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Novel Approaches for Electron Tomography to Investigate the Structure and Stability of Nanomaterials in 3 Dimensions.

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Nanomaterials are important for a wide range of applications because of their unique properties, which are strongly connected to their three-dimensional (3D) structure. Electron tomography has therefore been used in an increasing number of studies. Most of these investigations resulted in 3D reconstructions with a resolution at the nanometer scale, but also atomic resolution was achieved in 3D. However, the increasing complexity of nanomaterials has driven the development of even more advanced 3D characterization techniques, which will be discussed in this contribution.

For example, 3D characterization of structural defects in nanoparticles by transmission electron microscopy is far from straightforward since the presence of diffraction contrast in a tilt series of images violates the projection requirement for tomography. However, being able to visualize defects is of great importance to understand e.g. the initial growth of metallic nanoparticles or the effect of pulsed laser irradiation on the crystal structure. By simultaneous acquisition of tilt series using different annular detectors, we were able to visualize both the morphology and the defect structure of several types of nanostructures (Figure 1) [1,2]. Moreover, also a 3D characterization at the atomic scale could be performed. Such studies are of great importance to link the structure to the specific functionalities of the nanomaterials.

In order to preserve the carefully designed morphologies and functionalities, understanding the stability of nanomaterials during application is of equal importance. It is hereby important to note that most electron tomography investigations have been performed at the conventional conditions of an electron microscope. An emerging challenge is therefore to fully understand the connection between the 3D structure and properties under realistic conditions, including high temperatures as well as in the presence of liquids and gases. Therefore, innovative methodologies are required to track the fast 3D changes of nanomaterials that occur under such conditions.

Recently, we proposed an acquisition approach where a tilt series of 2D HAADF-STEM projection images is acquired within a few minutes. By continuously tilting the holder and simultaneously acquiring projection images while focusing and tracking the particle, we were able to reduce the total acquisition time for a tilt series by a factor of ten [3]. Moreover, a new approach was developed to compute high quality 2D virtual slices through nanoparticles in approximately 60 ms time. This technique drastically improves the efficiency of 3D characterization of nanomaterials by TEM. It enables explorative imaging and provides valuable information to dynamically adjust the acquisition parameters during an electron tomography experiment [4].

In this manner, we were able to study the 3D morphological evolution of anisotropic Au (Figure 2) and AuPd nanoparticles as a function of both heating time and temperature [3,5]. Moreover, we measured the elemental diffusion dynamics of individual anisotropic Au-Ag nanoparticles in 3D [6]. We conclude that for a given composition, the shape of the nanoparticle does not influence the alloying process significantly. Based on our analysis, it is clear that interdiffusion of metals at the nanoscale is more complex than predicted by simple Fickian diffusion and that other factors such as surface diffusion need to be taken into account [7].

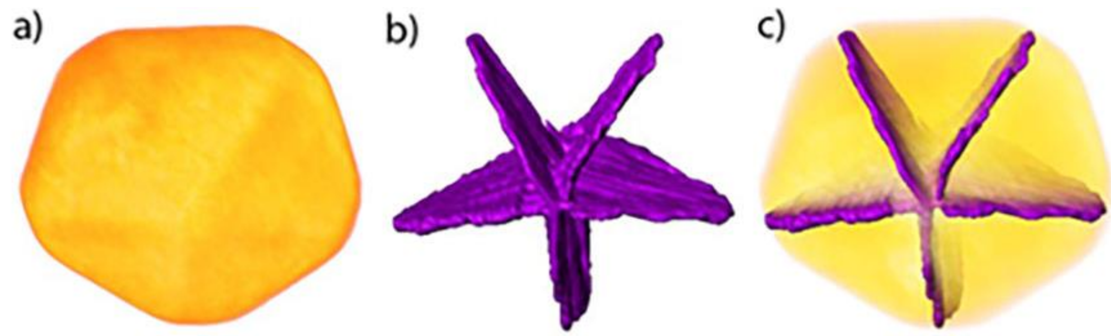

Figure 1. 3D visualization of the HAADF-STEM reconstruction (a) and the twin planes segmented from the LAADF-STEM reconstruction (b) of an Au decahedron. Both volumes are superimposed to evaluate the position of the twin planes in the correct volume (c).

