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SAMPLE PREPARATION FOR EBSD

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Dr. Grzegorz Cios has been working at the Academic Centre for Materials and Nanotechnology at AGH University of Krakow since defending his MSc thesis in 2014 in the group of Piotr Bała. During his PhD, he investigated nickel-based alloys with high-volume fractions of carbides for components with improved wear resistance. He defended his PhD thesis in Materials Science in 2019. From the early stages of his career, his main scientific interest has been electron backscatter diffraction (EBSD). He learned EBSD under the supervision of Tomasz Tokarski and later established a collaboration with Gert Nolze and Aimo Winkelmann, resulting in around a dozen joint publications that expanded the understanding and pushed the limits of EBSD methodology. At the EMAS 2019 Workshop in Trondheim, he received the award for the best early career scientist presentation for his work on resolving martensite tetragonality using EBSD. In 2019, he joined the European Microbeam Analysis Society Board as a student representative, and since 2023 he has served as a co-opted Board member.

He has co-authored over 100 peer-reviewed publications. His most important achievements include resolving the tetragonality of high-carbon martensite using EBSD, orientation analysis of quasicrystals using EBSD, the development of an improved methodology for 0° tilt EBSD, and, together with his team, the development in 2026 of a method for resolving overlapping EBSD patterns.

1. INTRODUCTION

A key question is what requirements a sample surface must meet to provide reliable information, both in terms of chemical composition and crystallographic data. The following sections describe the methodology of sample preparation for electron backscatter diffraction (EBSD) measurements. Although the article focusses specifically on EBSD, the presented preparation procedures are also applicable to other scanning electron microscopy (SEM) based techniques and microanalysis methods. This is because EBSD is one of the most demanding techniques in SEM in terms of surface quality. Therefore, a sample prepared to meet EBSD requirements is, in most cases, also suitable for conventional SEM imaging and microanalytical investigations.

2. WHAT MAKES A GOOD EBSD SAMPLE?

The first question that should be addressed is: what makes a good EBSD sample? The answer is not straightforward and continues to evolve with advances in both instrumentation and data analysis methods. In general, a sample can be considered well prepared when the EBSD measurement provides reliable information about the investigated material without introducing misleading artefacts. In practice, the answer often depends on the specific scientific question being asked.

From the author's experience, certain surface imperfections may be acceptable for some analyses. For example, a sample containing a few scratches can still provide reliable information about average grain size or texture. However, the same sample would not be suitable for analyses based on local orientation gradients, such as geometrically necessary dislocation (GND) estimation. In many cases, scratches and reduced indexing rates are less problematic than deformation introduced during improper cutting or grinding. A scratch-free sample with a 100 % indexing rate may still produce misleading results if subsurface deformation remains present. An example of such a case, where the surface appears acceptable but preparation artefacts are visible in the EBSD microstructure, is shown in Fig. 1.

Although some compromises are often unavoidable, a good EBSD sample should ideally exhibit the following characteristics:

- No additional deformation introduced during preparation (as in Fig. 1), typically caused by excessive pressure or overly aggressive grinding.
- No scratches. While minor scratches can sometimes be tolerated, they should generally be minimised.
- No pull-outs, including loss of structural constituents, cavities after dissolution of water-sensitive phases, or voids left by brittle inclusions detached from the matrix.
- No contamination by foreign material. Hard abrasive particles may become embedded in the surface, particularly in soft materials.

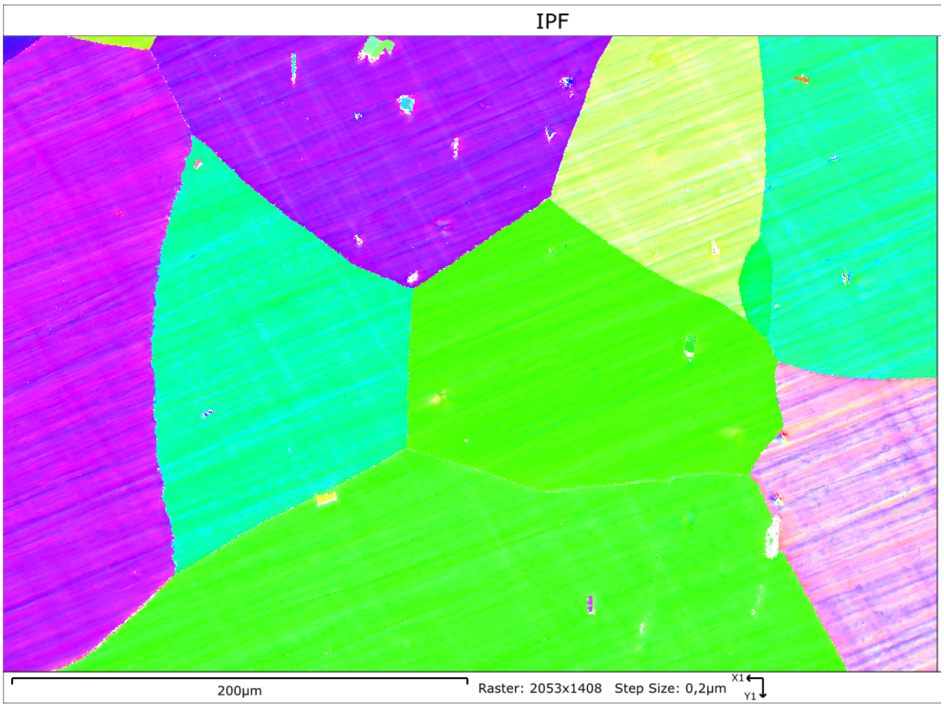


Figure 1. Improperly prepared ferritic stainless steel sample IPF map. Hit rate > 99 %.

