Use of cleaved wedge geometry for plan-view transmission electron microscopy sample preparation

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1 | INTRODUCTION

TEM is the most established tool for simultaneous acquisition of structural, crystallographic, and compositional analysis of micro- and nanoengineered films at the atomic level (Williams & Carter, 2009). Yet, TEM specimen preparation commonly presents a practical challenge for most samples and frequently limits the level of detail available through TEM characterization. The diversity of accessible preparation methods makes TEM sample preparation both an art and a science. It requires special skills and experience for achieving accurate control of the preparation process (Ayache, Beaunier, Boumendil, Ehret, & Laub, 2010).

To provide consistent analysis about the film structure, it is essential to observe the film from two (minimum) perpendicular directions. To achieve this, TEM samples of different geometries are prepared, which typically includes cross-sectional (side-view) and plan-view preparation of, for example, a film. TEM sample preparation of films deposited on brittle substrates typically involves conventional mechanical and Ar ion beam thinning or FIB techniques. A cross-sectional TEM specimen preparation is performed via the well-established mechanical-Ar ion procedures (Barna, Pécz, & Menyhárd, 1999; Dieterle, Butz, & Müllera, 2011; Süess, Mueller, & Wepf, 2011), cleavage (McCaffrey, 1993), or lift-out approaches (Anderson & Klepeis, 2005; Giannuzzi & Stevie, 2005; Langford & Petford-Long, 2000; Orloff, Utlaut, & Swanson, 2003; Schaffer, Schaffer, & Ramasse, 2012), while the plan-view TEM sample preparation currently presents a challenge for the films deposited on the brittle substrates. As an example, once the thickness of the sample approaches hundred micrometers, during mechanical thinning, the sample handling becomes an issue due to bending and breaking risks (Sáfrán & Grenet, 2002; Sáfrán, Szász, & Sáfrán, 2015). While during Ar ion beam thinning, very accurate control of the thinning process is required for revealing the intended structure (Radnóci & Pécz, 2006). The FIB lift-out of plan-view samples is not straightforward either and requires, for example, advanced cutting geometries, prolonged milling time, and processing (Giannuzzi & Stevie, 1999; Jublot & Texier, 2014; Kim, Nam, Choi, & Park, 2015; Langford, Huang, ...
Lozano-Perez, Titchmarsh, & Petford-Long, 2001; Lenrick, Ek, Jacobsson, Borgström, & Wallenberg, 2014; Li, Habler, Baldwin, & Abart, 2018; O’Shea et al., 2014; Stevie et al., 1998). Existing plan-view sample preparation approaches seems as rather complex, time consuming, limited throughput, and prone for specimen failures. Additionally, if depth sectioning of the plan-view sample is desired, FIB is the only method where lift-out of each section significantly reduces the throughput.

To rectify these challenges, I have developed a fast and flexible solution for plan-view TEM sample with an emphasis on films deposited on brittle substrates. It combines the simplicity of conventional cleaving with the precision of FIB. The preparation procedure is provided in detail step-by-step, and key factors to ensure success are elucidated.

2 | PREPARATION METHOD

The proposed plan-view TEM sample preparation procedure consists of mechanical and ion-beam treatment stages. The description of involved stages, composed of number of steps, is discussed and demonstrated in detail below in Figures 1 and 2. The method is verified using a ~2-µm-thick TiB$_2$ film deposited on the Al$_2$O$_3$ substrate (~500 µm thick), with an intentionally oxidized surface.

![Figure 1: Mechanical treatment steps: 1.1 sawing (a–e), 1.2 cleaving (f–h), 1.3 mounting (i–k) and 1.4 back polishing (l, m) involved in the proposed preparation method. (Color figure can be viewed at wileyonlinelibrary.com)
2.1 | Mechanical processing

The purpose of the mechanical processing is to obtain a sample that possess a wedge-shape geometry from the substrate-side, mounted and immobilized onto a support grid compatible with FIB processing and standard TEM holders. Tools and supplies used to exemplify this procedure included a low-speed diamond wheel saw (Model 650, South Bay Technologies), diamond blade (diameter: 76 mm and thickness: 0.2 mm), a razor blade (thickness: 0.15 mm), glue (Gatan G-1 epoxy), and conventional polishing paper. Optical micrographs of the mechanical processes are shown step-by-step in Figure 1. All in all, the mechanical processing of the sample, as shown below, required approximately 1.5–2 hr of work.

