The Application of SEM & EDS & EBSD for Aluminum Alloy and Alloy Steel

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Sample type : Aluminum Alloy and Alloy Steel

Done by: Jerry, Chu

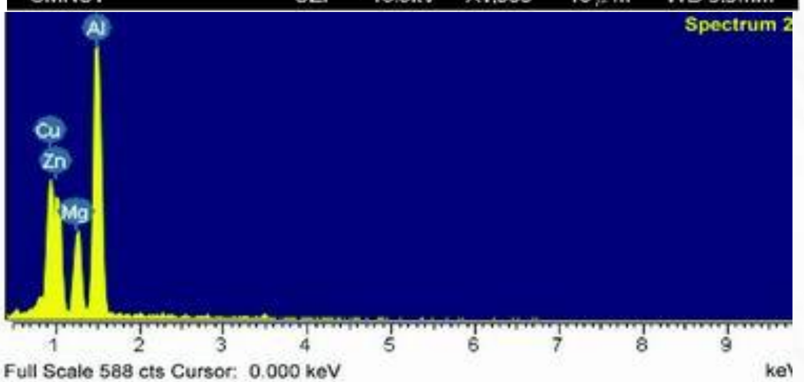
Sample preparation:

Ultimate Polishing

Compression Mounting

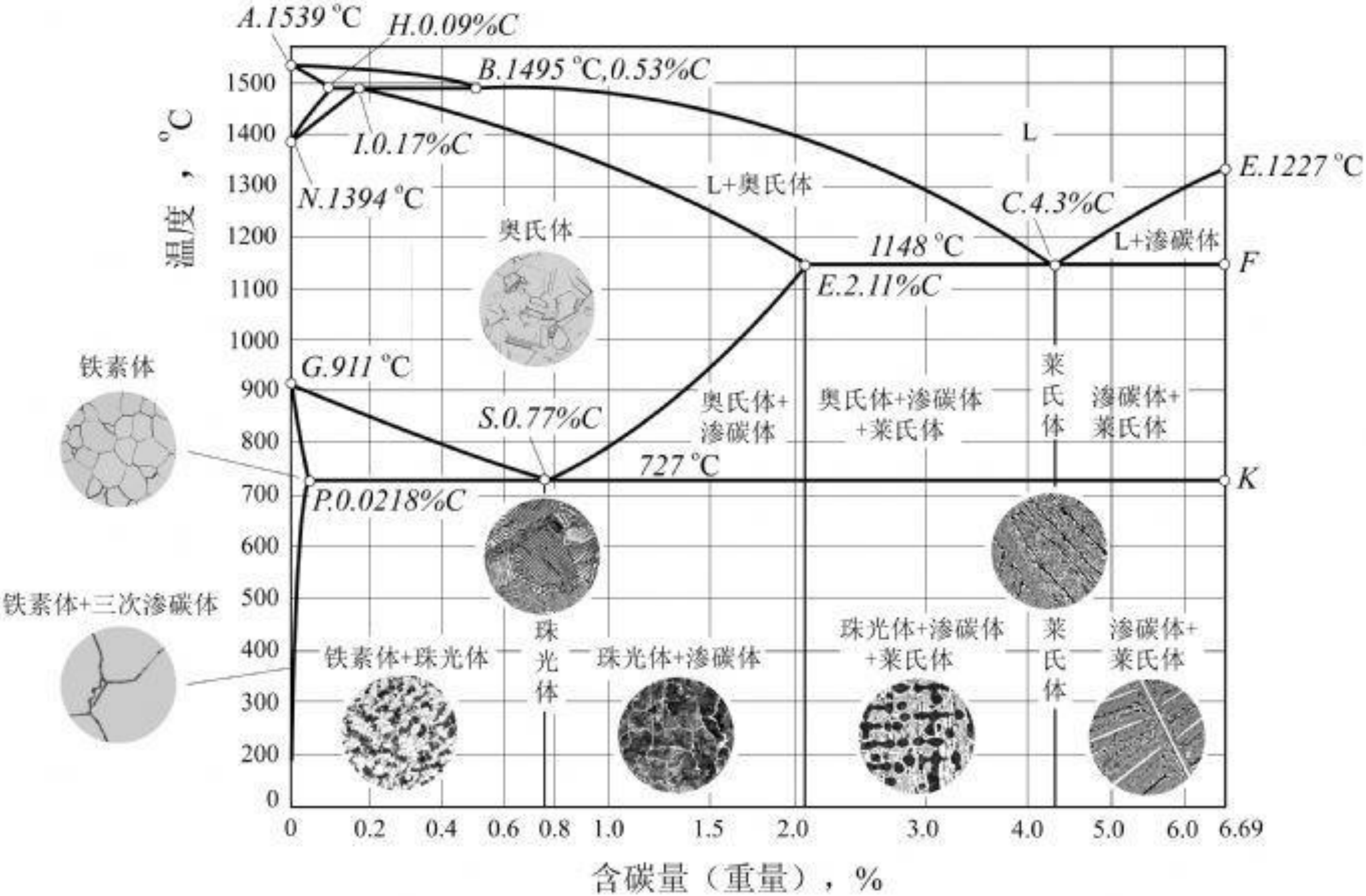
Sectioning

Grinding & Polishing



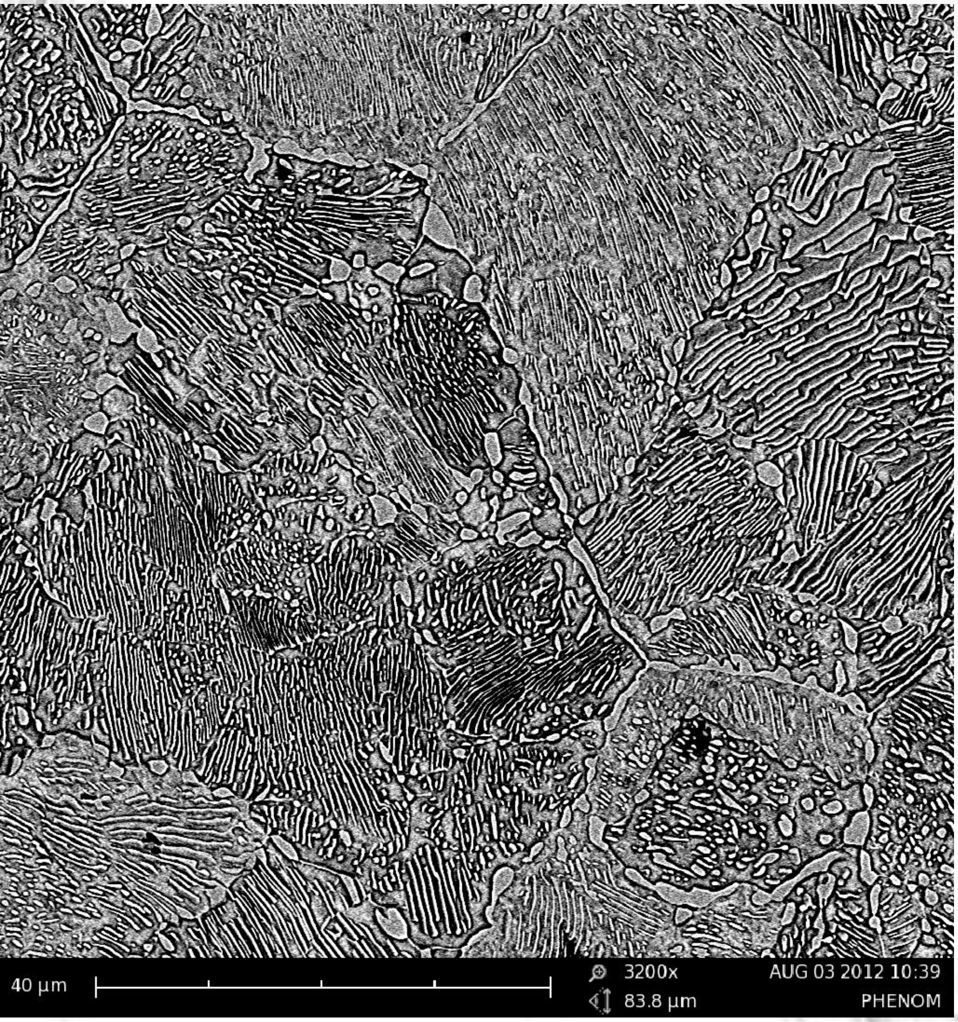
Element	Weight %	Weight %
Mg K A1 K Cu K Zu K	11.30 36.82 25.27 26.61	17.64 51.81 15.10 15.45
Totals	100.00	