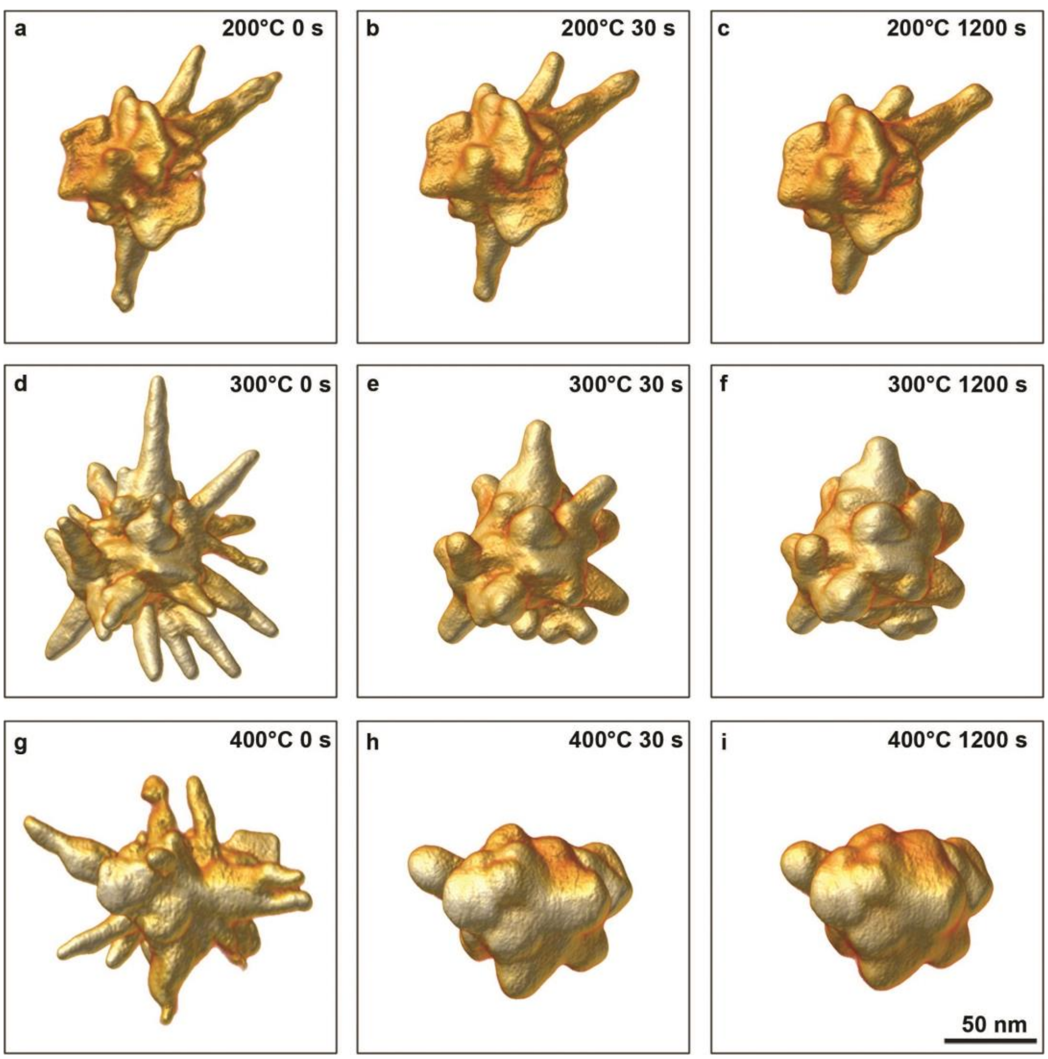

Figure 2. 3D visualizations, using fast tomography, of an Au NS before (a) and after heating for 30 s (b) and 1200 s (c) at 200 °C. 3D visualizations for the same time steps are shown for 300 °C (d-f) and 400 °C (g-i).

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