

# Effects of the Glass Bead Content and the Surface Treatment on the Mechanical Properties of Polypropylene Composites

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**ABSTRACT:** Polypropylene composites filled with glass beads (GBs) were prepared by means of a twin-screw extruder. The tensile properties and impact-fracture strength of the composites were measured at room temperature to identify the effects of the GB content and surface treatment on the mechanical properties. The results show that the relative elastic modulus increased nonlinearly, whereas the tensile strength decreased with increasing GB volume fraction ( $\phi_f$ ). The notched impact strength increased with increasing  $\phi_f$  when  $\phi_f$  was less than 11%, and then, it decreased; this might have been related to the GB aggregation in the case of higher concentration. The mechanical

properties of the composite systems in which the GB surface was treated with silane coupling agent were better than those of the composite systems filled with the untreated GBs under the same conditions. Furthermore, the impact-fractured surfaces were observed with a scanning electron microscope to understand the interfacial morphology between the inclusion and the matrix and to examine the toughening mechanisms. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 3054–3063, 2012

**Key words:** composites; interfaces; polyolefins; strength; toughness

## INTRODUCTION

Polypropylene (PP) is a thermoplastic resin with good performance in processing and practical applications and a low price; hence, it is used widely in industry and in daily life. However, the application range is somewhat limited to further expansion because of its poor impact toughness, especially at or under room temperature. Therefore, the toughening of PP resin has become a research focus since the 1980s. In industry, PP is usually filled with organic or inorganic particles, such as wood, flour,<sup>1</sup> talc,<sup>2</sup> mica,<sup>3</sup> glass beads (GBs),<sup>4–6</sup> or calcium carbonate ( $\text{CaCO}_3$ ),<sup>2,7,8</sup> to improve its mechanical properties and to reduce its cost. To fabricate some kind of functional composite material, PP may be filled with metal powder, such as alumina,<sup>9</sup> iron,<sup>10</sup> or hollow particles.<sup>11</sup> As to inorganic particles,  $\text{CaCO}_3$  is used extensively in the plastics industry because of its low cost. Recently, the structure/property relationship nanometer  $\text{CaCO}_3$ -filled PP composites has been paid extensive attention.<sup>8</sup> More recently, one of us<sup>12</sup> investigated the effects of the particle size and content of diatomite on the impact-fracture tough-

ness of filled PP composites, and got some interesting findings.

Because GBs have a smooth and spherical surface, the effects of the beads on the processing properties of the composites and the product surface quality should be insignificant. Moreover, the internal stress in the product should be less than that of other inorganic particles under the same conditions.<sup>13</sup>

The tensile and impact properties are important characteristics for the utilization of materials; the effects of the shape and size of inorganic particles on the tensile strength and impact strength of polymer composite materials are usually significant. Sometimes, a brittle–ductile transition phenomenon can also be observed in polymer composites filled with inorganic particles.<sup>14</sup> There are a number of complicated factors affecting the tensile and impact properties of polymer composites. It is generally believed that the mechanical properties of polymer composites are closely related to the compatibility between the filler and the matrix, the distribution and dispersion of the particles in the matrix, and the filler concentration, shape, and size. In general, the filler surface treated with a suitable coupling agent is beneficial in improving the compatibility between the filler and the matrix and the uniformity of the distribution and dispersion of the particles in the matrix of the polymer composites. Thus, studies on the surface treatment of the inclusions have been

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paid more and more attention in the past 2 decades.<sup>13–15</sup>

The objectives in this study were to prepare PP composites filled with GBs and measure their tensile and impact properties, including Young's modulus (i.e., tensile elastic modulus), tensile strength, elongation at break, and impact-fracture strength at room temperature, to investigate the effects of the particle content and surface treatment on the mechanical properties of the PP/GB composites.

## EXPERIMENTAL

### Raw materials

PP, with the trademark CJS-700G, was used as a matrix resin in this work. This resin was supplied by Guangzhou Petrochemical Works in Guangdong province (People's Republic of China), and its density in the solid state and its melt flow rate were 910 kg/m<sup>3</sup> and 10 g/10 min, respectively.

The GBs, supplied by Shanghai Huijing Nanometer New Materials Co., Ltd. (Shanghai, China), were used as the filler. Their mean diameter was 33.96  $\mu\text{m}$ , their density was 2.60 g/cm<sup>3</sup>, and their specific area was 74.41 m<sup>2</sup>/kg.

### Preparation

The surface of the GBs was first treated with a silane coupling agent, and the treated GBs were named GB1, whereas the untreated GBs were named GB2. The silane coupling agent, with the trademark KH-143, was supplied by the Xiangfei Institute of Chemistry (Nanjing, People's Republic of China). The GBs were first blended with PP in a high-speed mixer. The filler volume fractions were 5, 8, 11, 14, and 18%. Then, the PP/GB blends were extruded in the molten state of the resin by means of a corotating twin-screw extruder (model TSE-35A, Nanjing Ruiya Polymer Equipment Co. Ltd., Nanjing, China) under temperatures from 160 to 220°C; the screw rotation was 60 rpm, and the screw length-to-diameter ratio was 40/35.6. The feeding of the raw materials was done in the forced mode, and the feeding rate was the screw rotation of 25 rpm. Then, the extrudate was granulated to produce the PP/GB composites. These granular composites were dried for 5 h at 90°C. Finally, the specimens for tensile testing and impact testing, including Izod and Charpy testing, were molded with a plastics injection machine under temperatures ranging from 190 to 210°C, with the injection pressure varied from 6 to 8 MPa, a holding time of 14 s, and a cooling time of 20 s. It was a numerical-control, whole-automation, plastics-injection machine (model CJ80NC), and the screw diameter was 36 mm.

