EBSD Sample Preparation

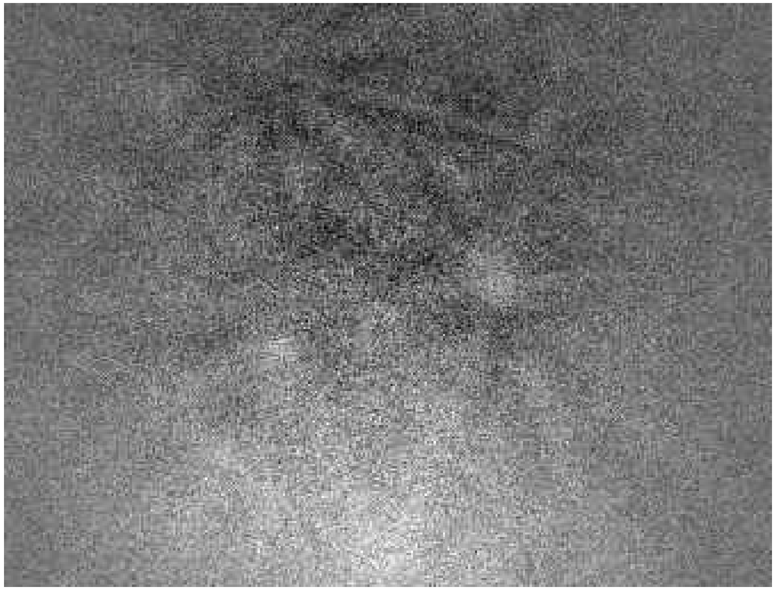
Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

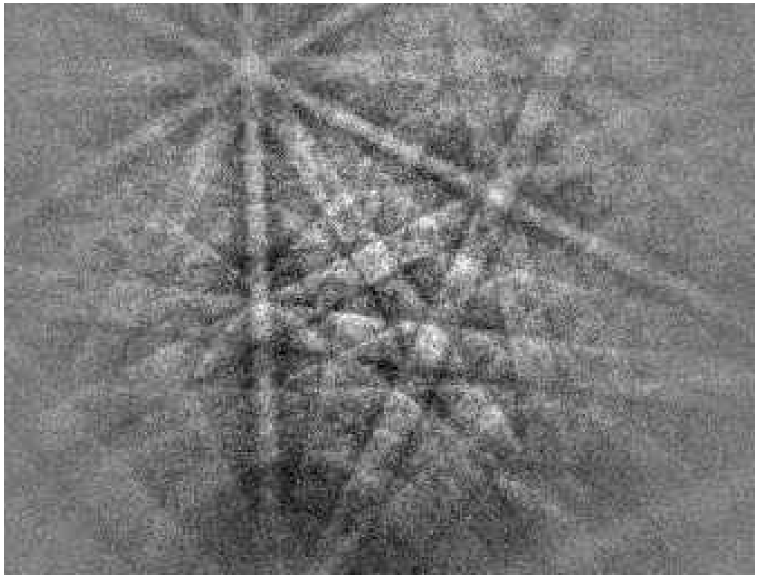
Sample preparation - Introduction

- EBSD is a surface technique – with the diffraction pattern signal coming from the top few nm
- Therefore sample preparation is absolutely critical!
- Any damage to the crystal lattice in the surface layer will worsen the EBSP (or prevent it altogether)
- Many conventional preparation techniques deform the crystal lattice – this will also result in poorer EBSPs
- Some samples do not need any preparation – e.g some as-deposited thin films

