**Digital image processing in nanoparticle size measurement using scanning electron microscopy**

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Nanoparticle (NP) size is a critical property that determines the performance of NP-products and that is potentially related to NP safety, so that accurate NP sizing is essential. Out of several characterizing tools, scanning electron microscope (SEM) is popularly used due to both of its unique surface sensitivity and convenience. In contrast, the electron beam-to-NP interaction in SEM is too complex to treat the data with a simple relation. SEM measurement of NP may be highly dependent on the experimental condition and analysis protocol. Further, since SEM often collects data as many as hundreds of NPs for the sizing, the measurement is time-consuming.

The measurement procedure consists of the following sequential steps: sample preparation, image acquirement, particle analysis (image processing), and data analysis (See Fig.1). Among them, the determination of SEM resolution and particle boundary from the background is a key step to make the measurement objective, rapid and automatized. Conventionally, it has been performed manually, which is subject to the personal error. Digital methods to resolve these issues are the objective of this study.

The resolution is a key performance of SEM. It should be considerably smaller than the particle to analyze. The smallest pixel size is chosen considering the resolution. Thus, the resolution should be known in the objective sense for the landing energy of the SEM probe, probe current and the working distance. An image of gold-on-carbon sample was taken, and the image sharpness was measured with a computer program. Algorithm to measure SEM image sharpness has been developed according to the derivative method as stated in ISO/TS 24597 document, as being implemented in a MATLAB-based program. The program was validated with a set of numerically generated reference images. Fig. 2 shows an example in a SEM condition used to measure NPs.

The particle analysis is divided into sub-steps including de-noising image, image thresholding, hole-filling, discarding unwanted NPs and artifacts, and obtaining sizes for all remained NPs. De-noising is the two-dimensional filtering of the taken image to suppress the image noise. Image thresholding has a significant impact on the analyzing process itself as well as the results. Otsu thresholding is used to separate NPs from background. The hollow region within any particle are filled with white against black background. The unwanted objects are removed type by type based on the suitable criterion. With the finally remained NPs, particle size-related parameters are determined digitally to get the statistical mean and other quantities.

We developed an algorithm for automatic particle analysis using MATLAB, and actually applied to the measurement of silica and gold NPs. The safety of the nanoparticle is still in dispute. The sample was prepared by dropping and drying the NP suspension on a silicon wafer chip treated clean and hydrophobic. At the sites full of isolated while densely located NPs, SEM images were taken until we collect many frames enough for the statistic. The mentioned procedure is quite reproducible and compatible with other programs, for example, ImageJ. Fig. 3 shows examples of thus-measured NP measurements.

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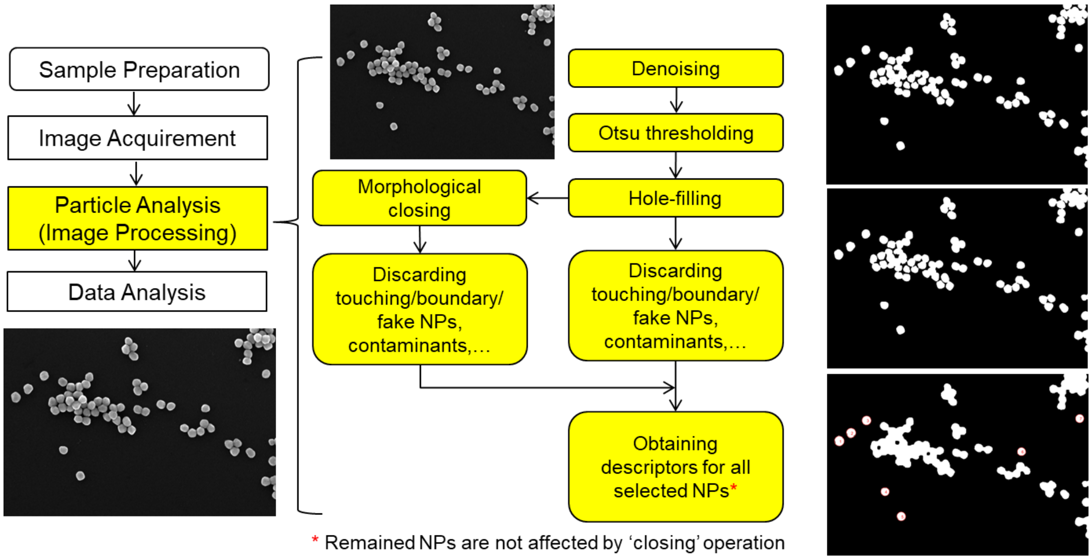


