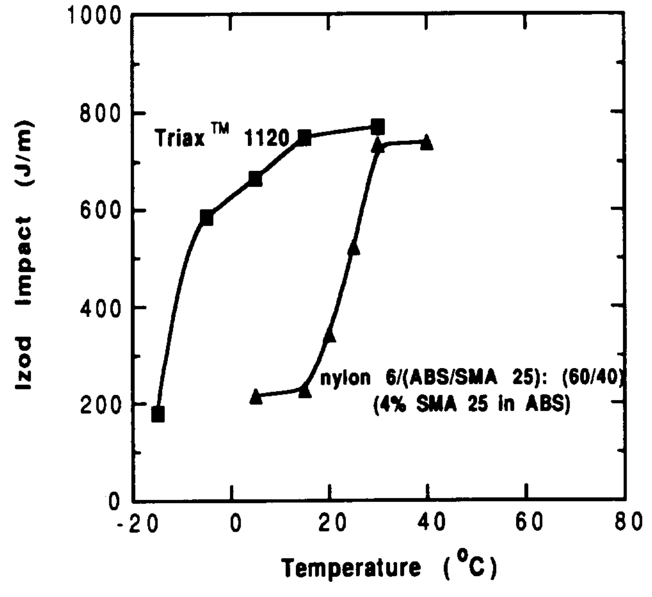

Figure 3 Izod impact strength *versus* temperature for the 60/40 nylon 6/(BL-65/SMA 25 96/4) blend and the commercial Triax™ 1120 material

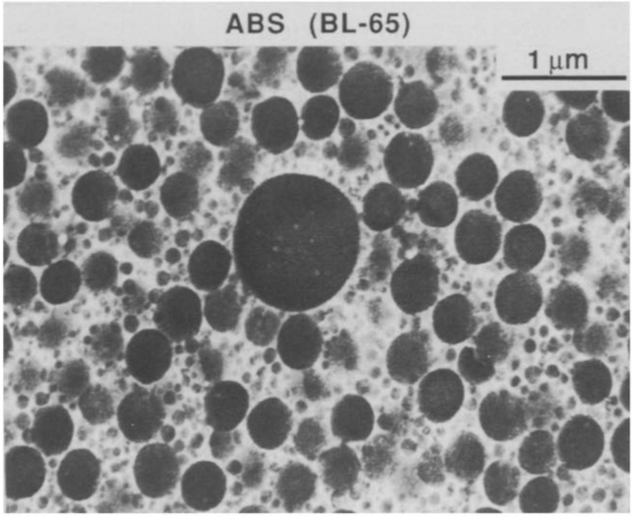

Figure 4 TEM photomicrograph of the commercial ABS material designated as BL-65. The sample was stained with OsO_4

re-examined. *Table 2* compares two compositions containing 4% and 6% SMA 25 pre-mixed with the ABS phase prior to blending with nylon 6. The current results agree rather well with the previous observations. The slightly higher average value for the impact strength obtained here at 4% SMA 25 is probably related to the large sample-to-sample variability, as reported previously at this composition[18]. As shown by the histograms in *Figure 2*, there is a significant variation in the impact strength among the limited number of samples prepared here. The injection-moulded Izod bars were cut in half and each end was tested. The gate end displays slightly higher values of impact strength than the end further away from the gate. Flexman[24] attributed similar effects in toughened polyamide blends to morphological differences which exist between the different ends of an injection-moulded Izod bar owing to thermal gradients within the mould.

Figure 3 shows the Izod impact strength for the nylon 6/ABS blend compatibilized with 4% SMA 25 (at the maximum room temperature toughness in *Figure 1*) as a function of temperature. At ambient temperature conditions this material is just above the ductile-to-brittle transition, which probably explains the wide variation in the impact strength values shown in *Figure 2*. *Figure 3* also shows the corresponding results for a commercial nylon/ABS blend[10], Triax™ 1120, which clearly has a much lower ductile-to-brittle transition temperature.

MORPHOLOGY

Figure 4 shows the TEM photomicrograph for the ABS material, BL-65, used in this work. The butadiene rubber particles in this blend were stained by placing the microtomed sections on a drop of 2% aqueous OsO_4 solution for a period of 3 min. It is clear from this TEM photomicrograph that the rubber particles have a broad distribution of sizes. There is a population of rubber particles with an average size of ~0.05 μm along with a separate population with an average particle size of ~0.2 μm. Occasionally, particles with diameters of 1 μm or more were also observed in some of the TEM photomicrographs.

