

Effect of *In Situ* Synthesized Macroactivator on Morphology of PA6/PS Blends via Successive Polymerization

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ABSTRACT: *In situ* polymerization and *in situ* compatibilization was adopted for preparation of ternary PA6/PS-g-PA6/PS blends by means of successive polymerization of styrene, with TMI and ϵ -caprolactam, via free radical copolymerization and anionic ring-opening polymerization, respectively. Copolymer poly(St-g-TMI), the chain of which bears isocyanate ($-\text{NCO}$), acts as a macroactivator to initiate PA6 chain growth from the PS chain and graft copolymer of PS-g-PA6 and pure PA6 form, simultaneously. The effect of the macroactivator poly(St-g-TMI) on the phase morphology was investigated in detail, using scanning electron microscopy. In case of blends with higher content of PS-g-PA6 copolymer, copolymer nanoparticles coexisting with the PS formed the matrix, in

which PA6 microspheres were dispersed evenly as minor phase. The content of the compositions (homopolystyrene, homopolyamide 6, and PS-g-PA6) of the blends were determined by selective solvent extraction technique. The mechanical properties of PA6/PS-g-PA6/PS blends were better than that of PA6/PS blends. Especially for the blends T10 with lower PS-g-PA6 copolymer content, both the flexural strength and flexural modulus showed significantly improving because of the improved interfacial adhesion between PS and PA6. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 1757–1765, 2007

Key words: macroactivator; TMI; PS-g-PA6; PS/PA6 blends; morphology

INTRODUCTION

Blending of polymers for production of new tailor made materials at low cost and for recycling mixed polymeric materials has gained great interest in recent years. The incompatibility of polyamides with many commercially interesting polymers, such as polyolefins or olefin elastomers, has already been solved by adding premade block or graft copolymers or by reactively forming them *in situ* at the interface during the compounding process.^{1–4} Recently, reactive compatibilization of immiscible blends has received more attention in terms of fine control over the phase morphology development in compatibilized blends. Polyamide 6 (PA 6) and polystyrene (PS) are commodity polymers each with unique properties. Thus, a PS/PA6 blend is expected to have commercial interest because it could provide a combination of the useful properties of its constituents.

Many studies deal with *in situ* chemical reaction compatibilization based on functionalized styrene copolymers, such as styrene maleic anhydride copolymer (SMA),^{5–7} or poly(styrene-co-sodium acrylate),⁸ which are capable of reacting with amine end groups of the polyamide during melt mixing.

The anionic polymerization of ϵ -caprolactam (CL) is a very useful method for producing articles. PA 6 can be synthesized in a few minutes by activated anionic polymerization of ϵ -CL in the presence of a catalyst (sodium caprolactamate, NaCL) and an activator. The activator is typically an isocyanate (NCO)-bearing compound. Hu and coworkers^{9–11} described a concept of *in situ* polymerization combined with *in situ* compatibilization for obtaining stabilized nanoblends based on polypropylene (PP) and PA6. It consists of polymerizing CL in the presence of a major PP phase, using a twin-screw extruder. PP bearing NCO groups was used as the macroactivator for CL anionic ring-opening polymerization. Similarly, PA6/Ethylene-Butylene (EB) elastomer blends having submicron phase morphology were prepared via polymerizing CL in the presence of EB elastomer bearing functional end groups of NCO.¹² 3-Isopropenyl- α,α -dimethylbenzene isocyanate (TMI) is a unique monomer because of its interesting

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bifunctional character: a free radically reactive double bond and an NCO group. Unlike classical NCOs, the NCO group of TMI is relatively insensitive to moisture under typical polymer processing conditions. As mentioned before, TMI have been used as a precursor for preparation of NCO-bearing macroactivators for the anionic polymerization of CL using reactive extrusion. However, the macroactivator was graft copolymer obtained via free radical grafting of TMI attached to the polymer backbones. It is well known that TMI can also be copolymerized with various vinyl monomers, for example, styrene.^{13–15} The copolymer of styrene and TMI are of great interest for compatibilizing the blends of PS and PA6.

The aim of this study was to develop a new method for synthesis of compatibilized blends of PS and PA6 via *in situ* polymerization starting with monomer styrene, TMI, and CL using the concept of *in situ* polymerization and *in situ* compatibilization. The process consists of firstly free radical copolymerization of styrene and TMI in reactive CL, and secondly anionic ring-opening polymerization of CL in the presence of PS-*g*-NCO copolymer. The basic idea is that NCO-bearing PS via free radical copolymerization was used as the macroactivators for the anionic polymerization of CL. Thus PA6 molecular chain was polymerized onto the PS backbone via initiating site of NCO incorporated into the PS chain. Additionally, the noncompatibilized PA6/PS system synthesized via successive *in situ* polymerization was used for comparison.

