Sample Preparation Methods

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Using the X^2-Holder to Create Thin TEM-lamella by FIB

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A new tiltable sample holder has been recently developed that facilitates the easy preparation of thin TEM lamellae with sufficient quality for HRTEM in our group [1]. Using this holder, two grooves in a direction at a high angle to each other are milled on either side of the lamella. Where both grooves overlap, an electron transparent window is created (figure 1). In contrast to the traditional method, however, the electron transparent window is on all four sides bounded by the thick lamella, which provides a sturdy frame around the electron transparent area. The frame increases the stability of the lamella significantly, making it possible to create large areas (tens of μm^2) of uniform thickness. The application of this technique to different sample types and geometries will be demonstrated.

To increase sample quality for very thin samples (< 20 nm), a post-FIB low voltage (500 V) argon polishing step can be performed. This however requires a slight modification of the lamella geometry to make the electron transparent area accessible to the argon beam and to reduce contamination. This modified geometry has been used in a Fishione Nanomill system to produce a high quality thin Lanthanum-Strontium-Aluminate sample. The 500 V final polishing step removed the ~ 5nm thick amorphous layer present after FIB preparation (5 kV final polishing) virtually completely as lattice fringes are visible up to the edge of the sample (figure 2).

As the electron transparent area is surrounded on all sides by thick material, the determination of the thickness of the electron transparent area is more difficult. A correct determination of the local thickness also greatly helps producing lamellae with a uniform thickness over a large area. We use a method based upon the intensity of the back scattered electron (BSE) signal [2]. For an accurate determination of the thickness it is essential that the background signal coming from the sample holder is minimized. Some modifications to the original design of the holder have been made, that minimizes the number of back-scattered electrons, but still ensure good sample holder conductivity to minimizing sample drift due to charging. In addition Monte Carlo simulations have been performed to determine the influence of detector position on the BSE signal as function of maximum electron penetration depth. These simulation show that the detector position has to be taken into account, otherwise this can lead to a ~ 20 % error in thickness determination (figure 3).

1. L. Lechner, J. Biskupek and U. Kaiser, *Microscopy and Microanalysis* 18 (2012), p. 379–384
2. R. Salzer, A. Graff, M. Simon and F. Altmann, *Microscopy and Microanalysis* 15 (2009), p. 340–341
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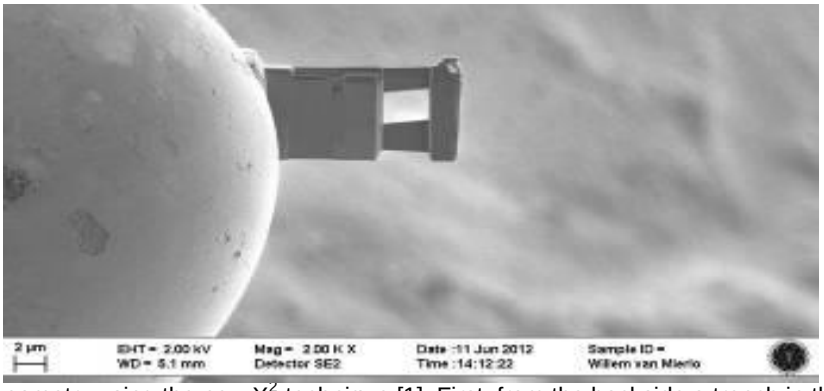

Figure 1: Milling geometry using the new X^2-technique [1]. First, from the backside a trench in the lamella is cut. After final polishing of the back side at a low voltage, the sample is tilted over ~ 90° and then is thinned from the front side the sample. An electron transparent window is created where both grooves overlap (see the bright window inside the lamella).

