

Effect of Al₂O₃ Fibers on the Thermal Conductivity and Mechanical Properties of High Density Polyethylene with the Absence and Presence of Compatibilizer

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ABSTRACT: Alumina (Al₂O₃) fiber/high density polyethylene (HDPE) composites were prepared by molding injection with or without compatibilizer, in which, maleic anhydride-grafted polyethylene (PE-g-MA) and acrylic acid-grafted polyethylene (PE-g-AA) were used as the compatibilizers. The thermal conductivities of the composites were anisotropic and the conductivities in the injection direction of the samples were higher than those in perpendicular direction of the injection. The anisotropic thermal conductivity for Al₂O₃/PE-g-AA/HDPE was the most obvious and this composite also gave the best mechanical performance.

The SEM and DMA test revealed that PE-g-AA was more effective than PE-g-MA in improving the matrix–filler interaction. The high interfacial interaction was more favorable for the viscous flow-induced fiber orientation, which resulted in the largest anisotropic degree of thermal conductivity of the Al₂O₃/PE-g-AA/HDPE among the studied composite. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 4874–4881, 2012

Key words: thermal conductive composites; injection molding; interfacial interaction; orientation; mechanical properties

INTRODUCTION

Polymers with excellent electrical insulation have been extensively used as packing materials in electrical devices because of their good process ability, light weight, and low cost. However, with the miniaturization and increasing power of equipment, the dissipation of heat has become a critical problem because of the low thermal conductivity (i.e., 0.10–0.25 W m⁻¹ K⁻¹) of polymers, which limits the performance improvement and causes the energy waste. It has been widely recognized that the thermal conductivities of the polymer-packing materials have to be enhanced.^{1,2}

Blending of polymer with inorganic thermal conductive fillers is an effective and convenient method to enhance the polymer thermal conductivity, but it is still a great challenge to obtain thermal conductive composites with good mechanical properties. The filler shape, size, and concentration influence the mechanical properties and the thermal conductivity of the composites.^{3,4} It has been found that the thermal conductivity of composites can be enhanced substantially if the fillers orient preferably along the

heat flow direction. Because of the geometry anisotropy of the fiber, the fiber-shaped fillers are more preferred to be oriented in polymer matrix under certain conditions than fillers of spherical shape.^{5,6} It was reported that the thermal conductivity of the aligned carbon fiber/epoxy composite could reach as high as 695 W m⁻¹ K⁻¹.⁷ For these reasons, the inorganic fiber-shaped fillers, such as carbon nanotube, carbon fiber, and alumina (Al₂O₃) fiber, are widely used in the preparation of the thermal conductive polymer composites.^{8–11}

It is well known that the fiber–matrix interaction has a significant effect in determining the properties of polymer composites. It was reported that the moderately improved interfacial adhesion was favorable for heat energy transfer because of the reduced interfacial thermal resistance in inorganic particle/polymer thermal conductive composite.^{12–14} However, research on the interfacial interaction effects on the thermal conductivities of the fiber/polymer composites has been seldom reported.

In addition, the inorganic fibers can also be used as effective reinforcements for the polymer.^{15–18} A good fiber–matrix interfacial interaction is essential to transfer the stresses from the matrix to the fibers and thus improve the strength properties of the composite.^{19,20} So the study of the effect of interfacial interaction on the properties of fiber/polymer composite is very important for the preparation of the

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thermal conductive polymer composites with good mechanical properties.

In this study, Al₂O₃ fiber/high density polyethylene (HDPE) composites were prepared and two types of compatibilizer (PE-g-MA and PE-g-AA) were used to modify the matrix-fiber interaction. The scanning electron microscope (SEM) and dynamic mechanical analysis (DMA) were conducted to elucidate the matrix-filler interaction. The orientation of fibers in the composites with different matrix-filler interaction was characterized by SEM and statistical method. The influences of matrix-filler interaction on the thermal conductivity and mechanical properties of Al₂O₃ fiber/HDPE composites were discussed.

EXPERIMENTAL

Materials

The polymer used in this study was HDPE 5000S (SinoPec Beijing Yansan Petrochemical, Beijing, China), with a density of 0.95 g cm⁻³ and a melt flow index of 0.9 g/10 min (2.16 kg at 190°C). The commercially supplied Al₂O₃ fiber with average diameter of 10 μm (Qin Xing Xian Wei, Zhejiang, China) was used as thermal conductive fillers. The Al₂O₃ fiber is of α-crystalline and the thermal conductivity is about 30 W m⁻¹ K⁻¹. The acrylic acid-grafted polyethylene (PE-g-AA) with 2 wt % acrylic acid (AA) was supplied by Shanghai Sunny New Technology (Shanghai, China). The compatibilizer maleic anhydride (MA)-grafted polyethylene (PE-g-MA) with 2 wt % MA was made in our laboratory.