EXPERIMENTAL

Materials

TMI (Sigma-Aldrich, USA; 95%) was used as received. Azodi-isobutyronitrile (AIBN) (Shanghai Chemical Reagents Company, China; analysis grade) was recrystallized twice from ethanol before use. Styrene (Shanghai Chemical Reagents Company, analysis grade) was freshly distilled at 40°C under vacuum before use. CL was obtained from Nanjing Oriental Chemical Company (Commercial Grade, China). NaCl was used without further treatment.

Copolymerization of styrene and TMI in CL

Firstly, the CL was vacuumed at 120°C to eliminate the trace water because that the NCO group of TMI is moisture sensitive. Secondly, 40 wt % styrene and 0.8 wt % TMI were dispersed in the CL at 80°C, and a homogeneous transparent mixture was observed. The polymerization was carried out using 0.2 wt % AIBN as the initiator at 80°C for 24 h. After it, a homogeneous and viscous mixture was obtained. The resulted copolymer of styrene and TMI was denoted

as poly(St-*co*-TMI), and the total polymerization system (both polymer and CL) was denoted as mother mixture.

Preparation of PA6/PS-*g*-PA6/PS blends and PA6/PS blends

Part of the mother mixture was took out and mixed with predetermined CL to desired composition. The system was vacuumed at 140°C for 20 min to remove residual styrene monomer. Then 0.2 wt % NaCl was added under stirring for another 5 min. Afterwards, additional TMI was added with stirring, and the total concentration of the TMI (both in the mother mixture and the supplementary) was maintained as 0.8 wt %, a typical amount of activator for the anionic polymerization of CL. Considering the influence of TMI concentration on the anionic polymerization of CL (for example the molecular weight of PA6), so the TMI content was kept as a constant in all experiment. The mixture was then immediately poured into a mold preheated to 180°C, polymerized in oven at 180°C for 20 min, and subsequently the PA6/PS-*g*-PA6/PS blends was obtained after cooling to room temperature. The PA6/PS blends via similar successive *in situ* polymerization were made in our laboratory.

Characterization

FTIR was carried out with a Nicolet AVATAR 360 FTIR spectrometer in the range 4000–500 cm⁻¹, with a resolution of 2 cm⁻¹. The radical copolymerization products were first precipitated in octane, and then the sediment was dissolved in tetrahydrofuran (THF). The solution was then charged dropwise to *n*-octane at room temperature. The polymer precipitated out while the unreacted TMI monomer and CL remained soluble. The dissolving and precipitate process was repeated five times for purifying completely. The resulted products were dried in vacuum at 150°C for 24 h before observation.

A Waters-991 model GPC was used to evaluate the weight-average molecular weight (M_w) of PS and poly(St-*co*-TMI) and their polydispersity (M_w/M_n), where M_n is the number-average molecular weight. PS standards were used for column calibration. The analyses were carried out in THF solution (20 mg mL⁻¹). For the copolymer poly(St-*co*-TMI), due to the low reactivity ratio of TMI, the amount of TMI incorporated into the copolymer was very little when compared with the styrene, so the molecular weight contributed by TMI can be ignored.

The morphology was examined with a JSM LV-5610 scanning electron microscope (JEOL). The samples were held in liquid N₂ and a brittle fracture was performed. Specimens were etched with either

THF or formic acid to dissolve the PS and PA6 homopolymers, respectively. The etched surface was coated with gold before observation.

The Izod notched impact strength was measured according to ASTM D256, using an IZOD machine Model CSI-137D by the pendulum method at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The flexural strength and modulus were determined according to ASTM D 790.

RESULTS AND DISCUSSION

Copolymerization of styrene and TMI

TMI is a bifunctional monomer with an unsaturation and an NCO group. Its NCO group provides an excellent chemical scaffold for further chemical modification. Unfortunately, TMI does not polymerize in free-radical bulk homopolymerizations because of its α -methyl group, which induces significant steric hindrance.^{14,16} However, TMI can be either free-radical copolymerize with various other vinyl monomers such as styrene^{13,15} or attached to polymer backbones by free radical grafting.^{9–11} Low reactivity ratio values for TMI were obtained for the copolymerizations with styrene as a result of the inability of TMI to undergo homopolymerization and the slower polymerize rate.¹⁴ Copolymers of styrene and TMI via radical copolymerizing, denoted as poly(St-co-TMI), are of great industrial potential. Copolymer poly(St-co-TMI) was prepared via AIBN initiated radical copolymerization of styrene and TMI in CL following by repeated dissolving and precipitating for completely purification, and the corresponding FTIR spectrum was given in Figure 1. In the spectrum, there was a peak at 2255 cm^{-1} , corresponding to the NCO group, and this indicates the presence of the copolymer poly(St-co-TMI). The quantitative

