

The effects of particle size and content on the thermal conductivity and mechanical properties of Al₂O₃/high density polyethylene (HDPE) composites

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Abstract. The influences of filler size and content on the properties (thermal conductivity, impact strength and tensile strength) of Al₂O₃/high density polyethylene (HDPE) composites are studied. Thermal conductivity and tensile strength of the composites increase with the decrease of particle size. The dependence of impact strength on the particle size is more complicated. The SEM micrographs of the fracture surface show that Al₂O₃ with small particle size is generally more efficient for the enhancement of the impact strength, while the 100 nm particles prone to aggregation due to their high surface energy deteriorate the impact strength. Composite filled with Al₂O₃ of 0.5 μm at content of 25 vol% show the best synthetic properties. It is suggested that the addition of nano-Al₂O₃ to HDPE would lead to good performance once suitably dispersed.

Keywords: polymer composites, mechanical properties, thermal conductivity

1. Introduction

Polymers with excellent electrical insulation have been extensively used as packing materials in electrical devices due to their good process ability, light weight and low cost. Nevertheless, the thermal conductivity of polymers are generally very low (i.e. 0.10–0.25 W/m¹·K⁻¹), and it has been widely recognized that the thermal conductivity of these polymers has to be enhanced to lower the energy loss and increase the stability of the devices. With the increasing demand for high density power and energy transmission of electronic devices, fabrication of insulating polymer materials with high thermal conductivity has become crucial [1–4].

High crystallization and orientation of polymer can greatly enhance the thermal conductivity along the orientation direction, but they usually have difficul-

ties of processing [5]. Blending of polymer with inorganic fillers is an effective and convenient way to enhance the polymer thermal conductivity while maintaining the electrical insulation, such as polymer composites with boron nitride (BN) [6, 7], aluminum nitride (AlN) [2, 3], silicon nitride (Si₃N₄) [8, 9], alumina [10], silicon carbide (SiC) [11] and silica (SiO₂) etc. [12] have been investigated. In the inorganic filler/polymer composites, the thermal conductivity increases with the filler content. Very high filler loading is often used to obtain high thermal conductivity. However, it seriously alters the polymer component and can form agglomerates, which induces stress concentration and decrease of the tensile strength, modulus and ductility of the material [13], so the practical application of thermal conductive polymer composites is limited.

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Filler size and shape are also important factors for the thermal conductivity and mechanical properties [14, 15]. Zhou *et al.* [16] used Al₂O₃ with different size to fill silicone rubber, and reported that nano-sized Al₂O₃ composite exhibited higher thermal conductivity and mechanical properties than the micro-sized one. The influence of hybrid fillers was also investigated [17, 18]. However, reports on how to enhance the thermal conductivity and mechanical properties of the polymer composites simultaneously are still quite limited.

The particle size and content in the composite determine the average interparticle distance [19, 20], which is close related to the thermal conductivity and mechanical properties. At the same particle content, smaller particle size leads to lower interparticle distance and more chances for the formation of thermal conductive ‘pathway’ [21–23]. The particle size and content affect the interparticle distance and the stress state of the matrix polymer surrounding the voids. When the average interparticle distance is in a suitable range, extensive plastic deformation in the matrix can be easily induced [24–26]. So the optimization of the particle size and content can be a convenient and feasible way to prepare composites with good synthetic properties. In this paper, Al₂O₃/HDPE composites with different alumina particle sizes (diameter of 10, 4.7, 0.5 μm and 100 nm, denoted as A10, A4.7, A0.5 and A0.1) were prepared. The influence of alumina content and particle size on thermal conductivity, impact strength and tensile strength are studied. The synthetic properties of the composites are optimized and the mechanism is also discussed.

2. Experimental

2.1. Materials

The polymer used in this study was high density polyethylene (HDPE) 5000S (SinoPec Beijing Yansan Petrochemical Co. Ltd, Beijing, China). It had a density of 0.95 g/cm³ and a melt flow index

of 0.9 g/10 min (2.16 kg at 190°C). Four types of alumina particles with average diameters of 10, 4.7, 0.5 μm and 100 nm (denoted as A10, A4.7, A0.5 and A0.1 respectively, >99 wt% purity) were used as fillers. The suppliers and parameters of the alumina particles were listed in Table 1. All of the alumina particles were of α-crystalline and the thermal conductivity was about 30 W/(m·K). The alumina particles were added to the HDPE without surface pretreatment.

2.2. Sample preparation

The blends of HDPE with various alumina particles were prepared by a two-roll mill (X(S) K-160, Double Elephant Group Company, Jiangsu, China) for 20 min. The temperature of the front roll and back roll were 140 and 130°C respectively. After extrusion, the samples were cut into small pieces.

