

Mechanical Properties of Glass Fiber and Liquid Crystalline Polymer Reinforced Polypropylene Hybrid Composites Toughened with Elastomers

S. C. Tjong,¹ S. A. Xu,^{1,2}

¹Department of Physics and Materials Science, City University of Hong Kong, Tat Chee Avenue, Kowloon, Hong Kong
²Institute of Polymer Science and Engineering, East China University of Science and Technology, 130 Mei Long Road, Shanghai, China

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ABSTRACT: A group of short glass fiber (SGF) and liquid crystalline polymer (LCP) reinforced polypropylene (PP) hybrid composites and toughened with maleic anhydride (MA)-grafted styrene-ethylene butylene-styrene (SEBS-g-MA) elastomers with controlled morphology were designed and injection molded. MA was also grafted to PP (PP-g-MA) in which the mPP blend was prepared by compounding 95% PP and 5% PP-g-MA. The matrix of hybrid composites consisted of 80/20 (wt %) mPP/SEBS-g-MA. The fibrillation of LCP minor phase depended on the injection-molded temperatures. The effects of LCP and SGF hybridization on the morphology and mechanical characteristics of quaternary hybrid composites were studied. Tensile measurements showed that hybridization of SGF and LCP fibrils were

beneficial in improving the tensile strength and stiffness of hybrid composites prepared at 265 and 285°C. This was attributed to the fact that LCP minor phase can deform into fine and long fibrils at these temperatures. However, LCP minor phase was deformed to nonuniform ellipsoids at 220°C, leading to poorer mechanical performance of the hybrid composites. The correlation between the processing temperature and compatibilizer with the structure-mechanical property of hybrid composites is discussed. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 1539–1546, 2004

Key words: polypropylene (PP); fibers; compatibility; elastomers; liquid crystalline polymer

INTRODUCTION

Polypropylene (PP) is a widely used commodity polymer because of its low cost, ease of processing, good mechanical properties, and acceptance to various types of fillers. However, its application as a structural material is somewhat limited because of its relatively poor strength under low-temperature or high-loading-rate conditions. The impact toughness of PP can be improved considerably by the addition of elastomers such as ethylene-propylene rubber (EPR), ethylene-propylene-diene monomer (EPDM), and styrene-ethylene butylene-styrene (SEBS) triblock copolymer.¹ Among these, SEBS can act as an impact modifier and a compatibilizer. The incorporation of elastomers into PP leads to a dramatic reduction in the modulus and the yield stress; the stiffness and yield strength generally decrease with increasing elastomer content. Considerable efforts have been devoted to achieve simultaneous reinforcing and toughening of PP. For example, the addition of inorganic fillers or short glass

fibers (SGF) into PP/elastomer blends provides an attractive means of maintaining its stiffness-to-toughness balance. The incorporation of both rigid reinforcement and elastomeric phase into PP leads to the formation of ternary or hybrid composites. Extensive studies have been conducted on the structure-property relationship of PP hybrid composites reinforced with inorganic fillers or particulates.^{2–4}

Polymer composites are generally fabricated by the injection-molding process because of its cost effectiveness, good dimensional accuracy, and excellent surface finish. The mechanical properties of the injection-molded fiber-reinforced composites are determined by the interfacial bonding of the fiber and polymer matrix, fiber content, fiber orientation, and distribution. A strong interfacial bond generally yields better tensile and impact properties for the composites. Good bonding between the SGF and the PP matrix ensures effective stress transfer from the matrix to the reinforcing phase during tensile deformation. The adhesion between the SGF and PP is very poor because the glass surface is polar but PP is a nonpolar polyolefin. Functional groups such as maleic anhydride (MA) are commonly grafted to PP or SGF to enhance the interfacial adhesion of the composites.^{5,6} Recently, MA-

Correspondence to: S. C. Tjong (aptjong@cityu.edu.hk).

grafted styrene-ethylene-butylene-styrene (SEBS-*g*-MA) triblock copolymer was used increasingly to improve the compatibility and toughness of the polyblends and hybrid composites.⁷⁻¹¹

