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Features of large-scale thin foils fabrication for transmission electron microscopy by focused ion beam

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Abstract. Focused ion beam (FIB) sample preparation for transmission electron microscopy is a well established technique. However there are some problems related with high-quality large-scale (over 10 um) sample preparation of thin foils. In this work we demonstrate the successful preparation of such samples on example of ferrite steel sample and present the refined lift-out preparation technique, which was applied here. All sample preparation has been carried out in conventional single-beam FIB Hitachi FB-2100 with additional low-kV Ar^{+} ion polishing.

1. Introduction

Transmission electron microscopy (TEM) is one of the main direct methods for materials structure studying down to the atomic level. For successful study TEM specimens, depending on the objective of the study, should be from a few atomic layers to 100 nm thick (high resolution TEM - from a few atomic layers to 20 nm, crystal lattice defects and diffraction analysis - from 20 to 60 nm, elemental analysis - from 60 to 100 nm) and minimum preparation artefacts.

Nowadays, focused ion beam (FIB) microscopy is a well established tool for TEM specimens preparation due to ability to remove material in a highly localized area. Sample preparation capabilities of FIBs can be further extended by introducing micro-manipulators and gas- injection systems (GIS) into the microscope chamber. The first allows in-situ manipulations such as lift-outs, rotations and transfers of microscopic sample parts during preparation, while the second enables both site-specific material deposition and material-specific preferential milling by introducing reactive gases in the vicinity of the electron or ion probe during operation. Fully-equipped dual-beam FIBs (DB-FIBs) offer great flexibility in TEM sample preparation and many preparation techniques have been developed and reviewed over the years [1-5]. There are several, often unique, advantages of FIB preparation. The ability to cut and extract small quantities of any hardness material (including such materials as sapphire and diamond) and mount these pieces at fairly exposed positions on dedicated support grids generally helps minimize detrimental bulk-material effects such as charging, magnetic fields or surface migration of contaminants. Finally, FIB sample preparation is generally considered a fairly fast technique, capable of producing a complete sample from a bulk specimen within hours. However, it is sometimes useful to keep in mind that the pursuit of speed often comes at the cost of reduced sample quality [6].

The disadvantages of this technique include amorphization of samples (damage layer thickness is

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up to 30 nm) [7], the introduction of defects and the locality of the method (TEM area is up to 2-3 μm). Another disadvantage of TEM sample preparation by FIB is the inability to prepare a thin foil (lamella) with a size larger than 5 um and thinner than 100 nm. Since, when trying to obtain a larger sample, lamella is bent due to internal mechanical stresses, as well as due to the impact of an ion beam with high energies, and the sample becomes unsuitable for further research.

Modern DB-FIBs allow low-kV Ga+ ion polishing, which removes the damage layer and preparation defects and obtain 20 nm thick lamella. However, not every science center has such facilities. Therefore, when preparing samples using a conventional FIB (including single-beam FIB), additional polishing with low-energy Ar + ions is used to remove the re-precipitated and destroyed layers, but lamella larger than 5 um will bend for the reasons described above.

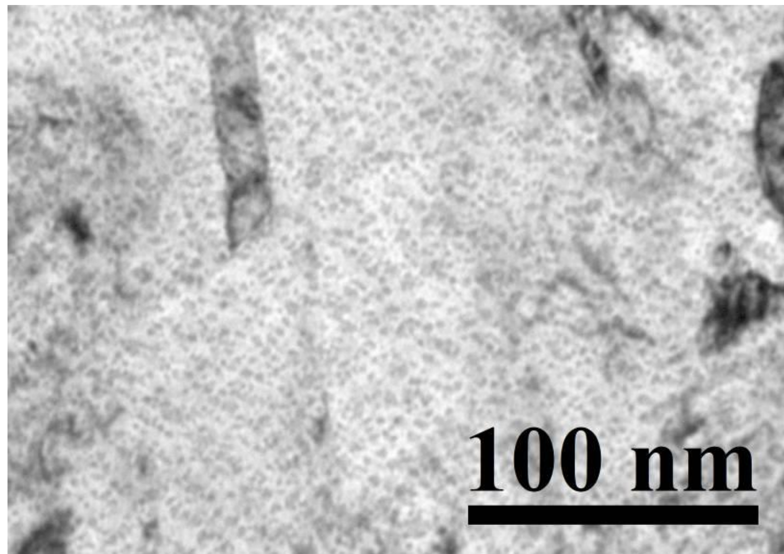

Figure 1. Re-deposition (nanoparticles) of the support grid material onto sample (austenite steel) surface.

