

## Preparation and Various Characteristics of Epoxy / Alumina Nanocomposites

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**Keywords** : nanocomposite, polymer nanocomposite, epoxy resin, alumina, nano-dielectric, electrical insulation

Polymer nanocomposites are defined as polymers in which small amounts of nano-scale fillers, or nanofillers, are homogeneously dispersed by only several weight (wt) percentages. In the dielectrics and electrical insulation field, several reviews and original papers have been reported actively on the dielectric properties of polymer nanocomposites in recent years.

Epoxy/ alumina nanocomposites were newly prepared by dispersing 3, 5, 7, and 10 wt% boehmite alumina nanofillers in a bisphenol-A epoxy resin using a special two-stage direct mixing method. Commercial epoxy resin used for experiments was Epikote 816B, which was a bisphenol-A epoxy resin, manufactured by Japan Epoxy Resins Co. Hardner was Epicure 113, which was a modified alicyclic amine manufactured by the same company. Boehmite alumina (AlO(OH)) particles used as nanofillers was DISPERAL OS-1 manufactured by Sasol Co. The nanofillers were of plate-like shape, and their crystallite size was about 10 nm in (120) plane. They were surface-treated by a para-toluenesulfonic acid as a solubilizer. A special two-stage direct mixing method consists of the two stages. Resin is mixed with nano-fillers by a blade rotation type mixer in the first stage, and the resultant mixture is passed through in a very fine channel, or an orifice, by high pressure using a high-pressure homogenizer in the second stage. It is postulated that the large shear force caused by high pressure would be much effective to reduce agglomeration of nano-particles. The blend was poured into aluminum cast moldings, and sufficiently defoamed, and then cured under suitable curing conditions. Specimens used were slab in shape for evaluation of several properties, and molding type of needle-plane electrode geometry for evaluation of electrical breakdown time. Epoxy specimens without nanofillers were also prepared in the same way for comparison. It was confirmed that the powder particles turned into about 50 nm through mixing processes, and those nanofillers were well dispersed in all the specimens as shown in Fig.1. Dielectric, mechanical, and thermal properties were investigated. Conclusion is drawn as follows as for nanostructure effects.

(1) PD resistance increases as the filler content increase as shown in Fig.2; e.g. 7 wt% nanofiller content creates a 60 % decrease in depth of PD-caused erosion.

(2) Weibull analysis shows that short-time electrical treeing breakdown time is prolonged to 265 % by 5 wt% addition of

nanofillers. But there was more data scatter in nanocomposites than in pure epoxy.

(3) Permittivity tends to increase from 3.7 to 4.0 by 5 wt% addition of nanofillers as opposed to what was newly found in the recent past.

(4) Glass transition temperature remains unchanged as 109 °C.

(5) Mechanical properties such as flexural strength and flexural modulus increase; e.g. flexural strength and flexural modulus are improved by 5 % and 8 % with 5 wt% content, respectively. Excess addition causes a reverse effect.

(6) Interfacial bonding seems to be rather weak in the nanocomposite specimens prepared this time.

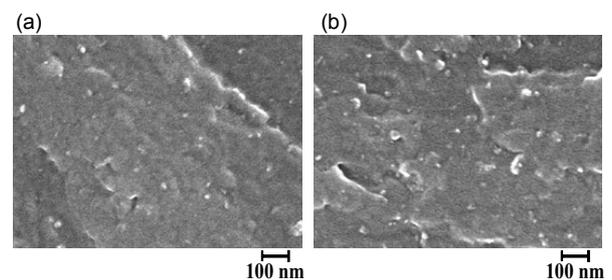


Fig. 1. SEM images of cracked surface of epoxy boehmite alumina nanocomposites with 5 wt% addition (a) and with 10 wt% addition (b)

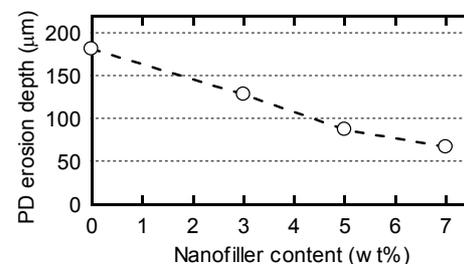


Fig. 2. Depth of PD-caused erosion on the specimens as a function of boehmite alumina nanofiller content