It is generally known that the addition of SGF into polymers can result in an increase in the viscosity of the materials. This leads to difficulties during processing of the composites. The melt viscosity of the fiber-reinforced composites can be reduced drastically by incorporation of a small amount of liquid crystalline polymers (LCPs).¹² The LCPs with high strength and stiffness, high chemical resistance, good dimensional stability, and low melt viscosity are attractive high-performance engineering materials. The LCPs can function as processing aids by reducing the viscosity of thermoplastic matrix during compounding, thereby easing the processibility of thermoplastics. Moreover, the mesogenic units of LCPs have a high degree of long-range order that enables them to orient along the flow direction during processing. This order leads to the formation of fine fibrils under certain processing conditions. The fine fibrils effectively reinforce the matrix of thermoplastics, giving rise to the development of polymer composites commonly known as *in situ* composites. The fibrillation, morphology, and distribution of LCP dispersed phase in the matrix is greatly affected by the processing conditions. Furthermore, other factors such as viscosity ratio of the components, LCP content, interfacial adhesion between the components, and the rheological characteristics of the matrix also play a crucial role in LCP fibrillation.¹²⁻²²

The development of hybrid composites based on the reinforcement of LCP and SGF fibers in polymeric matrices is of technological importance. More recently, several workers have attempted to achieve the desired rheological and mechanical properties of polymer composites via hybridization of LCP with SGF.²³⁻³¹ For example, Tjong and Meng have studied the morphology, rheology, and mechanical properties of *in situ* hybrid polyamide-6 (PA6) and PP composites reinforced with potassium titanate ($K_2Ti_6O_{13}$) whisker and LCP.^{23,24} $K_2Ti_6O_{13}$ whiskers are selected as reinforcement materials because of their low cost and superior mechanical strength as well as high stiffness. They reported that the addition of LCP into $K_2Ti_6O_{13}$ /PA6 or $K_2Ti_6O_{13}$ /PP composites decreases the melt viscosity, thereby improving the processibility of such composites. The tensile strength and modulus of the LCP/ $K_2Ti_6O_{13}$ /PA6 hybrid composites tend to increase with increasing whisker content. Kulichikhin et al. have investigated the effect of various concentrations of LCP additions to glass fiber reinforced PP composites. They reported that the addition of LCP concentrations > 20 wt % leads to a decrease of blend viscosity.²⁵ Kulichikhin et al. observed a similar ben-

eficial effect of LCP addition in reducing the melt viscosity of carbon fiber reinforced poly(ether sulfone) (PES) composites.^{26,27} More recently, Pisharath and Wong investigated the processibility of LCP-short glass fiber-PA6,6 hybrid composites.²⁸ They reported that such a hybrid composite offered better processibility over the glass fiber reinforced polymers alone. The thermal stability of glass fiber reinforced composite was improved by blending with LCP. The hybridization of PP, LCP, and silica (SiO_2) filler was attempted by Lee et al.^{29,30} They indicated that the silica particles promote the fibrillation of LCP in a LCP/PP blend. Such an improvement was attributed to an increase in the PP matrix viscosity. In previous studies, we investigated the mechanical properties of ternary SGF/(SEBS-*g*-MA/mPP) hybrid composite toughened with SEBS-*g*-MA elastomer.^{32,33} The 20/80 SGF/(SEBS-*g*-MA/Maleu anhydride compatibilized polypropylene [mPP]) composite toughened with SEBS-*g*-MA elastomer was shown to exhibit excellent impact toughness (essential work of fracture) under high-impact loading conditions.³² This article aims to investigate the combined effects of SGF and LCP fibers on the microstructure and mechanical properties of LCP-SGF/(SEBS-*g*-MA/mPP) hybrid composites. The properties of LCP-SGF/(SEBS-*g*-MA)/mPP quaternary hybrids are rather complicated because they consist of several phase components.

EXPERIMENTAL

Materials

The materials used in this study were polypropylene homopolymer (Profax 6331; Himont Co.), maleic anhydride grafted polypropylene (PP-*g*-MA; Epolene G3003; Eastman Chemical Co.), SEBS-*g*-MA (Kraton FG 1901X; Shell Co.), Vectra A950 (Ticona GmbH), and short glass fiber with a length of ~ 4 mm (144A-14C; Owens Corning). The glass fiber used was not treated with coupling agents. All materials used were dried separately in ovens for more than 48 h.

