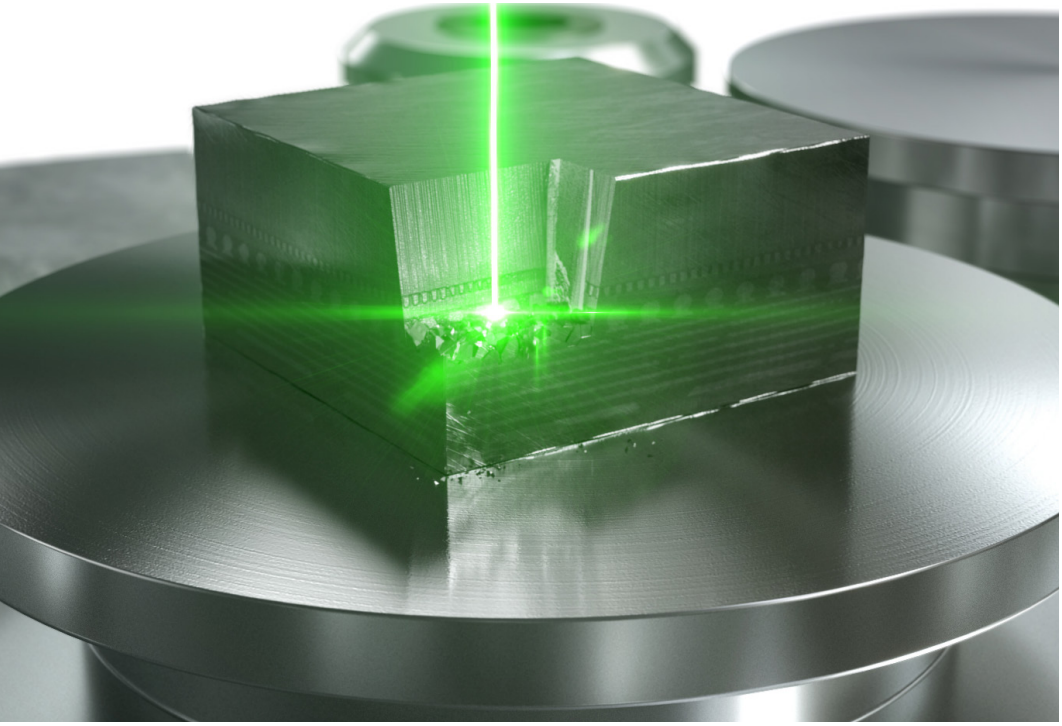
Application Note

Rapid Sample Preparation for EBSD-analysis

Enabled by the LaserFIB



ZEISS

Seeing beyond

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In the last years EBSD camera technology, pattern acquisition and indexing have been improved significantly and today acquisition rates of >3000 frames per second are becoming standard. With these improvements, EBSD has evolved from a purely scientific analysis method to be also applicable for industry and QA/QC purposes. The remaining time consuming and thus limiting factor is now sample preparation instead of acquisition and indexing. State-of-the-art preparation methods are mechanical polishing with a vibration-polish finish for large areas or focused ion beam (FIB) polishing for smaller areas and sensitive materials. Mechanical polishing requires less time than FIB polishing and is suitable for the preparation of large areas however it has limitations when it comes to target preparation. FIB polishing on the other hand is the method of choice for target preparation but is limited to small areas. To overcome limitations of both methods, the new femtosecond laser for ZEISS Crossbeam is used to rapidly prepare cross-sections in sheets of different metals and EBSD is performed on the laser-polished surfaces.

Introduction

Focused ion beam equipped scanning electron microscopes (FIB-SEM) have seen a strong increase in use in the field of materials science. Not just as a single purpose preparation tool for Transmission Electron Microscopy (TEM) but also as a versatile research instrument for direct microstructure preparation and materials characterization. A proven benefit of FIB preparation is location-specific cross-sectioning while maintaining a virtually deformation-free microstructure. With typical FIB beam diameters of about 5 nm to several micrometers, these systems are primarily used to section regions ranging from a few tens of atoms to a few tens of microns. Nevertheless, it is possible to prepare cross-sections that extend to several hundred micrometers in length and depth and thus provide better statistics in microstructure characterization. However, this has shown to be a time and cost intensive method.

