Synergetic Toughness and Morphology of Poly(propylene)/Nylon 11/Maleated Ethylene-Propylene Diene Copolymer Blends

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ABSTRACT: Polypropylene (PP)/nylon 11/maleated ethylene-propylene-diene rubber (EPDM-g-MAH) ternary polymer blends were prepared via melt blending in a corotating twin-screw extruder. The effect of nylon 11 and EPDM-g-MAH on the phase morphology and mechanical properties was investigated. Scanning electron microscopy observation revealed that there was apparent phase separation for PP/EPDM-g-MAH binary blends at the level of 10 wt % maleated elastomer. For the PP/nylon 11/EPDM-g-MAH ternary blends, the dispersed phase morphology of the maleated elastomer was hardly affected by the addition of nylon 11, whereas the reduced dispersed phase domains of nylon 11 were observed with the increasing maleated elastomer loading. Furthermore, a core-shell structure, in which nylon 11 as a rigid core was surrounded by a soft EPDM-g-MAH shell, was formed in the case of 10 wt % nylon 11 and higher EPDM-g-MAH concentration. In general, the results of mechanical property measurement showed that the ternary blends exhibited inferior tensile strength in comparison with the PP matrix, but superior toughness. Especially low-temperature impact strength was obtained. The toughening mechanism was discussed with reference to the phase morphology. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1344–1350, 2008

Key words: PP; ternary polymer blends; morphology; toughness

INTRODUCTION

Polypropylene (PP) is one of the most important commodity polymers and is widely used in automobiles, household appliances, and the construction industry because of its easy processability, low cost, and well-balanced physical and mechanical properties. Despite these advantages, the application of PP has one major drawback: its poor impact resistance, especially at low temperature. To improve the impact toughness of PP and to extend its application range, numerous and thorough researches on the toughening of PP have been performed. These traditional PP-toughening approaches included blending the PP matrix with elastomers,[1–17] rigid particles,[18–21] rigid polymer,[22–30] or combination of elastomers with rigid particles[31–35] or rigid polymer.[36–38] In the pursuit of the optimum balance of the stiffness and toughness, extensive attention was drawn to the PP/elastomer/rigid particle or PP/elastomer/rigid polymer ternary blends over the past few years. As demonstrated in the previous publications,[39–46] three distinct types of phase morphology were obtained in the ternary blends system: (a) elastomer and rigid fillers were independently dispersed in the PP matrix, (b) elastomer encapsulated fillers to form a core-shell inclusion structure, and (c) mixed phases of two minor components were formed without any ordered organization. The type of morphology and the size of dispersed phases, which were the important factors that determined the mechanical properties of polymeric blend, can be affected by composition, melt viscosity of the components, interfacial interaction, and processing parameters. For a certain PP/elastomer/filler system, a separated microstructure increased stiffness, whereas a core-shell microstructure improved toughness.[25]

For interpretation of the toughening mechanism, various conceptual models, including crazing, cavitation, and shear yielding, have been proposed and worked out during the last two decades.[47–50] On the basis of the experimental investigation of nylon-6/EPDM blends, Wu[51–53] proposed and demonstrated the "critical matrix ligament thickness" theory. The

theory proposed that the toughness of such systems depended neither on the rubber inclusion size nor on the rubber concentration alone but instead correlated with the thickness of the matrix ligament between rubber particles. The theory also stated that a critical surface-to-surface interparticle distance (ID), which was independent of the rubber volume fraction and particle size and was the property of the matrix alone for a given mode, temperature, and rate of deformation, was the key parameter determining whether a blend was brittle or tough. If the average matrix-ligament thickness (MLT) is lower than ID, the matrix yielding would propagate and pervade over the entire matrix, and then the blend would be toughened. On the contrary, if MLT is greater than ID, the matrix yielding could not propagate, and the blend would fail in a brittle manner. A physical explanation for Wu's theory was provided by Muratoglu et al.[54–56] They proposed that the incoherent particle-matrix interfaces stimulated a preferential form of crystallization over a definite distance around the particles with the lowest energy surfaces of crystalline lamellae also representing the crystallographic planes of lowest plastic resistance lying parallel to the interfaces. Argon and coworkers[57–59] further extended the criterion of interparticle distance beyond polymer/rubber toughening, proving that this property is intrinsic in each polymeric material and that instead of rubber, rigid mineral fillers like $CaCO_3$ can be used for semicrystalline polymer toughening. This indicated that regardless of whether the material introduced into the polymer matrix was rubber or mineral filler, tough materials were produced if the above criterion was satisfied. This model can be used for the prediction of the supertoughening behavior of semicrystalline polymers modified not only with rubber particles but also with rigid fillers.

