

Study on insulating thermal conductive BN/HDPE composites

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Abstract

Thermal conductivity of boron nitride (BN) reinforced high density polyethylene (HDPE) composites was investigated under a special dispersion state of BN particles in HDPE, i.e., BN particles surrounding HDPE matrix particles. The results indicated that the special dispersion of BN in matrix gives the composites high thermal conductivity at low filler content; moreover, the smaller BN particles can more easily form conductive chains of filler compared to the larger filler particles. Examining the dependence of electrical insulation and mechanical properties of the composites on BN content demonstrated that the reinforced composites containing 30% by volume of filler has good electrical insulation and mechanical properties.

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1. Introduction

With the miniaturization and increasing power of microelectronics, heat dissipation has become critical to the performance, reliability and further miniaturization of microelectronics [1]. The importance of thermal conductivity in polymer composites is associated with the need for appreciable levels of thermal conductance in circuit boards, heat exchangers, appliances, and machinery [2]. Commonly used plastics, such as polyethylene (PE), polypropylene (PP) and polyamide (PA) are excellent electrical insulators with a low thermal conductivity. Some new applications, like heat sinks in electronic packaging, require new composites with higher thermal conductivity. Plastics for electronic packaging require high thermal conductivity in order to dissipate heat generated in devices especially during turning on and turning off of the devices [3,4]. By the addition of fillers to plastics the thermal behavior of polymers can be increased remarkably. Such filled plastics with higher thermal conductivity than unfilled ones become more and more an important area of study due to the wide range of applications, e.g. in electronic packaging in applications with decreasing geometric dimensions and increasing output of power, like in computer chips or in

electronic packaging [5,6]. The higher thermal conductivity can be achieved by the use of suitable electrically insulating fillers such as alumina, boron nitride (BN) and alumina nitride (AlN) or other ceramic powder as filler.

BN has high thermal conductivity and excellent high temperature resistance, together with light weight and moderate cost compared to other ceramic fillers. Up to now, BN powder reinforced high density PE (HDPE) composites has been seldom studied before. So, the aim of this study was to investigate the thermal conductivity and ultimate mechanical properties of BN powder filled HDPE composites. Furthermore, the previous papers relating to ceramic powder reinforced polymers considered the effect on composite properties of particulate reinforcements introduced into a melted polymer matrix [7–9]. While, others types of dispersion state have not generally been considered. Agari and co-workers studied PE composites filled with graphite using four dispersion methods: (a) powder mix; (b) solution mix; (c) roll-milled mix; (d) melt mix. They found that for a given volume fraction of graphite the thermal conductivity of filled composites were ranked as follows: powder mixture > solution mixture > roll-milled mixture > melted mixture [10–12]. Therefore, the powder mixing was chosen to mix the BN and HDPE particles, and a special dispersion of BN in matrix was achieved in composites in which the HDPE particles are surrounded by BN particles. Thus, it is possible to improve the thermal conductivity of composites at low filler

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content using this kind of special dispersion, and to minimize the adverse effect of the filler on the dielectric properties of composites. So, physical behaviors of filled HDPE with BN, namely thermal conductivity and ultimate properties were investigated in this paper, together with the influence on thermal conductivity of particle sizes of filler used.

2. Experimental

2.1. Materials

High-density polyethylene was used as the matrix component. Qilu Petroleum Corporation, China, supplied the polymer. The HDPE has an MFI of 0.08 g/10 min, a melting point of 200 °C, a density of 0.95 g/cm³. Sanhe Chemicals in China supplied the BN powder, which was used as filler. It has a melt of over 2000 °C, a density of 2.27 g/cm³, platelet-shaped particles (see Fig. 1), and the particle sizes of BN used are 0.5 μm, 1 μm, 5 μm, 10 μm and 15 μm, respectively.

