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Thermal and dielectric properties of the AlN particles reinforced linear low-density polyethylene composites

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ABSTRACT

A thermally conductive linear low-density polyethylene (LLDPE) composite with aluminum nitride (AlN) as filler was prepared in a heat press molding. Differential scanning calorimeter results indicated that the AlN filler decreases the degree of crystallinity of LLDPE, and has no obvious influence on the melting temperature of LLDPE. Experimental results demonstrated that the LLDPE composites display a high thermal conductivity of 1.25 W/m K and improved thermal stability at 70 wt% AlN content as compared to pure LLDPE. The dielectric constant and dissipation factor increased with AlN content, however, they still remained at relatively low levels, i.e., <5 in wider frequency range from 10 to 10^6 Hz. The surface treatment of AlN particles had a beneficial effect on improving the thermal conductivity and dielectric constant, whereas, the dissipation factor was less affected. Additionally, the obtained AlN/LLDPE composites have possessed rather low dielectric constant and high electrical insulation, which is suitable for substrate and packaging materials.

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1. Introduction

Polymeric matrix composites have gained wide applications in various branches of industry because of their low density, ease of forming, chemical inertia, low cost, and electrical insulation [1]. However, high thermal conductivity is desirable for appreciable levels of thermal conductance in some cases such as printed circuit board, heat exchangers, thermal interface materials and phase change materials [2]. In the microelectronic systems great effect has been contributed to improving the integration density, fast performance, miniaturization, and low cost of electronics in recent year [3–5]. With further miniaturization and increasing power of microelectronics, heat dissipation has become critical to their performance, reliability since high integration of transistors has resulted in the escalation of power dissipation as well as an increase in heat flux at the devices [6–8]. So, the use of polymer is restricted to parts requiring high heat dissipation capability, however, cooling is often practically restricted by a low thermal conductivity of the molded polymers [9,10]. As the power density becoming larger and larger, better properties are required for substrate and packaging materials, such as a high thermal conductivity, a low coefficient of thermal expansion (CTE), and a low dielectric constant and loss [11]. Under this circumstance, highly thermal conductive and insulating polymers are emerging as one of the most economic and effective means to remove heat accumulated from microelectronics and to cope with thermal management issues [12–15].

Plastics filled with ceramic particles possessing a high thermal conductivity are attractive candidates in this area because they can provide a required thermal conductivity while maintaining a desired electrical insulation property [9,11]. There are many references in the plentiful literature concerning adding thermal conductive fillers to plastics to produce a more heat conductive composites. For example, many composite plastics have been investigated including different kinds of fillers such as boron nitride (BN) [16], aluminum nitride (AlN) [1,9,14,17,18], silicone nitride (Si_3N_4) [9,19,20], alumina (Al_2O_3) [21], silicon carbide (SiC) [6,22,23], and diamond [24], and various polymer matrix such as high density polyethylene (HDPE), low density polyethylene (LDPE), ABS, polyamide and polypropylene.

Compared with LDPE, linear LDPE (LLDPE) possesses better strength, toughness, cold-resistance, environmental stress cracking resistance, and tearing resistance properties [1,25]. To take advantage of the excellent manufacturing process performance of LLDPE and to more effectively increase the thermal conductivity of LLDPE, the use of intrinsic high thermal conductive AlN particles can be considered due to its high strength, reliable electric insulation, low dielectric constant and loss, and matching linear CTE like silicone, and are paid more and more attentions [1,9,14,17,18].

Up until now, the investigations of the dielectric properties of the heat conductive AlN/LLDPE composites over a wide frequency

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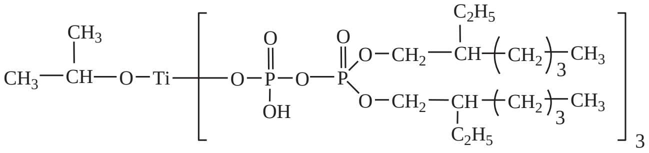

Scheme 1. Chemical structure of the titanate coupling agent.

range have seldom been reported before. So, our purposes expect to give a deeper insight into the influence of AlN content on the thermal and dielectric properties of AlN/LLDPE composite. In order to understand the fundamentals of interface effect on thermal conductivity and dielectric properties of the composites, a titanate coupling agent was used to functionalize the surface of AlN particles to improve the wettability and uniform dispersion of AlN particles in the LLDPE.

