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Application News

No. Q124

Powder Property Analysis

Evaluation of Concentration of Coarse Particles in High Concentration Silica Nanoparticle Slurry: Foreign Object Detection by Dynamic Image Analysis Method

Silica is used in a wide range for fields, for example, as an encapsulant for semiconductors and other electronic components and as a filler in paints. When coarse particles (foreign matter, aggregates) are unintentionally included in silica, they can become a cause of defects such poor forming, insulation, or electrical properties in electronic components and reduced strength or unevenness of the paint film in paints.

The techniques used in size evaluations of silica include laser diffraction method (LD) and scanning electron microscopy (SEM). However, as issues of these two methods, LD has low sensitivity for trace amounts of coarse particles, and SEM has a long measurement time owing to its narrow field of view in observation, and it is impossible to secure an adequate number of measurements. In addition, concentration information cannot be obtained with SEM. In contrast, the dynamic image analysis method makes it possible to acquire images of a large number of particles quantitatively and in a short time, and thus is suitable for rapid concentration evaluations and detection of trace amounts of coarse particles.

The Shimadzu iSpect™ DIA-10 dynamic particle image analysis system (Fig. 1), which is based on this dynamic image analysis method, acquires images of the particles in liquid samples and measures the particle size distribution and particle concentration and shape. It is possible to analyze tens of thousands of particles in a few minutes with few missed particles (image acquisition efficiency: 90 % or higher). Moreover, since the iSpect DIA-10 uses the microcell method, it is also possible to measure cloudy samples which do not transmit light in a sample container. As a result, it is possible to shorten the time required for pretreatment and minimize the effect of dilution on samples by measuring the stock solution or a low-dilution solution.

This article introduces an example in which the iSpect DIA-10 was used to evaluate the concentration of coarse particles in a silica nanoparticle slurry without diluting the stock solution.

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

Sample and Method

A commercially-available silica slurry (concentration: 50 mg/mL) with a nominal particle size of 0.2 µm was used as the sample material. Fig. 2 shows the appearance of the silica slurry. The stock solution of the sample liquid and a liquid obtained by filtering the stock solution with a syringe filter having a pore diameter of 0.8 µm were measured under the conditions in Table 1.

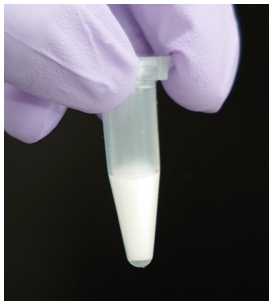


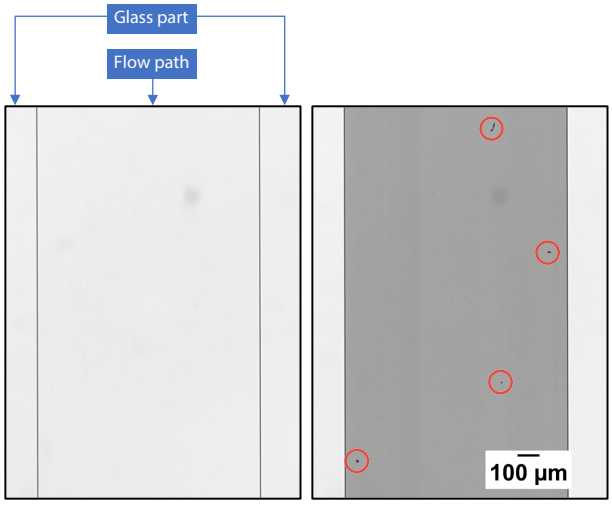
Fig. 2 Appearance of Silica Slurry with Nominal Particle Size of 0.2 µm

Table 1 Measurement Conditions

Frame rate	: 8 frame/s
Efficiency	: 97 %
Sample amount	: 50 µL
Threshold	: 130
Flow rate	: 0.1 mL/min

Measurement Results

Fig. 3 shows images of cells acquired with the iSpect DIA-10 (gray scale image). From Fig. 3(a) Water, it can be understood that light is transmitted through water similarly in both the flow path and the glass part, whereas in (b) Sample liquid, submicron particles are not distinguished as particles, and the entire flow path appears dark, corresponding to their concentration. From the appearance of the slurry in Fig. 2, the sample appears to be milky white and does not transmit any light whatsoever, but because of the thin thickness (120 µm) of the microcell, measurement is possible and coarse particles can be detected, as indicated by the red circles in Fig. 3(b).



