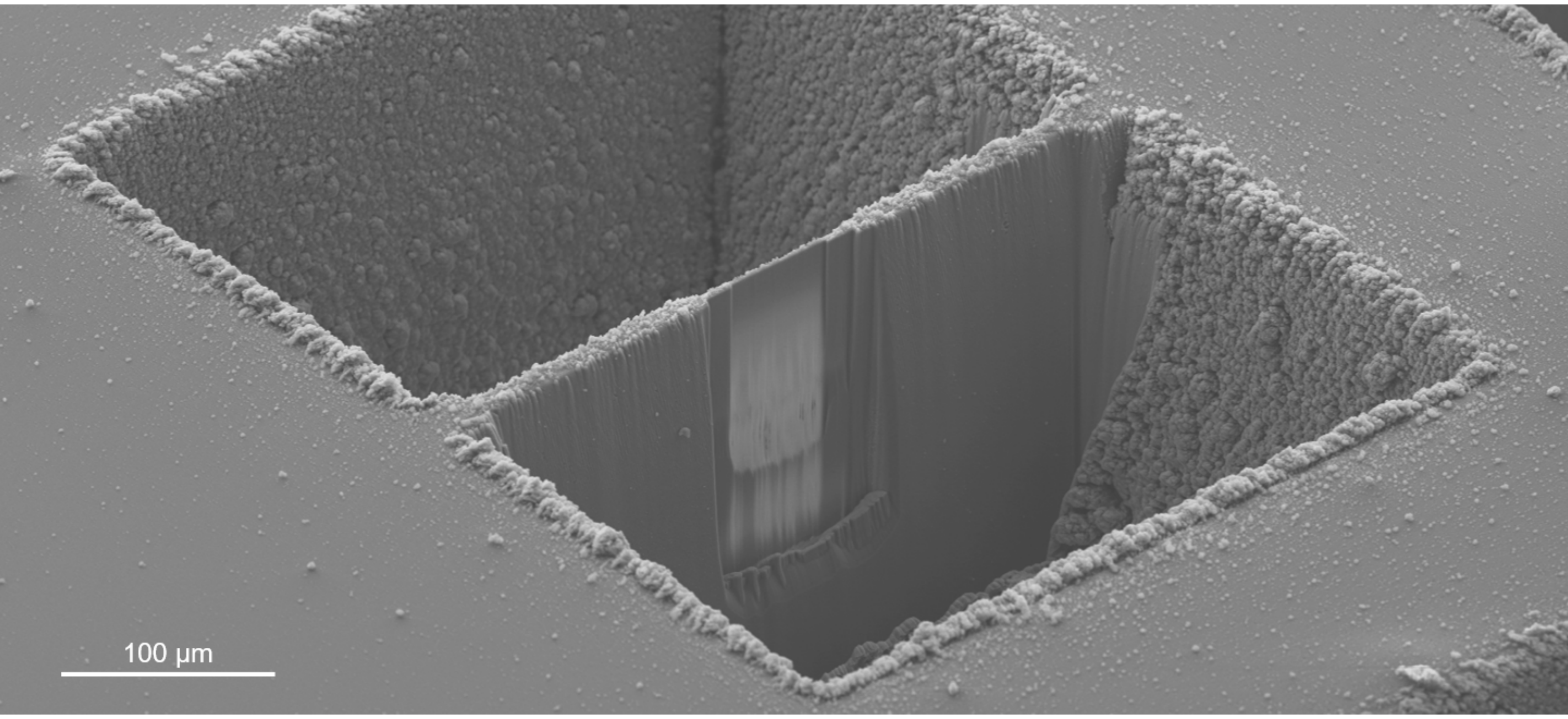
Exploring Laser-Assisted TEM Sample Preparation with ZEISS Crossbeam laser



Seeing beyond

Authors: Dr. Fabián Pérez-Willard, Tobias Volkenandt
Carl Zeiss Microscopy GmbH, Oberkochen, Germany
Date: 20th of November 2020

This note explores the use of femtosecond (fs) laser ablation for site-specific TEM sample preparation in a FIB-SEM. Different workflows are presented enabling TEM lamella preparation of regions of interest (ROI) buried so deeply in the sample that they cannot be accessed directly by FIB. As a best-known method, the so-called Cut-to-ROI workflow was developed and is explained in detail. General limitations when preparing large TEM lamellas are discussed, as well as the new possibilities the LaserFIB opens for microstructural characterization.

