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A simplified approach for measuring sample thickness and mis-tilt from position-averaged convergent-beam electron diffraction patterns

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In recent years, high-resolution scanning transmission electron microscopy (STEM) has been applied successfully to the analysis of the atomic structure of solid materials. Many approaches have been proposed for extracting quantitative information about local atomic structure from STEM images, including the matching of image simulations to experimental images on an absolute scale (1). It is highly desirable to determine as many unknown experimental parameters as possible from independent measurements in order to improve comparisons. Sample thickness and mis-tilt from a chosen crystallographic orientation are examples of parameters that are often difficult to measure experimentally. The analysis of position-averaged convergent beam electron diffraction (PACBED) patterns has been shown to provide accurate measurements of sample thickness and mis-tilt, with reduced requirements for a priori knowledge about other experimental parameters (2). However, it is still challenging to match the details of simulated and experimental PACBED patterns accurately and robustly. In the present work, we investigate possibilities for extracting quantitative sample information from PACBED patterns with reduced demands on prior knowledge. We focus on the determination of sample thickness and mis-tilt from integrated intensities rather than pattern recognition. For example, the integrated bright-field and low angle dark-field intensity is found to decrease monotonically with sample thickness (Fig. 1). This measure can be calibrated with respect to the intensity of the incident probe by recording reference patterns without the sample present (but under otherwise identical conditions) and used to determine the sample thickness with a precision of few nanometres. The evaluation is insensitive to small sample mis-tilt. For larger sample tilt angles, an estimate of the mis-tilt can be determined from the cross-overs of Kikuchi lines, which are visible in patterns recorded from thicker samples (e.g., Fig. 2). Our approach is particularly applicable to momentum-resolved STEM data, from which the sample thickness and mis-tilt can be obtained with unit cell resolution. Although sample thickness has previously been measured from averaged intensities recorded using a high-angle annular dark-field detector (3), quantification then requires careful prior calibration of the detector sensitivity to scale the experimental image intensity as a fraction of the incident probe intensity.

References:

(1) J.M. LeBeau et al., Phys. Rev. Lett. 100 (2008) 206101.

(2) J.A. Pollock et. al., Ultramic. 181 (2017) 86-96.

(3) A. Rosenauer et al., Ultramic. 109 (2009) 1171-1182.

Fig. 1: Integrated intensity of simulated PACBED patterns of SrTiO3 in the [001] zone axis orientation plotted as a function of sample thickness for 200 keV electrons. The integration area is a disk centred on the zero beam with a radius of 44 mrad, which corresponds to twice the semi-convergence angle of the incident probe. The intensity is plotted as a fraction of the incident probe current. The horizontal blue line marks the intensity value obtained from the experimental PACBED pattern shown in Fig. 2, for which the estimated sample thickness is 39 nm.

Fig. 2: Experimental (left) and simulated (right) PACBED patterns of SrTiO3 in the [001] zone axis orientation. The experimental pattern was recorded using a probe CS-corrected FEI Titan instrument operated at an accelerating voltage of 200 kV. The white circle marks the circular integration area used to measure the intensity value marked in Fig. 1.