2.1.1 | Sawing

The preparation procedure starts by cutting a segment, ~1.8 mm wide, from the as-received sample using a low-speed wheel saw (Figure 1a,c,e). Prior to cutting, the film side of the sample was glued to a glass slide for protecting the film surface during cutting and handling. The length of the strip will define the number of potential cleaved pieces and should not be shorter than ~1.6 mm. The collected segment (~1.8 mm wide) is remounted onto a fresh glass plate. A series of cuts into the sample is performed, 90° to the section sides, with a separation of ~0.8 mm. The dimension of segment (1.8 mm × 0.8 mm) is compatible with the grid size onto which segment will be mounted (see Figure 1i–m). The cuts are performed from the substrate-side and must not penetrate all the way through the sample (Figure 1b,d). The depths of these cuts are of paramount importance. If the cuts are too deep, the pieces will separate while removing the sample from the glass plate. If they are too shallow, it will be challenging to cleave the sample in the next step. Preferably, the cuts should penetrate more than half-way through the substrate thickness (Figure 1d). Important to note that the sample area under in the cuts will eventually become the electron transparent window(s) after FIB processing step (shown in Figure 2).

2.1.2 | Cleaving

The section now requires cleaving to obtain pieces exhibiting wedge-shaped geometries at the sample edge. The cleaving procedure is performed by using a razor blade, which is inserted in the produced cut, and consecutively bent by applying a force parallel to the film surface (see the schematic shown in Figure 1g). As a result of cleaving, a wedge-shape geometry is produced at the sample edge. The cleaving procedure included a low-speed diamond wheel saw (Model 650, South Bay Technologies), diamond blade (diameter: 76 mm and thickness: 0.2 mm), a razor blade (thickness: 0.15 mm), glue (Gatan G-1 epoxy), and conventional polishing paper. Optical micrographs of the mechanical processes are shown step-by-step in Figure 1. All in all, the mechanical processing of the sample, as shown below, required approximately 1.5–2 hr of work.

2.1.3 | Mounting

The cleaved sample piece needs to be mounted onto the standard half-moon grid (e.g., Cu) compatible with FIB sample preparation. The applied glue preferably needs to ensure fast hardening and for this reason G1 epoxy was chosen. A minute amount of G1 epoxy was applied to the central part of grid using a thin metal wire. The cleaved sample piece was then placed onto the grid with the film side of the sample facing the grid and wedge sticking out from the grid (Figure 1i–k). The surface of the wedge-shape must remain free from the applied glue. In case the sample gets contaminated with the glue (wrong positioning, excessive glue, or sliding of the piece), it may be cleaned using, for example, acetone and the gluing procedure repeated. For fast hardening of G1 epoxy, the glass plate holding grid and sample was placed onto a hotplate heated to ~200°C for ~2 min.

2.1.4 | Polishing

The sample requires thinning for easy handling both in the FIB and in the TEM (Figure 1l-m). Prior to back polishing, the sample was fixed onto a glass plate using wax with the film side of the sample facing the glass plate. The substrate side of the sample was consequently polished with an abrasive diamond paper of 30 μm roughness. The thickness of the sample was reduced from ~500 μm (initial substrate thickness) to ~150–200 μm. The sample was cleaned using acetone and isopropanol and ready for the FIB processing.

2.2 | Focused ion beam milling

The purpose of the FIB milling procedure is to prepare electron transparent window(s) located at the apex of the wedge-shaped sample which will be ready for TEM examination. The milling shown in the examples below was performed in a FIB instrument (Carl Zeiss cross beam 1540 ESB system). SEM images of the ionic preparation steps from major perspectives are shown in Figure 2. The procedure is straightforward and involves gentle milling of the sample apexes using low milling currents (20–200 pA). This part of the process required ~0.5 hr and will scale linearly with the number of electron transparent windows.

2.2.1 | Milling from the substrate-side

The sample was loaded in the FIB system standard way (the grid is standing up), once the site-specific area was identified along the apex, the sample was aligned through rotation for performing milling from the substrate-side. The sample stage was tilted to 54° (as in Carl Zeiss system FIB column leaned to 54° from SEM column) to reach the configuration in which the FIB beam is parallel to the film surface (Figure 2a–c). As the purpose of the procedure to obtain plan-view TEM sample from the as-grown film, the ion
The milling procedure was set to locally remove the Al₂O₃ substrate and part of the film in a ~3-μm-wide window (Figure 2d–f). The milling was performed initially using 200 pA (30 kV) currents while the final cleaning carried out using 20 pA (30 kV) currents. Additionally, from the SEM micrographs (Figure 2) it can be observed that the film has two different microstructures, where top film surface is caused by intentional partial oxidation of the film as indicated in Figure 2e.

