Living up to Life

TIC 3X - Ion milling system for cross sectioning and large area preparation of sample surfaces

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Leica MICROSYSTEMS

Ion Milling

Most common sample preparation method for Electron Microscopy

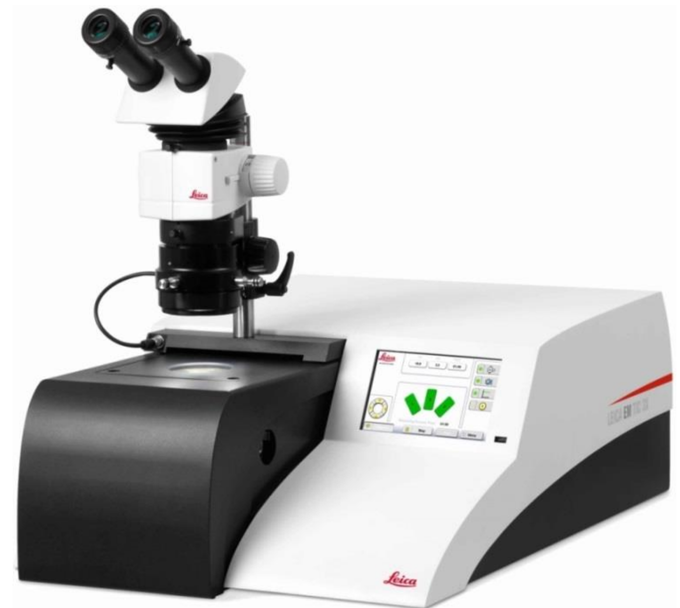
For porous and brittle materials or hard / soft material combinations ion milling is often the only possible method.

- TEM sample preparation
- **SEM sample preparation**
  - **Cross sections**
  - **Surface preparation**

Leica EM TIC 3X

Useable for:

- Cross sections (ion beam slope cutting)
- Large area preparation



EM TXP pre-preparation

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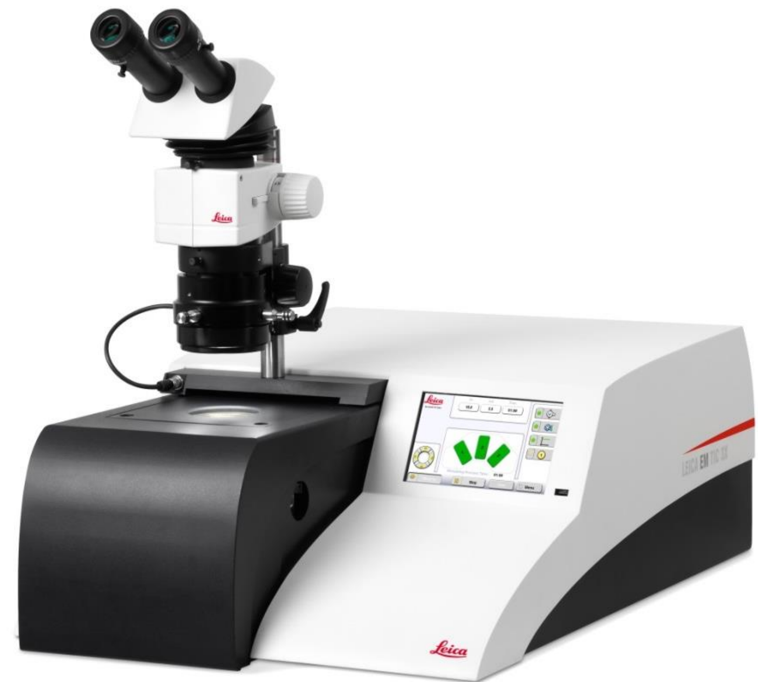
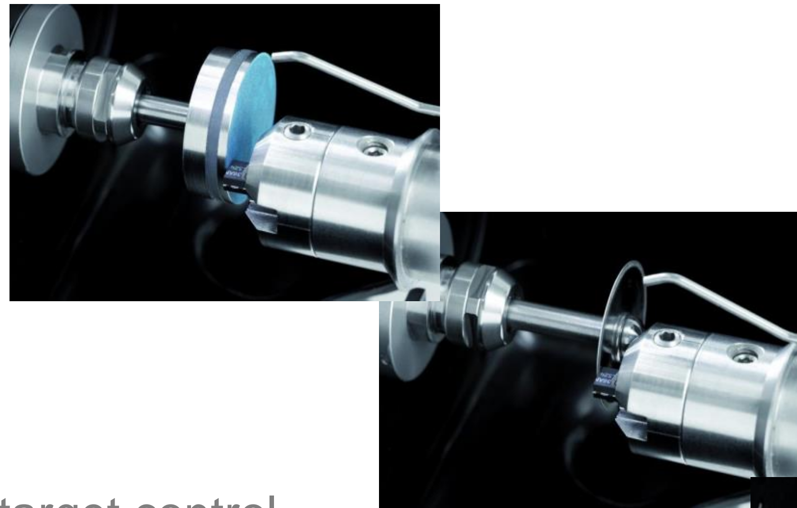
Leica EM TXP

- prior to ion beam slope cutting



EM TXP Unique Target Preparation Device

- Versatile:
  - Polishing
  - Sawing
  - Milling
  - Drilling
- Stereo microscope target control
- Perfect alignment of sample holder and tools
- Integrated automatic process control
- Wide range of sample holders



Specimen holders

Flat specimen holder

Universal specimen holder

AFM specimen holder

AFM specimen holder insert

Specimen stub insert

Specimen holder for
TIC 3X pre-preparation

Sample holders for samples from 1.5 mm x 1.5 mm to 25 mm x 25 mm

SEM PREPARATION WORKFLOW

- MAIN BENEFIT: One sample holder for all instruments in the process chain

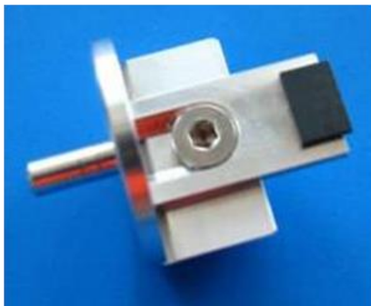
Al holder

TXP preparation

TIC holder

Angle adjustment

TIC - processing



SEM observation

Storage

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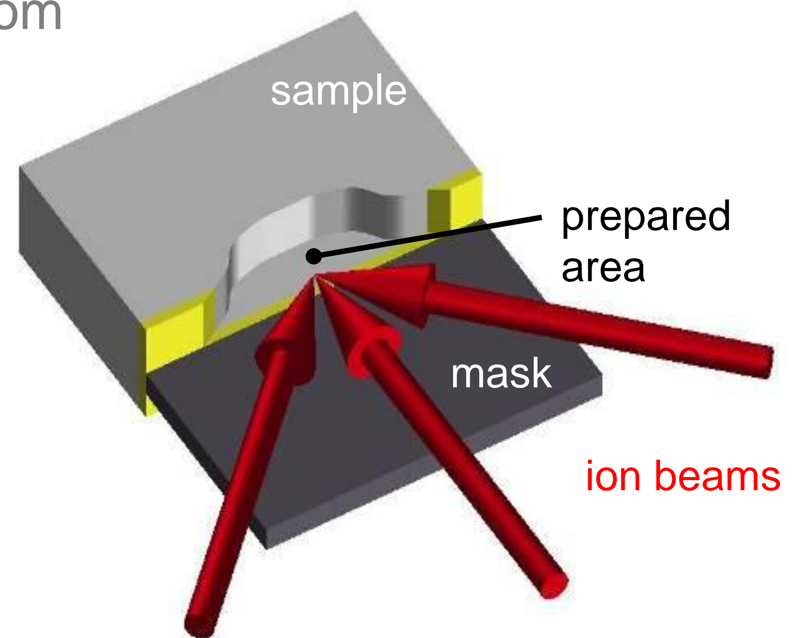
Leica EM TIC 3X

Cross sectional sample preparation

Triple ion beam slope cutting

Principle

- Partially masked sample
- Three ion beams hitting the sample from different directions
- Fixed sample



Features

- Cutting depth >1000µm
- Cutting width > 4000µm!
- Cutting speed > 350 µm/h (Si)

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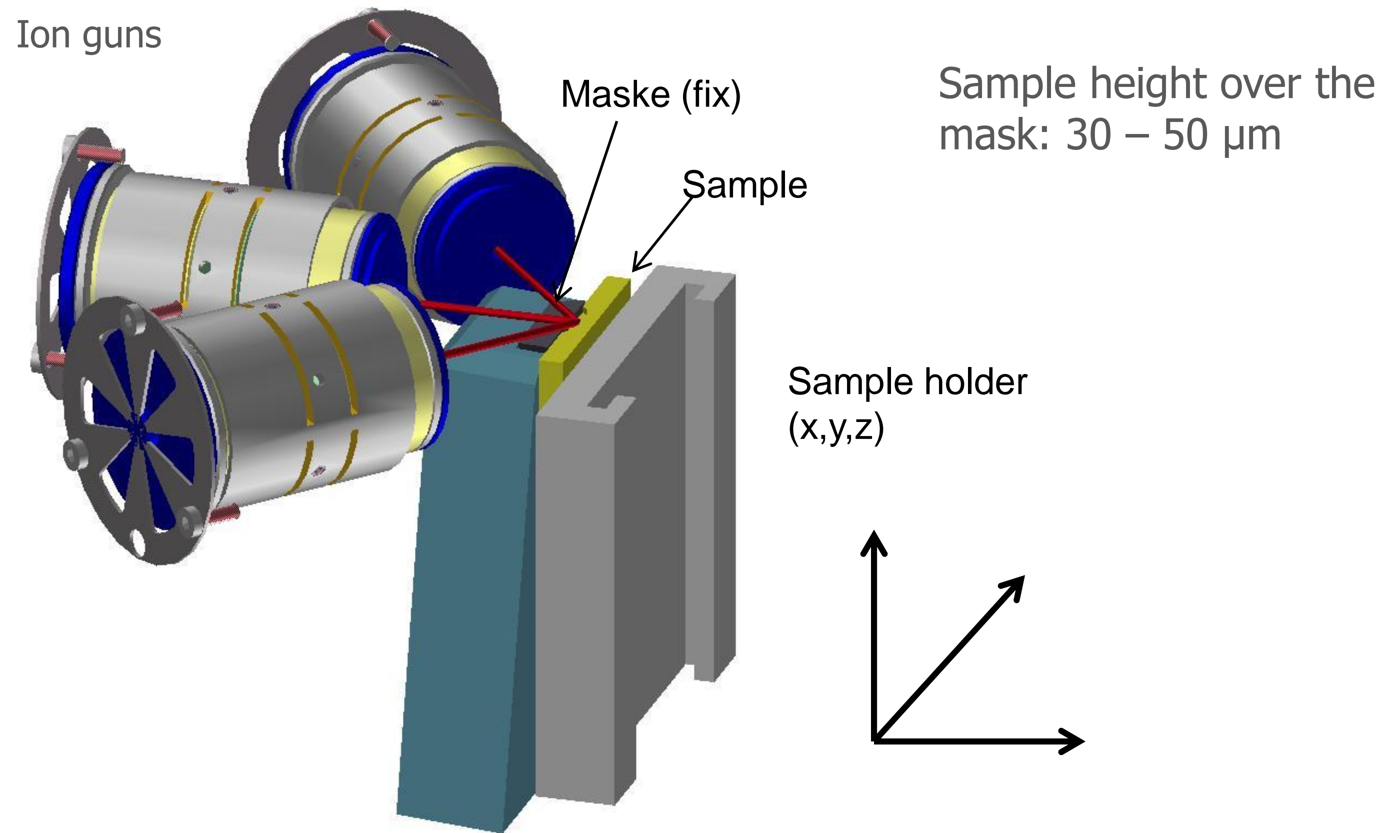
Advantages of the triple beam technique:

- No sample movement
- Optimum observation condition while milling
- Perfect heat transfer
- Sample temperature below 70° C (depending on the sample)

Sample adjustment regarding mask



Sample height over the mask: 30 – 50 µm

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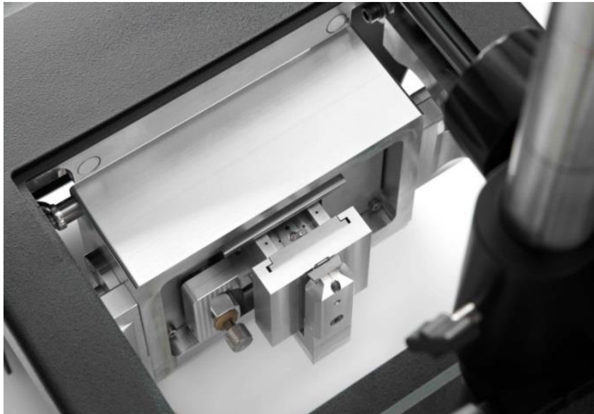
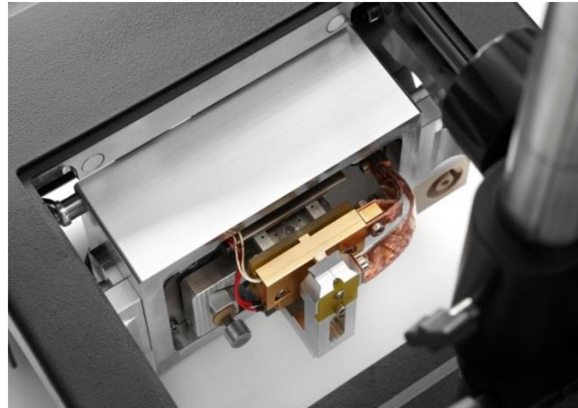
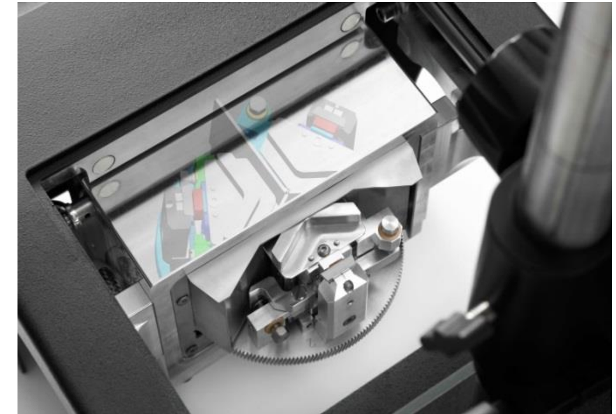
Three stages for cross sections

standard	cooling	multiple sample
		
largest sample size: 50x50x10mm	cooling of sample holder and mask temperature range 30° to -150° C	three loading positions

