

# Crystallization Behavior and Mechanical Properties of Polypropylene Random Copolymer/Poly(ethylene-octene) Blends

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**ABSTRACT:** New polymer blends of polypropylene random copolymer (PP-R) and poly(ethylene-octene) (POE) were prepared by melt-blending process using a corotating twin-screw extruder. The POE content was varied up to 35%. The toughening efficiency of POE for PP-R was evaluated by the mechanical properties of the resulted PP-R/POE blends. The crystallization behavior and morphology of the blends were also studied. Results show that POE acts as nucleation agent to induce the crystallization of PP-R matrix at higher crystallization temperature. Super-tough-

ened PP-R/POE blends (Izod impact strength more than 500 J/m) can be readily achieved with only 10 wt % of POE. The high toughness of PP-R/POE is attributed to cavitation and shear yielding of matrix PP-R, as revealed by the morphology studies. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 122: 461–468, 2011

**Key words:** polypropylene random copolymer; poly(ethylene-octene); polymer toughening; polymer blends

## INTRODUCTION

Melt blending of immiscible polymers has been a topic of great academic and industrial interest over the recent decades because of its versatility in preparing new multiphase polymeric materials with improved deficient properties of component polymers. Most commercial two-phase polymer blends have proven to afford large advantages over their individual polymers,<sup>1,2</sup> with the toughening of brittle plastics via blending with elastomer as one convincing example.<sup>3</sup> The toughening efficiency is highly dependent on the content, particle size, and architecture of elastomer, as well as the interactions between phases determining the mechanical properties of the polymer blends.<sup>4–8</sup>

Polypropylene (PP) is one of the most widely used engineering polymers because of its good mechanical and thermal properties, chemical resistance, and easy processing features.<sup>9</sup> However, its low fracture toughness at low temperature and, in particular, its high notch sensitivity at room temperature often limits its industrial applications. To address this issue, numerous researches have been conducted in prepar-

ing toughened PP blends over the last 20 years.<sup>10,11</sup> To date, elastomer-toughened PP blends are one of the most successful systems. Elastomers can be ethylene-propylene copolymer (EPR),<sup>12–15</sup> ethylene-propylene diene monomer (EPDM),<sup>16–19</sup> ethylene vinyl acetate,<sup>20–23</sup> styrene-*b*-butadiene-*b*-styrene (SBS), and styrene-*b*-hydrogenated butadiene-*b*-styrene triblock copolymers (SEBS).<sup>23,24</sup> More recently, poly(ethylene-octene) (POE) was used as the impact modifier for PP, and the results show that POE presented good toughening efficiency and better processability than EPDM in toughening PP.<sup>25–28</sup> Generally, elastomer-toughened PP blends achieved a great increase in impact strength and ductility, but a decrease in the elastic modulus and tensile strength.

As a new product of modified PP, polypropylene random copolymer (PP-R) has received a great deal of attention from academics and industry.<sup>29–31</sup> By copolymerization with propylene, ethylene was occasionally embedded into the long propylene sequences. Consequently, the crystallization of the propylene sequences is disrupted by the embedded ethylene units, leading to a decrease in total crystallinity, rigidity, and melting point of PP. As the copolymer is mainly composed of long propylene sequences and occasional ethylene units, PP-R has shown to have excellent thermal stability, aging resistance, and mechanical properties, making it attractive for piping systems for both domestic and

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industrial applications.<sup>32–34</sup> However, the impact-resistant properties of PP-R are still an issue in its application, especially at low service temperature. Surprisingly, there are only few researches to address this issue. Forte and coworkers<sup>24</sup> developed SBS- and SEBS-toughened PP-R blends, with the latter showing better toughening efficiency. Both SEBS and SBS can act as nucleating agent for matrix crystallization. The higher toughening efficiency was explained as the better dispersion of PS segment into PP matrix because of the miscibility of its EB segments in SEBS and the resulted smaller rubbery domains formed with lower coalescence levels.<sup>24</sup>

