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SAMPLE PREPARATION FOR SEM, MICROANALYSIS AND EBSD

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Dr. Grzegorz Cios is working in the Academic Centre for Materials and Nanotechnology at AGH University (Krakow, Poland), since his MSc thesis defence in 2014 in the group of Piotr Bała. During his PhD, he investigated nickel-based alloys with high-volume fractions of carbides for parts with improved wear resistance. He defended his PhD thesis in 2019 in Materials Science. Since early career, his scientific interest was EBSD. He learned EBSD under Tomasz Tokarski's supervision; later, together, they started cooperation with Gert Nolze and Aimo Winkelmann, which resulted in around a dozen joint papers expanding the understanding and shifting the limits of EBSD. At the EMAS 2019 Workshop in Trondheim, he was awarded the best early career scientist presentation about resolving martensite tetragonality using EBSD. In 2019 he joined the EMAS Board as a student representative. From 2023, he is a co-opted EMAS Board member. He co-authored over 90 peer-reviewed papers. His most important achievements are related resolving tetragonality of high-carbon steels martensite using EBSD, quasicrystals orientation analysis using EBSD and an improved methodology for 0° tilt EBSD.

1. INTRODUCTION

What are the requirements for the sample surface to be a reliable source of information, both in terms of chemical composition and crystallographic information? In the following text, the methodology for preparing samples for electron backscatter diffraction (EBSD) measurements will be outlined. The article's title states that sample preparation methods for SEM and microanalysis will be presented. So, the question is why the text is only about sample preparation methodology using the EBSD technique. The point is that the EBSD technique is one of the most demanding techniques in scanning electron microscope when it comes to sample preparation. Thus, in most cases, a sample prepared for EBSD will also be well prepared for SEM observation and microanalysis.

2. WHAT MAKES A GOOD EBSD SAMPLE?

First, define what makes a good EBSD sample. The answer is evolving. Is there a general answer? The answer would be that the sample is good when EBSD measurement answers the question about the investigated material (and does not lie about it). In other words, "it depends". From the author's experience, one can live with a sample with some scratches on the surface when the question is, what is the average grain size or texture? However, the author would not trust any geometrically necessary dislocation (GND) estimations from maps collected on severely scratched areas. The lowered hit rate and scratches are not that bad. Typically, the sample requires more time and more careful polishing. EBSD users should be more afraid of scratch-free samples with 100 % hit rates that are not properly prepared during cutting and grinding. An example of an EBSD map from the sample with no scratches but artefacts are present in the microstructure shown in Fig. 1. With some compromises possible, a good EBSD sample should possess:

- No additional deformation (like in Fig. 1, typically caused by too quick advancing during the grinding procedure).
- No scratches (sometimes is very hard to avoid, and for some analyses, one can live with some scratches).
- No pull-outs (no loss of structural elements, no cavities after dissolution of water-sensitive phases, no holes after brittle inclusions that break out of the matrix).
- No introduction of foreign elements (hard abrasive particles can be embedded into the surface, especially for soft materials).
- No smearing (smearing is the plastic deformation of large sample areas, typically related to lack of lubrication on polishing cloth).
- No relief (relief is the different height of different microstructure elements, often occurs even between different grains of the same material after long polishing with colloidal silica; it is hard to be fully avoided).
- No edge rounding (edge rounding may not be a problem at all when the edge area is not in the region of interest; typically occurs due to much faster resin material wear in comparison to sample wear or due to a gap between sample and resin).