Also, the re-deposition (figure 1) of the support grid material on the sample occurs when Ar^+ ion polishing, if the classic method of lamella mounting on support grid [6] is used. The re-deposition caused by different etching speed and angle of support grid and lamella.

2. Experimental and Results

FIB sample preparation was carried out on conventional single-beam FIB system Hitachi FB-2100 at 40 kV Ga^+ ion source. Final 2 kV Ar^+ ion polishing was carried out on Linda Technoorg ion mill system. TEM studies were carried out on conventional TEM Hitachi HT 7700 at 100 kV (W-sourse).

The proposed solution of described above problems is modification of the lamella support grid (figure 2 a). Immediately note that molybdenum support grid is better to use, because molybdenum sputtering ratio is much less than that of copper. Firstly is mechanically thinning the lamella support grid to ~ 5 μm. Secondly is high-intensity ion beam etching groove (figure 2 b) in which the sample is mounted. Mechanical thinning of lamella support grid avoids the excess consumption of gallium when etching the groove. In this position, the sample is firmly fixed on both sides with a deposition mask (figure 2 c) and is not deformed with further thinning. At the final stage of thinning lamella is tilted at a small angle (1-1.5 degrees) to obtain a uniform sample thickness throughout the depth.

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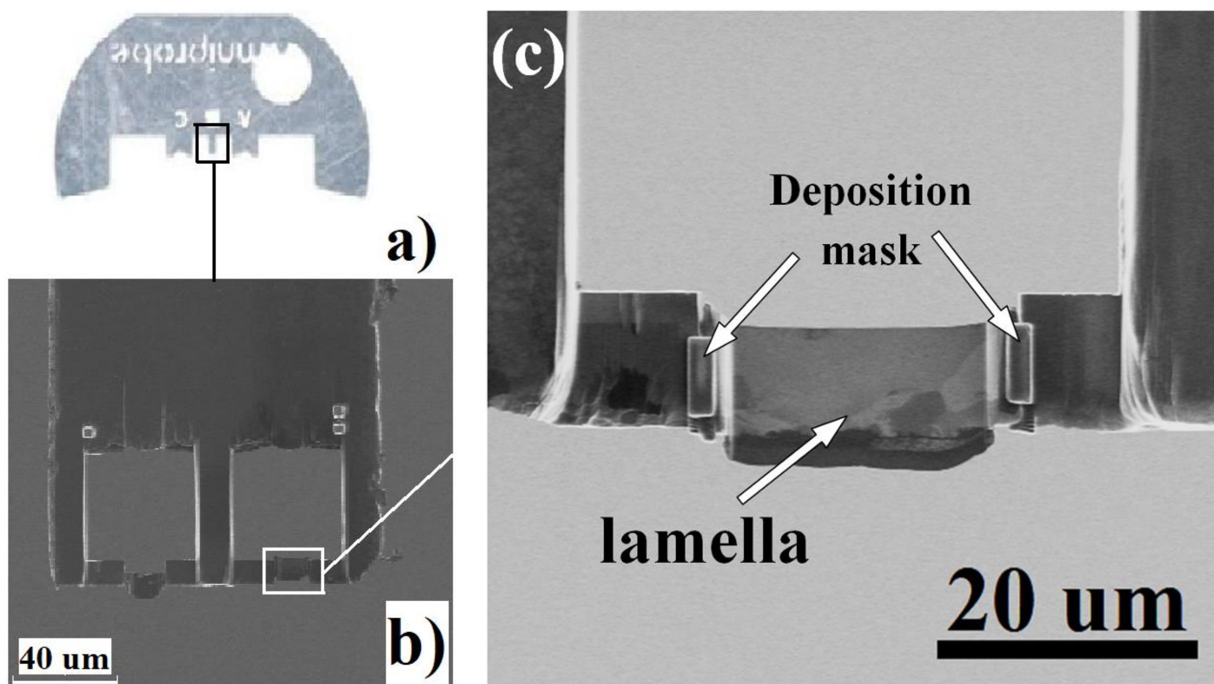

Figure 2. Modification of the lamella support grid: a) overview of support grid; b) enlarged fragment; c) the lamella mounting position.

