824
doi:10.1017/S1431927618004610

Microsc. Microanal. 24 (Suppl 1), 2018

An Improved FIB Sample Preparation Technique for Site-specific Plan-view Specimens: A New Cutting Geometry

Chen Li[1,2,*], Gerlinde Habler[2], Lisa C. Baldwin[2] and Rainer Abart[2]

[1.] Stuttgart Center for Electron Microscopy, Max Planck Institute for Solid State Research, Stuttgart, Germany.
[2.] Department of Lithospheric Research, University of Vienna, Vienna, Austria
* Corresponding author email: chen.li@fkf.mpg.de

Focused ion beam (FIB) in-situ lift-out sample preparation is a powerful technique on preparing specimens for transmission electron microscopy (TEM) studies. One significant advantage is that FIB allows precise selection of targeted areas with spatial accuracy given by the ion beam size. Currently, the most developed FIB sample-preparation technique is using a cross-section geometry, which produces a lamella perpendicular to the bulk material surface. However, the plan-view lift-out technique, in which the lamella is oriented parallel to the bulk surface, has an advantage over the cross-section lift-out method: it allows the direct correlation of microscale morphology or property to further atomic TEM studies. Fig. 1 shows a comparison between the FIB lift-out specimens prepared using cross-section (Fig. 1b) and plan-view (Fig. 1c) geometries on a spinel-corundum interface. The interface geometry observed in the plan-view specimen (Fig. 1c) clearly matches the electron backscatter diffraction (EBSD) orientation relationship map inside the red box in Fig. 1a where the specimen was extracted.

However the plan-view FIB sample preparation technique has not been widely applied yet, as the preparation technique is much more demanding as compared to the cross-section technique. In order to extract plan-view specimens, the bulk surface remains horizontal, and the material underneath the targeted specimen needs to be removed in order to entirely detach the specimen from the bulk material. One important issue is that, without removing a large enough amount of the material surrounding the targeted specimen, it is hard to directly observe whether or not the specimen has been cut free. However, in many cases the surrounding material is also of interest, for instance when it exhibits different properties. This paper presents a more general plan-view FIB lift-out technique, which is adapted to minimize damage to the surrounding material and to allow extraction of relatively large specimens. For this purpose, a new cutting geometry is introduced (Fig. 2). Pt layers are used to mark the region of interest. Subsequently channels are milled surrounding this region leaving a bridge for supporting the "isolated" cuboid-shaped specimen. Next the two long sides of the "isolated" cuboid are wedged, forming two triangles at the cross sections of the triangular prism-shaped specimen. A micromanipulator needle is then used for in-situ transfer of the specimen to a FIB TEM grid, which is mounted parallel to the specimen surface. Finally, the grid is transferred to the standard FIB grid holder for final thinning with standard procedures as for cross-section lift-out techniques. Preparing specimens in such an "isolated" cuboid geometry is the key for this improved technique, because it not only makes the wedging processes in the following steps efficient, but also provides clear viewing angles for monitoring the milling process in between the steps. This minimizes damages on surrounding material and solves the difficulty of judging whether the specimen is entirely detached from the bulk material, thus improving the success rate. Besides, this new cutting geometry provides a larger ratio of the usable specimen surface area to the volume of the material needed to be transferred by the needle.

This plan-view FIB technique has the advantage to allow for directly linking atomic scale TEM investigations with any SEM-based microstructure study including EBSD, cathodoluminescence (CL) and electron beam induced current (EBIC). It can also be extended to extract specimens mapped with other techniques such as optical microscopy, scanning tunneling microscopy (STM) and atomic force microscopy (AFM), after extra steps of location identification using features visible in SEM images. Several examples [2] on such material science application will be presented in the meeting [3].

References:

[1] C Li *et al*, Ultramicroscopy **184** (2018) 310–317

[2] C Li *et al*, Migration of metal oxide interfaces via the glide of partial dislocations, Under review.

[3] Research funded by EU's Marie Skłodowska-Curie grant No. 656378 – Interfacial Reactions (CL), and the Austrian Science Fund FWF: I1704-N19 in research group FOR741-DACH (GH).