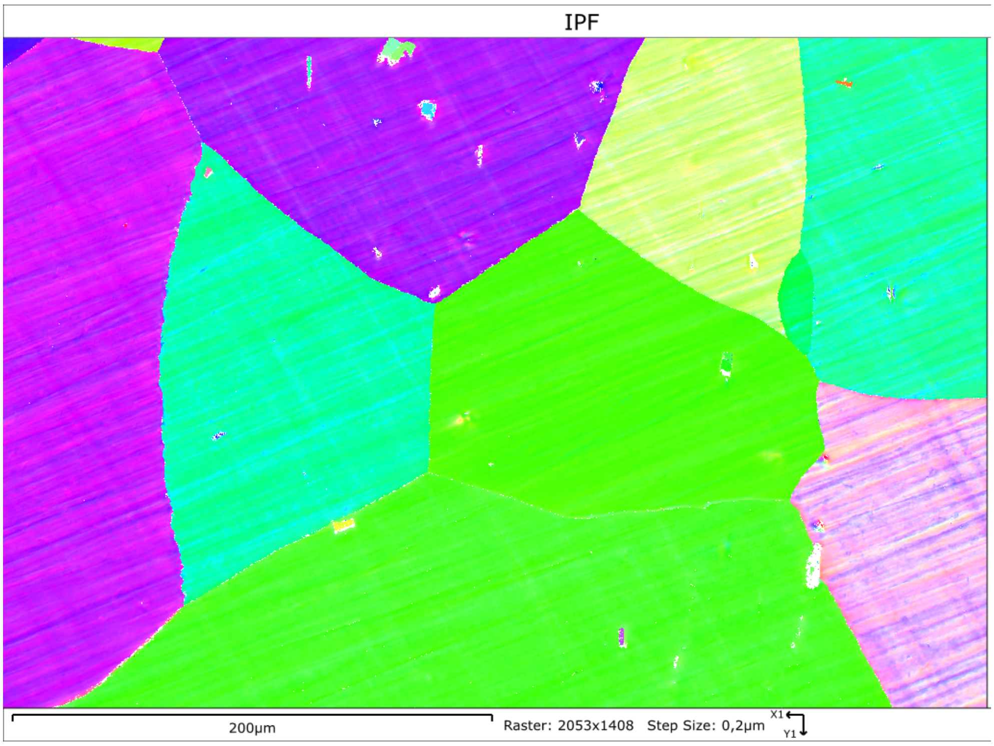


Figure 1. Improperly prepared ferritic stainless steel sample IPF map. Hit rate > 99 %.

- No contamination/staining (staining mainly happens after cleaning the sample.
- after polishing or etching, improper drying of the sample, or leaving it in a humid environment).
- No charging (charging, in general, is caused by insulating properties of analysed material but can be amplified by porosity or sharp edges).

3. SAMPLING

Taking a sample for the EBSD technique can be crucial for subsequent interpretation, particularly concerning the material's texture. Often, the samples studied will be from metallurgical processes such as rolling or drawing, after which a characteristic crystallographic texture is expected. It is good practice to take photographs during sampling. Taking rectangular sample cross-sections will make it easier to orient the texture and avoid errors in interpretation compared to square or circular samples. Sample preparation becomes even more complicated when taking a mineralogical or ice sample. Then, in addition to photographic documentation, it is useful to have a map and to be able to orient the sample on the map, considering the geographical directions. An example of an extreme case of a sample reference frame is presented in Fig. 2.

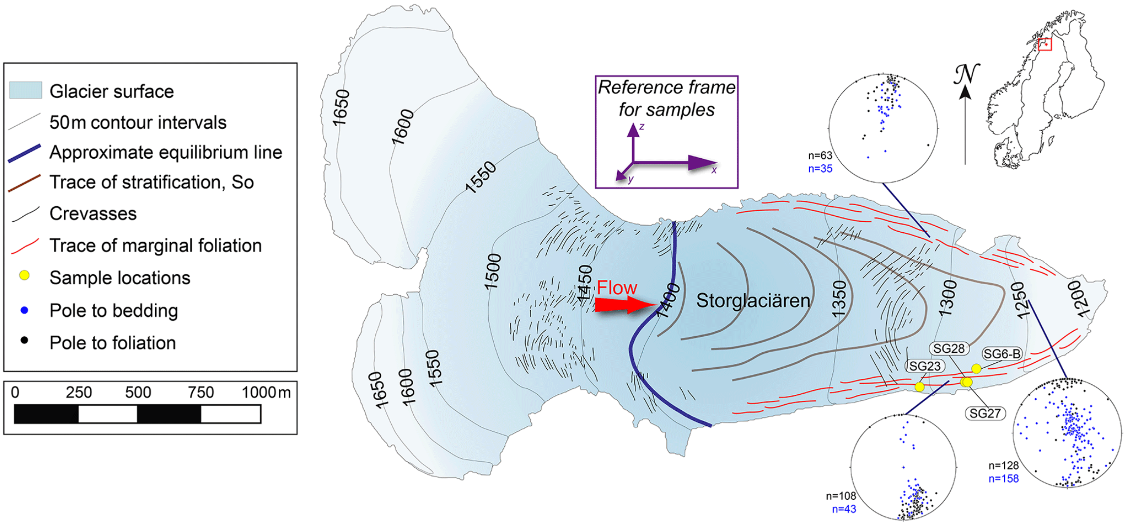


Figure 2. An example of documented sampling points with a reference frame overlaid on map of Storglaciären (https://tc.copernicus.org/articles/15/303/2021).

4. *CUTTING*

Typically, collected samples require further processing before being placed in the SEM and subjected to EBSD. The first step is cutting. Metallographers cut the samples using a method that will change the sample's microstructure the least. Plastic deformation and the heat-affected zone are the most common artefacts introduced during cutting. Wire sawing and low-speed diamond disk cutting are the most suitable techniques for cutting samples (Fig. 3). Nevertheless, they are low-performance, so water-cooled SiC disk cutting or wire EDM cutting seems an appropriate compromise. Wire EDM is especially effective for hard metals or metallic matrix composites. Band saws are known for their high efficiency in cutting low to medium-hard metals. However, they leave the thickest deformation zone under the surface.

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

5. DEMAGNETISATION

Ferromagnetic materials can have remanence, meaning they will behave more or less like permanent magnets. As is well known, magnetic fields interact with electrons: electrons move in circles at a constant speed if projected into a magnetic field at right angles to the field, and electrons move in straight lines at a constant speed if projected into a magnetic field along the direction of the field. A popular online example fulfils the situation that may happen inside the SEM chamber: "An electron is accelerated from rest through a potential difference of 5 kV and then enters a magnetic field of strength 0.02 T acting at right angles to its path. Calculate the radius of the resulting electron orbit". The resulting electron orbit is 1.2 cm (search this question online for detailed answers). In SEM, we can have electrons accelerated through a potential of 30 kV, and some of the neodymium magnets can create magnetic fields of 1.4 T. The magnetic field will interact with the primary beam and the backscattered electrons traveling to the EBSD detector. A permanent magnet close to the area of interest for EBSD analysis will make the quantitative analysis very hard due to the heavy distortions of the patterns. Examples of heavily and slightly magnetic field distorted EBSPs are shown in Fig. 4.

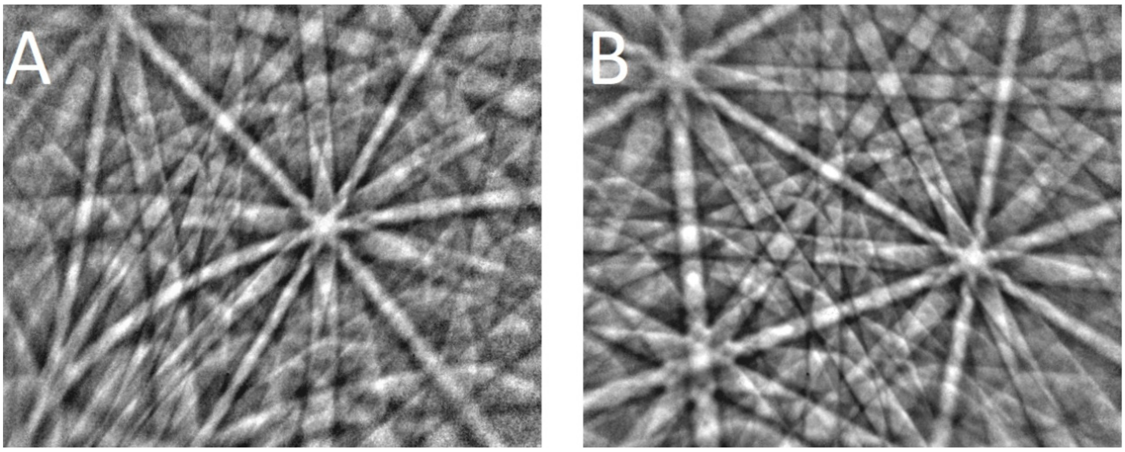


Figure 4. a) Ni patterns collected close, and b) far from permanent neodymium magnet (collecting pattern from magnet was not possible).

Different demagnetising methods may work well for different materials. For example, for magnets and materials with low Curie temperature, heating above it may be the best method (assuming heating will not affect the microstructure); for metals and alloys, permanent magnet based or induction-based demagnetizing tools might work better.

6. *PLATING*

Suppose the region of interest is the sample surface layer cross-section, like oxide scale or carburised/decarburised subsurface areas. In that case, an additional electrodeposited nickel coating may improve the sample edge quality. In the case of the oxide scale, electrodeposited nickel will support the oxides and prevent them from falling from the sample during preparation and the edge rounding. Plating typically precedes gold sputter coating to make the plated surface conductive. Later the sample is placed in a Ni-bath solution (Table 1) with a Ni plate as an anode. Ni layer of about 60 μm thickness is deposited after 6 hours at a current density of about 25 mA/cm^2.

