

# Reactive blending of poly(styrene-*co*-maleic anhydride) with poly(phenylene oxide) by addition of $\alpha$ -amino-polystyrene

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$\alpha$ -(3-Aminopropyl-1-amino)polystyrene ( $\alpha$ -amino-PS) was melt-blended with styrene/maleic anhydride copolymers (SMA) containing 28 wt% maleic anhydride groups. The terminal primary amino group can react with the maleic anhydride monomer units in SMA, forming imides. The resulting product turned out to be an efficient emulsifier for SMA blends with poly(phenylene oxide) (PPO). At constant weight fraction of added  $\alpha$ -amino-PS, the size of the dispersed PPO particles decreased with decreasing molar mass, i.e. increasing mole fraction, of the  $\alpha$ -amino-PS. In a second series of experiments,  $\alpha$ -amino-PS with  $\bar{M}_n > \bar{M}_{\text{entanglement}}$  was used to compatibilize SMA/PPO blends which also contained poly(acrylonitrile-*co*-butadiene-*co*-styrene) and some poly[styrene-*b*-(ethylene-*co*-butylene)-*b*-styrene] triblock copolymer. Although, in this case, it was not possible to detect major changes in the phase texture or morphology, it could be shown that the addition of  $\alpha$ -amino-PS improved the impact strength significantly. For comparison, blends were also prepared with unmodified PS of a similar  $\bar{M}_n$ . No corresponding improvement of the mechanical properties was observed in this case.

(Keywords: reactive blending; poly(phenylene oxide); amino-polystyrene)

## INTRODUCTION

Blending of different polymers is an important way to prepare materials with optimized properties. Since the entropy of mixing approaches zero for high molecular weight polymers, thermodynamic miscibility is an exception rather than the rule for synthetic macromolecules, which are mostly formed from building blocks whose interaction is not particularly favourable. An example exhibiting miscibility in the whole composition range is polystyrene (PS) and poly(oxy-2,6-dimethyl-1,4-phenylene) (PPO)<sup>1-3</sup>. The demixing temperature for a 1:1 (by weight) mixture of high molecular weight components is reported<sup>1-3</sup> to be in the range of 340–360°C. This fact has been commercially exploited and blends of PPO and PS are widely used. The addition of PS to PPO results in a more easily processable material but also inevitably in a large drop of the glass transition temperature ( $T_g$ ), which is often unfavourable. The  $T_g$  of a miscible blend is approximately described by the Fox equation:

$$\frac{1}{T_{g,\text{blend}}} = \frac{w(\text{PPO})}{T_{g,\text{PPO}} (= 217^\circ\text{C})} + \frac{w(\text{PS})}{T_{g,\text{PS}} (= 100^\circ\text{C})} \quad (1)$$

where  $w(X)$  = weight fraction of component  $X$ .

To limit the drop in  $T_g$ , which accompanies the increase in processability, PS must be substituted

by another material with a higher  $T_g$ , which is still easy to process. An example of such a material is a styrene/maleic anhydride random copolymer (SMA). For maleic anhydride (MA) contents of 8.5 wt% and lower, SMA is, like PS, homogeneously miscible with PPO<sup>4</sup>. The gain in  $T_g$  with respect to PS, however, is small in the case of such low contents of MA. It would be more interesting to blend PPO with an SMA containing 20–30 wt% MA. For example SMA containing 28 wt% MA has a  $T_g$  of 165°C and is easily processable. However, such an SMA is no longer miscible with PPO (see, for example, this work and ref. 4). Blending will result in a two-phase material in which the high  $T_g$  of the PPO remains unaffected. A compatibilizer which reduces the particle size of the dispersed phase by lowering the interfacial tension and which enhances the adhesion between both blend components by entanglement formation can be expected to help in preparing a blend with useful mechanical properties<sup>5-7</sup>.

In this work, PS samples of different molecular weights with primary amino end-groups are applied as 'reactive compatibilizers' for blends of PPO and SMA containing 28 wt% MA. During blending in the melt, the amine groups can react with the MA groups of SMA. The resulting SMA-*g*-PS graft copolymers are expected to be effective compatibilizers for the blends because of the miscibility of the PS grafts with PPO. The effectiveness of the compatibilizers is tested by studying both blend morphology and some mechanical properties.

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## EXPERIMENTAL

## Materials

SMA containing 28 wt% MA ( $\bar{M}_w = 110\,000\text{ g mol}^{-1}$ ), poly(acrylonitrile-co-butadiene-co-styrene) (ABS) of which the SAN phase contained 27 wt% acrylonitrile (Ronfalin TZ 220), and PPO with an intrinsic viscosity  $[\eta]^{25^\circ\text{C}} = 0.48\text{ dl g}^{-1}$  (in  $\text{CHCl}_3$ ) were provided by DSM. Polystyrene (PS-30) was prepared by anionic polymerization under high vacuum conditions ( $\bar{M}_n = 29\,600\text{ g mol}^{-1}$ ,  $\bar{M}_w = 31\,500\text{ g mol}^{-1}$ ).  $\alpha$ -(3-Aminopropyl-1-amino)polystyrene ( $\alpha$ -amino-PS) was prepared by anionic polymerization under high vacuum conditions. The polymer anion was deactivated with 1,5-diazabicyclo(3,1,0)hexane and treated with acetic acid to generate the primary amino end-group as described elsewhere<sup>7,8</sup>. The poly[styrene-*b*-(ethylene-co-butylene)-*b*-styrene] (SEBS; Kraton 1650<sup>TM</sup>, Shell) had a styrene content of 29 wt%.