Blending

The matrix of hybrid composites consisted of 20/80 (wt %) SEBS-*g*-MA/mPP blend in which mPP was prepared by compounding 4 wt % PP-*g*-MA and 76 wt % PP. The matrix blend was fabricated by mixing 20 wt % SEBS-*g*-MA and 80 wt % mPP in a Brabender twin-screw extruder. The operating temperature profiles were 180–220–220–210°C. The extrudates were pelletized and dried. The dried polymer pellets, SGF and LCP, were then loaded into a Brabender twin-screw extruder with operating temperature profiles of 275–295–295–295°C. The extrudates were also pellet-

ized and dried. Hybrid composite designated as the H_1 sample in this article was reinforced with 10 wt % LCP and 20 wt % SGF. The total reinforcement content in the hybrid was 30 wt % with respect to the polymer matrix of hybrid. The H_2 hybrid was reinforced with 20 wt % LCP and 10 wt % SGF. For the purpose of comparison, polymer composite (designated as L_3) containing no SGF and reinforced with 30 wt % LCP only was prepared under similar conditions. The dried composite pellets were finally fed into an injection molder at 220, 265, 280, and 310°C, respectively, to produce plaques of $200 \times 80 \times 3.2$ mm.

Mechanical measurements

The static tensile behavior of the polymer and hybrid composites was determined at 21°C by using an Instron tester (model 4206) with a crosshead speed of 10 mm/min. Five specimens of each composition were tested and the average value was reported. Izod notched-impact samples were cut from injection-molded plaques. Impact measurements were carried out with a Ceast pendulum impact tester at 21°C. The fracture surfaces of tensile specimens were examined in a scanning electron microscope (JEOL JSM 820).

RESULTS AND DISCUSSION

Mechanical behavior

In a previous study, we conducted a study on the tensile deformation behavior of the mPP copolymer, 20/80 SEBS-*g*-MA/mPP blend, and 20/80 SGF/(SEBS-*g*-MA/mPP) composite.³² The tensile results show that the 20/80 SEBS-*g*-MA/mPP blend undergoes extensive plastic deformation up to a strain exceeding 90% and no final failure associated with the addition of SEBS-*g*-MA into mPP. However, the incorporation of SEBS-*g*-MA in mPP leads to a sharp drop in the yield stress and stiffness. The introduction of SGF into the 20/80 SEBS-*g*-MA/mPP blend restores the yield stress, thereby maintaining the stiffness-to-toughness balance. Figure 1 shows the typical stress-strain curves for the PP homopolymer and H_1 , H_2 , and

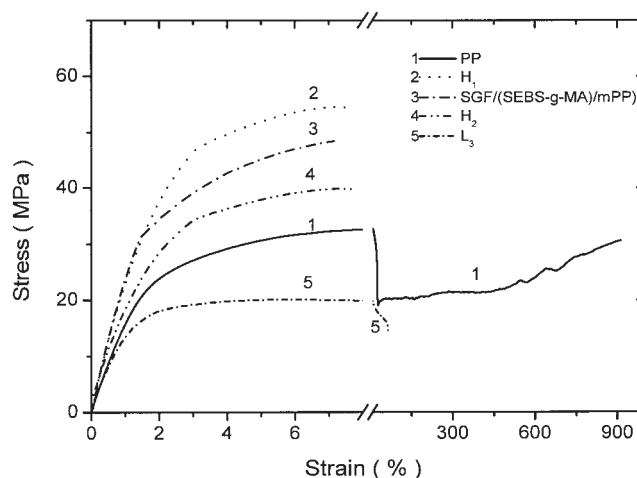


Figure 1 Stress-strain curves for the PP homopolymer and H_1 , H_2 , and L_3 composite specimens molded at 220°C.

L_3 composite specimens molded at 220°C. The stress-strain curve of the 20/80 SGF/(SEBS-*g*-MA/mPP) composite is also shown for the purpose of comparison. It can be seen that the yield stress of H_1 hybrid containing 20% SGF and 10% LCP (53.8 MPa) is slightly higher than that of the 20/80 SGF/(SEBS-*g*-MA/mPP) composite (49.7 MPa). As the LCP content in hybrid is increased to 20% (H_2), the tensile stress at break drops considerably. A dramatic drop in the stress at break is observed by further increasing the LCP content to 30 wt % in the composite specimen (L_3). The tensile ductility of hybrids is not impaired by associating with the incorporation of LCP. The mechanical properties of composite specimens molded at 220°C are listed in Table I. This table also reveals that the LCP addition to hybrids only leads to a slight decrease of the Izod impact strength. However, L_3 composite containing no SGF exhibits the lowest impact strength.

