Sample Preparation for EBSD

Because diffracted electrons only escape from the uppermost layer of the specimen surface (of the order of a few 10's of nanometers deep), specimen preparation for EBSD is consequently critical to achieve good results. If the specimen surface retains surface damage on a crystallographic scale, or has any surface contaminant/oxide/reaction product layers present, then EBSD pattern formation may be suppressed.

Standard preparation methods may be employed successfully with care and perhaps in conjunction with extra steps to achieve an adequate surface finish. As a general rule standard preparation methods can be progressed to the final polishing stage without any deviation from the normal route employed. Thereafter, an additional polishing stage using colloidal silica is all that is required to achieve a finish suitable for EBSD.

However, different materials respond differently to common preparation methods. Therefore the material under investigation should be considered on an individual basis and prepared appropriately. The manufacturers of preparation equipment should be consulted for the applicability of a given approach for a given material.

The following steps are critical for good specimen preparation:

Cutting

Mounting Samples

Grinding

Polishing

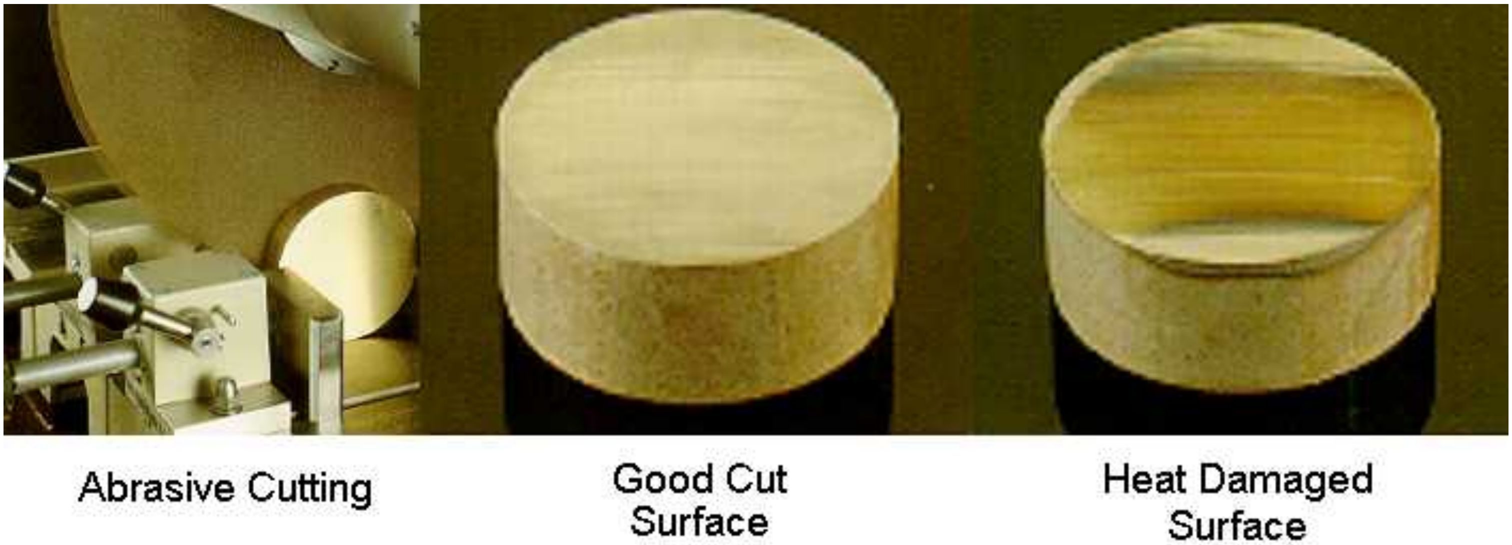
Etching

Electrolytic Polishing/etching

Alternative Preparation Methods Includes information on: Plasma Etching, Ion Milling, Attack Etching

Cutting Samples for EBSD

Avoid aggressive cutting methods that generate heat or cause deformation at the cut surface. Severe damage induced at this stage may extend so deep into the material that it is not removed by subsequent grinding and polishing. Heating caused during cutting may cause changes to the microstructure - phase transformations or precipitation/diffusions mechanisms may become active. Therefore heating must be avoided at all costs.



Abrasive cutters are common in the metallurgical industry and are suited to cutting larger sections of material. Often the sample is subjected to considerable force, and heating, although if used with care, good cuts can be performed with minimal damage. Heating is the main problem, and using excessive force with an inappropriate wheel can cause a sample to locally glow red with heat. The Next slide in the series helps to make the choice of wheel easier, but the importance of observing the manufacturer?s recommendations cannot be over-stressed. If over-heating of the sample occurs, is usually due to using a wheel that is too ?hard? for the material being cut. The wheel does not wear properly which causes the abrasive to become blunt. Another possibility is that the abrasive becomes clogged. Friction then causes excessive heating and damage to the sample. Heating often results in altered surface structures, which subsequent grinding and polishing do not remove. EBSD is very sensitive to damage caused in this manner and greater care than usual is warranted to avoid generating misleading results, or compromising pattern quality.

The selection of abrasive wheel is therefore important to avoid introducing unnecessary levels of damage when cutting materials.