SMA/ $\alpha$ -amino-PS/PPO blends

SMA/PPO blends, with the amino-terminated PS as a reactive compatibilizer, were prepared in two steps. First the  $\alpha$ -amino-PS was reacted with SMA. The resulting graft copolymer was then added to PPO. SMA (1.52 g, 80 wt%) and  $\alpha$ -amino-PS (0.38 g, 20 wt%) were put into the cylinder shaft of a home-built miniature single screw extruder (DSM) and mixed at  $400\text{ rev min}^{-1}$  for 4 min. The temperature of both the cylinder shaft and the die was set at  $220^\circ\text{C}$  and the screw had also been preheated to  $220^\circ\text{C}$ . Mixing was carried out under nitrogen in order to prevent degradation of the polymers. A sample (1.26 g, 70 wt%) of the graft copolymer thus prepared and 0.54 g (30 wt%) of PPO were mixed at  $400\text{ rev min}^{-1}$  for 4 min in the same small extruder. The temperature of both the cylinder shaft and the die was set at  $280^\circ\text{C}$  and the screw had also been preheated to  $280^\circ\text{C}$ . Mixing was carried out under nitrogen to avoid degradation of the polymers. After mixing, the blend was extruded into small glass vials and quenched in liquid nitrogen.

ABS/SMA/ $\alpha$ -amino-PS/PPO/SEBS blends

$\alpha$ -Amino-PS or PS and PPO were dissolved in 150 ml  $\text{CHCl}_3$ . A PPO/PS-( $\text{NH}_2$ ) blend was isolated by precipitation in 1.5 l methanol. In the second step, the PPO/ $\alpha$ -amino-PS mixture, SMA and SEBS were dispersed in a Brabender 50 ml plasticorder at  $50\text{ rev min}^{-1}$  for 3 min at  $220^\circ\text{C}$ . Then ABS was added to the premixture and mixed at  $50\text{ rev min}^{-1}$  for another 5 min at  $220^\circ\text{C}$ . The composition of the different blends is given in Table 1.

## Gel permeation chromatography

G.p.c. experiments were performed in tetrahydrofuran (THF) using  $\mu$ -Styragel<sup>TM</sup> columns with 500,  $10^3$ ,  $10^4$

and  $10^5\text{ \AA}$  particle size at room temperature. The concentration of the polymer solution was  $10\text{ mg ml}^{-1}$ . The detection was performed with a differential refractometer (Waters 410) and a low angle laser light scattering (LALLS) detector (Chromatix KMX-6).

The blends were pressed into plates on a Fontaine press at  $220^\circ\text{C}$ . Standard Izod impact test-bars were cut from the compression-moulded plates, and notched Izod impact tests were carried out according to ISO 180.

## Vicat temperature

The Vicat temperature was determined according to ISO 306, using method B. D.s.c. experiments were performed with a Perkin-Elmer DSC 7 at a heating rate of  $20^\circ\text{C min}^{-1}$ . The instrument was calibrated with high purity gallium and indium standards.

## Transmission electron microscopy

Transmission electron microscopy (TEM) of the SMA/ $\alpha$ -amino-PS/PPO blends was carried out with a Zeiss EM 902 80 kV microscope equipped with a Kontron image processor. Bulk samples were trimmed for microtoming and stained by placing them for 10 h into a freshly prepared aqueous solution of 10 wt% NaOCl and 0.2 g  $\text{RuCl}_3$ . Ultrathin sections were cut by means of a Reichert-Jung microtome Ultracut-E. Transmission electron micrographs of the ABS/SMA/ $\alpha$ -amino-PS/PPO/SEBS blends were obtained with a Philips TEM 420. After staining with  $\text{OsO}_4$ , thin sections were microtomed at  $-100^\circ\text{C}$  with a Reichert-Jung microtome Ultracut-E, supplied with a cryo-unit FC4E. Thin sections of blend 1 and blend 2 were additionally treated with  $\text{RuO}_4$  vapour to visualize the Kraton.

## RESULTS AND DISCUSSION

Since SMA containing 28 wt% of MA and PPO are not miscible, a compatibilizer is needed, whose constituent segments or blocks are compatible with the two polymers and which is therefore absorbed in the interphase. Due to the expected decrease in the interfacial tension, the dispersion and thus also the properties of the blends should be improved.