- No smearing. Smearing refers to plastic deformation over larger surface regions and is commonly associated with insufficient lubrication during polishing.
- No surface relief. Relief corresponds to height differences between individual microstructural constituents and may develop even between grains of the same material, particularly after prolonged polishing with colloidal silica. Complete elimination is often difficult.
- No edge rounding. Although not always critical, edge rounding can become problematic when the region of interest is close to the sample edge. It usually results from faster wear of the mounting resin relative to the sample or from gaps between the sample and resin.
- No contamination or staining. Staining most commonly occurs after polishing or etching due to improper cleaning or drying of the sample, or after exposure to a humid environment.
- No charging. Charging is generally related to the insulating nature of the analysed material, although the effect may be intensified by surface porosity, poor electrical contact, or sharp edges.

3. SAMPLING

Proper sampling for EBSD analysis can be crucial for the subsequent interpretation of results, particularly when crystallographic texture is of interest. In many cases, the investigated materials originate from metallurgical processes such as rolling, drawing, or extrusion, where characteristic textures are expected. For this reason, careful documentation of the sampling procedure is strongly recommended. Taking photographs during sampling is often helpful and may later assist in the interpretation of the results.

Whenever possible, samples with rectangular cross-sections should be prepared, as they simplify orientation of the reference frame and reduce the risk of misinterpretation compared to square or circular specimens.

Sample orientation becomes even more important in the case of geological, mineralogical, or ice samples. In such cases, photographic documentation alone is often insufficient. It is advantageous to record the sample position on a map and preserve information about its orientation with respect to geographical directions. An example of an extreme case of defining the sample reference frame is shown in Fig. 2.

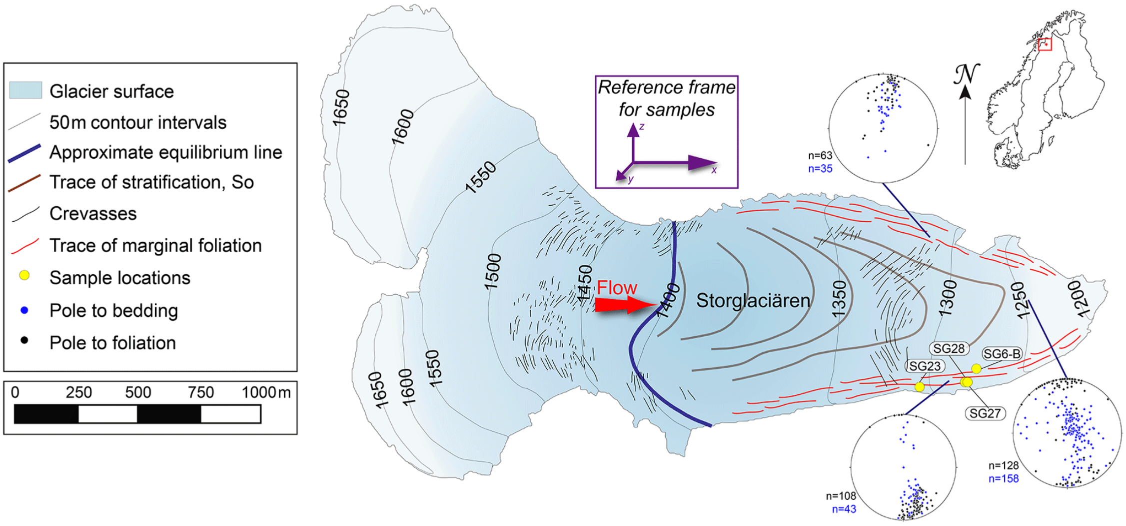


Figure 2. An example of documented sampling points with a reference frame overlaid on map of Storglaciären (https://tc.copernicus.org/articles/15/303/2021).

4. CUTTING

Collected samples usually require additional preparation before they can be placed in the SEM and analysed using EBSD. The first step is cutting. In metallography, samples should be cut using methods that introduce the smallest possible alteration to the original microstructure. The most common artefacts generated during cutting are plastic deformation and the formation of a heat-affected zone. Wire sawing and low-speed cutting with a diamond disc are generally considered the most suitable techniques for EBSD sample preparation (Fig. 3), as they minimise mechanical and thermal damage. However, both methods are relatively slow and have limited throughput. For this reason, water-cooled SiC disc cutting or wire electrical discharge machining (EDM) is often used as a practical compromise between preparation quality and efficiency. Wire EDM is particularly effective for hard metals and metal matrix composites. Band saw cutting is widely used because of its high efficiency when sectioning low- and medium-hardness metals. However, it also produces the thickest deformation layer beneath the cut surface and therefore usually requires more extensive subsequent grinding and polishing.

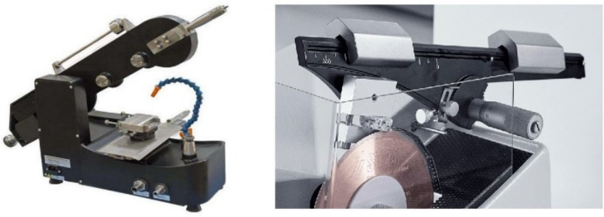


Figure 3. Examples of diamond wire saw and low speed diamond disc saw.