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Epoxy/ alumina nanocomposites were newly prepared by dispersing 3, 5, 7, and 10 weight (wt) % boehmite alumina nanofillers in a bisphenol-A epoxy resin using a special two-stage direct mixing method. It was confirmed by scanning electron microscopy imaging that the nanofillers were homogeneously dispersed in the epoxy matrix. Dielectric, mechanical, and thermal properties were investigated. It was elucidated that nanofillers affects various characteristics of epoxy resins, when they are nanostructured. Such nano-effects we obtained are summarized as follows. Partial discharge resistance increases as the filler content increases; e.g. 7 wt% nanofiller content creates a 60 % decrease in depth of PD-caused erosion. Weibull analysis shows that short-time electrical treeing breakdown time is prolonged to 265 % by 5 wt% addition of nanofillers. But there was more data scatter in nanocomposites than in pure epoxy. Permittivity tends to increase from 3.7 to 4.0 by 5 wt% nanofiller addition as opposed to what was newly found in the recent past. Glass transition temperature remains unchanged as 109 °C. Mechanical properties such as flexural strength and flexural modulus increase; e.g. flexural strength and flexural modulus are improved by 5 % and 8 % with 5 wt% content, respectively. Excess addition causes a reverse effect. It is concluded from permittivity and glass transition temperature characteristics that interfacial bonding seems to be more or less weak in the nanocomposite specimens prepared this time, even though mechanical strengths increase. There is a possibility that the nanocomposites specimens will be improved in interfacial quality.

**Keywords** : nanocomposite, polymer nanocomposite, epoxy resin, alumina, nano-dielectric, electrical insulation

## 1. Introduction

Polymer nanocomposites are defined as polymers in which small amounts of nano-scale fillers, or nanofillers, are homogeneously dispersed by only several weight (wt) percentages. In the dielectrics and electrical insulation field, several reviews and original papers have been reported actively on the dielectric properties of polymer nanocomposites in recent years<sup>(1)-(5)</sup>. There are several preparation methods for polymer nanocomposites such as intercalation, sol-gel, molecular composite, and direct dispersion that focus the separation of cohesive nanofillers from each other<sup>(2)</sup>. In the direct dispersion method, nanosized particles are chemically modified on their surfaces to increase compatibility with polymers, and are mixed with a polymer by adding shear force and dispersed homogeneously without agglomeration.

It was clarified by the authors that nanocomposite dielectrics are more resistant to partial discharges (PDs) than pure resin and conventional micro-particle-filled resin<sup>(6)-(12)</sup>. Epoxy/boehmite alumina nanocomposite was processed in laboratory scale. Boehmite alumina nanofillers addition to an epoxy resin was

expected to reinforce not only mechanical property and flame retardancy but also electrical insulation property of base resin. Nano-scale boehmite alumina particles were dispersed by 3, 5, 7, and 10 wt% in bisphenol-A epoxy resin by the special two-stage method that some of the authors had developed. Boehmite alumina particles were mixed with epoxy resin to be subjected to the shear force between them so as to be separated each other. It was confirmed that the nano-particles were homogeneously dispersed in the epoxy matrix by eye observation, the optical microscope and the electron scanning microscope. Dielectric, mechanical, and thermal properties were measured of pure epoxy and nanofiller-added epoxy for comparison.

## 2. Specimens

**2.1 Materials** Commercial epoxy resin used for experiments was Epikote 816B manufactured by Japan Epoxy Resins Co. It was a bisphenol-A epoxy resin that was made less viscous by adding a reactive diluting agent. Hardner was Epicure 113 manufactured by the same company. It was a modified alicyclic amine. Boehmite alumina (AlO(OH)) particles used as nanofillers was DISPERAL OS-1 manufactured by Sasol Co. The nanofillers were of plate-like shape, and their crystallite size was about 10 nm in (120) plane. They were surface-treated by a para-toluenesulfonic acid as a solubilizer.