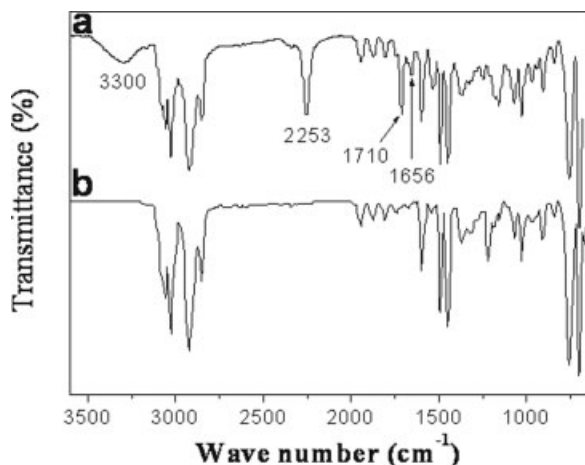


Figure 1 FTIR spectrum of (a) poly(St-co-TMI) synthesized via radical copolymerization in CL, and (b) pure PS for comparison.

studies of the weight percentages of the NCO group attached onto the copolymer are still underway, and we'll describe it elsewhere. In addition, two new bands at 1656 and 1710 cm^{-1} corresponding to the amide and the imide, were also found. At the same time, one peak appeared at 3300 cm^{-1} , corresponding to the formation of the acylactam.¹⁷ To illustrate this, the following explanation can be proposed. During the radical copolymerization of styrene and TMI in CL, a portion of the CL molecule may be activated by part of the NCO group on the molecule chain of poly(St-co-TMI) due to the comparative high temperature (80°C) and long time (24 h) for the radical polymerization. As a result, the CL monomer was attached to the copolymer, and the correlative chemical basis was shown in Figure 2, eq. (2).

The monomer conversion ratio was followed by gravimetry. The final value is 92.8%, and it can be deemed approximately as the conversion ratio of styrene. The reasons were as follows: First, the total amount of TMI was very tiny (St/TMI = 50/1), so the TMI units incorporated by copolymerization were neglectable. Second, CL units introduced to the copolymer chain via activated by the NCO group of TMI, considering the low TMI content, the contribution of the CL unit was negligible too.

Synthesis of PA6/PS-g-PA6/PS blends

Poly(St-co-TMI) formed *in situ* was used as macroactivators for anionic polymerization of CL to compatibilize and control the morphology structure of PS/PA6 blends. The whole grafting polymerization process of CL, including activation by the macroactivator and propagation process was shown in Figure 2. It is important to note that the NCO was permanently attached to the PS chain. The final product was expected to be a graft copolymer with PS as the backbone and PA6 as grafts and will be designated as PS-g-PA6.

Additionally, monomer TMI also existed in the system of the mixture before CL polymerization. The monomer TMI has two parts: First, during the radical copolymerization process, TMI might not completely incorporate into the copolymer because of the limitation of the reaction conversion degree. Furthermore, the unreacted monomer removed process can not eliminate the TMI monomer because of its higher boiling point. Second, additional TMI for retain equivalent concentration of TMI were also supplemented to the system before anionic polymerization of the CL. So the CL could be activated by two kinds of NCO: either the macroactivator of poly(St-co-TMI) or the microactivator of TMI monomer. Consequently, CL was also activated by TMI monomer and the PA6 was existed in form of both homopolyamide 6 (denoted as PA6) and copolymer PS-g-PA6.

