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Effect of the functional group inhomogeneity of an in situ reactive compatibilizer on the morphology and rheological properties of immiscible polymer blends

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Abstract

The effect of the poly(acrylic acid) homopolymer [PAA] existing in poly(propylene-*graft*-acrylic acid) [PP-*g*-AA] on the morphology and rheological properties of a PP-*g*-AA and polystyrene [PS] blend with varying amounts of poly(styrene-*ran*-glycidyl methacrylate) [PS-GMA] was investigated. Two different PP-*g*-AAs, one with PAA, [PP-*g*-AA(O)] and the other without PAA, [PP-*g*-AA(T)], were used. We have shown that when the amount of PS-GMA is increased, the dispersed domain size of PP-*g*-AA(O) in the PP-*g*-AA(O)/PS blend *does not* decrease, even when the amount of PS-GMA in the blend is as large as 25 wt%. Conversely, the dispersed domain size of PP-*g*-AA(T) in the PP-*g*-AA(T)/PS blend *does* decrease gradually and reaches a steady value. However, the reduction in the dispersed domain size (D_r) for PP-*g*-AA(T) in the PP-*g*-AA (T)/PS blend with PS-GMA was just 30%. Here, D_r is defined by the dispersed domain size of the blend without PS-GMA minus that with 15 wt% of PS-GMA divided by the former. The changes in rheological properties with the proportion of PS-GMA content in the PP-*g*-AA(T)/PS blend were different to those for the PP-*g*-AA(O)/PS blend, thus demonstrating that the presence of PAA significantly affects the rheological as well as the morphological blend properties.

Keywords: Reactive compatibility; Poly(propylene-*graft*-acrylic acid); Functional group inhomogeneity

1. Introduction

Many compatible polymer blends, exhibiting very fine morphology and thus good mechanical properties, have been developed. Among many compatibilization techniques, reactive melt blending methods that graft or block copolymers as compatibilizers formed in situ by the reaction between functional units in the blends, have been widely used [1–8].

It is well established that in the reactive compatibilization, the amount of the functional group in the reactive compatibilizer is very important in determining morphological and mechanical properties [9,10]. When the amount of the functional group is too high, the reaction results in the formation of graft copolymers with a very high molecular weight or even those of the cross-linked type; thus, these copolymers are not well distributed near the interface between two immiscible polymers because of high viscosity. In contrast, if the amount of the functional group is too small, few graft copolymers are formed and thus cannot play efficient roles as compatibilizers; the final physical properties of the polymer blend become poor.

Very recently, the authors [11] did indeed find that, in order to reduce effectively the dispersed domain size in a poly(ethylene-*ran*-acrylic acid) [PE-*r*-AA]/polystyrene [PS] blend compatibilized by poly(styrene-*ran*-glycidyl methacrylate) (PS-GMA), there exists an optimal amount of acrylic acid in PE-AA that promotes a reaction suitable for the formation of sufficient amounts of graft copolymers as compatibilizers between PE-AA and PS, as well as fast diffusion of the produced copolymers towards the interface.

However, in addition to the amounts of functional groups in an in-situ compatibilizer, the molecular architecture of an in-situ compatibilizer plays an important role in determining the compatibilization efficiency. In our previous paper [12], we reported that in two different in-situ compatibilizers, PE-*r*-AA and poly(propylene-*graft*-acrylic acid) [PP-*g*-AA], only PE-*r*-AA played an effective compatibilization role in reducing the dispersed domain size in a PE-*r*-AA/PS/PS-GMA blend system, while PP-*g*-AA *did not* play an effective role in a PP-*g*-AA/PS/PS-GMA blend system,

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even when the amount of PS-GMA added is up to 9 wt%. This is because of the existence of the homopolymer of poly(acrylic acid) [PAA] in PP-*g*-AA. Since many acrylic acid units are available in PAA, if even a small amount of PAA exists in PP-*g*-AA, it can act as a scavenger of GMA groups in PS-GMA. This suggests that, in the presence of PAA, the reaction between PS-GMA and PP-*g*-AA, which results in the formation of in situ poly(propylene-*graft*-styrene) [PP-*g*-PS] copolymers, is much suppressed. Therefore, in order to study the effect of the molecular architecture of an in-situ compatibilizer on the morphology of a blend with PP-*g*-AA, homopolymer PAA should first be removed from the PP-*g*-AA.

In this study, we investigate in detail the effect of the existence of PAA in PP-*g*-AA on the morphology and rheological properties of a PP-*g*-AA/PS/PS-GMA blend system.

