Nanocluster analysis using ultra high resolution scanning electron microscope

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Metal oxide nanocrystals have attracted much attention due to their unique properties including magnetism, luminescence, and ferromagnetism. The shape and the surface structure of the nanocrystals largely affect their properties and thus methods for their analysis should be developed.[1] In addition to the analysis of individual nanocrystals, the arrangement of nanocrystals should be also studied because the properties of nanoclusters comprising the primary nanocrystals largely depend on the arrangement of the nanocrystals. However, the analysis of the nanoparticles and nanoclusters remains difficult due to their small size. For a long time, transmission electron microscopy has been widely used to study materials at the nanoscale. Recently, we have developed an extremely high resolution scanning electron microscope (HR-SEM, JSM-7900F JEOL Ltd.,) equipped with EDS detectors to analyze composition of nanoclusters. By using this HR-SEM with automatic electron-optics adjustment function, we have successfully observed nano-structures smaller than 1.0 nm at a landing voltage of 1 kV [2]. The low landing-voltage technique suppressed the sample charging and damage, leading to clear observation of the shape and size of nanocrystals and nanoclusters. Figure 1 shows an SEM image of CeO_2 nanoparticles observed at a 1 kV landing voltage (Sample bias: -5 kV, Probe current: 8 pA, Detector: in-column detector). The shape and size of CeO_2 nanoclusters are clearly observed without coating.

We believe this low voltage SEM is very useful method to understand structures in surface. On the other hand, it is difficult to obtain average size of clusters and elemental information. Recently, we have succeeded in developing a method for analyzing nanoparticles using ultra high solid angle EDS (X-max 170mm^2 x 2 and Windowless type EDS Extreme 100 mm^2, Oxford instruments) and ultra high resolution SEM. Figure 2 shows the result of the particle analysis that can automatically measure the size of 848 particles smaller than 50 nm for 3 min.

In this talk we will discuss about high resolution impinging at low voltage and recent achievement of nanoparticle analysis using ultra high resolution SEM.

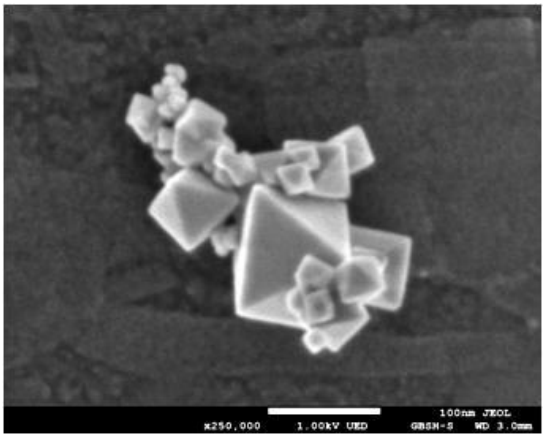

Figure 1 High resolution low voltage image of CeO_2 nanoclusters using SEM.

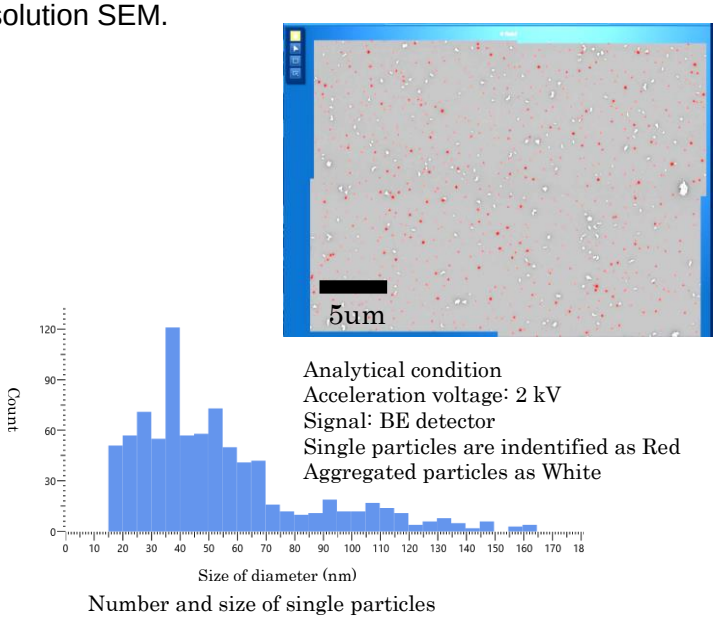

Figure 2 Result of nanoparticle analysis in CeO_2.