5. DEMAGNETISATION

Ferromagnetic materials may exhibit remanence and, therefore, behave, to some extent, like permanent magnets. Since magnetic fields interact strongly with electrons, this can significantly influence SEM and EBSD measurements. Electrons entering a magnetic field perpendicular to their trajectory follow curved paths, whereas electrons travelling parallel to the magnetic field direction continue approximately in straight lines.

A commonly used physics example illustrates the scale of this effect: an electron accelerated through a potential difference of 5 kV and entering a magnetic field of 0.02 T perpendicular to its trajectory follows a circular orbit with a radius of approximately 1.2 cm. In SEM, electron beam energies can reach 30 kV, while strong neodymium magnets may generate magnetic fields of up to 1.4 T. Under such conditions, magnetic fields can substantially influence both the primary electron beam and the backscattered electrons travelling toward the EBSD detector.

As a consequence, the presence of a permanent magnetic field near the analysed area can severely distort EBSD patterns and make quantitative analysis difficult or even impossible. Examples of EBSD patterns strongly and weakly distorted by magnetic fields are presented in Fig. 4.

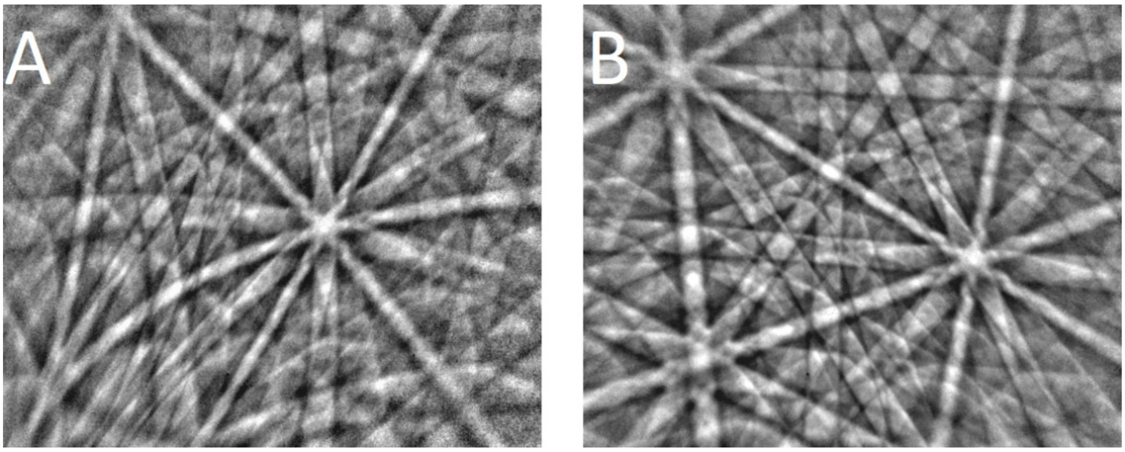


Figure 4. a) Ni patterns collected close, and b) far from permanent neodymium magnet (collecting pattern from magnet was not possible).

Different demagnetisation methods may be more suitable for different classes of materials. For example, in the case of permanent magnets or materials with relatively low Curie temperatures, heating above the Curie temperature may be the most effective approach, provided that the heat treatment does not alter the microstructure of interest. In contrast, for metallic materials and alloys, demagnetisation using permanent magnet systems or induction-based demagnetising devices is often more practical and effective.

6. PLATING

When the region of interest is located close to the sample surface, such as in the case of oxide scales or carburised/decarburised subsurface layers, applying an additional electrodeposited nickel coating can significantly improve edge retention during preparation. In the case of oxide scales, the deposited nickel mechanically supports the oxide layer, reducing the risk of cracking, pull-out, or detachment during grinding and polishing, while also limiting edge rounding.

The plating procedure is typically preceded by gold sputter coating to ensure sufficient surface conductivity. The sample is then immersed in a nickel plating bath (Table 1), with a nickel plate serving as the anode. Under typical conditions, a nickel layer approximately 60 µm thick can be deposited after about 6 hours using a current density of approximately 25 mA/cm².

Table 1. Composition of the Ni-plating solution (after [15]).

30 g	$H_4N_2NiO_6S.4H_2O$	(nickel (II) sulphamate)
31 g	$NiCl_2.6H_2O$	(nickel (II) chloride hexahydrate)
31 g	H_3BO_3	(boric acid)
1000 ml	H_2O	(distilled water)

7. FIXING AND MOUNTING

During cutting and mounting, EBSD users should carefully consider the geometry of the final SEM setup. This aspect is often overlooked during sample preparation, although it can strongly influence the feasibility and safety of EBSD measurements. Some of the most important factors are listed below.

i) Use of a load lock. When a load lock system is used, the maximum allowable sample dimensions are often restricted. This should be considered already at the cutting and mounting stage.

ii) Sample stage geometry. The stacking sequence of the stage axes is particularly important. Stages with the tilt axis located below the Z-axis generally accommodate taller samples more easily than stages in which the Z-axis is positioned below the tilt mechanism.

iii) Analytical working distance. Currently, two major EBSD detector manufacturers offer in-chamber tiltable detectors, while two others use fixed detector geometries. Most fixed-geometry detectors are positioned to provide optimal performance close to the preferred EDS working distance, typically between 4 and 15 mm. Under such conditions, using large sample holders may create a significant risk of collision with the SEM pole piece. For example, a sample mounted in the centre of a 30 mm diameter puck may not be movable to shorter working distances required for optimal EBSD acquisition (Fig. 5a).

