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Quantification of the thickness of TEM samples by low-energy scanning transmission electron microscopy

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Introduction

- Knowledge of the local sample thickness is important for analytical electron microscopy
- Established techniques: EELS, CBED, electron holography, thickness contours in TEM
- More recent and promising technique: high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) at low energies (E_0 ≤ 30 keV) [1-2]
  - → Strong material (Z-) contrast
  - → Negligible knock-on damage
- Contrast depending on sample thickness and composition → thickness determination if composition is known

Goals

- Precise thickness determination of samples within a large range of atomic numbers
- Comparison of measured intensities of HAADF-STEM images with Monte Carlo (MC) simulations
- Validation of the method by using samples with known thicknesses
- Determination of the most suitable scattering cross-section (CS) to be used in the simulation

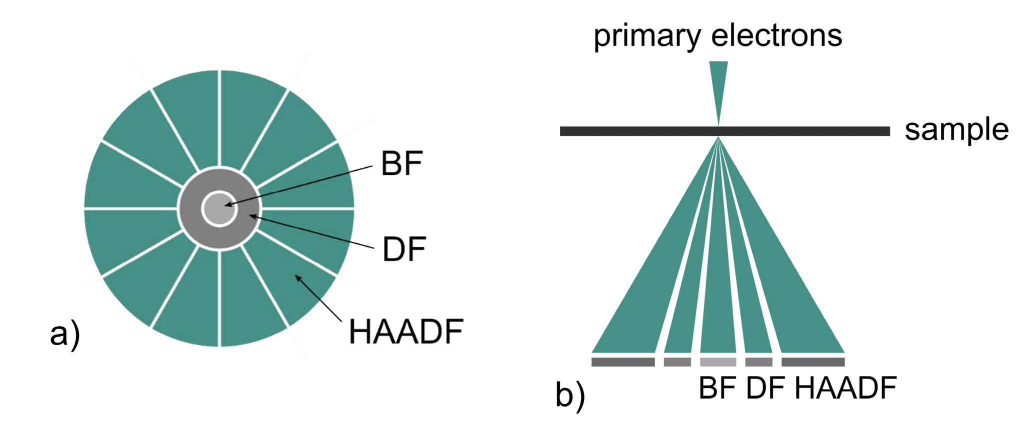

Fig. 1: Annular semiconductor STEM-detector (a) topview and b) sideview of the experimental setup.

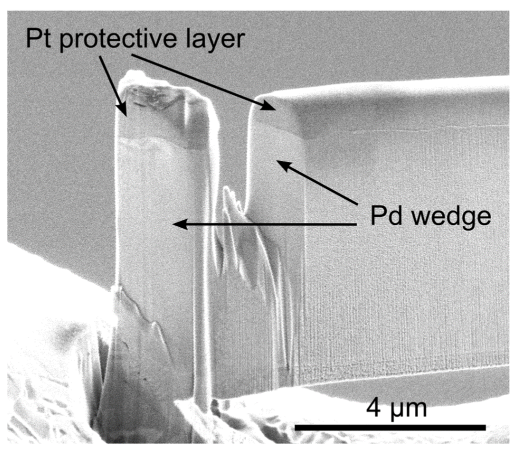

Fig. 2: FIB-prepared lamella with wedge-shaped samples (SE-image, 3 keV)

Experimental techniques and samples

- Wedge-shaped samples with defined thickness profile fabricated by focused-ion-beam (FIB) milling (Fig. 2)
- FEI DualBeam Strata 400S, combined FIB and scanning electron microscope (SEM)
- Annular semiconductor STEM-detector with bright-field (BF), dark-field (DF) and HAADF-segments below the sample (Fig. 1)
- Sample materials: MgO (Z = 10), Ge (Z = 32), Pd (Z = 46)

Imaging Conditions

- Measurement of normalized image intensities
- Reference intensites from direct imaging of the detector: Inner part of HAADF-segment visible at lowest magnification (Fig. 3a)
- Brightness and contrast kept constant for sample imaging
- Primary electron energy: 10 - 30 keV
- Minimum and maximum scattering angles: 0.187 – 0.683 rad

