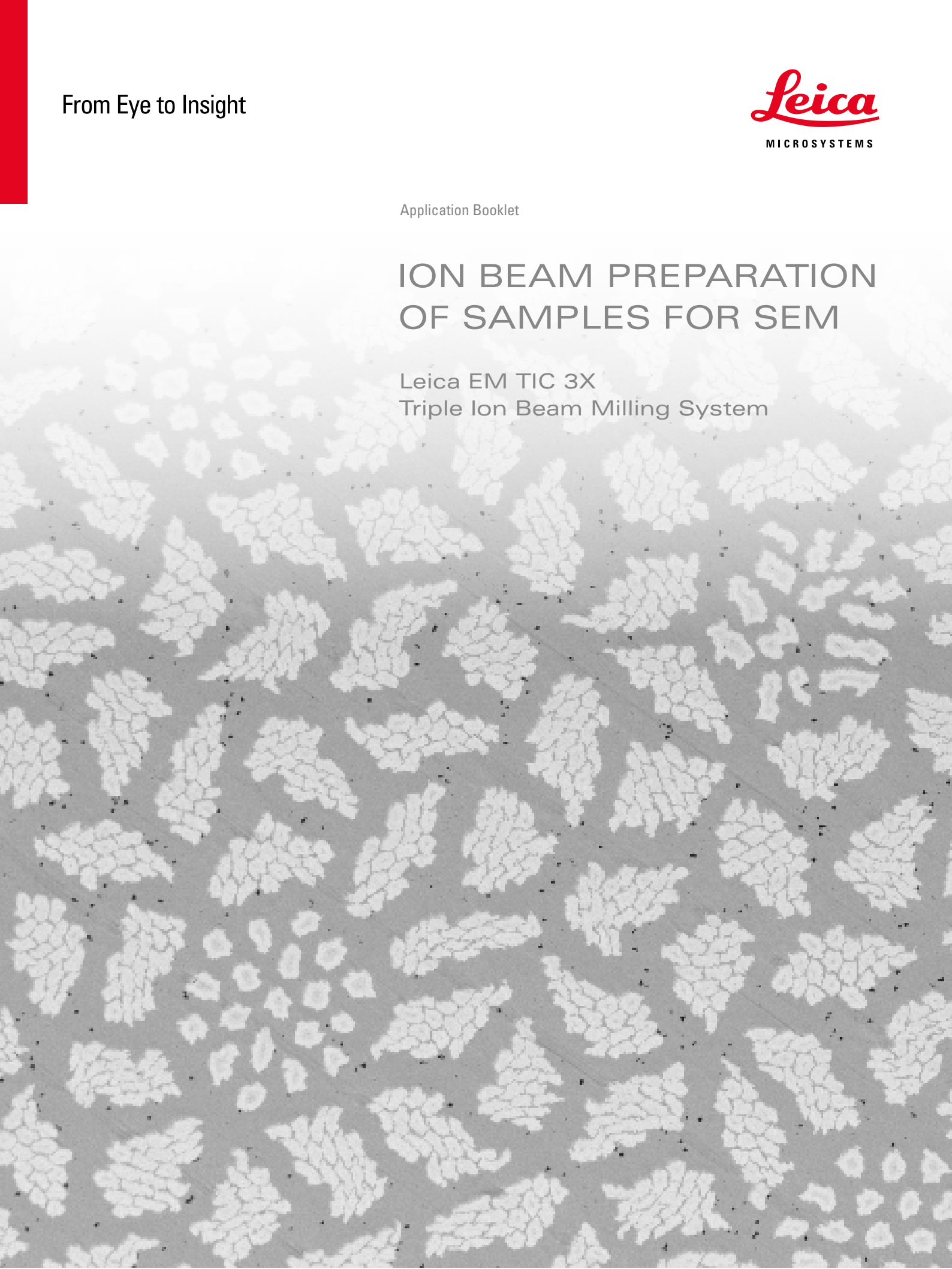
From Eye to Insight

Leica
MICROSYSTEMS

Application Booklet

ION BEAM PREPARATION OF SAMPLES FOR SEM

Leica EM TIC 3X
Triple Ion Beam Milling System



CONTENT

Author:
Wolfgang Grünewald
Leica Mikrosysteme GmbH
Laboratory at:
TU Chemnitz
Institut für Physik
Germany
Phone: +49 371 531 3 7755
Mobile: 0172 624 0800
Email: Wolfgang.Gruenewald@leica-microsystems.com

1. INTRODUCTION

Today, ion beam etching is the most widely-used method for preparing samples for electron microscopy. In this process, the sample material is bombarded with a high-energy argon-ion beam. The ion energy and the milling angle depend on the corresponding application.

For preparing samples for scanning electron microscopy (SEM), etching is used to improve or modify the quality of surfaces.

One can clean, polish or alter the contrast of these surfaces. The quality of the surfaces after the ion beam treatment is so good that they can also be used for surface sensitive methods, such as EBSD.

Another special application of ion milling involves the preparation of sample cross-sections using ion beam slope cutting.

The Leica EM TIC 3X is an ion beam etching unit that is used for preparing samples for scanning electron microscopy. Sample cross sections can be produced and larger surfaces can be processed.

The cross-sections can be treated again in an additional process using a low-energy ion beam. This applies both to cleaning as well as contrast enhancement.

Additionally, it is also possible to improve the quality of mechanically polished surfaces via a final ion polishing process. The maximum size of the processed sample surface is 25 mm in diameter, the maximum size of the samples to be processed is 38 mm in diameter.

The surface of samples can be cleaned, polished and have its contrast altered. This makes it possible for all preparation types for scanning electron microscopy to be carried out in a single instrument.

The instrument is setup in a modular system and uses 5 different samples stages for the respective applications: a standard stage, cooling stage, multiple stage, rotary stage and a vacuum lock to transfer cryogenic samples or samples sensitive to oxidation. These sample stages can be changed very easily.

Fig. 1: Leica EM TIC 3X ion beam etching unit with the unique triple beam technique

The standard stage is suitable for a wide application of samples that have different sizes, shapes and material. Samples with thermal sensitivity are prepared in a special cooling stage to prevent the samples from being destroyed due to overheating. Finally, the multiple stage makes it possible to prepare 3 samples successively without intervention by the operator. This guarantees a high level of sample throughput.

The rotary stage can handle the surface treatment of samples that measure up to 38 mm in diameter.

The preparation process and progress can be observed at any time using a stereomicroscope. As an option, images can also be stored using an integrated camera.

The TIC 3X is a compact tabletop instrument that combines all components in one housing. The instrument uses a unique triple beam technique. Three ion beams intersecting at the mask edge shape a milling sector of 100°. The instrument uses saddle-field ion sources that together attain milling rates of more than 150 µm/h at 10 kV and 3 mA.

The triple beam technique eliminates the need to move the sample during ion milling. This creates myriad benefits that will be discussed in greater detail below. All of these advantages guarantee a high level of preparation quality.

2. SAMPLE PREPARATION FOR SCANNING ELECTRON MICROSCOPY (SEM)

2.1. CROSS SECTIONAL SAMPLE PREPARATION

Ion beam slope cutting is a method for producing sample cross-sections for scanning electron microscopy (SEM).

This method has its origins in an idea from Wolfgang Hauffe, who initially used this method only for producing diagonal cuts. This is what led to this method being referred to as slope cutting.

The original slope cutting principle is shown in Fig. 2. The sample is covered with a sharp-edged mask so that only 50–100 µm of the sample material is exposed above the mask. A collimated ion beam facing the mask surface strikes the uncovered material and removes it. This produces a sample cross-section with an excellent surface quality.

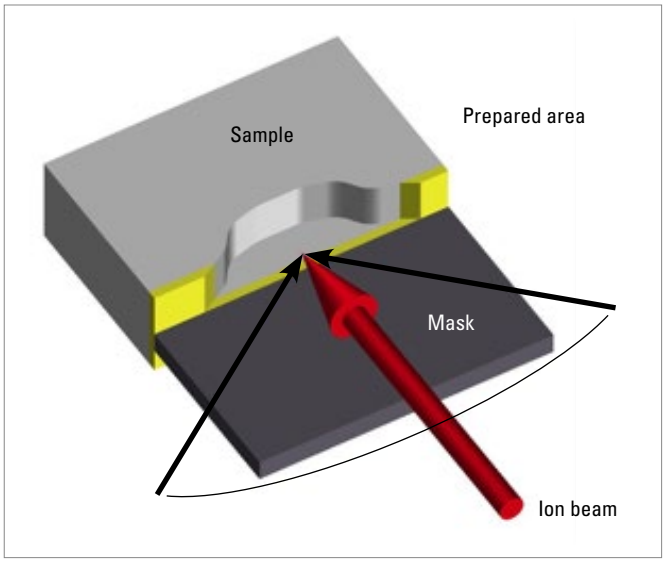


Fig. 2: Ion beam slope cutting principle with an ion beam and an oscillating sample

In terms of the prepared cross-section surface, preparation artifacts are rare due to the parallel incidence of ions. The sample is oscillated during the etching process to prevent etching structures caused by different milling rates in a non-uniform sample.

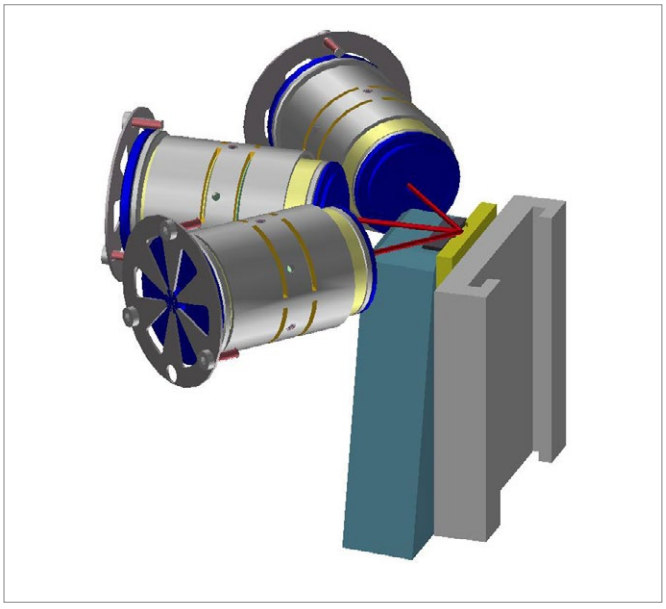


Fig. 3. Leica EM TIC 3X triple beam technique principle

The Leica EM TIC 3X uses an innovative triple beam technique (Fig. 3). Instead of one ion source and an oscillating motion on the sample, three ion sources are used that form an etching sector of 100° (Fig. 3, 4). The ion beams strike the mask edge directly.

The ion sources and the mask are fixed in place. A three-axis stage is used to adjust the sample with respect to the mask and the ion beams as a result.

The milling sector has the same effect as oscillating movement of the sample. A stationary sample, however, provides myriad advantages that will be discussed below.

The stationary sample can be observed in high quality during the cutting process by using a stereomicroscope. The heat dissipation between the sample and the environment during the cutting process is significantly better with a stationary sample. As a result, the sample temperature can be kept below 70 °C without active cooling. This does, however, depend on the heat transfer from within the sample. Three ion sources reduce the preparation time without exposing the sample to excessive thermal stress.

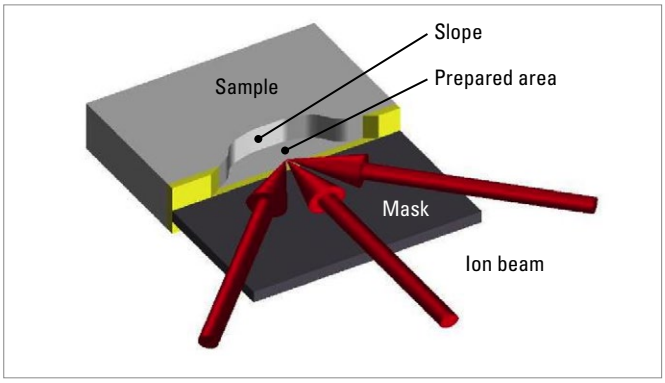


Fig. 4: Ion slope cutting with the triple beam technique

Finally, the continuous ion bombardment from multiple sides diminishes the danger of soiling the surface as a result of re-deposition.

All of these aforementioned advantages result in outstanding preparation surface quality.

In general, ion beam slope cutting does not require mechanical pre-preparation, unlike ion polishing. Some pre-preparation steps, however, can be necessary in the case of site-specific preparation. The excess material must be removed if the target is located deeper than 100 µm below the sample surface (Fig. 5). This can be done using a mechanical grinding process (also see Chapter 3), in which the following aspects must be observed:

> The ground surface should be smooth, but it does not need to be polished.
> The angle between the sample surface and the cross-section surface intended for milling should be 90°.
> The sample surface and the base area should be parallel to allow for optimal adjustment of the mask. If this is not the case, the adjustment holder (Fig. 7) can be used to compensate for these deviations.

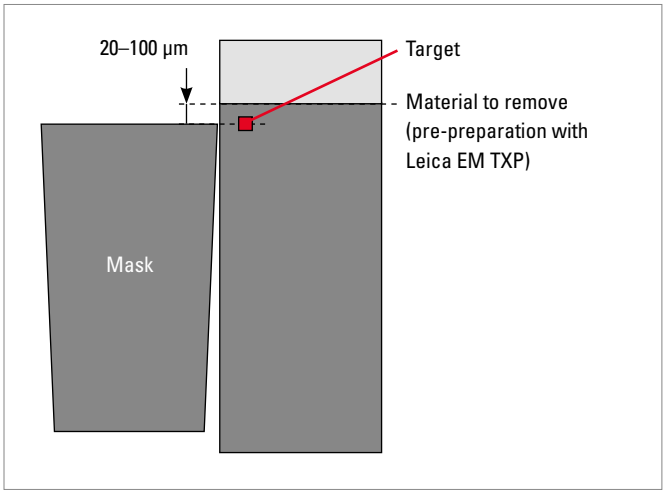


Fig. 5: Requirements for a successful cross sectional sample preparation using the ion slope cutting procedure

All of these requirements are important for ensuring that the preparation result is good.

Additionally, securing the sample to the sample holder and adjusting to the mask edge are a critical aspects.

Three different sample stages for the cross sectional sample preparation are available for the Leica EM TIC 3X. These stages can be swapped out with ease. The problem determines which is best for use.

Thermally stable samples are usually prepared using the standard sample stage. This has the advantage of allowing the use of various sample holders, which enable the preparation of samples that have different dimensions and shapes.

Samples with a regular shape, i.e. semiconductor materials, can be prepared using the standard aluminum holder for samples with a size of up to 50 mm × 50 mm × 10 mm.

Fig. 6. Standard stage with different sample holders

The adjustment holder (Fig. 7) is available for samples with irregular shapes. This enables internal adjustment of the sample in two directions in relation to the mask. As a result, samples such as crumbs from rubber or stones can also be aligned and prepared. In the event of a site-specific preparation of semiconductor structures, the holder can also be used to achieve optimal adjustment of the structures to the mask edge.

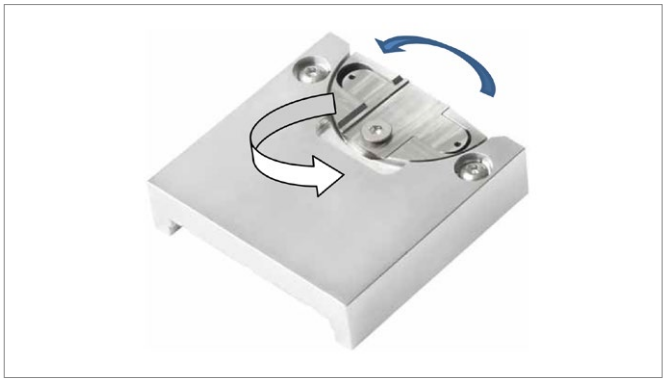


Fig. 7: Adjustment holder for the preparation of samples with irregular shapes. The sample can be adjusted in two directions.

Jigs that enable the pre-adjustment of the sample are used to apply the sample to the sample holder (Fig. 8). These jigs enable pre-adjustment of the sample height and ensure the mask and sample are parallel.

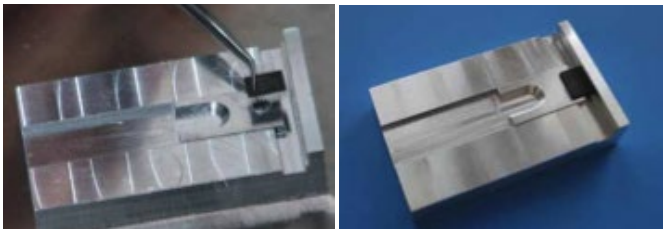
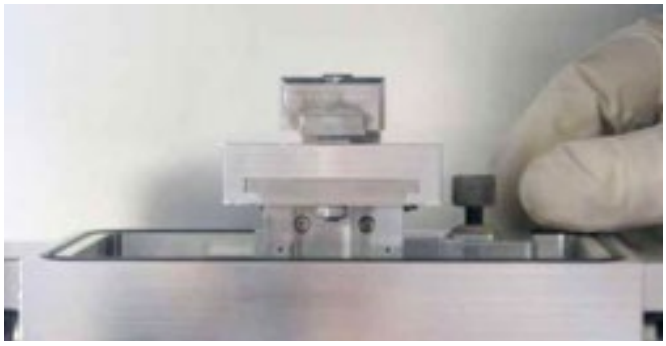


Fig. 8: Adjustment jig for the sample on the sample holder

After this adjustment, a fine alignment of the sample using the three-axis stage is typically all that is necessary.



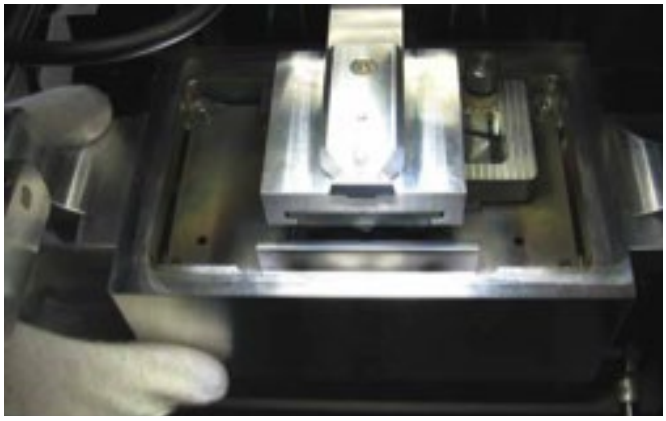


Fig. 9: Adjusting the sample using a three-axis stage: Distance of the mask to the sample (above) and sample height above the mask (below).

The orientation of the sample to the mask (parallelism) and the distance of the sample to the mask have a large impact on the preparation quality. The sample stage can be tilted in two different positions for the observation of the sample position during the adjustment process (Fig. 9.).

Multiple stage

The multiple stage (Fig. 10) can be used for a high volume of sample throughput. This stage combines three standard stages in one. Three samples can be prepared successively without intervention by the operator. The samples are installed successively and the corresponding preparation parameters are stored for each preparation. The samples can then, for example, be processed overnight in a preparation cycle.

The multiple stage, however, can use only the standard stage's small aluminum sample holder due to geometric reasons. Adjustment of the sample along the X axis (left/right movement) is not possible.

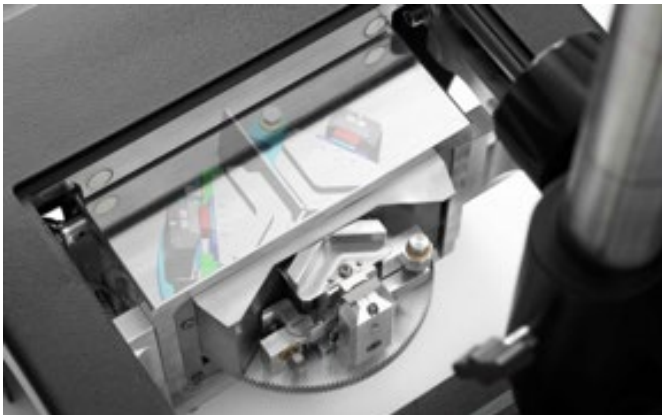


Fig. 10: Multiple stage for the preparation of 3 samples in a preparation cycle

The content below is intended to show examples of preparing samples with the standard stage.

2.1.1. Preparation of semiconductor material

Semiconductor samples usually have a regular shape and as a result, can be adjusted easily to the Leica EM TIC 3X mask. An additional adjustment, however, can often times be required in the case of site-specific preparation. This can be done using the adjustment holder (Fig. 7).

SOLDER BUMP

Aim of analysis

Solder bump cross-section

Preparation problem

Solder bumps consist of a very soft material that is usually difficult to prepare successfully using conventional mechanical polish. The polished surface is usually smeared with dirt and does not show structural details.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Affix the sample to a small Al holder on the Leica EM TIC 3X (e.g. using cyanoacrylate adhesive) and fasten it to a Leica EM TXP adapter.
- Sand the cut face using 9 µm diamond foil until the solder bump is visible
- ≥ 45° wedge of the upper edge of the sample for reducing the material to be etched (also refer to Chap. 3)

The sample's wedge-shaped polished section requires the use of a wedged mask for slope cutting.

The sample can be prepared without changing the sample holder in the Leica EM TIC 3X (also refer to Chap. 3) after mechanical pre-preparation.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	3 h
Mask	Wedged mask

Result

The surface of the solder bump is very smooth and clean. The structural details in the solder bump are all easily recognizable. However, spaces can be seen between solder bump components that are rich in lead and tin. These spaces can be explained by the shrinking of the components. This, in turn, can be traced back to the excessive heating of the sample during of the preparation process /1/.

As a result, the sample has also been prepared with a cooling process (refer to Chapter 2.1.5., image 42) to verify the effect of the temperature increase on the sample structure and to remedy the problem.

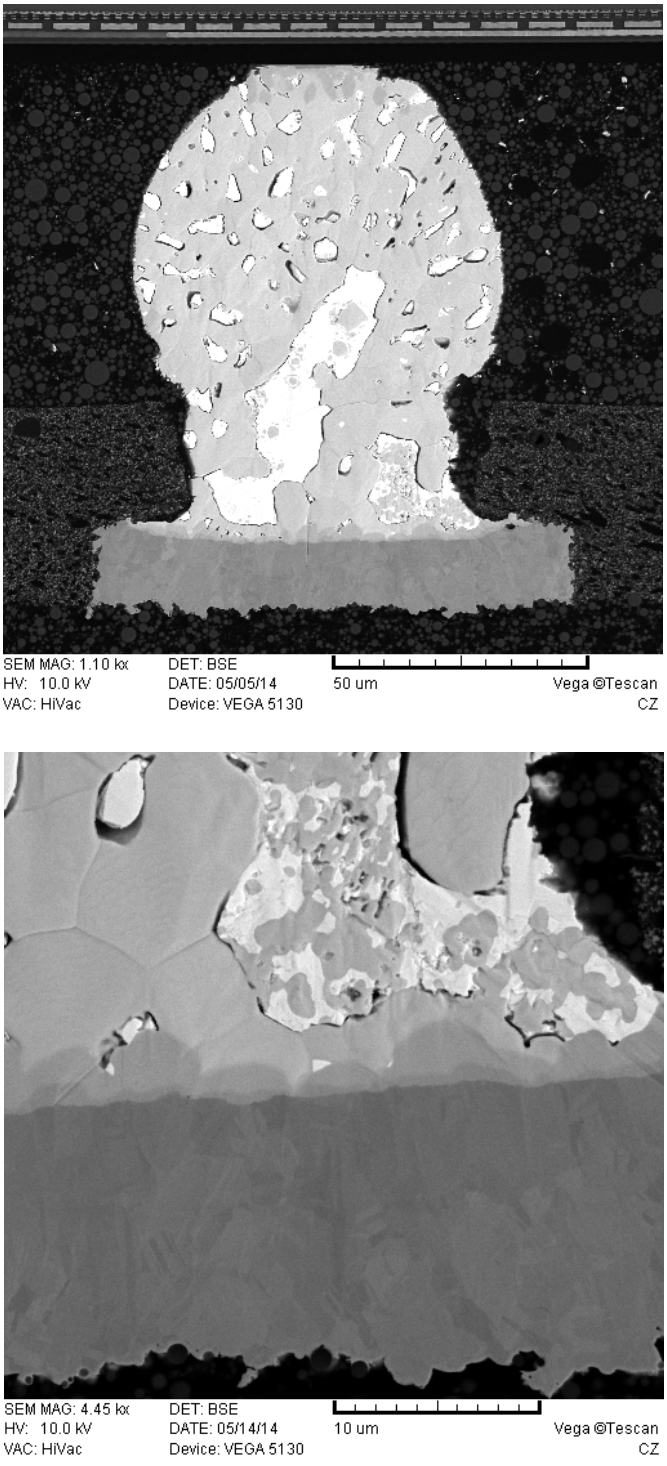


Fig. 11: Cross-section of a solder bump with shrinkage effects as a result of heating the sample during the etching process (compare with cooled sample in Fig. 42)

VIA STRUCTURE FILLED WITH COPPER

Aim of analysis

Cross-section through as many vias (site-specific preparation) as possible to examine the grain structure of the copper in the via.

Preparation problem

The vias have a diameter of only 5 µm. This puts the structure size at the limits of the setting accuracy for the sample in relation to the mask. The site-specific preparation of these vias requires optimum adjustment of the sample.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Affix the glass onto the sample surface to protect the structure.
- The sample is then glued to the Leica EM TIC 3X Al holder and screwed into the Leica EM TXP adapter.
- Sand the cross-section surface using 2 µm diamond foil until the structure is visible. While doing so, the sample can be fastened to the sample holder using an adjustment jig and adjusted exactly to the grinding disc.

ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	2 h 10 min

Result

The good adjustability in the Leica EM TXP made it possible to sand the sample parallel to the via structure.

This perfect mechanical pre-preparation also makes an almost parallel cut through several vias possible. The image below in Fig. 12 shows the section through 3 complete vias.

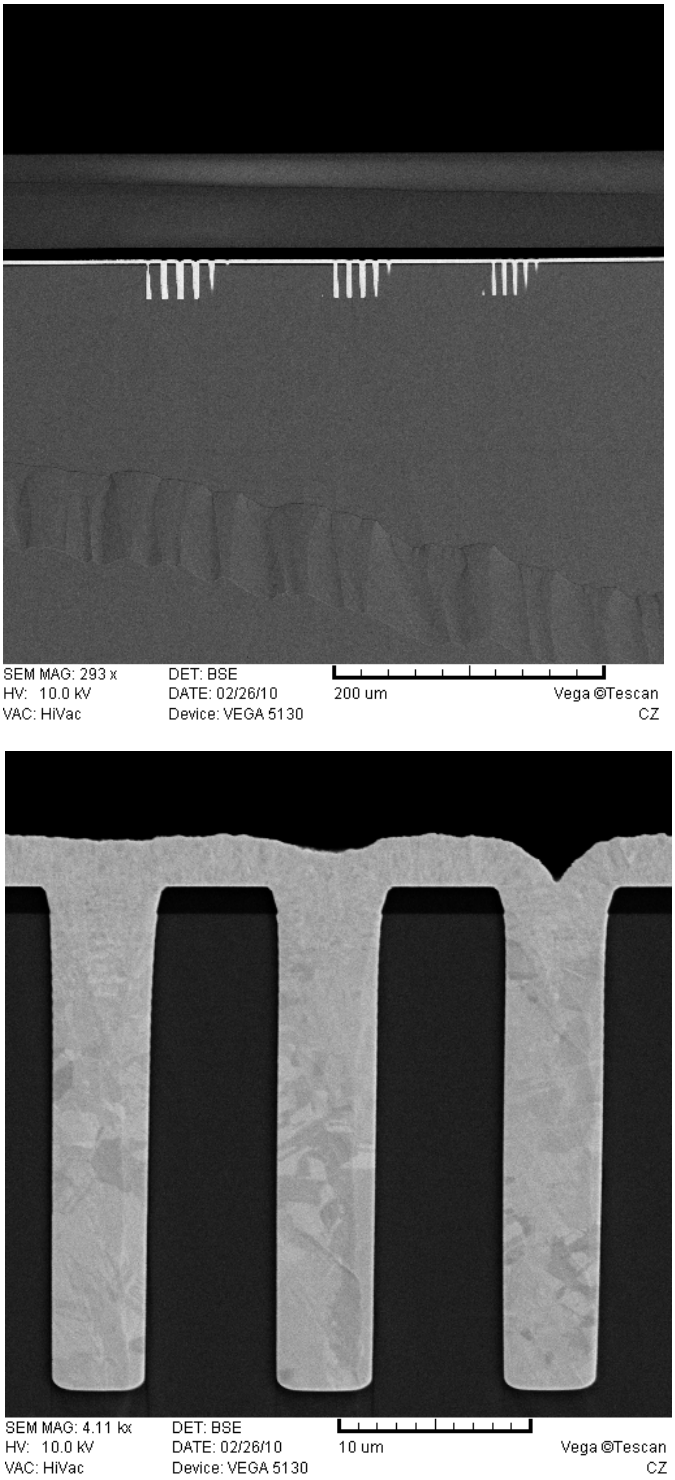


Fig. 12.: Cross-section through vias filled with copper

COPPER/TIN CONNECTOR (SOLAR CELL)

Aim of analysis

Cross-section of the copper/tin connector for the examination of layered structures and their interfaces.

Preparation problem

The materials are very soft and as a result, they become smeared with dirt during mechanical polishing.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Embedding between two pieces of cover glass
- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the cross-section surface using 9 µm

The sample remains on the sample holder and can be prepared in the Leica EM TIC 3X after the mechanical pre-preparation.

ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	6 h 30 min

Result

It was possible to cut through the sample completely in 6 h and 30 min. The grain structure of the copper is easily visible. Pores can be seen at the interface. The cut face is very smooth and clean. As a result of the parallel incidence of ions, it should be free of defects and very well suited for EBSD as a result. Because the sample is protected by being embedded between two pieces of cover glass, preparation effects on the edge of the Cu/Sn connector (see image above in Fig. 13) which would otherwise occur without the protection are prevented.