Sample preparation

The blends of HDPE/Al₂O₃ fibers with and without compatibilizers 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 rolling, the samples were cut into small thin pieces.

The contents of Al₂O₃ fibers in composites were 5, 10, 25, 40 and 50 wt %, respectively. The real composition was measured by calcinations of the sample and the difference between the theoretical and real composition was less than 2%. The concentration of the compatibilizer was an important parameter for the properties of the composites.^{21,22} In this study, the optimal concentration of the compatibilizer was 5 wt % of the matrix, which had been evaluated previously by mechanical property experiments in our laboratory.

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. Ten-

sile and impact bars were prepared according to ASTM D638 Type 1 and ASTM D256.

Characterization

Thermal conductivity

Thermal conductivity of the composites was measured using thermal conductivity analyzer (HC-110, EKO Instruments, Sasazuka Shibuya-ku, Japan), which was fabricated according to the ASTM-C518. The temperatures of the two sides of the samples were set as 30 and 50°C, respectively. The samples were clamped with good contact with the calorimeters. The average heat flux generated by temperature gradient was recorded when thermal equilibrium was achieved. It was assumed that the heat flow was one dimensional in the perpendicular direction and that no heat loss occurred in the lateral direction. The thermal conductivities of the samples were then calculated from Fourier's law.²³ The tolerance of the measurement is controlled within 5% and obtained by at least three samples.

The square samples for the thermal conductivity test (12 mm in side and 10 mm thick) were prepared by injection molding. The thermal conductivity in the injection and perpendicular direction can be obtained by altering the interfaces contacting with calorimeters.

Mechanical properties

The notched Izod impact strength was measured on a pendulum impact strength tester CSI-137C at 23°C (Wuzhongshi Material Tester, Ningxia, China), according to ASTM D256. The drop velocity was 3.5 m s⁻¹ and the testing results were the average values of 10 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 value of the tensile strength for each composition was obtained by five independent measurements.

The composite flexural strength was determined at 23°C using an Instron 3365 universal materials testing machine (Instron Corporation, Massachusetts, America), according to ASTM D-790-00 A test method. A span of 63 mm was used in a 5-kN load cell. The load was placed midway between the supports. The crosshead speed applied was 20 mm min⁻¹. The flexural strength was determined for five samples of each composition.

Scanning electron microscope

The prepared composites were cryogenically fractured in liquid nitrogen. The fracture surfaces show the central area of the cross-section of the molded

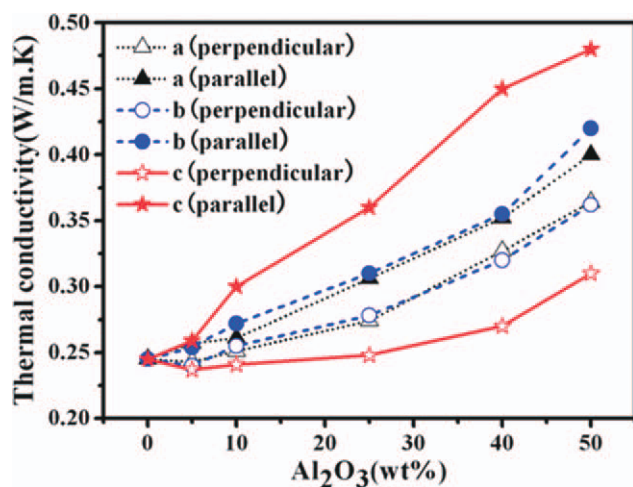


Figure 1 Dependences of thermal conductivity on alumina weight percentage in two directions: (a) Al₂O₃/HDPE; (b) Al₂O₃/PE-g-MA/HDPE; (c) Al₂O₃/PE-g-AA/HDPE. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

samples. The numbers of fibers in these two directions were counted manually by the assistance of Photoshop software. At least three SEM pictures were used to collect the fiber dispersion for one sample. Both the cryogenically fractured and impact fractured surfaces of composites were observed by SEM (JSM-6700F, Japan Electron Optics Laboratory, Tokyo, Japan) at an acceleration voltage of 3 kV. The surfaces of the samples were coated with a conductive platinum layer before observation.

