A combined laser ablation/focused ion beam approach to atom probe sample preparation

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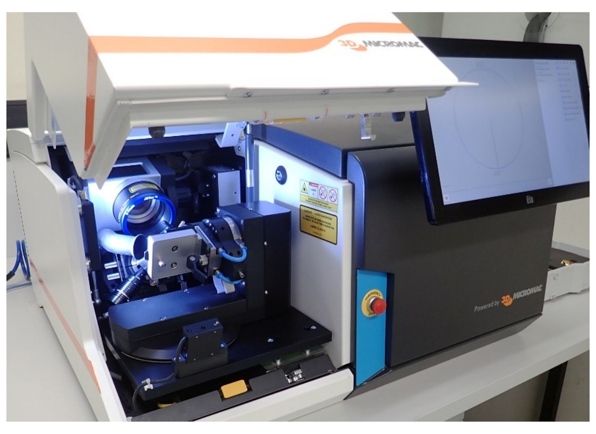


We have used 3D-Micromac's microPREP laser ablation tool to optimise a combined laser ablation/focused ion beam (FIB) atom probe sample preparation workflow. The laser cutting and thinning capabilities of the microPREP allowed for a range of atom probe specimens to be prepared directly from bulk material (<~350 µm thick) to a tip sharpness of <5 µm. Tips could then be taken to the FIB for minimal annular milling to a sharpness suitable for APT. The simplicity of operation and speed of preparation removes the need for user expertise in electropolishing and saves FIB beam time by removing the need to prepare specimens via the lift-out method.

Methodology:

Custom designed 3 mm half grids (Fig. 1) were laser cut from commercially bought sheets of tungsten, alumina, and silicon. Tungsten and alumina sheets were 50 µm and 100 µm thick respectively. The silicon started at a thickness of 575 µm, and was mechanically ground/polished to a thickness of ~300 µm. Sample sheets were then transferred to the microPREP cutting fixture (Fig. 2) and loaded into the system. Appropriate laser parameters for 'cutting' were set for the support and region of interest (see Table 1). The custom 3 mm grid design was chosen and cutting proceeded.

The cut grid was gold coated if necessary (alumina in this case), transferred to the microPREP thinning fixture (Fig. 3) and loaded into the system. Appropriate laser parameters for 'pillar thinning' were set (see Table 1) and an inner/outer pillar diameter was chosen. The inner diameter was kept at 0.5 µm to keep pillars as sharp as possible. The outer diameter was typically set to ~450-550 µm to eliminate material between adjacent pillars (Fig. 4b). This process was repeated along the long axis of the grid until 4-5 pillars remained (Fig. 4c and 4d). Finally, the grid was transferred to the Zeiss Auriga FIB-SEM where the specimen was annular milled to <~200 nm sharpness, ready for atom probe tomography (APT).

Table 1. Optimised laser parameters for grid cutting and pillar thinning.

Material	Process	Laser power (W)	Spot Diameter (um)	Pulse distance (um)	Line distance (um)	Area Dose (pulses/um2)	Number of Layers	Process time (min)
Alumina	Cutting Support	2	15	4	4	250	15	2
	Cutting ROI	0.5	12	4	4	250	15	
	Pillar Thinning	**0.75**	**18**	**26**	**0.1**	**5**	**30**	**3**
Tungsten	Cutting Support	2	15	12	5	300	4	6.5
	Cutting ROI	0.1	10	12	3	1200	20	
	Pillar Thinning	**0.2**	**20**	**25**	**0.3**	**25**	**20**	**11**
Silicon	Cutting Support	2.8	15	14	6	600	8	9
	Cutting ROI	0.5	8	8	2	1000	20	
	Pillar Thinning	**0.1**	**18**	**8**	**0.3**	**35**	**3**	**5**