2.2. Composites preparation

- Powder mix: HDPE powder and BN particles were mixed in a mixing container at different ratios at room temperature until the BN particles surrounded the HDPE particles, and the samples were weighed according to the required ratios. After that, the mixture was transferred to a stainless steel die and melt pressed at 200 °C in an electrically heated hot press machine (SL-45, Shanghai, China) with a pressure of 5.0 MPa for 15 min, and was allowed to cool for 20 min at room temperature. Composites with BN powder concentrations ranging between 0% and 40% by volume were prepared.
- Melted mix: the whole procedure was basically same as the one described above. But, the samples are weighed accord-

ing to the required ratios, and mixed in 45 cm³ chamber at 200 °C and a speed of 30 rpm for 15 min.

2.3. Characterization

Thermal conductivity of the composites was measured using a Hot Disk thermal analyzer (Hot Disk AB, Uppsala, Sweden). The hot-disk method utilizes a thin disk-shaped sensor as a heat source and a temperature sensor to measure the thermal conductivity, which is based upon a transient technique. This hot-disk sensor was made of nickel foil in the form of a double spiral covered on both sides with an insulating layer of Kapton. A constant electric power was supplied to the hot-disk sensor by the source meter to measure the sensor resistance. Computer controls source meter, records the data, and analyzes thermal response during measurement. In this paper, a minimum of eight individual measurements was performed on bulk specimens (20 × 20 × 6 mm³) with the sensor (3 mm diameter) being placed between two similar slabs of material. The sensor supplied a heat-pulse of 0.03 W for 20 s to the sample and the associated change in temperature was recorded. The thermal conductivity of the individual was obtained by fitting the data according to Gustavsson et al. [13]

Morphological observations on the composite were done by means of scanning electron microscope (SEM), Model KYKY-2000, Japan. Observations were carried out on the cross-section of the samples to study the BN dispersion and morphology affecting thermal conductivity of system greatly.

A Hounsfield H5KS tensile tester was used for the tensile analysis of the samples. The dumbbell samples were stretched at a speed of 50 mm/min under a cell load of 2500 N. The gauge length was 24 mm, the thickness ranged between 0.5 mm and 1.0 mm, and the width was 4.8 mm. The final mechanical properties were evaluated from 6 different measurements.

Volume resistivity, dielectrical constant and breakdown voltage were performed on ultrahigh electric resistor (ZC-36, China), dielectrical constant Apparatus (S914, China) and Dielectric Breakdown Voltage (TNC, China), respectively, adopting the corresponding measurement standards GB/T1410-1989, GB/T1409-1988 and GB/T1408-1999.

3. Results and discussion

3.1. Dispersion state of filler in matrix

The thermal conductivity of BN (33 W/m K) is much larger than that of HDPE (0.26 W/m K). Therefore, the addition of BN into HDPE matrix would increase the thermal conductivity, and the content of filler has an effect on the thermal conductivity of the composites. The dispersion state of filler in matrix influences the thermal conductivity to a great extent since the thermal conductivity is mainly dictated by the numbers of conductive pathways or networks formed from conductive filler in matrix. The different processing method of filled composites results in different dispersion state of filler. Therefore, according to Agari [11] the processing way has effect on the thermal conductivity of composites.

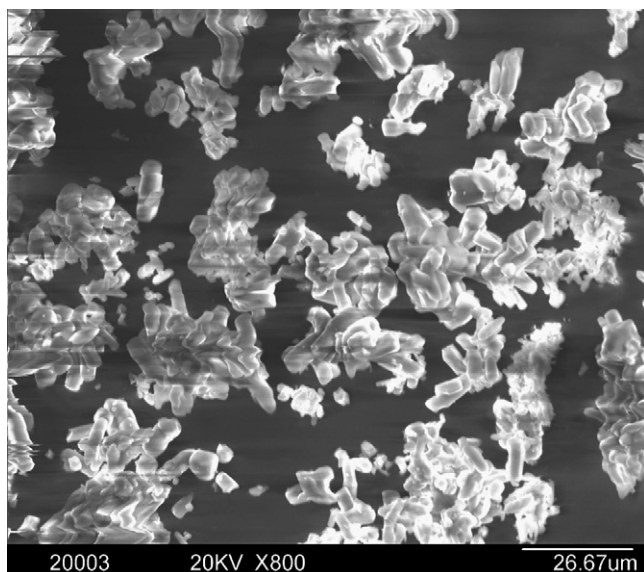


Fig. 1. SEM micrograph of boron nitride particles.

