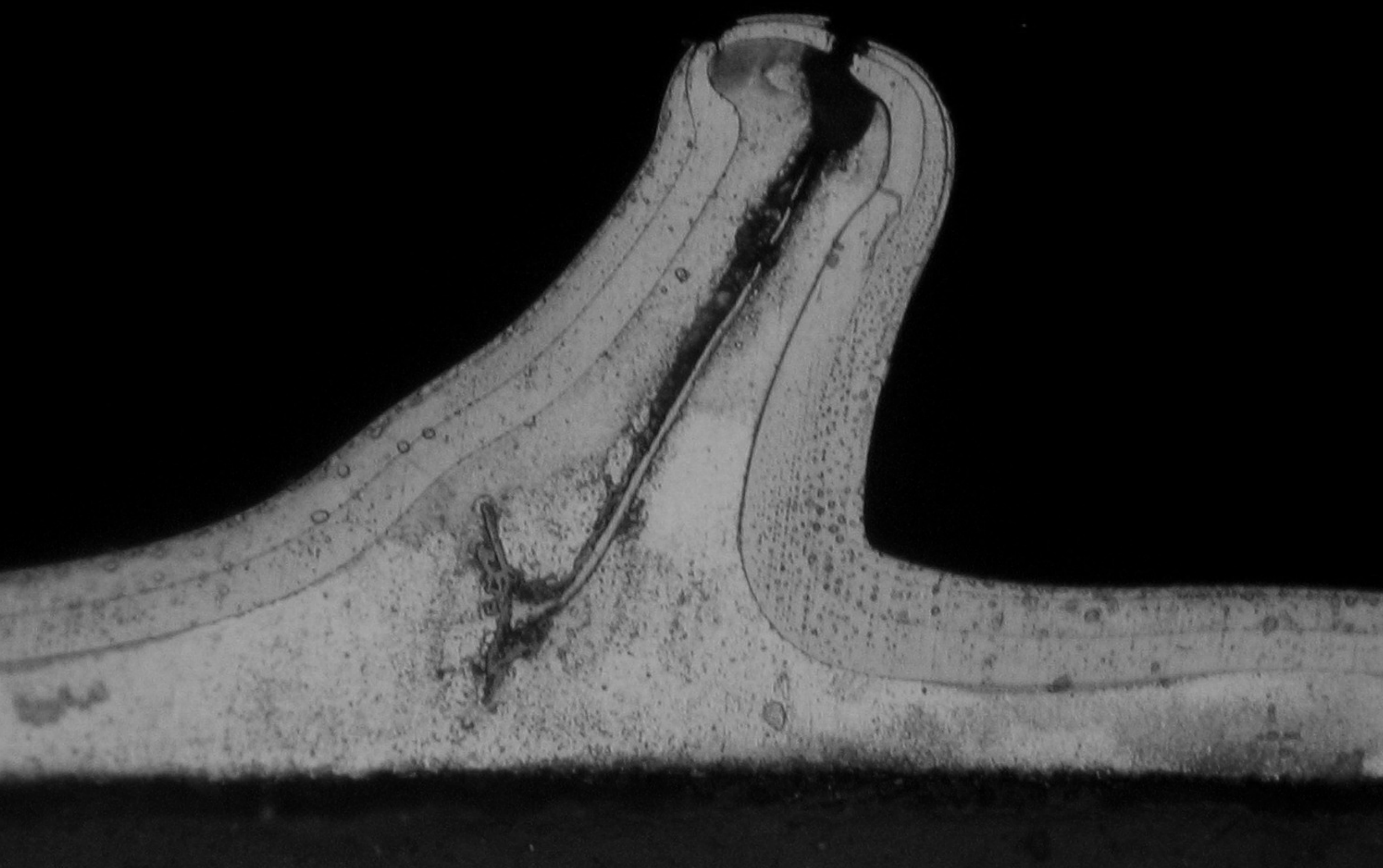
From Eye to Insight

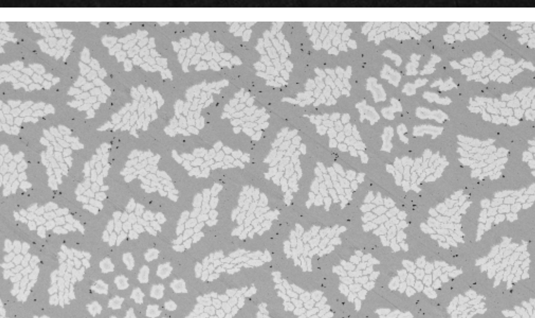
Leica MICROSYSTEMS

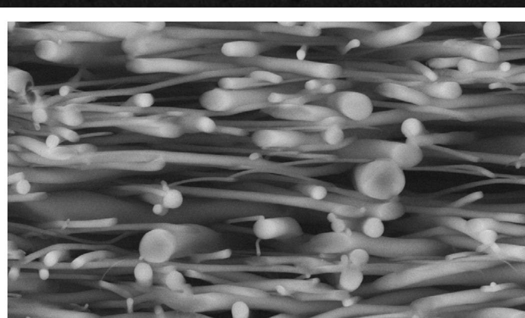
Sample Preparation at Leica Microsystems

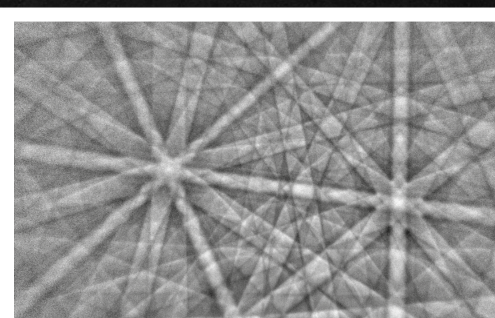
WORKFLOW SOLUTIONS FOR MATERIAL SCIENCE RESEARCH

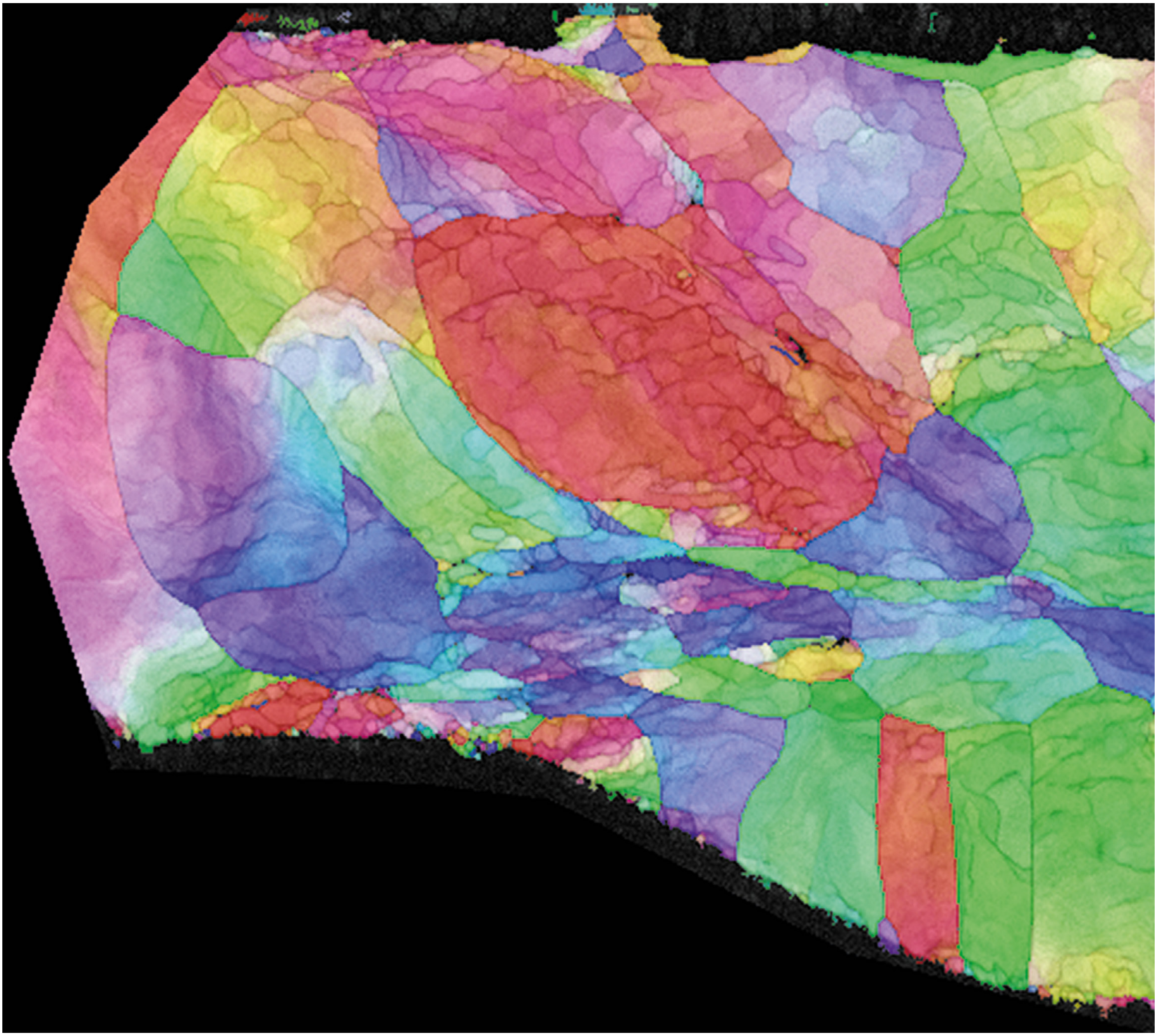


We focus on workflow solutions to provide a product range serving your needs in TEM, SEM, LM, and AFM investigations.





