Research Infrastructure QualityNano

Electron Microscopy and Focussed Ion Beam Nanostructuring

Technology:
Scanning Electron Microscopy (SEM)
Transmission Electron Microscopy (TEM)
Focussed Ion Beam (FIB) Nanostructuring

Equipment:

- ZEISS922 Omega
- Field-emission (FE)-SEM FEI Quanta 650 ESEM
- FE-SEM ZEISS LEO 1530
- FIB FEI Strata 400

Category:
C. Particle Characterisation in- and ex-situ

Institute:
KIT

Location:
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Short technology description/Overview:

Different electron-microscopic techniques, including scanning electron microscopy (SEM), transmission electron microscopy (TEM) as well as scanning TEM (STEM), are applied to characterise the properties of nanoparticles and agglomerates thereof. Detailed information about the particle size and surface topography is obtained by field-emission SEM with a lateral resolution down to approximately 1 nm, whereas the crystal structure can be elucidated by electron diffraction in TEM and direct imaging at atomic resolution via high-resolution TEM (HRTEM). Both SEM and TEM investigations are combined with energy dispersive X-ray spectroscopy (EDXS) to reveal the chemical composition of the material in a quantitative manner, which can also be characterised along a line (line profiling) and two-dimensionally (X-ray mapping). An additional analytical tool for TEM inspection of particles at a high spatial resolution in the order of 1 nm is electron energy loss spectroscopy (EELS), which allows their quantitative element analysis, but also chemical-bond analyses by characterising energy-loss near-edge structures (ELNES). Element distributions can be imaged through EELS line profiles and by energy-filtered TEM (EFTEM).

A target preparation of nanoparticle assemblies is facilitated by combined focused ion beam (FIB) milling and SEM imaging, allowing for example a stepwise sectioning of single particles. These experiments can be supplemented by in-situ EDXS analyses and low-kV HAADF (high-angle annular dark-field) STEM imaging, yielding strong atomic-number contrast.

Main Features (Equipment Capabilities):

FE-SEM ZEISS LEO 1530 and
FE SEM FEI Quanta 650 ESEM

- Accelerating voltages from 1 kV to 30 kV
- Secondary-electron (SE) imaging with optimum resolution of ~1 nm
- Backscattered electron (BSE) imaging with optimum resolution of~ 2.5 nm
- EDXS analysis (detection of elements with Z ≥ 5)
- Low-vacuum operation up to 4000 Pa

Liquid nitrogen cooled stage

Dual-beam FIB FEI Strata 400

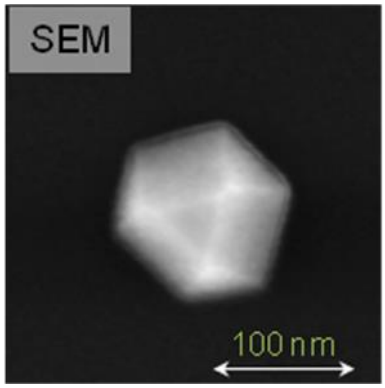
- Materials milling with Ga+ ions in the energy range from 1 to 30 kV - minimum structure size of ~10 nm
- Preparation and lift-out of TEM lamellae
- In-lens SE imaging (about 1 nm resolution with electron excitation), < 1 nm resolution in HAADF STEM mode
- Material deposition (Pt, W) with ion or electron beam

EDX spectrometer with silicon-drift detector (SDD), element detection for Z ≥ 5

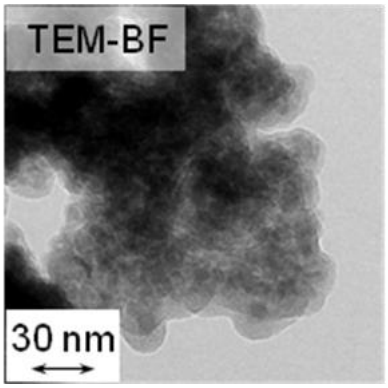
Limitations / constrains

For investigations, powder material can be directly put on an electron-conducting support. For TEM/STEM characterization the particle size should be smaller than 200 nm. All samples have to be stable under high-vacuum conditions (except for SEM imaging with the FEI Quanta ESEM). Depending on the composition, the sample might be damaged by the electron beam (this holds especially organic material and polymers). EDXS allows detection of elements with atomic number $Z \geq 5$ (EELS is able to detect $Z \geq 3$).

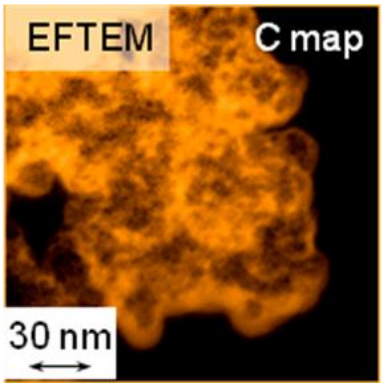
Typical structures & designs



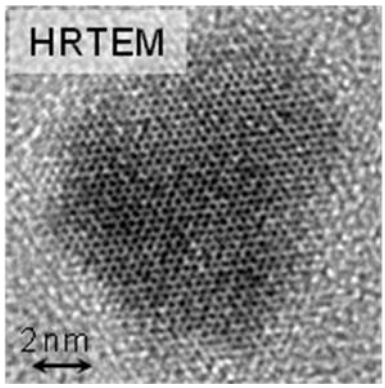
Secondary-electron image of a single Pt particle



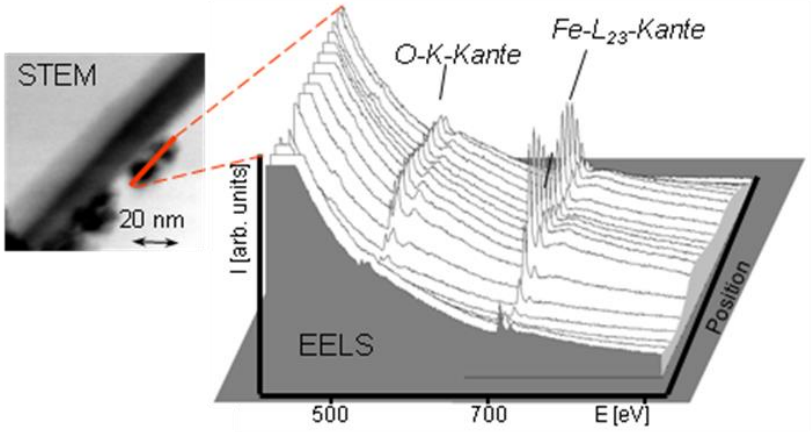
TEM bright-field image of a mixture of Fe_2O_3 nanoparticles in soot



Carbon map of a Fe_2O_3 nanoparticles/ soot mixture obtained by EFTEM



HRTEM image of an individual ZnO particle with about 10 nm size



Series of EEL spectra across two single Fe_2O_3 particles embedded in soot

Any further Information: