

Dielectric Properties of Epoxy/Alumina Nanocomposite Influenced by Control of Micrometric Agglomerates

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ABSTRACT

Introduction of metal oxide nanoparticles to polymer material is known to have unique dielectric behavior and significant advantages in electrical insulation performance in power apparatus. This paper presents an attempt to clarify the influence of dispersibility of nanoparticles, especially focusing on agglomerates, on dielectric properties of a nanocomposite system by changing particle dispersion processes. Experiments were carried out in epoxy/alumina nanocomposites with the particle dispersion techniques by applying ultrasonic wave and centrifugal force. For the dispersibility control of nanoparticles, we changed the duration of ultrasonic wave and centrifugal force. The experimental results clarified the effect of centrifugal force on the separation of agglomerates and the effect of ultrasonic wave on the disruption of agglomerates. Next, we examined the dielectric properties such as relative permittivity and $\tan \delta$ of the nanocomposites. As the result, we verified that the permittivity of epoxy/alumina nanocomposites became low due to separation and disruption effects of the agglomerates.

Index Terms — Epoxy nanocomposite, alumina nanoparticle, ultrasonic wave, centrifugal force, agglomerate, particle dispersibility, permittivity, $\tan \delta$.

1 INTRODUCTION

FOR the enhancement of electrical insulation reliability and compact design in electric power apparatus, the electrical properties of polymer composites filled with metal oxide particles will play an important role. Recently, it has been recognized that the introduction of metal oxide nanoparticles to polymer has unique dielectric behavior and advantages when compared to the base polymer and the polymer filled with conventional micrometric particles [1-6]. These properties would be mainly attributed to the large interfacial area of nanoparticles with homogeneous dispersibility and the specific interfacial region

between a nanoparticle and polymer [7-13]. On the other hand, it has been reported that the poor dispersibility of nanoparticles, e.g. the agglomerate of nanoparticles which might inevitably occur in fabrication process, might affect the dielectric and electrical properties [14-17]. Questions still remain as to how dispersibility of nanoparticles influences the specific properties of nanocomposites and what degree of dispersion is necessary to obtain excellent characteristics of the nanocomposite. Therefore, the influence of the dispersibility of nanoparticles on electrical and dielectric properties in nanocomposite materials needs to be clarified.

This paper focuses on the influence of agglomerates of alumina nanoparticles in epoxy resin on dielectric characteristics. Firstly, epoxy/alumina nanocomposites were fabricated with particle dispersion techniques using ultrasonic wave and centrifugal force. For the control of the dispersibility of nanoparticles, we changed the duration of ultrasonic wave and centrifugal force. We verified that the application of centrifugal force could separate the agglomerates and the application of ultrasonic wave could disrupt them. Then, we examined the dielectric permittivity and dielectric loss of nanocomposites with various dispersibilities. As the result, we verified that dielectric permittivity of epoxy/alumina nanocomposites was made low by separation and disruption of the agglomerates.

2 EXPERIMENT

2.1 FABRICATION OF NANOCOMPOSITE WITH PARTICLE DISPERSION PROCESS

We fabricated the nanocomposite material from epoxy resin matrix with alumina nanoparticles as shown in Table 1. The epoxy resin was based on bisphenol-A cured with anhydride-type hardener. The alumina nanoparticles were of spherical shape with an average diameter of 31 nm. The filler content was 0.5 vol%. As coupling agent, silane coupling was added in epoxy resin.

Table 1. Specifications of materials for nanocomposites.

Epoxy resin	Hardener	Alumina filler		Coupling agent
		Average diameter	Filler content	
Bisphenol-A	Anhydride-type	31 nm	0.5 vol%	Silane coupling

To improve the dispersibility of the nanoparticles in epoxy resin, we consecutively applied centrifugal force and ultrasonic wave. Figure 1 shows a schematic illustration indicating the expected effect of particle dispersion by application of centrifugal force and ultrasonic wave. By application of centrifugal force to the nanocomposite (see Figure 1a), no nanoparticles but the agglomerates of nanoparticles can be precipitated in the direction of centrifugal force, because the agglomerates are large in diameter. Next, by removing the precipitation in the fabrication process, we can obtain nanocomposites with fully dispersed particles.

On the other hand, by application of ultrasonic wave to the nanocomposite material (see Figure 1b), the agglomerates of nanoparticles can be disrupted due to the pressure oscillation with high frequency occurring in the nanocomposite material. Furthermore, by applying the ultrasonic wave and subsequently applying centrifugal force (see Figure 1c), residual agglomerates which remain after application of the ultrasonic wave can be clearly separated.