POE is characterized by a narrow molecular weight distribution and homogeneous octene distribution that exhibits the advantage of good processability and compatibility with PP.<sup>25–27</sup> When compared with conventional EPR or EPDM, POE exhibits the advantage of mechanical properties when blended with PP.<sup>25–28</sup> In the previous stage of our project, we have reported our efforts in preparing Ethylene Styrene Interpolymer (ESI)-compatibilized PS/PE blends,<sup>35,36</sup> SEBS- and SEBS-*g*-MAH-compatibilized Polyphenylene sulfide (PPS)/PA66 blends,<sup>37</sup> and ESI-toughened PP-R/ESI blends.<sup>38</sup> The possible consequences of blending PP-R matrix with certain amount of POE are intriguing to us and thus motivated this study. In this article, POE-toughened PP-R blends were prepared by varied content of POE. The toughening efficiency of POE was evaluated by mechanic properties of the resulted blends. In addition, the crystallization and fracture morphology of the prepared blends were also investigated.

## EXPERIMENTAL

### Materials

PP-R (C4420) was supplied by Yanshan Petrochemical Co. (Beijing, China) and has a melt-flow index (MFI) of 0.3 g/10 min (at 230°C under 2.16 kg load). The concentration of ethylene is 3 wt %. POE (Engage 8150) was procured from DuPont Dow Elastomers (Wilmington/Delaware, USA), and it has an octene content of 25 wt % and MFI of 0.5 g/min (at 230°C under 2.16 kg load).

### Sample preparation

The pellet polymers were dried at 80°C overnight in a vacuum oven. Melt blending was performed using a corotating twin-screw extruder (SLF-35B, L/D = 30; Keqiang Polymer Engineering Company, Sichuan, China) with a rotation speed of 200 rpm. The temperature along the barrel was increased from 180 to 210°C. The blends were cooled down with water bath before they were pelletized. The obtained blend

pellets were dried again at 80°C in a vacuum oven before the injection molding. The weight ratios of PP-R/POE were 100/0, 95/5, 90/10, 85/15, 80/20, 75/25, 70/30, and 65/35. Dumbbell tensile bars and notched impact specimens were molded using an injection-molding machine (SZ-160/80 NB, China). The cylinder temperature and molding temperature during molding injection were 200°C and 80°C, respectively.

### Thermal analysis

The thermal behavior of PP-R and PP-R/POE blends was analyzed using a Perkin-Elmer Differential scanning calorimetry (DSC) Pyris 1 in nitrogen atmosphere. The samples were heated from room temperature to 200°C, held for 3 min, and then cooled to room temperature. They were reheated to 200°C at the same heating rate. Crystallization and melting temperatures ( $T_c$  and  $T_m$ ) and melting enthalpy ( $\Delta H_m$ ) were taken from the second and third run curves, respectively. Sample crystallinity was calculated by taking PP fusion enthalpy 190 J/g as the reference value.<sup>24</sup>

### Mechanical property tests

The tensile properties were evaluated using an Instron tensile tester (model 3211) according to ASTM D 638. The testing speed was 10 mm/min. The notched Izod and Charpy impact strengths were determined with a pendulum impact testing machine (XJ-40A; Wuzhong Material Testing Machine Company, Hebei, China) according to ASTM D 256. Five blend specimens were tested for each composition of blends, and the mean value and standard deviation were calculated. All mechanical property tests were performed at 23°C  $\pm$  1°C.