SEM PREPARATION WORKFLOW

Pre-preparation

Leica EM TXP

Ion beam slope cutting

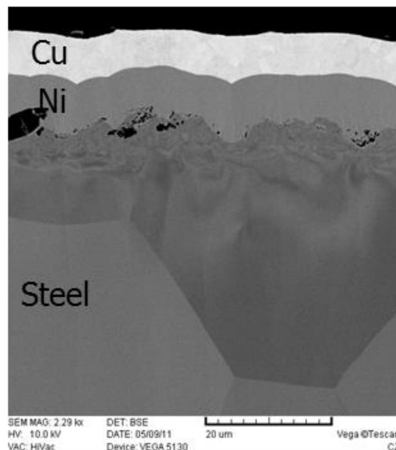
Leica EM TIC 3X

Standard stage

Cooling stage

Rotary stage with new CS holder

Scanning Electron Microscopy



Cross sectional image

Workflow of cross sectional sample preparation for SEM

Preparation problems

Problem:
• Interaction between mask and sample surface leads to contamination and preparation artifacts

Solution:
• Surface protection
• Preparation from the back side

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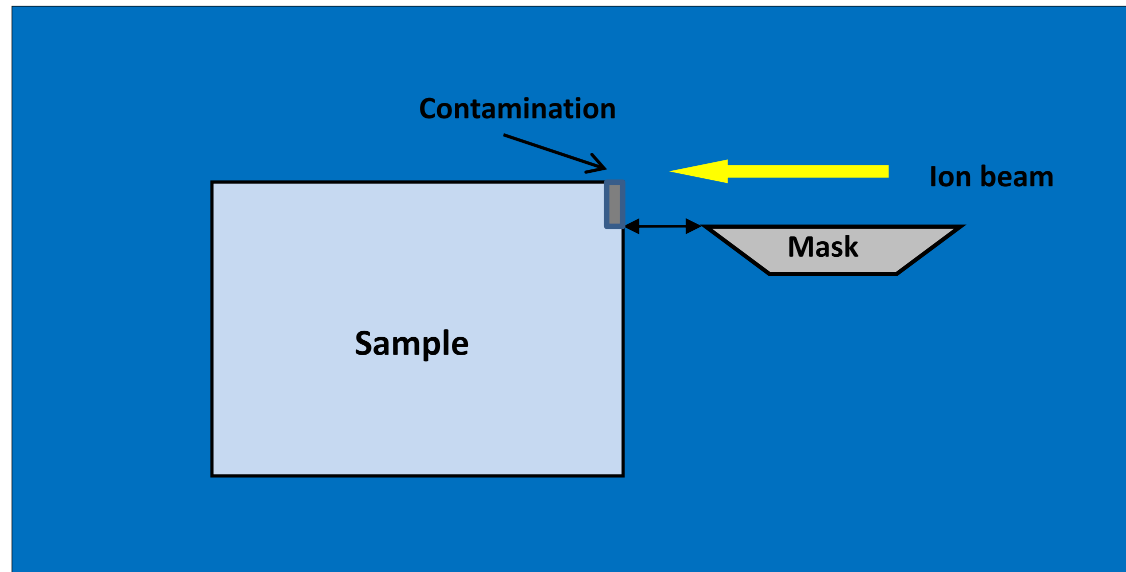
Interaction between sample and mask

- Distance mask-sample is too high



That leads to contamination on the sample edge

Surface protection

Surface protection is important:

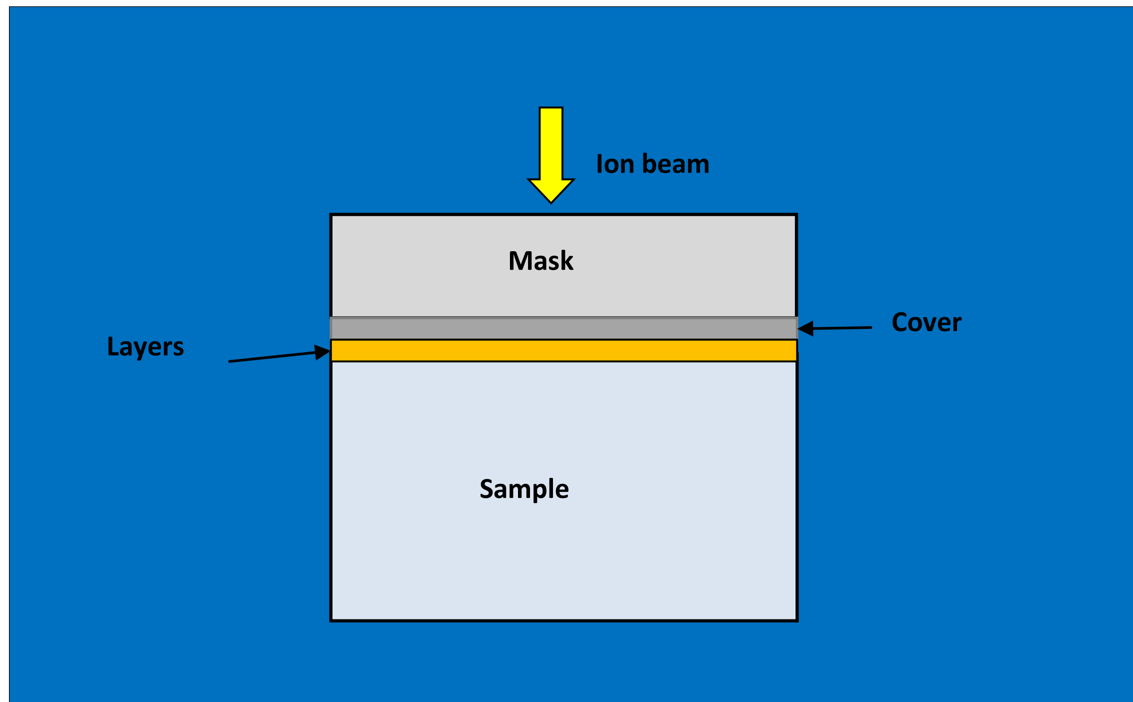
- If the sample surface is not flat
- If the sample has very thin layers (nm range) on top
- If the top layer is very soft

Surface protection through:

- Cover slips
- Si slice
- Different cover materials like layers

Surface protection

- Protection of the sample top to avoid any contamination or interaction



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Sample preparation from the back side

Goal:

- Avoid interactions or damages of the sample surface

Problem:

- Sample thickness is too high

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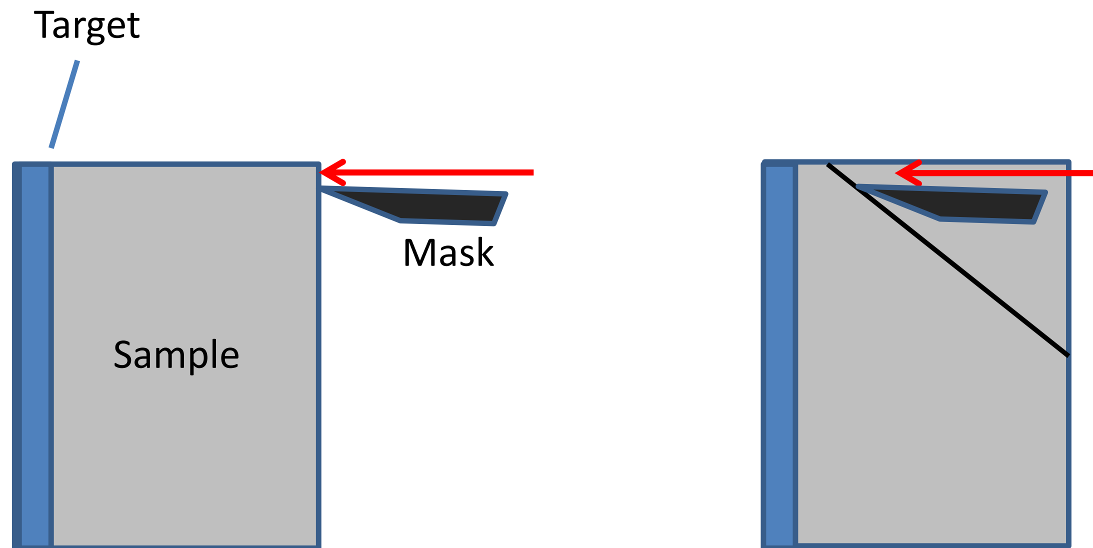
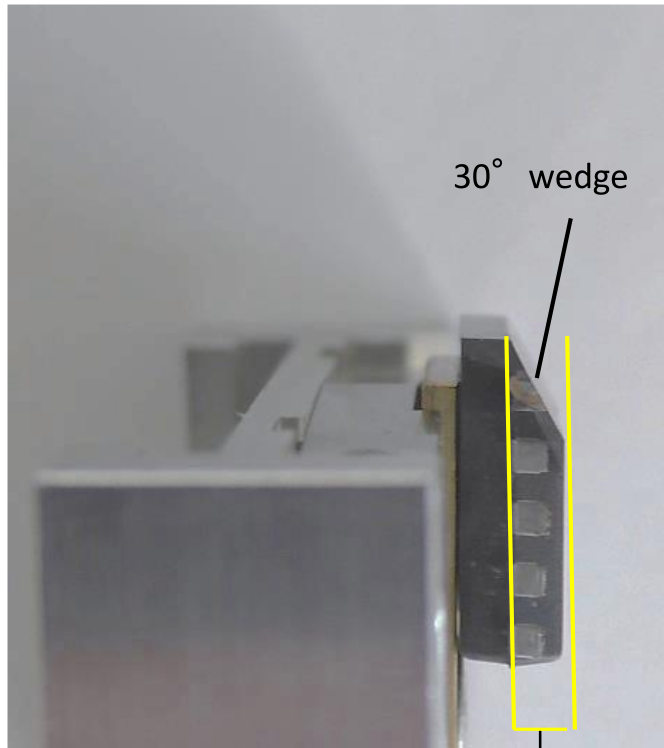
Preparation of a 30° wedge with the TXP

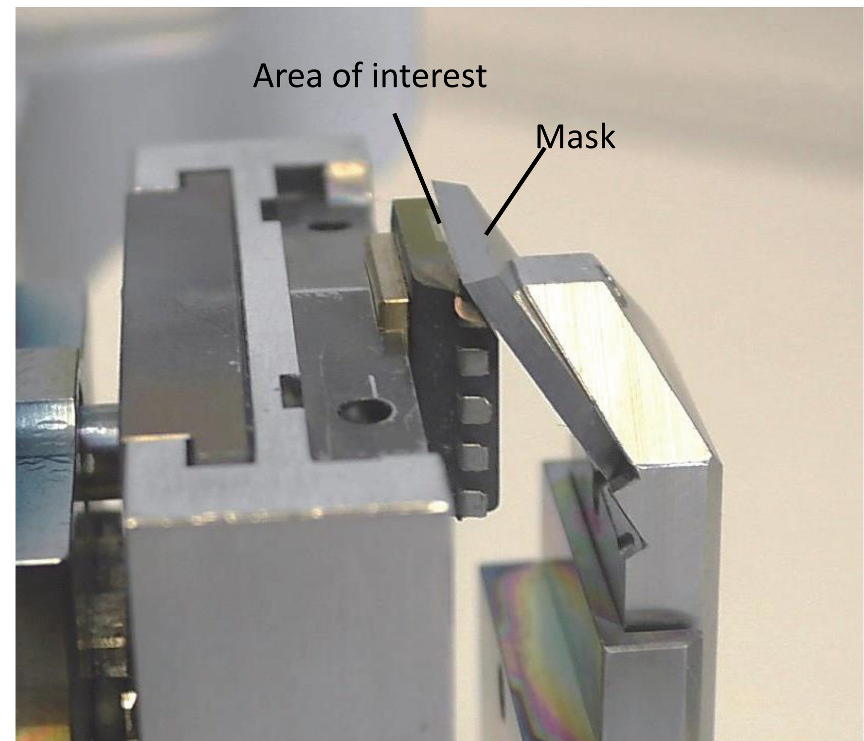


Solution:

Removing the redundant material to reduce the lead time using the TXP

Preparation of a 30° wedge with the TXP





Distance of redundant material removed with Leica EM TXP

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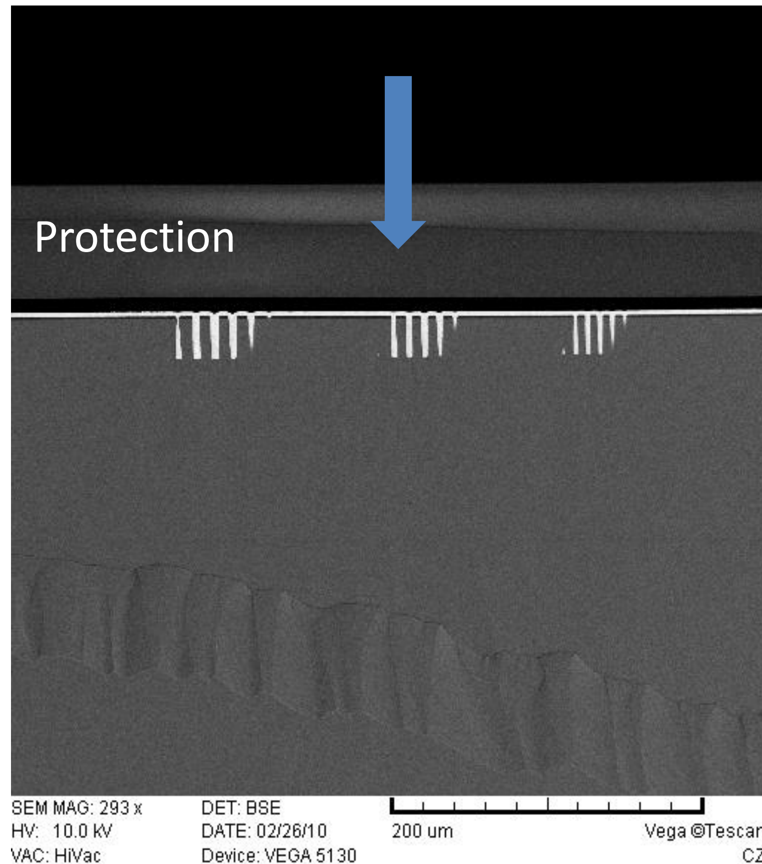
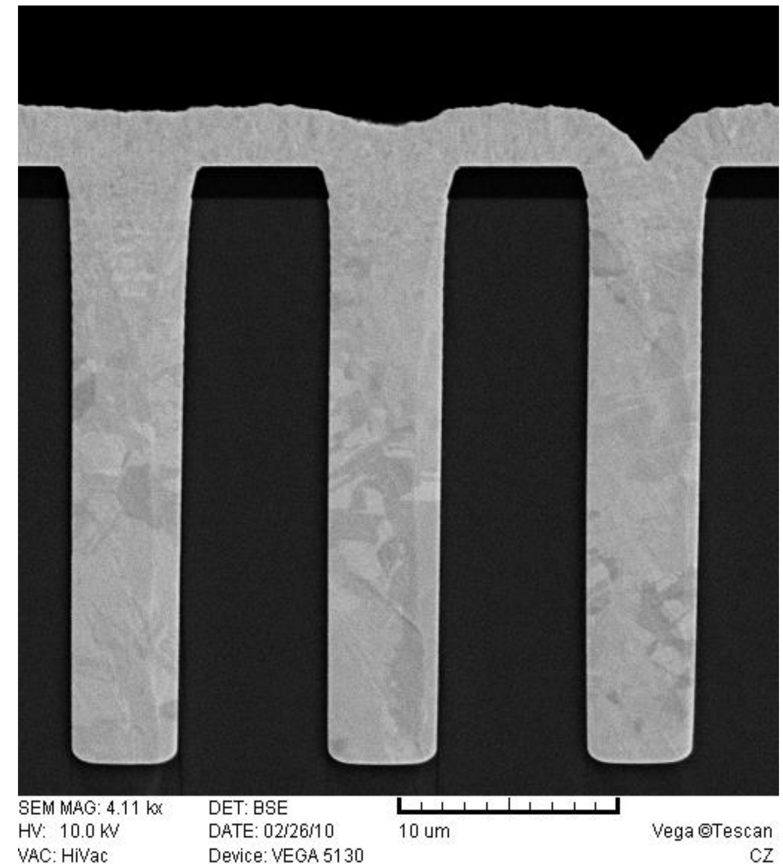
Applications