We fabricated the nanocomposite specimens with different particle dispersibility by changing the application conditions of

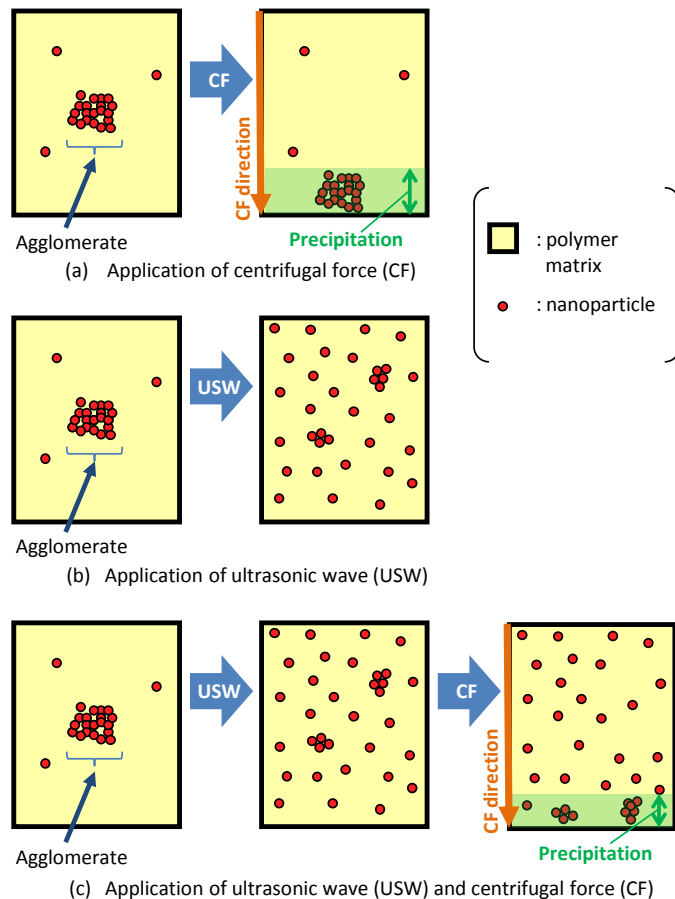


Figure 1. Illustration of particle dispersion effect.

centrifugal force and ultrasonic wave. The fabrication process of the nanocomposite specimens consisted of the following steps [16].

- (1) Epoxy resin was mixed with hardeners, silane coupling and fillers by a planetary mixer.
- (2) Centrifugal force and ultrasonic wave were applied to the mixture. The application conditions were varied as shown in Table 2. Especially, for N_UC, we applied the ultrasonic wave and subsequently applied the centrifugal force.
- (3) The upper part of the mixture was poured into casts for removing the precipitation of the agglomerates, and then this was mixed again for homogenizing the distribution of particle density caused by the application of centrifugal force.
- (4) The specimen was degassed sufficiently for removal of bubbles, and then was cured and postcured.

The specimens were sliced as discs with a thickness of 1.0mm for the measurement of filler contents, agglomerate diameters and dielectric properties.

Table 2. Specimen list with application conditions of centrifugal force and ultrasonic wave.

Specimens	Centrifugal force application		Ultrasonic wave application	
	Force	Duration	Frequency	Duration
N_MM	1 G (gravitation)	–	No operation	
N_CF1	200 G	1 hour		
N_CF2	2,000 G			
N_CF3	20,000 G			
N_US1	1 G (gravitation)	–	40 kHz	1 min
N_US2				5 min
N_US3				30 min
N_UC	20,000 G	1 hour		

2.2 QUANTITATIVE ANALYSIS OF PARTICLE DISPERSIBILITY

We quantified the particle dispersibility of nanocomposite by scanning electron microscope (SEM) image analysis and filler content measurement.

SEM image analysis was used from micro/nanoscope viewpoints on the particle dispersibility. We firstly focused on the diameter of the agglomerate as a typical parameter of the dispersibility of nanoparticles. By analyzing SEM micrographs using image processing and image analysis, we investigated the maximum diameter of agglomerate cross-section, which was assumed to be the same as the actual diameter of the agglomerate. In order to find the representative maximum diameter of all the agglomerates, we measured more than 1,000 particles with larger diameter from 1 disc of the sliced specimen, based on the statistical investigation of particle distribution by image analysis.

Filler content was measured from macroscopic viewpoints on the particle dispersibility. We focused on the filler content at the dispersed area without agglomerates, which was located above the precipitation area. If the nanocomposite has “many” agglomerates of nanoparticles (see Figure 1a), the filler content above the precipitation area can be decreased significantly by gravity force and centrifugal force. On the other hand, if the nanocomposite has few agglomerates of nanoparticles after application of ultrasonic wave (see Figure 1(c)), even if centrifugal force is applied, the filler content above the precipitation area can be decreased slightly. The filler contents above the precipitation area were estimated by measurement of the specific gravity of the sliced specimen.

2.3 DIELECTRIC PROPERTIES MEASUREMENT

Relative permittivity and dielectric loss tangent ($\tan \delta$) were estimated from the measurement of capacitance of the specimens at the frequency from 1 kHz to 1 MHz under 30 °C. The accuracy of the measurement was confirmed by measuring the permittivity and $\tan \delta$ of a standard PTFE sample provided.

3 RESULTS AND DISCUSSION

3.1 PARTICLE DISPERSIBILITY OF NANOCOMPOSITE SPECIMENS

3.1.1 SEM OBSERVATION

Examples of SEM micrograph of the fabricated specimens are shown in Figures 2 and 3 for magnification of 1,000 and 20,000, respectively. In the micrographs, white parts show alumina particles and black parts show epoxy resin. The distribution of the micrometric agglomerate of the nanoparticles can be discussed in Figure 2. By application of the centrifugal force, micrometric agglomerates are well separated. On the other hand, by application of ultrasonic wave, the number of micrometric agglomerates is decreased. These results can qualitatively bring about the confirmation of the particle dispersion effect for micrometric agglomerates by centrifugal force and ultrasonic wave. Furthermore, the particle distribution with less than 1 μm diameter can be discussed in Figure 3. Although we can see several small agglomerates with less than 1 μm diameter, the nanoparticles are well dispersed by application of ultrasonic wave.

