A new method for mapping the three-dimensional atomic distribution within nanoparticles by atom probe tomography

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Analyzing nanoparticles has been an emerging but also highly challenging field in atom probe tomography (APT). In this work, we present a new method for the preparation of APT specimens from metallic nanoparticles of less than 10 nm in size. This method is based on electrophoresis of nanoparticles on a substrate followed by electroplating of a metallic layer. Transmission electron microscopy (TEM) confirms that particle shape and size are well preserved after these two process steps. APT specimens can be routinely prepared from the deposited nanoparticle/metal films using focused-ion-beam (FIB) milling. Correlative TEM/APT analyses on APT specimens ad resharpening by FIB prior to APT analyses were found to increase the chances of detecting the nanoparticles within the volume probed by APT. In addition, such correlative analyses help to examine the size and shape of nanoparticles and optimize APT data reconstruction. In addition to the constituent elements of the nanoparticles, we were able to map the distribution of surfactants used during synthesis and deposition of the nanoparticles.