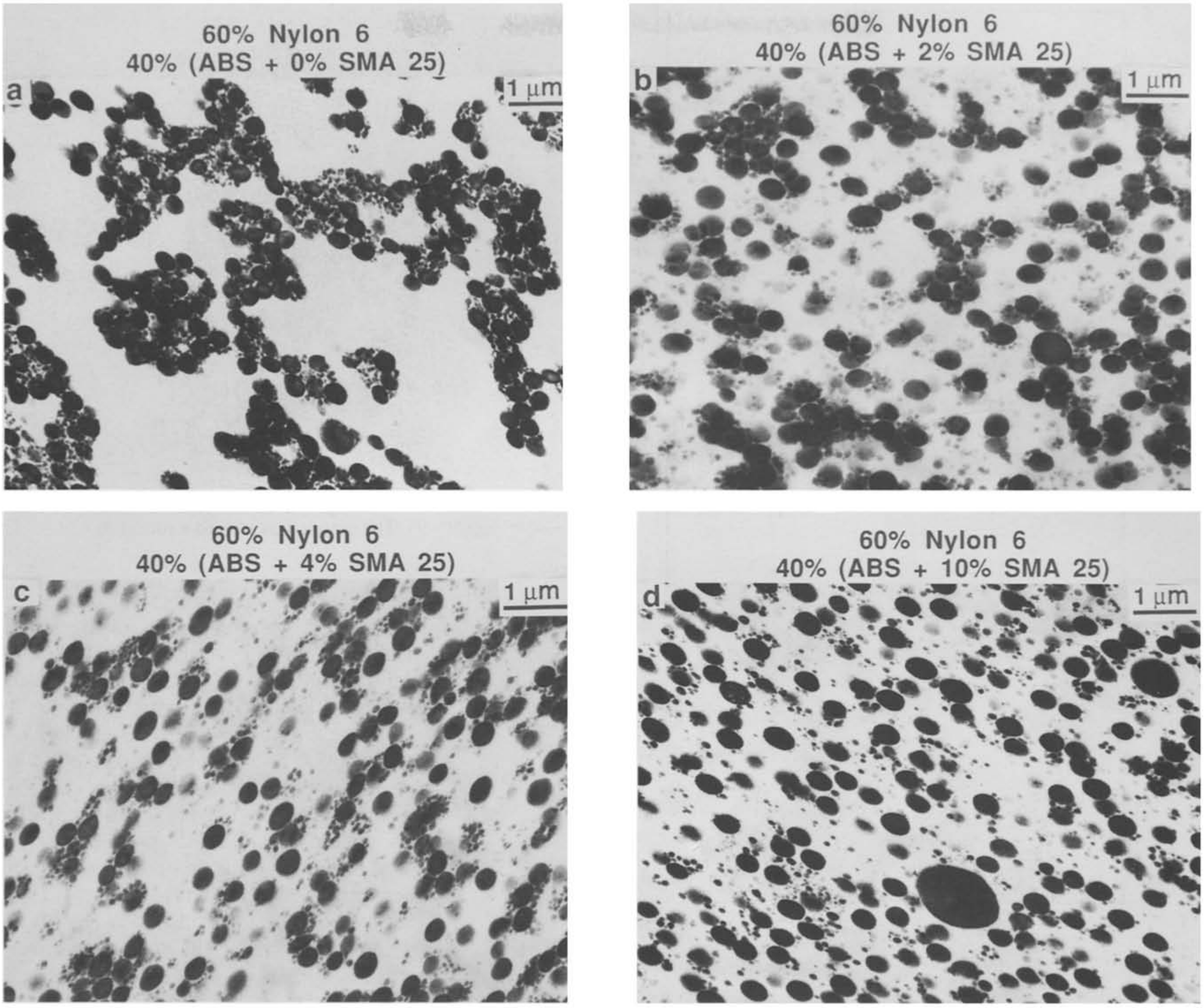

Figure 5 TEM photomicrographs for 60% nylon 6 blends with BL-65 containing (a) 0%, (b) 2%, (c) 4% and (d) 10% SMA 25 copolymer in the ABS phase. The samples were stained with OsO_4

Nylon 6/ABS blends compatibilized with SMA 25

In this section, the morphologies of nylon 6/ABS blends compatibilized with SMA 25 are examined by TEM utilizing several different staining techniques. *Figure 5* shows photomicrographs of the series of blends containing 60% nylon 6 with varying amounts of SMA 25. These samples were stained by exposure to vapours from a 2% aqueous OsO_4 solution for at least 8 h under ambient conditions. For the blend without any SMA 25 compatibilizer, the butadiene rubber particles of the ABS exist in certain regions, which are presumably ABS phases, and are entirely absent from other regions, which must consist primarily of nylon 6 (*Figure 5a*). *Figures 5b–d* show improved dispersion of the ABS domains with increasing SMA 25 concentration. However, it is not possible using this staining technique to distinguish clearly between the SAN phase in the ABS material and the nylon 6, since these materials are not stained by OsO_4.