    Several representative geometries are illustrated in Fig. 5. In Fig. 5a, the sample is mounted in the centre of the puck, which prevents safe movement to shorter working distances favourable for EBSD. More suitable configurations for simultaneous EDS and EBSD acquisition are possible. In Fig. 5b, the samples are positioned close to the edge of the puck, allowing combined EDS/EBSD measurements at a working distance of approximately 6 mm, assuming both detectors are optimised for this geometry. Measurements on the remaining mounted samples can still be performed using non-eucentric stage rotation. Figure 5c presents a side-view geometry optimised for a 6 mm working distance, whereas Fig. 5d demonstrates how detector movement (downward in this example) can improve accessibility and reduce the risk of collision with the pole piece. This configuration, however, may compromise simultaneous EDS and EBSD acquisition.

    Longer working distances may also be advantageous for large-area EBSD mapping, as they provide a larger field of view and increased depth of focus. Another important aspect to consider during mounting is the effect of temperature on the investigated material.

iv) Effect of mounting temperature. During hot mounting, samples are typically exposed to temperatures of approximately 180 - 200 °C for at least several minutes. Such thermal exposure may significantly alter sensitive microstructures. For example, it can lead to complete recrystallisation of zinc alloys or promote carbide precipitation in quenched steels. Cold-mounting resins may also generate substantial heat during polymerisation, in some cases reaching temperatures of around 110 °C for short periods. In general, longer curing times are associated with lower peak temperatures. Some resin manufacturers offer moderators that reduce the maximum curing temperature while extending the curing time.

    Hot mounting has the advantage of producing mounts with two parallel surfaces, which simplifies later preparation steps. In contrast, cold-mounted samples often require additional grinding or machining to obtain parallel planes. Cold mounting is also preferred for fragile or brittle materials, since hot mounting commonly involves pressures of up to approximately 250 bar, which may fracture the sample.

    After mounting, samples should be clearly labelled. Engraving the sample name onto the curved side surface of the mount is often preferable to using ink markings, which may dissolve during cleaning. Such engraved labels also remain visible in the SEM chamber infrared camera, even when the bottom surface is coated with conductive silver paint.

    The mounted sample should provide both good mechanical stability and reliable electrical conductivity in all directions. Attachment using silver, carbon, or nickel conductive cement is generally sufficient. In contrast, mounting the sample on conductive carbon tape is usually not recommended for EBSD analysis.

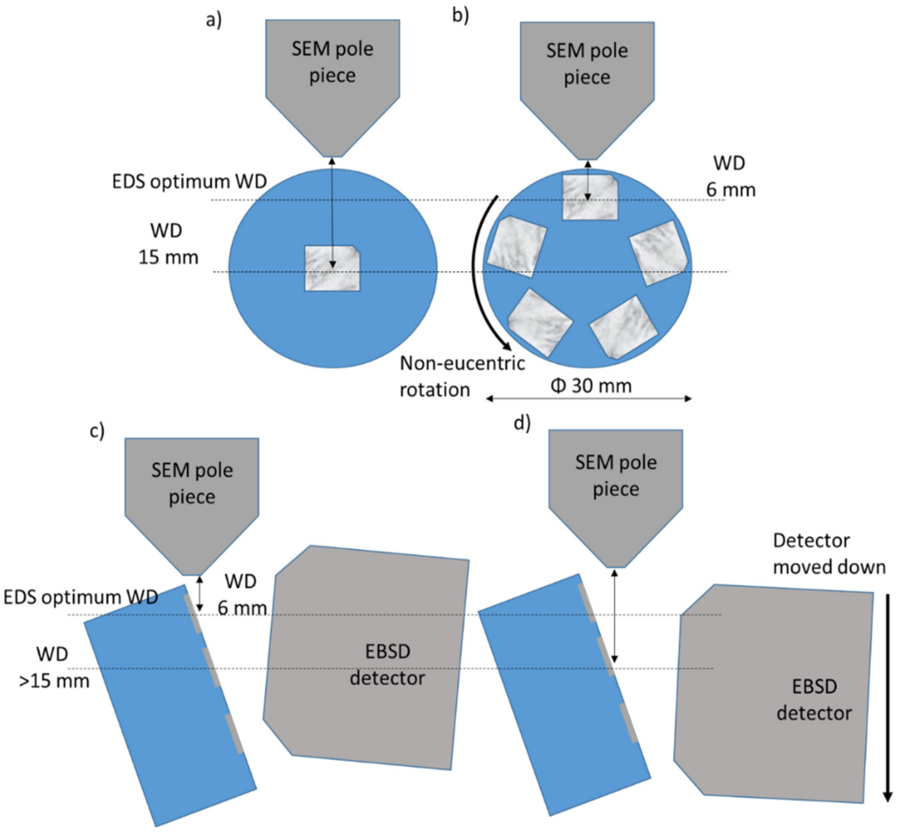


Figure 5. Different pole piece / sample / EBSD detector geometries.