Specimen preparation flow chart

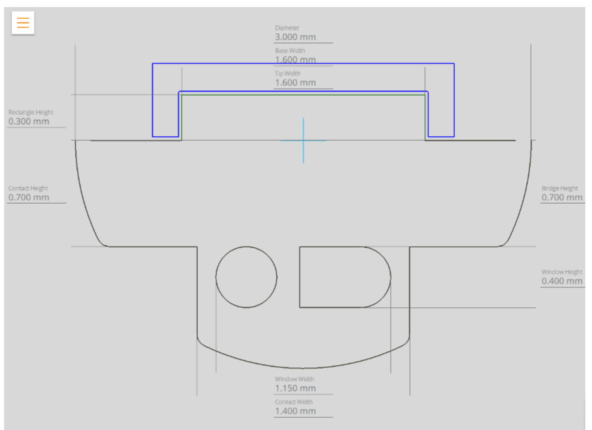


Fig. 1. Custom 3 mm grid design, taken from UI of microPREP.

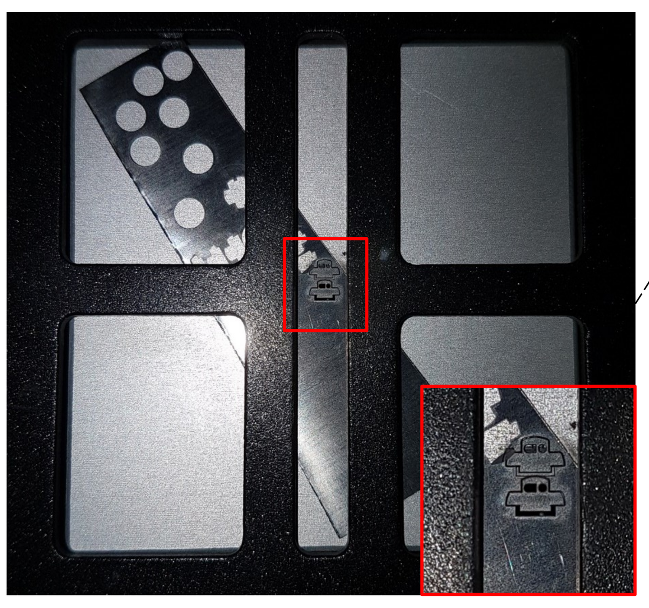
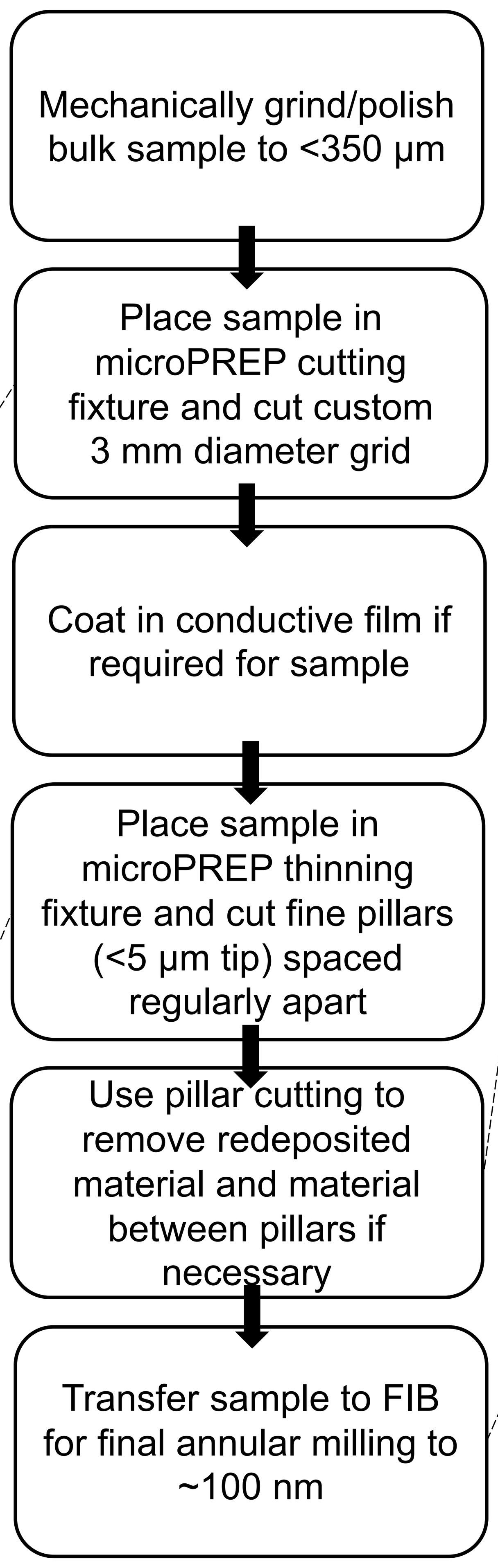


