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Original Paper

Particle size measurement of standard reference particle candidates and theoretical estimation of uncertainty region

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ABSTRACT

In order to confirm reliable particle size measurement technique and to prepare standard reference particles for calibrating particle size measurement devices, experimental and theoretical studies have been conducted about particle size measurement of 0.1–1 μm silica particles. The microscopic method with sample size greater than 90,000 particles was conducted for the size measurement.

Theoretical equation of uncertainty region over all particle diameter range is newly proposed and compared with computer simulation. Previous paper [H. Masuda, K. Iinoya, Theoretical study of the scatter of experimental data due to particle size distribution, J. Chem. Eng. 4(1) (1971) 60–67] reported the uncertainty region only for mass median diameter, but this paper presents the uncertainty region for all particle size range. The uncertainty region increases with the increase in particle diameter and also increases as the sample size decreases. Theoretical uncertainty region agreed with the results of computer simulation.
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1. Introduction

Particle size distribution is measured by various methods such as microscopy method, laser diffraction and scattering method, dynamic light scattering method, electrical sensing zone method and liquid sedimentation method. Though the laser diffraction and scattering method, dynamic light scattering method and electrical sensing zone method have the advantage of shorter measurement time and good repeatability, but they need complicated calibration by direct method. In order to calibrate particle size measurement devices, it is necessary to prepare standard reference particles. For the reference particles, mono-disperse and poly-disperse particles are proposed. For the poly-disperse reference particles, Yoshida et al. measured particle size distribution of three kinds of spherical glass beads by use of improved type sedimentation balance and microscopic methods with sample size greater than 10,000 particles [1,2]. Mori et al. reported the results of the round robin test for the two kinds of particles (MBP1-10, 10-100) [3]. This paper discusses the estimation method of uncertainty region for particle size distribution due to limited particle count number.

In order to represent particle size distribution by microscopic method, uncertainty region must be estimated. On this purpose, Masuda et al. derived analytical equation of the necessary sample size with known uncertainty region at mass median diameter [4]. However, in order to know better information of particle size distribution by microscopic method, it is necessary to estimate uncertainty region over all the range of particle diameter.

In this report, particle size measurement of 0.1–1 μm silica particles was conducted. The microscopic method with sample size greater than 90,000 particles was conducted for the measurement. Theoretical equation of uncertainty region over all particle diameter range is newly proposed and compared with computer simulation.

2. Microscopic method

Measurement of particle size distribution was carried out by use of silica particles produced by atomizing method of metal silicon solution under high temperature. Fig. 1 shows a photograph of silica particles measured by scanning microscope (SEM S-4800, Hitachi, Co., Ltd.). The magnification and acceleration voltage were set to 20,000 and 2 kV, respectively. In order to measure the length of particle size accurately, a certified scale shown in Fig. 2 (MRS-4.1, Geller Microanalytical Laboratory, Boston) was used for the measurement. The scale attached in the SEM apparatus was not used and maximum deviation between the certified scale and the SEM scale was about 3%. For the microscopic method, the following procedure was used to prepare the sample plate:

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Nomenclature

$f^{(\beta)}(x)$	size frequency distribution of parameter β (−/μm)
m,β	parameter used in Eq. (3) (−)
n	sample size (−)
u	reliability parameter (−)
x,x_0	particle diameter and reference particle diameter (μm)
$x_{50,3}$, $x^*_{50,3}$	mass median diameter and true mass median diameter, respectively (μm)
$\bar{x}(m,\beta)$	mean particle diameter defined by Eq. (3) (μm)
α	parameter used in Eq. (4) (−)
σ,σ_g	standard deviation of log-normal distribution and geometric standard deviation, respectively (−)
σ_1	uncertainty region due to limited sample size (μm)
σ_2	uncertainty region due to scale length measurement (μm)
σ_e	total uncertainty region defined by Eq. (11) (μm)
δ_1	uncertainty region due to limited sample size (−)
δ_2	uncertainty region due to scale length measurement (−)

(1) Acetone of 1 cm^3 and test silica particles of 0.001 g was mixed in a glass beaker.
(2) Ultrasonication using bath (100 W) about 1 h was applied to the solution.
(3) Adhesive tape was attached on the surface of a highly oriented pyrolytic graphite base plate (HOPG, GRBS grade, NT-MDT, Rossia) and the surface was treated to change into hydrophilic surface.
(4) The slurry of 6 μl was dropped on the tape and the plate was dried.
(5) The plate was inclined to 45° and Pt coating was applied from two directions.

Fig. 3 shows a photograph taken by SEM and each particle size was measured manually by marking a suitable sized circle on the particles. In order to eliminate counting error near the frames, size measurement was carried out only to the particles having the center positions inside the screen. Particle size measurement was not carried out for the non-spherical particles including strongly sintered or aggregated particles.