A two-step staining protocol using OsO_4 and RuO_4 gives further insights into the morphologies of these blends. The microtomed sections were first placed on a drop of 2% aqueous OsO_4 solution for a period of 3 min followed by exposure to vapours from a 0.5% RuO_4 solution for a period of 10 min under ambient conditions. The OsO_4 stains the butadiene rubber particles, while the RuO_4 is expected to stain the styrenic polymers (SAN and SMA)[25–28]. *Figure 6* shows TEM photomicrographs for a series of nylon 6/(ABS/SMA 25) blends stained in this way. Large ABS domains are clearly apparent in the uncompatibilized nylon 6/ABS blend (*Figure 6a*); this technique reveals the spherical butadiene domains (stained by OsO_4) inside the more lightly stained SAN phase (stained by RuO_4). Although there is a significant improvement in the dispersion of the ABS phase for the blend containing 2% SMA 25 in the ABS (*Figure 6b*) compared to the uncompatibilized blend, most ABS domains contain numerous rubber particles. The number of rubber particles per cluster is significantly diminished when the concentration of the SMA 25 copolymer in the ABS phase is further increased (*Figures 6c–e*).

A third strategy for examining the morphologies of these blends involved staining the polyamide phase by floating the samples on a 2% phosphotungstic acid solution[29–31] for 30 min at ambient temperature. The morphology of the uncompatibilized blend as revealed by this technique (see *Figure 7a*) is consistent with the observations made using the previous two techniques (see

