

Research Article

Microstructure and Properties of SCE- Al_2O_3 /PES-MBAE Composite

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SCE- Al_2O_3 was the nano- Al_2O_3 modified by supercritical ethanol and the surface of SCE- Al_2O_3 was coated with active group. 4,4'-diaminodiphenylmethane bismaleimide (MBMI) was used as matrix; 3,3'-diallyl bisphenol A (BBA) and bisphenol-A diallyl ether (BBE) were used as reactive diluent, polyethersulfone (PES) as toughening agent, and SCE- Al_2O_3 as modifier; SCE- Al_2O_3 /PES-MBAE nanocomposite was prepared through in situ sol-gel method. The mechanism of composite toughened by PES was observed and analyzed. FTIR indicated that the reaction between MBMI and allyl compound occurred and SCE- Al_2O_3 had doped into the polymer matrix. SEM showed that PES particle was inlaid in matrix and presented as a two-phase structure in matrix. The heat resistance, dielectric properties, and mechanical properties of SCE- Al_2O_3 /PES-MBAE nanocomposites were evaluated. The results showed that with the incorporation of PES, although the toughness of the material improved, the heat resistance and dielectric properties of material declined, meanwhile. The adulteration of SCE- Al_2O_3 could remedy the harmful effect caused by PES, while the content of SCE- Al_2O_3 was reasonable. The decomposition temperature, dielectric constant, and dielectric loss of composite were 441.23°C, 3.63 (100 Hz), and 1.52×10^{-3} (100 Hz); the bending strength and impact strength were 129.22 MPa and 13.19 kJ/mm², respectively, when the content of SCE- Al_2O_3 was 3 wt% and PES was 5 wt%.

1. Introduction

Bismaleimide (BMI) is a bifunctional compound and is derived from polyimide resin and is used in many fields such as aerospace, machinery, and electronics due to the outstanding processability, excellent insulativity, and high thermostability. But unmodified BMI cannot meet the requirements of processing and using because BMI has great brittleness and bad shock resistance. The toughening modification of BMI has become a hot topic at present. Polyethersulfone resin (PES) is a high-performance thermoplastic resin that has good toughness, high modulus, excellent heat resistance, and outstanding resistance to aging [1–3]. PES-BMI material will be synthesized and the thermostability will not decline and the toughness will increase. But BMI could not mix with PES well because BMI has high viscosity and the toughness of composite will become poor when the viscosity of mixture is lower. Allyl compound could react with BMI and form

cross-linked structure, and allyl compound could also adjust the viscosity of mixture. Additionally, nanocomposite has much large surface area and shorter interparticle distance at the same particle content and results in superior properties. Nanoparticles may have a significant impact on the electrical properties, heat resistance, and mechanical properties of the composites, but the nano- Al_2O_3 particles are prone to reunite in the matrix resin and scattered unevenly, since the surface energy of nano- Al_2O_3 is huge and the instability, and the surface of nano- Al_2O_3 is oleophobic, the excellent performances of nano- Al_2O_3 cannot develop entirely [4, 5]. The surface modification can enhance the dispersion of nanopowders and it becomes a hot point of nanocomposite manufacturing technology [6]. Supercritical ethanol (SCE) has excellent properties of supercritical fluid and the reaction condition is easy to achieve (241°C, 6.14 Mpa); ethanol molecules can adhere to the surface of nanocomposite by the special effects of SCE. 4,4'-diaminodiphenylmethane

bismaleimide (MBMI) was used as matrix and 3,3'-diallyl bisphenol A (BBA) and bisphenol-A diallyl ether (BBE) were used as reactive diluent, polyethersulfone (PES) as toughening agent, and SCE- Al_2O_3 as modifier in this paper. The microstructure and mechanism were studied by SEM and FT-IR; the heat resistance, dielectric properties, and mechanical properties of SCE- Al_2O_3 /PES-MBAE nanocomposites were evaluated and provided experimental data and theoretical basis for the development and application of BMI. Refer to Figure 1 for the chemical structure of MBMI, BBA, and BBE.

2. Experimental

2.1. Materials. 4,4'-diaminodiphenylmethane bismaleimide (MBMI), 3,3'-diallyl bisphenol A (BBA), and bisphenol-A diallyl ether (BBE) were all purchased from Laizhou laiyu chemical co., LTD. Polyethersulfone (PES, molecular weight 30,000, intrinsic viscosity 0.32) was from Changchun Jida Special Plastic Engineering Co., Ltd. Nano- Al_2O_3 was provided by Hangzhou Wanjing new materials co., LTD.