Fig. 2. 50 µm thick tungsten film mounted onto the microPREP cutting fixture. Custom 3 mm grid has been laser cut, shown outlined in red.

Fig. 3. Custom 3 mm grid mounted in the microPREP thinning fixture.



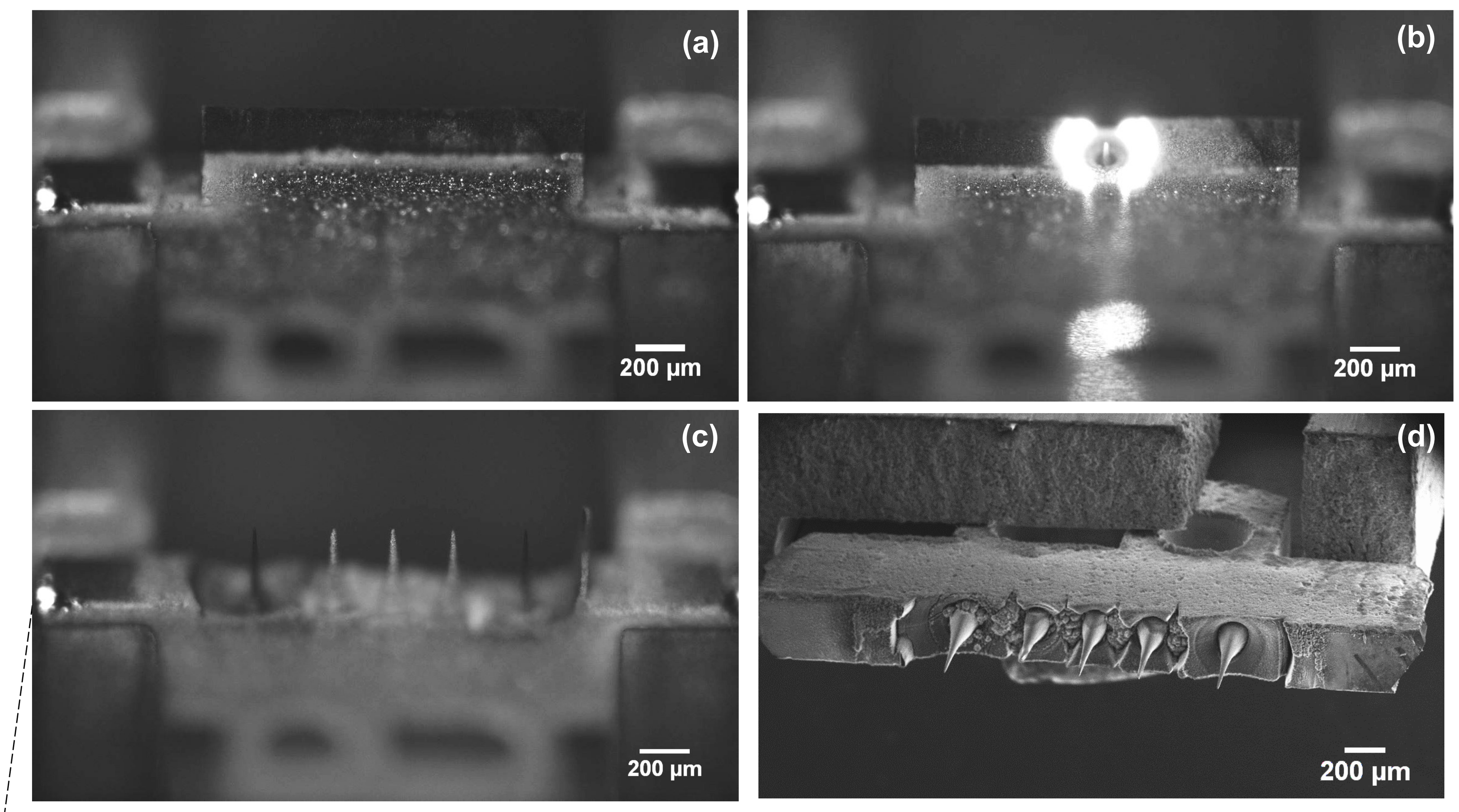


Fig. 4. (a) microPREP chamber view of ~300 µm thick silicon grid mounted in thinning fixture prior to annular laser cutting; (b) mid-process snapshot of pillar being