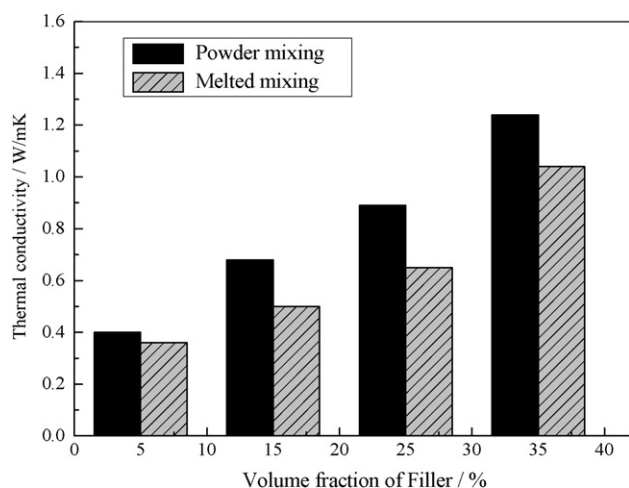


Fig. 2. Influence of dispersion ways on the thermal conductivity of composites.

The thermal conductivity of BN filled HDPE composites fabricated through powder and melted mixing, respectively, at different filler content, is displayed in Fig. 2.

It was found from Fig. 2 that with increasing the filler content the thermal conductivity of composites increased. At low filler content such as 5% by volume the composites prepared using the two mixing methods exhibited near same thermal conductivity. The reason may be attributed to the fact that at low filler concentration the filler particles would be segregated around the forming crystal structure by HDPE when processed from melt as it cools because the filler can be distributed only in the amorphous phase of HDPE due to its high crystalline degree. Therefore, filler particles could not form conductive pathways in matrix, and most of the filler were encapsulated by HDPE resin. Therefore, the mixing ways have little effects on the thermal conductivity [14]. When filler content continued to increase the composites made using powder mixture showed higher thermal conductivity compared to the one fabricated with melted mixture. For example, the thermal conductivities of the former were 0.68 W/m K, 0.89 W/m K and 1.24 W/m K at 15%, 25% and 35% by volume of filler content, respectively, compared to the corresponding 0.50 W/m K, 0.65 W/m K and 1.04 W/m K of the latter. Fig. 2 revealed that there must exist different internal structures between the two composites prepared through different mixing ways. The powder mixing method improved the thermal conductivity of composites compared to the melted mixing. Furthermore, the powder mixing could form more conductive channels in matrix, and might lead to an effective dispersion of filler in matrix.

To investigate the dispersion state of BN filler in the HDPE matrix, SEM photos of the composites made using the two mixing methods were obtained in Fig. 3.

It was seen clearly from the Fig. 3(a) that the HDPE particles were essentially surrounded by BN particles although there was some connectivity between adjacent HDPE particles in small regions. The most of adjacent BN particles could touch each other, and formed conductive pathways in composites. Since most of the connectivities of filler were not blocked by matrix, the thermal resistance of composites mainly resulting