Introduction

Recently, LaserFIB instruments have attracted substantial attention in the microscopy community. The term LaserFIB denotes the combination of a FIB-SEM with an ultra-short pulsed laser, typically a fs laser, within a single instrument. The laser adds additional capabilities to the FIB-SEM, expanding its already broad application space [1].

By means of fs laser ablation large volumes of material can be removed from a sample very rapidly with in most cases negligible sample damage. Thus, access can be gained to regions of interest deeply buried in the sample for their subsequent FIB-SEM analysis. Other applications of LaserFIBs include the fabrication of micromechanical devices for materials testing with dimensions of up to millimeters [2,3], the preparation of surfaces for EBSD analysis [4,5], and the production of pillar-shaped samples for the synchrotron or X-ray microscope [6].

In the LaserFIB setup implemented by ZEISS, with the product name ZEISS Crossbeam laser, the laser work is done in a dedicated chamber to prevent contamination of the main FIB-SEM chamber by laser ablated debris [7].

In this paper we explore the possibilities ZEISS Crossbeam laser offers for TEM sample preparation. We present different preparation workflows and discuss possible modifications to adapt to specific microstructural characterization challenges. The workflows are well suited for the TEM cross section preparation of deeply buried features of interest or the preparation of multiple thinned windows distributed over a large specimen area.

Experimental

All experiments were conducted on a ZEISS Crossbeam laser.

ZEISS Crossbeam laser is equipped with a diode pumped solid state (DPSS) 515 nm (green) laser with laser pulses of less than 350 fs duration. Pulse repetition rates can be varied in the range of 0.1 kHz to 1 MHz. The maximum output power is 10 W when operated at 1 MHz. With this laser, material removal rates of more than 15 mio $\mu m^3/s$ in silicon can be reached, representing at least three orders of magnitude improvement in speed compared to any focused ion beam [1]. When defining the laser milling strategy for a given layout, important parameters are laser power (in percentage), writing speed (in mm/s), pulse repetition rate and number of passes (hatches). The user can input these for individual shapes or groups of shapes in a CAD interface.

ZEISS Crossbeam laser features an Ion-sculptor gallium FIB column with beam currents of 1 pA to 100 nA. For the lamella lift-outs, an OP400 (Oxford Instruments plc) micromanipulator and a gas injection system (GIS) were used.

The samples used were <100> silicon (for most of the experiments) and a 99.9% pure copper foil (for the H-bar experiment). These materials were chosen to illustrate the different workflows because they are readily available, homogeneous, and a good reference for comparison with standard FIB work.

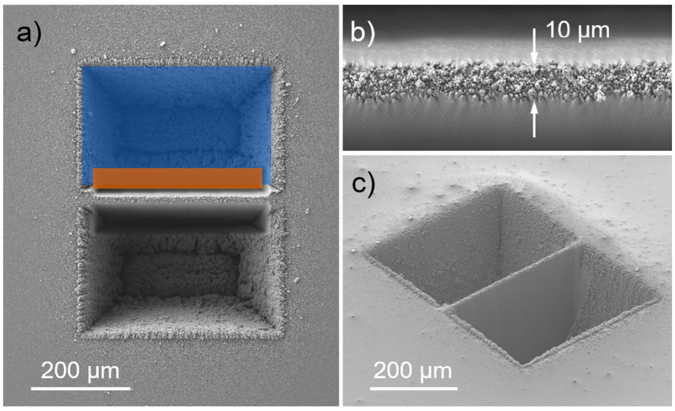


Figure 1: SEM images of a laser-machined chunk. a) Top view with laser milling shapes overlaid on one side of the chunk; blue for the rough milling and orange for the polishing step. b) Magnified top view. c) Sample tilted 54° and rotated.

Chunking with the Laser

Figure 1 shows a laser prepared “macro-lamella”, also known as a chunk, in silicon. The chunk was defined by first performing a coarse milling step of two opposing boxes. One of these boxes is schematically shown in Figure 1a) as a shaded blue rectangle. In a second laser machining step – corresponding to the orange shaded rectangle – both sides of the chunk were polished.