### 2.2.2 Milling from the film-side

To finalize the preparation process and to obtain an electron transparent window, the milling was performed from the film surface side. For this step, the sample was rotated 180° while maintaining the 54° tilt angle. Due to the limited thickness of the film remaining to be milled, a low-current ion beam of 20 pA (30 kV) was used to finalize the milling (Figure 2f–h), while additionally minimizing the surface damage caused by the ion beam (Giannuzzi & Stevie, 1999; Jublot & Texier, 2014; Mayer, Giannuzzi, Kamino, & Michael, 2007; Mehrtens, Bley, Satyam, & Rosenauer, 2012). After completing this step, the sample is ready for TEM analysis.

For ion beam sensitive samples, prior to ion milling, the targeted area is preferably protected by depositing a strip of, for example, protective Pt. Additionally, the width of the milling window (in this case ~3 μm) could be varied depending on the material. It was noted that some materials produce high quality lamellas for wider windows (e.g., ~5 μm) while for other, for example, film with high internal stresses, the lamellas bend, and thus the window width should be reduced. The same applies for optimizing milling angle within few degrees range and milling currents.

### 2.3 Depth sectioning

This approach enables the preparation of the multiple windows for depth sectioning of the film, as illustrated by the SEM images shown in Figure 3. Multiple windows are exemplified on a ~3-μm-thick Ti(Al) B₂₋₄ film deposited on the Si substrate, with an intentionally oxidized surface.
Figure 3 shows how five windows were prepared at varying depth for each individual window. The depth was varied gradually from substrate (I), to as-grown film (II), as-grown film-oxidized film interface (III), oxidized film (IV) all the way to the oxidized film top surface (V). This precise positioning enables sectioning of the sample with high accuracy and provides depth resolved information from the film along the growth direction without overlap from adjacent regions that cause interference and uncertainty.

2.4 Lift-out from the cleaved wedge

The plan-view FIB lift-out technique has limited applications, as the procedure for it is much more stringent than for lift-out cross-sections (Li et al., 2018; Stevie et al., 1998). It suffers from challenges related to, for example, the redeposition of sputtered material (causing problems to detach the specimen from the bulk sample) together with impeached monitoring of the milling processes underneath the target area resulting to specimen failures. Further, for such cases as preparing TEM sample (from bulk sample) on microelectromechanical systems (MEMS) chips—the FIB lift-out procedure is the only option (Duchamp, Xu, & Dunin-Borkowski, 2014). In the light of current challenges and needs, the cleaved wedge geometry offers the unique opportunities for efficient lift-out approach as demonstrated in Figure 4. The plan-view FIB lift-out technique is verified using a ~400-nm-thick TiB₂ film deposited on the Al₂O₃ substrate (~500 μm thick) onto MEMS heating chip.

### 2.4.1 Milling trench from the substrate-side

The sample was loaded in the FIB system standard way (the grid is standing up) and tilted to 54°, identical to Figure 2 configuration. The ion milling procedure was set to locally remove the Al₂O₃ substrate in a ~20-μm-wide and ~4-μm-broad area (Figure 4a). The milling was performed using 2 nA (30 kV) current.

### 2.4.2 Milling frame around lamella from the substrate-side

The sample was tilted to 0° and substrate-side orientated towards the FIB gun through stage rotation of 180°. The ion milling procedure was set to locally remove the material around the lamella in the frame fashion which makes the lamella hold to the bulk though the connecting bridge (Figure 4b). The frame milling was performed using 2 nA (30 kV) current.

### 2.4.3 Milling electron transparent window

The sample was tilted back to 54°, the same configuration as in 4.1 step. To finalize the pre-lift-out procedure, an electron transparent window was obtained by the milling a ~3-μm-wide window on the right end of lamella (opposite to the bridge) from the substrate and...
film (rotated 180°) surface side (Figure 4c, identical to Figure 2d–h). The milling was performed initially using 50 pA (30 kV) currents while the final cleaning carried out using 20 pA (30 kV) currents.

2.4.4 Lift-out onto MEMS chip

For executing lift-out procedure the sample needs to be reloaded in the FIB system with the grid laying down (instead of standing up) with the substrate-side facing the SEM column. The manipulator is inserted and welded to the lamella followed by cutting the connecting bridge. The lamella is transferred onto the laying down MEMS chip and welded to it (Figure 4d). Finally, the lamella is cut loose from the needle and procedure is complete (Figure 4e). Important to note, although the electron transparent window was obtained before the actual lift-out, it provides the high-quality STEM images after complete processing (Figure 4f). For achieving this, the electron transparent window should not be images with FIB/SEM during 4.4–4.5 steps as it might results in contamination. Alternatively, the lift-out procedure can be attempted after milling frame around lamella (4.2) and reloading the sample, while final milling can be performed after completing the sample transfer onto the MEMS chip.

