Transmission Electron Microscopy

Transmission electron microscopy (TEM) enables characterization of powders and thin films (which can be prepared in a target preparation from bulk materials) by direct imaging with up to atomic resolution. The image information can be locally correlated with spectroscopic techniques (EELS/EFTEM and EDX) to provide semi-quantitative elemental composition/maps with sub-nanometer resolution. All of these techniques can also be performed in-situ, e.g. during heating, electrical biasing or straining to directly correlate structural changes and materials properties. For complex three-dimensional structures, electron tomography can be used to generate a 3D representation of the material with a spatial resolution of 1–2 nm.

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Features

- FEI Titan 80–300 (aberration corrected TEM)
- Resolution:
  - 0.08 nm information limit TEM
  - 0.14 nm resolution in STEM
  - 0.7 eV energy resolution EELS
- Imaging and Analysis Techniques:
  - BF-TEM, aberration corrected HRTEM
  - HAADF-STEM, HRSTEM
  - EFTEM, EELS, EDX
  - (S)TEM tomography
  - electron diffraction, electron precession
  - orientation mapping
  - Lorentz imaging
  - low-dose techniques & cryo imaging
- In-situ Techniques:
  - Heating (Protochips Aduro: RT-1200 ºC; Gatan 652: RT-800 ºC)
  - Cooling (Gatan 915: LN2-80 ºC)
  - Straining (Hysitron Picoindenter PI 95 and Gatan 654)
  - Electrical Biasing (Protochips Aduro)
  - Electro chemistry (Protochips Poseidon 500)
- Sample preparation:
  - Thin films or nano powders can be directly analyzed without additional preparation
  - Target preparation by FIB lift-out (for details see FIB description) with final polishing by low-voltage Argon ion beam (Fischione 1040 NanoMill)
  - Electro polishing
  - Classical preparation by cutting, grinding, argon ion milling or microtomy

Limitations/constraints

- Sample has to be a solid at LN_2 temperatures and stable under high vacuum conditions
- Maximum sample thickness: 10–2000 nm (depending on resolution and technique)
- Depending on the structure and chemical composition, the sample might be sensitive to the electron beam resulting in changes during analysis
- Except in tomography, TEM always provides an image/analysis of the projected structure of a sample
- H, He und Li cannot be detected by our analytical techniques

Typical results

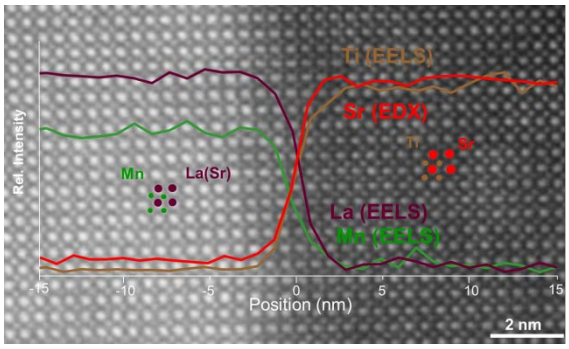

Fig. 1: HAADF-STEM image (filtered by NAD) of a $La_{1-x}Sr_xMnO_3/SrTiO_3$ interface with the individual atomic columns well resolved across the interface. Overlaid is an EELS/EDX intensity profile across this interface. P.M. Leufke and D. Wang et al., Thin solid films, 2012, 520, 5521-5527.

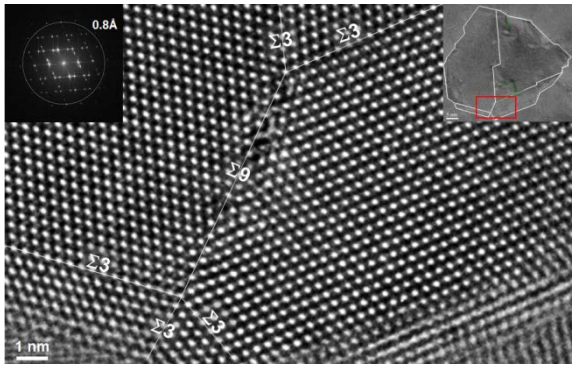

Fig. 2: Atomic resolution TEM image of a triple and a quadruple line at the interface between Σ3 boundaries and a Σ9 boundary in nanocrystalline palladium. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