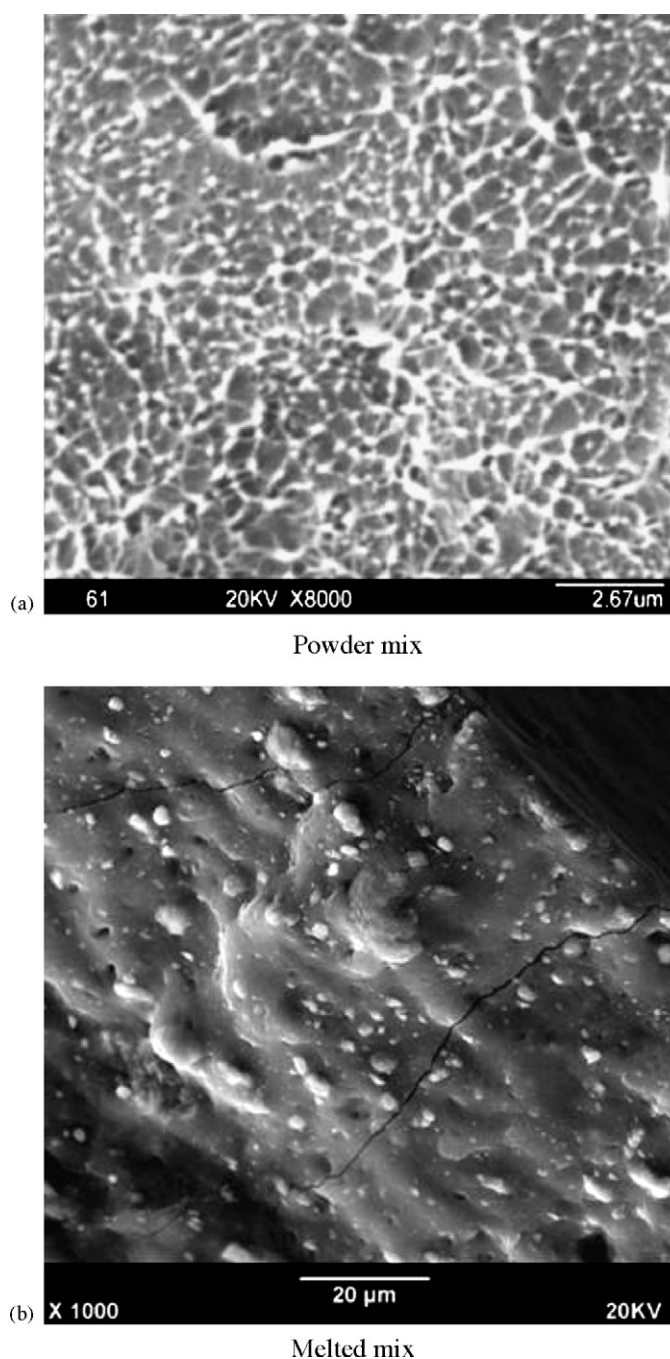


Fig. 3. SEM micrographs of cross section of BN/HDPE composites produced using different mixing ways.

from HDPE with low thermal conductivity was low, resulting in higher thermal conductivity. In addition, the deformation of the soft, platelet-shaped BN filler particles in composites provided improved particle-to-particle connectivity and allowed greater packing density, resulting in the ability to achieve much conductivity than was possible for hard and stiff particles, such as alumina, of similar initial morphology. From the viewpoint of network, some imperfect conductive networks of filler had formed in composites which aided enhancing the thermal conductivity [15–17]. While, from Fig. 3(b) it was found that the BN

filler homogeneously dispersed in HDPE in spite of some aggregations of filler owing to the high viscosity of melted HDPE. However, many filler particles were encapsulated completely by HDPE matrix. The thermal conductivity of BN is much greater than that of HDPE, when the samples are heated, just as electric charge flows where resistance is low, so the heat will flow through the BN particles. Thus, conductive channels can be formed in the composites under the dispersion state using powder mixing, leading to a higher conductivity than in a melted HDPE matrix [10].

The powder mixing gives the filler a special dispersion state in composites, facilitating to improving connectivity of filler in composites. Therefore, it could be concluded that at lower filler content higher thermal conductivity can be achieved. Thus, the adverse effect of filler content on the dielectrical and mechanical properties of composites may be minimized [18].

3.2. BN filler content

The effect of filler content on the thermal conductivity of the composites is shown in Fig. 4. It was seen that the thermal conductivity increased with increasing BN content and reached 1.12 W/m K, more than four times that of pure HDPE at 30% of BN by volume.

In order to predict the thermal conductivity of composites as a function of filler content, many theoretical models concerning the thermal conductivity of composites have been published. Among them the Maxwell-Eucken and Bruggeman models [4] were used here to evaluate the thermal conductivity of composites.