### Tensile property measurements

The tensile properties of the PP/GB composites were measured at room temperature by means of a universal materials testing machine (model CMT6104, Newsans Co., Ltd., Shenzhen, China). The tests were conducted according to ASTM D 638-91, and the crosshead speed was 50 mm/min.

### Impact property measurements

The Charpy notched impact properties of the PP/GB composites were measured at room temperature by means of a Charpy impact testing machine (model XJJ-5, Newsans). The tests were conducted according to ASTM D 256-93 to measure the Charpy V-notched impact-fracture toughness.

The Izod notched impact properties of the PP/GB composites were measured at room temperature by means of an Izod impact testing machine (model XJU-5.5, Newsans). The tests were conducted according to ASTM D 256-93 to measure the Izod V-notched impact-fracture toughness.

Each group specimens contained five pieces, and the average values of the impact properties were used from the measured data.

### Fractography

The specimen fracture surfaces from the impact tests were examined with a Leo 1530 VP scanning electron microscope (Zeiss Instrument Co., Oberkochen, Germany) to observe the impact-fractured surface morphology, including the interfacial debonding, interlayer structure, and filler dispersion or distribution in the PP matrix, to understand the toughening mechanisms of these composite systems. The specimens were gold-coated before the scanning electron microscopy (SEM) examination.

## RESULTS AND DISCUSSION

### Tensile properties

#### Tensile curves

Figure 1 shows the curves of the tensile stress ( $\sigma$ ) versus strain ( $\varepsilon$ ; i.e., tensile curves) of the PP/GB1 composites. It can be seen that the unfilled PP resin and the composites with low loading presented obvious necking phenomenon, and the fracture  $\varepsilon$  was large. When  $\phi_f$  was more than 11%, the fracture  $\varepsilon$  of the composites decreased with the addition of  $\phi_f$ ; namely the tensile ductility of these composite systems was weakened in this case. Figure 2 shows the  $\sigma$ - $\varepsilon$  curves of the PP/GB2 composites. Similarly, the composites with low loading presented obvious necking phenomenon, and the fracture  $\varepsilon$  of the

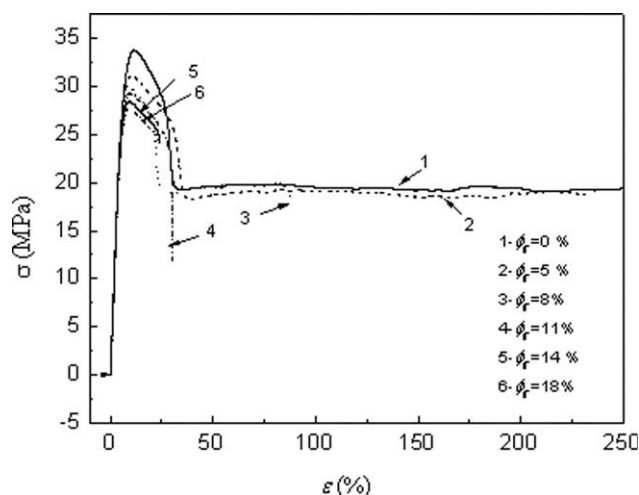


Figure 1  $\sigma$ - $\varepsilon$  curves of the PP/GB1 composite.

composites decreased with an increase of  $\phi_f$  when  $\phi_f$  was more than 11%. This was because part of the resin matrix decreased with increasing filler particle concentration; this led to an increase of the composite stiffness. In addition, because of the interaction between the inclusion and the matrix, the defects generated in the composites increased obviously in the case of higher concentration of the filler, and these defects first formed microcracks and developed quickly under a tensile load. Consequently, the ductility of the composites was weakened relevantly; this led to a reduction of the fracture  $\varepsilon$ .

One can see by comparing Figures 1 and 2 that the fracture  $\varepsilon$  of the PP/GB1 composites was greater than that of the PP/GB2 composites at the same GB content. This indicates that the tensile ductility of the composites increased to a certain extent as the GB surface was treated with the silane coupling agent. The reason might have been that the silane coupling agent made the chemical reaction, respectively, with the filler and the matrix; that is, the end

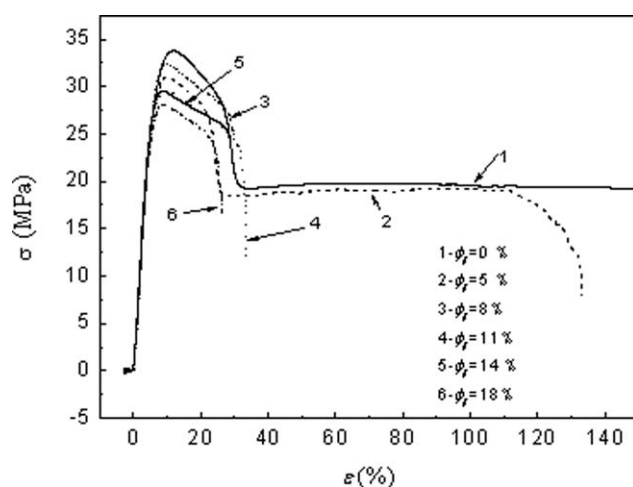


Figure 2  $\sigma$ - $\varepsilon$  curves of the PP/GB2 composite.

of the molecule of the silane coupling agent combined with the GB surface, whereas the organic function of the other end interacted with the PP molecule. This led to an enhancement of the interfacial bond strength between the matrix and the filler. Hence, the compatibility and interfacial adhesion between the GBs and the PP resin and the dispersion of the filler in the matrix may have been improved; this resulted in an increase of the tensile ductility of the composites, and the maximum increase of the fracture  $\varepsilon$  was more than 30%. Moreover, the maximum  $\sigma$  of the two composite systems decreased with an increase of the filler content.