Figure 1. *LaserFIB: ZEISS Crossbeam equipped with new femtosecond laser.*

To overcome said issues, nanosecond laser ablation systems were implemented in ZEISS FIB-SEM systems. Nanosecond lasers however have the disadvantage of heat input into the sample and thus altering of the microstructure, which requires extensive FIB post-polishing. To reduce the thermal impact of the laser ablation on the microstructure and to reduce the FIB post-polishing time, the nanosecond laser was recently switched to a femtosecond (fs) laser. The use of a femtosecond laser facilitates extremely fast sample machining and surface preparation and has also shown to enable EBSD analysis directly on the laser polished surface due to the pseudo athermal ablation process it provides.

Experimental Set-up, Results and Discussion

To prepare a cross-section suitable for direct EBSD measurement, the first step is to find suitable process parameters for milling the desired material with as little surface damage as possible. Parameters that can be adjusted and have the most influence on surface quality have shown to be laser power [%], scan speed [mm/s], repetition rate [Hz] and line-distance [µm]. Extensive preliminary tests on different metal samples led to a more or less universal 3-step preparation recipe shown in table 1. To achieve the maximum surface quality only the final step has to be individualized in further tests.

I	Laser power	100 %
	Scan speed	950 mm/s
	Repetition rate	300 kHz
	Line distance	4 µm
II	Laser power	25 %
	Scan speed	10 mm/s
	Repetition rate	12 kHz
	Line distance	4 µm
III	Laser power	20 %
	Scan speed	3 mm/s
	Repetition rate	800 Hz
	Line distance	4 µm

Table 1: *Universal 3-step parameter-set*

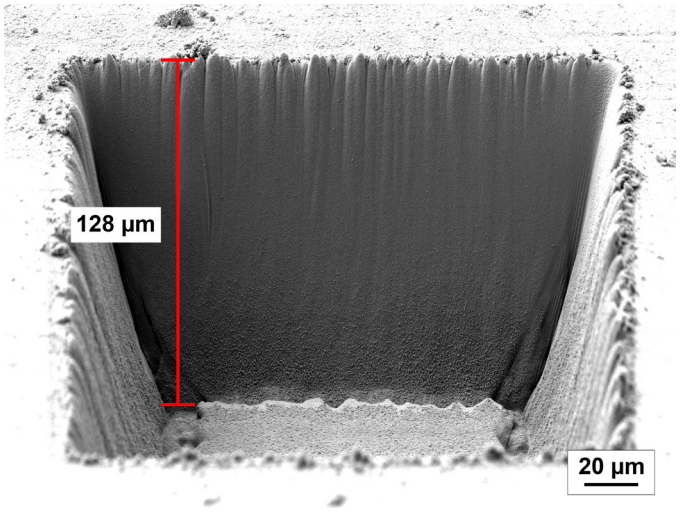


Figure 2: *Example of milled trench in copper to determine removal rate per raster*

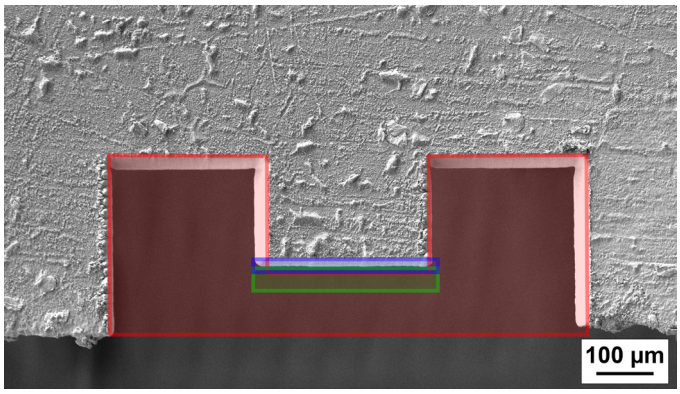


Figure 3: *Preparation routine for EBSD acquisition on laser polished cross-sections*

The three described steps can be divided into I – rough milling, II – rough polishing and III – fine polishing. To mill down to a desired depth, the laser-milling software is set to repeat the defined hatch pattern with the given parameters and remove a certain amount of material with each raster. Especially for EBSD measurement on laser-polished surfaces of sheets, it is important to mill through the whole sample to avoid obstruction of the EBSD signal by residual sample material. This is due to the geometric setup of sample and EBSD camera.