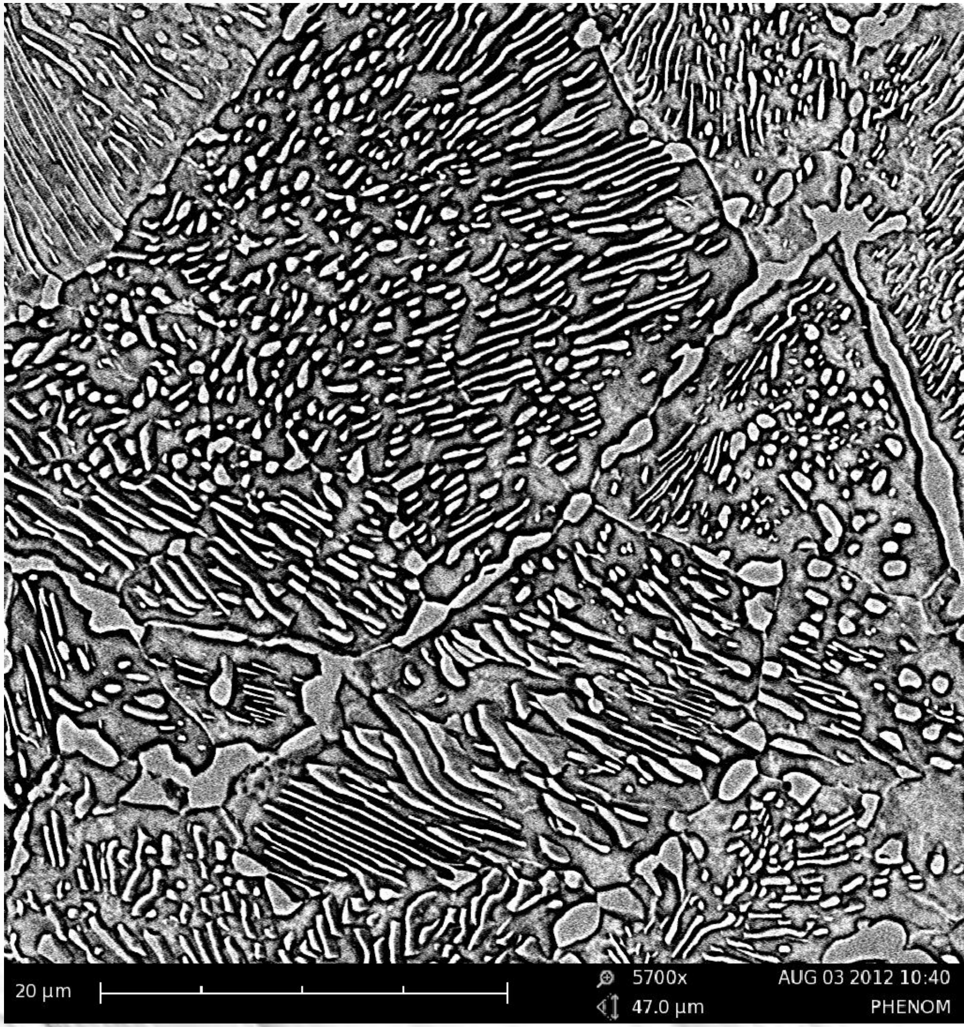
The SEM & EDS is used for the observation of high resolution microstructure and elemental analysis. You can fully apply it to understand how it effects on the microstructure under the casting parameters and homogenized process. Moreover, it also can be used for the structure observation of the afterward extrusion process, material analysis and the evaluation of the defect.


A.1539 °C
H.0.09%C
B.1495 °C,0.53%C
I.0.17%C
N.1394 °C
L
L+奥氏体
E.1227 °C
C.4.3%C
L+渗碳体
1148 °C
F
E.2.11%C
奥氏体
温度，°C
铁素体
G.911 °C
奥氏体+渗碳体
奥氏体+渗碳体+莱氏体
莱氏体
渗碳体+莱氏体
S.0.77%C
727 °C
K
P.0.0218%C
铁素体+三次渗碳体
铁素体+珠光体
珠光体
珠光体+渗碳体
珠光体+渗碳体+莱氏体
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
0
0 0.2 0.4 0.6 0.8 1.0 1.5 2.0 3.0 4.0 5.0 6.0 6.69
含碳量（重量），%

碳鋼的SEM金相分析：

鋼材的SEM金相分析:


40 µm
3200x
83.8 µm
AUG 03 2012 10:39
PHENOM


20 µm
5700x
47.0 µm
AUG 03 2012 10:40
PHENOM

鋼材的SEM的金相分析:

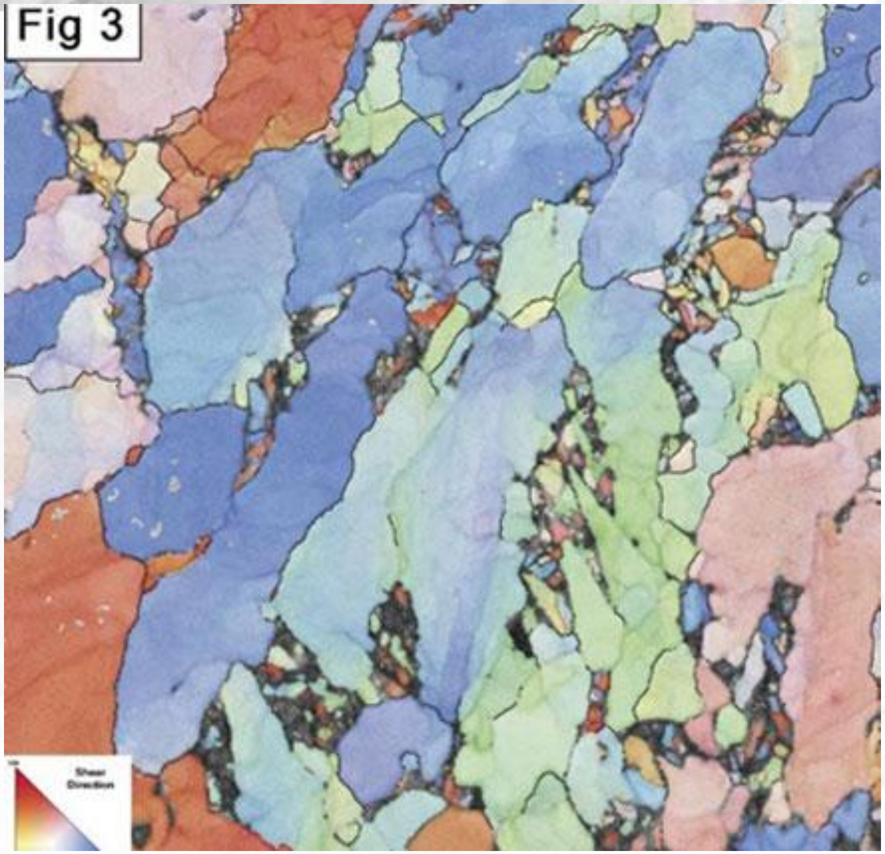
1. 藉由SEM金相分析，可清楚分辨共析鋼，亞共析鋼與過共析鋼的金相結構。
2. 完全退火組織的金相觀察。
3. 感應硬化處理的金相觀察。
4. 脫碳處理深度與金相結構觀察。
5. 滲碳處理與過滲碳處理的金相觀察。
6. 硬化處理與調質熱處理的金相觀察。
7. 正常化處理(細化晶粒)的金相觀察。
8. 退火處理與球化處理的金相觀察。

鋼材的SEM+EDS元素分析:

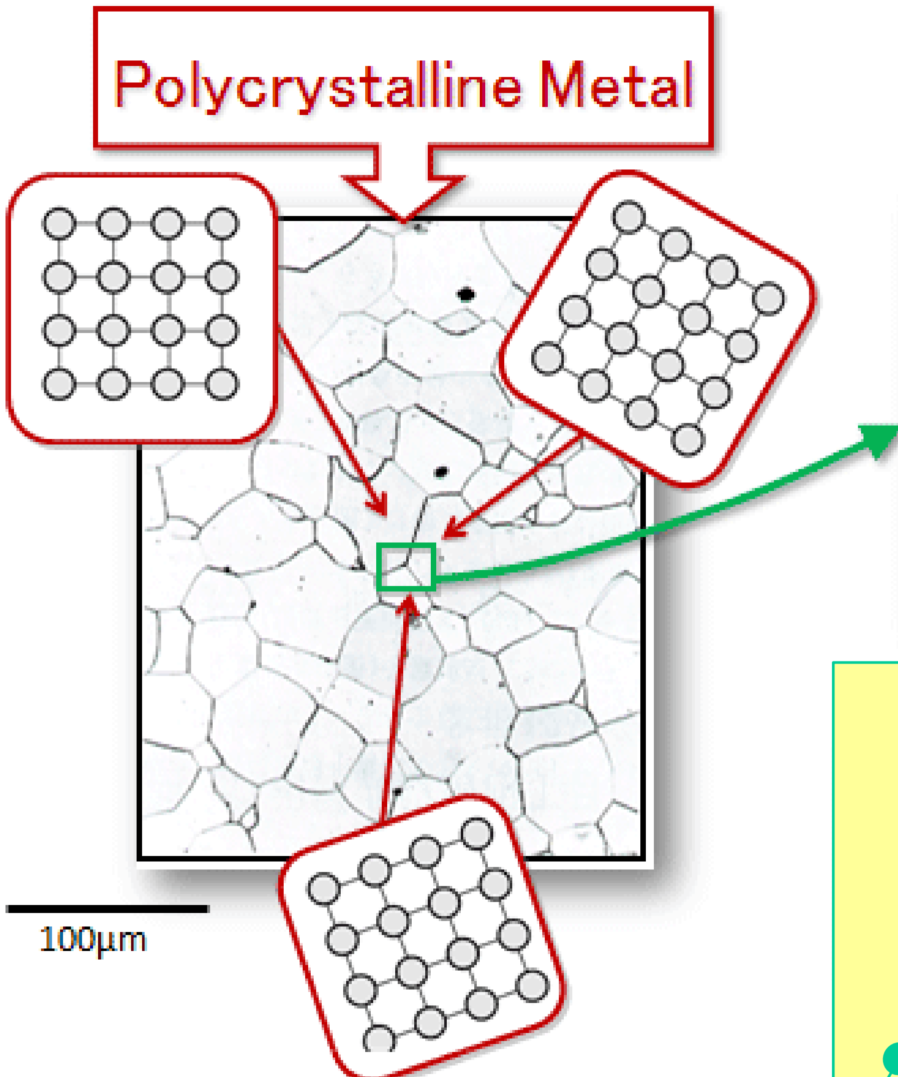
★藉由SEM+EDS元素分析，可清楚分辨鋼材種類，析出物成分，異物分析與微區材質變化。

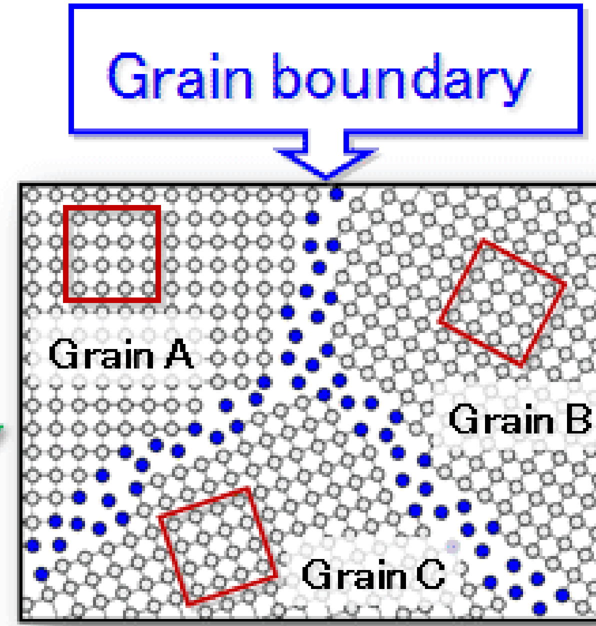
例如:

1. 304不鏽鋼，18%Cr+8%Ni。
2. 303不鏽鋼為304不鏽鋼加入少量的P和S。
3. 302不鏽鋼比304不鏽鋼，碳含量更高。
4. 304N不鏽鋼為304不鏽鋼含氮。
5. 309S不鏽鋼比304不鏽鋼，Ni,Cr含量更高。
6. 316不鏽鋼為304不鏽鋼添加了鉬。
7. 347不鏽鋼為304不鏽鋼添加了鈮。
8. 408不鏽鋼，11%Cr+8%Ni。
9. 409不鏽鋼為408不鏽鋼添加入Ti。

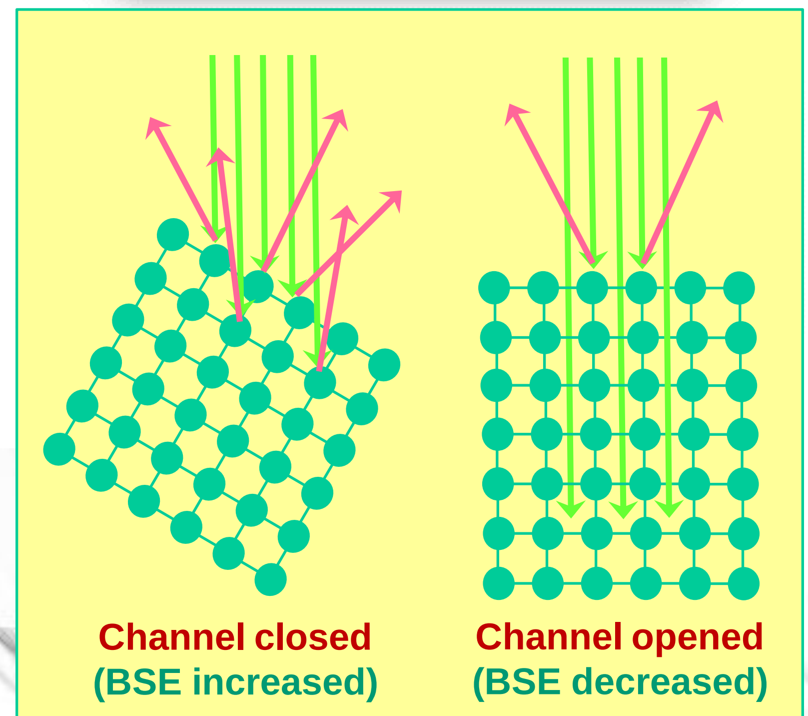


Characterization of steel using EBSD:
在鋼材的應變特徵中，裂紋的產生與擴展是非常重要的，高規格SEM & EBSD需求的樣品前處理變得十分的重要，使樣品更容易觀察到晶粒尺寸，晶界特徵，紋理，取向等資訊，使之更能夠理解機械應力與化學侵蝕下的裂紋行為。