#### Relative tensile elastic modulus

Figure 3 displays the relationship between the relative tensile elastic modulus ( $E_R$ ) of the PP/GB composites and  $\phi_f$ .  $E_R$  is defined as the Young's modulus ratio of the composite to the matrix. That is

$$E_R = E_c/E_m \quad (1)$$

where  $E_c$  and  $E_m$  are the Young's moduli of the composite and the matrix, respectively. It could be observed that the  $E_R$  increased nonlinearly with increasing  $\phi_f$  within the  $\phi_f$  range from 0 to 18%.  $E_R$  increased gently when  $\phi_f$  was less than 8% and then increased quickly with increasing  $\phi_f$ . The reason for this phenomenon might have been as follows. First, it might have been attributed to the stiffening effect of inorganic particles on the matrix; this was because when the modulus of the inclusions was much greater than that of the resin matrix, the stiffening effect of the composite would have been enhanced with increasing filler content. Second, the relative movement of the molecular chains would have been limited because of the filling of rigid particles into

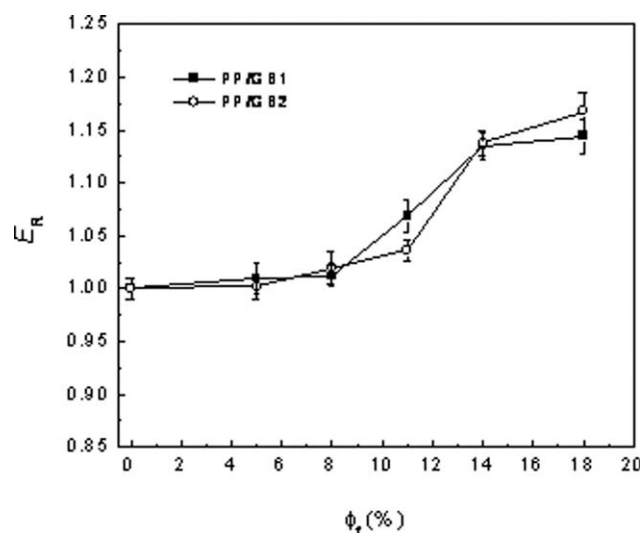


Figure 3 Relationship between  $E_R$  and  $\phi_f$ .

the resin. Consequently,  $E_c$  increased with increasing inorganic particles within a certain scope.<sup>4,13,16</sup>

Under the same conditions, the difference between the PP/GB1 system and the PP/GB2 system with regard to Young's modulus was not obvious. This indicated that the influence of the filler surface treatment on  $E_c$  of the filled PP composites was insignificant. This was because the tensile elastic modulus characterized the mechanical properties of the materials in the case of small deformation, and the filler dose did not generate a large deformation and, especially, did not produce a slipping or turning movement in the matrix. Therefore, the effect of the interfacial adhesion strength on  $E_c$  was relatively small. Demjen et al.<sup>17</sup> also noted that the Young's modulus described the mechanical properties of composites under the conditions with zero deformation, and it was not related to the interfacial adhesion strength. As a result, even though there was some effect of the silane coupling agent on the interfacial adhesion strength, it did not affect the tensile elastic modulus of the composite materials.

#### Tensile strength

If there was no interfacial adhesion between the filler and matrix, the external load supplied was only subjected by the matrix resin, whereas the inorganic particles could not be undertaken by the load. In this case, the tensile strength of the composites could be expressed as follows:<sup>18</sup>

$$\sigma_{yc} = \sigma_{ym}(1 - 1.21\phi_f^{\frac{2}{3}}) \quad (2)$$

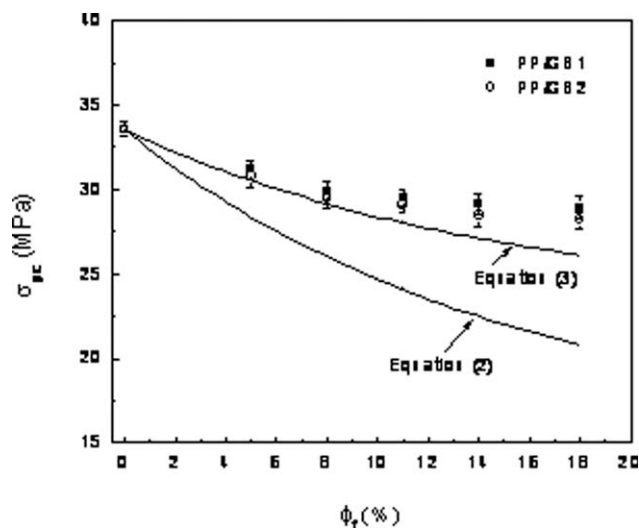
where  $\sigma_{yc}$  and  $\sigma_{ym}$  are the tensile strengths of the composite and the matrix resin, respectively, and  $\phi_f$  is the volume fraction of the filler.

In fact, there should have been a certain adhesion strength in the interface between the inclusions and the matrix for the polymer composites. On the basis of this, Liang and Li<sup>4,13</sup> introduced the concept of an interfacial adhesion angle ( $\theta$ ) and derived a modified tensile strength equation:

$$\sigma_{yc} = \sigma_{ym}(1 - 1.21 \sin^2 \theta \phi_f^{\frac{2}{3}}) \quad (3)$$

Obviously, eq. (3) will become eq. (2) when  $\theta$  is equal to  $90^\circ$ . In other words, the smaller  $\theta$  is, the better the interfacial adhesion state is. It is a good interfacial adhesion when  $\theta$  is equal to zero. In this case,  $\sigma_{yc} = \sigma_{ym}$ .

Figure 4 illustrates the relationship between  $\sigma_{yc}$  of the PP/GB composites and  $\phi_f$ . The  $\sigma_{yc}$  of the two composite systems decreased nonlinearly with increasing  $\phi_f$ . This was because when the effective matrix area of the specimen cross-section-supported



**Figure 4** Comparison of the predictions and measured data for the tensile strength.

tensile load decreased with increasing inorganic particle content, the tensile strength decreased when the interfacial adhesion strength between the GBs and the matrix was not strong. A similar conclusion could be obtained by analysis of eqs. (2) and (3).

