

Mechanical dispersion of alumina powder in D.I. water and ethylene glycol

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For increasing thermoelectric properties, making core-shell structure of alumina and thermoelectric material was studied. As the first step of this study, the nano alumina particle was dispersed in D.I. water and ethylene glycol without dispersant. The concentration of alumina and other enhanced methods were adapted in water and ethylene glycol respectively. In D.I. water, the dispersion of alumina was related to the alumina concentration and the re-dispersion was also possible by ultrasonic homogenizer. However, in the case of ethylene glycol, the mean particle size wasn't changed by the enhanced dispersion methods. After the dispersion, the mean particle size was changed significantly during the short keeping time. As a whole, the mechanical dispersion of alumina in ethylene glycol was possible in a few second. And, in the experiment of dispersion stability by the addition of commercial dispersants, the slurry using D.I. water has kept under submicron for a month. However, in the case of ethylene glycol, particles were coagulated for a moment regardless of the kind of dispersion agent.

Key words: Mechanical dispersion, Nano Particles, Alumina, Solvent.

Introduction

The dispersion of alumina powder has been studied mostly in ceramic processing [1]. They are adapted in the industry of the ceramic substrate, the package, the electrical module and the high strength bodies. In recent, the study of the thermoelectric material is increasing with the increasing interest of renewable-energy. For the enhanced thermoelectric properties, Ito *et al.* [2] reported that ceramic nano particle was dispersed in β -FeSe₂ to decrease thermal conductivity due to scatter phonon. In this report, they made dispersed material by mechanical alloying.

In the same approach, if nano ceramic powder dispersed in the synthesized thermoelectric material powder phonon scattering might be possible in the sintered grain. However, the study of dispersed nano ceramic particle in thermoelectric material powder hasn't been reported till now. It is possible that the dispersion of alumina particle into grain make the core-shell structure. Some report has been in the synthesis of core-shell structure but in the thermoelectric material [3]. To make core-shell, ceramic powder was dispersed in solvent without dispersant as possible. Though so many reports have been in dispersion, the study of the only mechanical dispersion was a few [4-6].

In this study, as the first step of the making core-shell, alumina nano particle was dispersed in D.I. water and ethylene glycol by mechanical method without

dispersant. The concentrate of alumina and another enhanced method was adapted in the water and the ethylene glycol respectively. We want to confirm possibility of nano particle in these solvent without dispersant. And, also, the stability of dispersed slurry was observed by the adding of commercial dispersion agents.

Experimental

Alumina (99%, N & A Materials Co. Inc., USA) was selected in starting material. The particle size of commercial powder was 10 nm and hardly aggregated. For the dispersion solvents were D.I. water and ethylene glycol (99.7%, J.T. Baker Co. Ltd., USA). Alumina and solvent were mixed by the ratio of 1 : 1. The slurry put into jar with zirconia grinding media. Dispersion process was carried by ball milling for 24 hours crushing hard agglomerate. And then, for additional dispersion, ultrasonic homogenizer (HU H-200, Han-Tech, Korea) was used for 10 min.

To define the effect of the concentration, the concentration of alumina was changed from 50 wt.% to 0.5 wt.%. In water, the slurry was re-dispersed by ultrasonic homogenizer after coagulation for 1 week. The high energy milling by attrition mill for 5 hrs, the pre-dispersion by ethanol for 24 hrs and dispersant addition were carried for the enhanced dispersion in the case of ethylene glycol. For the observation of dispersion stability, commercial dispersion agent mixed with dispersed slurry using ultrasonic homogenizer for 10 min. BYK-111, BYK-180, BYK-184 and BYK-190 were used by dispersion agent.

To evaluate the dispersion of slurry, particle size

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distributions of all dispersed slurry were measured during short keeping time and long keeping time respectively. Particle size distribution was measured by particle size analyzer (NPA250, Microtrac Inc., USA). Powder morphology was observed by scanning electron microscopy (JSM-6700F, Jeol Ltd., Japan).

