Application News

No. Q125

Powder Property Analysis

Evaluation of Shape/Coarse Particle Content of Spherical Silica Powder: Quality Control by Dynamic Image Analysis Method

Silica powder is used in a wide range of fields, from encapsulants for semiconductors and other electronic components to paints and fillers. However, when coarse particles (foreign matter or aggregates) are unintentionally included in silica powder, they can cause forming defects, insulation defects, and electrical property defects in electronic components and defects such as strength reduction or unevenness in paint films when present in paint. Moreover, the shape of particles also influences fluidity and performance.

Laser diffraction method (LD) and scanning electron microscopy (SEM) are used to evaluate the size and shape of silica powders. However, LD has low sensitivity for trace amounts of coarse particles, and in addition, it is theoretically impossible to acquire shape information, while SEM has the problems of a long measurement time due to its narrow observation field, and it is not possible to secure an adequate number of measured particles. On the other hand, because the dynamic image analysis (DIA) method can capture images of a large number of particles quantitatively in a very short time, DIA is suitable for rapid detection of trace amounts of coarse particles and evaluation of particle shape.

The Shimadzu iSpect™ DIA-10 dynamic particle image analysis system (Fig. 1), which is based on the dynamic image analysis method, measures the size distribution, concentration, and shape of particles by imaging particles in liquid samples, and makes it possible to analyze tens of thousands of particles in a few minutes with an optical system that ensures minimal omission (imaging efficiency: 90 % or more).

This article introduces an example in which the iSpect DIA-10 was used to evaluate the particle shape of silica powder and the contents of coarse particles before/after filtration.

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Fig. 1 iSpect™ DIA-10 Dynamic Particle Image Analysis System

Sample and Method

The sample material used here was a commercial spherical silica powder with a nominal diameter of 50 µm. Silicon oil (kinematic viscosity: 50 mm²/s) was used as the dispersion medium. The sample liquid and the sample liquid filtrate (obtained using a syringe filter with a pore size of 40 µm) were measured under the conditions in Table 1.

Table 1 Measurement Conditions

Frame rate	: 8 frame/s
Efficiency	: 97 %
Sample amount	: 50 µL
Threshold	: 200
Flow rate	: 0.1 mL/min

Measurement Results

Fig. 2 shows (a) the particle size distribution (volume basis) and (b) a scatter diagram (X-axis: area based diameter, Y-axis: circularity) of the silica powder before filtration. From the particle size distribution, it can be understood that the peak exists at 50 µm and the distribution displays a tail extending to about 10 µm. The scatter diagram shows that many particles with low circularity exist in the size distribution region of <40 µm in comparison with the region around 50 µm.