The coarse milling objects were nominally 400 µm wide. The ones for the polishing step 350 µm. The position of the boxes was chosen to yield a chunk thickness of around 10 µm as measured at the top, see Figure 1b). After the laser polishing step, the side walls of the chunk have a taper of (7±1)°.

The preparation of the chunk by laser is analogous to standard FIB preparation. The main difference is the scale. The dimensions of the laser-prepared chunk are a factor of 10 to 20× larger than for a typical gallium FIB chunk. Still, the laser fabrication process is very fast. For the chunk in Figure 1 the total laser machining time was 80 seconds.

In order to explore several different ways to use the laser to assist TEM sample preparation, an array of 3 × 2 chunks was prepared. This array is shown in Figure 2. The complete array spans an area of 1.6 × 1.3 mm² and was patterned in 240 seconds.

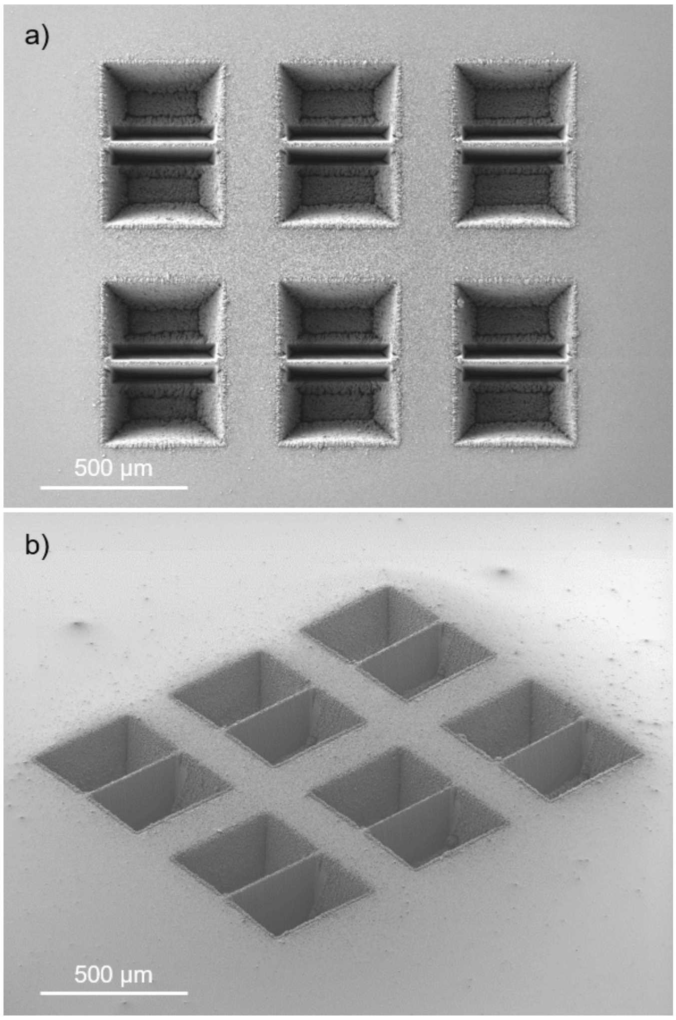


Figure 2: SEM images of a 3 × 2 array of laser-machined chunks. a) Top view. b) Sample tilted 54° and rotated. The FOV width is 2.3 mm for both images.

Thinning and Shaping by FIB

The laser preparation was followed by gallium FIB machining to planarize and prepare the samples for lift-out. Different approaches were tested to explore their practicability.

Figure 3 shows a first example. One of the laser-prepared chunks was thinned down to slightly less than 2 µm over an area 58 µm wide and more than 75 µm deep by gallium FIB milling. This area appears bright when imaged with the SEM at 15 keV landing energy, Figure 3a). The thinning of this lamella was done in three steps performed at FIB currents of 65 nA, 30 nA, and 7 nA. The sample was over-tilted and under-tilted when machining the front and back side, respectively. The over- and under-tilt angles were reduced from 4°, to 3°, to 2° with decreasing milling current to ensure a homogeneous thickness over the entire thinned window. The FIB thinning including all three steps took 3 h in total. This is a time we consider acceptable in light of the sample size and the fact that the FIB milling can be fully automated.

