TEM sample preparation workflow using laser ablation and broad ion beam milling

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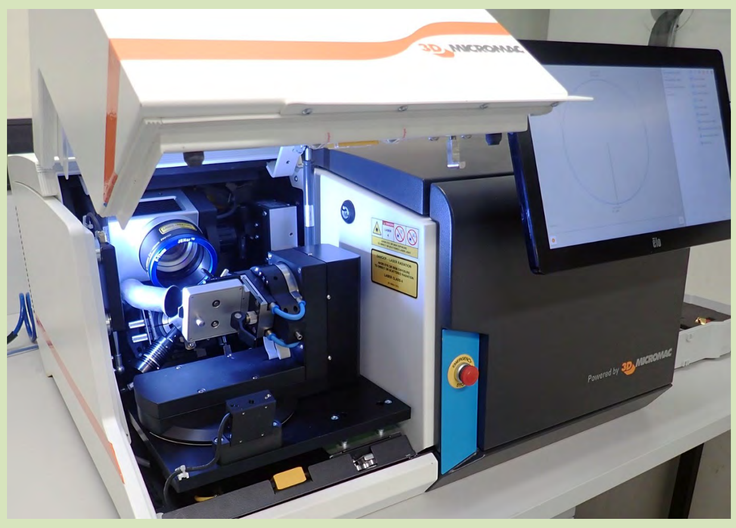
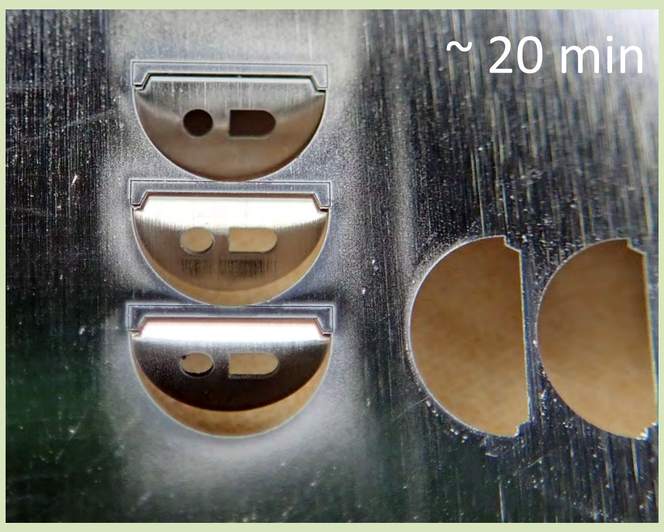
Introduction

TEM sample preparation can often become a bottleneck for any materials science research

Traditional methods (dimpler, electropolish, rotary broad ion beam) and FIB-SEM lift-out are still effective, but rely heavily on the operator skill, experience and time commitment

A new simple workflow is proposed, which minimises the process steps and skill levels required to create a high quality TEM sample from a solid bulk material





3D-Micromac MicroPREP

- Benchtop laser sample preparation tool
- DPSS laser, ± 5 micron process accuracy, up to 25 x 25 x 10 mm workpiece
- Pre-set microscopy sample patterns or import CAD drawings

Workflow

STEP 1

Bulk sample is cut into approx. 0.1 mm sheets

STEP 2

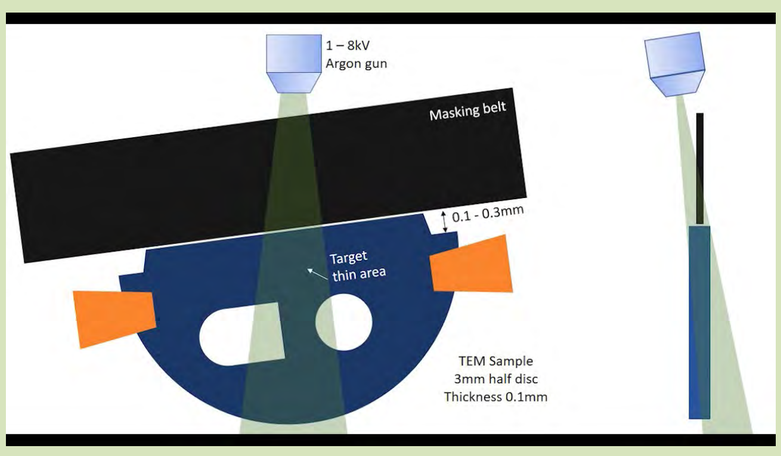
3D-Micromac MicroPREP cuts the sheet into 3mm Semi-circular TEM sample shapes

- Top edge is raised by 0.1 – 0.3 mm to avoid laser heat affecting the centre of the disc
- Tweezer holes are cut for convenience
- Laser cutting time depends on the material and thickness (e.g. 10-20 min to make 3 aluminium discs)

