

## Research Article

# Characteristics and Properties of TiO<sub>2</sub>/EP-PU Composite

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Polymer matrix of EP-PU was prepared by epoxy resin which was polyurethane toughened, and TCA201 coupling agent was used to modify nano-TiO<sub>2</sub>, and TiO<sub>2</sub>/EP-PU composite was synthesized using EP, PU, and TCA201-TiO<sub>2</sub>. The results of SEM and TEM showed that the surface of TiO<sub>2</sub> was coated with TCA201 coupling agent through the bonding between the hydroxyl of nano-TiO<sub>2</sub> particle and coupling agent molecules, the interaction would be beneficial to improve compatibility of inorganic and organic phases, and TCA201-TiO<sub>2</sub> would disperse evenly in composite and improve performance of composite materials. The mechanical properties, thermal stability, dielectric properties, and breakdown strength of composites were investigated by electronic tensile machine, TGA, dielectric spectrum, and CS2674C type voltage tester. The results indicated that appropriate amount of TCA201-TiO<sub>2</sub> could improve mechanical properties, the shear strength of 3 wt% TiO<sub>2</sub>/EP-PU reached the maximum value at 27.14 MPa, its thermal decomposition temperature was 397.82°C, enhanced 17.48°C more than that of EP-PU matrix, and its dielectric constant ( $\epsilon$ ) and dielectric loss ( $\tan \delta$ ) showed 4.27 and 0.02, respectively. Its breakdown field strength was 14 kV/mm. Its performance met the requirement of dielectric materials.

## 1. Introduction

Epoxy resin (EP) is one of the most commonly used thermosetting materials in high voltage apparatus as insulation due to its excellent mechanical, electrical properties and chemical stability. EP adhesive with excellent bonding properties is widely used in aerospace, military, electronics, construction, and many other fields [1]. The main purpose of this study was that polyurethane (PU) and inorganic nano-TiO<sub>2</sub> are used to increase the mechanical strength, thermal stability and improve the dielectric properties of the composites. Polyurethane (PU) was used as the flexibilizer that reinforced toughness of epoxy resin, and the interfacial theory of composite materials was used to analyze the relationship between material structure and properties of composite [2]. Owing to the poor compatibility existing between the interface of organic matrix and inorganic fillers, some efforts were usually necessary to enhance the compatibility between the inorganic fillers and the polymer matrix during the preparation of hybrid materials [3]. To achieve this

objective, TCA201 coupling agent is usually used to modify nano-TiO<sub>2</sub> and the surface of modified nano-TiO<sub>2</sub> has existed in active group (-OH, -COOH), so the relations of inorganic fillers and polymer matrix should be improved and were beneficial to interfacial energy in the formation of hybrid composites. In this research, EP-PU has existed in "island" structure; this structure is in favor of composites toughness [4]. At the same time, the performance of the composites was tested through mechanical properties, thermal stability, and dielectric properties.

## 2. Experimental

**2.1. Materials.** The chemicals used in this study such as epoxy resin (EP-51) and methyl tetrahydrophthalic anhydride (MTHPA) were industrial products and were purchased from the Lanxing New Chemical Materials Co., Ltd., China. Polyurethane (PU) was industrial products and obtained from Jindao Material Sci & Tech Co., Ltd., Beijing, China, and imidazole was industrial products and purchased from Jinyan

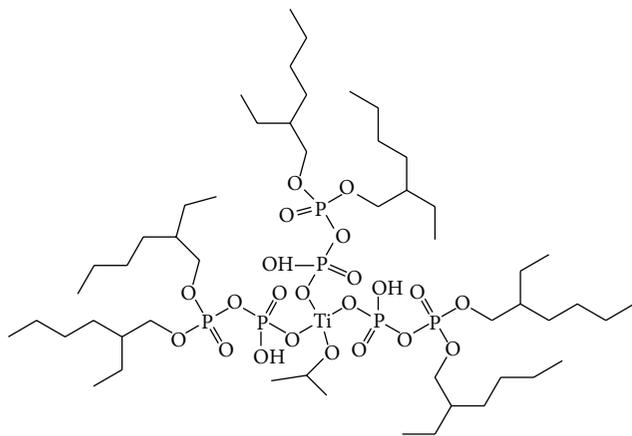


FIGURE 1: The chemical structure of TCA201.