Dynamic mechanical analysis

The dynamic mechanical properties were examined by single cantilever mode using dynamic mechanical analyzer (Q800, TA Instruments, USA) on rectangle specimens, which were taken from the impact bars (length 18 mm, width 12.5 mm, and thickness 2.3 mm). The loss modulus E'' was measured in the temperatures ranging from -130 to 100°C at a constant frequency of 1 Hz and a heating rate of 3°C min^{-1} .

RESULTS AND DISCUSSION

Thermal conductivity and mechanical properties of the composites

The dependences of the thermal conductivity with Al₂O₃ content for different composites are shown in Figure 1. The thermal conductivity parallel to the injection direction is higher than that perpendicular to the injection direction for all samples. The thermal conductivities all increase with the Al₂O₃ content, but the increase rate in the injection direction is greater, which is identical with the thermal conductivity in the published reports about the

fiber/polymer composite.^{24,25} Thermal conductivity of Al₂O₃/PE-g-AA/HDPE in the injection direction and perpendicular direction is the highest and lowest, respectively. The thermal conductivity of Al₂O₃/HDPE and Al₂O₃/PE-g-AA/HDPE with 50 wt % of Al₂O₃ in the injection direction is 0.40 and $0.47 \text{ W m}^{-1} \text{ K}^{-1}$, respectively, which is 66 and 96% higher than that of the HDPE. The thermal conductivities of Al₂O₃/PE-g-MA/HDPE are close to those of Al₂O₃/HDPE in both directions.

Figure 2 presents the dependences of the mechanical properties of the composites with Al₂O₃ weight percentage. It is found that the impact strength decreases with the Al₂O₃ content. The impact strength of Al₂O₃/PE-g-AA/HDPE is the lowest at the same filler concentration [Fig. 2(a)].

In contrast to the impact strength, the tensile strength and flexural strength of the composites increase with the Al₂O₃ fiber loading [Fig. 2(b,c)]. The tensile strength and flexural strength of composites at the same Al₂O₃ fiber content comply with the following order: Al₂O₃/PE-g-AA/HDPE > Al₂O₃/PE-g-MA/HDPE > Al₂O₃/HDPE. Al₂O₃/PE-g-AA/HDPE with 50% of Al₂O₃ presents the best strength properties, the tensile and flexural strength is 38.5 and 43.0 MPa, respectively, increasing by 88 and 81% when compared with the pure HDPE.

The thermal conductivity of Al₂O₃ fiber/HDPE composite at high filler content is improved notably especially in the injection direction. Addition of the PE-g-AA contributes to the further enhancement of thermal conductivity in this direction. It is notable that the composites are reinforced while the thermal conductivities are improved. The thermal conductivity and strength properties of Al₂O₃/PE-g-AA/HDPE are remarkably higher than those of the pure HDPE, the comprehensive properties of which are the best among the studied composites.

Dispersion of alumina fibers in the composites

The properties of the composite are related to the filler dispersion in the polymer matrix.^{26,27} The comparison of filler dispersions in Al₂O₃/HDPE, Al₂O₃/PE-g-MA/HDPE, and Al₂O₃/PE-g-AA/HDPE is characterized by SEM. The typical results are illustrated in Figure 3, which are the cryogenically fractured surfaces along the injection direction of composites containing 40 wt % Al₂O₃ fibers. As expected, for all the samples, most Al₂O₃ fibers orient in the injection direction.

The features of filler orientation in the injection direction make fibers have more chance to contact with each other and form chain-like structure or local Al₂O₃ network, which favors the heat energy transport in the composite. So the thermal conductivity of certain composite in the injection direction