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 their in-chamber geometry. Some of the most important things are:

i) The use of load lock. Typically, when load lock is used, sample dimensions are limited.

ii) The sample stage geometry. Here, the axes stacking sequence is important. Stages with a tilt axis at the bottom typically allow higher samples than stages with a Z-axis at the bottom.

iii) Analytical working distance. Currently, on the market, two vendors offer in-chamber tiltable EBSD detectors, and two others offer EBSD detectors with fixed geometry. Most fixed geometry detectors are positioned to provide a good signal at EDS optimum working distance, typically 4 to 15 mm. One can easily imagine operating with a sample mounted in a 30 mm diameter puck at a working distance of 4 mm can risk hitting the SEM pole piece (see Fig. 5a). In Fig. 5, a few typical geometries are considered. In Fig. 5a, the sample is mounted in the puck's centre, making the sample's movement to a shorter working distance, which may be optimal for EBSD, impossible. However, there are better positions than this for simultaneous EDS/EBSD collection (both detectors optimised for WD of 6 mm). In Fig. 4b, samples are distributed close to the edge of the puck; this geometry allows EDS/EBSD collection at WD of 6 mm. Measurements of the other four mounted samples are possible by non-eucentric rotation. Figure 5c shows the side view of the sample and detector optimised for 6 mm WD, and Fig. 5d shows how detector movement (down in this case) can improve geometry for comfortable sample movements, limiting the risk of collision with pole piece sacrificing, however the simultaneous EDS/EBSD acquisition. Long

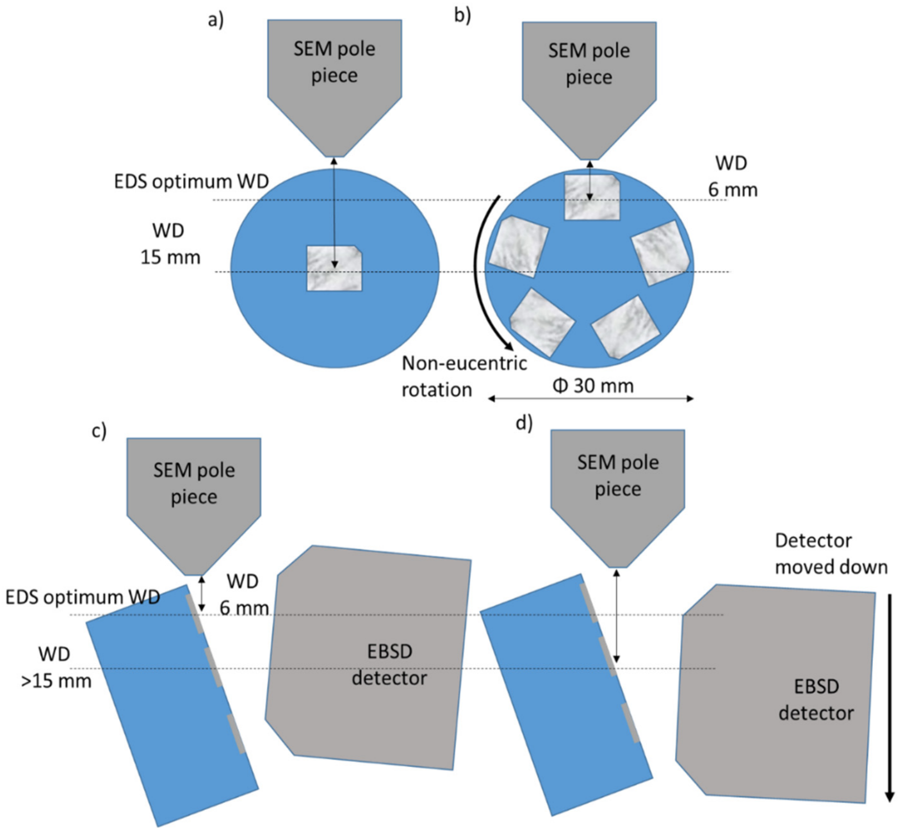


Figure 5. Different pole piece / sample / EBSD detector geometries.

working distances will also help acquire single scan large area maps thanks to the larger field of view and depth of focus. Another important problem with mounting is the effect of temperature.

iv) The hot mounting sample is typically heated up to 180 - 200 °C for at least 4 minutes. This "heat treatment" can lead to the complete recrystallisation of zinc alloys or cause carbide precipitation in quenched steels. Depending on the composition, cold mounting resins can also heat up to 110 °C for a short period. Typically, the longer the curation time, the smaller the temperature peak. Some manufacturers provide moderators for resins that lower the curation peak temperature and longer the curation time. While hot mounting delivers two parallel planes of the mount, cold mounting typically requires extra grinding or lathe milling to obtain two parallel planes. Cold mounting will also be a first choice for fragile and brittle materials because during the hot mounting pressure of 250 bar is applied and may cause a sample fracture. After mounting, it is worth labelling the samples. Engraving the curved and circular base with the sample name might be the right choice when any ink may be dissolved during sample cleaning. The curved surface engraved label is visible in the chamber IR camera and will remain visible even when the bottom surface is covered with silver paint. Fixing the sample should provide good mechanical stability and sufficient electrical conductivity in all directions. Gluing the sample with silver, carbon, or nickel cement should be sufficient. Fixing the sample on carbon tape is not recommended.