To assure the whole thickness is milled, the critical step is to determine the correct removal-rate per raster, so that the laser can adjust the focus accordingly. Therefore, a trench is milled into the sample material with each parameter set of the universal 3-step method and a fixed number of rasters. Afterwards the depth of the milled trench is measured (see Fig. 2) and the removal-rate per raster can be calculated for each set of parameters. In case of the examined copper sample, the removal rate for rough-milling and rough-polishing is 2.5 µm/raster, the rate for the fine-polishing is 1.6 µm/raster. Experiment, however, has shown that the final polishing step results in a better surface quality when no removal rate per layer is given and a fixed laser focus is applied instead.

It is crucial to determine the correct removal rate per raster for each individual sample as every metal, alloy and even every batch of the same alloy seems to show at least a slightly different specific removal rate.

For best practice EBSD detection, it is important to provide a free path of the diffracted electrons towards the EBSD camera. Figure 3 shows a suitable way to ensure said free path. With the first rough milling step, a free-standing “nose” is prepared into the sample (Fig. 3 red box) to avoid any redeposition and edge effects. The “nose” should have suitable dimensions for the intended EBSD scan. The next step is to rough polish the cross-section face to remove any redeposited material and to flatten the surface from the rough milling step (Fig. 3 green box). Here it is important to provide a certain overlap of the rough-polishing box with the rough-milling object. By following this method, any potential inaccuracy or misalignment can be avoided. The final step is the fine-polishing, and this is also done with a smaller milling box with overlap to the previous rough-polishing box. Given that all removal rates are determined correctly, the result should be as displayed in figure 4. No residual material is blocking the path of the diffracted electrons and the milled trenches show sharp edges and steep sidewalls with minimal sloping on the lower ends. The overall