2. Experimental

2.1. Materials

The matrix component was LLDPE with a brand of Hyundai SR646 from Hyundai Group (Seoul, South Korea), which has a density of 0.935 g/cm^3, a melt index of 45 g/10 min. AlN powder with hexagonal structure supplied by Sanhe New Material Co. (Sanhe, Hebei) has a density of 3.26 g/cm^3, a thermal conductivity of 170 W/m K, and the average particle sizes of 8–10 μm. The titanate coupling agent with a purity of 99% was from Nanjing Xiangfei Chemical Co. (Nanjing, China), whose chemical structure is shown in Scheme 1. Some additives such as polyethylene wax, stearic acid, tetra-[methylene-β-(3,5-di-tert-butyl-4-hydroxyphenyl)-propionate]methane(EVERNOX-10), and methylbenzene were all available from market. The polyethylene wax and stearic acid served as lubricants in the formulation, and the EVERNOX-10 served as antioxidant protecting LLDPE from being oxygenized and degradation at high processing temperature.

2.2. Samples preparation

2.2.1. Surface modification of AlN particles

The surface modification for the AlN particles with the titanate coupling agent involved the following steps: (a) dissolving the titanate coupling agent (1.2% of the AlN filler mass) in methylbenzene; (b) adding AlN particles to the solution made in step (a) and stirring for 20 min (min); (c) heating the mixture to 90 °C, refluxing it for 2 h while stirring, and then cooling it to room temperature and setting it for 10 h; (d) rinsing the mixture with methylbenzene by filtration at least three times; and (e) drying the mixture at 120 °C for 10 h.

2.2.2. Preparation of AlN/LLDPE composites

LLDPE and AlN particles were dried in vacuum at 50 °C for 5 h and at 120 °C for 8 h prior to use, respectively. The LLDPE was first mixed with polyethylene wax, stearic acid, antioxidant at 150 °C, followed by the addition of AlN fillers according to the designed mass-fraction ratio (as shown in Table 1). The compounding was carried out on a two-roll mixing mill (Type: SK-106B, China), and the total mixing time for all the different concentrations was kept at 20 min. After that, the resulting mixture was transferred to a stainless steel die and melt pressed at 170 °C in an electrically heated hot press machine (type: SL-45, Shanghai, China) with a pressure of 15 MPa for 15 min, and was allowed to cool for 30 min at room temperature. Composites with AlN powder concentrations ranging from 0 to 70 wt% were prepared.

Table 1
Formulation of AlN/LLDPE composites.

Ram materials	Content
LLDPE (g)	100
Polyethylene wax (g)	1
Stearic acid (g)	1.5
Antioxidant (g)	1.5
AlN (g)	Variant

2.3. Characterization

The differential scanning calorimeter (DSC) (Model, DSC 200PC, Netzsh Corp., Selb, Germany), was used to analyze the influence of AlN content on the melting temperature and melting enthalpy of the LLDPE samples (5–10 mg). Measurements were conducted in a nitrogen atmosphere, from 20 °C to 200 °C, at a heating rate of 10 °C/min.

Weight loss of the samples (~10 mg) upon heating was measured using a thermogravimetric analyzer (model: SDTA851, Swiss). Measurements were conducted in a nitrogen atmosphere, from 25 °C to 600 °C, at a heating rate of 15 °C/min.

Thermal diffusivity of the samples was measured on Netzsch system (Model, LFA427, Netzsh Corp., Selb, Germany). Then, the thermal conductivity was calculated from thermal diffusivity according to Eq. (1):

$$k = \alpha \cdot \rho \cdot c_p \tag{1}$$

where k, α, ρ, c_p are the thermal conductivity (W/m K), thermal diffusivity (cm^2/s), density (g/cm^3), and specific heat capacity (J/kg K) of the material under constant pressure. The thermal diffusivities of samples were measured at room temperature (in air) and elevated temperature (in argon). The specimens for thermal diffusivity measurement were made in the form of circular discs with ~1 mm in thickness and 12.7 mm in diameter.