The samples for thermal conductivity and mechanical property tests were prepared by injection molding (SZ-68/400, Liuzhou Rubber and Plastic Machinery Factory, Guangxi, China) at 200°C. The samples for thermal conductivity measurement were square bars of 50 mm in length and 4 mm in thickness. Tensile and impact bars were prepared according to ASTM D638 Type I and ASTM D256.

The maximum contents of alumina A4.7 and A0.5 in the composites were 50 vol%, but those of A10 and A0.1 were 30 vol%. The reasons for the difficulties of processing for 10 and 0.1 μm particle composites are different. The surface area of the 0.1 μm alumina particles is very large. When filler content is higher than 30 vol%, it is very difficult for HDPE to wet the surface of all nano-particles, and constrained the blending. On another hand, the 10 μm particles increase the roughness of the composite surface. When the 10 μm alumina content is higher than 30 vol%, the surface of the composite becomes too rough to measure the thermal conductivity for it can't make a good contact with heat source. The compositions were represented by the

Table 1. The suppliers and data of alumina fillers

| Filler code | Supplier | Surface area* [m ² /g] | Mean particle size* [μm] | Geometrical standard deviation* |
|-------------|---|--------------------------------------|-----------------------------|---------------------------------|
| A10 | Shanghai Yuejiang Titanium Chemical Manufacture Co. Ltd. CHN, Shanghai, China | 0.6 | 10.0 | 1.5 |
| A4.7 | Fujian Johnson Mining Co. Ltd. CHN, Xiamen, China | 1.5 | 4.7 | 2.0 |
| A0.5 | Wan Jing New Material Co. Ltd. CHN, Hangzhou, China | 5.0 | 0.5 | 2.8 |
| A0.1 | Wan Jing New Material Co. Ltd. CHN, Hangzhou, China | 12.5 | 0.1 | 3.6 |

*The values were given by the suppliers

alumina volume fraction. The real composition of the composites was measured by the calcination method, and the difference between the theoretical composition and the real one is less than 2%.

2.3. Characterization

2.3.1. Thermal conductivity

The through-plane thermal conductivity was measured at 30 and 50°C with thermal conductivity analyzer (HC-110, EKO Instruments Co. Ltd, Sasazuka Shibuya-ku, Japan).

2.3.2. Impact and tensile test

The notched Izod impact strength was measured on a ceast pendulum impact strength tester CSI-137C at 23°C (Wuzhongshi Material Tester Co. Ltd, Ningxia, China), according to ASTM D256. The drop velocity was 3.5 m/s and the testing results were the average of ten parallel measurements. The tensile measurements were carried out at 23°C using an Instron 3365 universal materials testing machine (Instron Corporation, Massachusetts, America), according to ASTM D638. The average values of the mechanical properties in this study were obtained by five independent measurements.

2.3.3. Scanning electron microscope (SEM)

The as prepared composites were cryogenically fractured in liquid nitrogen. Both the cryogenically fractured and impact fractured surfaces of composites were inspected by SEM (JSM-6700F, Japan Electron Optics Laboratory Co. Ltd, Tokyo, Japan) at an acceleration voltage of 3 kV. The surfaces of the samples were coated with a conductive platinum layer before inspection.

3. Results and discussion

3.1. Dispersion of Al₂O₃ in the HDPE matrix

The dispersion of the alumina particles differs with particle content and particle size, which determines the properties of the composites [27, 28]. The composites cryogenically fractured surfaces were inspected by SEM (Figure 1 and Figure 2).

For a certain size of alumina particle, the dispersion in the composite changes with content. Their trends are similar when the particle size $\geq 0.5 \mu\text{m}$. The composite of A4.7/HDPE is taken as a typical example to show the influence of content on the dispersion (Figure 1a–c). When the alumina content is of

5 vol% (Figure 1a), the alumina particle is dispersed in the matrix without serious aggregation. The smooth holes on the surface of the matrix should be the spaces occupied by the alumina particles. The average interparticle distance is about several microns and the alumina particles are mostly separated without contacting each other. When the alumina content increases to 15 vol% (Figure 1b), the average interparticle distance gets smaller, and some agglomerates are formed. When the alumina content increases to 30 vol% (Figure 1c), the average interparticle distance gets even smaller and the amount of agglomerates increased, resulting in the formation of the local alumina network. Of course, inter polymer layers are existed among the particles. The red lines were added to the SEM pictures to indicate the local Al₂O₃ network. The size of the local networks increases as the filler content increasing.

The dispersion of the particles differs with particle size when the content is the same. Figure 1(c–f) show the SEM micrographs of the composites with particle size of 4.7, 10 and 0.5 μm respectively at 30 vol% alumina content. The average interparticle distance reduces with the decrease of particle size, and smaller particle size affords more connected alumina particles. Alumina particles in A10/HDPE (30 vol%) are mostly dispersed separately (Figure 1d), while connected alumina particles can be found for A4.7/HDPE at the same content. In A0.5/HDPE (30 vol%) composite, more connected alumina particles are formed.

The dispersion of alumina particles in A0.1/HDPE is somehow quite different from others that the dispersed and aggregated particles are coexisted (Figure 2a). In most area, 100 nm particles dispersed uniformly, but in some parts the particles aggregated up to several micro-meters due to the large specific surface area and high surface energy. The enlarged SEM pictures of the two parts are shown in Figure 2b and Figure 2c. Figure 1c, e, f and Figure 2b show that the number of the local alumina networks increase as the decreasing of the particle size.

3.2. Thermal conductivity of the composite

Figure 3a shows the thermal conductivity curves with alumina volume content.

Thermal conductivities of all composites increase with filler content, and the trends are similar. The

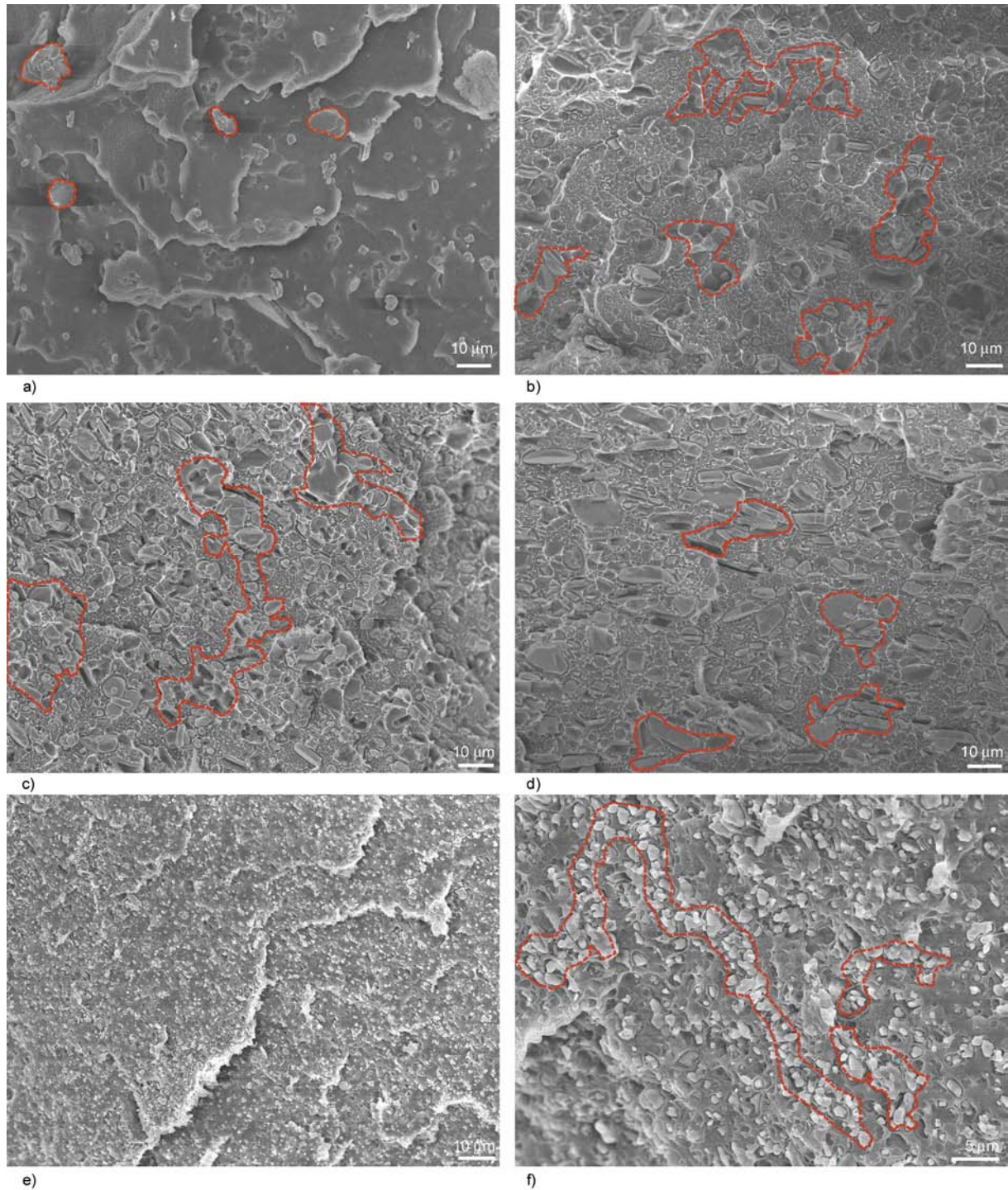


Figure 1. SEM micrographs of the cryogenically fractured surfaces of $\text{Al}_2\text{O}_3/\text{HDPE}$ composites: (a) A4.7/HDPE (5 vol%); (b) A4.7/HDPE (15 vol%); (c) A4.7/HDPE (30 vol%); (d) A10/HDPE (30 vol%); (e) A0.5/HDPE (30 vol%); (f) A0.5/HDPE (30 vol%) in high magnification. The percentages refer to the alumina volume fraction in the composites. The red lines are added to emphasize the local network of the samples.