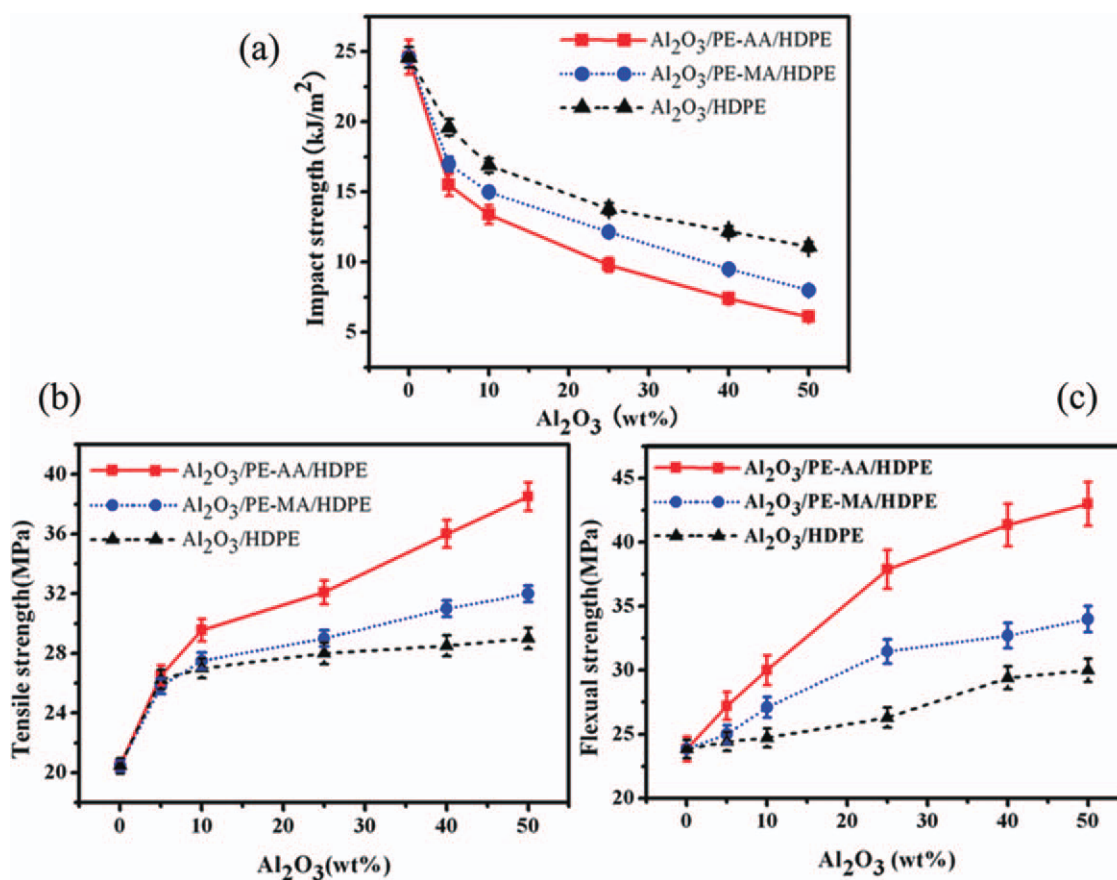


Figure 2 Dependences of the mechanical properties of the composites with alumina weight percentage: (a) impact strength; (b) tensile strength; (c) flexural strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

is higher than that in the perpendicular direction (Fig. 1).

To present the fiber orientation quantitatively, the numbers of fiber in the injection and perpendicular direction were counted from typical SEM pictures. The statistical analysis result of the number of the orientated fibers is shown in the Table I. It had to be noted that the fiber's orientation can be represented by the angle (0–90°) with the injection direction as reported by other researchers.²⁸ In this study, to make it simple, the fiber orientation was divided into two groups. The fibers oriented with the angle of 0–45° are considered along the injection direction and those with the angle of 45–90° are considered in the perpendicular direction.

In this study, the fiber orientation has been simplified to the above directions as a matter of research convenience. The number of the fibers in the injection direction is much higher than that in the perpendicular direction for all composites, but the amount of orientated fiber is varied with composites. The addition of the PE-g-MA has little effect on the fiber orientation compared with the composite Al₂O₃/HDPE, so the thermal conductivities of the Al₂O₃/HDPE and Al₂O₃/PE-g-MA/HDPE are very

close to each other. When PE-g-AA is used as the compatibilizer, the amount of orientated fiber dramatically increased, which causes the increase of difference between the thermal conductivities in the two directions (Fig. 1).

The fiber orientation is also an important factor influencing the strength of the composites. The composite strength can be greatly enhanced if the stress is applied along the fiber-orientated direction.^{29,30} In this study, the fiber orientation in the composite Al₂O₃/PE-g-MA/HDPE remains almost unchanged compared with Al₂O₃/HDPE, but the strength of Al₂O₃/PE-g-MA/HDPE is higher than Al₂O₃/HDPE. The interfacial interaction is then studied, which may be mainly responsible for the enhancement of the mechanical strength.