On the other hand, Maiti and Mahapatro<sup>19</sup> and Demjen et al.<sup>17</sup> believed that the interaction between the filler particles and the matrix might weaken the motion of the molecular chains and block the stress transition, leading to reduction of the tensile strength. It can also be seen that the reduction of the tensile strength for both of the composite systems was obvious when  $\phi_f$  was smaller than 8%, and the tensile strength decreased, respectively, 10.9 and 12.4% for the PP/GB1 and PP/GB2 composites at  $\phi_f = 8\%$ . With further increasing GB content, the reduction of  $\sigma_{yc}$  for the PP/GB1 system decreased and tended to be gentle, whereas  $\sigma_{yc}$  for the PP/GB2 system decreased further, and the reduction was obvious. Under the same GB content,  $\sigma_{yc}$  of the PP/GB1 system was higher than that of the PP/GB2 system, and the difference between them increased with increasing  $\phi_f$ . This illustrated that the GB surface treated with the silane coupling agent was beneficial for improving the interfacial adhesion and resulted in an enhancement of the tensile strength of the PP/GB composites. As stated previously, the silane coupling agent might have made the chemical reaction with the GB surface and PP resin to improve the compatibility between the filler and matrix, respectively. Consequently, the dispersion of GB in the PP matrix and the interfacial adhesion between them were improved correspondingly; this resulted in an increase in the tensile strength.

$\theta$  could be determined by means of eq. (3) from the experimental data of polymer composites. The expression is as follows:<sup>20</sup>



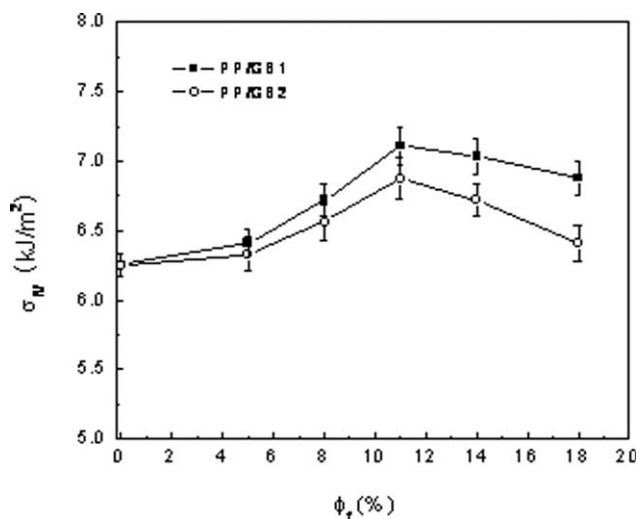


Figure 5 Relationship between the  $\sigma_{IV}$  and  $\phi_f$ .

$$\theta = \arcsin \left[ \frac{1 - \frac{\sigma_{yc}}{\sigma_{ym}}}{1.21\phi_f^{2/3}} \right]^{1/2} \quad (4)$$

$\theta$  of the PP/GB composite systems was about  $45^\circ$  under the testing conditions. Substituting the property parameters of the PP resin and GB in to eqs. (2) and (3), we estimated the tensile strength of the PP/GB composites in the concentration range of the filler and compared the predictions with the measured data, and the results are shown in Figure 4. It can be seen that the predictions of  $\sigma_{yc}$  by eq. (3) were closer to the measured data than those predicted by eq. (2). In addition, the measured tensile strength of the two composite systems was higher than the estimations by means of eq. (2). This indicates that the interfacial adhesion between the GBs and the PP matrix was good.

## Impact strength

### Notched Izod impact strength

Figure 5 shows the relationship between the V-notched Izod impact strength ( $\sigma_{IV}$ ) of the PP/GB composites and  $\phi_f$ . It can be seen that  $\sigma_{IV}$  of both the PP/GB1 and PP/GB2 composite systems at  $\phi_f = 5\%$  was slightly higher than that of the unfilled PP, and then, it increased obviously with increasing  $\phi_f$ .  $\sigma_{IV}$  reached up to a maximum at  $\phi_f = 11\%$ , and the  $\sigma_{IV}$  values for the PP/GB1 system and the PP/GB2 system increased 14 and 10%, respectively, compared to the unfilled PP resin. Then,  $\sigma_{IV}$  decreased with increasing  $\phi_f$ . Moreover,  $\sigma_{IV}$  of the PP/GB1 system was higher than that of the PP/GB2 system under the same conditions, and the difference in  $\sigma_{IV}$  between the two composite systems increased with increasing  $\phi_f$ .

### Notched Charpy impact strength

Figure 6 displays the relationship between the V-notched Charpy impact strength ( $\sigma_{CV}$ ) of the PP/GB composites and  $\phi_f$ . For the PP/GB1 system,  $\sigma_{CV}$  increased nonlinearly with increasing  $\phi_f$  when  $\phi_f$  was smaller than 8% and reached up to a maximum at  $\phi_f = 8\%$ ; then,  $\sigma_{CV}$  decreased with increasing  $\phi_f$ . Similarly,  $\sigma_{CV}$  for the PP/GB2 system increased nonlinearly with increasing  $\phi_f$  when  $\phi_f$  was smaller than 11% and reached up to a maximum at  $\phi_f = 11\%$ . Then,  $\sigma_{CV}$  decreased with increasing  $\phi_f$ . Furthermore, the difference in  $\sigma_{CV}$  between the two composite systems decreased with the addition of  $\phi_f$  when  $\phi_f$  was more than 11%. In brief, the toughening effect of these composite systems was significant under the experimental conditions.