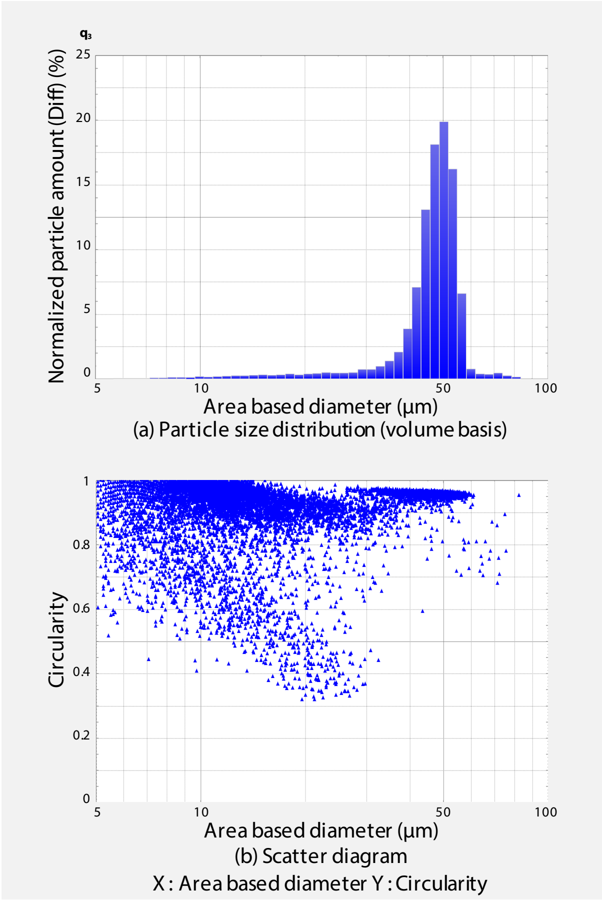
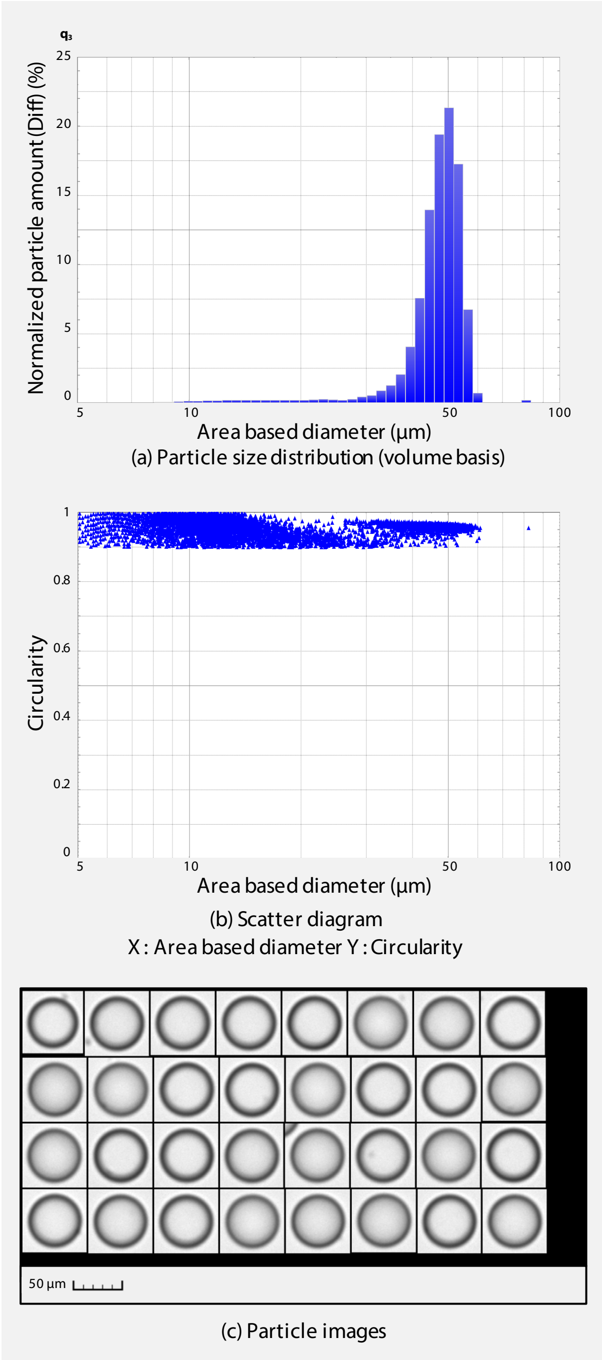


Fig. 2 Measurement Results of Spherical Silica (Nominal Diameter: 50 µm) Before Filtration