3.1.2 EFFECT OF CENTRIFUGAL FORCE ON PARTICLE DISPERSIBILITY

The agglomerate diameter of specimens with no application of ultrasonic wave is shown in Figure 4 as a function of centrifugal force. In this figure, the largest 10 data are sequentially plotted from the observed micrographs for each specimen. With the increase of centrifugal force, size reduction of agglomerates can be seen. In particular at 20,000 G application, the diameters of the agglomerates are reduced to 500–900 nm. Furthermore, the movement of an agglomerate in viscous liquid under centrifugal force is calculated with the assumption that the shape of the agglomerate is spherical, by the following equation [16, 18].

$$D_p^2 = \frac{18\eta_c A}{F\pi^2(\rho_p - \rho_f)(1 - e^{-At})} R_1 \ln \frac{R_1}{R_0} \quad (1)$$

Here, D_p is particle diameter, η_c is viscosity, A is the increase rate of the viscosity for the curing time, R_1 and R_0 are outer and inner radii of centrifuging, respectively, F is centrifugal force, t is duration, ρ_p and ρ_f are specific gravity of the particle and polymer matrix, respectively.

In Figure 4, the measured values of agglomerate diameter are close to the calculated value. This result confirms that the centrifugal force application is effective in separating the agglomerates with micrometric diameter. On the other hand,

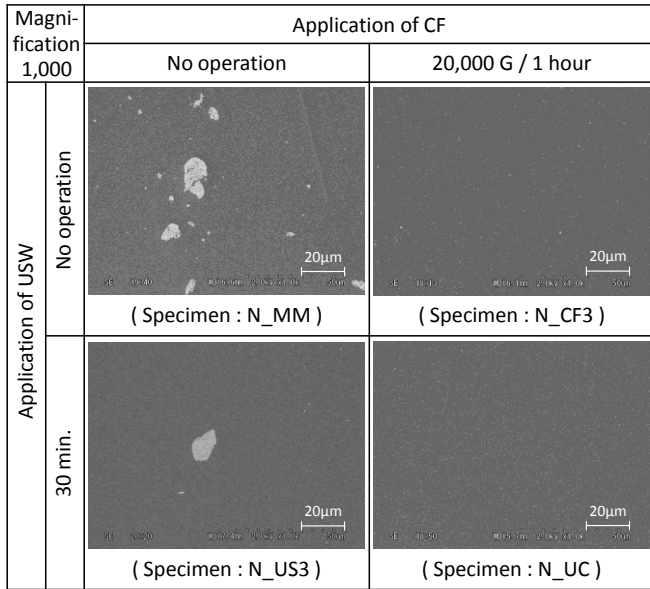


Figure 2. SEM micrographs of epoxy/alumina nanocomposite at magnification of 1,000.

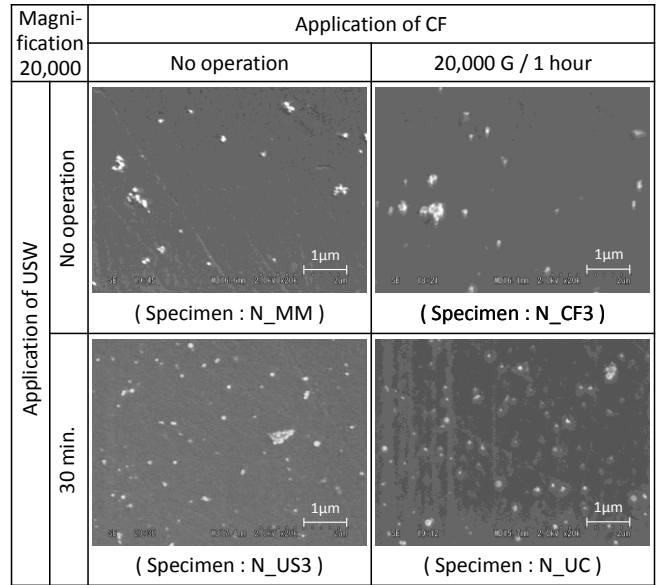


Figure 3. SEM micrographs of epoxy/alumina nanocomposite at magnification of 20,000.

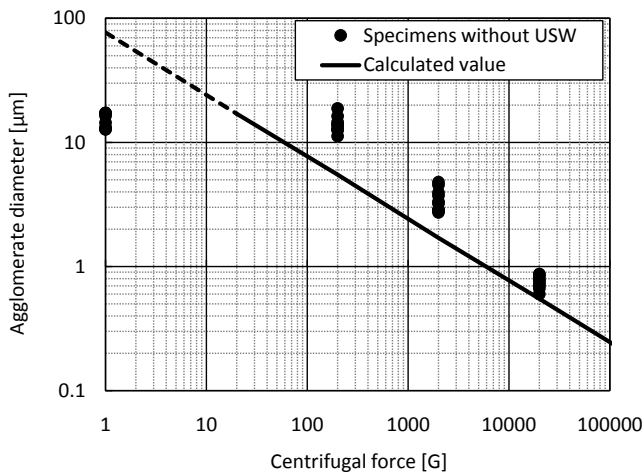


Figure 4. Agglomerate diameter as a function of centrifugal force.