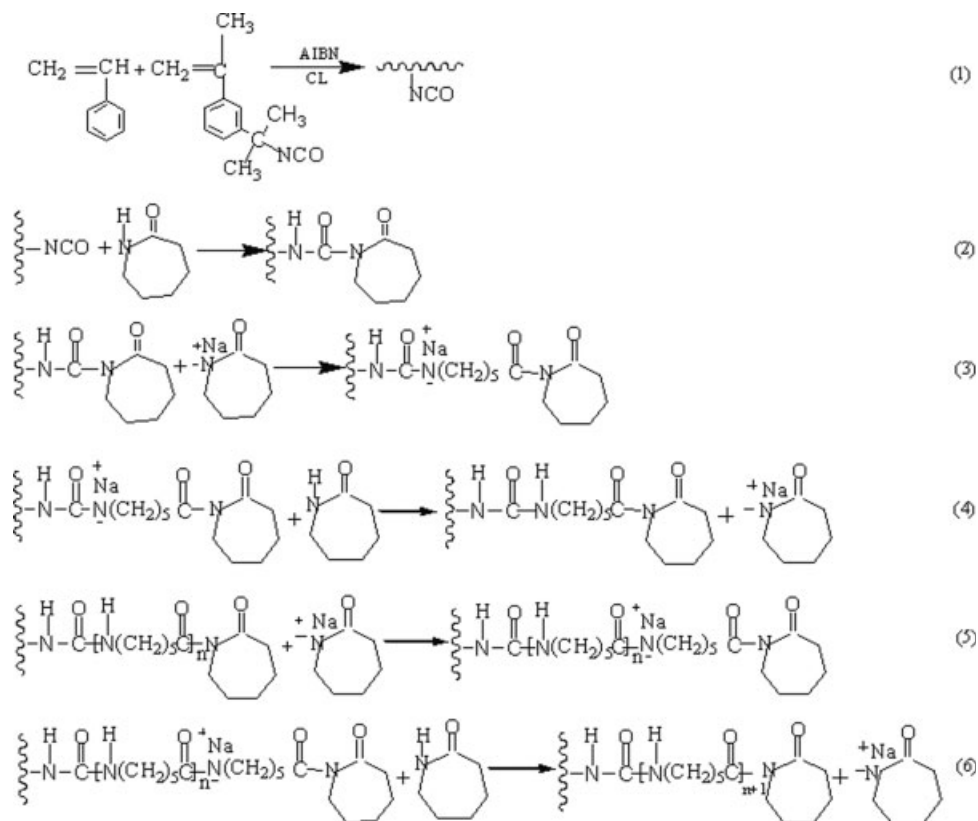


Figure 2 Mechanism of the formation of PS-g-PA6 copolymer.

PA6/PS-g-PA6/PS blends with different component weight ratio were prepared, as shown in Table I.

Morphology of PA6/PS blends

Prior to the study of the ternary compatibilized PS/PS-g-PA6/PA6 blends a complete view on the phase morphology development of the uncompatibilized PA6/PS blends is necessary. Figure 3 showed the SEM micrographs of the noncompatibilized binary PA6/PS blends. In Figure 3(a), the blends were etched by THF and the black domains corresponded to the extracted PS phase, and it showed that the PS was dispersed as spherical minor domains in blends. In case of PS content of 20 and 30 wt %, the blends

could be etched neither by THF nor by formic. THF made the blends fell apart while formic resulted in the greatly swollen blends. SEM micrographs of unextracted binary PA6/PS blends with PS 20 and 30 wt % were given in Figure 3(b,c). Spherical domain size increased dramatically to larger than 20 μm , but then the dispersed domain size decreased acutely to less than 10 μm with the fraction of PS in blends increasing, e.g., spherical domain size decreased with the fraction of PA6. These phenomena indicated that phase morphology showed strong dependence on the composition, from a dispersion system to an inverse dispersion system, through phase inversion (dual-phase continuity), and the phase inversion occurred when PS content was between 10 and 20 wt %. In case of PS content of 40 wt %, PA6 particles was thoroughly isolated by PS, i.e., the PS was matrix and PA6 was dispersed domain, which was confirmed by the SEM of formic etched binary PA6/PS blends in Figure 3(d). The detailed description of the morphology development of PA6/PS blends can also be found in our previous work.¹⁸

TABLE I
Composition of the PA6/PS-g-PA6/PS Blends

Trial	Mother mixture ^a /CL	St/CL	TMI ^b	NaCl
	wt/wt	wt/wt	wt %	wt %
T10	1/3	10/90	0.6	0.7
T20	1/1	20/80	0.4	0.7
T30	3/1	30/70	0.2	0.7
T40	1/0	40/60	0	0.7

^a Mother mixture denote the system of radical polymerization of styrene and TMI in CL.

^b Additional TMI added to keep the total TMI at 0.8%.

Selective solvent extraction of PS/PS-g-PA6/PA6 blends

Selective solvent extraction technique was used to evaluate the composition of the PS/PS-g-PA6/PA6