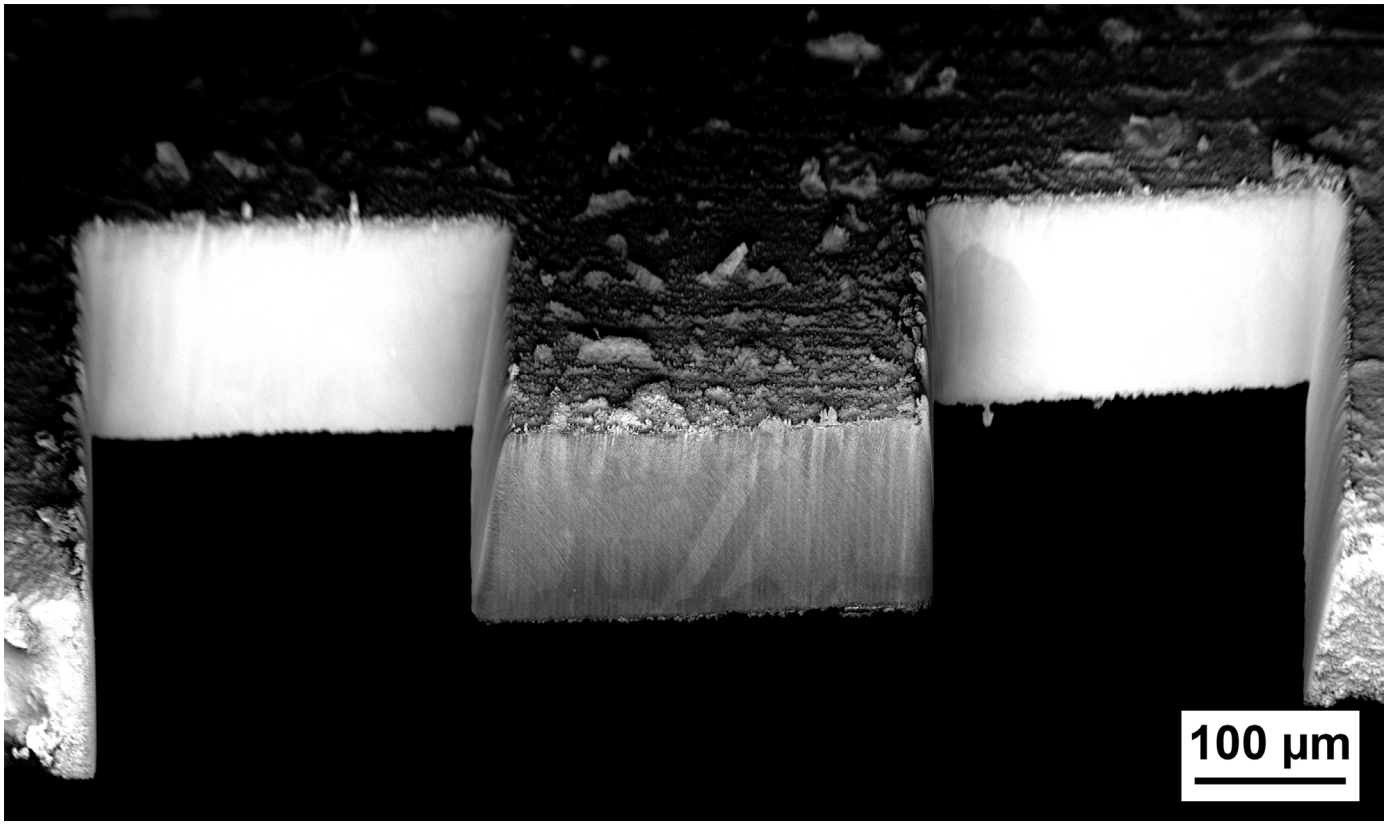


Figure 4: *Final laser-polished cross-section in copper sheet, visible microstructure/grain structure; SEM, imaged with Inlens SE detector, 130×*

cross-section size is 280 × 120 µm. The final fine-polishing step furthermore provides a smooth surface finish with the microstructure ideally being visible already. The shown 3-step process here takes about 15 minutes. The cross-section is now ready for imaging and EBSD acquisition.

Although the final polishing parameters were optimized for the material, there is still a superstructure present on the cross-section face. These periodic line structures are so-called LIPSS (Laser Induced Periodic Surface Structures) and are a phenomenon specific to ultra-short-pulsed laser-material-interactions. The formation of LIPSS is a matter of ongoing research and is debated in literature. Figure 5 shows a detailed view of LIPSS on the laser-polished copper sample. As the microstructure is visible through the LIPSS, it can be assumed that this does not affect the EBSD signal significantly. The negligible impact of the LIPSS can also be estimated from figure 6 where an area of the cross-section has been FIB polished for comparison. The FIB-polishing over the full width but only half the height of the cross-section took 4 hours using 7 nA of FIB probe current. FIB polishing the complete cross-section would have taken more than 8 hours and was regarded as impractical.

With the LIPSS present on the surface to be analyzed, it is best to use high acceleration voltage (20 kV) and high probe current (3 nA) to achieve a strong EBSD signal. The EBSD camera was set up with automatic optimization.

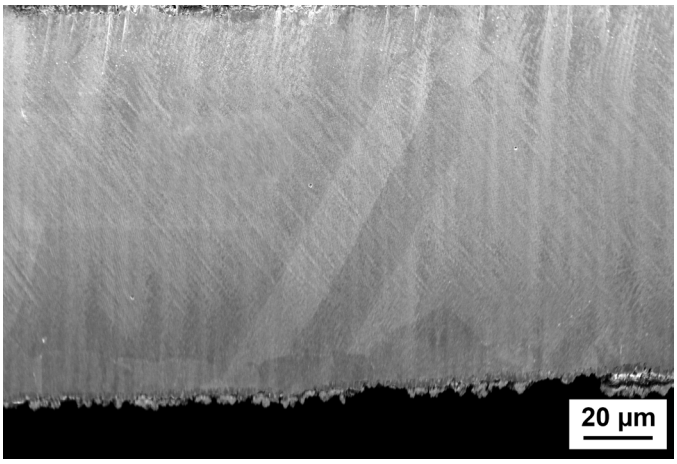


Figure 5: *Detailed view of LIPSS on laser-polished copper; SEM, Inlens SE, 500×*