2.2. Measurements. The fracture structures of samples were examined on the HITACHI S-4300 scanning electron microscope (SEM, JAPA). Samples were deposited on a sample holder with adhesive carbon foil and were sputtered with gold.

The FT-IR spectra, which were used to study the chemical structure of the polymer matrix, were performed with a EQUINOX-55 Fourier transform spectrometer (GER), in the 400–4000 cm^{-1} range and 5 scans were averaged for each spectrum. And it could be seen that there existed the characteristic absorption peaks of the material.

Thermogravimetric analysis (TGA), which not only could investigate the state of a material and the process of decomposition but also could provide useful information about the thermal stability of material, was recorded on a Perkin-Elmer 6 series thermal analysis system (USA); samples were heated from 200 to 600°C at a heating rate of 10°C/min in a nitrogen atmosphere and the weight of sample was about 10–15 mg.

Dielectric constant (ϵ) and dielectric loss ($\tan\delta$) of composite material were measured with Agilent-4294A precise impedance analyzer (Japan) in the frequency range of 100 Hz–100 kHz at room temperature according to GB/T 1409-89.

The bending strength of the composite materials was determined by CSS-44300 electronic testing machine (Shanghai technical instrument technology co., LTD, CHN) according to a native standard of GB/T 2918. Standard of bending strength experiments were performed at room temperature, under a speed of 2 mm/min. For each sample, five measurements were made at least and the average value was taken.

The impact strength of the composite materials was measured by CBJ-11J Charpy Impact Machine Tester (Jinan huaxing laboratory equipment co., LTD) according to a native standard of GB/T 1043-93. For each sample, five measurements were made at least and the average value was taken.

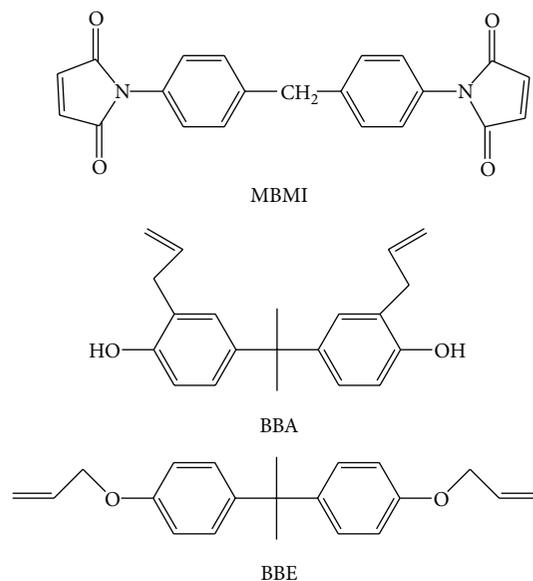


FIGURE 1: The chemical structure of MBMI, BBA, and BBE.

2.3. Surface Treatment of Nano- Al_2O_3 . Nano- Al_2O_3 was dried at 80°C for 10 h in an oven before being used; 5.00 g Al_2O_3 was added into a supercritical reactor at first, then 50 ml anhydrous ethanol was added and stirred and the reactor should be sealed. Put the reactor into heated pool, while the heated pool reached 241°C and was kept five minutes when the pressure of reactor reached 6.14 MPa. Then take out the reactor and put it into the cooling bay. Take out the Al_2O_3 and dry it for 8 h at 80°C in a vacuum oven while the reactor is cooled. The structure of supercritical device is shown in Figure 2.

2.4. Preparation of Composite Materials. A three-necked flask was charged with BBA (6 g) and BBE (4 g) and the solution was stirred for 10 min at 80°C. The SCE- Al_2O_3 was added into flask and stirred for 1 h at 80°C, under ultrasonic resonance. The mixed solution was heated to 170°C while the solution was uniform, then PES was added into the solution and stirring was kept until the PES was completely dissolved. The mixture was cooled to 140°C when the solution became homogeneous, then MBMI (20 g) was added and stirred for 30 min. The mixture was decanted into the mould which was already preheated well. Put the mould into oven for curing. Curing process was carried out at five stages, 130°C/1 h, + 150°C/1 h, + 180°C/1 h, + 200°C/1 h, + 220°C/2 h. The samples were numbered as in Table 1, in order to be expressed clearly. A represented the matrix and B meant the composite in which the content of PES was 5 wt%.

3. Results and Discussions

3.1. Analysis of SEM Patterns. SEM was always used to analyze microscopic structure of the composite material and the microstructures of composites are shown in Figure 3. Figures 3(a) and 3(b) were the SEM images of matrix, Figures

TABLE 1: The type and component of composite.

