

Permittivity Characteristics of Epoxy/Alumina Nanocomposite with High Particle Dispersibility by Combining Ultrasonic Wave and Centrifugal Force

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ABSTRACT

This paper proposes a novel technique to fabricate epoxy/alumina nanocomposites with nanoparticle composite process by combination of ultrasonic wave and centrifugal force. The particle dispersion effect of the nanoparticle composite process and its influence on dielectric permittivity were discussed quantitatively. Experimental results clarified that the combination of ultrasonic wave and centrifugal force was effective to increase dispersed nanoparticles and as well as to separate residual agglomerates. We verified that the improvement of particle dispersibility in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force could bring about lower permittivity of the nanocomposites, especially than that of unfilled epoxy material.

Index Terms —Epoxy nanocomposite, alumina particle, permittivity, particle dispersibility, ultrasonic wave, centrifugal force

1 INTRODUCTION

EPOXY composite material filled with metal oxide particles is widely used for insulating material in electric power apparatus [1]. As new composite materials with significant features of insulation performances, the nanocomposite through the incorporation of nanoparticles and functionally graded material (FGM) with grading distribution of filler density have been reported [2-4]. Recently, epoxy nanocomposite through the incorporation of nanometric particles (nanoparticles) is being increasingly investigated for several dielectric properties such as dielectric permittivity and loss tangent, electrical conductivity, space charge formation, electrical breakdown characteristics, long term voltage endurance [5-8]. Especially, dielectric

permittivity of the epoxy nanocomposite was reported to be higher and lower than that of unfilled epoxy material [9-14]. The higher permittivity of the nanocomposite would be mainly attributed to permittivity of metal oxide filler, which is higher than that of unfilled epoxy material and the inclusions, e.g. moisture included on the interface of nanoparticles and inside agglomerates. On the other hand, lower permittivity of the nanocomposite would be mainly attributed to the restriction of polymer chain movement in the specific interfacial region between a nanoparticle and polymer and also by the large interfacial area of nanoparticles with homogeneous dispersibility. The dispersibility of nanoparticles can be more or less essential properties in discussing the dielectric characteristics of nanocomposites [15-17]. However, it has not yet been made clear how the specific properties of nanocomposites are influenced by the dispersibility of nanoparticles. Moreover, the

difficulty still remains in how to control the dispersibility of nanoparticles and how to quantify the dispersibility of nanoparticles [18].

In our previous studies, we investigated the particle dispersion effect by ultrasonic wave and centrifugal force and its influence on relative permittivity of epoxy/alumina nanocomposites [19]. By quantifying the particle dispersibility with using SEM image analysis and filler content measurement, we clarified the influence of agglomerates with micrometric diameter on permittivity characteristics. However, the residual agglomerates with diameter of 600 nm- 20 μm still existed in the fabricated nanocomposites.

In this paper, we propose a novel technique with nanoparticle composite process by combination of ultrasonic wave and centrifugal force. Its influences on particle dispersion and dielectric permittivity of epoxy/alumina nanocomposites were discussed as main parameters of application duration of ultrasonic wave and centrifugal force in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

2 NANOPARTICLE DISPERSION TECHNIQUE COMBINING ULTRASONIC WAVE AND CENTRIFUGAL FORCE

In order to obtain the homogeneous dispersion of nanometric particle (nanoparticle) in polymer matrix, it is necessary to develop both surface treatment of particles and particle dispersion process. The surface treatment of particles is a method to improve the affinity of polymer and particle surface, e.g. by silane treatment of particles and addition of silane coupling agent. The particle dispersion process is a direct mixing method, e.g. by mechanical extruder and kneading machine to disperse nanoparticles by strong shearing force, and application of ultrasonic wave to disperse nanoparticles by the pressure oscillation with high frequency.

As a novel direct mixing method, we suggest nanoparticle composite process by combination of ultrasonic wave (USW) and centrifugal force (CF). Figure 1 shows a schematic illustration indicating the expectation of particle dispersion from the nanoparticle composite process by combination of USW and CF, which has a consecutive process of USW and CF. Applications of USW and CF are expected to disrupt agglomerates and to separate residual agglomerates, respectively.

3 EXPERIMENTAL

3.1 MATERIALS AND FABRICATION PROCESS

Nanocomposite material in this paper was composed of alumina nanoparticles filled in epoxy matrix as shown in Table 1. The alumina nanoparticles were of spherical shape with an average diameter of 31 nm as shown in Figure 2. Filler content was 0.5 vol%. The epoxy matrix was based on bisphenol-A cured with anhydride-type hardener. Silane coupling agent was added in epoxy resin.

