

Shapes and Atomic Arrangements of Pure Cu-Nanoparticles Grown in High Vacuum and in Non-stress Condition Observed by 3D-Tomography in a TEM

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The performances and the properties of nanoparticles are determined by those sizes, structures and elemental concentrations. The structures are also determined growing conditions and sizes of the particles[1]. In this work, pure copper (Cu) metal particles were prepared in ideal conditions, such as in ultra-high vacuum and in non-epitaxial growth, as fundamental experiments and analysis. The synthesized particles were observed and analyzed by high-resolution transmission electron microscopy (HRTEM) and tomography in TEM (TEM-Tomography) techniques. The shapes of the particles in morphological and atomic arrangements in both grains and grain boundaries were analyzed in detail as a function of particle sizes.

The Cu nanoparticles were prepared by pulsed laser deposition technique (PLD) with a KrF excimer laser ($\lambda=248$ nm) in an ultra-high vacuum (1×10^{-6} Pa order) chamber. This original approach is based on the deposition of Cu atoms from a vapour phase obtained by the interaction between the laser beam and the corresponding metallic targets. This technique provides a better control of parameters such as size, shape, local environment and structure of the nanoparticles [1] than conventional thermal evaporation technique. This control leads to a better understanding of the nanoparticles physical properties. The Cu atom vapour was deposited on amorphous carbon micro-grid. There are no interactions between Cu particles and amorphous carbon film, therefore, non-epitaxial growth of Cu particles and no stress generated among the Cu atoms.

The *JEM-2100F* 200 kV field emission TEM (FE-TEM) was used for high-resolution observations and tomograph auto-acquisition system installed in the TEM was used for data collection of the tilting series of the images. The 3D reconstruction and 3D data analysis were carried out by Tomography software (*TEMograph II*[®]).

The outer shapes (sizes, facet configurations) of the particles were analyzed by Tomography and atomic arrangements (grain sizes and orientations, grain boundaries) were analyzed by HRTEM image and nano-beam electron diffractions. Also selected area diffractograms were calculated by digital FFT processing from the HRTEM images..

Figure 1 shows a physiognomy of Cu single crystal, observed from various directions after 3D reconstruction. Data was recorded in tilting from -60° to $+60^\circ$ with 1 degree step. It has a polygonal column shaped in a size of 10 – 60 nm of the crystal.

Figure 2 shows a physiognomy of a multi-twined Cu crystal. A size of the particles was around 15 nm in diameter. A shape is sphere-like but there are a lot of facets on the surface. Figure 2(a) shows a high-resolution image. It provides projected information of grain boundaries in the lattice images. Figure 2(b) shows 3D reconstructed information. 3D information was reconstructed from TEM-image series acquired in tilting from -30° to $+65^\circ$ with 1° step. 3 dimensional structures of the grain boundaries in the crystal were analyzed from the information.

References

- [1] M. Cazayous, C. Langlois, T. Oikawa., C. Ricolleau and A. Sacuto; *Physical Review B* (2006) (in press).
 [2] V. Dureuil, C. Ricolleau, M. Gandais, C. Grigis, J.P. Lacharme, A. Naudon; *J. of Crystal Growth*, **233**, (2001) 737-748.



Fig 1. A physiognomy of Cu single crystal, observed from various directions after 3D reconstruction. TEM images were recorded in tilting from -60° to $+60^\circ$ with 1° step.

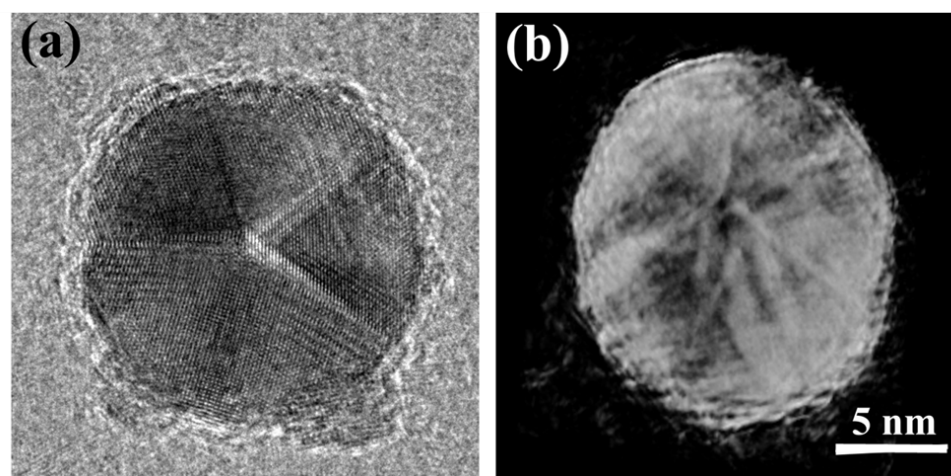


Fig. 2 A physiognomy of a multi-twined Cu crystal.

(a) A high-resolution observation showing grain boundaries in lattice images.

(b) Reconstructed 3D information from TEM images recorded in tilting from -30° to $+65^\circ$ with 1° step.

3-dimensional structures were obtained from the information.