A wide range of wheel types is available from a variety of manufacturers, and selecting the correct wheel can appear confusing. This frequently leads to the use of inappropriate wheel types, or simply using what happens to be fitted to the machine. It may help to be aware of the parameters that govern efficient cutting: The abrasive used in the wheel may blunt easily or break down, if cutting a hard material, or become clogged if cutting a softer or ductile material. These are overcome by selecting the abrasive, and the bonding medium, and the strength of the bond, to suit the material to be cut. Thus SiC is used for softer materials, being a sharp, but rather less durable abrasive compared to Al_2O_3. For a soft material having a sharp abrasive is most important. The bond of the wheel is formulated to provide a wear rate in use that keeps presenting fresh abrasive at the cutting edge. This gives rise to ?different grades? of wheel which have the same abrasive, but different bond strengths to control the wear rate. For harder ferrous materials, the abrasive of choice is Al_2O_3, This is because Al_2O_3 has adequate sharpness and is sufficiently durable to withstand cutting harder materials. For harder materials the durability of the abrasive is most important. Again a range of bond strengths are provided to allow selection to match the wear rate of the wheel to the hardness of the material being cut. A ?soft? bonded wheel wears fastest, stays sharp at the cutting edge, but provides the worst economy - wheels can be expensive, so extending their life is an issue. Using too hard a wheel leads to over-heating as the abrasive becomes blunt and friction causes excessive heating. Getting the balance correct provides quick, efficient cutting with minimal heating, consistent with reasonable economy. The manufacturer of wheels often provide charts or tables to assist selection, and may recommend different lubricants for different conditions or materials. Follow the advice given. Very hard materials dictate that abrasives with superior properties are used, such as Cubic Boron Nitride and Diamond. Again, some degree of wear is desirable for cutting materials with a degree of ductility, principally to avoid clogging. Metal bonded wheels, which are designed not to wear, are suitable with hard brittle materials when clogging is unlikely.

As a rough guide:

- Non Ferrous, soft and very ductile metals, hardness Hv 30 - 400 use a Silicon Carbide (SiC), Bakelite bonded abrasive wheel of appropriate grade.
- Ferrous materials Hv 80 - 850 use an Alumina (Al_2O_3), Bakelite bonded abrasive wheel of appropriate grade.
- Extremely Hard Ferrous Hv 500 - 1400 use a Cubic Boron Nitride, Bakelite bonded abrasive wheel of appropriate grade.
- Sintered Carbides, Ceramics Hv 800 - 2000 use a Diamond, Bakelite bonded wheel.
- Minerals, Ceramics, brittle materials Hv 800 - 2000 use a Diamond, metal bonded wheel.

Follow the manufacturers' guide when selecting cutting wheels and operating cutting equipment.

Precision, Low Deformation Cutting

There are many examples of cutters on the market designed for precision and low damage cutting. Such machines normally employ Cubic Boron Nitride and Diamond type cutting wheels, although other types may be available, depending on the machine.

These machines may be divided into two main groups:

- Simple low speed saws which normally use metal bonded CBN and Diamond wheels only, with gravity feed
- More sophisticated machines capable of a wide range of speeds and controllable cutting feed rates.

The latter machines can use the traditional ?abrasive? type wheels which operate at high speeds (designed to wear down to keep fresh abrasive exposed), as well as CBN and Diamond wheels, both metal and resin bonded, for low and high speed cutting.

Both types of machine can be used to obtain low deformation cuts from a wide range of materials, although the more sophisticated type offers a greater range of choice of abrasive media and cutting conditions. This generally leads to much greater cutting efficiency and shorter cutting times. If the facility to use a wide range of cutting wheels is available, then the same principles apply which were covered in the previous slide. Again the manufacturer?s instructions should be adhered to. If limited to using metal bonded CBN and Diamond type cutting wheels, clogging may be experienced when cutting ductile materials. Softer material can become adherent to the abrasive particles of the wheel and build up into a ?rim?. This can completely halt the cutting process. However, this problem is simple to overcome. CBN and Diamond wheels are often supplied with a carborundum ?dressing? stick. Cutting a harder material of this type cleans the cutting surface of the wheel. This is usually very effective and restores cutting efficiency, just by touching the stick to the cutting edge for a few seconds. However, frequent ?dressing? of the wheel may be necessary. Experimenting with the cutting load can optimize the the length of time between ?dressing?. Sometimes, cutting a softer material can be enhanced by arranging to cut a harder material or a dressing stick simultaneously, to keep the wheel clean of debris. When using low speed saws, efficient cutting can be assessed by the noise produced: the sound of cutting is quite distinct to that which occurs when the wheel is clogged, or the abrasive has become blunted. If there is little noise, there may be little cutting.

See also sample ***mounting*** and ***grinding***.

Sample Mounting for EBSD

Small samples generally require mounting such that the sample is supported in a stable medium for ***grinding*** and ***polishing***. The medium chosen can be either a cold curing resin or a hot mounting compound.

Cold mounting resins

A wide range of products are available on the market. Generally faster setting products including Acrylic resin types are less favorable, as these tend to develop low hardness and often suffer from 'shrinkage'. Shrinkage is the term given when the resin shrinks away from the sample surface during curing. This is undesirable as the gap which forms harbors contaminants, grit from grinding and polishing stages to cause cross contamination of polishing surfaces. It is difficult to obtain a good polished, scratch free surface, when gaps in the mounting material are present.

Epoxy resin types generally have the best characteristics with respect to hardness and shrinkage. However, epoxy resins tend to be slower curing and adequate time should be allowed to ensure that the material is fully cured before proceeding. Epoxies often take a considerable period of time after initial 'setting' to develop full hardness.

It is not generally possible to make cold curing resins conductive suitable for SEM examination.

To Summarize:

- Hot Mounting may be unacceptable, if the effect of temperature and pressure are expected to be inappropriate for the sample under investigation.
- Generally, the materials employed for cold setting cannot match the hardness of materials traditionally used in Hot Mounting. This may lead to compromises in the degree of edge protection and support that the mount provides for the sample. Further, the abrasion characteristics may need to be taken into account during the preparation.
- The material should be stable under vacuum. Out-gassing can be a major problem leading to high contamination

rates on the sample, and even microscope parts.