We fabricated nanocomposite specimens with different

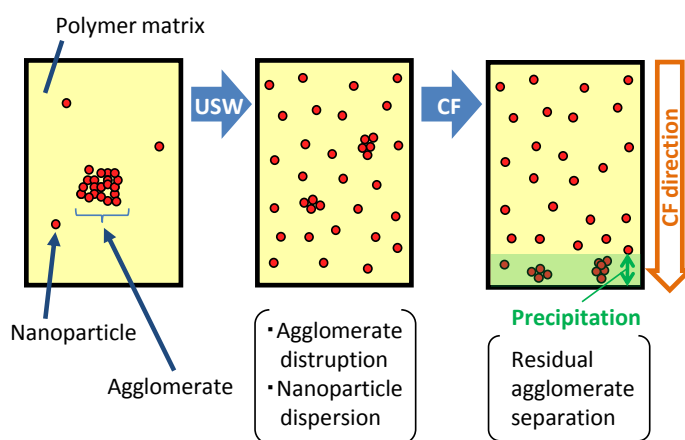


Figure 1. Schematic illustration for particle dispersion effect of nanoparticle composite process by combination of ultrasonic wave and centrifugal force. (USW: ultrasonic wave, CF: centrifugal force)

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

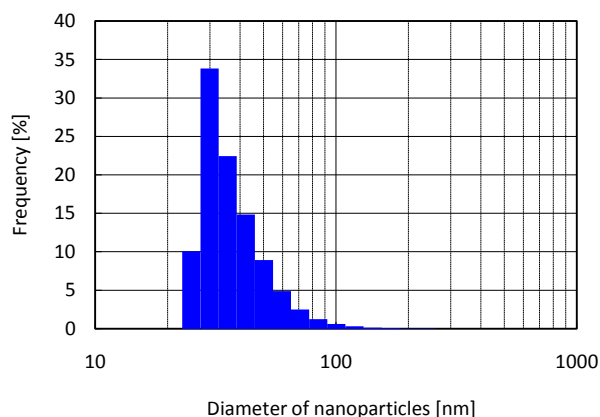


Figure 2. Particle distribution of alumina nanoparticles with an average diameter of 31nm.

particle dispersibility by changing particle dispersion process with USW and CF as shown in Table 2. As main parameters in the nanoparticle composite process by combination of USW and CF, application duration of USW was varied to 1-30 minutes and application duration of CF was varied to 1-12 hours. The specimens without USW (with neither USW nor CF, with only CF under low temperature of 20 $^{\circ}\text{C}$) have been obtained as nanocomposites with poor dispersibility [17].

The fabrication process of the nanocomposite specimens consisted of the following steps.

- (1) Epoxy resin is mixed with hardeners, silane coupling and alumina nanoparticles by a planetary mixer.

- (2) USW and CF are applied consecutively to the mixture. After application of the CF, the precipitation is removed from the mixture.
- (3) The mixture is poured into casts with a diameter of 50mm, and is degassed enough for removal of bubbles.
- (4) The specimen is cured and postcured.
- (5) The specimens were sliced as discs with a thickness of 1.0 mm for the measurement of filler contents and the dielectric permittivity.

Table 2. Application conditions of ultrasonic wave and centrifugal force for nanocomposites.

Specimens	Ultrasonic wave application		Centrifugal force application		
	Frequency	Duration	Force	Duration	Temperature
N_MM	No operation		1 G (gravitation)	–	–
N_Ca			200 G	1 hour	20 °C
N_Cb			2,000 G		
N_Cc			20,000 G		
N_U1	40 kHz	1 min	1 G (gravitation)	–	–
N_U1/C1			20,000 G	1 hour	47 °C
N_U1/C12				12 hour	
N_U5		5 min	1 G (gravitation)	Without	–
N_U5/C1			20,000 G	1 hour	47 °C
N_U5/C12				12 hour	
N_U30		30 min	1 G (gravitation)	–	–
N_U30/C1			20,000 G	1 hour	47 °C
N_U30/C3				3 hour	
N_U30/C6				6 hour	
N_U30/C9				9 hour	
N_U30/C12				12 hour	

3.2 PARTICLE DISPERSIBILITY QUANTIFICATION

As a quantitative analysis of particle dispersibility from micro/nanoscope viewpoints, SEM image analysis was used. We focused on the diameter of the agglomerate as a typical indicator of the dispersibility of nanoparticles. By analyzing SEM micrographs with using image processing, we obtained the maximum diameter of agglomerate cross-section which was assumed to be the same as the actual diameter of the agglomerate.