Type	Component	PES content (wt%)	SCE-Al ₂ O ₃ content (wt%)
A	MBMI/BBA/BBE	0	0
B0~B6	SCE-Al ₂ O ₃ /PES/MBMI/BBA/BBE	5	0.1.2.3.4.5.6

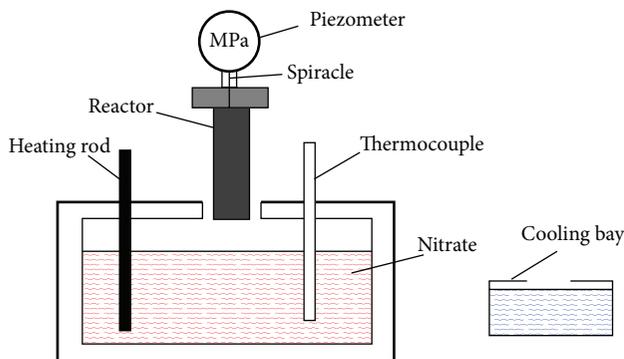


FIGURE 2: The structure of supercritical device.

3(c) and 3(d) were 5 wt% PES-MBAE, and Figures 3(e) and 3(f) were 1 wt% SCE-Al₂O₃/5 wt% PES-MBAE.

Figures 3(a) and 3(b) showed that the surface of fracture of matrix had no obvious microcracks and was typical morphology of brittle fracture, compared to all SEM images. The reason was that the chemical structure of BMI was symmetric and the crystallinity was high and this could lead the material to be brittle [7, 8]. There existed many particles in Figures 3(c) and 3(d) and the size of particle was about 1 μm and the particles were PES resin. It could be seen from Figures 3(c) and 3(d) that the PES particles were inlaid in matrix and presented as a two-phase structure in matrix. The fracture surface of PES-MBAE had obvious microcracks and the fracture was ductile; the reason was that the PES phase dispersed in continuous MBAE phase evenly and the interaction between two phases was strong. Material would crack along the direction of the force when the material is subjected to an external force. A large number of microcracks were formed because the interface between two phases combined well and played a role of stress raiser and shear zone was induced by PES particle, meanwhile. The formation of microcracks and shear zone could absorb a quantity of energy, which caused ductile fracture [9]. The images of SCE-Al₂O₃/PES-MBAE composite were different from others, while being compared to all images. The fracture surface of SCE-Al₂O₃/PES-MBAE composite had no obvious interface and obvious microcracks existed, which was different from PES-MBAE matrix. This phenomenon could be explained as follows: the nano-Al₂O₃ modified by supercritical ethanol would adsorb ethanol molecules, and ethanol molecules could combine PES, nano-Al₂O₃, and MBAE, which caused the absence of two-phase structure. The ductile fracture occurred also due to the mixture of PES resin and produced many microcracks.

3.2. FT-IR Spectral Analysis. FTIR spectra of composite materials were presented in Figure 4. The curves of a, b, c, and d are designated as PES-MBAE, SCE-Al₂O₃/MBAE, SCE-Al₂O₃/PES-MBAE, and MBMI powder.

The peak of curve b at 3100 cm^{-1} was due to the stretching vibration of C=C in MBMI, but it disappeared in curves a, b, and c. The absence confirmed the presence of a chemical reaction between MBMI and allyl compound. The reaction has taken place via Ene reaction and Diels-Alder reaction according to Figure 5. The Ene reaction took place at low temperature (130°C) and the intermediate formed in this reaction. The Diels-Alder reaction took place at high temperature (220°C) and cross-linked structure was formed. The small peak at 1299 cm^{-1} of curves a and c was due to the antisymmetric stretching vibration of SO₂ and the peaks at 1100 cm^{-1} and 710 cm^{-1} were due to stretching vibration of -S- and C-S stretching vibration, respectively [10–12]. All of these indicated that the material was mixed with PES. But these peaks were not obvious, because PES resin presented a two phase structure in MBMI resin, the particles of PES were coated with MBMI, and there was a strong interaction between the active groups of PES and MBMI. The peak of curves b and c at 599 cm^{-1} and 649 cm^{-1} were due to the stretching vibration of Al-O and it indicated that SCE-Al₂O₃ molecule could interact with the MBAE matrix. The results of FTIR suggested that SCE-Al₂O₃/PES-MBAE composite had been prepared.