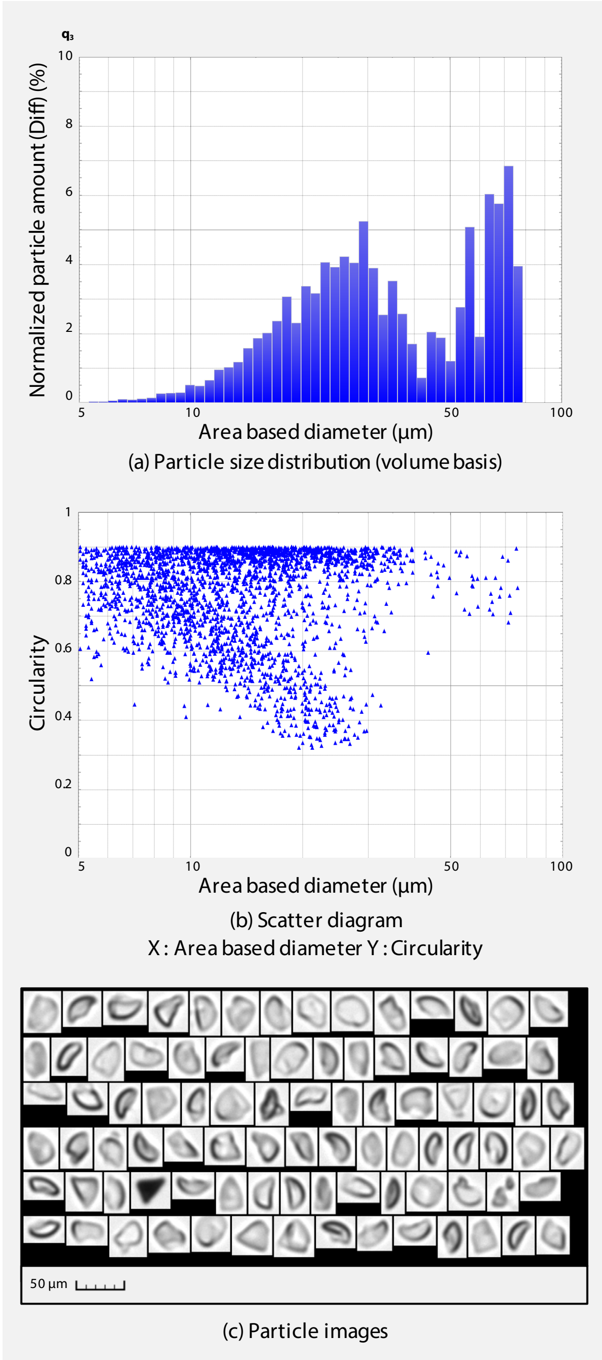


(a) Particle size distribution (volume basis)

(b) Scatter diagram
X : Area based diameter Y : Circularity

(c) Particle images

Fig. 3 Case of Classification by Circularity ≥0.9



(a) Particle size distribution (volume basis)

(b) Scatter diagram
X : Area based diameter Y : Circularity

(c) Particle images

Fig. 4 Case of Classification by Circularity <0.9

Fig. 3 shows the particle size distribution (volume basis), a scatter diagram (X-axis: area based diameter, Y-axis: circularity), and particle images for the measurement results of the spherical silica before filtration for classification by circularity of 0.9 or more. Fig. 4 shows the same results for classification by circularity of less than 0.9. From the particle size distribution, the large majority of particles with circularity of 0.9 or more have sizes around 50 µm, whereas particles with a distribution from 10 µm to 50 µm account for more than half of the particles with circularity of less than 0.9. Moreover, from the particle images, the particles with circularity of 0.9 or more are spherical particles, while those with circularity of less than 0.9 appear to be fragments of spherical particles. Fig. 5 shows the relative particle contents of the two classes of circularity. Based on the particle images, it can be thought that particles with circularity of 0.9 or more are defect-free, and the larger part of particles with circularity of less than 0.9 consists of cracked or chipped particles. This suggests that the defect ratio can be evaluated by this value.