The microstructures of composites were observed by scanning electron microscope (SEM) (model: JSM-7000F, JEOL, Japan). The fractured surfaces were prepared in liquid N$_2$. The samples were sputtered with gold in vacuum prior to observation. The observation was carried out on the cross-sections of samples to study fillers distribution.

The dielectric measurement was performed on a broadband dielectric spectrometer (Novocontrol Technology Company, Germany) with Alpha-A high performance frequency analyzer. The measurement was carried out in the frequency range from 10^{-1} to 10^7 Hz under room temperature. The specimens were made in the form of circular discs with ~1 mm in thickness and 20 mm in diameter. Both sides of the disks were suitably sprayed with gold powder to improve electrical contact.

The volume resistivity and surface resistivity measurements were performed on an ultrahigh electric resistor (model: ZC-36, China) at room temperature.

3. Results and discussion

3.1. Melting temperature and degree of crystallinity

The DSC curves of native LLDPE and its AlN composites are plotted in Fig. 1, and the corresponding thermal dynamic data values are listed in Table 2. It can be observed that the AlN content does not seem to have appreciable influence on the melting temperature of LLDPE, and the peak temperature of melting (T_m) very slightly shifts toward a lower temperature with increasing AlN loading. The reason may be that the AlN filler probably reduces of lamellar thickness of crystallites and thus it leads to a decrease of melting temperature [4,11], because some AlN particles will fill themselves in the

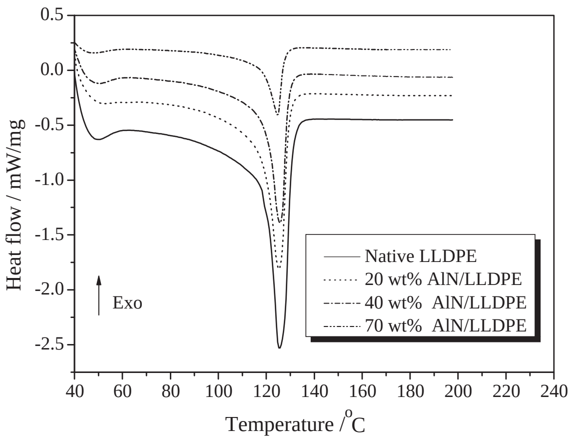

Fig. 1. DSC curves of native LLDPE and AlN/LLDPE composites.

interlamellar space due to a higher degree of crystallinity of pure LLDPE.

Table 2 indicates that with increasing AlN content the degree of crystallinity (X_c) of LLDPE decreases as compared to native LLDPE. The reason may be that the LLDPE has a relative high crystallinity, and has not a major amorphous part in which the AlN particles can be accommodated. At low filler loading the filler particles locate themselves in the interlamellar space, which leave little space for additional crystallization, and the presence of filler may even inhibit crystallization. At high filler content there is probably a change in crystallization mechanism [11]. It is therefore possible for the fillers to decrease the mobility of LLDPE chains in the formation of crystallites and, as a result, the domains of crystalline phase are reduced in size [26–28]. It is also likely that imperfection of crystals in the presence of the AlN inhomogeneities contributes to the decrease in crystallinity [28].

3.2. Thermal stability and thermal conductivity

The TGA curves for AlN/LLDPE composites are illustrated in Fig. 2. There is an obvious increase in the thermal stability of LLDPE with increasing AlN concentration. The reason may be ascribed to the higher heat capacity 0.738 J g^{-1} °C^{-1} of AlN, as compared to 0.18 J g^{-1} °C^{-1} of LLDPE and the high thermal conductivity of 170 W/m K, compared to 0.264 W/m K of native LLPDE, which will cause it to preferably absorb the heat. So, this will result in LLDPE chains starting to degrade at higher temperature [11].