2. Experimental

2.1. Materials

The PS and PS-GMA used in this study were the same as those described in our previous paper [12].

The PP-*g*-AA was a commercial grade (Polybond 1002, Uniroyal Chemical) in pellet form, kindly donated by Dr C. Mudrick. The amount of acrylic acid in the PP-*g*-AA was 5.5∼7.5 wt%, according to the manufacturers brochure.

However, as reported in our previous paper [12], Polybond 1002 contained some homopolymer PAA not grafted to PP chains. This PAA could be removed by dissolving Polybond 1002 into methanol at 40°C for 1 month since methanol is a good solvent for PAA but not for PP or PP-*g*-AA. In order to facilitate the dissolving of PAA effectively, the pellet form of Polybond 1002 was crushed into very fine powder under a liquid nitrogen environment, using a grinder. The molecular weight of the original Polybond in pellet form did not change after grinding. We found that ∼2.0 wt% of PAA was extracted from Polybond 1002, determined by subtraction of the final weight of Polybond 1002 after dissolving into methanol from the original weight of Polybond 1002. Hereafter, Polybond 1002 as received, and Polybond 1002 after removing PAA are referred to as PP-*g*-AA(O), and PP-*g*-AA(T), respectively.

The number-average molecular weight (M_n) and the polydispersity index (M_w/M_n) of PP-*g*-AA(O) were 81,000 and 2.52, respectively, and those of PP-*g*-AA(T) were 84,000 and 2.5, respectively, both determined by high temperature gel-permeation chromatography (GPC) (Waters 150C) using calibration curves for standard PE. It should be mentioned that due to using different sample lots, the molecular weight of Polybond 1002 employed in this study (PP-*g*-AA(O)) was different from that employed in our previous study [12], where the M_n and the polydispersity index were reported to be 73,000 and 2.5. This was inevitable since a relatively large amount of PP-*g*-AA(T) was needed for this experiment.

The M_n and the polydispersity index of PAA extracted from PP-*g*-AA(O), determined by GPC and using a solvent of tetrahydrofuran, were 1430 and 1.67, respectively. The molecular weight was calculated on the basis of the calibration curve obtained from standard polystyrenes.

2.2. Melt blending

All materials employed in this study were dried in a vacuum oven at 100°C for 1 day. Two blend compositions, namely, 10/90 wt/wt and 30/70 wt/wt of PP-*g*-AA(O)/PS and PP-*g*-AA(T)/PS blends with various amounts of PS-GMA (0–25 wt% based on the total PS phase consisting of neat PS and PS-GMA) were prepared by an internal mixer (Brabender Plasticorder) with a capacity of 30 cm^3 operating at a speed of 50 rpm for 10 min. In order to minimize the oxidative degradation of polymers during the melt mixing, nitrogen gas was purged into the mixer. It is known that GMA in PS-GMA reacts easily with acrylic acid units (AA) in PP-*g*-AA at higher temperatures [11–16]. Pellets of PP-*g*-AA(O) were also crushed into powders by a grinder under the same conditions as for PP-*g*-AA(T). The wall temperature inside internal mixer was 220°C for both blend systems.

2.3. Morphology

A scanning electron microscope (SEM; Jeol JSM-840A) was used to observe the morphology of specimens fractured at the cryogenic temperature, and then coated with a thin layer of gold. The number average dispersed domain size (D_n) was obtained by a Quantimet 570 image analyzer (Cambridge Instrument), using about 200–300 particles for each blend. The cross-sectional area (A_i) of each particle on the micrograph was measured, and then converted to the diameter (D_i) of a circle having the same cross-sectional area ($D_i = 2(A_i/\pi)^{1/2}$).

2.4. Rheological measurement

The complex viscosities (η^*), storage and loss moduli (G' and G'') of PP-*g*-AA/PS blends with varying amounts of PS-GMA as well as neat PS, PS-GMA, PP-*g*-AA(O), and PP-*g*-AA(T) were measured at various angular frequencies (ω) by an Advanced Rheometrics Expanded System (ARES) with 25 mm parallel plates under an oscillatory shear mode at 220°C. To avoid any oxidative degradation of the samples, all measurements were conducted under nitrogen atmosphere.