- Charging. This can be overcome by covering all exposed surfaces with conductive foils or carbon type conductive tapes or paints. Silver paint or other metal loaded paints can be used. Implications if other analytical techniques i.e. EDS or WDS: Coating using the traditional coating metals deposited by sputtering or evaporation is a possibility, although steps must be taken to protect the surface of the sample from becoming coated. This can be accomplished simply - glass microscope coverslips can be very effective for this. Glass coverslips are very thin and can be easily broken down into small, appropriately shaped pieces. Usually it is sufficient to lay the cover slip over the area to be protected. However, if the initial rush of air that occurs when pumping is started dislodges the coverslip during the coating process,use a few small spots of conductive paint to secure it. After coating, remove the coverslip to expose the uncoated surface.

Hot mounting

Hot mounting uses a thermosetting compound, cured in a mounting press which exerts both heat and high pressure. This mounting method produces hard mounts in a short space of time. However the heating (generally in the order of 120°C) and considerable pressure applied may be unsuitable for delicate/soft/low melting point samples. Techniques may be used to protect a delicate sample from the effects of pressure, such as placing the sample under a supporting structure within the molding cavity. Such a supporting structure can protect the sample from the initial pressure applied when the mounting material is in a granular form, and most likely to inflict damage. When the mounting material becomes fluid, infiltration should occur to encapsulate the sample which will then be subject to hydrostatic pressure. Hydrostatic pressure can be applied to all but the most delicate of samples without problem. In the case of very soft or thermally sensitive materials, Hot mounting is not appropriate. Conductive mounting resins are available, which are good for SEM examination, although the adhesion and hardness characteristics are not as good as those of epoxy hot set compounds. If the edges of the sample are not of interest, then non conductive mounting materials can be used. The sample mount can be made conductive after polishing by using conductive tapes or paints, applied to cover exposed areas of mounting material, and to make contact with the sample itself.

To Summarize:

- Hot Mounting is generally preferable to cold setting resins, when the sample is not affected by temperature and pressure (200ºC & 50kN). However, not all samples can tolerate this.

- The material selected should have: Good abrasion characteristics and sufficient hardness such that the edges of the sample are protected, i.e., the rate at which abrasion takes place should be even across the face of the mount and the sample.
- Stable and adherent to sample. This is important. If the mounting material has poor adhesion or high shrinkage, gaps may open up between the mounting material and the sample surface. When this happens, it is very difficult to prevent cross-contamination of one abrasive to another, causing heavy scratching in the finished section. Also any friable surface layers (oxide layers etc.) should be held adhered to the surface and not pulled off.
- Proper curing - insufficient time and temperature can lead to partially cured sample mounts. Under these conditions the properties of the mounting material are not properly developed and the material may be loose and powdery. Generally, if the material is improperly cured, the hardness and abrasion characteristics are poor and the material is adversely affected by etches and solvents. Further, the characteristics under vacuum are very poor with out-gassing a major problem. If the mounting stage is suspected to be at fault, it is best to break the sample out and start again.
- Stable in vacuum - no out-gassing or vapour to cause contamination. Particularly important for high magnification work, long map acquisition times and microscopes with high vacuum requirement (FEGSEMs)
- Non-conductive mounts must be covered with adhesive conductive tape or coated with a conductive medium (the sample area can be masked if sputter coating, or using an evaporator. Aluminum foil or glass cover slips are useful for this purpose). Note: many adhesive metal tapes have non-conductive adhesive, so the use of carbon/silver paint may be required at seams. Although very thin films of carbon can be tolerated on the sample, ideally the sample surface should be bare.

Grinding Samples for EBSD

Grinding can be achieved in a variety of ways, using a variety of abrasives. Fixed abrasive surfaces are available using Diamond or Cubic Boron Nitride (CBN) abrasives. The method used to bind the abrasives to the wheel affects the grinding characteristics, the harder or more rigid the bonding medium, the more aggressive the grinding action of the surface. Therefore metal bonded fixed abrasive wheels are the most aggressive grinding surfaces, whereas resin bonded wheels are less aggressive.

Silicon Carbide (SiC) paper is the traditional method used for grinding and is adequate when used properly. SiC paper blunts quickly and therefore should be discarded after a short period of grinding in order to maintain efficient 'stock' removal. Grinding on a surface that has blunt abrasives causes a great deal of surface damage by smearing, 'burnishing' and local heating.

Thus whatever approach adopted, ensure that sharp abrasives are used and follow the manufacturers' instructions with regard to grinding speeds, direction, force, times and lubricants used. Damage injected during grinding may be invisible in the polished surface, but serve to distort the EBSD result or even completely suppress ***pattern formation***.

After every grinding stage, it is advisable to inspect the ground surface using a light microscope in order to ensure that all damage from the previous stage, whether that was a cutting or grinding stage, is completely removed. Advance in this manner to the finest abrasive size required, ready for polishing. Care at this stage will greatly reduce the amount of polishing required to achieve a good surface.

