Atomic resolution electron tomography of catalytic particles

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Recent advancement of the aberration corrector enables us to observe and to analyze samples in atomic resolution. However, TEM and STEM images are two dimensional projections of three dimensional (3D) objects. It is becoming increasingly important to obtain 3D information of samples in atomic scale. For example, catalytic properties depend on atomic arrangements in the catalytic particles. So, there are demands to investigate 3D atomic structure of the particles. The electron tomography is useful technique to observe 3D structure in nanometer scale and has been successfully employed in the various fields. Combination of electron tomography and aberration corrected STEM has potential to extract 3D structural information with atomic resolution. In this study, trials of atomic resolution tomography using an aberration corrected STEM was made to clarify atomic arrangements in nanoparticles.

The material of interest is a catalyst containing Au particles (Haruta Gold Incorporated). STEM observation was performed using an HF5000 TEM/STEM (Hitachi High-Technologies Co.) with a probe forming aberration corrector operated at 200 kV. The probe corrector with automated correction allows us to obtain aberration-less STEM images with several operations. This instrument has three detectors for scanning imaging: secondary electron, bright field STEM and annular dark field STEM detectors. SEM and STEM images are recorded simultaneously. In the electron tomography observations, tilt series of dark filed STEM images were acquired at intervals of 4° from +50° to -50° of the tilt angles. While recording the images, fast scan images were acquired to avoid electron beam damage. The 3D images were reconstructed from the series of dark field STEM images by EMIP software. The 3D reconstructed images were displayed by Image Pro Premier 3D (Media Cybernetics).

Fig.1 shows a tilt series images of dark field STEM of an Au nanoparticle. Even when the sample was tilted to higher angles, lattice images and atomic columns are clearly observable. The particles did not change shape significantly after finishing the tilt series image acquisition. Fig.2 demonstrates projection of 3D images reconstructed by simultaneous iterative reconstruction technique. 3D distribution and shape of Au atoms in the particle are visualized. Artifact of the reconstruction process makes it harder to distinguish atoms arranging in the particle in one direction because the missing wedges of information caused by the limited range of tilting at acquisition of original STEM images stretch the image of each atom. Although conventional reconstruction method was used in this study, a new reconstruction method suitable for atomic column images will be applied. There are problems to obtain high quality 3D image with atomic resolution left. However, this study demonstrates some of capability of the HF5000 TEM/STEM to perform atomic resolution electron tomography.

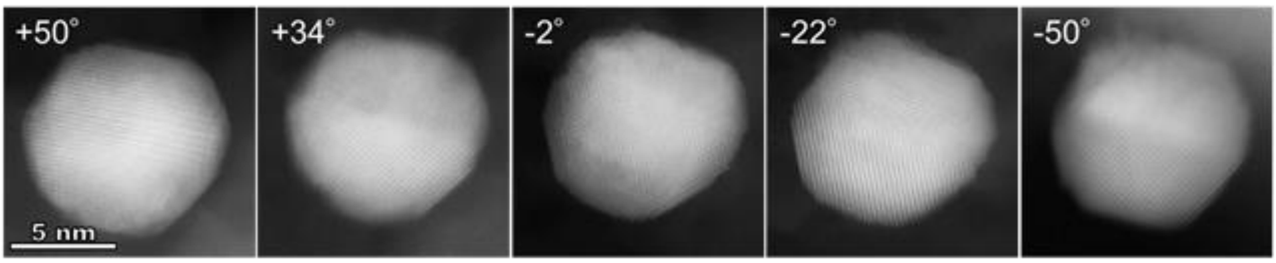

Fig.1 Tilt series images of dark field STEM images of an Au nanoparticle

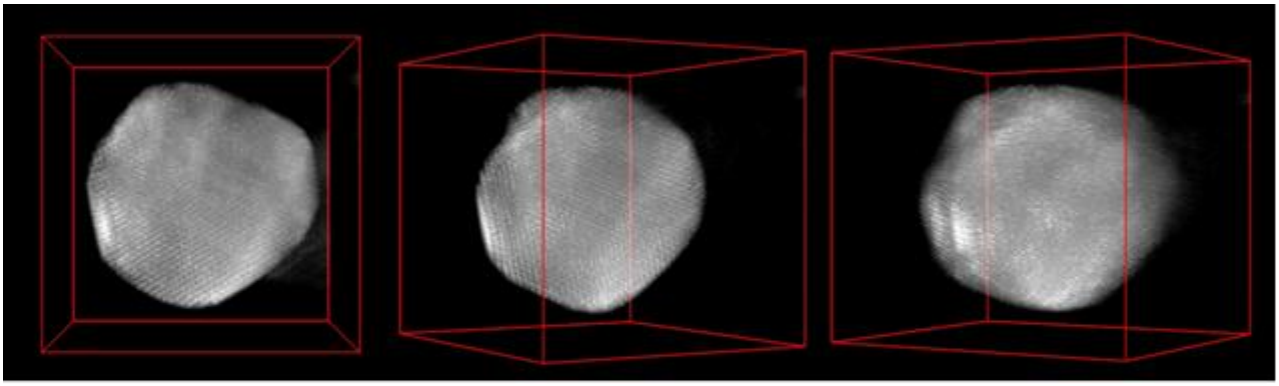

Fig.2 3D reconstruction images of Au nanoparticles. The left, center and right images show regions viewed from normal, 30° and 60° directions, respectively.