

**Leica**  
MICROSYSTEMS



## LEICA EM TIC 3X

Triple Ion-Beam Milling System

High Quality Surface Finishing For Almost Any Material

# Leica EM TIC 3X

## Triple Ion Beam milling system

Would you like to prepare surfaces of hard, soft, porous, heat-sensitive, brittle and /or heterogeneour materials for microstructure analysis in scanning electron microscopy (SEM) and investigations in the Atomic Force Microscope (AFM)? The unique broad ion beam milling system of the Leica EM TIC 3X is the systems of choice for EDS, WDS, Auger and EBSD, because ion beam milling is often found to be the only method capable of achieving high quality cross-sections and planed surfaces of almost any material. The process reveals the internal structures of a sample whilst minimizing deformation or damage.

With its three ion beams, rotary stage, cooling stage, and multiple sample stage the Leica EM TIC 3X can mill at high rates, cut broad and deep into the sample, and create smooth surfaces resulting in quality cross-sections for almost any material, quickly and easily. Connectivity with the Leica EM VCT environmental transfer system provides perfect cryogenic surfacing of biological, geological, or industrial samples subsequently transferred under cryo and vacuum conditions to coater and (cryo) SEM.

### Leica EM TIC 3X – INNOVATIVE FEATURES IN DESIGN AND OPERATION

#### High throughput, cost-efficient

- › Cuts high-quality cross-sections with large areas of  $>4 \times 1$  mm
- › Multiple sample stage capable of processing up to three samples in one run
- › Samples up to a size of  $50 \times 50 \times 10$  mm or up to 38 mm diameter can be inserted for processing
- › Various sample holder sizes can be used

#### › Total workflow solution saves user interaction

- Ease of use, high process accuracy**
- › Easy and accurate sample mounting and alignment
- › Simple operation via touch screen, no special skills necessary
- › Process monitoring via stereomicroscope or HD-TV camera
- › LED illumination for optimal specimen viewing and alignment

#### › Integrated, decoupled roughing pump provides vibration-free observation

- › Contrast enhancement at  $90^\circ$  to the prepared surface
- › Parameter and program upload and download on USB stick

#### Usable for almost any material

- › Cooling stage provides temperatures of mask and sample down to  $-160^\circ\text{C}$



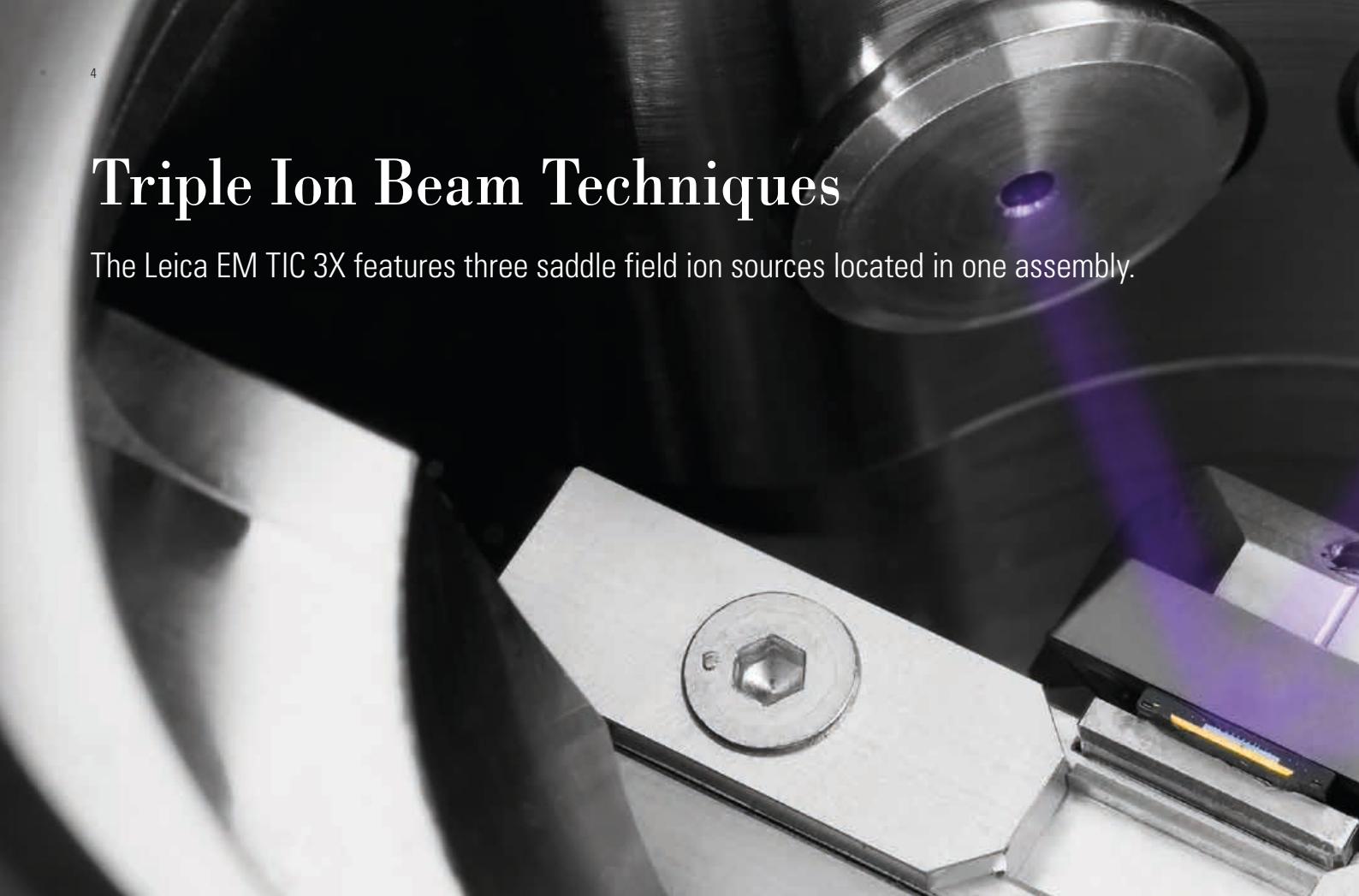
## ERGONOMIC WORKPLACE

A specially designed instrument table is offered, featuring numerous additional benefits such as:

- › Passive damping elements reduces vibrations from the environment
- › LN<sub>2</sub> pump storage
- › Clamping device for the Argon gas bottle for safe installation
- › Elbow rests allow an ergonomic posture during sample handling
- › A specially designed chair completes the ergonomic concept of this exceptional instrument table

# Triple Ion Beam Techniques

The Leica EM TIC 3X features three saddle field ion sources located in one assembly.