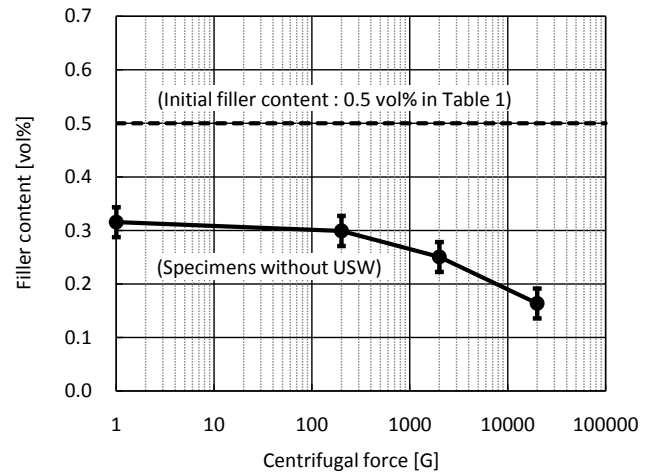


Figure 5. Filler content as a function of centrifugal force.

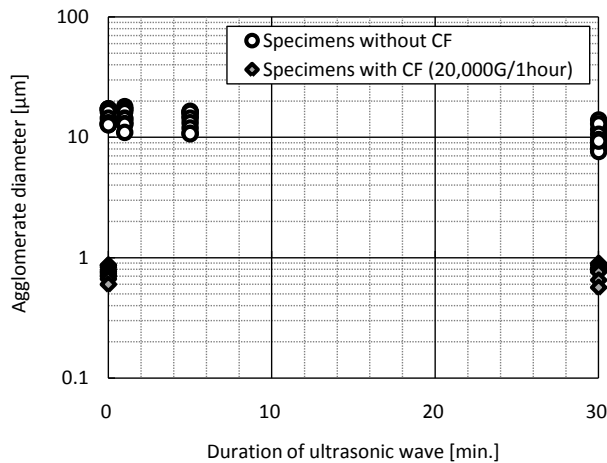


Figure 6. Agglomerate diameter as a function of duration of ultrasonic wave.

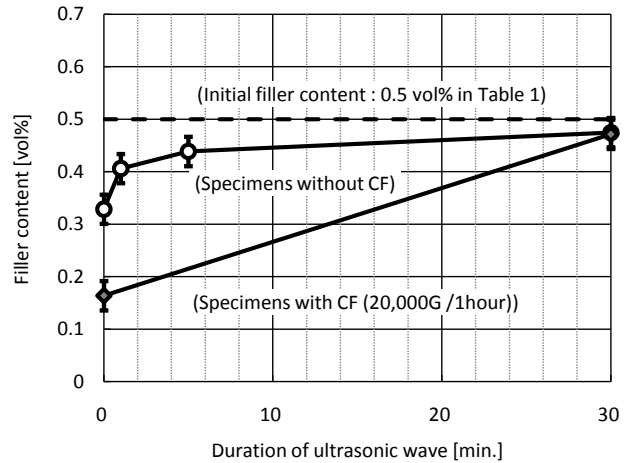


Figure 7. Filler content as a function of duration of ultrasonic wave.

the measured values at 1G are significantly smaller than the calculated value. This may be explained by the fact that agglomerates as large as 20 μm in diameter can precipitate in curing process at high temperature. The measured values at 200–20,000 G are a bit larger than the calculated value. The reason is presumed to be the inclusion of epoxy matrix and impurities (e.g. moisture) inside the agglomerates

The filler content of the specimens with no application of ultrasonic wave is shown in Figure 5 as a function of centrifugal force. The filler contents of specimens (0.16–0.32 vol %) are lower than the initial filler content (0.5 vol% in Table 1). From the results, it is confirmed that a lot of agglomerates can be precipitated by both gravitation and centrifugal force and thus, the precipitation can be removed from the specimens in the fabrication process. Furthermore, the filler content of specimens is decreased with the increase of centrifugal force, because the stronger centrifugal force can separate smaller diameter agglomerates.

3.1.3 EFFECT OF DURATION OF ULTRASONIC WAVE ON PARTICLE DISPERSIBILITY

Figure 6 shows the largest 10 diameters of the agglomerate as a function of the duration of ultrasonic wave. With the increase of the duration of ultrasonic wave, the agglomerate diameter of the specimens without centrifugal force is not significantly decreased. On the other hand, the size reduction of agglomerates can be seen by application of centrifugal force, even if ultrasonic wave was applied. This result indicates that the agglomerate diameter is mainly determined by the centrifugal force or the gravitation.

Figure 7 shows the filler content as a function of the duration of ultrasonic wave. The filler content of specimens (0.33–0.48 vol %) without centrifugal force is lower than the initial filler content (0.5 vol%) and increases with the increase of the duration of ultrasonic wave. The result indicates that the application of ultrasonic wave can increase the number of dispersed nanoparticles in spite of the fact that the maximum diameter of agglomerate is not significantly decreased (see also Figure 6). Furthermore, the filler content of specimens with duration of ultrasonic wave for 30 min (0.47 vol%) is almost the same as those without centrifugal force. The result exhibits that the application of the ultrasonic wave for 30 min can disrupt almost all of the micrometric agglomerates into nanometric particles.