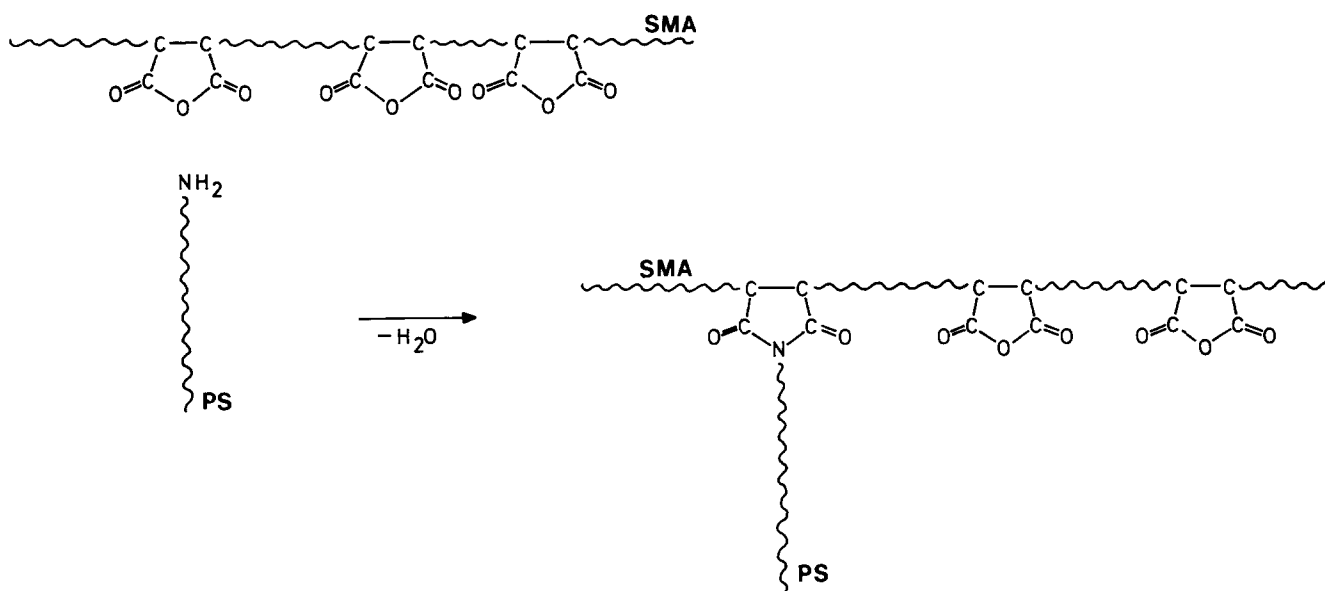
While SMA is at least partly miscible with a number of styrene/acrylonitrile copolymers (SAN)<sup>9</sup>, PS homopolymers are fully miscible with PPO<sup>1-3</sup>. MA units in SMA are reactive moieties on which, for example, amino-terminated macromolecules can be grafted<sup>10</sup>. Reaction of PS molecules carrying primary amino end-groups with SMA yields graft copolymers which are potentially useful as compatibilizers for blends of styrene-containing polymers (SMA and/or the SAN part of ABS) and PPO (Scheme 1).

Such an approach has the advantage that the compatibilizer can be generated *in situ* when the two prepolymers are reacted during the actual blending process<sup>1</sup>. Efficient preparation of PS with primary amino end-groups has been described recently<sup>7</sup>. As shown in Scheme 2, PS of defined molecular weight can be synthesized in high yield (>75%) by adding 1,5-diazabicyclo[3,1,0]hexane (1) to polystyryllithium. Acidolysis with acetic acid converts the resulting *N*-polystyryl-1,3-diazacyclohexane (2) to  $\omega$ -polystyryl-3-amino-1-propylamine (3).

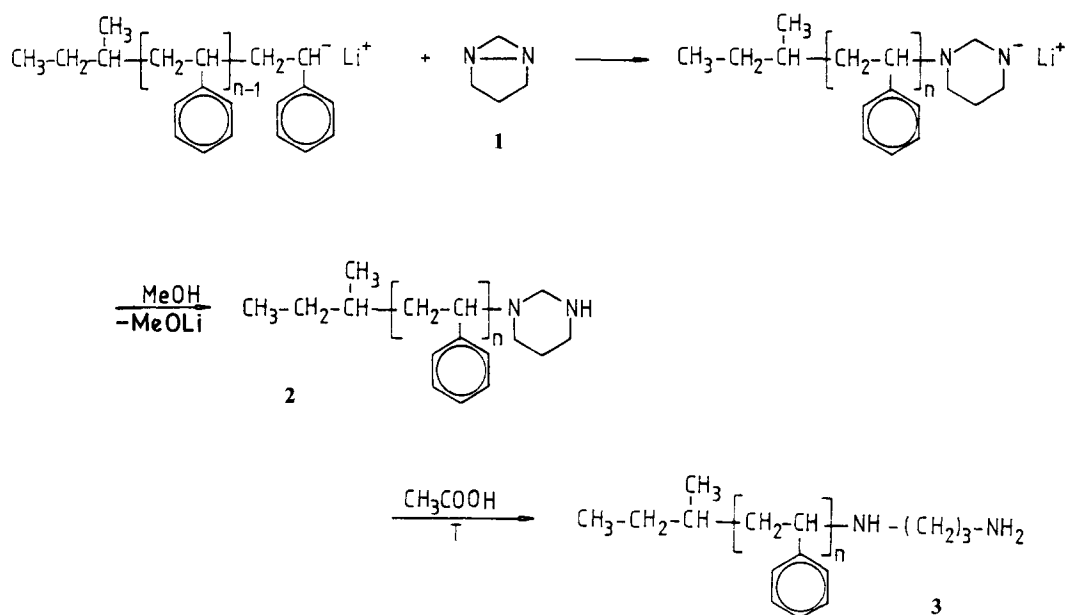
Amino-terminated PS samples of different molecular weight were mixed with SMA (28 wt% MA) and PPO.

**Table 1** ABS/SMA/ $\alpha$ -amino-PS/PPO/SEBS blends (composition in wt%)

Compound	Blend 1	Blend 2	Blend 3	Blend 4
SMA	25.2	25.2	35.0	35.0
ABS TZ 220	25.2	25.2	30.0	30.0
PPO	25.6	25.6	17.5	17.5
Kraton G 1650	10.0	10.0	7.5	7.5
PS-30	14.0	—	10.0	—
PS-DAP-40.I	—	14.0	—	10.0



Scheme 1



Scheme 2

SMA and PPO itself are immiscible and form a simple model blend which allows investigation of the influence of the chain length as well as the number of side-chains of the PS-*g*-SMA graft copolymer upon dispersion of PPO in SMA. A reference blend was prepared using unmodified PS (PS-30). A list of  $\alpha$ -amino-PS samples is given in Table 2, giving the respective molecular weights and degrees of functionalization. The degree of functionalization gives the number fraction of PS molecules which actually carry a terminal 3-aminopropyl-1-amine group; it was determined by high performance liquid chromatography over silica columns using a chloroform/ethanol (99.5:0.5) mixture as eluent, which allowed a baseline separation as long as molecular weights did not exceed  $\bar{M}_n = 40\,000$  (ref. 8).