## ARRANGEMENT

The assembly is arranged perpendicular to the sample surface, so the sample (mounted on a holder) does not require an oscillating movement to reduce shadowing /curtaining effects. Also, it enables an efficient heat transfer from the sample when doing cross-sectioning.

## CROSS SECTIONING

The three ion beams intersect at the center edge of the mask, forming a milling sector of 100° cutting the exposed sample (~20 to 100 µm above the mask) until the area of interest is reached. The design of the ion guns develops a milling rate of 150 µm/hour (Si 10 kV, 3.5 mA, 50 µm from edge) which is one of the highest value considering the removed volume of the material.

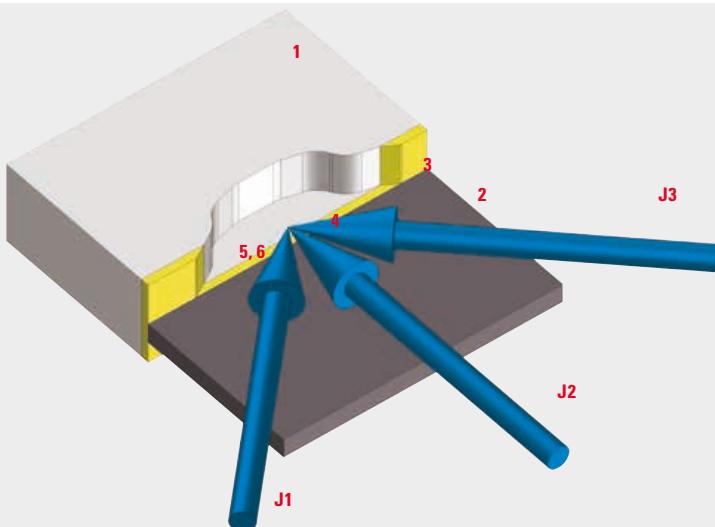
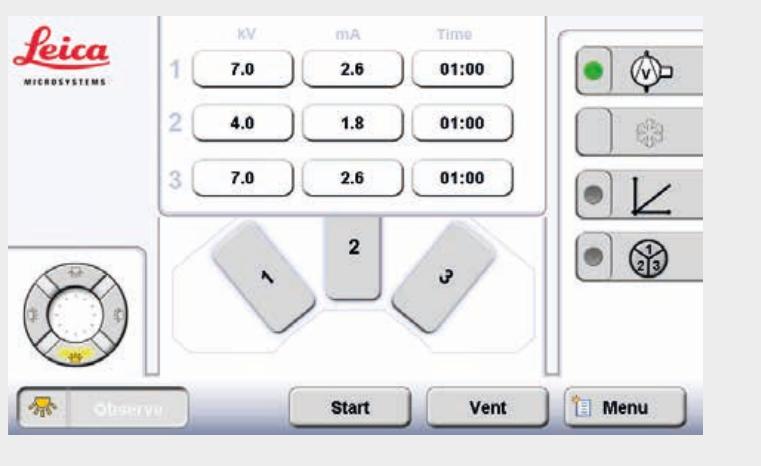
This unique technique produces a vast cross-sectioning area of >4 × 1 mm at a very high material removal rate with a high quality finish.

For flat milling the rotary stage is used. Due to the gun assembly and additional lateral movement of the sample an area larger than 25 mm diameter can be prepared.

## USAGE

For processing, each gun can be controlled and switched separately. This enables users to set different gun parameters depending on the application, e.g. for contrast enhancement or gentle milling.

Due to the gun design, no time consuming gun alignment has to be done and shortens the down time and maintenance costs to a minimum.



- |            |                               |
|------------|-------------------------------|
| 1          | Sample                        |
| 2          | Mask                          |
| 3          | Sample surface                |
| 4          | Cross over point of ion beams |
| 5          | Area of interest              |
| 6          | Direction of observation      |
| J1, J2, J3 | Ion beams                     |

# Configure Your System

Our customers seek faster and simpler methods of sample preparation without having to forgo quality. The innovative technology of the Leica EM TIC 3X triple ion beam milling system offers solutions to help laboratories with high expectations achieve their goals. Depending on the preparation needs, the Leica EM TIC 3X can be configured for applications of standard preparation, high throughput processing, and the preparation of extremely heat sensitive samples such as polymers or rubbers at low temperatures. Samples can be transferred to the (cryo) SEM under cryo-vacuum condition.

Five easily interchangeable stages are offered to fulfill individual application demands:

- STANDARD STAGE
- MULTIPLE SAMPLE STAGE
- COOLING STAGE
- ROTARY STAGE
- VCT DOCKING STATION

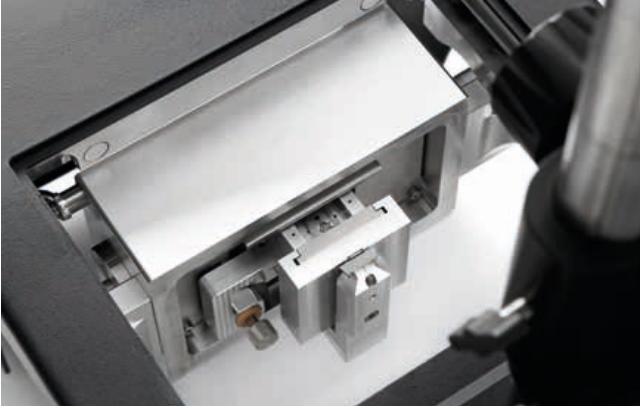


Various sample holders for almost every sample size and a wide range of use are available e.g. one sample holder for the complete process from mechanical pre-preparation (Leica EM TXP) to ion beam milling process (Leica EM TIC 3X) to SEM investigation to the point of storage.