3.1.4 CONTROL OF MICROMETRIC AGGLOMERATE DISTRIBUTION

The maximum diameter of agglomerates and the filler contents for all of the tested specimens are compared in Figure 8. The particle dispersion effect by centrifugal force and ultrasonic wave is confirmed from this figure as below.

- (1) Application of centrifugal force can reduce both of the maximum diameter of agglomerates and the filler content, because the micrometric agglomerates are separated.

- (2) Application of ultrasonic wave can increase the filler content, because almost all the agglomerates can be disrupted into nanometric particles. It is notable that the agglomerate disruption and nanoparticle dispersion could occur simultaneously by application of ultrasonic wave.

According to the above results, we clarify that the application conditions of centrifugal force at 200–20,000 G for 1 hour and duration of ultrasonic wave for 1–30 min can improve the dispersibility in micrometric particle distribution. Furthermore, by applying the ultrasonic wave and subsequently applying the centrifugal force, we can obtain the nanocomposite with “better” dispersibility of nanoparticles, i.e. all dispersed particles were less than 1 μm in diameter. However, particles of several hundred nanometers still exist and are expected to be improved by optimization of application conditions of ultrasonic wave and centrifugal force.

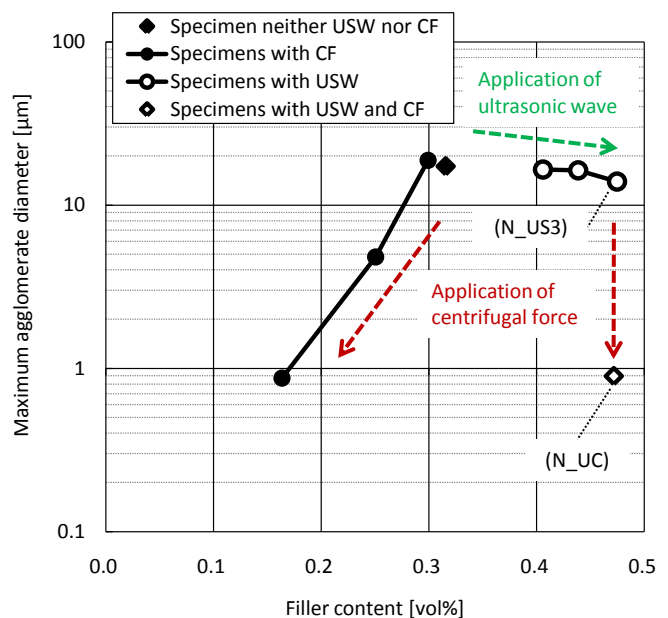


Figure 8. Relationship between maximum agglomerate diameter and filler content under ultrasonic wave and centrifugal force.

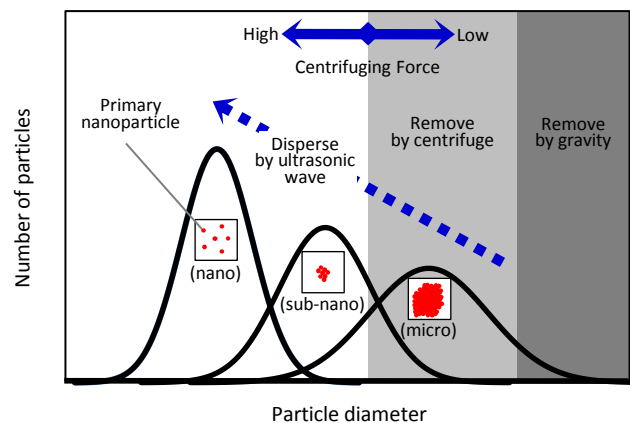


Figure 9. Illustration of improving dispersibility by ultrasonic wave and centrifugal force.

From the viewpoint of particle distribution, Figure 9 illustrates the further reducing effect of agglomerate diameter and number by application of the ultrasonic wave and the centrifugal force. From this figure, it is understood that the application of ultrasonic wave can move the average particle size distribution to smaller diameter, i.e. improvement of dispersibility, thus filler content after applying the ultrasonic wave does not change even if centrifugal force is applied.

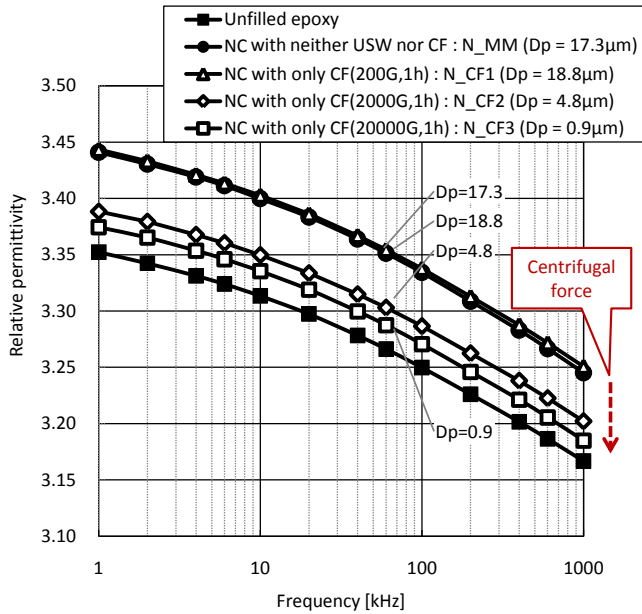


Figure 10. Frequency dependence of relative permittivity with respect to centrifugal force (Dp: maximum diameter of agglomerate).