As mentioned above, the fibrillation, morphology, and distribution of the LCP dispersed phase in the matrix of composites is greatly affected by the processing conditions such as processing temperature. In the case of 20/80 SGF/(SEBS-*g*-MA/mPP) composite,

TABLE I
Mechanical Properties of PP, Its Blends, and Injection-Molded Composites at 220°C

Sample	Young's modulus (MPa)	Yield stress (MPa)	Elongation at break (%)	Impact strength (kJ/m ²)
Pure PP ³²	1890 ± 165	32.6 ± 0.2	>910	2.71 ± 0.10
mPP ³²	1760 ± 140	31.9 ± 0.1	>910	2.75 ± 0.10
SEBS- <i>g</i> -MA/mPP ³²	1330 ± 130	24.2 ± 0.5	>910	25.38 ± 0.44
SGF/SEBS- <i>g</i> -MA/mPP ³²	4150 ± 522	49.7 ± 0.3	7.3 ± 0.2	10.77 ± 0.58
H_1	4143 ± 296	53.8 ± 0.4	7.6 ± 0.2	9.56 ± 0.50
H_2	3426 ± 267	39.4 ± 0.6	7.4 ± 0.6	7.89 ± 0.23
L_3	1859 ± 83	20.0 ± 0.1	62.0 ± 11.7	3.83 ± 0.47

TABLE II
Mechanical Properties of Injection-Molded Composites at 265°C

Sample	Young's modulus (MPa)	Yield stress (MPa)	Elongation at break (%)	Impact strength (kJ/m ²)
H ₁	4378 ± 109	60.1 ± 0.6	7.4 ± 0.1	9.95 ± 0.32
H ₂	3601 ± 191	54.6 ± 0.6	7.7 ± 0.3	8.08 ± 0.15
L ₃	2644 ± 84	44.4 ± 0.6	9.1 ± 0.4	3.98 ± 0.30

processing temperature much greater than 220°C could lead to a deterioration in mechanical properties because of the degradation of polymer molecules. Thus, the 20/80 SGF/(SEBS-*g*-MA/mPP) composite is injection molded at 220°C. However, fibrillation of LCP does not take place in H₁ and H₂ hybrids at a low processing temperature of 220°C. In the absence of LCP fibrils, short glass fibers act as main load-bearing elements in these hybrids during tensile loading. The yield stress of H₁ hybrid containing 20 wt % SGF content exhibits a slightly higher yield than that of 20/80 SGF/(SEBS-*g*-MA/mPP) composite. However, the yield stress of H₂ hybrid containing 10 wt % SGF and 20 wt % LCP is lower than that of the 20/80 SGF/(SEBS-*g*-MA/mPP) composite. The L₃ specimen containing no SGF exhibits the lowest yield tensile stress and Izod impact strength.

To improve the tensile performance of hybrids, the composite specimens were injection molded at 265, 285, and 310°C, respectively. Their tensile and impact properties are summarized in Tables II and III. From these, it appears that the H₁ and H₂ hybrids molded at 265°C exhibit superior mechanical performance owing to the fibrillation of LCP on the basis of SEM observation that will be discussed later. It should be noted that the inorganic filler also promotes the fibrillation of LCP minor phase under appropriate processing conditions.^{29,30} In this respect, fine LCP fibrils can bear the applied load. Therefore, the stiffness and stress at break of H₁ hybrid are higher than those of the 20/80 SGF/(SEBS-*g*-MA/mPP) (Tables I and II). Pisharath and Wong²⁸ reported that the LCP not only served as a processing aid but also as a reinforcing agent in