The thermal conductivity values of AlN/LLDPE are shown in Fig. 3, as can be seen that they increase obviously with an increase in filler loading. The filler particles at low volume fraction can disperse randomly in the LLDPE matrix and has weak interaction each other to present a little increase of thermal conductivity. As the filler loading increases the particles begin to touch each other, and to form partial conductive path or network which enhance heat conductance. For example, at the 40 vol% AlN content, the thermal conductivity reached 1.25–1.39 W/m K, five times that of pure LLDPE.

Table 2
DSC data of native LLDPE and AlN/LLDPE composites.

AlN/LLDPE (w/w)	ΔH_m (J/g)	T_m (°C)	X_c (%)
0/100	152.5	125.7	52.6
20/80	110.7	125.3	47.7
40/60	89.33	125.4	51.3
70/30	36.67	124.9	41.8

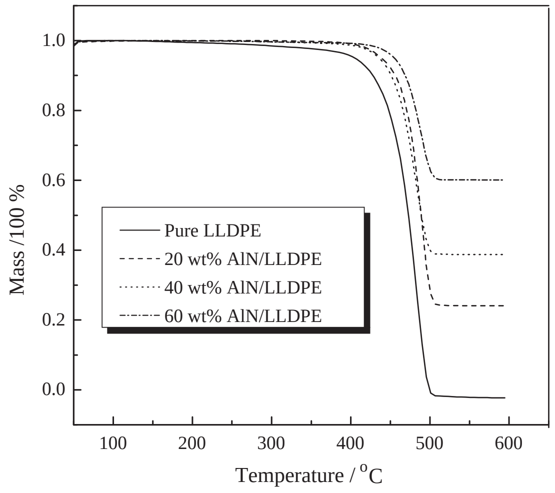

Fig. 2. TGA curves of native LLDPE and AlN/LLDPE composites.

The experimentally determined thermal conductivity values and those predicted from Maxwell model are shown in Fig. 3. The Maxwell's equation is an exact solution for the effective conductivity of randomly distributed and non-interacting spheres in a continuous medium. However, it does not take into account the mutual interaction of the particles; thus it is not a satisfactory treatment for composites in the high volume fraction range (>20%). Form Fig. 3 we can see that at high filler content Maxwell model tends to underestimate thermal conductivity of the composites because they begin to touch one another.

Fig. 3 suggests that the use of titanate coupling agent to functionalize the surface of AlN obviously improved the thermal conductivity compared to the untreated filler reinforced LLDPE at AlN content >20 vol%. It is well known that the transport of heat in all non-metals is by the flow of lattice vibration energy [8], or phonons, along the temperature gradient within the specimen; for a two phase system like AlN/LLDPE composites, interfacial physical contact between polymer and filler is very critical, since phonons are very sensitive to surface defects [29,30]. Thermal resistance is caused by various types of phonon scattering processes, and the interfacial thermal barriers in composites is mainly due to the scattering of phonons resulting from acoustic mismatch and flaw associated with the matrix–filler interface [27]. The interface between the two-phase composites acts as a barrier of heat transmission. So, the surface treatment of AlN with titanate improves the interfacial bonding between AlN and matrix, and reduces the

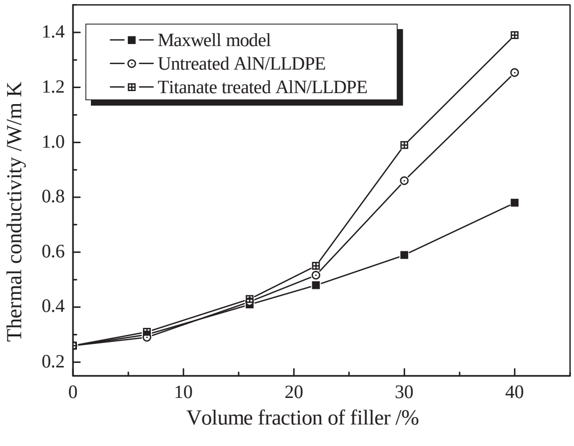

Fig. 3. The experimentally thermal conductivity values and theory values of Maxwell model of the composites.