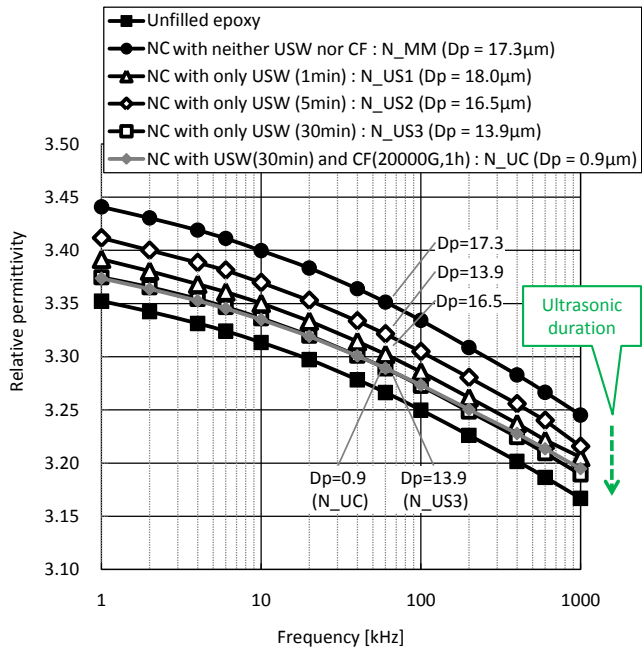


Figure 12. Frequency dependence of relative permittivity with respect to duration of ultrasonic wave (Dp: maximum diameter of agglomerate).

3.2 DIELECTRIC PROPERTIES OF NANOCOMPOSITE SPECIMENS

3.2.1 INFLUENCE OF DURATION OF ULTRASONIC WAVE AND CENTRIFUGAL FORCE

The frequency dependence of relative permittivity is shown in Figure 10 for different centrifugal force conditions without ultrasonic wave. The specimen of unfilled epoxy is used for comparison with nanocomposites. Relative permittivity of the nanocomposite applied with neither ultrasonic wave nor centrifugal force (N_MM) is higher than that of unfilled epoxy.

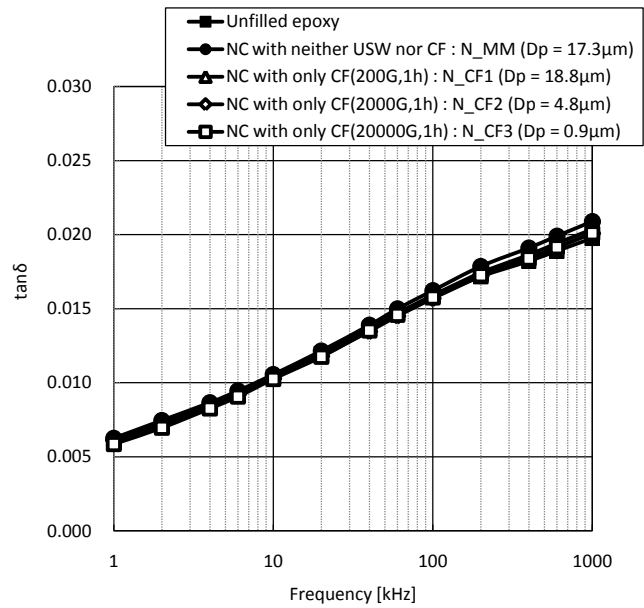


Figure 11. Frequency dependence of $\tan \delta$ with respect to centrifugal force (Dp: maximum diameter of agglomerate).

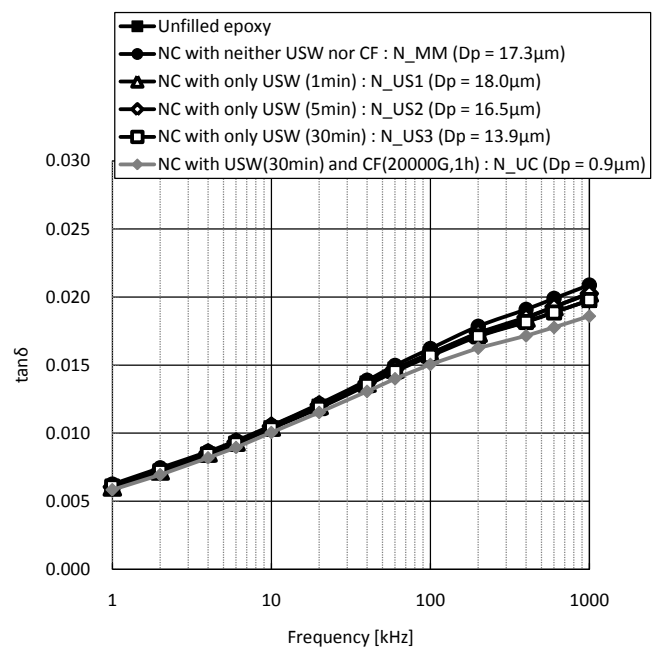


Figure 13. Frequency dependence of $\tan \delta$ with respect to duration of ultrasonic wave (Dp: maximum diameter of agglomerate).

