

CRYO-ELECTRON TOMOGRAPHY OF MAGNETIC AND CATALYTIC CLUSTERS, POLYMERS AND HUMAN CELLS

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Three-dimensional(3D) electron tomography is one of the key techniques for developing new possibility of electron microscopy of nano-materials. In the present talk, 3D observation data of iron-platinum(FePt) ferromagnetic dots(clusters)[1], photocatalytic films of titania(TiO₂)[2], catalytic platinum(Pt) supercrystals having tunnels[3], triple-mixed polymers and tissues in human-cells[4] are presented in order to show the present state of art. The observation was performed with a helium or liquid-nitrogen cooled 300 kV FEG-TEM with an automated tilting stage for 3D electron tomography. Sample cooling below 10 K was very much effective for reducing irradiation damages during many exposures for the tomography. Other essential points for 3D observation are sample preparation and overcoming the so-called missing cone effect, the former of which is indeed important for polymers and biological samples and the latter is studied by using a self-developed simulation routine of 3D images in the present study.

The TEM observation was performed with a 200 kV TEM (JEM-2010) and a 300 kV TEM(TECNAI F30) with a post-column energy filter(Gatan Imaging Filter). Image recording for 3D reconstruction was made by using the latter with automated tilting and recording systems. The data recording was made in a single tilt mode from -70 to $+70$ degree with increments of 1 degree in small tilting angles up to ± 50 degree and 0.5 degree in larger ones from ± 50 degree to ± 70 one. 3D reconstruction was then performed by using IMOD software as an internationally standard one[5], and 3D visualization, with a commercial AMIRA software (Mercury Computer Systems Inc.).

Image simulation of 3D reconstructed images was realized with a combined use of a multi-slice image simulation program(MachREM)[6] and the IMOD software. First, we constructed a supercell for a MgO single crystal including an FePt dot. Second, the image intensity at an exit surface of the supercell was calculated including dynamical diffraction and lens effects by using the multislice program. In tilted incident cases, we took different supercells for each of the cases. Third, more than 100 simulated image data from the supercells were combined in the IMOD in order to create 3D reconstructed images.

Fig. 1 shows a 3D image of FePt dots embedded in a MgO single crystal film[1]. The sample was prepared by a successive UHV deposition of MgO, Pt, Fe and MgO onto heated NaCl substrates. In spite of dots embedded in the crystalline MgO film, spatial resolution less than 1 nm is realized in the 3D reconstructed image[1]. This result is gifted from more than 100 good images with a little irradiation damage by cooling samples. Fig. 2 shows image simulation of 3D reconstructed images of a FePt dot in MgO and the actual image; (a)model for calculation, (b)multislice simulation data as thin slabs against tilting angles, (c)2D representation of the 3D reconstructed image and (d)actual image with two FePt dots. By using the present simulation routine, we have clarified origins of various kinds of artifacts appearing in electron tomography[7]. Fig. 3 shows a surface-rendering image of catalytic Pt supercrystals prepared from meso-porous MCM-48 caged crystals as the mold[3]. The inner tunnels are beautifully reconstructed in spite of obtaining the initial data from the bright field TEM images[8], not from ADF-STEM images[3]. 3D electron tomography is indeed useful for biological samples. Fig. 4 shows a 3D reconstructed image

of a tissue inside the cell of urinary bladder[4]. The sample is a replica of the tissue located on backside surfaces of the cell, prepared by a freeze-etching technique. 3D reconstruction is successfully made from many good images accumulated without strong damage due to cooling by helium. We have found the cooling is essential for successful 3D reconstruction of also inorganic samples as well as biological ones.

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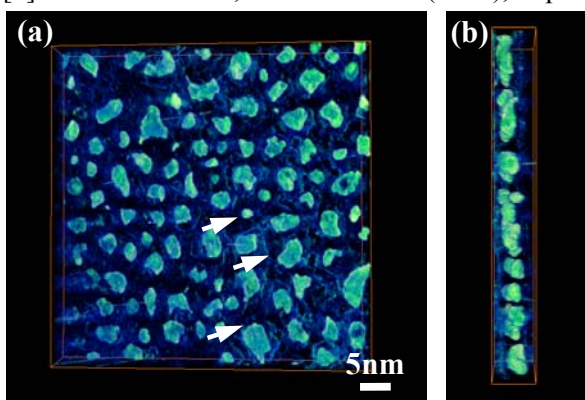


Figure 1: 3D image of FePt dots embedded in MgO thin films. (a)plane view, (b)cross-sectional view.

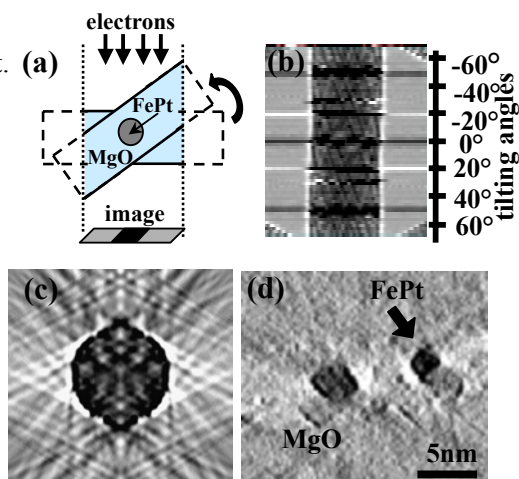


Figure 2: Simulation of 3D reconstructed image(b,c) and actual image of FePt dots(d).

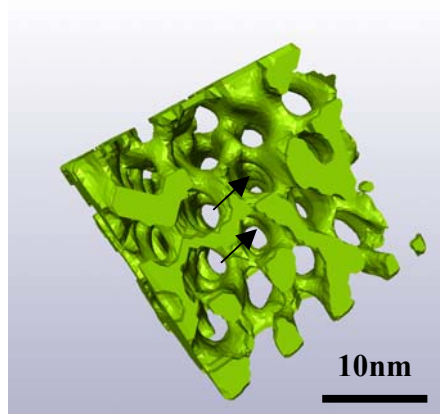


Figure 3: 3D image of a Pt catalytic caged supercrystal.

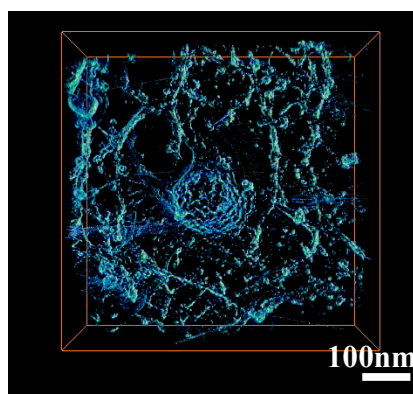


Figure 4: 3D reconstructed image of a tissue attached on the cell wall of urinary bladder.