Although conductive paints and cements require drying time before insertion into the SEM chamber, many dedicated holders provide both good mechanical stability and reliable electrical conductivity. Several examples of dedicated EBSD holders are presented in Fig. 6.

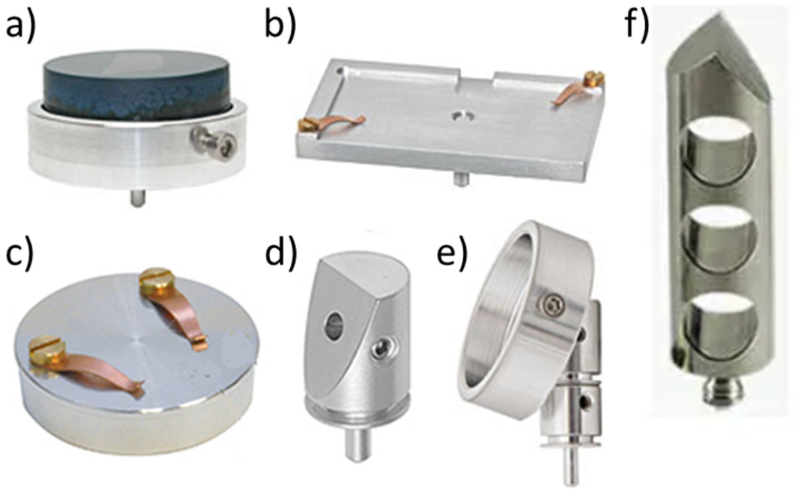


Figure 6. Examples of EBSD holders: a) Metallographic mount holder; b) Geological slide holder; c) Clamp holder; d) 70° pre tilted holder; e) Combination of a) and c); f) 3D EBSD holder.

Pre-tilted EBSD holders offer certain advantages, but they also have important limitations. In many SEM systems, such holders are mounted on standard specimen pins, which may introduce additional rotational misalignment. As a consequence, the geometric distortion of the EBSD map changes from an isosceles trapezoid to a non-isosceles trapezoid. In addition, the assumed pattern centre geometry is no longer strictly valid, leading to systematic shifts in the calculated pattern centre position.

8. MECHANICAL GRINDING AND POLISHING

Before beginning the grinding and polishing procedure, the author chamfers the edges of the mount using coarse sandpaper. This improves handling during manual grinding and helps extend the lifetime of polishing cloths. Several practical rules can also simplify sample preparation and improve reproducibility:

- Replace grinding paper once it becomes dull.
- Thoroughly clean samples before proceeding to a finer abrasive size.
- Label polishing cloths according to abrasive size and material type.
- Clean polishing cloths after use.
- Do not allow colloidal silica to dry on the sample, polishing cloth, or polishing system, as dried residues can accumulate and later break off during polishing.

For many materials, cleaning the sample with dish soap after polishing effectively reduces the number of residual colloidal silica particles on the surface. More commonly, however, samples are cleaned in an ultrasonic bath for approximately 5 minutes. In most cases, the goal is to remove all colloidal silica particles from the sample surface. Interestingly, a few isolated particles remaining on the surface may actually facilitate SEM focussing and beam alignment.

It is also important to note that both colloidal silica and Al_2O_3 suspensions are available with different pH values, which may influence the polishing behaviour and affect the resulting microstructure differently. In addition, various additives and modifiers are commonly used with colloidal silica suspensions to improve the polishing performance for specific alloys. Examples of such modifiers are presented in Table 3.

9. ELECTROPOLISHING

When properly applied, electropolishing can provide excellent EBSD surface quality. Since the process requires good electrical conductivity, its application is generally limited to metallic materials. Even among metals, however, not all materials respond well to electropolishing, and optimisation of the electrolyte composition and polishing conditions is often necessary.

Table 2. A "start with" grinding/polishing procedure (disc rotation 150 rpm and spindle co-rotation 150 rpm).

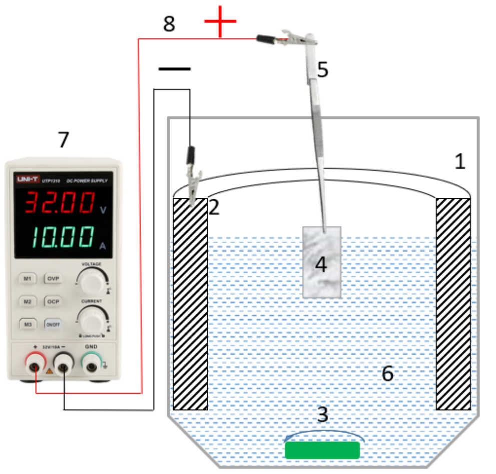
Particle size (µm)	**FEPA P**	**US**	**Particle material**	**Cloth**	**Lubricant**	**Time**	**Force [N]**[a]
125	#120	#120	SiC	-	water	4 min.	25
82	#180	#180	SiC	-	water	4 min.	25
46	#320	#280	SiC	-	water	4 min.	25
30	#500	#320	SiC	-	water	4 min.	25
22	#800	#400	SiC	-	water	4 min.	25
15	#1200	#600	SiC	-	water	4 min.	25
10	#2000	#800 - #1000	SiC	-	water	4 min.	25
3	-	-	Diamond/Al_2O_3 [b]	MD-Mol	water / ethanol [d]	4 min.	25
1	-	-	Diamond/ Al_2O_3 [b]	MD-Nap	water / ethanol [d]	4 min.	25
0.05	-	-	Colloidal silica	MD-Chem	water / ethanol [d]	10-30 min.	25
0.05 [c]	-	-	Colloidal silica [c]	MD-Chem [c]	water / ethanol [d]	6-48 h [c]	~5 [c]

a) Force applied to 30 mm diameter mount.
b) For titanium alloys, Al_2O_3 typically gives better results than diamond.
c) Vibration polishing (not always needed).
d) Ethanol or oil-based diamond suspensions for water reacting materials.