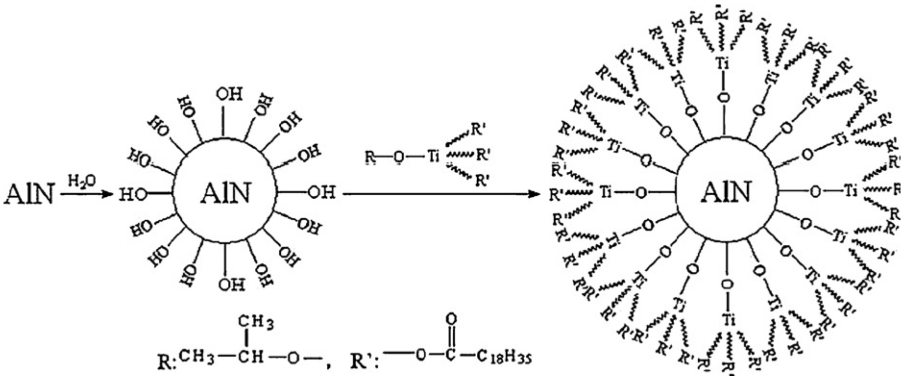

Fig. 4. The modification mechanism of titanate on the surface of AlN.

voids at the filler–matrix interface, which facilitates enhancing the thermal conductivity.

Chemical bonding theory and adsorption model of monomolecular layer have been proposed as the mechanism of titanate coupling reagent [31]. The possible structure model of titanate treated AlN particle is shown in Fig. 4 [1]. It can be supposed that titanate can form an organic active monomolecular layer between the interface of AlN and LLDPE. Just like a bridge, one end reacts with free proton on the AlN surface to form firm chemical bond, and the crosslinking reaction occurs or tangles with LLDPE chains by Van der Waals force at the other end. Thus, the organic interface layer between AlN and LLDPE is generated [1]. So, the titanate enhances the phase interfacial bonding, and improves the thermal conductivity.

The influence of temperature on the thermal conductivity of composites for different AlN filler content is presented in Fig. 5. It is noticed that the thermal conductivity of the composites decreases slightly as temperature increases within the testing temperature range. The reason for the decrease might be that the volume of composites expands slightly when heated, and the distance of adjacent conductive filler particles increases slightly, thus leading to a decreased thermal conductivity.

The micro-structure images of LLDPE filled with 50 wt% and 70 wt% of filler are depicted in Fig. 6. The AlN powder distributions are found to be relatively rather uniform. At high filler loading the most of adjacent AlN particles could touch each other, and form particles clusters or partial conductive pathways in composites. The thermal resistance is therefore reduced owing to the connectivity of some filler particles, leading to a high thermal conductivity [14].

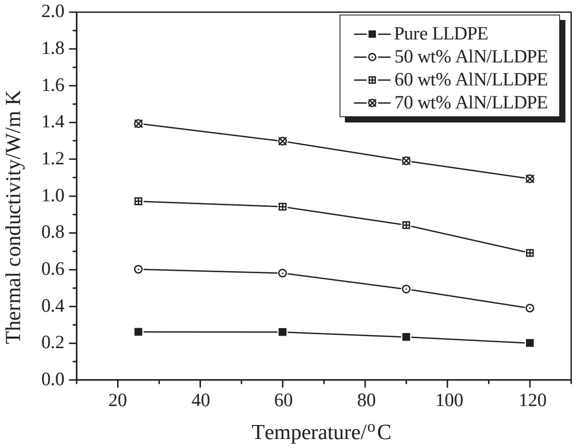

Fig. 5. The thermal conductivity of the composites as a function of temperature.

3.3. Dielectric property

The dielectric properties of AlN/LLDPE composites as a function of filler weight percentages at different frequencies at room temperature are plotted in Fig. 7. As shown in Fig. 7(a) the dielectric constants were almost independent of the frequency in the measured range from 10^2 to 10^7 Hz, and they increased with increasing the AlN particles loading, moreover, they kept at rather low level, i.e., generally <4.0. When the frequency of applied electric field is above the relaxation frequency of a polarization mechanism, the