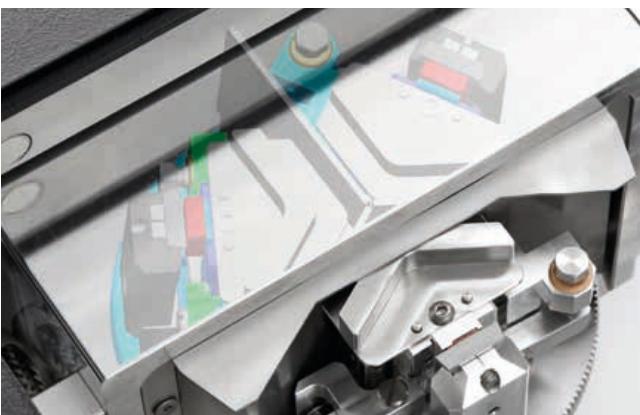


Various sample holders for samples up to 12 mm height as well as adaptors for commercial available SEM stubs are available for the rotary stage.

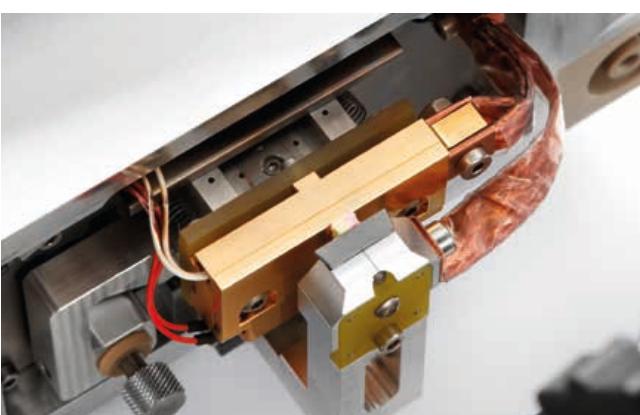
**Standard stage** for routine applications and contrast enhancement of the prepared surface.



**Multiple sample stage** is used if high throughput is desired. Three samples can be loaded and automatically processed in one session (e.g. overnight) without any user interaction.



**Cooling stage** provides very low temperature processing. With temperatures of the sample holder and mask down to  $-160^{\circ}\text{C}$ , extremely heat-sensitive samples such as rubber, water-soluble polymer fibers or even marshmallows (if desired) can be processed to a high quality. The 25 liter dewar provides at least enough  $\text{LN}_2$  for a full working day without refilling. Warming up of the sample is automatically carried out at the selected temperature under low vacuum, thus avoiding moisture contamination.



Using the **VCT docking configuration** the sample can be transferred from the Leica EM TIC 3X into the (cryo) SEM under optimal conditions. It is perfect for surfacing environmentally sensitive samples which can be subsequently transferred to coating and /or SEM systems under inert gas / vacuum conditions. Due to the cooling possibility of the VCT docking stage cryogenic sample preparation and transfer offers investigation possibilities which were unknown until now.



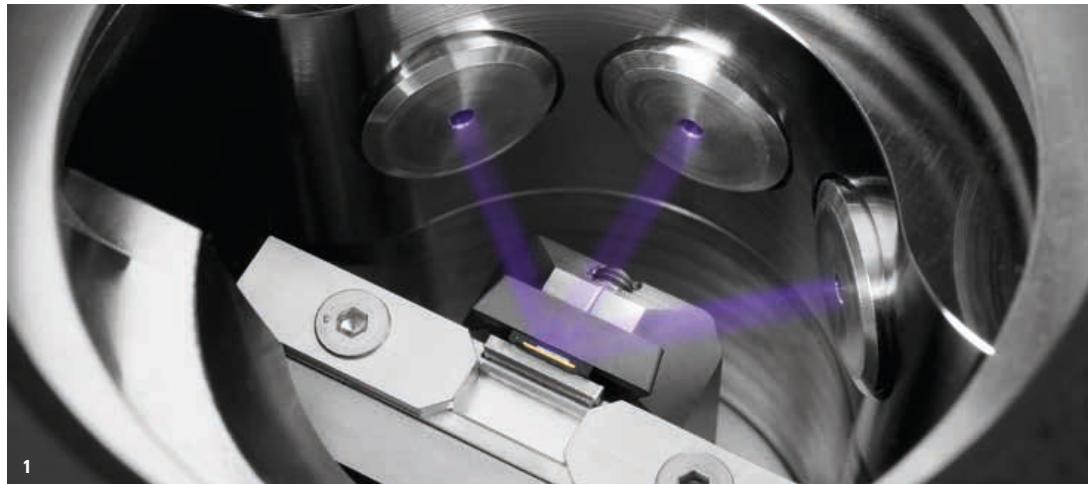
**Rotary stage** is used for ion beam flat milling of already mechanically polished surfaces. During ion beam processing the sample can be rotated or oscillated. The additional lateral movement of the sample provides for a prepared area of more than  $\varnothing 25\text{ mm}$ . Thus mechanical artifacts e.g. smearing and fine scratches are removed. It reveals the structure of the sample as native as possible.



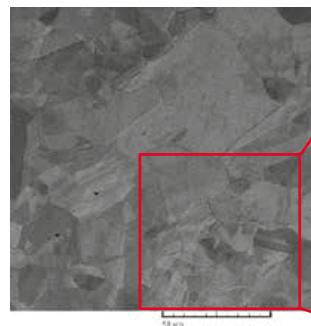
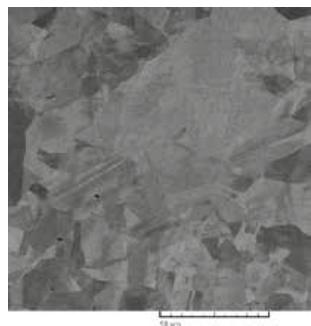
# Contrast Enhancement

In addition to cross-sectioning, the same holder can be used for cleaning and contrast enhancement to provide clear visualization of the surface topography (e.g. grain boundaries).