**2.2 Preparation Procedure** Epoxy/alumina nanocomposites were prepared in laboratory scale by a new two-stage direct mixing method, as shown in Fig.1. It consists of

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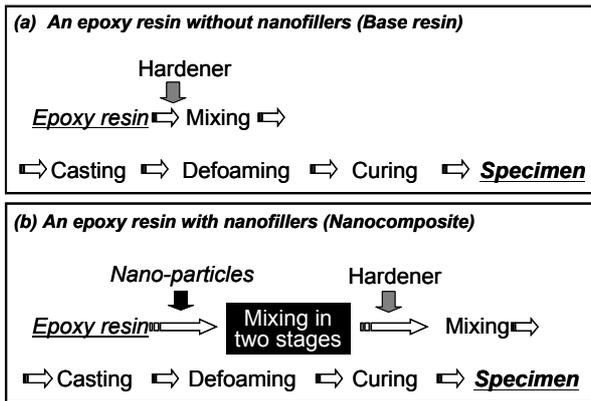


Fig. 1. Preparation scheme of epoxy specimens without and with nanofillers

Table 1. Curing conditions of the epoxy specimen synthesis

	Slab	Molding type
step 1	60°C for 2 h	70°C for 3 h
step 2	90°C for 2 h	120°C for 3 h
step 3	120°C for 12 h	non

the two stages. Resin is mixed with nano-fillers by a blade rotation type mixer in the first stage, and the resultant mixture is passed through in an orifice, by high pressure using a high-pressure homogenizer in the second stage. It is postulated that the large shear force caused by high pressure would be much effective to reduce agglomeration of nano-particles. The mixer in the first stage used for experiments was T.K. Homo Mixer Mark II *f*-model which was manufactured by Primix Co. The number of rotations was from 600 rpm to 12,000 rpm. The high-pressure homogenizer in the second stage used for experiments was Nanomizer for laboratory scale which was manufactured by Yoshida Kikai Co. Inner diameter of the orifice was 150  $\mu\text{m}$ . The available maximum pressure was 150 MPa. Actually, the blend was mixed at 7,000 rpm for 2 hours in the first stage, and then the pressurized process at 140 MPa was repeated 5 times in the second stage.

The blend was poured into aluminum cast moldings, and sufficiently defoamed, and then cured under such curing conditions as shown in Table 1. Specimens used were slab in shape for evaluation of several properties, and molding type of needle-plane electrode geometry for evaluation of electrical breakdown time. Epoxy specimens without nanofillers were also prepared in the same way for comparison.

### 3. Measuring Methods

**3.1 Glass Transition Temperature** The glass transition temperature ( $T_g$ ) of the specimens was measured by dynamic mechanical spectroscopy (Seiko Instrument Inc., DMS-200). The measurements in three-point bend mode were taken at a driving frequency of 1 Hz and a heating rate of 2  $^{\circ}\text{C}/\text{min}$  in air. The specimens of rectangular slab shape were approximately 1 mm thick, 5 mm wide, and 30 mm long.

**3.2 Mechanical Properties** Mechanical properties such as flexural strength and flexural modulus were measured by a universal bending testing machine (ORIENTEC Co., TENSILON RTC-1350A) according to JIS-K7171 using a crosshead speed of 2 mm/min. The specimens of rectangular slab shape were approximately 1 mm thick, 10 mm wide, and 50 mm long. Five

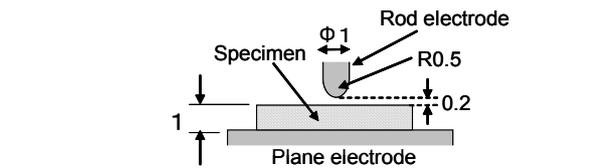


Fig. 2. Rod-gap-plane electrode geometry for partial discharges degradation test

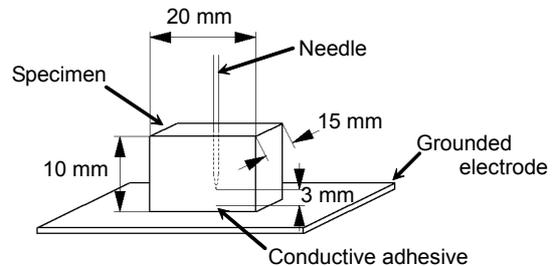


Fig. 3. Needle-plane electrode geometry for electrical treeing breakdown test

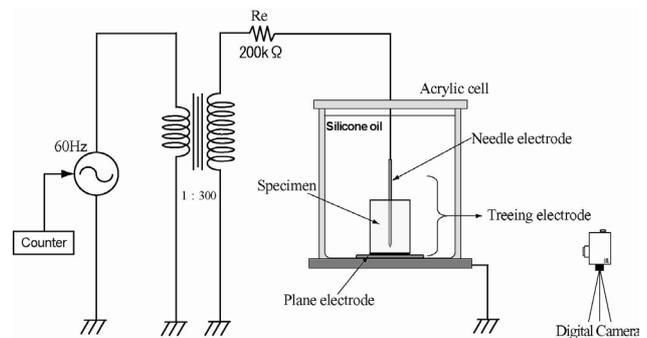


Fig. 4. Experimental setup for electrical treeing breakdown test

specimens were used for one condition.

**3.3 Dielectric Permittivity** Relative permittivity was measured by an LCR meter (Agilent Technologies Co., 4262A) at measurement frequency of 60 Hz. The specimens of square slab shape were approximately 1 mm thick and 50 mm wide.

**3.4 PD Resistance** PD resistance property was evaluated by a rod-gap-plane electrode system as shown in Fig.2. A flat specimen was inserted with 0.2 mm air gap held between a tungsten rod and a plane electrode. The rod was 1 mm in diameter with curvature of 0.5 mm radius on its tip. The specimens used were approximately 1 mm thick and 50 mm wide. They were subjected to 720 Hz ac voltage (4  $\text{kV}_{\text{rms}}$ ) for 120 h so that PDs were generated at the tip of the rod electrode. In order to confirm that PD quantity in each specimen was almost equal, PD pulses were measured. PDs created erosion on the surface of the specimens, which was measured by a mechanical surface profilometer.

**3.5 Electrical Treeing Breakdown** Electrical treeing breakdown time was measured by a needle-plane electrode system as shown in Fig.3. An iron needle electrode was 1 mm in diameter and 60 mm in length, with a tip angle of 15 $^{\circ}$  and curvature of 5  $\mu\text{m}$  radius on the needle tip. The needle was set with a 3 mm separation between the needle tip and the bottom of the specimen. The specimens were fixed on the grounded electrode by a conductive adhesive, and then set in silicone oil-filled vessels. A

constant ac voltage (30 kV<sub>rms</sub>) was applied to the needle electrode at the rate of ac 1.5 kV/s, and then time to electrical breakdown was measured by a setup as shown in Fig.4. At the same time, a digital camera with a telephoto lens, which was set in a distance of about 1 m from the specimen, was used to take photos of electrical trees every 1 minute after 30 kV<sub>rms</sub> was applied. Ten specimens were used for one condition.

**4. Experimental Results**

**4.1 Confirmation of Nanofiller Dispersion** Figures 5 (a) and (b) show two SEM (scanning electron microscopy) images of boehmite alumina powder particles used. They were originally

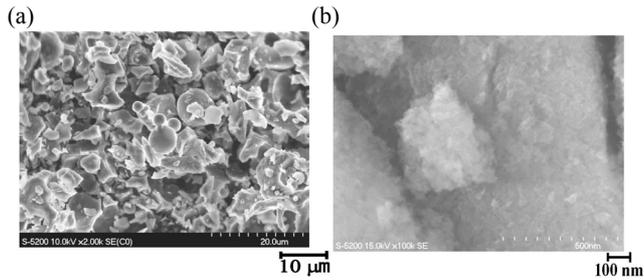


Fig. 5. SEM images of boehmite alumina powder particles

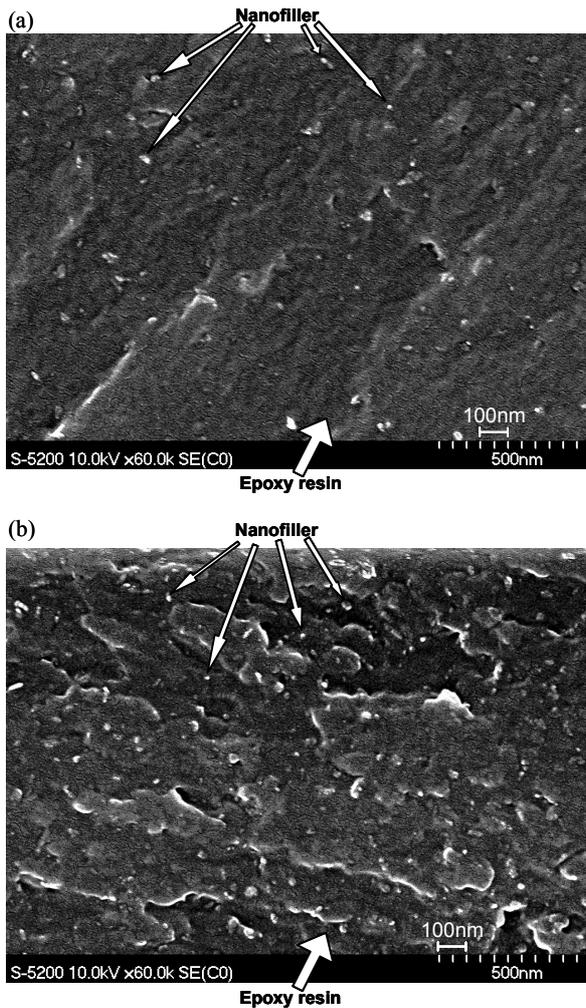


Fig. 6. SEM images of cracked surface of the nanocomposite specimens with 5 wt% addition (a) and with 10 wt% addition (b)

agglomerated into lumps of several μm in size before mixing. It seems difficult to clearly confirm the size and shape of the nano-particles as shown in Fig. 5 (b). Figures 6 (a) and (b) show two SEM images of cracked surface of nanocomposites with 5 wt% and 10 wt% additions, respectively. It was confirmed that the powder particles turned into about 50 nm through mixing processes, and those nanofillers were well dispersed in all the specimens. Their number density is much larger in nanocomposite with 10 wt% addition than in nanocomposite with 5 wt% addition. Distance between the neighboring particles, i.e. inter-filler distance seemed to be in the range of several tens or several hundreds nm in two dimensional SEM observation. In addition, the nanocomposite specimens prepared in laboratory scale kept high transparency similar to its base resin according to eye sight.

**4.2 Glass Transition Temperature** It was found that glass transition temperature is higher in epoxy/layered silicate nanocomposites than base epoxy<sup>(5)</sup>. For epoxy/boehmite alumina nanocomposite specimens prepared this time, data for glass transition temperature were obtained as shown in Table 2. This table indicates that glass transition temperature is kept almost unchanged after dispersing several wt% of the nanofillers and even decreased a little bit in the case of 7 wt% content. It might be related to the degree of binding of nanofillers with surrounding polymer matrix in interaction zone whether it increases or decreases.

**4.3 Mechanical Properties** Figure 7 shows mechanical properties such as flexural strength and flexural modulus as their dependence on the content of boehmite alumina nanofillers. It is clearly observed in the figure that 3 to 5 wt% additions of the nanofillers would improve flexural strength and flexural modulus.

Table 2. Relation between boehmite alumina nanofiller content and glass transition temperature (T<sub>g</sub>) of the specimens

Nanofiller content (wt%)	0	3	5	7
T <sub>g</sub> (°C)	109	106	109	105

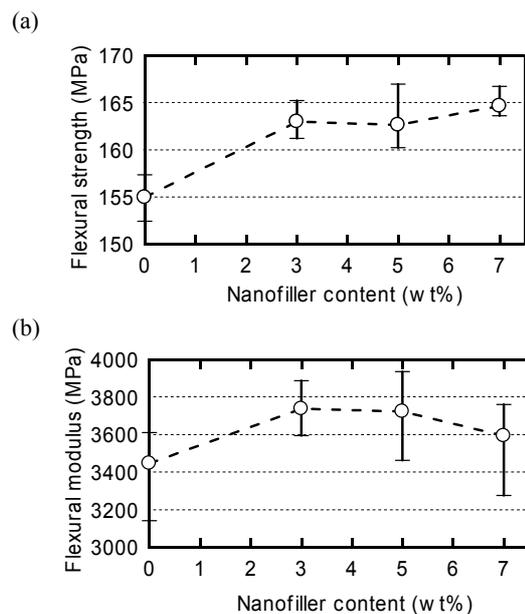


Fig. 7. Flexural strength (a) and flexural modulus (b) of the specimens as a function of boehmite alumina nanofiller content

In the case of 7 wt% content, flexural strength tends to increase, while flexural modulus tends to decrease. The latter worse effect must be related to the observed decrease in glass transition temperature in the case of 7 wt% nanofiller addition. This observation is considered to be affected by the following phenomena;

- (i) Loose coupling between nanofillers and matrices.
- (ii) Stress concentration around possibly agglomerated nanofillers.

**4.4 Dielectric Permittivity** Permittivity was generally considered to decrease by nanostructuration in low frequency range at least around 50/60 Hz, as far as nanocomposites were properly formed<sup>(5)(11)-(14)</sup>. Frequency dependence observed thus far exhibits more or less complicated performances. In the case of epoxy/ boehmite alumina nanocomposite specimens prepared this time, permittivity tends to increase, as shown in Table 3. This apparent contradiction must come from how nanofillers are dispersed and how the interfaces between nanofillers and polymer matrices are formed or whether coupling agents are used or not. Furthermore, attention is drawn to original absolute values of permittivity that base resins possess. Loose inorganic and organic couplings might be involved in this mechanism, too.

**4.5 PD Resistance** It was clarified that PD resistance is much enhanced by nanostructuration in the case of polyamide/layered silicate nanocomposite<sup>(6)-(12)</sup>. The same is almost true of other nanocomposites generally. Figure 8 shows surface profiles of the specimens without nanofillers, with 3, 5, and 7 wt% nanofillers after the PD degradation test is made.

Table 3. Relation between boehmite alumina nanofiller content and relative permittivity of the specimens

Nanofiller content (wt%)	0	3	5	7
$\epsilon_r$ [60Hz]	3.7	3.9	4.0	4.0

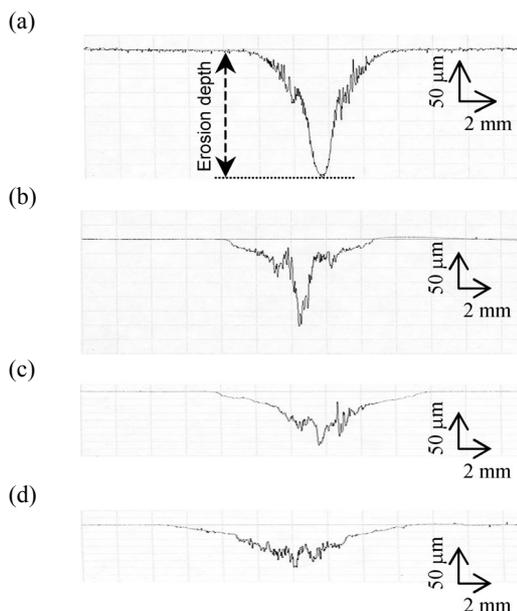


Fig. 8. Surface profiles of the specimens after partial discharge degradation test; (a) pure epoxy, (b) nanocomposite with 3 wt% addition, (c) nanocomposite with 5 wt% addition, and (d) nanocomposite with 7 wt% addition

Erosion depth was defined as height between the non-degraded part and the most deeply eroded part as shown in Fig.8 (a). The erosion depth was selected as index of PD resistance in this paper. Figure 9 shows nanofiller content dependence of erosion depth due to PDs. Erosion depth decreases with increasing nanofiller content; e.g. it is 2 times shallower for the specimen with 5 wt% addition of nanofillers than for the specimen without nanofillers. This result shows that the nanocomposite specimens are more resistant to PDs than the specimen without nanofillers. It could be related to the effects of nanofillers on PD resistance as mentioned previously<sup>(7)</sup>, i.e.

- (i) Bonding strength between fillers and matrices.
- (ii) Inter-filler space or matrix volume surrounded by neighboring fillers.
- (iii) Morphology in the inter-filler space.
- (iv) Mesoscopic interaction.

**4.6 Electrical Treeing Breakdown** It is generally understood that nanostructuration would enhance lifetime for electrical treeing type (long-time) failure in many kinds of polymers. It was also confirmed that short-time treeing breakdown time is elongated by nanostructuration. Figure 10 shows a Weibull plot for time to electrical treeing breakdown of base epoxy and epoxy/boehmite alumina nanocomposites with 5 wt% and 10 wt% additions. Table 4 shows scale and shape parameter values ( $\alpha$  and  $\beta$ ) obtained by the Weibull analysis as shown in Fig. 9. Scale parameters in Table 4 indicate that electrical breakdown time at 63.2 % probability increases from 437 sec for base epoxy to 1,160 sec and 980 sec for nanocomposites with 5 wt% and 10 wt% additions, respectively. From the shape parameter analysis, data for 5 wt% added nanocomposite shows most scattered data among

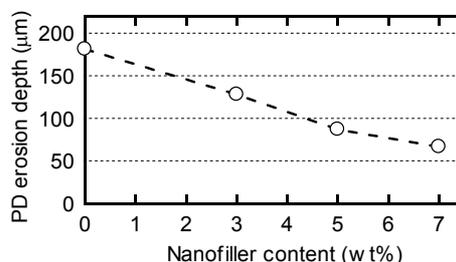


Fig. 9. Depth of PD-caused erosion on the specimens as a function of boehmite alumina nanofiller content

