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Application News

Dynamic Particle Image Analysis System
Laser Diffraction Particle Size Analyzer

Evaluation of Microscopic Foreign Matter in CMP Slurry Using Dynamic Image Analysis and Machine Learning

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User Benefits

- Foreign matter in the micron range can be detected and evaluated at near-undiluted concentration using the microcell method.
- Telecentric optical system minimizes missed microscopic foreign matter (imaging efficiency: 90 % or higher).
- Particle images and morphological information obtained by dynamic image analysis can be used for particle classification, which enables a more detailed evaluation of microscopic foreign matter in CMP slurry.

■ Introduction

Foreign matter in CMP* slurry used for precision polishing of semiconductor wafers may cause defects. Therefore, abrasive particles and foreign matter must be carefully controlled. In studies of filter-based foreign matter removal and process control, it is important to detect and evaluate even trace levels of foreign matter.

However, laser diffraction and dynamic light scattering, which are commonly used for particle size distribution analysis, may require adjustment of the sample concentration to match the main component, making it difficult to detect trace amounts of microscopic foreign matter.

In this study, colloidal silica used in CMP slurry was analyzed using the iSpect DIA-10 dynamic particle image analysis system to evaluate trace amounts of microscopic foreign matter. Machine learning-based clustering was then applied to assess the detected foreign particles.

* Chemical Mechanical Planarization/Polishing

■ Sample

A commercially available polishing-grade colloidal silica slurry was used as the sample, with a solids concentration of approximately 30 wt% and water as the dispersion medium. Laser diffraction analysis using the SALD-7500nano nanoparticle size distribution analyzer showed a monodisperse particle size distribution with a median diameter of 88 nm. The dilution factor during measurement was 1000-fold. Since the particles used in precision polishing are typically tens to hundreds of nanometers in size, the laser diffraction/scattering results indicated a particle size distribution suitable for precision polishing.

Fig. 1 Sample Appearance

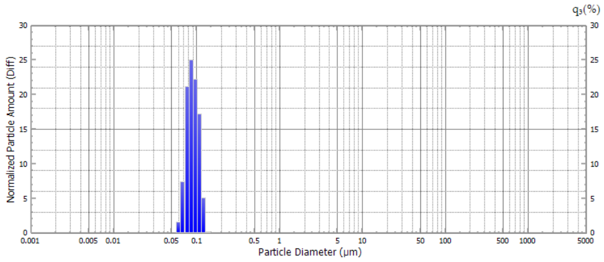

Fig. 2 Particle Size Distribution Obtained by Laser Diffraction
(Dilution: 1000-fold; measured with the SALD-7500nano Nanoparticle Size Distribution Analyzer)

However, conventional laser diffraction/scattering methods generally require sample concentrations on the order of several hundred ppm, and because the concentration must be adjusted to the main component, it can be difficult to detect trace microscopic foreign matter. Therefore, to detect and evaluate trace foreign matter, measurements were performed by dynamic image analysis (DIA) using the iSpect DIA-10.

DIA captures images of a microcell channel in which particles flow, extracts each particle image from the channel images, and evaluates particle shape and concentration. In addition, by using the microcell method, the iSpect DIA-10 achieves highly reliable foreign matter detection and accurate concentration evaluation. Furthermore, when evaluating micron-scale foreign matter in samples containing relatively high concentrations of submicron particles, measurement can be performed without dilution or at a low dilution factor.

(Related application: Application News No. Q124)

Fig. 3 The iSpect DIA-10 Dynamic Particle Image Analysis System

■ Methods and Results

The DIA measurement conditions are shown in Table 1, and the results are shown in Figs. 4 and 5. Measurements were performed using the undiluted sample. The number concentration of micron-scale particles was 3315 particles/mL, and the largest detected particle had an area-equivalent diameter of approximately 73 µm.

The volume concentration, calculated by determining the sphere-equivalent particle volume from the area-equivalent diameter for each particle and summing the volumes of all particles, was 2.337 ppm. Since laser diffraction/scattering measurements are typically performed at concentrations of several hundred ppm, and the sample was measured after a 1000-fold dilution, it is clear that detecting micron-scale foreign matter in this sample by laser diffraction/scattering would be difficult.

Table 1 DIA Measurement Conditions

Frame Rate:	8 fps
Efficiency:	96.5 %
Luminance:	249
Threshold of Binary Image:	90 %
Pump Volume:	250 µL
Background Correction:	Enable

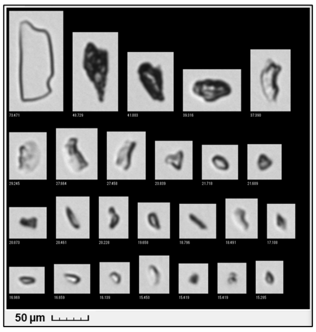

Fig. 4 Particle Images Obtained by DIA (Undiluted Sample)

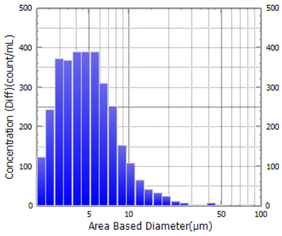

Fig. 5 Particle Size Distribution Obtained by DIA (Undiluted Sample)

Particle images obtained by DIA are shown in Fig. 4, while the corresponding particle size distributions are shown in Figs. 5 and 6. Even with DIA, only a very small number of foreign and coarse particles were detected at a 1000-fold dilution factor. This indicates that evaluation under undiluted conditions is preferable when assessing trace foreign matter.