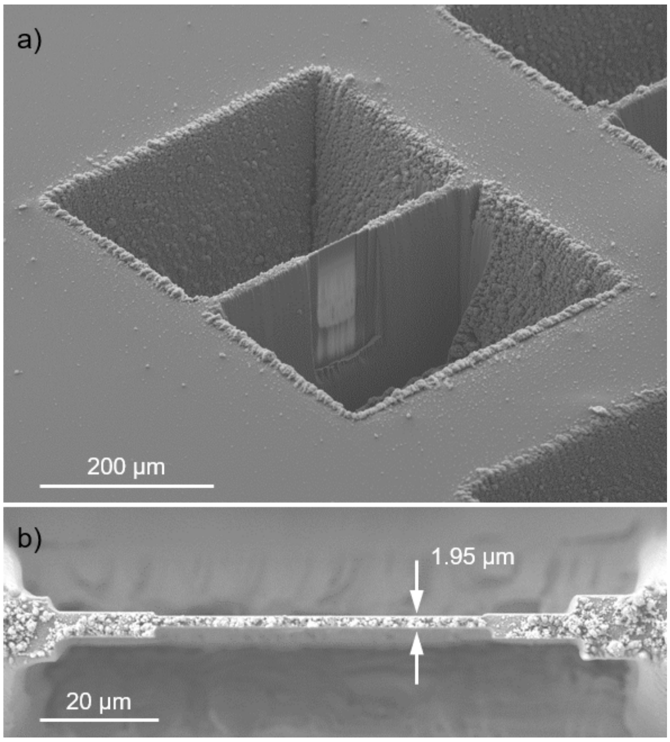


Figure 3: a) SEM images of a large FIB thinned lamella. a) Sample tilted 54° and rotated. b) Top view of the thinned region.

For the next sample, the same amount of time was invested to thin a 220 × 104 µm² area to 4.5 µm thickness including cut-out, see Figures 4a) and b). This time all FIB milling operations were conducted at 65 nA. The over/under-tilt angle was 3°. Note that these settings would allow to produce a lamella of the dimensions in Figure 3 in well below 1 h.

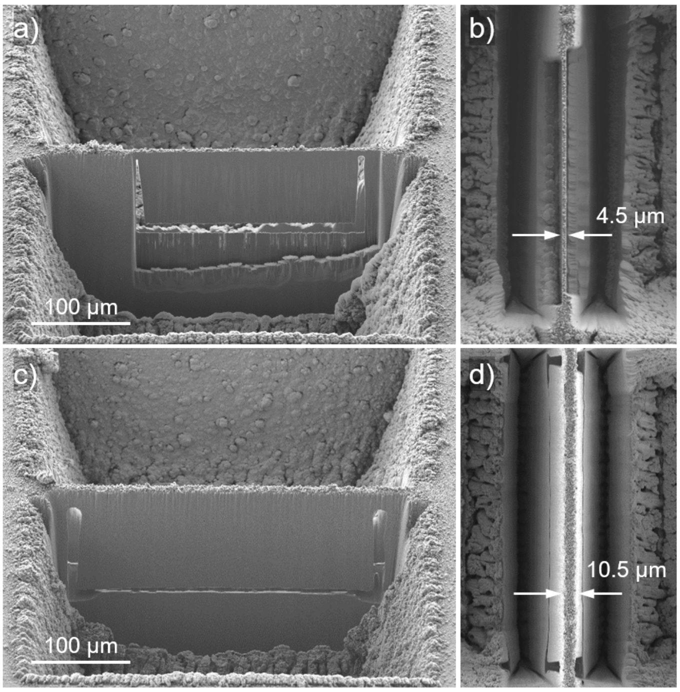


Figure 4: SEM images of two large lamellas after cut-out. a) and b) show different SEM perspectives – 0° and 45° stage tilt – of a 220 µm × 104 µm × 4.5 µm lamella. c) and d) show a bigger lamella with dimensions of 300 µm × 140 µm. This one is 10.5 µm thick at the top with a side wall taper of 7°.

Alternatively, the 3 h time budget can be allocated just to the cut-out step of the as-lasered sample, without any FIB thinning/polishing. This case is shown in Figures 4c) and d). Again, a FIB current of 65 nA was used. The sample is now 300 µm × 140 µm in size, with a thickness of 10.5 µm at the top and getting thicker towards the bottom due to the side wall taper of 7°.