3.3. Thermal Stability of Composites. Thermal degradation behaviors of composites were evaluated by thermogravimetric analysis (TGA). TGA curves of materials were shown in Figure 6 and the obtained T_d , T_d^5 , and T_d^{10} temperatures (onset decomposition temperature and the decomposition temperatures at 5 wt% and 10 wt% weight losses of composites under nitrogen atmosphere) are listed in Table 2.

Thermal stability of composites was subject to PES and SCE-Al₂O₃ and was declined when MBMI resin was mixed with PES, and the residual weights of SCE-Al₂O₃/PES-MBAE composites were higher than that of MBAE in Figure 6. Through research, it was found that the heat resistance of nanocomposites increased to some extent with the increasing of SCE-Al₂O₃ content. The reasons were as follows: the effect of nanoparticles on composite thermal performance was multifaceted [13]. It was commonly believed that the introduction of inorganic components in polymer matrix resin would help to improve the thermal stability of polymer material, but this must be related to binding force among the two phases, interface properties, and inorganic doping quantity [14]. The structure of nanoparticles determined

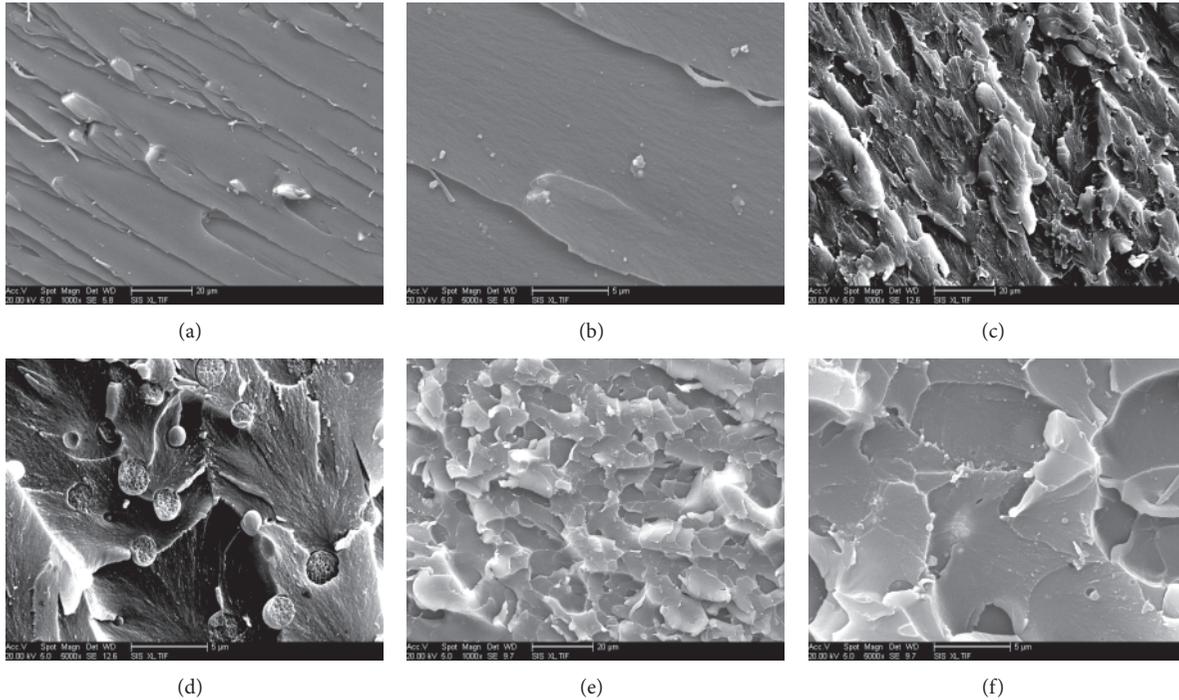


FIGURE 3: SEM images of composite materials.

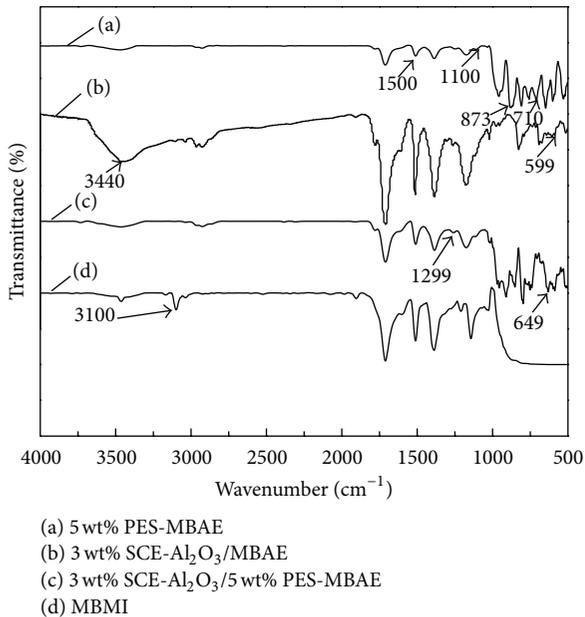


FIGURE 4: FTIR spectra of composite materials.

their excellent physical and chemical properties and Al₂O₃ had very high bond energy, so Al₂O₃ could contribute to the excellent thermostability of the composite. Above all, using SCE-Al₂O₃ to modify PES-MBAE could replenish the thermostability of SCE-Al₂O₃/PES-MBAE.