As another quantitative analysis of particle dispersibility from macroscopic viewpoints, filler content was estimated. We focused on the filler content above the precipitation of the specimen. The filler contents can be estimated by the measurement of specific gravity of the sliced specimen at the fixed height from the bottom of each specimen. If “many” agglomerates of nanoparticles exist (see the left side of Figure 1), the filler content above the precipitation can be decreased significantly by gravity force and CF. On the other hand, if few agglomerates of nanoparticles exist after applying USW (see the right side of Figure 1), even if CF applied, the filler content above the precipitation can be decreased slightly.

3.3 DIELECTRIC PERMITTIVITY MEASUREMENT

Relative permittivity (ϵ_r) in the frequency of 1 kHz to 1 MHz under 30 °C was estimated from the measurement of capacitance of the sliced specimens. A specimen was placed between parallel circular electrodes with the guarded electrodes. We measured ϵ_r of a standard PTFE sample in order to confirm the accuracy of the measurement.

4 RESULTS AND DISCUSSIONS

4.1 EFFECTS OF COMBINATION OF ULTRASONIC WAVE AND CENTRIFUGAL FORCE TO PARTICLE DISPERSIBILITY

4.1.1 SEM OBSERVATION

The structural morphology of the nanocomposite specimens was observed by using SEM as shown in Figure 3. In the SEM micrographs, white and black parts show alumina particle and epoxy matrix, respectively. In the micrographs at the magnification of 1,000, we found that the number of agglomerates was reduced by application of USW and the agglomerates were well separated by application of CF after applying USW. In the micrographs at the magnification of 20,000, we found that the number of nanoparticles was increased by application of USW and then agglomerates with several hundred nanometer was well removed by application of CF after applying USW.

4.1.2 AGGLOMERATE DIAMETER

The agglomerate diameter of nanocomposite specimens is shown in Figure 4 as a function of ultrasonic duration in the nanoparticle composite process by combination of USW and CF. In this figure, the largest 10 data are sequentially plotted from the observed micrographs for each specimen. With the increase of ultrasonic duration, the maximum diameter (D_p) of agglomerate was not significantly decreased. On the other hand, the size reduction of agglomerates can be seen by the CF application. This result indicates that the agglomerate diameter is mainly determined by CF conditions in the nanoparticle composite process by combination of USW and CF.

The agglomerate diameter of nanocomposite specimens under application of USW for 30 minutes is shown in Figure 5 as a function of centrifugal duration in the nanoparticle

