Sample thickness determination by HAADF-STEM in a scanning electron microscope

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Low-energy scanning transmission electron microscopy (STEM) in a scanning electron microscope is a promising method for the determination of the sample thickness which is an important property for many quantitative TEM-based techniques [1]. Thickness determination relies on comparing measured high-angle annular dark-field (HAADF)-STEM intensities with simulated ones, which require a realistic model of the electron scattering. Most approaches for modeling the HAADF-STEM intensity are based on Monte Carlo simulations with different types of elastic scattering cross-sections. However, the HAADF intensity can also be obtained from the solution of electron transport equation which provides the angular distribution of multiply scattered electrons [2]. Within this study, we used the latter approach by numerically solving the electron transport equation which has the benefit of being faster than Monte Carlo simulations. HAADF-STEM intensities at 30 keV were calculated by the in-house developed software CeTE1.4 (Computation of electron Transport Equation) written in Java (http://openjdk.java.net/, version 1.8.0). Corrections for detector threshold energy and efficiency in the charge collection were considered [3]. Angular distributions of electrons for different primary energies, sample thicknesses and compounds were calculated and compared with experimental data obtained from wedge-shaped samples with known thickness.

Experiments were performed with a scanning electron microscope of the FIB-SEM Thermo Fisher Strata 400 equipped with a STEM detector. To compare experimental and simulated HAADF intensities, measured HAADF intensities I_{EXP} were normalized by the intensity of the incident electron beam I_0 by directly scanning over the HAADF detector plane. The normalized HAADF-STEM intensity is calculated by $I_{HAADF}=(I_{EXP}-I_{DARK})/(I_0-I_{DARK})$ with I_0 and the dark background I_{DARK}. Experimental I_{HAADF} for ZnO are displayed by the red line in Figure 1b as a function of sample thickness. Simulations using the Mott cross-section and Screened Rutherford cross-section are included in the graph. A reasonable match between simulation and experiment could be achieved. Thickness determination is applied to measure the local thickness of ZnO nanoparticles and generate in this way a three-dimensional profile of the particle shape.

1. Volkenandt, T., Müller, E. & Gerthsen, D. (2014). Sample thickness determination by scanning transmission electron microscopy at low electron energies. Microsc Microanal 20, 111-123.
2. Goudsmit, S. & Saunderson, J.L. (1940). Multiple scattering of electrons. Phys Rev 57, 24-29.

3. Reimer, L. (1998). Scanning Electron Microscopy: Physics of Image Formation and Microanalysis. IOP Publishing.

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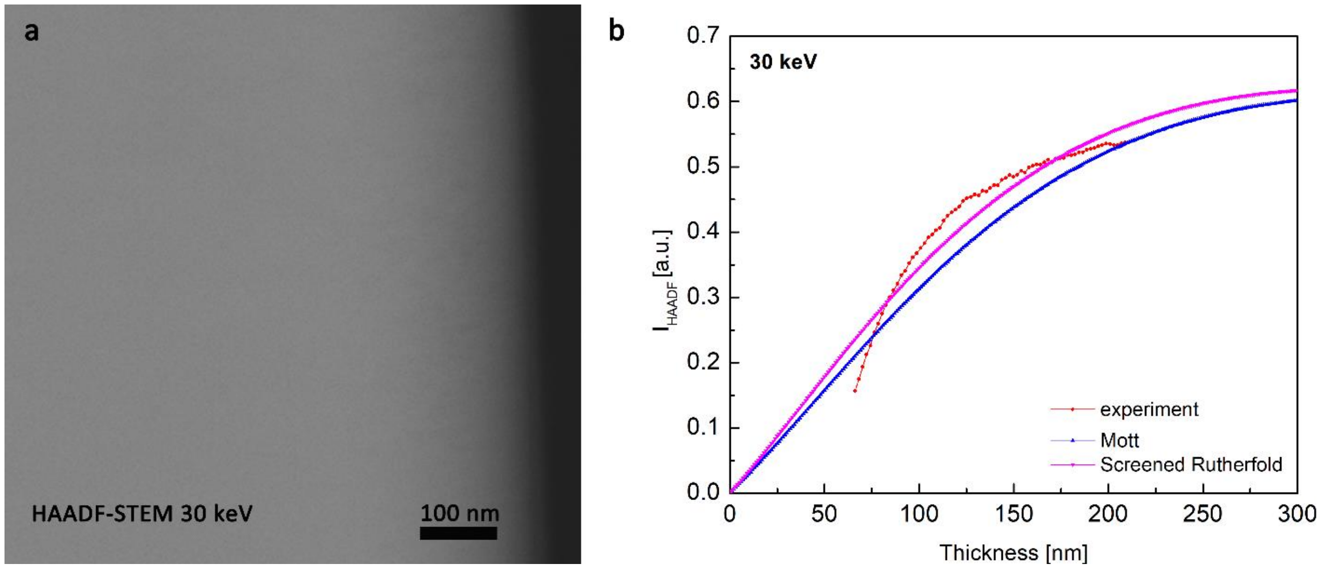

Fig. 1a HAADF-STEM image of the ZnO wedge sample; Fig. 1b Comparison of 30 keV experimental (red line) and simulated I_{HAADF} for ZnO (blue line: simulations based on Mott scattering cross-section, purple line: simulation based on screened Rutherford cross-sections) as a function of the sample thickness from a wedge-shaped specimen.