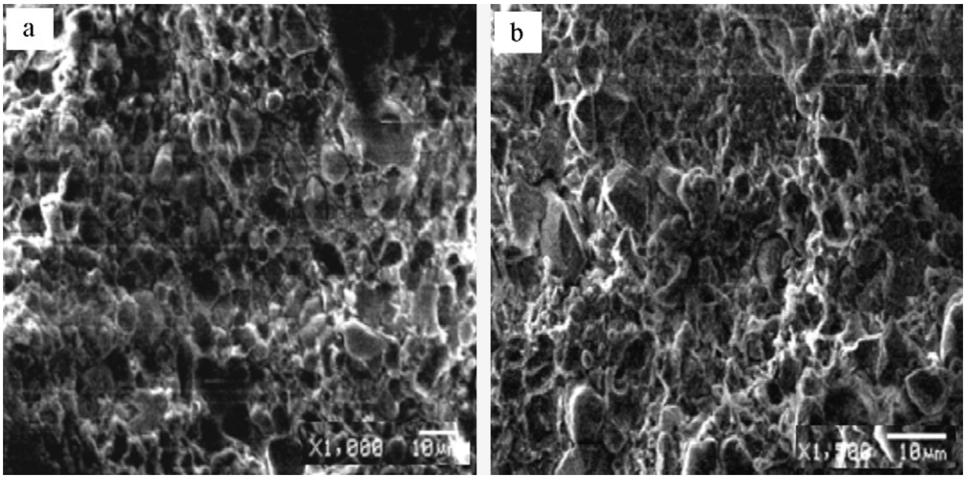

Fig. 6. Microstructure of AlN/LLDPE composites at the AlN weight of (a) 50% and (b) 70%.

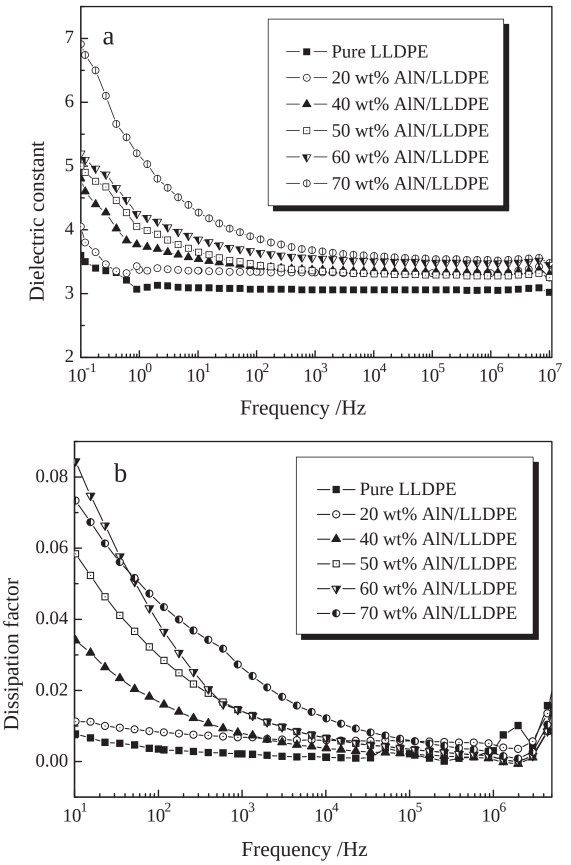

Fig. 7. Dielectric properties of filled LLDPE as a function of AlN concentration: (a) Dielectric constant and (b) dissipation factor.

contribution from this polarization mechanism will be dramatically reduced because this polarization is too slow to completely follow the oscillation of the applied electric field. The frequency independence behavior of AlN/LLDPE composites indicates the major polarization mechanism contributing to the dielectric constant does not change over the measured range. In contrast, the dielectric constant of the composites containing filler content >40 wt% began to rise below 100 Hz, and continued to rise to 5, 5.2 and 6.9 at 0.1 Hz, for the 50, 60, and 70 wt%, respectively. The decrease in the dielectric constant with an increase in frequency is ascribed to the reason that the interfacial dipoles have less time to orient themselves in the direction of the alternating field.

The data in Fig. 7(b) indicate that the dissipation factors of the composites with AlN filler (<20 wt%) were almost independent of frequency in the measured range from 10 to 10^7 Hz, whereas those of the other systems increased with decrease in the frequency in the measured range. In generally, the dissipation factors of the prepared composites remained at a rather low level, i.e., <0.085 at frequency of 10 Hz for the composites with 60 and 70 wt% AlN.