Fig. 4 shows the change of particle size distributions for different sample size. As the sample size increases, the shape of size distribution tends to converge to a specific distribution. The total sample size is 93535 and size distribution curve tends to converge for sample size greater than about 20,000. It is found that particle size distribution ranges from 0.1 to 1.0 μm.

Fig. 5 shows the relation between mass median diameter and sample size. The mass median diameter approaches to about 0.34 μm as sample size increases, and the experimental results are indicated inside the calculated uncertainty region of the following equation. Assuming true particle size is represented by log-normal distribution with mass median diameter $x^*_{50,3}$ and geometric standard deviation σ_g, the mass median diameter obtained from sample size n indicates the following uncertainty region [3].

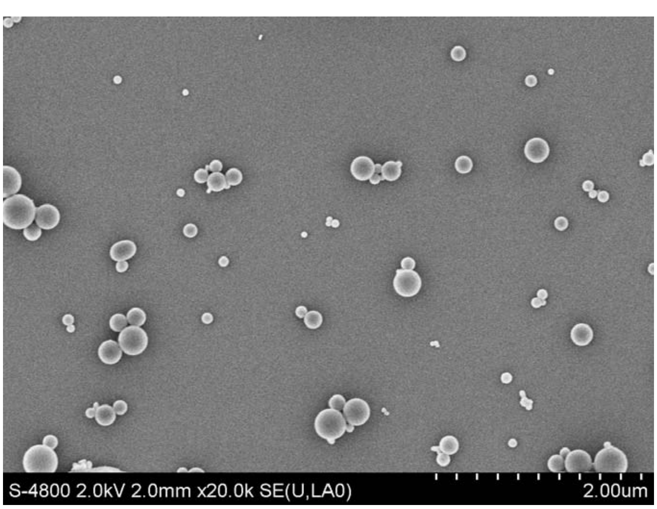

Fig. 1. Photograph of silica particles (SEM).

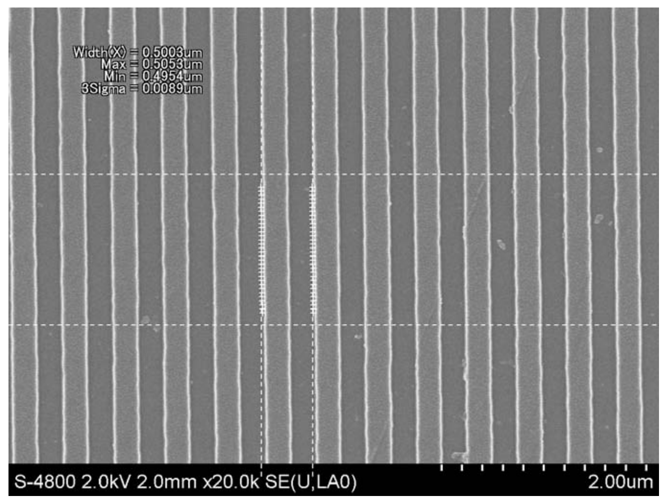

Fig. 2. Certified – scale for SEM measurement.

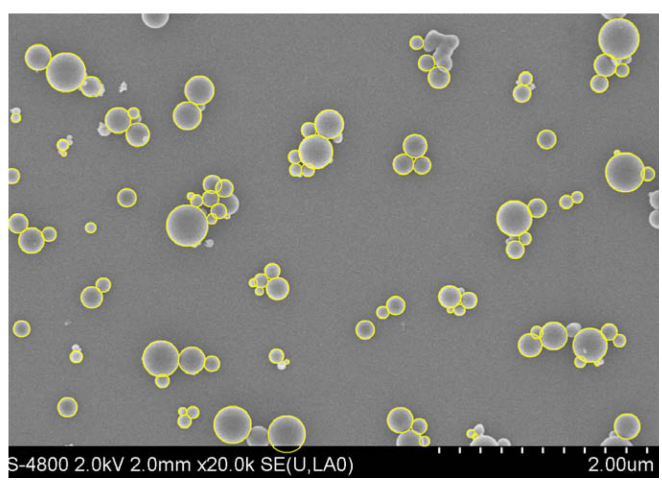

Fig. 3. Photograph of silica particles by SEM.

$$(1-\delta_1)x^*_{50,3} \leqslant x_{50,3} \leqslant (1+\delta_1)x^*_{50,3} \tag{1}$$

Assuming 95% confidence level, the uncertainty region is as follows:

$$\delta_1 = 1.96\sigma\sqrt{\frac{36(1+18\sigma^2)}{n}} \tag{2}$$

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