Interfacial interaction between the fibers and matrix

SEM observation and DMA measurements were used to analyze the interface of the composites. The morphologies of the impact fracture surfaces of the composites containing 40 wt % Al₂O₃ fibers are shown in Figure 4. There are holes in the fracture

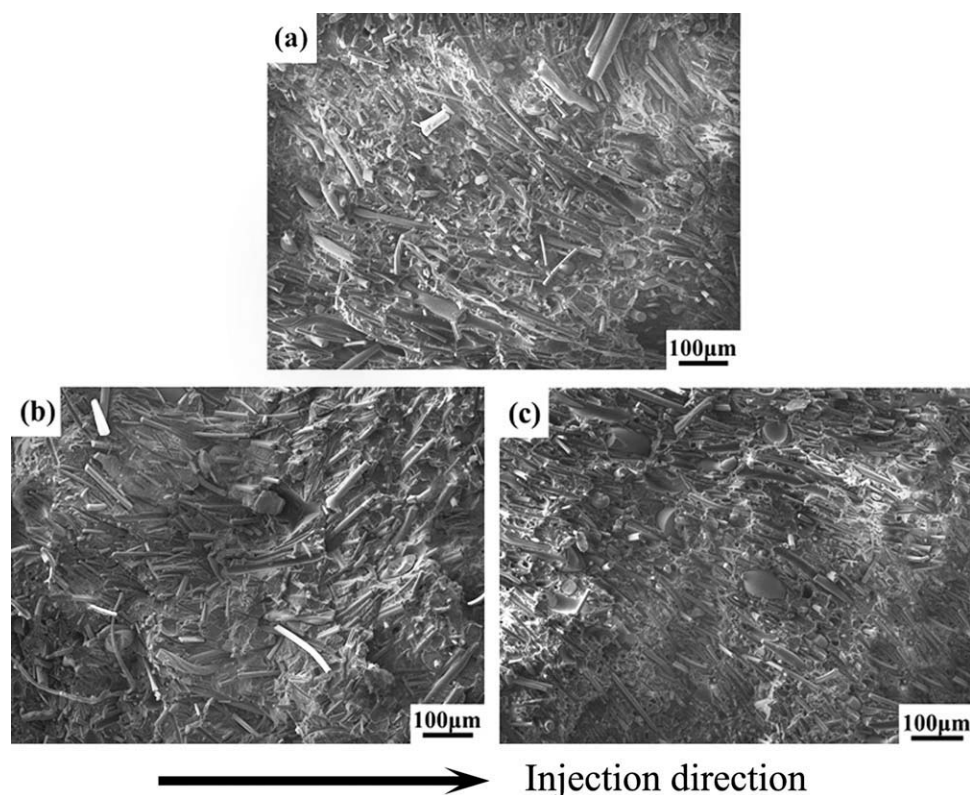


Figure 3 SEM micrographs of the brittle fracture surfaces in the injection direction: (a) $\text{Al}_2\text{O}_3/\text{HDPE}$; (b) $\text{Al}_2\text{O}_3/\text{PE-g-MA}/\text{HDPE}$; (c) $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$. The injection direction is from left to right.

surface of the $\text{Al}_2\text{O}_3/\text{HDPE}$ [Fig. 4(a)], which were left after the fibers were pulled out for the weak interfacial adhesion. The number of the holes decreased when compatibilizers were used, and there is no holes observed in the fracture surface of the $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$ [Fig. 4(e)].

The surface of the fiber in the $\text{Al}_2\text{O}_3/\text{HDPE}$ is smooth, and there is space between the fiber and HDPE [Fig. 4(b)]. The adhesion between the Al_2O_3 fibers and HDPE is improved when PE-g-MA is used [Fig. 4(d)], but the fibers' surface is still smooth when detached from the matrix. In the composite $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$, the matrix still adheres to the surfaces of the fibers even when they are fractured [Fig. 4(f)].

The addition of compatibilizer shows beneficial influences on the improvement of matrix–filler interaction, and PE-g-AA is more effective in enhancing the interaction than PE-g-MA.

TABLE 1
Statistical Results of the Fiber Orientation

Composite	Fibers perpendicular to injection direction (%)	Fibers in the injection direction (%)
$\text{Al}_2\text{O}_3/\text{HDPE}$	37	63
$\text{Al}_2\text{O}_3/\text{PE-g-MA}/\text{HDPE}$	34	66
$\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$	15	85

The loss modulus curves of HDPE, $\text{Al}_2\text{O}_3/\text{HDPE}$, $\text{Al}_2\text{O}_3/\text{PE-g-MA}/\text{HDPE}$, $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$ (with 40 wt % of Al_2O_3) are presented in Figure 5.

The pure HDPE shows two transitions, known as α and γ transition peaks.³¹ The α -relaxation is related to the motion in the crystalline phase, and occurs at a temperature where the folded chains are formed during melting-recrystallization processes. The γ -relaxation is because of long CH_2 sequences in the amorphous phase or in the defect lattice of crystal and can be considered as the glass transition temperature of HDPE.