The T_d^5 ranged from 421.18 to 441.18°C and the T_d^{10} was in the range of 446.28–457.09°C, along with the doping

TABLE 2: Decomposition temperature of composite.

Type	T_d (°C)	T_d^5 (°C)	T_d^{10} (°C)
A	438.35	432.84	449.06
B0	423.89	421.18	446.28
B1	437.00	431.80	448.68
B2	439.91	432.70	449.61
B3	441.23	435.11	452.01
B4	444.41	441.18	457.09
B5	443.31	442.04	456.25
B6	442.85	439.56	454.46

quantity of SCE-Al₂O₃ which changed from 0 to 6 wt%. The great changes of T_d^5 and T_d^{10} implied that SCE-Al₂O₃ had great influence to some extent on the thermal stability of composites, which could have also resulted in the change of the decomposition temperatures (423.89–444.41°C).

The T_d , T_d^5 , and T_d^{10} of MBAE matrix were 438.35, 432.84, and 449.06°C, respectively. The thermostability of PES-MBAE material declined when the MBAE matrix was mixed with PES, because the PES resin itself was thermal plastic resin including more flexible chain and had low thermostability. It could be seen from Table 2 that the decomposition temperature of composites increased with the increasing of SCE-Al₂O₃ content at first and declined while the content of SCE-Al₂O₃ was beyond 4 wt%. The decomposition temperature of SCE-Al₂O₃/PES-MBAE composites was higher than that of MBAE matrix and it reached the highest (444.41°C) when the content of SCE-Al₂O₃ was 4 wt%. This phenomenon can be explained as follows: on one

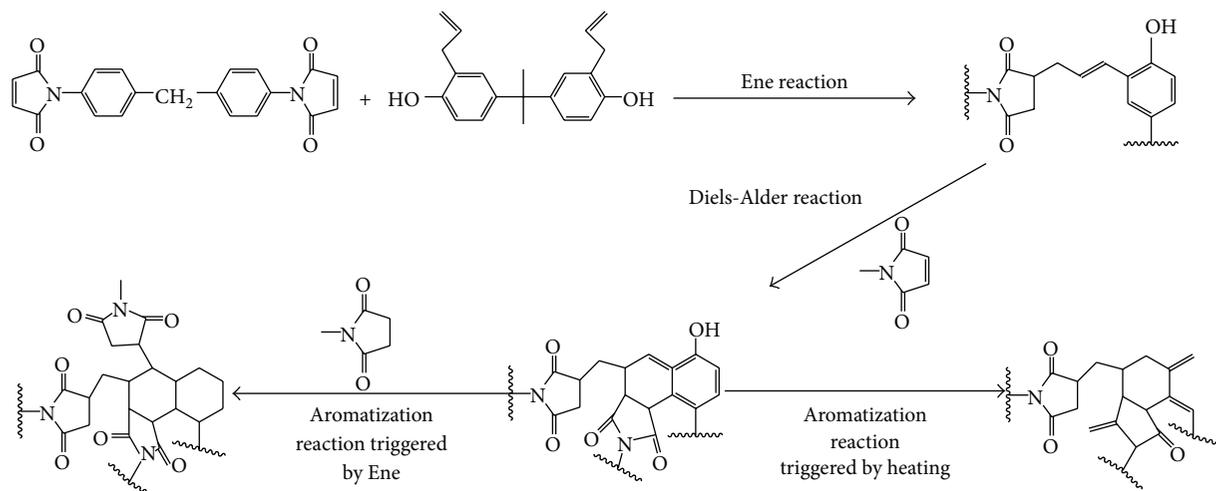
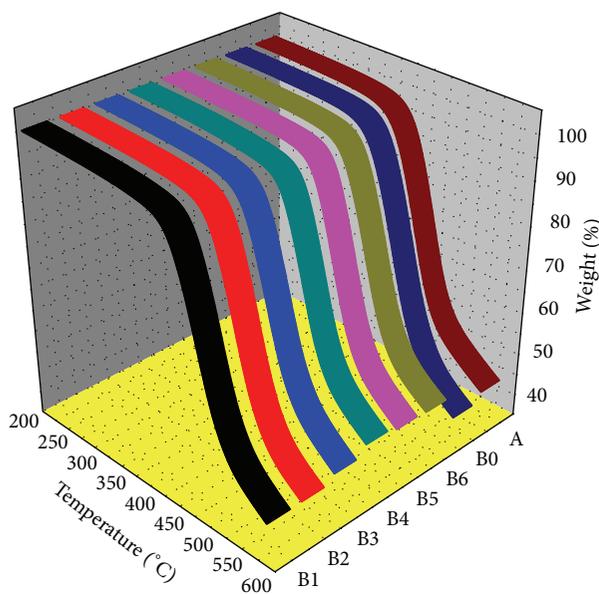