Example EBSPs



Poorly prepared sample – deformed / damaged crystal lattice – gives blurred EBSP

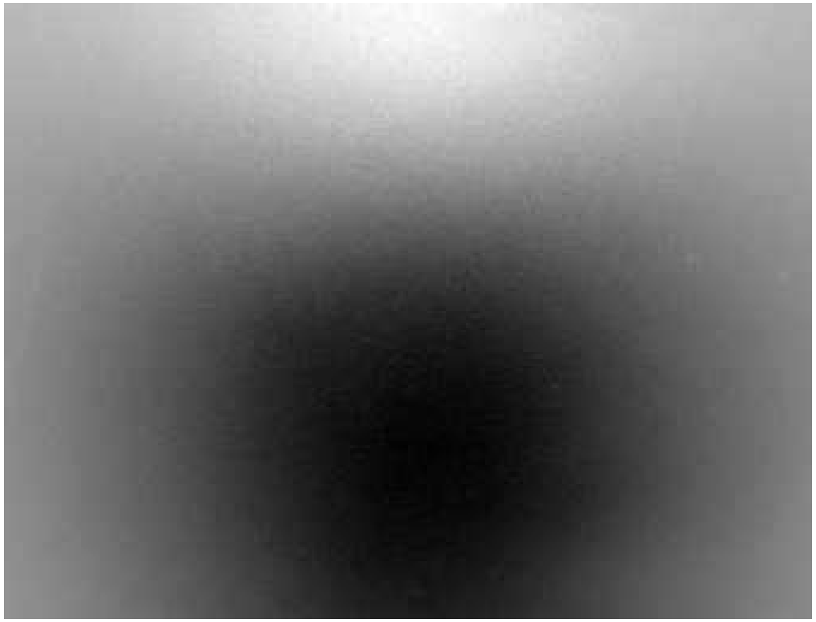


Well prepared sample – pristine crystal lattice – gives sharp EBSP

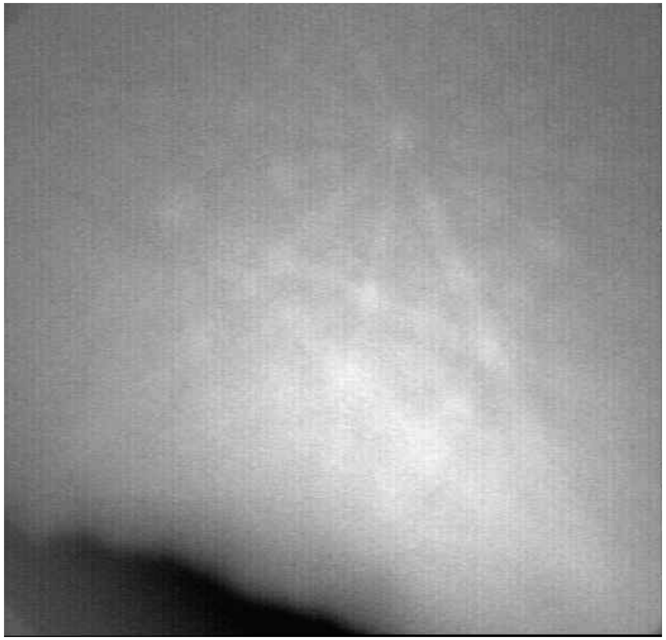
Other Problems

- Due to the high tilt angle, any surface topography (or particles) will cause shadowing – the surface must be free from relief and clean
- You cannot get EBSPs from samples with a normal thickness conductive coat – generally EBSD samples are uncoated...
- This can lead to charging problems with insulators