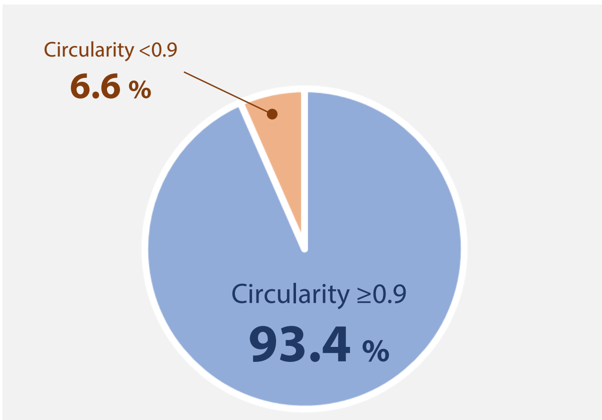


Fig. 5 Relative Particle Contents (Volume Basis) by Circularity Class

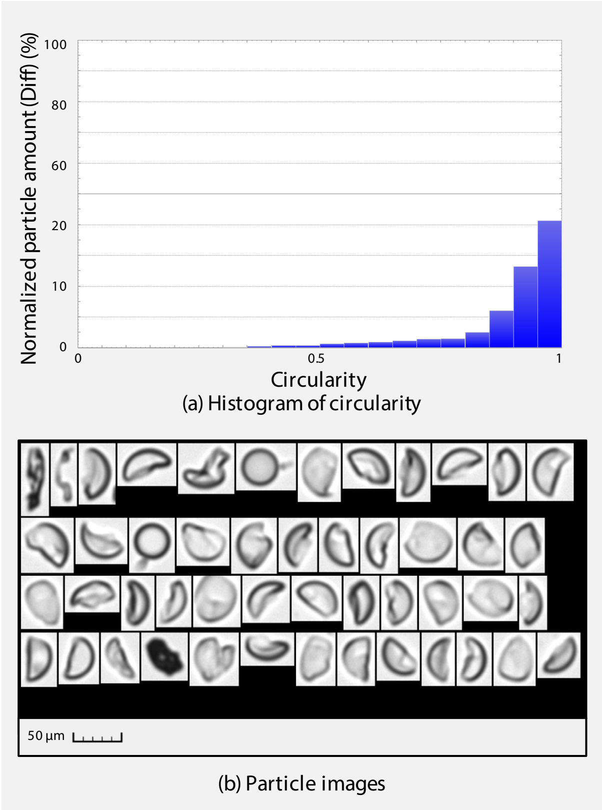


Fig. 6 Case of Classification by Area Based Diameter <40 µm

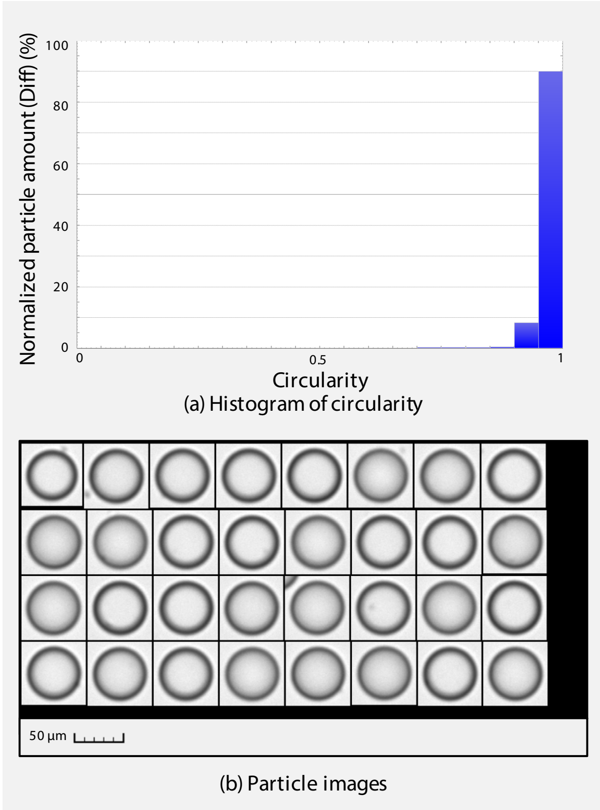


Fig. 7 Case of Classification by Area Based Diameter ≥40 µm

Fig. 6 and Fig. 7 show the histograms of circularity and particle images for the measurement results of the spherical silica before filtration for the area based diameters of less than 40 µm and 40 µm or more, respectively. From the particle images, almost all of the 40 µm or more particles are spherical particles, but among the particles with area based diameters of less than 40 µm, many particles which are considered to be fragments of spherical particles can be seen. Likewise, from the circularity histograms, the <40 µm particles include many with low circularity. Fig. 8 shows the average values of circularity for the two area based diameter classes. The average circularity of the <40 µm particles is 0.893, while that of the ≥40 µm particles is 0.956.