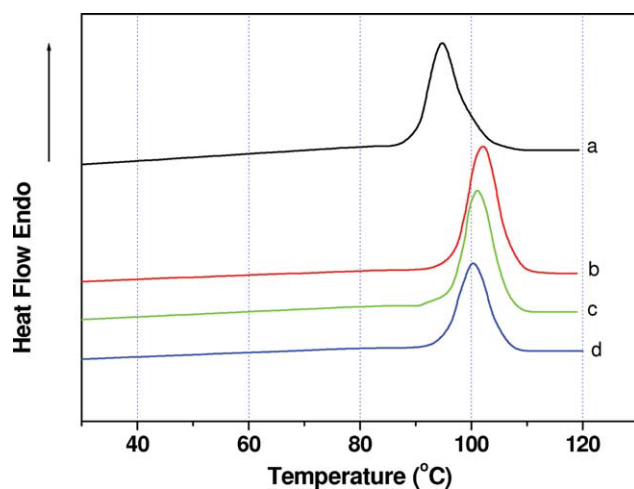
### Morphological observations

The morphological characteristics of the blends were examined by Scanning electron microscope (SEM) (Cambridge Instrument Co., Cambridge, UK). Samples were freeze fractured in liquid nitrogen. The cryogenically fractured surfaces were coated with a thin layer of gold to increase the contrast between the matrix and the dispersed phase in morphology study. In addition, fractured surfaces of PP-R/POE blends near the notch were directly sputter coated with a thin layer of gold for SEM observation.

## RESULTS AND DISCUSSION

### Thermal behaviors

Figure 1 presents the crystallization peaks of exothermic DSC curves for PP-R and its blends with POE blends. In general, the crystallization peaks



**Figure 1** Crystallization peak of exothermic DSC peak curve of (a) pure PP-R, (b) PP-R/POE 90/10, (c) PP-R/POE 80/20, and (d) PP-R/POE 70/30 blends. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

of all polymer blends were displaced to higher temperatures when compared with pure PP-R, indicating that the POEs act as nucleation agent for PP-R. The highest crystallization temperature ( $T_c$ ), measured as the apex of the crystallization peak, was found in the blends with 5 wt % addition of POE. PP-R blends with higher content of POE (e.g., 15 or 30 wt %) demonstrate an exothermic peak falling between that of PP-R and its blends with 5 wt % POE. Similar crystallization behavior was also observed for PP/SBS, PP/SEBS, and PP-R/ESI blends.<sup>38,39</sup> These results may be explained as the PP-R molecular chains aggregated to the long POE molecular chains and crystallized. With POE content increase in certain blends compositions, dispersed POE particles aggregated to form continuous zone and the crystallization of the blend tended to bulk crystallization of POE; thus, the crystallization of PP-R was hindered with  $T_c$  decreasing again. It is noteworthy that PP-R/POE blends exhibit slightly lower crystallization temperature ( $T_c$ ) than PP-R/ESI blends with the same blend composition.

Table I summarizes the  $T_m$ ,  $T_c$ ,  $\Delta H_m$ , and crystallinity values for all PP-R/POE blends. PP-R showed lower melting temperature (142°C) than *i*-PP (163°C) because of the ethylene insertion in the PP chain, which disrupted the isotactic sequences, decreased the size of lamella, and introduced defects in the crystallite.<sup>24</sup> In general, PP-R/POE blends presented lower  $T_m$  than pure PP-R. For the fusion enthalpy, there was an increase in  $\Delta H_m$  up to 30 wt % POE, mainly due to the high affinity of POE with PP-R matrix. PP-R matrix presents higher crystallinity with the addition of POE, and this crystallinity decreases with increasing POE content in the range of 5–30 wt % (Table I). In comparison, PP-R/POE

blends exhibit lower crystallinity in comparison with PP-R/ESI blends with same elastomer content.<sup>38</sup>

The influence of POE as a nucleation agent can be related to its molecular structure. POE is a copolymer of octene with 75 wt % ethylene. Both POE ( $T_g \sim -52^\circ\text{C}$ ) and PP ( $T_g \sim -15^\circ\text{C}$ ) possess a  $T_g$  below ambient temperature, the flexible polyolefin blocks (octene segments) of POE show high affinity with the macromolecule chains of PP-R, thus POE achieves good dispersion in PP-R matrix. When the certain octene segments move into PP-R macromolecules, they may aggregate to form micelles, which act as nuclei for the PP-R macromolecule segments to crystallize at high temperature than usual. However, with increase in the POE content, the octene segments could hinder the access of PP-R segments to the growth nucleus, resulting in a less crystalline matrix.