Planar Grinding

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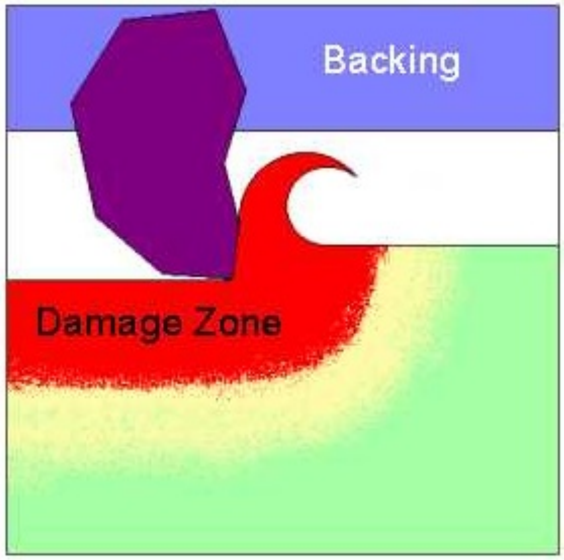


Planar grinding should be progressed until all cutting damage has been removed and the surface is flat.

The choice of media is varied.

The type of abrasive and the method by which it is bonded to the supporting backing is critical.

Rigidly fixed abrasives are more aggressive than those in a shock-absorbing backing.

In general, maintaining sharp abrasives promotes good grinding characteristics with minimal damage.

Fine Grinding

Grinding (& polishing) occurs when the abrasive acts as a cutting 'tool'

Fine and coarser scratches together indicate insufficient fine grinding

Each stage with successively finer grit size should entirely remove the damage left by the preceding step.

This should be assessed by inspection using a light microscope at each step.

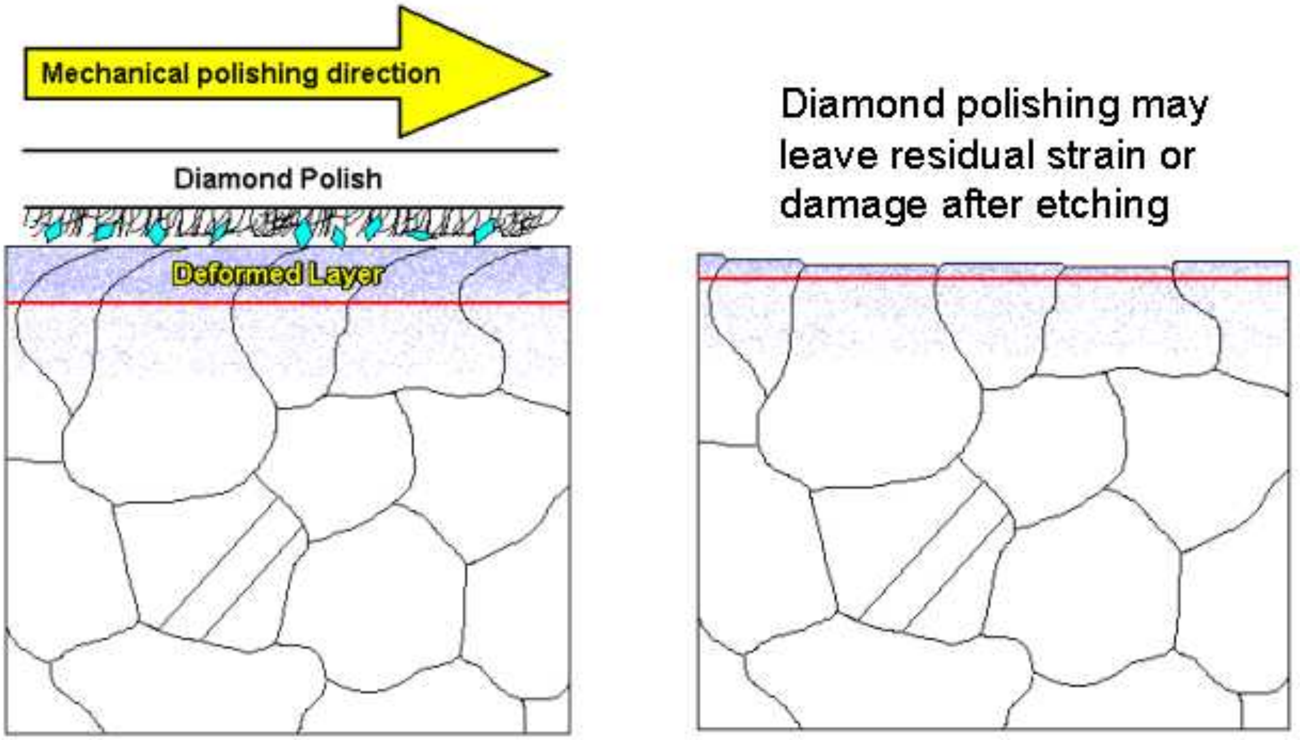
Polishing Samples for EBSD

Polishing is a similar action to grinding, accept that the supporting medium used to hold the abrasive is capable far greater 'shock absorbency' i.e. the ability of the medium to allow the abrasive to move to some degree and conform to the surface asperities of the sample. Thus different polishing surface materials have differing characteristics: soft cloths allow the greatest shock absorbency and therefore allow for gentle polishing with little damage associated. However soft cloths allow the abrasive to abrade different areas at different rates, giving rise to 'relief'. 'Relief' is the term used to describe the undulations that form in a polished surface. Extreme undulations or relief in the polished surface is to be avoided, although a certain amount can be tolerated (or even desirable) because the SEM generally has high depth of field. Harder polishing surfaces or cloths, conversely, produce a flatter or 'plane' surface, but may leave polishing damage in the surface of the material, and promote superficial scratching.

