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Experimental analysis of sample preparation conditions for particle size measurement

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Abstract

The influence of deagglomeration on an agglomerated sample, the dispersion media for a non-oxide sample, and the measuring conditions for a mixed sample on the particle size distribution are investigated in the present paper. It is found that deagglomeration is effective for dispersing an agglomerated powder well, regardless of the type and output power of the ultrasonic disrupters used. Ethanol is a useful dispersion medium for achieving stable measurements of non-oxide samples. In the measurement of a mixed sample, the measured distributions by X-ray sedimentation do not coincide with the distribution calculated from the weight ratio for each original sample. However, they coincide with the modified distribution from the X-ray absorption ratio for each original sample, because the cumulative mass % in this measuring principle is determined on the basis of the relationship between the X-ray transmittance ratio and the suspension concentration, depending on the sample materials.

Keywords: Particle size measurement; Sample preparation conditions; Deagglomeration; Non-oxide; Mixed sample

1. Introduction

Particle size analyzers based on many kinds of measuring principles have been developed for the measurement of fine particles. The particle size distribution measured by this commercial equipment does not always agree, even if the same sample is measured. The cause of the discrepancy is attributed to the following two reasons. One is the difference in the sample preparation conditions and the other is the difference in models and measuring principles.

For comparison of the particle size distribution measured by different equipment, some studies have been conducted [1–8]. The authors have also carried out round robin tests to elucidate the data scatter among different models and measuring principles [9–12].

However, there have been few studies conducted on the method of sample preparation for particle size analysis although there are many technical problems to be solved. For example, it has been pointed out that the deagglomeration conditions for agglomerated powders and the dispersion media for non-oxides should be investigated. Furthermore the conditions for analyzing the samples using various measuring principles have not been systematically made clear for mixed samples.

To answer some of these questions, this paper sets out suitable methods of sample preparation and measuring conditions for an agglomerated sample, a non-oxide and for mixed samples for experimental examination.

2. Experimental

2.1. Samples

The scanning electron micrographs are shown in Fig. 1a–c. Titanium dioxide in the crystalline form of rutile made by the chloride process [13] was used as the agglomerated powder. Aluminum nitride, having high reactivity to water, was selected as the non-oxide [14]. This was made by the direct nitridation of aluminum [15]. For the mixed sample, titanium dioxide, as used before, and barium titanate were used. Each sample was mixed in a 1:1 ratio by weight in a dispersion medium. Barium titanate was made by a solid phase reaction of barium carbonate and titanium dioxide [16], as used in the previous studies [9,11].

The sample properties are shown in Table 1. The true density was measured by a helium gas pycnometer method

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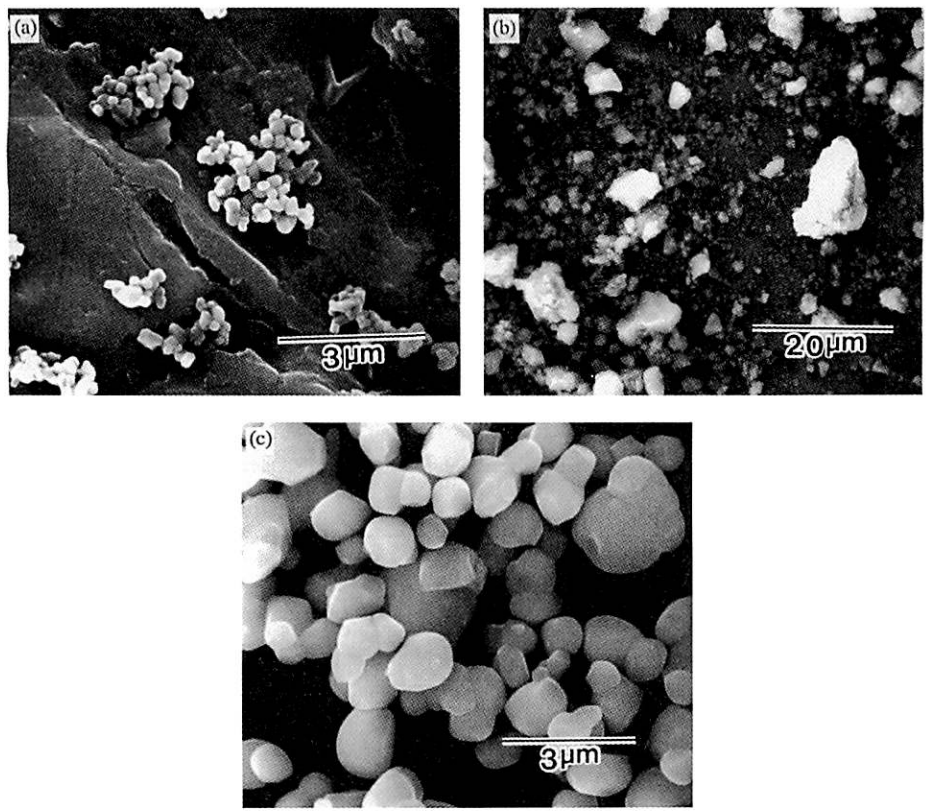