After final Ga^+ ion milling lamella thick is consist ~ 150 nm. The sample of this quality is unsuitable for TEM studies (figure 3 a). Additional low-kV Ar^+ ion polishing at small angle (5-7 °) allows to thin the sample to the desired thickness (figure 3 b) also remove amorphous layer and Ga^+ ion beam artefacts.

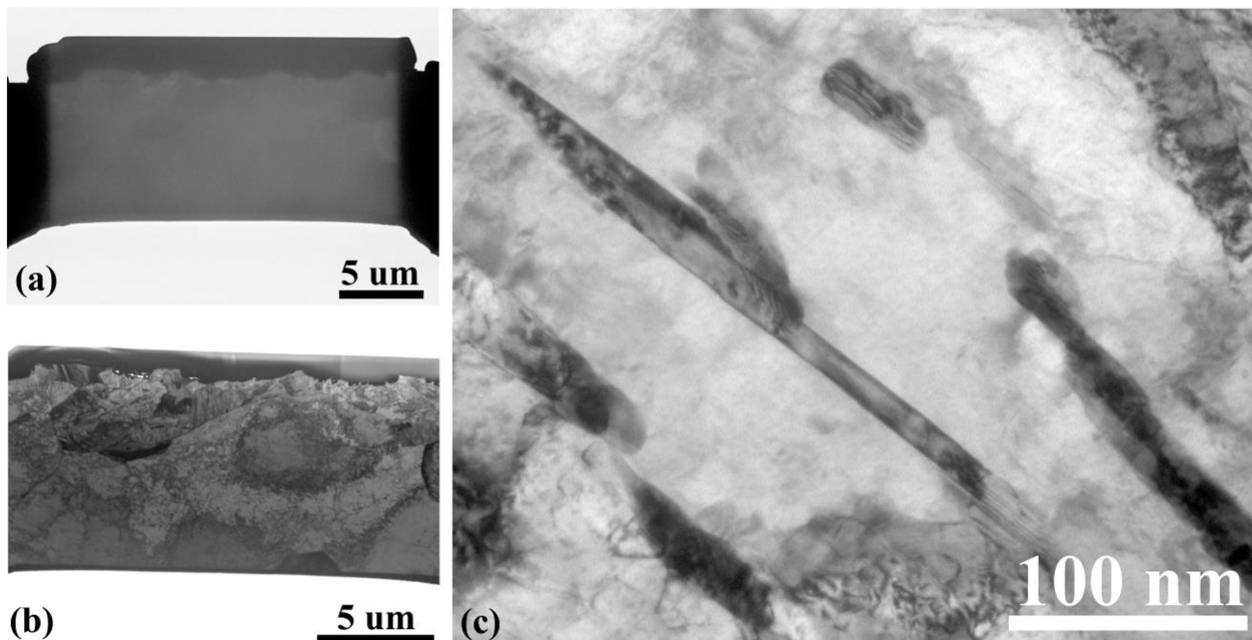

Figure 3. TEM studies of ferrite steel lamella: (a) Low-magnification TEM image after FIB preparation, (b) Low-magnification TEM image after 2 kV Ar^+ ion polishing, (c) Medium magnification TEM image of austenite grain fragment with carbide phase inclusions.

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Medium magnification TEM image (figure 3 c) clearly demonstrate high-quality sample preparation without amorphization, Ga^+ ion beam artefacts and re-deposition.

3. Conclusion

In this work, we demonstrate that conventional FIB system together with low-kV Ar^+ ion polishing allows to obtained high-quality large-scale (~ 20x10 um and more) samples for TEM examinations. It is possible due to special samples support grid modification.

FIB and TEM studies were carried out at the Center for Collective Use of the Krasnoyarsk Scientific Center of the Siberian Branch of the Russian Academy of Sciences.

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