Fig. 1. Procedure for NP size measurement using SEM.

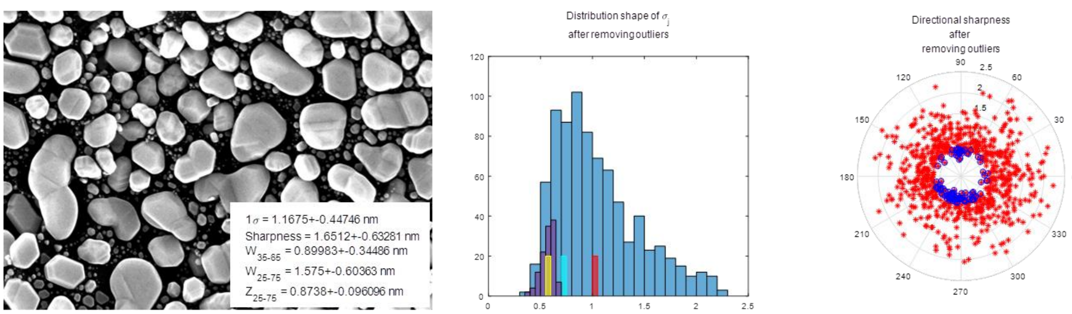


Fig. 2. Image sharpness measurement using derivative method.

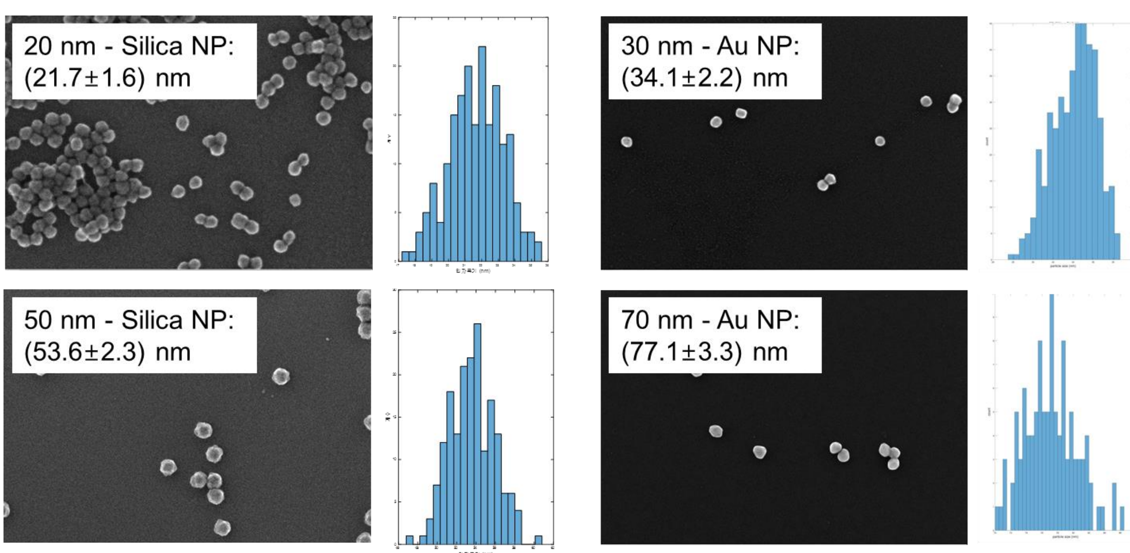


Fig. 3. SEM measurement results for silica and gold nanoparticle size.