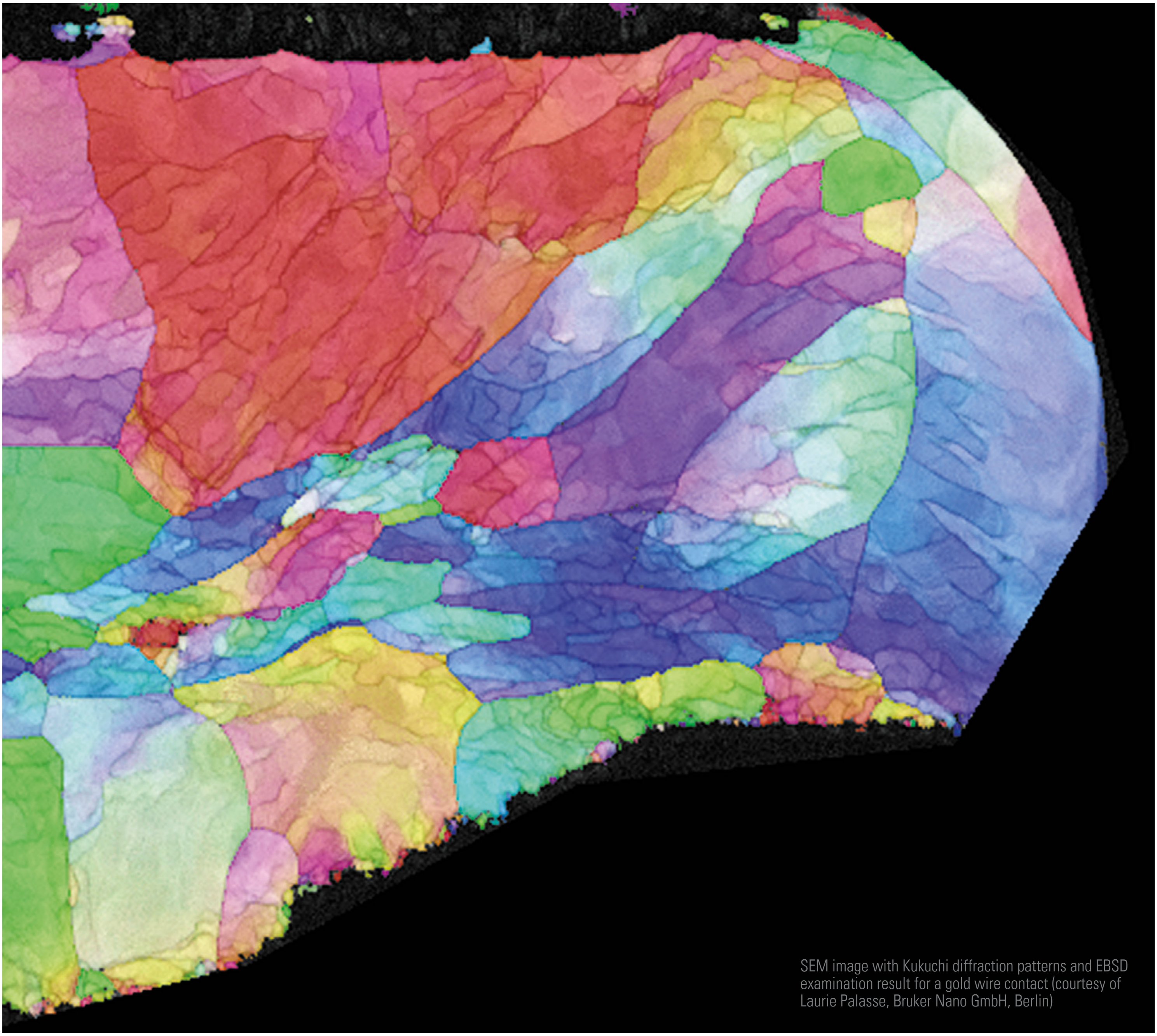




Sample Preparation Workflows for Material Science research

Sample Preparation is critical to obtaining the best possible results when investigating materials via TEM, SEM, LM, or AFM. For TEM observation the sample needs to be extremely thin, while analysis with LM, SEM and AFM requires the sample to have a smooth surface. To achieve maximum accuracy in investigation and analysis, it is crucial to minimize the artifacts introduced in the preparation process.

This brochure presents Leica's solutions to the most frequently used sample preparation workflows. Our products will help you achieve the results you need for your TEM, LM, SEM, and AFM analysis. If you have special requirements or questions, our Leica experts will be happy to assist you at any time.



SEM image with Kukuchi diffraction patterns and EBSD examination result for a gold wire contact (courtesy of Laurie Palasse, Bruker Nano GmbH, Berlin)

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Note: all images in this brochure, with no explicitly mentioned courtesy are courtesy of Leica Microsystems.

LEICA MICROSYSTEMS FOR MATERIAL SCIENCES WORKFLOWS – PORTFOLIO ALIGNED WITH YOUR NEEDS

EM TIC 3X

The Triple Ion Beam Milling System, EM TIC 3X, allows production of cross sections and planar surfaces for scanning electron microscopy (SEM), microstructure analysis (EDS, WDS, Auger, EBSD), and AFM investigations.

EM TXP

The EM TXP is a target preparation device for milling, sawing, grinding, and polishing samples prior to examination by SEM, transmission electron microscopy (TEM), and light microscopy (LM) techniques. An integrated stereomicroscope allows pinpointing and easy preparation of barely visible targets.

EM RAPID

The EM RAPID is a high speed milling system with an integrated stereomicroscope and LED ring illuminator. It is designed for trimming and surfacing of biological, pharmaceutical, and industrial samples prior to ultramicrotomy or NIR-investigations.

EM UC7

The EM UC7 is an ultramicrotome for ultrathin sectioning of biological and industrial samples for TEM, SEM, AFM, and LM examination. For sample preparation for array tomography the ARTOS 3D can be used.

EM VCT500

The versatile vacuum cryo transfer system, EM VCT500, enables the connection of different preparation techniques to various analysis devices.

DM2700 M

The DM2700 M materials microscope combines high-quality Leica optics with state-of-the-art universal white light LED illumination. With the Flexacam C5, you can document the fine sample details with precise colors and high resolution, no PC required.


Leica

WORKFLOW & EQUIPMENT IMAGE GLOSSARY

Sample Transfer

Cutting

Broad Ion Beam Thinning

LM Investigation

High Pressure Freezing

Surfacing

Staining / Contrasting

SEM Investigation

Grid Plunging

Freeze Fracture

Grinding

TEM Investigation

Critical Point Drying

Cross Sectioning

Polishing

FIB

Microtomy

Trimming

Ion Beam Milling (High End Polishing)

AFM Microscopy

Ultramicrotomy

Target Preparation

Ion Beam Milling

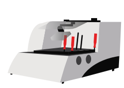
Glove Box

Slush Freezing

Coating

IR Spectroscopy


EM ACE200

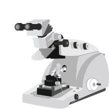

EM VCM

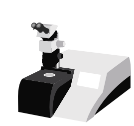

DM2700 M


EM GP2


EM ACE600


EM ICE


EM UC7

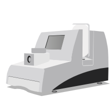

EM TIC 3X

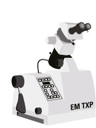

EM ACE900


EM RAPID

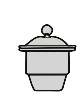

EM UC7 with EM FC7


Fume Hood

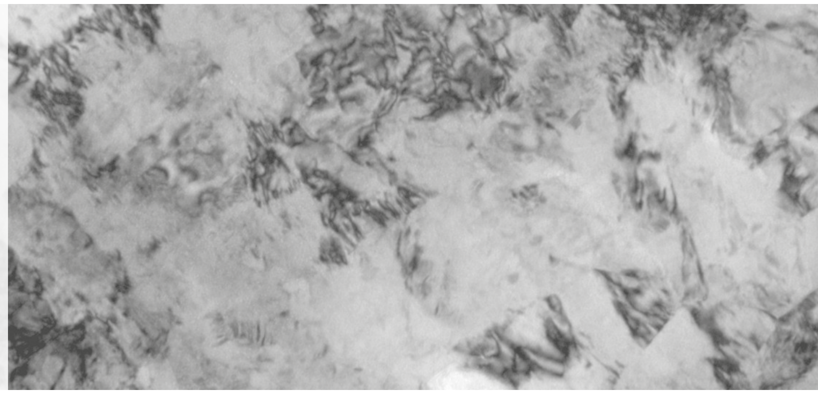

EM CPD300


EM TXP


EM VCT500

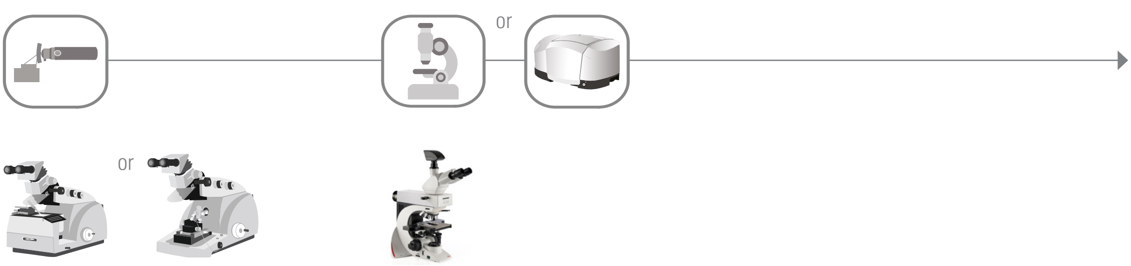

Desiccator

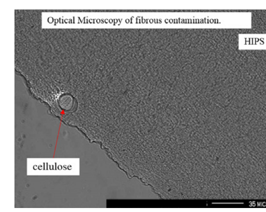
SOLID STATE SAMPLE PREPARATION



LM / FT-IR Sample Preparation - For Incident and Transmitted Light Investigations

This technique answers morphological questions and allows screening and identifying of contamination in e.g. polymers. For FT-IR micro-spectroscopy the section thickness is between 5 µm and 15 µm.

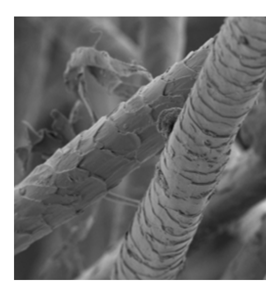




Optical microscopy of fibrous contamination of HIPS.

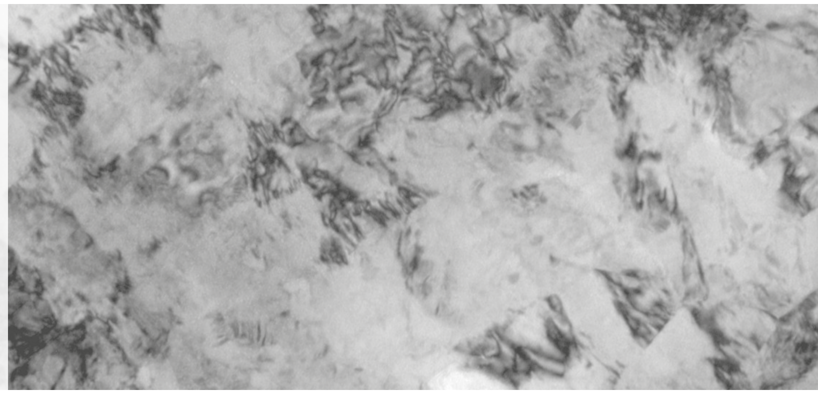
SEM Sample Observation

Coating is a necessary, routine processing step for non-conducting samples, as it can eliminate the charging effect during SEM observation. The coating method is critical for high resolution SEM because improper coating may induce several pseudo structures or phases.



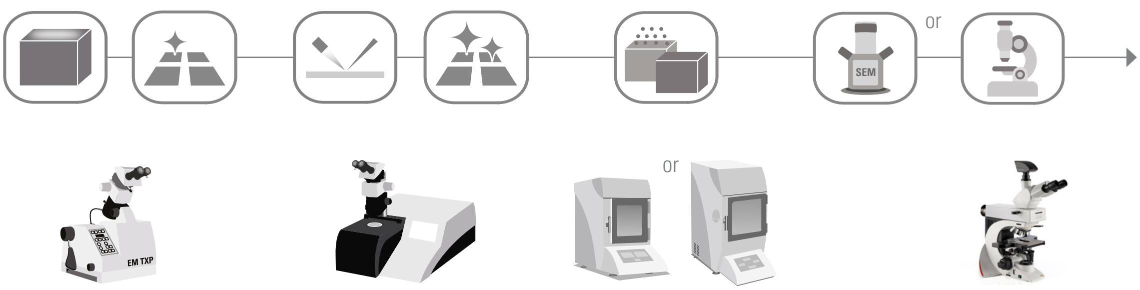
Wool 25 nm Tungsten coated with the EM ACE200. Courtesy of DI Dr. Evelin Fisslthaler, ZFE Graz, ACR, FELMI, Graz University of Technology

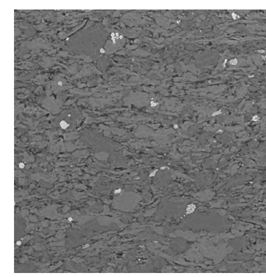
SOLID STATE SAMPLE PREPARATION



SEM / LM Solid State Preparation - Broad Ion Beam Polishing

This technique is used for large solid-state SEM or LM samples. When a large area (several cm^2) has to be investigated at a highest quality level (e.g. EBSD) the sample is mechanically pre-prepared (mirror-like finish) and finally prepared with the ion beam polishing method.