curves can be modeled into three stages (Figure 3b). The thermal conductivity increases more rapidly in second stage than in the first and third stages. The turning point from stage 1 to stage 2 appears at lower alumina content for composites with the smaller particle size. Different from others, the increase of

the thermal conductivity of the A10/HDPE only shows the character of the first stage up to the alumina content of 30 vol%.

The isolated particles have little effect on the enhancement of the thermal conductivity until the particle form continuous pathway through the mate-

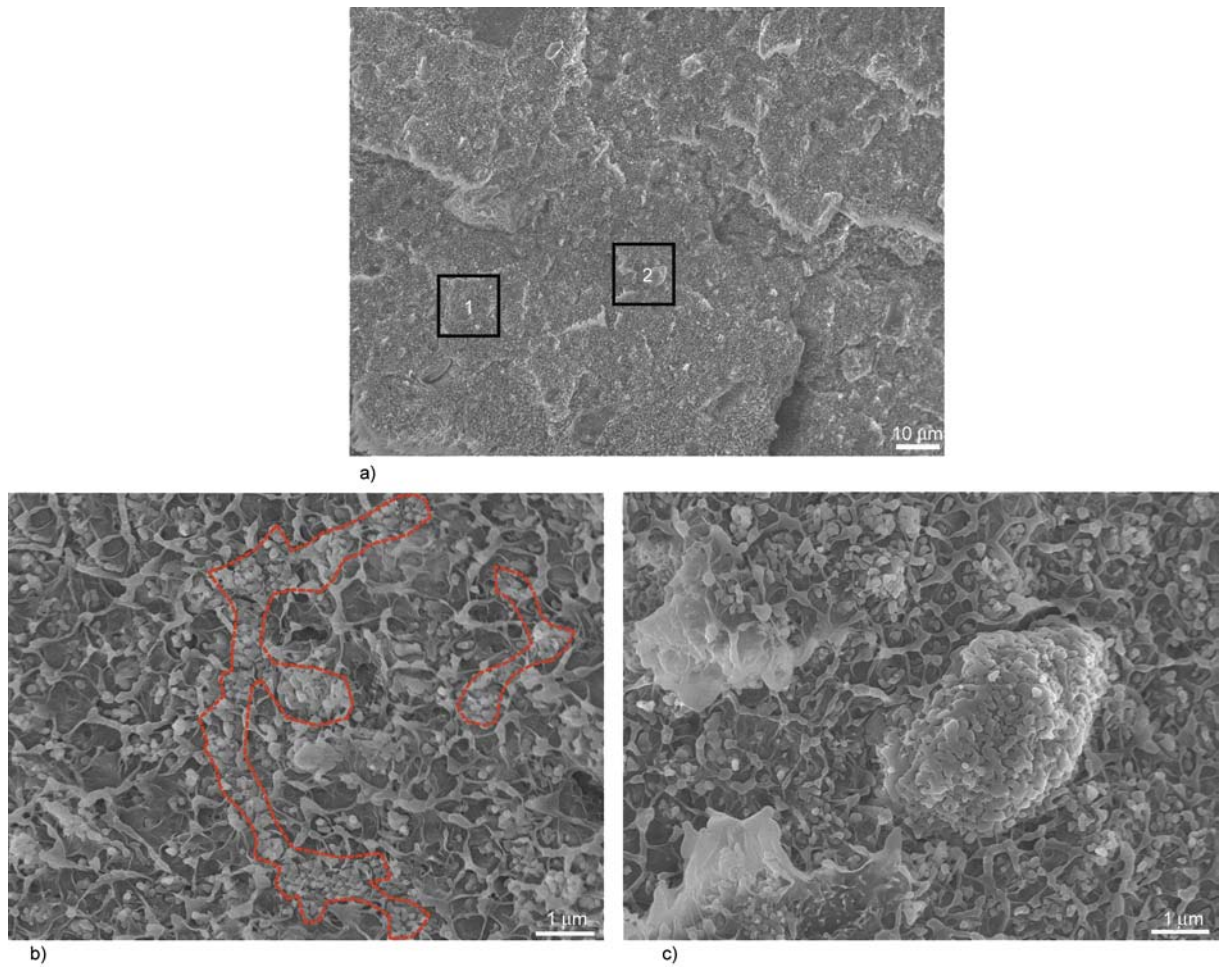


Figure 2. (a) SEM micrographs of the cryogenically fractured surface of A0.1/HDPE (30 vol%); (b) magnified image of part (1) in (a); (c) magnified image of part (2) in (a). The percent is referred to the alumina volume fraction in the composites. The red lines are added to emphasize the local network of the samples.

