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Investigation of the HAADF-STEM contrast thickness for different camera lengths in metallic glasses

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Shear bands are of utmost importance for understanding the plastic deformation behaviour of metallic glasses. They are associated with a structural change compared to the surrounding matrix due to local dilatation most likely resulting in enhanced free volume [1]. Thus the quantification of free volume inside shear bands is an important issue.

In this respect, we have developed a new experimental approach [2] to measure density changes using the relation of the dark-field intensity I/I_0 (scattered electrons collected by a high-angle annular dark-field (HAADF) detector) and the mass thickness:

$$\frac{I}{I_0} = \left[1 - \exp\left(-\frac{N_A \cdot \sigma \cdot \rho \cdot t}{A}\right)\right] = \left[1 - \exp\left(-\frac{\rho \cdot t}{x_k}\right)\right]$$
(Eq. 1)

Where N_A is the Avogadro's number, σ is the total scattering cross-section, ρ is the density, t is the foil thickness and A is atomic weight. x_k is the contrast thickness, which is defined as $A/(N_A \cdot \sigma)$.

The foil thickness *t* is obtained from the low-loss region of the electron-energy loss spectrum (EELS) [3], which is acquired simultaneously to the HAADF signal.

To obtain a small argument of the exponential function, which allows the linearization of Eq. 1 to $I/I_0 \approx \rho \cdot t/x_k$, the contrast thickness should preferably be large. This can be achieved by choosing small camera lengths. However, the camera length affects the scattering cross-section and thus the HADDF-STEM signal. Larger camera lengths show a better signal-to-noise-ratio. However, the collection angle for the EELS measurement has to be smaller than the convergence angle [3]. In the present study gain-corrected and normalized HAADF-STEM line scans using different camera lengths were compared taken on a FEI Titan 80-300 operated at 300 kV. Different systems of metallic glasses were examined using melt-spun $AI_{88}Y_7Fe_5$ as well as $Pd_{40}Ni_{40}P_{20}$.

We found that the HAADF-STEM signal was constant within the noise level for different camera lengths from 48 mm to 130 mm. Thus the linear approach works sufficiently accurate within this range. However for larger camera lengths a deviation in the HAADF-STEM intensity was observed.

Thus, it is concluded that a camera length of 130 mm was the best choice for simultaneous measurements of the HAADF-STEM and EELS signal.

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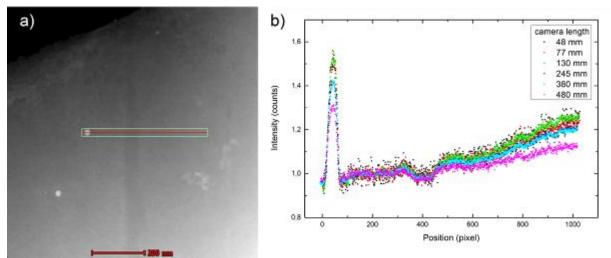


Figure 15. a) HAADF-STEM image of a shear band in Al₈₈Y₇Fe₅. The position of the line scan is indicated by the frame and integrated over the area. b) Gain-corrected and normalized intensities of the line scans shown for different camera lengths. Note the deviation for large camera length.

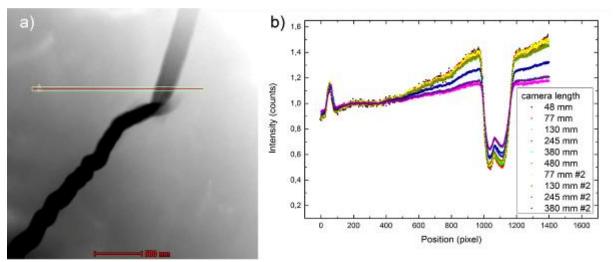


Figure 16. HAADF-STEM image (a) of a crack-initiated shear band in Pd₄₀Ni₄₀P₂₀. The line scan is integrated over the boxed area. b) Profiles repeated and compared with respect to different camera lengths. Larger camera lengths reveal an underestimation of the intensity change compared to the smaller ones.