In recent decades, extensive interests have been focused on the use of functionalized rubbers as both toughners and compatibilizers for PP/PA6 blends. A core-shell morphology occurred[37,38] in PP/nylon 6 blends with a continuous PP matrix, wherein nylon formed the rigid core encapsulated by an elastomeric shell. However, no published research has addressed toughening in PP/nylon 11/EPDM-g-MAH ternary blends. Of the possible rubbers, EPDM-g-MAH might serve as both a coupling agent and an impact modifier arising from its elastomeric segments. Additionally, it is believed that the blending of nylon 11 into PP can improve the low-temperature resistance of PP matrix because nylon11 has excellent cold-tolerance. In our previous work,[60] we reported the melting behavior, nonisothermal crystallization kinetics, and morphology of PP/nylon 11/EPDM-g-MAH ternary blends. In this article, the phase morphology of PP/nylon 11/EPDM-g-MAH ternary blends in the case of various nylon 11 or elastomer content was investigated by scanning electron microscopy (SEM). The effect of nylon 11 or elastomer content on the mechanical properties is also discussed, and the toughness is related to the phase morphology.

EXPERIMENTAL

Materials

Nylon 11 was manufactured in our laboratory with weight-average ($\overline{M}_w$) and number-average ($\overline{M}_n$) molecular weights of 19.8×10^4 and 4.0×10^4 g/mol, respectively. PP (T30S, melt index 3.0 g/10 min) and EPDM-g-MAH with 1% MAH was supplied by Sinopec Qilu Company (Jinan, Shandong, China) and Shanghai Sunny New Technology Development (Shanghai, China), respectively. Nylon 11 and EPDM-g-MAH were dried at 80°C in vacuum for at least 12 h before use.

Preparation of blends

Melt blends were obtained by using a TSSJ-25 ∼ 33 twin-screw extruder (Nanjing, Jiangsu, China). The three different polymer chips or powder were fed together under the screw speed at 80 rpm, and the extruding temperature at various zones was between 185 and 205°C. The extrudate passed through a cooling water bath and was palletized. It was dried in vacuum for 24 h at 100°C. For comparison, pure PP was also extruded under the conditions listed earlier.

Scanning electron microscopy

The electron microscope (Hitachi S530, Tokyo, Japan) was employed to examine the morphology of the blends. The fractured surface was previously etched for 24 h with n-heptane (a good solvent for EPDM-g-MAH but not for nylon 11) or with m-cresol (a good solvent for nylon 11 but not for EPDM-g-MAH), and then coated with a thin layer of gold.

Measurement of mechanical properties

The tensile properties were determined at room temperature using an Instron Universal Testing Machine (Model 1130) at a crosshead speed of 50 mm/min according to ASTM D 638. The notched Izod impact strength was measured with a SUMITOMO impact tester according to the ASTM D 256. Five measurements were carried out to obtain each data point.