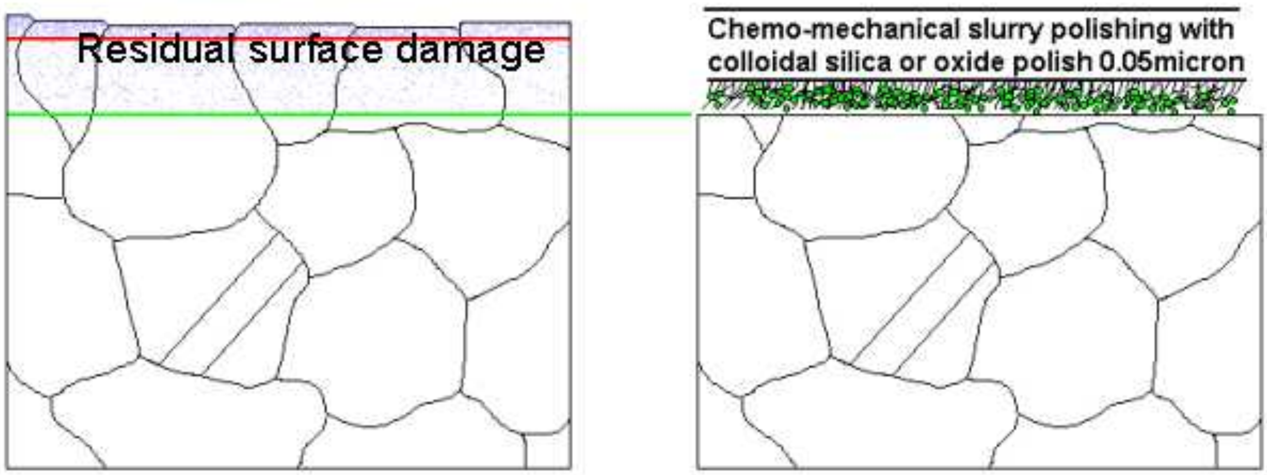
Therefore, it is usually the case that polishing is started on a hard cloth with a coarser abrasive and finished on a softer cloth with a finer abrasive. Final polishing should not be prolonged, but just sufficient to achieve the desired surface finish without causing excessive relief. For EBSD, it is generally necessary to use an additional final polishing stage using colloidal silica.

Colloidal Silica is a chemo-mechanical polish, i.e., it combines the effect of mechanical polishing with ***etching***. This type of stock removal is ideal in many cases for EBSD, as a damage free surface can be obtained with little effort. Typical abrasive size 0.05 micron. Note: Colloidal Silica crystallizes readily and will ruin polishing cloths if left to dry. Further, a film can form on the polished surface of the sample which must be removed. A convenient method to achieve this is to flush the polishing wheel with water during the last few seconds of polishing to clean the sample surface. Remove and dry the sample in the usual manner, using a solvent with low water content and not so volatile as to cause water condensation on the surface. Alcohol is ideal, whereas Acetone is not. Flush the polishing wheel with water until all traces of Colloidal Silica is washed away, spin to drain and store in a suitable container such that contamination of the wheel cannot occur. Meticulous attention to avoiding contamination of wheels is an important aspect to achieve the best results.

Polishing is an extension of grinding i.e., the abrasives are held by the cloth and not free to rotate (lapping)



Diamond polishing may leave residual damage



Chemo-mechanical polishing with Colloidal Silica can remove residual damage

To Summarize:

- Diamond Good for preliminary stages for most materials
- Diamond polishing is not an appropriate final stage due to residual strain left in the sample surface, unless removed by appropriate etching.
- Oxides and Suspensions good for final polishing:
- Alumina based suspensions Suitable for most materials. Scratch free surface with minimal relief. May need to be followed by:
- Colloidal Silica - Chemo-mechanical action - etch combined with abrasive. Especially suited to non-ferrous, ductile (Ti, Zr) also ceramics. Scratch free surface with relief introduced. Develops ***grain*** delineation. Can be used with additional etchants while polishing.

Cloths:

Hard Cloths

- High planarity
- Fast abrasion rates

- Best edge retention
- Minimum relief
- Low risk of ?pull-out?

Soft Cloths

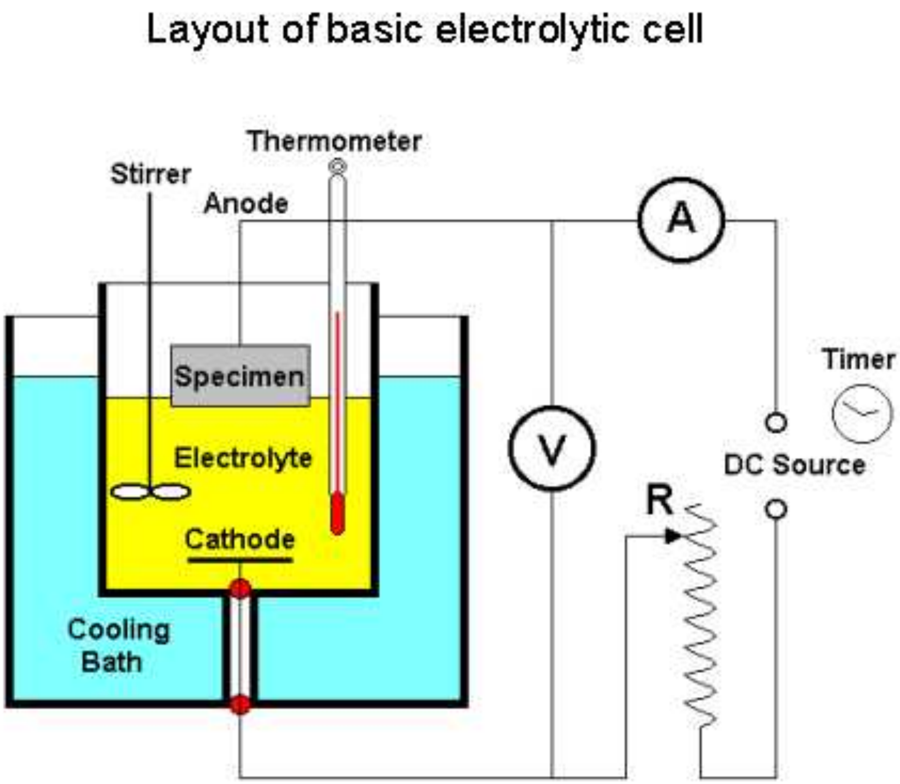
- Superior scratch removal
- Risk of generating surface 'Relief'