While the liquid paint or cement must dry for some time, many holders provide good mechanical stability and electrical conductivity. A few examples of dedicated EBSD holders are shown in Fig. 6. There are pros and cons to using the pre-tilted EBSD holders; nevertheless, in most of the SEMs, these are mounted on standard pins, which can cause additional misalignment in rotation, and as a result, the trapezoidal distortion of the EBSD map is changed from isosceles trapezoid to non-isosceles trapezoid. Moreover, the pattern centre shifts are no longer correct.

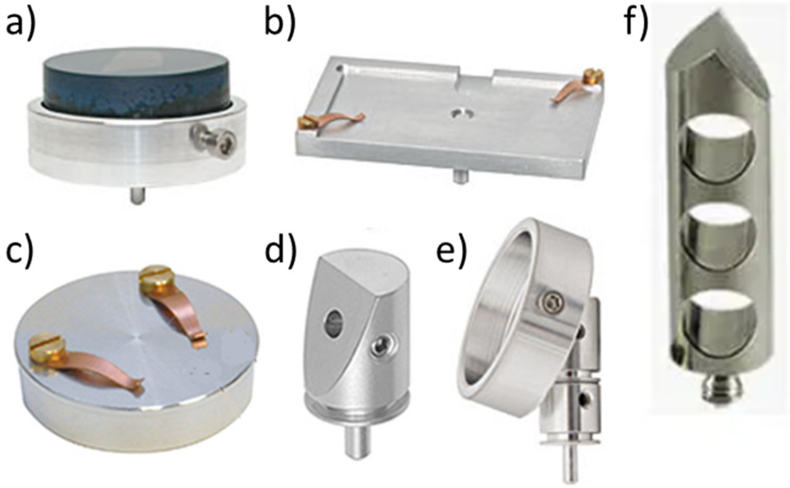


Figure 6. Examples of EBSD holders: a) Metallographic mount holder; b) Geological slide holder; c) Clamp holder; d) 70° pre tilted holder; e) Combination of a) and c); f) 3D EBSD holder.

8. *MECHANICAL GRINDING AND POLISHING*

The general idea behind grinding and polishing is to go from coarser to finer particles. The subsurface deformation and scratches from the previous step are removed with every further step. A "start with" grinding and polishing procedure is presented in Table 2. (Please note the polishing cloths' names are according to the Struers catalogue. However, any consumables supplier delivers equivalent cloths under different names). Before starting the polishing process, the author chamfered all edges of the mount using coarse sandpaper. This makes manual grinding more comfortable and prolongs the life of the polishing cloths. Applying a few rules will make sample preparation easier. Replace the sandpaper when it is dull. Clean the samples before moving to a smaller grain size. Keep cloths labelled with abrasive grain size and materials it is used for. Clean your clothes after polishing. Do not let colloidal silica dry on your sample, cloth, or polishing device (it will build up and crumble during polishing). For many materials, cleaning with dish soap after polishing will reduce the number of colloidal silica particles. More popular, however, is ultrasonic bath cleaning for around 5 minutes. Typically, we want to remove all the colloidal silica particles from the sample surface. However, single-separated particles make focussing and beam alignment much easier. It is important to mention that both colloidal silica and Al_2O_3 suspensions may be delivered with different pH values and affect the microstructure differently. There are also common modifiers added to colloidal silica suspension for improvement of polishing of some alloys are presented in Table 3.

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 $^{c)}$

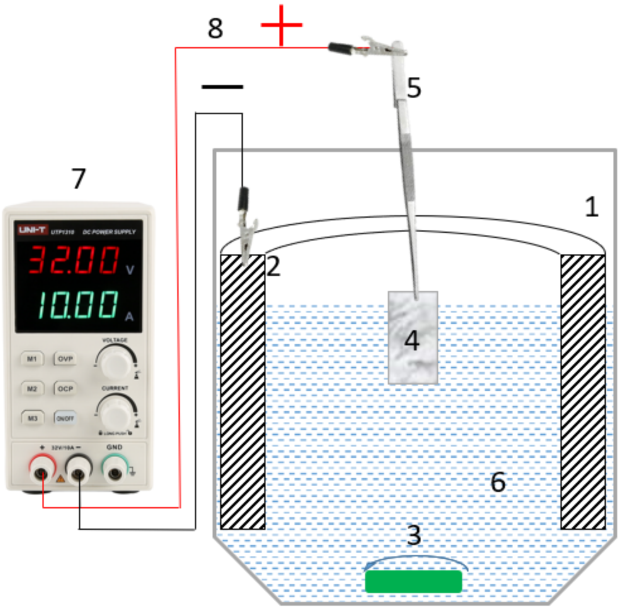
a) Force applied to 30 mm diameter mount.
b) For titanium alloys, Al_2O_3 typically gives better results than diamond.
c) Vibration polishing (not always needed).
d) Ethanol or oil-based diamond suspensions for water reacting materials.