Fig. 1. Scanning electron micrographs: (a) titanium dioxide; (b) aluminum nitride, (c) barium titanate.

Table 1
Sample properties

Samples	True density (g/cm^3)	Specific surface area (m^2/g)	Refractive index[a] (−)
Titanium dioxide	4.21	6.9	2.71 [17]
Aluminum nitride	3.22	3.7	1.8 <[b]
Barium titanate	5.97	1.2	2.40 [18]

[a]Literature value.
[b]Measured value.

(Auto Pycnometer 1320, Micromeritics Instrument). The samples were heated at 110°C for 2 h and cooled down in a desiccator to room temperature. After that, the density was measured. The specific surface area was measured by the BET gas adsorption method (Quantasorb, Quantachrome). The powder was degassed at 200°C for 2 h. The refractive index was mainly taken from the literature [17,18]. Only the refractive index of aluminum nitride was measured by the liquid immersion method (RIMS ($\lambda = 588$ nm), Kyoto fission track) because the data could not be found in the literature.

2.2. Equipment

The particle size analyzers used for this investigation were the SediGraph 5100:Micromeritics for the X-ray sedimentation method, the SA-CP3:Shimadzu, CAPA-700:Horiba for the photo-sedimentation method; the MasterSizer:Malvern, SALD-2000:Shimadzu, LA-700:Horiba for the laser diffraction and scattering method.

2.3. General sample preparation conditions

Fig. 2 shows the general procedure for particle size measurement in a liquid. The conditions to be considered in the measurement are also listed in this figure. The agglomerated samples were stabilized by deagglomeration since the ultrasonification effects depend on the dispersibility of the samples. For the non-oxide, if water is used as the dispersion medium, the bubbles formed by the reaction with the water or particle dissolution will affect the data scatter of the measured results. For the mixed sample, the measured results will vary with the physical properties of the particles and therefore the size distribution will vary according to the measuring principles used. This is discussed in Section 3 in detail.

Other conditions were determined for each sample as follows. For the dispersion in water system, commercially available distilled water (Wako, analytical grade) was used. The dispersant agent and its proper concentration were selected by the measurement of the zeta potential of the sample suspension [9,10,19]. The zeta potential was measured by electrophoresis (Lazer Zee Meter Model 501, Pen Kem). For the dispersion of each sample, an ultrasonic homogenizer (Nissei model us-300: 300 W) and ultrasonic baths (Sharp UT-604: 600 and 300 W, and Honda W103T: 40 W) were used. Regardless of disrupters and output power used, the disper-

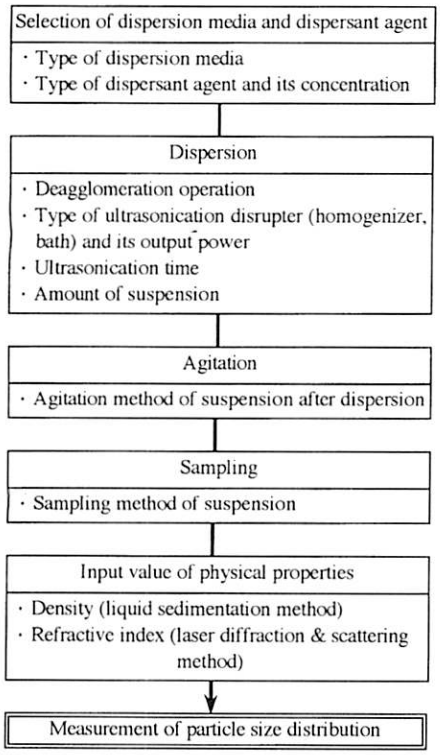

Fig. 2. The main factors in sample preparation conditions which influence the results of particle size measurement.