RESULTS AND DISCUSSION

Morphological microstructure of EPDM-g-MAH in the PP/EPDM-g-MAH binary blends

Figure 1 shows the impact-fractured SEM micrographs of PP/EPDM-g-MAH blends after erosion of
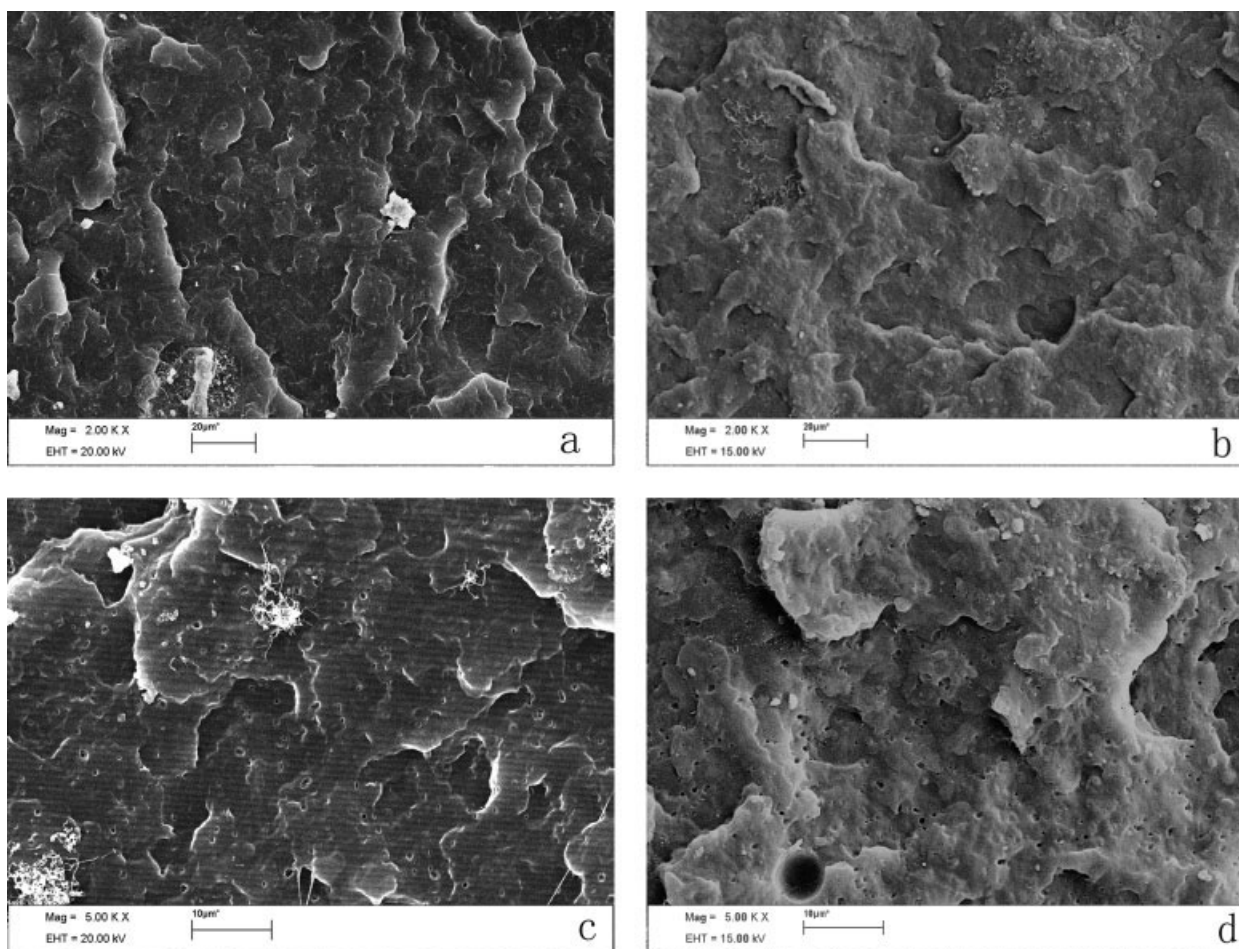
Figure 1 SEM micrographs of impact-fractured surfaces of PP/EPDM-*g*-MAH binary blends with various EPDM-*g*-MAH concentration: (a) 97/3; (b) 95/5; (c) 92/8; (d) 90/10 (etched by *n*-heptane).

EPDM-*g*-MAH. The black voids visible in these micrographs were due to the EPDM-*g*-MAH droplets that were etched by *n*-heptane. As the nonpolar olefinic rubber within the EPDM-*g*-MAH had good compatibility with PP matrix, the dispersed EPDM-*g*-MAH phase was hardly distinguished from the PP matrix at low EPDM-*g*-MAH content, as shown in Figure 1(a,b). When the content of EPDM-*g*-MAH was increased up to 8 wt %, a fine EPDM-*g*-MAH dispersion appeared [shown in Fig. 1(c)]. Furthermore, there was a clear phase separation in the case of 10 wt % EPDM-*g*-MAH, which may attribute to the increasing polarity (including the polar-grafted anhydride group and its density).

face of these blends was etched by *n*-heptane. The black holes visible in these micrographs were thus ascribed to EPDM-*g*-MAH droplets. As shown in the micrographs, the morphological structure of EPDM-*g*-MAH dispersion seemed to be not affected by the variation of nylon 11 content. It exhibited typical sea-island-type structure, and the dispersed EPDM-*g*-MAH domain size had no evident change in comparison with that of PP/EPDM-*g*-MAH (92/8, w/w) binary blends. It can be inferred that the reaction between anhydride group in EPDM-*g*-MAH and terminal amine group in nylon 11 had no significant influence on the compatibility between EPDM-*g*-MAH and PP.