9. ELECTROPOLISHING

Electropolishing, when applied properly, may provide very good results. The sample should conduct the electricity well, so electropolishing is limited to metallic materials. Nevertheless, for many metallic materials electropolishing will not provide good results. A typical requirement for electropolishing is a plane parallel sample after grinding with SiC papers down to 10 µm particle size. A simple electropolishing setup is shown in Fig. 7. It is worth mentioning electropolishing for EBSD can be done using twin-jet machines used commonly for TEM sample preparation. The prepared area will have a 3 mm diameter, but typical EBSD maps are smaller

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



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.

than that. The situation is even more interesting if we let the sample perforate. Then, on one 3 mm diameter disc, we can perform EBSD, TKD, and TEM. The reason is that many metallic materials are multiphase, and typically, only the metallic matrix will react properly to electropolishing. In general, cast, slowly cooled steels or aluminium alloys with large primary Si or carbide phases are not prone to electropolishing for EBSD. The problem is mainly due to surface topography. These insoluble during electropolishing phases will stick out from the polished surface, causing much shadowing. In extreme examples of eutectic Al-Si alloys, silicon may form a continuous scaffold that covers the whole field of view after electropolishing.

The situation changes when the second phase precipitate/particle size is much smaller. Some particles in such a situation will fall from the sample during electropolishing, but the remaining ones may not stick out too much, and proper EBSD analysis can be performed.

On the other hand, some materials may react with electrolytes in a way that the matrix around the particles will be removed faster, and particles will fall from the sample, leaving craters behind. For example, one can prepare 304/316L austenitic stainless steels for EBSD by electropolishing in less than 5 minutes per sample, and the sample may have a complex shape like, for example sample for in-situ tensile tests. Electrolytic polishing often leads to visible waviness parallel to the rolling or drawing direction; this is a sign of the segregation of the alloying elements. A list of a few common electrolytes with materials and parameters is presented in Table 4. When looking for a electrolyte for a particular material, it is worth looking at the TEM analysis papers. Most electrolytes for TEM sample preparation will work for EBSD sample preparation proper.

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 applied as a final or intermediate preparation step for challenging materials. Chemical etching will typically etch the grain boundaries or phase interfaces. Colour and tint etching methods will not work well for EBSD as these may leave residue on the sample surface. Chemical etching is very effective for large-grain single-phase materials. For such samples, the typical EBSD analysis step size is in the range of micrometres. Therefore, the etched grain boundary topography will be fine. The typical requirement for chemical etching is a 1 µm diamond finish.

11. BROAD BEAM ION POLISHING

Broad beam ion polishing is a technique where, in most cases, Ar-ions with energies from a few hundred eV up to 16 keV are sputtering the sample surface material, removing the deformed material layer. The two most common geometries for broad beam ion polishing are flat milling and cross-section milling. During the flat milling beam is directed towards the rotating sample surface and removes the material from the sample's surface (Fig. 8a). In cross-section polishing, the specimen is glued to the specimen stub and placed behind the mask (Fig. 8b). Properly set up sample is sticking out over the mask. The beam is aligned so that it partially removes the mask and the material above it. In practice, it leads to a Gaussian-shaped groove in the sample surface or, after more time, to cut through the sample. Despite applied wobbling by 10°- 45°, the cut can contain curtains, but generally, it is EBSD-ready. Flat milling is typically applied as the last step of sample polishing after initial colloidal silica polishing. Broad beam ion milling will remove the sample oxidation and contamination. Cross-section polishing, on the other hand, is typically applied after grinding the sample down to 10 µm particles. Parameters set up in the machine are beam energy, beam irradiation angle, and polishing time, and each of these may need adjustment for different sample types. An affected round area forms due to the Gaussian distribution of the beam intensity. While for many cases, the centre of the round area is too heavily polished, moving a bit outside of the area, one can find the proper place for EBSD mapping. Broad beam ion polishing may be the only reliable method for some samples whose mechanical properties are similar to chalk or for samples with phases that pull-out during mechanical polishing.