sion time over which the size distribution becomes the same was determined for each disrupter. The suspension volume was kept at 200 cm^3 in a 200 cm^3 beaker. For treatment of the dispersed suspension, the suspension was continuously stirred by a magnetic stirrer to prevent reagglomeration due to the settling of the particles. For sampling, the suspension was sampled quickly with a pipette from an arbitrary position in the stirred suspension. The true density or the refractive index for each sample was used for the inputted value of the physical properties.

2.4. Deagglomeration for the agglomerated sample

The measured distribution of the agglomerated sample depends on the type and output power of ultrasonic disrupter used. It is said that data scatter due to the difference in ultrasonication is reduced by deagglomeration of the sample with a mortar and pestle. However, this has not been systematically investigated before, so was investigated in this study.

The deagglomeration experiment is done by the following procedure: (i) 2 g of the sample is put into a mortar; (ii) It is strongly rubbed with the pestle, until most of the powder adheres onto the inner wall of the mortar; (iii) It is scraped off and collected to the bottom of the mortar. This procedure is defined as one deagglomeration operation. Operations (ii) and (iii) are repeated as required. The specific surface area was measured before and after deagglomeration. After the suspension was prepared with different types and output powers of ultrasonic disrupters according to the procedure of Section 2.3, the particle size distribution was measured by X-ray sedimentation; laser diffraction and scattering; and photo-sedimentation.

2.5. Dispersion media for the non-oxide sample

The effect of the dispersion media on the measured results of the non-oxide sample was investigated. Ethanol (Nacalai Tesque, analytical grade) and sodium hexametaphosphate, 0.025 wt.% in distilled water, were used as the dispersion media. The size distribution was measured by X-ray sedimentation because this method can detect the bubbles produced by the reaction of water and sample more sensitively compared to the other measuring principles.

2.6. Physical properties of the particles in the mixed sample

For X-ray sedimentation and photo-sedimentation, the particle settling velocity depends on the given particle density. For laser diffraction and scattering, the distribution is influenced by the given particle refractive index. Therefore, the effect of these values on the results for the mixed sample was also investigated.

3. Results and discussion

3.1. Deagglomeration operation for the agglomerated sample

Table 2 shows the change in particle size and specific surface area with repeated deagglomeration operations. The specific surface areas are almost constant, regardless of the number of deagglomeration operations which means that the size reduction of primary particles does not occur by deagglomeration. The 95% diameter gradually decreases for the first five deagglomeration operations and then becomes more or less constant, the 50% diameter remains almost constant after three deagglomeration operations.

Fig. 3 shows the effect of the deagglomeration operations on the dispersibility of the agglomerated powder. The size distribution of the deagglomerated sample is finer than that without the deagglomeration operation. The measured distri-

Table 2
Effect of the number of deagglomerations on particle size and specific surface area

Number of deagglomerations	50% diameter[a] (μm)	95% diameter[a] (μm)	Specific surface area (m^2/g)
0	0.38	1.16	6.9
3	0.35	0.82	7.0
5	0.35	0.72	6.9
10	0.35	0.74	6.9

[a]Ultrasonic was applied 10 min by homogenizer.

