3D characterization of the structure and stability of complex nanomaterials using electron microscopy

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Electron tomography (ET) is an outstanding technique for investigating nanomaterials [1]. There are however, still challenges that require further progress. For example, the time-consuming nature of ET makes it challenging to collect statistically representative data, necessary to establish structure-property connections. In additional to “fast” electron tomography, where we reduce the acquisition time for electron tomography from approximately 1 hour to a few minutes [2], we explored imaging by "secondary electron electron beam induced current" or "SEEBIC". SEEBIC enables a significant gain in data collection efficiency in comparison to conventional ET while providing necessary topographical information with superior resolution in comparison to scanning electron microscopy (Figure 1) [3]. Another challenge is related to the conventional conditions of a TEM: ultrahigh vacuum and room temperature. We therefore combined dedicated in situ holders with optimised acquisition schemes and 3D reconstruction algorithms. We could investigate the organisation of ligands at the surface of nanoparticles and characterise the organisation of nanoparticles in an assembly in liquid. Moreover, the stability of gold nanoparticles, exposed to relevant environmental conditions, such as high temperature could be determined [4,5]. Finally, by combining aberration corrected electron microscopy with a quantitative interpretation and modelling approaches, we can perform quantitative measurements of the coordination numbers of the surface atoms of catalytic nanoparticles at high temperatures and in gas [6].

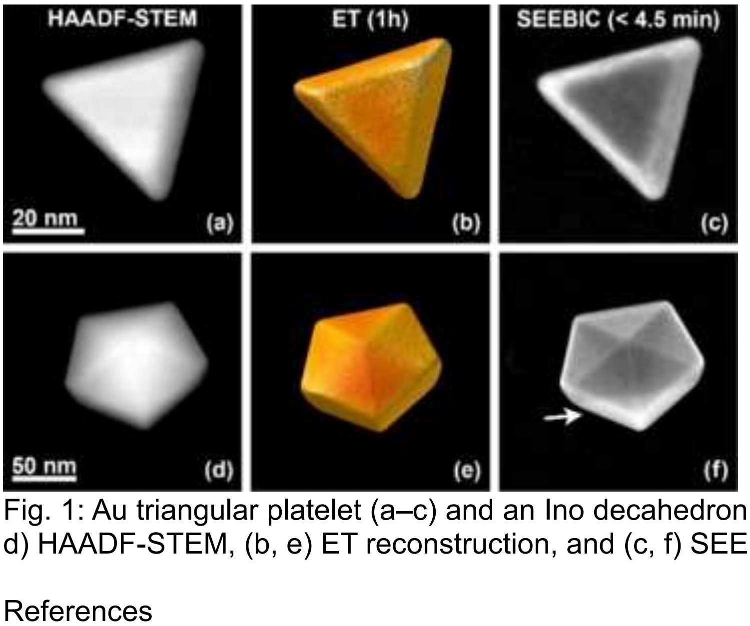

Fig. 1: Au triangular platelet (a–c) and an Ino decahedron (d–f) imaged in different modes: (a, d) HAADF-STEM, (b, e) ET reconstruction, and (c, f) SEEBIC. Reprinted from [4]

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Interpreting short range order with electron microscopy

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This presentation will describe our recent results utilizing energy filtered diffraction, 4D-STEM and in situ TEM mechanical testing to provide insight into the structure and role of short range order (SRO) in materials. Examples will be presented from structural alloys such as Ti-6Al [1] and the CrCoNi medium entropy alloy [2-5], as well as semiconductors such as a SiGeSn/GeSn multilayer [6]. We will show how coordinated computational methods to simulate diffraction patterns for direct comparison with experimental measurements can help to interpret the ambiguous diffuse intensities in both dilute alloys and concentrated solid solutions. Lastly, we will discuss both the strengths and limitations of electron microscopy methods for analyzing SRO in these systems.

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Recent advances and applications of magnetic-field-free atomic-resolution electron microscopy

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In atomic-resolution scanning transmission electron microscopy (STEM), atomic-resolution observation and analysis of magnetic materials is normally very difficult since high perpendicular magnetic field is always exerted on the samples inside the magnetic objective lens. In recent years, we have succeeded in developing a magnetic objective lens system that realizes a magnetic field free environment at the specimen position [1]. Using this new objective lens system combined with a higher-order aberration corrector, atomic-resolution imaging of magnetic materials is realized. This electron microscope (Magnetic-field-free Atomic Resolution STEM: MARS) is now used for research and development of many magnetic materials and devices [2,3]. By combining MARS with differential phase contrast (DPC) imaging, it is shown that real-space visualization of atomic-scale magnetic fields in an antiferromagnet is possible [4]. In addition, newly developed tilt-scan averaging system for DPC imaging is installed in MARS [5,6], which enables local electromagnetic field imaging at heterointerfaces and grain boundaries by minimizing diffraction contrast [7-9]. In this presentation, several material applications of MARS will be reported. In addition, I will also report on the progress of our new development project, MAgnetic field-free Cryogenic Atomic resoLUtion electron microscope: MACALU. In MACALU project, we aim to install liquid He cooling stage in the magnetic field free objective lens. The base microscope is built for 300kV accelerating voltage. We obtained 78 pm spatial resolution for the base microscope under magnetic field free sample condition.