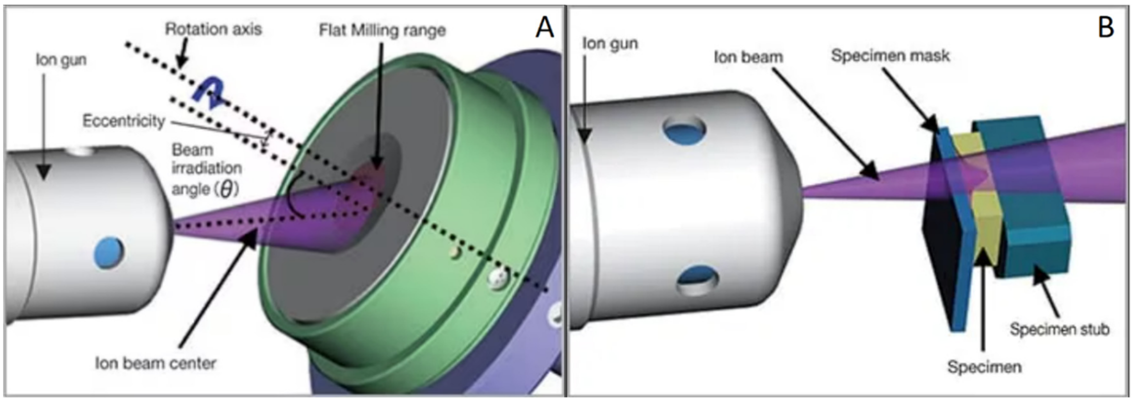


Figure 8. Broad beam ion polishing: a) Flat milling; b) Cross-section milling (from [16]).

12. FOCUSSED BEAMS

Sample preparation for EBSD is also possible in situ in the chamber of an SEM. Currently, there are more than three focussed beams available for sample preparation. Standard Ga^+-ions FIB, Xe^+-ions pFIB, and lasers. All three beams will provide site-specific EBSD ready sample area.

Typically, after laser preparation, further Xe^+-ions cleaning is required. Ga FIB will deliver the smallest areas, and Xe pFIB intermediate and laser will provide the largest EBSD-ready areas. Of course, all mentioned focussed beams allow 3D data collection, but this is out of the scope of the present overview. Figure 9 shows 150 x 150 µm EBSD ready area and mapping of Sn film prepared with Xe pFIB. An interesting application of Xe pFIB is the “spin mill”. In this technique, Xe pFIB is used very similarly to the Ar broad beam. The rotating sample is milled at a low incidence angle. However, the operator can choose the precise area to be polished; everything happens in the SEM/pFIB chamber. The principles of “spin mill’ are shown in Fig. 10. Typical areas prepared using “spin mill” are smaller than 0.9 mm in diameter. For better understanding of focussed beams for EBSD watch video [9] from by Bart Winiarski entitled “Focused beams for use in EBSD”.

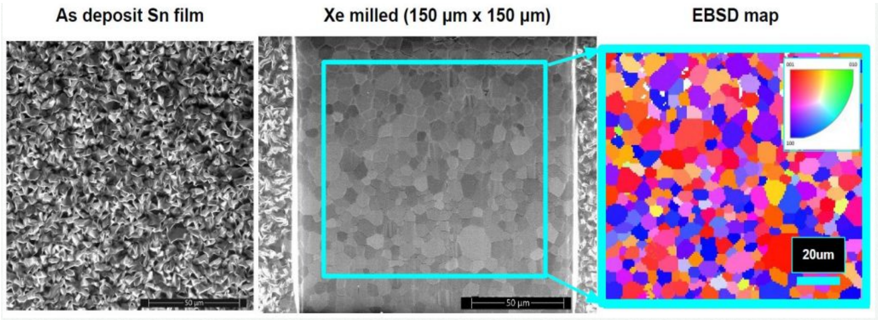


Figure 9. Example of sample surface prepared with Xe pFIB.
https://www.nanofab.ualberta.ca/2021/news/multi-ion-beam-fib-sem-is-operational/.