FIGURE 5: Reaction of MBMI and allyl compound.

FIGURE 6: TG curves of SCE-Al₂O₃/PES-MBAE composites.

hand, the heat resistance property of inorganic nanoparticle was excellent and the introduction of SCE-Al₂O₃ would make a contribution to the heat resistance of composite. On the other hand, the surface of Al₂O₃ would adhere with ethanol molecules when Al₂O₃ was modified by supercritical ethanol and the reunion of SCE-Al₂O₃ particles would reduce because the active groups of SCE-Al₂O₃ particle could adsorb ethanol molecules. Many effective interface areas would form by the interaction between SCE-Al₂O₃ and MBAE matrix, while SCE-Al₂O₃ particles dispersed in MBAE matrix, evenly, and the interface areas would absorb energy when the material was heated. The decomposition temperature of the material became higher when the interface areas absorbed energy [15–17]. The collision between nanoparticles would

increase and lead reunion to occur when the content of SCE-Al₂O₃ was too much. The reunion of inorganic particles could destroy the cross-linked network structure and the thermostability of composites, therefore, declined.

3.4. Dielectric Properties of SCE-Al₂O₃/PES-MBAE Nanocomposites. MBMI resin had low dielectric constant (ϵ) and dielectric loss ($\tan\delta$), which was regarded as important characteristics of the dielectric materials. Although MBMI had plenty of polar groups in its molecular chains, the chains movements were restricted due to their location on the benzene ring and their symmetry position. Generally, MBMI was regarded as a nonpolar resin and had good dielectric properties [18, 19].

The internal factors of influencing dielectric constant were the condition of dielectric polarization and polarization divided into electronic polarization, atomic polarization, orientation polarization, and interfacial polarization according to different molecular mechanisms of polarization [20]. The dielectric constant of polar molecule depended on the orientation of polarization and increased with the increasing of polarization extent.

Figure 7 displayed dielectric constant curves of SCE-Al₂O₃/PES-MBAE nanocomposites. It was observed that the dielectric constant of the composite materials had decreased slightly with the increase of frequency (from 10¹ to 10⁷ Hz) in the same doping quantity. This phenomenon was because the diversion of dipole could keep pace with changes in the electric field and when the electric field was in a low frequency, the diversion of dipole would fall behind the changes in the electric field due to the viscous effect, in high frequency (higher than 10⁵ Hz), and then the orientation polarization decreased and caused dielectric constant decline.

The dielectric constant increased rapidly, while MBAE resin mixed with PES resin. The reasons were that PES was polar polymer and increased polar group density of the composite material and the polar groups could make a material have higher polarization strength, to cause the

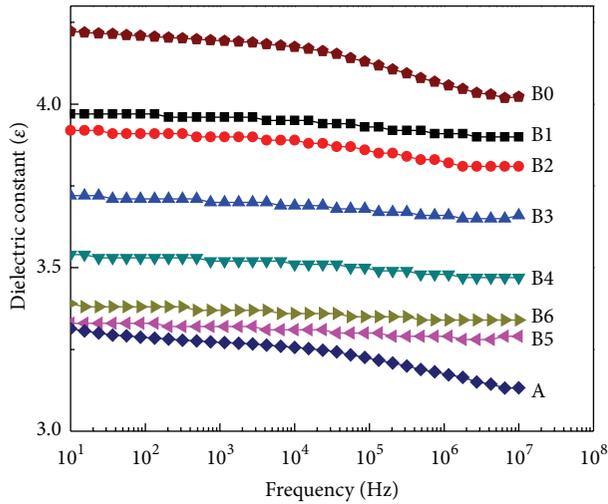


FIGURE 7: The dielectric constant curves of SCE- Al_2O_3 /PES-MBAE composites.

increase of dielectric constant in electric field. And as PES resin presented as a two-phase structure in MBAE resin, the interface between PES and MBAE matrix could cause interfacial polarization. Therefore, the dielectric constant increased due to the comprehensive action of orientation polarization and interfacial polarization.