Etching Samples for EBSD

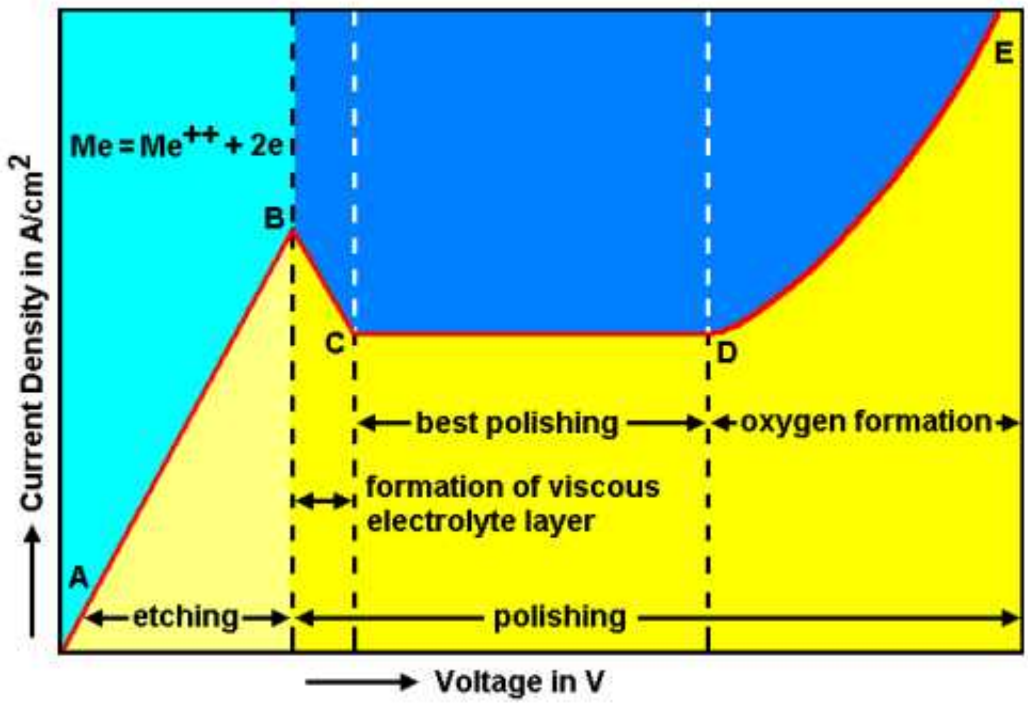
Directly polished surfaces can be inspected using EBSD, but in many cases the pattern quality is improved by etching. Additionally, etching delineates the grain structure, which is of obvious benefit. However, etching may attack a second phase preferentially, or attack grain boundaries excessively. Caution should be exercised when choosing and using etches. Inspect the sample surface using a light microscope before and after etching to assess the effect. Materials that are difficult to polish may benefit from repeated etching and repolishing. This method can expose an undamaged surface suitable for EBSD when conventional polishing and etching fails to achieve an adequate surface. Using special acid or alkali resistant cloths, it is also possible to add dilute etchants to the polishing wheel during polishing. This can be effective, but can be difficult to control. Some experience is required. Any etchant that is used must dissolve the sample surface in an even manner, and not leave behind any oxide or reaction product layers. Such layers can completely suppress diffraction. Many etchants listed in metallographic text books are 'contrast etches' which rely on the formation of different thickness oxide layers to generate colors visible using a light microscope. Therefore such etches are generally not suitable for EBSD.

Electrolytic Polishing/Etching

Electrolytic preparation uses an electrolytic reaction cell containing a liquid electrolyte with two electrodes: an Anode and Cathode. The sample to be polished/etched forms the anode. The electrodes are connected to an external power supply and voltage applied to cause reaction within the cell.