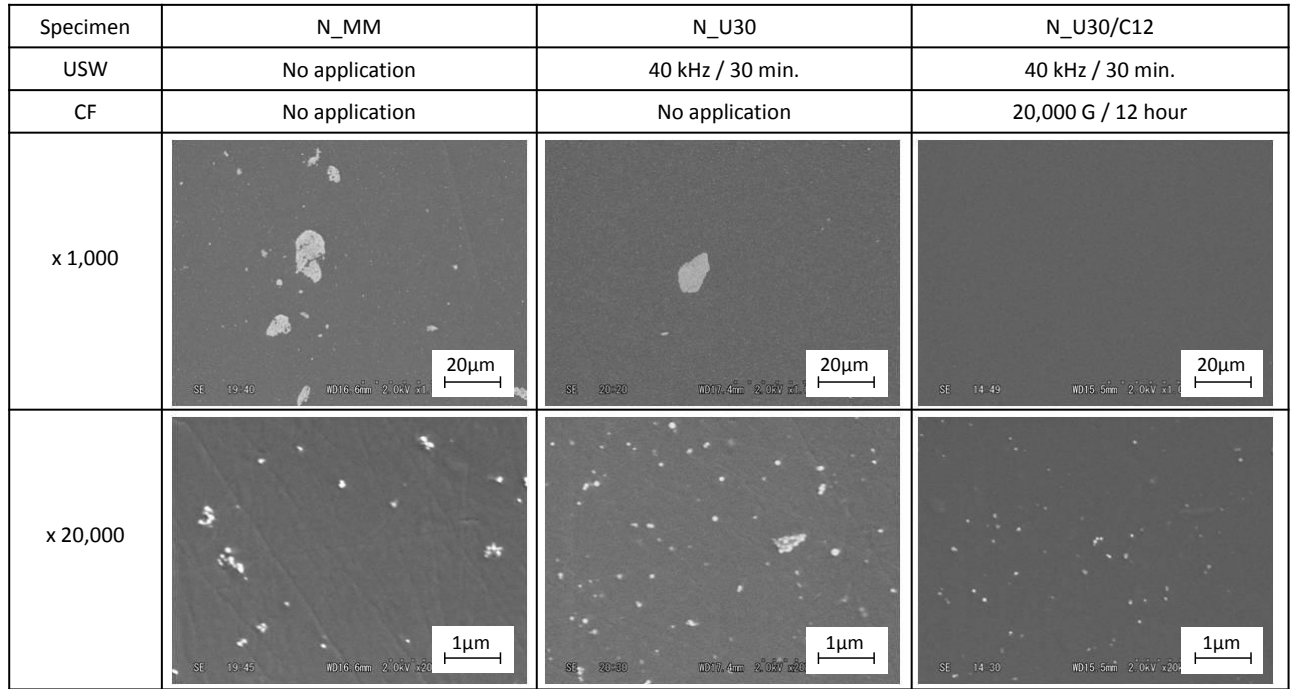


Figure 3. SEM micrographs of epoxy/alumina nanocomposite processed by the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

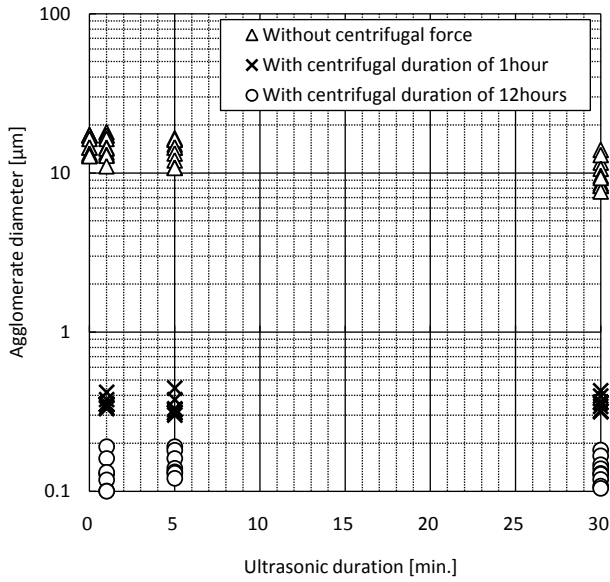


Figure 4. Agglomerate diameter of nanocomposite specimens as a function of ultrasonic duration (CF: 20,000G) in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

composite process by combination of USW and CF. With the increase of centrifugal duration, D_p decreased, especially down to 180 nm at the duration of 12 hours. Furthermore, the movement of an agglomerate in viscous liquid under CF application was calculated by the following equation [19].

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

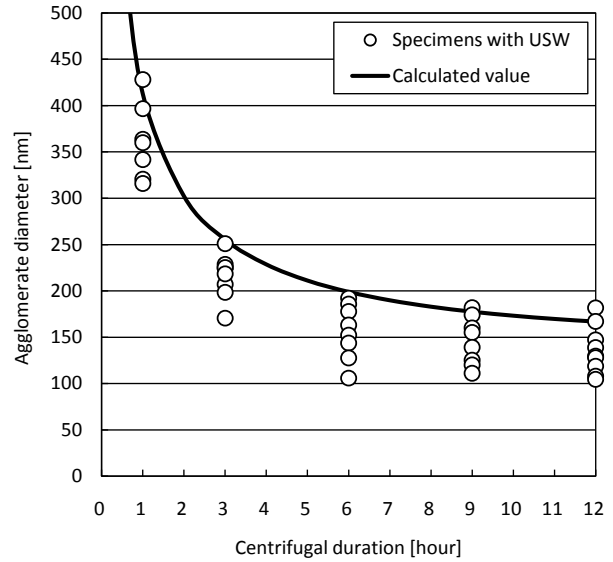


Figure 5. Agglomerate diameter of nanocomposite specimens under application of USW for 30 minutes as a function of centrifugal duration (CF: 20,000G) in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

Here, D_p is a particle diameter, η_c is a viscosity, A is an increase rate of the viscosity for the curing time, R_1 and R_0 are outer and inner radius of centrifuging, respectively, F is a centrifugal force, t is a duration, ρ_p and ρ_f are a specific gravity of the particle and polymer matrix, respectively. In this figure, the values of measured D_p are close to the calculated value. These results confirm that the CF application for 12 hours at 20,000 G can well separate the agglomerates larger than 180 nm in diameter.

4.1.3 FILLER CONTENT

Figure 6 shows the filler content (V_p) of nanocomposite specimens for different centrifugal duration as a function of ultrasonic duration in the nanoparticle composite process by combination of USW and CF. The filler content of the specimens was lower than the initial filler content (0.5 vol%) and increased with the increase of ultrasonic duration. The result indicates that the application of USW can increase the number of dispersed nanoparticles, in spite of the fact that D_p is not significantly decreased, as shown in Figure 5. On the other hand, with the increase of centrifugal duration, V_p decreased. The result also confirms that the CF application can well separate the agglomerates from macroscopic viewpoints of particle dispersibility. Accordingly, extension of ultrasonic duration under centrifugal duration for 12 hours in the nanoparticle composite process by combination of USW and CF can increase the number of dispersed nanoparticles of $D_p = 180$ nm.