Fig. 1

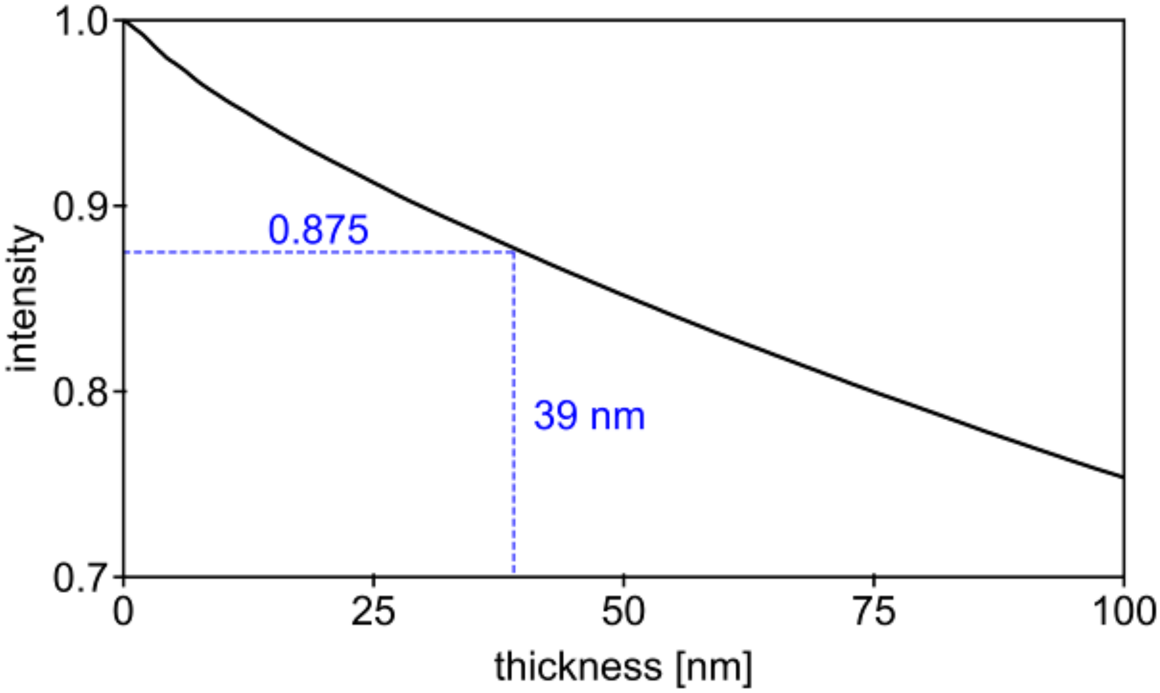
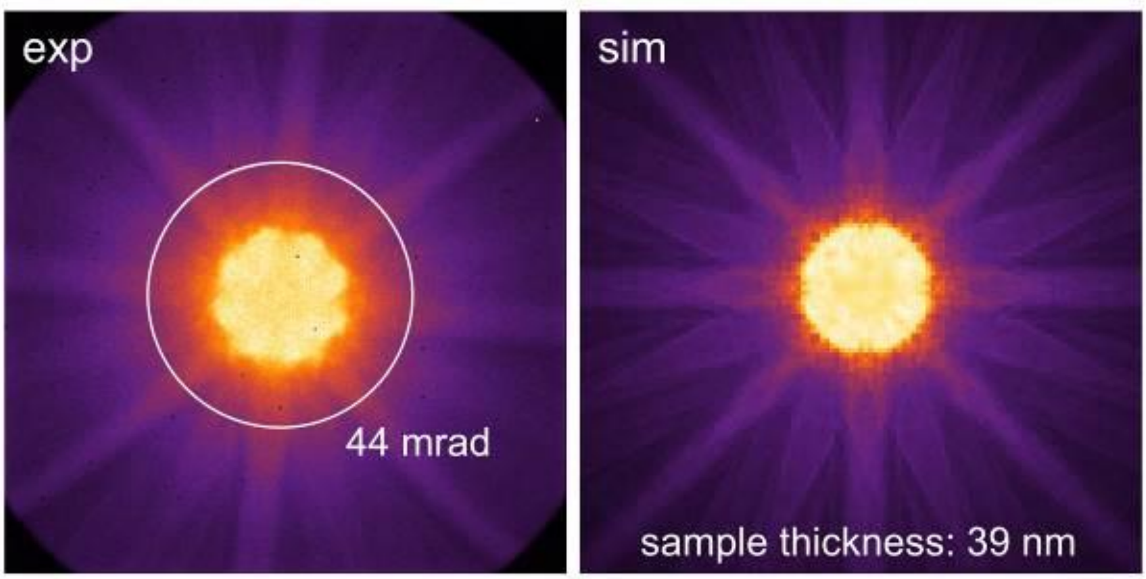

1.0
0.9
0.8
0.7
intensity
0.875
39 nm
0
25
50
75
100
thickness [nm]

Fig. 2


exp
44 mrad
sim
sample thickness: 39 nm