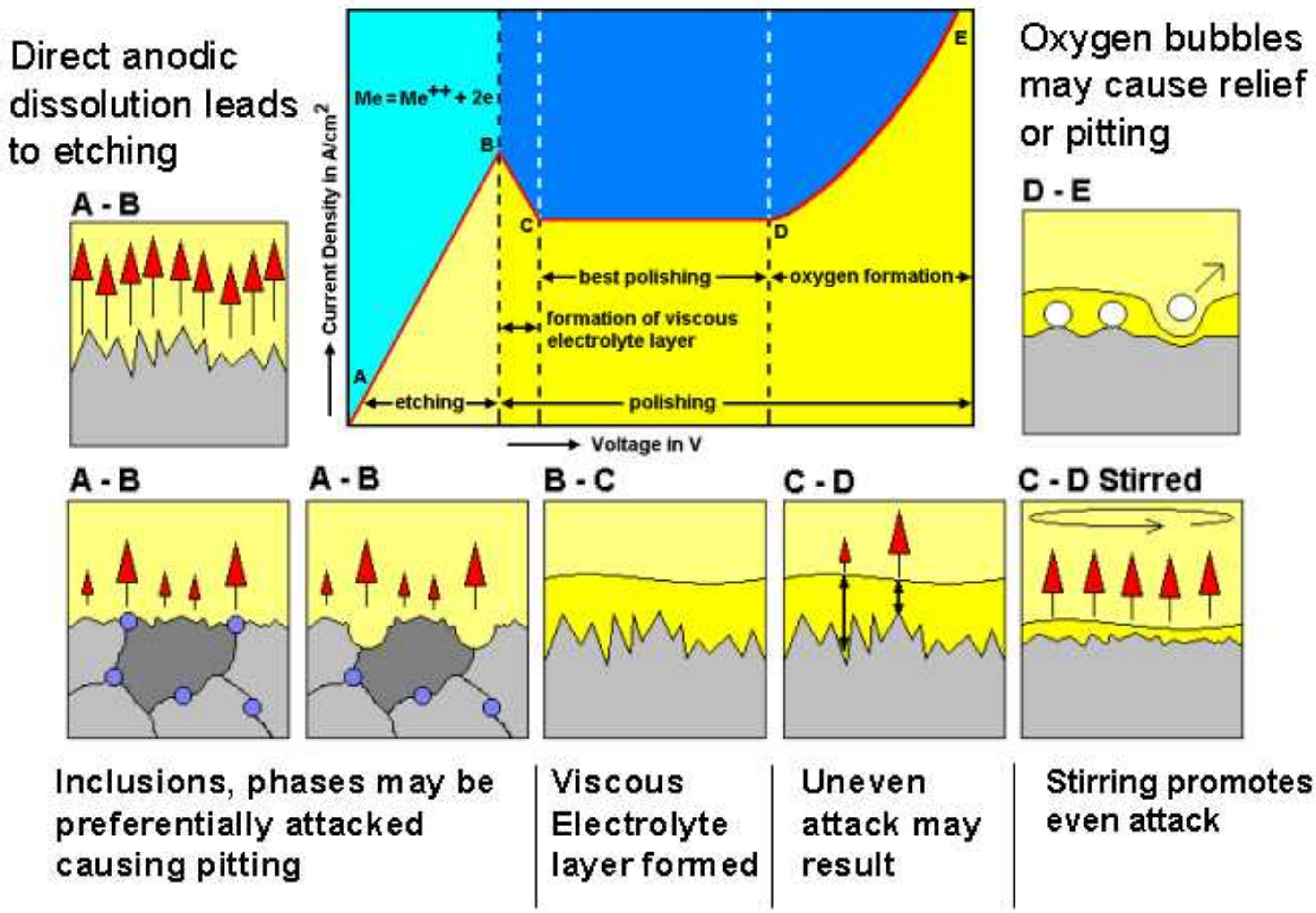
Control of the voltage and current density at the anode, plus electrolyte composition, temperature and agitation are all critical in achieving the desired etching/polishing characteristics.



Electrolytic Cell - Characteristic Curve

Note: this curve is dependent on the electrolyte used and will vary for different electrolytes.

Establishing adequate control of these parameters can be difficult and further, many of the electrolytes are hazardous or even explosive. In the case of the latter, temperature control is critical. Do not attempt electro polishing/etching without the necessary experience and safety measures in place.



Effects at Sample Surface during Electrolytic Attack

To Summarize:

- Do not attempt electro polishing/etching without the necessary experience and safety precautions in place.

Factors controlling etching/polishing characteristics:

- Electrolyte composition
- Electrolyte temperature
- Electrolyte stirring
- Area to be polished/etched (current density)
- Voltage

Advantages:

- Etching or Polishing possible
- Fast
- Can be reproducible
- No mechanical deformation
- Can be automated

- Can produce excellent surfaces for EBSD

Disadvantages:

- Conductive samples only
- Not all alloys can be polished
- Preferential attack or pitting can occur
- No edge retention
- Limited polishing area
- Limited scratch/material removal
- Hazardous Electrolytes
- Temperature control vital
- Establishing correct conditions can be difficult

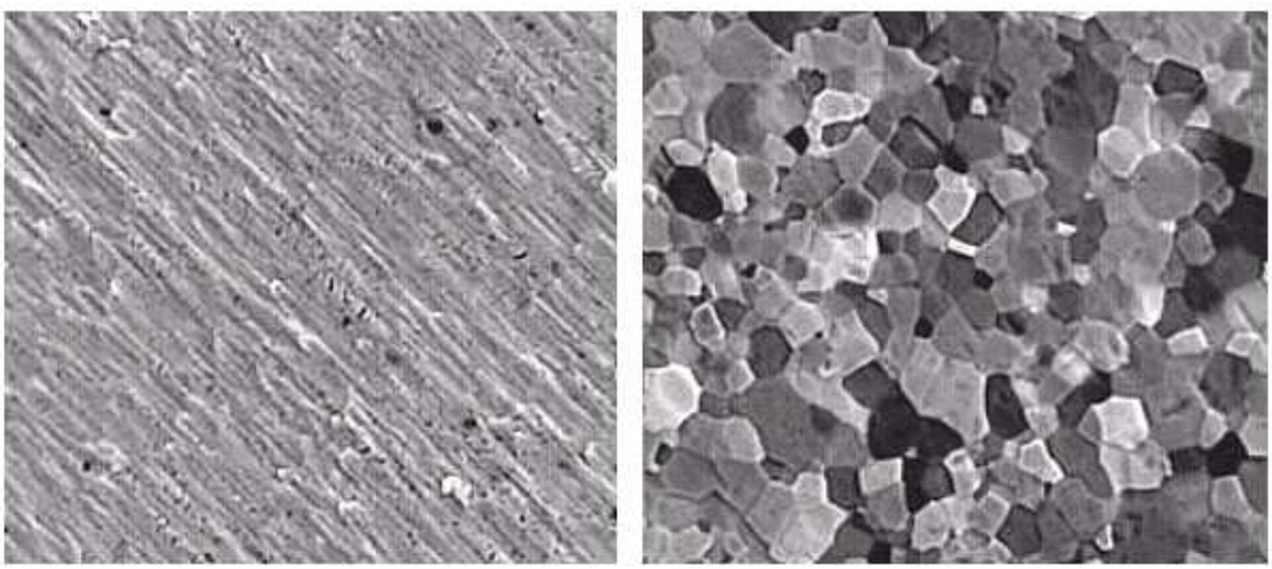
Alternative Sample Preparation methods for EBSD