Table 3. Colloidal silica modifiers (from [4]).

Alloys	**Modifier**	**Mixing ratio Modifier: Colloidal silica**
Ti	30 % hydrogen peroxide	1:5
Ti	10 g ammonium persulfate per 100 ml distilled water	1:5
Zr/Hf	30 % hydrogen peroxide	1-2:8
Zr/Hf	chromium trioxide	5:95
Cu	add a solution of ammonium hydroxide or copper ammonium persulfate solution	-
Pb	ammonium acetate	5:1000
Refractory alloys	chromium trioxide	5:95

A typical requirement before electropolishing is a flat, plane-parallel sample surface prepared by grinding with SiC papers down to approximately 10 µm abrasive size. A simple electropolishing setup is presented in Fig. 7.



1. Beaker/plastic bottle 2. Stainless steel pipe
3. Magnetic stirrer 4. Sample 5. Tweezers
6. Electrolyte 7. DC power supply 8. Cables

Figure 7. Simple electropolishing setup.

It is also worth noting that electropolishing for EBSD can be performed using twin-jet electropolishers commonly employed for TEM sample preparation. Although the polished area produced by such systems is usually limited to approximately 3 mm in diameter, this is sufficient for many EBSD applications, as typical EBSD maps are often considerably smaller than that.

The situation changes when the second-phase particles or precipitates are much smaller. In such cases, some particles may still detach from the surface during electropolishing, but the remaining ones usually do not protrude significantly above the matrix, allowing reliable EBSD analysis to be performed. In other materials, the electrolyte may preferentially dissolve the matrix surrounding the particles. As a result, the particles become unsupported and eventually detach, leaving small craters behind. For some alloys, electropolishing can be extremely efficient. For example, 304 and 316L austenitic stainless steels can often be prepared for EBSD in less than 5 minutes per sample, even when the specimen has a complex geometry, such as samples designed for in-situ tensile testing. Electropolishing frequently produces visible surface waviness aligned parallel to the rolling or drawing direction. This effect is commonly associated with segregation of alloying elements within the material. Examples of commonly used electrolytes together with suitable materials and polishing parameters are listed in Table 4. When searching for an appropriate electrolyte for a particular material, it is often useful to consult TEM sample preparation literature, since many electrolytes developed for TEM electropolishing are also suitable for EBSD preparation.

Table 4. A list of most common electrolytes.

Materials	Electrolyte	Voltage [V]	Temperature
Steels, Stainless steels, Ni alloys, many HEAs	10 % perchloric acid (60 %) in glacial acetic acid	25	RT
Al-Cu alloys	30 % nitric acid (65 %) in methanol	10	-30 °C
Al alloys general	10 % perchloric acid in ethanol	25	10 °C
Cu alloys	500 ml distilled water 250 ml phosphoric acid (85 %) 250 ml ethanol 50 ml propanol 5 g urea	10	10 °C
Ti alloys	600 ml methanol 360 ml butoxyethanol 60 ml perchloric acid (60 %)	35 - 45	-20 °C

10. CHEMICAL ETCHING

Chemical etching can be used as either an intermediate or a final preparation step for particularly challenging materials. In most cases, chemical etchants preferentially attack grain boundaries or phase interfaces, thereby revealing the microstructure. Colour etching and tint etching methods are generally not suitable for EBSD preparation, as they often leave reaction products or residues on the sample surface that degrade EBSD pattern quality. Chemical etching is especially effective for coarse-grained, single-phase materials. In such cases, EBSD analyses are typically performed with step sizes in the micrometre range, meaning that the surface relief introduced by grain boundary etching remains sufficiently small and does not significantly interfere with data acquisition. A common requirement before chemical etching is a final mechanical polishing step using a 1 μm diamond suspension.

11. BROAD BEAM ION POLISHING

Broad beam ion polishing is a preparation technique in which, most commonly, Ar ions with energies ranging from a few hundred eV up to approximately 16 keV sputter material from the sample surface, thereby removing the mechanically deformed surface layer. The two most common broad beam ion polishing geometries are flat milling and cross-section milling.

In flat milling, the ion beam is directed toward the rotating sample surface and gradually removes material from the top surface of the specimen (Fig. 8a). In cross-section polishing, the specimen is attached to a holder and positioned behind a shielding mask (Fig. 8b). The sample is intentionally mounted so that a small portion protrudes above the mask edge. The ion beam is then aligned in such a way that it partially mills both the mask and the exposed sample region. In practice, this produces a Gaussian-shaped milled groove on the sample surface or, after sufficiently long polishing times, may completely cut through the specimen.