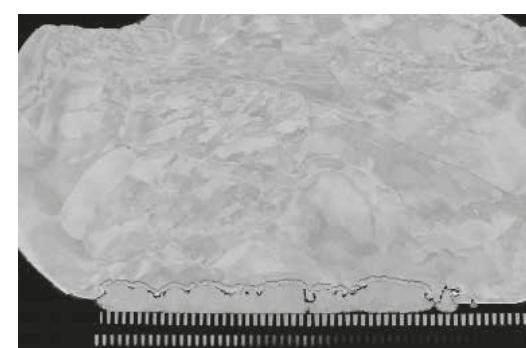
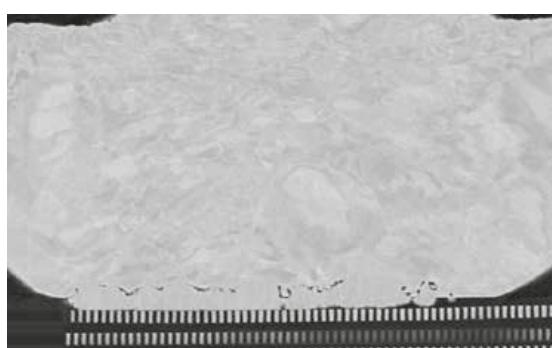
Contrast enhancement  
holder of the standard  
stage  
1) sample in cross  
sectioning position;  
2) contrast enhance-  
ment at low kV for few  
minutes



Copper sample  
Left: after ion beam  
cross sectioning;  
middle: after addi-  
tional contrast en-  
hancement step;  
right: enlarged view



Gold-wire bond  
Left: after ion beam  
cross sectioning;  
right: after addi-  
tional contrast en-  
hancement step



## Ergonomic and Easy to Use

The stereo microscope of the Leica EM TIC 3X is not only used for aligning and process observation. A work-plate attached beneath the microscope can be used for handling the sample or attaching the sample to its holder. Thus, no additional observation system is needed for manipulating small samples.

The outstanding performance of the integrated touch screen controller is not only reflected in its intuitive operation but hints and helpful information are also displayed to allow the most effective use of the instrument. A USB stick can be attached to upload and download parameters for reporting and processing.

## High Process Accuracy

Nowadays, smaller and smaller details need to be revealed within the sample. Achieving a cross-section through, for example, small TSVia holes, has become easier than ever before. All stages are designed to adjust the sample within  $\pm 2 \mu\text{m}$ . Not only does the stage accuracy provide this fine target adjustment, but also is an observation system offered to resolve sample details of around  $3 \mu\text{m}$  which is needed for such accurate alignment. To help orientate the sample, the segment-controlled ring LED or LED coaxial illumination is an additional feature to provide a clear image via the stereo microscope or HD-TV camera.

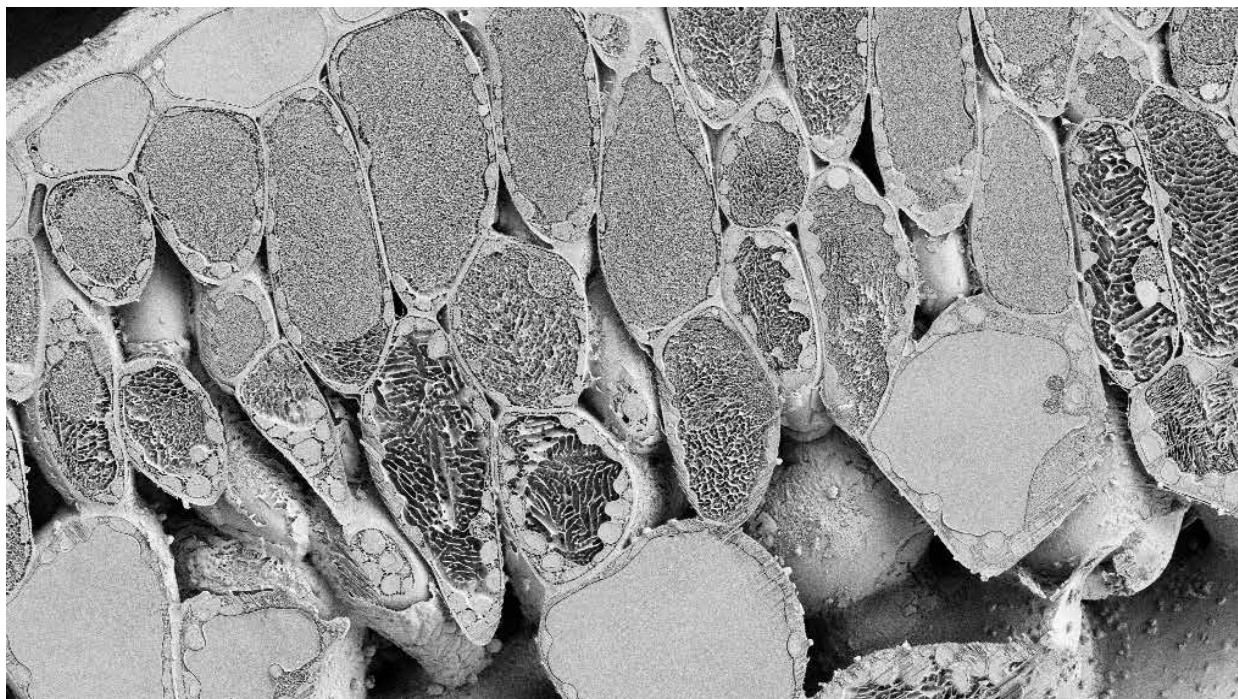
As the roughing pump is integrated into the instrument, a separate space in the laboratory is no longer required. Due to the decoupled design, sample observation during the process is not influenced by annoying vibrations caused by the roughing pump.