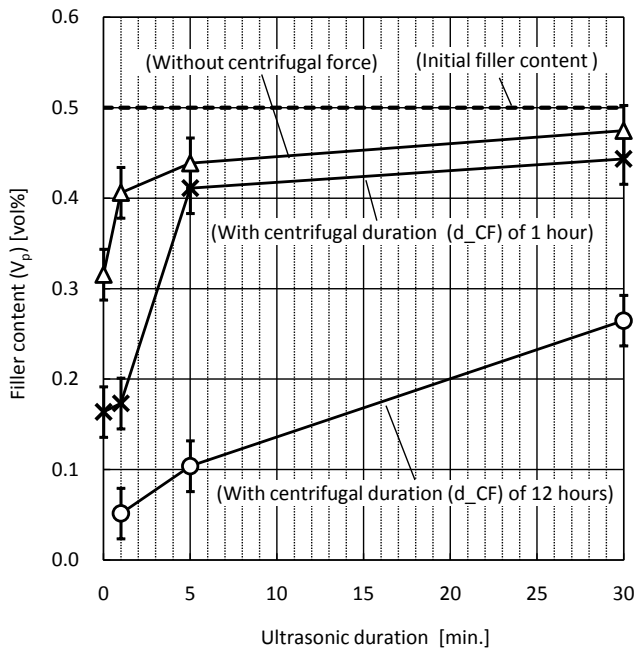


Figure 6. Filler content (V_p) of nanocomposite specimens as a function of ultrasonic duration (CF: 20,000G) in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

Figure 7 is the relationship between D_p and V_p for all of the tested specimens, which is derived from the vertical axes of Figure 4, Figure 5 and Figure 6. In this figure, we could obtain the nanocomposite specimens with different dispersibility at the same filler content, e.g. N_Cb and N_U30/C12, by controlling application conditions of USW and CF. N_Cb is the nanocomposite with D_p of 4.8 μm and V_p of 0.25 vol%, i.e. poor dispersibility. N_U30/C12 is the nanocomposite with D_p of 180 nm and V_p of 0.26 vol%, i.e. good dispersibility. In the following section 4.2.1, we will investigate the influence of particle dispersibility on relative permittivity of nanocomposites, by comparison of N_Cb and N_U30/C12.

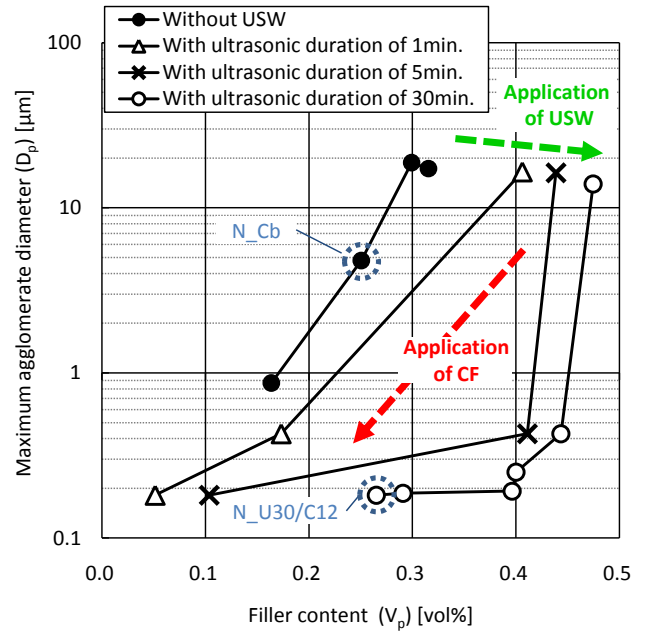


Figure 7. Relation between maximum agglomerate diameter (D_p) and filler content (V_p) of nanocomposite specimens.

4.2 PERMITTIVITY CHARACTERISTICS INFLUENCED BY COMBINATION OF ULTRASONIC WAVE AND CENTRIFUGAL FORCE

4.2.1 EFFECTS OF COMBINATION OF ULTRASONIC WAVE AND CENTRIFUGAL FORCE TO PERMITTIVITY

In order to focus on the influence of particle dispersibility on ϵ_r of nanocomposites, we compared the nanocomposite specimens (N_Cb and N_U30/C12) with different dispersibility of nanoparticles at the same filler content as shown in Figure 7. The frequency dependence of ϵ_r of the nanocomposites is shown in Figure 8 for different dispersibility of nanoparticles. From this figure, we found that ϵ_r of the nanocomposites became low with improving particle dispersibility. This result indicates that particle dispersibility can influence on ϵ_r of the nanocomposites effectively.