Maxwell-Eucken model

$$\lambda = \lambda_p \frac{[2\lambda_p + \lambda_f + 2V_f(\lambda_f - \lambda_p)]}{[2\lambda_p + \lambda_f - V_f(\lambda_f - \lambda_p)]} \quad (1)$$

Bruggeman model

$$1 - V_f = \frac{(\lambda_f - \lambda)(\lambda_p/\lambda)^{1/3}}{(\lambda_f - \lambda_p)} \quad (2)$$

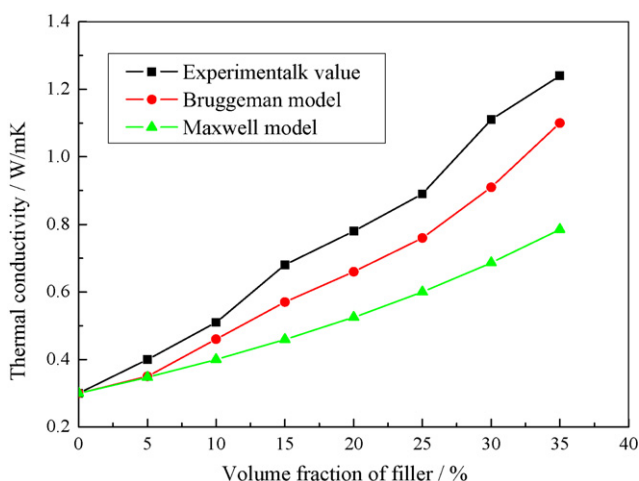


Fig. 4. Thermal conductivity of the composites as a function of filler content.

where λ , λ_p and λ_f are the thermal conductivity of composites, polymer and filler, respectively; V_f is the volume fraction of filler.

It was shown that the experimental data did not agree with the Maxwell-Eucken and Bruggeman models well, although the Bruggeman models is suitable for predicting the thermal conductivity of composites containing greater than 20% volume fraction of filler. From the Fig. 4 the experimental data always are higher than those predicted. That is because that the two models both assume that the shape of filler is sphere, and the dispersion state of filler is confined to common dispersion state of filler in the composites that is filler mixed into melted matrix. However, the special dispersion state of filler formed here obviously exceeds the presuming limits of the models. So, it is this special dispersion that increases the thermal conductivity of system effectively at lower filler content.

3.3. Particle sizes of filler

The filler particle size has an effect on the thermal conductivity of composites when BN particles were dispersed around HDPE particles. Different sizes of BN will result in a different filler layer surrounding the HDPE at the same BN content.

In order to examine the influence of particle size of filler on the thermal conductivity of composites, the five BN fillers with different average particle sizes of 0.5 μm , 1.0 μm , 5.0 μm , 10 μm and 15 μm , respectively, were used to reinforce HDPE in this study. The result is illustrated in Fig. 5.

The Fig. 5 revealed that the smaller particles filled composites showed higher thermal conductivity than that of the larger particles at the same filler content. For a given volume fraction of BN with various sizes the thermal conductivity of filled systems were ranked as follows: 0.5 μm > 1.0 μm > 5.0 μm > 10 μm > 15 μm . The thermal conductivities of reinforced composites using 30% by volume of the five kinds of particles above were 1.20 W/m K, 1.12 W/m K, 1.08 W/m K, 1.06 W/m K and 1.03 W/m K, respectively. Since the particle size of HDPE is fixed, the number of particles increases with decreasing the particle size at the same volume fraction of filler. For smaller particles, the absorption