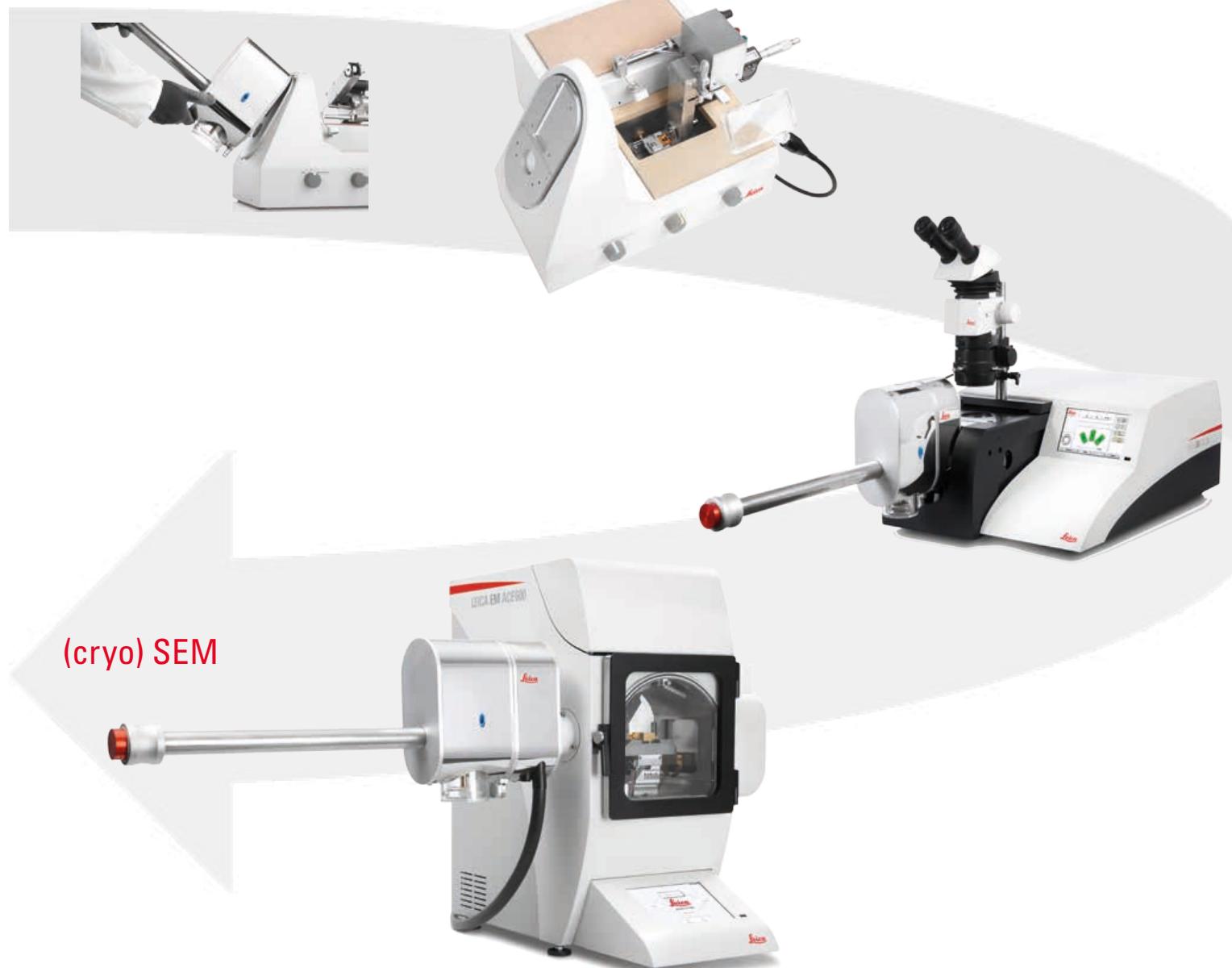
# Preparation workflow under cryogenic conditions

Samples which need to be prepared and transferred under cryo conditions also have to be pre-prepared under cryogenic conditions. A special VCT loading station with cryo-saw has been developed to fulfill these conditions. The sample is inserted in the VCT sample holder and mechanically pre-prepared using a diamond disc cutter in LN<sub>2</sub>.

Users can transfer the pre-prepared sample to the Leica EM TIC 3X – VCT for further broad ion beam processing with the Leica EM VCT. Thus prepared, the sample can be subsequently transferred to the coating system EM ACE600 and / or to the cryo SEM for investigation.



Taxus, cross sectioned at -120°C, investigated in a cryo SEM



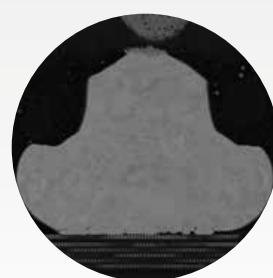
(cryo) SEM

# Create synergies with the Leica EM TXP

Prior to use of the Leica EM TIC 3X, mechanical preparation is often required to get as close as possible to the area of interest. The Leica EM TXP is a unique target surfacing system developed for cutting and polishing samples prior to follow-on techniques with instruments such as the Leica EM TIC 3X. The Leica EM TXP is specially designed to pre-prepare samples by sawing, milling, grinding, and polishing. It excels with challenging specimens where pinpointing and preparing difficult targets becomes easy.

In addition, the Leica EM TXP can be used to pre-prepare environmentally sensitive samples. These pre-prepared samples can be subsequently transferred with the Leica EM VCT shuttle to the ion beam milling system Leica EM TIC 3X with VCT port without changing the sample holder. After ion beam surfacing, the sample can be subsequently transferred to the follow-on procedures / techniques such as coating and /or SEM investigation without exposing the sample to environmental changes.

ADVANTAGES	SPECIAL EDGED MASK	SPECIAL HOLDER
Using a dedicated sample holder, the sample can be left in situ on the holder from mechanical pre-preparation with Leica EM TXP through ion beam milling with the Leica EM TIC 3X to SEM examination.	A special edged mask (optional) has been designed to intensify this synergy. Now the Leica EM TXP can be used to remove "redundant" material within minutes, just by beveling the sample. By doing so, the material around the area of interest can be easily removed without changing the specimen holder. Thus, not only the ion-beam processing time is drastically reduced, which could save many hours of lead-time, but also is the gun's lifetime per sample significantly increased as well.	Additionally, a special holder for the Leica EM TXP is offered. This holder is used to clamp commercially available SEM stubs. Hence, the sample can be mechanically polished to be suitable for an additional ion beam polishing process using the rotary stage without removing the sample from its SEM stub.



**Special edged mask:** Around 300 µm of "useless" material above the wire bonding was removed with the Leica EM TXP. In conjunction with the specially designed edged mask the sample was completely prepared within 1 hour (including pre-preparation).