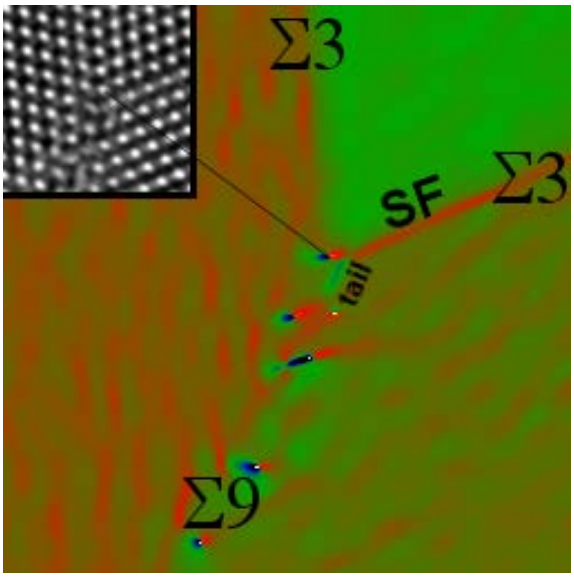

Fig. 3: Geometric phase analysis reveals the local strain distribution around the triple line in the image above. H. Rösner and C. Kübel et al., Acta Mat., 2011, 59, 7380-7387.

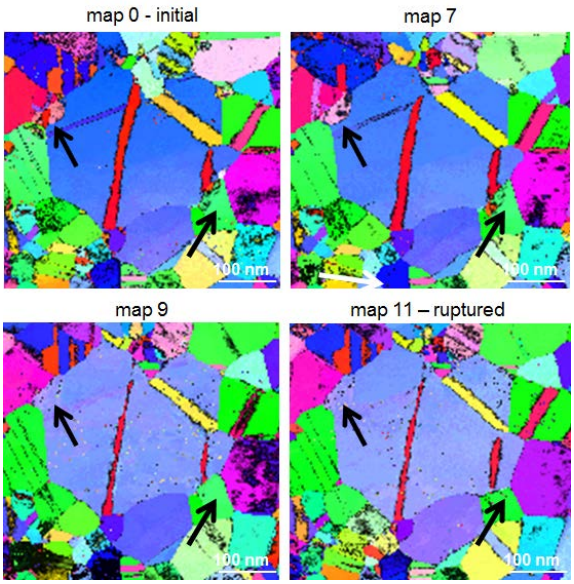

Fig. 4: In-situ orientation mapping (different color correspond to different crystal orientations) of the grain structure changes in nanocrystalline gold during straining – selected images of the straining series showing anomalous grain growth. A. Kobler and C. Kübel et al., Ultramicroscopy, 2013, 128, 68-81.

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Typical results *(continued)*

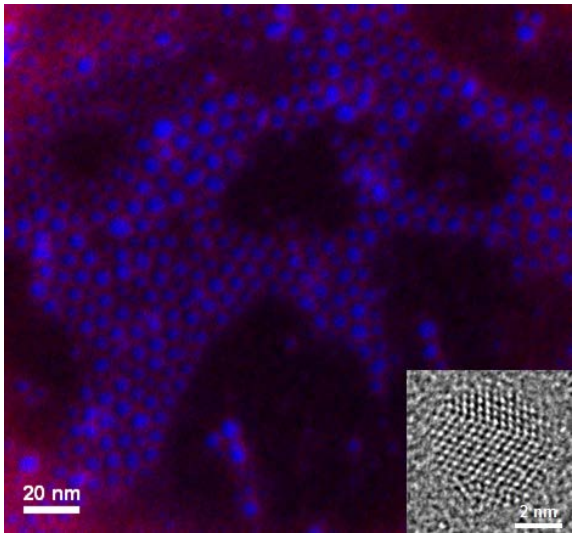

Fig. 5: EFTEM mapping (Si-blue, C-red) and HRTEM image of nanocrystalline silicon particles with a covalently bound C18 shell. The EFTEM maps reveal the ~1.2 nm wide carbon shell around the silicon core. Sample provided by G. Ozin, University of Toronto.

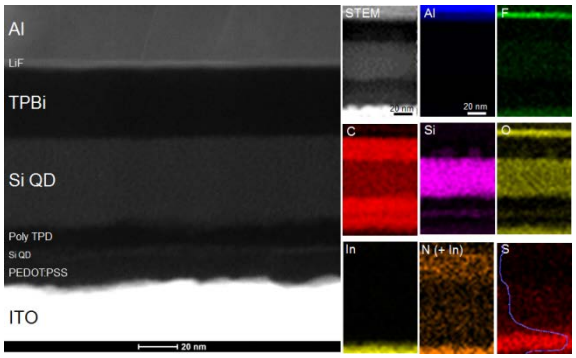

Fig. 6: HAADF-STEM image with EDX compositional mapping of the different layers in a silicon quantum dot based organic LED (SiLED). F. Maier-Flaig and C. Kübel et al., Nano Letters, 2013, online; DOI: 10.1021/nl400975u.