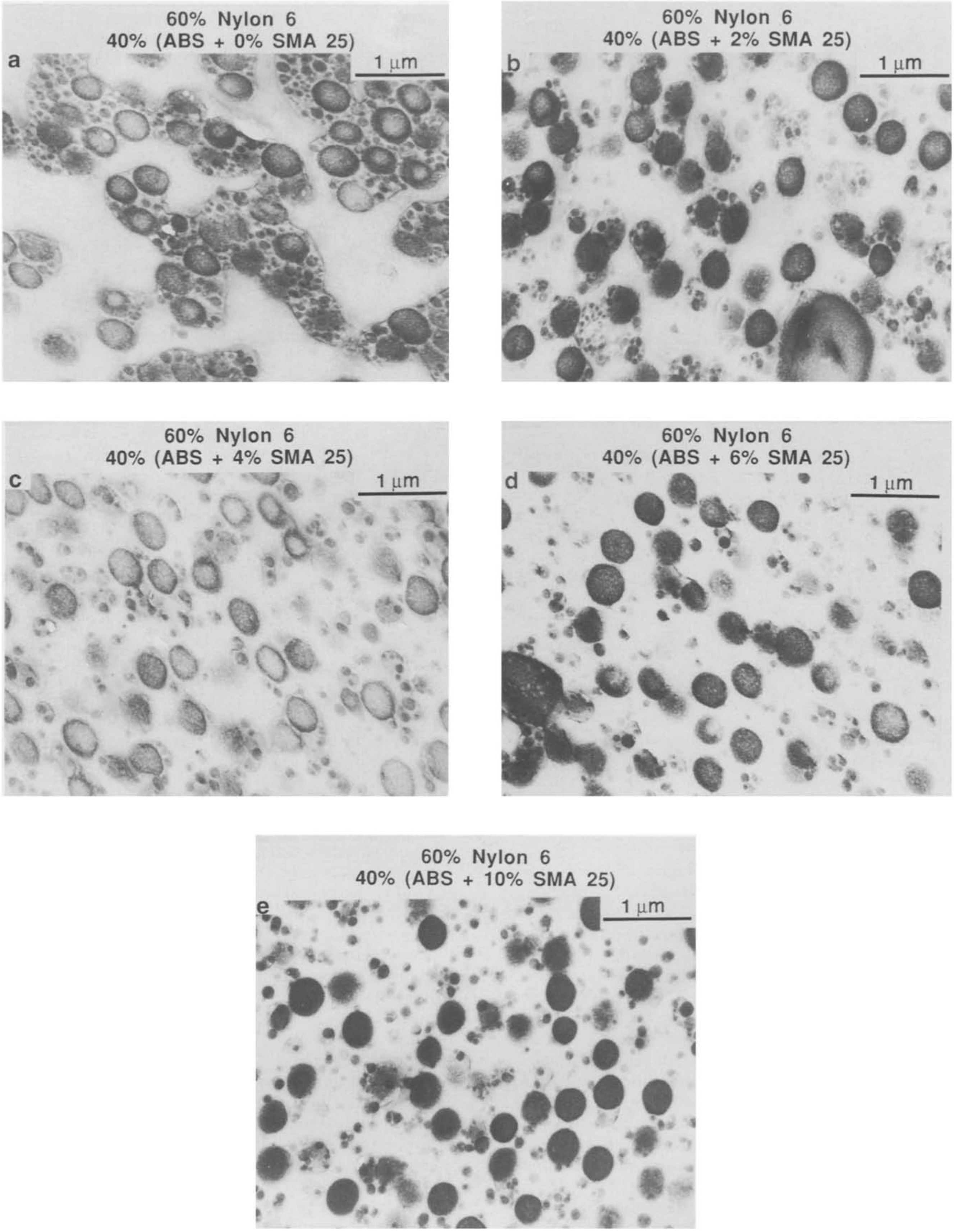

Figure 6 TEM photomicrographs for 60% nylon 6 blends with BL-65 containing (a) 0%, (b) 2%, (c) 4%, (d) 6% and (e) 10% SMA 25 copolymer in the ABS phase. The samples were dual stained with OsO_4 and RuO_4