(cryo) SEM

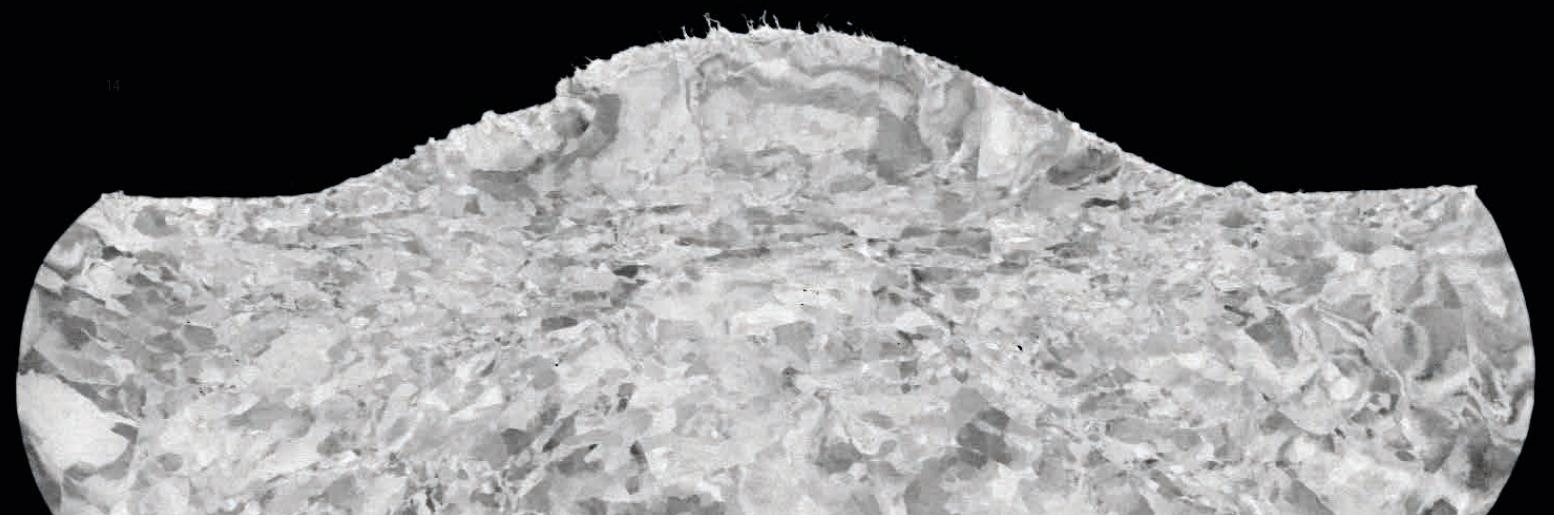


#### Synergies with the Leica EM TXP

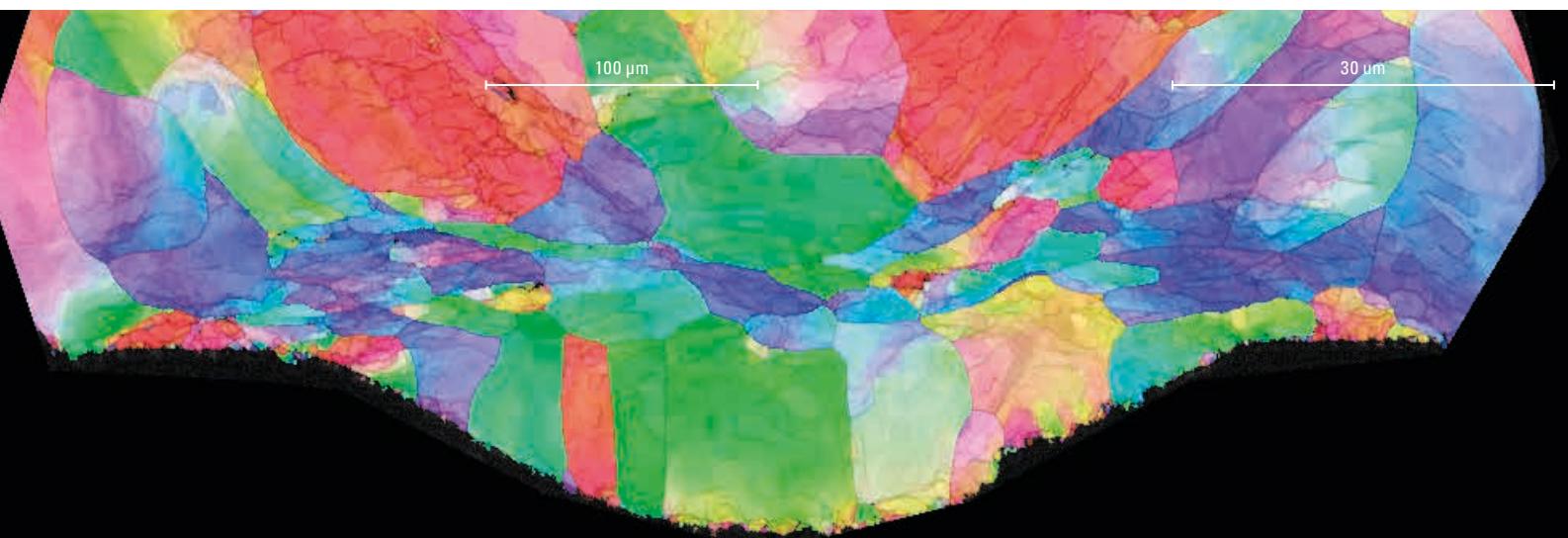
Left: Leica EM TXP

Right: Leica EM TIC 3X

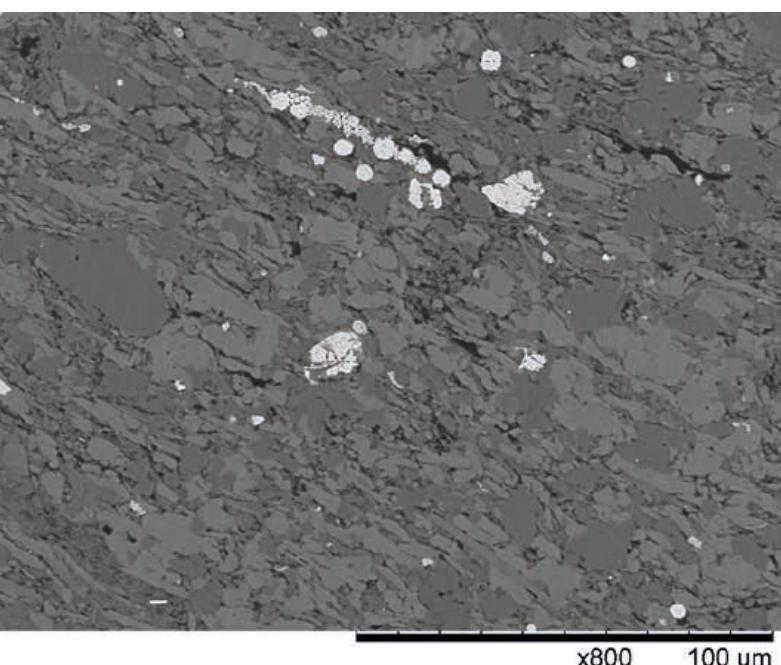




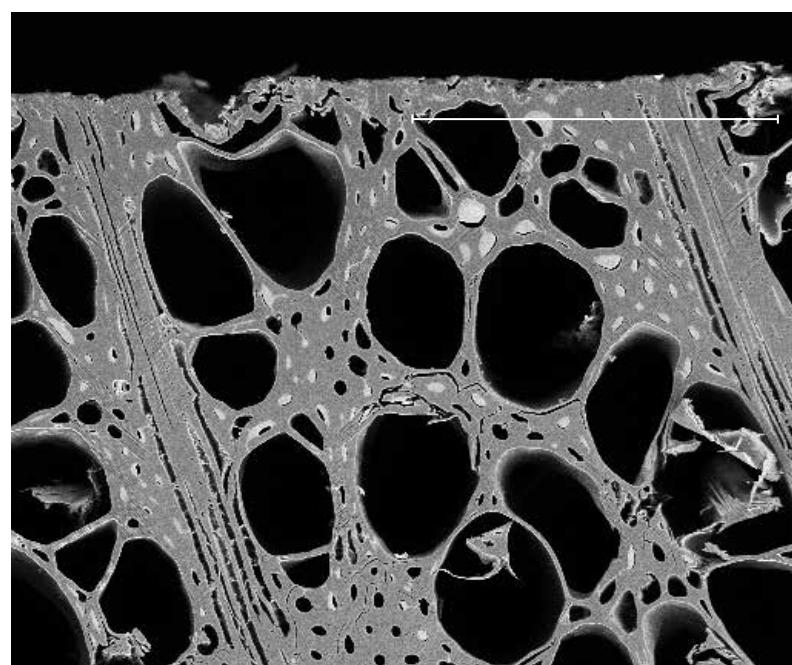
Cross section of a gold wire bond; mechanically pre-prepared with the Leica EM TXP. The final surface preparation was carried out with the Leica EM TIC 3X



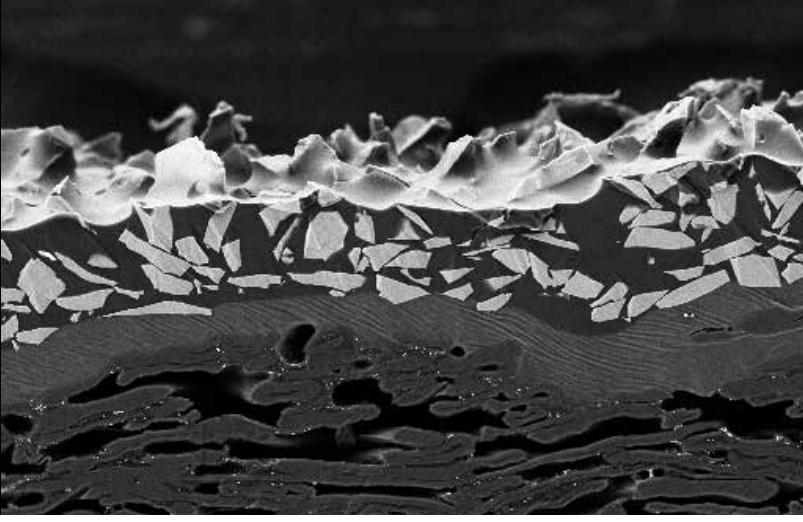