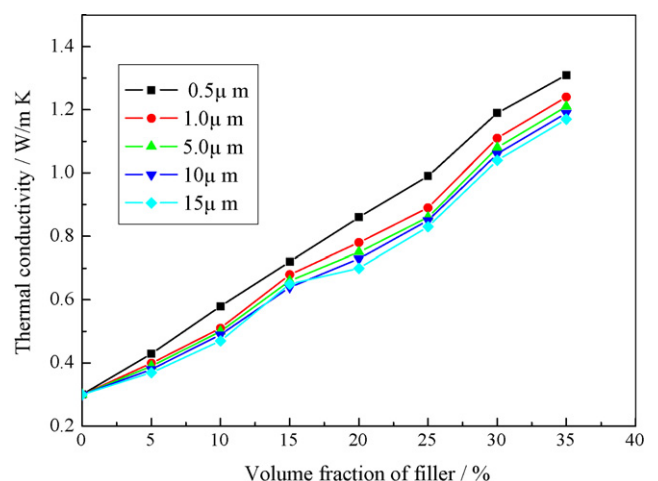


Fig. 5. Effect of particle sizes of BN on the thermal conductivities of composites.

Table 1
 C_1 and C_2 of Agari model for BN/HDPE containing 30% of filler by volume

Average particle size of BN (μm)	C_1	C_2	Thermal conductivity (W/m K)
0.5	1.040	0.987	1.18
1.0	1.090	0.951	1.11
5.0	1.110	0.813	1.08
10	1.121	0.784	1.06
15	1.156	0.762	1.05

energy between the filler particle and HDPE particle is higher compared with the larger particles, due to high surface energy of smaller particle. Therefore, much more filler particles surround the HDPE particles as particle size decreases, and more HDPE particles are encapsulated and segmented by filler in composites. In other words, the number of conductive pathways or channels formed in the smaller particles reinforced composites is higher than that of the larger particles reinforced one. So, the smaller particles filled composites shows higher thermal conductivity compared to the larger particles.

In addition, when decreasing the filler size, the thickness of filler layer surrounding HDPE will increase, so, there may be more BN particles around each HDPE particle at the same filler content compared to the larger particles. Therefore, the thermal conductive pathways can be considered more stable for smaller filler particles because the thicker conductive channels have less chance of being disrupted by contacting grains.

The model of Agari et al. [19,20] considered the effect of dispersion state by introducing two factors C_1 and C_2 :

$$\lg \lambda = V_f C_2 \lg \lambda_f + (1 - V_f) \lg (\lambda_p C_1) \quad (3)$$

where λ , λ_p and λ_f are the thermal conductivity of composites, polymer and filler, respectively; V_f is the volume fraction of filler; C_1 is a factor relating to the structure of polymer, such as crystallinity of matrix, and C_2 is a factor relating to the measure of ease for the formation of conductive chains of filler. According to Agari, the values of C_1 and C_2 should be in between 0 and 1, the closer C_2 values are to 1, the more easily conductive chains are formed in composites. So, if the dispersion state of filler is different, the thermal conductivity of composites may be different even if the composites are the same. By fitting the experimental data into the model, a possible explanation of the dispersion state of filler in composites could be obtained. Through data fitting, C_1 and C_2 for the above five systems are listed in Table 1.

From Table 1, it was observed that the addition of fillers with various sizes into the HDPE affected the C_2 values more strongly than the C_1 values. This observation indicated that particle sizes did not affect the secondary structure of HDPE. It was also observed that the C_2 values for the five composites were affected by the particle sizes of filler used, the smaller particles reinforced composites showed a higher C_2 value compared to the larger particles reinforced one. That is because a better state of filler dispersion was achieved in the smaller particles reinforced composites compared to the larger particles. This demonstrated

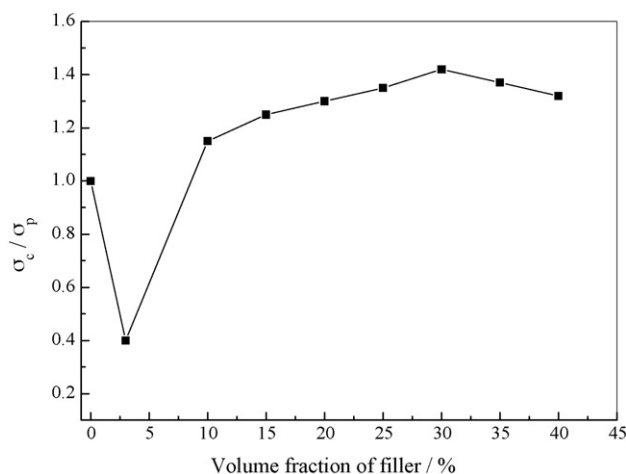


Fig. 6. Stress at break of the composites as a function of volume fraction of filler.

that the formation of conductive channels in the smaller particles filled composites is more likely [21–23].