Except the above two peaks, the composites filled with Al_2O_3 fiber show an additional peak, which is related to the interface between the matrix and fillers as reported in the published reports.³² The temperatures corresponding to interface relaxation are -40.4 , -39.6 , and -32.6°C for composites $\text{Al}_2\text{O}_3/\text{HDPE}$, $\text{Al}_2\text{O}_3/\text{PE-g-MA}/\text{HDPE}$, $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$, respectively. The interface relaxation peaks shifts toward higher temperature with the increase of the interfacial interaction because of the increased restriction for the motion of interphase polymer segments.

The SEM and DMA results suggest that the interaction between Al_2O_3 and HDPE in $\text{Al}_2\text{O}_3/\text{PE-g-AA}/\text{HDPE}$ is the strongest and the $\text{Al}_2\text{O}_3/\text{HDPE}$ is the weakest among the composites. The acrylic acid groups of the compatibilizer PE-g-AA can react with

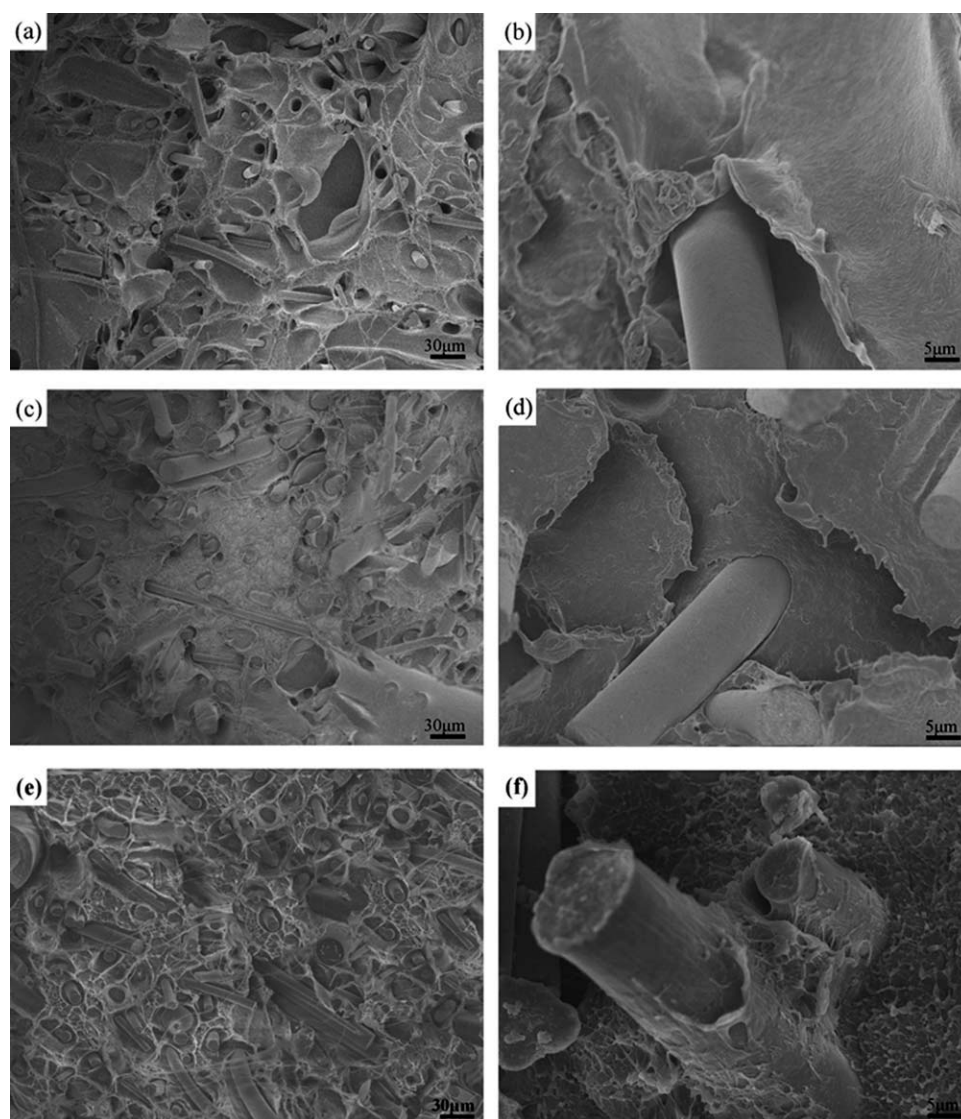


Figure 4 SEM micrographs of the impact fracture surfaces of composites: (a) Al₂O₃/HDPE (low magnification); (b) Al₂O₃/HDPE (high magnification); (c) Al₂O₃/PE-g-MA/HDPE (low magnification) (d) Al₂O₃/PE-g-MA/HDPE (high magnification); (e) Al₂O₃/PE-g-AA/HDPE (low magnification); (f) Al₂O₃/PE-g-AA/HDPE (high magnification).