Morphological microstructure of EPDM-*g*-MAH in the PP/nylon 11/EPDM-*g*-MAH ternary blends

Figure 2 illustrates the impact-fractured SEM micrographs of PP/nylon 11/EPDM-*g*-MAH ternary blends, in which the EPDM-*g*-MAH concentration was fixed at 8 wt % whereas the nylon 11 content ranged from 2 to 10 wt %. The impact-fractured sur-

Morphological microstructure of nylon 11 in the PP/nylon 11/EPDM-*g*-MAH ternary blends

Figure 3 presents the effect of EPDM-*g*-MAH concentration on the morphology of PP/nylon 11/EPDM-*g*-MAH blends. The impact-fractured surface of these blends was etched by *m*-cresol. The black holes visible in these micrographs were thus
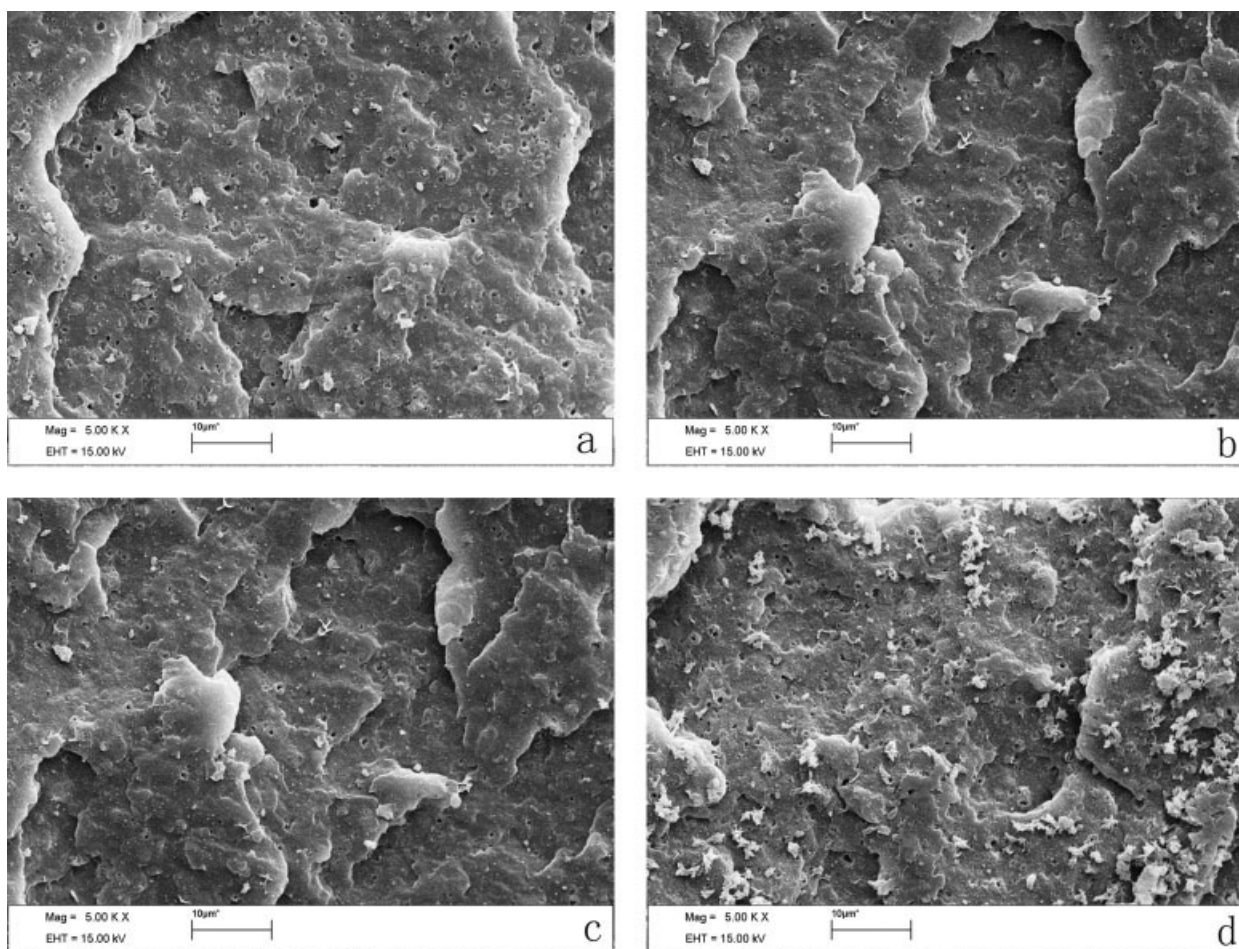
Figure 2 SEM micrographs of impact-fractured surfaces of PP/PA11/E PDM-*g*-MAH blends with various nylon 11 content: (a) 2%; (b) 5%; (c) 8%; (d) 10% (PDM-*g*-MAH was etched by *n*-heptane).

ascribed to nylon 11 droplets. It was found from Figure 3(a) that there was a distinct interface between PP and nylon 11 for the PP/nylon 11 binary blend, and the dispersed nylon 11 phase particles were large and nonuniform because of the inherent immiscibility. In comparison with that in the binary blends without EPDM-*g*-MAH, the size of the dispersed nylon 11 phase domain drastically decreased even in the case of the lower EPDM-*g*-MAH loading (5 wt %). A further increase of the EPDM-*g*-MAH concentration reduced the dispersed nylon 11 particles size to a greater extent. More of greater interest, a core-shell morphology containing nylon 11 cores (black hole) encapsulated by EDPM-*g*-MAH (brighter interfacial layer) was formed. A similar core-shell structure was also observed in PP/PA6/elastomer blends.[18,37,38,61,62] As reported in our previous studies,[63,64] the anhydride units of EPDM-*g*-MAH could react readily with the terminal amine groups of nylon 11 to form block or graft copolymers, whereas EPDM-*g*-MAH had a good compatibility with PP. EPDM-*g*-MAH had a Young's modulus of only 4 MPa,[37] therefore it formed very

soft interlayers around the rigid nylon 11 cores during extrusion. Moreover, the thickness of interfacial layer and degree of encapsulation of dispersed nylon 11 particles seemed to increase with the increasing EPDM-*g*-MAH concentration.

Therefore, the multiphase morphologies for the PP/nylon 11/EPDM-*g*-MAH ternary blends are outlined schematically in Figure 4. The systems with low EPDM-*g*-MAH concentration formed two separate dispersed phases in the PP matrix [as shown in Fig. 4(a)], whereas the blends with high EPDM-*g*-MAH concentration formed complex dispersed phases [as shown in Fig. 4(b)], including separate dispersed phases and core-shell structures.
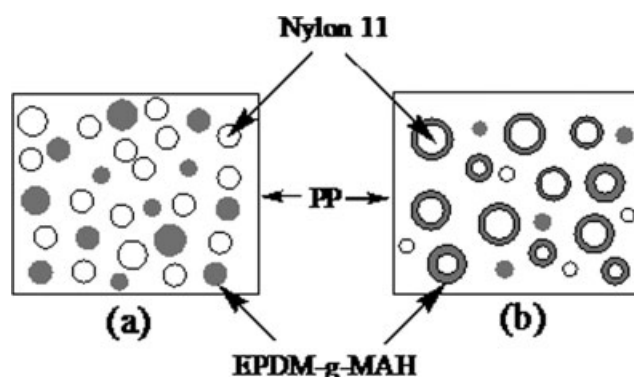
Mechanical properties

