microPREP™ PRO - Workflow for XRM Sample Preparation

Introduction

Initially developed at synchrotron radiation sources, X-ray microscopy (XRM) is coming to industrial and research labs where it will foster materials development, exploration, as well as failure analysis due to its high resolution (down to 50 nm) and non-destructive imaging capability. Since XRM requires samples of several tens of microns in diameter, out-of-the-box approaches to target sample preparation are required. microPREP™ PRO, the versatile, laser-based tool developed by 3D-Micromac, can provide samples of the required geometries at significantly higher rates than existing established sample preparation methods.

Motivation

Depending on the specific X-ray absorption length of a given material, the X-rays used in XRM can penetrate a depth between a few microns and several hundred microns. Ideally, an XRM sample is cylindrical in shape, facilitating tomographic rotation about its axis. This raises the question of how such samples can be prepared, especially when the target structure can only be a few microns in diameter. Focused ion-beam (FIB) micromachining is an established technique for preparing tiny samples, but the high precision inherent with FIB is accompanied by a severely limited ablation rate. This prevents FIB from being used for making XRM samples, since the time required to prepare a typical cylindrical sample of 60 µm diameter and 100 µm height would take about a week and is thus not practical or economic for a FIB system. Laser micromachining of the same geometry, however, takes less than 5 minutes. In addition to their high rate of ablation (up to 10,000x greater compared to FIB), lasers have the major advantage that photons are free from any elemental contaminations. Using the right setup, the width of the laser-affected zone can be tightly controlled to ensure micron-level accuracy.

Non-dedicated laser micromachining tools are available on the market, but lack the implementation of workflows to prepare required XRM geometries or do not use laser sources well-suited for XRM sample preparation. Another approach is to use plane-parallel samples, assessable by mechanical grinding and polishing, but samples prepared this way suffer from the missing-wedge issue inherent to tomography of non-rotational samples.

Laser-Based Approach to Sample Preparation

microPREP™ PRO was developed jointly by 3D-Micromac and Fraunhofer IMWS to serve a multitude of workflows. While sample preparation for scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atom probe tomography (APT) is always two-staged, with the laser providing the coarse milling at massive ablations rates and an ion-beam polishing the sample at much smaller rates, XRM sample prep can be performed entirely using the ultra-short pulsed laser integrated into microPREP.

There are three different workflows applicable to XRM preparation. The easiest, but often sufficient method includes mounting the sample onto a cutting holder, finding the target position using the integrated optical microscope on the 1-micron scale, and then exposing the flat sample to a circular laser trajectory, resulting in slightly conical samples that can be taken off the starting material (Fig. 1).

The second workflow involves taking off what is called an XL-Chunk™ (Fig. 2), mounting it onto a supporting structure (Fig. 3) and, if needed, reducing its diameter using workflow #1.

The third workflow is the most sophisticated, yielding non-conical, rotation-symmetrical samples. It is characterized by mounting the sample onto a dedicated, motorized jig (Fig. 4) that resembles the mount of a turning machine. The laser is then guided along a trajectory perpendicular to the rotation axis of the mount, while the mount hosting the sample is rotated non-continuously.

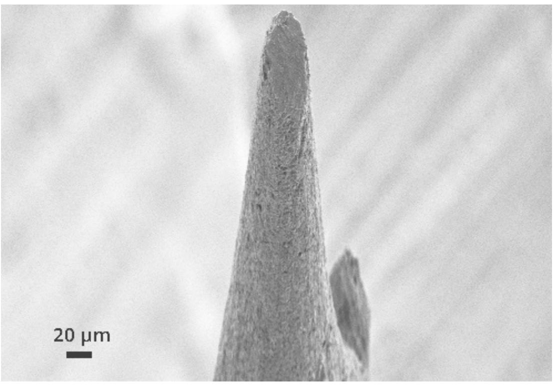

Fig. 1: SEM micrograph of an XRM-pillar in oil-shale

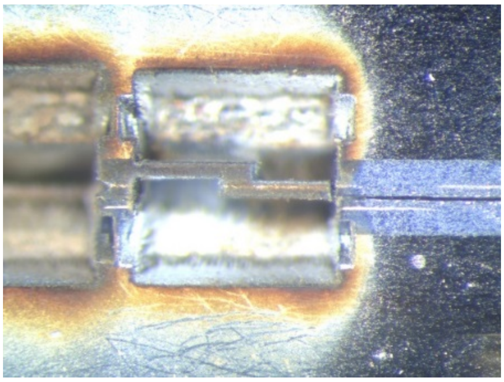

Fig. 2: Light microscopy micrograph of an XL-Chunk prepared from a mechanical cross section

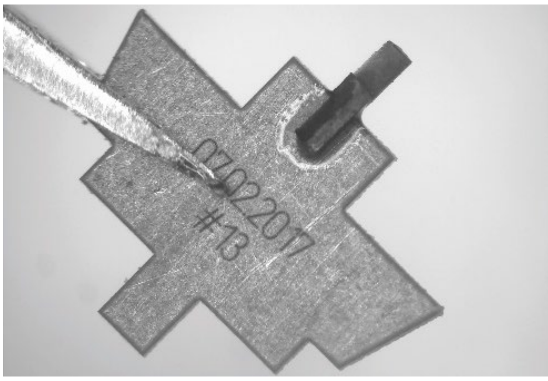

Fig. 3: XL-Chunk for XRM mounted on a carrier structure prior to final Laser-thinning

Fig. 4: Motorized XRM-holder for preparation of rotation-symmetrical samples

Conclusion

XRM workflows implemented into microPREP enable the preparation of artifact-free, geometry-adopted samples for XRM from target positions. The three workflows described take advantage of the ultra-short pulsed laser integrated into microPREP as well as means to observe laser micromachining and reduced thermal load to the sample.

For XRM users involved in materials development, failure analysis, or exploration, microPREP is a versatile, time-saving tool for obtaining optimal samples.

For more information, visit https://3d-micromac.com/laser-micromachining/products/microprep/.

Contact:

3D-Micromac AG

Technologie-Campus 8
09126 Chemnitz, Germany

Phone: +49 371 40043-0
E-Mail: sales@3d-micromac.com
Website: www.3d-micromac.com