3. Results and discussion

3.1. Characterization of PAA in PP-g-AA

We reported in our previous paper [12] that homopolymer PAA ungrafted to PP chains existed as phase-separated

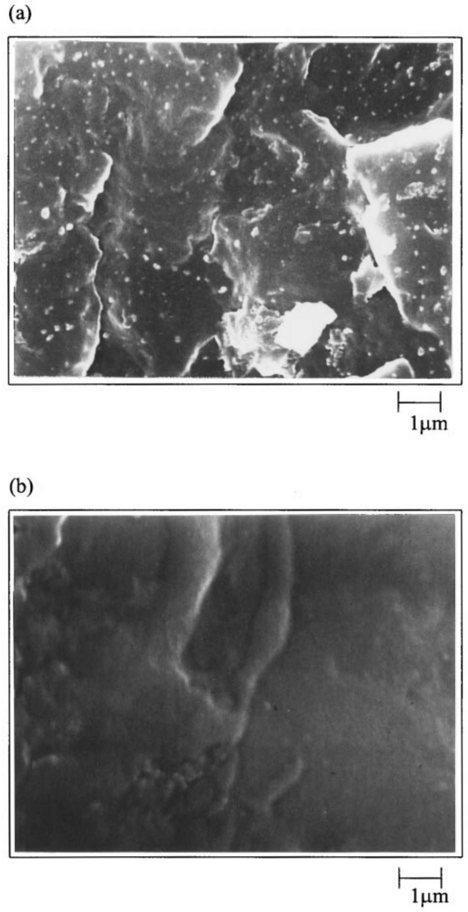

Fig. 1. Scanning electron micrographs of (a) PP-*g*-AA(O), as received PP-*g*-AA sample with PAA and (b) PP-*g*-AA(T), PP-*g*-AA without PAA. Here, dispersed domains consisting of PAA homopolymers are clearly visible as white spots in (a); these are seldom seen in (b).

domains in PP-*g*-AA(O), and that this could be confirmed by a scanning electron micrograph (SEM) image of the cryogenically fractured surface (transmission electron micrographs (TEM) give better images than SEM). Fig. 1 presents SEM images of PP-*g*-AA(O) and PP-*g*-AA(T) demonstrating that the phase-separated domain of PAA existing in PP-*g*-AA(O) (white spots in Fig. 1(a)) cannot be seen in PP-*g*-AA(T) (Fig. 1(b)) at this magnification scale. Thus, almost all portions of the PAA homopolymer in PP-*g*-AA(O) are considered to have been effectively removed by the solvent extraction.

Fig. 2 presents the FT-IR spectrum of the film of PAA extracted from PP-*g*-AA(O) by methanol. It can be seen that the IR spectrum of this film is essentially the same as that of neat PAA [17]. For instance, peaks appearing at the wave numbers (ν) of 3500~2500 cm^{-1} representing OH groups, ν of 1730 cm^{-1} representing C=O, and ν of 1200 cm^{-1} representing C–O are clearly seen.

Fig. 3 gives DSC thermograms of PP-*g*-AA(O), PP-*g*-AA(T), and PAA extracted from PP-*g*-AA(O). It can be seen that at ~100°C a small but distinct transition is detected for PP-*g*-AA(O), while no transition is detected for PP-*g*-AA(T) (see inner box in Fig. 3). The glass transition temperature (T_g) of PAA extracted from PP-*g*-AA(O) was 98°C. Al-Najjar et al. [18] showed that the T_g of the PAA homopolymer with a molecular weight of 2000 was 95°C but that at molecular weights greater than 250,000, it was 134°C. The molecular weight of the PAA extracted from PP-*g*-AA(O), determined by GPC, was 1430; thus the T_g of PAA given in Fig. 3 is reasonable. If the molecular weight of PAA grafted onto the PP main chain is assumed to be comparable to that of the PAA homopolymer, ~20 acrylic acid monomer (AA) units are grafted on to the PP main chain during the graft reaction.

PAA exists as phase-separated domains in the matrix of PP or PP-*g*-AA because PAA is highly immiscible with PP due to large differences in their solubility parameters [12]. Since PAA has many functional monomer units, it reacts very easily with other functional groups of other blend constituents. For instance, for the blend of PP-*g*-AA with PAA and PS with PS-GMA, most GMA can react with AA groups in PAA not PP-*g*-AA, thus the amount of PAA-*g*-PS becomes much larger than PP-*g*-PS. Of course, the in-situ formed PAA-*g*-PS copolymers are difficult to locate near the interface between PP-*g*-AA and PS, which suggests that PP-*g*-AA with PAA cannot be an efficient compatibilizer. It should be mentioned, however, that for a graft reaction of a maleic anhydride (MAH) onto a polyolefin chain at temperatures above 150°C, which is known as the ceiling temperature for MAH polymerization, only one (or a