In a first series of experiments, the generated SMA-*g*-PS copolymers were mixed with PPO. In all cases, the final blend consisted of 56 wt% SMA, 14 wt% amino end-functionalized or non-functionalized PS and 30 wt%

Table 2 Polystyrenes used in blending experiments

Sample	$\bar{M}_n$ (g mol <sup>-1</sup> )	$\bar{M}_w$ (g mol <sup>-1</sup> )	Degree of functionalization (%)
PS-30	29 600	31 500	0
PS-DAP-20.II	16 300	17 300	74
PS-DAP-40.I	30 200	36 900	80
PS-DAP-80.I	57 800	70 600	78
PS-DAP-150.I	280 000	330 000	70

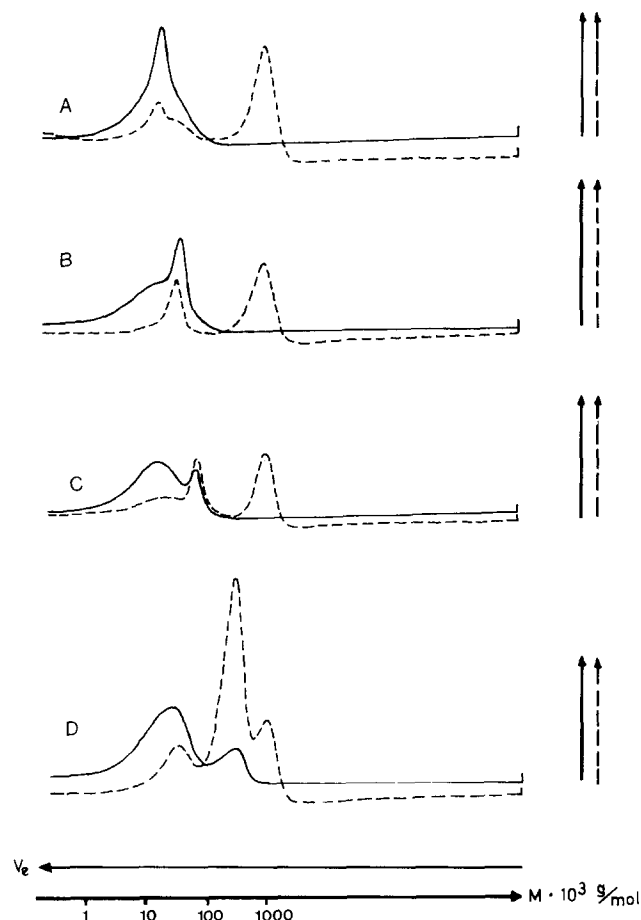
PPO. The blends were prepared in a two-step procedure. In the first step, 80 wt% SMA and 20 wt% of the respective PS were blended in a miniature single screw extruder. In the second step, 70 wt% of the prepared graft copolymer was blended with 30 wt% PPO in the same small extruder.

FTi.r. measurements were made in order to check whether the blending conditions were suitable for the

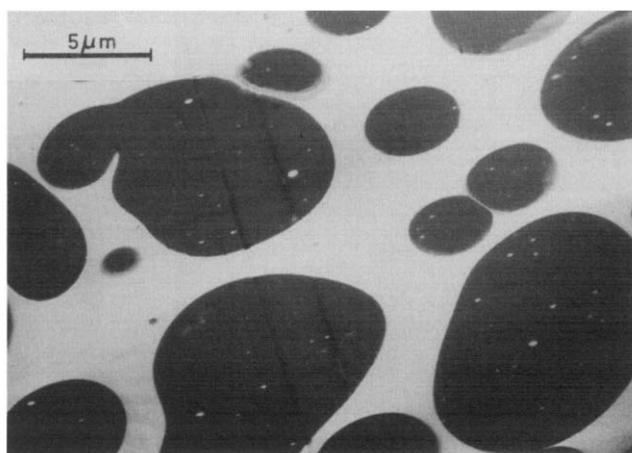
desired imide formation. Because the concentration of the imide grafts in the blends was too low for their determination by i.r., a model experiment was made. PS-DAP-20.II and 10 mol equivalent phthalic anhydride were mixed in a minimum amount of THF. After evaporation of the THF, the homogeneous mixture was heated for 5 min at 220°C. FTi.r. measurements were performed on the reaction product. An intense band at 1715  $\text{cm}^{-1}$  was found in the FTi.r. spectrum, which can be assigned to the imide group. This signal was not observed in the i.r. spectra of the educts, phthalic anhydride and PS-DAP-20.II.

The degree of grafting was investigated by g.p.c./LALLS experiments using THF as a solvent. PPO was separated from the blend by extraction with THF, which is a good solvent for SMA and PS but not for PPO. SMA/ $\alpha$ -amino-PS/PPO (0.5 g) was extracted with 5 ml THF for several hours and filtered. The solvent of the filtrate was evaporated and the residue dissolved in a small volume of THF. After precipitation in methanol, SMA/ $\alpha$ -amino-PS was dried overnight under vacuum at 50°C.