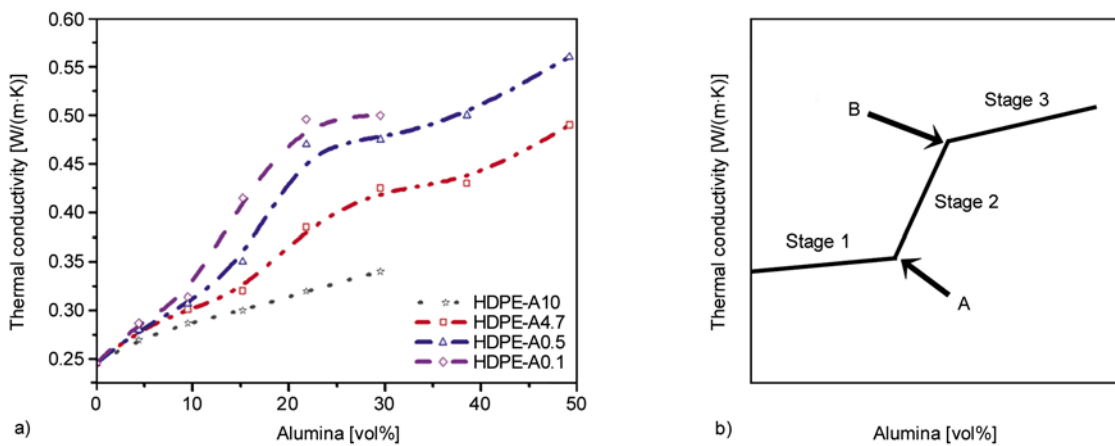


Figure 3. (a) Dependence of thermal conductivity with volume percentage for different particle size in alumina/HDPE composite; (b) Schematic illustration for the three stages of thermal conductivity variation with alumina volume fraction

material, which can lead to the abrupt increase of the thermal conductivity. The first stage should correspond to the composites, in which most alumina dispersed separately, so the thermal conductivity increases slowly with content. The abrupt increase

of conductivity of the second stage indicates the formation of the continuous pathway [29, 30]. The continuous alumina pathway is a network in 3D and the SEM picture is in 2D. Although the abrupt increase of the thermal conductivity indicates the

formation of the alumina pathway, the continuous pathway can't be observed in the SEM pictures [31].

In the second stage, the increase of alumina content increases the number of the alumina pathways, so the rapid increase of the thermal conductivity was continued. After that, further increase of the alumina content has little effect on the increase of the pathway [32], so the increase rate of thermal conductivity slows down, which starts the third stage. With a given particle content, the smaller the particle size, the higher the composite's thermal conductivity would be. The values of thermal conductivity (K_s) with different particle size at alumina content 30 vol% comply with the following order: $K_{0.1}$ ($0.49 \text{ W}/(\text{m}\cdot\text{K})$) > $K_{0.5}$ ($0.47 \text{ W}/(\text{m}\cdot\text{K})$) > $K_{4.7}$ ($0.42 \text{ W}/(\text{m}\cdot\text{K})$) > K_{10} ($0.37 \text{ W}/(\text{m}\cdot\text{K})$). $K_{0.1}$ and $K_{0.5}$ at alumina content 30 vol% is about two times of that of HDPE ($K_{\text{HDPE}} = 0.249 \text{ W}/(\text{m}\cdot\text{K})$).

3.3. Mechanical properties of the composite

3.3.1 Impact strength

The variations of impact strength of composite with alumina volume content are shown in Figure 4. The toughening efficiencies of the alumina particles are dramatically different. The $\text{Al}_2\text{O}_3/\text{HDPE}$ compos-

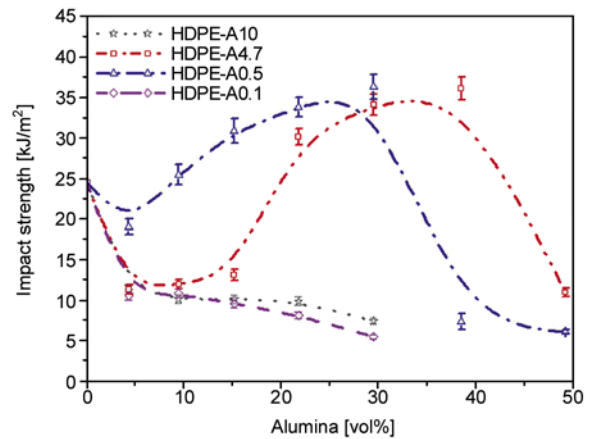


Figure 4. Variations of the composite impact strength with alumina volume fraction