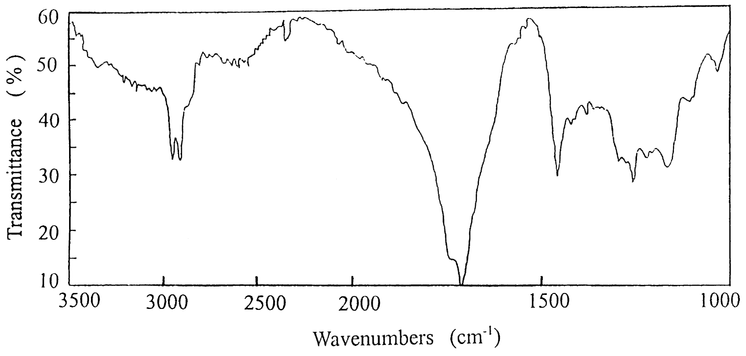

Fig. 2. FT-IR spectrum of PAA homopolymer extracted from PP-*g*-AA(O) using methanol.

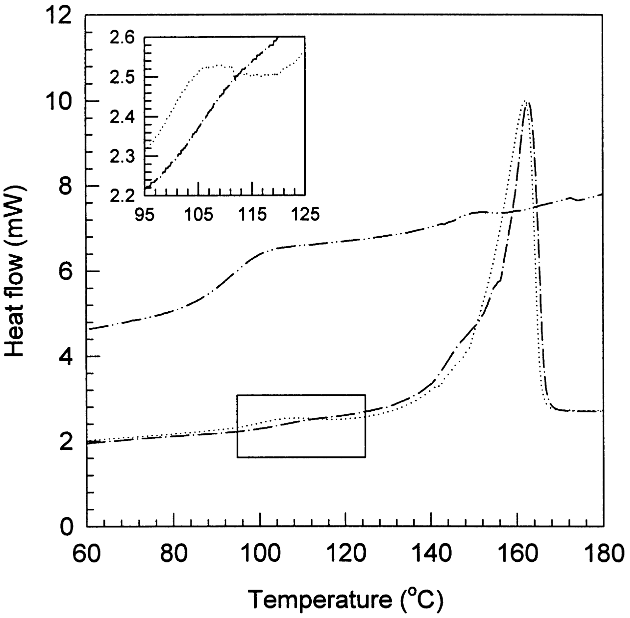

Fig. 3. Differential scanning calorimeter thermograms for PP-*g*-AA(O) (……), PP-*g*-AA(T) (— · —) and extracted PAA homopolymer (— ·· —).

maximum of two) monomer groups of MAH can be grafted onto the polyolefin chain, thus one does not worry about the existence of MAH oligomers or its homopolymers [19,20].

3.2. Morphologies

Fig. 4 presents SEM images for 30/70 wt/wt PP-*g*-AA(O)/PS with varying amounts of PS-GMA (wt% based on neat PS and PS-GMA). It can be seen in the figure that the dispersed domain size of PP-*g*-AA in the PP-*g*-AA(O)/PS blend *does not* decrease with increase in the amount of PS-GMA, even when the amount of PS-GMA is increased up to 15 wt% of the PS phase (i.e., 10.5 wt% of the total blend), which is consistent with our previous results [12]. SEM images for the 30/70 wt/wt PP-*g*-AA(T)/PS blend with varying amounts of PS-GMA are shown in Fig. 5, from which we note that, with increase in the amount of PS-GMA, the dispersed domain size in the PP-*g*-AA(T)/PS blend decreases until the amount of PS-GMA reaches 7 wt% and then levels out.

The summary of changes in the number average dispersed domain size (D_n) of PP-*g*-AA in 10/90 and 30/70 (wt/wt) PP-*g*-AA(O)/PS and PP-*g*-AA(T)/PS blends with varying amounts of PS-GMA is presented in Fig. 6 and the numerical values of D_n are given in Table 1. Note that D_n in the 10/90 (wt/wt) PP-*g*-AA(O)/PS blend in this study is ~1.1 μm which is slightly larger than that (~0.7 μm) reported in a previous paper [12]. This may be attributed to the fact that the sample lot of Polybond 1002 employed in this study (namely PP-*g*-AA(O)) was different from that used in our previous study. As explained in Section 2, the M_n and the