TABLE III
Mechanical Properties of Injection-Molded Composites at 285°C

Sample	Young's modulus (MPa)	Yield stress (MPa)	Elongation at break (%)	Impact strength (kJ/m ²)
H ₁	4285 ± 202	58.5 ± 0.5	7.2 ± 0.2	8.83 ± 0.27
H ₂	3188 ± 139	50.4 ± 0.5	8.2 ± 0.1	6.68 ± 0.31
L ₃	2168 ± 84	38.3 ± 1.0	10.6 ± 0.5	4.83 ± 0.32

TABLE IV
Mechanical Properties of Injection-Molded Composites at 310°C

Sample	Young's modulus (MPa)	Yield stress (MPa)	Elongation at break (%)	Impact strength (kJ/m ²)
H ₁	4183 ± 224	53.1 ± 0.6	6.5 ± 0.2	7.38 ± 0.33
H ₂	2950 ± 94	43.6 ± 0.4	7.6 ± 0.2	6.25 ± 0.63
L ₃	2055 ± 207	27.48 ± 1.9	10.2 ± 0.9	2.39 ± 0.53

LCP/SGF/PA6,6 hybrid composite when its domains were oriented along the load-bearing direction. They also reported a similar reinforcing effect of LCP in the carbon fiber/PES composites. Such *in situ* hybrid composites contain two kinds of fiber reinforcements with diameters at two orders of magnitude (i.e., carbon fibers with their diameters on the order of 1–10 μm and LCP fibrils of 10⁻¹ μm). Carbon fibers act as the main load bearer while LCP fibrils block the propagation of microcracks in the hybrids.^{26,27} From Table III, it is evident that the mechanical properties of the H₁, H₂ hybrids are slightly lower than those of similar samples processed at 265°C. Extensive LCP fibrillation is also observed. However, the mechanical characteristics of H₁, H₂, and L₃ composite specimens molded at 310°C are inferior to those of H₁, H₂, and L₃ composites molded at 265°C (Table IV). This is attributed to the degradation of polymer matrices at high processing temperature. Degradation of PP occurs readily when PP is processed above the rheological transition temperature of Vectra A-950 (290°C). This leads to poor mechanical performance of the Vectra A-950/PP blends.^{19,34} Thus, H₁, H₂, and L₃ molded composites are processed at 265–285°C to trade off the mechanical strength against degradation.

SEM fractography

Figure 2(a–b) is the SEM micrographs showing the tensile fractured surfaces of H₁ and H₂ hybrids fabricated at 220°C. It is evident that the LCP minor phase disperses as ellipsoid domains in the matrices of hybrids. The glass fiber surfaces are partially bonded with a small piece of the matrix material in H₁ hybrid [Fig. 2(a)]. This indicates that the bonding between the SGF and PP is enhanced somewhat by the MA functional group grafted to PP. It can be seen from Figure 2(b) that the LCP phase of H₂ hybrid fabricated at 220°C deforms into ellipsoids instead of fine fibrils. Moreover, the adhesion or wetting between reinforcing LCP phase and the polymer matrix is relatively poor. As mentioned above, the matrix of the hybrids is functionalized with maleic anhydride compatibilizer (i.e., mPP and SEBS-*g*-MA). It is suggested that the