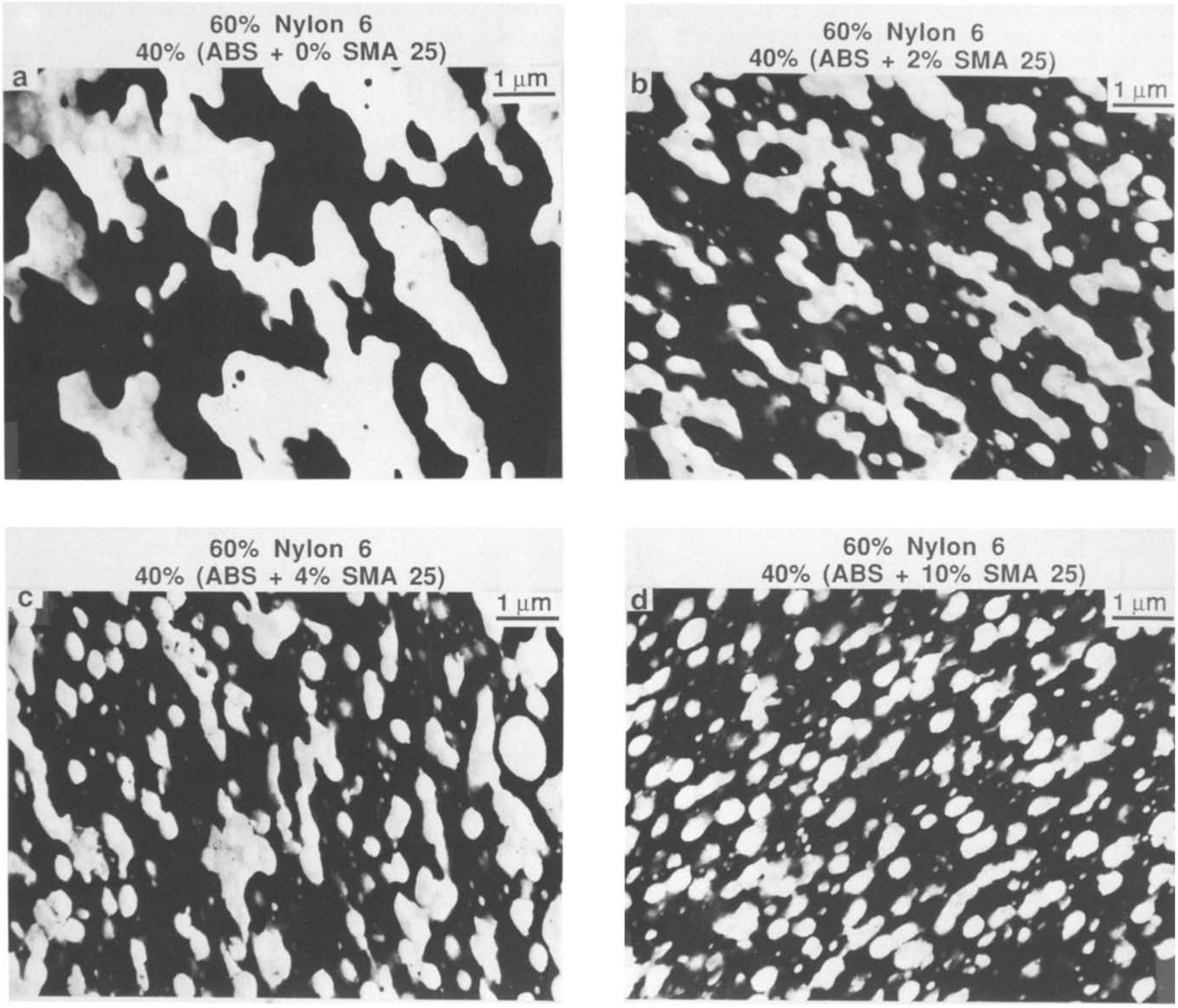

Figure 7 TEM photomicrographs for 60% nylon 6 blends with BL-65 containing (a) 0%, (b) 2%, (c) 4% and (d) 10% SMA 25 copolymer in the ABS phase. The polyamide phase was stained with phosphotungstic acid (PTA)

Figures 5a and *6a*). In all cases, careful examination of the unstained ABS phase reveals the outline of the butadiene rubber particles encapsulated by the SAN phase. The addition of 2% SMA 25 (based on ABS content) again causes a reduction in the size of the ABS domains (*Figure 7b*). Increasing the SMA 25 content to 4% further reduces the size of these ABS domains, although some relatively large ABS domains (2–4 μm) are still present (*Figure 7c*). Increasing the SMA 25 content to 10% further decreases the size of the dispersed domains, as shown in *Figure 7d*. From the series of TEM photomicrographs in *Figure 7*, it is clear that this staining technique provides a very effective method for defining the polymide/ABS interfacial boundary and is quite useful for revealing subtle changes in the phase morphologies of these blends. This staining technique allows us to calculate an average ABS domain size, except in the case of the uncompatibilized blend which has a co-continuous morphology (*Figure 7a*). *Figure 8* shows the steady decrease in the average ABS domain size (computed from the TEM photomicrographs in *Figures 7b–d*) as the concentration of SMA 25 is increased. While it is not possible to assign a value for the average domain size in the case of the uncompatibilized blend in *Figure 7a*, the domains appear to be at least 4–5 μm in width in most cases. Adding just 2% SMA 25 leads to a significant dispersion of the ABS domains by breaking down co-continuous clusters into domains which are mostly less than 1 μm wide. The average domain size is reduced by a factor of two as the SMA 25 content in the ABS is increased from 2% to 10%.

Using the above techniques it is not possible to determine whether there are rubber particles in all the unstained domains. This can be addressed by a dual staining technique involving sequential exposure of the sample to phosphotungstic acid (PTA) followed by osmium tetroxide. This should stain both the polyamide phase and the butadiene rubber particles in the ABS, leaving only the SAN plus SMA 25 phase unstained. The samples were floated in a 2% phosphotungstic acid solution for 30 min under ambient conditions and then exposed to vapours from a 2% OsO_4 solution for at least

8 h. The blend containing 10% SMA 25 is of particular interest since in spite of the increased dispersion observed here, this material has a dramatically lower Izod strength than the one containing 4% SMA 25 (*Figure 1*). *Figure 9* shows the TEM photomicrograph of the 10% SMA 25 blend stained in this manner. Both the polyamide and the spherical butadiene particles are indeed stained dark. The unstained (or white) regions in this blend should represent the miscible phase formed by SAN and SMA 25, which is clearly seen to encapsulate the butadiene domains. There is little evidence of there being SAN/SMA 25 domains without any butadiene rubber particles inside.

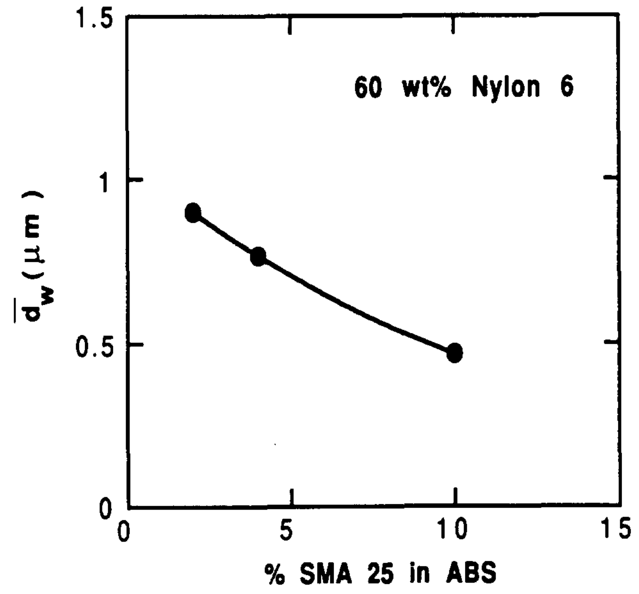

Figure 8 Effect of compatibilizer content on ABS domain size for blends containing 60% nylon 6. Values of $\bar{d}_w$ were computed on an equivalent area basis for the irregular ABS domains. A simple diameter at 0% SMMA 25 cannot be computed since the nylon 6 and ABS appear to be co-continuous