With the decrease of the maximum diameter of agglomerate (D_p) by increasing the centrifugal force, relative permittivity of nanocomposites becomes low and close to that of unfilled epoxy. These results indicate that relative permittivity of nanocomposite with micrometric agglomerates is higher than that of unfilled epoxy and can be decreased by separating micrometric agglomerates. $\tan \delta$ for different centrifugal force conditions is shown in Figure 11. $\tan \delta$ of nanocomposites is almost the same, even if the centrifugal force is increased. The data shows that the influence of centrifugal force to $\tan \delta$ is small.

Figure 12 shows relative permittivity for different duration of ultrasonic wave conditions. With the increase of the duration of ultrasonic wave, even if maximum diameter of agglomerates does not significantly change, relative permittivity of nanocomposites without centrifugal force becomes decreased and close to that of unfilled epoxy. In contrast, relative permittivity of the nanocomposite with only ultrasonic wave (N_US3) is almost the same as that of the nanocomposite with ultrasonic wave and centrifugal force (N_UC) in spite of the fact that N_US3 and N_UC have different maximum diameters of agglomerate. These results indicate that influence of ultrasonic wave on relative permittivity of nanocomposites cannot be explained by maximum diameter of agglomerates. $\tan \delta$ for different duration of ultrasonic wave is shown in Figure 13. With the increase of the duration of ultrasonic wave, $\tan \delta$ of nanocomposites tends to be slightly decreased. The data shows that the influence of the duration of ultrasonic wave to $\tan \delta$ is small.

3.2.2 INFLUENCE OF FILLER CONTENT

Figure 14 shows the relative permittivity at 1 MHz as a function of filler contents for all of the tested specimens. Relative permittivity of nanocomposite specimens with the application of centrifugal force only is higher than those of unfilled epoxy and become close to those of unfilled epoxy with decreasing filler contents. This result is attributed to the agglomerate separation effect by centrifugal force and indicates that relative permittivity of agglomerates is higher than that of unfilled epoxy.

On the other hand, relative permittivity of nanocomposite specimens with the application of ultrasonic wave only is higher than unfilled epoxy and becomes close to those of unfilled epoxy with increasing the filler content. It was presumed that, by the application of ultrasonic wave, agglomerates can be disrupted and nanoparticles can be dispersed simultaneously, which could affect permittivity of nanocomposites. Therefore, agglomerate disruption and nanoparticle dispersion need to be separated for confirmation of the influence of the dispersion effect by ultrasonic wave to dielectric properties.

Furthermore, relative permittivity of nanocomposite specimens with the application of ultrasonic wave only (N_US3) and with the application of both ultrasonic wave and centrifugal force (N_UC) were almost the same. N_UC is the

nanocomposite which separates a small number of micrometric agglomerates from N_UC3 by centrifugal force as shown in Figure 8. This result indicates that a small number of micrometric agglomerates does not affect relative permittivity if nanoparticles are dispersed.

However, the fabricated nanocomposites by ultrasonic wave and centrifugal force still have agglomerates of several hundred nanometers in diameter and thus, unique dielectric properties due to the specificity of nanoparticle, i.e. dual layer model and multi-core model, exists behind permittivity characteristics of agglomerate [8, 11]. Besides, the optimization of centrifugal force technique for separating smaller agglomerates could verify not only specific dielectric properties by nanoparticles but also the dispersion degree of nanoparticles necessary to obtain excellent characteristics of the nanocomposite.

Figure 15 shows $\tan \delta$ at 1 MHz as a function of filler contents for all of the tested specimens. $\tan \delta$ tends to be slightly decreased by improvement of particle dispersibility as shown in Figure 8 but the change of $\tan \delta$ is small.

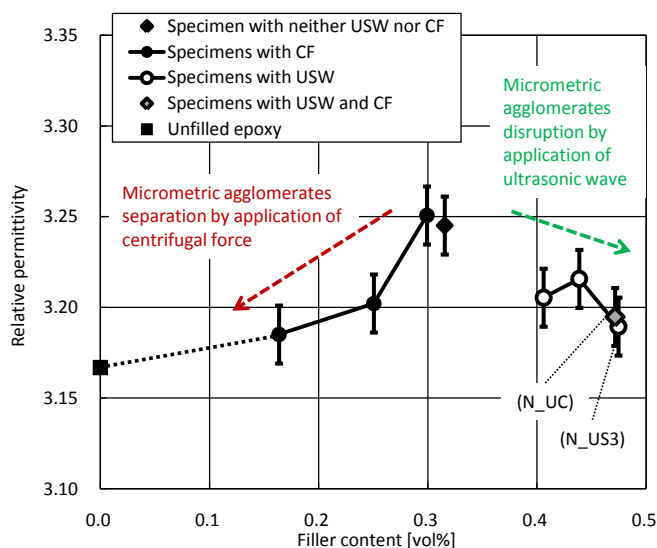


Figure 14. Filler content dependence of relative permittivity.

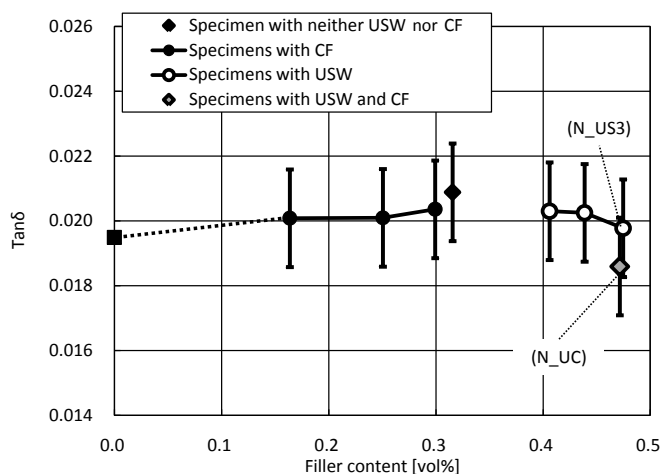


Figure 15. Filler content dependence of $\tan \delta$.

4 CONCLUSIONS

In order to derive the influence of the dispersibility of nanoparticles on electrical properties of epoxy nanocomposite material, we investigated the dielectric properties of epoxy/alumina nanocomposites with agglomerates control. Firstly, we fabricated epoxy/alumina nanocomposites and evaluated the agglomerate diameter by an image processing and analysis of SEM micrographs and measured filler contents of specimens. Next, we examined the relative permittivity and $\tan \delta$ for different application conditions of the ultrasonic wave and the centrifugal force. The main results are summarized as follows.

- (1) For improvement of dispersibility of nanoparticles in the fabrication process by direct mixing, the applications of centrifugal force and ultrasonic wave are strongly effective. Application effects of centrifugal force and ultrasonic wave are separation and disruption of agglomerates of nanoparticles, respectively.
- (2) The distribution of micrometric agglomerates can be controlled by changing the duration of ultrasonic wave and the amount of centrifugal force.
- (3) Relative permittivity of nanocomposite in frequency range of 1 kHz – 1 MHz can be made lower by the application of centrifugal force and ultrasonic wave.
- (4) The lower permittivity of nanocomposite by centrifugal force is caused by the separation of micrometric agglomerates. This effect is consistent with the fact that dielectric permittivity of micrometric agglomerate was higher than that of particle material.
- (5) The lower permittivity of nanocomposite by ultrasonic wave is caused by disruption of agglomerates and dispersion of nanoparticles.

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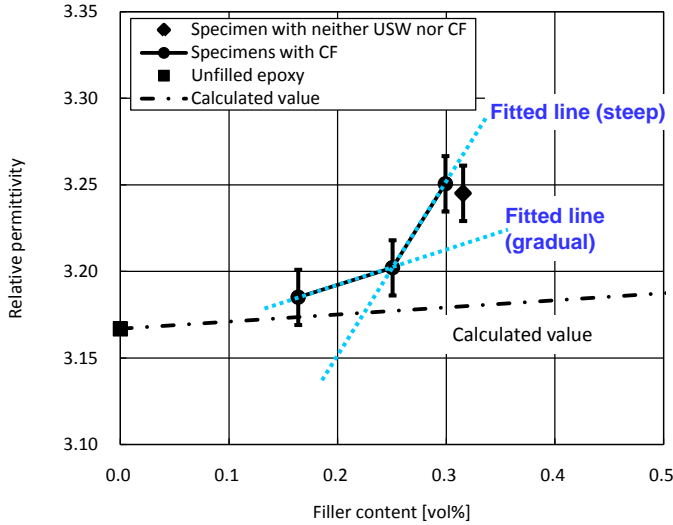


Figure 16. Filler content dependence of relative permittivity at 1MHz (NC without USW).

3.2.3 EQUIVALENT PERMITTIVITY OF MICROMETRIC AGGLOMERATE

Tendency of relative permittivity with application of centrifugal force only is considered to be determined by micrometric agglomerate, because the nanocomposite specimens without ultrasonic wave have plenty of micrometric agglomerates. In Figure 16, we compared the measured values of relative permittivity of nanocomposite (application of centrifugal force only) with the calculated value of the effective permittivity. For calculation of the effective permittivity, we used the equation (2) with the random arranged model of alumina particle material and epoxy material [19].

$$\epsilon_a = \sqrt{\frac{(\epsilon_p - \epsilon_m) * V_p + \epsilon_m}{\left(\frac{1}{\epsilon_p} - \frac{1}{\epsilon_m}\right) * V_p + \frac{1}{\epsilon_m}}} \quad (2)$$

Here, ϵ_p is relative permittivity of alumina (9.3), ϵ_m is relative permittivity of epoxy (3.17), V_p is the volume fraction of alumina to epoxy matrix. From this figure, the measured values are found to be higher than the calculated value. This result indicates that the micrometric agglomerate has higher permittivity than alumina material.

Furthermore, we presumed an equivalent permittivity of micrometric agglomerate by assigning derivatives of approximation line of measured values to the differentiation value by V_p of equation (2). It is 39.8–214.8 with higher than alumina material. This higher value may be attributed to the inclusion of moisture and other specific effect of agglomerate structure [20, 21].

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