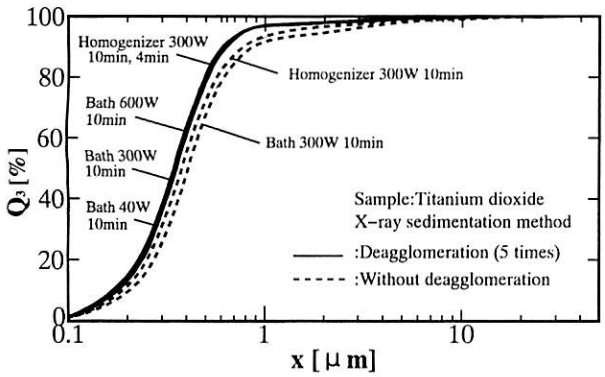

Fig. 3. Change of the dispersibility for titanium dioxide with the deagglomeration operation.

bution without the deagglomeration operation depends on the type of ultrasonic disrupters although the same ultrasonication time of 10 min is applied. Besides, the measured distribution with the deagglomerated sample hardly ever changes with the type and the output power of the disrupters. Particularly, in the case where the homogenizer is used, the measured distributions coincide with each other regardless of the different ultrasonication time. This means that the deagglomeration operation is effective in stabilizing the dispersion effect of the agglomerated sample.

Fig. 4 shows the change in 10%, 50% and 90% diameters measured by three methods before and after the deagglomeration operation. The discrepancy among the methods for the 50% and 90% diameter decreases with the deagglomeration operation. As shown in Fig. 1a, this sample consists of agglomerates formed by submicron primary particles. The large agglomerates not fully dispersed into primary particles affect the difference in data among obtained from the different measuring techniques [10,12]. In this experiment, since some portions of the agglomerates are deagglomerated over the more coarse size range, it is supposed that the results obtained by different measuring principles are close. It is found that the deagglomeration operation is effective for

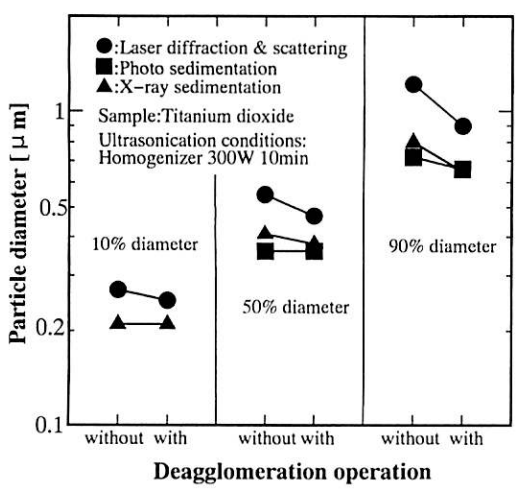

Fig. 4. The effect of deagglomeration operation on the scattering among measured results with three kinds of measuring principles.

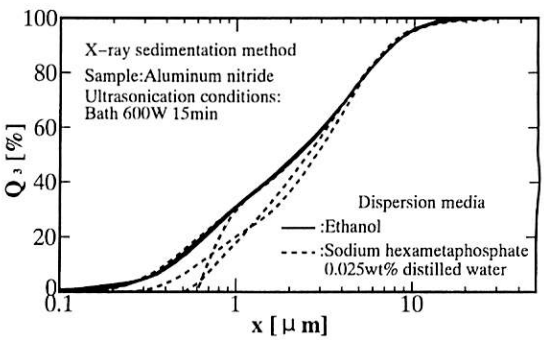

Fig. 5. Reproducibility of particle size measurement for aluminum nitride by using ethanol and distilled water as dispersion media.

decreasing this difference in data from the measuring principles.

The deagglomeration operation proposed in this experiment was also applied to another agglomerated powder, silicon nitride and silicon carbide [10]. The results from this confirmed that the deagglomeration operation is effective for decreasing the data scatter due to the difference of the ultrasonication effect.

3.2. Dispersion media for the non-oxides

Fig. 5 shows the four results for each dispersion media. In the case where water is used, the reproducibility is poor in the fine range of the particle size distribution. Some results obtained with distilled water do not approach 0% gradually. These phenomena show that bubbles are produced by the reaction between the sample and water [20]. In the case where ethanol is used, good reproducibility is obtained as shown in solid lines.