The EBSD result shows a perfect cross section of the target, without introducing damage. Diffraction pattern are obtained from the Si, W, Al Si and Au layers. Very fine deformation structures in the Au layer (of less than 40 nm) are revealed by the EBSD analysis. Courtesy of: Bruker Nano GmbH



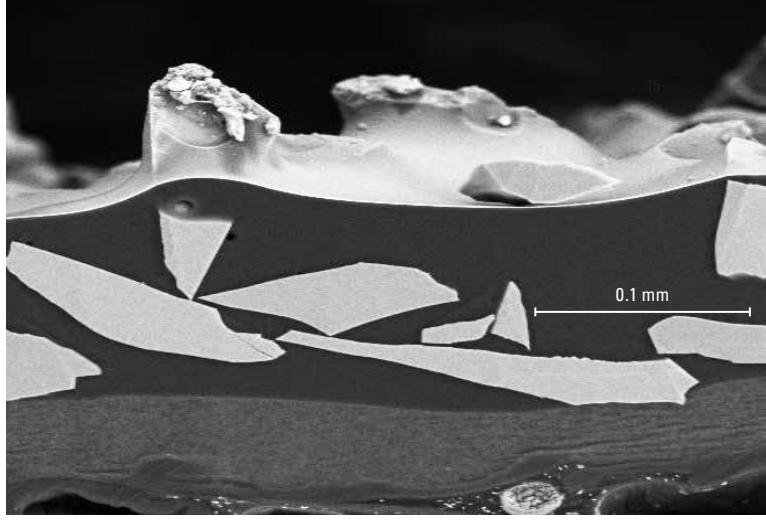
Oil shale (nano pores), revealed with the Leica EM TIC3X (rotary stage) total sample size Ø 25 mm