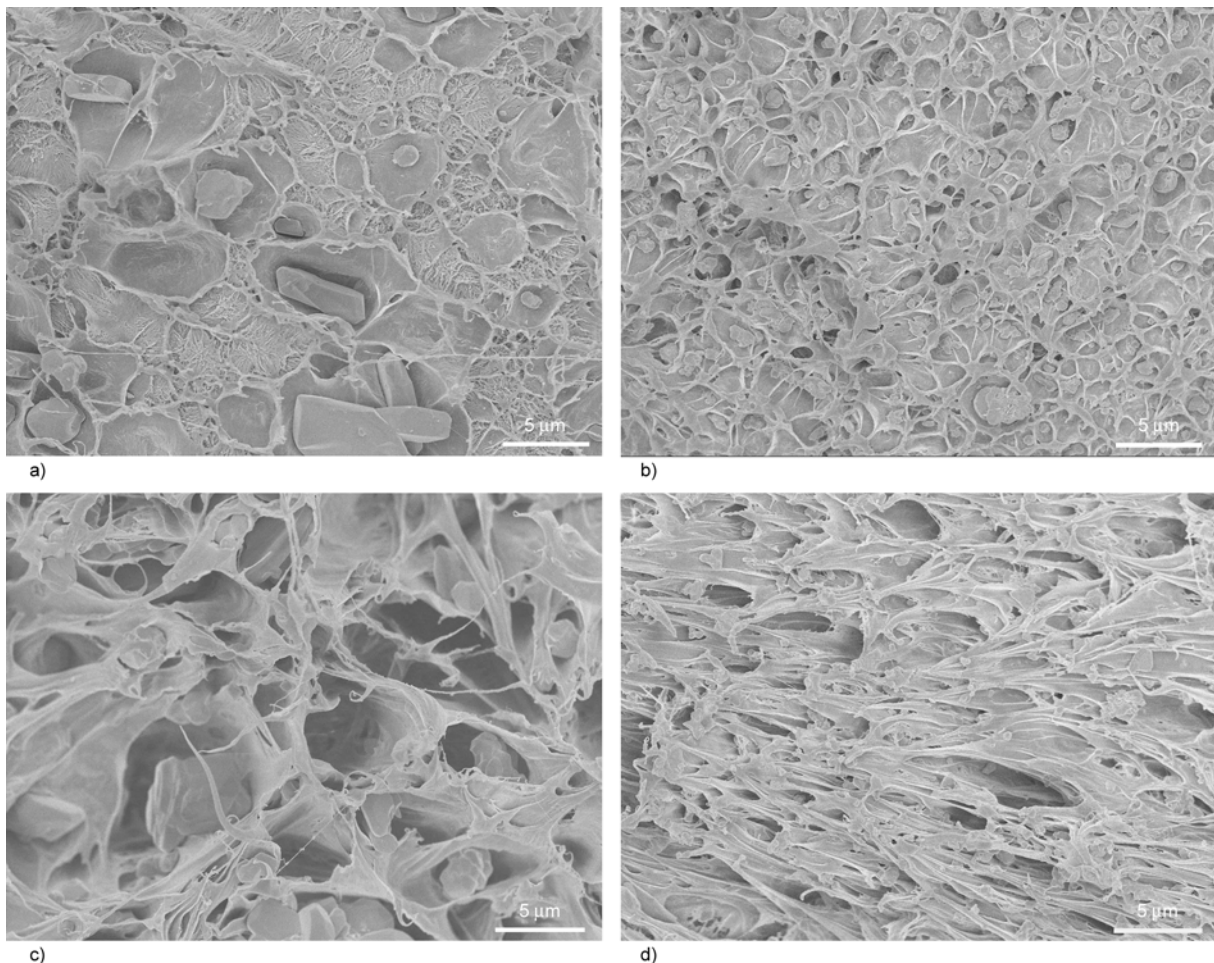


Figure 5. SEM micrographs of the fracture surfaces of Izod samples: (a) A4.7/HDPE (5 vol%); (b) A0.5/HDPE (5 vol%); (c) A4.7/HDPE (30 vol%); (d) A0.5/HDPE (30 vol%); The impact direction was from right to the left. The percents refer as the alumina volume fraction in the composites.

ites can not be toughened by A10 and A0.1 with the content up to 30 vol%. The composites can be toughened by A4.7 and A0.5 when the contents are in the range around 10–30 vol%. The maximum impact strength of A0.5/HDPE is about 36 kJ/m², which is 1.44 times of that of the pure HDPE.

