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Sample thickness determination by low-energy scanning transmission electron microscopy

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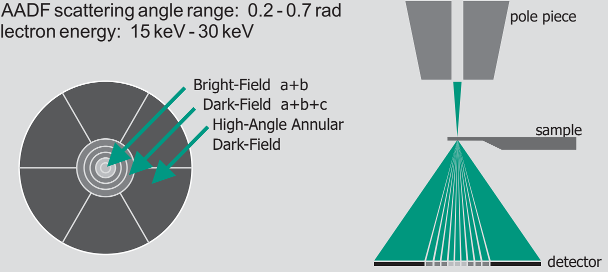
Motivation

- Scanning transmission electron microscopy (STEM) in high-angle annular dark-field mode (HAADF) ⇨ strong material (Z-) contrast
- Energies ≤ 30 keV ⇨ negligible knock-on damage
- Important parameter in analytical electron microscopy ⇨ sample thickness
- Contrast depending on sample thickness and composition ⇨ determination of local sample thickness if composition is known [1]

Instrumentation

- Combined focused ion-beam (FIB)/SEM: FEI Strata 400S
- Annular semiconductor STEM-detector with bright-field (BF), dark-field (DF) and six azimuthal HAADF segments
- HAADF scattering angle range: 0.2 - 0.7 rad
- Electron energy: 15 keV - 30 keV

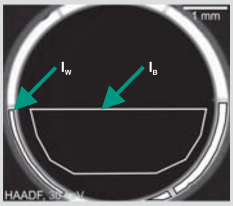


Imaging Conditions

- Measurement of normalized image intensities
- Reference intensities for normalization from detector STEM images
- Inner part of HAADF segment visible at lowest magnification
- Brightness & contrast tuned to avoid over- and undersaturation and kept constant for sample imaging

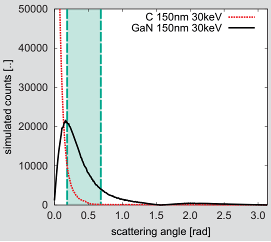
$$I_{HAADF} = \frac{I_S - I_B}{I_W - I_B}$$

I_{HAADF}: normalized image intensity
I_S: measured image intensity
I_W: intensity of incident beam (white-level)
I_B: offset intensity (black-level)



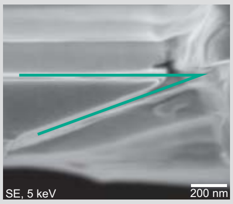
Monte-Carlo Simulations

- Use of NISTMonte package [2]
- Consideration of screened Rutherford (SRCS) and Mott (MCS) scattering cross-sections
- Normalization for comparison with measured intensities
- Corrections for detector-related effects concerning collection current [3] and geometry [4]
- HAADF intensity by integration of scattering angle histogram



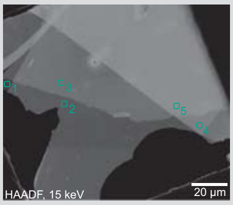
Initial Thickness Measurements

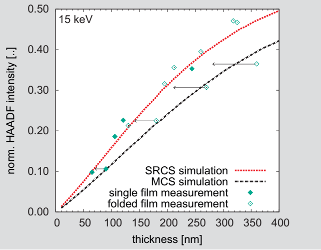
- In case of film sample via Dektak profilometer
- In case of wedge-shaped lamellas prepared by FIB milling via top-view SE SEM imaging
- Measurement of thickness offset at the edge t_0 and wedge angle α



Results: FHBC films

- Five films of fluorenyl hexa-peri-hexabenzocoronene ($C_{100}H_{98}$) with different nominal thickness ($\overline{Z}$=3.5, ρ=1.1 g/cm³)
- Measurements at discrete multiples of nominal film thickness due to folding
- Correlation with nominal thickness and comparison with simulations
- Agreement with simulations based on SRCS
- Deviations for one film explaned by uncertainty of nominal thickness
- Improved agreement for shift of -20nm for nominal thickness (arrows)



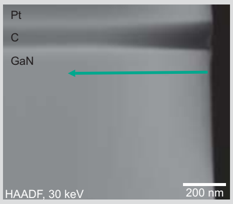


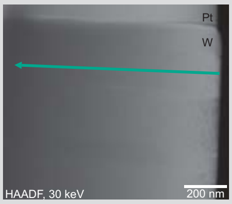
Results: GaN- and W-lamellas

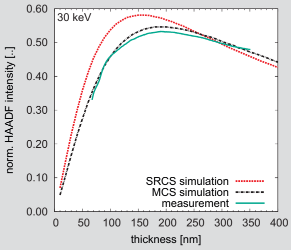
- GaN- and W-lamellas with FIB-prepared wedges for a defined thickness profile ($\overline{Z}$=19, ρ=6.1 g/cm³ and Z=74, ρ=19.3 g/cm³)
- Intensity measurement along line with increasing thickness due to wedge shape
- Transformation of position coordinate x of linescan to sample thickness t via:

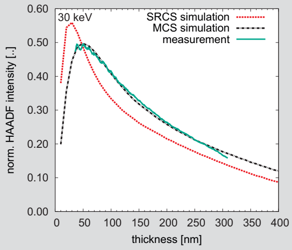
$$t = t_0 + x \cdot \tan\alpha$$

- Fit to simulated curve by adjusting t_0 and α
- Agreement with simulations based on MCS
- Agreement of fit parameters with measurements in top-view SE SEM images









Summary

- Low-kV HAADF STEM intensity measurements enable local sample thickness determination by comparison with Monte-Carlo simulations
- Adequate adjustment of brightness and contrast necessary
- Normalization of measured intensities with intensity of the incident beam
- Consideration of detector-related effects during evaluation of simulations
- Materials with Z<6 described by screened Rutherford cross-sections, materials with Z>14 described by Mott cross-sections
- Results avaiable for FHBC (Z=3.5), GaN (Z=19) and W (Z=74), as well as C (Z=6) and Si (Z=14) [1]
- Initial thickness measurements by profilometer and top-view SE SEM imaging

References and Acknowledgement

[1] T. Volkenandt et al. (2013), Microsc Microanal, *submitted*
[2] N. Ritchie (2005), Surf Interface Anal **37**, p. 1006
[3] L. Reimer (1998), Heidelberg: Springer-Verlag
[4] T. Volkenandt et al. (2010), Microsc Microanal **16**, p. 604
[5] This work is funded by the Deutsche Forschungsgemeinschaft (DFG)

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