Microsc. Microanal. 28 (Suppl 1), 2022
doi:10.1017/S1431927622001167

Advanced Large Area Sample Preparation for Electron Microscopy using Initial Notches

Richard Busch[1*], Michael Krause[1] and Thomas Höche[1]

[1.] Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle / Saale, Germany
* Corresponding author: Richard.Busch@imws.fraunhofer.de

Sample preparation is a decisive step for successful microscopy and microanalysis. High demands on sample geometry and surface quality need to be satisfied in order to achieve the best resolution and data quality. Targeted sample extraction or sectioning of samples with depth-dependent microstructure is often desired and routinely tackled using highly-resolved but laborious focused ion beam technology [1]. Here, we propose a novel approach using a particular sample geometry: initial notches (iNotch, [2]). Initial notches utilize the idea of preferential erosion at local surface elevations in order to facilitate the formation of receded terraces upon glancing angle ion beam erosion (Fig. 1). Crucially, this allows for targeted material removal for the purpose of milling or thinning in a self-aligned way at high ion beam currents. As a consequence, larger sample regions can be prepared more rapidly.

The process-relevant aspects of notch milling and terrace formation as well as the underlying physics were studied in detail. Initial notches were implemented with different technological approaches, using either ultra-short pulsed laser ablation or focused ion beam milling for notch cutting. Terrace formation was achieved using glancing-angle erosion of the notched surfaces with broad noble gas ion beams. The applicability to different materials (semiconductor, glass, glass ceramic) was demonstrated. Surface quality was characterized with optical profilometry and SEM and the ion-induced amorphization was studied with TEM. Terrace growth kinetics were analyzed using numerical simulations based on a deterministic model of the sputtering process. As proof-of-concept, initial notches were applied to depth-resolved electron backscatter diffraction studies and TEM lamella preparation (Fig. 2).

We found that high-quality samples can be prepared even for challenging materials. Surface roughness crucially depends on notch quality. Highly smooth terraces are created when notch faces are free of curtaining artefacts and suitable glancing-angle ion milling parameters are selected. Initially rough surfaces were successfully smoothed using initial notches. The terrace geometry could successfully be described by a linear model as a two-stage process of initial terrace formation and stationary terrace growth. The dependence of terrace formation and growth speed on process parameters like the ion incidence angle was determined. Based on these findings, guidelines for applications of initial notches are given.

Initial notches provide a lot of promise for flexible and efficient sample preparation work flows, with potential use case ranging from thinning of TEM lamella over sample sectioning for depth-resolved microstructure analysis to surface smoothing. In order to exploit this potential, future research will focus on integration of iNotches into existing workflows and instrumentation [5].

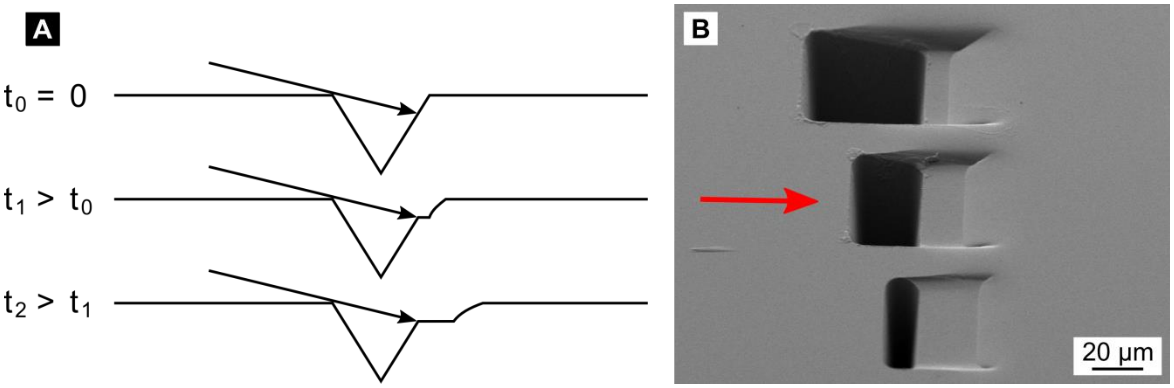

Figure 1. Sketch of the basic principle behind initial notches (A) and SEM micrograph of terraces formed on a notched Si surface upon gracing-incidence ion beam erosion (B). The arrow is indicating the incidence direction of the ion beam. (A) reprinted from [2], (B) reprinted from [3].