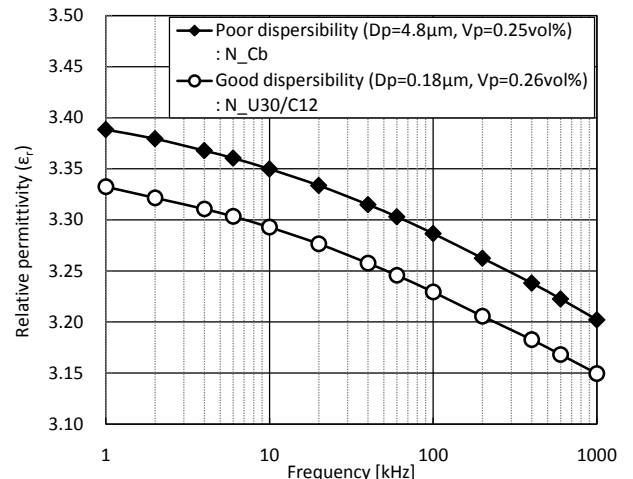


Figure 8. Frequency dependence of relative permittivity (ϵ_r) of nanocomposite specimens for different dispersibility of nanoparticles at the same filler content.

The frequency dependence of ϵ_r of nanocomposites is shown in Figure 9 for consecutive application steps in the nanoparticle composite process by combination of USW and CF. The specimen of unfilled epoxy is used for comparison with nanocomposites. The ϵ_r of the nanocomposite with neither USW nor CF (N_MM) was higher than that of unfilled epoxy. By application of USW for 30 minutes (N_U30), ϵ_r became low and close to that of unfilled epoxy. By application of CF for 12 hours after applying USW for 30 minutes (N_U30/C12) in the nanoparticle composite process by combination of USW and CF, ϵ_r became lower than that of unfilled epoxy.

By comparing ϵ_r of nanocomposites with different application duration of USW and CF in the nanoparticle composite process by combination of USW and CF, the lower ϵ_r characteristics can be discussed in detail. Figure 10 shows ϵ_r at 1MHz as a function of ultrasonic duration in the nanoparticle

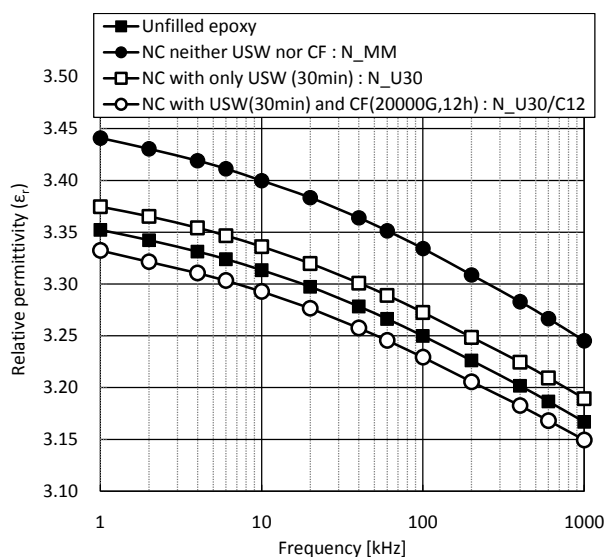


Figure 9. Frequency dependence of relative permittivity (ϵ_r) of nanocomposite specimens for different application conditions in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

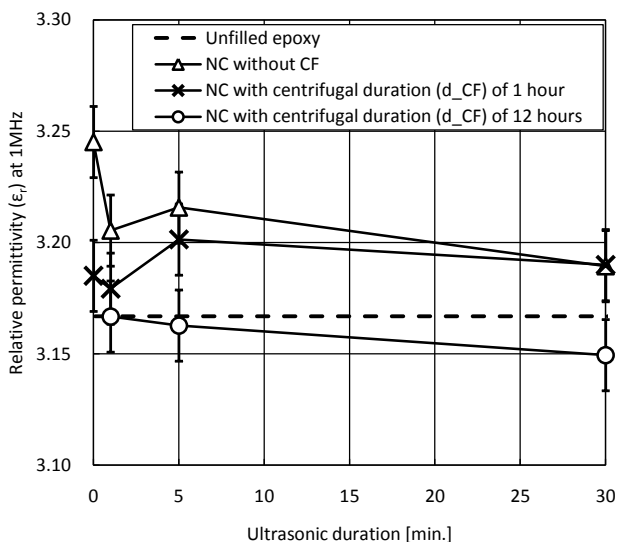


Figure 10. Relative permittivity (ϵ_r) at 1MHz as a function of ultrasonic duration (CF: 20,000G) in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

composite process by combination of USW and CF. With the increase of ultrasonic duration, ϵ_r decreased. These results could be attributed to both effects of the disruption of agglomerates and the dispersion of nanoparticles by application of USW. On the other hand, with the increase of centrifugal duration, ϵ_r decreased, which was attributed to the separation of residual agglomerates by application of CF after USW.