Results and Discussion

Fig. 1 shows particle size distributions of alumina powder in D.I. water with alumina concentration and re-dispersion. The mean particle size of alumina slurry of 50 wt.% was about 3 μm . With decreasing alumina concentration, mean particle size was changed to 300 nm and 20 nm at the concentration of 3 wt.% and 0.5 wt.% respectively. These result shows that the dispersion of nano particle is related to the concentration of particle and that the coagulation of nano particle is increased with increasing concentration. In the colloid, the coagulation of particles is explained by Van der Waals forces between two particles. Hamaker showed that attraction potential energy between two particles can be expressed as.

$$V_A = -\frac{A}{\pi} \int \frac{dv_1 dv_2}{x^6} \quad (1)$$

where A is Hamaker constant, v_1 and v_2 are the volume of particles respectively and x is the distance between two particles [7]. The concentration of slurry is the distance in the dispersed slurry. So, with the decrease of alumina particle concentration, coagulation rate should be also decreased. The slurry was kept for 1 week after mechanical dispersion of Al_2O_3 -D.I. water slurry with 0.5 wt.% and then, it was re-dispersed by ultrasonic homogenizer for 10 min. The particle size distribution of re-dispersed slurry was similar to that of early dispersed slurry. Though mechanical dispersed particle was coagulated and settled out the D.I. water, re-dispersion was possible by simple operation. Fig. 2 shows particle size distributions to evaluate the dispersion stability of dispersed slurry. The Al_2O_3 -D.I. water slurries were added by BYK-180 dispersant of 2 wt.% to alumina powder. As shown in Fig. 2, the mean particle size was under 100 nm for two weeks. Even until a month, main distribution of alumina particle was observed under 100 nm. In the D.I. water, we conformed that nano alumina particle was well dispersed by only mechanical dispersion and the dispersion stability of slurry also was kept for a long time.

The dispersion of alumina particle in the ethylene glycol was experimented and the particle size was shown in Fig. 3. In this figure, four different conditions were displayed, such as as-dispersed like D.I. water, high energy milling using attrition mill for 5 hrs, pre-dispersed in ethanol and the addition of dispersion agent. The mean particle sizes of these are similar from 2 μm to 4 μm respectively. They are also the same

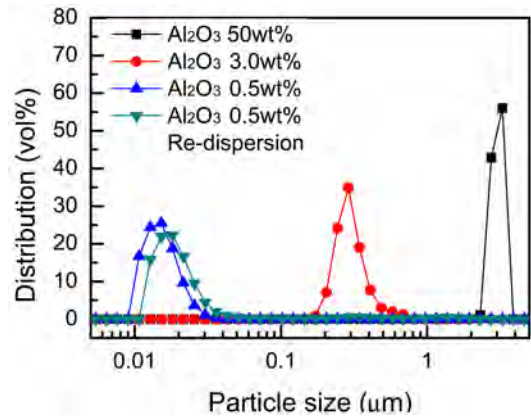


Fig. 1. Particle size distribution of Al_2O_3 -D. I. water slurries with alumina concentration and re-dispersion after 1 week.

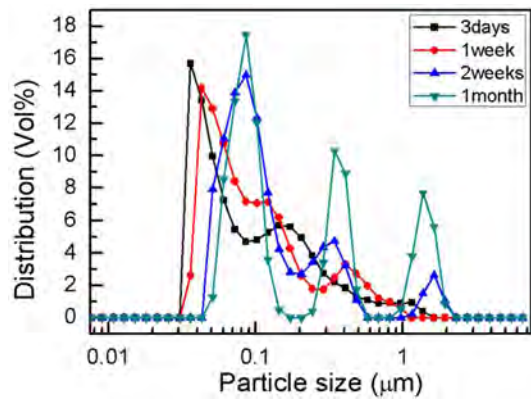


Fig. 2. Dispersion stability of Al_2O_3 slurries added by 2 wt.% of BYK-180.

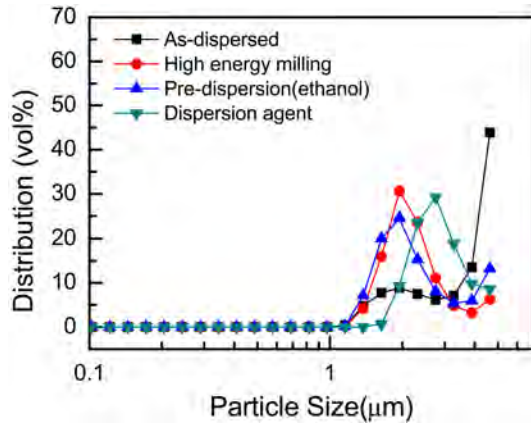


Fig. 3. Particle size distribution of Al_2O_3 -ethylene glycol slurries with various dispersion methods.

tendency with changing Al_2O_3 concentration. As a whole, particle size distribution of alumina particle wasn't changed in ethylene glycol. Though 4 methods which we used were enough to crush aggregate particle size distributions were not any more decreased. It was thought that particle was coagulated in ethylene glycol in a second.