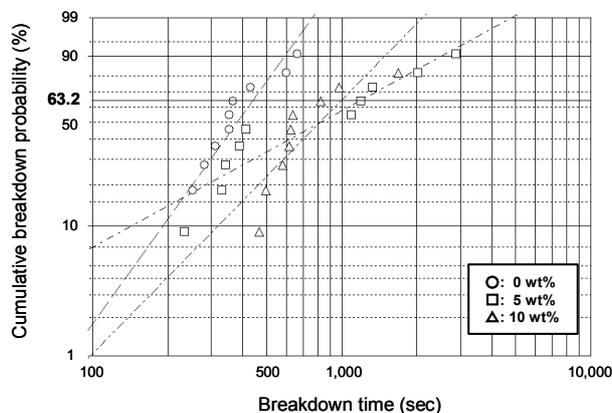
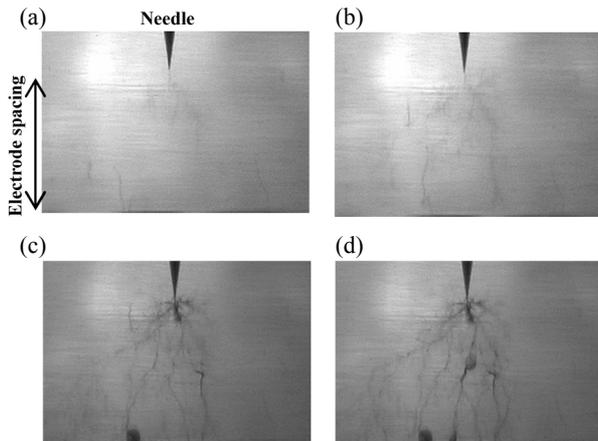


Fig. 10. Weibull plot for time to electrical treeing breakdown of pure epoxy and nanocomposites with boehmite alumina nanofillers

Table 4. Scale and shape parameter values obtained by the Weibull analysis of Fig. 10

Specimen	Scale parameter $\alpha$ [sec]	Shape parameter $\beta$ [-]
base epoxy resin	437	2.73
nanocomposite with 5 wt% nanofillers	1,160	1.07
nanocomposite with 10 wt% nanofillers	980	2.00

Fig. 11. Electrical treeing in pure epoxy at 30 kV<sub>rms</sub> 1 min (a), 5 min (b) and 10 min (c) after applied voltage, and 1 min before breakdown, or 11 min (d)

three kinds of specimens. There are many factors to affect data scattering, and some of them are;

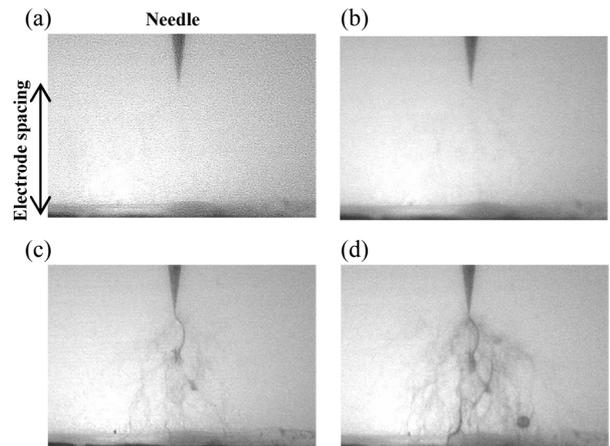
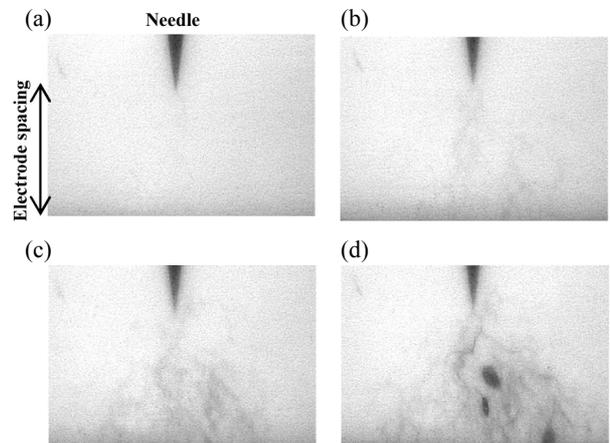
- (i) Insufficient homogeneity of nanofiller dispersion in epoxy matrix.
- (ii) Defects unexpectedly introduced during nanocomposite preparation process.