(a) Water (b) Sample liquid

Fig. 3 Images of Cells

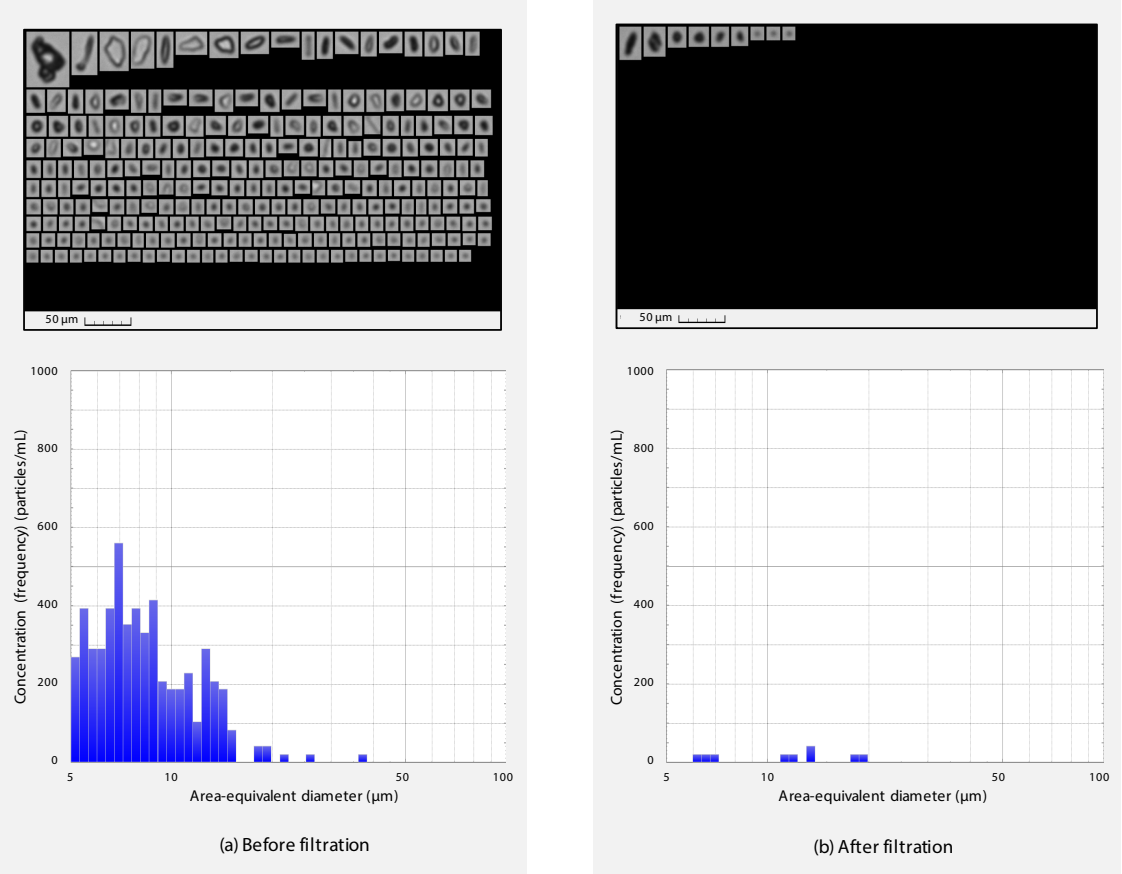


Fig. 4 Particle Images and Particle Size Distribution
(a) Before and (b) After Filtration of Spherical Silica Slurry with Nominal Particle Size of 0.2 µm

Fig. 4 shows images of the particles and the particle size distribution before and after filtration of the silica slurry with the nominal particle size of 0.2 µm. The particle images before filtration with the filter reveal the presence of scale-like particles with a long-side length exceeding 10 µm and a refractive index close to that of water. (As the refractive index of particles approaches that of the solvent, the images become increasingly transparent.) From the particle size distribution, many particles with sizes mainly around 10 µm were detected, and the size distribution was up to an area-equivalent diameter of somewhat less than 40 µm at maximum. On the other hand, after filtration, the number of particles decreased in comparison with before filtration. Moreover, the presence of particles with sizes exceeding the filter pore diameter of 0.8 µm indicates that particles larger than the filter pore diameter were not completely removed even after filtration.

Table 2 Particle Concentration Before/After Filtration

	Observed particle count (particles)	Particle concentration (particles/mL)*
Before filtration	266	5511
After filtration	9	186
Particle removal rate (%)	96.6	

* The particle concentration was calculated from the observed particle count, cell volume, and number of images.

Table 2 shows the concrete change in the particle concentration. The particle concentration was 5,511 particles/mL before filtration and 186 particles/mL after filtration, and the particle removal rate by the filter was 96.6 %.

Conclusion

By using an iSpect DIA-10 dynamic particle image analysis system, it was possible to measure a commercial silica slurry with a nominal particle size of 0.2 µm and a concentration of 50 mg/mL without diluting the stock solution and quantitatively evaluate the concentration of coarse particles of µm order size before and after filtration. Moreover, it was also found that coarse particles were not necessarily removed as expected from the filter pore diameter, demonstrating the importance of measurements to confirm the particle size after filtration. With the iSpect DIA-10, it is also possible to measure slurries with a high concentration of submicron particles by utilizing the microcell method, using the undiluted stock solution or a low-dilution solution. Because quick measurement with high detection efficiency is possible, the iSpect DIA-10 is an effective tool for concentration evaluations and detection of coarse particles (foreign matter and aggregates) in quality control.

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