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TEM sample preparation of metal/alloy nanotubes

The most common sample preparation technique for TEM/STEM observations of one-dimensional (1D) nanostructures, such as nanotubes and/or nanorods, is to disperse them on a holey and/or lacy carbon coated grids. This simple method usually enables only observation along their length since 1D nanostructures deposit flat on the carbon film due to their large aspect ratio. For more complete structural and chemical characterization, however, one has to be able to prepare and analyze also cross-section thin slices of these 1D nanostructures as well. In present procedure we describe the preparation of Co-Pt alloy nanotubes thin slices for TEM/STEM observation by ion-milling method. The Co-Pt nanotubes were synthesized by electrophoretic deposition from solutions into polycarbonate-based templates and annealed in reducing atmosphere at 700^{o}C. Typical polycrystalline Co-Pt nanotubes are shown in Figure 1.

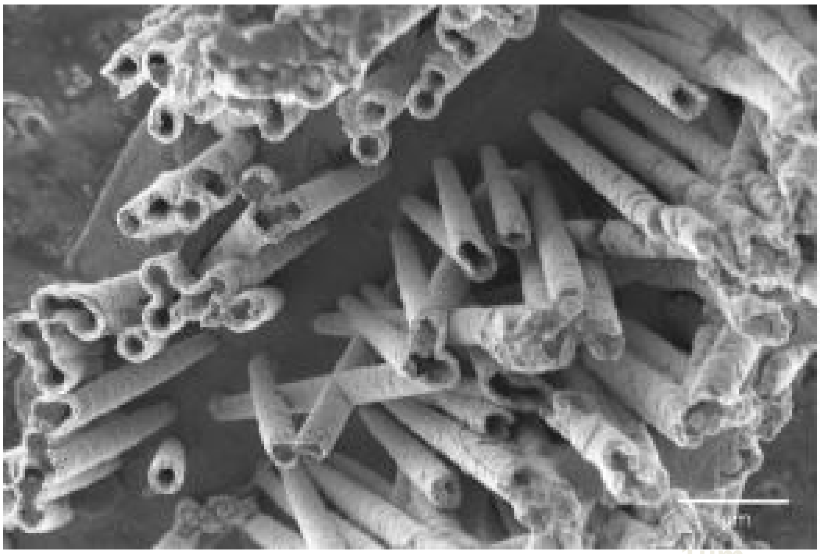

Figure 1. FEG-SEM micrograph of the as-deposited Co-Pt nanotubes.

For the preparation of Co-Pt nanotubes for TEM observation, the Co-Pt nanotubes were first mixed with an epoxy (G1) resin (Fig. 2a). It is advisable to ensure high nanotubes/epoxy volume ratio in the mixture. Then the mixture was deposited between two silicon substrates. The assembly was inserted under the press during polymerization at 130^{o}C in order to ensure thin and compact layer of polymerized nanotubes&epoxy mixture between both substrates. Cut slabs of the silicon/polymerized mixture/silicon sandwich were filled with the epoxy into a metal tube (Fig. 2b) and again polymerized on hot plate at 130^{o}C. The metal tube containing the specimen in cross-section geometry was sliced into app. 500 µm thick discs using a diamond wire saw (Fig. 2c). The specimen were afterwards grinded from both sides down to a thickness of app. 80 µm by using 20, 6, 4, 2 µm grit silicon carbide papers moistened with water in order to achieve a planar surface (Fig. 2d). After grinding, the specimens were mechanically dimpled. The specimen's thickness in the center of the disc was reduced from app. 80 µm to app. 15 µm (Fig. 2e). The final thinning can be achieved by ion-beam thinning techniques. In our case the sample was Ar+ ion-beam milled in an ion-miller (Bal-Tec RES 010) at 4 keV and 10^{o} incident angle (Fig. 2f). The TEM thin foil specimen after ion-milling is shown in an optical micrograph (Fig. 3).

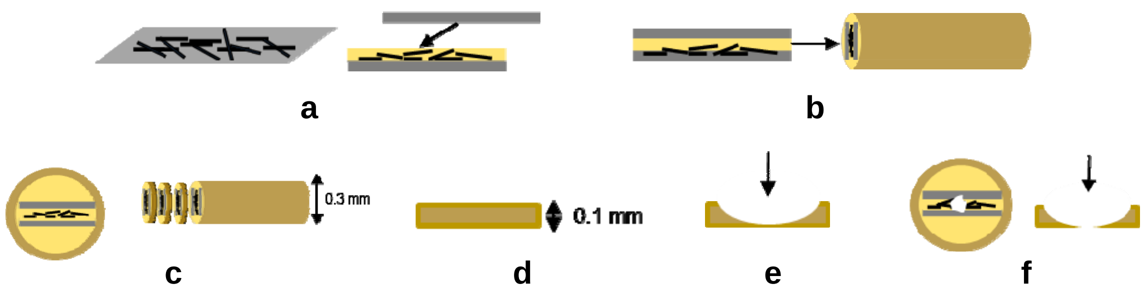

Figure 2. Schematic procedure showing different steps of Pt-Co nanotubes thin foils specimens preparation: (a) Mixing, (b) embedding into metal tube, (c) slicing, (d) grinding, (e) dimpling, and (f) ion-milling.