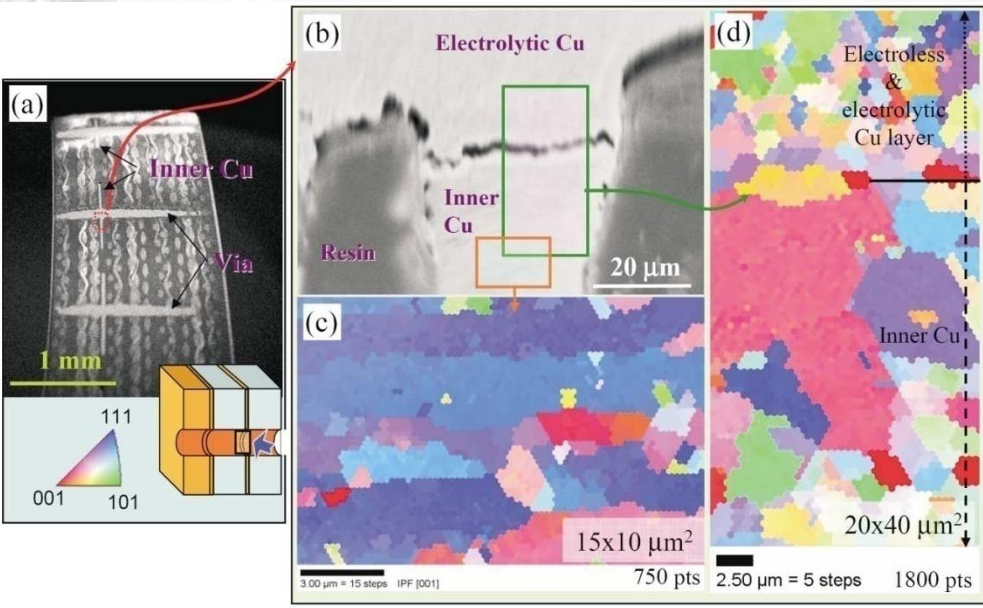


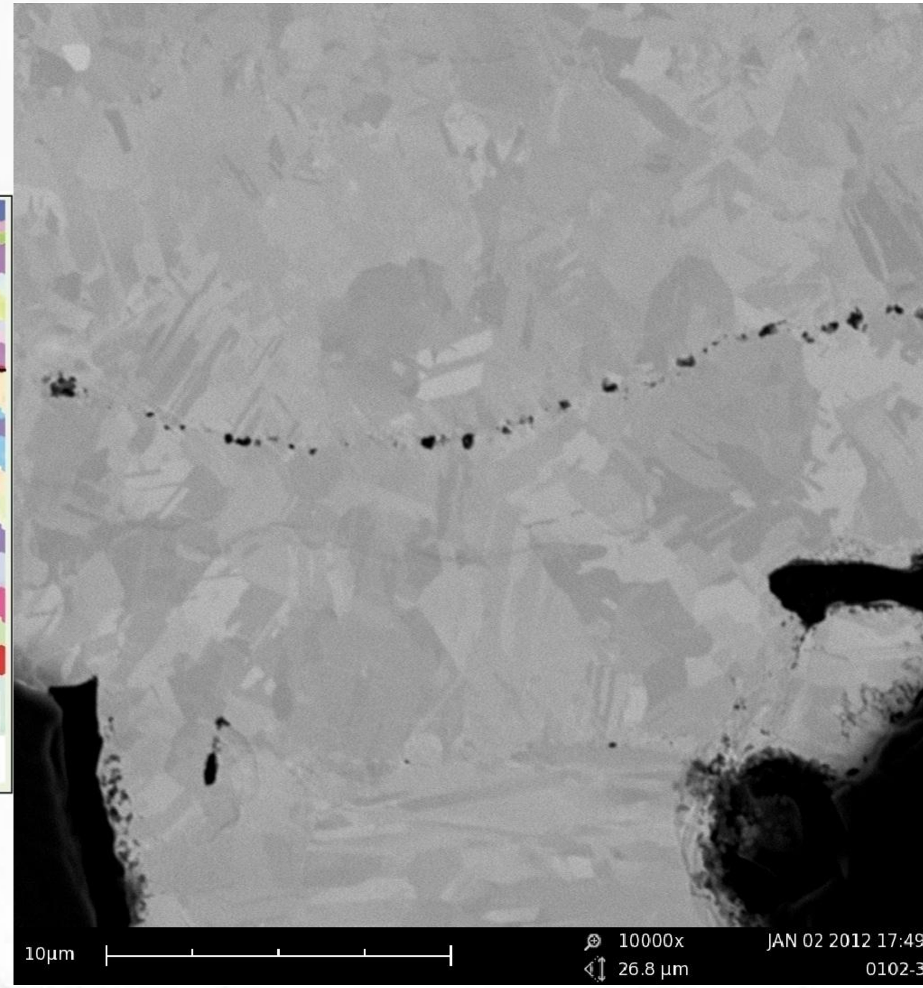


The regular arrangement of atoms in crystalline materials can influence the backscattering of electrons. (ECCI) Electron Channeling Contract Imaging