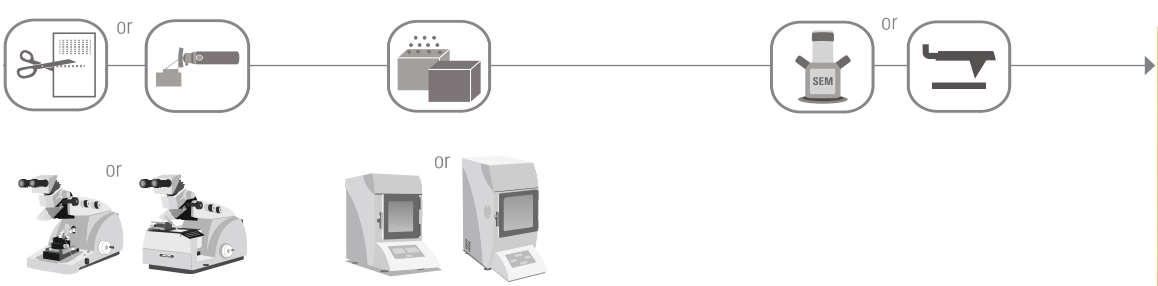


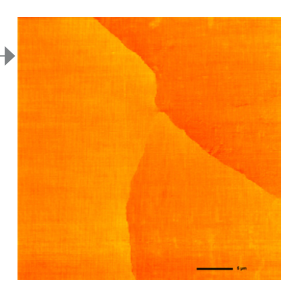


Oil shale, diameter 25 mm

SEM / AFM Solid State Preparation - Ultramicrotomy

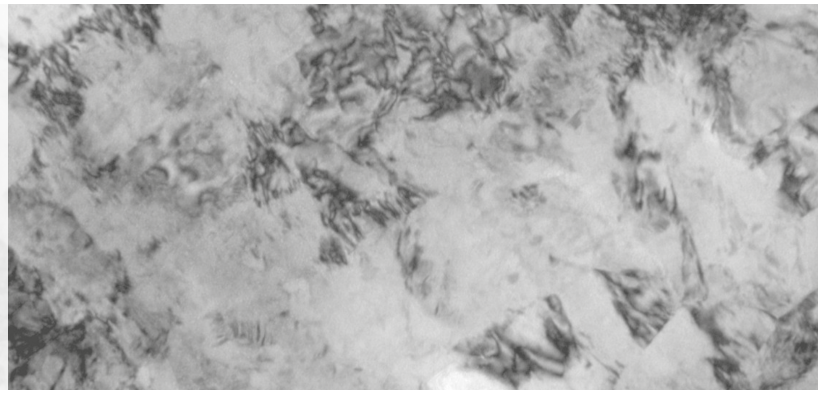
Ultramicrotomy can be used to prepare a high-quality surface on ductile metals, polymers, rubbers or paper. The ultramicrotome allows the user to set the feed for sections in a nanometer range. The smaller the feed, the higher quality the surface will be. Afterwards, for non-conductive samples, coating is necessary to make the sample suitable for SEM observation.





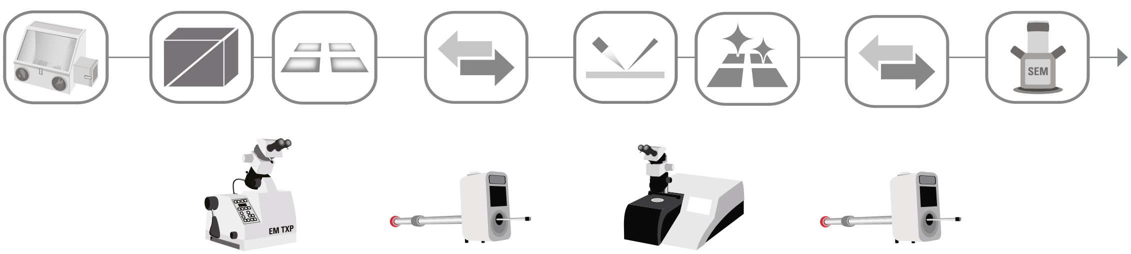
Gold grains after sectioning

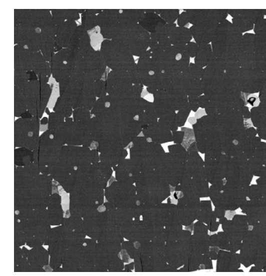
SOLID STATE SAMPLE PREPARATION



SEM of Environmentally Sensitive Materials - Broad Ion Beam Cross Sectioning

In the case of batteries and other environmentally sensitive materials, it is necessary to keep samples under vacuum or inert environment during processing. With the EM VCT500, samples can be transferred from a glovebox to the ion mill under vacuum. Afterwards they can be ion milled to prepare the desired cross section and subsequently transferred under vacuum directly to the SEM.

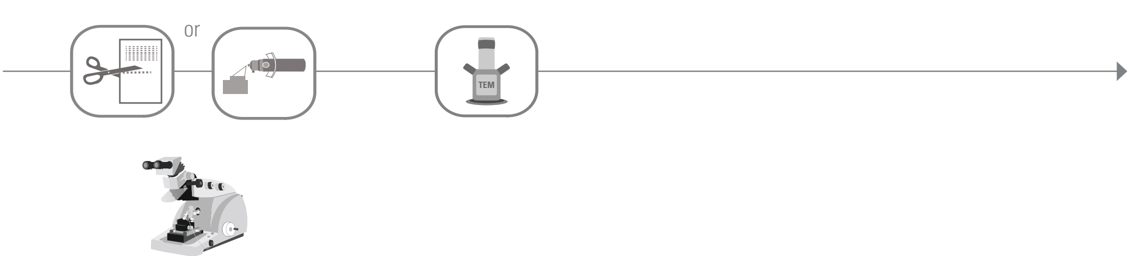


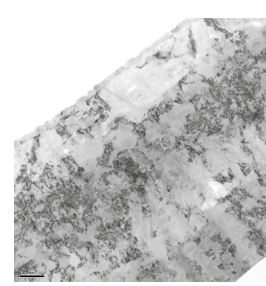


Cross section of Yttrium; Praseodym, Neodym, Promethium, Dysprosium

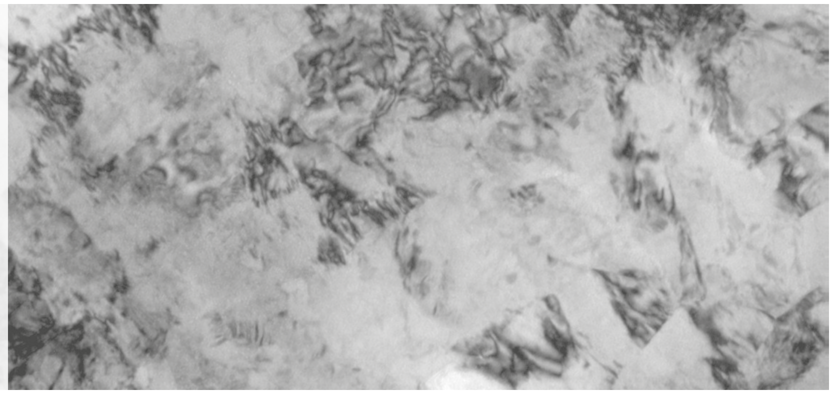
TEM Solid State Preparation - Ultramicrotomy

This workflow can be used instead of the ion beam thinning method. Many solid-state samples such as ductile material (Al, Au, Ms, Cu and their alloys) can be cut by a diamond knife. This method is a way to get nanometer thin sections for TEM within minutes. In comparison: the ion beam thinning process takes several hours and the electron transparent area measures only a few micrometers.





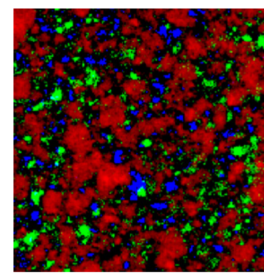
TEM image of Cu_Au layers



SOLID STATE SAMPLE PREPARATION

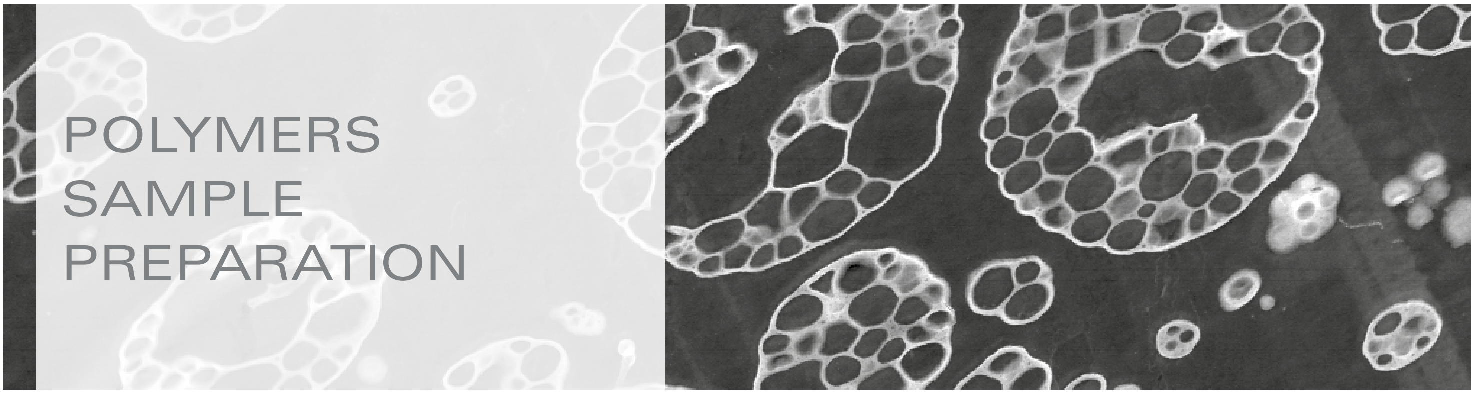
Milling of Pharmaceutical Tablets - Mechanical Milling

For certain NIR analysis applications of pharmaceutical tablets it is necessary to produce optically flat surfaces. Mechanical milling offers a very fast way to prepare these surfaces.



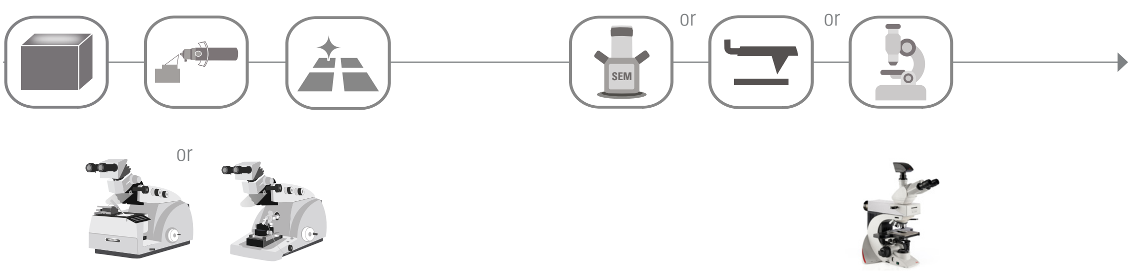
NIR picture of a surfaced tablet

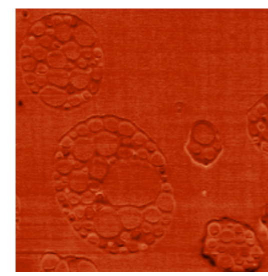
POLYMERS SAMPLE PREPARATION



LM / AFM / SEM Polymer Sample Preparation - Ultramicrotomy

This technique is used for LM, AFM and/or SEM investigation with a demand of a high degree surface quality. After trimming, the sample is sectioned in the ultramicrotome which provides for such prerequisite. The smaller the feed (nm) is set the better the surface quality of the block face will be. The prepared block face is then used in LM, AFM and/or SEM analysis.