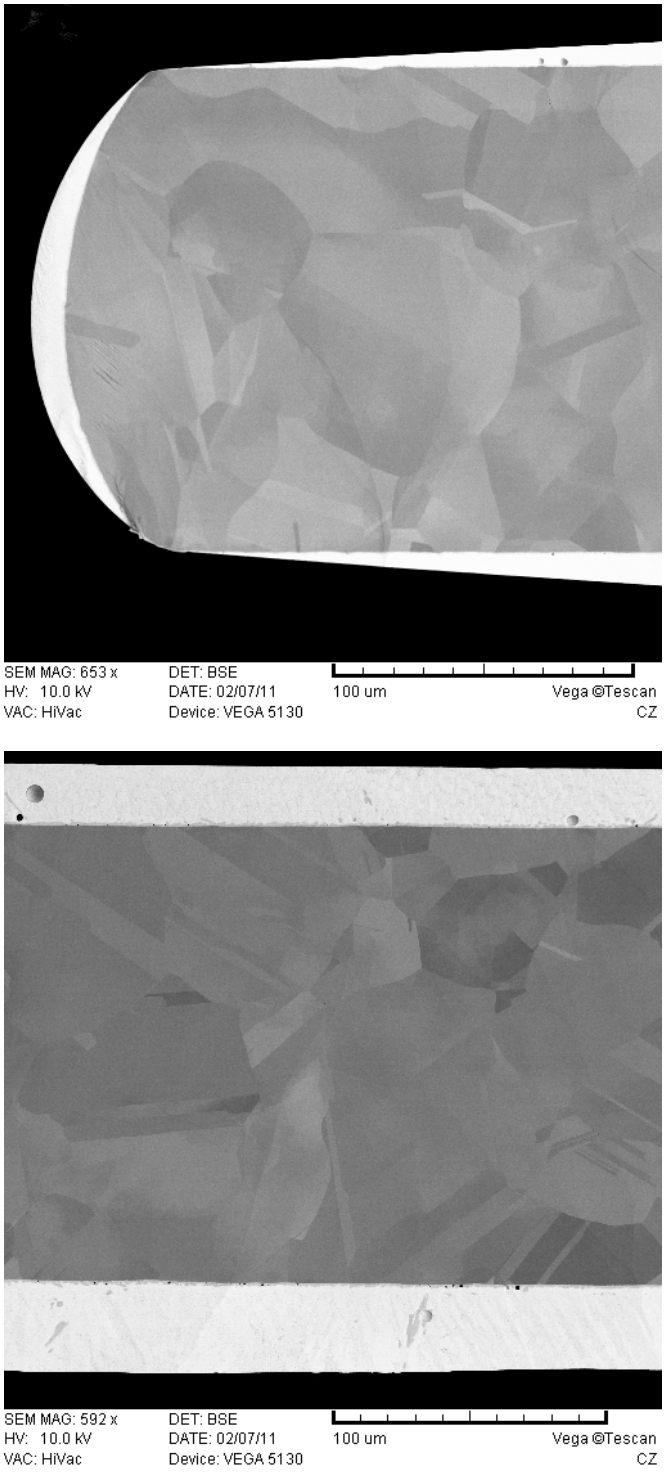


Fig. 13: Cross-section of a copper/tin connector on a solar cell

2.1.2. Preparation of metals

NICKEL / COPPER ON STEEL

Aim of analysis

Examination of the layer and interface structure

Preparation problem

Compared to the substrate, the layers are very soft and become smeared with dirt during mechanical polishing. The layered structures and the interfaces are then not completely visible.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP

> Affix a thin cover glass to the sample surface
> Affix the specimen to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
> Sand the sample's cross-section surface using 9 µm diamond foil to obtain a smooth cross-section surface.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	4 h

Result

The cross-section is very clean and flat. The structure of the layers and of the steel substrates are easily visible. Many pores can been seen at the interface to the steel.

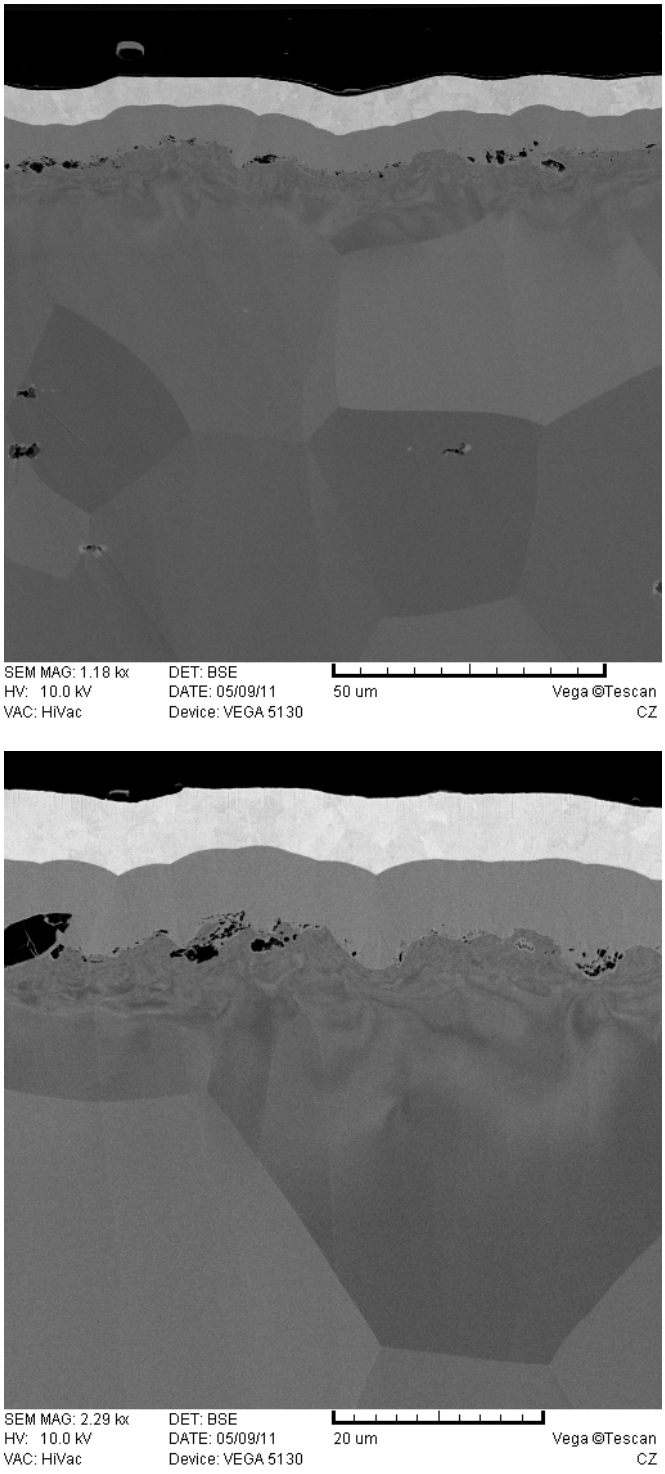


Fig. 14: Cross-section of a steel sample, coated with copper and nickel

CORRODED STEEL

Aim of analysis

The sample has been oxidized at 400 °C for 24 h. Cross sectional sample preparation is intended to facilitate examination of the layer of corrosion on the steel surface.

Preparation problem

The layer of corrosion is sensitive to water and adheres very poorly to the steel surface. Mechanical polishing is excluded for this reason.

Preparation conditions

Mechanical pre-preparation:

> A Si wafer has been glued to the sample surface to protect the sensitive layer of corrosion during preparation.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	5 h

Result

The cross-section surface is very large at 6 mm long and 0.9 mm deep. The surface is smooth and clean. The layer of corrosion is easily visible and can be evaluated. Significant cavities between the layer and the steel substrate can be seen.

The images show a steel substrate coated with KCl and ZnCl2.

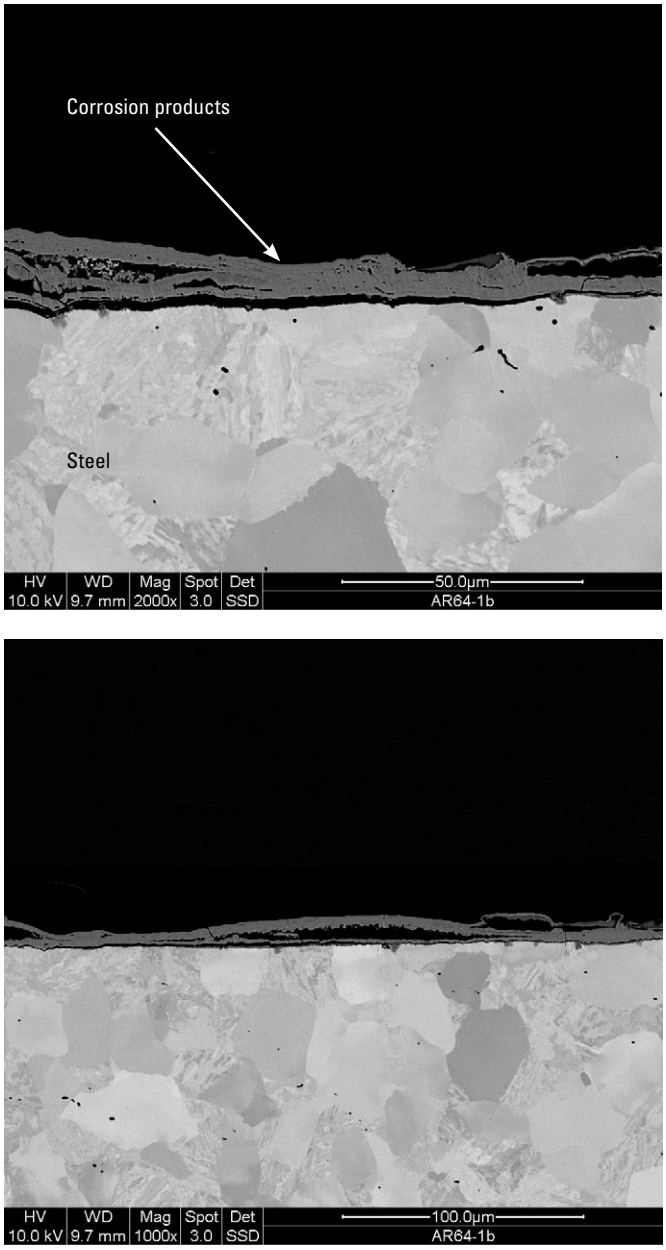


Fig. 15: Cross-section of KCl and ZnCl2 on steel. The sample has been oxidized at 400 °C for 24 h (Courtesy of Torbjörn Jonsson, Chalmers University of Technology, Gothenburg, Sweden).

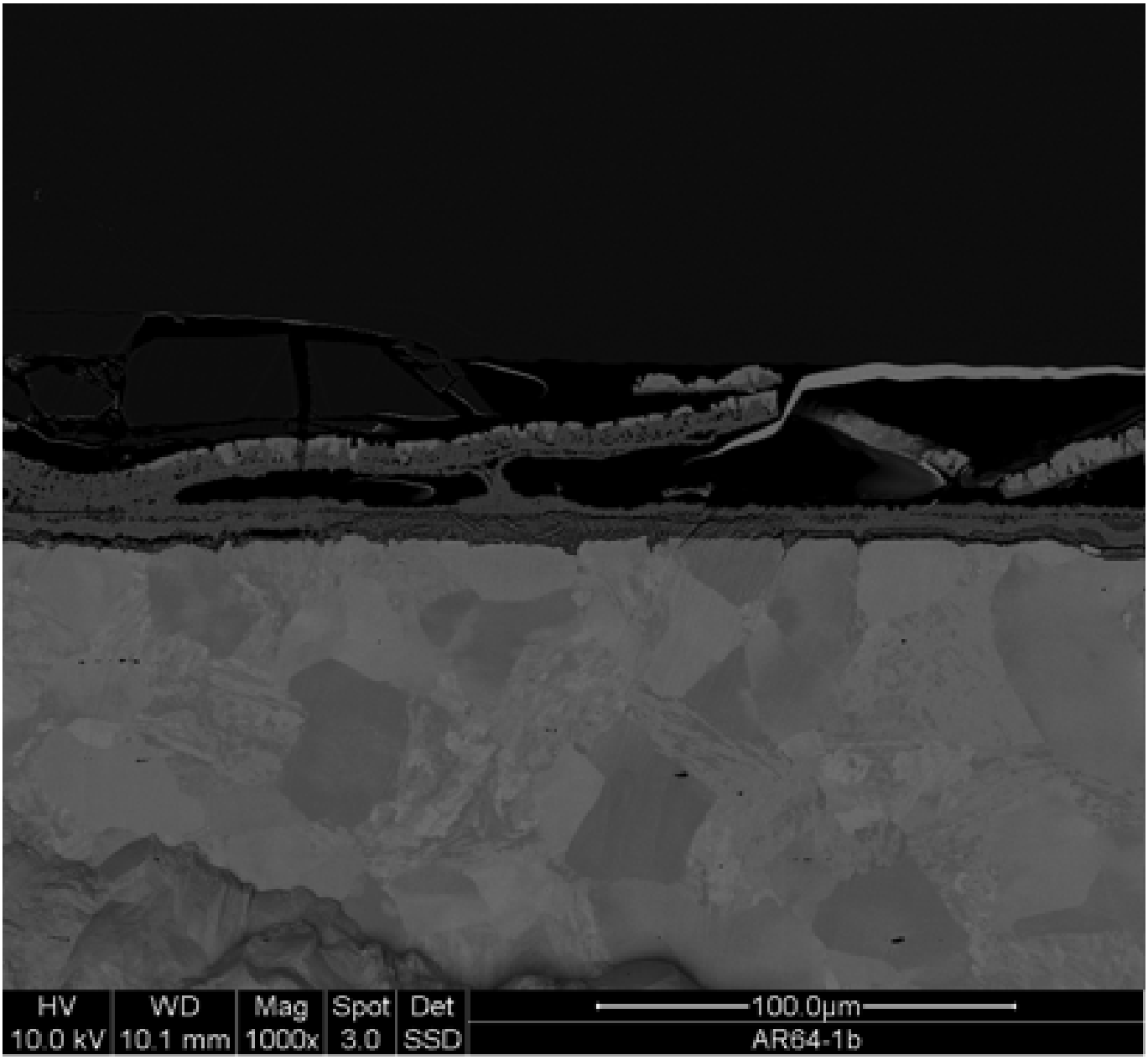


Fig. 16: Cross-section of KCl and ZnCl2 on steel. The sample has been oxidized at 400 °C for 24 h (Courtesy of Torbjörn Jonsson, Chalmers University of Technology, Gothenburg, Sweden)

ZINC ON STEEL

Aim of analysis

Cross sectional investigation of the zinc layer on steel

Preparation problem

Zinc is very soft compared to steel and would become smeared with dirt during mechanical polishing. This would make an examination of the layered structure impossible.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- A Si wafer has been glued to the sample surface to protect the zinc layer
- Affix the specimen to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sawing the sample
- Polish using 9 µm diamond foil

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	6 h

Result

The cross-section shows the structure of the zinc layer. The layer is very clean and smooth. No preparation artifacts are visible. The grain structure of the zinc is clearly visible. Pores and partial inclusions can be found in the layer.

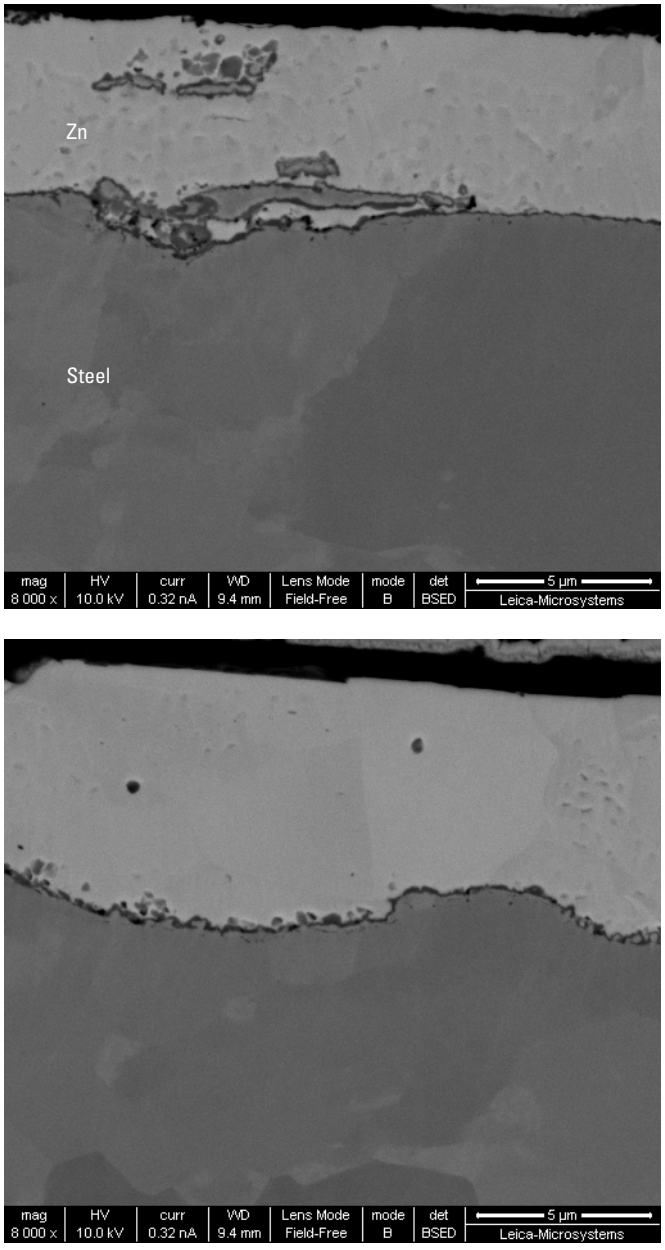


Fig. 17: Cross-section of a zinc layer on steel with a clearly visible grain structure.

ZINC ON STEEL

Aim of analysis

Cross sectional sample preparation for EBSD examination

Preparation problem

Difference in hardness between the layers and the substrate material

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Two samples were glued together on the layer side to protect the zinc layers during preparation.
- Affix the specimen to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- The cross-section of the sandwich sample was sanded using 9 µm diamond foil to obtain an even cross-section surface.

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	13 h

Result

Both samples were able to be sectioned via ion beam etching.

Two different zinc layers are visible. The Kikuchi diffraction patterns show the quality of the prepared sample surfaces.

The samples have been protected optimally during preparation, thanks to the “face to face” style of adhesion. The long etching time is calculated from the thickness of the steel substrate.

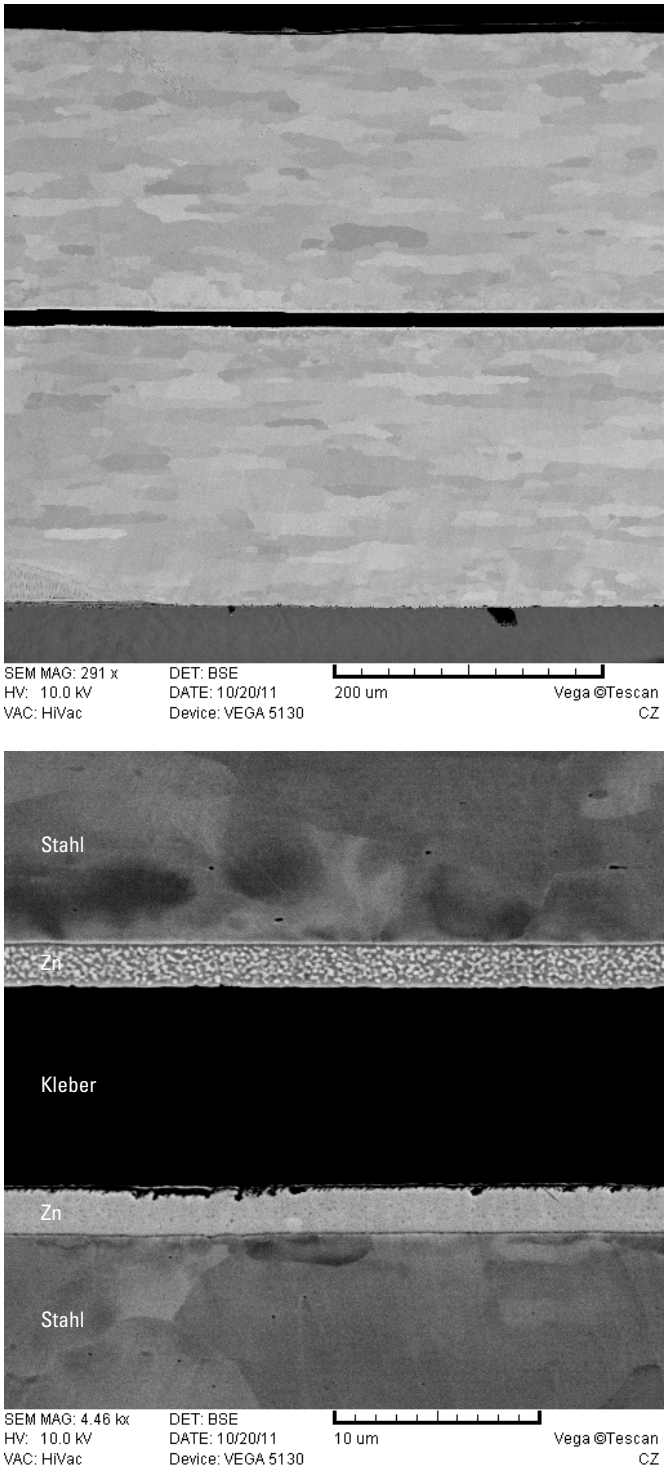
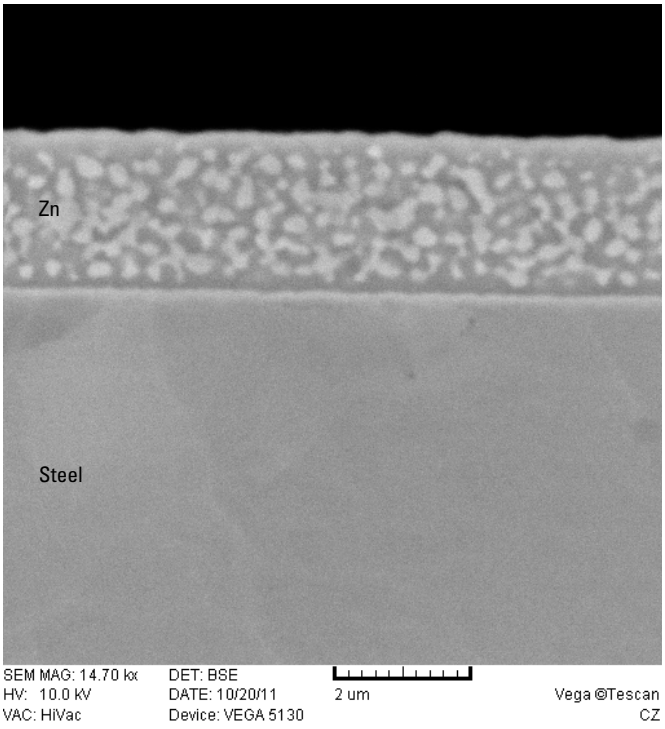
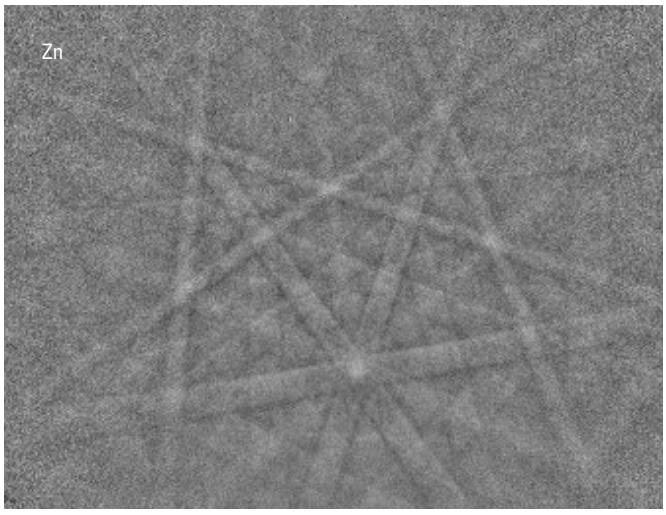
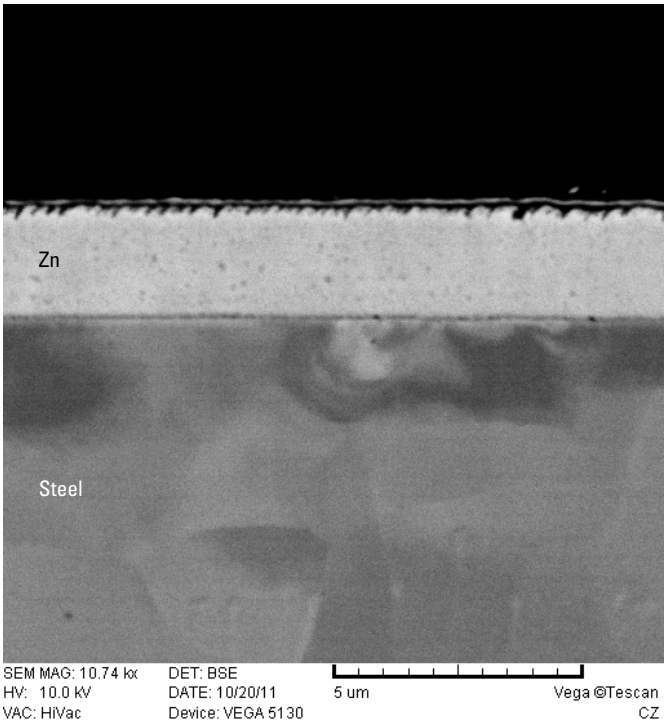


Fig. 18: Cross-section of two steel samples coated with Zinc







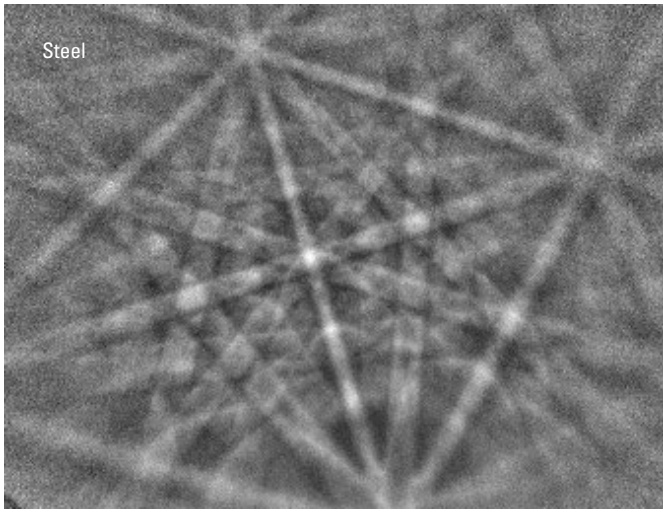


Fig. 19: Zinc layers on steel with the corresponding Kikuchi diffraction patterns

TUNGSTEN CARBIDE ON STEEL

Aim of analysis

Compared to the FIB (focused ion beam) technique, a significantly larger sample area should be prepared using cross sectional sample preparation. The pore structure in the tungsten carbide, in particular, was of interest.

Preparation problem

The pores are usually clogged during mechanical polishing and are not visible in the scanning electron microscope.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the cross-section of the sample using 9 µm diamond foil
- Sand the 45° wedge of the cross-section surface using 9 µm diamond foil to reduce the sample material to be etched as well as the etching time

ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	6 h

Result

It was possible to prepare a surface over 2 mm long and several 100 µm deep. The images show very small pores and larger filled pores that have been made visible by this preparation for the first time.

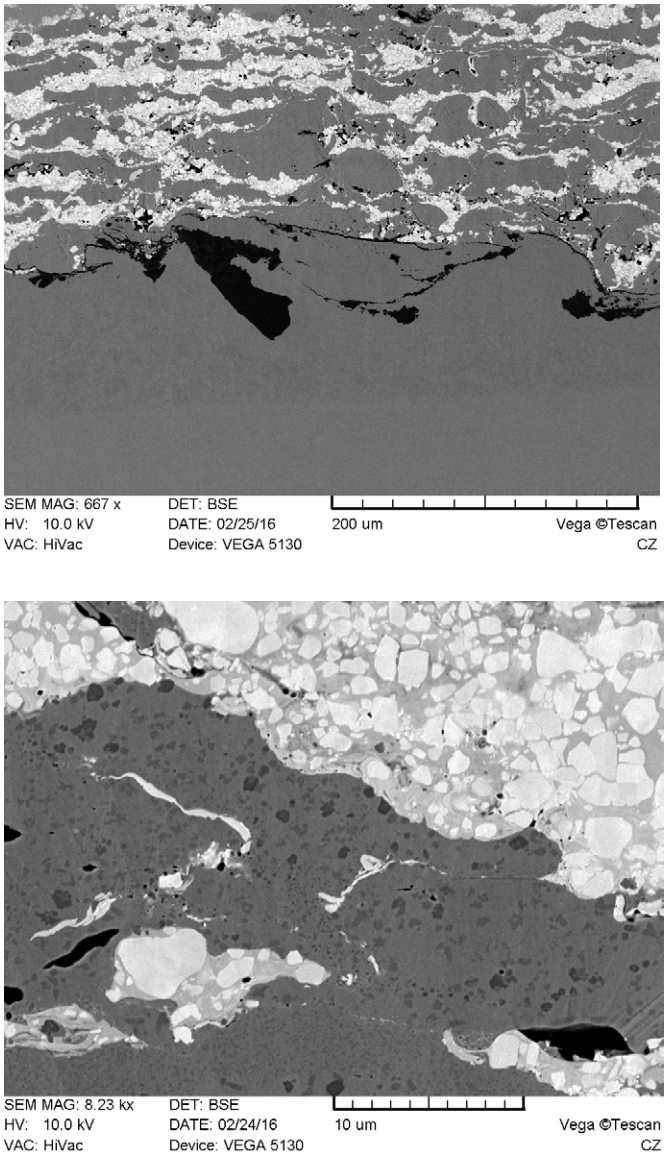


Fig. 20: SEM cross-section image of WC-coated steel with many pores in the tungsten carbide

GALVANIZED STEEL WITH LAYERS OF PAINT

Aim of analysis

Cross sectional investigation of the metal/paint interface

Preparation problem

The adhesion of the first layer of paint to metal is of major importance. Very diligent sample preparation is necessary to avoid delamination.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the cross-section of the sample using 9 µm diamond foil
- 45° wedge of the cross-section surface using 9 µm diamond foil for reducing the etching time

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	3 h

Result

The sample has been etched from the substrate side.

The cross-section shows the entire layer structure on the steel. The interfaces are easily visible. The sample surface is smooth and clean. No preparation-related defects are visible. Only minimum delamination are visible between the zinc layer and the first layer of paint.

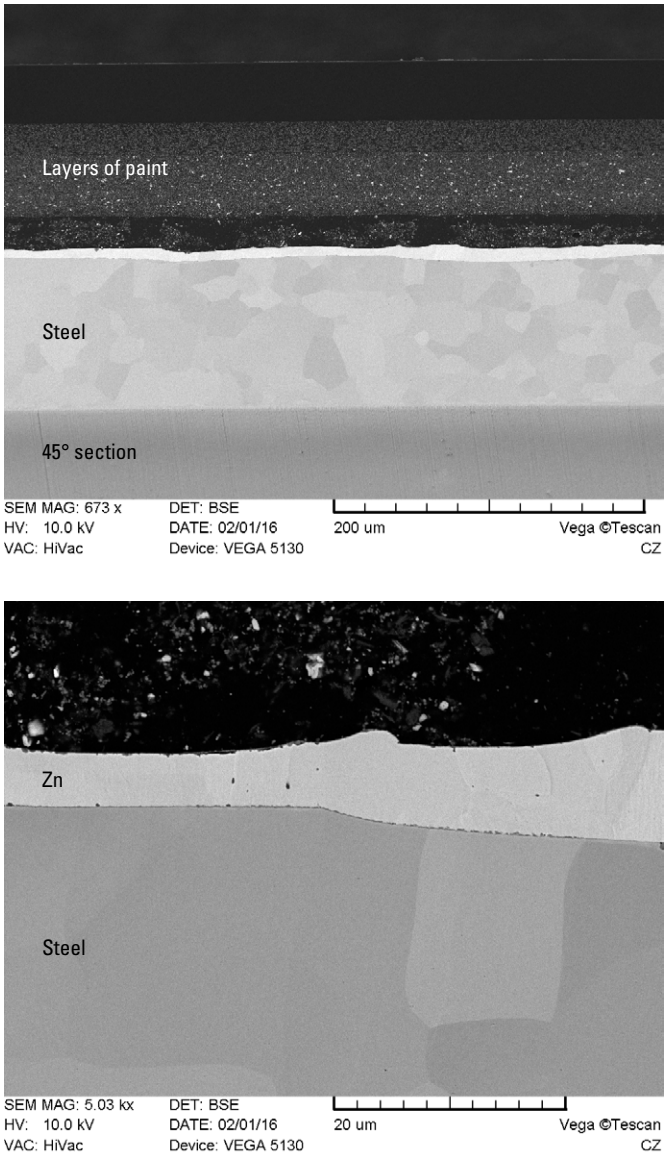


Fig. 21: Cross-section of galvanized steel with layers of paint

TIN LAYER ON COPPER FOIL

Aim of analysis

Cross-section for examining the tin layer and its interface to the copper

Preparation problem

Both materials are very soft and are very difficult to polish mechanically.