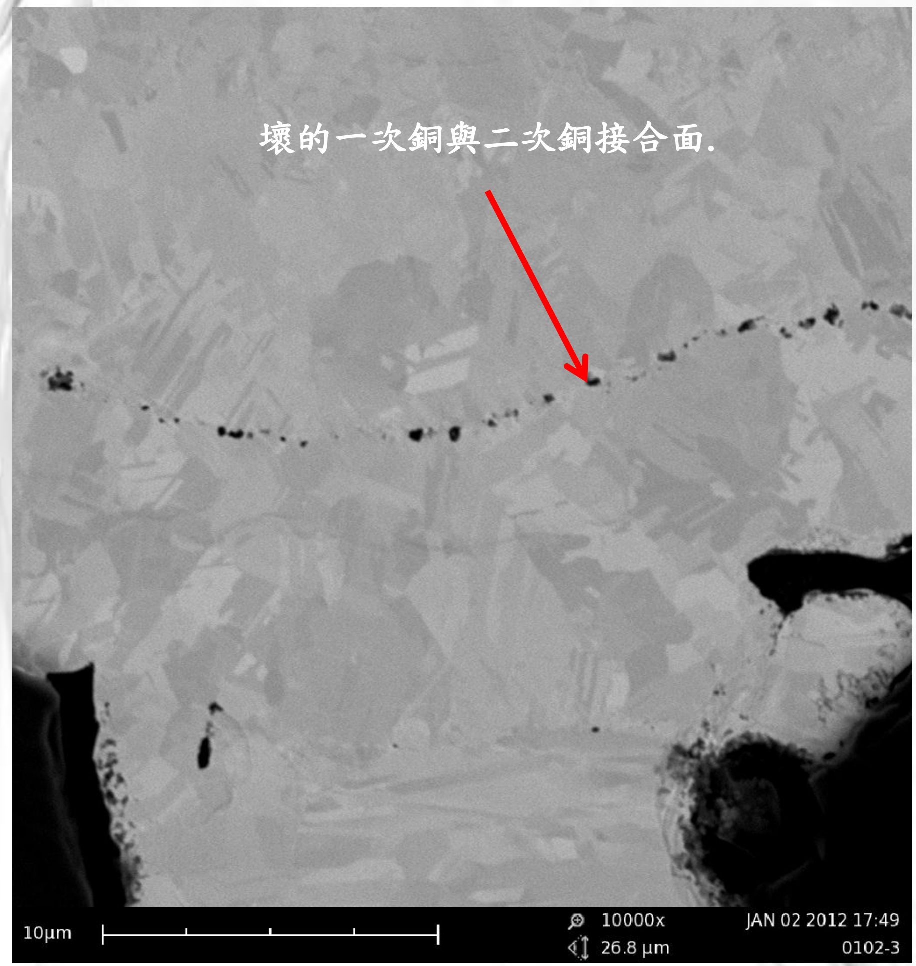


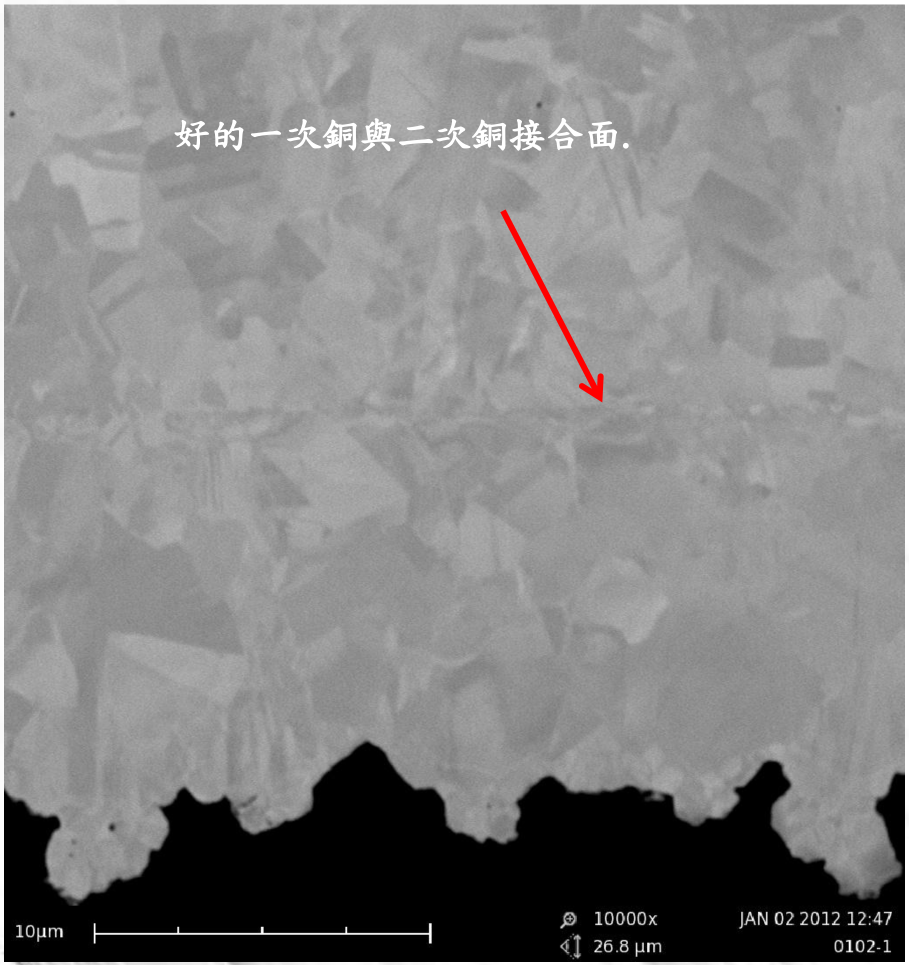
SEM Sample Preparation System – Demo result
Final Polishing for Orientation Imaging





SEM Sample Preparation System – Demo result
Final Polishing for Orientation Imaging

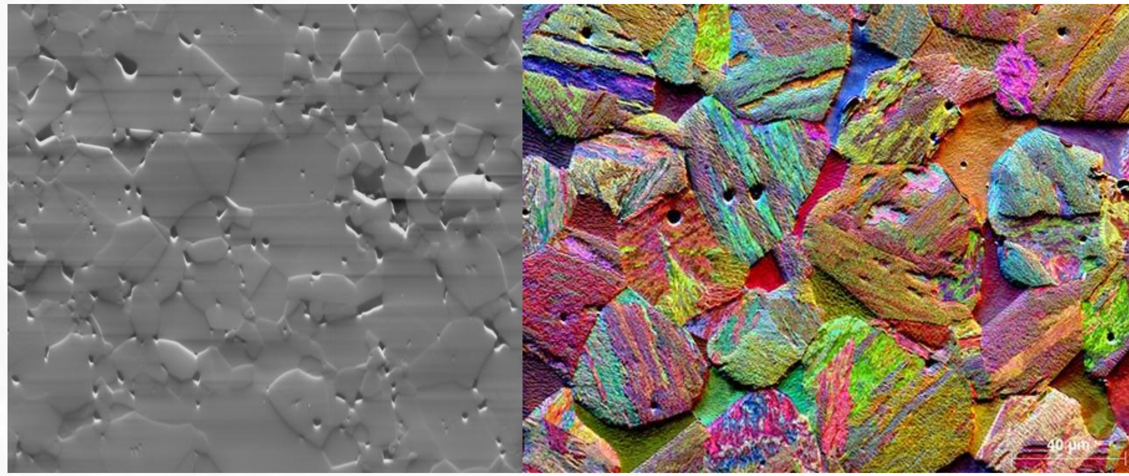


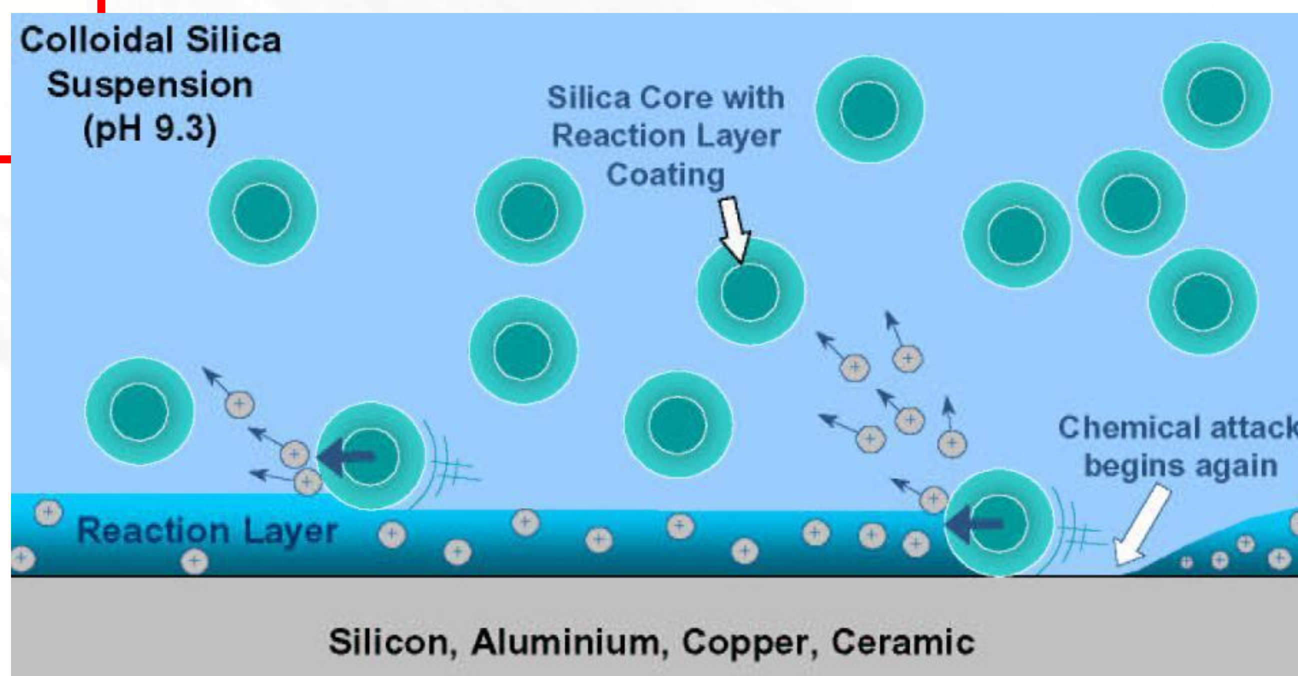


SEM Sample Preparation System

Ultimate Polishing

MasterMet, 0.06um SiO2最終拋光懸浮液。





Talk Outline

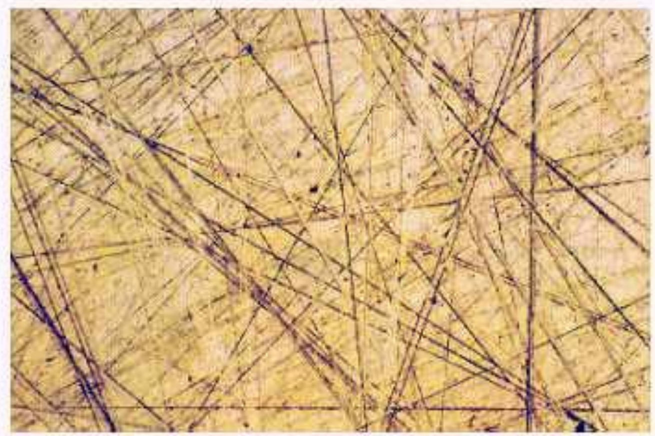
- Introduction
- Mechanical Polishing
- Electropolishing
- Chemical Etching
- Ion Beam Milling / Ion Etching
- Coating
- Sample storage

Scanning electron microscopy applications

Why we need the SEM sample preparation: Rapid slope cutting and gentle surface cleaning