Trading Co., Ltd., Guangzhou, China. The density of  $\text{TiO}_2$  is  $0.42 \text{ g/cm}^3$ , the size of particle is 25 nm, and the melting point of  $\text{TiO}_2$  is 1830–1850°C and comes from Yicheng Jingrui New Material Co., Ltd., China. Coupling agent of titanate (TCA201) is brought from Jing Tianwei Chemical Co., Ltd., Nanjing, China. The structure of isopropyl tri(dioctylpyrophosphate) titanate (TCA201) was shown in Figure 1.

**2.2. Modification of Nano- $\text{TiO}_2$ .** TCA201 was dissolving in 17 mL toluene and its concentration was 2.6%.  $\text{TiO}_2$  was added into above solution until the system was evenly about 2–4 hours under 80°C and then was leached.  $\text{TiO}_2$  was 10 g. Washing modified  $\text{TiO}_2$  solution with toluene 3–4 times and leaching, put it into the oven and let it dry for 10 hours at 80°C and get TCA201- $\text{TiO}_2$ .

**2.3. Preparation of Material Composites.** Technological process of composite materials was described as follows. A certain percentage of the EP-51 and PU were mixed at 80°C until they dispersed evenly, and a certain amount of TCA201- $\text{TiO}_2$  powder was added into above systems. The solution was dissolving in the ultrasonic condition, cooled to 50°C, and added into MTHPA and imidazole until the system was mixed completely. The mixture was degassed to remove air bubbles and pour into preprepared molds for molding in the oven and curing temperature was 80°C for 2 hr, 120°C, 150°C, and 180°C for 1 hr, respectively.

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

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

The state of aggregation and morphology of nano- $\text{TiO}_2$  were tested by JEM-2100 transmission electron microscopy

(TEM, Japan), the test temperature was 18°C, and voltage was 120 kV.

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

The shearing strength of the composite materials was determined with CSS-44300 electronic testing machine (China) according to a native standard of GB/T7124-1986. Standard tensile shear experiments were performed at room temperature, under a speed of 5 mm/min. For each sample, at least five measurements were made, and the average value was taken.

Thermogravimetric analysis (TGA), which could not only investigate the state of a material and the process of decomposition but also provide useful information about the thermal stability of material, was recorded on a Perkin-Elmer 6 series thermal analysis system. Samples were heated from 200°C to 800°C at a heating rate of 20°C/min in a nitrogen atmosphere and weight of sample was about 10–15 mg.

The dielectric constant ( $\epsilon$ ) and dielectric loss ( $\tan \delta$ ) of composite materials were measured with Agilent-4294A precision impedance analyzer (Japan) in the frequency of 50 Hz at room temperature according to GB/T 1409-89.

CS2674C pressure tester (GER) was used to test the breakdown strength of the composites.

### 3. Results and Discussions

**3.1. FT-IR Spectral Analysis.** FT-IR spectra of nano- $\text{TiO}_2$  were presented in Figure 2. The curves ((a) and (b)) were designated as unmodified  $\text{TiO}_2$  and TCA201- $\text{TiO}_2$ , respectively.

It was seen from Figure 2 that the curve of TCA201- $\text{TiO}_2$  was different from the curve of unmodified  $\text{TiO}_2$ . The peak of curve (b) at 2864  $\text{cm}^{-1}$  and 2934  $\text{cm}^{-1}$  was due to the stretching vibration of C–H in TCA201, and the peak at 1039  $\text{cm}^{-1}$  was the characteristic peak of P–O–P [5]. The peak at 1724  $\text{cm}^{-1}$  was the absorption band of P–O–H, and the peak at 1464  $\text{cm}^{-1}$  was the characteristic peak of P=O. It could prove that TCA201 has been grafted to the surface of  $\text{TiO}_2$ . Chemical interaction of  $-\text{OCH}(\text{CH}_3)_2$  in titanate coupling TCA201 and polar group for nano- $\text{TiO}_2$  would form organic active monolayer, and this could help to facilitate two-phase action between inorganic and organic phase and improve the properties of composites.

