

Dispersion of TiO2 Nanoparticles by using Focused Ultrasound

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1. Introduction

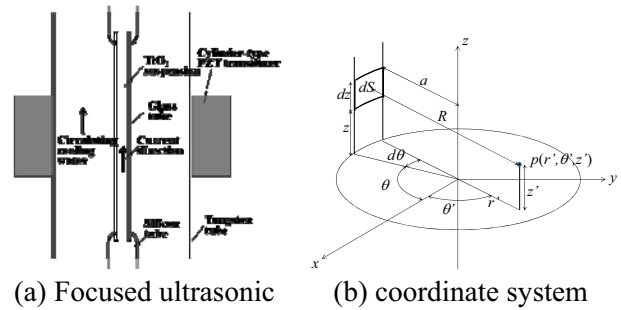
Nano sized particles of ceramics or metal are expected to be a functional material of the next generation in various fields because their characteristics that are different from those of micro sized particles. The examples of the application of nano materials are photocatalyst, fuel cell, gas sensor, and fine electric circuit¹⁾. However, nanomaterials usually contain a large amount of aggregation or agglomeration. Therefore, in the application fields, the aggregation or the agglomeration causes serious problems and limitation. There have been several dispersion methods using ultrasonic power for nanoparticles such as the ultrasonic homogenizer and ultrasonic water bath^{2,3)}. We have reported a non-contact type dispersion method using focused ultrasound in order to have pure nano particles suspension without any foreign substance⁴⁾. However, the design principle and the driving method to optimize the dispersion system have not been sufficiently investigated. In this study, to optimize the system, we extend our investigation to cover the calculation of sound field and the dispersion effect depending on ultrasonic exposure time. For the nano particle, we used titanium dioxide (TiO2) particle.

2. Construction of the dispersion system

The construction of the focused ultrasound system is shown in Fig. 1(a). Ultrasonic field is focused in a glass tube, which is fixed at the center of a cylindrical transducer. The cylindrical transducer is 38.5 mm in inner radius, 46.5 mm in outer radius, and 19.2 mm in height. To prevent the piezoelectric transducer from heating, a tungsten tube filled with cooling water was inserted and circulated by a water pump in the inner space of the cylindrical transducer. The ultrasonic field from the transducer can transferred to the glass tube by the cooling water. The thickness of the glass tube and the tungsten tube is selected as thin as possible so as not to affect the ultrasound from the transducer. The DI water is circulated through a silicon tube that is connecting the glass tube to the water pump.

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The piezoelectric transducer has a resonant frequency of 495 kHz, and it is driven by the frequency. The suspension of TiO2 and the cooling water are circulated at flow rates of 3.0 and 2.0 mL/s, respectively. The thickness of the glass tube and the tungsten tube are 0.3, and 0.5 mm, respectively.



(a) Focused ultrasonic dispersion system (b) coordinate system

Fig. 1 Schematic of focused ultrasonic system.

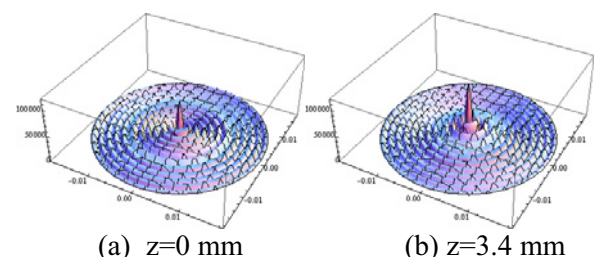
To decide the optimum size of the glass tube, the ultrasonic field should be calculated. The coordinate system to calculate the field inside the cylindrical vibrator is shown in Fig. 1(b). In a point inside the cylindrical vibrator, the acoustic field from the vibrator with radius a is given by

$$p(r', \theta', z') = \int_{-h/2}^{h/2} \int_0^{2\pi} \frac{qe^{-ikR}}{R} ad\theta dz, \tag{1}$$

Here,

$$R = \sqrt{(a \cos \theta - r' \cos \theta')^2 + (a \sin \theta - r' \sin \theta')^2 + (z - z')^2}$$

, h is the height and q the source strength of the vibrator. In Eq. (1), k is the wave length of the ultrasound. The acoustic field distributions inside the cylinder are calculated using Eq. (1) as shown in Fig. 2. In the calculations, the source strength was $1 \text{ m}^3/\text{s}$.