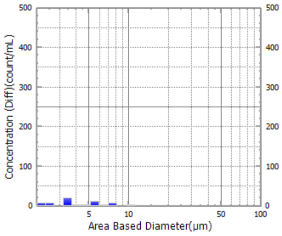

Fig. 6 Particle Size Distribution Obtained by DIA (Dilution: 1000-fold)

Particle type is important for identifying particle origin, developing reduction measures, and evaluating impact, and particle images provide useful clues for such identification. In addition to shape information, brightness contains information related to thickness in the observation direction, the relative refractive index between the particle and the dispersion medium, and surface roughness. To understand what particle types were present, particles larger than 10 µm, which accounted for more than 90 % of the total volume, were classified into six particle populations based on the results of clustering obtained by machine learning, using dimensionality reduction by UMAP and clustering by HDBSCAN (Fig. 7 and Table 2).

To remove particle groups a, b, and d, which have small aspect ratios and are elongated, it is important to select a filter pore size that takes the minor axis into account. In addition, groups a and d, which have higher average brightness than particle groups of similar size, may be flake-like particles that are thin in the observation direction. In this way, more advanced foreign-matter control is possible by using the obtained particle images and shape information.

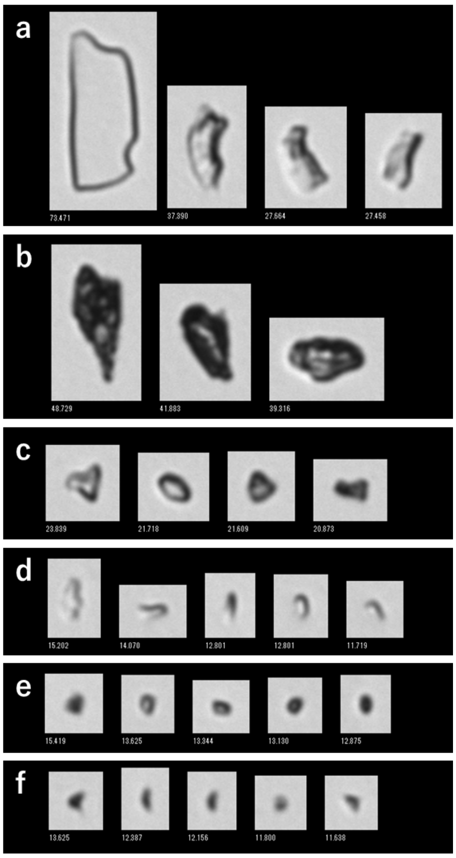

Fig. 7 Grouping Results for Particles Larger than 10 µm

Table 2 Morphological Information for Each Particle Group

Cluster	Particle Amount		Morphology Parameter (Average)			
	Volume	Count	ABD	Circ.	AR	Br. Avg
a	54.7 %	8.8 %	35.909	0.505	0.485	144
b	26.1 %	4.4 %	43.310	0.681	0.536	82
c	9.0 %	17.6 %	18.903	0.753	0.611	127
d	3.8 %	22.1 %	12.974	0.576	0.524	155
e	2.8 %	22.1 %	11.992	0.970	0.754	131
f	3.5 %	25.0 %	12.230	0.858	0.610	144

[ABD]:	Area Based Diameter	[Circ.]:	Circularity
[AR]:	Aspect Ratio	[Br. Avg]:	Average Brightness

■ Conclusion

To evaluate trace foreign matter and coarse particles contained in polishing-grade colloidal silica used as CMP slurry, DIA measurements were performed. Images and concentrations of micron-scale particles that could not be detected by laser diffraction were successfully measured. In addition, the microcell method enabled measurement to be performed without dilution. On the other hand, even with DIA, only a small number of particles were detected at a 1000-fold dilution factor, demonstrating the effectiveness of evaluating trace foreign matter in the undiluted sample. Furthermore, machine-learning-based clustering enabled quantitative evaluation of the particle types present. Thus, DIA is an effective analytical technique for evaluating trace foreign matter and coarse particles in CMP slurry.

References
1) Syuhei Kurokawa: The Overview and Future Prospects for Planarization CMP Technology, Journal of the Japan Society for Precision Engineering, Vol. 84, No. 3, pp. 213–216 (2018)

Related Applications
1. Evaluation of Concentration of Coarse Particles in High Concentration Silica Nanoparticle Slurry: Foreign Object Detection by Dynamic Image Analysis Method, Application News No. Q124

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