Preparation conditions

Mechanical pre-preparation:

> Reducing the size of the sample using a razor blade

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	2 h 30 min

Result

It was possible to section the sample completely. The structure of the tin layer is clearly visible. The layer indicates large cracks. Cracks and pores are also visible on the interface area to the copper.

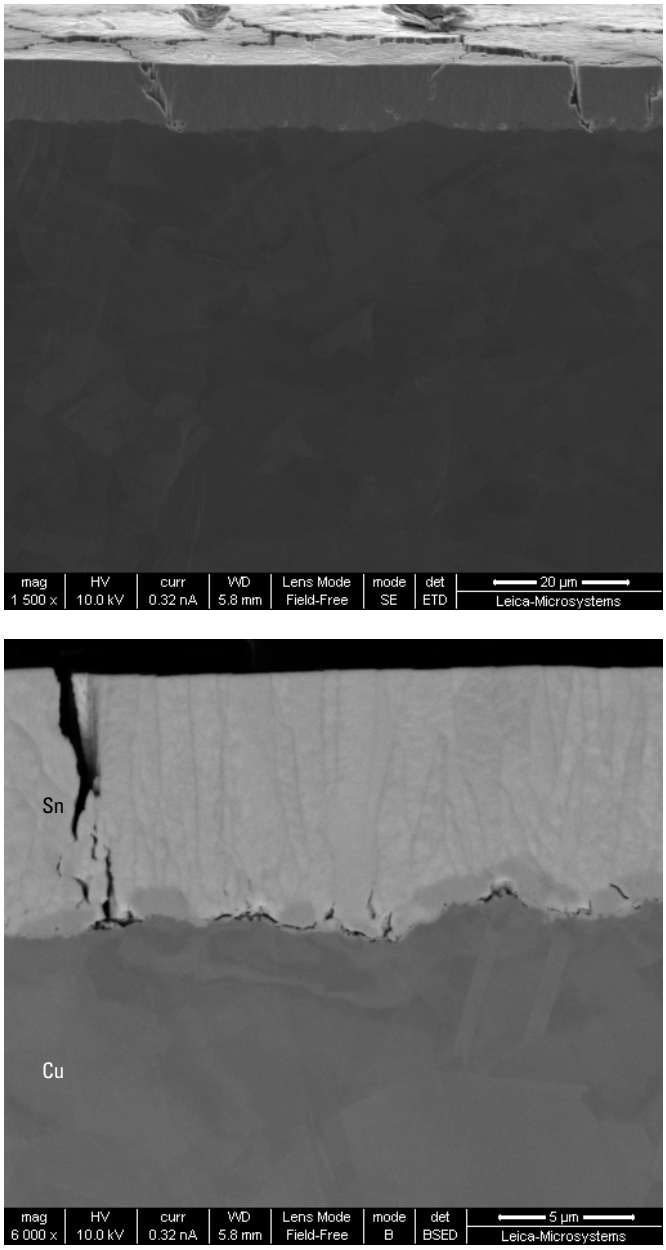


Fig. 22: Cross-section of a tin layer on copper with clearly visible cracks in the layer and in the interface area

CONNECTOR FROM THE AUTOMOBILE INDUSTRY WITH LASER WELDED CONNECTION

Aim of analysis

Cross-section for the examination of the laser welded connection of tin-coated copper

Preparation problem

The sample cannot be prepared mechanically due to its shape and the soft material.

Preparation conditions

Mechanical pre-preparation using the Leica EM TXP:

> Cut the regions of interest out
> Affix the sample to an Al holder
> Sand the sample using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	9 h

Result

It was possible to prepare the laser-welded area of the plug successfully using slope cutting.

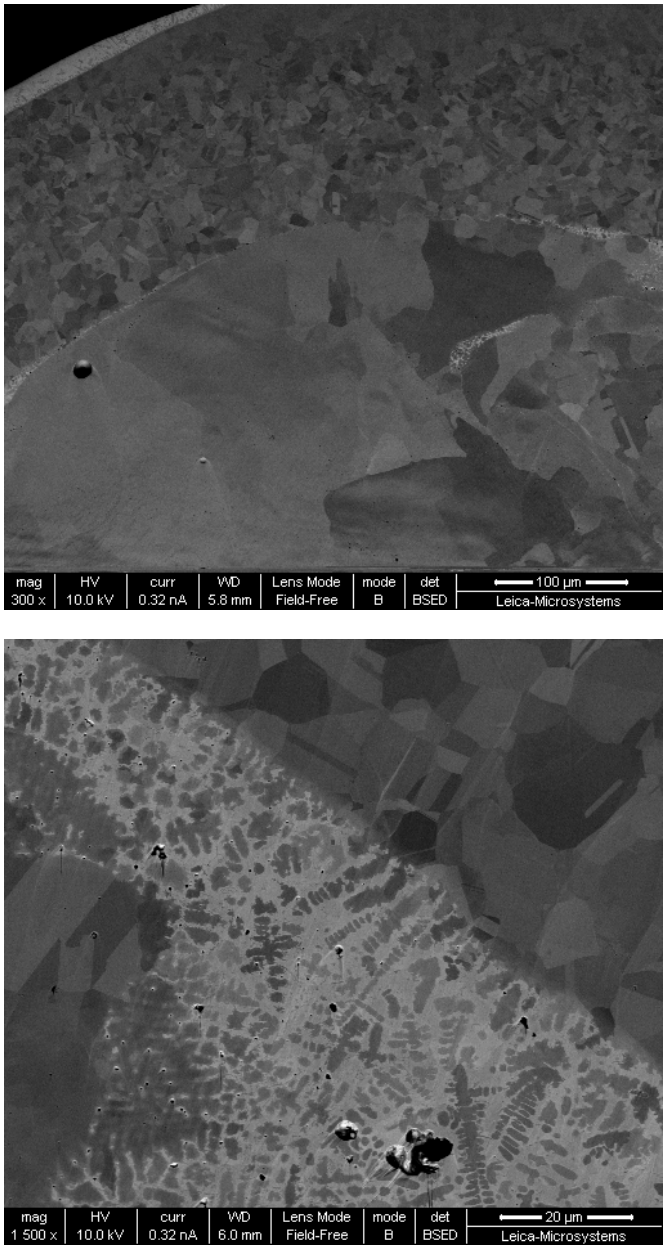


Fig. 23: Cross section of a connector used in automotive industry

DIAMOND/AL COMPOSITE

Aim of analysis

Examination of the interface structure between the diamond particles and the Al matrix

Preparation problem

Due to the extremely high difference in hardness between Al and diamond, the preparation is very difficult. Mechanical polishing is almost impossible.

Preparation conditions

Mechanical pre-preparation:

> Because of the extreme hardness, mechanical pre-preparation was only possible in a very limited sense. An attempt was made to produce a sample that was as square as possible.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	6 h

Result

Ion milling made it possible to prepare a relatively smooth sample surface. The comparison to a mechanically polished surface can be seen in Fig. 25 a and b. The sample has been tilted by 45° during the REM examination to be better able to compare the topography.

The ion-etched sample (Fig. 25 c) is largely free of artifacts and allows the examination of the interface of the diamond/Al composite /2/.

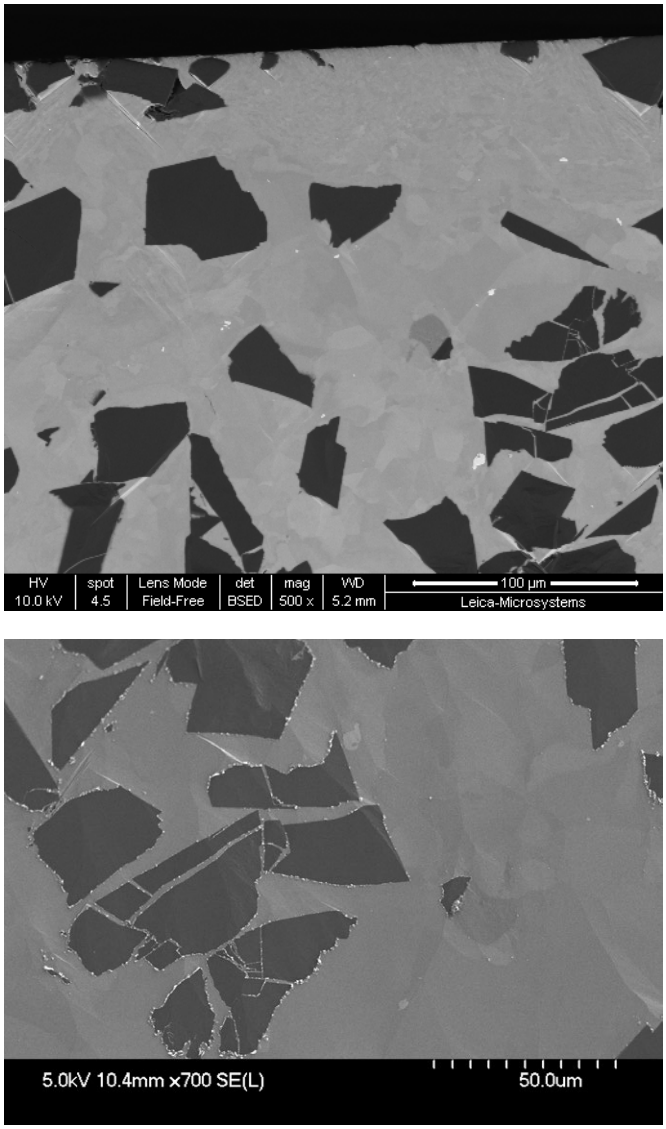


Fig. 24: Querschnitt eines Diamant / Al Verbundes.

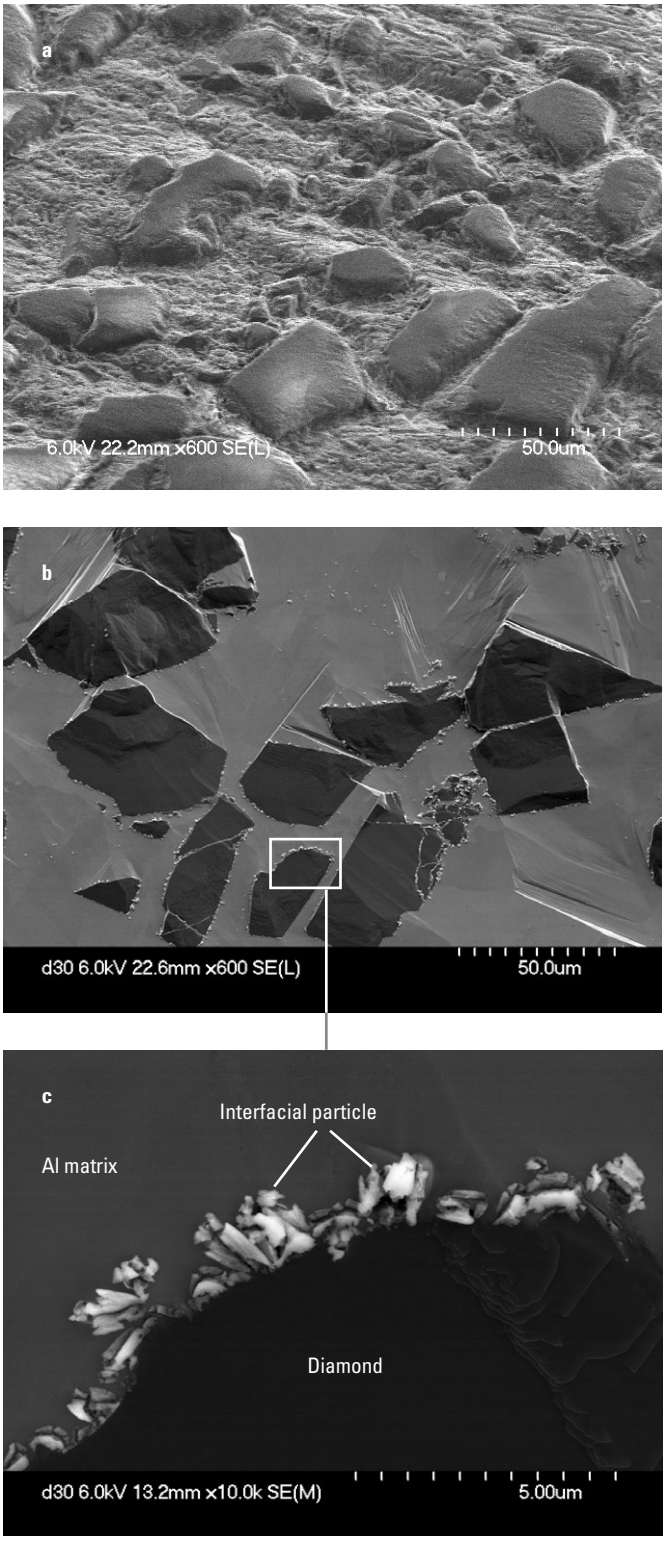


Fig. 25: SEM images of a diamond/Al composite:
(a) Rough surface as a result of mechanical polishing using the tripod,
(b) nearly perfect surface as a result of ion milling using the Leica EM TIC 3X,
(c) detail enlargement with interfacial particles at the interface.
The sample was tilted by 45° in Fig. a and b to compare the surface roughness.

NB3SN SUPERCONDUCTOR

Aim of analysis

Cross-section of the wire to investigate the structure of the Nb3Sn superconductor

Preparation problem

The shape of the wire makes cross sectional sample preparation difficult.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Embed the wire between two Si wafers using epoxy resin
- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the sample using Leica EM TXP to obtain a smooth cross-section surface

ION MILLING	
Acceleration voltage	6 kV
Gun current	2 mA
Milling time	5 h

Result

The cross-section of the superconductor wire is smooth and clean. This is mainly due to its embedding before the ion milling process.

The structures of the Nb3Sn and Cu matrix are easily visible.

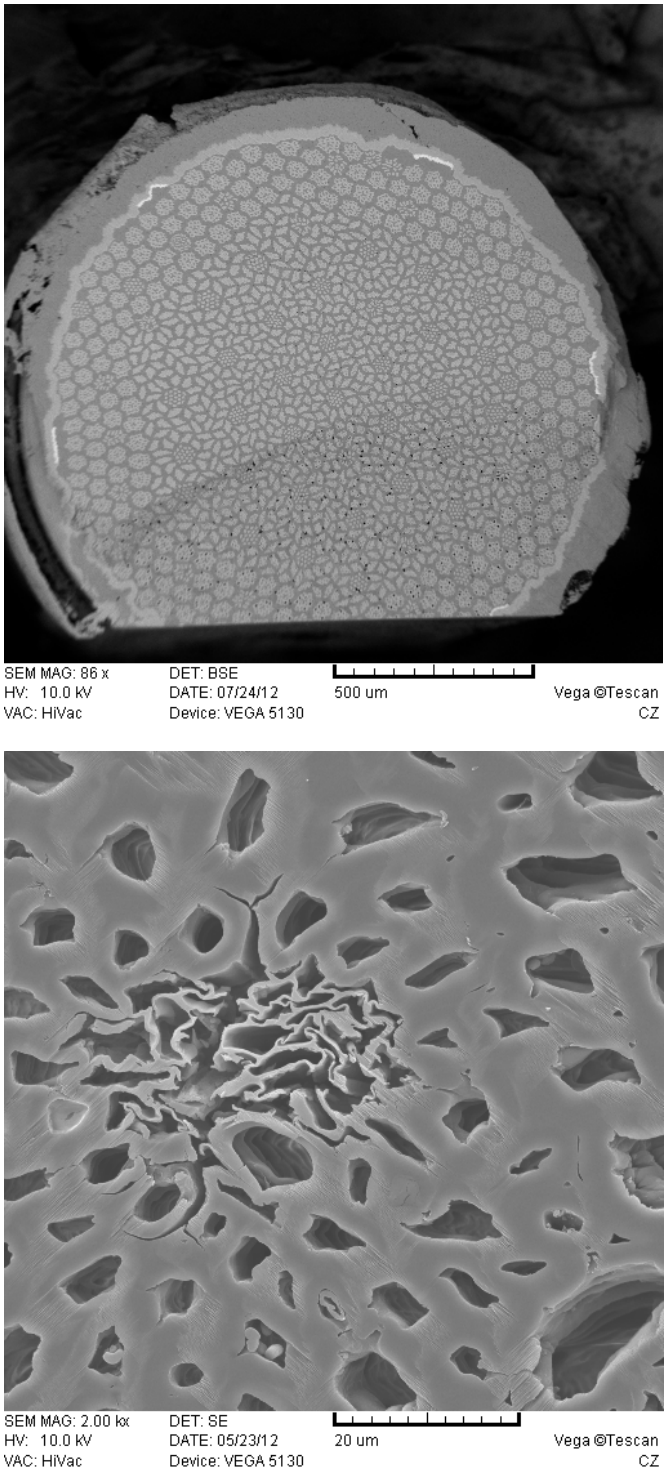


Fig. 26: Cross-section of a Nb3Sn superconductor

2.1.3. Preparation of stones

Rock samples usually have an irregular shape. They must be prepared using mechanical pre-preparation in such a way that adjustment to the Leica EM TIC 3X mask is possible. A wedged mask is often used.

CARBONATE MINERAL

Aim of analysis

Cross-section for the examination of the pore structure in a carbonate mineral

Preparation problem

The carbonate mineral sample is porous and brittle. The fine pore structure can become clogged during conventional mechanical grinding.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
> Sand the product using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	3 h

Result

The surface of the carbonate mineral sample is very smooth. The pore structure is easily visible. Even very small pores can be seen. Ion milling is consequently very well suited for preparing porous rock samples.

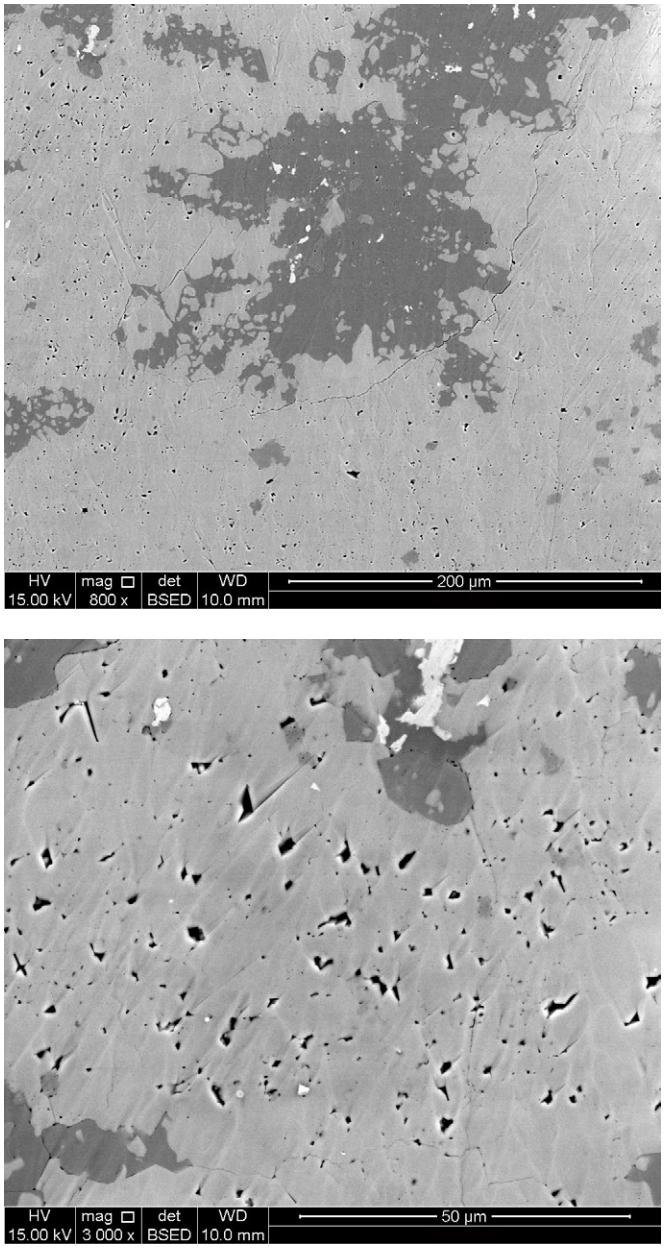


Fig. 27: Cross-section of a carbonate mineral with a pronounced pore structure (Repsol)

OIL SHALE

Aim of analysis

Cross-section of the oil shale sample for the examination of the organic and inorganic compounds in the sample

Preparation problem

The oil shale contains organic compounds and is very brittle.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter
> Sand the cross-section surface using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	4 h

Result

The cross-section is very clean and smooth. The individual components like clay, pyrite and quartz are easily visible.

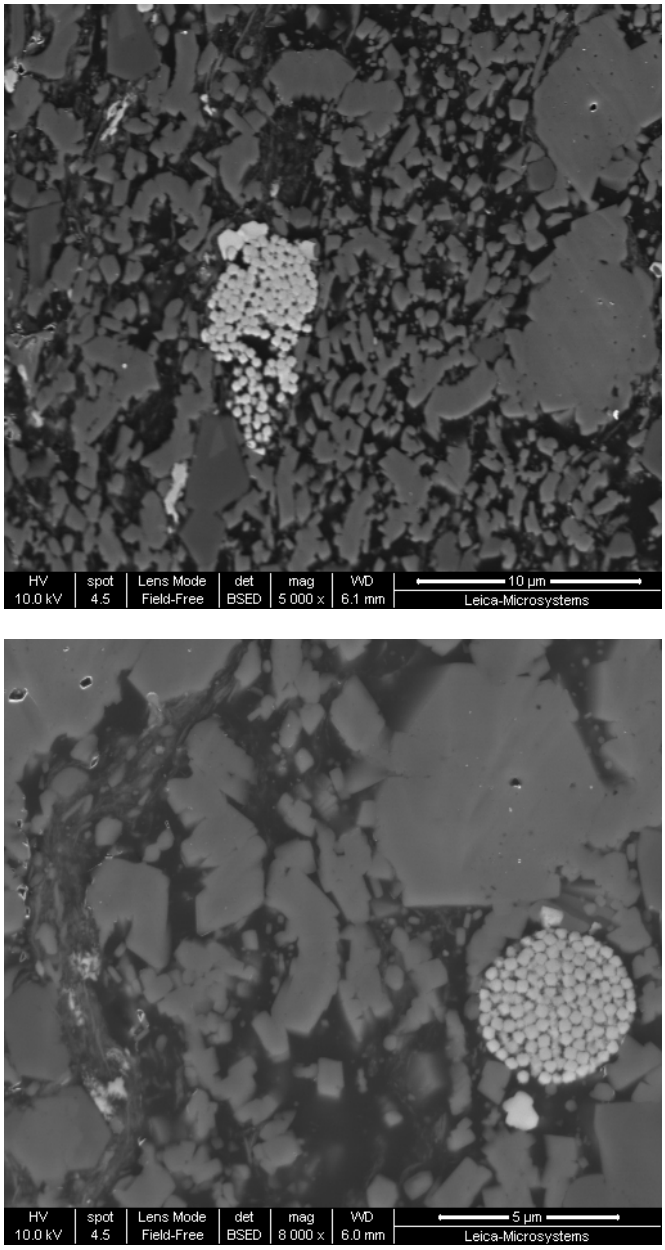


Fig. 28: Ion beam slope section of oil shale with clay, pyrite and quartz

SHALE

Aim of analysis

Shale sample structure, organic and inorganic components

Preparation problem

The sample has components with different levels of hardness and is brittle.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
> Sand the product using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	3 h

Result

It was possible to prepare a very smooth surface without any damage to the structure.

The images show a crystallized matrix that is rich in clay with debris exclusions.

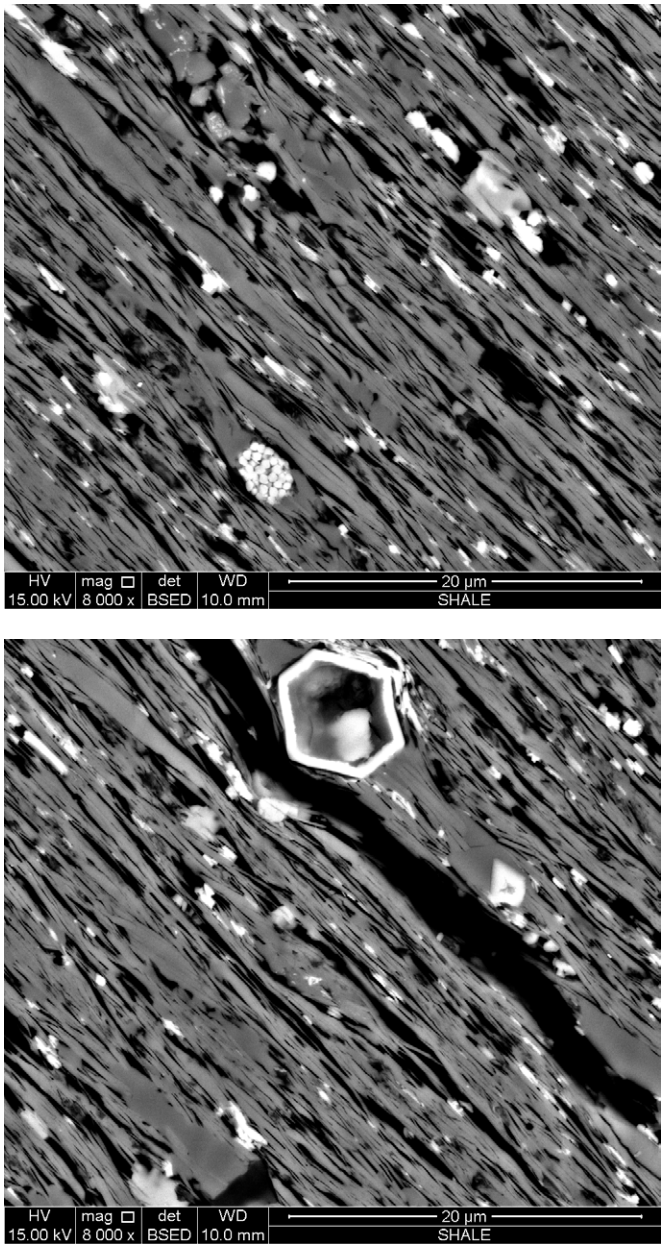


Fig. 29: SEM image using Backscattered Electron (BSE) of ion-milled surface which provides a general overview of the abundant, recrystallized clay-rich matrix surrounding detrital constituents. Detailed observation of the general abundance of organic matter (Repsol).

PAINTED CONCRETE

Aim of analysis

Cross sectional sample preparation for the examination of the interface between the paint and concrete

Preparation problem

The soft layer of paint on the hard concrete is very difficult to polish mechanically.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
> Sand the product using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	6 h

Result

The cross-section is clean and smooth. No preparation effects are visible. Organic binders and inorganic pigment can clearly be seen. The interface between the paint and the concrete can be evaluated.

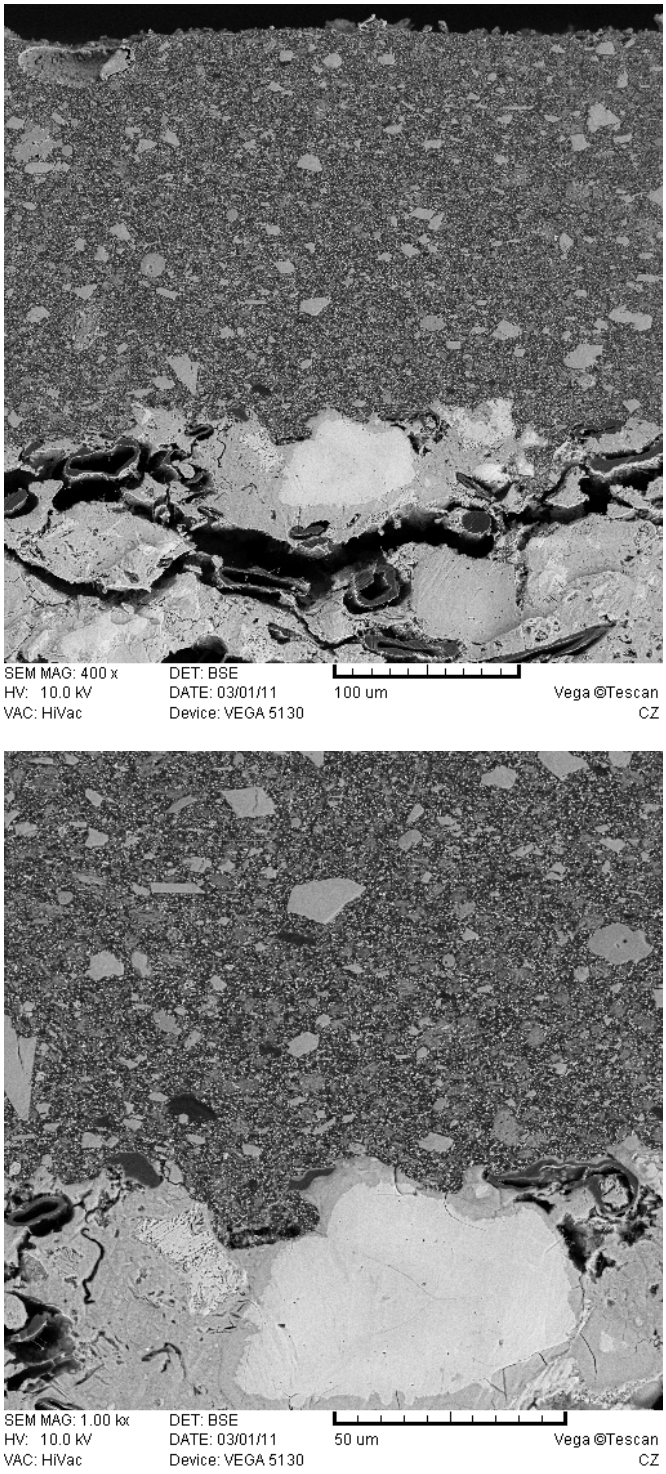


Fig. 30: Cross-section of painted concrete

2.1.4. Preparation of paper, wood, rubber and other materials

Pre-preparation is usually necessary for this group of materials to produce a sample shape that permits an optimal adjustment of the sample to the mask. In this section, applications are presented that represent the limit range for ion milling applicability.