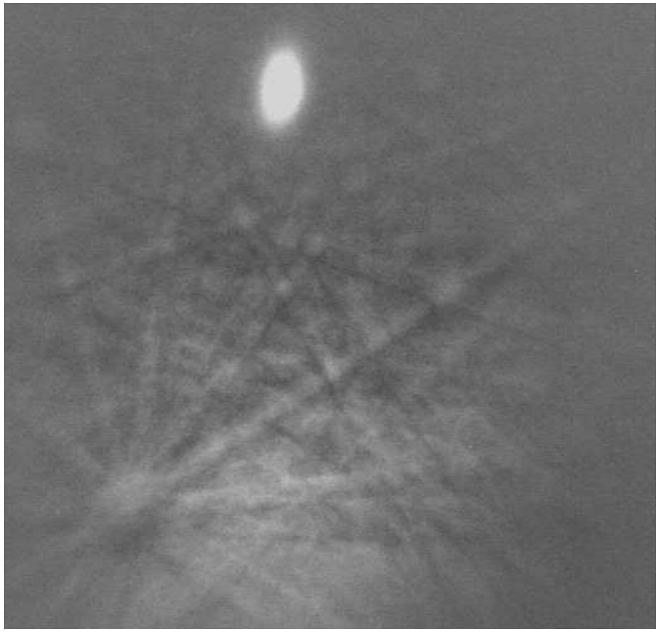
Example EBSPs



"EBSP" from a void or crack



EBSP with some shadowing



EBSP with CL flare

4 ways to polish your sample:

- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion etching / milling

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Mechanical Polishing

- Probably needed on most samples to some extent, to remove relief
- Very useful for polyphase samples, or for analysis of harder particles in a softer matrix
- Ideal for ceramics and geological samples
- Similar process for all materials - easier

Stage 1 - Mounting

- Easiest for subsequent polishing if samples are mounted
- No single mounting technique is best
- Conductive mounting material can be useful to dissipate charge
- BUT – beware - some hot mounting processes may cause some materials (such as many geological minerals) to expand and possibly fracture
- Samples on thin sections can also be polished (with care)

Stage 2 - Grinding

- This is the first mechanical stage of preparation,
- Primarily removes the deformation layer produced in sectioning and produces a flat surface.
- Start with 120 or 240 grit SiC paper
- Proceed to 1200 grit SiC, using water as a lubricant to remove and waste material.

Stage 3- Polishing

- This removes most of the damage caused by grinding
- Can be performed with many types of abrasive and suspension mediums
- Use 4 or 5 steps, with self lubricating diamond suspension or diamond paste
- Work down from15 µm to 0.25 µm grit size on a general purpose cloth

Stage 4 – Colloidal Silica

- This final stage of polishing involves both mechanical and chemical polishing together
- The colloidal silica solution is generally an alkali solution, and slightly etches the sample during the mechanical polishing process
- Ideally you should polish with colloidal silica on an automatic lap for anything from 10 minutes to several hours (depending on the material and the state of the polish)
- Colloidal silica has a number of commercial names – e.g. Syton fluid, OPS …

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Electropolishing - general

- Removes material from the surface of the sample by electrolytic action
- Works very well on many metals.
- Removes any deformation layer on the surface, as well as most surface irregularities
- Unfortunately there is no single electrolyte that will work with all materials
- Necessary to use the correct solution for any given material.

Electropolishing - tips

- Use some of the commercially available packages – come complete with suitable recipes and solutions for different materials
- Examples are Buehler and Struers: also look on their websites for more information
- Look at www.metallography.com, often enquiries about ideal electropolishing recipes for specific materials
- There are many variables that alter the polishing rate: the solution used, the operating voltage, the solution temperature, the sample size and the time of contact – vary the settings to find the best result for your materials

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Chemical Etching

- Chemical etching is a simpler preparation technique than electropolishing
- Variations in the set-up are not so critical
- Sample is immersed in an etchant for a few seconds, before being rinsed (usually with ethanol) and blow-dried

Chemical Etching - tips

- It is important to use an etchant that leaves no residue, and to choose an etchant suited to your material
- One of the most commonly used ones is Nital (5% Nitric acid, 95% Ethanol)
- It is usually necessary to go through at least some of the stages for grinding and fine polishing (maybe down to 1 μm or even 0.25 μm diamond)
- Note: any topography will be further enhanced by the chemical etching process

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Ion beam milling / etching

- Routinely used for the preparation of samples for TEM analysis
- Relatively new preparation technique for EBSD
- Works on almost all types of material – the ion beam removes material from the surface at a rate determined by the voltage, ion gun current, gun-sample geometry and the material itself.
- Can very accurately remove a given thickness of material from the surface, so this technique can also be used for serial sectioning
- Note, however, that these instruments are generally expensive (EUR 30,000 +)