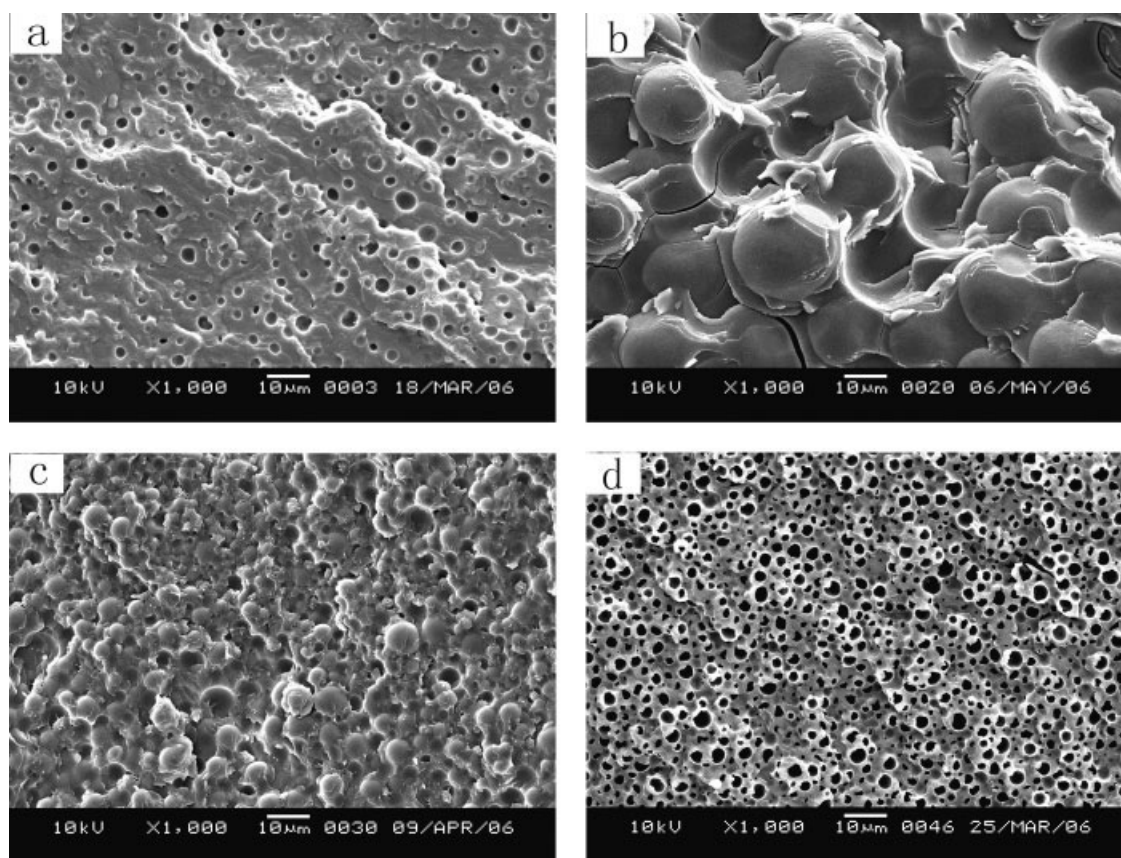


Figure 3 SEM micrographs of the PA6/PS blends (a) S10 (90PA6/10PS) etched by THF, (b) S20 (80PA6/20PS), (c) S30 (70PA6/30PS), and (d) S40 (60PA6/40PS) etched by formic acid.

blends. The process consisted in using THF and formic acid as extracting solvents. Known amounts of predried films were placed in the solvents under reflux for 72 h. Formic acid is a solvent for PA6 and a nonsolvent for PS and PS-*g*-PA6, while THF is a solvent for homopolystyrene and a nonsolvent for PA6 and PS-*g*-PA6. After successive extraction in THF and then in formic acid, there were still some residue that could not be dissolved. They corresponded to PS-*g*-PA6 copolymer. The weight difference before and after every extraction trial was corresponded to the PS and PA6, respectively. The results were summarized in Table II. The amount of the PS homopolymer in the T10 was found to be 2.53 wt %, and it was increased as expected with

increasing of the content of poly(St-*co*-TMI) added before anionic polymerization of the CL. For T40, PS homopolymer was 9.36 wt %. The weight percentage of PS-*g*-PA6 copolymer increased expectedly from 13.40 to 54.91 wt % with increasing content of poly(St-*co*-TMI). Contrarily, the PA6 existed in form of homopolymer was reduced, from 84.07 wt % in T10 to 35.73 wt % in T40. The reason will be given in the next part.

Morphology of PS/PS-*g*-PA6/PA6 blends

The SEM micrographs of PS/PA6 blends were given in Figure 3. Our goal is to study the effect of the poly(St-*co*-TMI) on the morphology of these blends. It is important to note that PS ($M_w = 11.1 \times 10^4 \text{ g mol}^{-1}$) and poly(St-*co*-TMI) ($M_w = 9.8 \times 10^4 \text{ g mol}^{-1}$) have a very comparable molecular weight and melt viscosity because this can also influence the characteristic of the morphology. Because the blends were constituted of homopolystyrene, PA6, and PS-*g*-PA6, the cryofracture surfaces may show complex phase morphology where it is not possible to identify which phase is located where. To confirm how the homopolystyrene, PA6, and PS-*g*-PA6 copolymer existed,

TABLE II
Data Determined from Selective Solvent Extraction Technique

Trial	PS wt %	PA6 wt %	PS- <i>g</i> -PA6 wt %
T10	2.53	84.07	13.40
T20	4.83	68.09	27.08
T30	7.95	52.43	39.62
T40	9.36	35.73	54.91