Lift-Out

Despite their size, all three lamellas discussed in the previous section could be easily lifted out of the bulk sample and attached to a 3 mm copper grid compatible with standard TEM sample holders following the standard *in situ* lift-out procedure [8].

For visual comparison, Figure 5a) shows a standard-sized FIB lamella (15 µm × 14 µm), and Figures 5b) to d) show the lamellas from the previously described experiments, following the lift-out step. The FOV is the same for all four images in this figure.

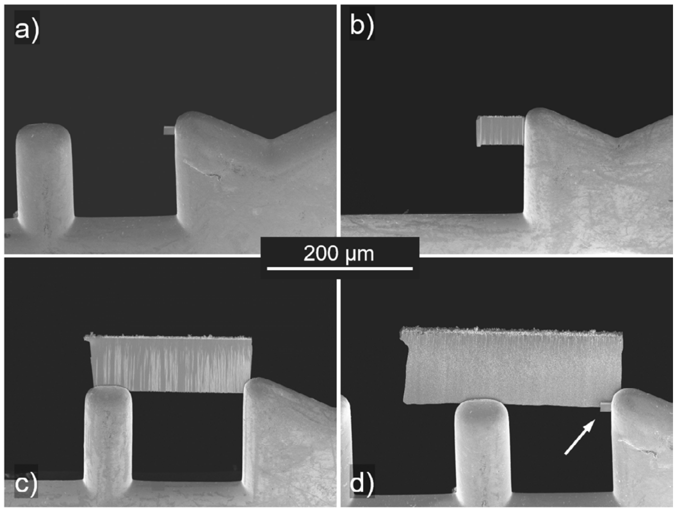


Figure 5: a) SEM image of a standard FIB lamella after in situ *lift-out. b) to d) SEM images of laser prepared lamellas of increasing size after* in situ *lift-out. The lamella in d) was attached to the same grid as the lamella in a), which is highlighted by the arrow.*

Limitations

The next step after the *in situ* lift-out is to thin the lamella towards electron transparency. TEM analyses require very thin samples with a thickness of 100 nm or less. It is a challenge to obtain a uniformly thinned window of this thickness covering a large area.

Depending on the sample material, FIB-thinned windows are typically of the order of 10 µm × 10 µm or smaller. This is because at some point during thinning the membrane starts to bend. This renders further thinning impossible without destroying the sample. The bending is due to intrinsic stress release in the sample combined with its lower mechanical stability. Thus, the material itself imposes limits to the achievable size of an electron transparent window. This means that even in large chunks only small windows can be thinned. However, numerous small TEM windows can be prepared to cover a large specimen area.

We see the main application of the LaserFIB for TEM sample preparation, as providing three-dimensional (3D) site-specific preparation of deeply buried structures of interest. The next section describes how this can be accomplished using what we have named the Cut-To-ROI workflow.

The Cut-To-ROI Workflow

The starting point or pre-requisite of the Cut-To-ROI workflow is a laser-machined chunk. The chunk contains the ROI. Further, the position of the ROI in the depth of the chunk (direction of the bulk sample surface normal, Z) is known.

The XYZ coordinates of a ROI can result from the sample layout itself (e.g. the known CAD layout of an electronics or semiconductor sample), from a previous characterization by a non-destructive imaging technique (e.g. by X-ray microscopy) [9], or any combination of both (e.g. lock-in thermography combined with CAD chip layout). Any feature of interest that has been located in the sample can be targeted by the laser with an accuracy of about 2 µm [10].

The chunk can be trimmed by FIB as shown in Figure 6a). Here, the grid was flipped by 90° after the lift-out, so that FIB incidence was perpendicular to the chunk side face for trimming. Three ROIs were assumed at Z = 45, 65 and 25 µm, from left to right. The FIB was used to remove the top of the chunk to form 40, 60, and 20 µm deep steps. This process took 13 min for the three steps at a FIB current of 30 nA.