Fine Grinding

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



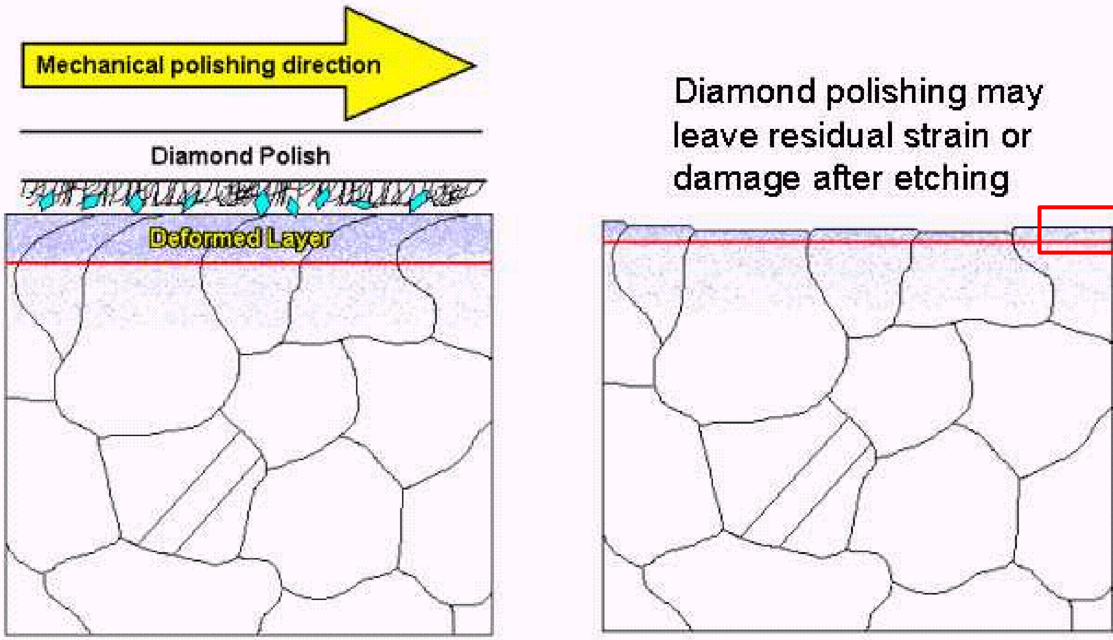
Fine and coarser scratches together indicate insufficient fine grinding

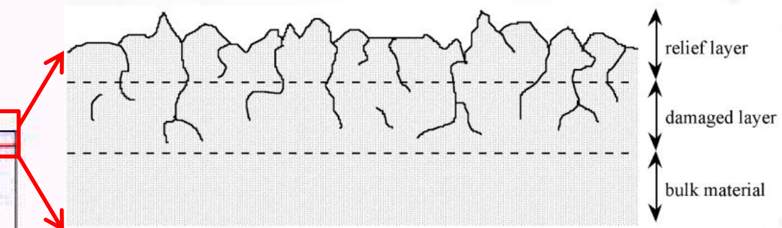
- Each stage with successively finer grit size should entirely remove the damage left by the preceding step
- This should be assessed by inspection using a light microscope at each step

Scanning electron microscopy applications

Why we need the SEM sample preparation:
Rapid slope cutting and gentle surface cleaning

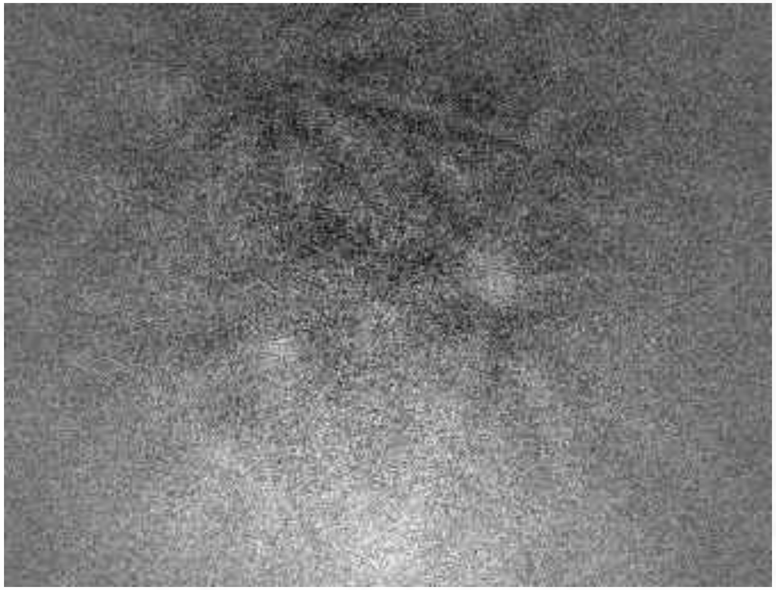
Polishing is an extension of grinding i.e., the abrasives are held by the cloth and not free to rotate (lapping)

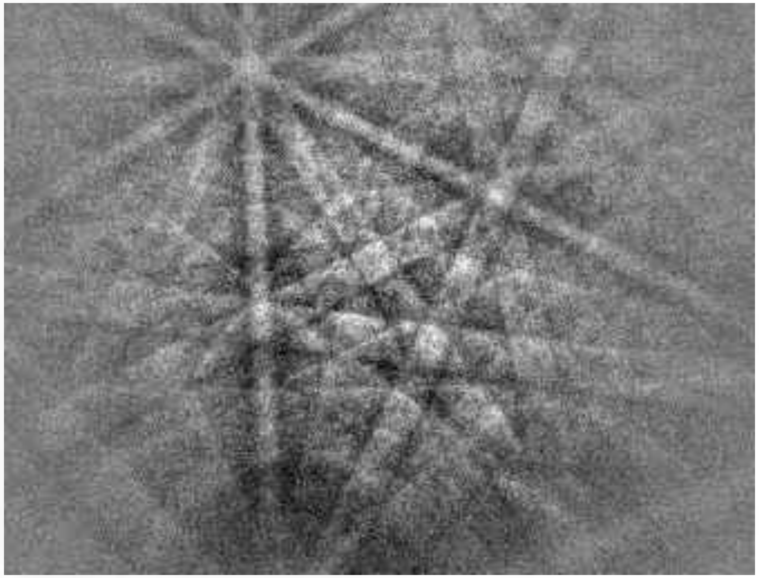




所以就電顯樣品製備的角度而言，只是傳統研磨拋光是不夠的，如需更清晰的影像與分析結果，就必須採用更進一步的拋光方式。

Example EBSPs





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

Well prepared sample – crystal lattice – gives sharp EBSP

Other problems

- **Any surface topography will cause shadowing.**
- **Normal thickness conductive coat.**
- **Charging problem.**

Preparation techniques

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

There are a number of techniques for preparing samples for EBSD, and choosing the correct technique is very dependent on the composition and structure of sample.
Unfortunately there is no single technique that works with all materials.

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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)

- Samples on thin sections can also be polished.(with care)

Tripod Polisher Kit:

SBT EBSD-150 Polishing Kit:

Thin sections

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

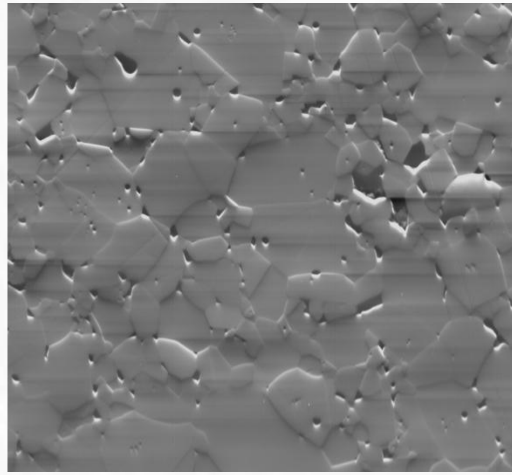
- This removes most of the damage caused by grinding.
- Can be performed with many types of abrasive and suspension mediums.
- Use 4 or 5 steps, with self lubricating diamond suspension or diamond paste.
- Work down from15 μm to 0.25 μm grit size on a general purpose cloth.