So, the BN filler with average size of 0.5 μm was chosen as conductive filler for preparing composite BN/HDPE plastics. Furthermore, the mechanical and electrical properties of the filler (0.5 μm) filled HDPE were studied as follow.

3.4. Mechanical properties

3.4.1. Stress at break

Fig. 6 revealed the dependence of stress at break on filler concentration (where σ_c and σ_p are the stress at break of composites and polymer, respectively). It was found that with increasing filler concentration the stress at break increased compared to that of pure HDPE. However, at very low filler content the stress at break decreased; the stress at break of pure HDPE decreased from 26 MPa to 10.1 MPa, when filled with 3.0% of BN by volume. Because the filler can be distributed only in the amorphous phase of HDPE due to its high crystalline degree, thus, the local concentration of the filler in amorphous phase in HDPE is high. In this case, the filler served as defects in fact [3]. The higher concentration of the defects caused by the higher local concentration of the filler results in steeper decrease in stress at break since the presence of defects in front of the neck has critical influence on the drawability [4].

The reinforcing effect of BN showed remarkable at the filler content of 30% by volume. Since filler was located only in the amorphous phase of HDPE, the consequence of this fact is that the amorphous part of HDPE was reinforced due to high local concentration of filler in the amorphous phase.

3.4.2. Elongation at break

The dependence of elongation at break on the filler content is shown in Fig. 7. A steep decrease of elongation at break at near 2.6% volume fraction of filler was observed. The elongation at break decreased from 650 to 45 at the 30% volume fraction of filler. For description the decrease of elongation at break with

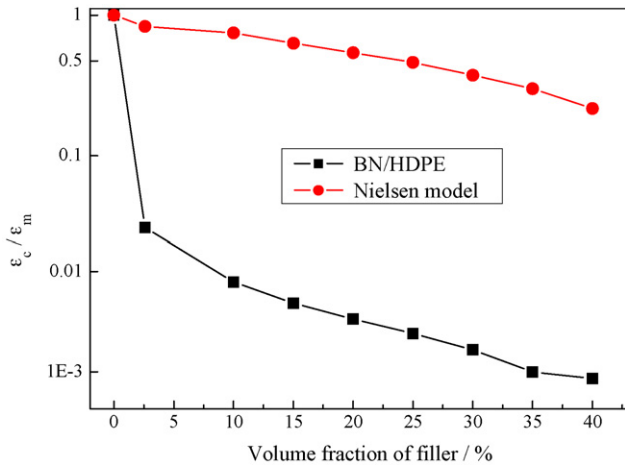


Fig. 7. Elongation at break of the composites as a function of volume fraction of filler.

an increase in filler content, Nielsen model given by Eq. (4) was used here.

$$\frac{\varepsilon_c}{\varepsilon_p} = 1 - V_f^{1/3} \quad (4)$$

where ε_c , ε_p are the elongation at break of composites and polymer, respectively; V_f is the volume fraction of filler.

From Fig. 7 it was seen that the predictions of the model deviated sharply from the experimental data. The reason for the deviations is because the Nielsen model is valid for filler having spherical shape assuming perfect adhesion between phases. As seen from Fig. 1, the used BN filler have very irregular shape and show platelet shape. Moreover, the filler are not uniform. These geometrical factors are expected to contribute significantly to a dramatic decrease of elongation at break. In addition, the use of Nielsen model is confined to the case where the deformation is homogeneous. As for HDPE, a neck is created during drawing and therefore stretching is not homogeneous [1,4].

3.5. Electrical properties

The dielectrical constant of BN is about 5.0, compared to the value of 2 for HDPE. So, the addition of filler into HDPE will increase the dielectrical constant of the composites. In addition, the filler concentration has influence on the volume resistivity of the composites. The effect of amount of filler on the dielectrical constant and volume resistivity of the composites is shown in Fig. 8.