3.3. Physical properties of the particles in the mixed sample

3.3.1. X-ray sedimentation method

Fig. 6 shows the particle size distribution of the mixed sample measured by X-ray sedimentation. The results, which are obtained by using the true density of titanium dioxide, barium titanate and the calculated density of the mixed sample ($4.94\ g/cm^3$), are indicated as solid lines numbered 1, 2 and 3, respectively. The measured distribution of each original

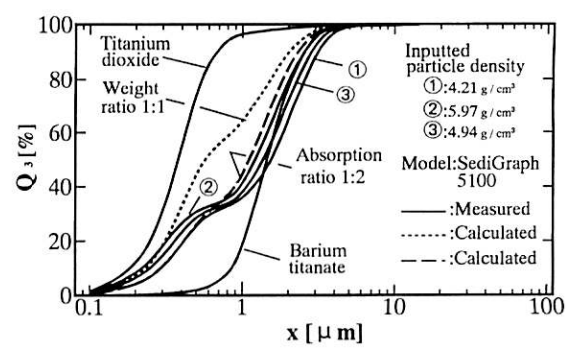

Fig. 6. Particle size distributions of mixed sample measured by SediGraph 5100 based on the X-ray sedimentation method.

sample is also shown in this figure. The dotted line is the calculated distribution from the weight ratio 1:1 (the volume ratio 6:4 corresponds to titanium dioxide and barium titanate). The broken line is the calculated distribution from the X-ray absorption ratio of the each original sample. The absorption ratio 1:2 was obtained as follows. Fig. 7 shows the relationship between the transmittance ratio of X-ray and the suspension concentration for mixed and each original sample. The absorption ratio which is expressed by (100—transmittance ratio) increases with the suspension concentration. At the same suspension concentration, the absorption ratio of barium titanate is twice that of titanium dioxide as shown in Fig. 7. For the suspension preparation of the mixed sample, each 0.5 g original sample was put into 100 cm^3 dispersion media, and an approximate 1 wt.% suspension was prepared. Since the absorption ratio of this 1 wt.% mixed sample suspension coincides with the sum of the absorption ratios for each 0.5 wt.% original sample suspension, the absorption ratio of each original sample in the 1 wt.% mixed sample suspension is 1 (titanium dioxide):2 (barium titanate).

The measured distributions of the mixed sample are little affected by the different particle densities, as shown in Fig. 6 and do not coincide with the calculated distribution from the weight ratio shown by the dotted line. However, they do coincide with the modified distribution from the absorption ratio shown by the broken line. This means that the cumulative mass % of X-ray sedimentation is determined on the basis of the relationship between the X-ray transmittance ratios and the suspension concentration depending on the sample materials. In the X-ray sedimentation method, this relationship is independent of particle size and refractive index of the sample.

However, because the particle density for each sample used in this experiment is relatively close, the measured distributions of the mixed sample are little affected by the inputted particle density.

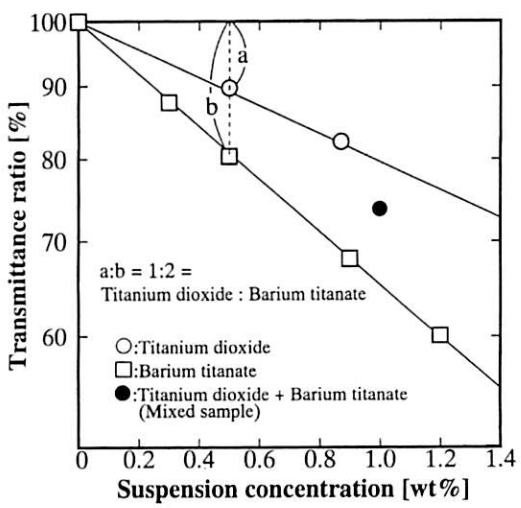

Fig. 7. Relationship between transmittance ratio of X-ray and suspension concentration for mixed and each original sample (X-ray sedimentation method).

3.3.2. Photo-sedimentation method

Fig. 8 shows the relationship between the transmittance ratio of light and the suspension concentration for the mixed and each original sample measured by the photo-sedimentation method (SA-CP3). It shows that the absorption ratio of the light for titanium dioxide is eight times larger than that for barium titanate at the same suspension concentration. The absorption ratio of 180 g/m^3 mixed sample suspension coincides with the sum of the absorption ratios for each 90 g/m^3 original sample suspension. Therefore, the absorption ratio of each original sample in the 180 g/m^3 mixed sample suspension is 1 (barium titanate):8 (titanium dioxide).