### PP-R/POE fractography

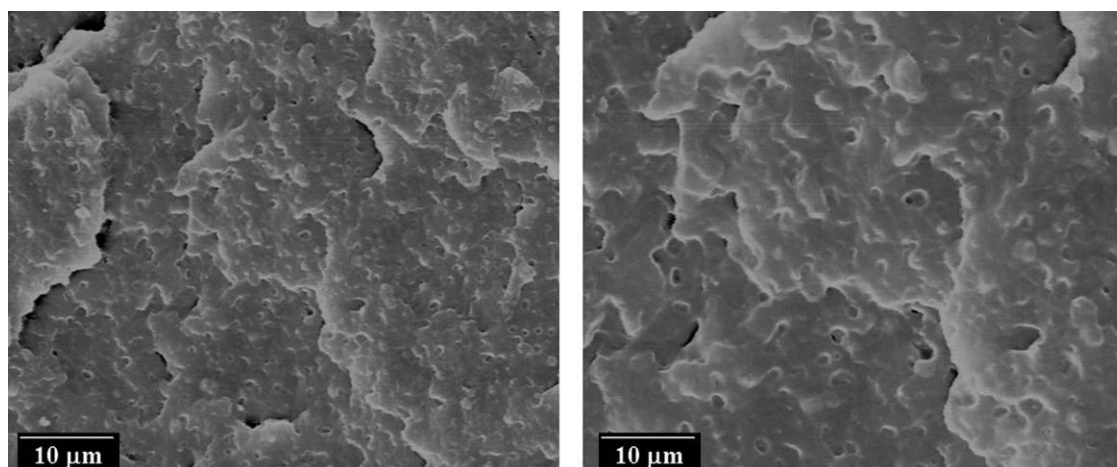
Many toughening researches have focused on the brittle–ductile transition (BDT) of PP and nylon. Three factors are reported to account for BDT, namely, inter-particle distance,<sup>4,5</sup> temperature,<sup>18,19</sup> and strain rate.<sup>11,40</sup> Impact and high-speed tensile test of PP/POE blends showed that notched impact deformation was actually high-speed tensile deformation near notch tip.<sup>40</sup> The formation and breakup of droplets or domains in viscoelastic fluids depend on the molecular weight and viscosity of the components, the viscosity ratio, the interfacial tension, and the blend composition.<sup>41,42</sup> Like other rubber-modified plastics, PP-R/POE blends separate into two distinct phases. The size, shape, and distribution of elastomer droplet were dependent on the elastomer (ESI<sup>38</sup> or POE) used, resulting in different morphologies.

As shown in Figure 2, typical droplet–matrix micrographs were observed from cryogenically fractured surfaces of PP-R/POE blends (80/20) after impact tests. The POE droplets were smaller than those of ESI in PP-R matrix, also with lower droplet coalescence levels.<sup>38</sup> It is already reported that average particle size of elastomer increases with increasing its content, attributed to rubber droplet coalescence.<sup>43</sup> The average diameter of POE particles in PP-R/POE blends

**TABLE I**  
Melting ( $T_m$ ) and Crystallization ( $T_c$ ) Temperatures, Melting Enthalpy ( $\Delta H_m$ ), and Crystallinity of PP-R/POE Blends

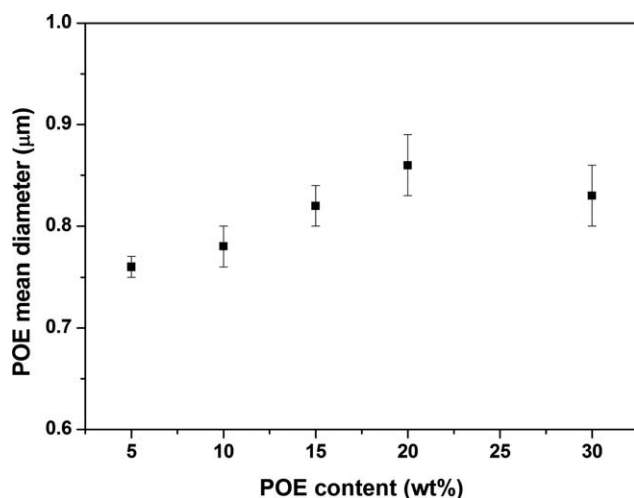
PP-R/POE	$T_c$ (°C)	$T_m$ (°C)	$\Delta H_m$ (J/g)	Crystallinity (wt %)
100/0	94.9	142.9	68.6	36
95/5	102.1	142.8	79.8	45
90/10	101.6	141.6	76.3	42
85/15	101.2	140.9	73.2	40
80/20	100.8	140.8	70.6	38
70/30	100.4	140.3	69.5	36





