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Beam broadening measured in transmission mode at low electron energies in a scanning electron microscope

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Introduction

- Primary electron energies $E_0 \leq 30$ keV yield high material contrast for weakly scattering materials and reduce knock-on damage
- Mean free path length decreases with lower E_0
  → Increased number of scattering events
  → beam broadening even for small sample thicknesses
- Tracking of electron paths impossible → beam broadening not directly visible
  → comparison with models necessary

Goals

- Experimental determination of beam broadening using low-energy-STEM
- Comparison with theoretical models by Goldstein [1] and Gauvin/Rudinsky [2]

Analytical model by Gauvin/Rudinsky

- Based on elastic Rutherford scattering cross-section [2]
- Hurst exponent H allows distinction between different scattering regimes depending on number of scattering events [2]
  - $H = 1$, thin samples, single scattering ($t / \lambda_{el} \rightarrow 0$)
    → agrees with previous measurements for thin samples [3]
  - $H = 0.5$, "thick" samples, plural scattering ($1 < t / \lambda_{el} < 25$)
- Analytical equation for beam broadening b

$$b = K' \frac{Z^{(4H+1)/3}}{E_0^{(2H+1)/2}} \left(\frac{\rho}{A}\right)^H t^{1+H} \quad \text{in units of cm}$$

with $K' = (0.1167)^{1-2H} (39437)^H \sqrt{\frac{R}{1-R}}$

Z: Atomic number
A: Atomic mass
ρ: Material density
t: Sample thickness in units of cm
E_0: Primary electron energy in units of keV

- Important parameter R: beam diameter defined to contain $R \cdot 100\,\%$ of the total beam intensity (in our work 68%) [4]

Experimental techniques

- Use of samples with known material properties (atomic number, density): MgO, Si, $SrTiO_3$, Ge
- Preparation of wedge-shaped specimens with defined thickness by FIB-milling (Fig. 1)
- Microscope: FEI DualBeam Strata 400S
- Primary electron energy $15 \leq E_0 \leq 30$ keV

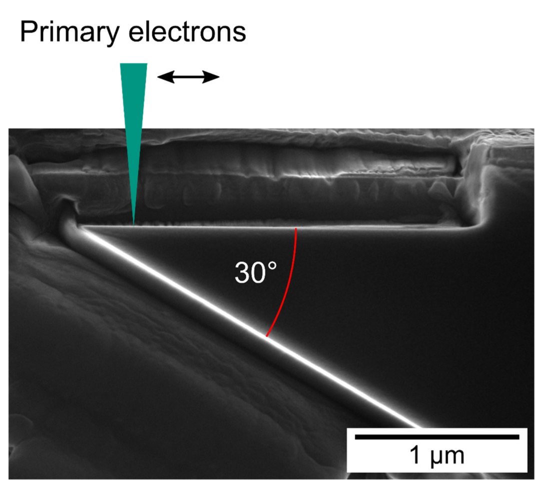

Fig. 1: MgO wedge with a wedge angle of 30° imaged by secondary electron SEM.

Procedure to measure beam broadening

- Assumptions and definitions
  - Beam diameter b: diameter that contains 68% of all electrons [4]
  - Diameter of the incident electron beam ≪ b → total beam diameter can be approximated by b
- Measurement of transmitted intensity I_{exp} up to scattering angle θ determined by HAADF detector as a function of the sample thickness (Fig. 2a)
- Variation of θ by changing the specimen-detector distance
- Normalization of I_{exp} with respect to the intensity of the incident electron beam I_0 and the black-level intensity I_{bl}

$$I = \frac{I_{exp} - I_{bl}}{I_0 - I_{bl}}$$

- Plot I as a function of sample thickness (Fig. 2b)