DOUBLE-COATED PAPER

Aim of analysis

Cross-section to display the coating structure

Preparation problem

Paper is a material that is difficult to prepare

Preparation conditions

Mechanical pre-preparation:

> Reduce the size of the sample using a razor blade

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	2 h

Result

The cross-section through the complete paper sample is very clean and smooth. Isolated small etching structures are visible.

You can see the grain structure of both coatings and the interfaces very well.

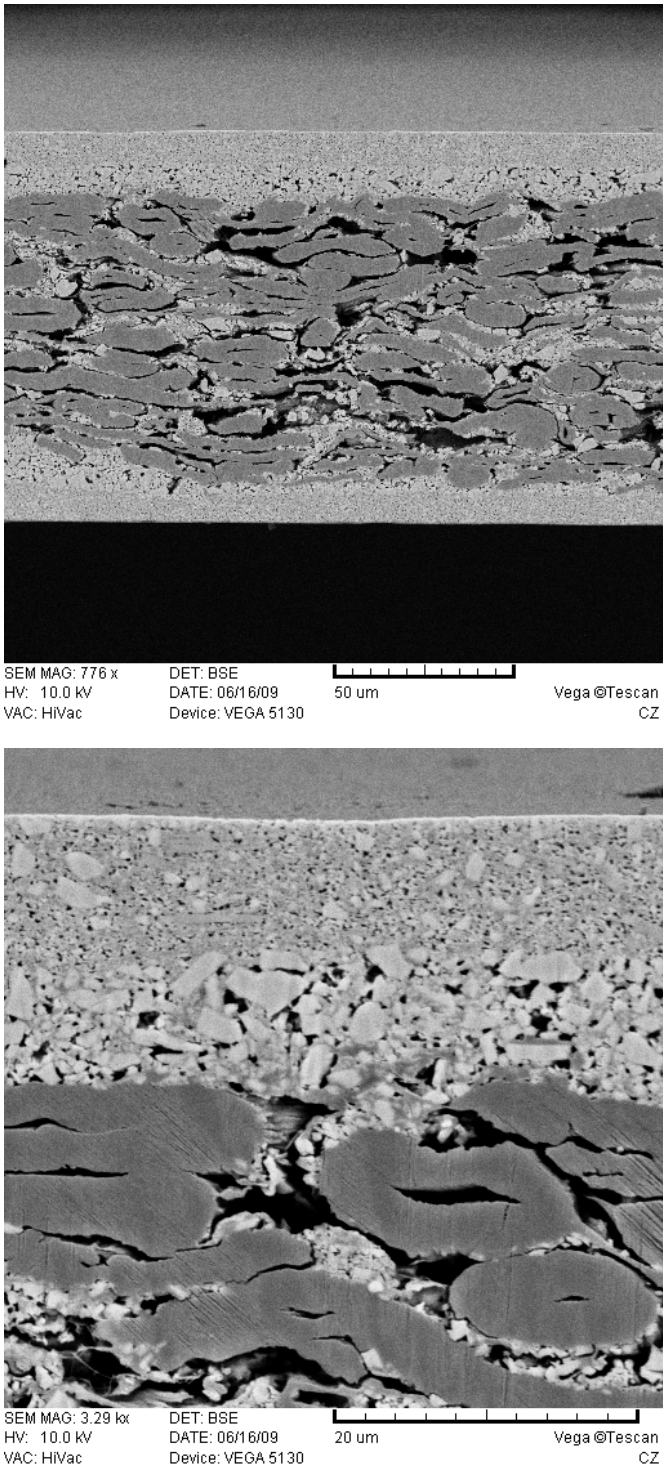


Fig. 31: Cross-section through double-painted paper

LAPPING FOIL (SIC)

Aim of analysis

> Examination of the structure of SiC lapping foil
> Investigation of problems when sanding with 2400 grit paper.

Preparation problem

The lapping foil has a very heterogeneous structure which makes preparation using conventional methods extremely difficult.

Preparation conditions

Mechanical pre-preparation:

> Cut the sheets into small pieces using a scissors

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	2 h – 2 h 10 min

Result

It was possible to use slope cutting to section two pieces of lapping foil with different grain sizes starting from the back side. It was possible to achieve a very good result this way. The entire vertical structure of the lapping foil is easily visible. It is possible to draw conclusions regarding the size and shape of the SiC grains as well as their distribution.

The 2400 grit paper has large cavities.

The surface quality of the processed sample deteriorates during sanding with 2400 grit paper. The reason for this can be traced back to the cavities.

Slope cutting is well suited for quality control in the event that lapping foil is used.

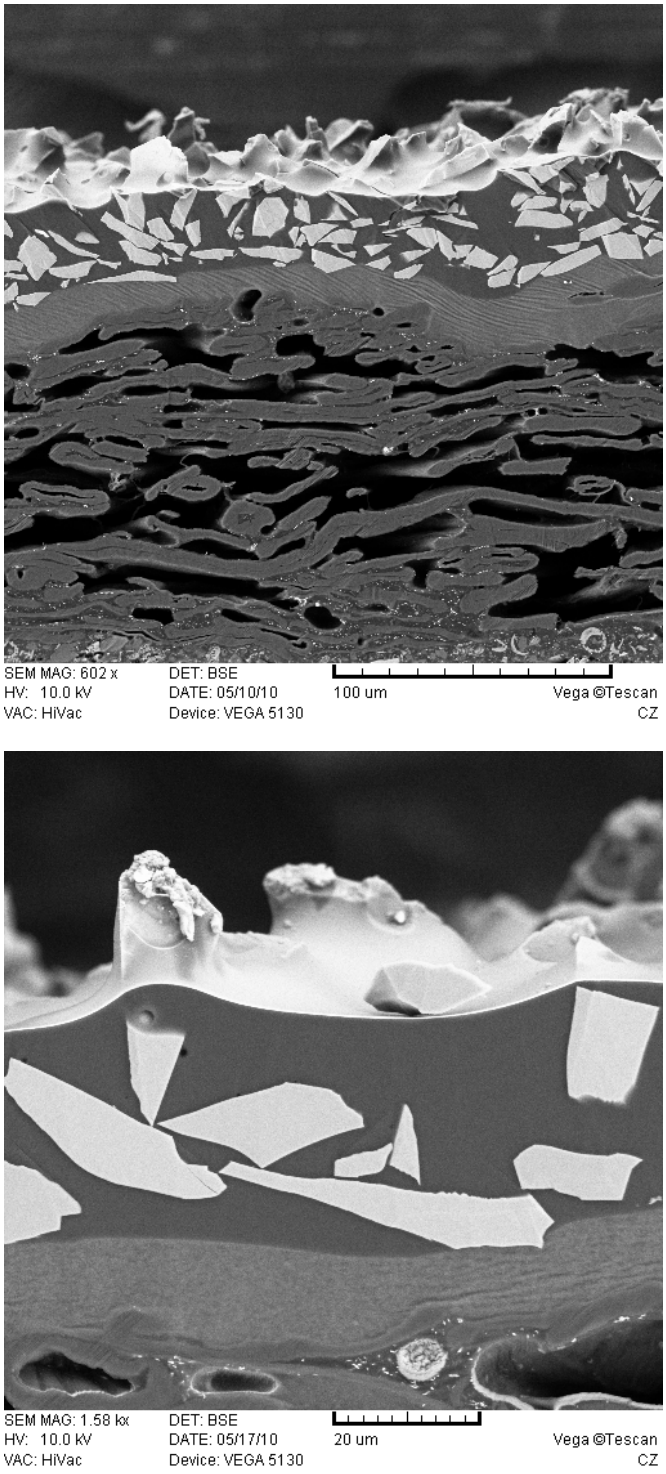
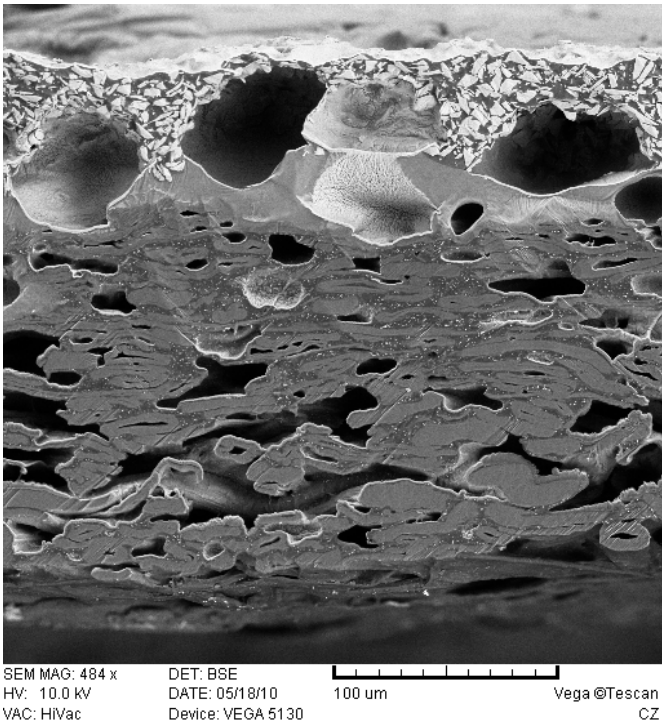


Fig. 32: Cross-section of 1200 grit SiC lapping foil



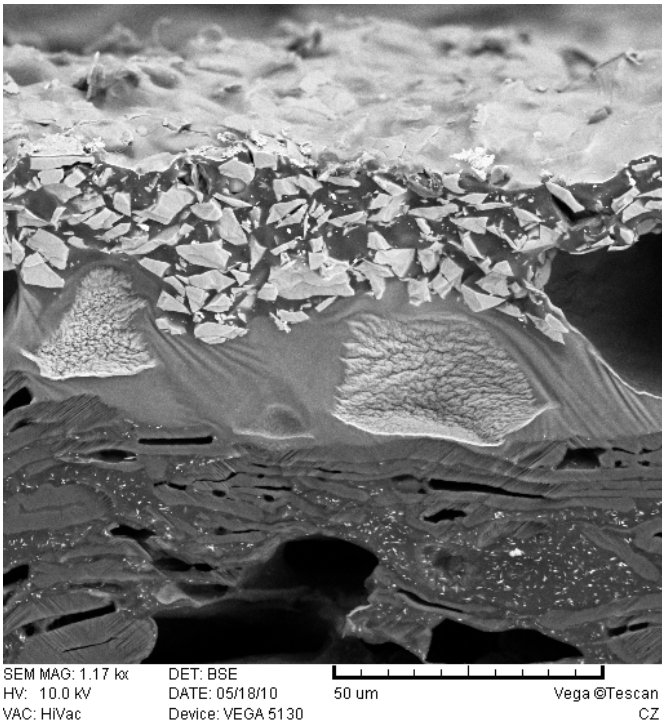


Fig. 33: Cross-section of 2400 grit SiC lapping foil with large cavities

VENEER

Aim of analysis

Cross-section for the examination of the veneer structure

Preparation problem

Veneer is very difficult to prepare due to its large pores.

Preparation conditions

Mechanical pre-preparation:

> Reduce the sample size and produce a rectangular sample using a razor blade.

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	3 min

Result

The cross-section of the veneer samples are smooth and clean. Smaller etching structures result from the large cavities that have an affect similar to that of the mask edges.

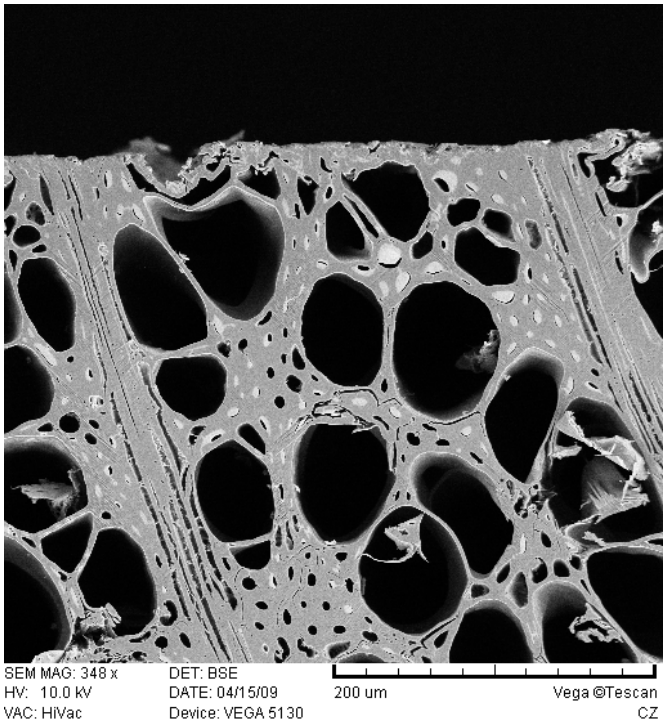


Fig. 34: Cross-section of veneer

BAMBOO

Aim of analysis

Cross-section to display the internal structure of bamboo

Preparation problem

Bamboo is an exotic sample for ion milling. One question raised here was whether the sample would experience thermal damage.

Preparation conditions

Mechanical pre-preparation:

> Reducing the size of the sample

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	5 h

Result

The cross-section looks very good. You can immediately see the bizarre internal structure of the bamboo. Almost no preparation effects are apparent. As a result, the sample is not subjected to excessive thermal stress under the aforementioned preparation conditions.

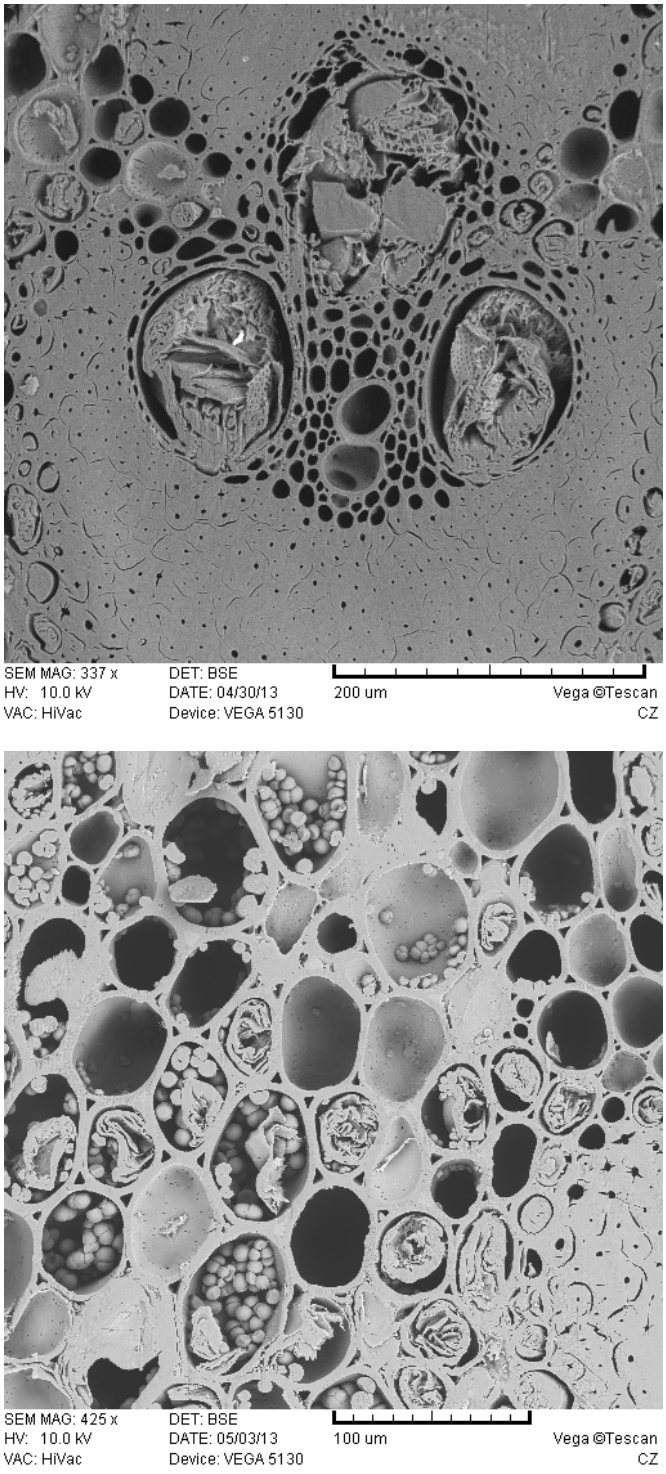


Fig. 35: Cross-section of bamboo

SILVER ON GLASS (REAR SCREEN HEATING ELEMENT)

Aim of analysis

> Interface between glass and the layer of silver
> Pore structure in silver

Preparation problem

The extreme difference in hardness between glass and silver makes mechanical polishing impossible.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> A thin glass plate has been glued to the sample surface to protect the layer of silver.
> The sample has been glued to a Leica EM TIC 3X Al holder and clamped in the Leica EM TXP using an adapter.
> The sample was ground using 2 µm diamond foil to obtain a smooth cross-section surface.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	3 h

Result

It was possible to prepare a perfect sample cross-section. The structural details are easily visible.

The layer of silver has the expected pore structure. An intermediate layer has formed on the interface to the glass.

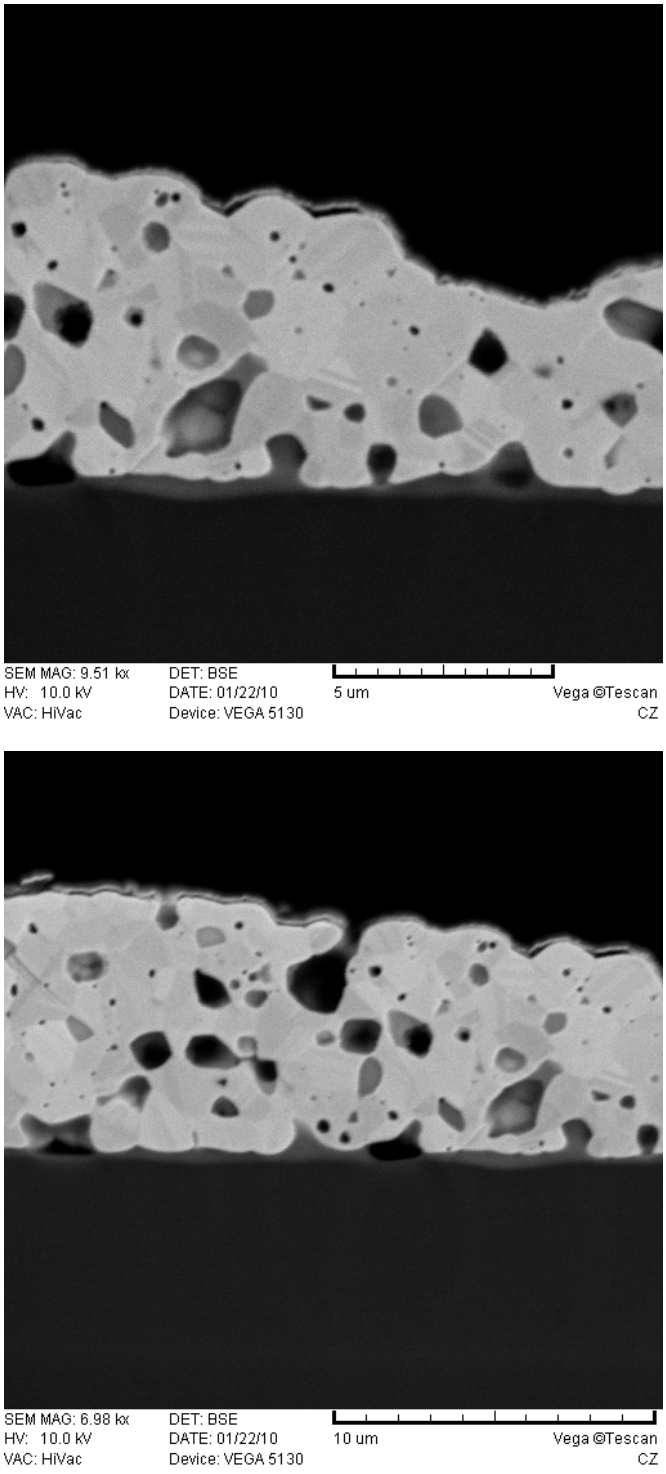


Fig. 36: Cross section from sections of rear screen heating element (automotive industry)

CERAMICS COATED WITH SILVER AND COPPER ON BOTH SIDES

Aim of analysis

Cross-section for the examination of the pore structure in the silver layer

Preparation problem

Extreme difference in hardness between silver, copper and ceramic

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- A thin glass plate was glued to the top layer to protect it.
- The sample has been glued to a Leica EM TIC 3X Al holder and clamped in the Leica EM TXP using an adapter.
- The sample has been sanded using 9 µm diamond foil to obtain a smooth cut face.

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	6 h

Result

It was possible to produce a section through the entire layer structure. The images show the cross-section of the ceramic substrate with a fine-pored layer of silver and a large-pored layer of copper. In the bottom image, the interface between the layers of silver and copper is visible. The layer of silver has an unwanted, fine pore structure.

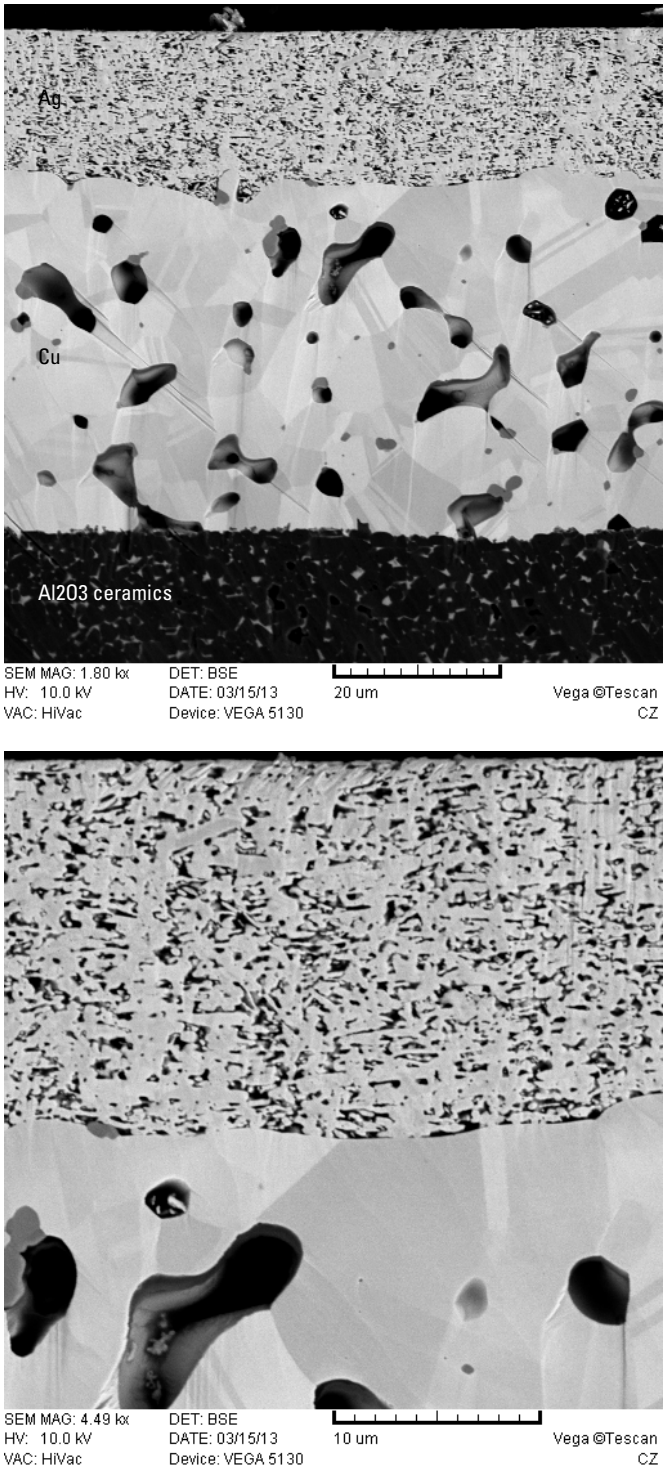


Fig. 37: Cross-section of a fine-pored layer of silver and a large-pored layer of copper on a Al2O3 ceramic substrate

TIMING BELT

Aim of analysis

Cross-section to display the interface between the glass cords and the embedding

Preparation problem

The glass fibers are extremely hard and have a low sputter rate.

Preparation conditions

Mechanical pre-preparation:

> Preparation of a virtually rectangular sample shape

ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	14 h

Result

It was possible to generate a clean cross-section of the interface between the glass fibers and the embedding. The overview image shows the glass fibers and the embedding with polyamide fibers. The etching time was very long due to the low sputter rate.

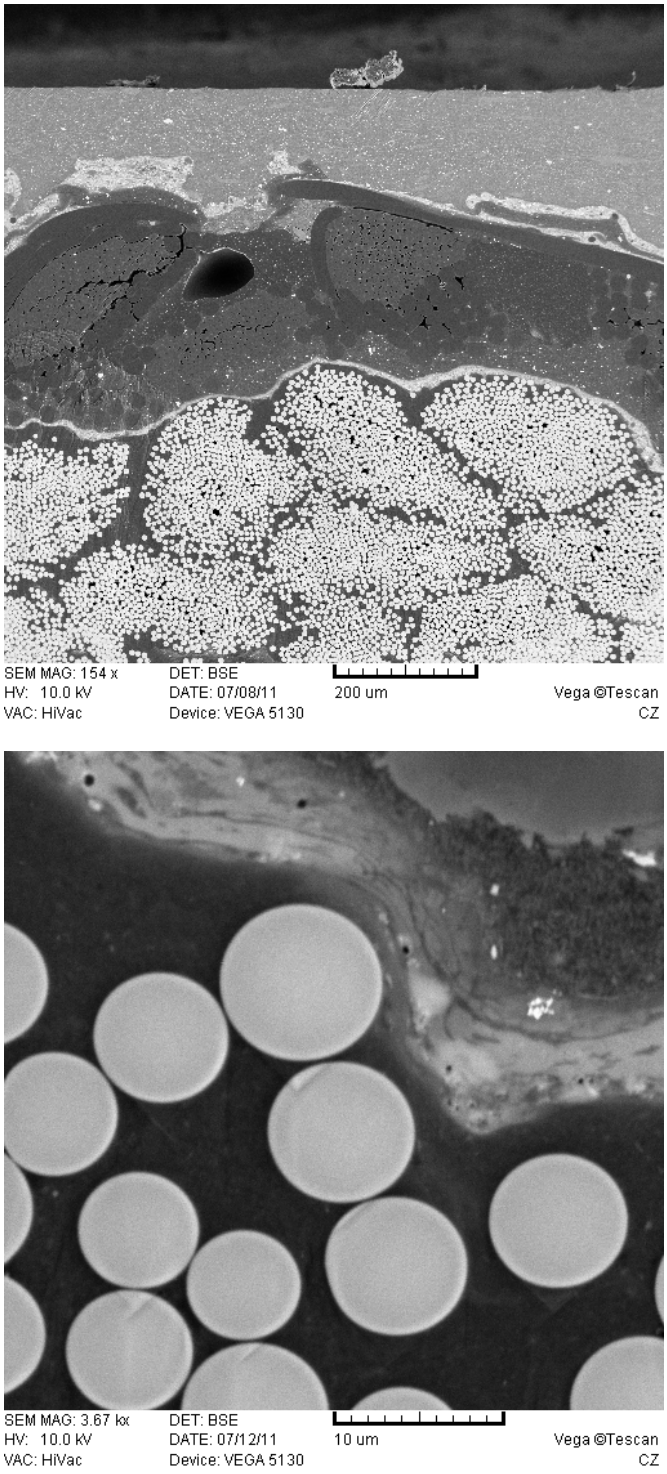


Fig. 38: Cross-section of a timing belt with glass cords and polyamide fibers

FCC PARTICLES (FLOW CRACKING CATALYTIC)

Aim of analysis

Cross-section of the particles

Preparation problem

It was not possible to prepare the sample embedded in epoxy resin successfully using the ultramicrotome as a result of the difference in hardness between the epoxy resin and the particle. Whether or not ion beam etching is better suited for this application should be tested.

Preparation conditions

Mechanical pre-preparation:

> The sample has been embedded in the epoxy resin and trimmed.
> After that, the sample was sanded on one side using the Leica EM TXP to ensure there is a flat side for the adjustment of the mask.

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	3 h

Result

It was possible to prepare a very smooth cross-section with slope cutting. The surface is free of preparation artifacts. The structure of the FCC particles is visible.

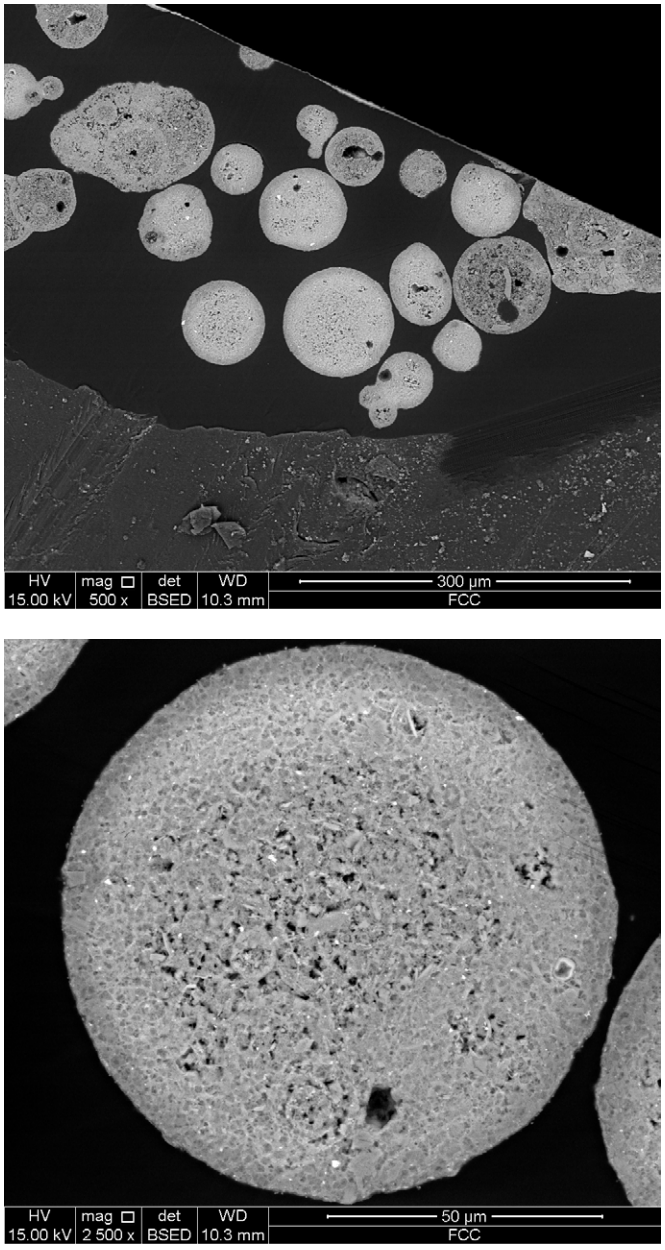


Fig. 39: SEM cross sectional image using Backscattered Electron (BSE) of ion-milled FCC particles (Flow Cracking Catalytic) included within a block of epoxy resin (Repsol).

TONER

Aim of analysis

Cross sectional sample preparation to display the components of the toner powder

Preparation problem

Fixing the powder is necessary to process the sample.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Embedding the powder between two glass plates
- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the sample cross-section using 9 µm diamond foil

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	4 h

Result

The cross-section is very smooth and clean. Toner powder components like toner grains and pyrite are easily visible.