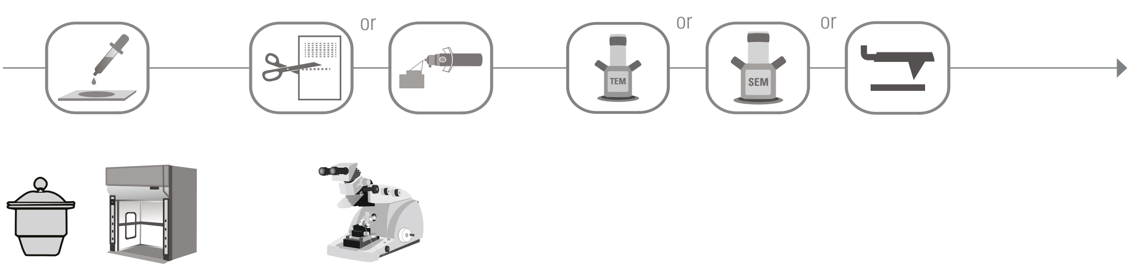


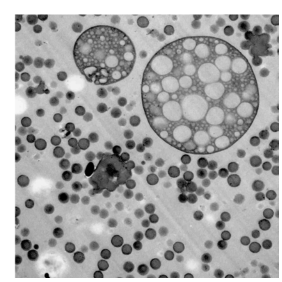


HIPS block faces section with 70 nm feed; AFM image

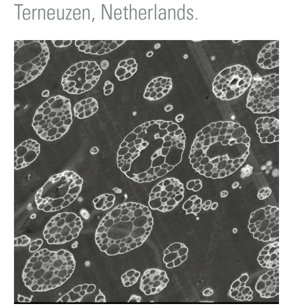
RT sectioning for Soft Polymers - Ultramicrotomy

In some cases, soft polymers can be processed at room temperature if they are hardened by chemical techniques first. Staining with OsO_4 or RuO_4 (depending on the polymer) causes the polymer to harden. The polymer can then be trimmed, sectioned/planed, and analysed.



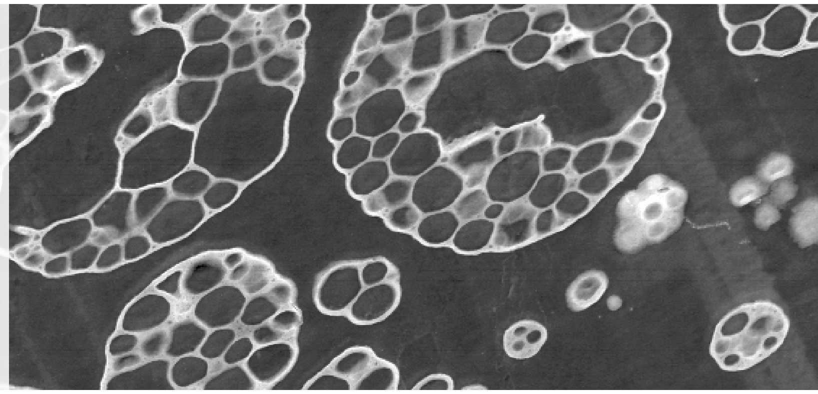


APS block stained, sectioned with an oscillating diamond knife; TEM image.Courtesy of Bob Vastenhout, Dow Chemicals, Terneuzen, Netherlands.



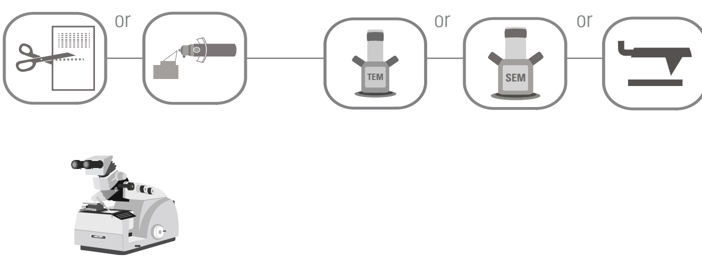
HIPS block stained HAADF TEM image

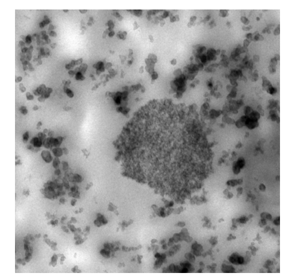
POLYMERS SAMPLE PREPARATION



Cryo sectioning for Soft Polymers - Ultramicrotomy

Soft polymers cooled down in the cryo chamber (LN_2) can become sufficiently stiff for sectioning with an ultramicrotome. Sectioned or planned samples are ready for TEM, SEM, or AFM investigation.

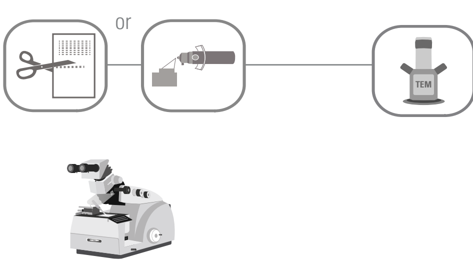




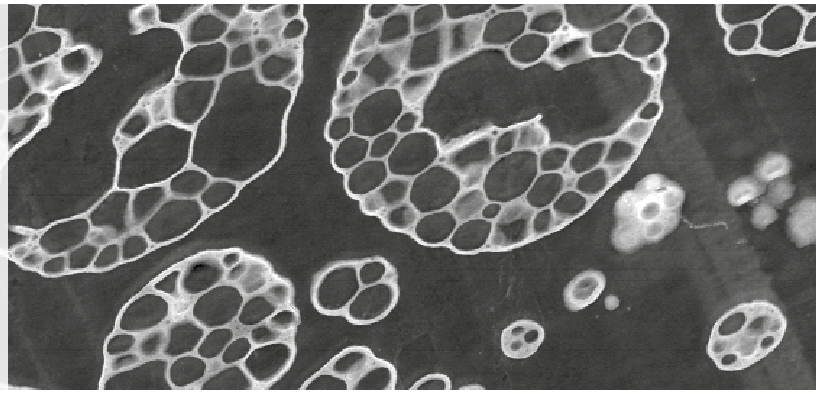
Car tire; dry sectioned with Ultra-Sonic Diatome knife at -100 °C ; TEM image courtesy of FELMI-ZFE Graz Austria

Wet Cryo sectioning for Soft Polymers - Ultramicrotomy

For wet cryo sectioning a suitable liquid must be chosen (usually DMSO). This method allows for thin sections to be floated off on a liquid, making the collection of sections easier comparing to dry cryo sectioning.

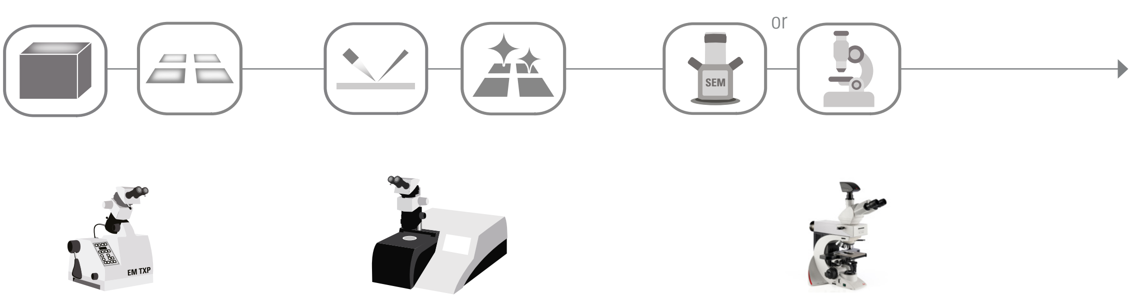


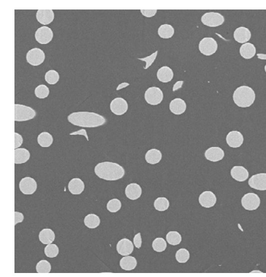
POLYMERS SAMPLE PREPARATION



Ion Beam Milling of Polymers - Broad Ion Beam Milling

Ion milling offers an alternative to ultramicrotomy approach for polymer preparation. It is especially useful for porous or multi-layered samples, or polymers with hard inclusions. Grinding and surfacing can be performed with EM TXP, followed by ion milling with EM TIC 3X.

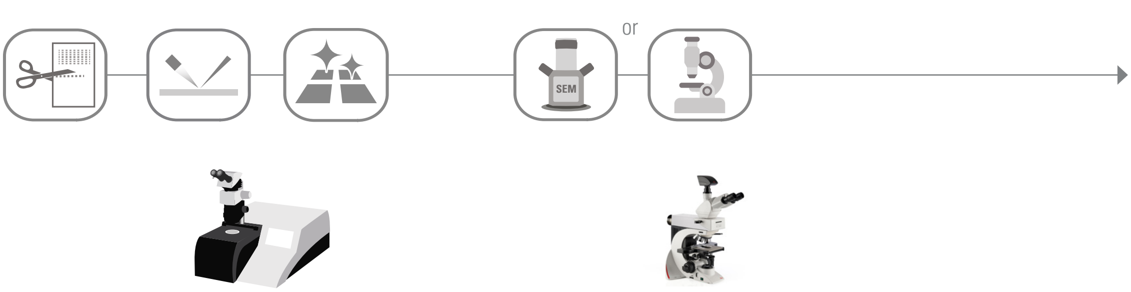


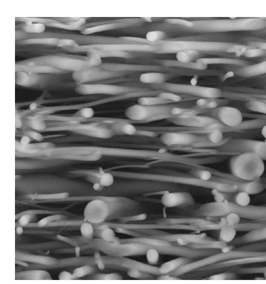


Glass fibers reinforced polyamide

Cryo Ion Beam Milling of Polymers - Broad Ion Beam Milling

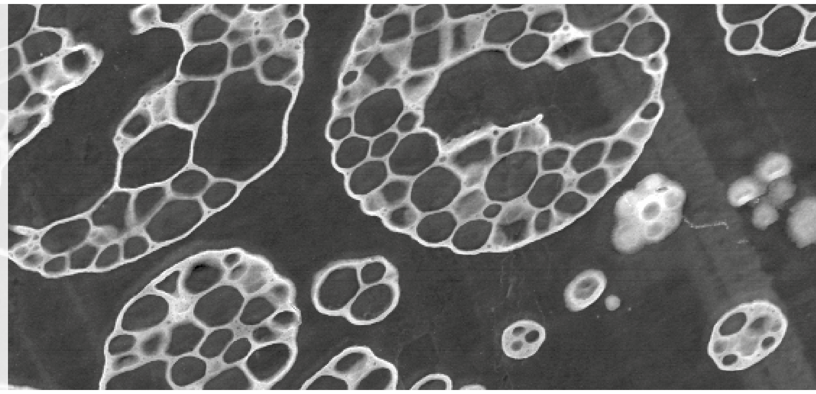
Ion milling offers an alternative approach to ultramicrotomy for polymer preparation. It is especially useful for soft polymers with multiple layers, porosity, or hard inclusions. Pre-preparation can be done using scissors or razor blades to shape the soft material. The sample is then milled in the ion beam milling instrument using the cryo stage.