Figure 11 shows ϵ_r at 1MHz, V_p and D_p as a function of centrifugal duration for nanocomposites under ultrasonic duration for 30 minutes in the nanoparticle composite process by combination of USW and CF. With the increase of centrifugal duration, ϵ_r decreased and became lower than that of the unfilled epoxy material at the duration for 12 hours. In terms of V_p and D_p , it is presumed that ϵ_r decreased significantly by separating plenty of agglomerates with D_p larger than 180 nm. It could be attributed to the nanoparticle distribution of the fabricated specimens, which was estimated from V_p and D_p .

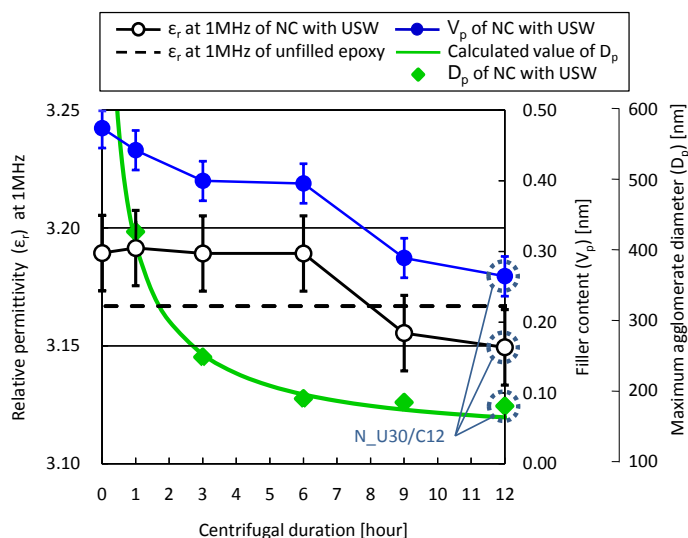


Figure 11. Relative permittivity (ϵ_r) at 1MHz, filler content (V_p) and maximum agglomerate diameter (D_p) of nanocomposite specimens under ultrasonic duration for 30 min as a function of centrifugal duration (CF: 20,000G) in the nanoparticle composite process by combination of ultrasonic wave and centrifugal force.

Figure 12 summarizes ϵ_r at 1MHz, V_p and D_p as a function of ultrasonic duration under CF conditions of 20,000 G for 12 hours in the nanoparticle composite process by combination of USW and CF. With the increase of ultrasonic duration, ϵ_r decreased and became lower than that of the unfilled epoxy material even if V_p was increased. In general, ϵ_r of epoxy composites filled with alumina material has been reported to increase with increasing filler content due to higher ϵ_r of alumina material than that of epoxy matrix [20]. Therefore, the increase of dispersed particles of $D_p = 180$ nm could bring about the lower ϵ_r characteristics of nanocomposites than that of the unfilled epoxy material. The lower ϵ_r characteristics than unfilled epoxy could be explained by the restriction of polymer movement as unique properties of a nanoparticle, e.g. by the dual layer model and multi-core model [10, 12].

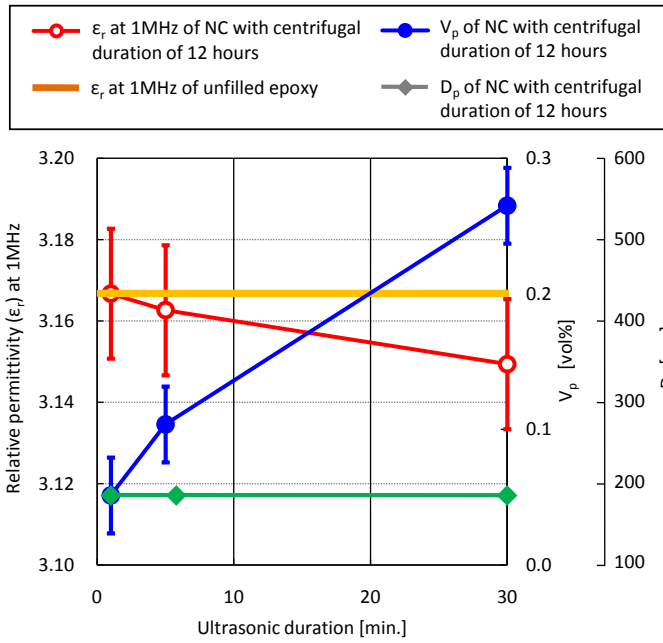


Figure 12. Relative permittivity (ϵ_r) at 1MHz, filler content (V_p) and maximum agglomerate diameter (D_p) of nanocomposites as a function of ultrasonic duration under CF conditions of 20,000G for 12 hours.