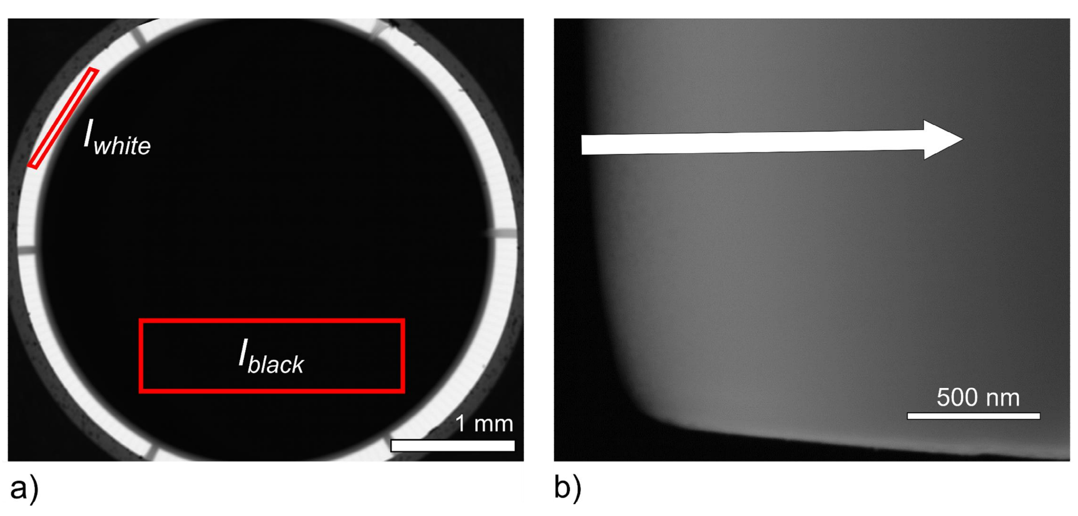

Fig. 3: a) HAADF STEM image of STEM-detector. Marked areas illustrate I_{white} and I_{black} used for normalization of the measured HAADF STEM intensities.
b) Cross-section HAADF STEM image of the MgO sample at 20 keV with indicated position of the line scan. The thickness increases from left to right.

Normalization of measured intensities

- Normalization of measured intensities I_s according to

$$I_{nor,\,HAADF} = \frac{I_s - I_{black}}{I_{white} - I_{black}}$$

$I_{nor,\,HAADF}$: normalized measured intensity, I_{black}: background intensity, I_{white}: intensity without sample (direct imaging of the detector)

Simulation of HAADF STEM intensities

- Monte Carlo simulations by CASINO software [3]
- Screened Rutherford CS (SR-CS) and different Mott CSs (M-CS)
- Normalization of simulated intensities

$$I_{nor,\,sim} = \frac{N}{N_0}\frac{E_{trans} - E_{off}}{E_0 - E_{off}}$$

$I_{nor,\,sim}$: normalized simulated intensity, N: number of electrons on the detector, N_0: number of simulated electrons, E_{trans}: average energy of transmitted electrons, E_{off} = 3 keV: offset energy of the detector

Results

- Measured and simulated intensity line profiles of the normalized HAADF STEM intensity as a function of thickness t (Fig. 4)