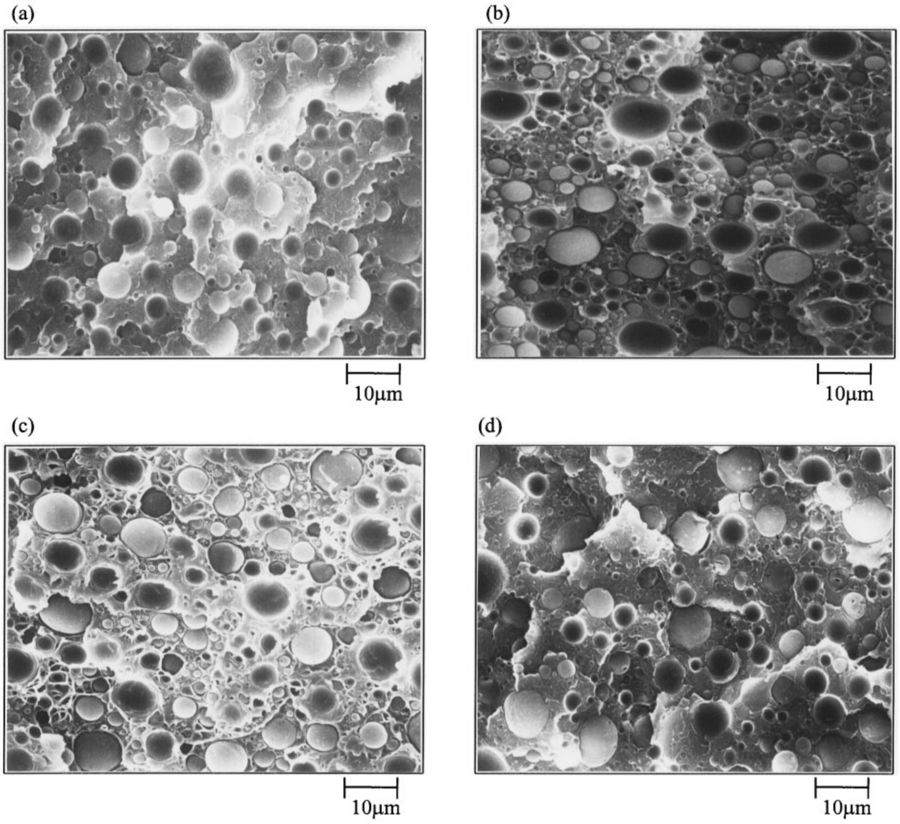

Fig. 4. SEM images of 30/70 (wt/wt) PP-*g*-AA(O)/PS blend with varying amounts of PS-GMA (wt% based on neat PS and PS-GMA): (a) 0; (b) 3; (c) 7; (d) 15.

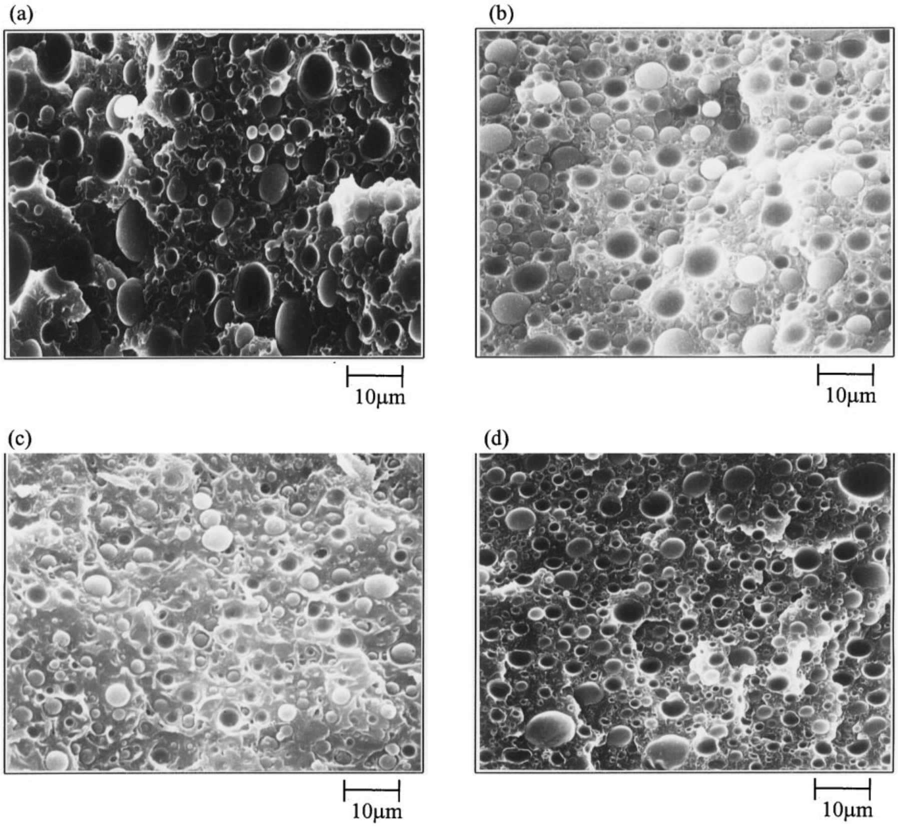

Fig. 5. SEM images of 30/70 (wt/wt) PP-*g*-AA(T)/PS blend with varying amounts of PS-GMA (wt% based on neat PS and PS-GMA): (a) 0; (b) 3; (c) 7; (d) 15.