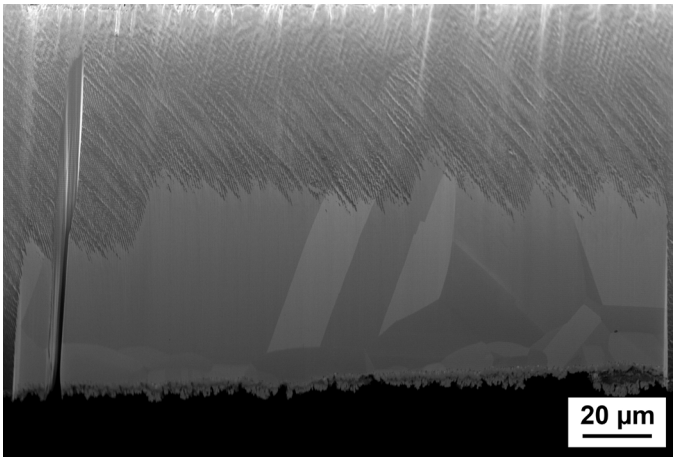


Figure 6: *FIB post-polish (4h, 7nA) of lower half of laser-polished cross-section; SEM, Inlens SE, 500×*

Figure 7 shows the acquired EBSD raw data. Points where the EBSD signal was not sufficient for indexing are represented by black pixels in the IPF-Map. The periodicity of these structures resembles the LIPSS on the laser-polished surface and it can be assumed that the LIPSS are thicker in these specific areas and thus hinder the generation of proper electron diffraction patterns. As long as the non-indexed pixels are sparse and surrounded by properly indexed points, various filters can be applied for correction without altering the analysis results significantly. In figure 8, a Neighbor Orientation Correlation (NOC) filter is applied to the raw EBSD dataset. With this filter operation, the majority of non-indexed pixels can be assigned.

To remove all non-indexed pixels and further improve the mapping result, a second filter can be applied in software. Figure 9 shows the results after the additional application of a 2-pixel grain dilatation filter.

The unfiltered EBSD results already show a decent quality that can be further improved by filtering. When the aim of EBSD analysis is grain size measurement or to get a rough idea of grain orientation, laser polishing is a suitable method for rapid preparation of EBSD quality surfaces. Whether the signal quality is also sufficient for strain measurement has to be evaluated by further experimentation.