(a) z=0 mm (b) z=3.4 mm
 Fig. 2 Acoustic pressure inside cylindrical vibrator.

This result shows that the acoustic field distribution has z-axis symmetry. The acoustic pressure along z-axis has a tendency to decrease as it is far from the center ($z=0$). However, as shown in Fig. 2(b), the maximum pressure does not appear in the center. To confirm the acoustic field profile along the z-axis, the acoustic pressure on the z-axis is calculated as shown in Fig. 3(a). In these results, we can see that the pressure increases by 3.4 mm from the center and then decreases along the center axis of the cylinder. This tendency can also be confirmed in Fig. 2. This calculation results that over than 20 mm length glass tube is enough to be used in this dispersion system. In this study, the length of glass tube was chosen as 80 mm. To decide the optimum inner diameter of the glass tube, the pressure profile along the y-direction is calculated as shown in Fig. 3(b). The result shows that 50% of peak pressure is distributed within 1 mm from the center. According with these results, 2 mm was chosen for the inner diameter of the glass tube.

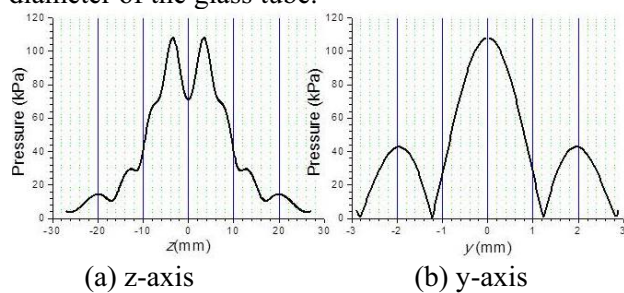


Fig. 3 Acoustic field profile

3. Particle distribution

The nanoparticle size distribution was measured by Scanning Mobility Particles Sizer (SMPS, TSI 3080). The measurement range of particle size is from 9.31 to 437.1 nm. In order to investigate the dispersion effect with focused ultrasound depending on the exposure time, the nanoparticle distribution of the TiO_2 suspension without any dispersing agent was measured as shown in Fig. 4. The particle size distribution before the dispersion shows low number concentration as shown in Fig. 4(a) because most of the nanoparticles are agglomerated into bigger size than given range. After 1 hour exposure to ultrasound, there are still a large number of agglomerated nanoparticles around 100 nm although the main peak appears at around 40~50 nm, as shown in Fig. 4(b). As the exposure time elapse, the dispersion effect improve because the main peak in the range of 40~50 nm becomes sharp and the number concentration of the peak increases as shown in Fig. 4(c). However, in Fig. 4(d), the main peak value decreased and the number concentration in the range of 20~30 nm increased.

This shows a tendency that particles are broken into smaller particles. Even though the dispersion effect increased in the range of 100~200 nm for 50 hr ultrasound exposure time, this result is not desirable for obtaining uniform sized nanoparticle. There is an optimum exposure time to disperse TiO_2 nanoparticle with focused ultrasound, and 10 hr was the optimum exposure time in this study.

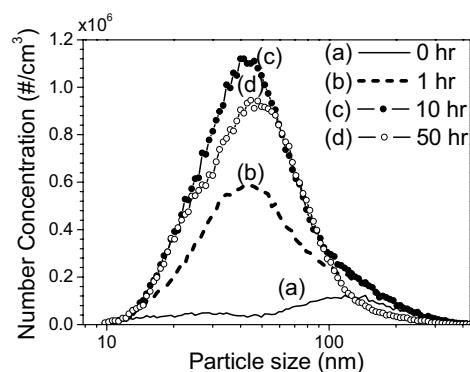


Fig. 4 Dispersion results by focused ultrasound for different exposure time.

4. Summary

A non-contact dispersion method is suggested for TiO_2 nano particles using focused ultrasound from a cylindrical piezoelectric vibrator. To keep the purity of the suspension, the ultrasound energy from the vibrator transfers into a glass tube filled with the nano suspension through cooling water. The line-focused ultrasound in the glass tube disperses the TiO_2 nano particles effectively without any dispersing agent. The dispersion effect was examined depending on the ultrasound driving time with particle size analyzers. From our results, we found that an optimum ultrasound exposure time was 10 hr for TiO_2 nanoparticle.

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