There are several mechanisms for toughening of polymer. For the inorganic particles toughening polymer, at least three factors are necessary: inherent ductility of the matrix, weak interphase supporting the filler/matrix debonding and suitable interparticle distance. The stress concentration first leads to debonding of the filler particles and voids formation. The particle content affects the interparticle distance and the stress state of the matrix polymer surrounding the voids [19, 20].

At low alumina content, the interparticle distance is long and the interparticle matrix ligament lies in plane strain state which is hard to yield, as a result the impact strength cannot be improved. When the alumina content increases and the interparticle dis-

tance reach a suitable range, the interparticle matrix ligament lies in plane press state, which can be plastic yield easily and the impact strength can be improved. But when the alumina content is too high, the interparticle distance becomes too small and even leads to large size agglomerates, which provide convenient triggers for brittle behavior.

The morphologies of the fracture surfaces of A4.7/HDPE and A0.5/HDPE after impact test are shown in Figure 5. When the alumina content is 5 vol%, the surfaces are generally even and smooth. The plastic yield increases with the increase of filler content or the decrease of particle size as shown in Figure 5c–d. The increase of particle content decreases the average interparticle distance of the matrix and results in the apparently plastic yield, which leads to high impact strength of the composites. Compared to that of A4.7, A0.5 is more efficient in toughening the composite. As shown in Figure 5d, stable fibril structure was formed in the A0.5/HDPE (30 vol%) sample at the particle–polymer interface.

