Nanoparticle Sample Preparation for Atom Probe Tomography: Electroplating Fixation

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Atom probe tomography (APT) is an enabling characterization tool for mapping the elemental distribution in nanostructured materials due to its unique combination of three-dimensional (3D) capability, sub-nanometer spatial resolution, and ppm-level detection sensitivity for all elements irrespective of their mass.[1] But in APT, a needle-shaped specimen is the primary optic and as such specimen preparation forms a critical step in a successful atom probe tomography.[2]

For nanoparticle characterization, some of the key questions which have remained mostly unanswered with respect to nanoparticles are: What are the exact chemical compositions of nanoparticles? What is the exact three-dimensional atomic-scale distribution of elements within nanoparticles? Does elemental segregation or partitioning prevail? Are any impurities incorporated into nanoparticles during their synthesis? Addressing these questions is of great importance, for instance when it comes to designing nanoparticles for catalytic applications. Since chemical composition, impurities and elemental distribution may affect lattice strains, electronic band structure and eventually catalytic activity and selectivity of nanoparticles, three-dimensional sub-nanometer resolution elemental mapping is of key interest.[3,4]

Yet only a limited number of APT results has been reported for nanoparticle systems, and this is essentially due to the fact that APT sample preparation from nanoparticles is highly challenging. Several methods have been proposed for the investigation of nanoparticles by means of APT. Although the feasibility of these techniques has been demonstrated in specific cases, none of them seem to have general applicability to all nanoparticle systems. Two major issues still pose great challenges for most of the sample preparation methods developed so far. First, nanoparticles must be well embedded in a matrix without any residual nano- or micro-voids. This is because voids can cause premature fracture during APT analyses and artifacts in APT data reconstruction. Second, the structure and composition of the nanoparticles should be preserved as good as possible during the embedding process in order to acquire meaningful data that can be related to the original as-synthesized nanoparticles.

Herein, we demonstrate the development of preparing specimens from freestanding nanoparticles using electroplating method.[5] The method developed yields reliable APT data for both noble and metal-oxide nanoparticles. We expect the method contributes to an improved general understanding of the structure-composition-property relationship of nanoparticles.
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Figure 1. Schematic diagram of nanoparticle characterization using atom probe tomography (APT).