Nylon 6/ABS blends compatibilized with an imidized acrylic polymer

Figure 10 shows TEM photomicrographs of a nylon 6/ABS blend, stained by two different techniques, where the ABS phase was pre-mixed with 4% of the imidized acrylic polymer described by Triacca *et al.*[18]. This blend is brittle at room temperature, whereas the corresponding blend containing the SMA 25 copolymer approached supertoughness. Both staining techniques show that the ABS domains are significantly larger when the blend is compatibilized with the imidized acrylic polymer (see *Figure 10*) rather than the same amount of SMA 25 (see *Figures 5c*, *6c* and *7c*). This no doubt is related to the lower content of reactive functionalities of the imidized acrylic polymer compared to SMA 25 (see *Table 1*).

Commercial nylon/ABS blend

Figure 11 shows the morphology of a commercial nylon/ABS blend sold by Monsanto[10] under the trade

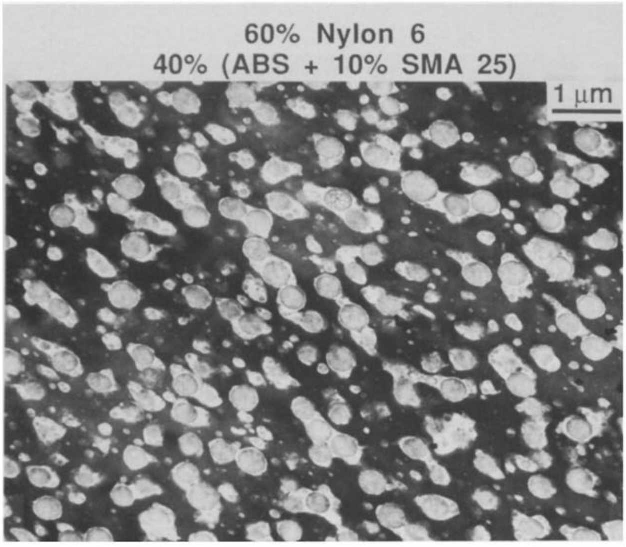

Figure 9 TEM photomicrograph for the 60/40 nylon 6/(BL-65/SMA 25 90/10) blend. The sample was dual stained with PTA and OsO_4