It is generally believed that the toughness effect of particulate-filled polymer composites will be the best in a certain range of the inorganic particle concentration. In the case of low filler contents, the interaction between the filler and the matrix will be small because of the quite small number of particles in it; thus, the toughening effect will be insignificant. With increasing particle concentration, the inclusion might induce crazes in the matrix around it to absorb the impact deformation energy when the material undertakes an impact load and to block the development of the cracks; this will lead to an obvious increase in the impact-fracture strength.

However, in the case of a high concentration of filler particles, the microcracks and plastic deformation produced in the composite materials will be very great under an impact load; thus, they might be considered macroscopic stress cracks and result in a reduction of the toughness in this case. Moreover, the accumulation or agglomerate phenomenon of the filler in the matrix will be generated easily because

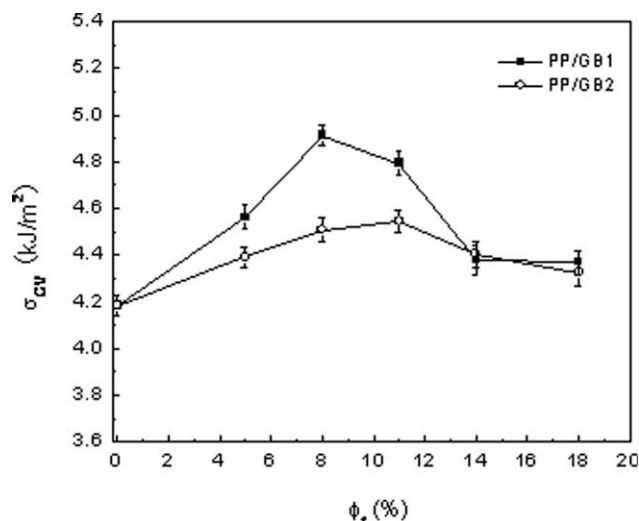
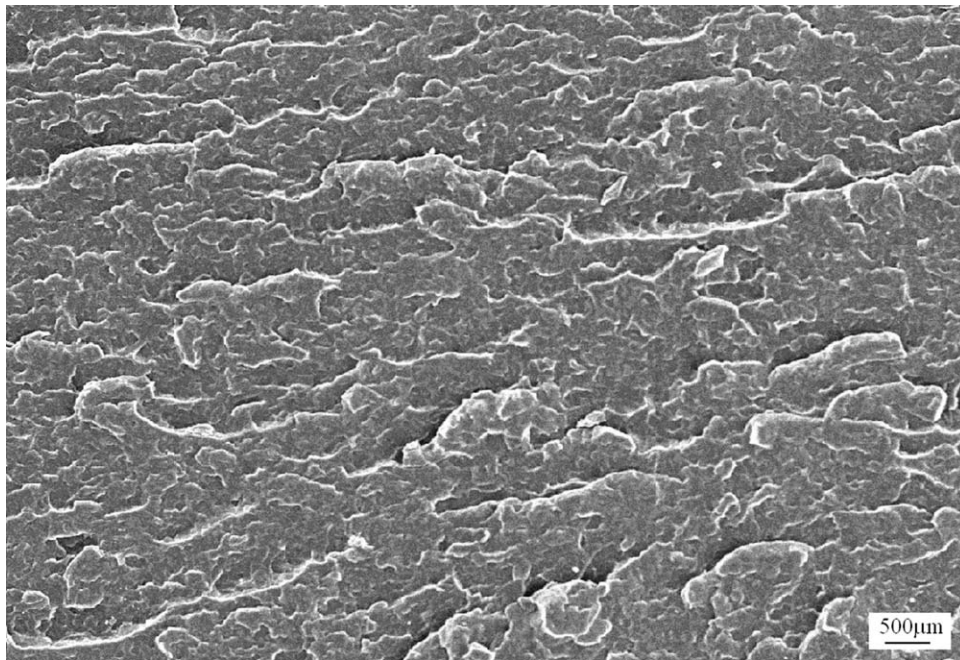


Figure 6 Relationship between  $\sigma_{CV}$  and  $\phi_f$ .



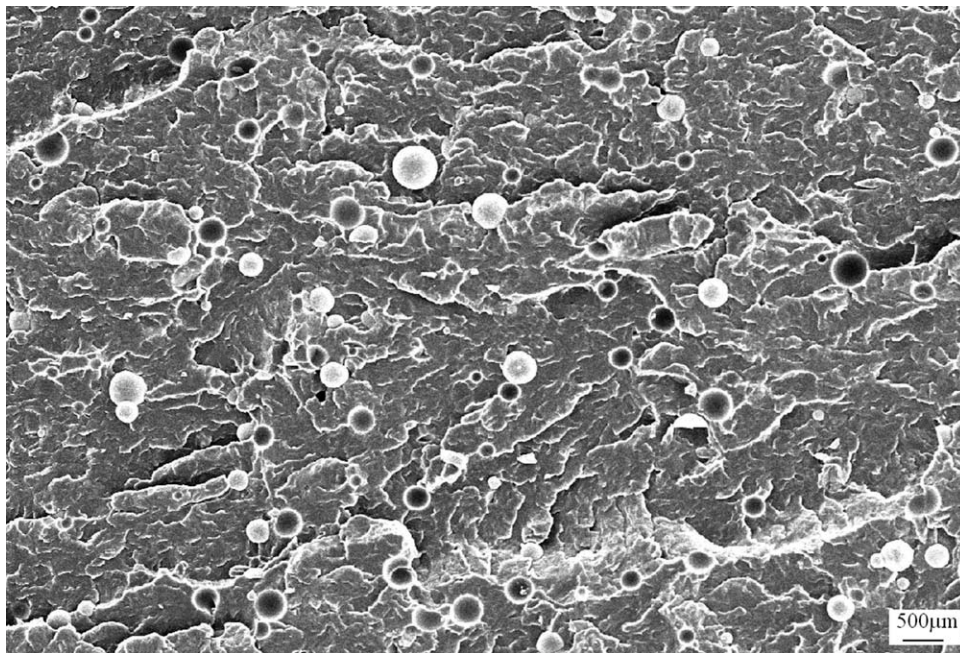
**Figure 7** SEM photograph of the impact-fractured surface of neat PP.