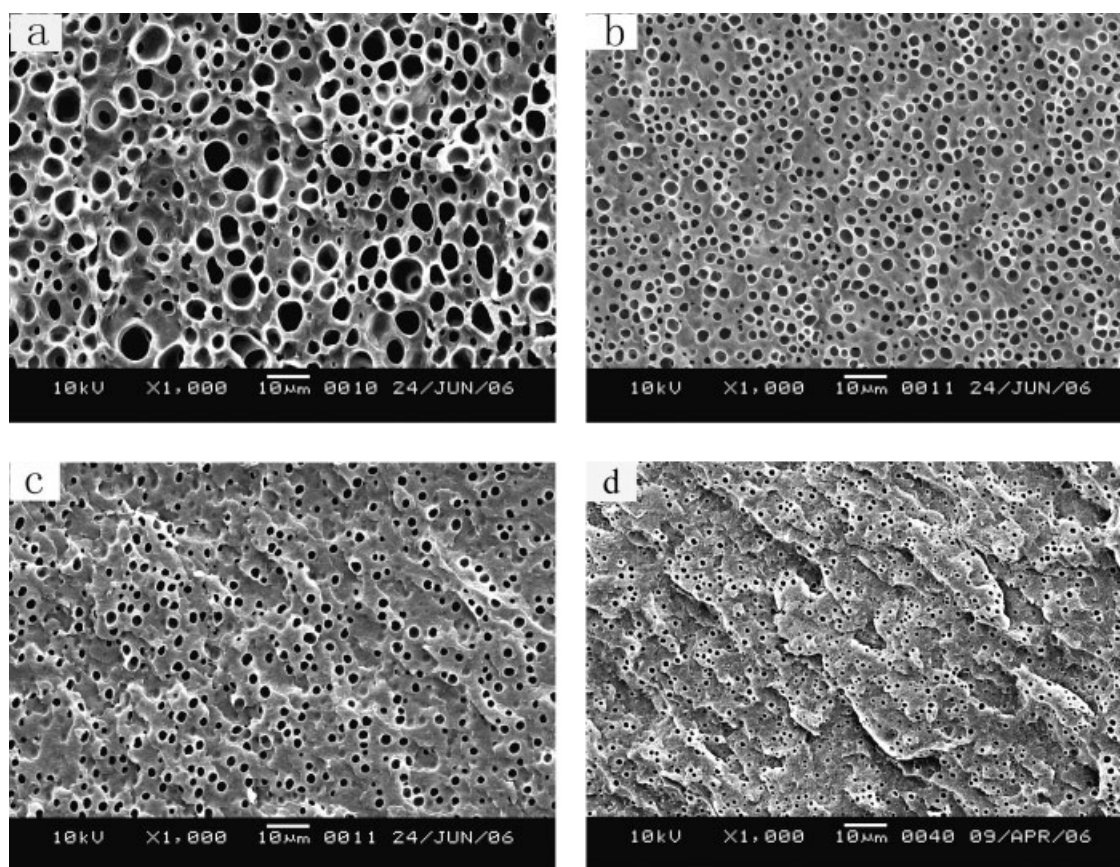


Figure 4 SEM micrographs of the blends PA6/PS-g-PA6/PS etched by formic acid. (a) T10, (b) T20, (c) T30, (d) T40.

the two parts of the same blends were selective etched by THF and formic acid, respectively.

As shown in Figures 4 and 5, the morphology of the PA6/PS-g-PA6/PS blends obtained from the polymerization of CL in presence of poly(St-co-TMI) copolymer was very interesting. In Figure 4, the formic acid treatment revealed that the homopolymer PA6 phase was in the form of minor phase as indicated by the dark holes. As can be seen from it, a further increase of the mother mixture (copolymerization product of the styrene and TMI in CL) content (i.e., decrease of the CL content) resulted in a further decrease of the PA6 phase size. This can be contributed to the following factors: firstly, the total amount of the PA6 (both copolymer PS-g-PA6 and homopolymer PA6) was decrease. Secondly, due to the supplemented amount of the microactivator TMI was reduced, the total amount of the microactivator TMI reduced (both from mother mixture and supplemented), the amount of the PA6 resulted was reduced too. Additionally, the content of poly(St-co-TMI) copolymer constituent was increased, so more CL was activated and more PS-g-PA6 was resulted. These results obtained from TEM were in well agreement with those of the selective solvent extraction given in Table II. The PA6 phase size decrease with

decreasing of its content is expected since the process of particle–particle coalescence is suppressed.

The SEM picture of Figure 5 was obtained from samples where only PS was selectively extracted by THF. It revealed that there were no obvious holes or hollow spaces. Dose the PS phase form a dispersion phase or is it continuous? By carefully observation, it can be found that in cases of T20, T30, and T40, bimodal particle size distribution was presented, i.e., the systems were composed with both larger spherical particles with sizes of micrometer scale and smaller particles with sizes of nanometer scale. Compared with the blends that etched by formic acid (Fig. 4), the dispersed larger spherical particles were corresponded to the homopolymer PA6. So the dense and smaller nanometer particles were corresponded to the remaining PS-g-PA6 copolymer phase. The formation of nanometer particles has been ascribed to the phase separation of the PS-g-PA6 copolymer at higher concentrations. The graft copolymer PS-g-PA6 is repelled from the interface of matrix/dispersed particle and forms a separate micelle phase, instead of assembling at the interfaces.^{3,5} Interestingly, copolymer nanoparticles can not be found in the SEM micrograph that etched by formic acid (Fig. 4), but it can be observed clearly in the

