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Transmission Electron Microscopy of Metal Nanodendrites Grown on Insulator Substrates by Electron-Beam-Induced Deposition

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Electron-beam-induced deposition (EBID) is one of the most promising techniques to fabricate small-sized structures on substrates. A variety of nanometer-sized structures have been fabricated [1-3]. Due to stable fabrication, electric conductive substrates are generally used [3] and compact structures are usually fabricated. On the other hand, little attention has been paid for the nanofabrication with insulator substrates. In the present study, metal (W, Pt) nanodendrites were fabricated on insulator substrates using an EBID process in transmission electron microscopes (TEMs). The as-fabricated microstructure was investigated in detail using conventional and high-resolution TEM (CTEM and HRTEM) and X-ray energy dispersive spectroscopy (EDS).

For the fabrication of W-nanodendrites, EBID was carried out in a JEM-ARM 1000 TEM. Accelerating voltages were 400, 600, 800, and 1000 kV. A gas introduction system including a nozzle and a reservoir of $W(CO)_6$ precursor was installed on this microscope. The nozzle was located near specimen within 2 mm. The inner diameter of the nozzle was smaller than 100μm. Crystalline SiO_2 TEM thin films were used as substrate. The fabrication of Pt-nanodendrites were carried out on crystalline Al_2O_3 substrates. $Me_3MeCpPt$ powder was a precursor. EBID experiments were done with a JEM-2010F TEM operated at 200 kV. All the experiments were carried out at room temperature.

Fig. 1 shows images of W-nanodendrites grown on SiO_2 substrate at 400 kV. The electron beam is defocused in size of about 2000 nm. The dendrites show a tendency to grow at the edge of the substrate in the irradiated area (Fig. 1a). They are grown self-standing at positions separated from each other in distance of several nanometers. Branches are observed at tips, which thickness is less than 3 nm (Fig. 1b). The diameter become thicker near the substrate, which imples that the deposition takes place at both tip and trunk part. This growth and morphology are attributed to a mechanism involving electric charge-up produced and accumulated on the surface of the substrate and tips of the deposits [4]. It is confirmed by EDS that W has been effectively deposited. Further characterization for the as-fabricated dendrites with HRTEM reveals that nano-sized W grains in bcc structure are contained in the dendrites. Fig. 2 shows HRTEM images of some branches of W-dendrites fabricated with various accelerating voltages. Lattice fringes, which are observed at the most places, indicates the formation of nano-sized crystals. The typical lattice spacing measured from images is 0.22 nm which is close to the lattice spacing, 0.224 nm, of {110} of bcc W crystals. Moreover, the inter-fringe angles of 60 degrees (grain A) and 90 degrees (grain B) agree well with the zone axis of [111] and [001] of bcc W structure, respectively. Furthermore, some lattice fringes shown by arrows in Fig. 2a are not clear because of the co-existence of amorphous state. Fig. 2b shows an image of W-dendrites grown at 1000 kV. Lattice fringes are clearly observed in almost all of the grains. The results indicated that the crystallinity of the dendrites was improved as increase in accelerating voltage. High-energy electron irradiation may enhance the diffusion of W atoms in the dendrites and promotes the crystallization of W grains.

With the EBID process, Pt depositions were also carried out on an Al_2O_3 substrate. Pt dendrites were observed to grow in the irradiated area, such as the case of W deposition. The dendrites consist of many nano-grains and amorphous state. Fig. 3 is an HRTEM image of tip-part of Pt-dendrite. The size of nano-grains is about 3 nm. Lattice fringes are observed clearly. Combined with diffraction pattern and EDS analyses, it is determined to be Pt crystal in fcc structure.

References

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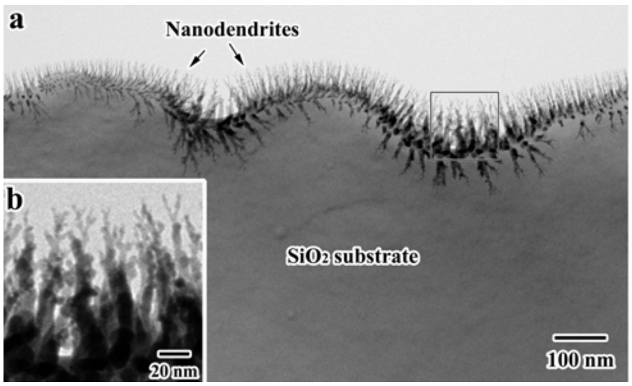

Fig. 1 (a) TEM image of W-nanodendrites grown on SiO_2 substrate at 400 kV to a fluence of 6.0× 10^{21} e cm^{-2}. (b) Enlargement of the square area in (a), showing the nanodendrites in more detail.

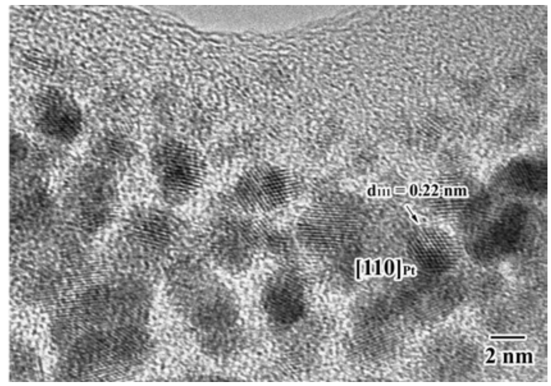

Fig. 3 HRTEM image of tip part of Pt-nanodendrite fabricated at 200 kV to a fluence of 2.6×10^{21} e cm^{-2}.

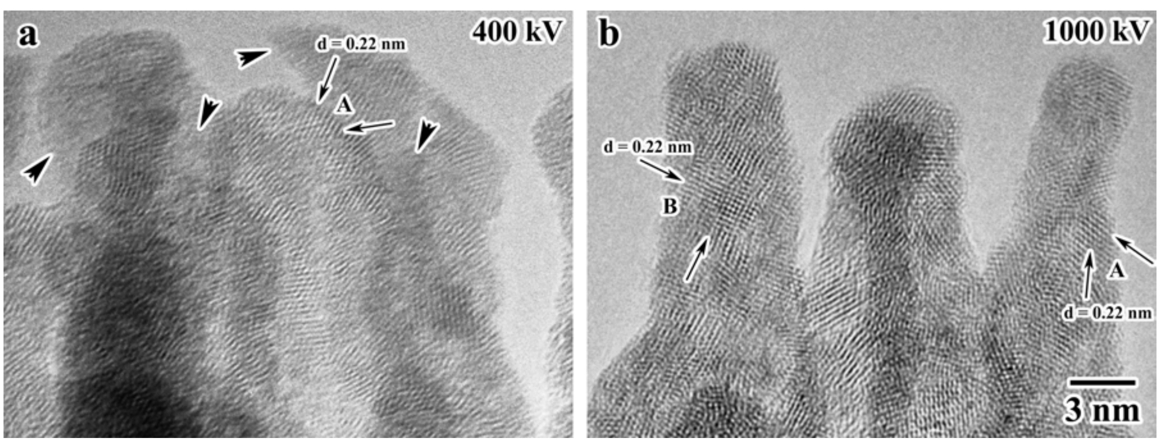

Fig. 2 HRTEM images of some branches of W-nanodendrites grown on SiO_2 substrate to a fluence of 6.0×10^{21} e cm^{-2} with various accelerating voltages. (a) 400 kV; (b) 1000 kV. Arrows indicate lattice fringes in grains A and B, which have inter-fringe angles of 60 (A) and 90 (B) degrees, respectively.

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