Cross sectional SEM images of vias filled with Cu (surface protection)

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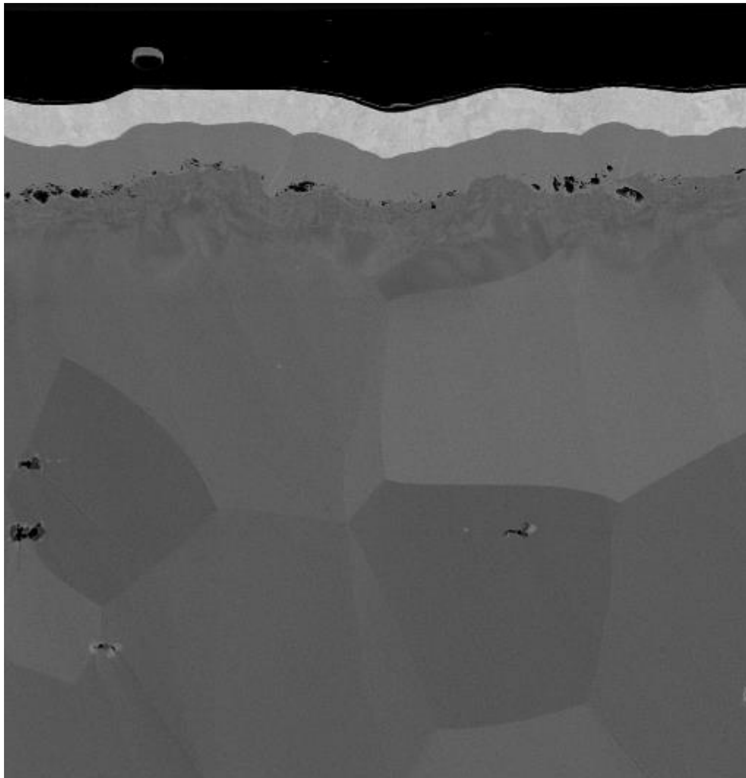
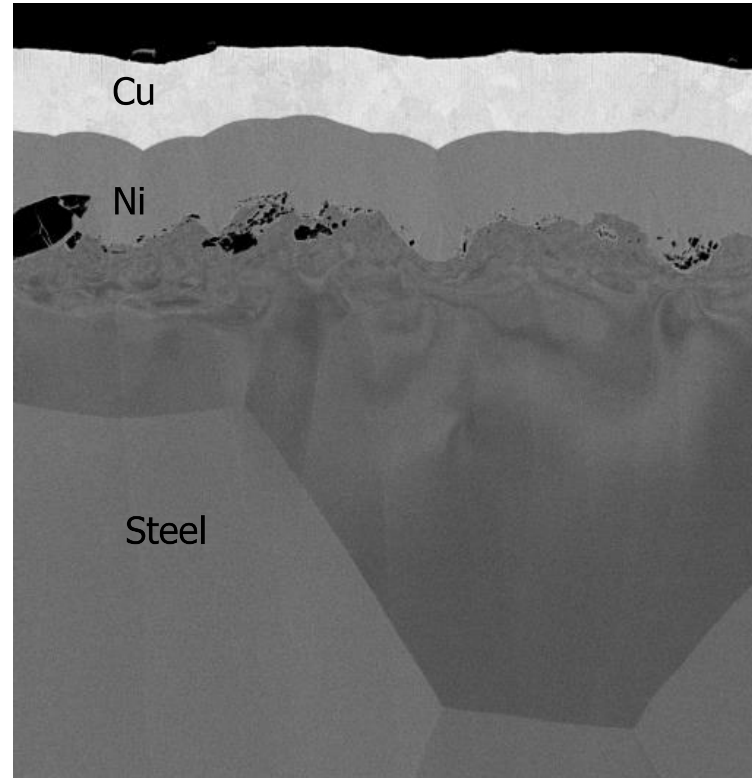
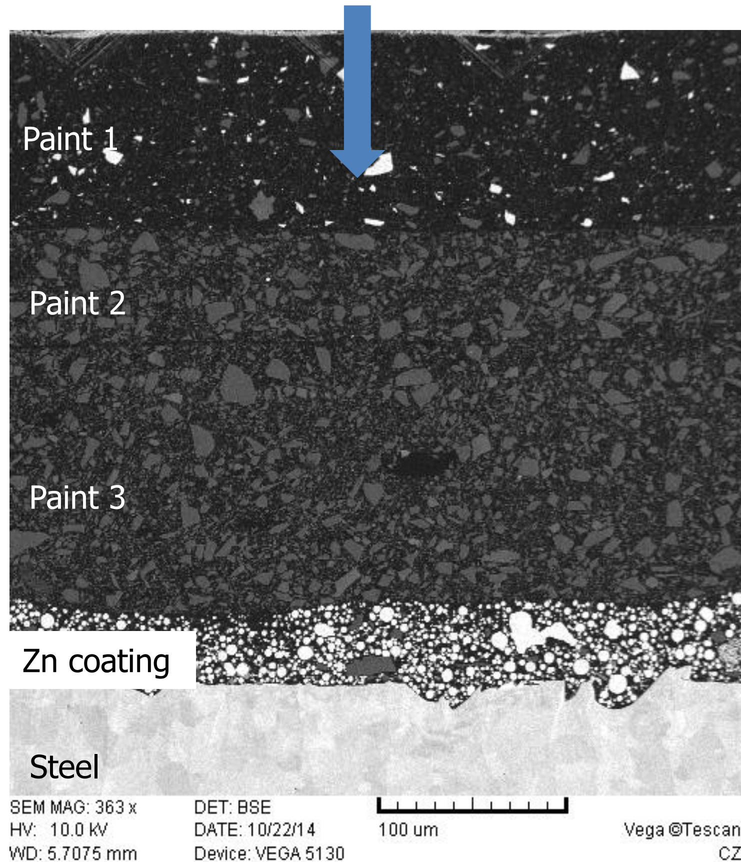
Applications

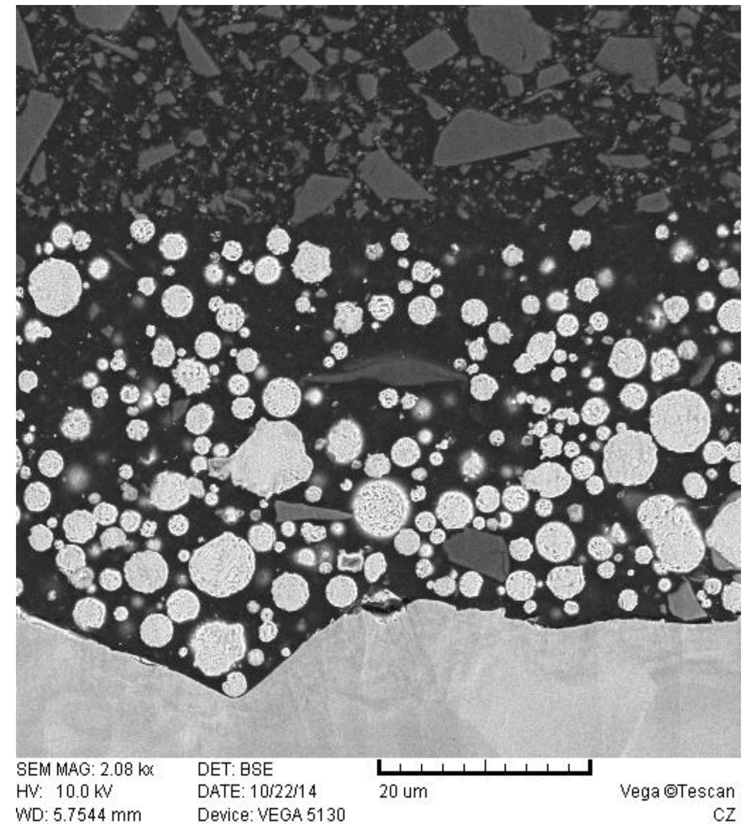




90° slope cuts of Cu / Ni on steel (surface protection)

Applications





Carbon steel coated with Zn based coating and painted with 3 paintings

(surface protection)

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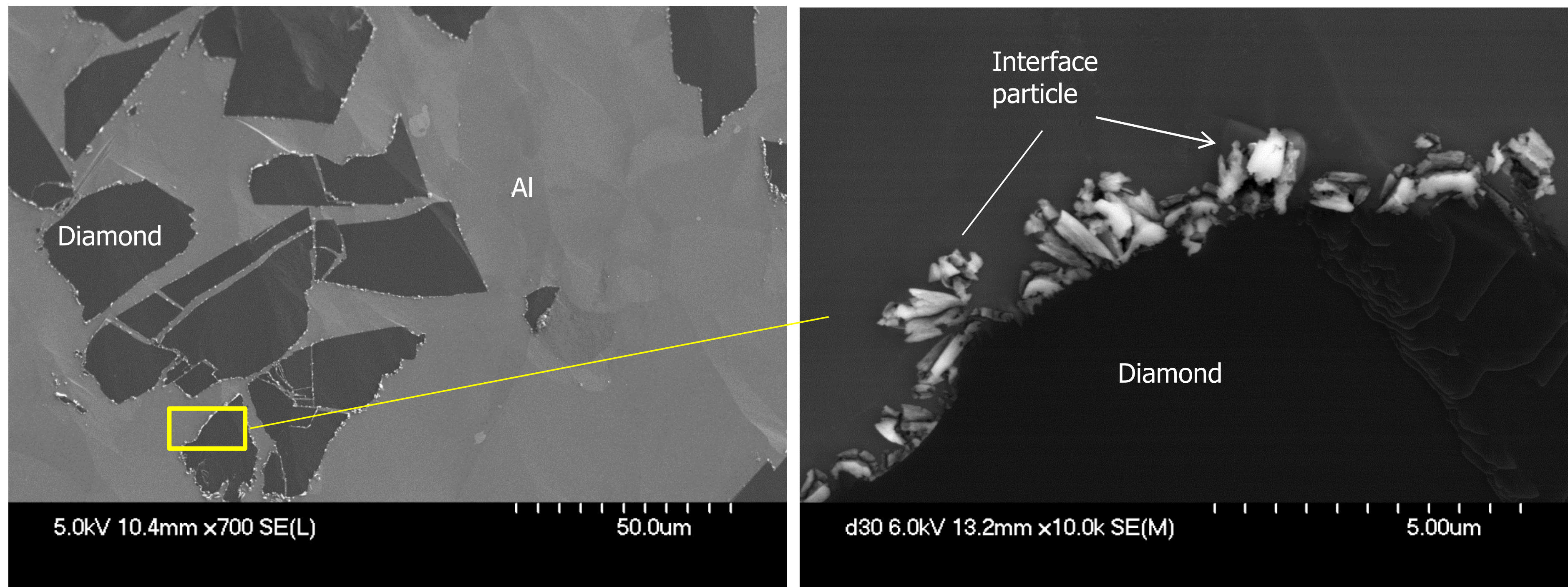
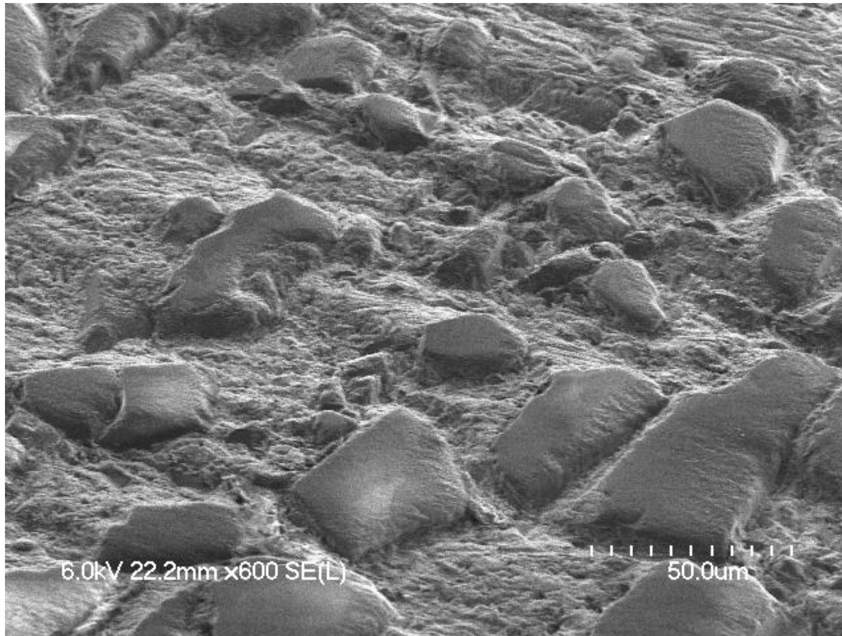
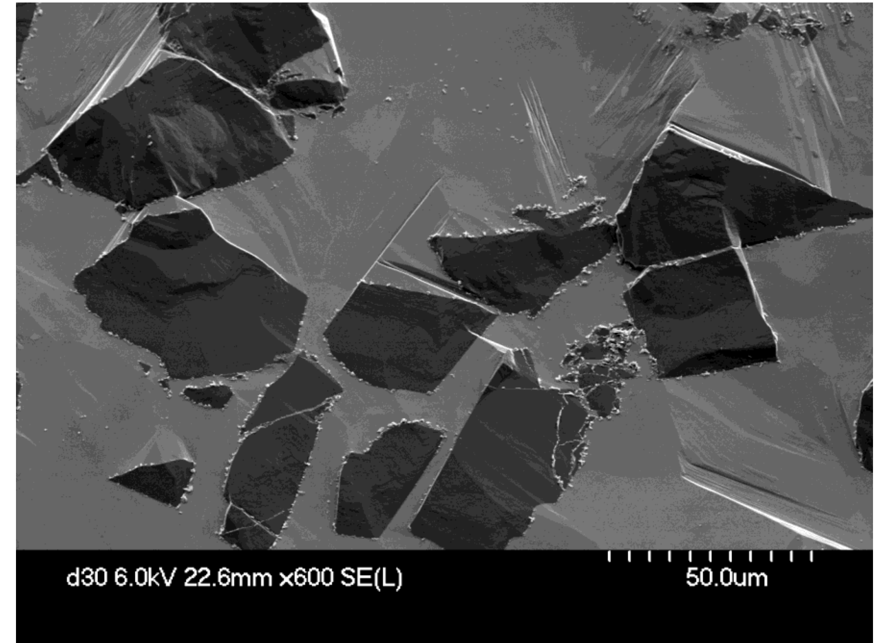
Applications



Cross section of a diamond / aluminium composite

Applications





SEM images of a diamond/Al composite:

(left) Rough surface as a result of mechanical polishing using the tripod,
(right) nearly perfect surface as a result of ion milling using the Leica EM TIC 3X,

The sample was tilted by 45° to compare the surface roughness.