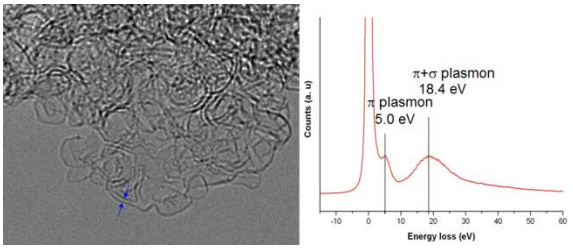

Fig. 7: HRTEM image of nano graphene with the corresponding low-loss EELS spectrum showing the characteristic π and π+σ plasmon losses. J. Biener and D. Wang et al., Adv. Mater. 2012, 24, 5083–5087.

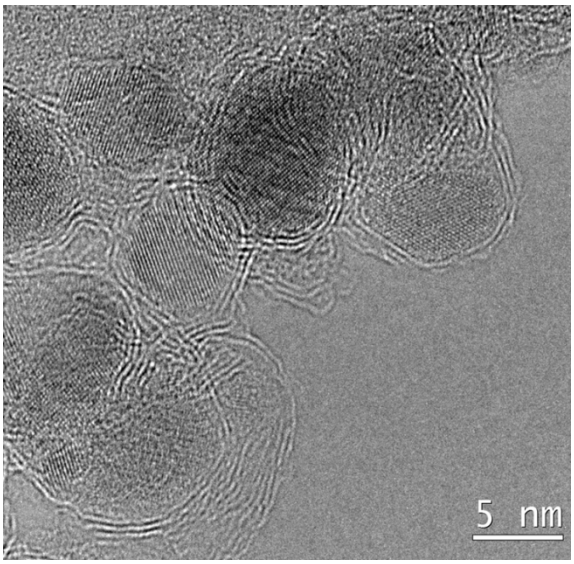

Fig. 8: HRTEM image of a Fe/LiF/C anode for lithium ion batteries revealing α-iron nanoparticles each surrounded by a few graphene layers. R. Prakash and C. Kübel et al., J. Power Sources, 2011, 196, 5936-5944.

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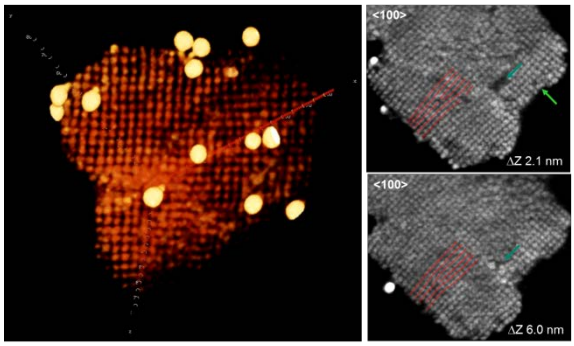

Fig. 9: Electron tomographic reconstruction of a self-assembled CdS nano cluster superlattice (with additional 5 nm gold particles in yellow). The two digital slices, one unit cell apart, show a single vacancy, an extended vacancy and dislocations in 3D. T. Levchenko and C. Kübel et al., Chem. Eur. J., 2011, 17, 14394-14398.

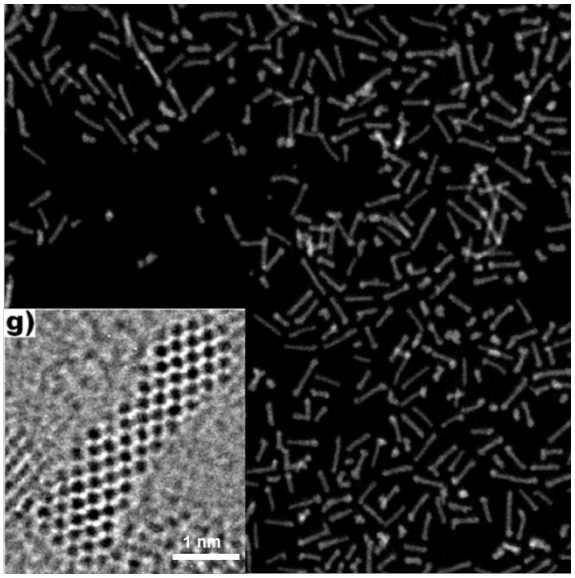

Fig. 10: HAADF-STEM and HRTEM imaging of uniform ThO_2 nanorods. D. Hudry and E. Courtois et al., Chem. Eur. J, 2013, 19(17), 5297–5305.

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