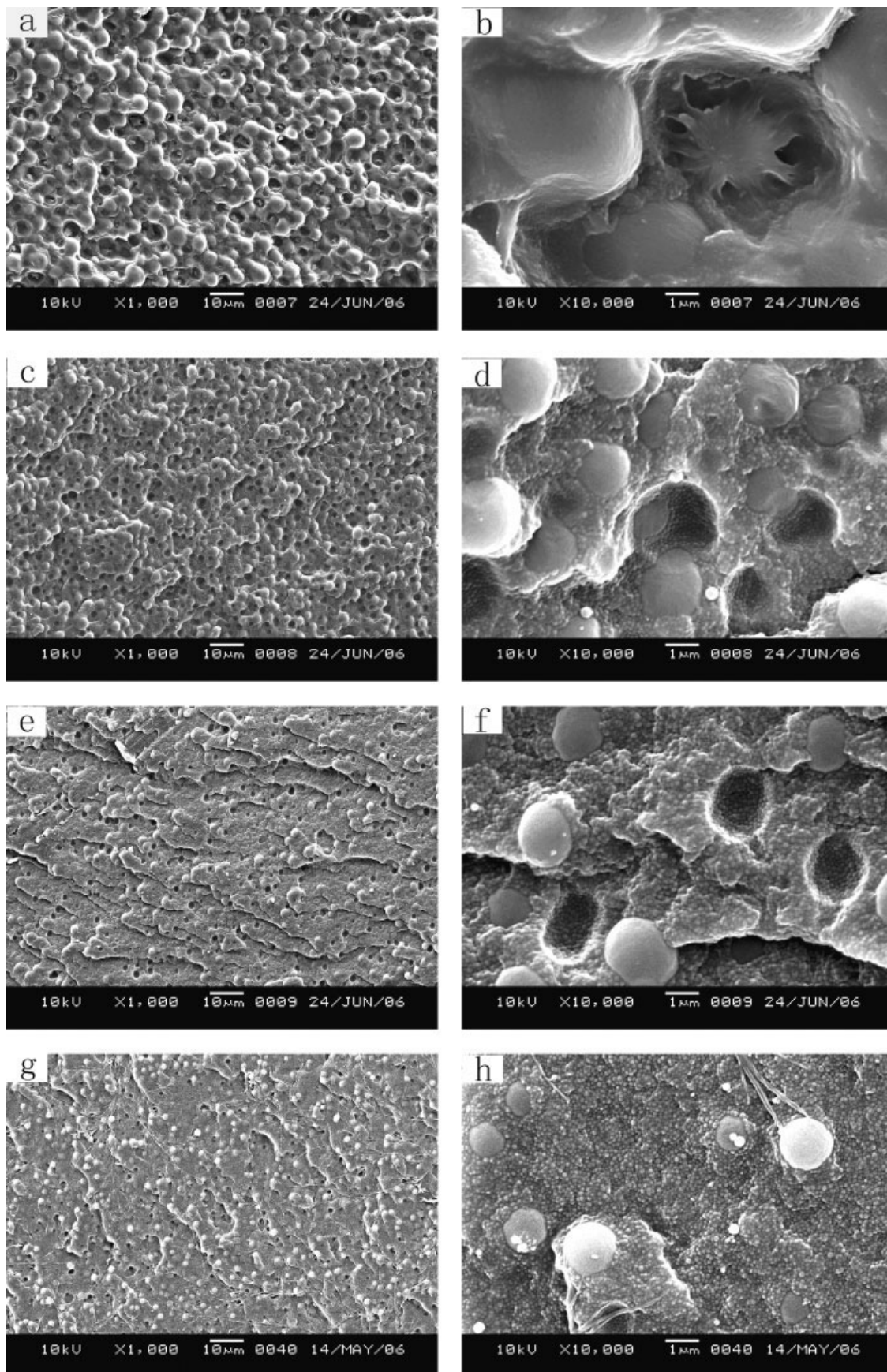


Figure 5 SEM micrographs of the blends PA6/PS-g-PA6/PS etched by THF. (a,b) T10, (c,d) T20, (e,f) T30, (g,h) T40.

micrograph etched by THF. Such a system of *in situ* polymerization and *in situ* compatibilization is very complicated, and from the phenomena mentioned earlier, we can draw a conclusion that the PS-*g*-PA6 graft copolymer may coexist with the PS forming the matrix phase, in which the PS was exist in the inter-spaces among the dispersed copolymer nanoparticles. While in case of T10 [Fig. 5(a,b)], the copolymer nanoparticles was hardly found even at higher magnification. It can be attributed to the fact that little amount of the poly(St-*co*-TMI) was introduced and hence proper amount of PS-*g*-PA6 copolymer was gained. The PS-*g*-PA6 copolymer concentrated at the interface of matrix and PA6 dispersed particles and played the role of a compatibilizer, which improved interfacial adhesion between PS and PA6.