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EBSD Application Graphite flakes/diamond/Al composite

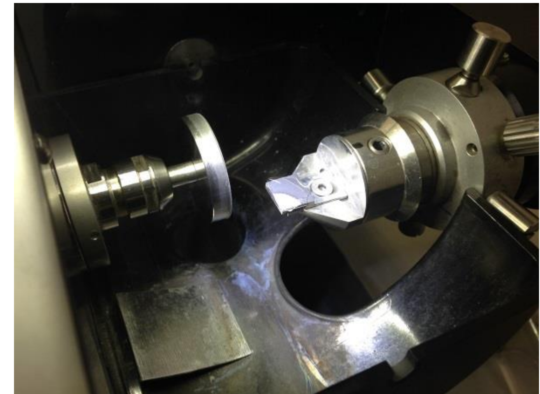
Goal: EBSD,

Problems: Big difference in hardness

Preparation conditions:

Mechanical pre-preparation:

- Grinding of the cross sectional surface with TXP

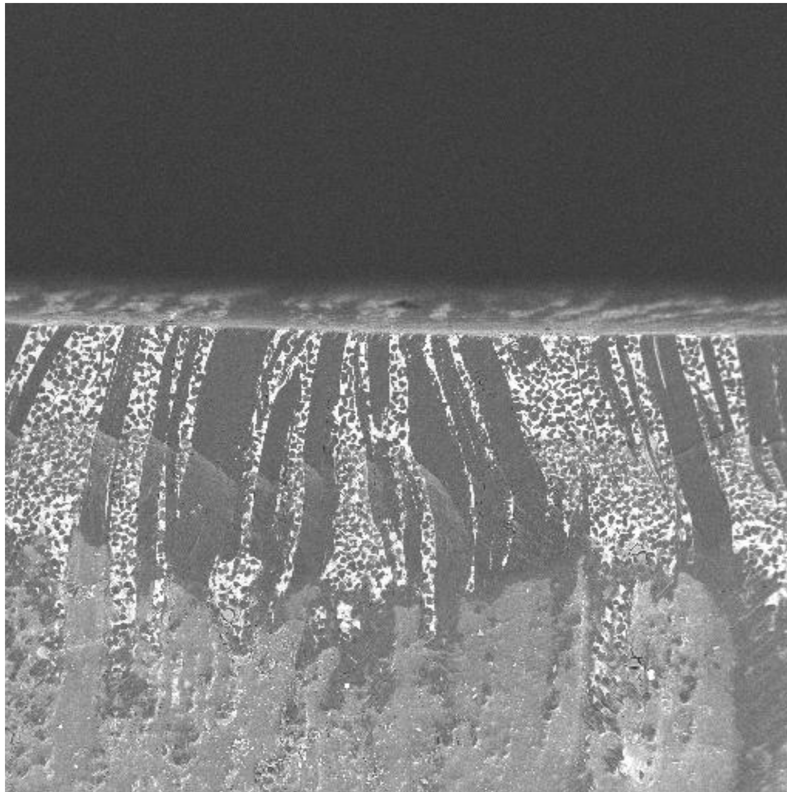


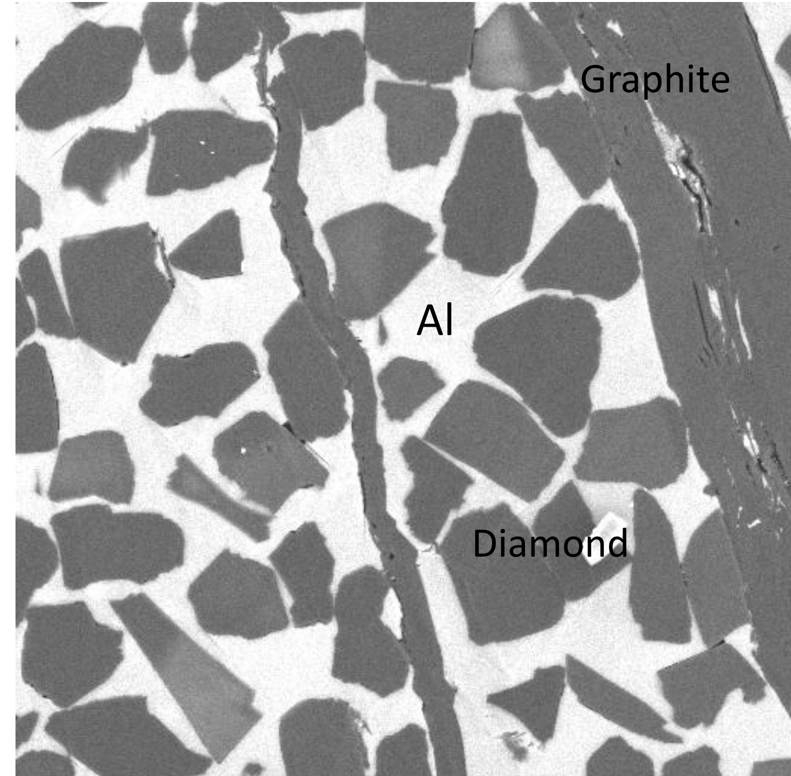
Ion milling:

Acceleration voltage: 6 kV

Milling time: 8 h

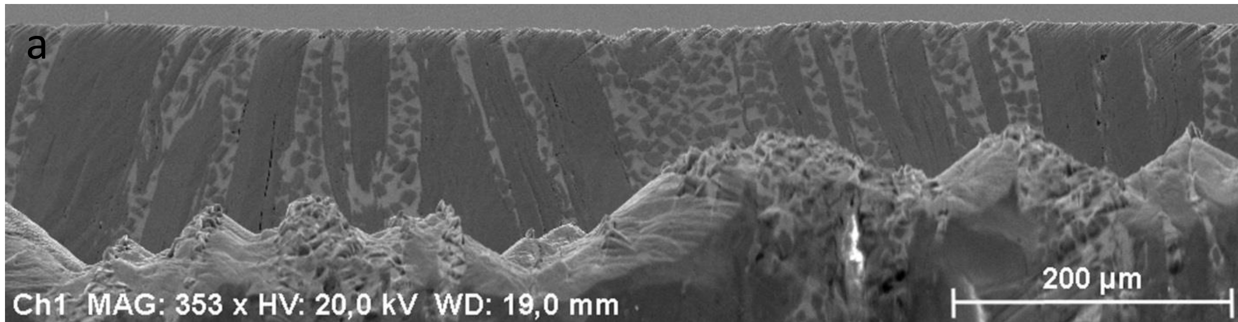
EBSD Application

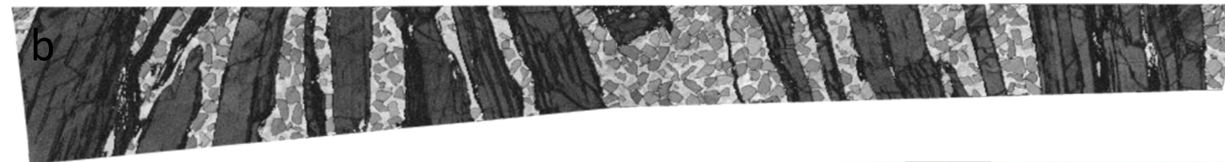


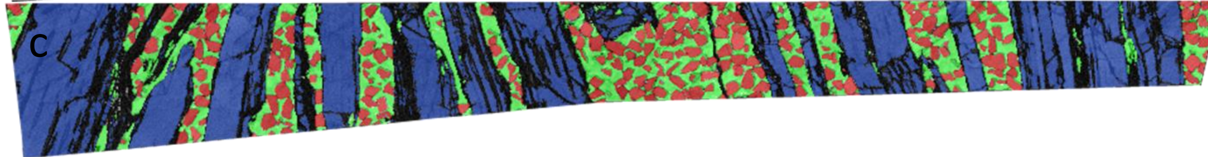


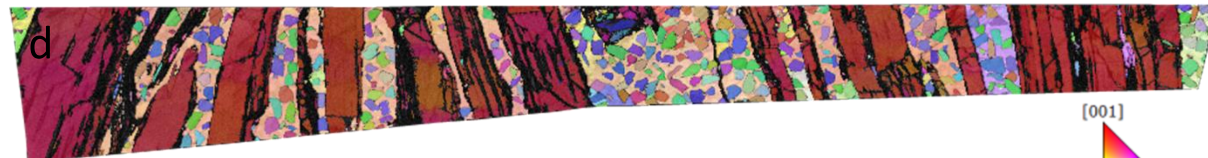
Graphite flakes / diamond / Al composite

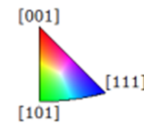
Applications EBSD

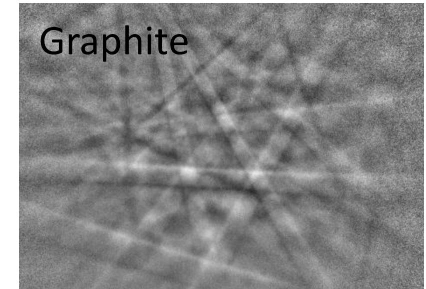


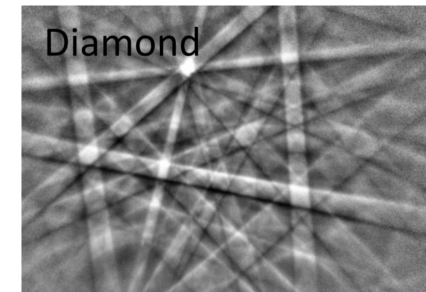


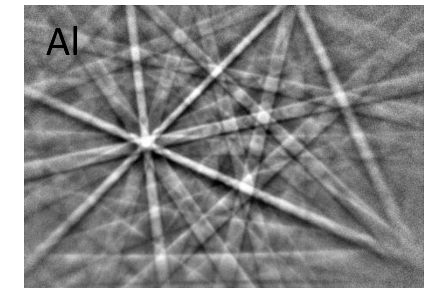






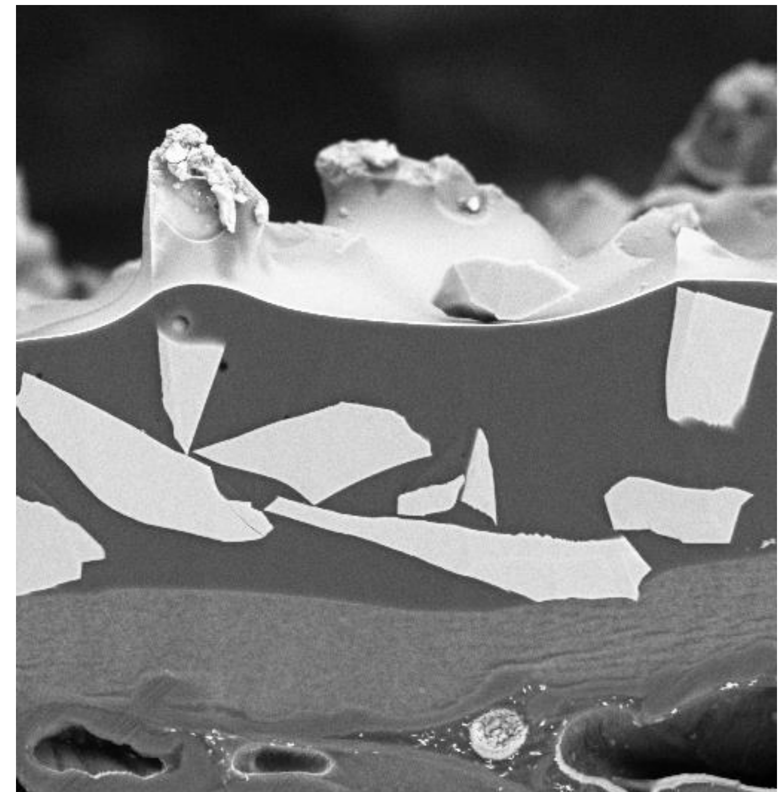






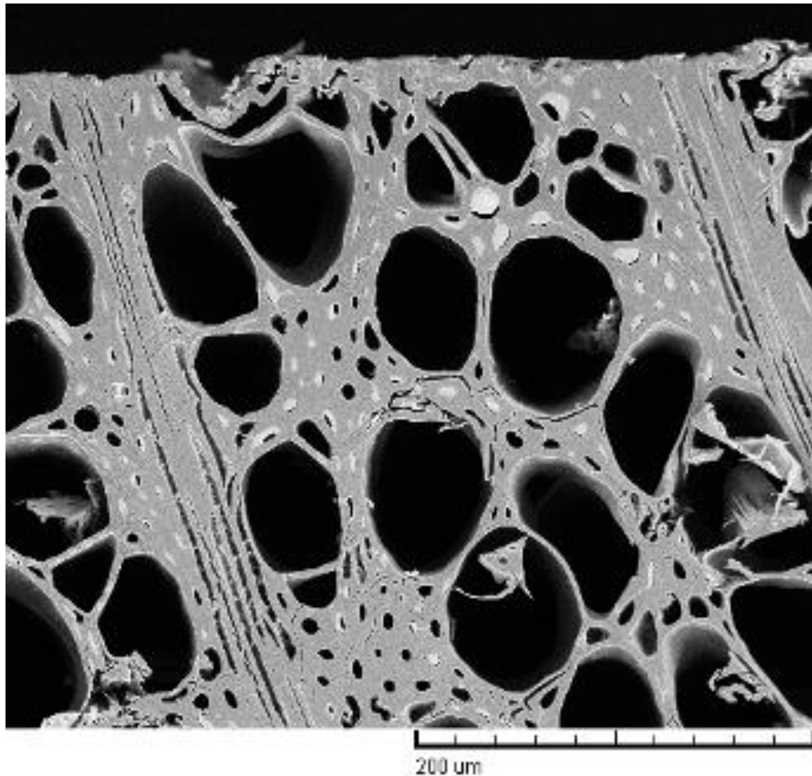
Cross section of a Aluminum / Diamond / Graphite Composite with SEM image(a), pattern quality map (b), phase map (c), orientation map along the x-axis (d) and the corresponding diffraction patterns (Laurie Palasse, Bruker Nano)