**3.2. Analysis of SEM Patterns.** SEM was always used to analyze microscopic structure of the composite material, and the microstructures of nano- $\text{TiO}_2$  and composites were shown in Figure 3.

Figures 3(a) and 3(b) show microtopography of unmodified nano- $\text{TiO}_2$ , TCA201- $\text{TiO}_2$  and Figures 3(c) and 3(d) show fracture SEM picture of 3 wt% (TCA201- $\text{TiO}_2$ )/EP-PU and 7 wt% (TCA201- $\text{TiO}_2$ )/EP-PU, respectively. Unmodified  $\text{TiO}_2$  particles had an irregular shape but uniformly disperse.  $\text{TiO}_2$  particles were of nanoscale and the white area was relatively big, so the clusters phenomenon existed (see Figure 4(a)). The surface that TCA201- $\text{TiO}_2$  was uniformly

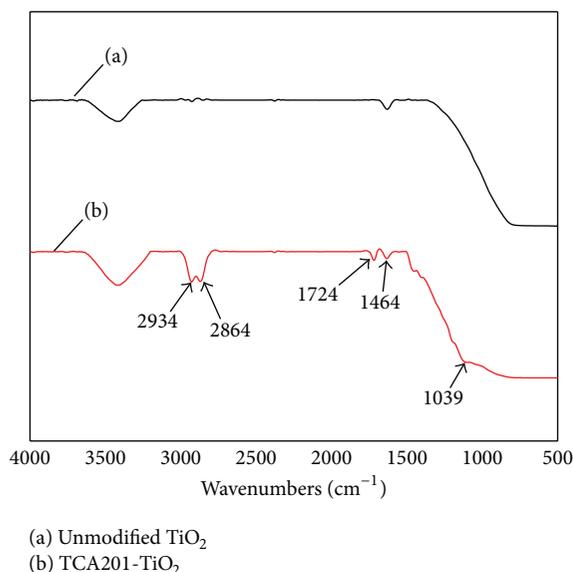


FIGURE 2: FT-IR spectra of nano-TiO<sub>2</sub>.

coated by coupling agent had relatively flat, smooth, and small spherical particles, and white areas were more dispersed in Figure 3(b). Agglomeration tendency was weakened; the interaction between the particles was reduced and uniformly dispersed. It also indicated that particle size decreased, and the surface area increased [6–8]. Figures 3(c) and 3(d) showed that the relatively flat area was EP matrix, which was continuous phase, and PU molecule had uniformly formed “island structure” in EP matrix. Inorganic particles were dispersing phase and were evenly dispersed in EP-PU matrix, and the interaction of inorganic components and PU matrix was very strong and they were distributed in PU matrix “island structure” in Figures 3(c) and 3(d), and the inorganic particles in “island structure” were of nanoscale. Nanoparticles had a larger surface area [9], and the surface of modified nanoparticles had active groups and compatibility with organic phase, to promote the mutual penetration of the two-phase interface; the interpenetration could help to improve the mechanical properties.

**3.3. Analysis of TEM Patterns.** The morphology of TCA201-TiO<sub>2</sub> was investigated by TEM in order to observe the effect of modification and verified the result of FT-IR in Figure 4.