STEP 3

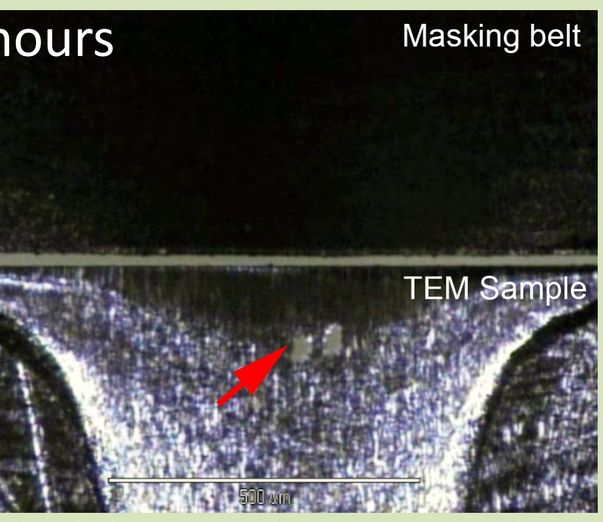
JEOL Cryo Ion Slicer makes an electron transparent thin area near the centre of the disc

- Example for aluminium alloys, 2.5 hours at 6kV, followed by ~30 min at 2kV until perforation
- Final thin area quality is highly dependent on the initial smoothness of the edge facing the masking belt. This is where laser cutting is advantageous
- Ion gun tilt angle can be adjusted to control the distance from the masking belt edge to the thin area, which is useful for avoiding the laser heat-affected region



JEOL CRYO ION SLICER IB-09060CIS

- Broad ion beam TEM sample thinning
- 1-8kV rocking argon ion gun
- Shallow angle milling with a masking belt
- Start from a 100 micron thick sample without pre-thinning
- Optional liquid N cryo cooling



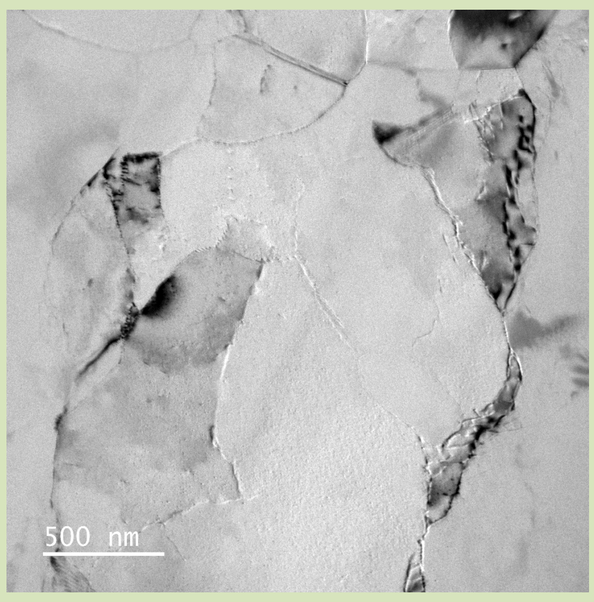
Gradual thin area formation and perforation

Key advantages

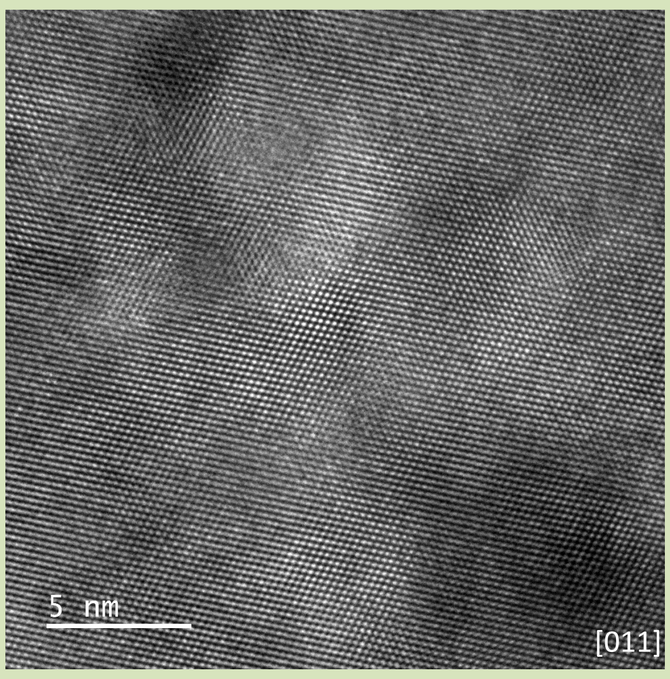
Compared to the traditional TEM sample preparation techniques:

- Reduced contamination (less TEM-beam damage)
- Reduced mechanical strain
- Uniform, extremely wide thin area
- Excellent grain/precipitate retention
- Repeatable process parameters
- Simple workflow, minimal training required, minimal risks of accidental damage during manipulation

Particularly applicable for:
High resolution TEM, SEM-TKD, hard brittle samples, advanced metallurgy, routine inspection



Finished Al-alloy sample (JEOL JEM-2200FS)



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