**Figure 2** SEM micrographs of cryogenically fractured surfaces for (A) PP-R/POE 90/10 blend and (B) PP-R/POE 80/20 blend.

(80/20) was  $\sim 0.8 \mu\text{m}$ . When the POE content was further increased up to 30%, the POE domains increased dramatically in size and presented a number of irregular shapes. This behavior is attributed to the coalescence of POE droplets. Because of increased coalescence of POE droplets at higher addition of POE, the POE domains increased accordingly. This evolution of rubber phase in size and shape is also observed in EPR-toughened PP blends.<sup>15</sup> Figure 3 depicts the mean diameter values of POE phase for PP-R/POE blends at varied POE. Because of the occurrence of POE droplet coalescence at POE content more than 20%, their droplet sizes are more than one time larger than those in PP-R/POE blends with POE content below 20%. The POE droplet size of PP-R/POE (POE% below 20%) blends was in the range of 0.7–0.9  $\mu\text{m}$ . The average size of POE droplets was much smaller than that of ESI droplets (1–3  $\mu\text{m}$ ) in PP-R/ESI blends.<sup>38</sup> This phenomenon can be explained by



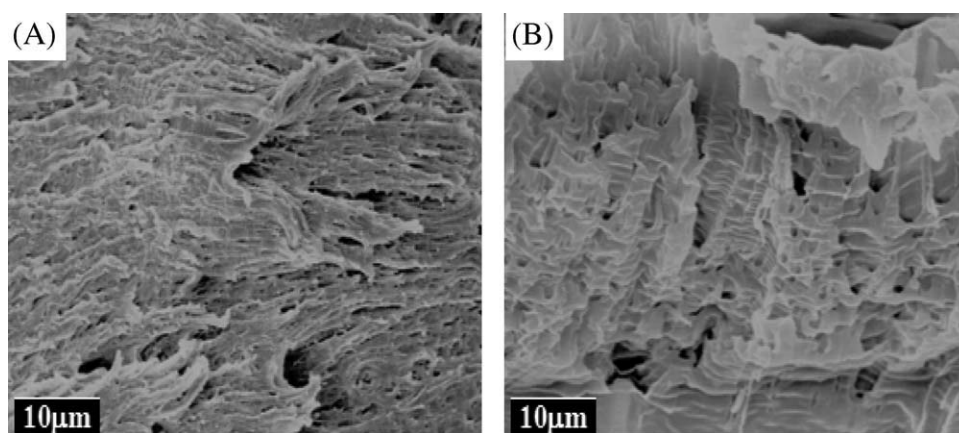
**Figure 3** Variation of mean diameter of POE droplets ( $\mu\text{m}$ ) in PP-R/POE blends.

the higher affinity of POE with PP-R matrix, leading to a better dispersion of POE in PP-R matrix during melt processing.