hydroxyl groups on Al₂O₃ fibers surface through esterification reaction.³³ The PE-g-MA increases the interfacial interaction mainly by the polar interactions such as the hydrogen bonds between the MA groups of the compatibilizer and surface hydroxyl groups of the Al₂O₃ fibers.^{34,35} The covalent bonds are stronger than the polar bonds, as a result, the PE-g-AA is more effective in improving the matrix-filler adhesion than PE-g-MA.

The effect of interfacial interaction on properties of the composites

The impact strength of composite is influenced by the polymer matrix, the fiber content, and the interfacial interaction. The inorganic fibers, which act as stress concentrator in composites are very brittle and

have very low elongation to break, so the impact strength decreases drastically with the increase of the filler content.²⁴ The differences of the impact strength among the studied composites are primarily due to the different matrix-filler interaction. With the existence of good interfacial adhesion, the external impact load can be transferred from matrix to fibers easily, which initiates the fiber fractures. And then the fractures expand through the composite rapidly, resulting in the drastically decreased impact strength. So the higher interfacial interaction, the lower is the impact strength of the composite.

The strength of the Al₂O₃ fibers is much higher than the polymer matrix. The strong adhesion can easily transfer the stress from matrix to fibers and the fibers act as the main stress carrier in the composite, leading to the significant increase of the

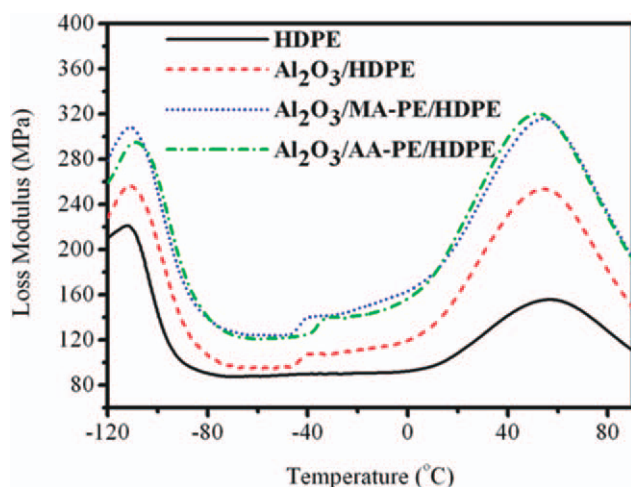


Figure 5 DMA loss modulus curves of HDPE, Al_2O_3 /HDPE, Al_2O_3 /PE-g-MA/HDPE, Al_2O_3 /PE-g-AA/HDPE. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

tensile strength and the flexural strength.³⁶ Contrary to the impact strength, the tensile and flexural strength increase with the improvement of the matrix–filler adhesion.

It has been found that the moderately improved matrix–filler interaction favors heat energy transfer because of the reduced interfacial thermal resistance in the inorganic particle filled polymer composites.^{13,37} However, the enhanced interfacial adhesion in Al_2O_3 /PE-g-AA/HDPE brings an increase of thermal conductivity in the injection direction and a decrease of thermal conductivity in perpendicular direction. This means that the reduced interfacial thermal resistance is the secondary reason for the rise of the thermal conductivity and the fiber orientation in the composite is the main factor determining the thermal conductivity of the fiber-filled composites. The interfacial adhesion influences the thermal conductivity of the composites indirectly by altering the orientation of the fibers in the composites.