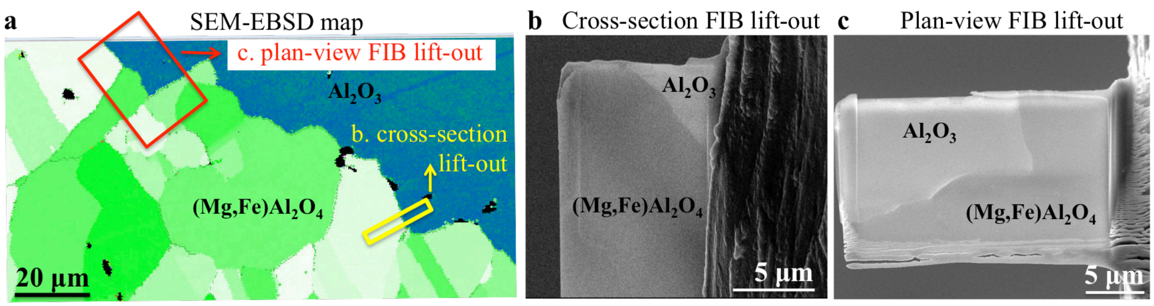

Figure. 1 Comparison of FIB lift-out techniques with cross-section and plan-view cutting geometries. (a) SEM-EBSD inverse pole figure map providing information on the orientation relationships between polycrystalline (Mg,Fe)Al_2O_4 spinel grains (light to dark green) and single crystal Al_2O_3 corundum (blue), where the color schemes are used to indicate different grain orientations. (b) and (c) are SE images of the TEM specimens prepared in cross-section and plan-view geometries, which were extracted from the areas marked by the yellow box and the red box in (a), respectively. [1]

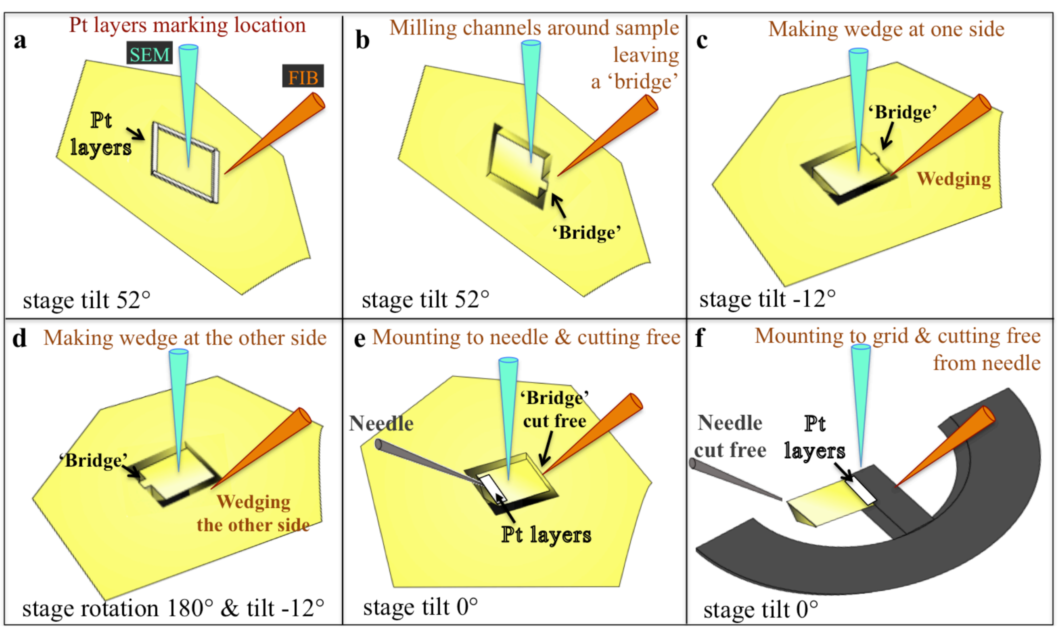

Figure. 2 Sketches illustrating a new cutting geometry for plan-view FIB lift-out sample preparation. [1]

https://doi.org/10.1017/S1431927618004610 Published online by Cambridge University Press