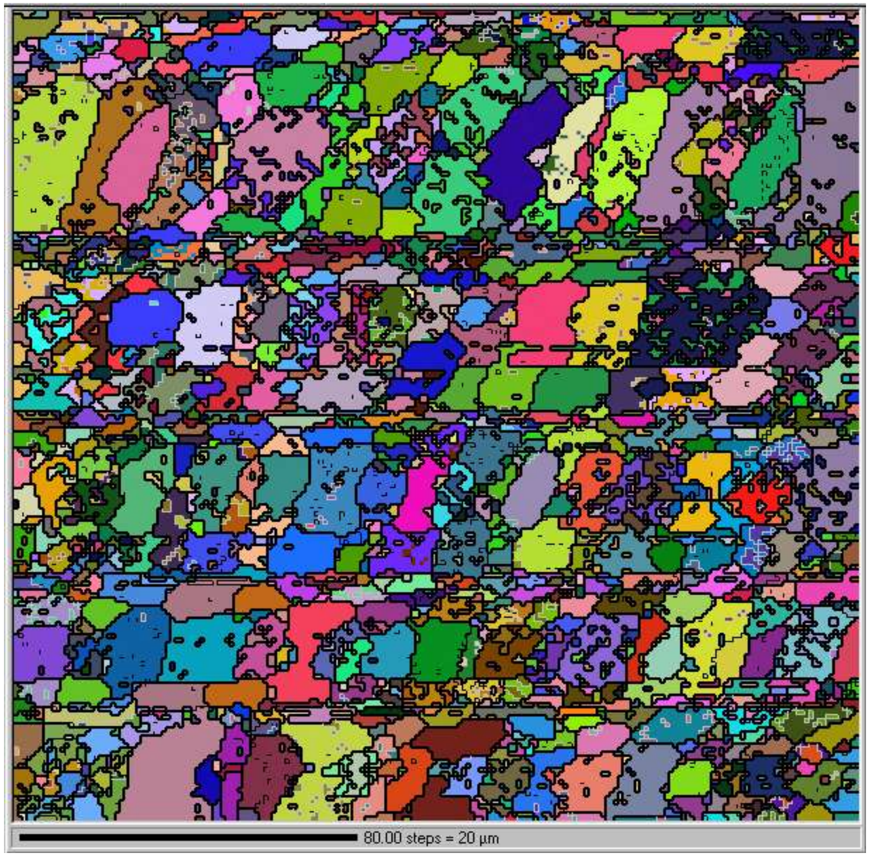
Ion beam milling - tips

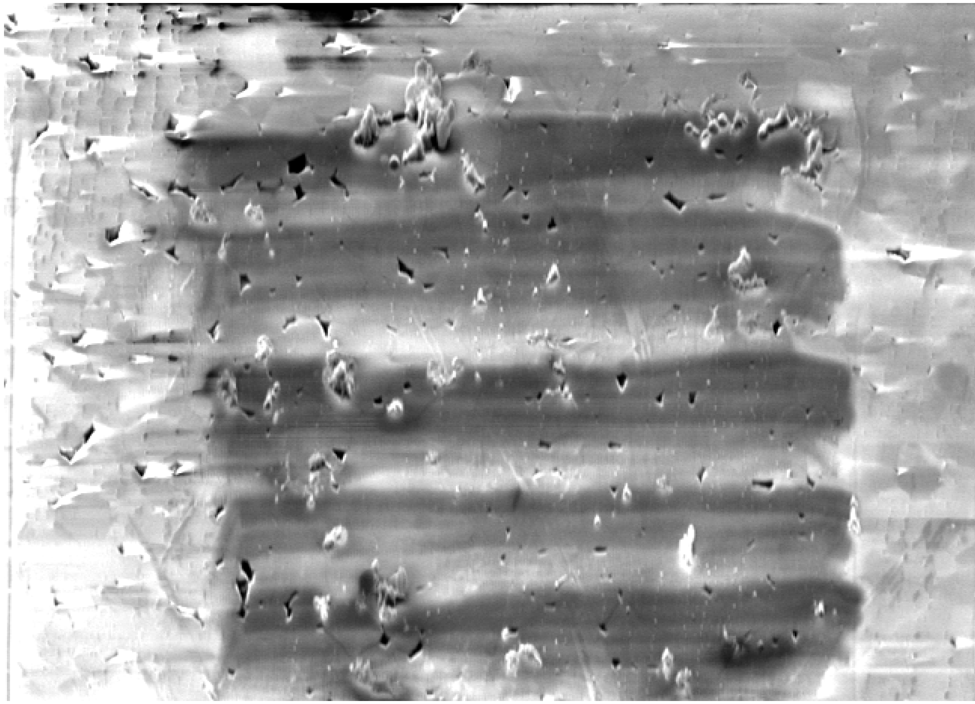
- It is important that the ion beam energy is low, otherwise this can introduce damage to the crystal lattice – this often happens with focused ion beam (FIB) instrument
- Therefore work at low voltages/currents
- Work at high tilts (45° - 70°)
- Better to etch slowly for a long time, than quickly for a short time
- Can also be used to remove layers on the surface, such as oxides or contamination
- Usually limited to relatively small areas (< 1cm^2), making it unsuitable for preparing coarse grained geological samples, for example

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Charging – a worse case scenario





Preventing charging

There are a number of ways to reduce or even eliminate charging on insulators:

- Ensure that there is no surface topography
- Ensure that the polish is very good
- Only turn the beam on once the sample has been tilted to 70°
- Coat with gold before the final polishing stage – this will fill cracks and voids with a conducting network

Preventing charging

- Work in variable pressure / low vacuum mode (if your SEM has this capability): ideally in the pressure range of 10-50 Pa. Any higher and the signal of the diffraction pattern will be too weak
- Work at higher speed so that the beam does not stay in one area for a long time
- Work at lower probe currents and/or accelerating voltages
- Prepare a conductive tract from the sample to the stub/holder – using some conductive paint or metallic tape

But sometimes you will have to apply a conductive coat…

Coating – key points

- Keep the coat very thin – typically in the range of 2-5 nm
- Too thick - the signal to noise ratio will decrease significantly and very poor EBSPs will result