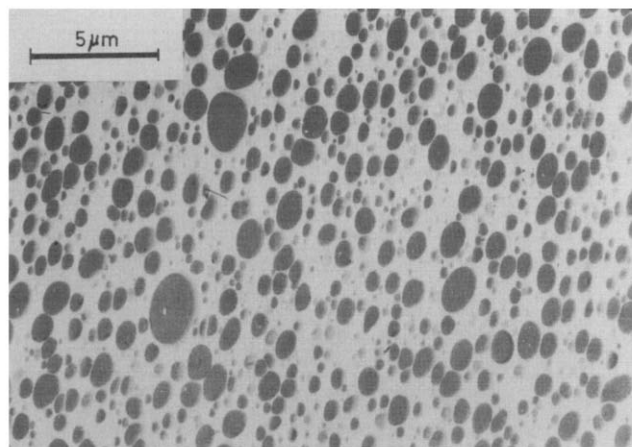
The g.p.c./LALLS diagrams of the different SMA/ $\alpha$ -amino-PS are given in Figure 1. The solid line shows the r.i. signal and the dashed line the LALLS signal. In all diagrams a high molecular weight LALLS signal can be detected at low elution volumes. This signal is more intense in the blends which were made with low molecular weight  $\alpha$ -amino-PS. The other signals in the LALLS curves can be assigned to unreacted SMA (broad) and



**Figure 1** G.p.c./LALLS diagrams of SMA/ $\alpha$ -amino-PS blends; solid line indicates the r.i. signal, dashed line the LALLS signal. A, SMA/PS-DAP-20.II; B, SMA/PS-DAP-40.I; C, SMA/PS-DAP-80.I; D, SMA/PS-DAP-150.I



**Figure 2** TEM photograph of SMA/PS-30/PPO, 56/14/30 (wt%)

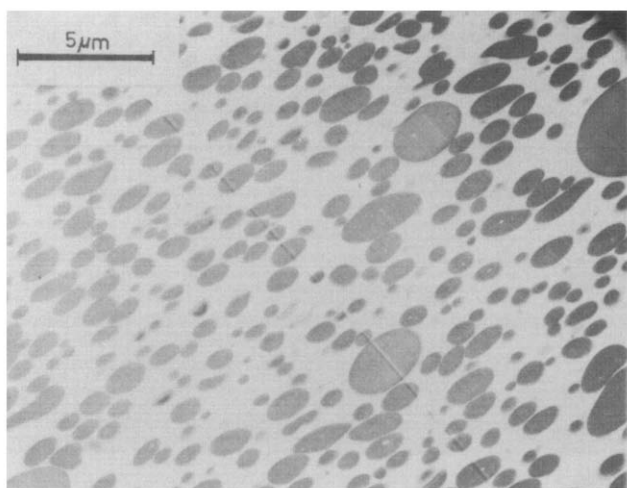


**Figure 3** TEM photograph of SMA/PS-DAP-20.II/PPO, 56/14/30 (wt%)

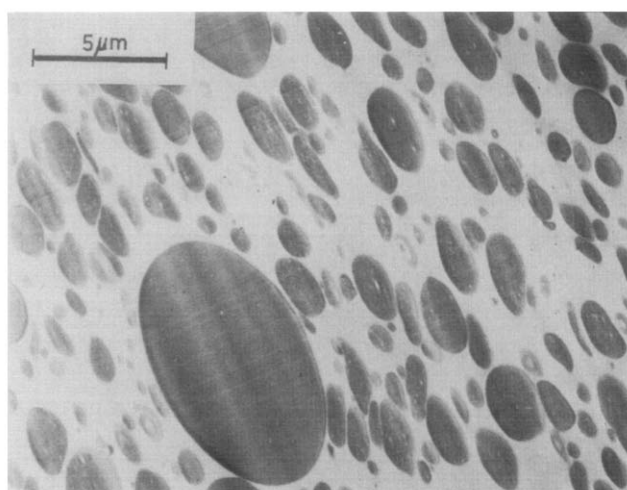
$\alpha$ -amino-PS (sharp and shifted towards lower elution volumes with increasing molecular weight of the  $\alpha$ -amino-PS). The high molecular weight fraction can be assigned to graft copolymers (SMA-*g*-PS). In all cases, the amount of the high molecular weight fraction is very low, so that this compound can only be detected by LALLS which is especially sensitive for high molecular weight compounds. In some cases, an even smaller high molecular weight fraction was also observed when only non-functionalized PS was added in the blending step in place of  $\alpha$ -amino-PS. Thus, it cannot be excluded that some grafting of PPO chains upon SMA can occur via the phenolic end-groups of PPO.

The blend morphology was investigated by TEM. It can be seen in Figures 2–5 that blends prepared with PS-30, PS-DAP-20.II, PS-DAP-40.I and PS-DAP-80.I, consist of an SMA matrix (light coloured phase in Figures 2–5) in which PPO domains (dark spheres in Figures 2–5) are dispersed.

Clearly, a significant decrease of the particle size of the PPO domains was observed with decreasing molecular weight of the amino end-functionalized PS. For the blend prepared with non-functionalized PS — where imide formation could not occur — an approximately five-fold domain diameter was determined as compared to the blend prepared with amino end-functionalized PS of comparable molecular weight. Thus,  $\alpha$ -amino-PS serves as an efficient reactive compatibilizer for SMA/PPO blends.



**Figure 4** TEM photograph of SMA/PS-DAP-40.I/PPO, 56/14/30 (wt%)



**Figure 5** TEM photograph of SMA/PS-DAP-80.I/PPO, 56/14/30 (wt%)

It should be noted that even the blend without  $\alpha$ -amino-PS is not very coarse, indicating that the interfacial tension between the SMA and PPO is relatively low. This is not surprising, in view of the miscibility of PPO and SMA containing up to 8.5 wt% MA. Another explanation might be that a small amount of SMA-*g*-PPO polymers has also been formed.