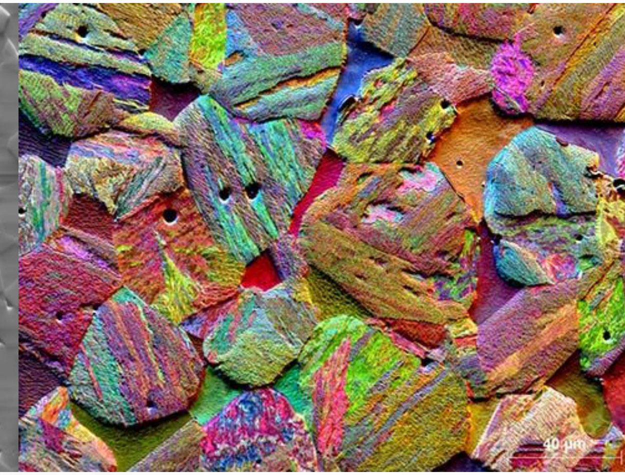
Stage 4 – Colloidal Silica

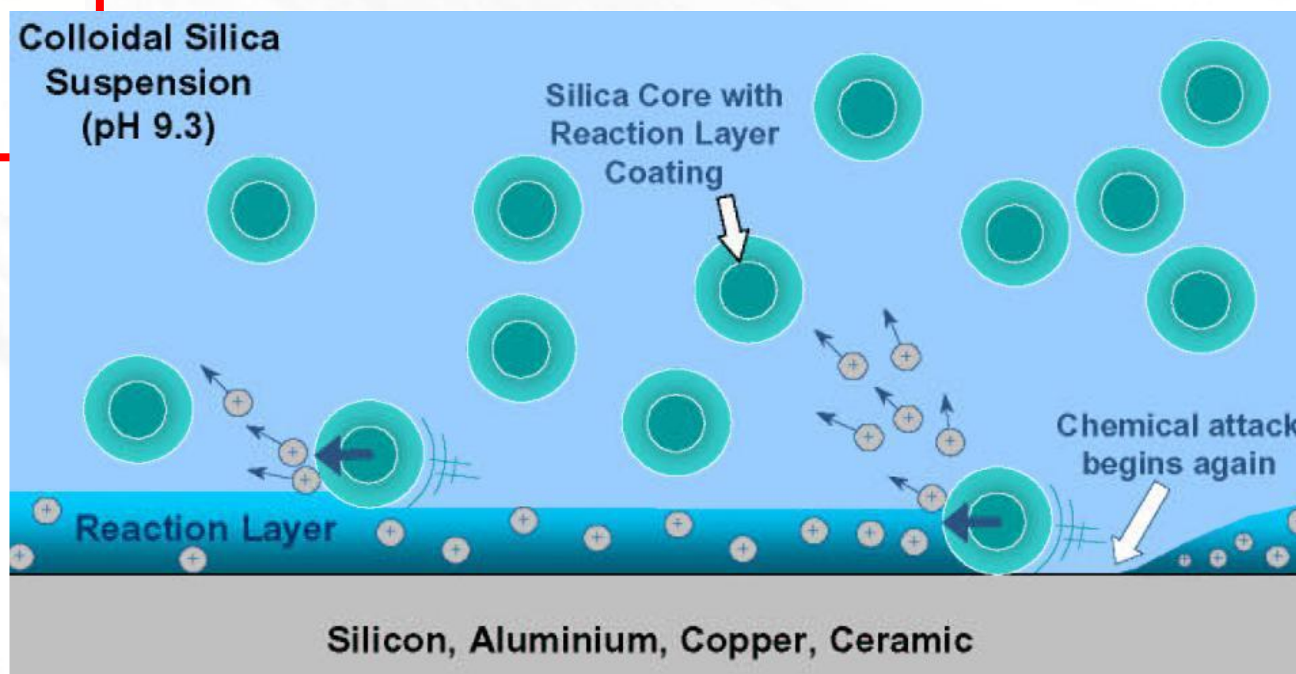
- This final stage of polishing involves both mechanical and chemical polishing together
- The colloidal silica solution is generally an alkali solution, and slightly etches the sample during the mechanical polishing process
- Ideally you should polish with colloidal silica on an automatic lap for anything 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 …

SEM Sample Preparation System

Ultimate Polishing







MasterMet, 0.06um
SiO2最終拋光懸浮液。

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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: also look on Buehler 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

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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

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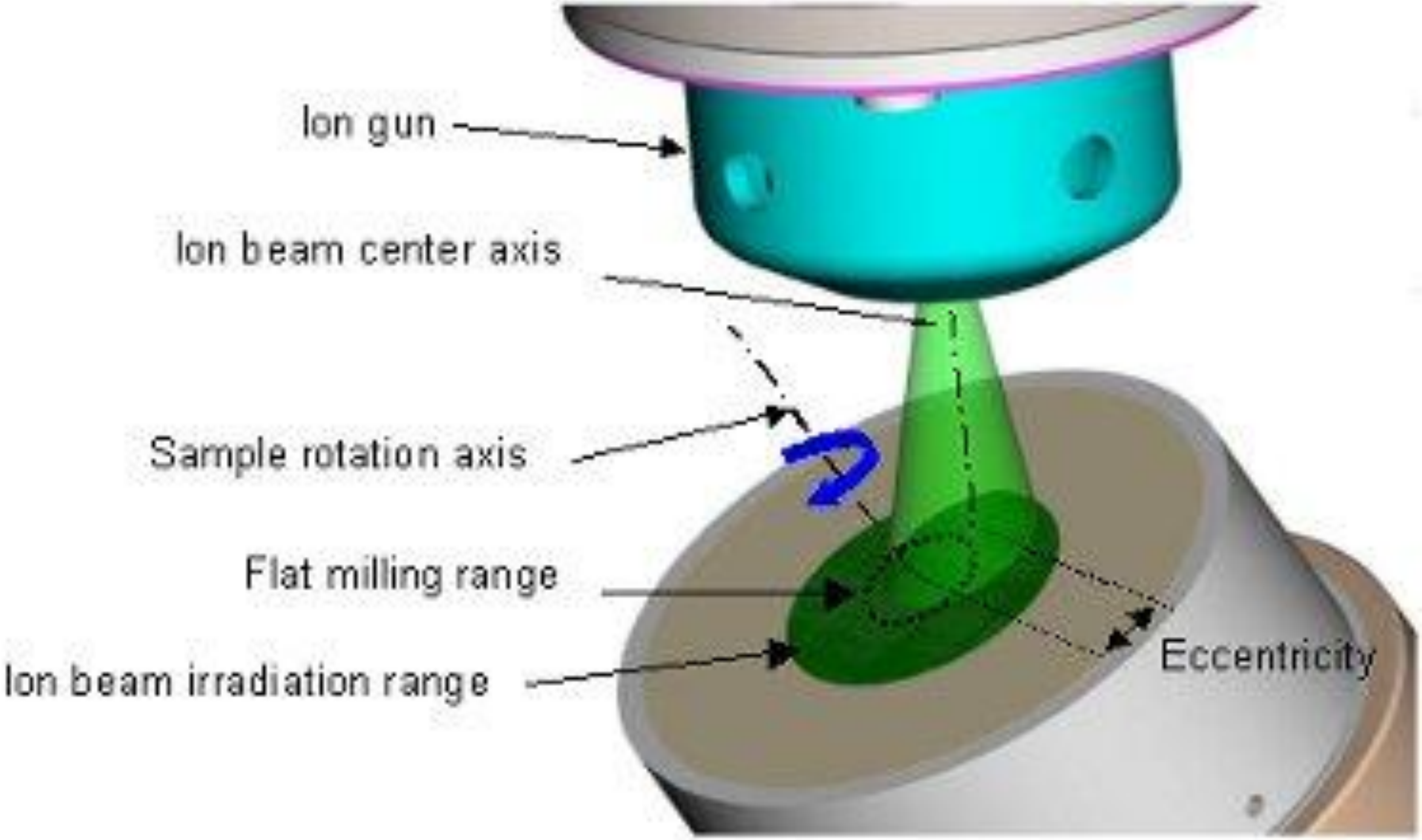
Ion beam milling / etching