The dielectric constant of composites was declined with the increasing of SCE- Al_2O_3 content, entirely. This phenomenon was opposite to theory and the reasons were as follows: on one hand, the dielectric constant depended on orientation polarization since SCE- Al_2O_3 was polar molecule. The surface of SCE- Al_2O_3 was coated with ethanol molecule and the polar groups of SCE- Al_2O_3 would adsorb ethanol molecules, which led the polarity of SCE- Al_2O_3 to decline. The orientation polarization would reduce due to the decrement of polarity and the dielectric constant of composite would decrease, finally. On the other hand, the active groups that adhered to the surface of SCE- Al_2O_3 played a role of bridge. Nanoparticles, PES, and MBAE matrix were linked by ethanol molecule and formed a structure like lattice orientation. The orientation polarization reduced and led dielectric constant to diminish [21]. The polar groups and high dielectric constant (10.51) of Al_2O_3 influenced the dielectric constant and exceeded the effect of ethanol molecule when the content of SCE- Al_2O_3 was too much.

Figure 8 exhibited the relations of the dielectric loss of composites with the SCE- Al_2O_3 quantity, while the frequency was between 10^2 Hz and 10^5 Hz. The dielectric loss of SCE- Al_2O_3 /PES-MBAE generally increased with the increase of frequency, and the change was insignificant for the composites of various doping quantities while the testing frequencies were between 10^2 and 10^4 Hz. The dielectric loss of the composites increased significantly when the frequency changed from 10^4 to 10^5 Hz. This was because the dielectric loss of the composites mainly came from the conduction loss of polar groups and relaxation loss [22]. The dielectric

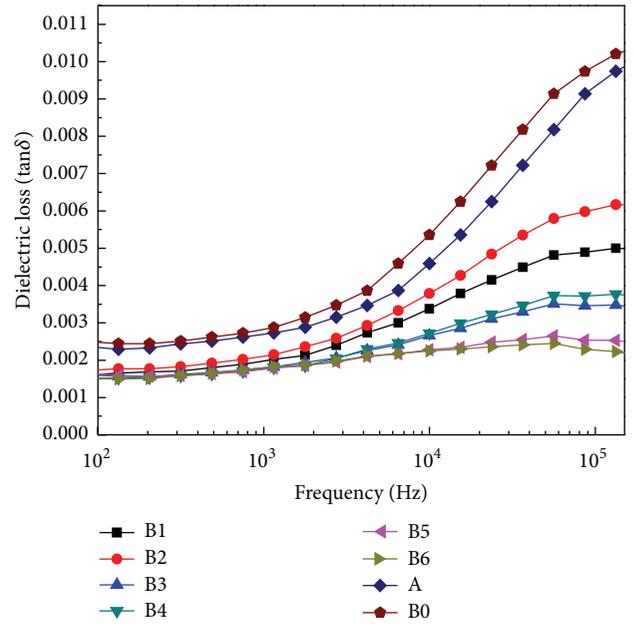


FIGURE 8: The dielectric loss curves of SCE- Al_2O_3 /PES-MBAE composites.

loss increased slowly at low frequency range, because the dipole steering could completely keep up with the change of the electric field; the energy that was absorbed by dipole could return to electric field and dielectric loss would increase slowly. The polarization could not keep up with electric field change in high frequency because dipole steering was affected by internal viscosity and friction resistance; dielectric would generate heat as dipole steering consumed energy, when dipole steering was overcoming friction resistance; this resulted in the augment of dielectric loss.

It could be seen from Figure 8 that the dielectric loss of PES-MBAE material was the highest, MBAE was the second, and the dielectric losses of SCE- Al_2O_3 /PES-MBAE nanocomposites were lower than that of others. This phenomenon could be explained as follows: the cross-linking density declined because the PES resin presented as a two-phase structure in MBAE resin, and the friction resistance increased due to the interaction between PES resin and MBAE matrix. The more energy would be needed while the dipole steering was overcoming friction resistance and the dielectric loss of PES-MBAE, therefore, increased [23]. The surface of SCE- Al_2O_3 would be coated with ethanol molecules while Al_2O_3 was modified by supercritical ethanol; the dispersion phases in the system were combined by ethanol molecule and formed a structure like lattice orientation, while the matrix mixed with SCE- Al_2O_3 . The ability of dipole and dielectric loss decreased, but SCE- Al_2O_3 itself was a conductive carrier, and conductivity loss would occur because the conductive carrier would move and consume energy. The effect of conductive carrier would exceed that of ethanol molecule while the content of SCE- Al_2O_3 was too much and it produced a similar trend with dielectric constant.