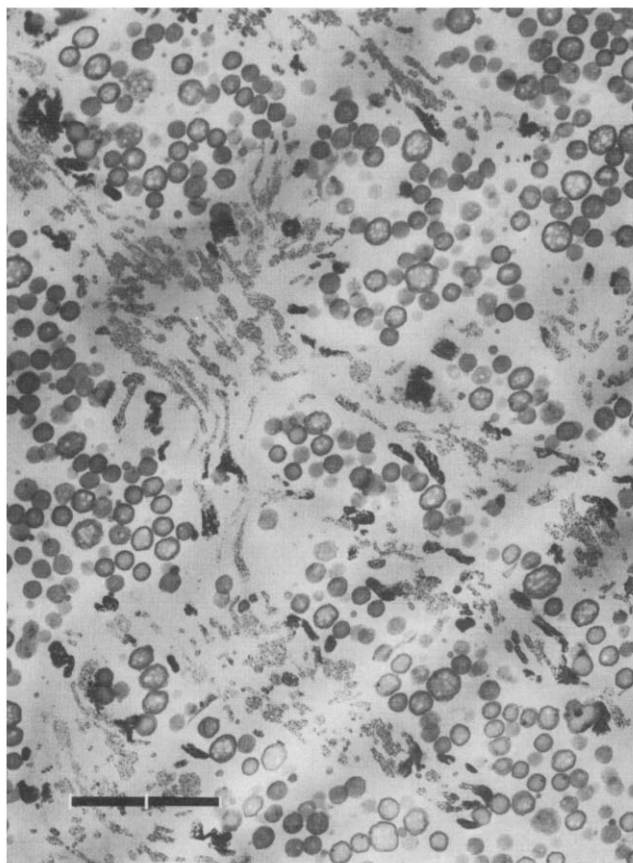
In a second attempt,  $\alpha$ -amino-PS graft copolymers were tested for their ability to modify the impact strength of SMA/PPO blends. In this case,  $\alpha$ -amino-PS was reacted with SMA during blending of SMA and PPO, which should result in *in situ* formation of SMA-*g*-PS. SEBS and ABS were used as additional additives to improve the toughness of both phases. Since SAN is miscible with SMA of comparable styrene content, ABS is expected to toughen the SMA phase, whereas SEBS is compatible with PPO (PS blocks are miscible with PPO). Blends with two different compositions were prepared. For each composition two blends were made, the first with PS-DAP-40.I as compatibilizing agent, the other with unmodified PS-30 as a reference blend (Table 1). PS-DAP-40.I was chosen because its use in the SMA/PPO blends led to a finely dispersed texture, as shown in Figure 4, and because its molecular weight of  $30\,000\text{ g mol}^{-1}$  is higher than the entanglement molecular weight of PS

( $\bar{M}_e = 18\,700\text{ g mol}^{-1}$ ) and thus considerably higher than  $M_e$  of a PS/PPO/SEBS mixture ( $\bar{M}_{e,\text{PPO}} = 3600$ )<sup>11</sup>.

In contrast to the experiments with SMA and PPO only, no significant difference in the morphology could be observed in the case of the impact-modified blends when PS-30 was replaced by the aminated PS-DAP-40.I (Figures 6–9). The TEM images of blends 1 and 2 (Figures 6 and 7) are very similar. The morphology appears to be co-continuous. The SMA/SAN phase can be assigned to the lighter areas in the micrograph, denoted by B in Figure 7, in which dark spherical inclusions, the polybutadiene particles of ABS, are incorporated. PPO forms the basis of a second, mostly co-continuous phase which is slightly darker in the micrographs, denoted by A in Figure 7. The size of the domains is roughly 1–3  $\mu\text{m}$  in diameter. The TEM images of blends 3 and 4 are also very similar. Comparison of Figures 6 and 7 with Figures 8 and 9 shows that the co-continuous PPO phase of blends 1 and 2 became dispersed when the weight fraction of SMA+ABS was increased (see blends 3 and 4 in Figures 8 and 9).

Lamellar structures, which are embedded within the darker PPO phase, can be observed in the TEM pictures of blends 1 and 2 (Figures 6 and 7). In this case the thin sections were treated with  $\text{RuO}_4$ . Due to their thickness and location, these lamellar structures can be assigned to the SEBS triblock copolymer. Because the corresponding thin sections were not stained with  $\text{RuO}_4$ , the block copolymer lamellae are not visible in the TEM pictures of blends 3 and 4 (Figures 8 and 9).