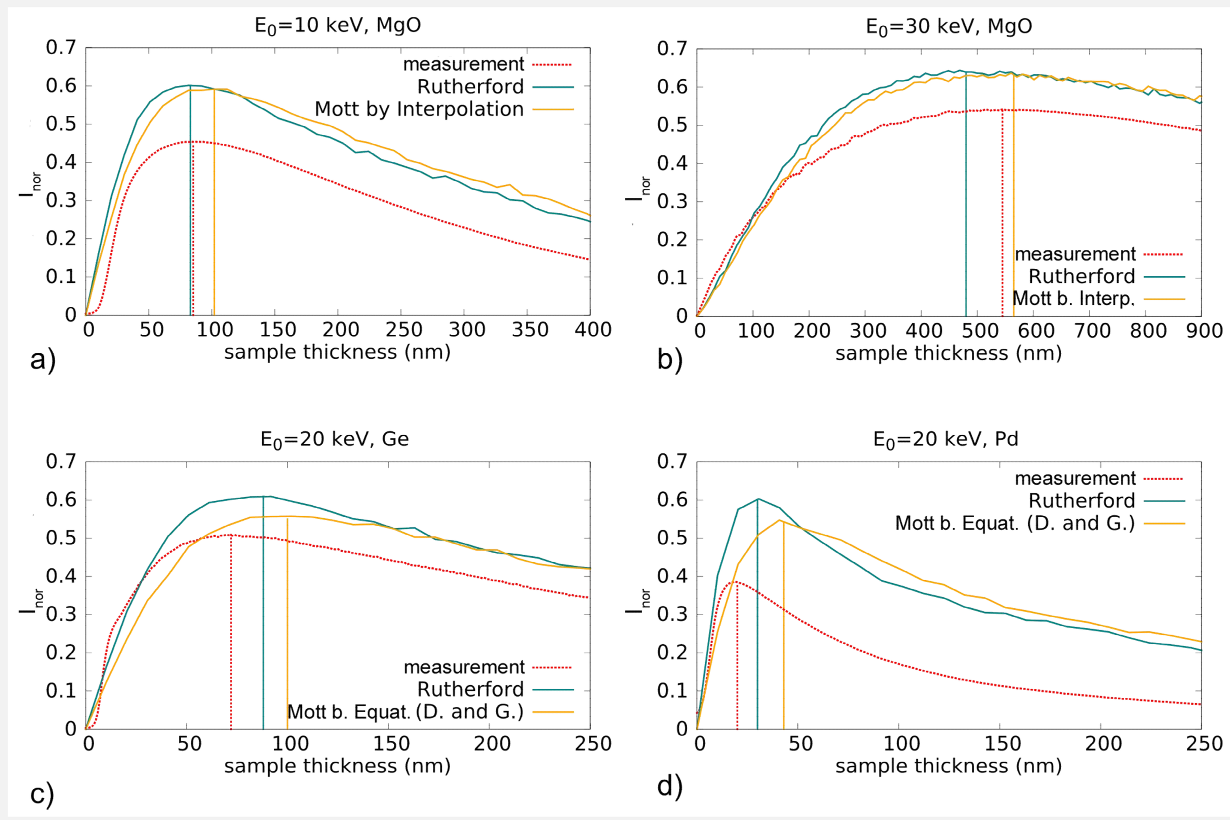

Fig.4: Simulated (SR-CS and most suitable M-CS, Mott by Interpolation or by Equation from Drouin und Gauvin [4]) and measured I_{nor} for samples with different Z and E_0 as a function of the sample thickness. Intensity maxima are indicated by lines.
a) MgO, E_0 = 10 keV,
b) MgO, E_0 = 30 keV,
c) Ge, E_0 = 20 keV,
d) Pd, E_0 = 20 keV.

- Maximum of I_{nor} shifts to lower t for lower E_0 and higher Z
- Lower values of measured I_{nor} due to uncertainties concerning I_{white} and the response of the detector
- Best fit between experiment and simulations determined by comparing the maxima positions of I_{nor}
  - Low-density materials (MgO) and high primary electron energies (30 keV)
    → Mott CSs probably better choice
  - Screened Rutherford CS better choice for all other E_0 and Z (Pd and Ge)

Summary

- Quantification of the local sample thickness by comparison of measured HAADF STEM intensity with Monte-Carlo simulations
- Adequate choice of scattering cross-sections necessary
- Light materials at high energies probably better described by Mott cross-sections, all other cases better described by screened Rutherford cross-section
- Uncertainties concerning I_{white} and response of the detector

References

[1] P.G. Merli et al., *Ultramicrosc.* 94 (2003), p. 89.
[2] T. Volkenandt et al., *Microsc. Microanal.* 16 (2010), p. 604.
[3] H. Demers et al., *Scanning* 33 (2011), p.135.
[4] D. Drouin et al., *Scanning* 19 (1997), p. 20.