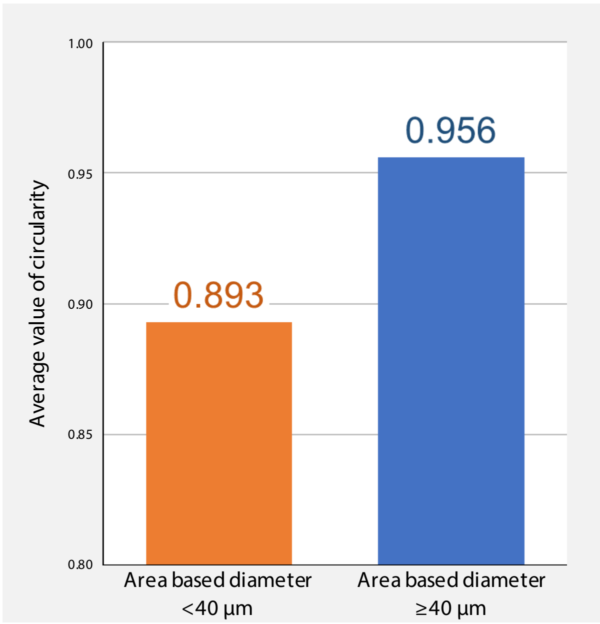


Fig. 8 Average Value of Circularity by Area Based Diameter Class

Next, Fig. 9 shows the particle size distribution (volume basis) before and after filtration. The scale of the ordinate (normalized particle amount) was set so that the heights of the frequency distributions of the <40 µm particles before and after filtration were the same. Fig. 10 shows the particle contents (content of <40 µm particles = 1) and the removal rate of particles by filtration. From the particle size distribution, it can be understood that particles in the range of 40 µm to 50 µm remained after filtration, indicating that particles with the filter pore size (40 µm) or more were not completely removed by filtration. The removal rates of the particles by area based diameter class were 43.7 % for particles with area based diameters in the range of 40 µm to 50 µm and 97.1 % for particles with area based diameters of 50 µm or more, showing that it was possible to remove almost all particles with sizes of 50 µm or more.

Conclusion

By using the iSpect DIA-10 dynamic particle image analysis system, it was possible to capture images of particles of commercial silica powder and quantitatively evaluate their shape parameters. A comparison of the content of coarse particles after filtration showed that it is important to check the results of filtration by actual measurement, as it is not necessarily possible to remove all particles as expected by the pore size of the filter. Because the iSpect DIA-10 enables rapid measurement with high detection efficiency by the microcell technique, this dynamic image analysis system is an effective tool for evaluations of particle shape and the content of coarse particles in quality control and research and development.

