Advanced sample preparation for quantitative TEM down to the atomic level

Butz, B.[1]

[1] Micro- and Nanoanalytics and –Tomography Group & Micro- and Nanoanalytics Facility (MNaF), University of Siegen, 57076 Siegen, Germany

To make use of nowadays exceptional TEM capabilities, researchers all over the world need samples of exceptional quality. Although, many of those people spend a lot of effort on sample preparation, systematic publications are still rare. By this contribution, we recap highly versatile techniques for conventional TEM sample preparation as well as the preparation/handling of nanoparticulate materials:

1) Functional properties of a Nanoparticles (NP) are commonly derived from a large ensemble of those because the correlation of the nanoscopic characteristics of an individual NP with its functional properties is hardly accessible. However, for many advanced materials such direct correlation becomes more important as the level of functionality increases. Here, we present a versatile and simple method to transfer selected NP after inspection in the TEM from a common TEM support grid onto a tailored tip (metal, glass, or other) for further characterization by, e.g., 360° electron tomography, atom probe tomography, nanoCT or optical measurements (Fig. 1). A conventional scanning electron microscope equipped with a micromanipulator is needed only. The technique is damage- and contamination-free and covers particles in the size range of a few m down to a few 10 nm.

Ref. 1: T. Przybilla, B. Apeleo-Zubiri, A. Beltran, B. Butz, A. Machoke, A. Inayat, M. Distaso, W. Peukert, W. Schwieger, E. Spiecker, Transfer of individual micro- & nanoparticles for high-precision 3D analysis using 360° electron tomography, Small Methods (2017), DOI: 10.1002/smtd.201700276

2) State-of-the-art TEM studies down to the atomic scale necessitate high-quality samples with exceptional surface quality. Although many researchers prefer FIB-lamellae, conventionally prepared samples provide by far larger electron transparent areas and may have better surface quality. However, the conventional cross-sectional preparation of complex devices and thin-films or the preparation of bulk materials often fails, because optimal Ar-ion milling parameters for the most critical preparation step are unclear. This study (Fig. 2) focusses on the optimization of Ar-ion milling parameters to prepare TEM samples of complex materials and layered structures with well-defined symmetry and exceptional quality. It includes hands-on experience and a milling scheme to generate plane-parallel as well as wedge-shaped samples systematically.

Ref. 2: L. Dieterle, B. Butz, E. Müller, Optimized Ar^+-ion milling procedure for TEM cross-section sample preparation, Ultramicroscopy 111 (2011), 1636 - 1644, DOI: 10.1016/j.ultramic.2011.08.014

3) For the comprehensive characterization of anisotropic NP, cross-sectional investigation on the atomic scale by analytical and high-resolution TEM is indispensable to gain information on structure and chemistry along all important projections. Here, we present a FIB method for site- and orientation-specific cross-sectioning of arbitrary nanoparticles that are dispersed onto a substrate. By adopting a shadow geometry originally developed for thin sensitive films, protection of the specimen by a platinum layer is avoided. This enables simultaneous observation by the electron beam and ion-beam sectioning of an individually selected particle with excellent accuracy on the nanometer scale.

Ref. 3: B.F. Vieweg, B. Butz, W. Peukert, R.N. Klupp-Taylor, E. Spiecker, TEM preparation method for site- and orientation-specific sectioning of individual anisotropic nanoparticles based on shadow-FIB geometry, Ultramicroscopy 113 (2012), 165 - 170, DOI: 10.1016/j.ultramic.2011.11.015

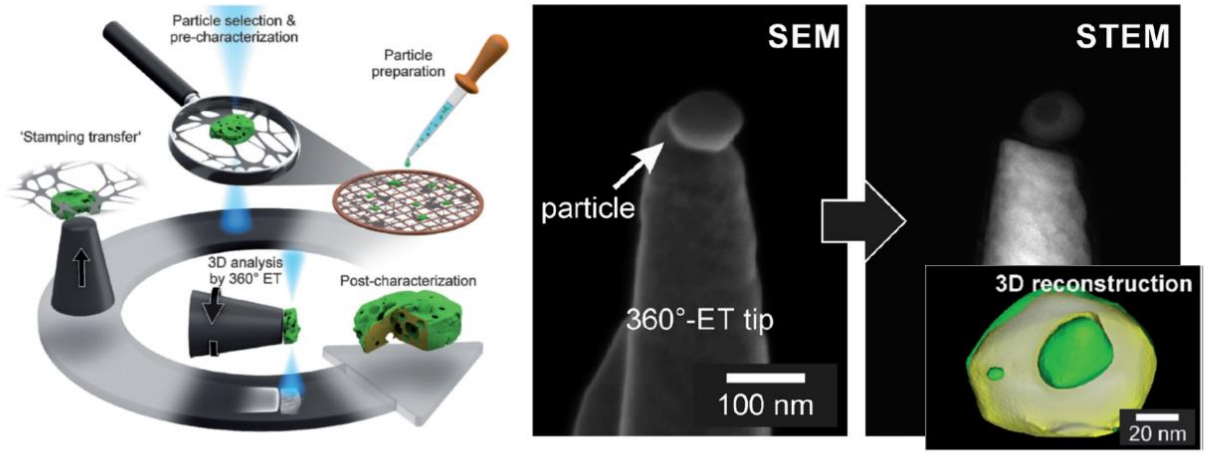

Figure 1: left: transfer scheme for individual NP, right: individual hematite NP on tapered tomography tip after transfer plus 360° tomography reconstruction. (Ref. 1)

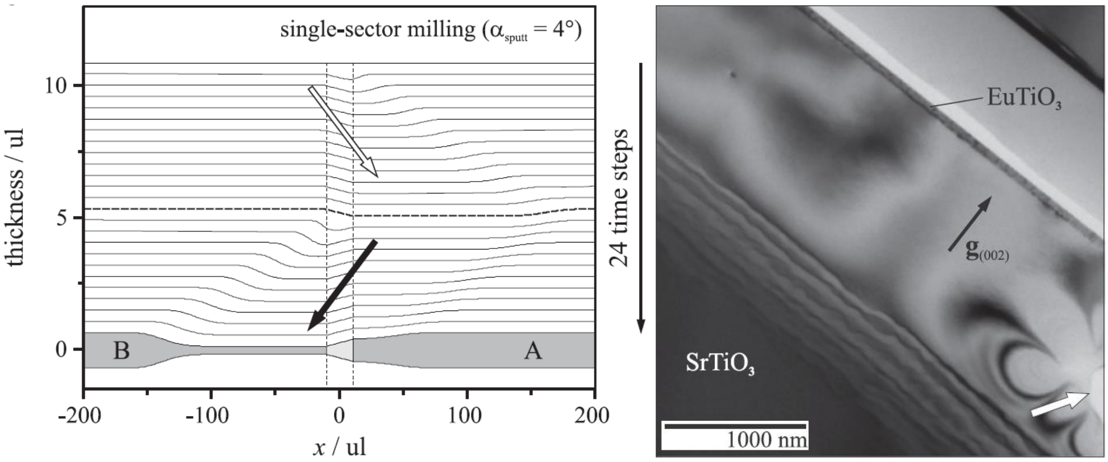

Figure 2: left: sequential Ar-ion milling of plane-parallel TEM sample, right: exemplary sample of $EuTiO_3$ film on STO substrate (prepared using Gatan PIPS). (Ref. 2)