The interpretation of the phase morphology given above is supported by d.s.c. experiments on the glass



**Figure 6** TEM photograph of ABS/SMA/PS-30/PPO/SEBS, 25.2/14/25.6/10 (wt%); blend 1. Scale bar 2  $\mu\text{m}$

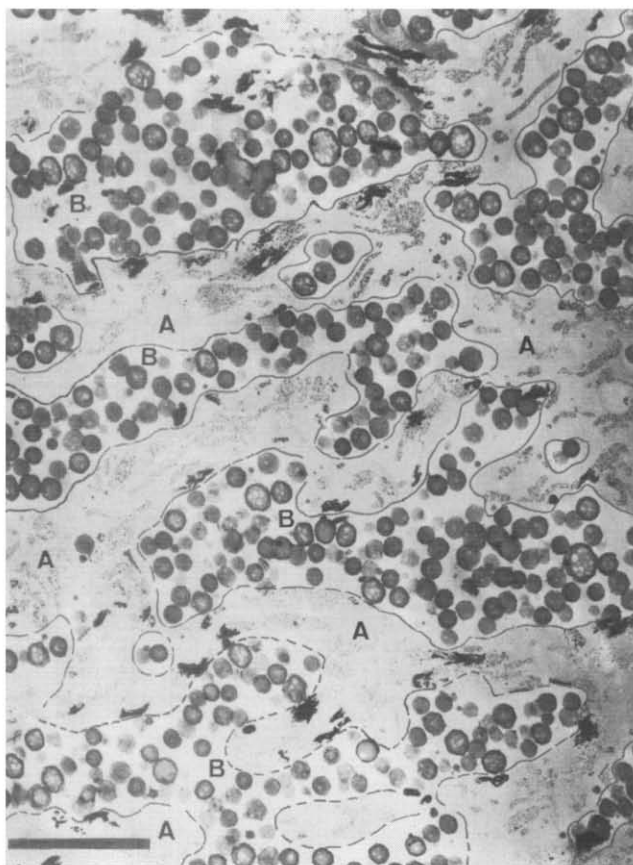


Figure 7 TEM photograph of ABS/SMA/PS-DAP-40.I/PPO/SEBS, 25.2/25.2/14/25.6/10 (wt%); blend 2. Scale bar 2  $\mu\text{m}$

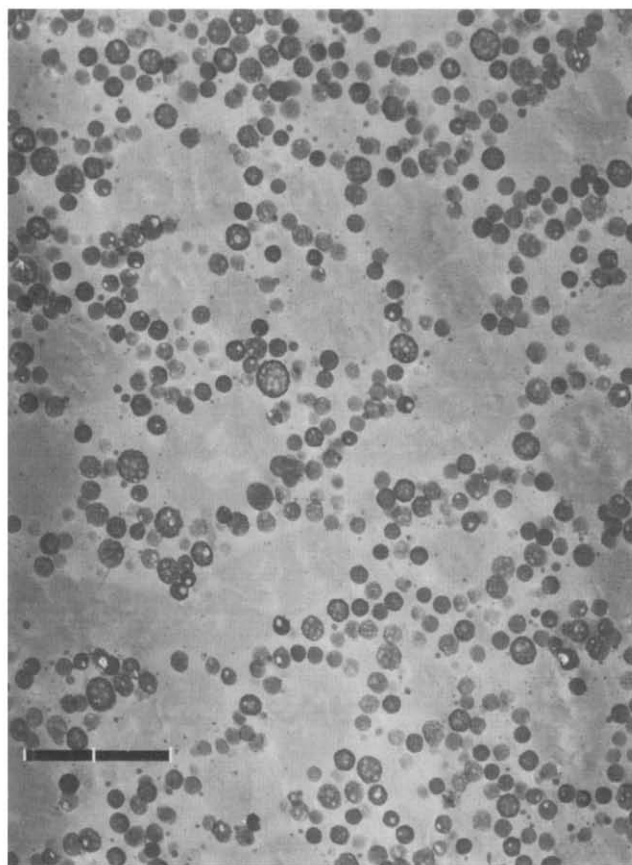


Figure 9 TEM photograph of ABS/SMA/PS-DAP-40.I/PPO/SEBS, 30/35/10/17.5/7.5 (wt%); blend 4. Scale bar 2  $\mu\text{m}$

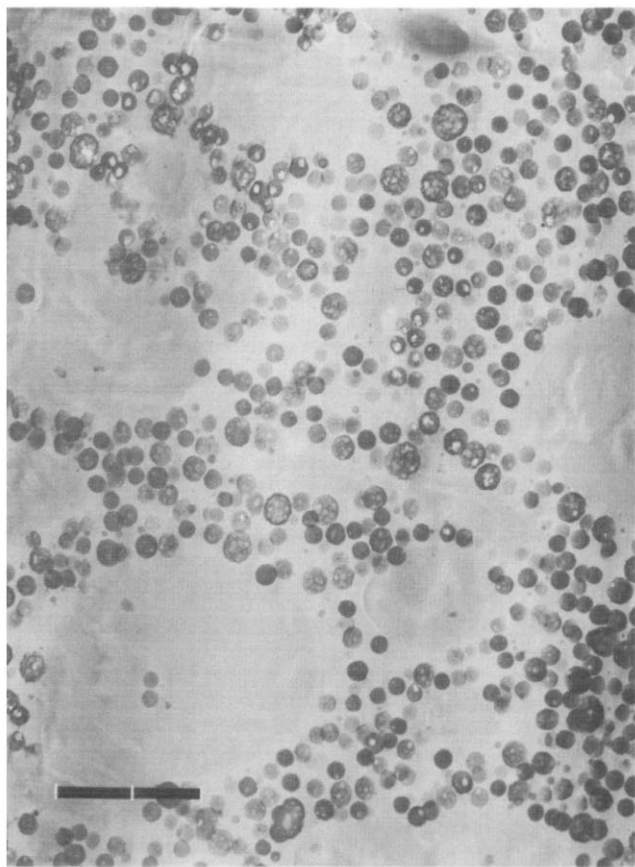


Figure 8 TEM photograph of ABS/SMA/PS-30/PPO/SEBS, 30/35/10/17.5/7.5 (wt%); blend 3. Scale bar 2  $\mu\text{m}$