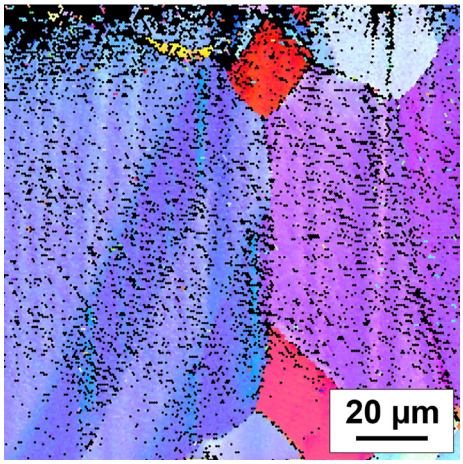


Figure 7: *Laser polished copper, IPF-Map, unfiltered raw data*

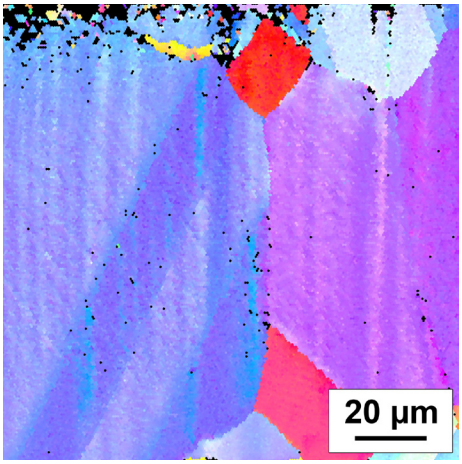


Figure 8: *Laser polished copper, IPF-Map, NOC filtered*

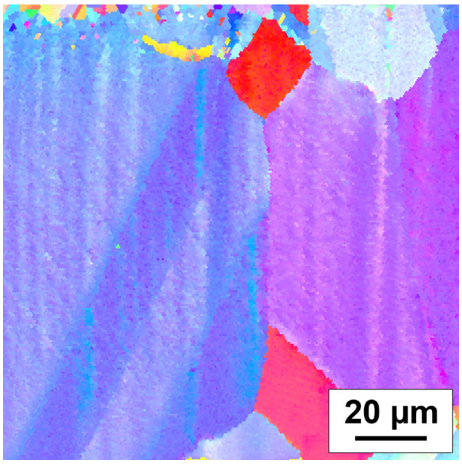


Figure 9: *Laser polished copper, IPF-Map, NOC and grain dilatation filter*

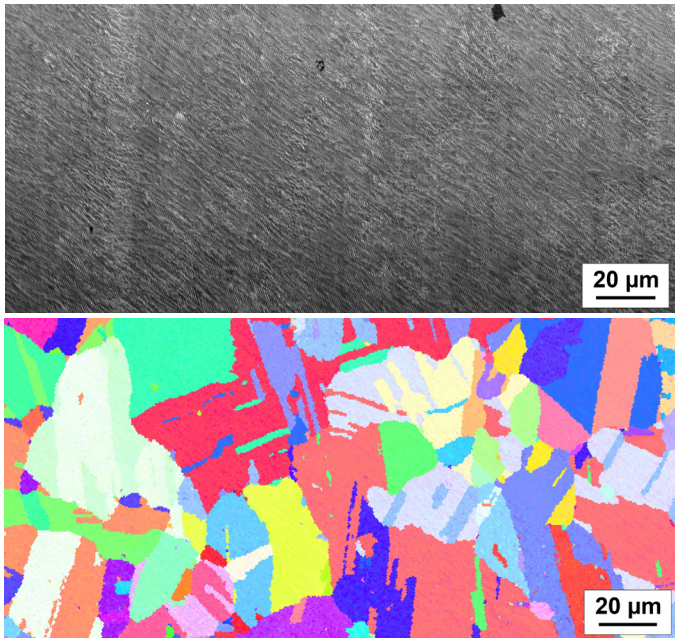


Figure 10: *Alloy600 sheet, top: laser-polished surface, no microstructure visible; bottom: EBSD IPF-map*