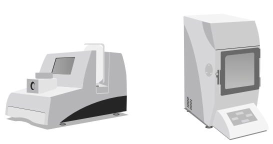
Cross section of coaxial polymer fibres with water soluble portion

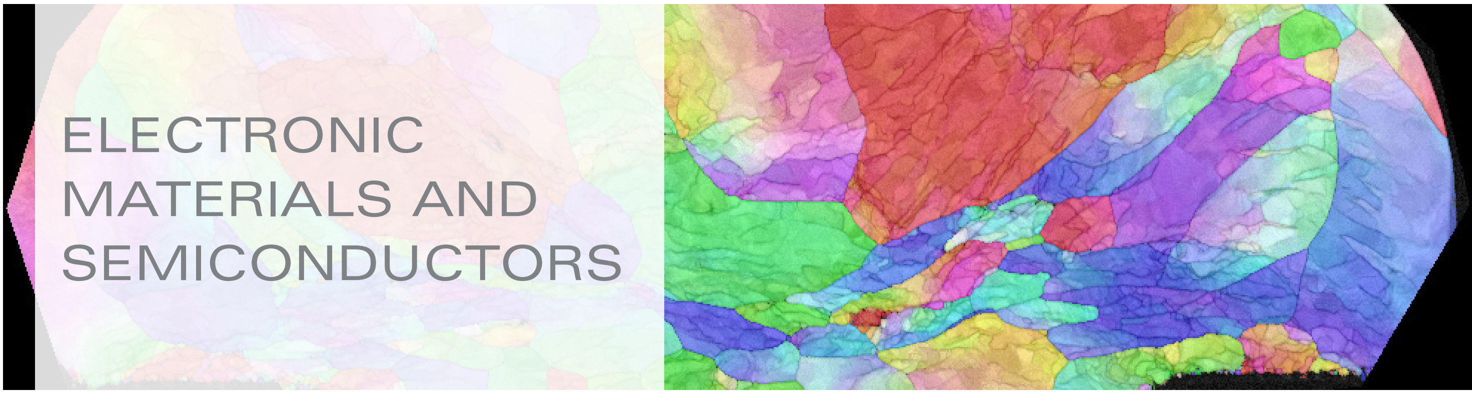
POLYMERS SAMPLE PREPARATION



Drying of Aerogels / Hydrogels

Ash-fabricated aerogels and hydrogels must be dried prior to analysis in the SEM. Critical point drying offers a thorough method to dry the gel sample without compromising its structure. If necessary, the gel can be coated prior to imaging to reduce charging and heat damage to the sample.



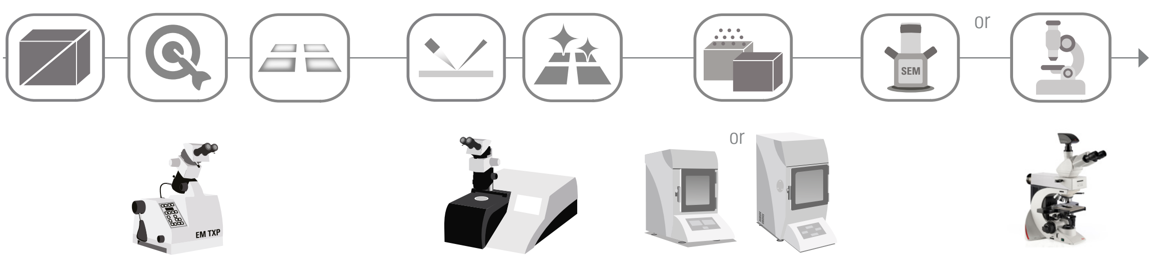


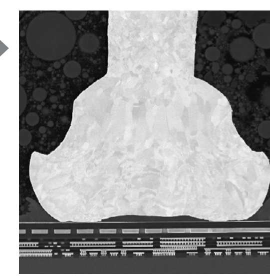
ELECTRONIC MATERIALS AND SEMICONDUCTORS

SEM / LM Solid State Preparation - Broad Ion Beam Cross Sectioning

The technique is most commonly used for solid-state SEM samples but can be applied for LM investigations as well. When the sample has a certain target to be exposed for observation, it needs to be well-cut to get the inner structure of the ROI.

For non-conductive samples subsequent coating is necessary to make the sample suitable for SEM observation.

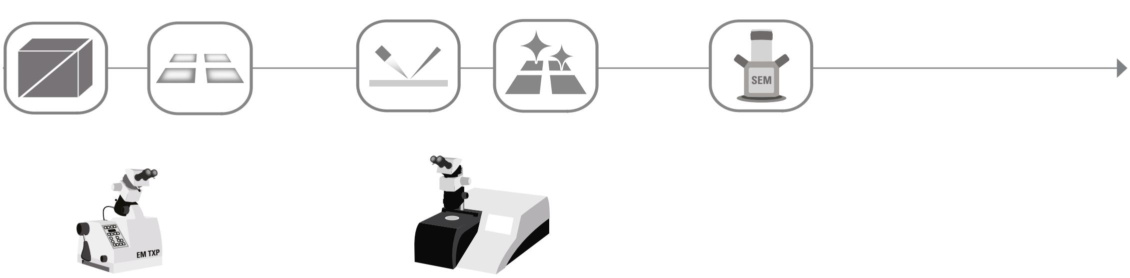


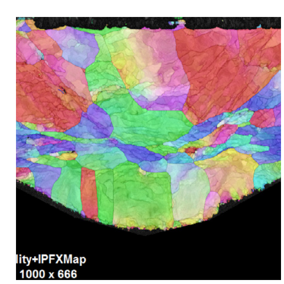


Cross section of IC gold wire bonding

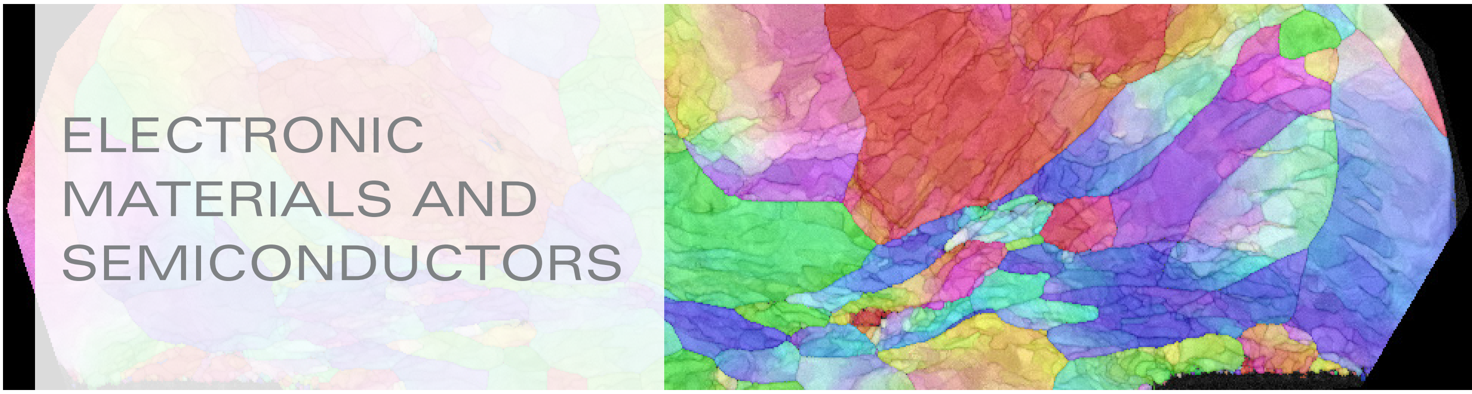
Cross Sections for SEM (EDS & EBSD) - Broad Ion Beam Cross Sectioning

This workflow provides cross sectional surfaces necessary for SEM imaging and EBSD analysis. Mechanical pre-preparation is done by cutting/grinding with the EM TXP followed by ion milling with the EM TIC 3X.



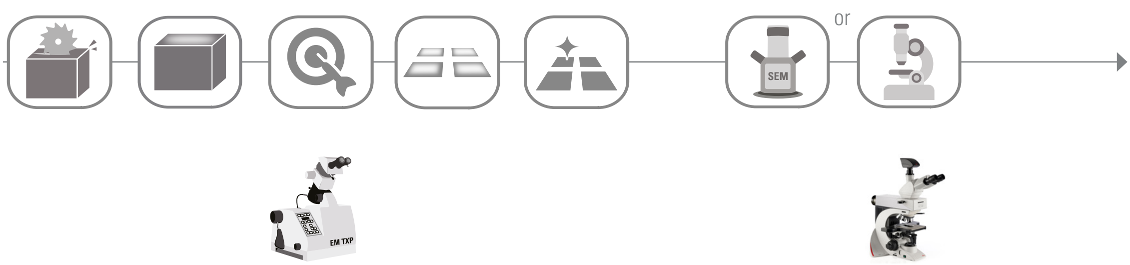


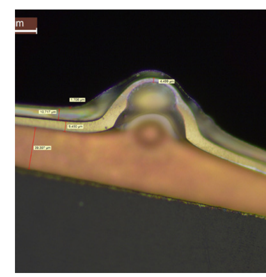
EBSD OIM image of cross sectioned wire bond



Surface Preparation for SEM and LM - Mechanical Preparation

Complete mechanical preparation can be accomplished using the EM TXP. Initial rough cuts can be made with a diamond saw, followed by grinding and polishing with lapping foils. Final polishing can be done using neoprene pads with pastes or colloidal media. This method is especially powerful if small samples have to be prepared. Ductile and brittle material can be milled using a diamond miller for fast preparation and defect inspection.

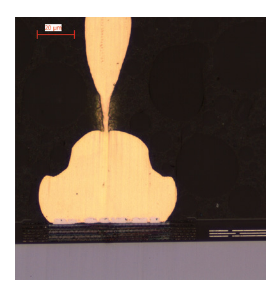




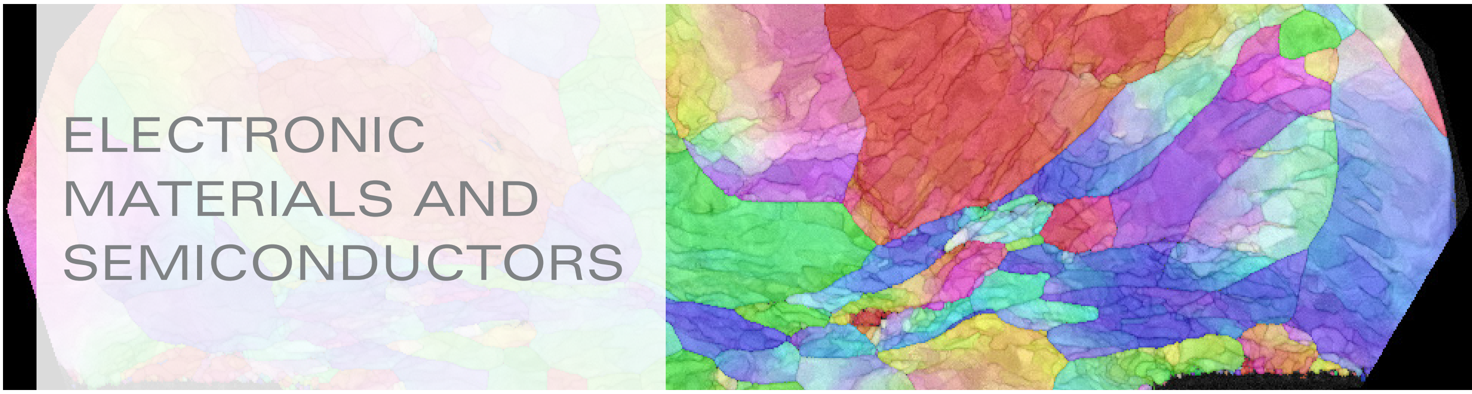
Defect on the lower layer due to a round cupper metal particle deposited, which produces a bump on the upper layer. Picture was taken with a DM2700 M

LM Solid State Preparation - Mechanical Cross Sectioning

This cross-sectioning technique provides for a very fast approach and preparation of the region of interest (ROI). A diamond miller can be used for ductile material whereas a 0.5 µm diamond foil can be used for the final finish of hard and brittle material. The achieved surface quality is mostly sufficient for subsequent LM investigation.