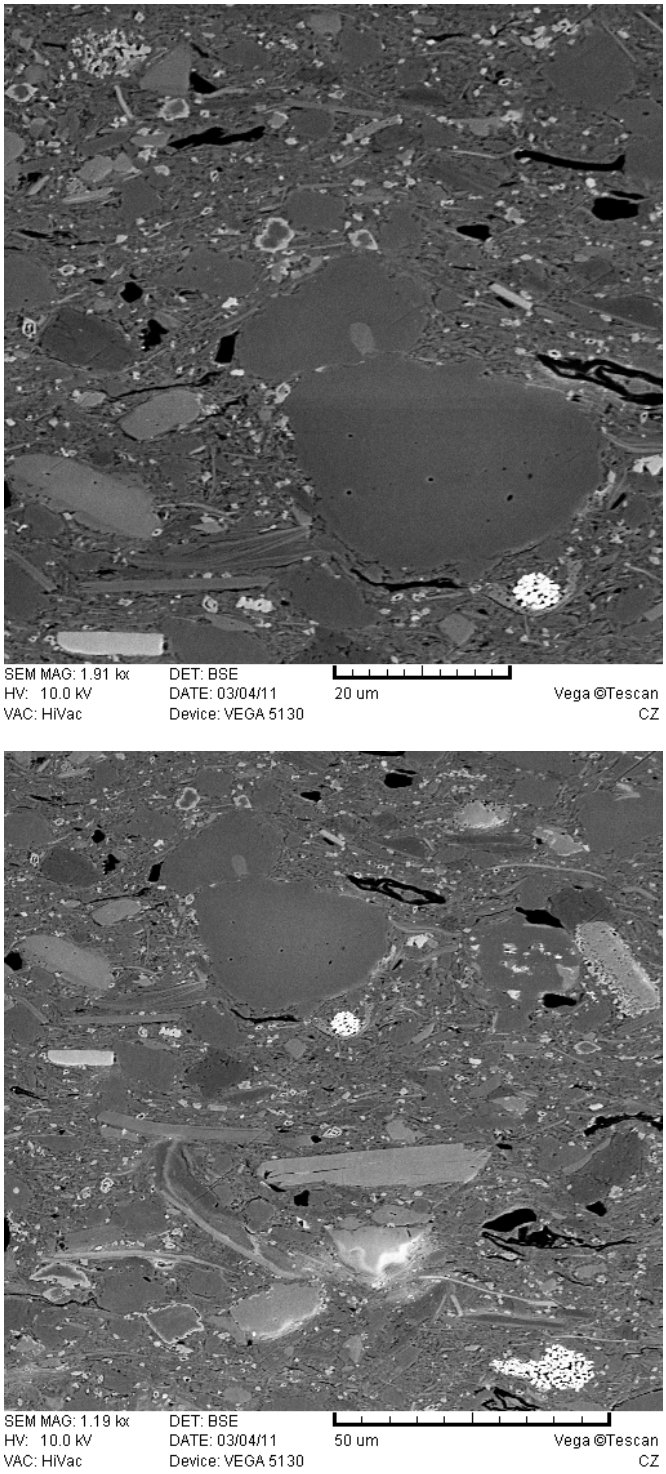


Fig. 40: Cross-section of toner powder

2.1.5. Preparation of thermally sensitive samples

The cooling stage is available for preparing thermally sensitive materials. Liquid nitrogen is used for cooling.

The cooling stage can be used to cool the samples down to a temperature of –150 °C during the milling process. The thermal conductivity in the sample itself is critical to the success of the preparation. Both the sample holder and the mask are cooled. After the preparation process, the sample is warmed up to room temperature again before the vacuum chamber can be opened. The maximum size of the samples to be processed is 25 × 25 × 5 mm.

Some application examples are shown below:

Fig. 41: Cooling stage with a 25 L dewar and liquid nitrogen pump

SOLDER BUMP

Aim of analysis

Cross-section of the solder bump and comparison of the results with the sample prepared without cooling

Preparation problem

The semiconductor structure with solder bumps is a very soft material that is rarely prepared successfully using conventional mechanical polishing. The polished surface is smeared with dirt and does not show structural details. During ion beam preparation without cooling, shrinkage effects of the individual components appeared in the solder bump. Therefore the sample was prepared again with cooling.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- Affix the sample to a Leica EM TIC 3X Al holder and fasten it in a Leica EM TXP adapter.
- Sand the cut face using 9 µm diamond foil until the solder bump is visible
- 45° wedge of the upper edge of the sample for reducing the material to be etched (also refer to Chap. 3)

The sample's wedge-shaped polished section requires the use of a wedged mask for slope cutting.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	3 h
Cooling	–50 °C
Mask	Wedged mask

Result

It was possible to prevent the shrinkage effects through cooling. Now the sample has a very smooth surface (compare to Fig. 11) /1/.

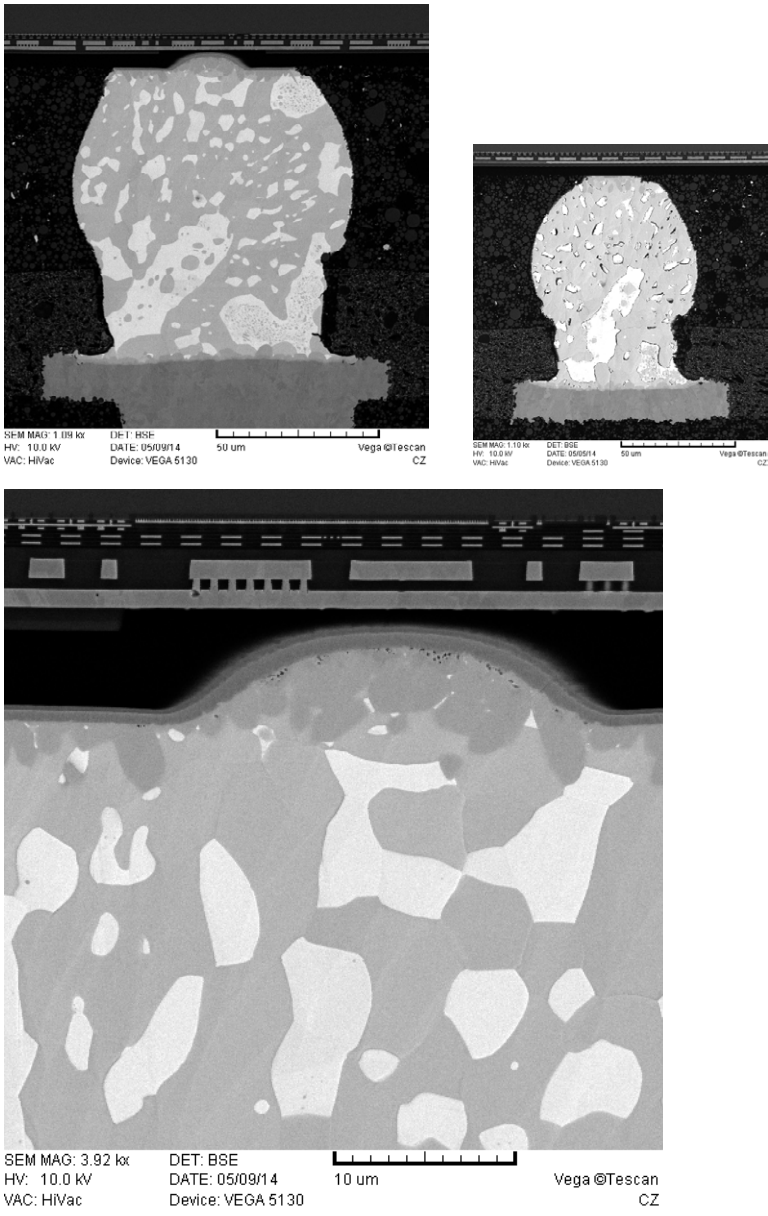


Fig. 42: Cross-section of a solder bump without thermal effects. For the sake of comparison, the small image shows the shrinkage effects of an uncooled sample (compare to the uncooled sample in Fig. 11).

ZINC ON STEEL

Aim of analysis

Cross-section for an EBSD examination

Preparation problem

Zinc is very sensitive and therefore should be prepared with cooling to prevent thermal destruction of the structure. This is especially true in the case of an EBSD examination.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

- A thin glass plate was glued to the zinc layer to protect it.
- The sample has been glued to a Leica EM TIC 3X Al holder and clamped in the Leica EM TXP using an adapter.
- The sample was ground using 9 µm diamond foil to obtain a smooth cut face.

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	5 h
Cooling	–70 °C

Result

It was possible to prepare a smooth and clean cross-section. The structure of the zinc layer is easily visible. Pores and fine cracks can be seen.

The Kikuchi diffraction patterns show that the sample can be used for EBSD examinations.

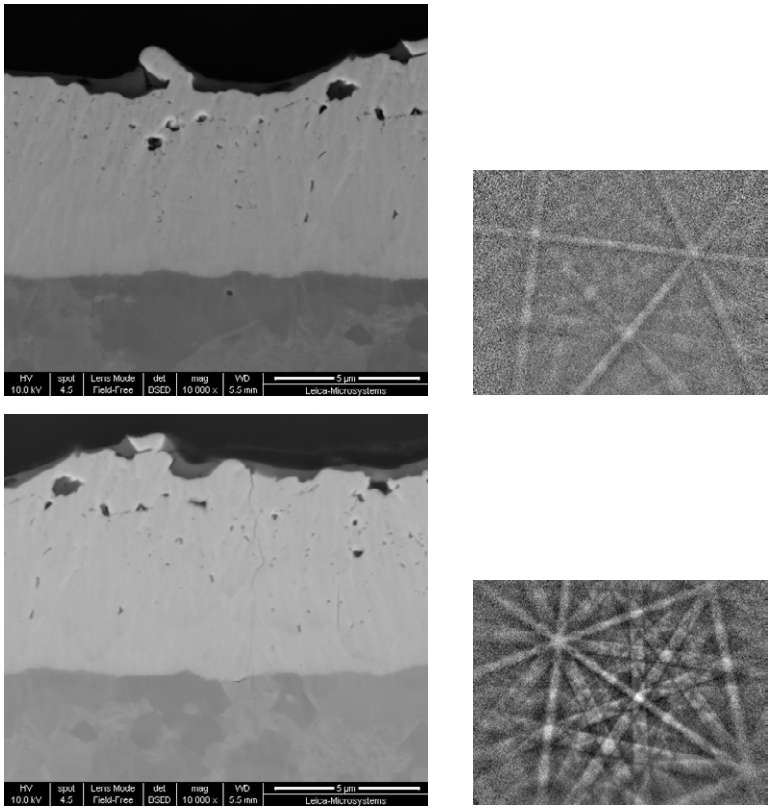


Fig. 43: SEM cross section image of a zinc layer on steel with the Kikuchi diffraction patterns of tin and steel

LIGNOCELLULOSE

Aim of analysis

Preparation of a smooth surface

Preparation problem

Lignocellulose, an organic material, is very temperature-sensitive. The dimensions of the sample are 0.5 mm × 1 mm. This makes fastening the sample to the sample holder extremely difficult.

Preparation conditions

Mechanical pre-preparation:

> Preparing a flat sample side using a razor blade.
> Affix the sample to a Leica EM TIC 3X Cu holder

ION MILLING	
Acceleration voltage	5 kV
Gun current	2 mA
Milling time	4 h
Cooling	–120 °C

Result

It was possible to prepare the sample surface to be very smooth. The surface quality is exceptionally good. Light contamination of the sample can be seen on just the top end of the sample due to interaction of the ion beam with the mask (top image, Fig. 44).

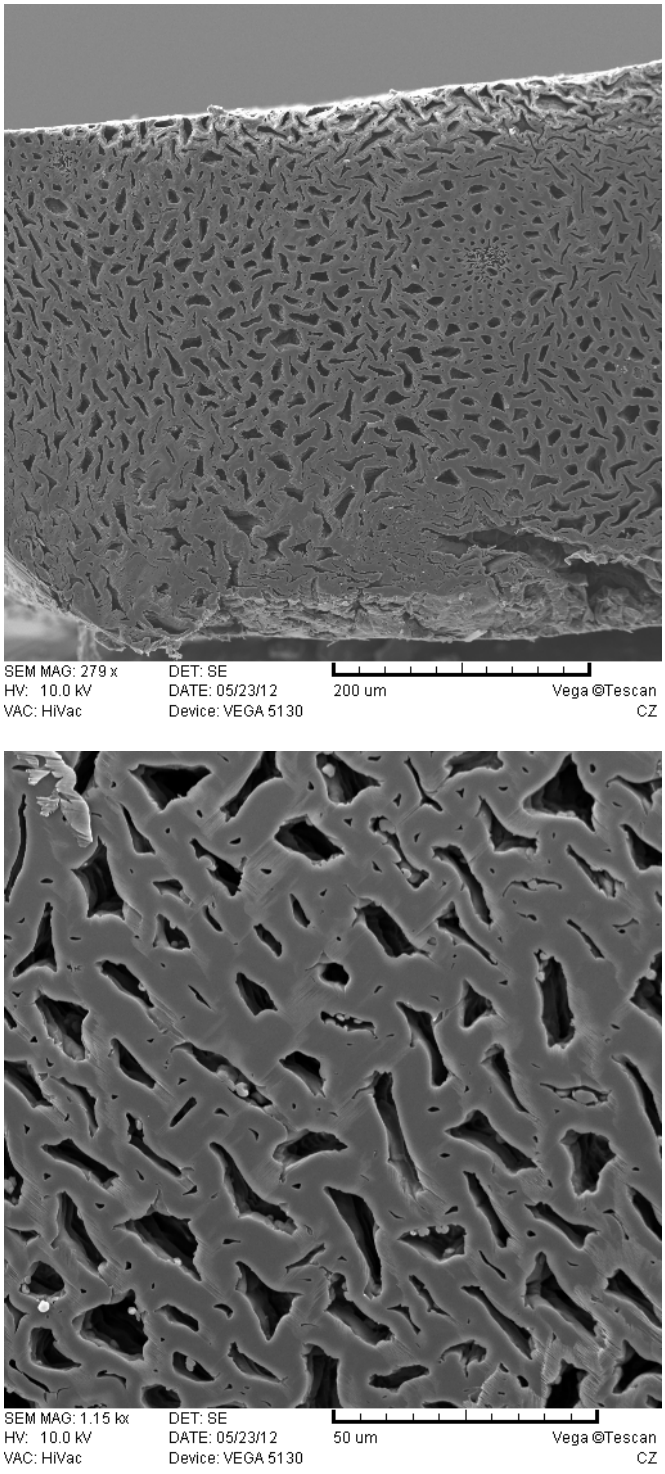


Fig. 44: Cross-section of ion-milled lignocellulose

TOUCHSCREEN WITH GLASS, POLYMER LAYERS AND LAYERS OF ADHESIVE

Aim of analysis

Cross-section to display all layers (polymer layers, layers of adhesive) on the touchscreen's glass substrate

Preparation problem

The layer system is extremely sensitive to thermal and mechanical influences

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

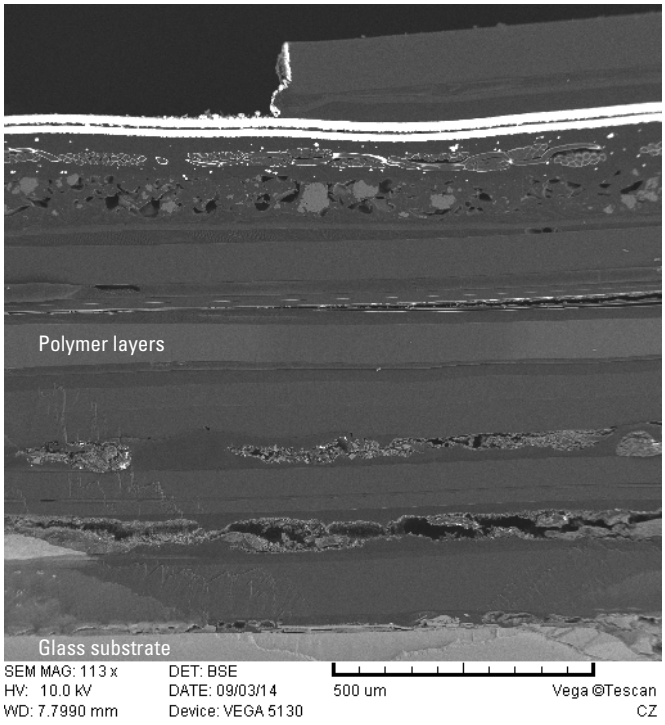
> Protect the sample surface by adhering a cover glass
> Affix the sample to a Leica EM TIC 3X Cu holder
> Sharpen the sample cross-section using 2 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	17 h
Cooling	–60 °C

Result

The preparation result is very good. It was possible to produce a section through the entire structure. The cut face is very smooth. The interfaces of the individual layers are easily visible.



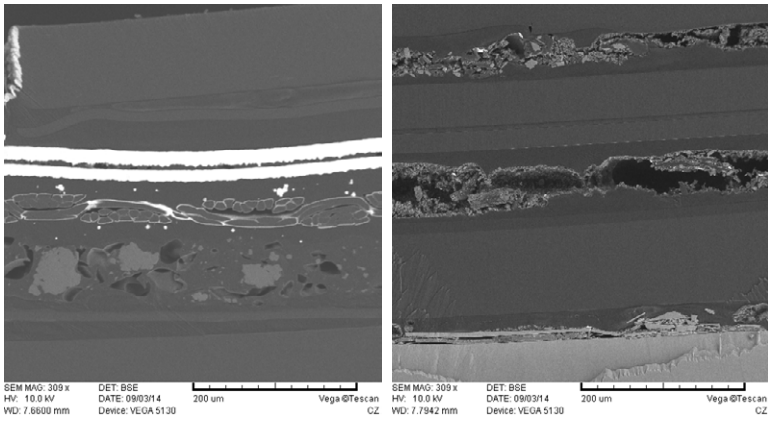


Fig. 45: Cross-section of a polymer/adhesive layer system on glass (touch-screen)

PAINT PARTICLES EMBEDDED IN EPOXY RESIN

Aim of analysis

Preparation of a flat and nondestructive sample surface for the examination of the organic binders and inorganic pigments in the paint with EDS and EBSD.

Preparation problem

The paint is very sensitive to temperature. The maximum possible temperature is 50 °C. As a result, the sample must be cooled during preparation.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Cu holder
> Sharpen the sample cross-section using 2 µm diamond foil

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	4 h
Cooling	–90 °C

Result

The cross-section of the paint particle is very smooth and clean. The organic binders and the inorganic paint pigment are easily recognizable.

The sample is suitable for EDS and EBSD examinations.

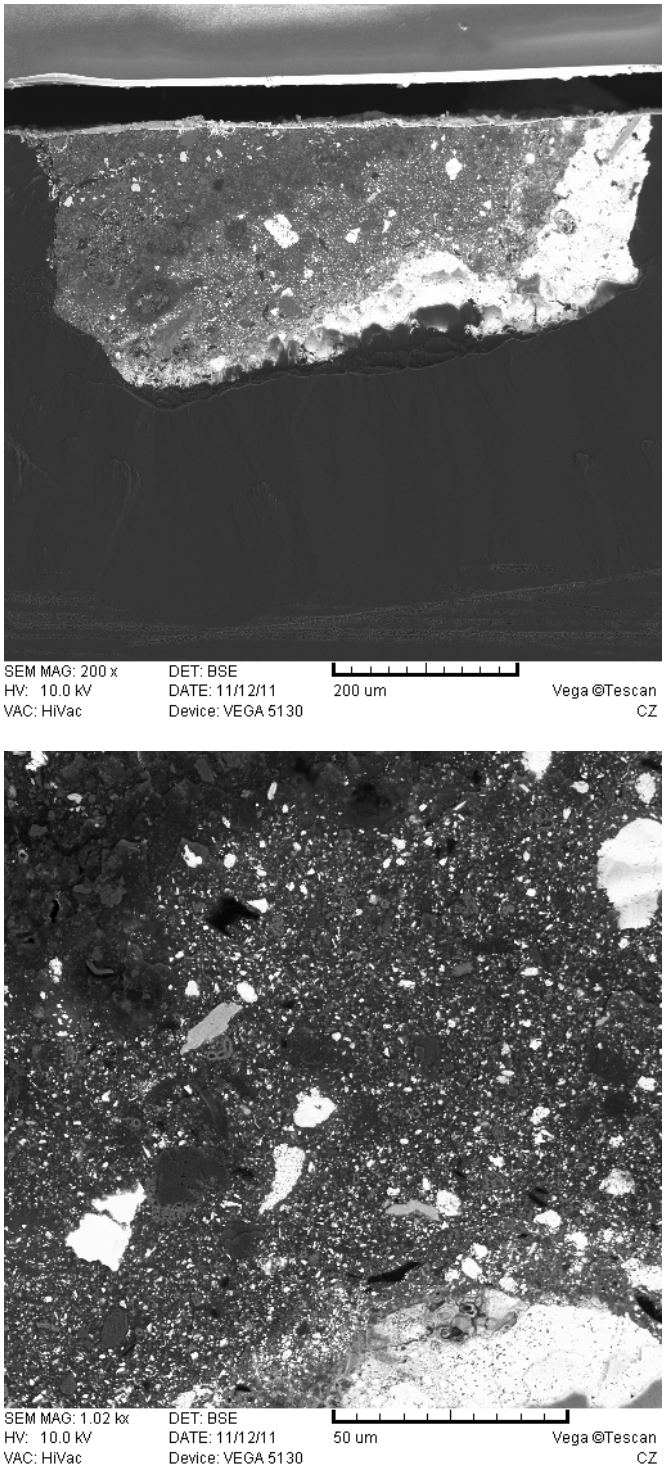


Fig. 46: Cross-section of a paint particle embedded in epoxy resin

AGAVE FIBERS EMBEDDED IN VINYL ESTER

Aim of analysis

Cross-section for the examination of the fiber structure

Preparation problem

The sample is a sensitive, organic material.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Sand the sample to reduce its size
> Affix it to a Cu sample holder
> Polish the cross-section surface using 9 µm diamond foil

ION MILLING

Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	10 h
Cooling	–100 °C

Result

The cross-section looks very good. The structure of the agave fibers are visible. EDS examinations identify the white surfaces as calcium (Ca).

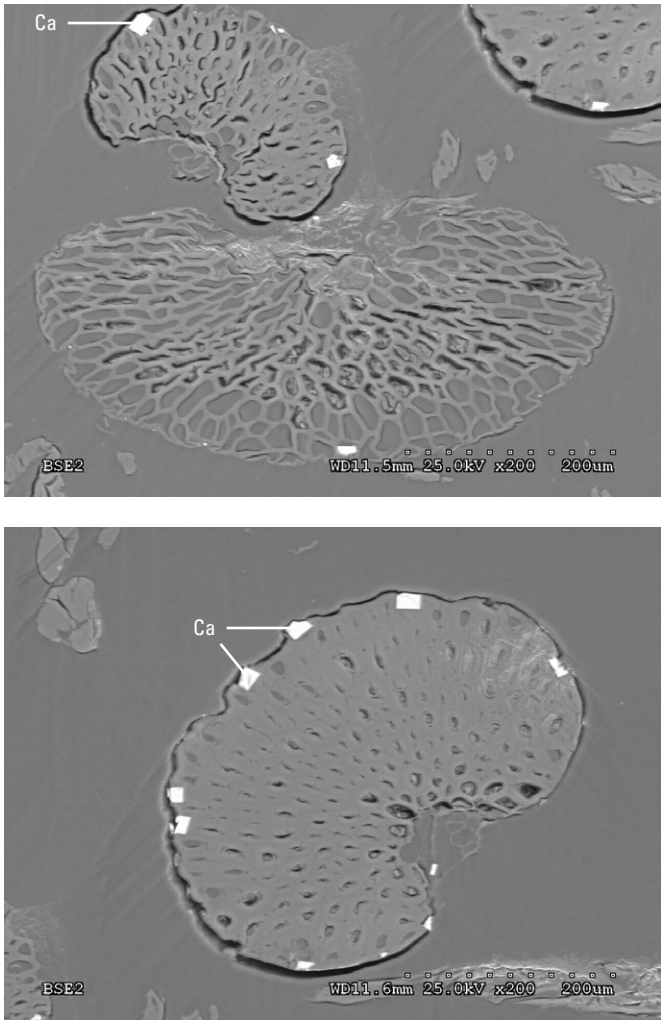


Fig. 47: Cross-section of agave fibers embedded in vinyl ester

CONCRETE

Aim of analysis

Investigation of the concrete's structure

Preparation problem

Figure out whether the heating of the sample during the milling process leads to cracks.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to a Leica EM TIC 3X Cu holder
> Sand the cross-section surface using 9 µm diamond foil

ION MILLING

Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	8 h
Cooling	–40 °C

Result

The preparation of the concrete was successful. It was possible to prepare a surface 0.9 mm deep and 2 mm long. The surface is smooth and clean. Cracks in the concrete are visible; these are apparent both during preparation with cooling as well as without cooling.

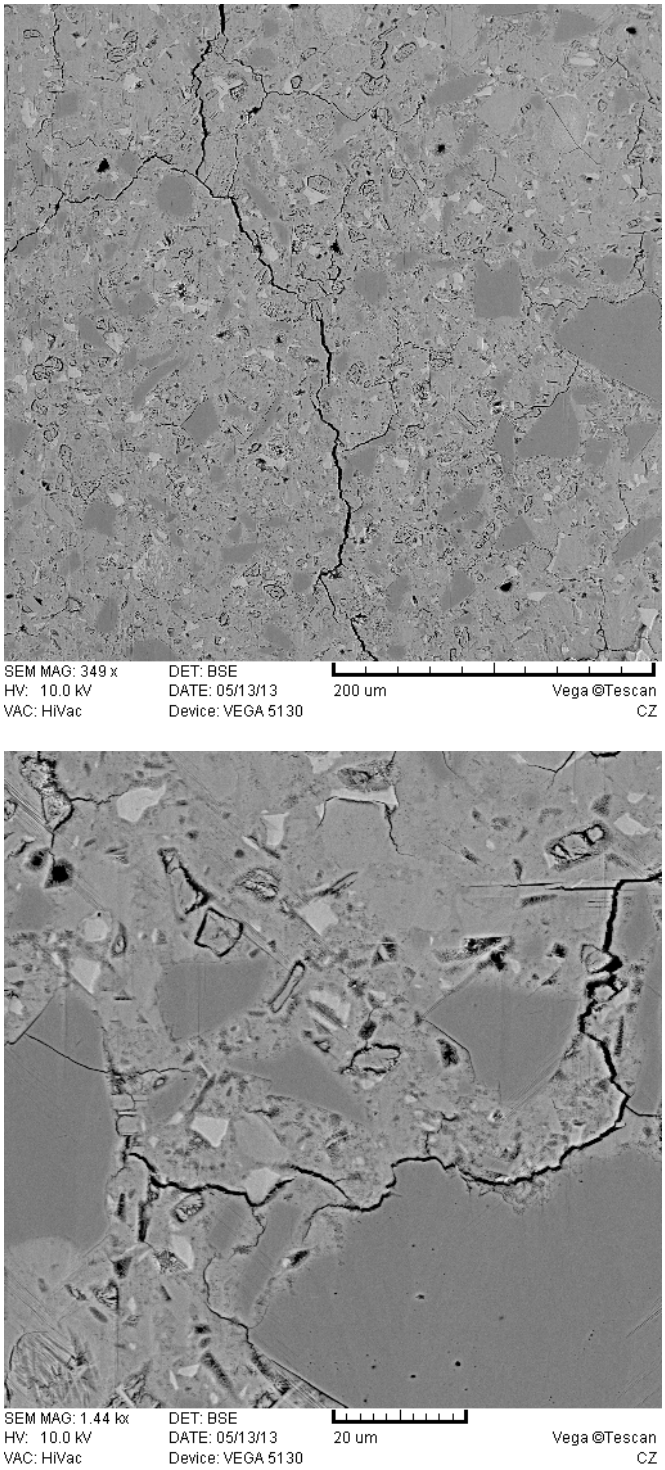


Fig. 48: Cross-section of concrete prepared with cooling

CAR TIRES (STEEL CORD IN RUBBER)

Aim of analysis

Cross-section of the steel cords embedded in rubber

Preparation problem

The preparation problem is a result of extreme differences in the materials contained in the sample.

Preparation conditions

Mechanical pre-preparation:

> Reduction in sample size

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	10 h
Cooling	–60 °C

Result

The interface between the bras, rubber and steel cord is clearly visible.

Two layers are visible on the steel cord, one very thin, light layer and one fibrous, dark layer. Both layers have varying thicknesses. Part of the light-colored layer penetrates deep into the steel.

Caution is required when cooling the sample. If the sample is cooled too much, the rubber will separate from the steel cord. The sample can be destroyed as a result of the cooling.

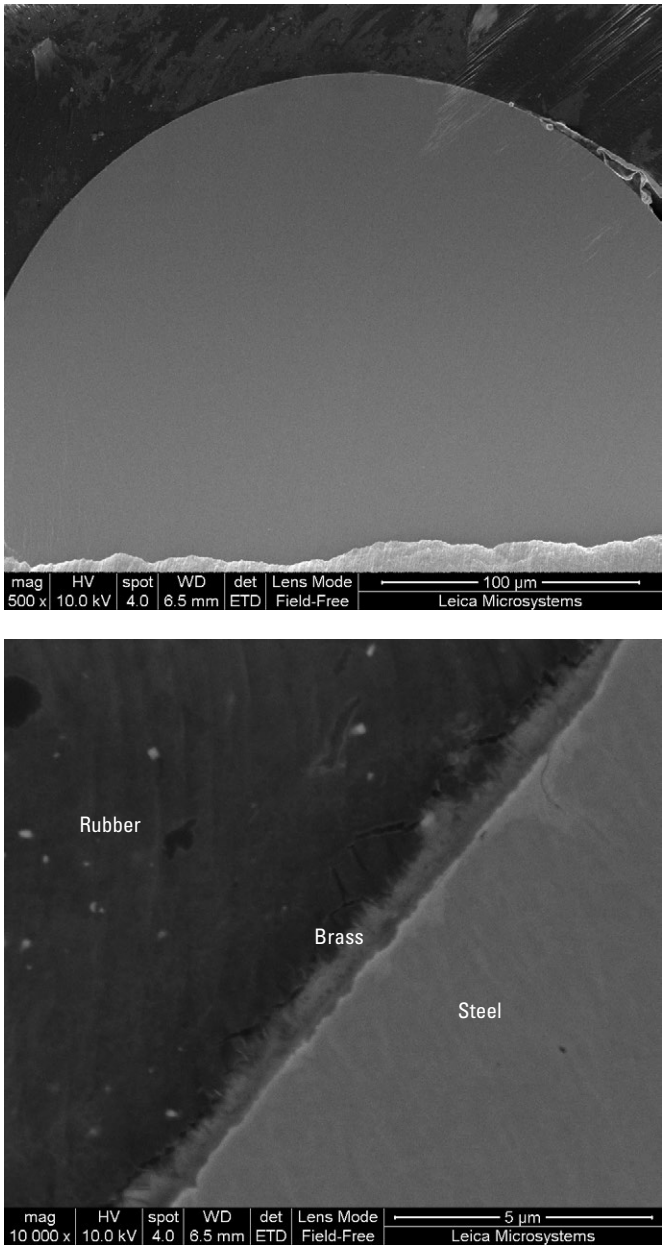


Fig. 49: Cross-section of a rubber/steel cord composite (car tires)

PVC ID CARD

Aim of analysis

A crack is starting to form in the PVC ID card. The crack and its path in the form of forks should be displayed using the cross sectional sample preparation.