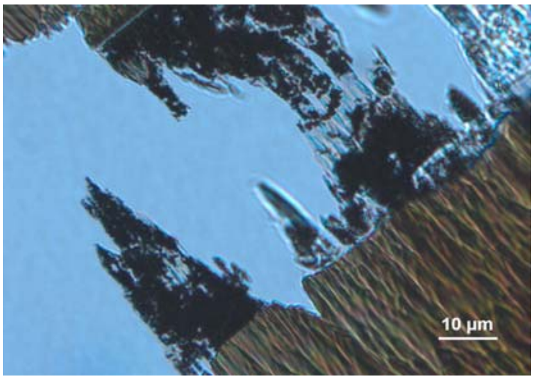

Figure 3. Optical micrograph of ion-milled thin foil specimen.

Due to random orientation of Co-Pt nanotubes embedded into the epoxy resin the regions showing longitudinal and cross-sections of the nanotubes can be readily found as shown in bright-field TEM micrographs (Fig. 4).

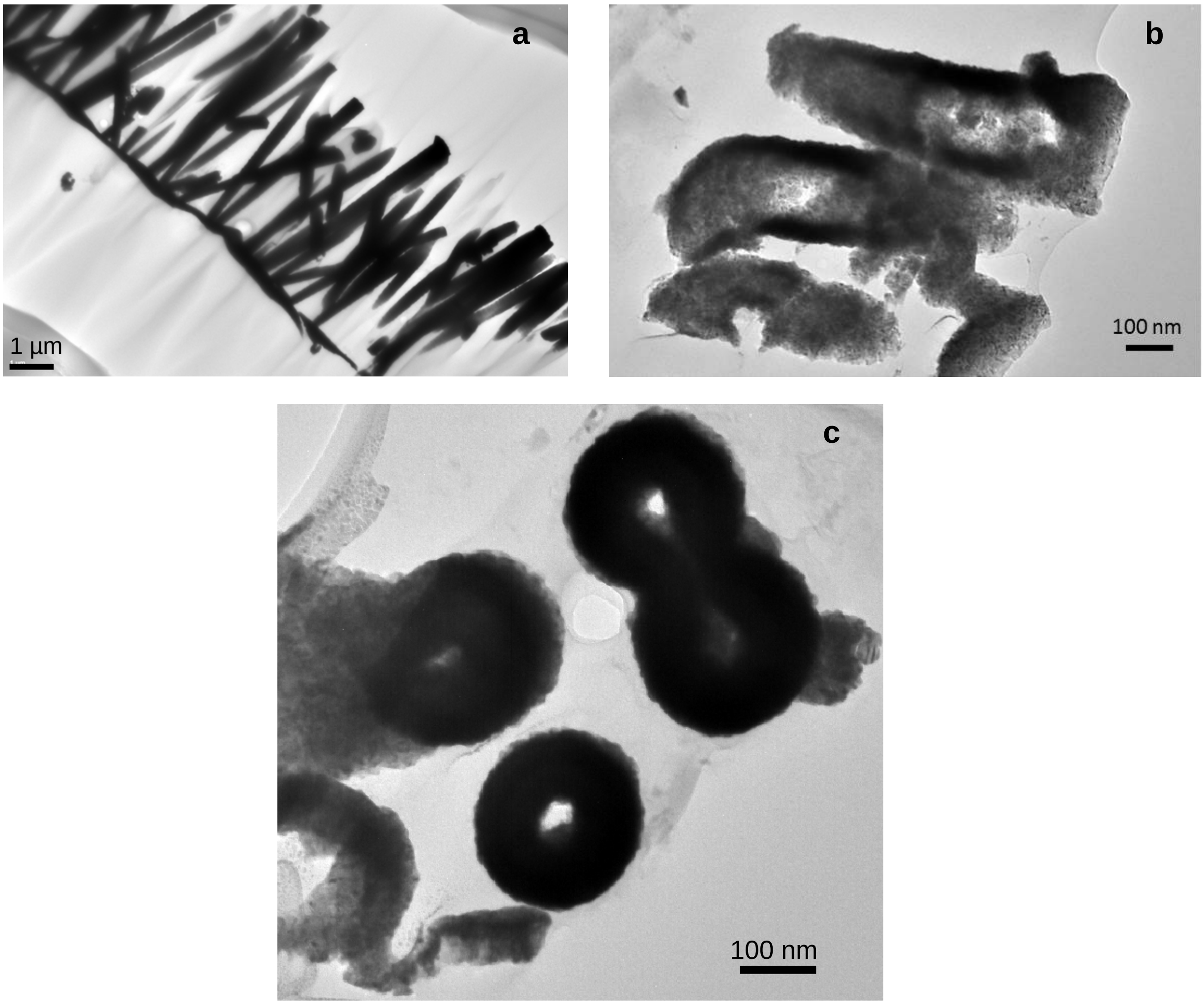

Figure 4. TEM BF micrographs of (a) longitudinal view of Co-Pt nanotubes within epoxy, (b) out of epoxy, (c) TEM BF micrographs of Co-Pt nanotubes cross-sections.

The described procedure can be implemented not only to prepare various cross-section views of metal and/or alloy nanotubes but can be also used to prepare thin foil specimens of different nanotubes/nanorods materials.