of bad dispersion when the particle concentration is quite high; this will lead to a decrease in the impact strength.<sup>13,15</sup>

### Morphology

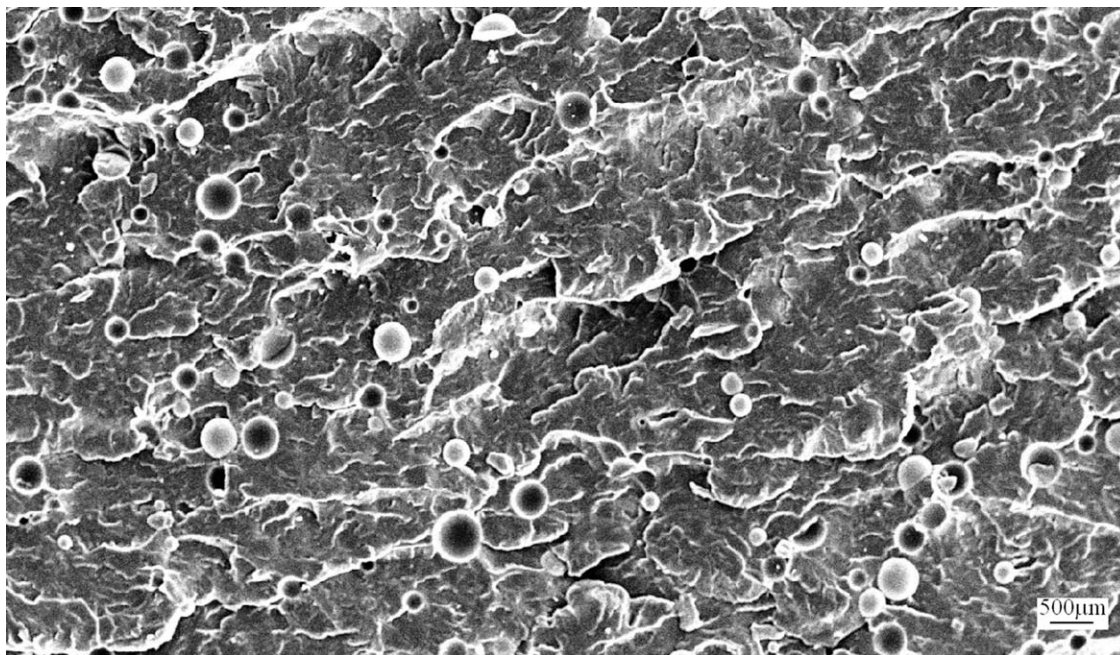
Figure 7 is the SEM photograph of the V-notched Izod impact-fractured surface of the unfilled PP resin. It may be observed that the impact-fractured surface is like sea waves, and the wave crest and the

trough are very clear. In addition, the arrangement and direction of the waves are regular and perpendicular to the impact direction. This means that the crack (e.g., the notch) developed from crazes might have propagated toward the whole cross section in a pattern of the wave until complete fracture of the specimen under the impact load. As a result, the specimen fractured rapidly, and the impact strength of the unfilled PP was relatively low (see Figs. 5 and 6).



**Figure 8** SEM photograph of the impact-fractured surface of the PP/GB1 system ( $\phi_f = 11\%$ ).



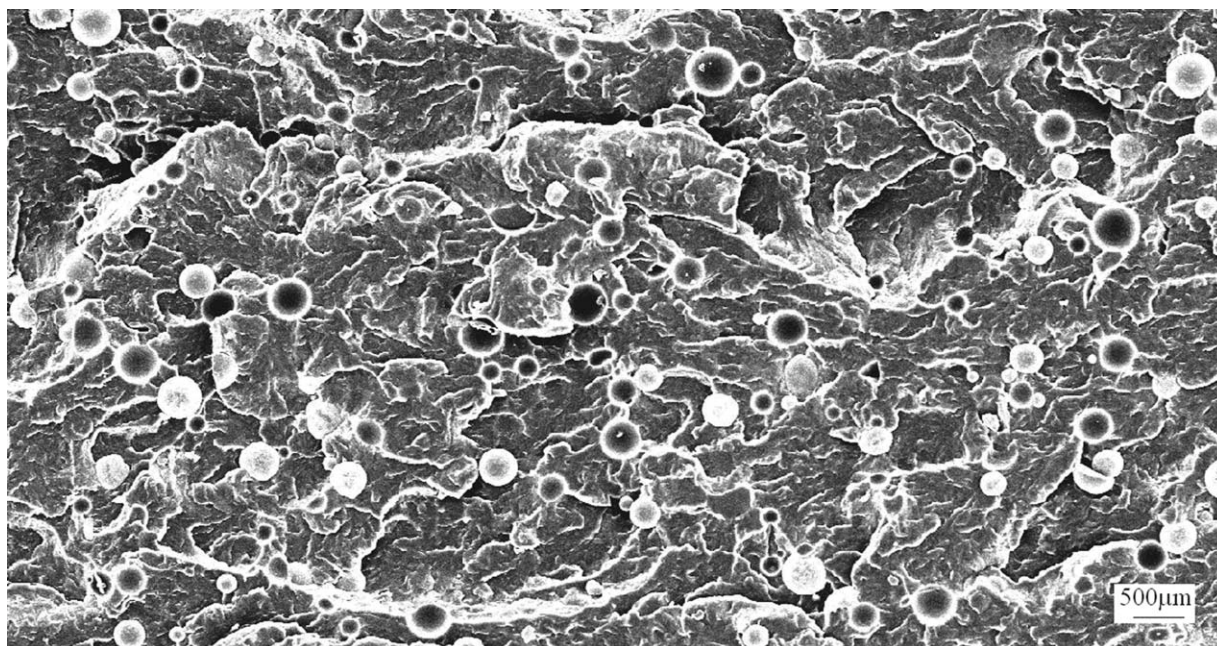


**Figure 9** SEM photograph of the impact-fractured surface of the PP/GB2 system ( $\phi_f = 11\%$ ).

Figure 8 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB1 composite when the filler volume fraction was 11%. It may be seen that the dispersion of the GBs in the PP matrix was roughly uniform, and the fracture surface was very uneven. This indicates that the matrix around the inclusions yielded first under the impact load because of stress concentration to generate plastic deformation and to form some morphology, such