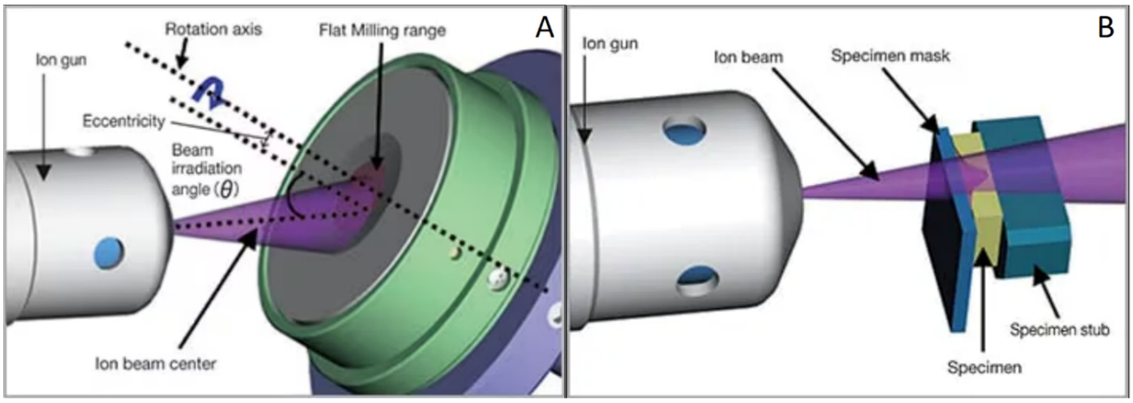


Figure 8. Broad beam ion polishing: a) Flat milling; b) Cross-section milling (from [16]).

Although beam wobbling in the range of approximately 10° - 45° is commonly applied to improve polishing uniformity, curtain artefacts may still appear in the cross-section. Nevertheless, the resulting surface is generally suitable for EBSD analysis.

Flat milling is most often used as a final preparation step following conventional mechanical polishing, typically after colloidal silica finishing. In addition to removing residual deformation, broad beam ion milling effectively removes thin surface oxides and contamination layers. Cross-section polishing, in contrast, is usually performed after grinding the sample down to approximately 10 µm abrasive size.

The most important processing parameters are ion beam energy, irradiation angle, and polishing time, all of which may require optimisation depending on the material. Because the ion beam intensity follows an approximately Gaussian distribution, the polished region typically has a circular shape with a heavily milled centre. In many cases, the optimal region for EBSD mapping is therefore located slightly away from the centre of the polished area.

Broad beam ion polishing may be the only reliable preparation method for particularly fragile materials with mechanical properties comparable to chalk, as well as for multiphase materials in which some constituents tend to pull out during conventional mechanical polishing.

12. FOCUSSED BEAMS

Sample preparation for EBSD can also be performed in situ inside the SEM chamber. Currently, several focussed-beam techniques are available for this purpose, including conventional Ga^+ focussed ion beam (FIB) systems, Xe^+ plasma FIB (pFIB) systems, and laser-based preparation methods. All of these approaches can produce site-specific areas suitable for EBSD analysis.

In most cases, laser preparation requires an additional Xe^+ ion cleaning step to remove the damaged surface layer generated during laser ablation. Conventional Ga FIB systems typically provide the smallest prepared regions, Xe pFIB systems produce intermediate-sized areas, while laser-based approaches can prepare the largest EBSD-ready surfaces. All of these focussed-beam methods also enable serial sectioning and 3D microstructural reconstruction, although this topic is beyond the scope of the present overview. Figure 9 presents a 150 × 150 µm EBSD-ready area together with EBSD mapping of a Sn film prepared using Xe pFIB. An interesting application of Xe pFIB is the so-called “spin mill” technique. In this approach, the Xe plasma beam is used in a manner conceptually similar to broad-beam Ar ion polishing. The sample is rotated while being milled at a shallow incidence angle. Unlike conventional broad-beam polishing, however, the operator can precisely define the region to be polished, and the entire preparation process is carried out directly inside the SEM/pFIB chamber. The basic principles of the “spin mill” method are illustrated in Fig. 10. Typical prepared areas are smaller than approximately 0.9 mm in diameter. For a broader introduction to focussed-beam preparation methods for EBSD, the reader is referred to the presentation by Bart Winiarski entitled “Focussed beams for use in EBSD” [9].

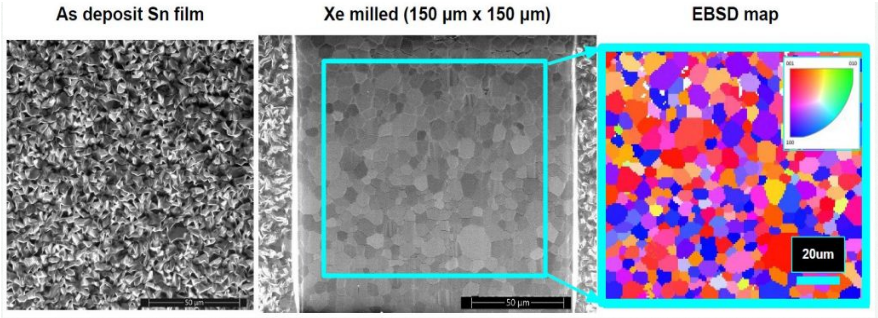


Figure 9. Example of sample surface prepared with Xe pFIB. https://www.nanofab.ualberta.ca/2021/news/multi-ion-beam-fib-sem-is-operational/.