Plasma Etching

Plasma etching is a process similar to sputter coating, commonly employed for depositing conductive films for SEM inspection. However the process is working in reverse with the sample forming the cathode in the vacuum reaction cell. High voltage is applied between the anode and cathode which oppose each other with a small gap between. The cathode is the 'target' on which the sample is placed. Gas, usually an inert gas such as Argon is leaked under controlled conditions into the vacuum. The gas atoms become positively ionized in the high electrostatic field between the electrodes. The positively charged gas ions accelerate toward the cathode and bombard the sample, eroding the surface in the process. There are many different suppliers and models of plasma etching equipment on the market. Those with low power rating are generally referred to as cleaners, whereas those with higher power or wattage are termed etchers. Plasma etching should generally be used as a means of cleaning or improving a mechanically or electrolytically prepared surface.

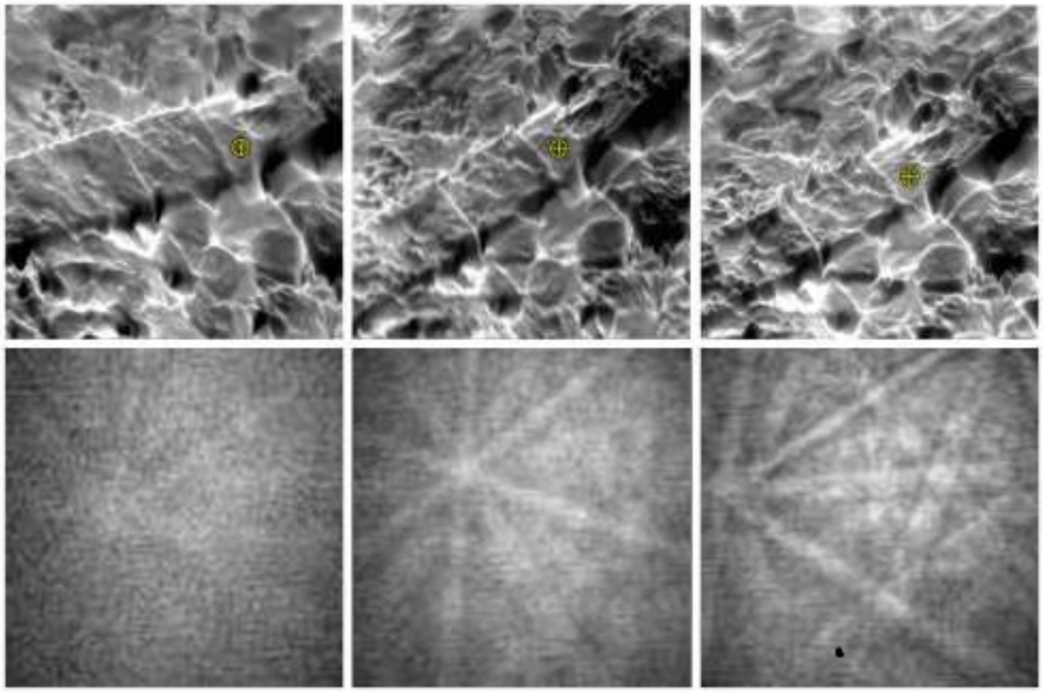
Ion Milling

Ion milling is a process applied to a sample under vacuum whereby a selected area of the surface can be bombarded by an energetic beam of ions. The bombardment erodes the surface, but can also cause damage by ion implantation which can lead to an amorphous layer being formed. Thus rotation and angle of attack is important. Generally using rotation and grazing bombardment angles promote even erosion of the sample surface and minimizes damage effects. However certain grain orientations, grain boundaries and phases my erode at different rates. This should be taken into consideration. Ion Milling can give reasonably high rates of material removal. This can be enhanced by the use of reactive gases, such as Iodine in the reaction chamber. Exhaust gases are a safety hazard and should be safely exhausted. Follow the manufacturer's instructions. Ion milling can produce surfaces suitable for EBSD with minimal prior preparation, especially on materials that are difficult to prepare by conventional metallography, such as zirconium and Zircaloy



Effect of ion milling Titanium. Mechanically prepared surface on left, ion milled surface on right.

Note good diffraction contrast of grains.



No ion milling 5 minutes 15 minutes

Showing the effect of different ion milling times on the quality of the EBSP acquired on a copper sample.

1mA ion beam at 6kV, 60° Tilted and rotated during milling

Attack Etching

Certain materials can be prepared for EBSD by Attack Etching. This is the immersion of a sample in a strong acid or alkali which reacts readily with the material in question. Reaction can be extremely vigorous and therefore this approach should not be attempted without expert chemical knowledge, or without adequate safety precautions. However, it is possible by this method to produce a surface suitable for EBSD in a matter of seconds. However, this approach may remove phases of interest or excessively attack grain boundaries.