Figure 4 shows the TEM images of TCA201-TiO<sub>2</sub>; Figures 4(a) and 4(b) were magnified to  $4 \times 10^4$  times and  $8 \times 10^4$  times, respectively. In the picture, it would be seen that TCA201-TiO<sub>2</sub> dispersed evenly, and the particle size was in the nanosized and modified nano-TiO<sub>2</sub> uniformly connected at both ends of the coupling agent, which demonstrated that the interaction of the active group in TiO<sub>2</sub> molecular and the active group in TCA201 was existing. The interaction could be intermolecular cross-linking, inhibit reuniting of TiO<sub>2</sub>, and improve the interaction of two phases, titanate made the epoxy resin and nano-TiO<sub>2</sub> surface connection strengthen, and organic functional groups of titanate and the epoxy resin should produce chemical bonding, so the nano-TiO<sub>2</sub> well dispersed in an epoxy resin and TCA201-TiO<sub>2</sub>

could greatly improve and enhance their overall performance. Transmission electron microscopy showed how nano-TiO<sub>2</sub> and TCA201 connected.

**3.4. Mechanical Properties.** The diagram of the shear strength of TCA201-TiO<sub>2</sub>/EP-PU composite was shown in Figure 5.

With the increase of TCA201-TiO<sub>2</sub> filler, the shear strength of the composites gradually increased and then declined. The shear strength of 3 wt% TCA201-TiO<sub>2</sub>/EP-PU composite was the maximum value for 27.14 MPa. The content of TCA201-TiO<sub>2</sub> was more than 3 wt% and the shear strength of composite materials decreased but still was higher than that of pure polymer matrix EP-PU.

Generally, TCA201-TiO<sub>2</sub> modified by coupling agent was not simply physically blending with polymer matrix. TCA201-TiO<sub>2</sub> and polymer matrix could form chemical bonds through coupling agent; as the surface of TCA201-TiO<sub>2</sub> has existed in much active groups, these groups could improve fracture energy and adhesive strength of the composites [10]. The structure of coupling agent had two kinds of functional group; one was active group and the other was nonactive group; this structure would play the role of a bridge which associated with the nano-TiO<sub>2</sub> and the epoxy matrix. The function enhanced the interfacial adhesion and consumed more energy; thereby mechanical properties of composites were improved [11].

Nanoparticles had large specific surface area and high superficial energy; it could help to improve interactions with epoxy matrix. When the content of nanoparticles was more excessive, the interactions of inorganic particles could be enhanced, but the interactions of polymer matrix and inorganic component would weaken, and inorganic TiO<sub>2</sub> would be reunion and the dispersion of two phases would be much difficult. The phenomenon of agglomeration occurred, which resulted in stress concentration point, and the mechanical properties of composites could decrease, but the shear strength of composites was still higher than that of pure epoxy resin matrix [12, 13].

**3.5. Heat Resistance Properties.** Thermal degradation behaviors of composites were evaluated by thermogravimetric analysis (TGA). TGA curves of composite materials were shown in Figure 6. The results demonstrated that  $T_d$  of TCA201-TiO<sub>2</sub>/EP-PU was firstly increased and then decreased with the increase of TCA201-TiO<sub>2</sub> doping. When the amount of TCA201-TiO<sub>2</sub> was 3 wt%,  $T_d$  that reached up to 397.82°C was higher 17.48°C than that of pure materials. The main reasons were that firstly heat resistance of inorganic TCA201-TiO<sub>2</sub> was stronger and to increase its content in the polymer matrix was bound to enhancing the heat resistance of the composite. Secondly, there were active groups in the TCA201-TiO<sub>2</sub> particles structure, and there were a large number of hydroxyl groups in the organic phase; the two kinds of active groups could have contact with cross-linking agent and exist in strong interactions. This interaction enhanced fracture energy of composites and became beneficial to the heat resistant properties of composite [14, 15]. Thirdly, inorganic TCA201-TiO<sub>2</sub> particles possessed a large specific surface area, contacting area of organic matrix was larger,