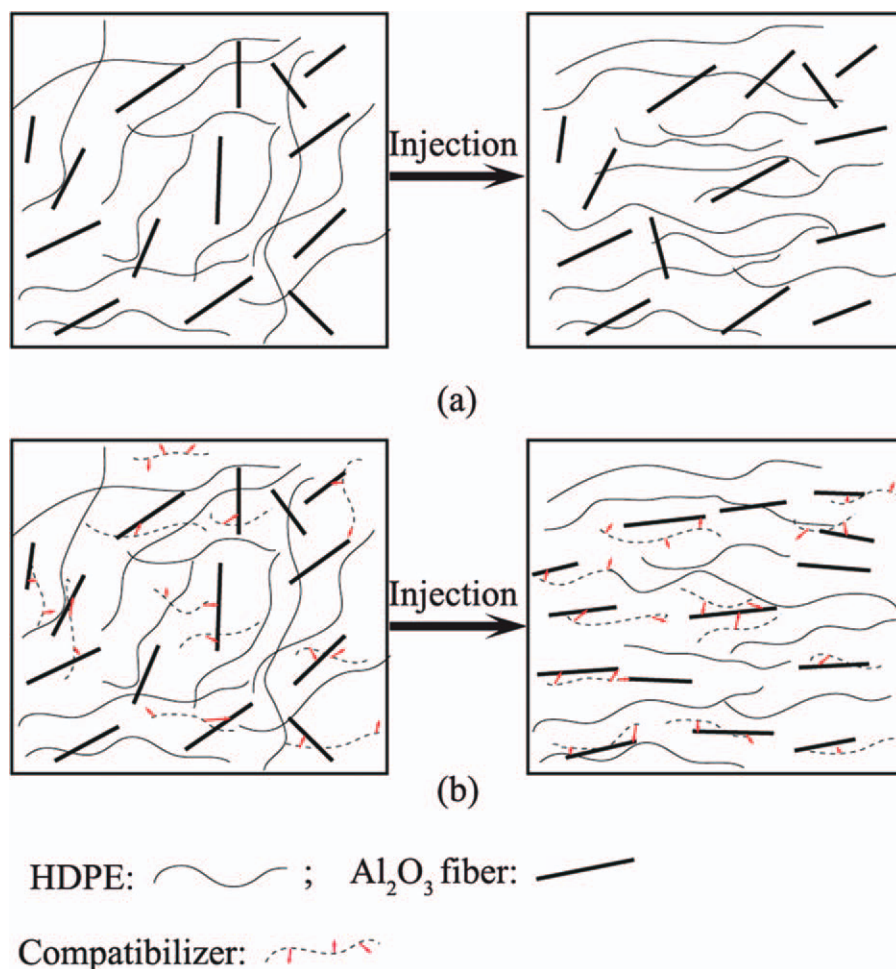


Figure 6 Schematic illustrations for the fiber orientation during the injection process: (a) Al_2O_3 /HDPE; (b) Al_2O_3 /Compatibilizer/HDPE. The dotted line stands for the matrix of the compatibilizer and the arrow stands for the grafted polar group in the compatibilizer. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The formation of the oriented fibers in different composites is schematically illustrated in Figure 6. In the melt state, the fibers are dispersed in the polymer matrix randomly (the left pictures in the Fig. 6). During the injection process, the polymer chains will orient in the injection direction under high pressure and the fibers will also prefer to orient in this direction because of the high viscosity of the polymer.³⁸ As a result, the injection-molded Al₂O₃/HDPE is anisotropic. In the composite of Al₂O₃/PE-g-AA/HDPE, the compatibilizer increases matrix–filler interaction and acts as a bridge, which favorably drags the fiber orientation in the injection direction. The improvement of the interaction with the addition of PE-g-MA is limited, so the orientation of the fibers in the composite Al₂O₃/PE-g-MA/HDPE is almost the same as that in the Al₂O₃/HDPE.

CONCLUSIONS

In this study, the effect of Al₂O₃ fibers on the thermal conductivity and mechanical properties of the HDPE with the absence or presence of compatibilizer was studied. The thermal conductivity in the injection direction was larger than that perpendicular to injection direction for all the investigated composites, which was attributed to the viscous flow-induced fiber orientation in the injection direction. The addition of PE-g-AA was more effective in improving the interfacial adhesion than PE-g-MA. The improved interfacial interaction was favorable for the viscous flow-induced fiber orientation in the injection direction, so the thermal conductivity anisotropy was also increased. In addition, the strength of the composites was enhanced with the increase of the interfacial adhesion.

The comprehensive properties of Al₂O₃/PE-g-AA/HDPE were the best among the studied composites. The thermal conductivity of Al₂O₃/PE-g-AA/HDPE with 50 wt % of Al₂O₃ in the injection direction was 0.47 W m⁻¹ K⁻¹, which was 96% higher than that of the HDPE. The tensile and flexural strength of Al₂O₃/PE-g-AA/HDPE with 50% of Al₂O₃ was 38.5 and 43.0 MPa, respectively, increasing by 88 and 81% when compared with the pure HDPE. These results suggested that the improvement of the matrix–filler interaction was an effective method to prepare the injection-molding fiber/polymer composite with good thermal conductivity and strength properties.

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