The changes of particle size distribution in ethylene glycol were shown in Fig. 4 with short keeping time.

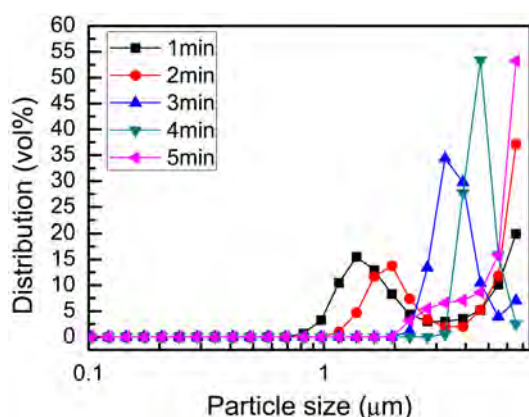


Fig. 4. Particle size distribution of Al_2O_3 -ethylene glycol slurries with keeping time after mechanical dispersion.

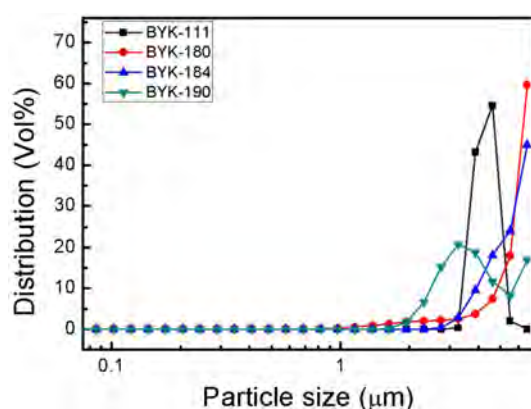


Fig. 5. Particle size distribution of dispersed slurries with the kinds of dispersion agent in ethylene glycol.

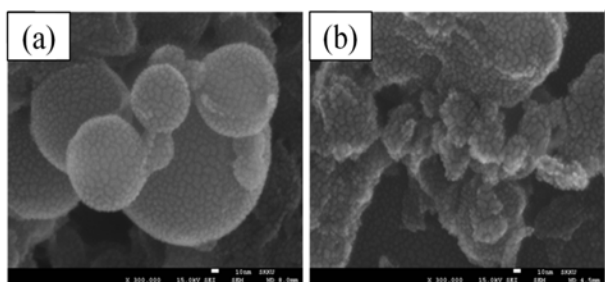


Fig. 6. SEM images of dispersed alumina particle in (a) D. I. water and (b) ethylene glycol.

From the result of Fig. 3, we supposed that the alumina particle was dispersed by mechanical treatment and it was coagulated in a second. Therefore, the particle size distribution of slurry might be changed in the short keeping time. When it was changed from 1 to 5 min. after dispersing by ultrasonic homogenizer, mean particle size was changed from 1 μm to 5 μm dramatically. It was thought that the mechanical dispersion of alumina powder in ethylene glycol was possible in a second and that the dispersion stability of nano alumina should be improved. Fig. 5 shows particle size

distributions of dispersed slurries with addition of four different dispersion agents. The solvent was ethylene glycol and the amount of commercial dispersion agent was 2 wt.% to alumina powder. As the Fig. 3, particle size distribution was also similar to previous data from 2 μm to 4 μm respectively. Therefore, in ethylene glycol, we conformed that dispersion of nano alumina particle was not improved by dispersion agent.

Fig. 6 is the morphologies of the dispersed alumina powder in water and ethylene glycol. In this figure, the primary particle size was similar with about 10 nm, however, secondly particles was different shape and size. In the case of D.I. water, the shape of secondly particle was spherical. It was thought that slurry made droplet during dry due to high surface tension of water. In the other side dispersed in ethylene glycol, the shape and the size of secondly particle were random, though the same drying process.

Conclusions

Mechanical dispersion of alumina powder was observed in D.I. water and ethylene glycol as solvent. In the D.I. water, dispersion of alumina was related to alumina concentration and re-dispersion was also possible by ultrasonic homogenizer. However, in the case of ethylene glycol, the mean particle size wasn't changed by concentration and the enhanced dispersion methods weren't effective in dispersion. After the dispersion, the mean particle size was changed significantly during the short keeping time. As a whole, the mechanical dispersion of alumina in ethylene glycol was possible in a second however, the stability wasn't sufficient in a long time. In the dispersion stability, also D.I. water was useful solvent for nano alumina particle but ethylene glycol was not stable even for a few minute.

References

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