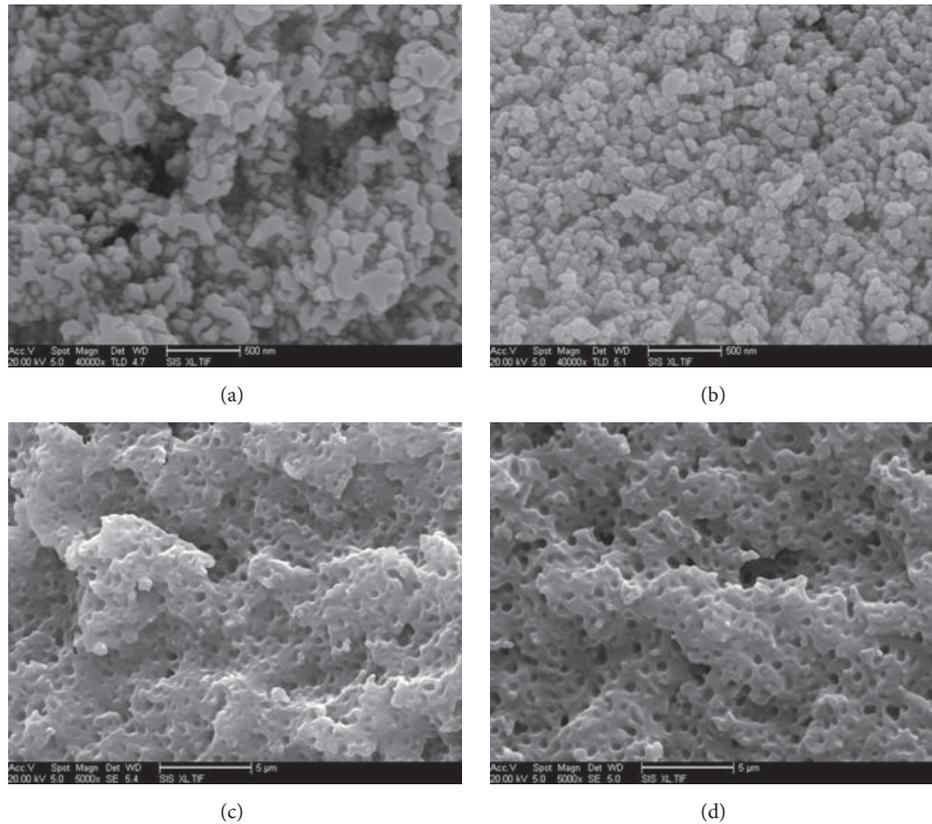


FIGURE 3: SEM photos of  $\text{TiO}_2$  and TCA201- $\text{TiO}_2$ /EP-PU composite.

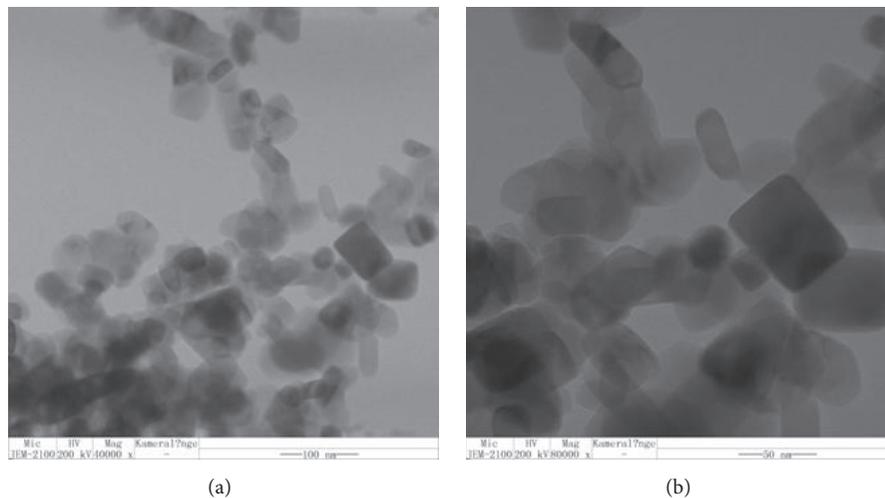


FIGURE 4: TEM patterns of TCA201- $\text{TiO}_2$ .

and there were nanoeffects. So the thermal stability of composites is enhanced. Fourthly, when the content of inorganic component was excessive, the interaction forces of inorganic particles would be enhanced and dispersion of two phases weakened and easily led to agglomeration in the epoxy resin.