The data from tensile and Izod impact testing are summarized in Table I. The mechanical performance data measured for the blends were compared with those measured for the pure PP matrix. It was worth noting that the Izod impact strength at low temperature (LT, −40°C) for the pure PP matrix was too low
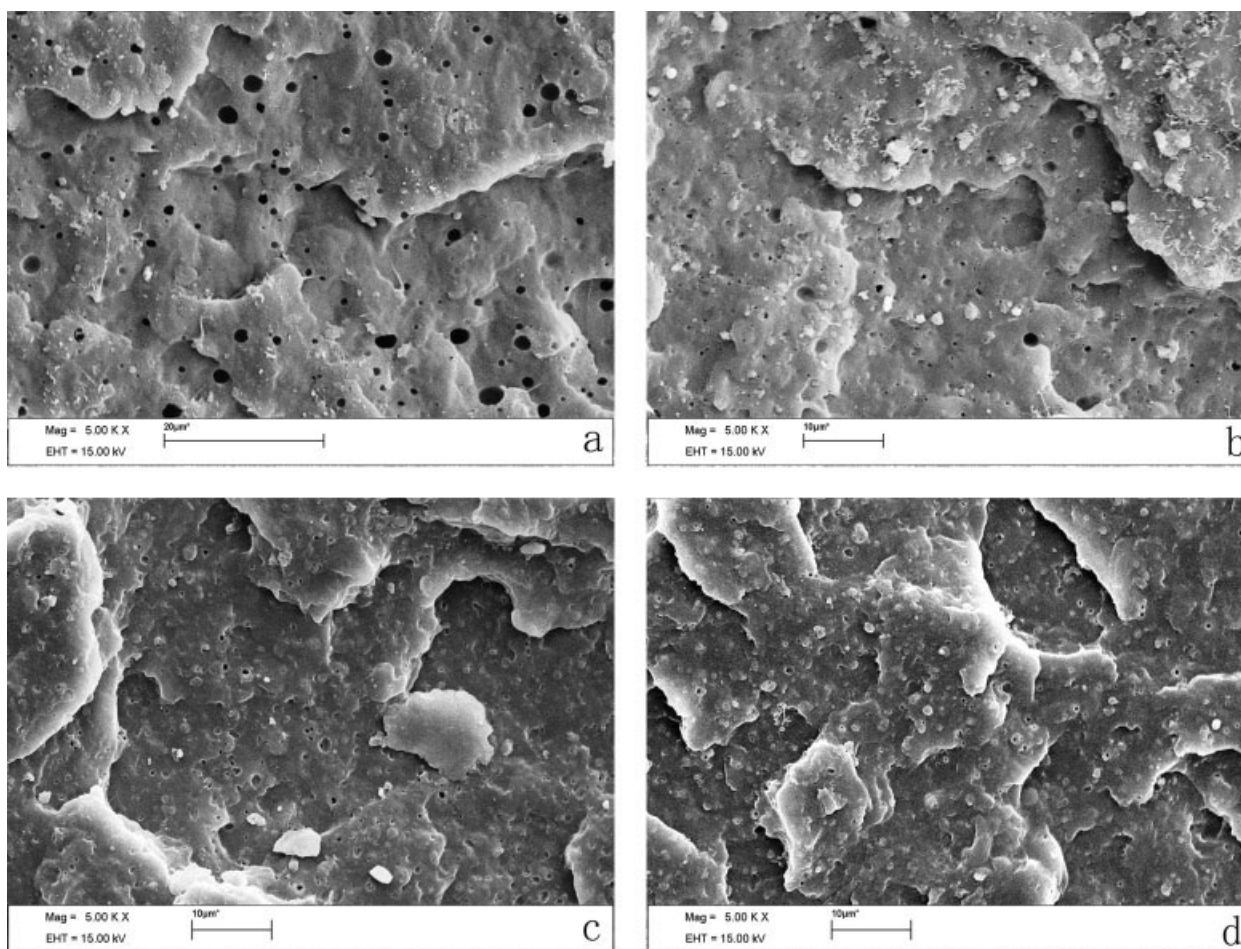
Figure 3 SEM micrographs of impact-fractured surfaces of PP/nylon 11/EPDM-*g*-MAH blends with various EPDM-*g*-MAH concentration: (a) 0%; (b) 5%; (c) 8%; (d) 10% (nylon 11 was fixed at 10 wt % and etched by *m*-cresol).

to be detected, which constrained the application of pure PP under cold conditions. The blends containing exclusively 10 wt % nylon 11 exhibited 1.89 kJ/m^2 of Izod impact strength at LT. By contrast, blending of 10 wt % of EPDM-*g*-MAH improved Izod impact strength both at RT and LT as well as the elongation at break while maintaining the tensile strength at almost same level of the pure PP matrix. The ternary blends with the same nylon 11 content (10 wt %) displayed inferior tensile strength but superior impact strength and elongation at break as compared with the pure PP matrix. The impact strength at RT was still twofold than that of the pure PP and the impact strength at LT increased to 2.29 kJ/m^2 even at low EPDM-*g*-MAH loading level. The impact strength improved significantly with the further increase of EPDM-*g*-MAH. For the PP (80)/nylon 11(10)/EPDM-*g*-MAH(10) ternary blends, the Izod impact strength at RT was fourfold as that of pure PP and LT Izod impact strength increased to 3.42 kJ/m^2, respectively. The similar trend of improvement of the mechanical properties with the nylon 11 content was also observed for the ternary

blends with the same EPDM-*g*-MAH content (10 wt %). For example, the Izod impact strength at LT increased from 2.01 kJ/m^2 for the PP(90)/EPDM-*g*-MAH(10) to 2.69 kJ/m^2 for PP(82)/nylon 11(8)/EPDM-*g*-MAH(10) ternary blend.