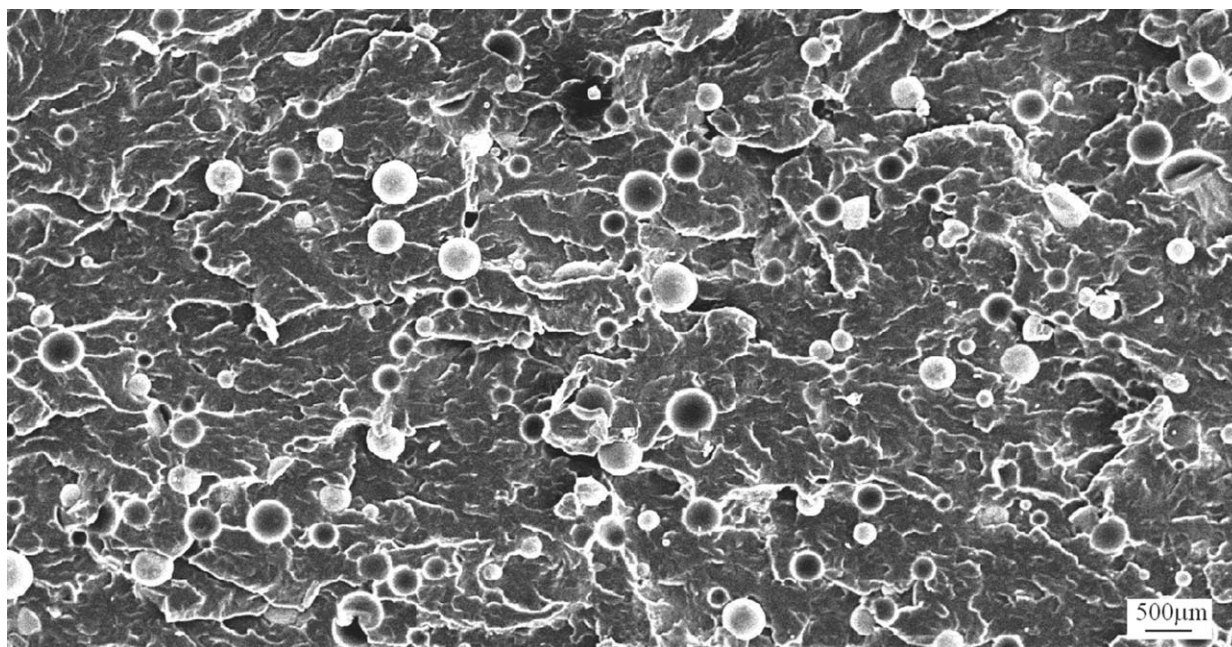
as small terraces. This shape, like small terraces, absorbed relevant impact deformation energy or fracture energy and led to an obvious improvement in the impact-fracture toughness of the composite systems (see Figs. 5 and 6).

Figure 9 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB2 composite when the filler volume fraction was 11%. Similarly, the dispersion of the GBs in the PP matrix



**Figure 10** SEM photograph of the impact-fractured surface of the PP/GB1 system ( $\phi_f = 18\%$ ).



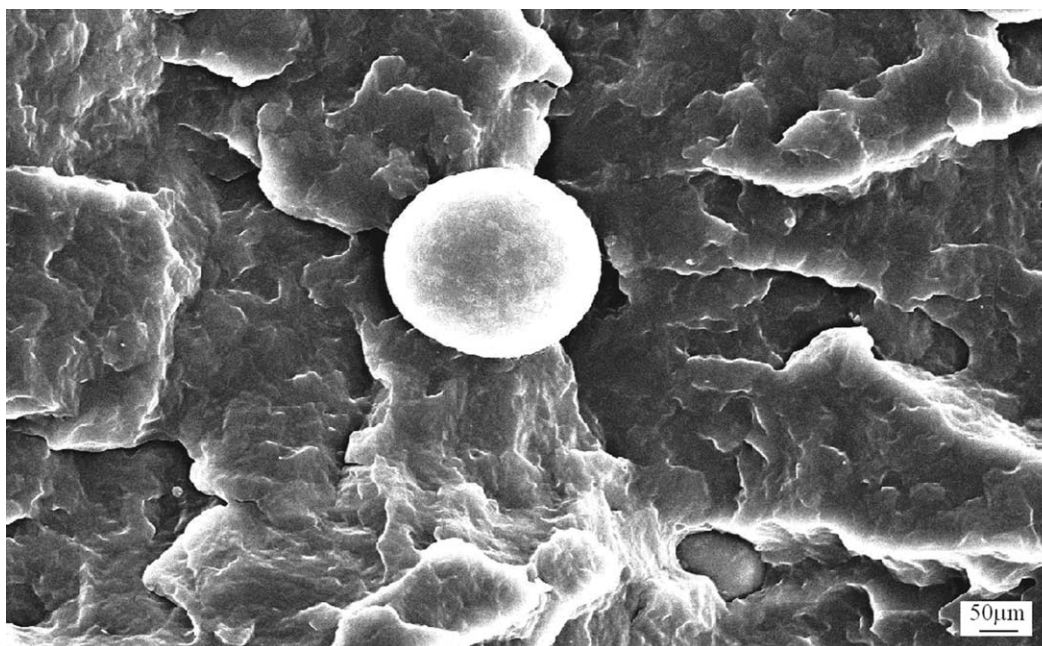


**Figure 11** SEM photograph of the impact-fractured surface of the PP/GB2 system ( $\phi_f = 18\%$ ).

was generally uniform, and the fracture surface was very uneven. Namely, there were a number of small terraces on the impact-fractured surface, and these small terraces absorbed relevant impact deformation energy or fracture energy; this led to an obvious improvement in the impact-fracture toughness of the composite systems (see Figs. 5 and 6). Moreover, one can see by comparing Figures 9 and 10 that the distribution of GBs in the matrix for the PP/GB1 system was better than that for the PP/GB2 system.