Preparation problem

The sample is temperature-sensitive. Site-specific preparation is necessary to display the cracks.

Preparation conditions

Mechanical pre-preparation:

> Reduction in sample size

ION MILLING	
Acceleration voltage	6 kV
Gun current	2.2 mA
Milling time	4 h
Cooling	–60 °C

Result

It was possible to prepare a cross-section through a PVC ID card using ion beam slope cutting. The sample has been sectioned at the start of the crack. The crack has forks that mainly run parallel to the main crack.

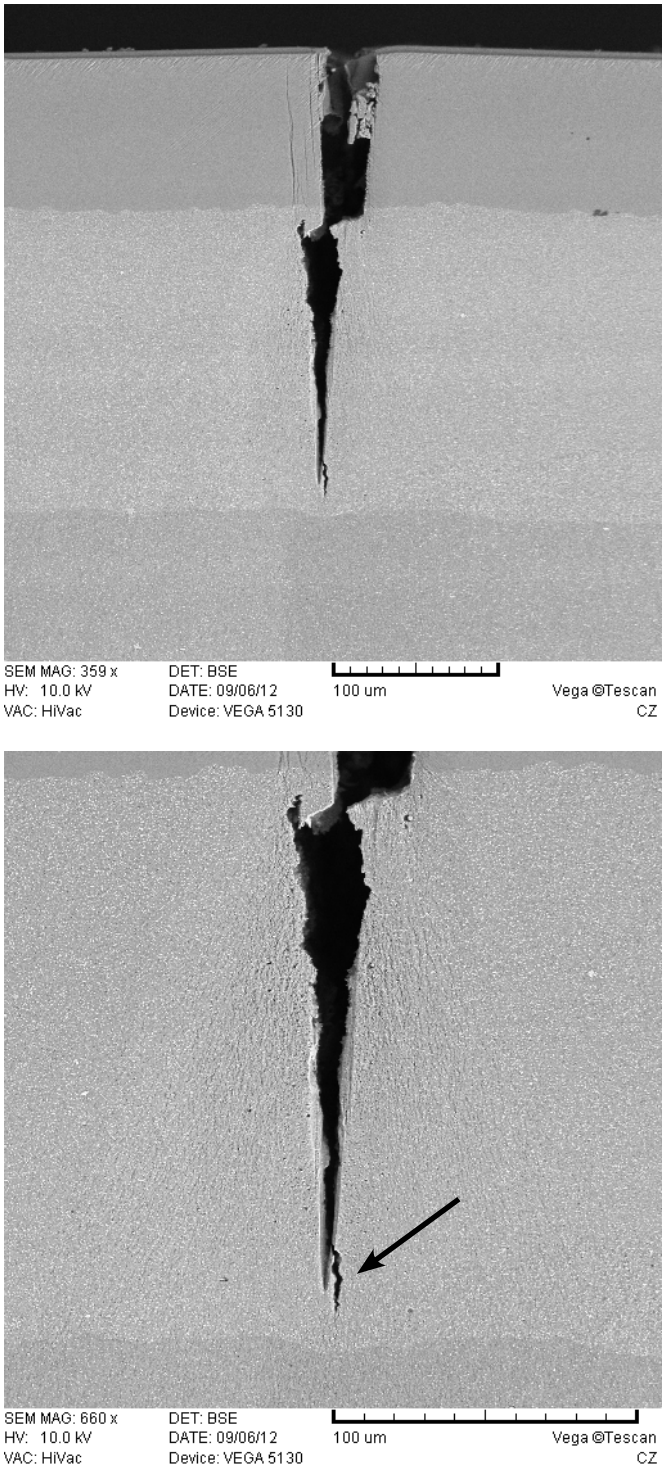


Fig. 50: Cross-section of a PVC ID card with a crack formation and its forks

2.2. CLEANING SAMPLE SURFACES AND CONTRAST ENHANCEMENT

For better showcasing the grain structure in polycrystalline materials, a chemical etching process where the individual grains are etched to varying degrees is normally used. This results in topography that enhances contrast.

This contrast enhancement, however, can also be generated by ion milling. Milling angles in a range of 30° to 90° are used in relation to the sample surface in this process. The angle depends on the material being prepared and its milling rate.

The ion etching process, unlike conventional chemical etching, has a number of advantages. It is cleaner, safer and, above all, can be controlled better.

In the Leica EM TIC 3X, this contrast enhancement of slope cuts is incredibly easy to implement. After preparation of the cross-section surface, the sample holder together with the sample is placed in a position tilted 90° relative to the original position.

Fig. 51 shows the sample in the position for cross sectional sample preparation (left image) and in the position for contrast enhancement (right image).

The three ion beams then come into contact with the prepared cross-section surface at an angle of 40° (source 1 and 3) and 90° (source 2). Because the ion sources can be controlled individually, it is also possible to use only ion sources 1 and 3 or ion source 2.

The sample holder that can be used for the contrast enhancement can be seen in Fig. 51 along with the various sample positions.

Fig. 52 shows the contrast enhancement created by ion milling using a copper sample. The grain structure is very easy to see.

Prepared cross-section surfaces can also be cleaned using this method if necessary. This involves the use of very low energies and very short etching times.

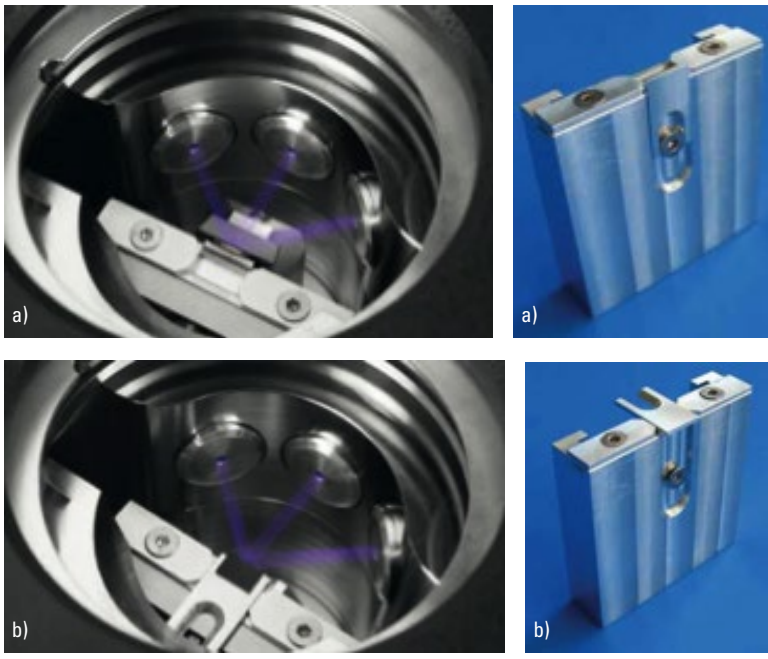


Fig. 51: Sample holder: (a) sample position for cross sectional sample preparation, (b) sample position for contrast enhancement or cleaning the prepared area

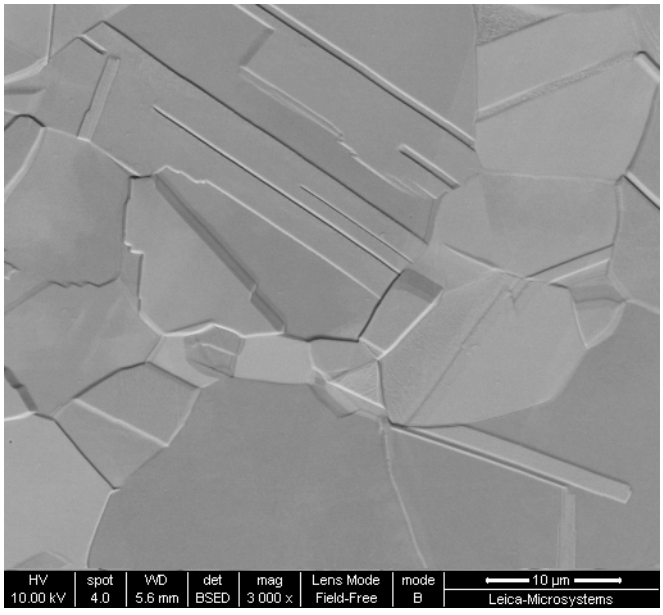


Fig. 52: Copper surface after contrast enhancement with ion milling

2.3. PROCESSING LARGE SAMPLE SURFACES WITH THE ROTARY STAGE

The rotary stage is available for processing large sample surfaces. The Leica EM TIC 3X is capable of covering the entire area of the SEM sample preparation using this stage and other stages mentioned earlier.

The rotary stage represents a significant expansion to Leica EM TIC 3X applications. It makes it possible to process the surfaces of large samples. The maximum sample size is 38 mm in diameter, the maximum surface that can be processed is 25 mm in diameter. By overlapping two sample movements (rotation/oscillation and translation), it is possible to uniformly process the surface. Because of the potential sample tilting from 0° to 48°, the surfaces can be polished, cleaned and have its contrast enhanced. The specifications of the stage are displayed in the following table.

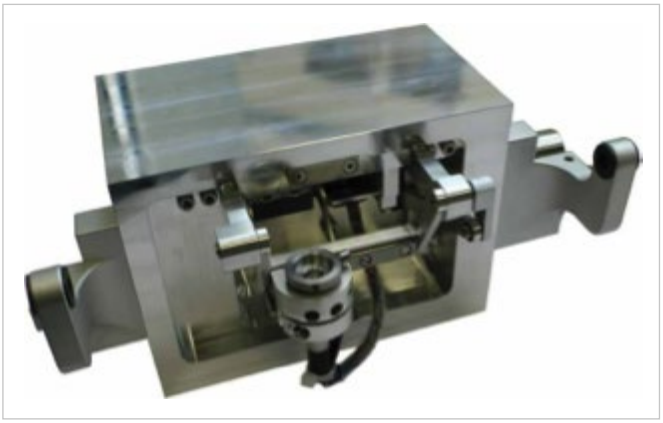


Fig. 53: Leica EM TIC 3X rotary stage for processing large sample surfaces up to 25 mm in diameter

SPECIFICATIONS

Max. sample diameter	38 mm
Max. preparation area	Ø 25 mm
Max. sample height	12 mm
Lateral movement	± 12.5 mm
Milling angle	0–48° (1.5° step size)
Oscillation	45°, 90°, 180°, 360°

The translation movement range of the sample depends on its diameter.

LIMITATIONS

Sample size	Translation movement range
Ø 38 mm	± 3 mm
Ø 35 mm	± 5 mm
Ø 33 mm	± 6 mm
Ø 30 mm	± 8 mm
Ø 28 mm	± 9.5 mm
Ø 25 mm	± 11.5 mm
< Ø 20 mm	± 12.5 mm

This also applies for the adjustment of the sample's tilting angle.

Sample size	Milling angle range
Ø 38 mm	0–12°
Ø 35 mm	0–13°
Ø 33 mm	0–14°
Ø 30 mm	0–15°
Ø 28 mm	0–16°
Ø 25 mm	0–18°
Ø 23 mm	0–36°
< Ø 20 mm	0–48°

The maximum height of a sample to be processed is 12 mm. Different sample holders and stub adapters are available for different sample heights.

Fig. 54: Sample holders and stub adapter for samples with different heights

The eucentric height of the sample must be adjusted using a special device (Fig. 55) before processing.

Afterwards the sample, together with the holder, is screwed onto the sample stage.

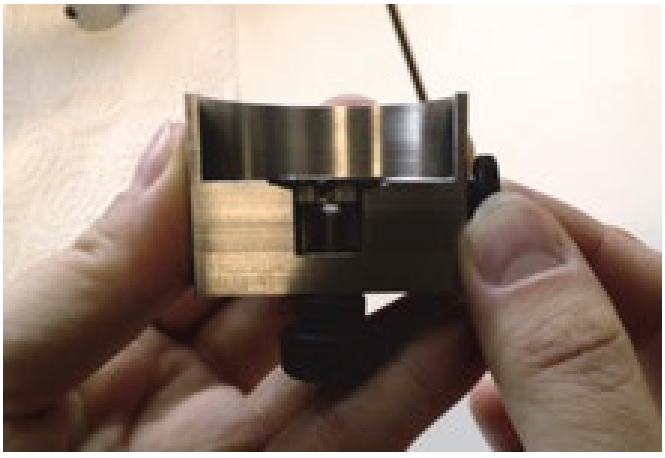


Fig. 55: Adjusting the sample surface to the eucentric height

Fig. 56: Fastening the sample holder to the sample stage

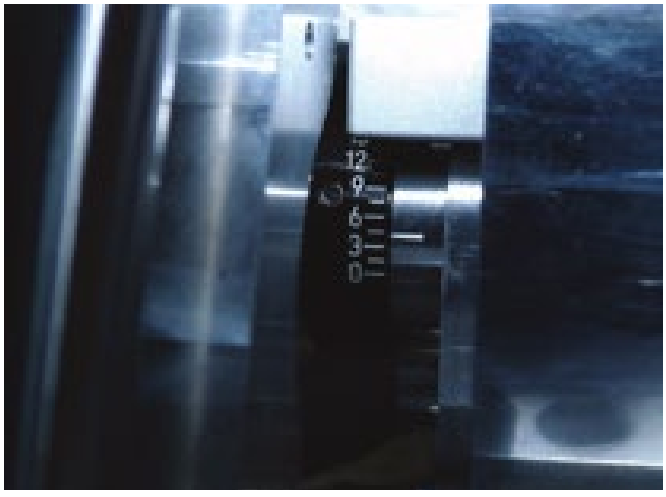


Fig. 57: Setting the milling angle (tilting angle of the sample)

A milling angle between 0° and 48° can be selected for the different surface processing methods. The angles can be adjusted in 1.5° increments using the adjustment knob shown in Fig. 57. The milling angle depends on the corresponding application.

The ability to adjust the milling angle is a significant advantage of the Leica EM TIC 3X because it enables the sample to be polished and cleaned, as well as have its contrast enhanced.

For polishing, use an angle between 3° and 5°; use an angle between 10° and 15° for surface cleaning and an angle larger than 30° for contrast enhancement.

If samples are processed using the rotary stage, it must be polished mechanically beforehand. The Leica EM TXP can be used for this (see Chap. 3).

The quality of the mechanical polishing should be as high as possible because ion polishing only represents the final step towards additional improvement to the surface quality. The basic operating principle behind ion polishing is displayed in Fig. 58. The sample surface is hardly affected by the extremely flat milling angle. In an area with a rougher surface, the ion beam strikes the sample at a milling angle that results in a higher sputter rate. As a result, the roughness is removed and the surface is leveled.

If the sample has deep scratches, all surrounding material must be removed in order to obtain a smooth surface.

Mechanical polishing of soft materials or material combinations with soft and hard components leads to the surface becoming smeared with dirt. In a case like this, it is extremely important to clean the surface of the smeared material before ion polishing. A cleaning step with a milling angle of approximately 10° is used for this.

The following is intended to show a few application examples that illustrate the options available for the Leica EM TIC 3X.

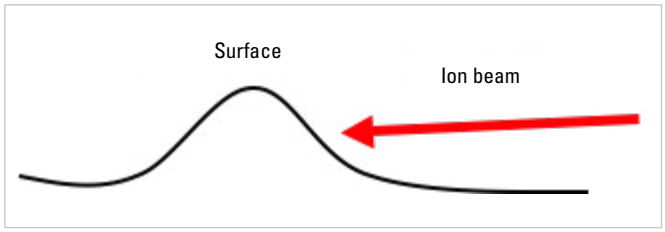


Fig. 58: Ion polishing of rough surfaces

KEROGEN AND SHALE

Aim of analysis

Preparing a perfect sample surface using ion milling

Preparation problem

The samples have received only very rough mechanical polishing. A test should be conducted to see if the surface quality can be improved.

Preparation conditions

Mechanical pre-preparation:

> Unknown

ION MILLING KEROGEN	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	2 mA	2.2 mA
Milling angle	10.5°	3°
Sample movement	Rotation	Rotation
Milling time	10 min	2 h

ION MILLING SHALE	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	2 mA	2,2 mA
Milling angle	10.5°	3°
Sample movement	Rotation	Rotation
Milling time	10 min	2 h

Result

It was possible to improve the surface quality substantially using the two-level ion beam preparation (cleaning and polishing). All contamination on the sample surface has been removed and the surface roughness has been significantly reduced. Structural details such as fine pores can now been seen clearly.

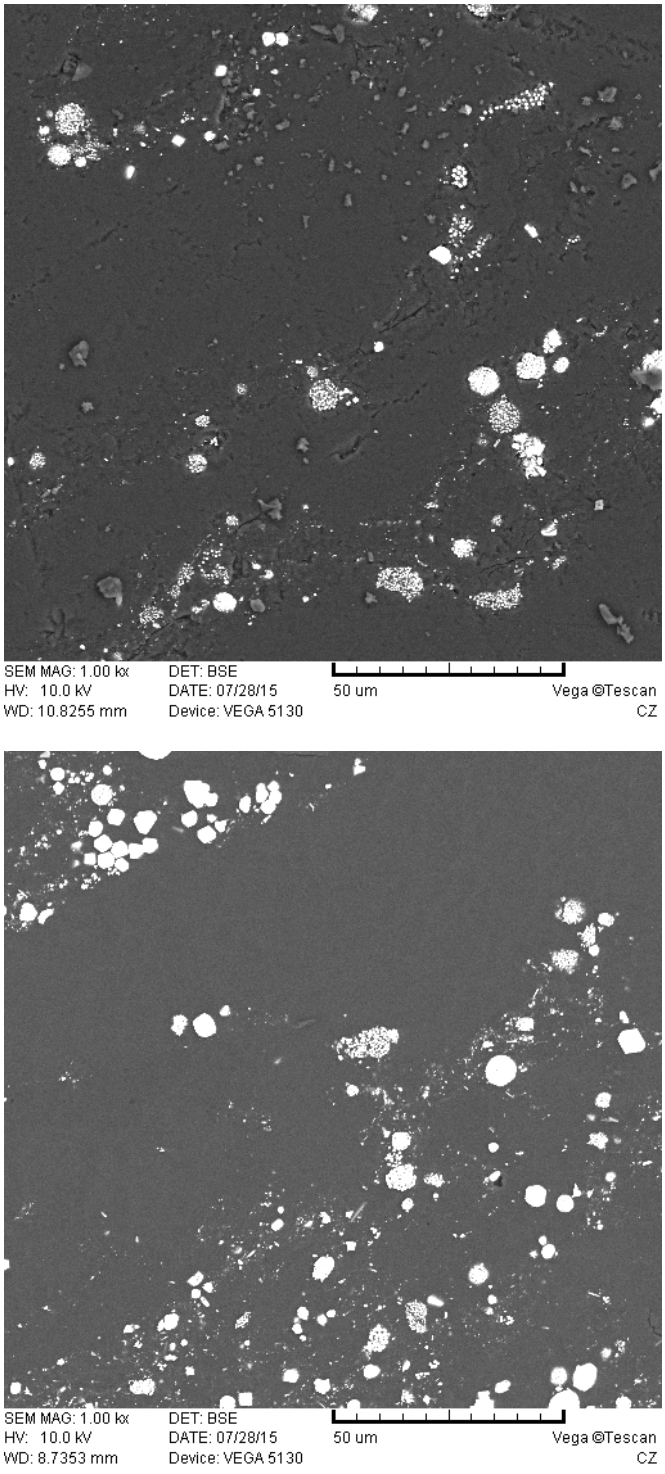


Fig. 59: Kerogen after mechanical polishing (above) and after additional ion polishing (below)

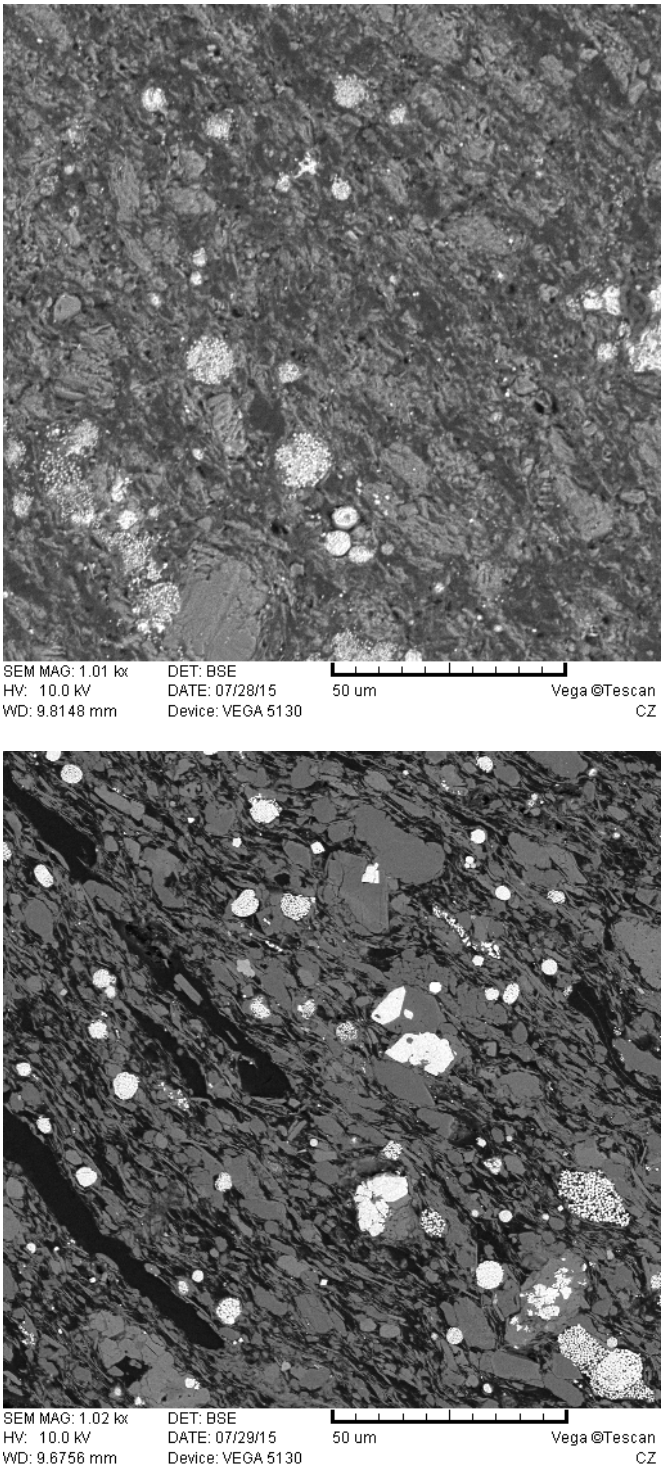


Fig. 60: Shale after mechanical polishing (above) and after additional ion polishing (below)

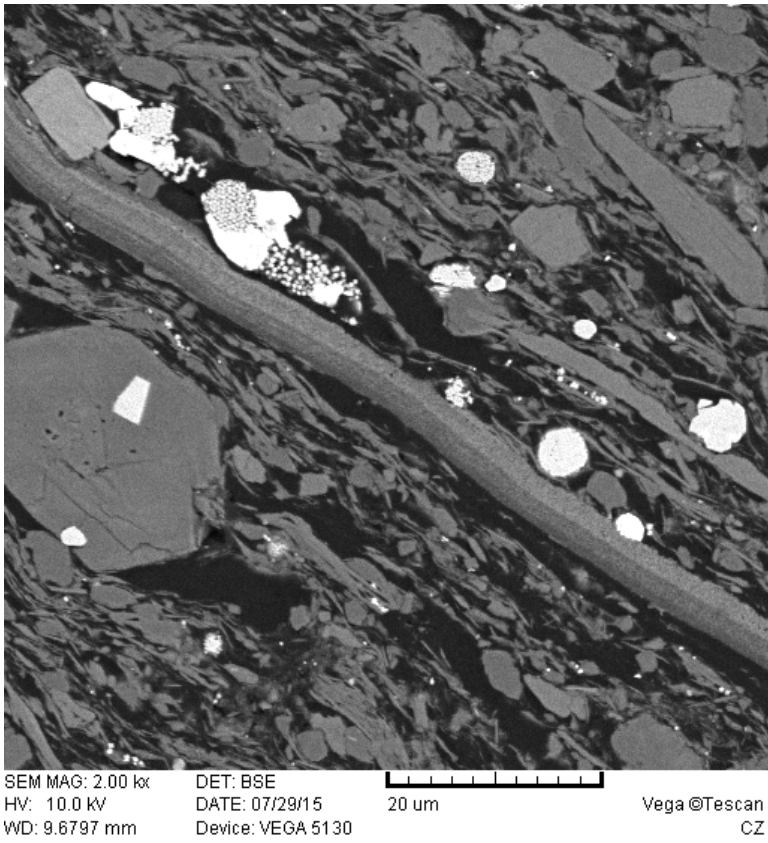


Fig. 61: Shale after additional ion polishing with pyrite, quartz, feldspar and clay.

SYNTHETIC ROCK SALT

Aim of analysis

Displaying the grain boundaries and substructure in the grains of synthetic rock salt

Preparation problem

Understanding the mechanical properties of rock salt and its deformation behavior is of major importance for the prediction of long-term stability of nuclear waste repositories, and our understanding of the dynamics of salt-related sedimentary basins which host the majority of oil and gas accumulations on Earth. The results of mechanical polishing are not satisfactory.

Preparation conditions

Mechanical pre-preparation:

> Mechanical polishing with SiC (4000)

ION MILLING	Cleaning	Polishing	Contrasting
Acceleration voltage	4 kV	6 kV	3 kV
Gun current	2 mA	2.2 mA	1.5 mA
Milling angle	10.5°	3°	30°
Sample movement	Rotation	Rotation	Rotation
Milling time	10 min	2 h	3 min

Result

Ar-ion polishing using the Leica EM TIC 3X and the rotary stage at low angle towards the triple beam produced a smooth surface (Fig. 62 c and Fig. 63 a) of the entire surface area (Ø 2 cm). SEM imaging allowed determination of the grain structure based on the density contrast caused by different mineral orientations. The grain boundaries are exposed as thin grooves and are only visible at very high magnification (Figs. 63 a1 and a2). The pores showed sharp etches and some of them were filled with small salt grains, which were presumably grinding dust.

Grain boundary decoration was done by means of etching using the rotary stage at high angle with respect to the orientation of the triple beam (Figs. 63 b1 and b2). This treatment caused the exposure of grain boundaries as grooves that were visible also at low magnification in SEM. The bulk grains showed a distinct surface pattern that varies from grain to grain and could be interpreted to be dependent on the crystal lattice orientation. However, this assumption could only be validated employing EBSD analysis /3/.

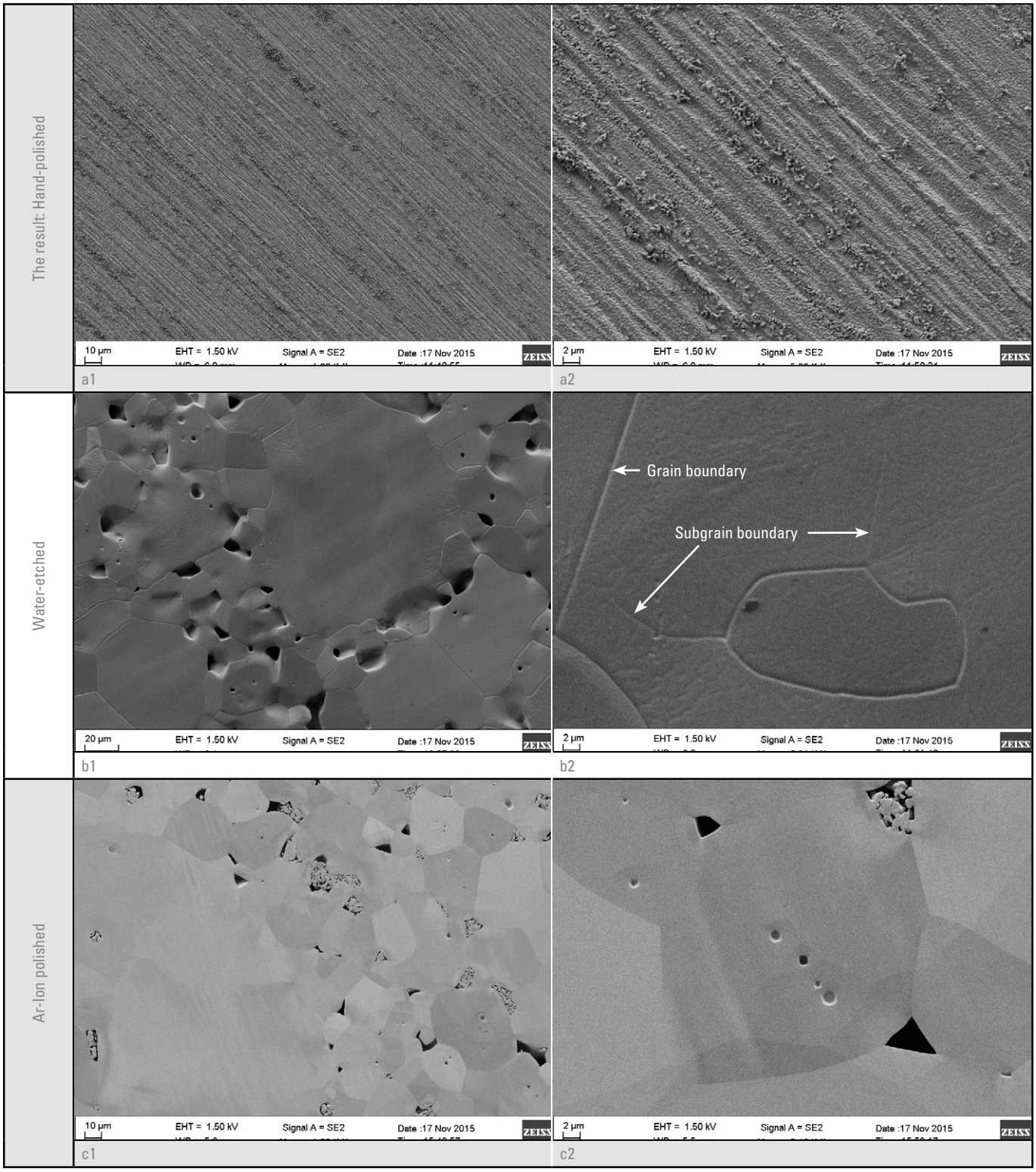


Fig. 62: Secondary Electron Micrographs of a stepwise a) hand-grinded, b) water-etched and c) hand-grinded and subsequently Ar-ion polished polycrystalline sample of rock salt (left column: low resolution; right column: high resolution).