- Too thin - there will not be sufficient conductive material to dissipate the charge
- Ideally the coat should be carbon (either sputtered or evaporated onto the sample)
- But it is possible to use other coating materials such as gold or tungsten
- In cases where the coat may be a little too thick, it may be possible to obtain good EBSPs by increasing the accelerating voltage of the electron beam in order to penetrate through the coat

Talk Outline

- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Sample Storage

As sample preparation is so critical, it make sense to store your samples so that you do not need to repolish them:

- The sample surface does not accumulate dust or other particles
- The sample surface is not scratched or subjected to further deformation
- The sample does not acquire an oxide layer (on some materials this may be impossible, such as Magnesium, and a quick repolish will be needed before further analyses)
- The sample does not acquire moisture. This is especially relevant for materials such as halite that are very sensitive to moisture
- The sample is kept at a cool temperature. Note that even room temperature may cause recrystallisation over a long time in many metals.
- These considerations indicate that a suitable place to store samples is in a desiccator, or at the very least in a clean room in appropriate containers or drawers. If you need to move samples, avoid contact with the surface and use suitable sample containers.

Sample Storage - conclusions

- These considerations indicate that a suitable place to store samples is in a desiccator
- Alternatively in a clean room in appropriate containers or drawers
- If you need to move samples, avoid contact with the surface and use suitable sample containers

And you should be able to get EBSPs like this....!