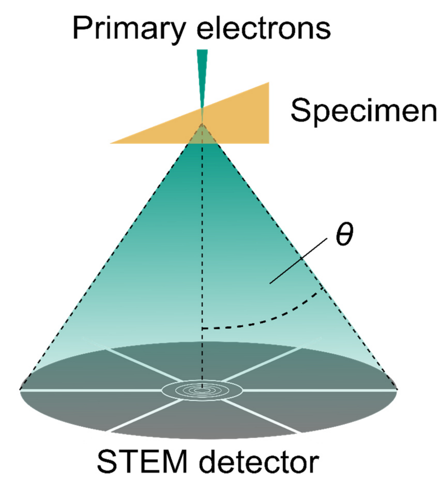

Fig. 2a: Experimental setup with annular semiconductor STEM-detector.

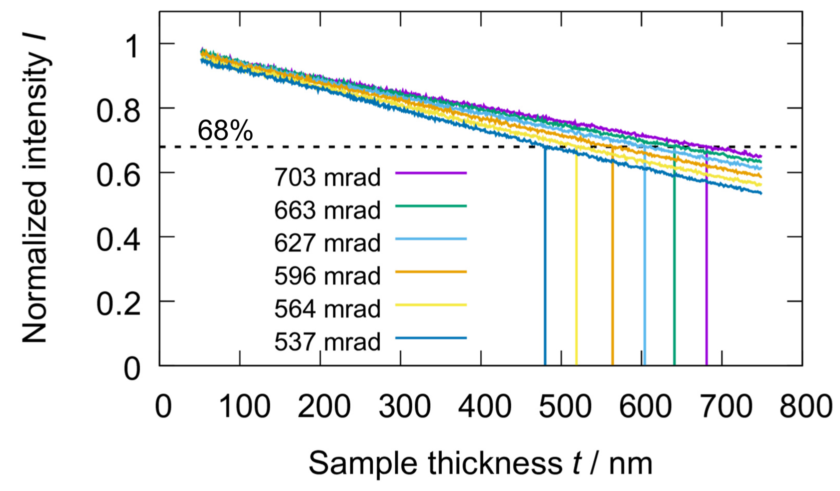

Fig. 2b: Normalized STEM intensity for Si at 25 keV. The intensity within the scattering angles indicated in the legend is drawn in different colors. The horizontal line indicates the thickness where the measured intensity corresponds to 68%.

Evaluation of measurement

- Extraction of pairs (t, θ), marked by vertical lines, from Fig. 2b: t is determined from the intersection of the intensity/thickness curves for a certain θ at 68% total beam intensity (dashed line in Fig. 2b)
- Calculation of beam broadening by (cf. Fig. 3)

$$b = t\ \tan\theta$$

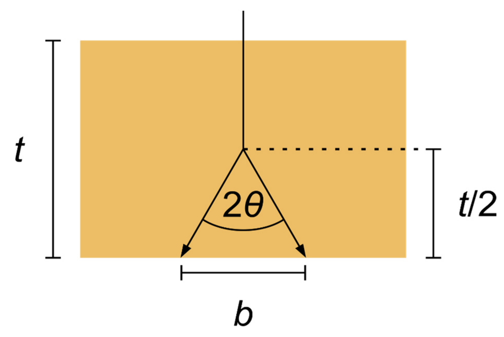

Fig. 3: Determination of beam broadening b in a sample of thickness t after [1].

Results

- Beam broadening for MgO, Si, $SrTiO_3$ and Ge (e.g. at 25 keV, Fig. 4)
- Merge all data and fit results for different materials with parameter H as fit parameter (Fig. 5)

$$b \frac{E_0^{(2H+1)/2}}{c_{mat}(H)} = K'(H)\ t^{1+H}$$

- Experimental results best described by the theoretical model with $H = 0.75$ for $R = 0.68$