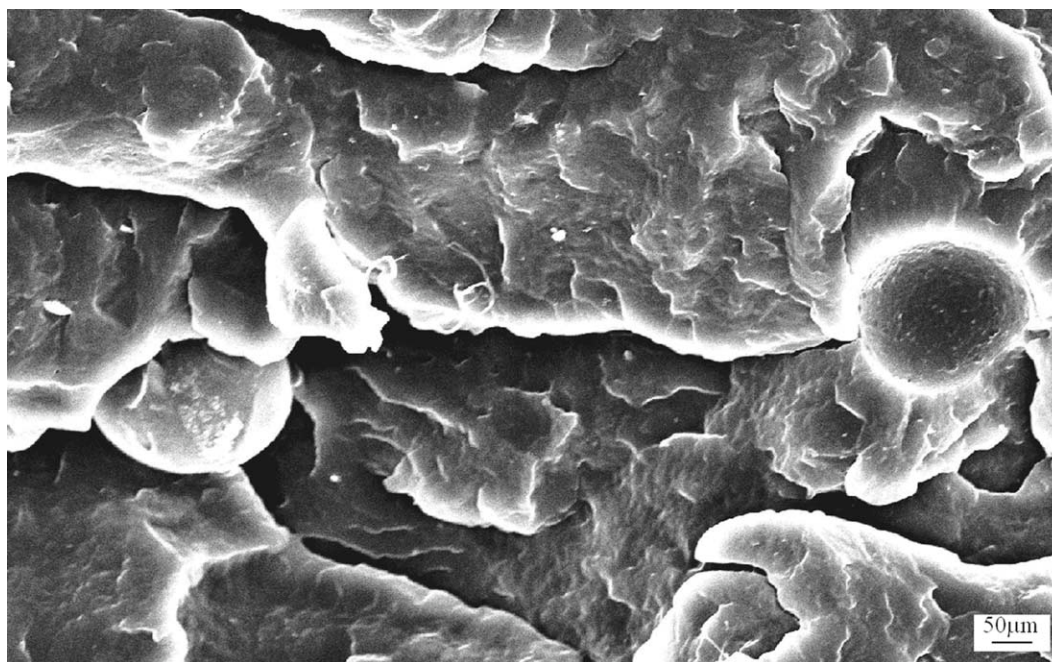
This should be one of the reasons that the impact strength for the PP/GB1 system was higher than that for the PP/GB2 system.

Figure 10 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB1 composite when the filler volume fraction was 18%. Figure 11 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB2 composite when the filler volume fraction was 18%. It was also observed that there was a slight aggregation



**Figure 12** SEM photograph of the impact-fractured surface of the PP/GB1 system ( $\phi_f = 14\%$ ).





**Figure 13** SEM photograph of the impact-fractured surface of the PP/GB2 system ( $\phi_f = 14\%$ ).

phenomenon of the GBs in the PP matrix, and the fracture surface was relatively smooth in a comparison of Figures 9 and 10. As stated previously, in the case of a high concentration of the filler particles, the microcracks and plastic deformation produced in the composite materials was very large under impact load; thus, they might have been considered as macroscopic stress cracks, resulting in obvious reduction of the toughness in this case (see Figs. 5 and 6).

Figure 12 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB1 composite when the filler volume fraction was 14%. It can be seen that the interfacial adhesion between the GBs and the matrix was good, and the impact load could be effectively transferred in this case; thus, the matrix around the particle generated the stress yield, and the relevant plastic deformation and stress-whitening phenomenon were produced to absorb the impact energy. Figure 13 is the SEM photograph of the V-notched Izod impact-fractured surface of the PP/GB2 composite when the filler volume fraction was 14%. The interfacial adhesion between the GBs and the matrix for the PP/GB2 system was relatively poor compared with that of the PP/GB1 system shown in Figure 12, and the matrix around the inclusion presented fracture phenomenon. However, there was still the stress-whitening zone at the crack acme; this indicated that there were some plastic deformations before fracture. In other words, there were still certain toughening effects for this composite system under the test conditions, and the impact strength was higher than that of the unfilled PP (see Figs. 5 and 6).

## CONCLUSIONS

The mechanisms of the stiffening and toughening phenomenon of the GB content and surface treatment on the filled PP composites under the experimental conditions were proposed. The major toughening mechanisms for these composites might have been that the matrix around the inclusions yielded first under impact load because of stress concentration to generate plastic deformation and absorbed the relevant impact deformation or fracture energy. The relative elastic modulus increased nonlinearly with an increase of  $\phi_f$ . Both  $\sigma_{IV}$  and  $\sigma_{CV}$  increased with an increase of  $\phi_f$  when  $\phi_f$  was less than 11% and then decreased. Namely, the toughening effect of these composite systems was significant under the experimental conditions, and the interfacial adhesion between the GBs and the matrix was roughly good. The GB surface treated with the silane coupling agent was beneficial to improving the interfacial adhesion and the dispersion and distribution of the particles in the PP matrix; hence, the mechanical properties, including the tensile and impact properties of the composites, were better than those of the composites filled with untreated GBs under the same conditions.

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