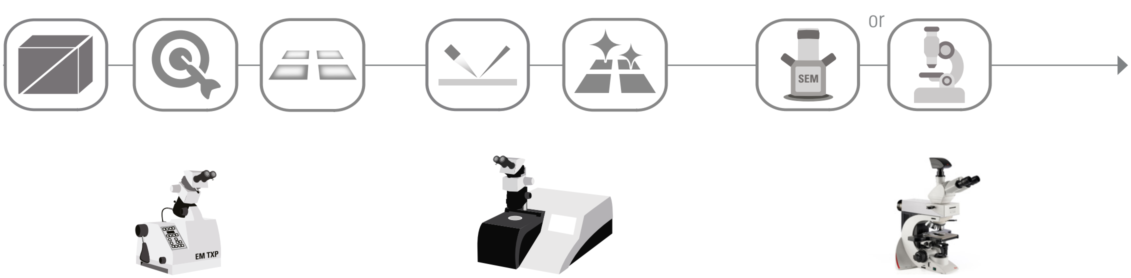
Cross section of gold wire bonding

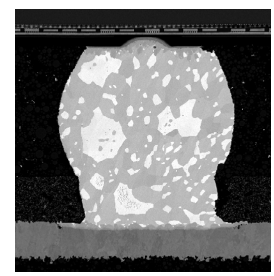


ELECTRONIC MATERIALS AND SEMICONDUCTORS

Target Preparation for SEM and LM - Broad Ion Beam Cross Sectioning

In this workflow, a micro target (or series of targets) within a sample is prepared in a cross section. The EM TXP is used for approaching/aligning the targets and in-situ observation. The sample is then milled in the EM TIC 3X to achieve the desired surface.

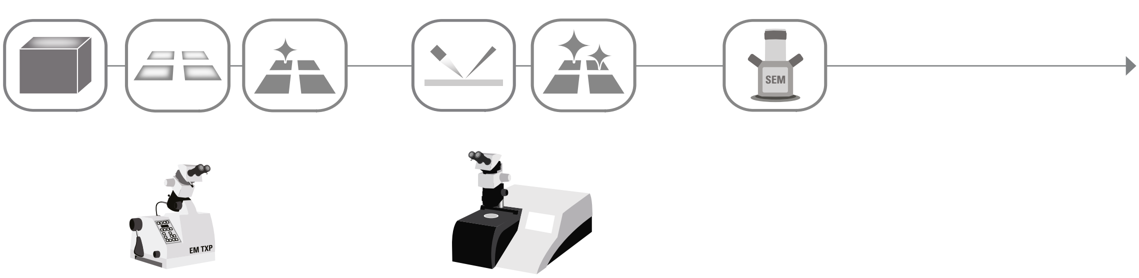


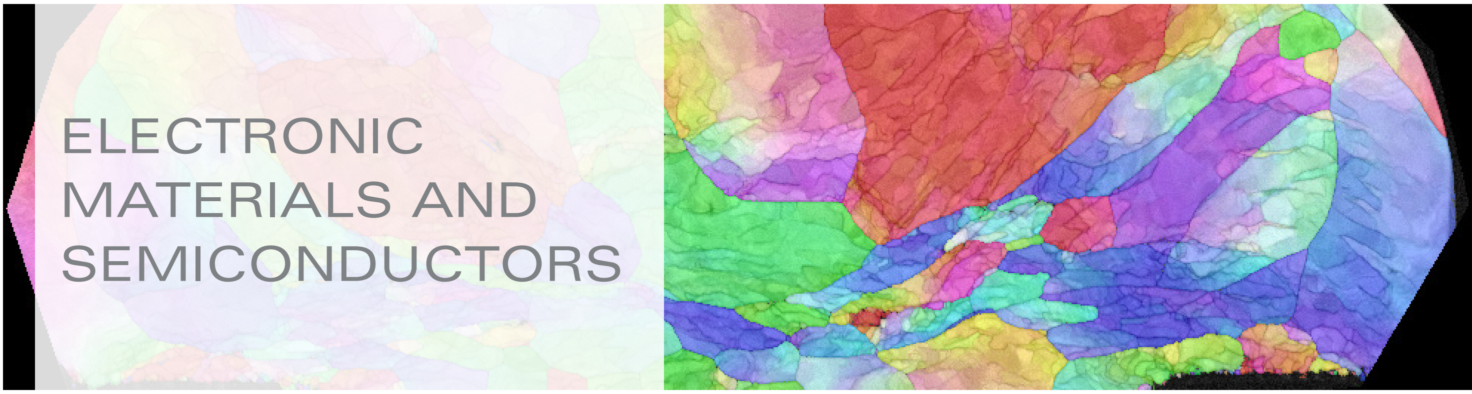


Cross section of a solder bump without thermal effects. Prepared at a cooling temperature of -50 °C

Ion Milling of large Surfaces for SEM - Broad Ion Beam Polishing

A large surface can be prepared to a high surface finish with this workflow. Pre-preparation is done with the EM TXP, which allows for grinding and polishing to a mirror-like finish. Final polishing is carried out by ion milling with the EM TIC 3X.

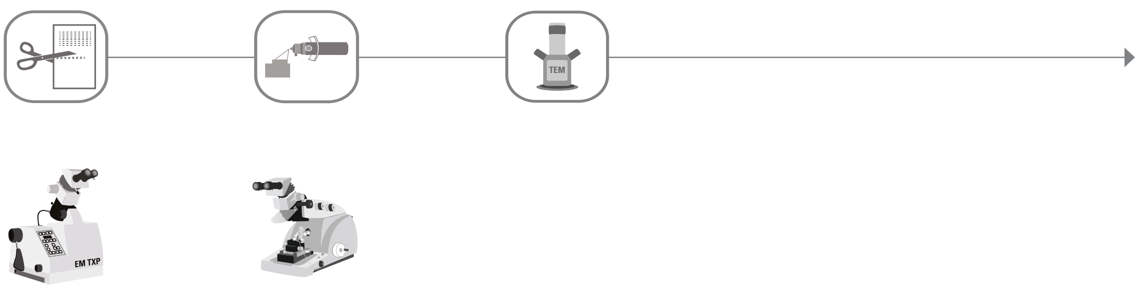


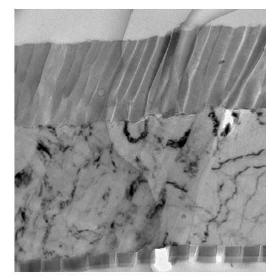


ELECTRONIC MATERIALS AND SEMICONDUCTORS

TEM Preparation - Ultramicrotomy

The EM UC7 offers a quick, cost-effective alternative to ion beam thinning to produce electron transparent sections of semiconductor materials for TEM. A small piece of a semiconductor can be sectioned within a few minutes.

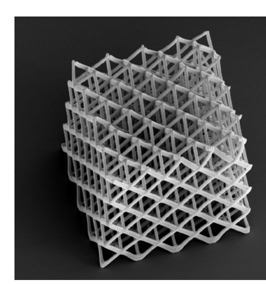




Si wafer, sectioned with 45nm; TEM image

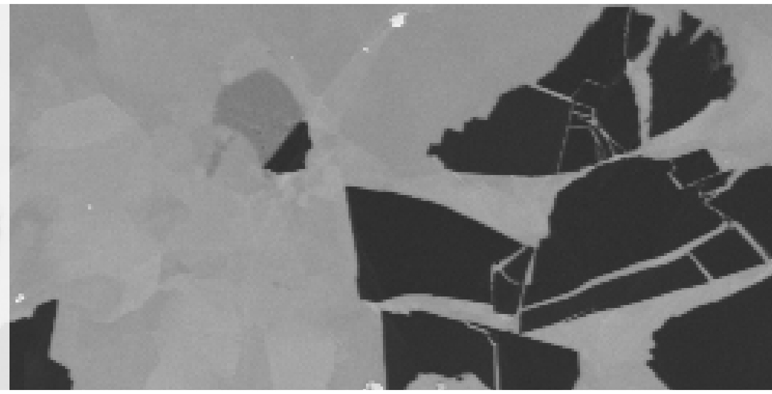
Drying of MEMS and Microfluidics

MEMS and microfluidic devices often need to be dried before further analysis. Critical point drying is one technique that can be used without altering the structure of the device.



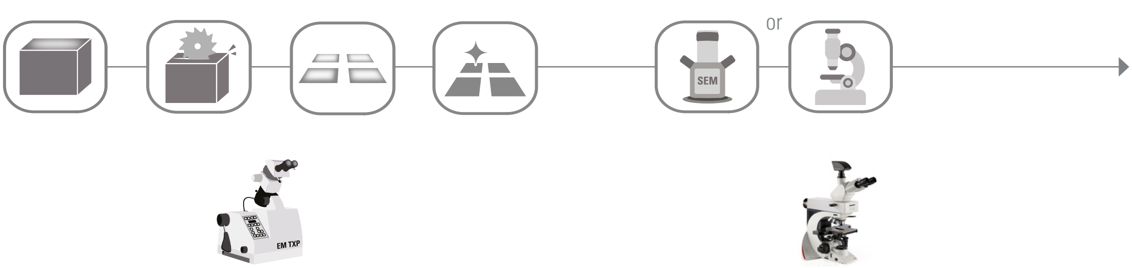
Lightweight nano structure metamaterial. Courtesy of J. Bauer, Karlsruhe Institute of Microstructure Technology (IMT), Germany

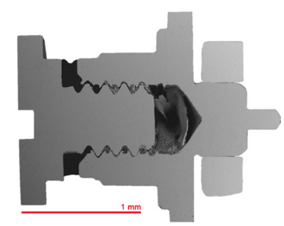
METALS, CERAMICS MATERIAL, HARD MATERIAL COMPOSITES PROCESSING



Surface Preparation for SEM and LM - Mechanical Preparation

Complete mechanical preparation can be accomplished using the EM TXP. Initial rough cuts can be made with a diamond saw, followed by grinding and polishing with lapping foils. Final polishing can be done using neoprene pads with pastes or colloidal media.