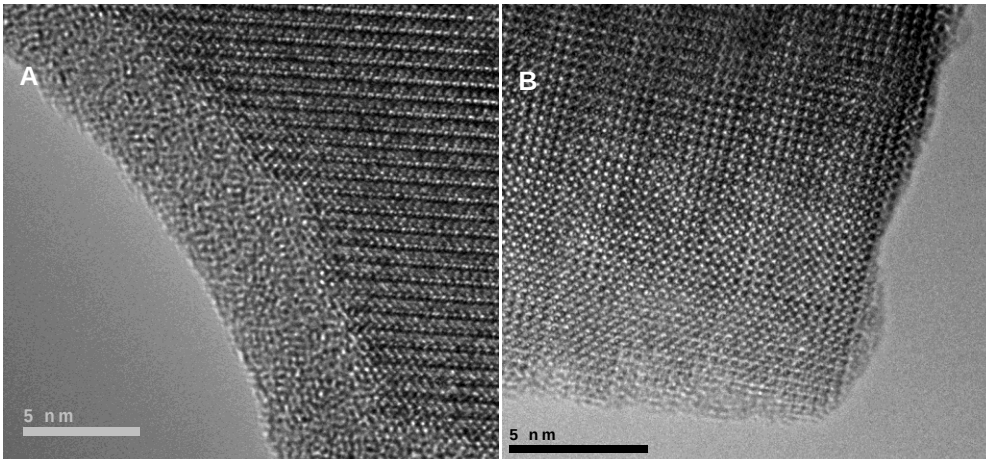

Figure 2: Improvement of sample quality after low-voltage argon-milling. A) Edge of the electron transparent window after final polishing with 5 kV Ga. B) After final polishing with argon at 500 V using the Fishione nanomill. The effect of low voltage Ar thinning step is seen by the complete reduction of the amorphous layer at the edge.

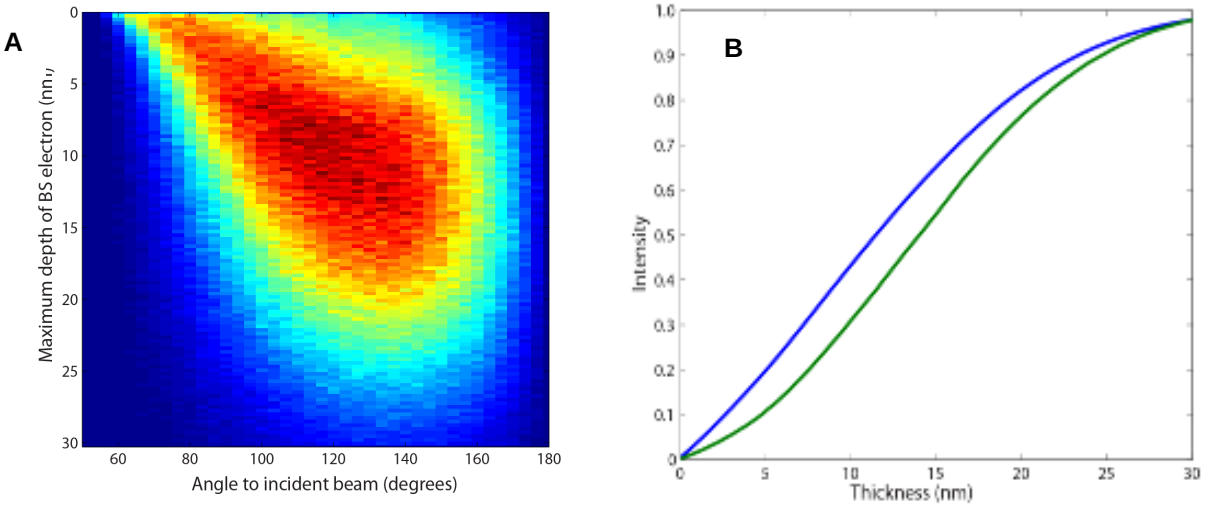

Figure 3: Results of Monte Carlo simulations showing the influence of the detector position on the maximum depth in the sample from which a detected back-scattered electron originates. A) Intensity distribution of back scattered electrons as function of back scattered angle (relative to incident beam) and maximum depth the back scattered electron penetrated into the target. Blue is low intensity, red is high intensity B) Difference between determined thickness with (green line) and without (blue line) taking into account the finite collection angle of the detector Not taking into account the finite collection angle leads to a ~ 20% underestimating of sample thickness.