Figure 7 shows SEM patterns for the breakdown point of 7 wt% TCA201- $\text{TiO}_2$ /EP-PU. Figures 7(a), 7(b), and 7(c) show back breakdown point of composite and Figure 7(d) shows face breakdown point of composite.

It was seen that a large number of inorganic components were separated (in Figures 7(a), 7(b), and 7(c)). This was because electrical energy would transform into heat energy in the breakdown processing; the heat energy caused epoxy resin matrix to decompose and inorganic component of composite was separated [16, 17]. And the heat energy of back breakdown could not spread rapidly; therefore, it would produce thermal loss. The produced heat energy during face breakdown of composite radiated its heat away into space and

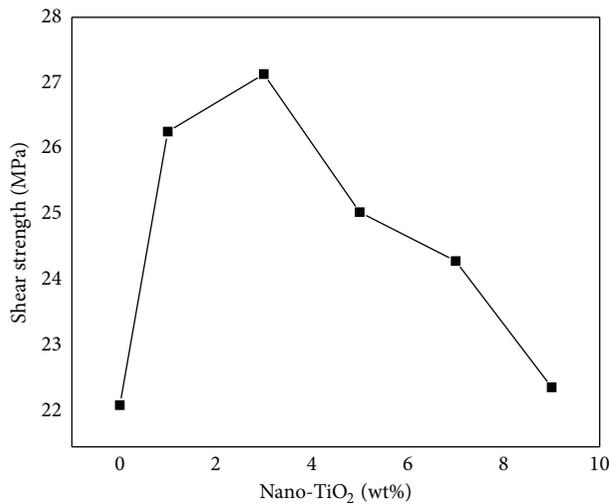


FIGURE 5: Shear strength curve of TCA201-TiO<sub>2</sub>/EP-PU.

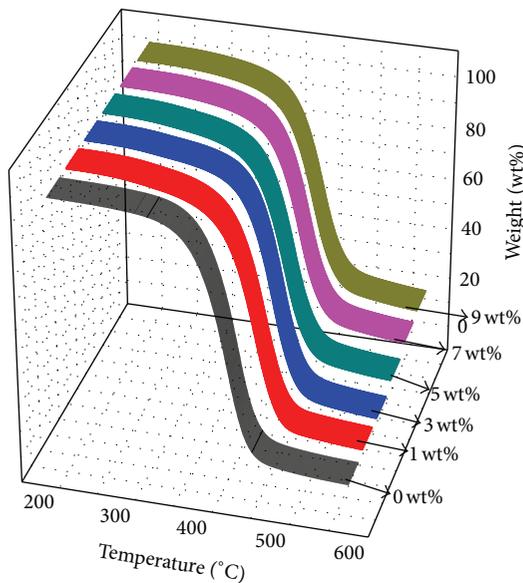


FIGURE 6: TG curve of TCA201-TiO<sub>2</sub>/EP-PU.

could not make two-phase inorganic and organic component separate in Figure 7(d).

**3.6. Dielectric Properties.** Figure 8 shows the dielectric constant and dielectric loss curve of TCA201-TiO<sub>2</sub>/EP-PU composites. It was noted that dielectric constant of TCA201-TiO<sub>2</sub>/EP-PU increased with the increasing of amount of TCA201-TiO<sub>2</sub>. As polar functional groups increased, it would result in the degree of polarization of the composite material to enhance and led to the increase of the dielectric constant eventually. At the same time, because nano-TiO<sub>2</sub> particles in epoxy resin had a good surface effect, other polarization also could occur in the two-phase interface of composite and led to increasing the degree of polarization of the composite [18].