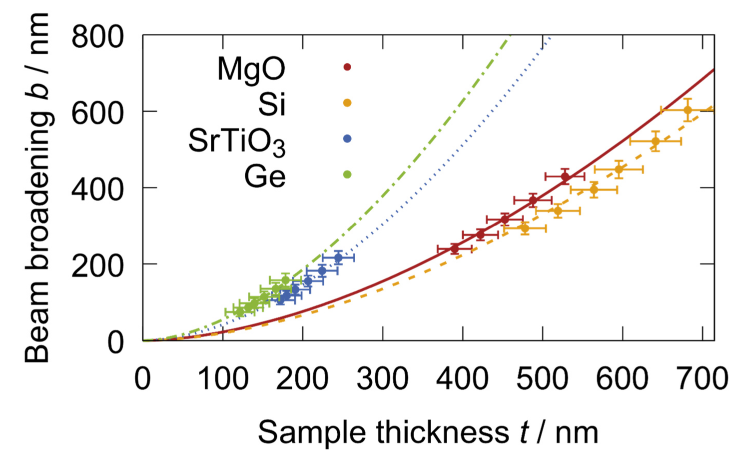

Fig. 4: Beam broadening for MgO, Si, $SrTiO_3$ and Ge at 25 keV. Fitted curves with $H = 0.75$ (MgO), 0.75 (Si), 0.81 ($SrTiO_3$) and 0.76 (Ge).

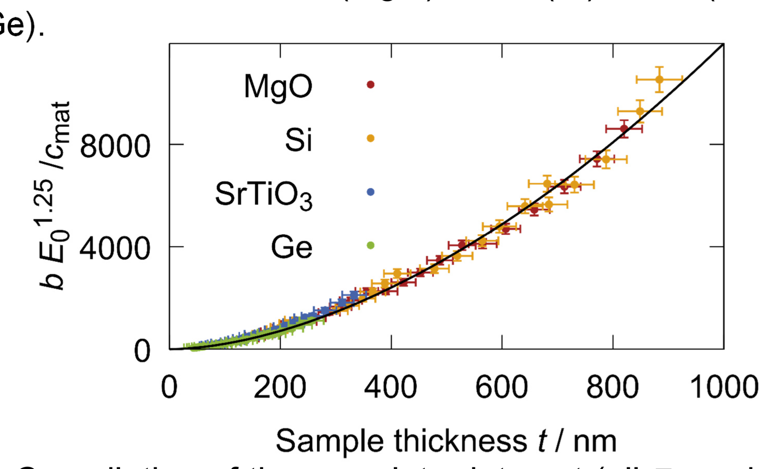

Fig. 5: Compilation of the complete data set (all E_0 and materials) as a function of t with $c_{mat} = Z^{(4H+1)/3}(\rho/A)^H$ for the corresponding material. Experimental data and fit curve with $H = 0.75$.

Conclusions

- Electron beam broadening was measured at low electron energies (15 to 30 keV) for materials with average atomic numbers between 10 and 32 and sample thicknesses up to 900 nm
- Electron beam broadening b can be well described by the Gauvin/Rudinsky model with $H = 0.75$ determined for $R = 0.68$

$$b = 0.1167^{-0.5}\ 39437^{0.75} \sqrt{\frac{R}{1-R}} \frac{Z^{4/3}}{E_0^{1.25}} \left(\frac{\rho}{A}\right)^{0.75} t^{1.75}$$

- For plural scattering as in our work, $H = 0.5$ was expected. Simulations [2, Fig. 5] however indicate that H increases with decreasing R. $H = 0.75$ is therefore consistent with small $R = 0.68$ in our work.

References

[1] Goldstein et al., Scanning Elecron Microscopy 1 (1977), p. 315.
[2] Gauvin and Rudinsky, Ultramicroscopy 167 (2016), p. 21.
[3] Drees et al., Ultramicroscopy 185 (2017), p. 65.
[4] Hugenschmidt et al., Journal of Microscopy 274 (2019), p. 150.

The authors acknowledge funding by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) under contract number Ge 841/20-2 and under Germany`s Excellence Strategy – 2082/1 – 390761711