Mechanical property

The morphological of polymer blends significantly affects the final mechanical properties (Table III). For PA6/PS binary blends, the flexural strength, flexural modulus, and the notched impact strength show a decline first and then a slight rise with increasing of the PS content. These phenomena can be contributed to the varied phase morphology. In case of S10, PS minor phase was dispersed in the PA6 matrix, which contributed substantially to the mechanical properties. In cases of S20 and S30, phase inversion was occurred, and the PA6 was dispersed as dozens of micrometer spheres, but the PS content was relatively too small to enclose the PA6 microspheres entirely, so the mechanical were all very poor. While for S40, the PA6 minor phase was dispersed in the PS matrix, so the properties show a little improvement.

For PA6/PS-*g*-PA6/PS blends, the mechanical properties were better than those of the PA 6/PS blends as a whole, and it can be contributed to the differences in morphology characteristic of them. In case of PA 6/PS blends, the PA6 phase was dispersed in the PS matrix when the PS was up to 20 wt %; both the PS matrix and the poor binding

resulted in the inferior mechanical property. While in case of PA6/PS-*g*-PA6/PS blends, the copolymer PS-*g*-PA6 together with the PS formed the matrix, and at the same time, finer PA6 dispersed phases were formed, hence better mechanical properties were obtained comparing with that of noncompatibilized PA 6/PS blends. Noticeably, the best properties corresponded to T10, in which the copolymer PS-*g*-PA6 content was the lowest. Flexural strength and flexural modulus improved 50 and 27% than that of pure PA6, respectively. What led to such great improvement of mechanical properties? From the SEM [Fig. 5(b)], we can find that the interfacial adhesion between PS matrix and PA6 was pretty well. The probably reason may be that these copolymer locating at the interface played a good compatibilization role for the incompatible PA6 and PS to improve the interfacial adhesion, thus favorable properties was gained. However, with increasing of the copolymer PS-*g*-PA6 content, the mechanical properties of PS/PS-*g*-PA6/PA6 blends began to decline. Especially, flexural strength decreased 54% when increasing copolymer content from 10 to 20 wt %. Probably reason maybe that with more PS-*g*-PA6 introduced, the compatibilizer PS-*g*-PA6 was overloaded and it was repelled from the interface of PA6 and PS. As a result, phase separation arose and a great quantity of discrete compatibilizer domains formed as shown in Figure 5(c-h). Therefore, the mechanical properties showed unsatisfactory performance to some extent when compared with that of T10.

CONCLUSIONS

In summary, macroactivator poly(St-*co*-TMI) was synthesized *in situ* via free radical copolymerizing of styrene and TMI in the reactive monomer CL, and then ternary PA6/PS-*g*-PA6/PS blends were prepared after *in situ* anionic ring-opening polymerizing of CL in presence of both macroactivator poly(St-*co*-TMI) and micro-activator TMI. The morphology of the ternary PS/PS-*g*-PA6/PA6 blends indicated that both PA6 and copolymer PS-*g*-PA6 were dispersed as spherical particles with bimodal particle size distribution, in which the size of the PA6 was several micrometers and the copolymer PS-*g*-PA6 was in the nanometer scale. Compared with binary PA6/PS blends, the size of the PA6 minor phase was decreased dramatically upon poly(St-*co*-TMI) copolymer introducing. Extraction techniques showed that the amount of the PS homopolymer and PS-*g*-PA6 copolymer were all increased while the PA6 homopolymer was decreased with increasing of the mother mixture. The phase morphology of the blends dominated their ultimate mechanical

TABLE III
Mechanical Properties of PA6/PS and PA6/PS-*g*-PA6/PS Blends with Different Composition

Sample	Flexural strength (MPa)	Flexural modulus (MPa)	Notched impact strength (J/m)
PA6	82.3	2428	43
S10	53.9	2037	37
S20	31.8	1702	13
S30	24.3	1857	18
S40	27.2	2100	20
T10	123	3077	45
T20	56.6	2789	32
T30	58.3	2670	27
T40	53.2	2438	22

properties. For PA6/PS blends, weak adhesion led to the phase separation, and as a result, inferior mechanical properties arose. For T20, T30, and T40 in PA6/PS-*g*-PA6/PS blends, mechanical properties showed improved results due to that PS-*g*-PA6 together with PS formed the matrix. For T10 with lower copolymers PS-*g*-PA6 content, best mechanical properties were found. This can be attributed to the compatibilizer concentrating at the interface improved the interfacial adhesion.

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