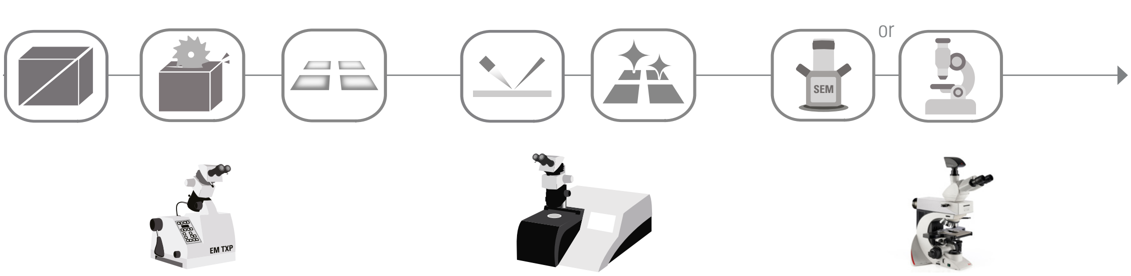


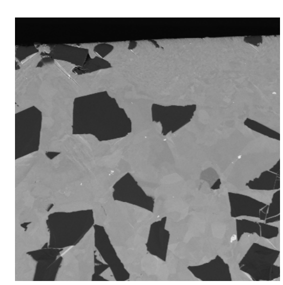


Cross section of a small screw and nut from a wrist watch; prepared without embedding - SEM image

Cross Sections for SEM (EDS & EBSD) and LM - Broad Ion Beam Cross Sectioning

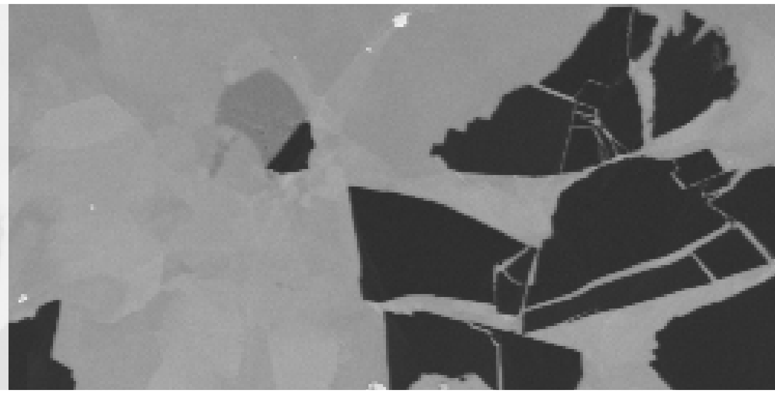
This workflow provides cross sectional surfaces necessary for SEM imaging and EBSD analysis. Mechanical pre-preparation is done by cutting/grinding with the EM TXP, followed by ion milling with the EM TIC 3X.





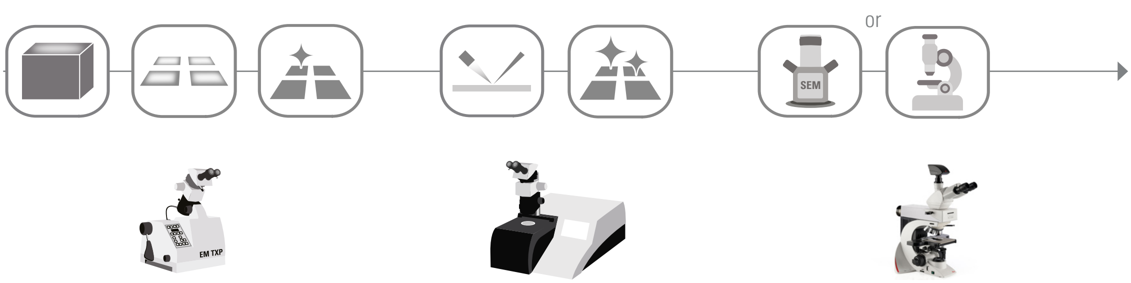
Cross section of diamond particles in an Al matrix

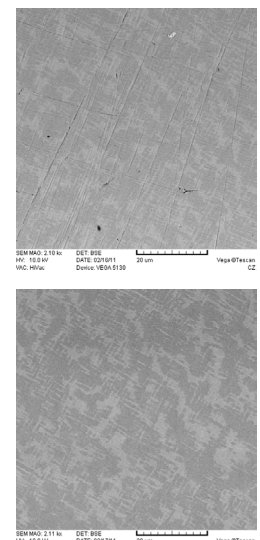
METALS, CERAMICS MATERIAL, HARD MATERIAL COMPOSITES PROCESSING



Ion Milling of large Surfaces for SEM and LM - Broad Ion Beam Polishing

With this workflow a large surface can be prepared to a high finish. Pre-preparation is done with the EM TXP, which allows for grinding and polishing to a mirror-like finish. The final polishing is accomplished by ion milling with the EM TIC 3X to provide a high quality surface e.g. for EBSD.

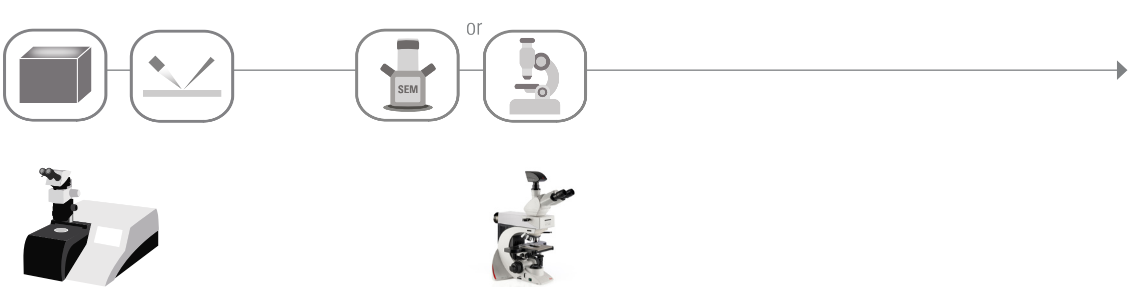


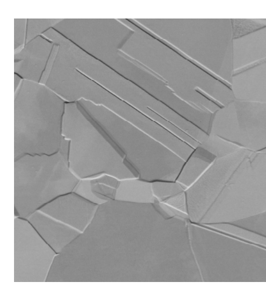


Nickel based super alloy (CSMX-4) after mechanical polishing (top) and after additional ion milling (bottom)

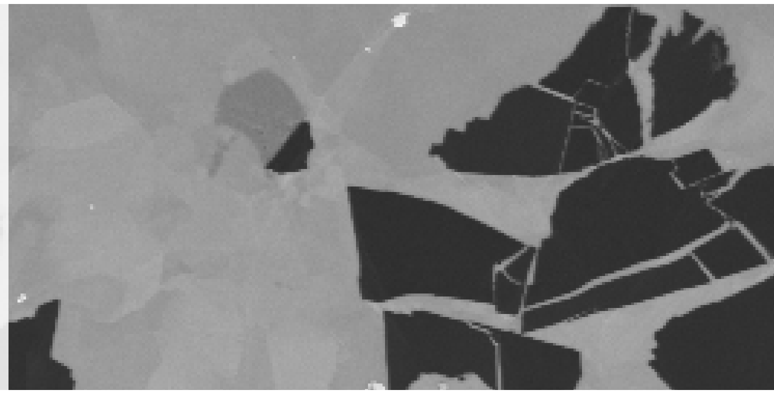
Contrast Enhancement for SEM - Broad Ion Beam Polishing

When working with polycrystalline materials it is often desirable to process the sample in a way that the grain structure is easily discernible. In many cases this can be achieved by chemical etching. However, after surface preparation in the ion mill, an additional brief milling step can be conducted at normal or near-normal incident to etch grain boundaries and increase the contrast.





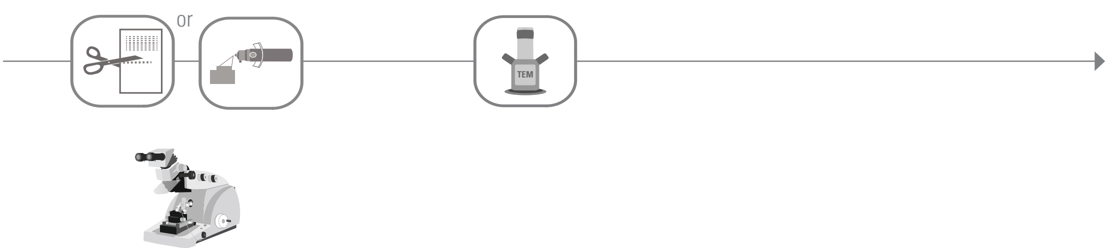
Cu surface after ion beam milling contrast enhancement; SEM image

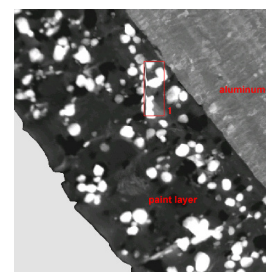


METALS, CERAMICS MATERIAL, HARD MATERIAL COMPOSITES PROCESSING

TEM Solid State Preparation - Ultramicrotomy

This workflow can be used instead of the ion beam thinning method. Many solid state samples such as ductile materials (Al, Au, Ms, Cu and their alloys) can be cut by a diamond knife. This method is a way to get nanometer thin sections for TEM within minutes. In comparison: the ion beam thinning process normally lasts for several hours and the electron transparent area measures only a few micrometers.

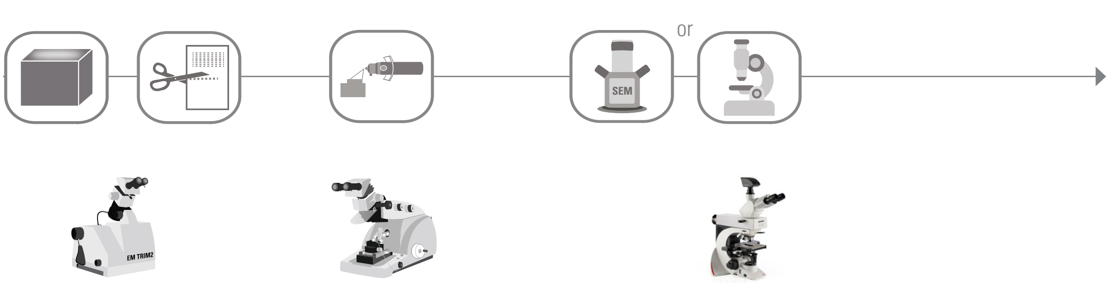


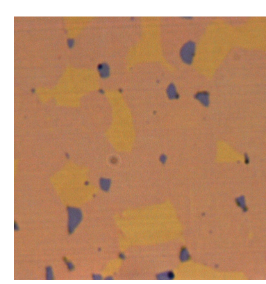


Cross section of a pop can, TEM with EDX

Surfacing for SEM and LM

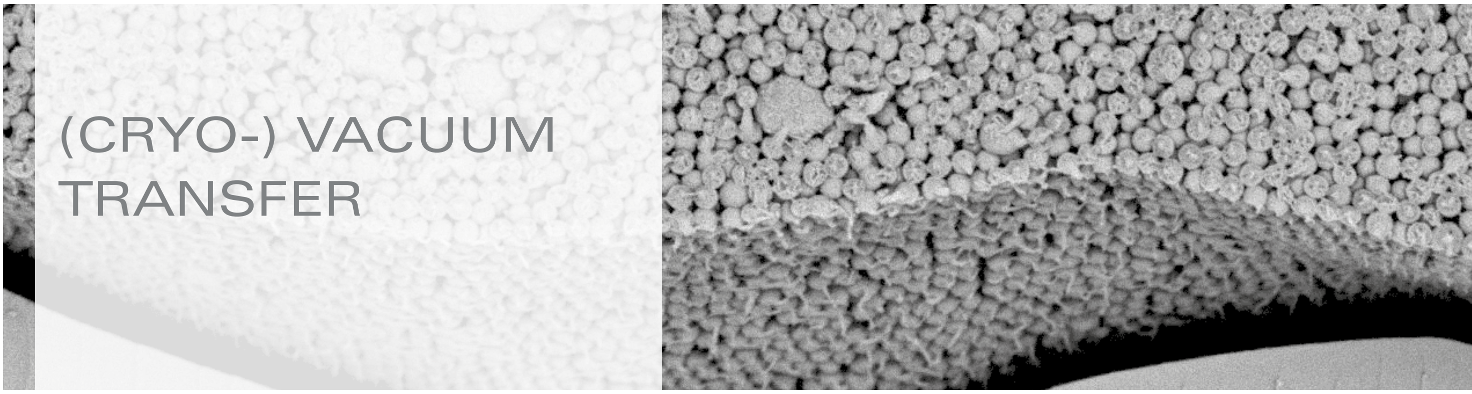
This method prepares a high quality surface of ductile metals such as Ag, Au, Al, Cu and its alloys. The ultramicrotome allows the user to set the feed for sections in a nanometer range.