4.2.3 INFLUENCE OF PARTICLE DISPERSIBILITY IN TERMS OF FILLER VOLUME FRACTION

In general, ϵ_r of polymer composite filled with metal oxide particles with greater than nanometric size could be explained in terms of the two-media composite equation which consisted from filler volume fraction and ϵ_r of polymer and filler material such as the Lichtenecker-Rother logarithmic law of mixing and random arranged model [20]. By comparing the measured value of ϵ_r of nanocomposites with the calculated value from the random arranged model, we investigated the influence of particle dispersibility of nanoparticles on ϵ_r of polymer composites from viewpoints of filler volume fraction.

Figure 13 shows the ϵ_r at 1MHz as a function of V_p for all of the tested specimens. The ϵ_r of nanocomposites with poor dispersibility, i.e. with only CF and with neither USW nor CF, is higher than that of the calculated value. The higher ϵ_r characteristics than the calculated value are mainly attributed to the agglomerate, which could have higher permittivity than alumina material due to the inclusions, e.g. moisture. On the other hand, by application of USW and CF in the nanoparticle composite process by combination of USW and CF, ϵ_r of nanocomposites is decreased, especially to lower than the calculated value with decreasing V_p . The lower ϵ_r characteristics than the calculated value is mainly attributed to the decrease in residual agglomerates as well as the relative increase in dispersed nanoparticles. From these results, we clarified that agglomerate of alumina-nanoparticles and dispersed alumina-nanoparticles have different influences on ϵ_r of epoxy composites from alumina-particle material.

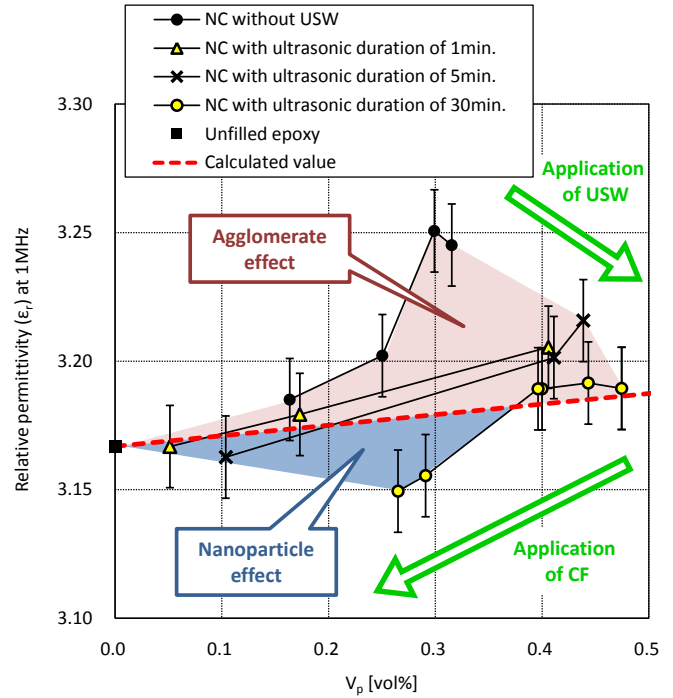


Figure 13. Filler content (V_p) dependence of relative permittivity (ϵ_r) at 1MHz.

5 CONCLUSIONS

Nanoparticle composite process by combination of ultrasonic wave (USW) and centrifugal force (CF) provided a process to efficiently disperse the nanoparticles in epoxy/alumina nanocomposites. Experimental results revealed that extension of application duration of USW and CF in the nanoparticle composite process by combination of USW and CF were effective to increase the number of the dispersed nanoparticles and as well as to separate residual agglomerates. Relative permittivity of nanocomposites processed by the nanoparticle composite process by combination of USW and CF could be lower than that of unfilled epoxy with increasing dispersed nanoparticles.

Furthermore, by controlling the application conditions of the nanoparticle composite process by combination of USW and CF, nanocomposites with various dispersibility of nanoparticles were obtained. Comparison of dielectric permittivity of the nanocomposites and calculated value of composite material revealed that higher permittivity characteristics by agglomerates and lower permittivity characteristics by dispersed nanoparticles than particle material.

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