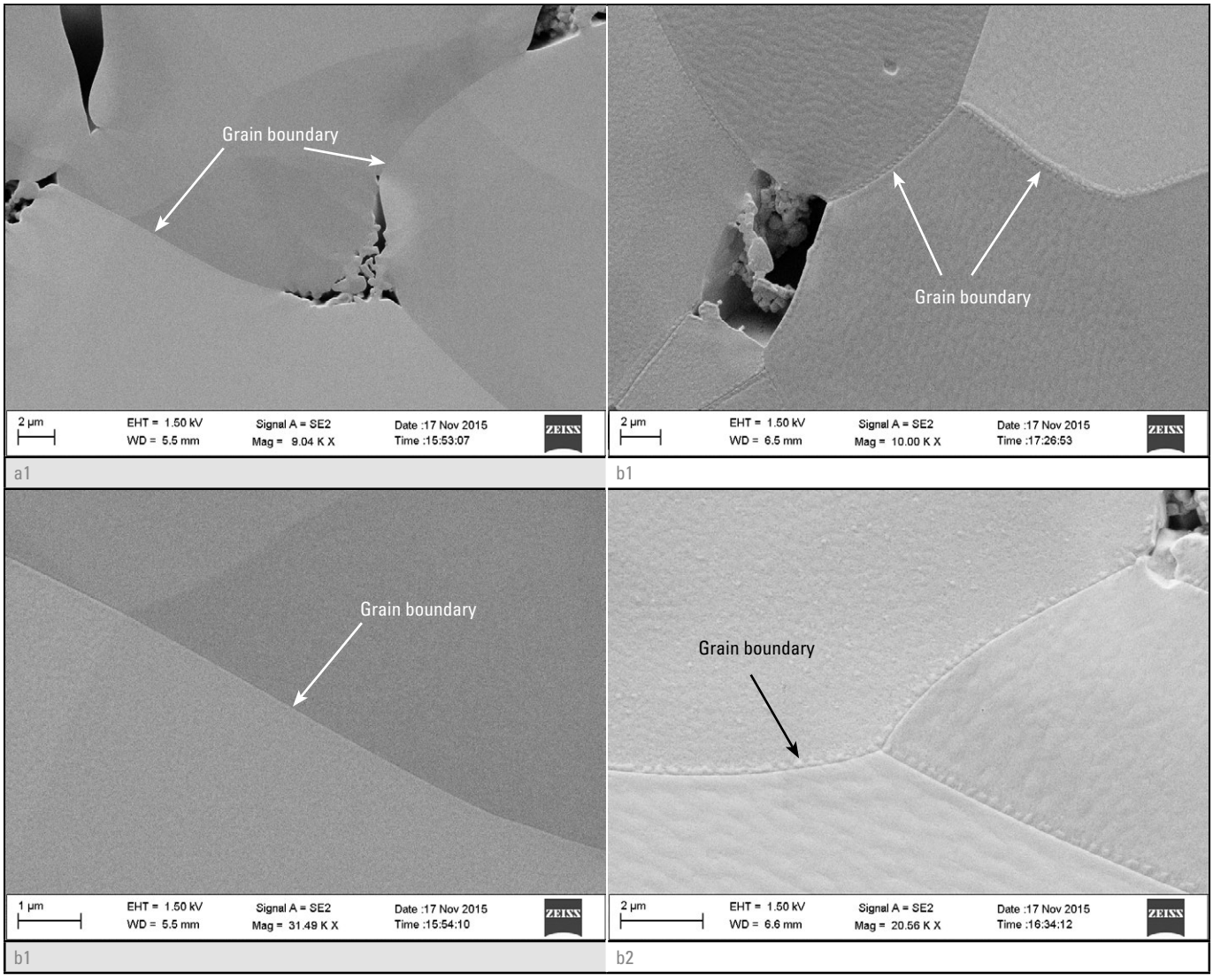


Fig. 63: Secondary Electron Micrographs a stepwise (a) low angle Ar-ion polished rocks salt and subsequently (b) high-angle Ar-ion etched surface of polycrystalline rock salt.

SHALE SAMPLE

Aim of analysis

Investigation of the pore structure in the shale

Preparation problem

The micropores become clogged with dirt during mechanical polishing.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Polish the surface using U-type diamond films: 9 µm, 2 µm and 0.5 µm
> Preparation time: 10–15 min

ION MILLING	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	2 mA	2.2 mA
Sample movement	Rotation	Rotation
Milling angle	10.5°	3°
Translation movement	± 3 mm	± 3 mm
Milling time	10 min	1 h 30 min

Result

The surface quality of the sample is very good after ion beam preparation. The micropores can be made clearly visible during SEM.

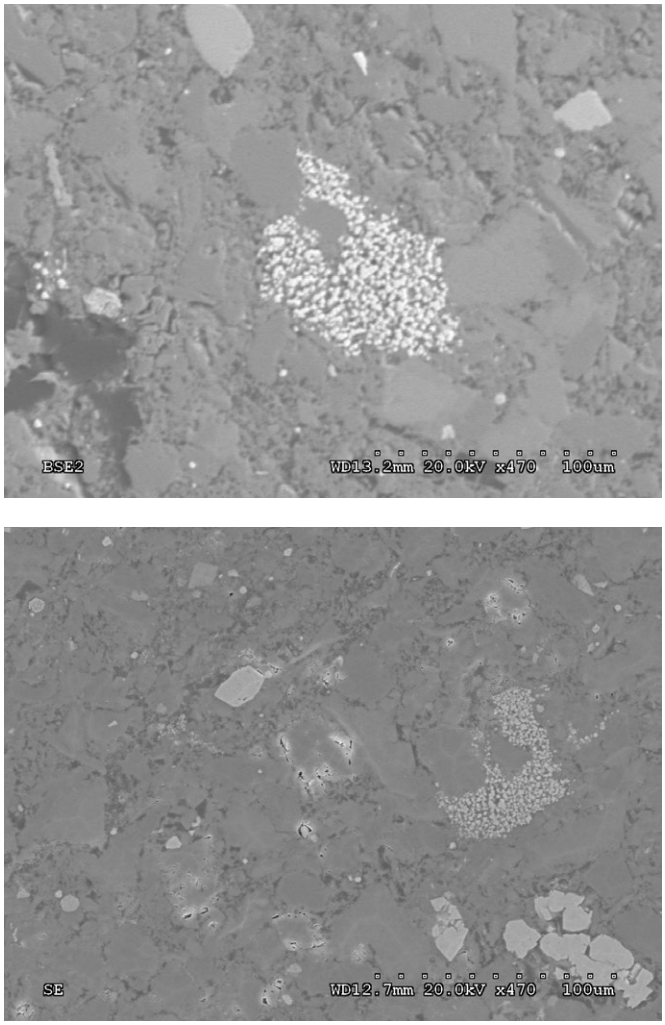


Fig. 64: Shale sample after mechanical polishing using the Leica EM TXP (above) and after additional ion polishing (below).

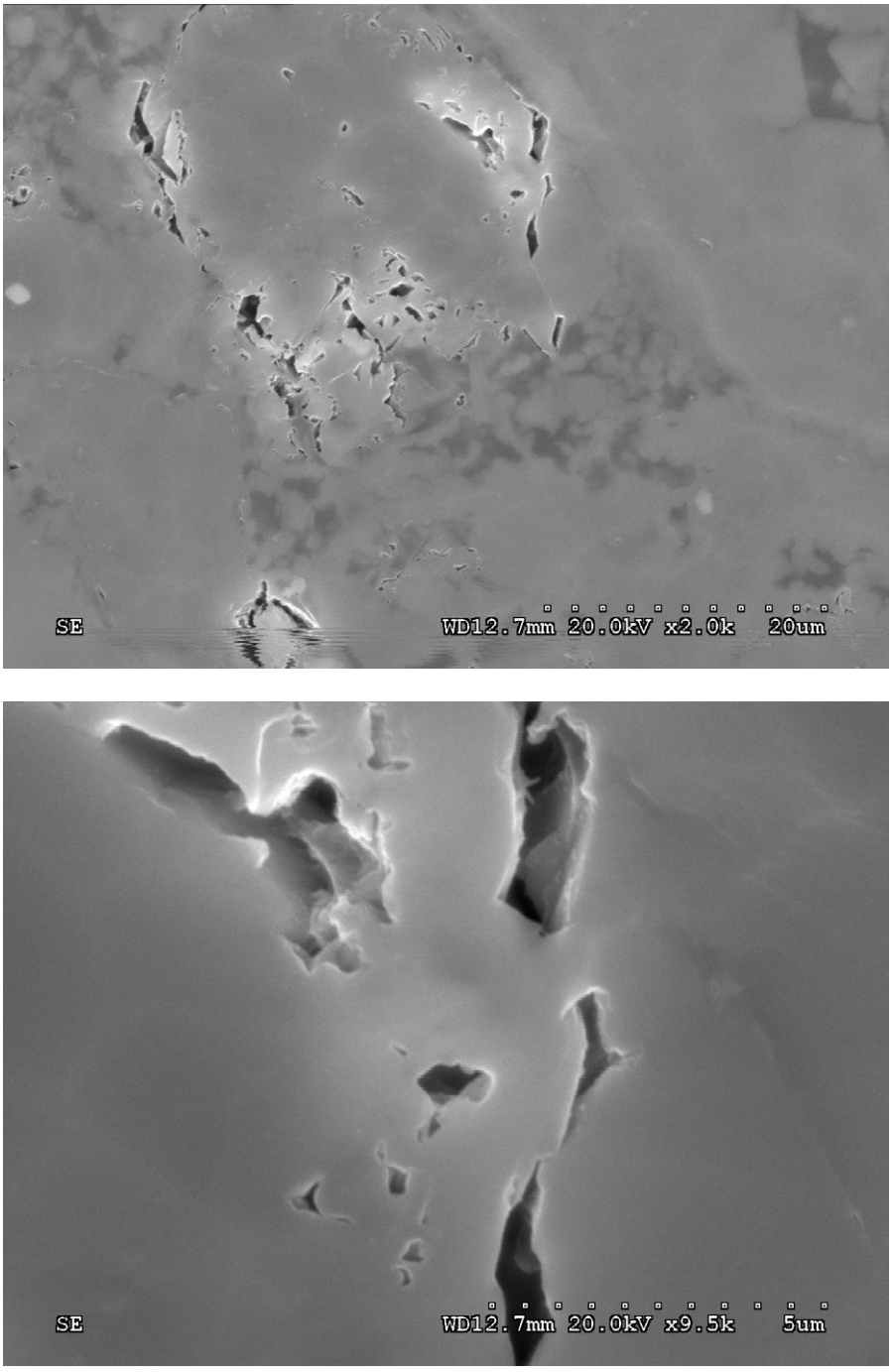


Fig. 65: Shale sample after ion polishing with clearly visible micropores.

ALUMINUM / SILICON ALLOY

Aim of analysis

Preparation of a smooth sample surface for EBSD that is free of defects.

Preparation problem

Mechanical polishing for an EBSD examination is not sufficient

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Polish the surface using U-type diamond films: 9 µm, 2 µm and 0.5 µm
> Preparation time: 20 min

ION MILLING	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	2 mA	2.2 mA
Milling angle	10.5°	3°
Sample movement	Rotation	Rotation
Translation movement	± 3 mm	± 3 mm
Milling time	10 min	1 h 30 min

Result

The sample surface looks very good after ion polishing. It was possible to remove all of the scratches caused as a result of mechanical polishing. The sample should be applicable for EBSD examinations.

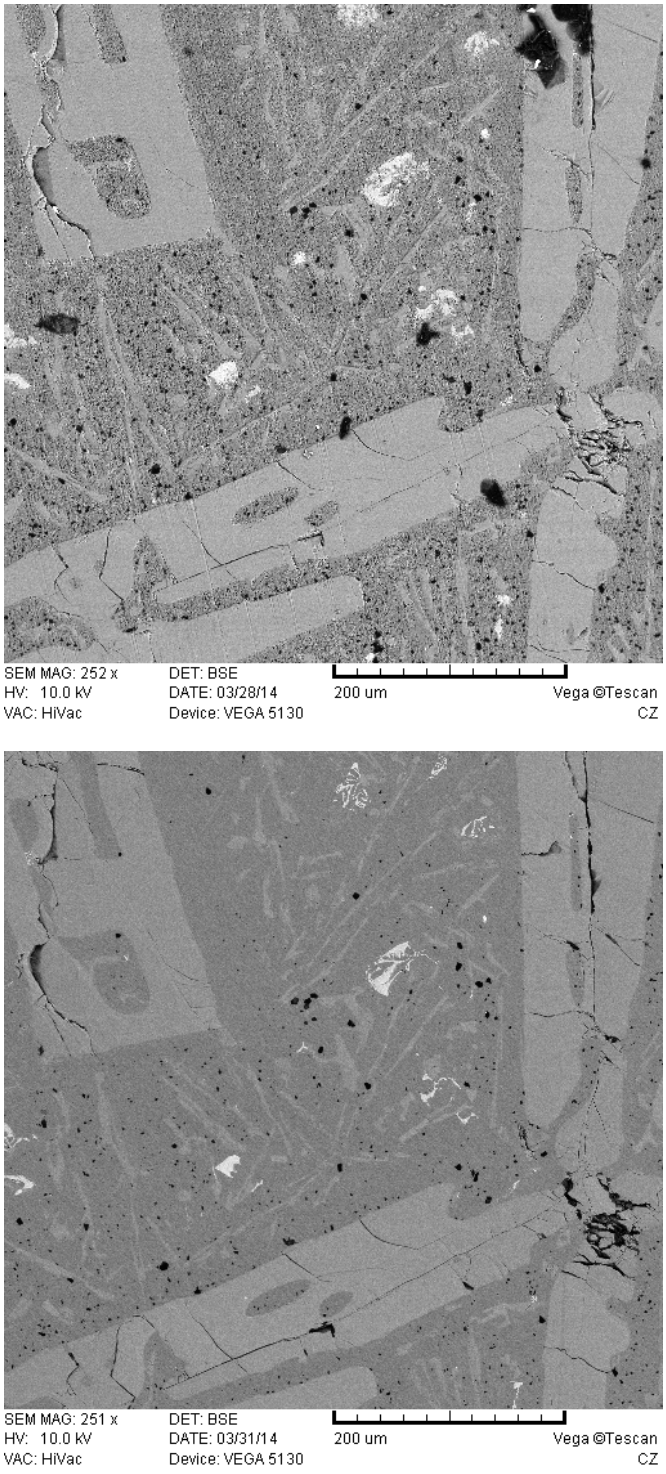


Fig. 66: Al/Si alloy after mechanical polishing using the Leica EM TXP (above) and after additional ion polishing (below)

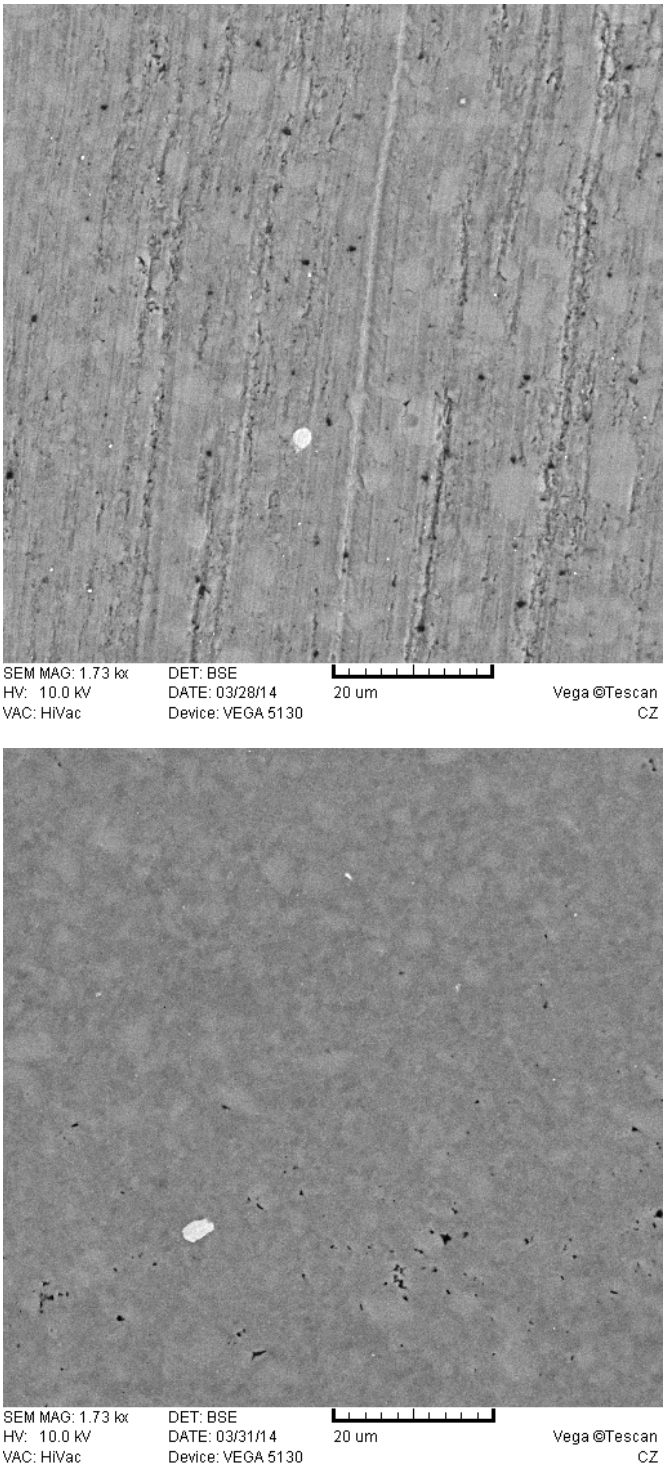


Fig. 67: Al/Si alloy after mechanical polishing (above) and after additional ion polishing (below)

CALCIUM CARBONATE (CALCITE AND ARAGONITE)

Aim of analysis

Preparation of a perfect sample surface for EBSD examinations

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Polish the surface using U-type diamond films: 9 µm, 2 µm and 0.5 µm
> Preparation time: 20 min

ION MILLING	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	1.5 mA	2.2 mA
Milling angle	9°	3°
Sample movement	Oscillation (±45°)	Oscillation (±45°)
Translation movement	± 4 mm	± 4 mm
Milling time	15 min	2 h

Result

The surface was still rather rough after mechanical preparation. It was possible to improve the surface quality significantly with ion polishing. Both calcite and aragonite are visible. The sample should then be used for EBSD.

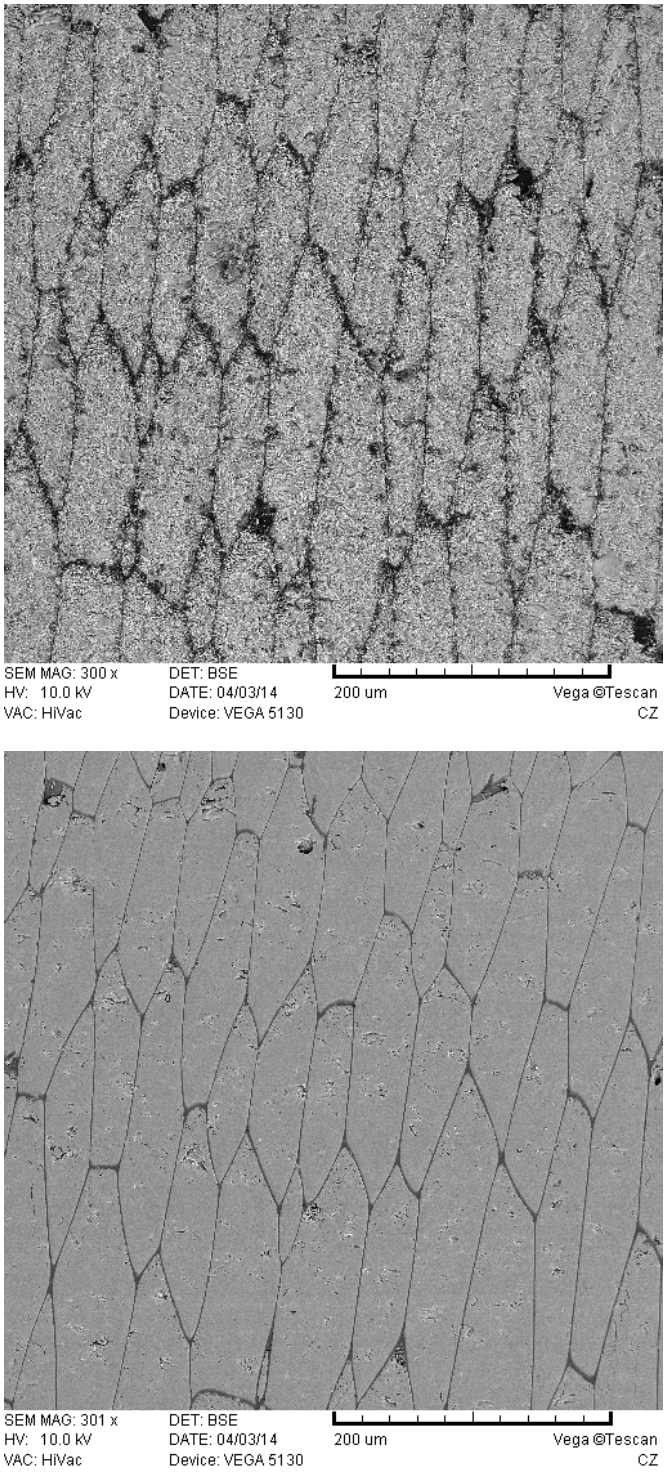
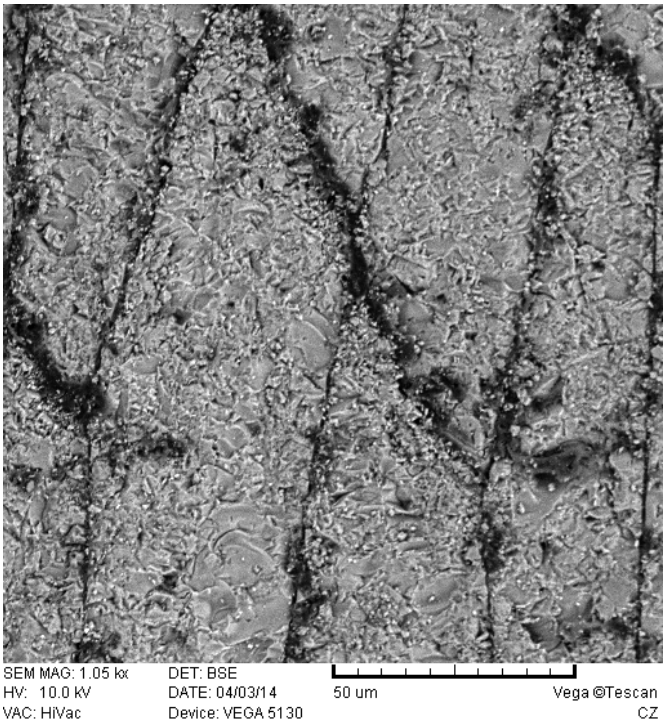


Fig. 68: Calcium carbonate after mechanical polishing (above) and after additional ion polishing (below)



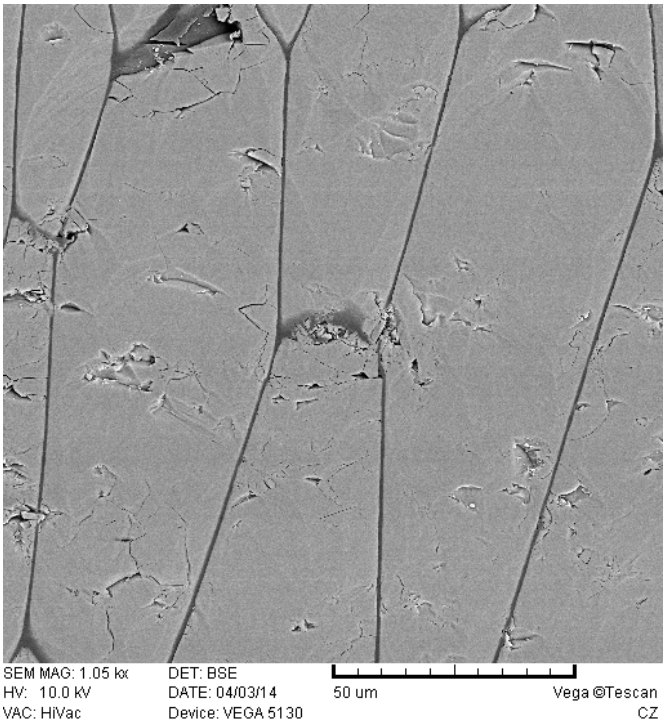
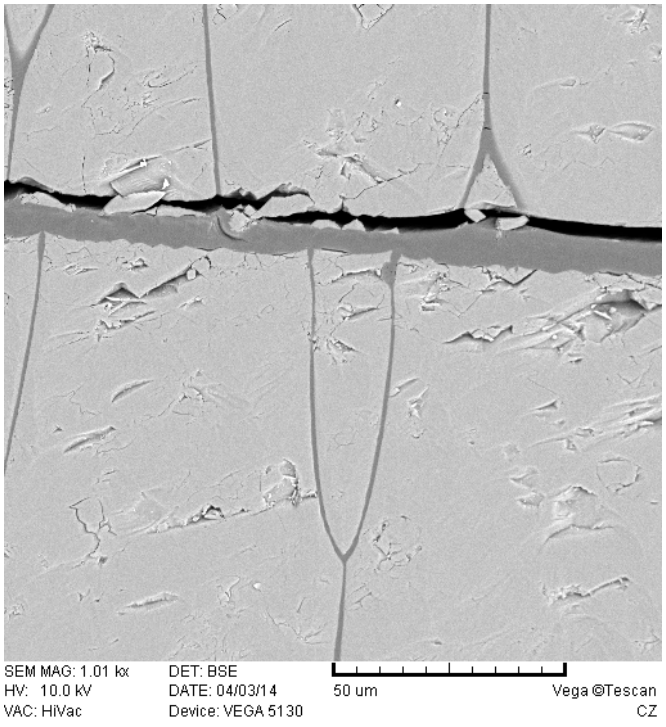


Fig. 69: Calcium carbonate after mechanical polishing (above) and after additional ion polishing (below)



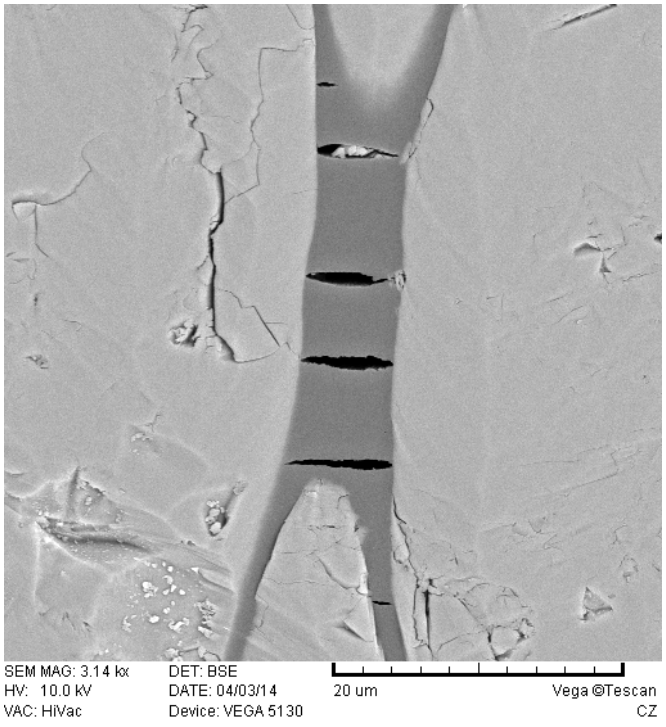


Fig. 70: Calcium carbonate after additional ion polishing

FLINT

Aim of analysis

Preparation of a smooth surface

Preparation problem

The small pores become clogged with dirt during mechanical polishing.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Polish the surface using U-type diamond films: 9 µm, 2 µm and 0.5 µm
> Preparation time: 20 min

ION MILLING	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	1.5 mA	2.2 mA
Milling angle	9°	3°
Sample movement	Oscillation (±45°)	Oscillation (±45°)
Translation movement	± 4 mm	± 4 mm
Milling time	15 min	2 h

Result

The surface of the flint is nearly perfect after ion polishing. There are no visible contaminants or scratches. The small pores are clearly visible.

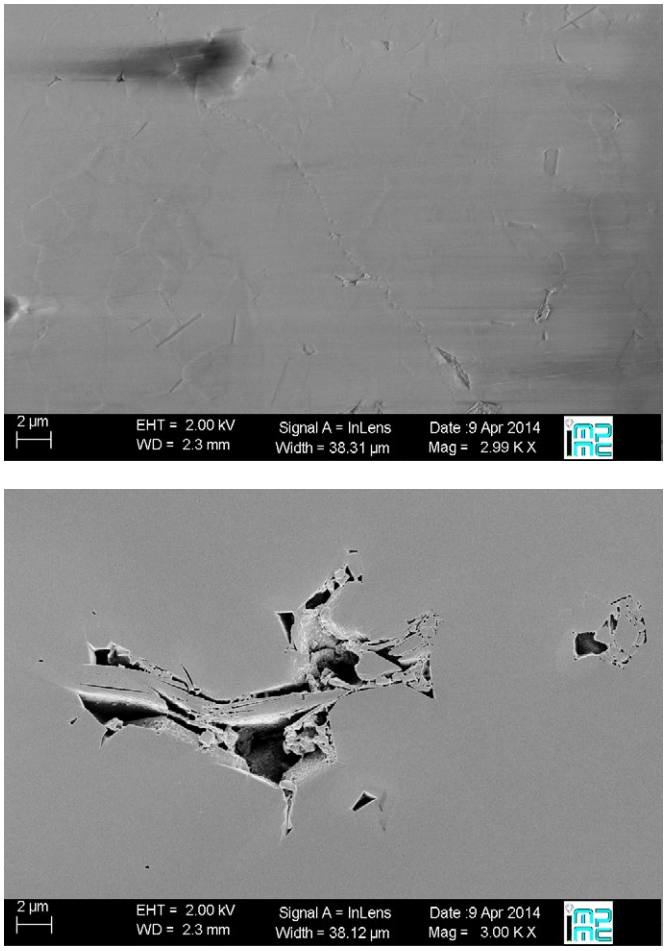


Fig. 71: Flint after mechanical polishing (above) and after additional ion polishing (below)

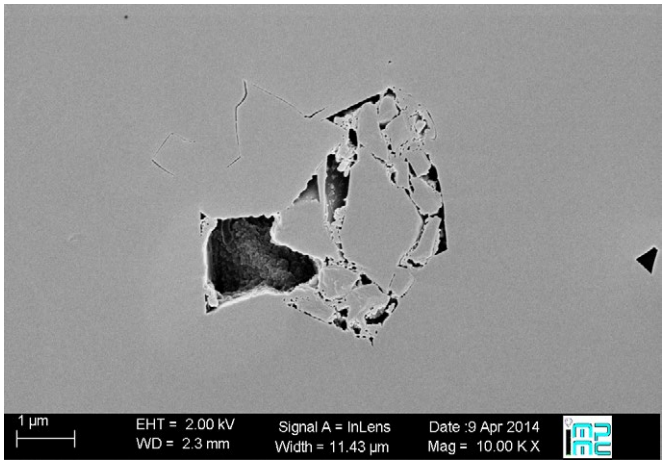


Fig. 72: Flint after additional ion polishing

GOLD

Aim of analysis

Preparation of a smooth surface, nondestructive surface for EBSD

Preparation problem

Gold is a very soft material that is hard to polish mechanically. The sample surface often becomes smeared with dirt.