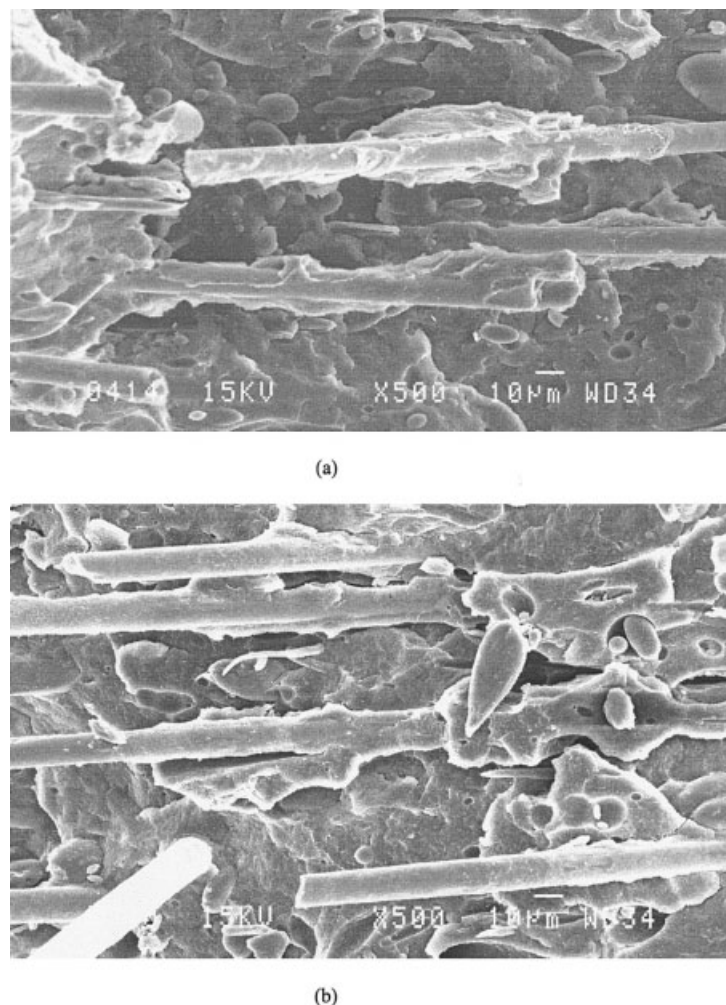


Figure 2 SEM fractographs showing the fracture surfaces of (a) H₁ and (b) H₂ hybrid molded at 220°C.

maleic anhydride functional group tends to react completely with the hydroxyl group of glass fiber during compounding at low processing temperature of 220°C. Such interaction results in better bonding between the glass fiber and matrix of the composites. This is evidenced by the adherence of matrix material on the glass fiber surfaces, as shown in Figure 2. Consequently, fewer maleic anhydride functional groups grafted to PP and SEBS are available to react with reinforcing LCP phase at a low-processing temperature of 220°C. At this temperature, the MA compatibilizer does not facilitate the fibrillation of LCP in PP matrix of hybrids. The LCP phase deforms only into ellipsoids rather than fine fibrils. Poor wetting is resulted accordingly as evidenced by the pull-out of LCP ellipsoids. The compatibilizing effect of maleic anhydride on the adhesion mechanism of the glass fibers and LCP phase is discussed in the next section. For the L₃ composite fabricated at low processing temperature of 220°C, LCP ellipsoids with various lengths are observed (Fig. 3).

Figure 4(a–b) shows the SEM micrographs showing the tensile fractured surfaces of H₁ and H₂ hybrids fabricated at 265°C. At such higher processing temperature, fine and long LCP fibrils are formed in the hybrids at 265°C. The SGF with a diameter of ~ 10 µm and LCP with ~ 1 µm acts synergistically during tensile loading. The tensile properties of hybrids molded at 265°C are improved accordingly. It is worth noting that the interfacial adhesion between the reinforcement-matrix phases plays a decisive role in enhancing the mechanical properties of hybrid composites. Strong interfacial bonding promotes effective stress transfer from the matrix to reinforcement fibers during loading.

Compatibilizing effect

Several interfacial interactions could occur in the LCP-SGF/(SEBS-*g*-MA)-PP hybrids (i.e., between the SEBS and mPP, SGF and SEBS, SGF and mPP, LCP and mPP). According to the literature, SEBS has good com-

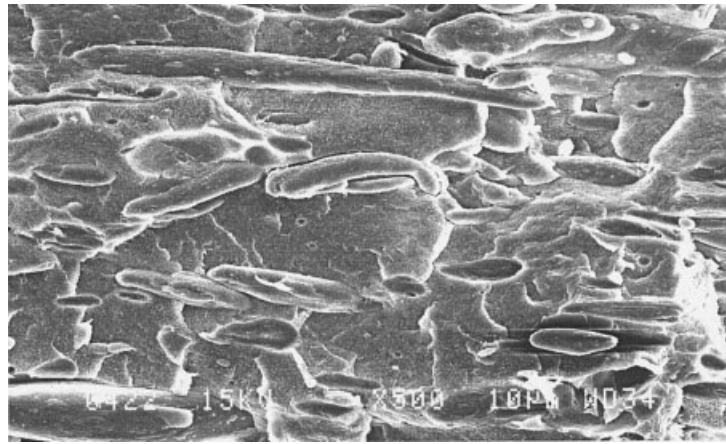
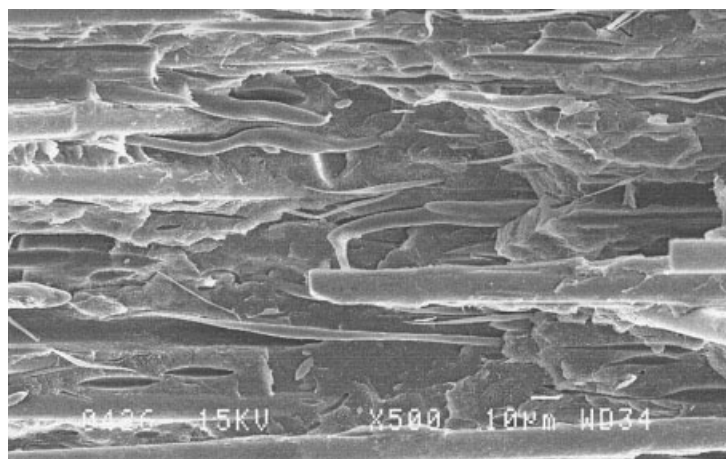