polydispersity of PP-*g*-AA(O) employed in this study were 81,000 and 2.52, respectively, while those of Polybond 1002 in the previous study were 73,000 and 2.5, respectively [12].

It can be seen in Fig. 6 and Table 1 that D_n of PP-*g*-AA(O) in PP-*g*-AA(O)/PS blends is larger than that of PP-*g*-AA(T) in PP-*g*-AA(T)/PS blends when PS-GMA is not added to either blend. This is attributed to the fact that the ratio of viscosity of PP-*g*-AA(T) to PS is greater than that of PP-*g*-AA(O) to PS, since lower viscosity PAA exists in PP-*g*-AA(O). This was verified experimentally and will be discussed later.

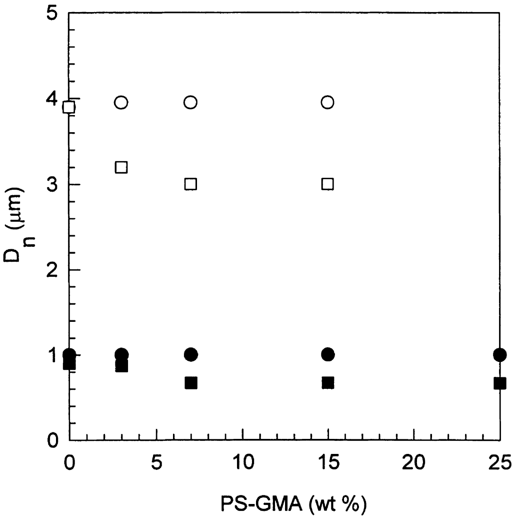

Fig. 6. Plots of the number average diameter (D_n) of the dispersed phase versus the amount of PS-GMA (wt% based on neat PS and PS-GMA) for two different blend systems with and without PAA: ●, 10/90 PP-*g*-AA(O)/PS blend; ■, 10/90 PP-*g*-AA(T)/PS blend; ○, 30/70 PP-*g*-AA(O)/PS blend; □, 30/70 PP-*g*-AA(T)/PS blend.

One can argue that the reason for the smaller decrease in D_n in PP-*g*-AA(O)/PS with increasing amount of PS-GMA compared to PP-*g*-AA(T)/PS, can be attributed to the fact that, compared with the latter blend, the reaction between AA and GMA in the former blend does not occur to a sufficiently great extent, thus producing only a small amount of PP-*g*-PS copolymer. Fig. 7 gives torque change versus blending time for 10/90 and 30/70 PP-*g*-AA(O)/PS-GMA and PP-*g*-AA(T)/PS-GMA, measured at a wall temperature of 220°C. It is seen that the torque increment (torque plateau appearing after 5 min minus minimum torque appearing at 2 min) for the 30/70 PP-*g*-AA(O)/PS-GMA blend is *greater* than that of the 30/70 PP-*g*-AA(T)/PS-GMA blend. Similarly, the torque increment for the 10/90 PP-*g*-AA(O)/PS-GMA blend is greater than that of the 10/90 PP-*g*-AA(T)/PS-GMA blend. Thus, we conclude that more reaction occurs between PP-*g*-AA(O) and PS-GMA than PP-*g*-AA(T) and PS-GMA. However, for the PP-*g*-AA(O)/PS-GMA blend, the large increment in torque is due to the reaction between AA in PAA and GMA producing PAA-*g*-PS, not due to the reaction between AA in PP-*g*-AA and GMA producing PP-*g*-PS. Since PP is very immiscible with PAA, PAA-*g*-PS does not play an effective compatibilizer role between PP and PS.

Table 1
The number average domain size (D_n) for two blend compositions (10/90 and 30/70 wt/wt) of PP-*g*-AA(O)/PS and PP-*g*-AA(T)/PS with varying amounts of PS-GMA

	Amount of PS-GMA				
Blend Systems	0	3	7	15	25
10/90 PP-*g*-AA(O)/PS	1.10 ± 0.40	1.10 ± 0.40	1.09 ± 0.38	1.06 ± 0.38	1.06 ± 0.38
10/90 PP-*g*-AA(T)/PS	0.97 ± 0.38	0.91 ± 0.36	0.68 ± 0.34	0.68 ± 0.30	0.68 ± 0.30
30/70 PP-*g*-AA(O)/PS	4.05 ± 0.74	4.04 ± 0.71	4.00 ± 0.73	4.00 ± 0.70	–[a]
30/70 PP-*g*-AA(T)/PS	3.72 ± 0.70	3.20 ± 0.68	2.85 ± 0.60	2.90 ± 0.62	–[a]

[a] not measured.

It should be mentioned that although D_n in 10/90 wt/wt and 30/70 wt/wt PP-*g*-AA(T)/PS blends decreases with increase in the amount of PS-GMA, the reduction in the dispersed phase (D_r) for PP-*g*-AA(T)/PS with PS-GMA is at most 30%, which is much smaller than that (about 80%) for poly(ethylene-*random*-acrylic acid) [PE-*r*-AA]/PS with PS-GMA [12]. Note that D_r for a blend is defined by D_n without PS-GMA minus that with 15 wt% of PS-GMA divided by the former. Although the chemical structure of PE-*r*-AA is different from PP-*g*-AA(T), the main difference in D_r between the two blend systems is attributed to the different molecular architectures between PP-*g*-AA(T) and PE-*r*-AA, namely a graft copolymer versus a random copolymer. Recently, Kramer and co-workers [21,22] investigated the effect of the molecular architectures of poly(styrene-*co*-4-hydroxy styrene) copolymers (PSHS) on the interfacial strength of PS/poly(vinyl pyridine) blends. They showed that the maximum fracture toughness of an interface with a random-type PSHS is several times higher than that with a graft-type PSHS, even if the amount of the functional group (OH) in the former is smaller than that in the latter. This suggests that when the amount of a functional group is similar, the interfacial activity of the random-type copolymer is more efficient than that of the graft-type copolymer.

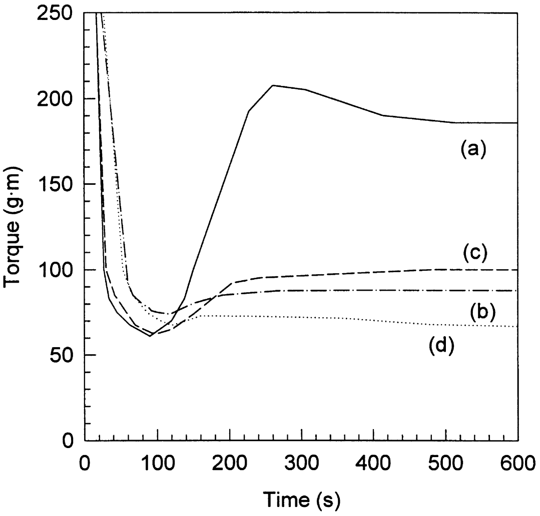

Fig. 7. Torque change with time at a temperature setting of 220°C: (a) 30/70 PP-*g*-AA(O)/PS-GMA; (b) 30/70 PP-*g*-AA(T)/PS-GMA; (c) 10/90 PP-*g*-AA(O)/PS-GMA; and (d) 10/90 PP-*g*-AA(T)/PS-GMA.

3.3. Rheological properties

Fig. 8(a) and Fig. 8(b) give plots of $\eta^*(\omega)$ versus ω at 220°C for 30/70 PP-*g*-AA(O)/PS and PP-*g*-AA(T)/PS blends with varying amounts of PS-GMA, respectively. It can be seen in Fig. 8(a) that neat PP-*g*-AA(T) has slightly