Preparation conditions

Mechanical pre-preparation:

> Mechanical polishing

ION MILLING	Cleaning	Polishing
Acceleration voltage	4 kV	6 kV
Gun current	2 mA	2.2 mA
Milling time	10 min	2 h
Milling angle	10.5°	3°
Sample movement	Rotation	Rotation
Translation movement	± 5 mm	± 5 mm

Result

It was possible to improve the surface quality of the gold significantly with ion polishing. The surface is now free of scratches generated during mechanical polishing. EBSD examinations are possible now.

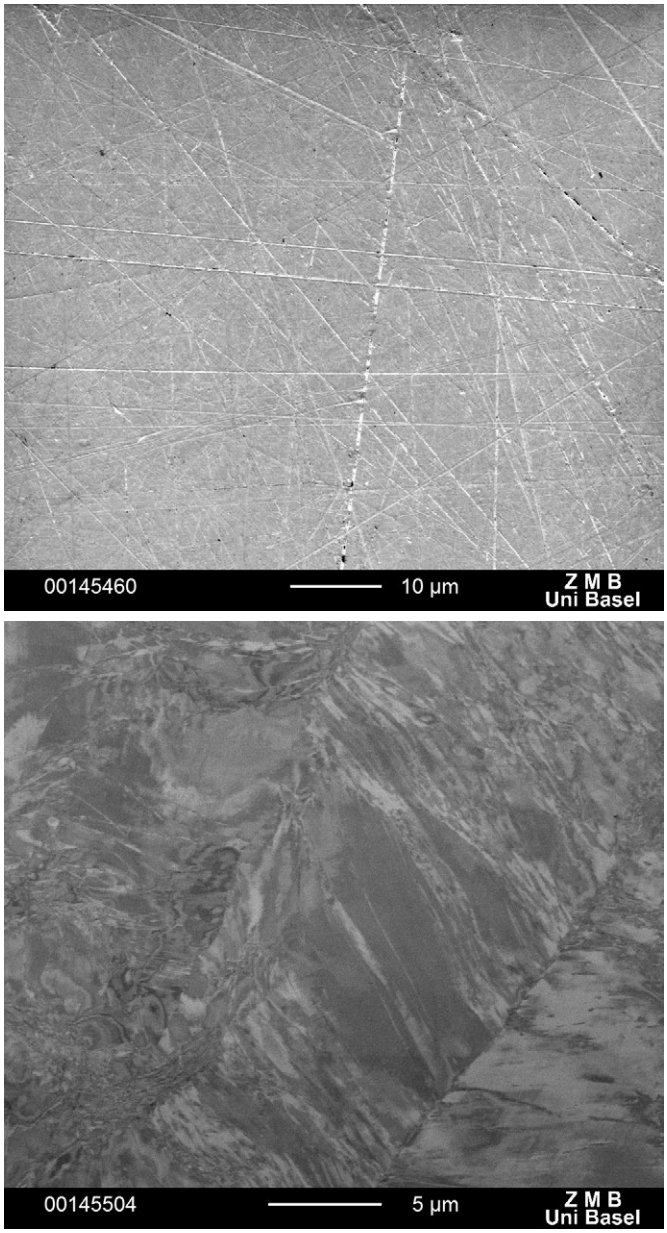


Fig. 73: Gold with clearly visible scratches after mechanical polishing (above) and after additional ion polishing (below) (Images: Markus Dürrenberger, ZMB, University of Basel).

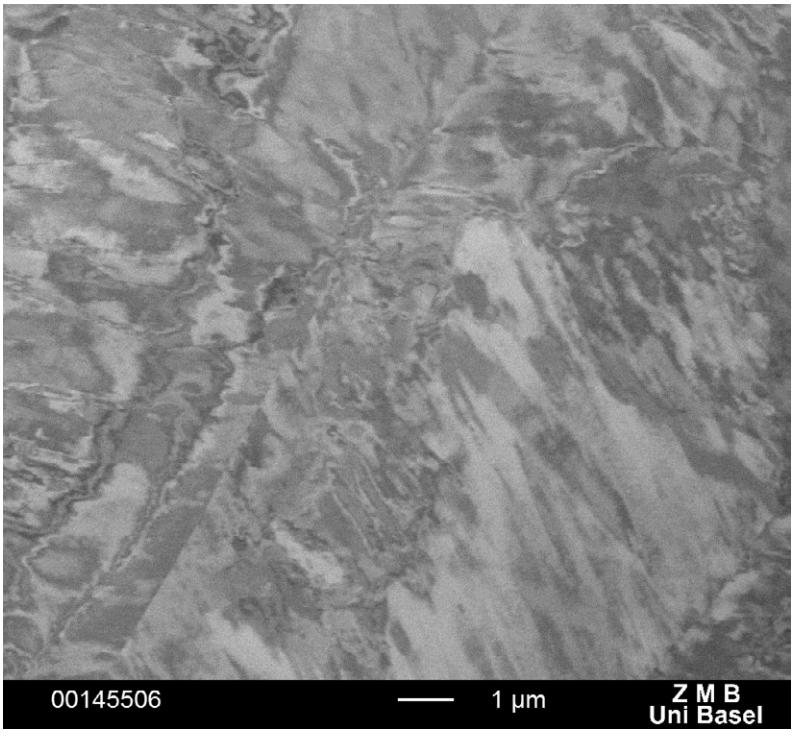


Fig. 74: Gold surface after additional ion polishing (Images: Markus Dürrenberger, ZMB, University of Basel).

2.4. Sample preparation for EBSD

EBSD is a surface sensitive examination method with an information depth of just a few nanometers. For sample preparation, this results in very high requirements. The sample surface should be very smooth and free of damage of any kind.

These requirements can be met using two different methods of ion beam preparation: ion beam slope cutting and ion beam polishing.

Prepared areas of no more than 8 mm in length and 1–2 mm in depth are achieved during ion beam slope cutting. Mechanical pre-preparation is necessary only for samples with an irregular shape or in the event of site-specific preparation.

Ion beam polishing, on the other hand, requires perfect mechanical pre-preparation.

Here, however, with the use of a Leica EM TIC 3X rotary stage, a surface with a diameter of 25 mm can be prepared uniformly to ensure that the samples can then be used for an EBSD examination.

Some preparation results are displayed in the following section:

GRAPHITE FLAKES/DIAMOND/AL COMPOSITE MATERIAL

Preparation goal

Perfect and nondestructive composite material surface for the EBSD examination

Preparation problem

Extreme differences in the hardness of materials makes preparing the samples very complicated.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the samples to a Leica EM TIC 3X Al holder
> Sand the cut face using 9 µm diamond foil

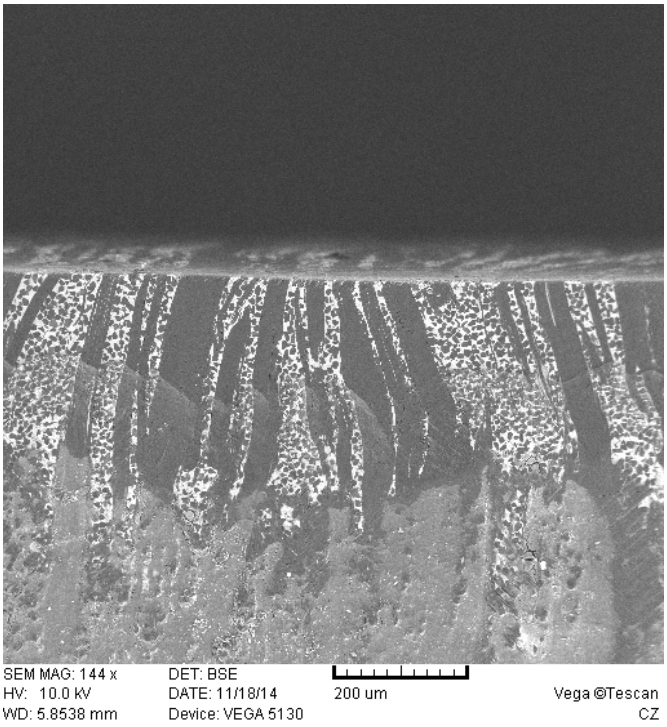
ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	10 h

Result

Despite the extreme difference in hardness among the different materials in the composite, it was possible to prepare a smooth section.

Fig. 75 shows the prepared surface with the Al matrix, the diamond grains and the graphite flakes.

EBSD examinations of the surface provide excellent Kikuchi diffraction patterns of all components (Fig. 76). Kikuchi diffraction patterns attained from graphite for the first time showcase the extraordinary quality of the sample preparation /4/.



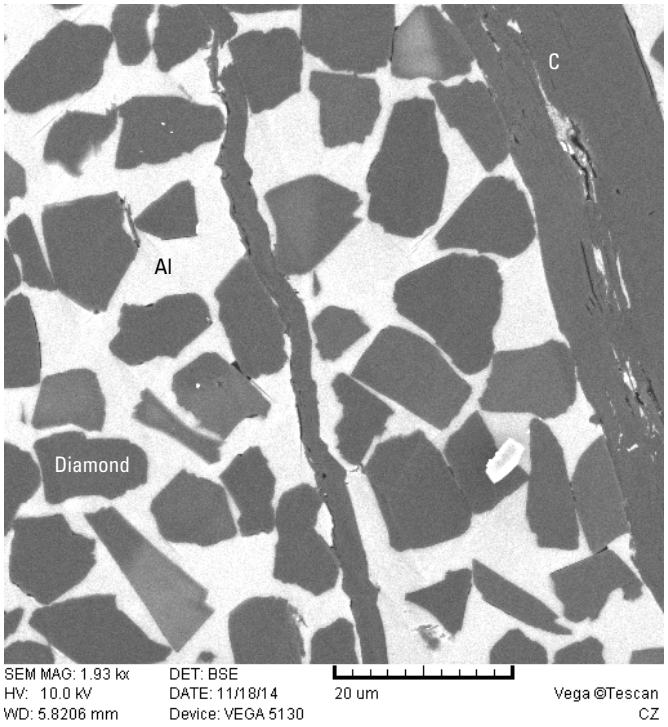


Fig. 75: The cross-section of the composite material shows a flat surface with the Al matrix, the diamond grains and the graphite flakes.

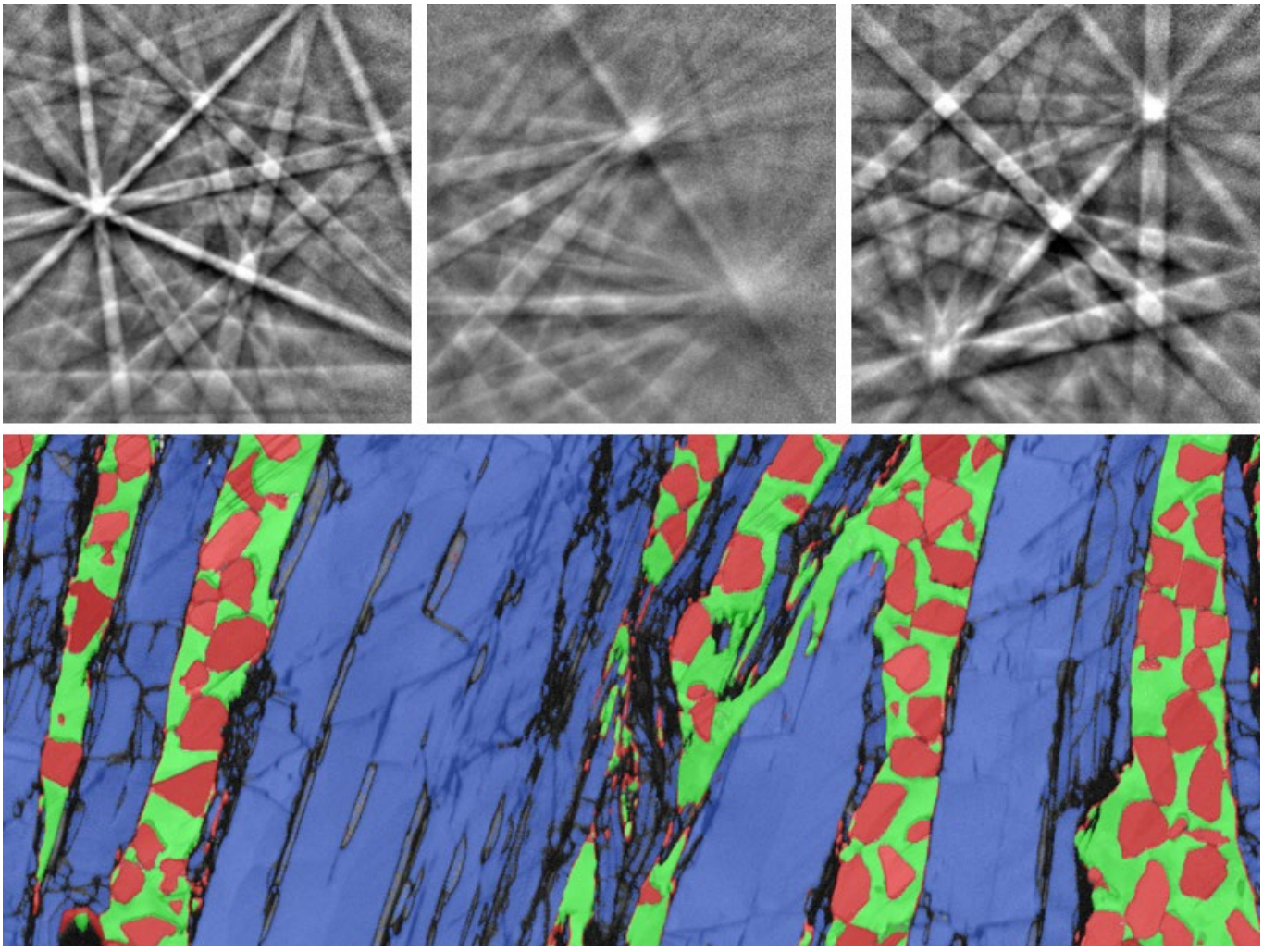


Fig. 76: EBSD map phase of Al, graphite and diamond and the corresponding Kikuchi diffraction patterns (Laurie Palasse, Bruker Nano GmbH Berlin)

GOLD WIRE CONTACT

Aim of analysis

EBSD examination on a gold wire contact (site-specific preparation)

Preparation problem

Site-specific preparation is necessary for the preparation of this semiconductor structure.

Preparation conditions

Mechanical pre-preparation with the Leica EM TXP:

> Affix the sample to the Leica EM TIC 3X Al holder and fasten it in the adapter for the Leica EM TXP.
> Sand the cut face using 9 µm diamond foil until the gold contact is visible
> 45° wedge of the upper edge of the sample for reducing the material to be etched (also refer to Chap. 3)

Preparation time

> 20 min

ION MILLING	
Acceleration voltage	7 kV
Gun current	2.6 mA
Milling time	3 h

Result

It was possible to prepare a very smooth, nondestructive cross-section of the gold wire contact. The results of the EBSD examination demonstrate the high quality of the sample preparation /4/.

It was possible to drastically reduce the preparation time for the site-specific preparation by using the Leica EM TXP for the pre-preparation.

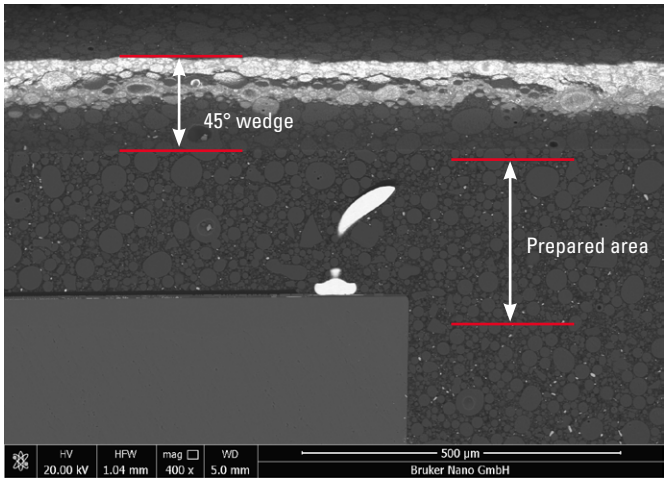


Fig. 77: Cross-section of gold wire contact with a 45° wedge on the upper edge of the sample to reduce the milling time

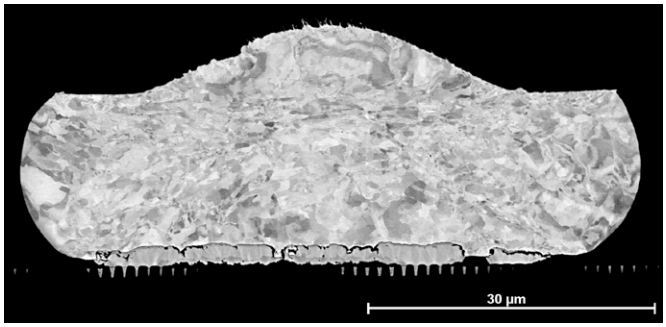
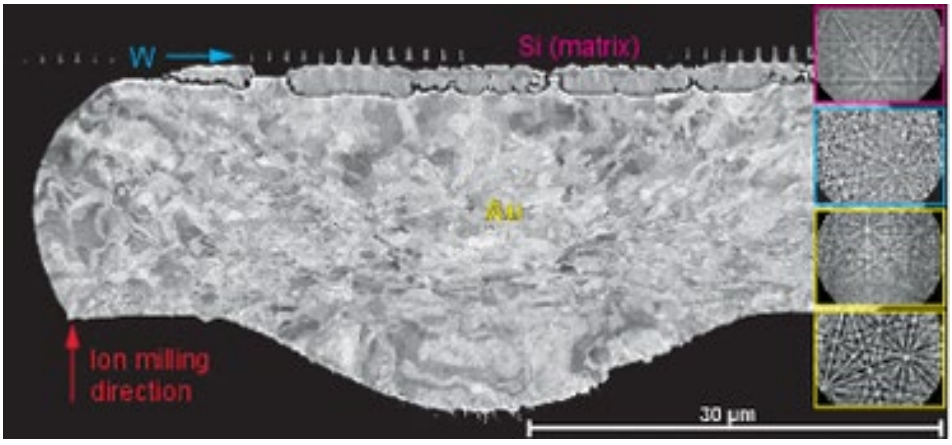
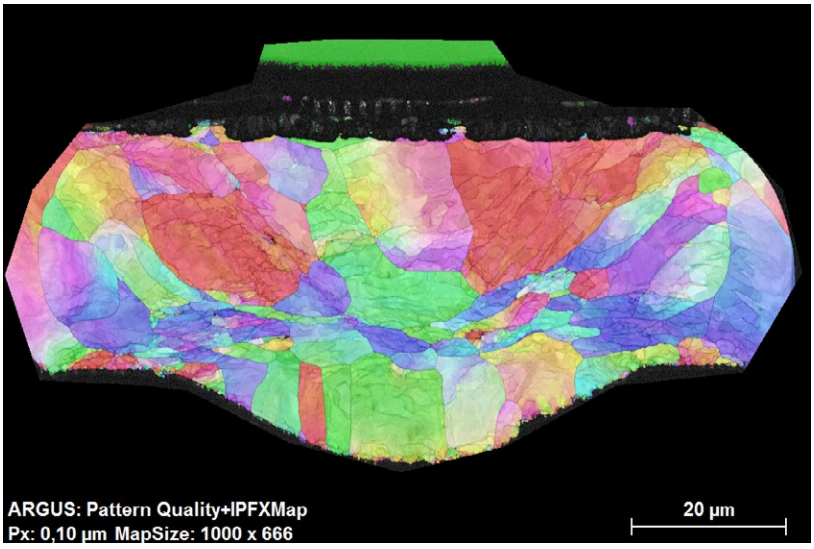


Fig. 78: Cross-section of the gold wire contact (Laurie Palasse, Bruker Nano GmbH, Berlin)





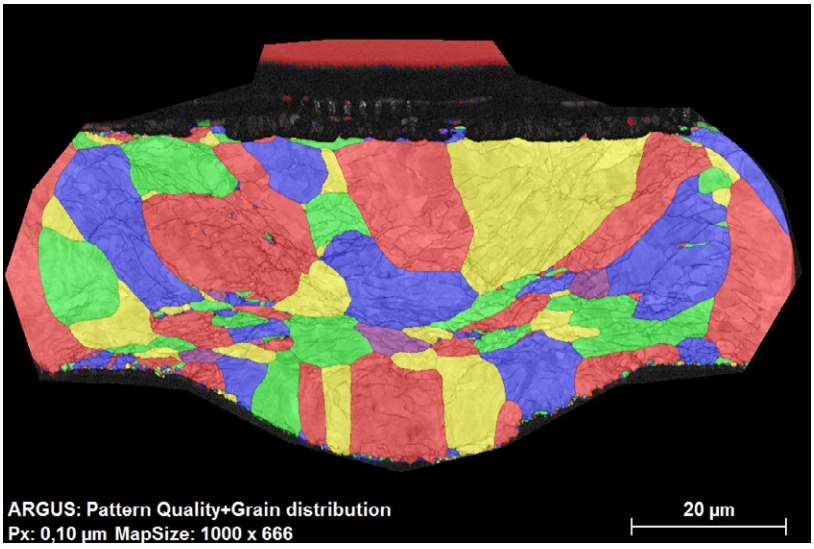


Fig. 79: SEM image with Kukuchi diffraction patterns and EBSD examination result for the gold wire contact (Laurie Palasse, Bruker Nano GmbH, Berlin)

3. SYNERGY WITH THE LEICA EM TXP

The Leica EM TXP is an instrument for the mechanical site-specific preparation of samples for optical and electron microscopy.

Samples can be sawed, polished, milled and drilled.

The preparation process can be observed at any time using a stereo-microscope. The arm to which the sample holder is fastened can be tilted. As a result, the sample surface as well as the cross-section surface are easily visible.

Samples and tools can be adjusted to match each other precisely. This is of major importance, particularly for site-specific preparation.

Samples can be completed by mechanical preparation or pre-prepared for ion milling in the Leica EM TIC 3X using the Leica EM TXP. Sample surfaces can be prepared for ion polishing through mechanical polishing. In the case of cross-section sample preparation, approaching the target area also requires mechanical pre-preparation of the sample in the event of site-specific preparation (see also Fig. 5).

If the structure is located well below the sample surface and the material above the structure is not of any interest, the following technique is also used. The edge of the material located above the structure of interest is ground using a 45° wedge. This is possible because the sample holder can be installed on a tiltable arm. The 45° wedge also requires a specifically shaped mask (wedged mask, Fig. 82). The preparation time for ion beam milling can be drastically reduced using this technique.

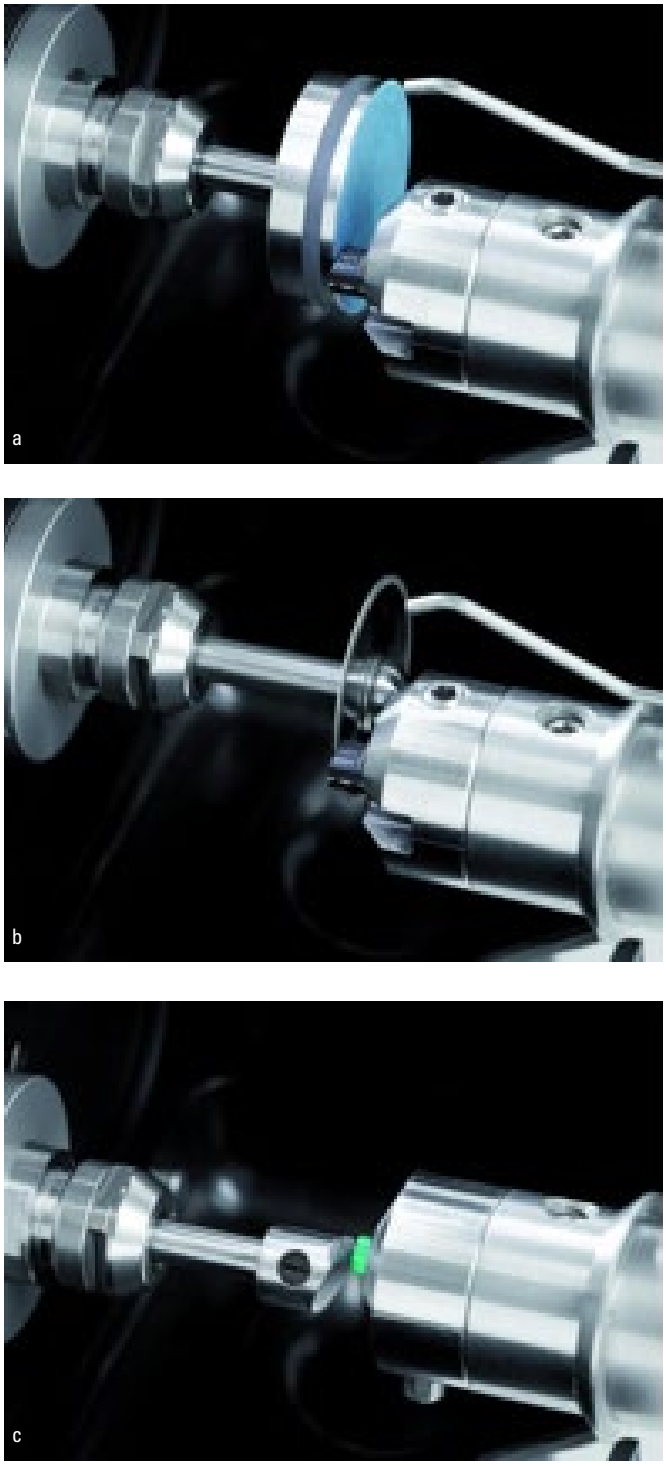
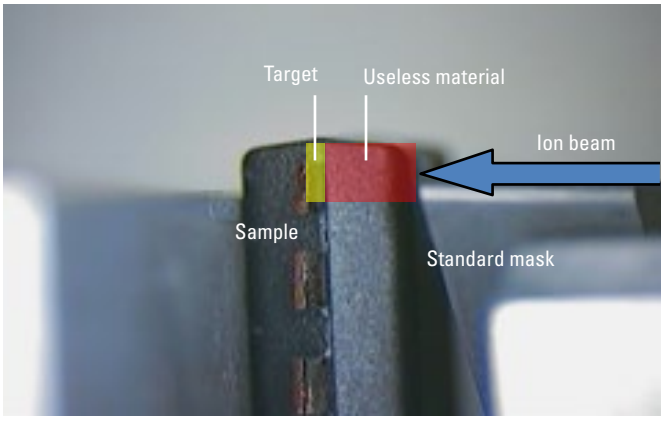
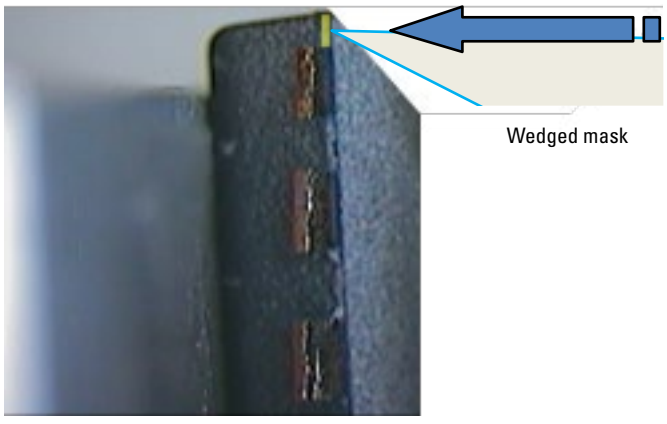


Fig. 80: Leica EM TXP with tools for polishing (a), sawing (b) and milling (c)

Fig. 81: Leica EM TXP for the site-specific preparation of samples for optical and electron microscopy



Preparation with a standard mask



Preparation with a wedged mask

Fig. 82: 45° wedge on the sample edge to remove unnecessary material. The milling time is reduced drastically as a result.

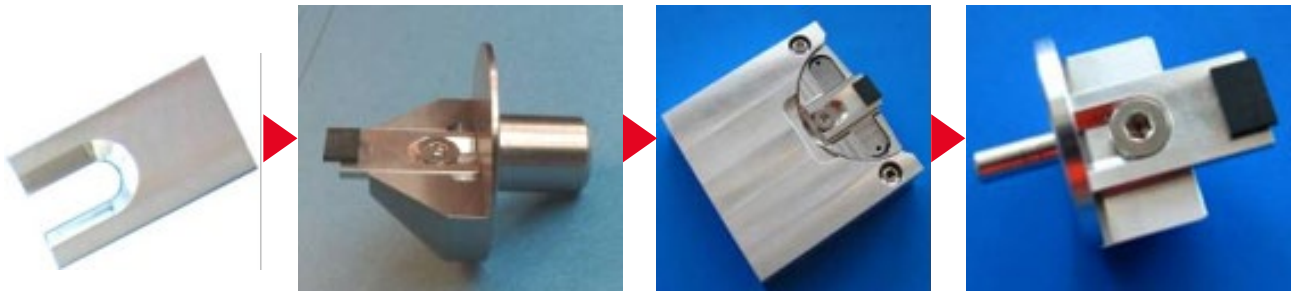


Fig. 83: Preparation process with the same specimen holder

Synergy between the Leica EM TXP and the Leica EM TIC 3X is also readily apparent in the use of sample holders for the entire preparation procedure. This enables the sample to be fastened and processed in the Leica EM TXP after it is fastened to an Al holder using an adapter. After pre-preparation in the Leica EM TXP, the sample remains on the Al holder and is prepared further in the Leica EM TIC 3X.

The sample, along with the Al holder, can finally be fastened to an SEM holder and examined using SEM. This ensures it is possible to conduct repeated SEM examinations of the sample after different processing steps without changing the sample holder. Additionally, the sample always remains at the pre-configured adjustment.

Reference

/1/ W. Qiu, B. Zee, F. J. Foo, W. Grünewald:
Alternative Flip Chip Sample Preparation Technique Using Triple Ion Beam Milling. 16th Electronics Packaging Technology Conference, Singapore, December 2014 – Proceedings.

/2/ Gang Ji, Zhanqiu Tan, Rajashekhara Shabadi, Zhiqiang Li, Wolfgang Grünewald, Ahmed Addad, Dominique Schryvers, Di Zhang:
Triple ion beam cutting of diamond/Al composites for interface characterization. Materials Characterization 89 (2014) 132–137.

/3/ Joyce Schmatz, Oliver Schenk, Jop Klaver, Janos Urai, Wolfgang Grünewald:
Triple-beam Ar-Ion-Milling with a Rotary Stage to Decorate Grain Boundaries and Substructures in Rock Salt. Leica Microsystems Science Lab.

/4/ Laurie Palasse, Wolfgang Grünewald:
High quality sample preparation for EBSD Analysis by broad beam ion milling. Poster EBSD 2015, University of Strathclyde, Glasgow, UK.
Leica Microsystems Science Lab

Leica
MICROSYSTEMS

CONNECT
WITH US!

qualityaustria
SYSTEM CERTIFIED
ISO 9001:2008 No.00805/0
ISO 14001:2004 No.02783/0

Leica Mikrosysteme GmbH | Vienna, Austria
T +43 1 486 8050-0 | F +43 1 486 8050-30

www.leica-microsystems.com