Figure 4 The schematic representation of morphologies for the PP/nylon 11/EPDM-*g*-MAH ternary blends.

TABLE I
Mechanical Properties of the Pure PP Matrix and PP-Based Blends

Sample (PP/nylon 11/ EPDM-*g*-MAH)	Izod impact strength (kJ/m^2)		Tensile strength (MPa)	Elongation at break (%)
	Room temperature (RT)	−40°C (LT)		
100/0/0	2.51 ± 0.11	/	36.67 ± 1.09	450 ± 3
90/10/0	2.48 ± 0.08	1.89 ± 0.05	32.55 ± 1.02	435 ± 5
87/10/3	3.86 ± 0.15	2.03 ± 0.07	31.94 ± 0.81	447 ± 4
85/10/5	5.23 ± 0.21	2.29 ± 0.11	29.81 ± 0.36	461 ± 3
82/10/8	9.10 ± 0.16	3.01 ± 0.17	28.53 ± 0.42	539 ± 5
80/10/10	10.68 ± 0.31	3.42 ± 0.11	27.98 ± 0.28	549 ± 5
90/0/10	5.71 ± 0.18	2.01 ± 0.06	36.43 ± 0.76	560 ± 10
88/2/10	6.49 ± 0.13	2.20 ± 0.08	31.69 ± 0.34	582 ± 7
85/5/10	7.28 ± 0.21	2.33 ± 0.02	30.42 ± 0.26	548 ± 3
82/8/10	8.78 ± 0.29	2.69 ± 0.15	29.85 ± 0.18	532 ± 7

Toughening mechanism

For the blends studied in this work, EPDM-*g*-MAH may act as sites of local stress concentration; these sites are capable of initiation of craze or shear band. Crazes might also be terminated at shear bands that are initiated by individual small particles or by mutual termination of several crazes, where the relative rubber concentration was high. Crazes are then kept from propagating into destructive cracks. Therefore, the initiation and propagation of craze or shear band effectively dissipates the applied energy, which leads to high toughness. Furthermore, the core-shell structure in the PP/nylon 11/EPDM-*g*-MAH ternary blends (nylon 11 as a rigid core surrounded by a soft EPDM-*g*-MAH shell, illustrated in Fig. 3) extends the effective volume of the rubber particles, thus effectively decreasing the average matrix-ligament thickness. Moreover, the stress fields around EPDM-*g*-MAH particle encapsulated with nylon 11 particles might interfere or overlap with those dispersed in the PP matrix. In this case, the stress fields around EPDM-*g*-MAH shell-particles seem to serve as a bridge between two neighboring rubber particles. Therefore, the synergetic effect between the reduction of the average matrix-ligament thickness and the overlap of the stress field between EPDM-*g*-MAH and core-shell structure is believed to result in the observed growth in the toughness of PP/nylon 11/EPDM-*g*-MAH ternary blends.

CONCLUSIONS

In this article, the morphology and mechanical properties of PP/nylon 11/EPDM-*g*-MAH blends were investigated. It was found that the maleated EPDM dispersed invisibly within the PP matrix at the low EPDM-*g*-MAH loading, while there was clear phase separation in the case of 10 wt % EPDM-*g*-MAH. For the PP/nylon 11/EPDM-*g*-MAH ternary blends,

the dispersed phase morphology of EPDM-*g*-MAH was hardly affected by the addition of nylon 11, but the size of nylon 11 dispersed phase domains was significantly reduced by EPDM-*g*-MAH incorporation. Moreover, a core-shell structure, in which nylon 11 was a rigid core surrounded by a soft EPDM-*g*-MAH shell, was formed. It was found from the mechanical properties that the ternary blends exhibited inferior tensile strength as compared with the PP matrix, but superior toughness. Especially excellent low-temperature impact strength was obtained. For the PP (80)/nylon 11(10)/EPDM-*g*-MAH(10) ternary blends, the synergetic toughness occurred because of the formation of the core-shell structure, the Izod impact strength at room temperature and −40°C was fourfold as that of pure PP and increased to 3.42 kJ/m^2, respectively. The toughening mechanism was intimately related with the phase morphology.

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