Figs. 9 and 10 show the size distributions of the mixed sample measured with two different instruments. The measured distributions significantly differ with each model and do not coincide with the distribution calculated from the weight ratio. With CAPA-700 as shown in Fig. 10, the measured distributions coincide with the modified distribution from the light absorption ratio. However, with the SA-CP3 as shown in Fig. 9, this is not observed. The effect of the particle density on the variation of the measured distribution is not observed as well as the result of X-ray sedimentation method.

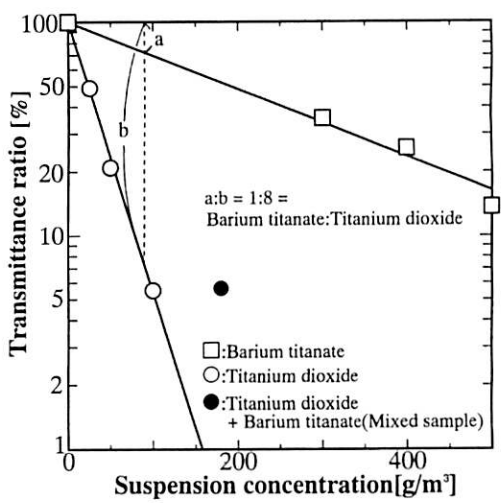

Fig. 8. Relationship between transmittance ratio of light and suspension concentration for mixed and each original sample (photo-sedimentation method).

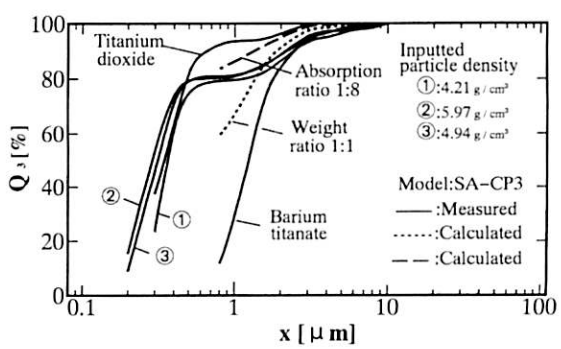

Fig. 9. Particle size distributions of mixed sample measured by SA-CP3 based on photo-sedimentation method.

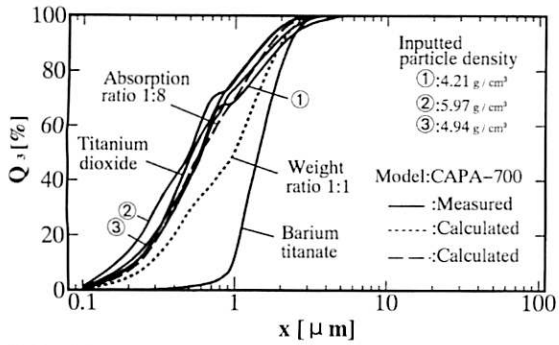

Fig. 10. Particle size distributions of mixed sample measured by CAPA-700 based on photo-sedimentation method.

The CAPA-700 does not make a correction for extinction coefficient correction [11,12,21,22] whereas the SA-CP3 does make this and it supposed that this is the reason for this difference.

3.3.3. Laser diffraction and scattering method

Fig. 11 shows the relationship between the transmittance ratio of light and the suspension concentration for mixed and each original sample measured by laser diffraction and scattering method (MasterSizer). A similar result to that shown in Fig. 8 is obtained.

Figs. 12–14 show the size distributions of a mixed sample measured by three different models. With the MasterSizer as shown in Fig. 12, the measured distributions do not coincide with the modified distribution from the absorption ratio, although they approach the distribution calculated from the weight ratio. The SALD-2000 and LA-700, the opposite tendency is observed as shown in Figs. 13 and 14.

In the laser diffraction and scattering method, the scattered light intensity is detected which is different from the method of detecting the transmitted light. The results measured by this method are also affected by the configurations of the detectors and arithmetic algorithms that differ with the manufacturer. Therefore, it is difficult to explain the difference in the results shown in Figs. 12–14.

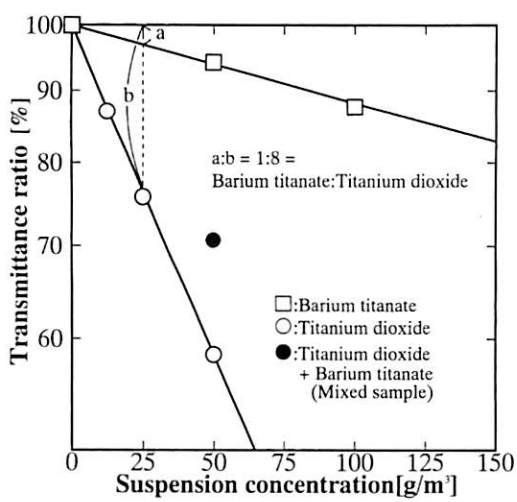

Fig. 11. Relationship between transmittance ratio of light and suspension concentration for mixed and each original sample (laser diffraction and scattering method).

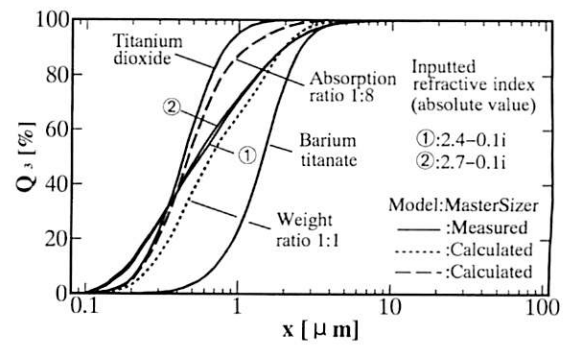

Fig. 12. Particle size distributions of mixed sample measured by MasterSizer based on laser diffraction and scattering method.

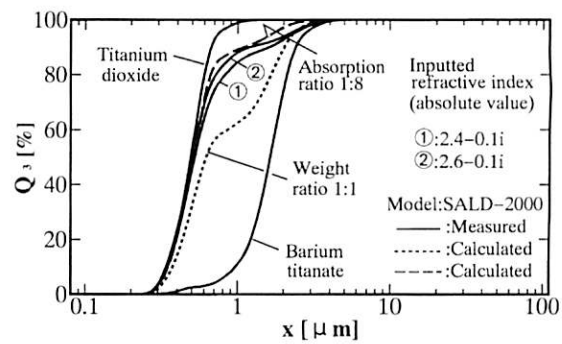

Fig. 13. Particle size distributions of mixed sample measured by SALD-2000 based on laser diffraction and scattering method.

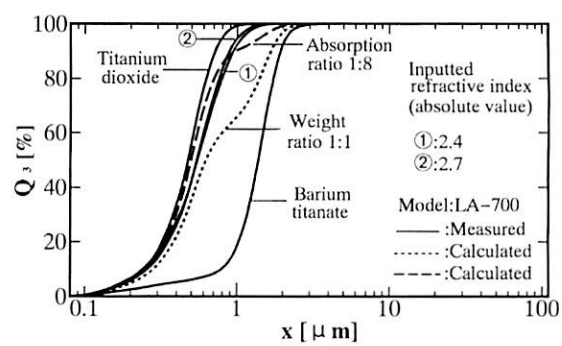

Fig. 14. Particle size distributions of mixed sample measured by LA-700 based on laser diffraction and scattering method.

The measured distributions of the mixed sample hardly change with the refractive index for each original sample, because the particle refractive index for each sample is relatively close.

4. Conclusions

(1) The deagglomeration operation with a pestle and mortar before ultrasonication is effective for dispersing the agglomerated powder well. In the case when deagglomerated powder is used, the measured distribution is finer than that without the deagglomeration, and it does not change with type and output power of the ultrasonic disrupters. In addi-

Table 3
The main sample preparation and measuring conditions determined for round robin test