When directly picturing the impact-fractured surfaces of PP-R/POE blends, one can observe a typical SEM micrograph as shown in Figure 4(A), which displays two regions: slow growth region initiated at the notch root, indicated as void zone, and the subsequent fast crack growth region, indicated as fibrillar zone [Fig. 4(B)]. Extensive fibrillation of the PP-R matrix can be observed to occur in the fast crack growth region. Moreover, a few small cavities (dimension of 1–5  $\mu\text{m}$ ) can also be observed in the fractograph. Such cavitation originates from shedding of POE particles from the PP-R matrix during impact deformation. As the size of the dispersed POE particles is below 1  $\mu\text{m}$ , the larger voids observed in this fractograph should be attributed to the coalescence of individual voids initiated by the dispersed POE particles. Furthermore, ductile tearing of the ligaments between particles can lead to the formation of fibrils once the voids are initiated. As shear yielding of the ligaments between particles dissipates a large amount of the energy, the PP-R/POE 80/20 blend exhibits excellent impact toughness. This phenomenon is analogous to the shear yielding of thermoplastic matrix induced by fine rubber particles during impact deformation.<sup>44,45</sup>

### Mechanical properties

The impact property of materials is closely related with their morphology, crystallinity, and fractography. In general, a suitable morphology with smaller, well-dispersed rubber domains is desirable to yield toughened polymer blends. The results of Izod impact strength, tensile strength, elongation at break, and elastic modulus for PP-R/POE blends are



**Figure 4** SEM micrographs of notch tip of the impact-fractured surface of PP-R/POE 80/20 blends: (A) overview and detail fibrillar features are shown in (B).

summarized in Table II. For POE-toughened PP-R blends, it is necessary to correlate the toughness with the matrix crystallinity and POE domain size when increasing POE content. As effective nucleation agents, POE-toughened PP-R blends enjoyed higher crystallinity than pristine PP-R matrix. Meanwhile, the average size of POE droplets increases with its content and reach its maximum at 20 wt % addition when no droplet coalescence occurs (Fig. 3). Therefore, the impact strength of PP-R/POE blends improved gradually with the increase in the POE content. The super-toughened PP-R/POE blends (notched Izod impact strength >500 J/m) can be easily obtained with 10 wt % POE. Nearly 2.4-fold increment in impact strength of PP-R (from 180 to 606 J/m) was achieved with 20 wt % POE. With a close examination at the variation of Charpy impact strength with POE content (Fig. 5), we found that the Charpy impact strength of PP-R/POE blends enjoyed significant improvement with POE content before reaching 10%. Afterward, this increment slowed down and the Charpy impact strength of PP-R/POE blends even leveled off after 25% addition of POE. This behavior may be explained by the change of POE particle size and POE role in toughening

PP-R. When increasing POE content up to 20%, the average size of rubber domain increases dramatically due to the coalescence of rubber droplets. The larger the addition of POE, the more frequent coalescence of rubber droplets occurs. When subject to external force, the rubber domains form cavitations to absorb impact deformation energy and cause shear yielding of matrix. After the addition of up to 20% of POE, the impact energy absorbed by rubber domains deformation is not increased proportionally with its increment in content because of the coalescence, as more addition of POE results in larger rubber domain size and distribution. The net result of POE addition is a slow-down enhancement in impact strength for POE-toughened blends.

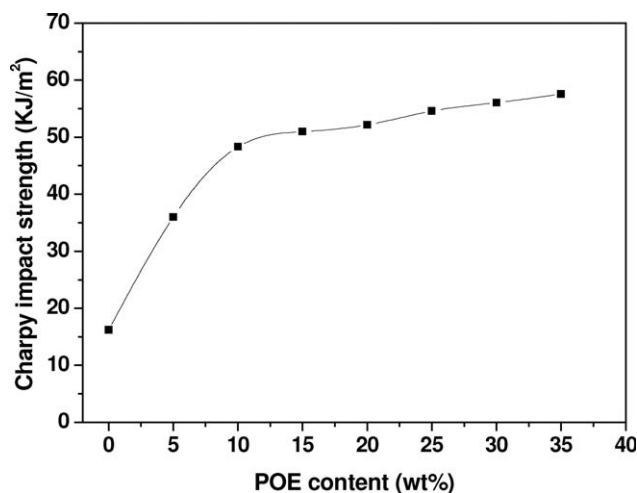
For the ductility of PP-R/POE blends, it is noted that the elongation at break increased with the increment in POE content. However, because of higher affinity of POE with matrix, POE-toughened blends presented higher ductility than PP-R/ESI blends.<sup>38</sup>