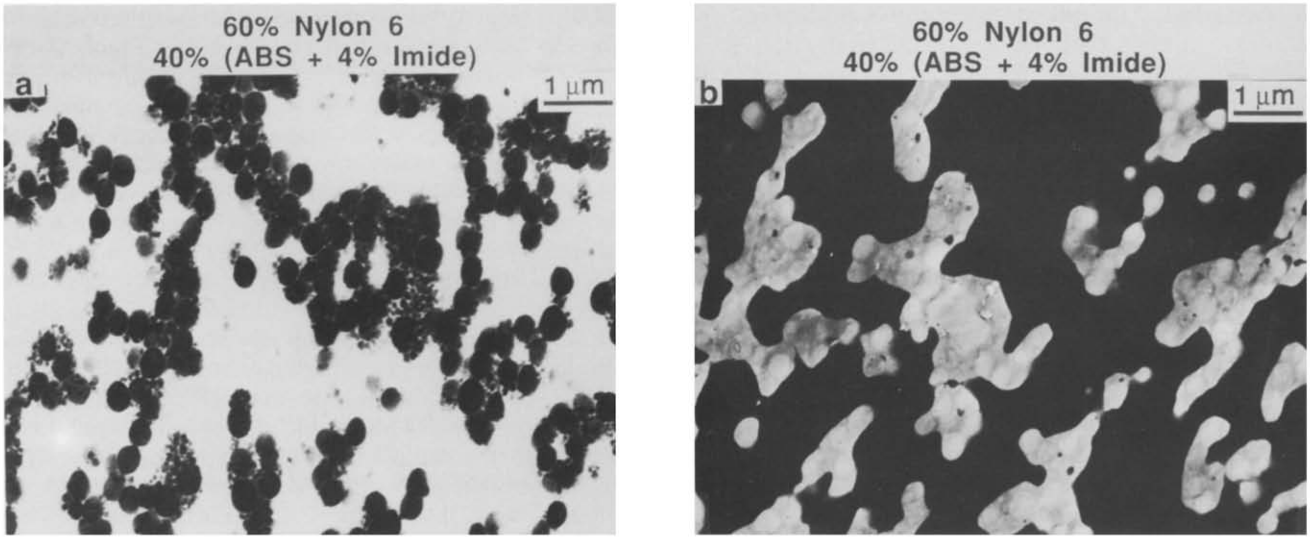

Figure 10 TEM photomicrographs for the 60/40 nylon 6/(BL-65/imide 96/4) blend stained with (a) OsO_4 and (b) PTA

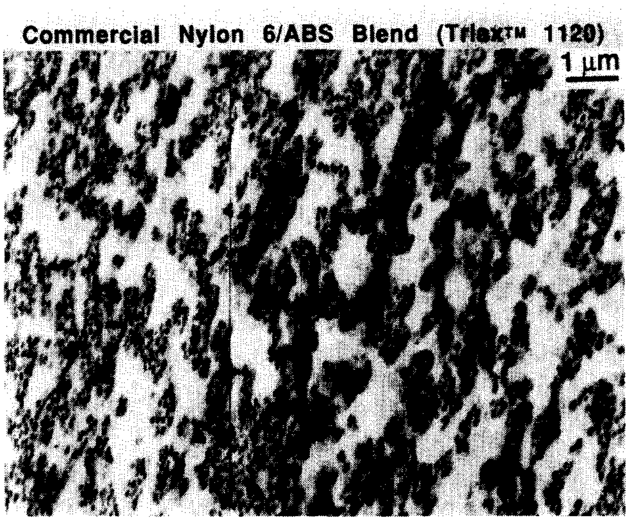

Figure 11 TEM photomicrograph for the commercial Triax™ 1120 material. The sample was dual stained with OsO_4 and RuO_4

name Triax™ 1120, which was stained by exposure to vapours from a 2% aqueous solution of OsO_4 for 8 h followed by exposure to vapours from a 0.5% RuO_4 solution for a period of 10 min under ambient conditions. It appears that the ABS material in this blend contains significantly smaller butadiene rubber particles that are more nearly uniform in size than is the case for the ABS material employed in this work. *Figure 11* also suggests that the ABS material and the polyamide essentially form co-continuous phases. In fact, the degree of clustering of the rubber particles within the SAN domains is comparable to that observed in the case of the uncompatibilized nylon 6/BL-65 blend (see *Figures 5a*, *6a* and *7a*). In the case of Triax™, however, the individual rubber particles ($\sim$0.14 μm) are much smaller than the main population of particles present in BL-65. The scale of the ABS domains in the uncompatibilized nylon 6/BL-65 blend is in the 2–4 μm range, whereas it is less than 1 μm in most cases for the Triax™ material. Interestingly, for the nylon 6/BL-65 blend compatibilized with 4% SMA 25 (leading to the maximum impact toughness in this series), the average size of the ABS domains is also slightly less than 1 μm but the nylon 6 and ABS phases are not co-continuous (see *Figure 7a*).

DISCUSSION

The results of Triacca *et al.*[18] and the results presented here show that nylon 6/ABS blends compatibilized with SMA 25 can approach the supertough range at an optimum SMA 25 content. However, at this optimum composition there is a wide distribution of impact values from sample to sample and within samples and poor low temperature toughness. It is quite possible that by varying the amounts of ABS and SMA 25 in the blend and by varying the processing conditions one could obtain wider composition windows for generating tough materials than is shown in *Figure 1*; however, it appears that the high functionality of the SMA 25 restricts the utility of this material as a compatibilizer. As a result of this conclusion, our future efforts will shift from the highly functional SMA 25 copolymer to polymers which have a lower functionality level. Specifically, we will demonstrate in forthcoming papers[32,33] that nylon 6/ABS blends with excellent toughness can be produced using the imidized acrylic polymer as the compatibilizer by optimizing the functionality characteristics and the concentration of such materials in the blend.

The main focus of the present work, however, was to characterize the morphologies of nylon 6/ABS blends containing varying amounts of SMA 25 as the compatibilizer. Several different staining techniques were utilized for this purpose. There is a distinct improvement in the dispersion of the ABS phase as the SMA 25 content is increased from 0% to 4%. The substantial increase in impact strength over this range no doubt results from a combination of the improved dispersion and the enhanced interfacial coupling at the nylon 6/SAN interface. Increasing the SMA 25 content beyond 4% leads to further improvements in the dispersion of the ABS domains (*Figure 8*) but results in an abrupt decline in the impact strength. Several factors may contribute to this sharp decrease in toughness[18]. Some of these issues are briefly examined in the following discussion.