The dissipation factors of AlN/LLDPE all exhibited initial decreases with increasing frequency, followed by an increase to a high frequency. As the frequency increased further, the dipole polarization effects reduced, and the values of the dissipation factor declined accordingly. The dielectric loss tangent tends to increase again when the frequency is above10^6 Hz, this is an obvious relaxation loss process related to the LLDPE [32]. The increase in dissipation factor with an increase in AlN content is considered a consequence of interfacial polarization [33].

The effect of surface modification of AlN particles on the dielectric properties of AlN/LLDPE as a function of filler concentration and frequency is illustrated in Fig. 8. Fig. 8(a) suggests that the surface treated AlN particle filled LLDPE composites exhibits a slight higher dielectric permittivity at high filler content, as compared to that without surface treatment filled one. At low filler content, i.e., <20 wt%, the surface modification almost has no appreciable influence on the dielectric permittivity. The reason may be that, at low filler concentration the filler particles were segregated or encapsulated completely by matrix resin, the interfacial polarization effect was weak despite of the surface treatment of filler particles [8]. However, at a high filler concentration the surface modification of filler particles has an appreciable effect on the dielectric permittivity. For example, the dielectric permittivities of the composites with 60 and 70 wt% AlN filler at 0.1 Hz are 6.4 and 7.3, as compared to 5.2 and 6.9 for the untreated filler reinforced those. The main reasons can be ascribed to the fact that the use of coupling agent improved the interfacial adherence strength between filler particles and LLDPE, and decreased the voids and defects at the phase interfaces, thus, leading to an enhanced dielectric permittivity.

Fig. 8(b) presents the dielectric loss factor of AlN/LLDPE composites containing various content of AlN with surface treatment. It can be seen that the dissipation factors exhibited almost unchanged compared with the untreated filler reinforced one. The surface modification of AlN particles slightly decreased the dielectric loss factor values at high filler loading. The reason may be that the motor ability of the polarization units at the interface area has been enhanced due to the improved interface adherence, which leads to a decreased energy loss used to overcome the internal friction in composites.

Therefore, the dielectric analysis demonstrates that the AlN/LLDPE composites have possessed rather low dielectric constant and dissipation factor in the measured frequency range from

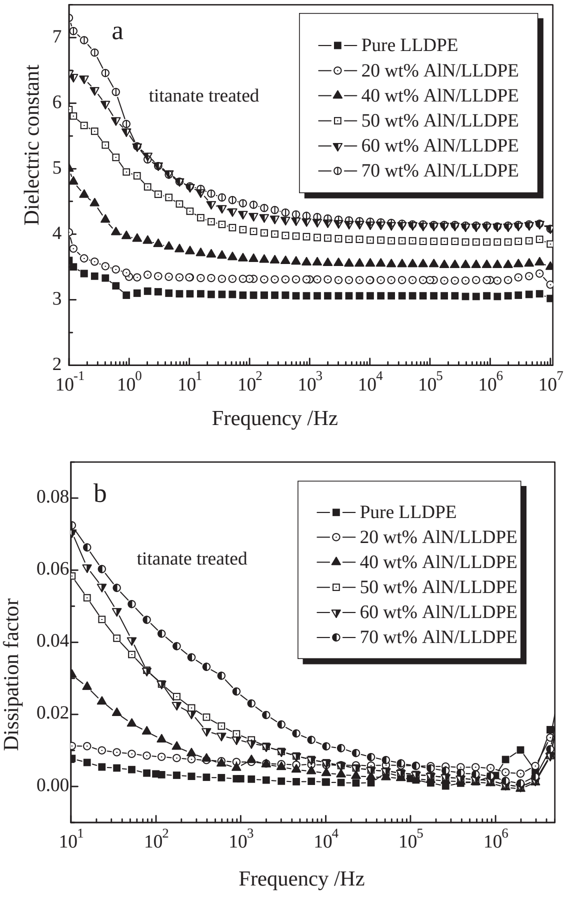

Fig. 8. Effect of surface modification of AlN on the dielectric properties of AlN/LLDPE: (a) dielectric constant and (b) dissipation factor.