Samples	Dispersion medium	Deagglomeration operation with a mortar and pestle	Ultrasonication time (min)	Given value of physical properties	
				Density (g/cm^3)	Refractive index (−)
Titanium dioxide	Sodium hexametaphosphate 0.1 wt.% distilled water	5 times	4 (homogenizer) 10 (bath)	4.21	2.71
Aluminum nitride	Ethanol	Without	10 (homogenizer) 15 (bath)	3.22	1.8<
Mixture of titanium dioxide and barium titanate	Sodium hexametaphosphate 0.1 wt.% distilled water	5 times (without for barium titanate)	4 (homogenizer) 10 (bath)	4.94	2.71 and 2.40

Table 4
Particle size analyzers used for round robin test

Principle	Model	Manufacturer
Laser diffraction and scattering	Helos	Sympa Tec
	MasterSizer	Malvern
	LA-700, 500	Horiba
	Microtrac FRA, SPA	Leeds and Northrup
	HR 850B	Cilas
	SALD-2000	Shimadzu
	LMS-24	Seishin
Photo-sedimentation	CAPA-700	Horiba
	SA-CP4L	
	SA-CP3L	
	SA-CP3	Shimadzu
	SA-CP2	
X-ray sedimentation	SediGraph 5100	Micromeritics
	SediGraph 5000D	
	BI-XDC	Brookhaven
Light obscuration	CIS-1	Galai
Electrical sensing zone	Multisizer II	Coulter

tion, the deagglomeration operation seems to decrease the difference in data among the measuring techniques.

(2) For the measurement of aluminum nitride, ethanol is a useful dispersion medium achieving good reproducibility of the measured results.

(3) In the measurement of a mixed sample with three different methods, the measured distributions do not coincide with the distribution calculated from the weight ratio for each original sample. However, as for the X-ray sedimentation method, the measured distributions coincide reasonably with the modified distribution from the X-ray absorption ratio for each original sample because the cumulative mass % in this method is determined on the basis of the relationship between the X-ray transmittance ratio and the suspension concentration depending on the sample materials.

5. Information of the round robin test results based on this paper

By knowing the conditions of the sample preparation used in this paper, a round robin test was conducted to evaluate the data scatter among different models and measuring techniques. The samples used in this test are the same in this paper. The main sample preparation and measuring conditions for the round robin test are shown in Table 3. These conditions are summarized as common instructions for each sample. The instructions and samples were distributed to 33 organizations and all measurements were carried out under the same sample preparation conditions. All results measured by these organizations were represented by the average 10%, 50% and 90% diameters, and the data scattering of their diameters was evaluated by the variation coefficient. The particle size analyzers used in the round robin test are shown in Tables 4. The results are listed in Tables 5–7.

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The authors gratefully acknowledge all participants who joined the round robin test for their great cooperation. And we also wish to thank the members of the advisory committee of this project for their beneficial support.

Table 5
Results of round robin test for titanium dioxide

Principle	10% diameter	50% diameter	90% diameter
Laser diffraction and scattering	0.26	0.42	0.76
	18.7	12.2	26.8
Photo sedimentation	0.22	0.39	0.93
	7.6	14.0	30.6
X-ray sedimentation	0.22	0.38	0.66
	7.0	7.6	5.8
maximum average diameter/minimum average diameter (−)	1.18	1.11	1.41

Upper: average diameter (μm), lower: variation coefficient (%).

Table 6
Results of round robin test for aluminum nitride

Principle	10% diameter	50% diameter	90% diameter
Laser diffraction and scattering	0.47	2.17	7.80
	23.9	25.5	22.9
Photo-sedimentation	0.39	1.49	11.5
	20.1	32.7	61.7
X-ray sedimentation	0.52	2.26	7.23
	13.7	8.6	7.5
maximum average diameter/minimum average diameter (−)	1.33	1.52	1.59

Upper: average diameter (μm), lower: variation coefficient (%).

Table 7
Results of round robin test for mixed sample of titanium dioxide and barium titanate

Principle	10% diameter	50% diameter	90% diameter
Laser diffraction and scattering	0.29	0.59	1.64
	23.2	41.2	47.8
Photo-sedimentation	0.21	0.37	2.10
	8.45	25.8	19.9
X-ray sedimentation	0.26	1.76	3.03
	23.8	28.1	8.5
maximum average diameter/minimum average diameter (−)	1.38	4.76	1.85

Upper: average diameter (μm), lower: variation coefficient (%).

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