It was also elucidated that the addition of boehmite alumina nanofillers improves electrical treeing breakdown time of epoxy resin. Electric field around a tip of the needle can be estimated from the following formula

$$E = \frac{2V}{r \ln(1 + 4d/r)} \dots\dots\dots (1)$$

where  $r$ ,  $d$  and  $V$  are the radius of a needle tip, an electrode spacing, and applied voltage, respectively. When they are 5  $\mu\text{m}$ , 3 mm, and 30 kV<sub>rms</sub>, the estimated electric field strength (peak value) is 2,180 kV/mm. As the electric field is rather high, the observed phenomenon indicates more or less short-time breakdown.

Figures 11, 12 and 13 show several photos of treeing processes for pure epoxy and epoxy nanocomposite specimens with 5 wt% and 10 wt% nanofiller content. Photos (a) (b), (c) and (d) in the three figures show the shapes of trees generated in 1, 5, and 10 min after 30 kV<sub>rms</sub> voltage application, and in 1 min before breakdown, respectively. Thin trees seem to be generated from a tip of the needle in all the specimens within the first 1 min after voltage application. The thin trees are grown up to be branched and expanded like dendrites and reach the opposite electrode within about 5 min in all the specimens, but does not cause any breakdown. After this process, the tree channels become thick and

Fig. 12. Electrical treeing in nanocomposite with 5 wt% addition at 30 kV<sub>rms</sub> 1 min (a), 5 min (b) and 10 min (c) after applied voltage, and 1 min before breakdown, or 20 min (d)Fig. 13. Electrical treeing in nanocomposite with 10 wt% addition at 30 kV<sub>rms</sub> 1 min (a), 5 min (b) and 10 min (c) after applied voltage, and 1 min before breakdown, or 16 min (d)

thick progressively, and then breakdown takes place some time after. What we observed on treeing is a short time breakdown, and is certainly different from a long-time treeing breakdown process in which tree channels grow step by step. Trees do not seem to be different in shape irrespective of pure and nano epoxy specimens within our experimental condition and observation method. Some explanation is given in a companion paper<sup>(15)</sup>, but further investigation is needed to clarify how prolonged tree breakdown time due to nanostructuring.

## 5. Discussion

It is confirmed that boehmite alumina nanofillers are homogeneously dispersed in the epoxy matrix by using a special two-stage direct mixing method developed this time. However, organic-inorganic bonding force seems to be relatively weak in epoxy/boehmite alumina nanocomposite specimens prepared this time. It can be postulated from performances of their glass transition temperature and mechanical properties. Permittivity and glass transition data are apparently contradictory to what have been obtained recently. This indicates that fine structures for interaction zones are different in different nanocomposites and even different preparation procedures. On electrical insulation

properties, the addition of boehmite alumina nanofillers improves both PD resistance and electrical treeing breakdown time of epoxy resin. It is expected that electrical treeing breakdown time can be more prolonged if the surface of nanofillers can be performed perfectly physical-chemical treatment. That would be strongly depend on compatibility between a nanofiller and a polymer used for experiments. These fine structures can be analyzed by using a multi-core model (16, 17) proposed as fine structures of interaction zones. Especially the first layer (binding layer) and the second layer (bound layer) of this model are deeply concerned with characteristics of specific interfaces formed.

## 6. Conclusion

Epoxy/boehmite alumina nanocomposites were prepared in laboratory scale by a two-stage direct mixing method, and investigated on various characteristics such as glass transition temperature, mechanical properties, dielectric permittivity, partial discharge resistance, and electrical treeing breakdown. Specimens were a bisphenol-A epoxy resin with 0, 3, 5, 7, and 10 weight % boehmite alumina nanofillers loading. It was confirmed that a two-stage direct mixing method is effective to prepare good quality nanocomposites in laboratory scale. Conclusion is drawn as follows as for nanostructuration effects.

(1) PD resistance increases as the filler content increase; e.g. 7 wt% nanofiller content creates a 60 % decrease in depth of PD-caused erosion.

(2) Weibull analysis shows that short-time electrical treeing breakdown time is prolonged to 265 % by 5 wt% addition of nanofillers. But there was more data scatter in nanocomposites than in pure epoxy.

(3) Permittivity tends to increase from 3.7 to 4.0 by 5 wt% addition of nanofillers as opposed to what was newly found in the recent past.

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(6) Interfacial bonding seems to be rather weak in the nanocomposite specimens prepared this time.

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