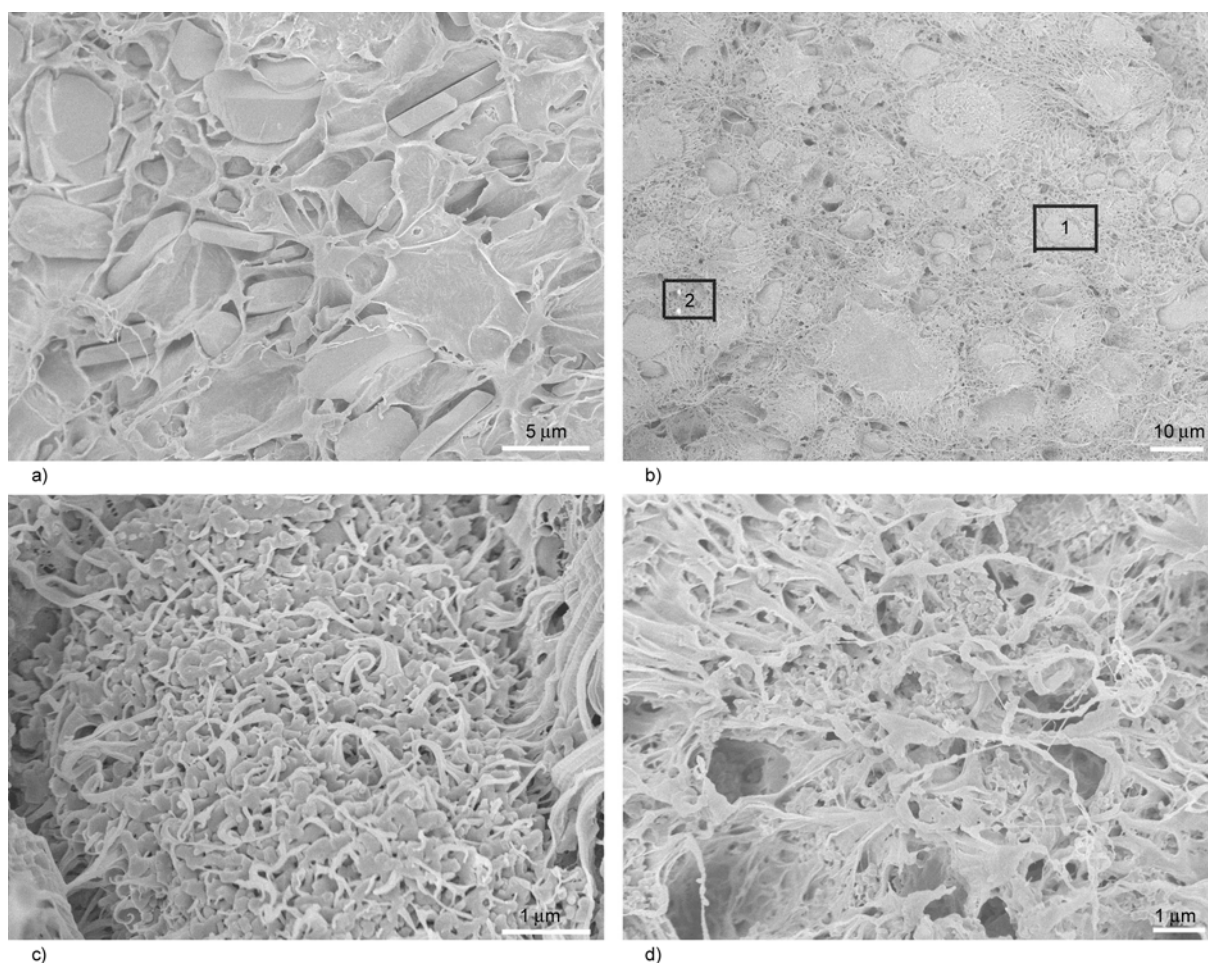


Figure 6. SEM micrographs of the fracture surfaces of Izod samples: (a) A10/HDPE (30 vol%); (b) A0.1/HDPE (30 vol%); (c) magnified image of part (1) in (b); (d) magnified image of part (2) in (b). The percents refer as the alumina volume fraction in the composites.

The SEM micrographs of Figure 6 reveal that the A10, A0.1/HDPE are brittle fractured. Most parts of the fracture surfaces of A10/HDPE are even and smooth, and only a small part of the HDPE at interfaces is microductile. It indicates that because of its size, A10 particles serve as the initiation sites for the brittle fracture of the composite [33].

A0.1 does not toughen the composite efficiently because of the aggregation shown in Figure 6b and Figure 6c. In this case, when the amount of aggregated clusters (Figure 6b, part (1)) is large enough, the fracture mode would be a brittle fracture. Nevertheless, in the Figure 6b, part (2) area without large alumina clusters, the HDPE matrix forms fibrils structure possessing plastic deformation as shown in Figure 6d. The result suggests that if the nano-alumina particles can be uniformly dispersed in matrix they could be used as effective toughening agent.

3.3.2 Tensile strength

The tensile strength of composites increases with the decrease of the alumina particle size (Figure 7). When the A0.1 content is 30 vol%, the tensile strength can be enhanced to 1.12 times of the pure HDPE.

Compared to the thermal conductivity and impact strength, tensile strength is more dependent on the filler-matrix interaction. Strong interaction benefits the tensile stress transfer from matrix to filler, thus reinforces the composite. The interfacial action increases with the decrease of the particle size.

In A0.1/HDPE, the results of the impact and tensile strength seem controversial because the agglomer-

ates play different roles in these loading processes. The agglomerates can bring strong interaction between the matrix and fillers, because some of the matrix is wrapped into it (Figure 6c), which doesn't favor the debonding. Furthermore, the agglomerates provide defects that can develop quickly and trigger brittle fracture. For tensile strength, the interfacial action is favored for the stress transfer from the matrix to the alumina particles, so the tensile strength can be enhanced.

4. Conclusions

In this study, the thermal conductivity and mechanical properties (impact strength and tensile strength) of 4 types of Al₂O₃/HDPE composites with alumina filler size from 10 μm to 100 nm were investigated. The alumina particle with smaller particle size can form thermal conductive alumina pathway more easily and has higher toughening efficiency for A10, A4.7 and A0.5. However, nano alumina particles can form aggregation and destroy the toughness as that of A0.1/HDPE in this study. In the tensile process, the size of voids decreases with the alumina particle size, thus alumina with smaller particle size can bring higher tensile strength. A0.5/HDPE at alumina content of 25 vol% owns the best integrated thermal conductivity and mechanical properties (impact strength and tensile strength). Its thermal conductivity is nearly two times of the pure HDPE, and the impact strength and tensile strength is 1.44 times and 95% of the pure HDPE respectively. This study would provide practical and theoretical supports for thermal conductive polymer composites with good synthetic properties.

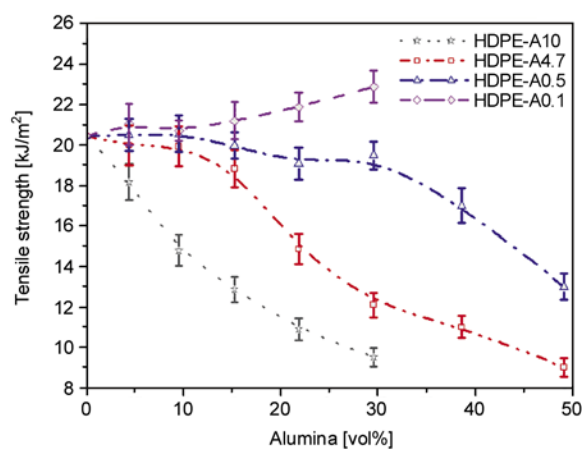


Figure 7. Variations of the composite tensile strength with alumina volume fraction

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