Dielectric loss ( $\tan \delta$ ) curve in Figure 8 displayed that  $\tan \delta$  increased, too, with the increase of TCA201-TiO<sub>2</sub> filler. There could be two reasons; one was electrical conductivity loss of polar functional groups and the other was the relaxation loss of the polar functional groups. From the above analysis, conclusions were obtained that the polar functional groups of the composite materials were driven by electric field strength to generate strong polarization. And the content of TCA201-TiO<sub>2</sub> in composite increased and the number of the conductive carriers increased, too. Under the influence of an external electric field, it made the carrier bring about the directional movement phenomenon of the heat loss of dielectric material, ultimately.

**3.7. Breakdown Field Strength.** Breakdown strength of insulating material generally generates thermal breakdown and electrical breakdown at the breakdown point, and simultaneously electric energy was changed to thermal energy, so breakdown phenomenon would happen and the composite is destroyed. Though the modified nanoparticles and the organic polymer could be compatible well, and there were certain defects between the inorganic phase and the organic phase interface. It was seen in Figure 9 that the breakdown field strength of TCA201-TiO<sub>2</sub>/EP-PU composite was reduced with the content of TCA201-TiO<sub>2</sub>. Because insulating materials might release heat and convert part of the electrical energy into heat under an external electric field, the dielectric loss happened and the energy was lost. Therefore, the increase of dielectric loss gave the media issue more heat and enhanced temperature, which would break the balance of heating and cooling. In addition, TiO<sub>2</sub> had the semiconductor, so the conductivity of composites improved rapidly with the increase of temperature. This resulted in the decline of material breakdown strength [19].

It would aggravate the second reunion of nanoparticles in the composite curing process, which caused deformities of the electric field and made the temperature at test point of the local area slightly higher than the others. And this part of heat could not be well distributed out and make the thermal breakdown occur in this region. Secondly, there were some compatibility issues between the two-phase interfaces when inorganic particles and the epoxy matrix were mixed, at which some tiny bubbles and trap appeared in composite materials, and the defects also increased with the increase of TiO<sub>2</sub>. These factors brought about the decline of breakdown field strength of composite material. Breakdown field strength of 3 wt%-TCA201-TiO<sub>2</sub>/EP-PU composite was 14 kV/mm; it met the requirement of insulating material.

## 4. Conclusions

The results of FT-IR, TEM, and SEM demonstrated that TCA201 coupling agent had connected with TiO<sub>2</sub> particle through chemical cross-linking, and TCA201-TiO<sub>2</sub> could disperse well in EP-PU matrix. This would improve the properties of composite. When the amount of TCA201-TiO<sub>2</sub> was 3 wt%, shear strength gained maximum value for 27.14 MPa, the thermal decomposition temperature reached

