Transfer of area of interest in thin films to a 360° Electron Tomography sample holder

Hayashida, M.[1], Phengchat, R.[2] and Delgado, F.P.[3]

[1] Nanotechnology Research Center, Canada, [2] Kobe University, Japan, [3] Centro de Investigación en Materiales Avanzados, Mexico

Electron tomography (ET) in a transmission electron microscope (TEM) enables structure of materials and nanodevices to be visualized in three dimensions (3D). If the sample of interest is on a substrate or embedded in a matrix, an area of interest can be plucked out and placed onto a 360° ET sample holder. A rod shaped specimen can be then fabricated by focused ion beam (FIB) to observe over a full tilt range. It allows us to avoid missing wedge of data and obtain an isotropic resolution in 3D [1]. However, most samples for TEM are on thin films such as carbon films. Therefore, we developed a pluck out method to enable 3D ET on objects extracted from thin film TEM samples. The method has been implemented in a Hitachi NB-5000 FIB / SEM.

The method is as follows. An area of interest (1.5 m×3 m) is selected using an SEM image. Carbon is then deposited using a low FIB deposition from phenanthrene precursor and a low FIB current. Two adjacent sides of the area are cut by FIB. A micro sampling probe is attached to the area by FIB carbon deposition. The remaining two sides are cut by FIB and the sampled area is released. The position of the probe is adjusted in STEM mode of the SEM column. FIB is used at low magnification only for final adjustment to avoid Ga^+ damage. The plucked area is transferred and attached to the 3D TEM sample holder by carbon deposition. The shape of the sample is finalized by removal of unnecessary regions of the sample by FIB. The width and thickness of the final sample is cut by FIB to approximately 200-300 nm and 100 nm. During the procedure, radiation damage by FIB should be avoided as much as possible.

Gold nanoparticles [2] on a carbon film were used as an example to demonstrate the method and to show its suitability for 3D TEM by ET. Only an area with a star-shaped gold nanoparticle was transferred on a Hitachi 3D TEM holder and a tilt series was taken with 3° step over 180° tilt range. Electron beam radiation damage of the area was not detectable during acquisition of the tilt series as estimated by comparing the projected shape in images collected at -90° and 90° tilt.

A new method for preparation of samples for 3D electron tomography by plucking out regions of thin film TEM samples has been demonstrated. A sample consisting of Au nanoparticles embedded in carbon matrix and isolated chromosomes are shown here. The method has been successfully applied to various samples including biological samples.

[1] Barnaed, D. P., Turner, J. N., Frank, J.,McEwen, B. F., (1992) Journal of Microscopy 167, 39-48.

[2] Morla-Folch, J.; Guerrini, L.; Pazos-Perez, N.; Arenal, R.; Alvarez-Puebla, R. A., Synthesis and Optical Properties of Homogeneous Nanoshurikens. Acs Photonics 2014, 1 (11), 1237-1244.