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FIB Target Preparation for 20 kV STEM - A Method for Obtaining Ultra-Thin Lamellas

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Recently, scientists have rediscovered the advantages of using low energies in transmission electron microscopy (TEM). It dramatically reduces knock-on damage for imaging low-Z number material and enables electron energy loss spectroscopy up to very high energy losses with exceptionally low background noise [1]. Alas, low voltage TEM requires extremely thin specimens virtually free of preparation artifacts. Conventional focused ion beam (FIB) preparation methods cannot be employed to create high quality specimens much thinner than 25 nm. We have developed a straightforward method for in-situ target preparation of ultra-thin TEM lamellas by FIB milling. With this method we are able to routinely obtain large area co-planar lamellas thinner than 10 nm. The resulting specimens are suitable for low kV TEM as well as scanning transmission electron microscopy.

TEM sample preparation by FIB has advanced dramatically over the past years and starts to replace conventional techniques when speed and site specificity are required [2]. Despite of these improvements the specimen thickness and quality obtainable is not satisfactory for the latest corrected microscopes let alone low voltage TEM. Scaling down lamella thickness using conventional FIB techniques is hampered by three detrimental effects: warping, amorphization, and shrinkage. Warping is caused when a lamella is thinned below a certain threshold due to intrinsic or processing induced strain. Special mounting and adaptive milling techniques have been proposed to reduce the effect [3]. Nevertheless mechanically stable large coplanar transparent areas have not been obtained. Amorphization is a side effect of ion milling. Depending on incident angle and energy the lamella is amorphized to a certain depth (~30 nm in Si for 30 kV Ga ions). Naturally the amorphization depth has to be much smaller than half of the lamella thickness. This is achieved by polishing the lamella with reduced ion energy. In conventional processing this causes difficulties since the beam shape degrades significantly with reduced energy. Shrinking of the lamella is observed during the final thinning process. With the removal of only a few tens of nanometers in thickness the height of the lamella's thinned area can be reduced by several micrometers. This effect is typically counteracted by the deposition of very thick protective layers — a very time consuming process.

We propose an elegant method based on conventional lift-out technique [4] that addresses the problems mentioned above. The essential difference is that the final thinning of the lamella is not performed with the same milling direction for both sides. Instead, after the first side is thinned the sample is rotated around its TEM observation direction by ~90 degrees. This creates a thin window where the two milling grooves overlap (Fig. 1). The process is repeated in several steps while reducing ion energy until the desired thickness is reached. In our Zeiss NVision 40 Ar a final low kV Argon polishing step can be applied to remove residual Ga contamination. The process yields a sturdy lamella with one or several electron transparent windows. Due to the window geometry even a very large region of interest can be milled to extremely small thicknesses without bending. In addition, shrinkage of the lamella does not occur since geometrical sputtering yield effects are efficiently suppressed. This also dramatically lifts milling angle restriction for low polishing with broadened beam diameter. A special specimen holder was designed to perform the sample rotation with each 180 degrees rotation of the microscope stage. Thus the switching between milling

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direction takes place reliably and the milled side is always conveniently facing the SEM for visual process control. Planarity of the window can be effortlessly adjusted to well below one degree deviation — enough to theoretically obtain an atomically flat specimen over the relevant field of view.

Fig. 2 shows an HRTEM image obtained with a Cs-corrected TEM at 20 kV on a silicon lamella prepared using the described method. The specimen thickness at this location was determined at 4 nm by EELS measurement.

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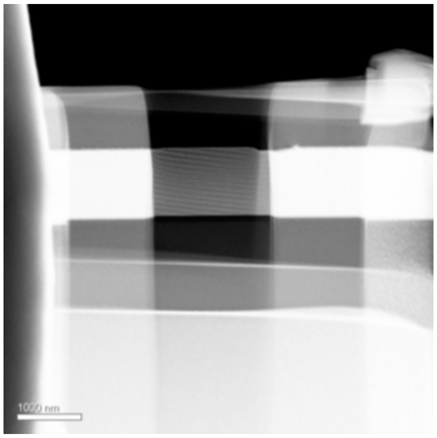

FIG. 1. HAADF STEM image of an x-ray mirror. The frontside of the lamella was thinned vertically, the backside horizontally. The overlap region is electron transparent and a Moiré pattern can be seen from the layered mirror structure.

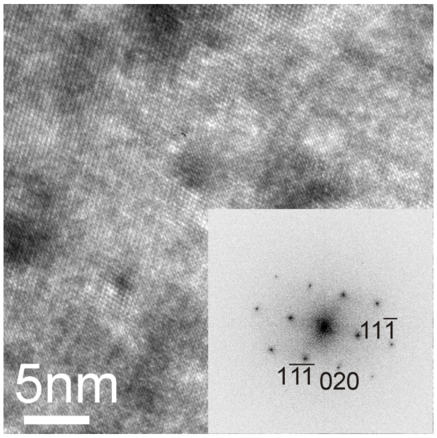

FIG. 2. HRTEM image and corresponding Fourier transform of a 4 nm thick FIB prepared Si lamella demonstrating atomic resolution at 20 kV.

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