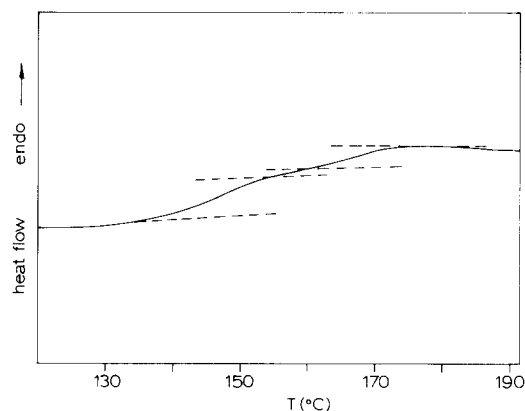
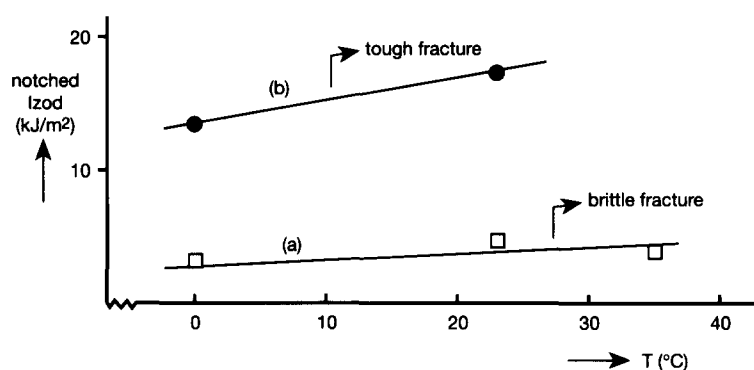


Figure 10 D.s.c. trace of ABS/SMA/PS-DAP-40.I/PPO/SEBS, 25.2/25.2/14/25.6/10 (wt%); blend 2

transitions of the blends. *Figure 10* shows a d.s.c. diagram of blend 2. Two  $T_g$ s are observed at 147 and 167°C, which can be assigned to the SMA/SAN phase and the PPO/PS phase, respectively. The composition of the SMA/SAN domains is 62.5/37.5 according to the fractions of the components which were added to form the blend. Correspondingly, the  $T_g$  is calculated with equation (1) to be 140°C ( $T_{g,SMA} = 165^\circ\text{C}$ ,  $T_{g,SAN} = 105^\circ\text{C}$ ). Assuming that PPO ( $T_{g,PPO} = 217^\circ\text{C}$ ) is mixed with the  $\alpha$ -amino-PS and the PS fraction of the SEBS (29%), the PPO/PS composition is 60.5/39.5 and the  $T_g$  is calculated to be 163°C. Thus, experimental  $T_g$ s are in fairly good agreement with the values calculated for the expected composition of the different phases.





**Figure 11** Notched Izod impact as a function of temperature for blend 3 (□), ABS/SMA/PS-30/PPO/SEBS, 30/35/10/17.5/7.5 (wt%); and blend 4 (●), ABS/SMA/PS-DAP-40.I/PPO/SEBS, 30/35/10/17.5/7.5 (wt%)

**Table 3** Properties of ABS/SMA/ $\alpha$ -amino-PS/PPO/SEBS blends

	Vicat temperature (°C)	Notched Izod impact (kJ m <sup>-2</sup> )		
		0°C	23°C	35°C
Blend 1	136		8.6 ± 1.2	
Blend 2	136		20.1 ± 2.4	
Blend 3	134	3.9 ± 0.4	4.7 ± 1.0	4.0 ± 0.5
Blend 4	134	13.4 ± 0.3	17.3 ± 0.3	

Although the morphology of the blends did not indicate particular improvement in the phase dispersion, the impact properties of the blends were improved significantly when the aminated PS was added. *Table 3* lists the notched Izod impact values together with the Vicat B temperatures for the four blends. Compared to blends 1 and 3, which were made with the unmodified PS-30, a significant improvement of the impact strength was observed for the blends when amino end-functionalized PS was added (blends 2 and 4). The Izod impact strength of blend 2 was twice that of blend 1, while the Izod impact strength of blend 4 was more than three times that of blend 3. *Figure 11* gives the notched Izod values at different temperatures for blends 3 and 4. When SMA was partly grafted by  $\alpha$ -amino-PS during blending, the material remained tough down to temperatures where the corresponding blend with non-functionalized PS became brittle. The tough-brittle transition is clearly shifted to lower temperatures.

## CONCLUSIONS

Addition of well-defined PS with primary amino end-groups has been demonstrated to influence the dispersion of SMA/PPO blends. A significant decrease of the PPO domain particle size in the SMA matrix was observed.

The observed compatibilization can be explained by the reaction of  $\alpha$ -amino-PS with MA monomer units in SMA. Imide formation results in formation of a grafted copolymer. The improvement in dispersion depends on the degree of grafting of SMA.

Although only a minor effect on the morphology could be observed when  $\alpha$ -amino-PS was added to blends of (SMA/SAN/ABS)/(PPO/SEBS), grafting of SMA by PS

had a significant influence on the Izod impact strength of the blends. When PS-DAP-40.I was used as a compatibilizing agent, the Izod impact strength was strongly improved with respect to blends of identical polymer composition. It can be concluded that the morphology and the impact strength are not directly correlated in this case. The generated amount of graft copolymer at the interface is probably in agreement with the g.p.c.-LALLS results: low (minor effect on the interfacial tension), but still sufficient to improve the interfacial adhesion and consequently the impact toughness. This idea is supported by results in the literature<sup>12</sup>, where it was found that only a few copolymer chains per surface area are required at the interface to obtain considerable interfacial strength.

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