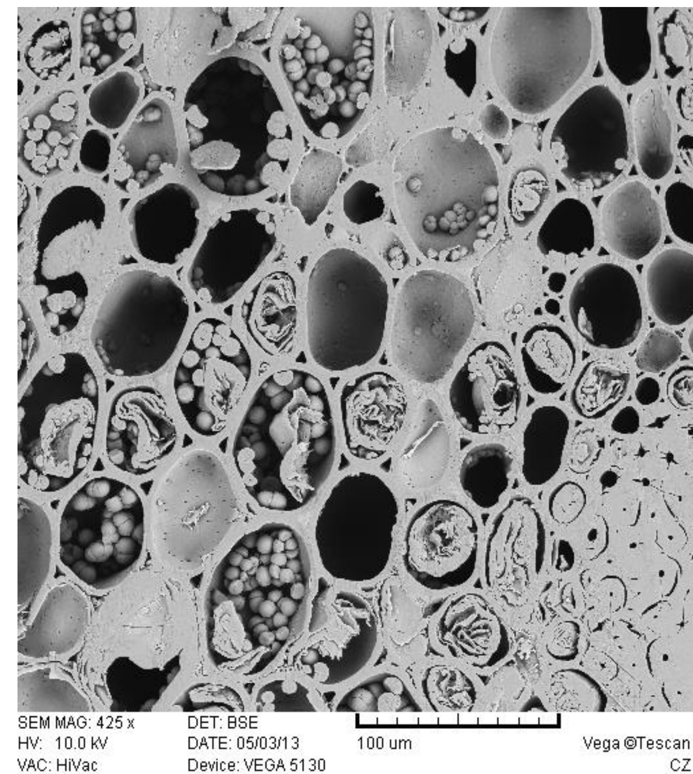
Applications



90° slope cuts of 1200 grit SiC foil with information about grain size, shape and orientation (prepared from the back side)

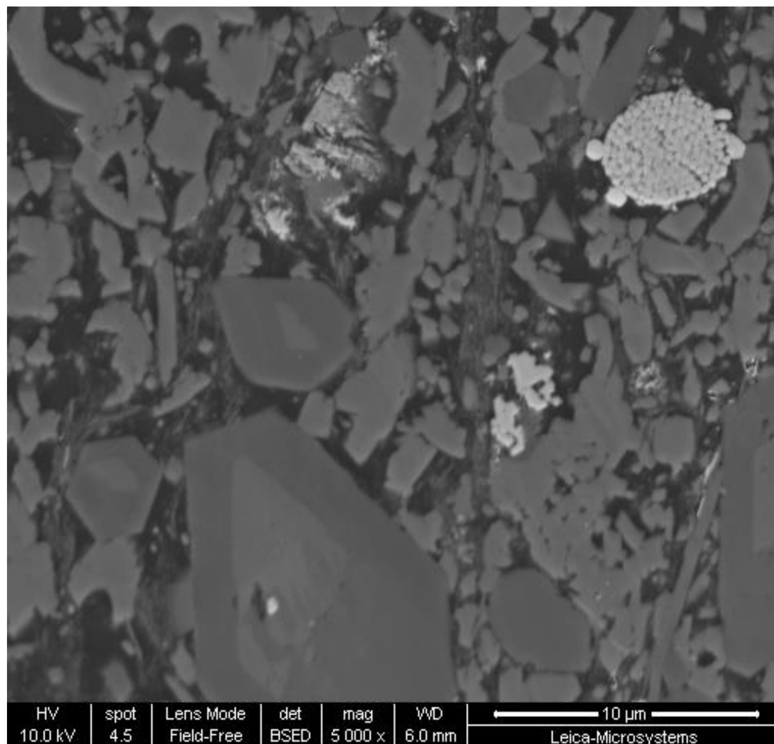
Applications

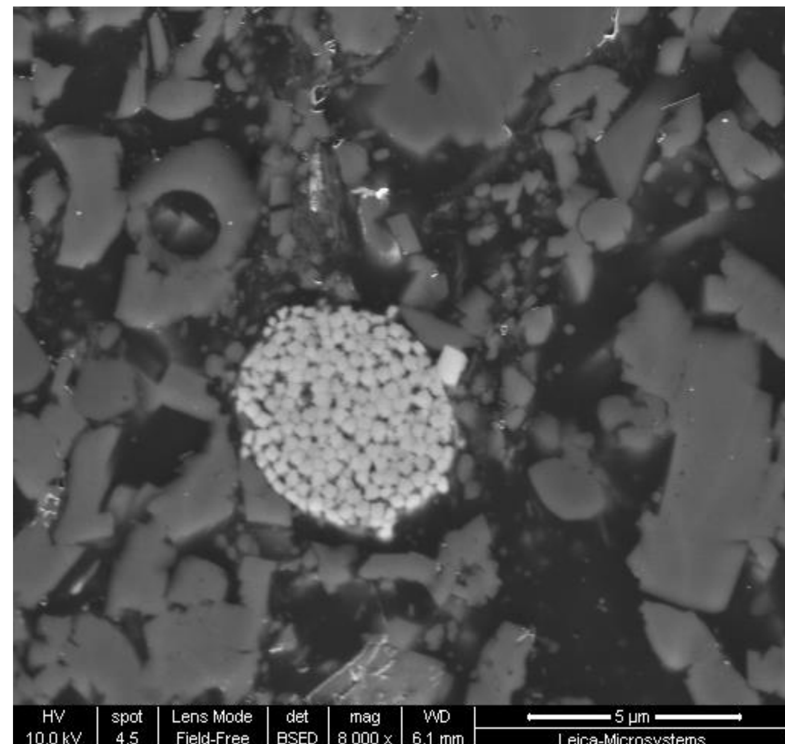




Cross section of veneer (left) and bamboo (right)

Applications

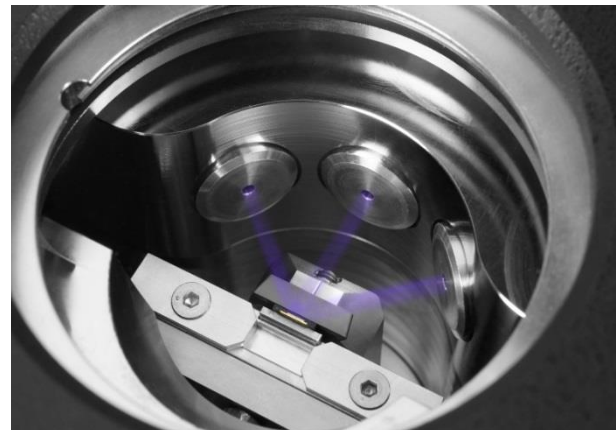


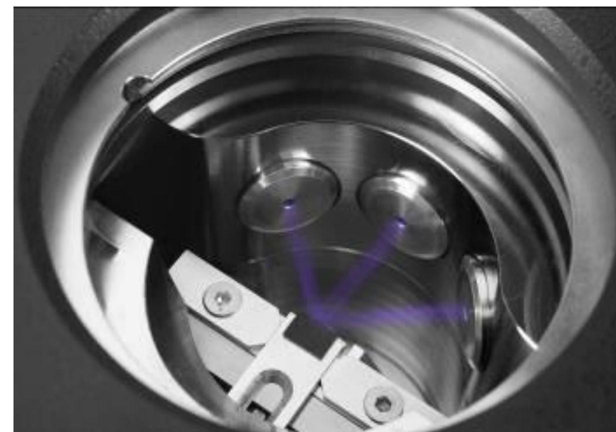


Cross section of oil shale

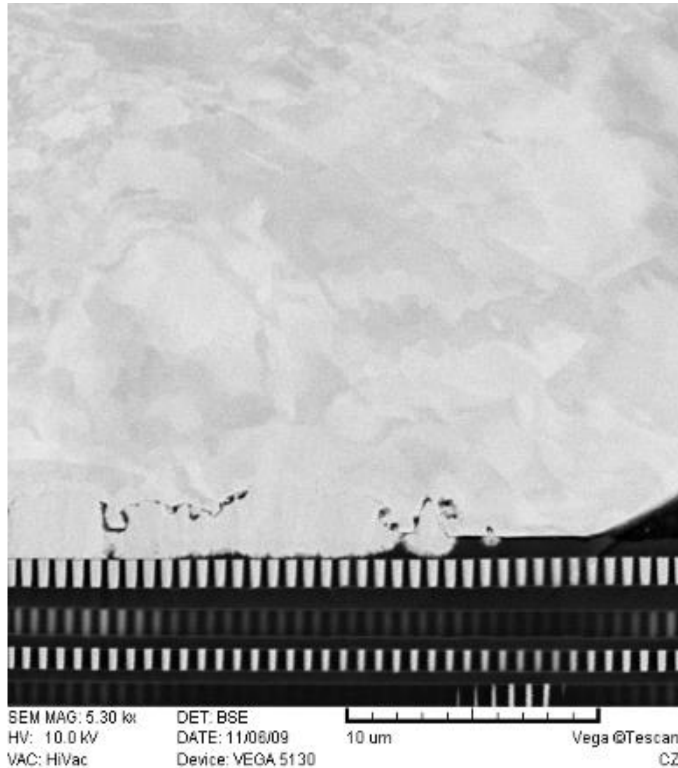
Standard stage

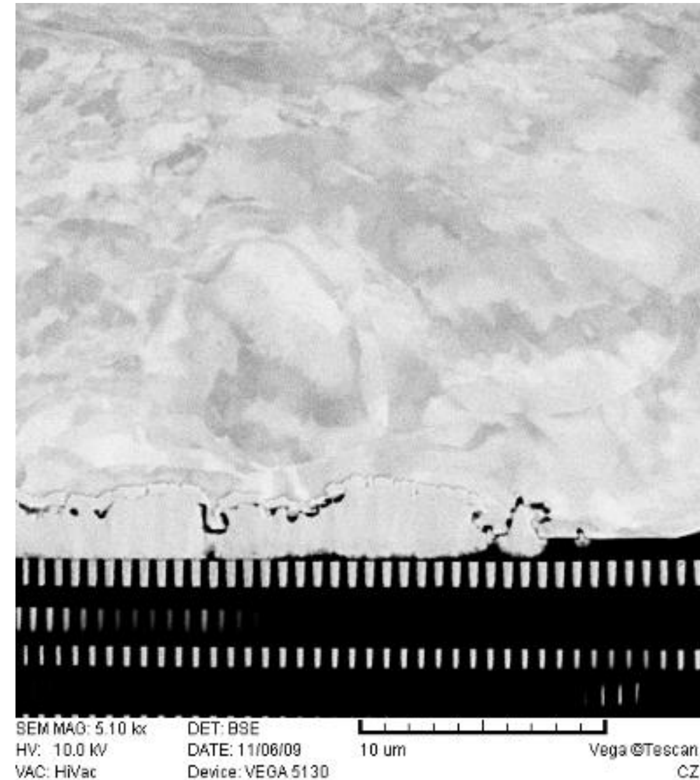
- contrast enhancement





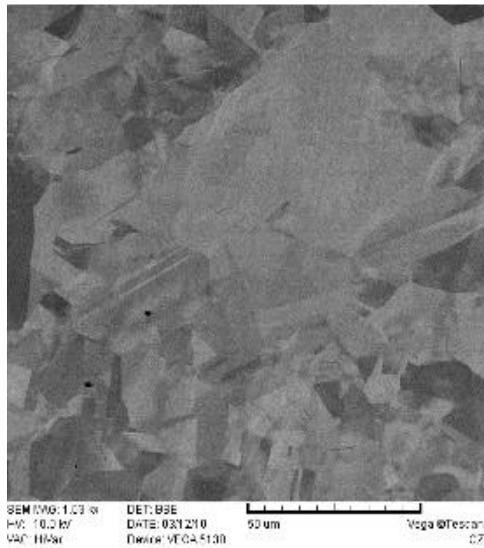
Application contrast enhancement



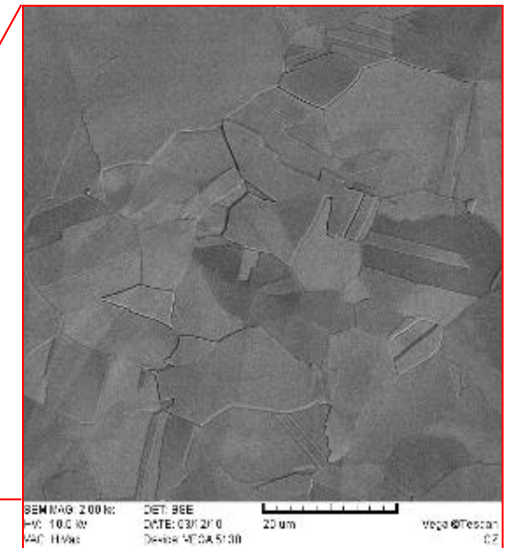


Gold wire bond after slope cutting (left) and after additional contrast enhancement (right)

Application contrast enhancement

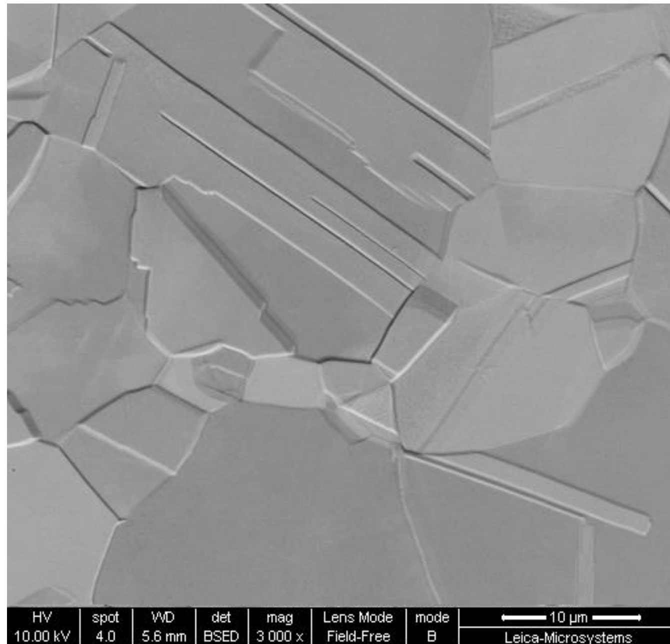


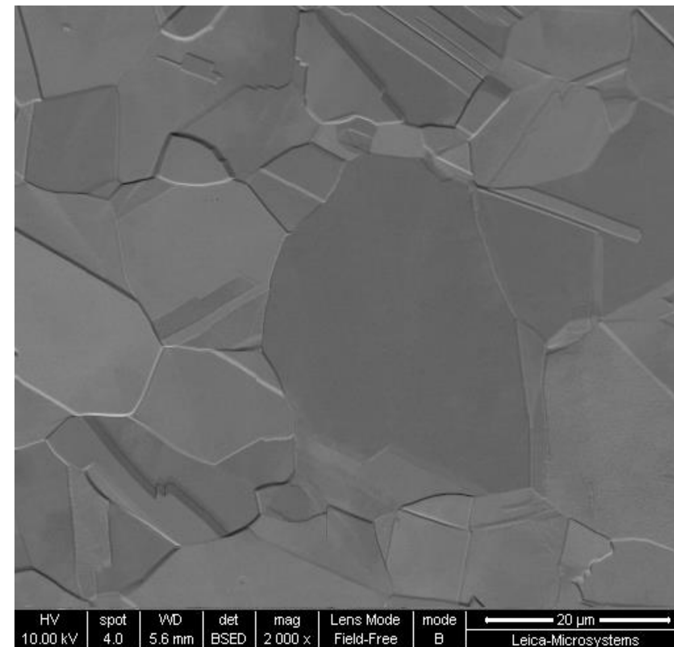
Cu sample after ion beam slope cutting



Cu sample after additional contrast enhancement step
U = 3 kV, t = 2 min

Application contrast enhancement

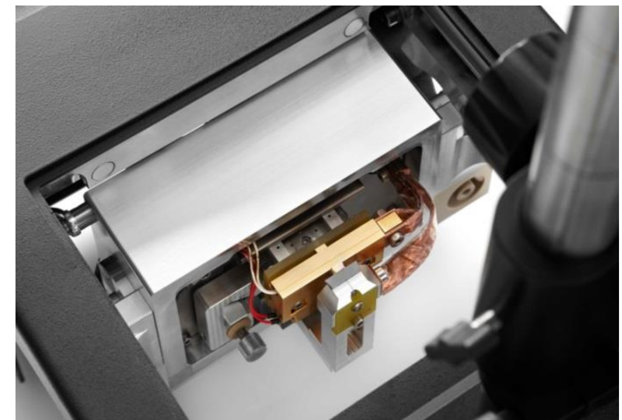




FEG-SEM images of Cu sample after additional contrast enhancement step
U = 3 kV, t = 2 min