cut; (c) final pillar array along length of silicon grid. Middle 3 pillars show some redeposition of material from adjacent pillar cutting process. (d) SE2 image of 5 pillar array along length of tungsten grid.

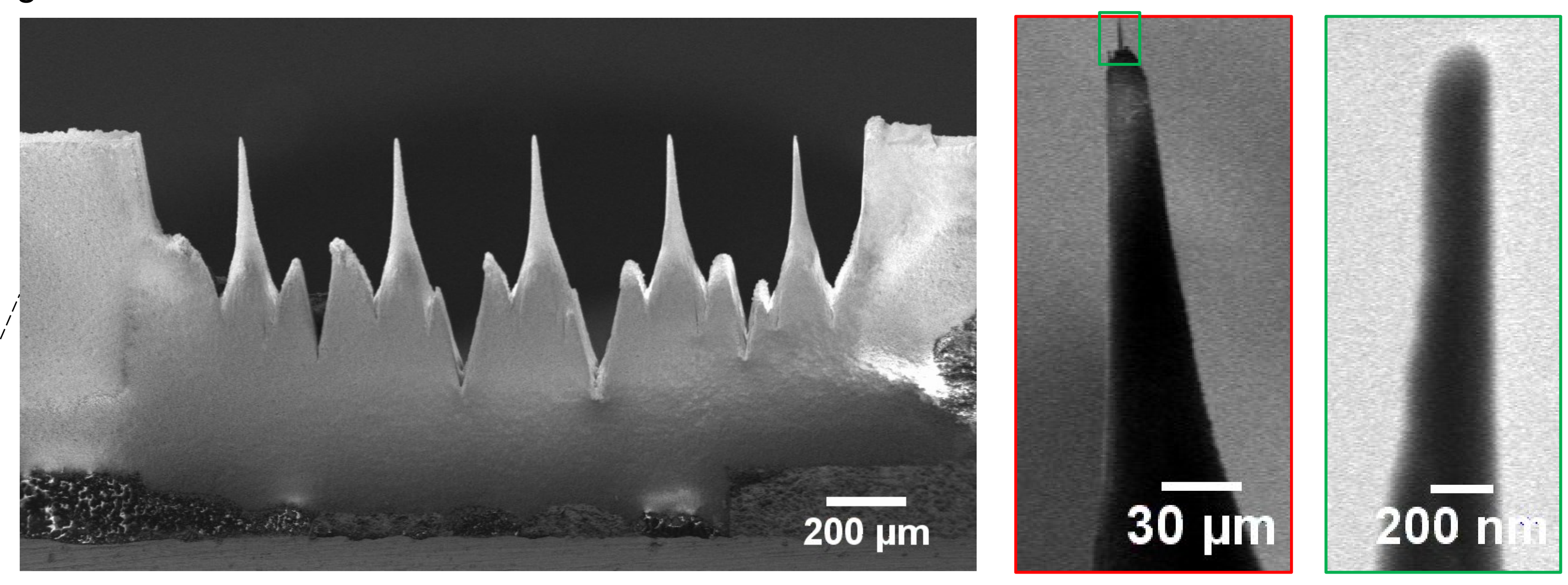


Fig. 5. SE2 image of alumina pillars prior to annular milling via FIB. Red ROI highlights ~5 µm tip following annular milling. Green ROI shows the final specimen ready for APT.