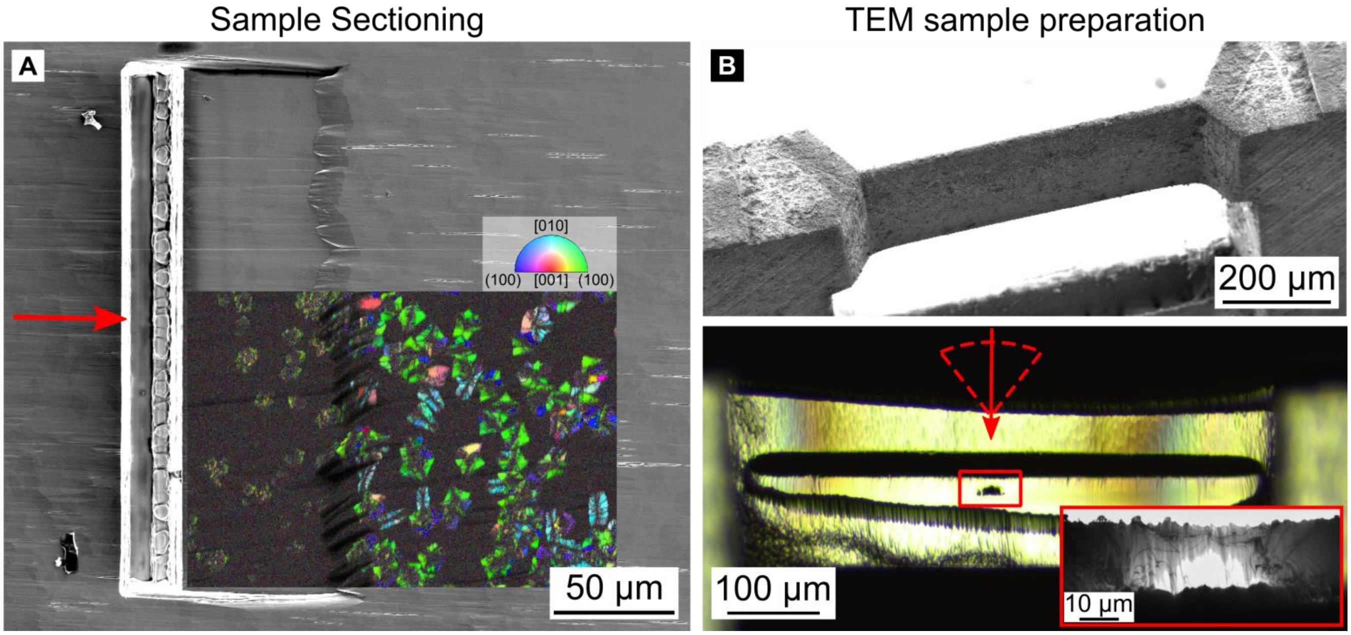

Figure 2. Two possible use case of initial notches: sectioning for analytical SEM measurements and TEM lamella preparation. (A) inverse pole figure map of a surface crystallized diopside glass ceramic after sectioning with initial notches, measured with electron backscatter diffraction. (B) TEM sample, where a base H-bar sample structure was created with laser machining (top) and thinned to electron transparency using an initial notch and glancing-angle broad ion beam erosion (bottom). Arrows are indicating the incidence directions of the ion beam. (A) adapted from [4], (B) adapted from [2].

References

[1] N Bassim, K Scott and L Giannuzzi, *MRS Bulletin* **39** (2014), pp. 317-325, doi:10.1557/mrs.2014.52
[2] R Busch, M Krause, S Coyle and T Höche, Micron **107** (2018), pp. 35-42, doi:10.1016/j.micron.2018.01.002
[3] R Busch, M Krause, and T Höche, Nucl. Instrum. Methods Phys. Res., Sect. B **509** (2021), pp. 12-20, doi:10.1016/j.nimb.2021.09.008

[4] R Busch, C Tielemann, S Reinsch, R Müller, C Patzig, M Krause, and T Höche, Micron **150** (2021), 103090, doi: 10.1016/j.micron.2021.103090

[5] Financial support from Deutsche Forschungsgemeinschaft (DFG) in Bonn Bad Godesberg (Germany) under grant PA 3097/1-1 and by the European Union via the Europäischer Fonds für Regionale Entwicklung (EFRE) and the Investitionsbank Sachsen-Anhalt (Germany) through the project FIBNotch under the grant number 2004/00071 is gratefully acknowledged.