13. PLASMA CLEANING

External plasma cleaning, as well as in-chamber plasma cleaning, generally improves EBSD measurement quality by reducing the amount of hydrocarbons present on both the sample surface

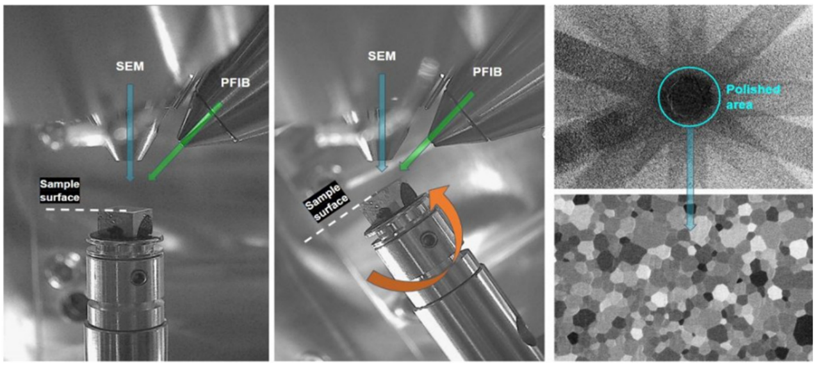


Figure 10. Principle of "spin milling" for EBSD. https://www.nanofab.ualberta.ca/2022/news/spin-mill-with-plasma-fib/.

and within the microscope chamber. Plasma cleaning is particularly beneficial for low-current EBSD measurements and for analyses performed at step sizes approaching the physical spatial resolution limits in both EBSD and TKD geometries.

During EBSD mapping, carbon contamination gradually accumulates at each scan point as a result of electron-beam-induced hydrocarbon deposition. Over the course of a long scan, this contamination layer may progressively degrade EBSD pattern quality and reduce indexing performance.

14. CONDUCTIVE COATING

Conductive coatings and low-vacuum SEM operation make EBSD mapping of non-conductive materials possible by reducing or eliminating surface charging. Operating the SEM under low-vacuum conditions, typically in the range of approximately 10 - 50 Pa, can effectively suppress charge accumulation on the sample surface.

When low-vacuum operation is not available, a conductive coating is usually deposited onto the sample surface by sputtering or carbon evaporation. The choice of coating material and thickness is critical for EBSD analysis. Since the EBSD signal originates from only the uppermost few tens of nanometres beneath the surface, excessively thick coatings may significantly attenuate the diffraction signal. The signal depth also depends on the atomic number of the coating and substrate material, with higher atomic number elements causing stronger attenuation of the EBSD signal as coating thickness increases.

Carbon is one of the most commonly used coating materials for EBSD, typically applied with a thickness of approximately 2 - 10 nm. Figure 11 illustrates a two-step coating procedure. In the first step, part of the sample is masked using a 12 mm specimen stub and coated with a relatively thick conductive layer. In the second step, the mask is removed and the entire sample is coated with a thin carbon layer of approximately 2 - 10 nm thickness.

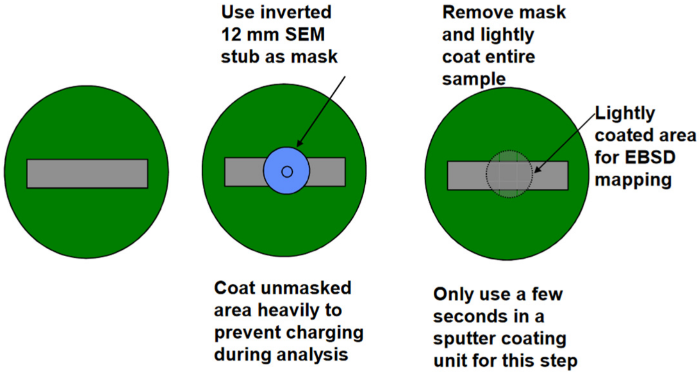


Figure 11. Two-step carbon coating process [17]

If the conductive coating becomes too thick and significantly weakens the EBSD signal, the accelerating voltage can be increased to improve electron penetration through the coating layer. In cases where charging occurs mainly inside cracks or pores, sputter coating the sample with gold before the final polishing steps may improve electrical conductivity within these features.

Several additional practical approaches can help reduce charging during EBSD analysis of nonconductive materials. It is advantageous to switch on the electron beam only after the sample has been tilted to the typical EBSD geometry of approximately 70°. At high tilt angles, the backscattered electron yield is significantly higher than at normal incidence, meaning that more electrons leave the sample surface instead of accumulating as charge.

Lower accelerating voltages and reduced beam currents are also recommended, while maintaining the highest practical mapping speed. Beam currents commonly used for metallic materials, typically in the range of 10 - 30 nA, are often unsuitable for nonconductive samples. In most cases, starting with beam currents of approximately 2 - 5 nA provides more stable operating conditions.

15. SAMPLE STORAGE

Sample storage can be just as important as the preparation procedure itself. Improper storage may lead to surface oxidation, contamination, or moisture adsorption, all of which can significantly degrade EBSD pattern quality. In many cases, storage in cabinets with controlled humidity is sufficient to preserve the prepared surface condition. Vacuum chambers commonly used for resin degassing can also serve effectively as storage containers for prepared samples.

For air-sensitive materials, temporary storage directly inside the SEM chamber may be advantageous, as it minimises exposure to oxygen and humidity before analysis.

16. REFERENCES AND FURTHER READING / WATCHING

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