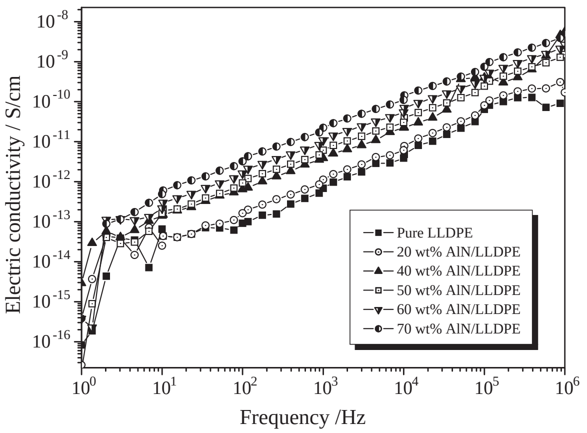

Fig. 9. Electric conductivity of filled LLDPE as a function of AlN concentration and frequency.

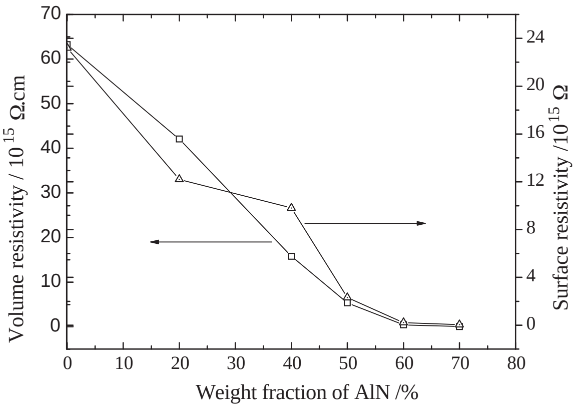

Fig. 10. Electrical insulation property of filled LLDPE as a function of AlN concentration.

10^1 Hz to 10^7 Hz, which is very important for the LLDPE packaging materials.

The ac electric conductivity is presented in Fig. 9. Fig. 9 reveals that the electric conductivity increased with increase in AlN concentration and frequency. For the 70 wt% AlN/LLDPE composites the electric conductivity increased from 2.36×10^{-17} S/cm at 1.0 Hz to 4.96×10^{-9} at 10^6 Hz. At lower frequency the AlN/LLDPE composites exhibited a low electric conductivity because of the excellent electrical insulation of AlN and LLDPE.

The dependence of electrical insulation property of AlN/LLDPE composites on the weight percentage of filler is presented in Fig. 10. It suggests that both the volume and surface resistivity continuously declined with increasing filler concentration, and reached 6.2×10^{13} Ω cm and 3.1×10^{13} Ω at 70 wt% AlN content. The reason may be because that the electric conductivity of LLDPE is greater than that of AlN and, according to the rules of mixture, the electric conductivity of the composites should lie in between those of the filler and matrix. The composites created for the present study still possesses rather good electrical insulation and should be suitable for substrate and packaging materials.

4. Conclusion

A thermal conductive and insulating plastic composite was prepared using LLDPE as matrix and AlN as filler by heat press molding.

The AlN particle decreases the degree of crystallinity, whereas, improves the thermal stability of LLDPE, and it has no appreciable influence on the melting temperature of LLDPE. The thermal conductivity of the LLDPE composites reached 1.25 W/m K at 70 wt% AlN content. Moreover, the surface treatment of AlN with titanate coupler enhanced the thermal conductivity. The thermal conductivity slightly decreased with raising temperature.

Both the dielectric constant and dissipation factor increased slowly as an increase in AlN content, however, they still remained at relatively low levels in wider frequency range from10 to 10^6 Hz. The AlN/LLDPE composites possessed very low ac electric conductivity and high electrical insulation properties at high filler concentration. The use of titanate coupler has an influence on the dielectric properties of the AlN/LLDPE composites at high filler concentration, the surface treatment of AlN improved the dielectric permittivity, whereas, the dissipation factor was less affected.

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