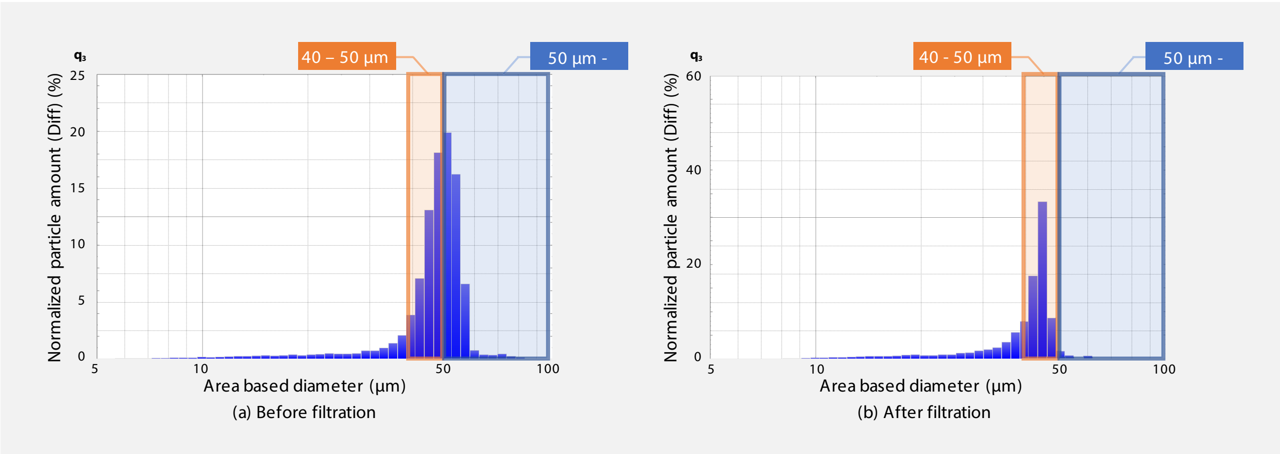


Fig. 9 Particle Size Distribution (Volume Basis) Before/After Filtration

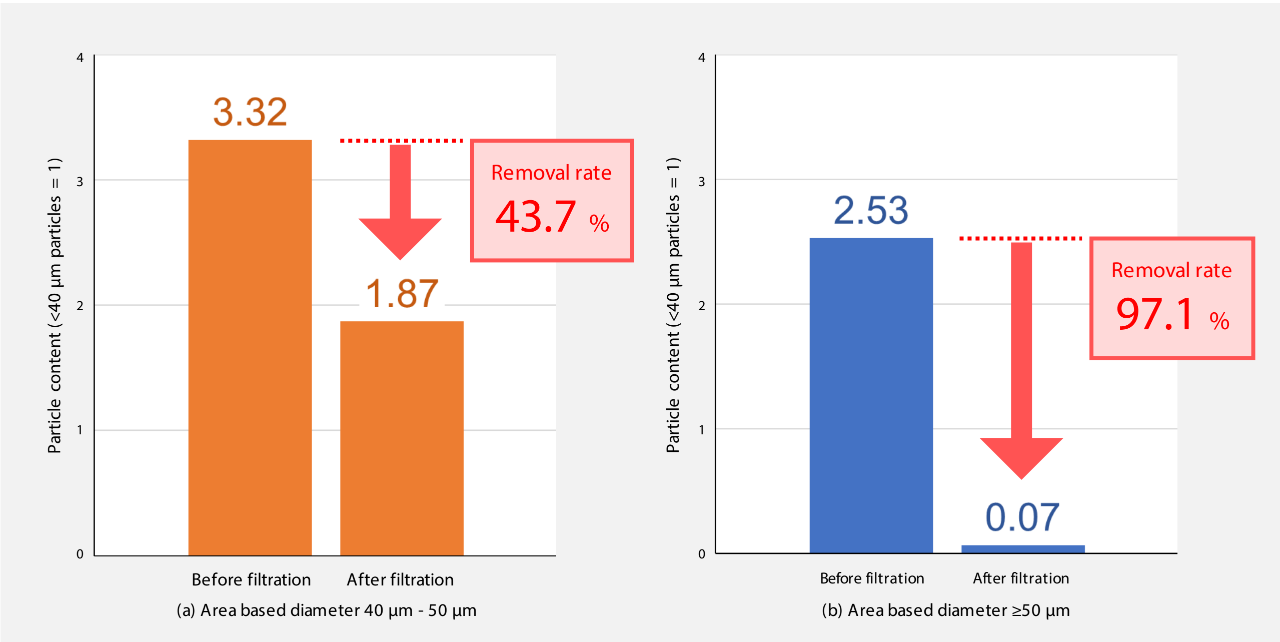


Fig. 10 Removal Rate of Particles by Area based diameter Class

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