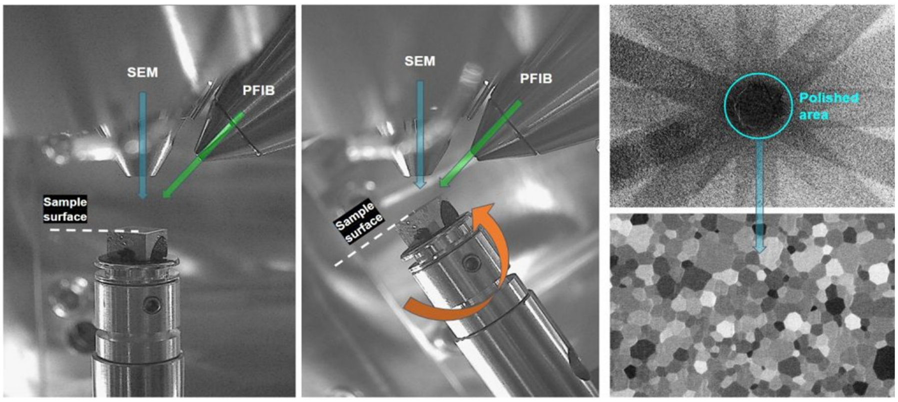


Figure 10. Principle of “spin milling” for EBSD.
https://www.nanofab.ualberta.ca/2022/news/spin-mill-with-plasma-fib/.

13. PLASMA CLEANING

External plasma cleaning or in-chamber plasma cleaning will generally improve the results. Plasma cleaning reduces the amount of hydrocarbons on the sample surface and in the microscope chamber. It is especially recommended for EBSD measurements at low rates and when the mapping step size reaches the physical resolution both in EBSD and TKD geometry. During an EBSD scan, carbon contamination builds up at every scan point and can gradually degrade the signal as the mapping proceeds.

14. CONDUCTIVE COATING

Conductive coating and low vacuum operation make nonconductive sample EBSD mapping possible, thanks to removing the charge from the sample surface. A low vacuum in the 10 - 50 Pa range successfully decreases sample surface charging. When low vacuum operation is unavailable, the conductive coating is sputtered on the sample surface. Considering the coating thickness and material, it is worth remembering that the EBSD signal comes from a maximum few ten of nanometres under the surface; thus, applying too thick coating may weaken the diffraction signal too much. The depth of the signal in EBSD is also a function of the atomic number of the sample (or coating), so a higher atomic number of elements will decrease the EBSD signal quicker with the increase of the thickness. A typical coating material is carbon with a 2 - 10 nm thickness. Fig. 11 shows a two-step coating process. In the first step, part of the sample is masked with a 12 mm sample stub and coated with a thick coat, in the second step the mask is removed and a thin 2 - 10 nm carbon coating is applied.

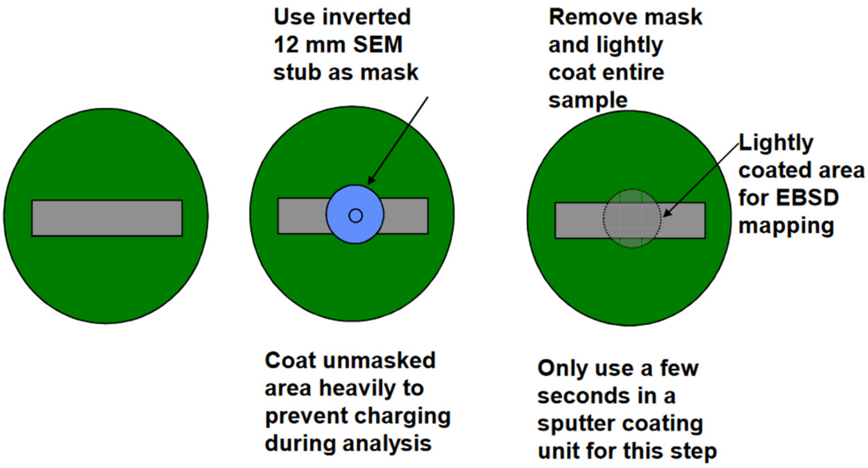


Figure 11. Two-step carbon coating process (from [17]).

When the coating is too thick, and the signal becomes too low, one can increase the accelerating voltage to penetrate the coating. When charging happens in cavities and cracks, gold sputtering before the last steps of polishing will help. A few more tips will help with nonconductive sample charging. Turn the beam on once the sample is tilted to 70°. At 70° tilt, the backscattered electron yield is higher than at 0° tilt, so more electrons will leave the sample instead of building up the charge on the surface. Use lower accelerating voltage and beam currents while keeping the mapping speed as high as possible. Beam currents typical for metallic samples 10 - 30 nA will not work; 2 - 5 nA are good to start with.

15. SAMPLE STORAGE

Sample storage can be often as important as the preparation procedure. Typically, cabinets with controlled humidity are enough. Resin degassing vacuum chambers can be successfully used as sample storage cabinets. Air sensitive samples can be stored for some time in the back of the SEM chamber.

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