In the case, plan-view FIB lift-out from the cleaved wedge is desired onto the standard (e.g., half-moon Cu) grid, the lift-out procedure needs be executed after milling frame around lamella (4.2) without the need to reload the sample.

3 RESULTS AND DISCUSSION

The resulting plan-view TEM sample characteristics together with microstructure of the film were explored using scanning TEM high angle annular dark field (STEM-HAADF) imaging and selective area electron diffraction (SAED). Microscopy was performed in double-corrected Linköping FEI Titan3 60–300, operated at 300 kV. Figure 5 represents a series of STEM images, with increasing magnification, acquired from the plan-view TEM sample shown in Figure 2.

Figure 5a shows an overview STEM image of the final window. The width and height of the lamella was estimated ~3 and ~5 μm, respectively. The homogenous STEM contrast within the lamella region indicates no bending artifacts (consistent with SEM observations in Figure 2g) or pronounced thickness variations. Figure 5b shows a higher magnification STEM image from the TiB$_2$Δ film. The lamella thickness was rather uniform although monotonically increasing while moving away from the edge, as judged by the STEM intensity increase, indicated as a line profile in Figure 5b. Additionally, the tiny amorphized layer was present on the top of the lamella which comes as an artifact from the milling process and is typical for the employed approach. Although it does not affect the structure below, it can be avoided by depositing protective layer before ion milling procedure.

The microstructure of the TiB$_{2}$Δ film is easily accessed and owns a dense nanocolumnar grain structure. SAED pattern shows that the film is constituted of the TiB$_2$ phase with pronounced (0001) texture. In Figure 5c, high-resolution STEM image reviewed that nanocolumns are composed of the subcolumns which are separated with dark-contrast regions attributed to high boron content typically observed in overstoichiometric TiB$_{2}$Δ films (Mayrhofer, Mitterer, Wen, Greene, & Petrov, 2005).

STEM images in Figure 5b,c reveals the atomic scale characteristics of the film and proves the proposed preparation method to be capable of delivering a high-quality specimen for plan-view TEM analysis. In fact, the maturity of the proposed preparation method is verified by its successful application in a handful of the studies elsewhere.
Further, it was observed that for films with a weak adhesion to the substrate, proposed sample cleaving produce (Figure 1) produces the cleave wedge with the substrate-free films sticking out from the wedge. In such cases, FIB milling procedure is even more time efficient as enables to prepare electron transparent window(s) without the need to mill the substrate.

Depth sectioning is commonly neglected in plan-view TEM investigations. The applied approach facilitated the multiple-windows sectioning of this partly oxidized film for decoding the oxidation mechanisms in understoichiometric Ti(Al)B$_2$-$_\Delta$ films (see Figure 6) and further described elsewhere (Bakhit, Palisaitis, Persson, et al., 2020; Bakhit, Palisaitis, Thörnberg, et al., 2020).

A range of films deposited on Si, SiC, and Al$_2$O$_3$ brittle substrates have been successfully prepared for plan-view TEM investigations by the proposed preparation method. Plan-view samples of films as thin as ~100 nm have been successfully realized; however, additional...
attention must be paid during milling of thin film samples, and milling from the film-side could be omitted if needed.

The relatively big sample dimensions (1.8 × 0.8 × 0.15 mm³) and immobilization onto the half-moon grid reduces the mechanical stress and supports “curved in” electron transparent windows after the sample preparation. This, in turn, ensures the TEM sample's rigidity and minimizes the failure risk. In very rare case of sample separation from the grid, the sample can easily be retrieved and glued back onto the grid. If a window gets broken, a new one can be easily produced from the existing piece.

Finally, the plan-view FIB lift-out of lamella from the cleaved wedge shape apexes were successfully demonstrated for preparing TEM samples on MEMS heating chips (Figure 4). Cleaved wedge geometry eases lift-out approach due to minute amount of material which needs to be removed (before the lift-out) and uninhibited monitoring of the milling process.

4 | CONCLUSIONS

The proposed preparation method was shown to provide a high throughput and yet informative route for examining films deposited on brittle substrates in plan-view. Few steps are needed to achieve electron transparency, and the method requires only rudimentary sample handling and FIBing skills. The substantial sample dimensions reduce sample bending from internal stresses, immobilize the curved-in windows, and benefit sample handling. Multiple side-by-side windows enable depth resolved sectioning of the sample, with the precision of the FIB system. Beyond traditional plan-view TEM sample preparation, cleaved wedge geometry proves to facilitate the plan-view FIB lift-out procedure. The approaches are compatible with the established TEM sample preparation techniques and can be easily adopted by laboratories equipped for TEM preparation.

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DATA AVAILABILITY STATEMENT

Data available on request from the authors.

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