It was found that with increasing the filler content the dielectrical constant of the composites increased, while the volume resistivity decreased. That was because the electrical insulation of HDPE is super to that of BN, and according the rules of mixture, the electrical insulation of the composites should lie between the filler and matrix. From the Fig. 8 the electrical insulation and volume resistivity of the composites containing 30% by volume of filler were 4 and 10^{15} , respectively, compared to 2 and 10^{17} of the pure HDPE. That proved that the composites still has good electrical insulation.

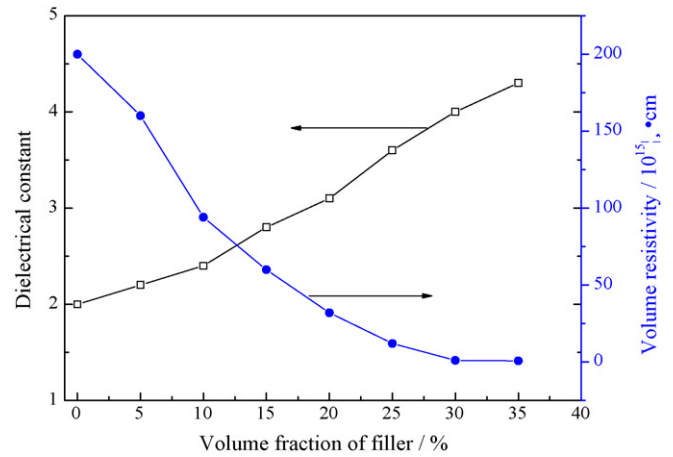


Fig. 8. Dielectrical constant and volume resistivity of the composites as a function of filler content.

Table 2

Properties of the composites containing 30% by volume of BN

Physical properties	HDPE	BN/HDPE
Thermal conductivity (W/m K)	0.26	1.20
Tensile strength (MPa)	26	30
Elongation at break (%)	650	45
Breakdown voltage (kV/mm)	38	20
Dielectric constant	2.0	4.0
Volume resistivity (Ω cm)	10^{17}	10^{15}

3.6. Overall physical properties [24,25]

From the discussion mentioned above, the filler content should be under a desired level in order to avoid deteriorating physical properties of the composites. According to the experiment, the preferable filler content is 30% by volume. Therefore, the physical properties of the composites are summarized in Table 2, with the pure HDPE as a comparison.

From the Table 2 the BN reinforced HDPE exhibited good thermally conductive and electrically insulating properties, along with improved tensile strength.

4. Conclusions

The BN reinforced HDPE composites prepared through powder mixing of BN and HDPE particles, obtained higher thermal conductivity at low filler content, compared to that made using melted mixing. There existed a special dispersion state of BN particles in the composites, i.e., BN particle surrounding HDPE matrix particles and forming more conductive pathways of filler, which led to higher thermal conductivity of the composites.

Thermal conductivity of the composites significantly exceeds the predictions from the Maxwell-Eucken and Bruggeman models owing to this special dispersion of filler in matrix

The particle sizes of the filler have an effect on the thermal conductivity of composites. The smaller filler particles reinforced composites showed higher thermal conductivity than that of the larger particles filled one. Examining the parameters of the Agari model indicated the smaller BN particles can more

easily form conductive channels of filler compared to the larger filler particles.

The stress at break of the composites increased with increasing the filler concentration. However, at low filler content the stress at break sharp decreased because the higher concentration of the defects, caused by the higher local concentration of the filler, results steeper decrease in stress at break. The elongation at break of the composites decreased with increasing filler content, furthermore, the steep decrease in elongation at break occurred at low filler concentration. The Nielsen model could not predict the elongation at break of the composites owing to the very irregular shape of filler.

The addition of BN into HDPE decreased the volume resistivity of the composites, while increased the dielectrical constant of the system. The reinforced composites containing 30% by volume of filler has good electrical insulation and mechanical properties.

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