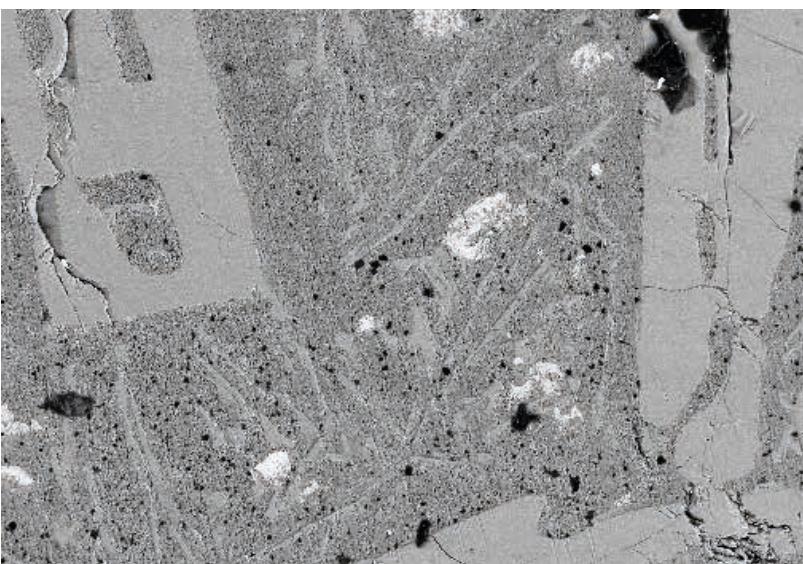
Cross section of veneer



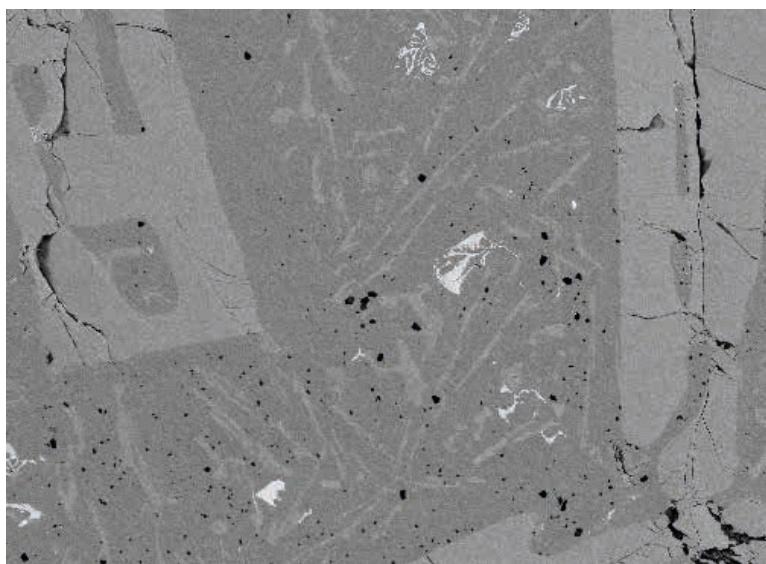
Cross section of SiC abrasive paper



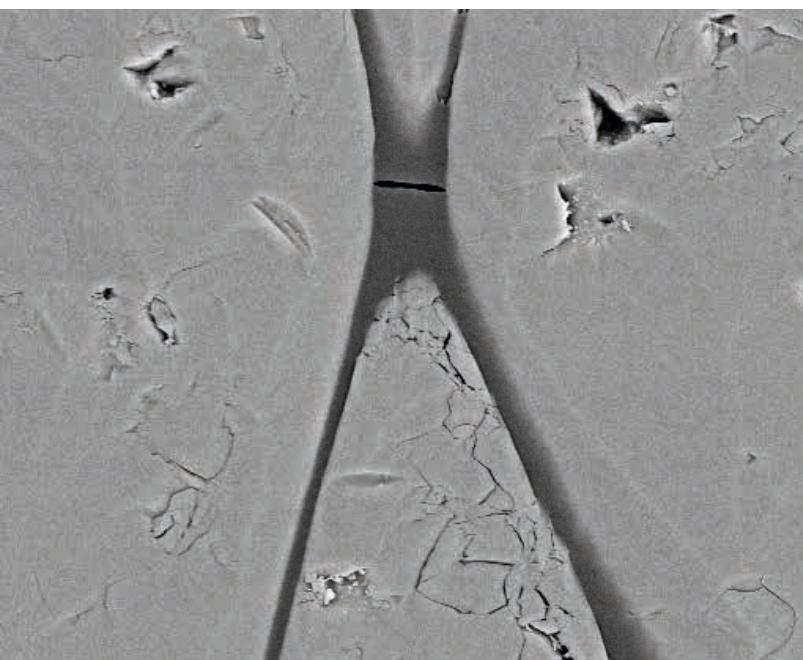
Cross section of SiC abrasive paper



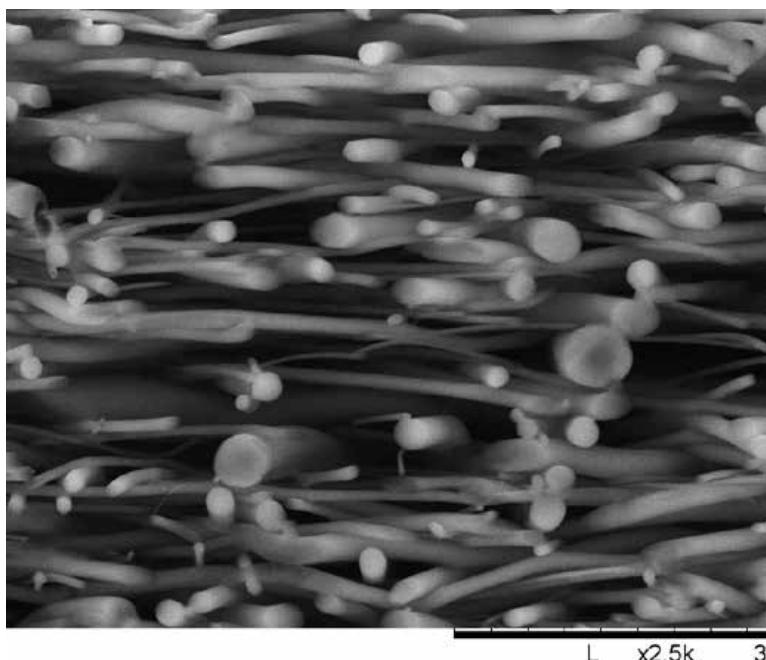
Friction Stir processes Al-30Si Alloy after mechanical polishing ...



... and after additional ion polishing with the Leica EM TIC 3X (Rotary Stage)



Calcium Carbonte (Calcite and Aragonite) with organic membranes between the carbonate, Ion beam polished with the Leica EM TIC 3X (rotary stage)



Coaxial polymer fiber (water soluble) prepared at -120 °C

L x2.5k 3



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Leica Mikrosysteme GmbH | Vienna, Austria  
T +43 1 488 99-0 | F +43 1 488 99-350

[www.leica-microsystems.com](http://www.leica-microsystems.com)