Cooling stage

- LN_2 flow design with external Dewar and pump
- Temperature range 30° C to -150° C
- Automatic sample warm-up before venting chamber



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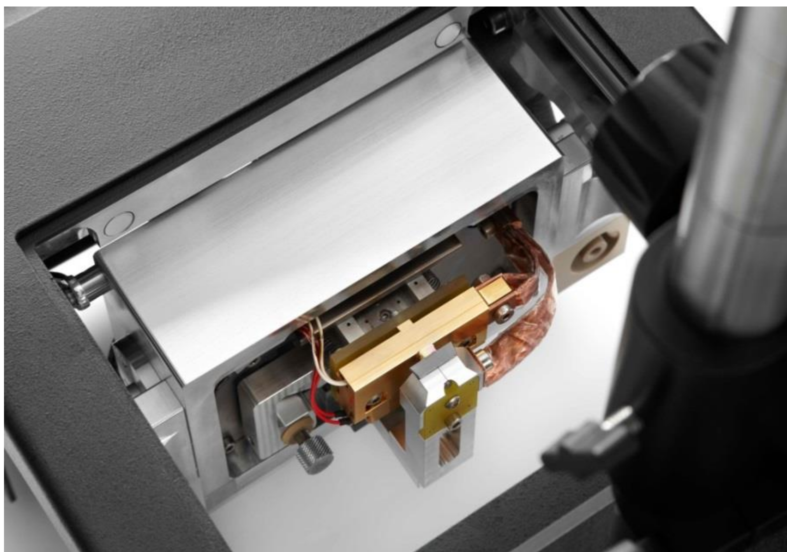
Cooling stage

- temperature range 30° to – 150° C (holder and mask)



sample thickness:

2 to 7mm

0 to 4mm

sample width: ~10mm

max. sample size: 25x25x0-5mm

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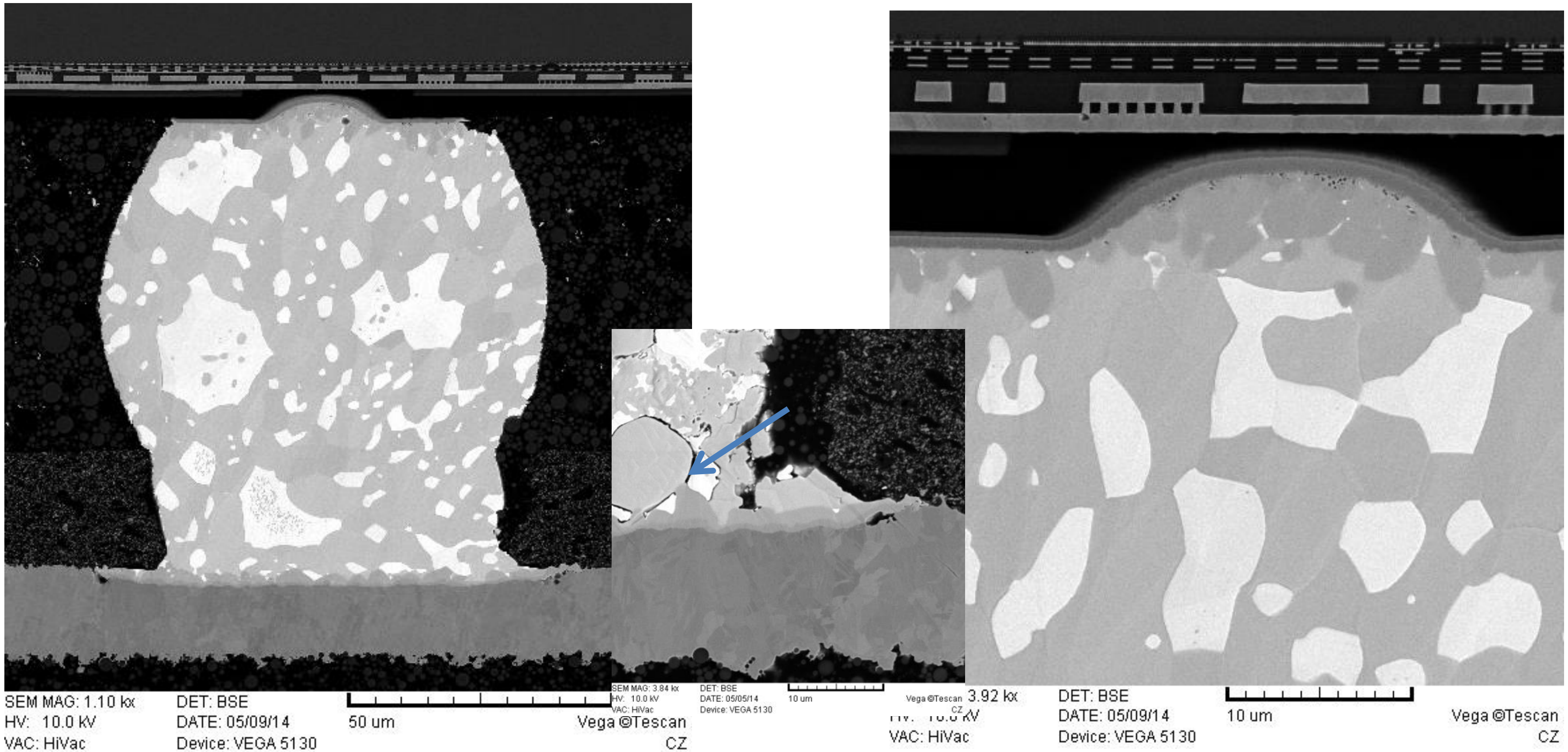
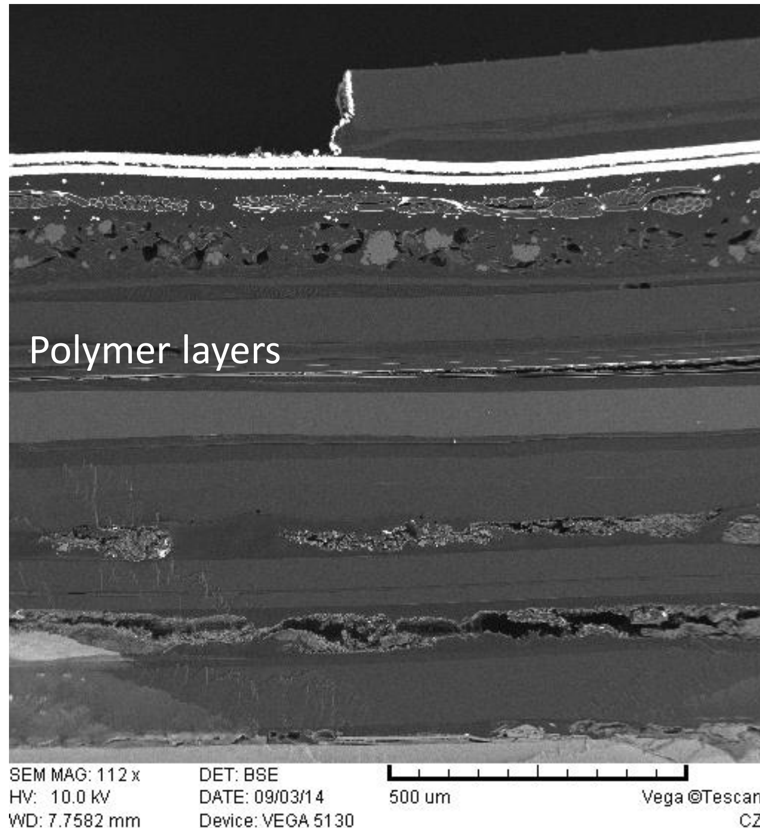
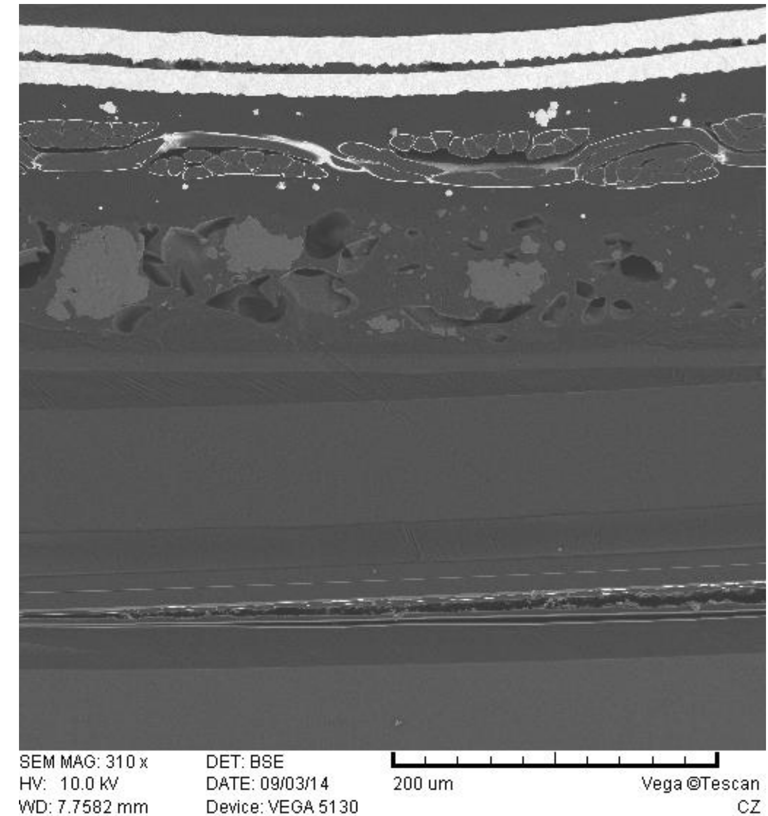
Applications Cooling stage T= -80° C



Cross section of a heat sensitive solder bumps and the thermal effect of a preparation without cooling (small image)

Applications Cooling stage T= -50° C





Cross section of a touch pad with a lot of polymer layers

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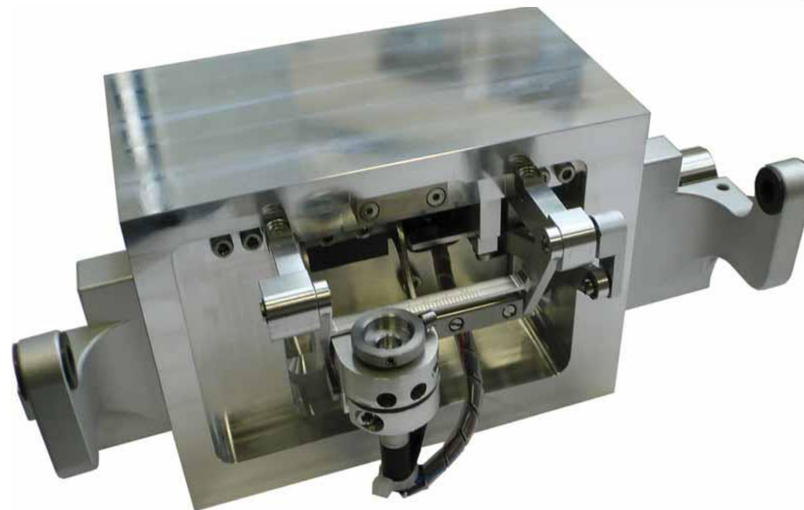
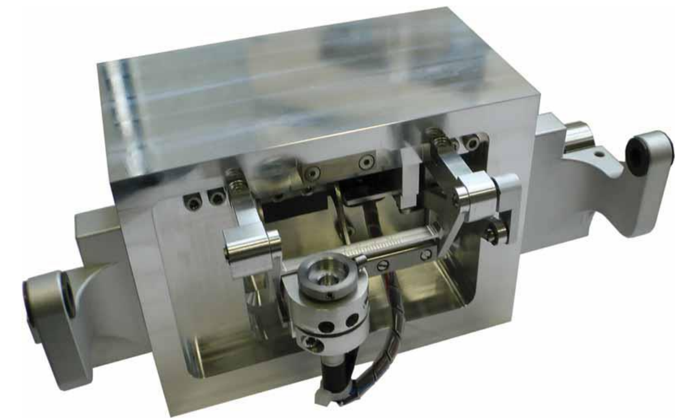
Large area surface preparation with rotary stage

Application:

- Polishing
- Cleaning
- Contrast enhancement



Rotary stage - Large area preparation



Specifications

Max. sample diameter:	38 mm
Max. ion beam prepared area:	Ø 25 mm
Max. sample height:	12 mm
Lateral movement:	+/- 12.5 mm
Incident angle:	0° to 48° (1.5° increments)
Oscillation:	20°, 45°, 90°, 180°, 360°

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Workflow large area preparation

Mechanical Polishing (e.g. with the Leica EM TXP)

Polishing with TXP:

- Diamond foils U type: 9 µm, 2 µm, 0.5 µm
- Preparation time: 0.5h -2h

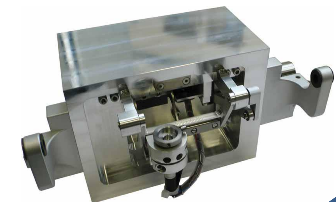
Ion Polishing (Leica EM TIC 3X, Rotary stage)

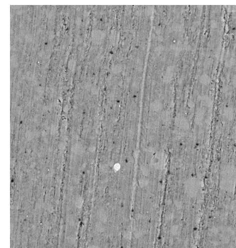
Cleaning:

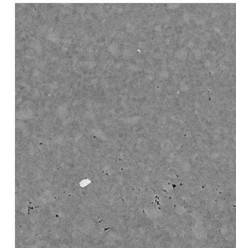
Acceleration voltage:	4 kV
Gun current:	2 mA
Milling time:	10 min
Milling angle:	10.5°
Sample movement.	Rotation
Lateral movement:	± 3 mm

Polishing:

Acceleration voltage:	6 kV
Gun current:	2.2 mA
Milling time:	1 h to 2 h
Milling angle:	3°
Sample movement.	Rotation
Lateral movement:	± 3 mm







SEM images

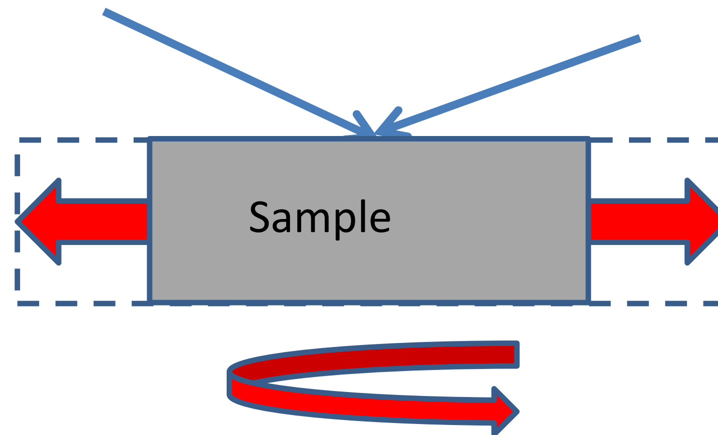
Large area sample preparation

- Sample surface should be almost perfect before ion milling
- Ion polishing is just the final step to remove damages, induced by mechanical polishing
- Hard / soft material combinations need a cleaning step before ion polishing to remove the smeared material

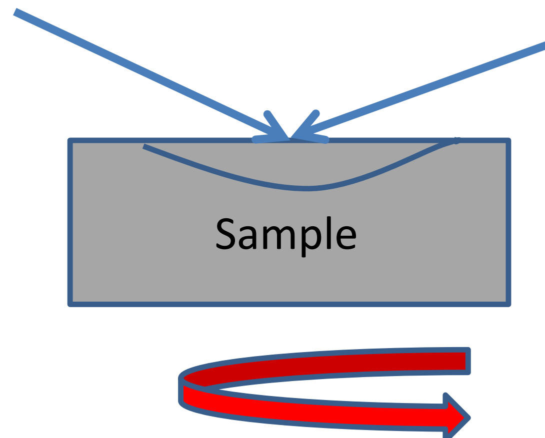
Sample surface is flat after mechanical polishing due to the smearing effect.
There is no polishing effect!

Leica MICROSYSTEMS

Superposition of two movements

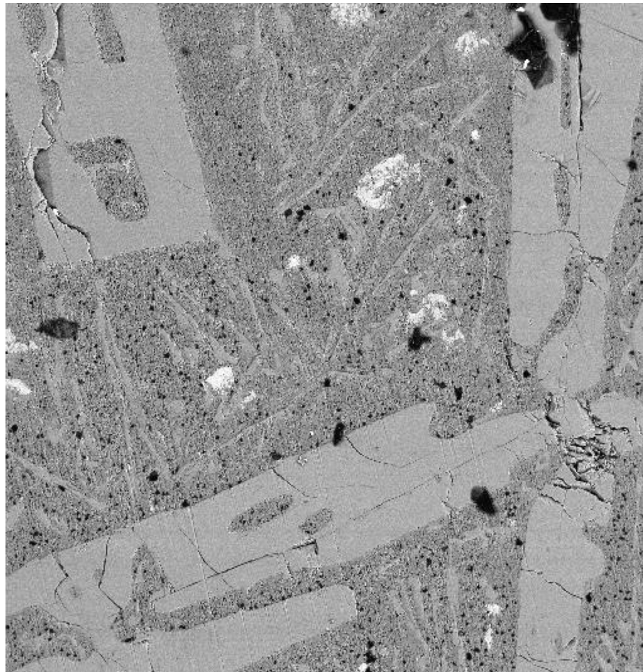


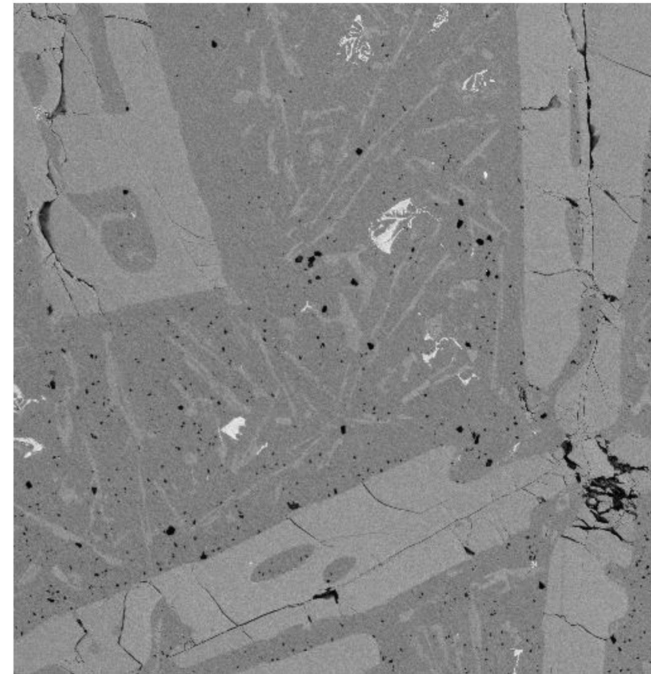
Uniform surface preparation of areas up to 25 mm in diameter



Not uniform surface preparation

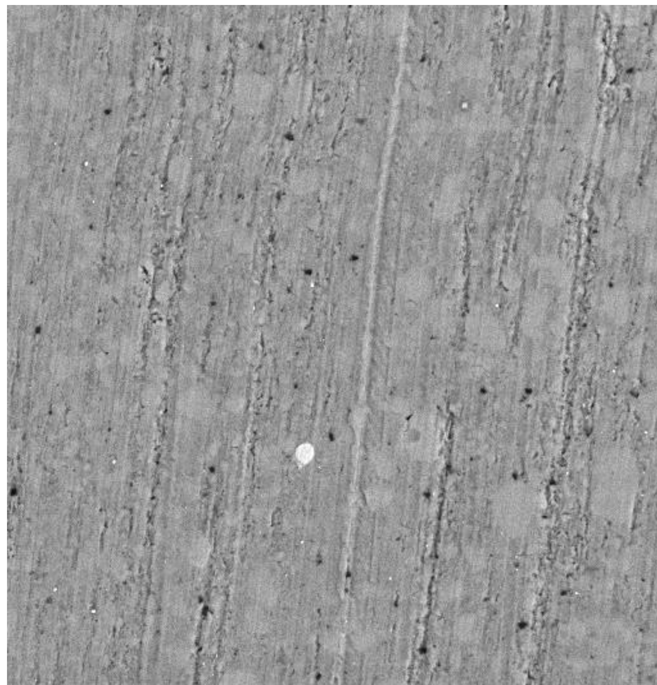
Application Rotary Stage: Friction Stir processed Al-3OSi Alloy

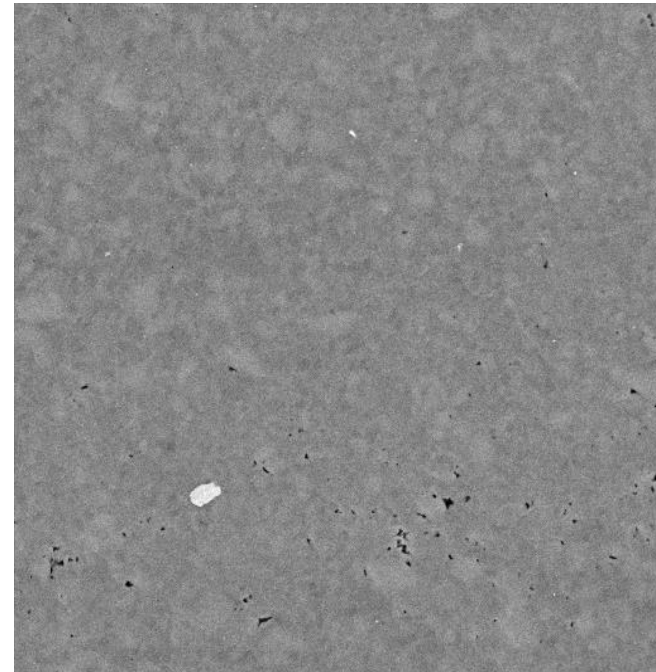




Friction Stir processed Al-30Si Alloy after mechanical polishing with the TXP(left) and after additional ion polishing

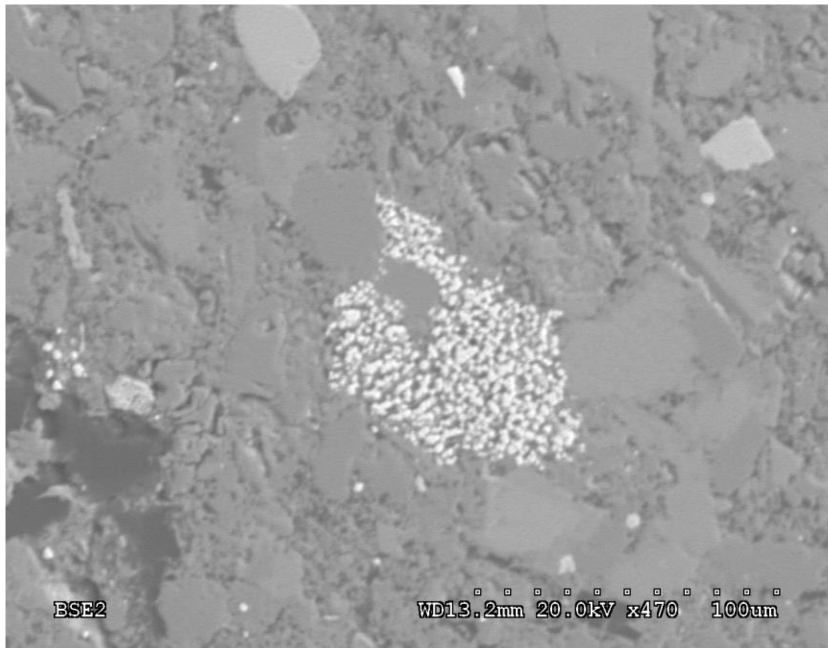
Application Rotary Stage: Friction Stir processed Al-3OSi Alloy

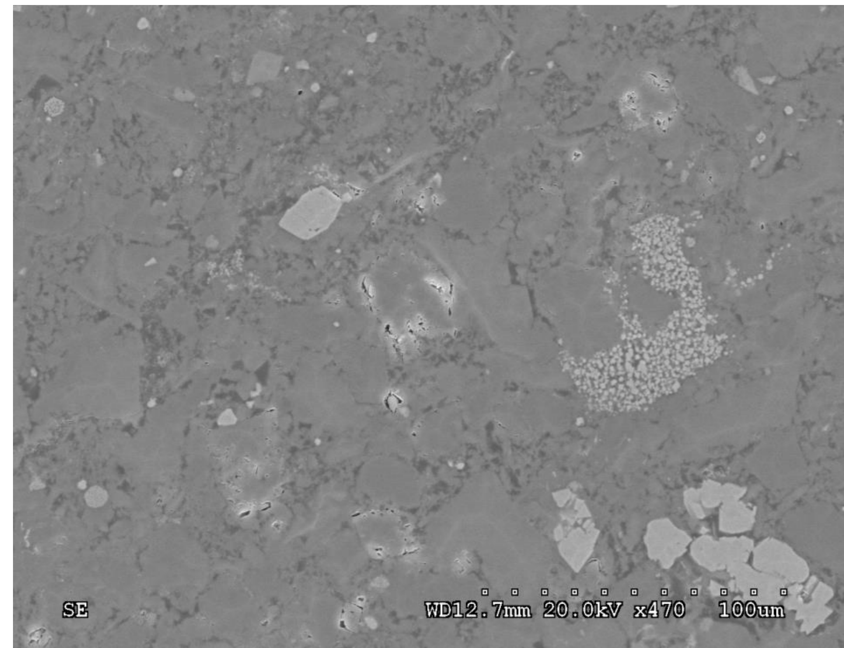




Friction Stir processed Al-30Si Alloy after mechanical polishing with the TXP(left) and after additional ion polishing

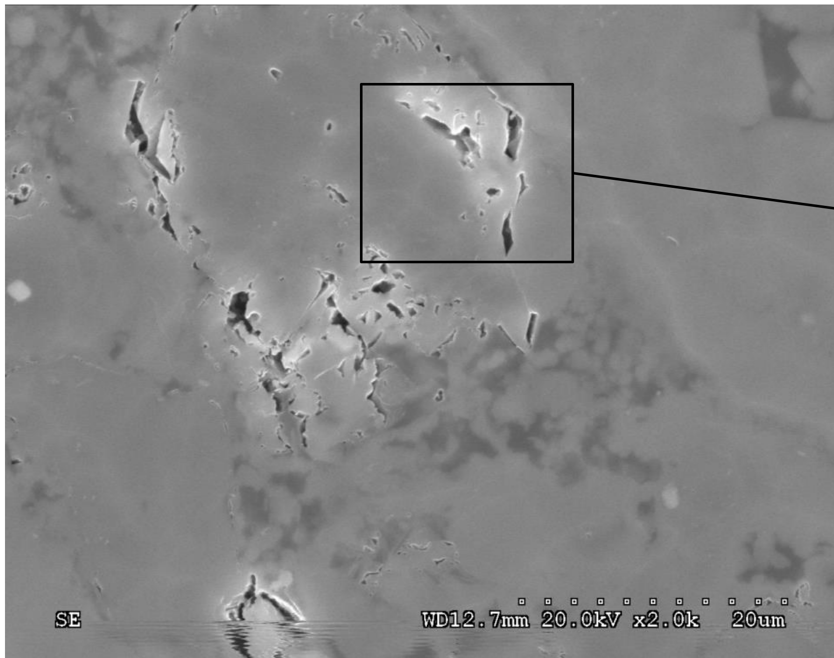
Application Rotary Stage: Shale

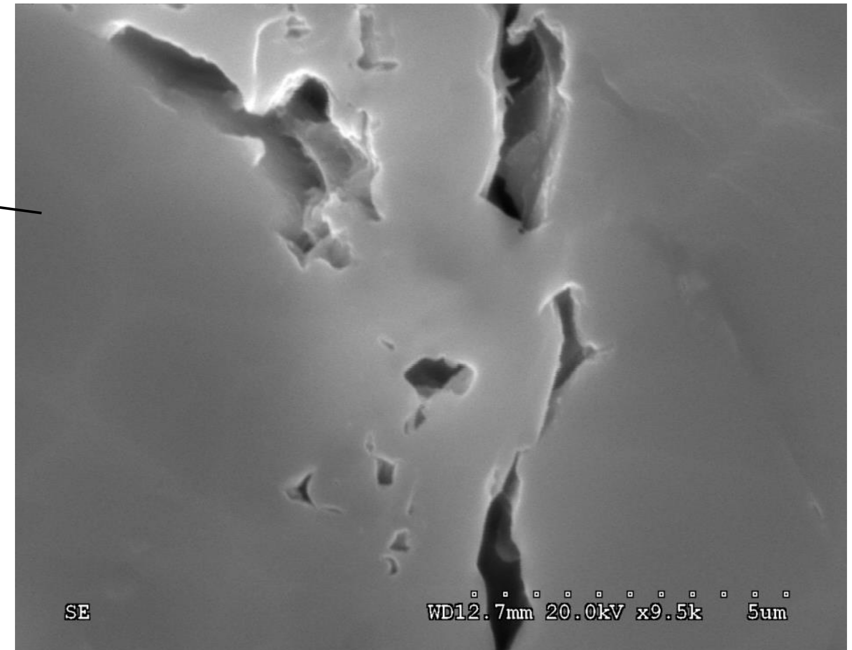




Shale sample after mechanical polishing with the TXP (left) and after additional ion polishing