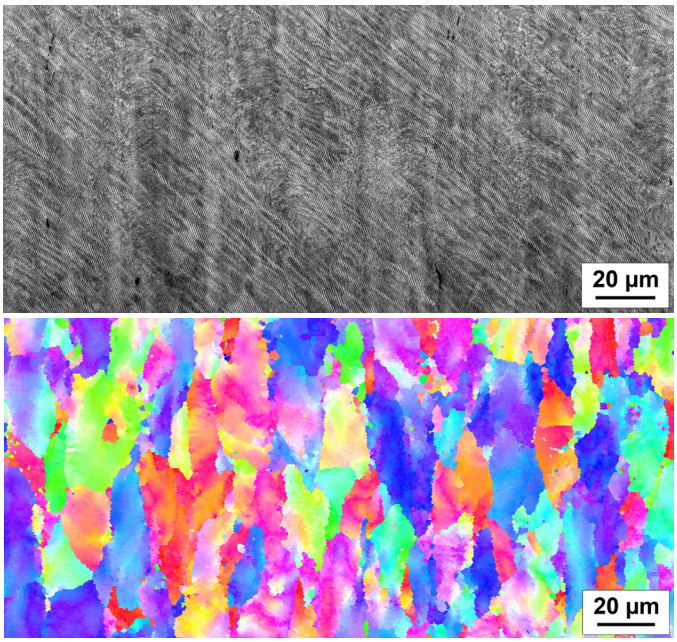


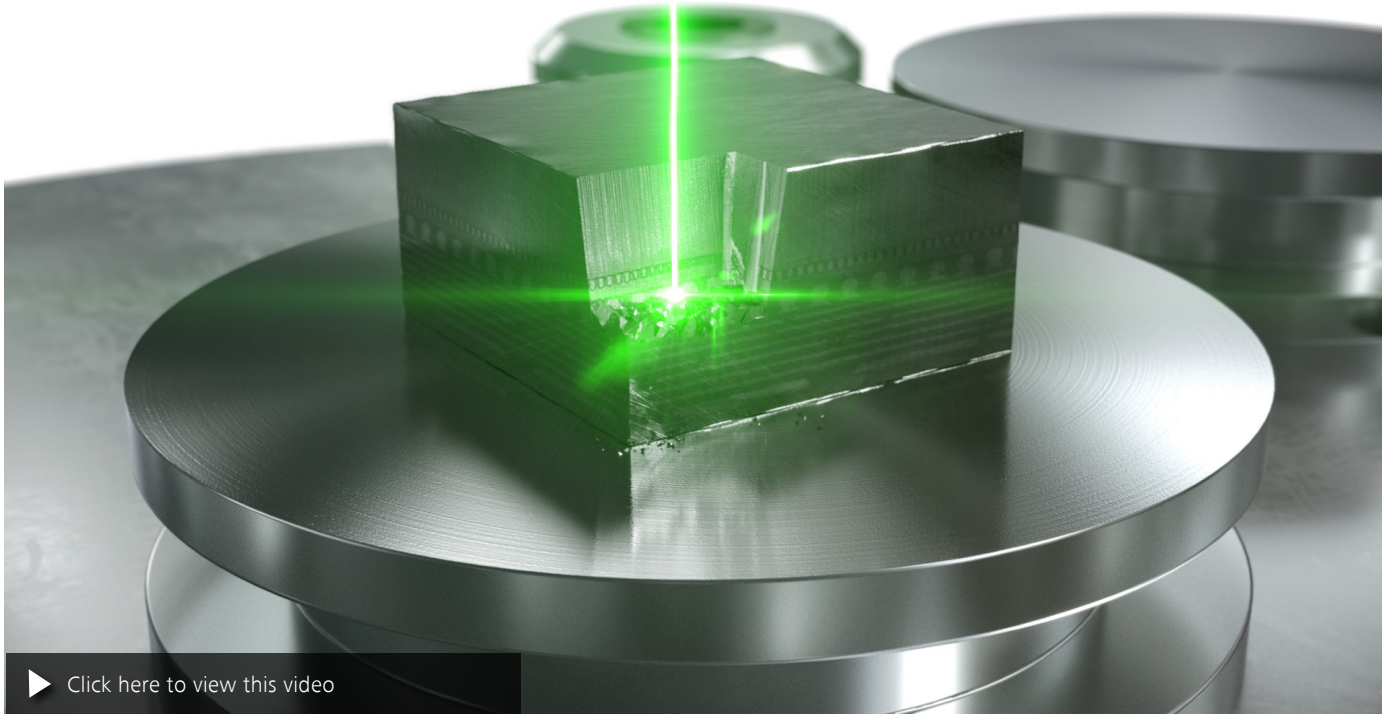
Figure 11: *Mild steel sheet, top: laser-polished surface, microstructure slightly visible; bottom: EBSD IPF-map*

The procedure to find optimum parameters and rapidly prepare a cross-section suitable for EBSD analysis by utilizing a femtosecond laser is of course not limited to the example of copper shown here. Figure 10 shows the polishing and EBSD results for an annealed Alloy600 sheet. The laser polished surface does not show the microstructure directly but still gives a good EBSD signal and reveals the individual grains, including twinning. In figure 11, the result for a mild steel sheet is shown. The laser polished surface shows a slight hint of the microstructure only, but again the polishing quality is sufficient to obtain an EBSD map of the individual grains. As the sample was cut from a rolled sheet, strain can be seen in the individual grains indicated by the blurry color transitions.

Conclusion

The presented work shows that ZEISS Crossbeam laser is a very suitable tool to rapidly prepare cross-sections in different metals without major defects or heat dissipation into the cross-section surface. With robust parameter development, it is also possible to achieve a preparation quality that is suitable for direct EBSD measurement on the laser polished surface. These results, when combined with recent advances in Laboratory Diffraction Contrast Tomography (LabDCT) in X-ray microscopy, represent a potential disruptive breakthrough in large statistics crystallography.

Reference



Watch this animation and discover the LaserFIB workflow. In this correlative experiment a defect buried in an electronics sample was located non-destructively with XRM (X-ray microscopy). After relocation in the LaserFIB, the ROI was exposed using the femtosecond laser, fine polished by FIB and finally analyzed with SEM in high resolution.

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