TABLE 3: Mechanical properties of composites.

Type	Bending strength (Mpa)	Increasing rate (%)	Impact strength (kJ/mm ²)	Increasing rate (%)
A	100.98	0	9.13	0
B0	135.99	34.67	11.34	24.21
B1	141.21	39.83	11.55	26.51
B2	138.62	37.27	12.99	42.27
B3	129.22	27.96	13.19	44.47
B4	116.12	14.99	11.1	21.58
B5	100.27	-0.71	9.57	4.82
B6	97.55	-3.39	8.66	-5.15

3.5. Mechanical Properties of Composites. Bending strength and impact strength were two important indicators of mechanical properties. The values of bending strength and impact strength are presented in Table 3.

The bending strength ranged from 97.55 to 141.21 MPa and the impact strength was in the range of 8.66~13.19 kJ/mm². The great changes of bending strength and impact strength indicated that SCE-Al₂O₃ had a great influence to some extent on the mechanical properties of composites.

The bending strength and impact strength of the matrix were 100.98 MPa and 9.13 kJ/mm², respectively. The reason was that the chemical structure of MBMI was symmetric and the crystallinity was high and the brittleness of unmodified MBMI was great. The increasing rate of bending strength and impact strength were 34.67% and 24.21%, and the mechanical properties of composites enhanced obviously while the matrix was mixed with PES, compared A and B0 in Table 3. PES resin could inlay in MBMI matrix and it presented as a two-phase structure according to the SEM results, and the toughening theory of PES was a two-phase toughening mechanism. The interface formed due to the interaction between continuous phase (MBAE) and dispersed phase (PES), and microcracks generated in the interface when the material was under external force and the shear zone would be induced by PES particle, meanwhile. The formation of microcracks and shear zone could absorb a quantity of energy, and the interface would also assume a certain amount of fracture energy, which prevented the formation of destructive cracks. It could be seen from Table 3 that the bending strength and impact strength of composite would be enhanced in further step when mixed with SCE-Al₂O₃, and the content of SCE-Al₂O₃ should not be too high. The bending strength of B1 was the highest of all samples to reach 141.21 MPa and was 39.83% higher than that of matrix. The impact strength of B3 was the highest, and the increasing rate was 44.47%. The increasing rate was small or even decreased while the content of SCE-Al₂O₃ exceeded 5 wt%. The reason was that the structure of material would be regular and orderly when mixed with SCE-Al₂O₃, and the influence of incorporation of SCE-Al₂O₃ on mechanical property was twofold; on one hand, the mixing of SCE-Al₂O₃ could combine PES and nano-Al₂O₃ and MBAE was due to the ethanol molecules that adhered in the surface of SCE-Al₂O₃ particles, which made a contribution to mechanical property; on the other hand, the mixing of SCE-Al₂O₃ would produce defects and cause

macroscopic fracture because the reunion would occur while the content of SCE-Al₂O₃ was higher, and the reunion could lead the mechanical property of composite to decline, finally.

4. Conclusion

- (1) The fracture morphology of bismaleimide (MBMI) is typical brittle fracture and it becomes ductile fracture while MBMI mixed with polyethersulfone (PES). PES presents as a two-phase structure in MBMI matrix and the two-phase structure would disappear when mixed with SCE-Al₂O₃.
- (2) The cross-link structure has formed by the reaction between MBMI and allyl compound. Incorporation of polyether ulfone (PES) could improve the toughness of the material, the heat resistance, and dielectric properties and voltage resistance of material would decline, meanwhile. SCE-Al₂O₃ could remedy the harmful effect caused by PES, while the content of SCE-Al₂O₃ is reasonable. When the content of SCE-Al₂O₃ is 3 wt% and PES is 5 wt%, the decomposition temperature, dielectric constant, and dielectric loss of composite are 441.23°C, 3.63 (100 Hz), and 1.52 10⁻³ (100 Hz); the bending strength and impact strength are 129.22 MPa and 13.19 kJ/mm², respectively.
- (3) Inorganic nanoparticles modified by supercritical fluid should be explored further and the effect of nanoparticles and PES should be studied in deep step. The toughness of composite material should be improved in future by changing the chemical structure of MBMI, using whisker, and others.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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