Results and Discussion:

Atom probe specimens were prepared for APT (Fig. 6) using a combined laser ablation/FIB approach. Tip sharpness and shank angle were highly dependent on laser power, pulse distance and line distance (Table 1). Lowering the power limits potential localised heating of specimens but often resulted in poor shank angles and rounder, flatter tips. Line distances <1 µm were essential to achieving the resolution necessary for tips <~5 µm in diameter. Tips may need to be spaced further apart than demonstrated to limit redeposition for material during adjacent pillar cutting (Fig. 4c). Further, tips must be of similar height to prevent adjacent tips hitting the local electrode during APT. The authors believe further optimisation of laser parameters and grid design will allow finer laser cut tips to be prepared.

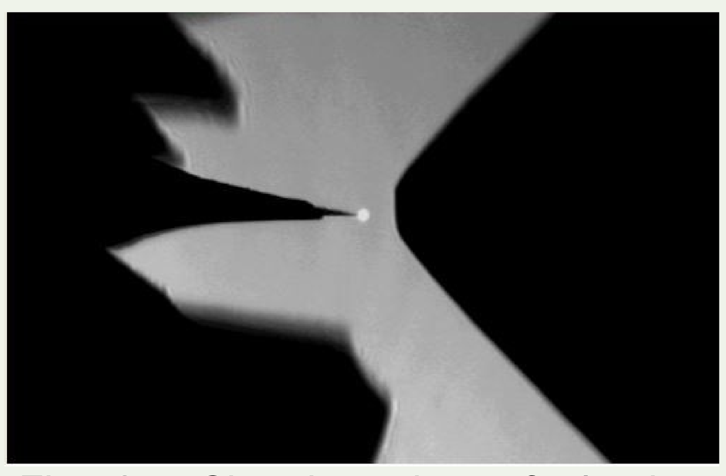


Fig. 6. Chamber view of alumina sample in atom probe LEAP4000XSi.

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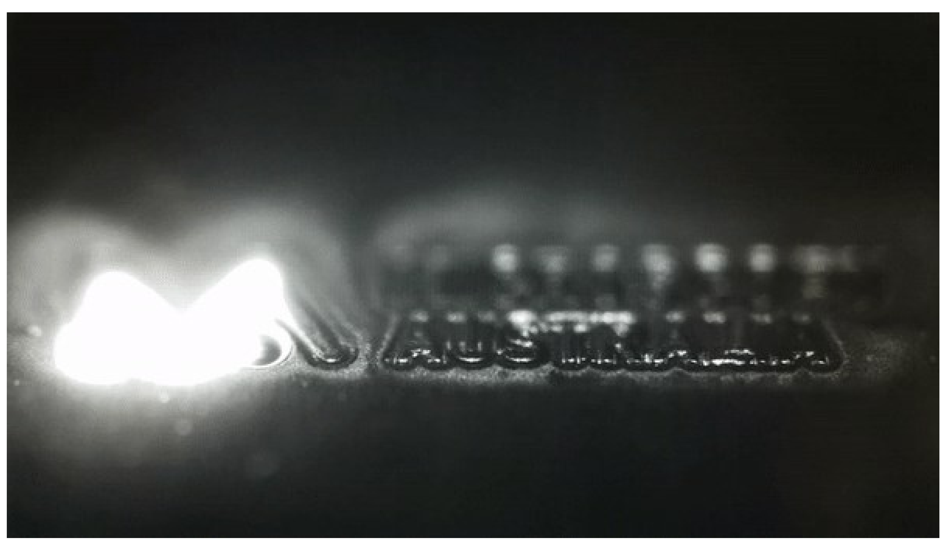

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