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MILESTONES IN HEXAPOLE ABERRATION CORRECTOR DEVELOPMENT

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After 30 years of successful hardware aberration correction in transmission electron microscopy, two of the inventors celebrated their 75th anniversary: Max. Haider and Ondrej Krivanek. They both achieved first resolution improvements with a two-hexapole design in TEM (CETCOR) [1] and a quadrupole-octupole design in STEM [2], respectively. This is a perfect moment to review some important development steps since then. Here, we will concentrate on the lineage of the hexapole aberration corrector.

For STEM, the two-hexapole design was very successfully used as well and became well known as CESCOR [3]. A careful redesign led to the advanced two-hexapole corrector (DCOR/ASCOR) with full fourth-order correction and effectively vanishing fifth-order aberrations [4]. In up-to-date microscopes with cold field emission guns (CFEG) the unavoidable three-lobe aberration in sixth order D6 becomes visible but is usually not limiting the STEM probe size [5]. Recently, a three-hexapole design (LASCOR) has been introduced which is by design free of sixth-order aberrations and allows to double the semi-aperture angle compared to ASCOR [6]. As one application this allows tilting of a high-resolution STEM probe above the corrector, e.g., for zone axis alignment of the specimen [7]. In combination with monochromated illumination we expect better spatial and depth resolution.

For TEM, the three-hexapole corrector BCOR was introduced first. It allows for off-axial aberration correction up to third order including azimuthal off-axial coma B2Gy [8]. Later on, the advanced two-hexapole correctors (ATCOR/CETCORPRIME) were developed in order to avoid limited phase contrast transfer due to six-fold astigmatism A5 in CFEG-equipped microscopes [9]. At the same time, diffraction distortions are largely reduced as well as fourth-order off-axial aberrations. In third order solely the azimuthal off-axial coma remains. Second-order off-axial aberrations are correctable.

As the first LASCORs and the first advanced two-hexapole TEM correctors arrive at the customer sites, we want to use the opportunity to review the optical improvements compared to the previous designs.

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Atomic spatial resolution in vibrational and magnetic properties from theoretical perspective

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Since the beam electrons interact strongly with the fields in the sample, this gives rise to multiple scatterings and consequently dynamical effects in the electron diffraction [1], which complicate the interpretation of experiments. For this reason, simulations play an important role in the applications of STEM-EELS, aiding the interpretation as well as optimization of experimental conditions.

Recent developments in STEM-EELS instrumentation have allowed to explore spectral features with energy resolution below 10 meV [2], while operating at atomic resolution and having just a counting noise [3]. Vastly improved quantitativeness has enabled to explore fine spectroscopic signals, such as circular and linear dichroism [4,5]. High energy resolution gave access to atomic-scale studies of atomic vibrations, and recently also to magnons [6]. These instrumental advances have been recently complemented with cooling of the sample down to 10K region [7], while maintaining spatial and energy resolution, extending the possibilities to study, e.g., phase transitions down to low temperatures.

Opening of new areas to be explored by STEM-EELS most often calls for widening the theoretical tool-set. For instance phonons were traditionally treated as a part of the zero-loss peak in frozen phonon calculations [1]. Now we need efficient methods to simulate their spectroscopic signatures, which led to a development of new computational methods [8,9]. Scattering of electrons on microscopic magnetic fields [10] is another such field, which experienced significant progresses in simulation methods during the last few years. New challenges may include couplings of electrons, phonons and magnons, temperature and time dependencies of scattering on phonons, just to name a few.

We will discuss our recent advances in theory and the simulations of dichroic spectra, and signatures of vibrational and magnon scattering intensities.

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10y of developing cryo-atom probe tomography

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In 2016, the group that I was leading at the Max-Planck Institut für Eisenforschung, in 2024 rebranded as Max Planck Institute for Sustainable Materials, embarked on a long (long) journey to enable workflows for handling samples at low temperature in a moisture-free environment, and transfer them into a scanning-electron microscopes for imaging and focused-ion beams to prepare specimens for atom probe tomography (and other fun techniques where necessary) and then transfer them for analysis. These workflows are now often referred to as cryo-atom prob tomography, to surf on the cryo-hype maybe as atom probe is somehow always performed at low temperature, but also, in a way to highlight that this could really enable new applications beyond what had so far been achieved.
In this presentation, I will showcase some of the work that the group has done, establishing the infrastructure [1] and developing methods for specimen preparation and handling [2,3], and enabling the analysis of frontier materials, from frozen liquids [4] to Li-containing materials for possible application in the broad scope of batteries [5,6] to liquid-solid interfaces [7] and building the grounds for investigating soft biological materials [8].

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