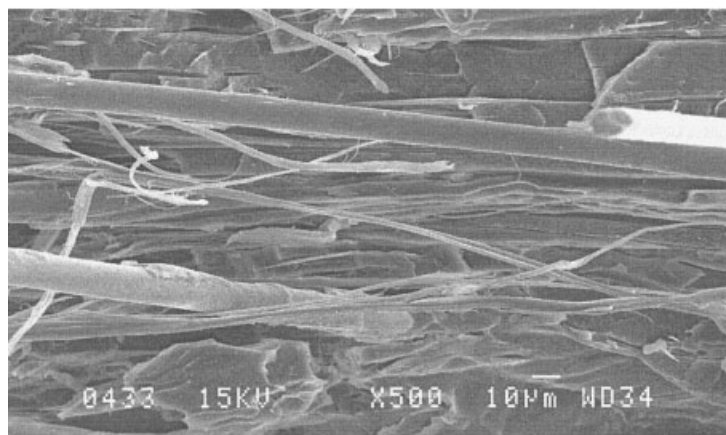
Figure 3 SEM fractograph showing the fracture surface of L₃ composite molded at 220°C.

patibility with PP because ethylene butyl (EB) mid-block of SEBS can diffuse into the PP phase, forming small micelles. The interdiffusion between the EB

block of SEBS and PP contributes to a certain extent to improvement in the interfacial between the SEBS and PP.¹¹ Grafting PP with MA can further strengthen the



(a)



(b)

Figure 4 SEM fractographs showing the fracture surfaces of (a) H₁ and (b) H₂ hybrid molded at 265°C.

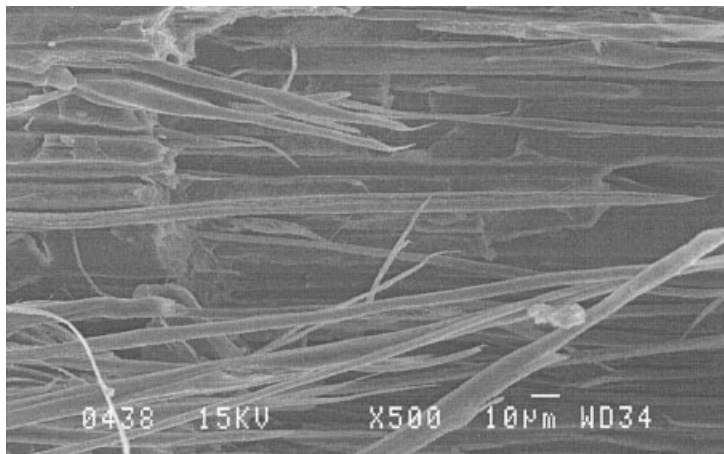
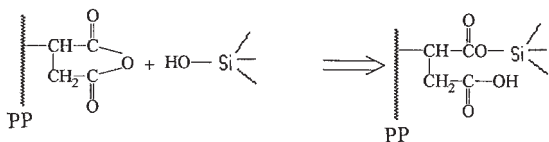
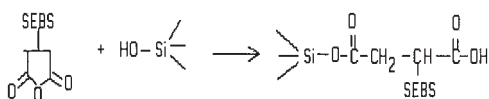


Figure 5 SEM fractograph showing the fracture surface of L₃ composite molded at 265°C.

interfacial bonding via a chemical reaction between the SEBS and PP. SGF has poor bonding with SEBS particles. The interfacial bonding between SGF and PP is also very poor because SGF exhibits a polar surface and PP is a nonpolar polymer. Functional MA grafted to PP can react with the hydroxyl group on the SGF surfaces during compounding, thereby enhancing the bonding between them. The chemical reaction can take place as follows:



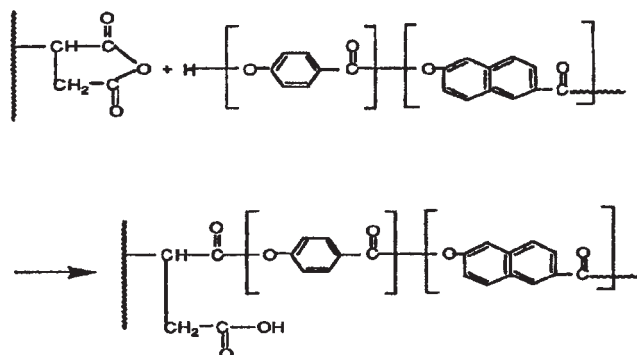
Although SEBS exhibits poor bonding with the SGF, the MA functional group grafted to SEBS can react with the hydroxyl group on the SGF surface during compounding. The chemical reaction that takes place between SEBS-g-MA and SGF is shown as:



The *in situ* graft copolymer formation between MA and the hydroxyl group of SGF is responsible for enhancement of interfacial bonding. Therefore, the glass fiber surfaces of the hybrids are partially adhered with the matrix materials owing to improved interfacial adhesion between mPP matrix and SGF, and between SEBS and SGF fiber.

We now consider the interfacial bonding between LCP and the matrix of hybrids. The properties of LCP/PP blends compatibilized with PP functional-

ized by maleic anhydride (MA-g-PP) are well documented in the literature.^{19,35-37} Seo³⁸ reported that the chemical reaction between the MA functional group grafted to elastomer and the hydroxyl group of Vectra A-950 copolyester can take place during compounding via:



The existence of graft copolymers at the LCP/matrix interface in *in situ* composites was confirmed by Fourier transform Raman spectroscopy.³⁸ It is considered that that MA functional group grafted to PP and SEBS can react with LCP of hybrids during compounding at higher processing temperatures of 265°C and above. The MA compatibilizer can facilitate the fibrillation of LCP in PP matrix of hybrids under appropriate processing conditions.²⁴ Tjong and Meng reported that fine LCP fibrils with a large aspect ratio are formed in the compatibilized PP/mLCP blends containing LCP content ≥ 10 wt %. However, LCP fibrils do not develop in uncompatibilized LCP/PP blends.¹⁹ The SEM micrograph of L₃ composite containing no SGF is depicted in Figure 5. It can be seen that the LCP minor phase can deform into long fibrils in the matrix of the LCP/(SEBS-g-MA)-PP composite.

CONCLUSION

LCP and SGF reinforced PP hybrids toughened with SEBS-*g*-MA were designed and produced via injection molding at various temperatures. The morphology and mechanical properties of the quaternary hybrids were characterized by means of SEM, tensile, and Izod impact tests. Tensile testing showed that the tensile strength and stiffness of H₁ quaternary hybrid molded at composites molded at 220°C are considerably lower than those prepared at 265 and 285°C. This is attributed to the finding that the LCP minor phase does not deform into fine fibrils at 220°C. SEM revealed that the LCP minor phase was dispersed as nonuniform ellipsoids in the matrices of composites. Thus, the glass fiber acts as the main load bearer during tensile loading. As the injection-molding temperatures are increased to 265 and 285°C, excessive fibrillation of LCP minor phase takes place as evidenced by scanning electron microscopy. MA compatibilizer can facilitate the fibrillation of LCP in PP matrix of hybrids at higher processing temperatures. The LCP fibrils act synergistically with SGF to carry the applied load, thereby improving the mechanical strength of the H₁ hybrid. However, hybrid composites prepared at higher temperatures of 310°C exhibit poorer mechanical performance owing to the degradation of polymer matrices.

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