As for the stiffness of PP-R/POE blends, both tensile strength and elastic modulus were observed to decrease with increasing POE content in comparison with PP-R matrix. However, with a careful inspection of the tensile strength data, one can observe

**TABLE II**  
Izod Impact Strength, Tensile Strength, Elongation at Break, and Elastic Modulus of PP-R/POE Blends at 23°C

PP-R/POE	Izod impact strength (J/m)	Tensile strength (MPa)	Elongation at break (%)	Elastic modulus (MPa)
100/0	180	28.7	422	354.3
0/100 <sup>26</sup>	–	2.3	1953	–
95/5	421.6	27.4	721	291.3
90/10	525	26.1	848	278.6
85/15	586.7	24.8	906	263.1
80/20	606.2	23.6	973	251.8
75/25	610.9	21.3	1169	235.0
70/30	614.5	18.1	1280	214.7
65/35	628.5	16.5	No break	203.9

Mechanical property data for POE were obtained from Ref. 26.



**Figure 5** Variation of Charpy impact strength of PP-R/POE blends with increasing content of POE.

that the tensile strength only dropped 18% for PP-R/POE system with the addition of 20 wt % POE, whereas the elastic modulus only suffered 29% loss in the same blend.

Figure 6 depicts the stress–strain curves of neat PP-R and PP-R/POE blends. A typical ductile plastic fracture behavior with a yield stress and following large strain was observed in all curves. However, the incorporation of POE significantly changed the nature of the curves. It is noted that the yield stress decreased continuously with the increase in the content of POE, whereas the strain increased dramatically with POE content. It is obvious that strong interfacial affinity was achieved between the matrix PP-R and POE. The reduction in yield stress and increase in strain with increasing POE content was also observed for PP-R/ESI blends,<sup>38</sup> similarly with PP-R/SEBS and PP-R/SBS blends.<sup>24</sup> When compared with PP-R/ESI blends,<sup>38</sup> because of better affinity of POE with PP-R, the PP-R/POE blends present higher strain than PP-R-ESI blends with the same content of elastomer.

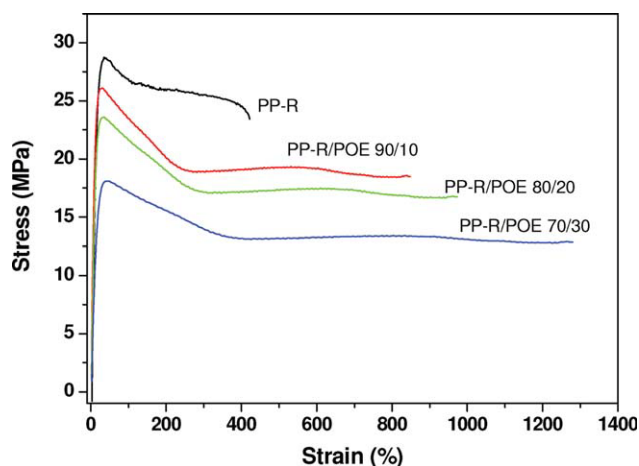
Taking into account the toughness (impact strength), ductility (elongation at break), and stiffness (tensile strength and elastic modulus) of PP-R/POE blends, toughness and ductility were drastically improved with the addition of POE. However, the stiffness decreased slowly with the increase in POE content. Overall, POE-toughened PP-R blends exhibited much improved toughness than pristine PP-R matrix, which may promote PP-R into more applications at room temperature.