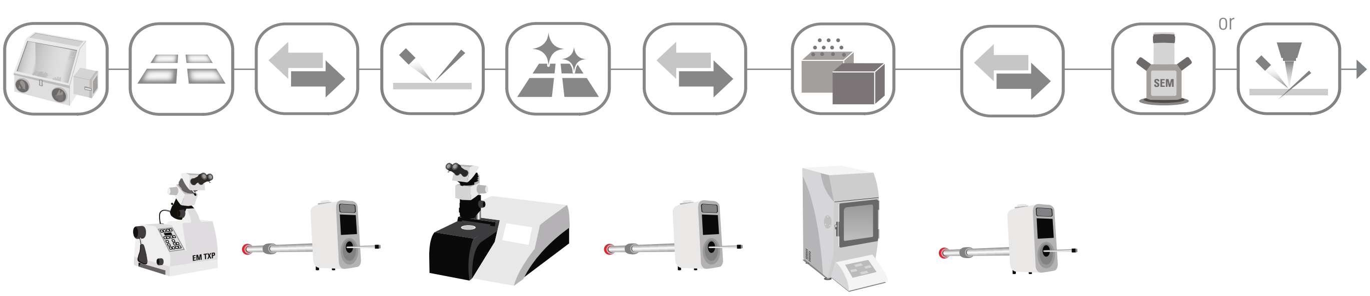
Block-face of surfaced brass; LM Image

(CRYO-) VACUUM TRANSFER



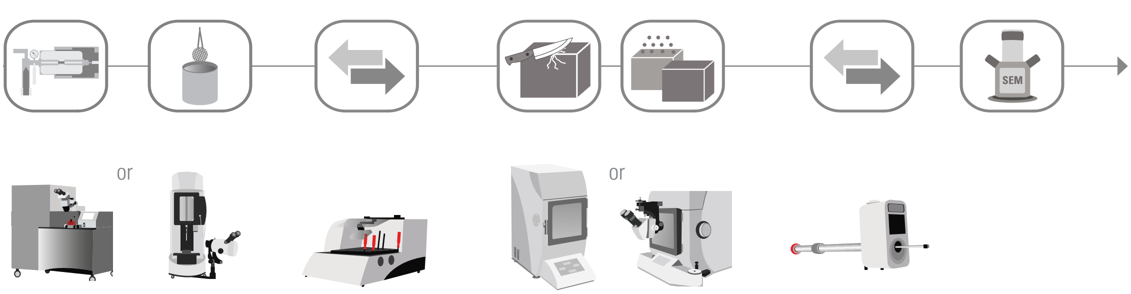
SEM Sample Preparation and Transfer under Vacuum or certain Atmosphere

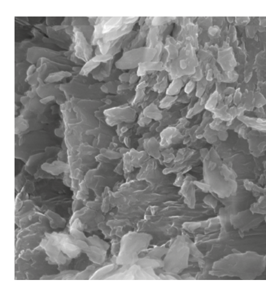
This is a specific workflow for air-sensitive samples which are known to oxidize fast. The EM VCT500 shuttle provides a well protected atmosphere for the sample, keeping it in vacuum or certain inert gases. The shuttle can then be connected to several sample preparation instruments and to microscopes such as an SEM or a FIB.



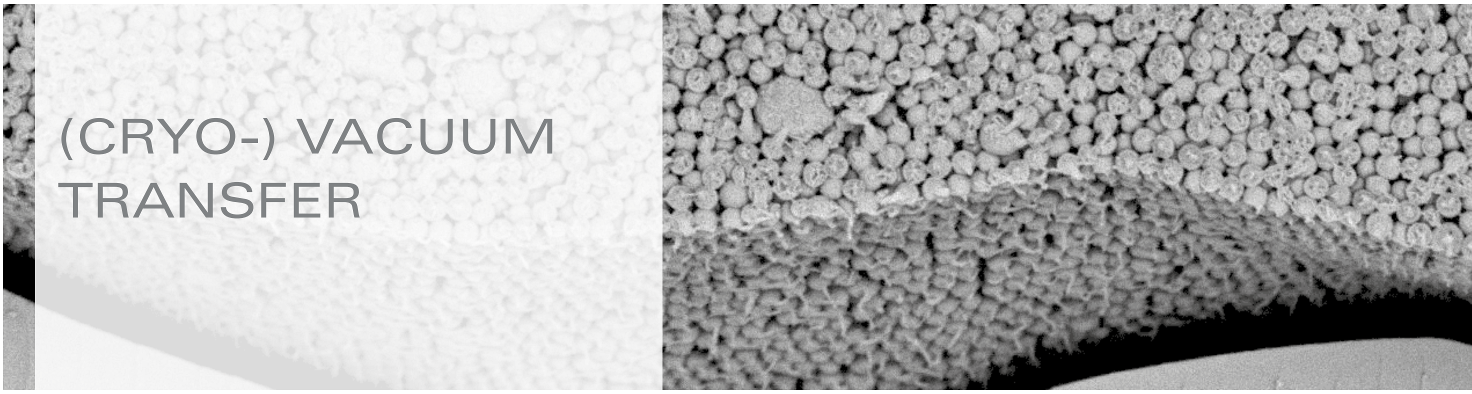
Cryo-SEM Sample Preparation and Transfer - Freeze Fracturing

This cryo transfer workflow is used for fluid or liquid samples, e.g. cosmetics, food, collochemistry samples etc. The freezing method depends on the sample character, e.g. water content, form etc. It is followed by freeze fracturing to reveal the inner structure of the sample for further imaging of the surface in the Cryo-SEM.





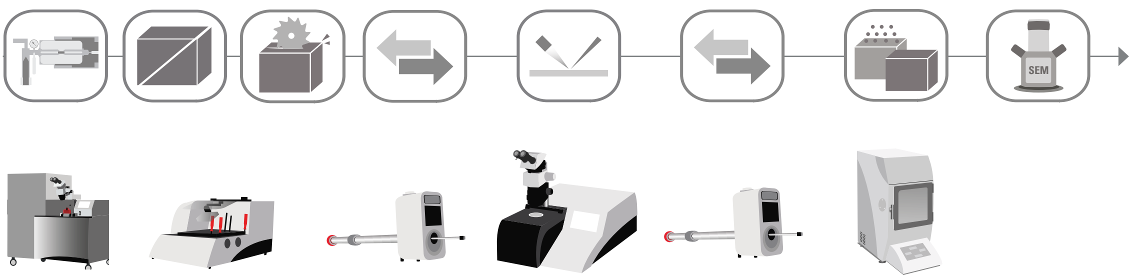
Sun Screen Lotion high pressure frozen without UV light exposure. Courtesy of Dietmar Pum, PhD, University of Natural Resources and Life Sciences, IAGZ, Austria

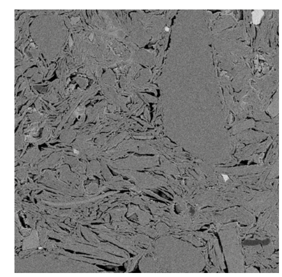


(CRYO-) VACUUM TRANSFER

Cryo-SEM Sample Preparation and Transfer - Broad Ion Beam Cross Sectioning

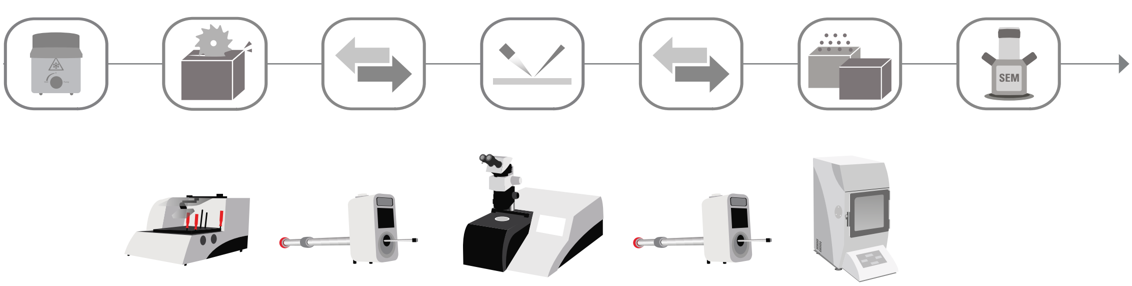
This cryo transfer workflow is used for high pressure frozen samples including hard and brittle material (e.g. clay with quartz). The sample has to be kept at -150 °C during the complete preparation process, from high pressure freezing, over mechanical pre-preparation using the cryo-saw, followed by an ion beam milling step and finally transferring it to the Cryo-SEM.

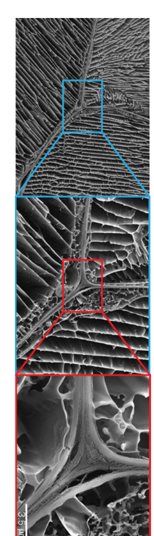




Compact clay

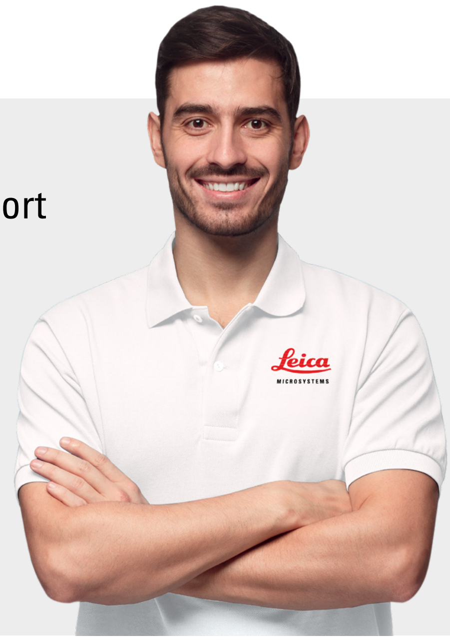
This workflow uses cryogenic broad ion beam milling in combination with scanning electron microscopy (cryo-BIB-SEM) for imaging and analyzing the microstructure of cryogenically stabilized soft polymers. It enables the examination of delicate, natural polymers, such as tomato skin or wood, with a cryo-BIB-SEM. The polymer surface morphology and various microstructural features can be analyzed.





High resolution cryo-BIB-SEM images of tomato skin cross section showing fine details of the microstructure, courtesy of J. Schmatz, MaP - Microstructure and Pores GmbH, Aachen, Germany

WHY LEICA SERVICE?



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