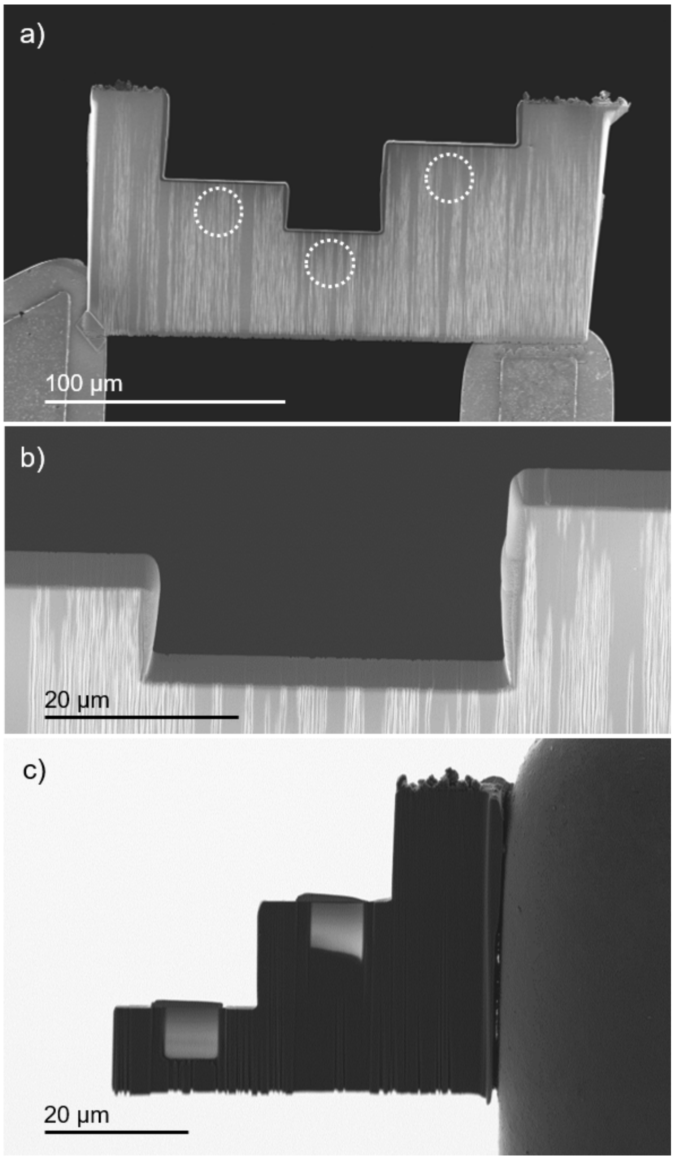


Figure 6: *Laser processed lamella after FIB shaping to access ROIs 45 µm, 65, and 25 µm below the surface (dotted circles). b) The top surfaces after FIB milling are flat and smooth. c) SEM image at 5 kV of a different lamella after thinning of two ROIs.*

After trimming, the grid was flipped back to the upright position. Figure 6b) shows the top surface of one of the steps. The surface is flat and smooth, which facilitates the subsequent preparation of the TEM-thin areas by FIB. Figure 6c) shows another exemplary chunk after final thinning of two ROIs 25 and 45 µm below the surface.

Figure 7 illustrates a variation of the Cut-To-ROI workflow. In this case, the laser machined chunk was not lifted out of the bulk but was trimmed in the Z-dimension directly by FIB. For this purpose, the sample stage was at 0° tilt. The FIB trimming then takes place at an angle of 36° to the bulk sample surface (XY plane). In this case, our assumed ROI was at Z = 80 μm. The top 75 μm of the chunk were removed over the entire width of the chunk (65 nA FIB current, 1h 36min FIB milling time) and then two standard FIB *in situ* lift-out specimens were prepared. For most applications, this alternative Cut-To-ROI workflow can be sped up easily by preparing a thinner chunk, or even allowing some material removal from the top of the chunk by the laser beam tail.

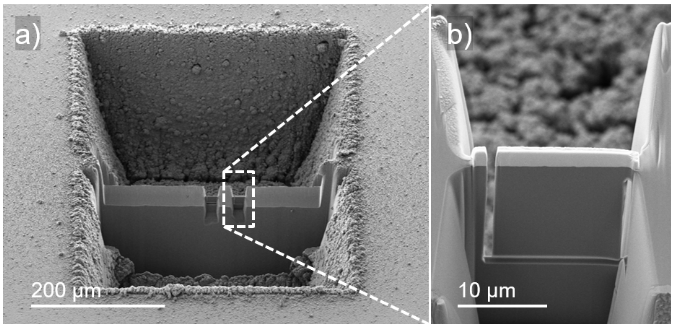


Figure 7: *a) Laser-processed chunk. After laser cutting, the top 75 μm were removed by FIB milling and two standard FIB lamellas were prepared. b) Detail of the right-most lamella corresponding to the area in the white frame in a).*

H-Bar Preparation by Laser

Another option is to combine H-bar [11] and Cut-To-ROI preparations. This technique can be applied e.g. on small pieces of metal foils or wafers. These pieces are mounted on edge for laser and FIB machining. This is shown in Figure 8. Two chunks were patterned into a 300 μm thick copper foil using the laser. The laser processing time for the larger chunk of 400 μm × 215 μm was 34 seconds.