### Rheology

As discussed above, the addition of POE (especially up to 10%) results in significant improvement in toughness of PP-R. Meanwhile, the introduced rub-

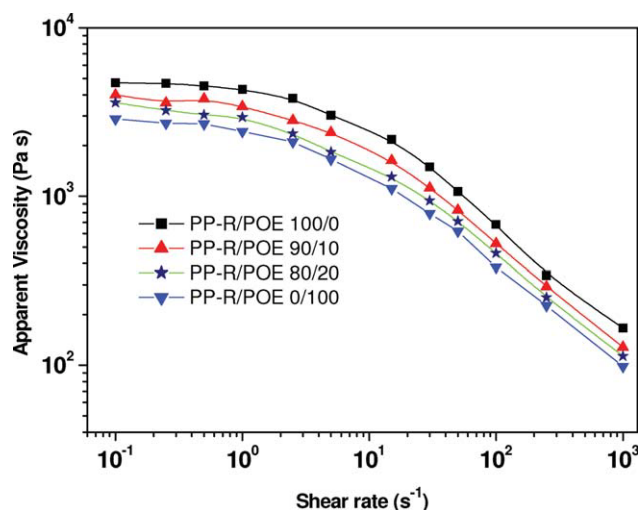
ber phase will affect not only the mechanical properties but also the rheological properties of the matrix resin. The rheological properties of polymers/blends provide crucial guidance in optimizing the processing conditions.<sup>46–48</sup> For the neat PP-R matrix resin, its melt viscosity displays the shear-thinning behaviors in the processing temperature ranging from 200 to 230°C,<sup>38</sup> i.e., the viscosity of PP-R melts gradually decreased when increasing the melt-processing temperature. Interestingly, at processing temperature higher than 200°C, the viscosity of PP-R melt decreased slowly with increased shear rate (ca.  $>50$  s<sup>-1</sup>), i.e., PP-R melt viscosity is less sensitive to shear rate than common polymers at processing temperature above 200°C. This behavior offers us options to achieve low viscosity of PP-R melt by selecting suitable processing temperature and shear rate: either increasing shear rate up to 50 s<sup>-1</sup> at processing temperature below 200°C or increasing processing temperature at shear rate below 50 s<sup>-1</sup>.<sup>38</sup>

With the addition of POE, the viscosities of PP-R/POE blends lie between the value of pure PP-R and POE (Fig. 7). As indicated from their MFI values, the rubber phase of POE presented much lower complex viscosity than PP-R matrix at all shear rates. Moreover, the viscosity of the blends decreases when increasing the POE content in the blend. This viscosity decrease behavior may be explained by emulsion character of polymer blends in terms of effects of rheological properties of the blend components and concentration of the dispersed rubber phase. As mentioned, POE has high affinity with PP-R matrix because of the compatibility of its ethylene segments with PP matrix, thus, the droplet–matrix microstructure morphology can be stabilized in the blend. The emulsion effect at the interface increases with increasing POE concentration. As a result, the higher



**Figure 6** Stress–strain curves of pure PP-R and PP-R/POE blends with varied concentration of POE. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]





**Figure 7** Plots of apparent viscosity versus shear rate for PP-R/POE blends. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

the content of POE in the blends, the higher viscosity the blends present. It should be mentioned that all blends possess lower viscosities than that of PP-R matrix. The decrease in viscosity is due to the disentanglement caused particularly by the ethylene segments in POE. On the other hand, the blends' viscosity decreases with increasing shear rate. This behavior is attributed to the disentanglements occurring at the interface between PP-R and POE when increasing the shear rate (ref. to Figure 7).

## CONCLUSIONS

POE-toughened PP-R blends were prepared by melt extrusion. The crystallization, fracture morphology, and mechanical properties of PP-R/POE were investigated. Crystallization results showed that POE behaved as a nucleation agent in the crystallization of PP-R, resulting in higher  $T_c$ . PP-R/POE blends exhibited significant enhancement in toughness and ductility, but slow decrease in stiffness such as tensile strength and elastic modulus. Impact measurements indicated that the impact strength of the blends increases significantly with POE content up to 10 wt % to achieve super-toughened polymer blends. Tensile tests showed that the yield stress of the PP-R/POE decreases substantially but the elongation at break appeared to increase dramatically with increasing POE content. SEM observations revealed that the improved impact strength of PP-R/POE blends is attributed to cavitation and shear yielding of matrix PP-R.

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