As shown earlier, the dispersion of the ABS domains continues to improve progressively as SMA 25 is added to the blend (see *Figure 8*), even beyond the composition where the maximum impact strength is obtained. It is well known[34–37] that there is an upper limit on the size of rubber particles that will toughen nylon 6. More recently[38,39], it has been shown that there is also a lower limit ($\sim$0.2 μm) on the rubber particle size which causes toughening of nylon 6. By analogy, one might propose that the increase in toughness in nylon 6/ABS blends observed with the addition of small amounts of SMA 25 is the result of reducing the ABS domains below some upper limit, while further addition of SMA 25 drives the domain size below a lower limit. The average size of the ABS domains for the blend containing 10% SMA 25 (*Figure 8*) is well above this critical value[38,39]; however, it is not unreasonable to expect that the presence of the very brittle styrenic materials (SMA 25 and the SAN matrix of ABS) could lead to an upward shift of this lower critical limit. Our recent work[32] on nylon 6/ABS systems using other compatibilizers (also brittle like SMA 25) has shown, however, that supertough blends can be obtained even when the average ABS domain size is smaller ($\sim$0.36 μm) than that observed for the 10% SMA 25 blend ($\sim$0.47 μm).

There is a rubber particle size distribution within the ABS material used here. From the TEM photomicrographs for the blend in which the ABS contains 10% SMA 25, it is clear that even the smallest rubber particles ($d_w \approx 0.05$ μm) become separately dispersed (see *Figures 5d*, *6e*, *7d* and *9*). It could be argued that the ABS domains containing these very small rubber particles are not effective for toughening nylon 6 for the reasons outlined above. This proposal could be tested by using ABS materials with rubber particles that are more uniform in size.

It might also be argued that the dramatic drop in impact strength seen in *Figure 1* is related to the dilution of the rubber concentration in the blend as the concentration of the highly brittle SMA 25 copolymer is increased. This explanation is difficult to justify fully in the light of recent results where the rubber content was independently varied over a wide range and imidized acrylic polymers were used as the compatibilizers[32]. The blend based on 60% nylon 6 and 10% SMA 25 in the

ABS contains 18% rubber. This is above the critical limit for obtaining supertough nylon 6/ABS blends[32].

Since the SMA 25 molecule has a large number of reactive sites, an extremely high level of grafting at the nylon/SAN interface[17,18] is possible. An abundance of highly branched polymers at high SMA 25 concentrations could be envisioned as one cause for the reduction in blend toughness. On the other hand, nylon 6 can be effectively toughened by core/shell impact modifiers dispersed through the aid of the SMA copolymer containing MA levels of up to 25%[40]. In these systems, there was no evidence of a rapid decline in the impact strength of the blends, even when the SMA 25 content was raised significantly above the levels employed in the present work.

Recent studies on the morphology generation in model nylon 6/SAN systems compatibilized with highly functional polymers have shown that some of the compatibilizing polymer leaves the SAN phase and resides as micellar structures in the polyamide phase[41]. It is reasonable to envision that this phenomenon extends to the nylon 6/ABS system compatibilized with SMA 25. During processing, some of the anhydride groups in SMA 25 are converted to imide units through reaction with the nylon; ultimately this could change the molecular structure of the SMA 25 such that it would no longer be miscible with the SAN matrix of the ABS phase. As the modified SMA 25 molecules lose affinity for the SAN phase, the graft copolymers could form micelles in the polyamide phase. The existence of finely dispersed aggregates of brittle SMA 25 in the polyamide phase could be a cause for the loss in blend toughness. However, samples of nylon 6/ABS blends stained by both PTA and OsO_4 did not provide any concrete evidence for the existence of such micellar structures (see *Figure 9*).

From the above discussion, it is clear that no single factor can be identified yet as the dominant mechanism for the rapid decline in the impact strength of these blends as the concentration of the SMA 25 copolymer is raised beyond a certain level. It is possible that there is a significant degree of interplay among the various factors outlined above which ultimately leads to this dramatic loss in the ductility of these blends. However, our experience with different types of compatibilizers for nylon 6/ABS systems seems to indicate that the limitations of SMA 25 as a compatibilizer largely stem from its excessive degree of reactive functionality[32,41].

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