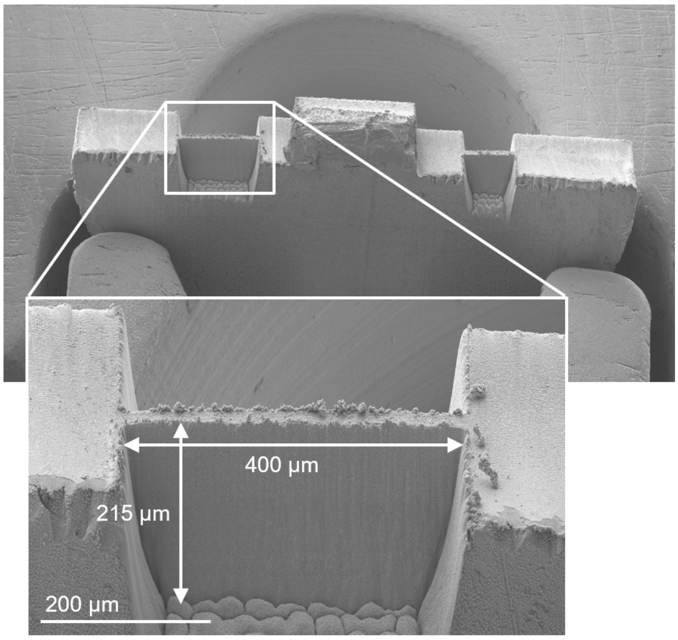


Figure 8: *H-bar preparation in a piece of copper foil by laser. Two large H-bars were prepared. The one in the inset was machined in 34 seconds.*

Summary

In this work different workflows were discussed combining laser and FIB machining for the preparation of TEM specimens. ZEISS Crossbeam laser systems allow the 3D-site-specific preparation of deeply buried regions of interest, or the preparation of multiple TEM-suitable windows spread over a large sample area.

References

[1] F. Pérez-Willard et al., Expanding the application space of ion microscopy, submitted.

[2] M. J. Pfeifenberger et al., The use of femtosecond laser ablation as a novel tool for rapid micro-mechanical sample preparation, Materials and Design 121 (2017), pp 109-118.

[3] M. J. Pfeifenberger et al., Electron Irradiation Effects on Strength and Ductility of Polymer Foils Studied by Femtosecond Laser-Processed Micro-Tensile Specimens, Materials 12 (2019), pp. 1468-1483.

[4] M.P. Echlin et al., The TriBeam system: Femtosecond laser ablation *in situ* SEM, Mater. Charact. 100 (2015), pp 1-12.

[5] T. Schubert et al., Rapid Sample Preparation for EBSD-Analysis Enabled by the LaserFIB, ZEISS Application Note (2020), available online.

[6] B. Tordoff et al., The LaserFIB: New Application Opportunities Combining a High-Performance FIB-SEM with Femtosecond Laser Processing in a Second Integrated Chamber, Appl. Microsc. accepted.

[7] H. Doemer et al., Processing System, US8115180B2 (2012).

[8] L. A. Giannuzzi and F.A. Stevie, Introduction to Focused Ion Beams: Instrumentation, Theory, Techniques, and Practice, New York, Springer (2005).

[9] M. Kaestner et al. Novel Workflow for High-Resolution Imaging of Structures in Advanced 3D and Fan-Out Packages, 2019 China Semiconductor Technology International Conference (CSTIC) (2019), pp. 1-3.

[10] ZEISS Crossbeam Product Specifications, available on request at www.zeiss.com/crossbeam

[11] J. Li, “Advanced Techniques in TEM Specimen Preparation”, in The Transmission Electron Microscope, Ed. Dr. K. Maaz, IntechOpen (2012).

Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/crossbeam