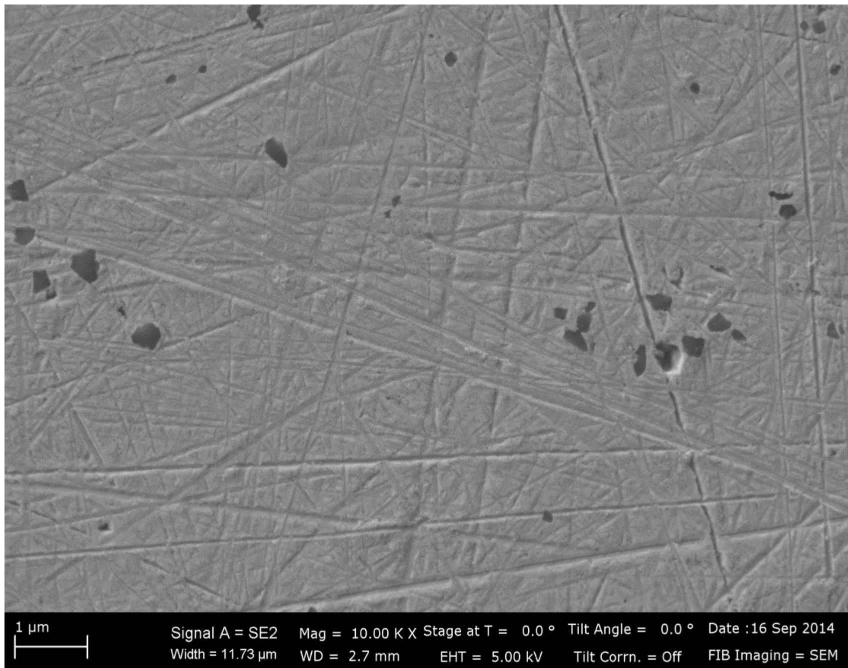
Application Rotary Stage: Shale

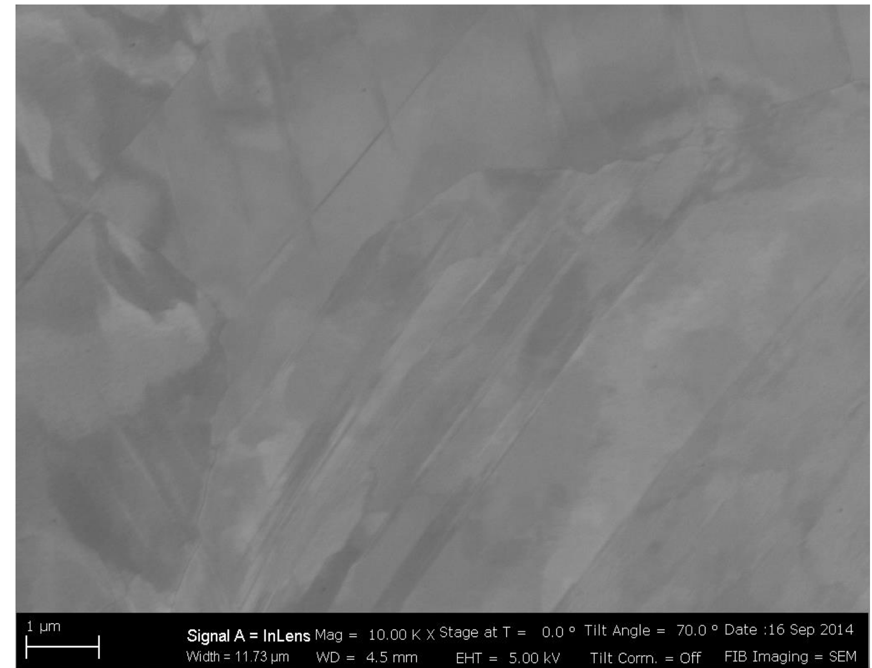




Shale sample after mechanical polishing and additional ion polishing

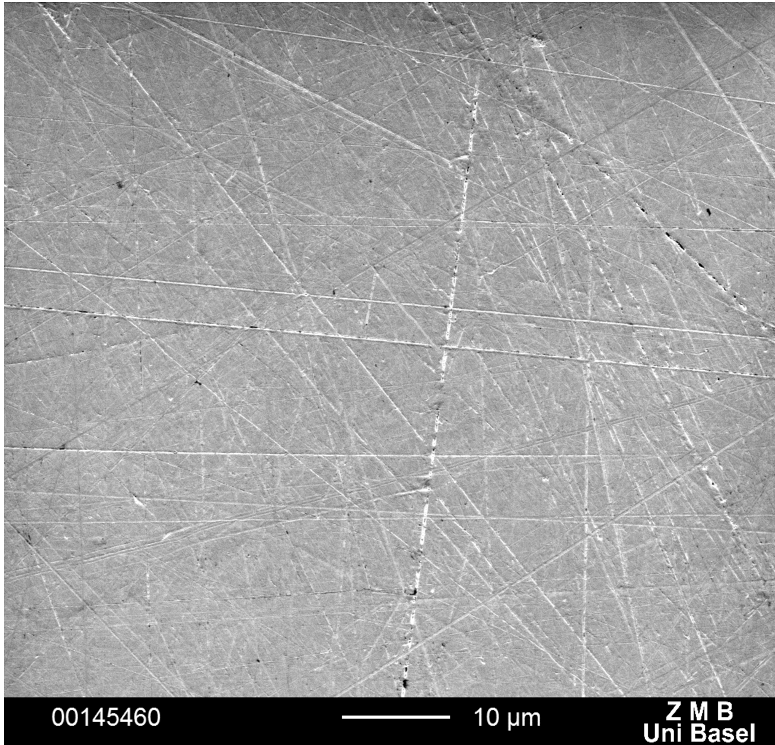
Application Rotary Stage: Silver

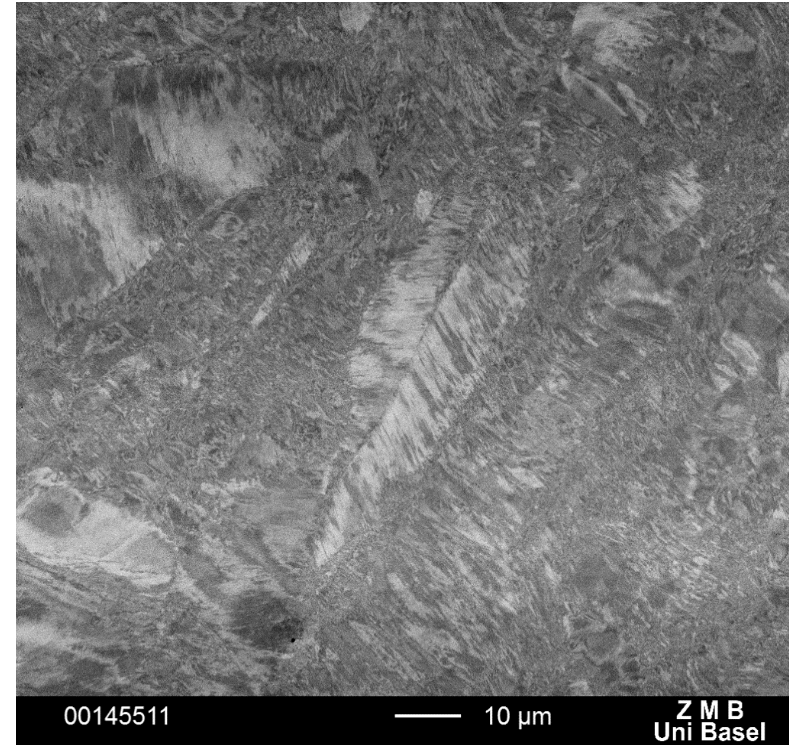




Silver sample after mechanical polishing (left) and after additional ion polishing

Application Rotary Stage: Gold

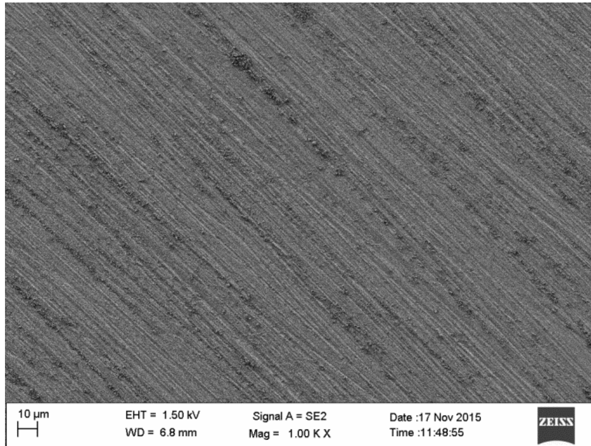


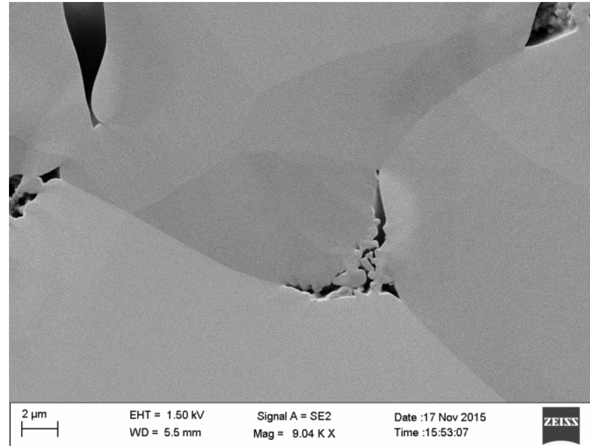


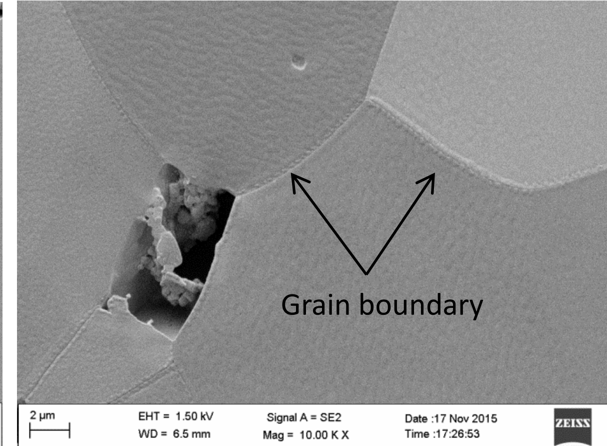
Gold sample after mechanical polishing (left)
and after additional ion polishing

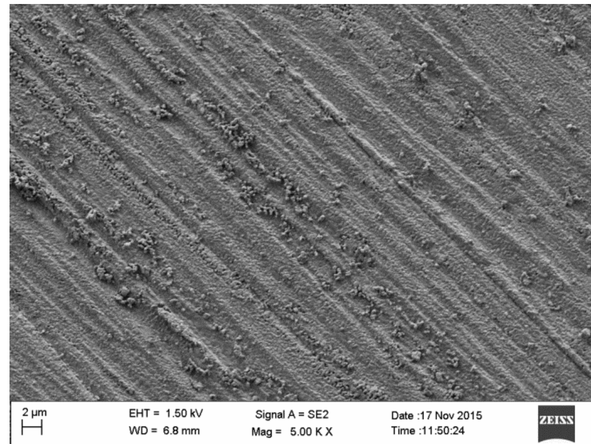
MICROSYSTEMS

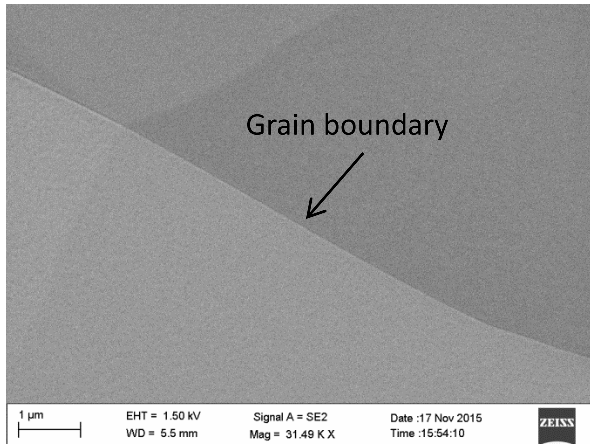
Applications rotary stage

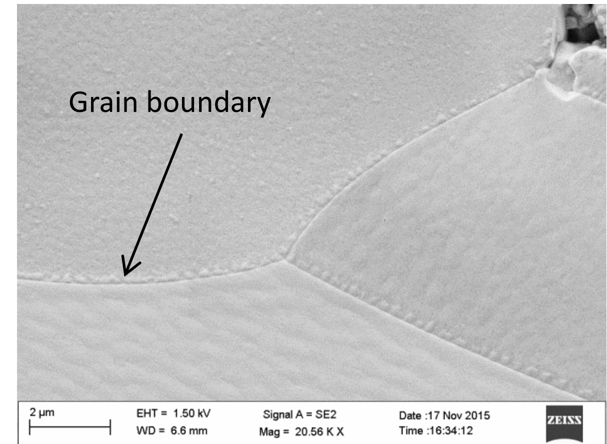












Synthetic rock salt mechanical polished (left), after ion polishing (middle) and after contrast enhancement (right)
(Images: Joyce Schmatz, RWTH Aachen)

Leica MICROSYSTEMS

Leica EM TIC 3X advantages:

- Large area cross sections (>4x1mm)
- High throughput design (multiple sample stage)
- Cooling Stage (temperature range 30° to -150° C)
- Total system solution (EM TXP / EM TIC 3X package)
- Large area surface preparation (Ø up to 25 mm):
  - Cleaning
  - Polishing
  - Contrast enhancement

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