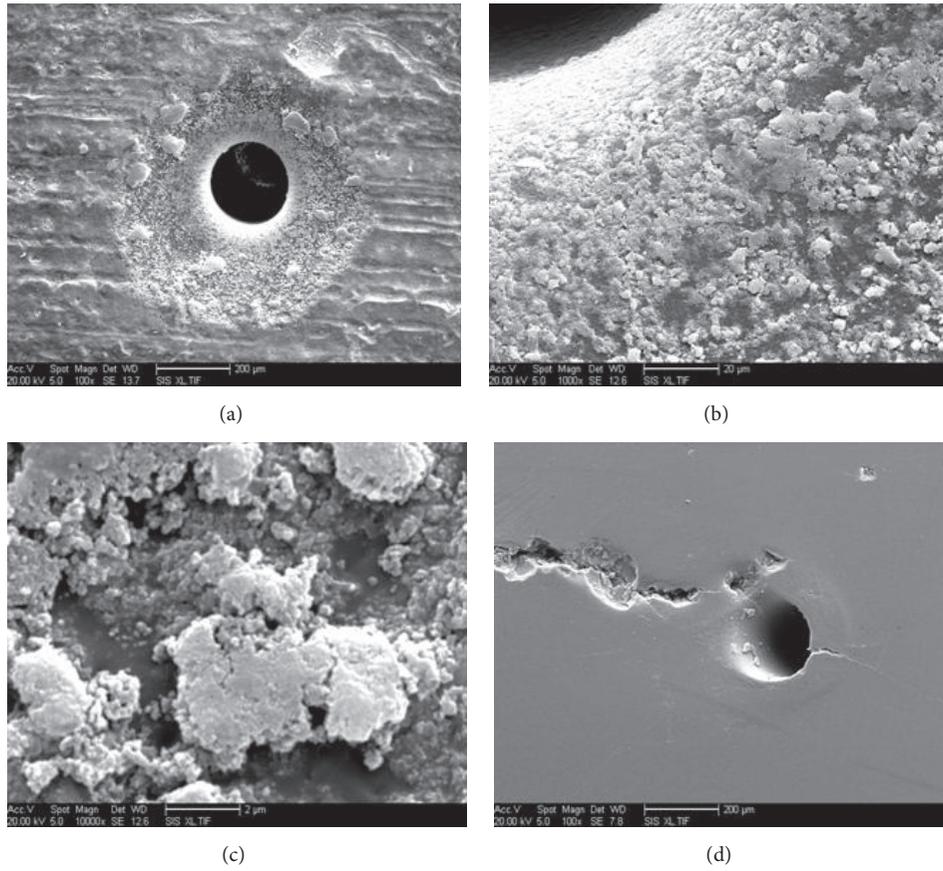


FIGURE 7: SEM of TCA201-TiO<sub>2</sub>/EP-PU breakdown point.

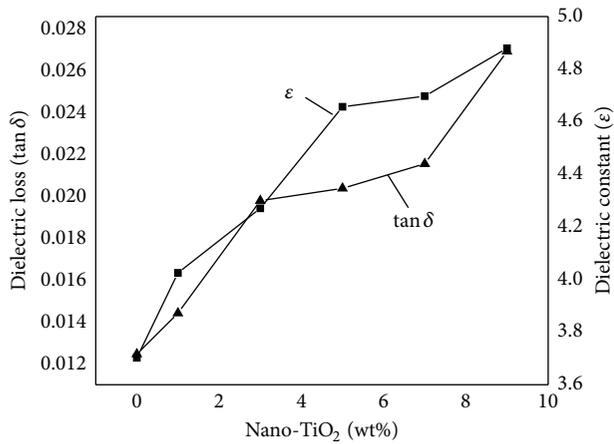


FIGURE 8: Dielectric constant and dielectric loss curves of TCA201-TiO<sub>2</sub>/EP-PU.

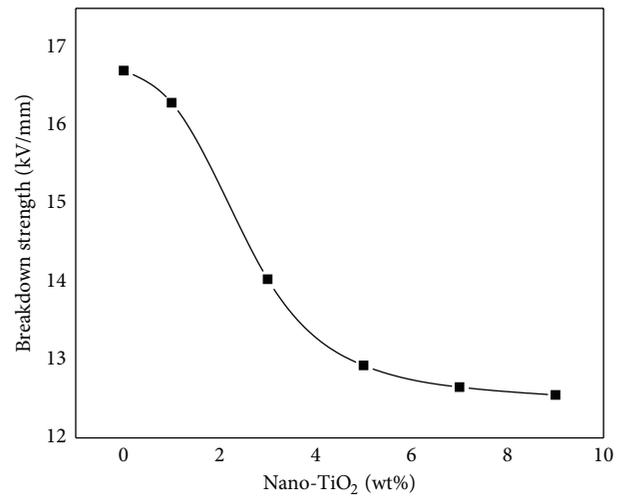


FIGURE 9: Breakdown strength curve of TCA201-TiO<sub>2</sub>/EP-PU.

up to 397.82°C, which was higher 17.48°C than pure polymer matrix, breakdown field strength of composite was 14 kV/mm, and the performances could meet the requirement of insulating material.

### Conflict of Interests

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

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