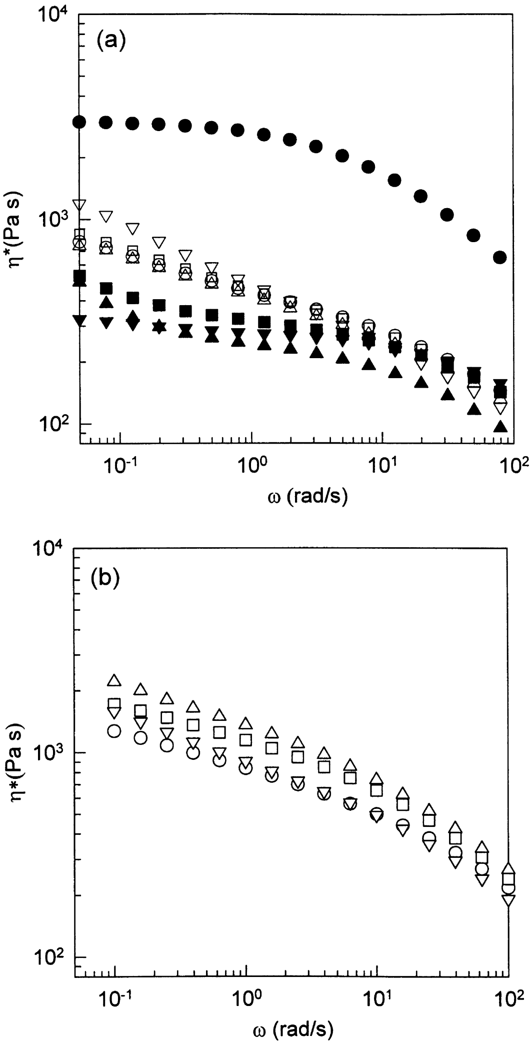

Fig. 8. The complex viscosity ($\eta^*(\omega)$) at 220°C as a function of ω for 30/70 (wt/wt) PP-*g*-AA(O)/PS blends (a) and PP-*g*-AA(T)/PS blends (b) with varying amounts of PS-GMA: ○, 0; □, 3; △, 7; and ▽, 15. Also, $\eta^*(\omega)$ for neat PS (●), PP-*g*-AA(T) (■), PP-*g*-AA(O) (▲), and PS-GMA (▼), are included in (a).

higher viscosity than neat PP-*g*-AA(O). This is due to the existence of the low molecular weight PAA homopolymer in PP-*g*-AA(O). For the PP-*g*-AA(O)/PS blend, $\eta^*(\omega)$ hardly changes with increase in the amount of PS-GMA up to 7 wt%, then rises at 15 wt% PS-GMA at low shear frequencies. The change in $\eta^*(\omega)$ with PS-GMA content resulted from the counter-balance between the lowering viscosity of the blend due to the low viscosity of PS-GMA and increasing viscosity due to the reaction between PAA and PS-GMA. Here, we consider that the major reaction would occur between PAA and PS-GMA, not PP-*g*-AA and PS-GMA. It should be mentioned that since the dispersed domain size does not change with the addition of PS-GMA, as shown in Figs 4 and 6, the effect of the morphological change on the $\eta^*(\omega)$ of PP-*g*-AA(O)/PS blend is negligible.

However, it can be seen in Fig. 8(b) that $\eta^*(\omega)$ of the 30/70 PP-*g*-AA(T)/PS blend increases with increase in the amount of PS-GMA up to 7 wt%. This is because the dispersed domain size becomes smaller with increase in the amount of PS-GMA, which results from graft copolymers formed in situ at the interface. However, when the amount of PS-GMA is further increased to 15 wt%, the viscosity begins to decrease. This is attributed to the fact that although the amount of graft copolymers formed in situ increases with increase in the amount of PS-GMA, the amount of the lower viscosity material PS-GMA is also increased. In other words, in determining $\eta^*(\omega)$ for 30/70 PP-*g*-AA(T)/PS blends, the role of PS-GMA as an in-situ compatibilizer, (the ability to enable the formation of in situ graft copolymers) dominates over the low viscosity properties of the PS-GMA homopolymer for blends with a PS-GMA content of less than 7 wt%, while for the PP-*g*-AA(T)/PS blends with 15 wt% PS-GMA, the low viscosity of the PS-GMA has the dominating effect on $\eta^*(\omega)$.

4. Concluding remarks

In this study, we have shown that with increase in the content of PS-GMA, the dispersed domain size of PP-*g*-AA with homopolymer PAA in a blend *does not* decrease even though the content of PS-GMA is as large as 25 wt%. Conversely, the dispersed domain size of PP-*g*-AA without PAA homopolymer in the blend *does* decrease gradually and reaches a steady value with increasing PS-GMA content. However, the reduction in the dispersed domain size (D_r) of PP-*g*-AA in a blend of PP-*g*-AA without PAA and PS in the presence of PS-GMA is at most 30%. The small reduction in the dispersed domain size may be due to the molecular architecture of graft copolymer PP-*g*-AA itself.

The change in rheological properties with the amount of PS-GMA for PP-*g*-AA(T)/PS blends was different from those for PP-*g*-AA(O)/PS blends, thus the existence of PAA in the blend significantly affects the rheological properties as well as the morphology of the blends.

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