- 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



Principle of Flat Milling

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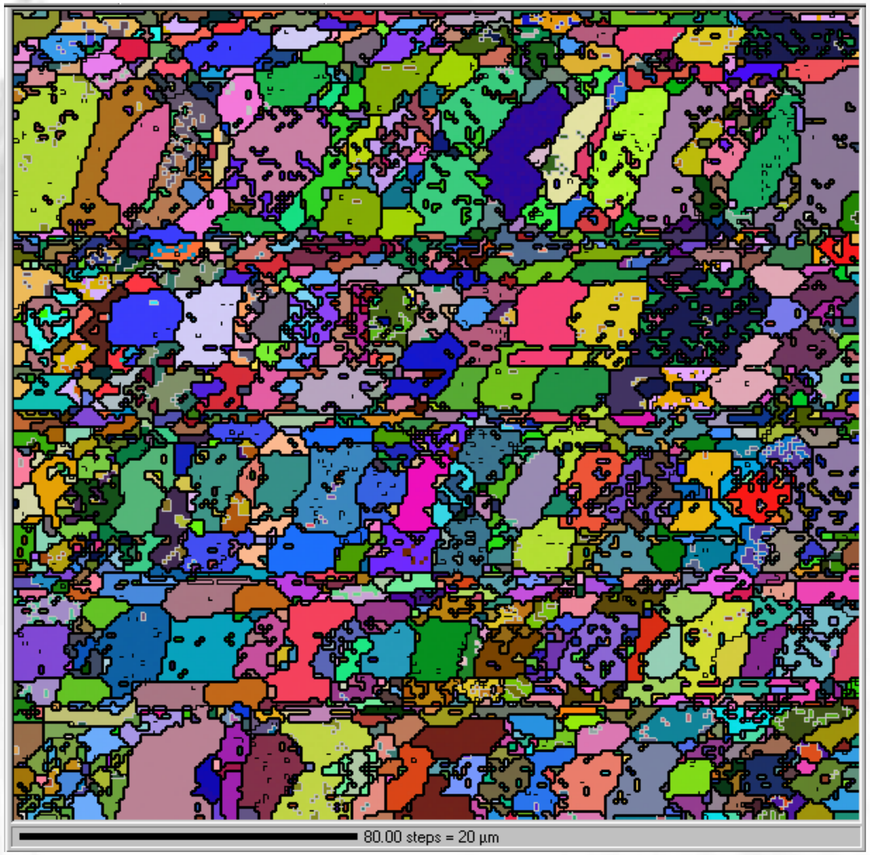
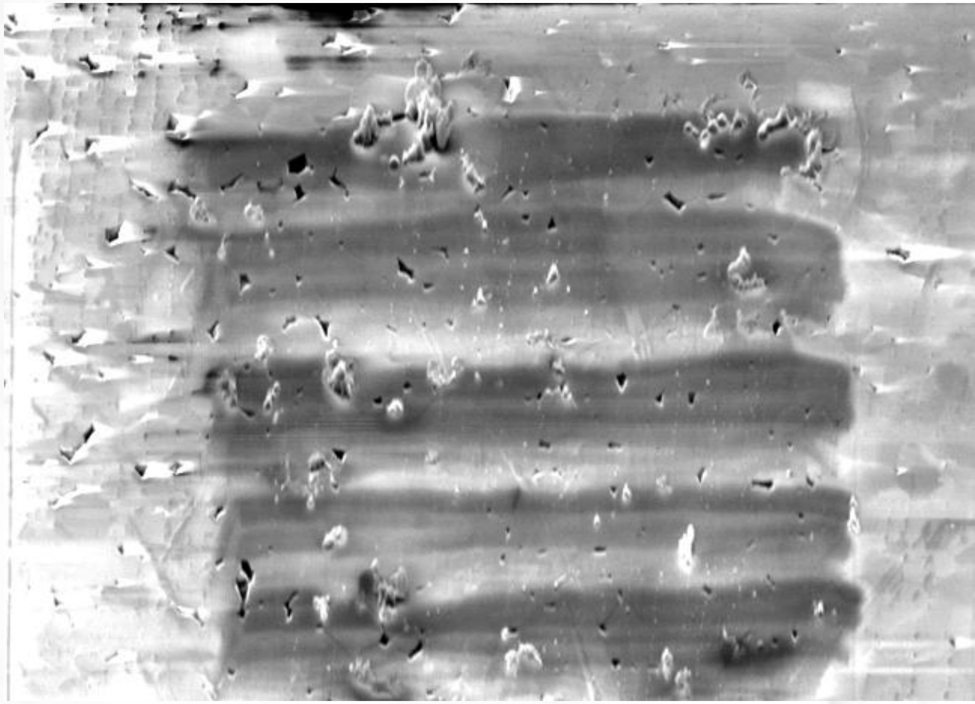
Charging – a worse case scenario





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

208C High Vacuum Turbo Carbon Coater - for TEM, Microprobe, FESEM and EBSD

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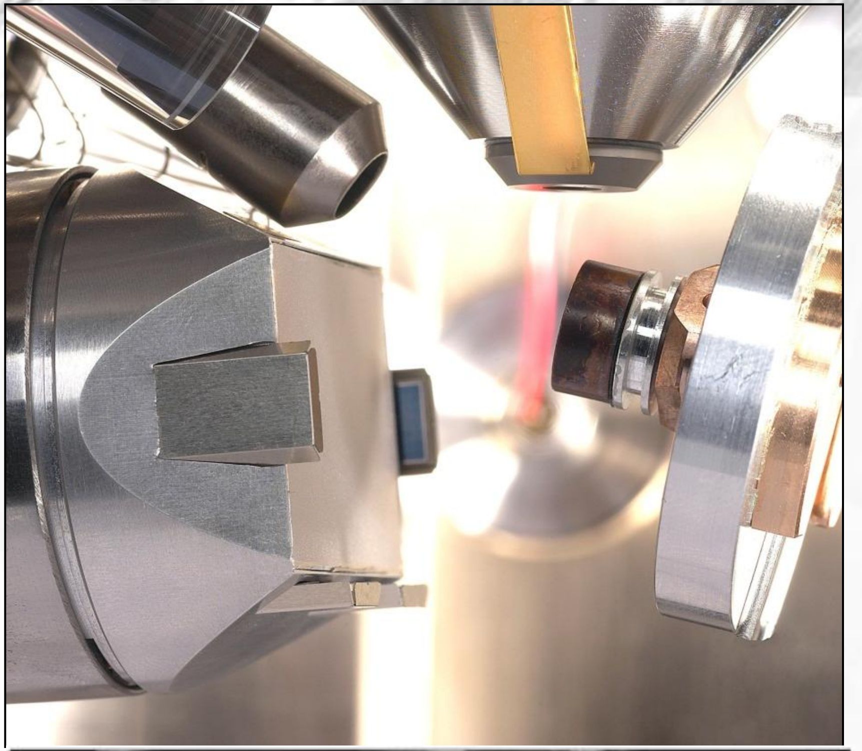
Sample Storage

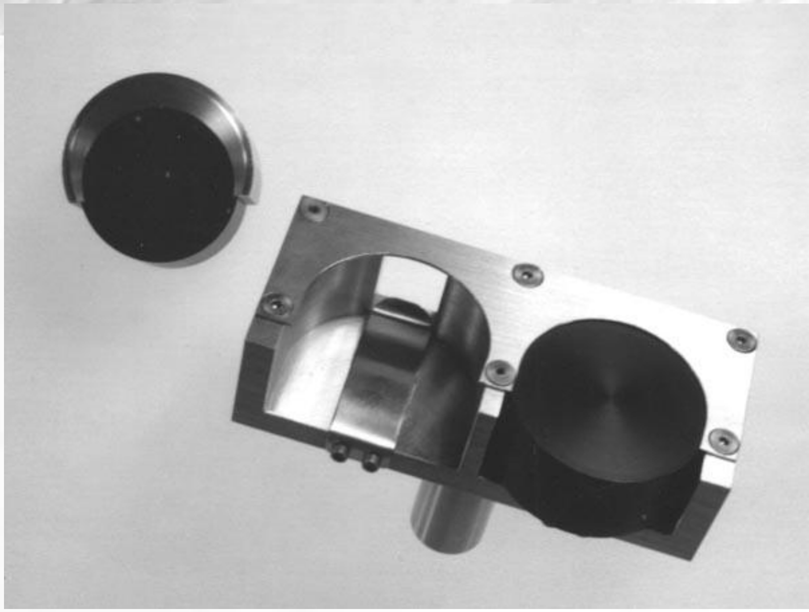
As sample preparation is so critical, it make sense to store your samples so that you do not need to repolish them:

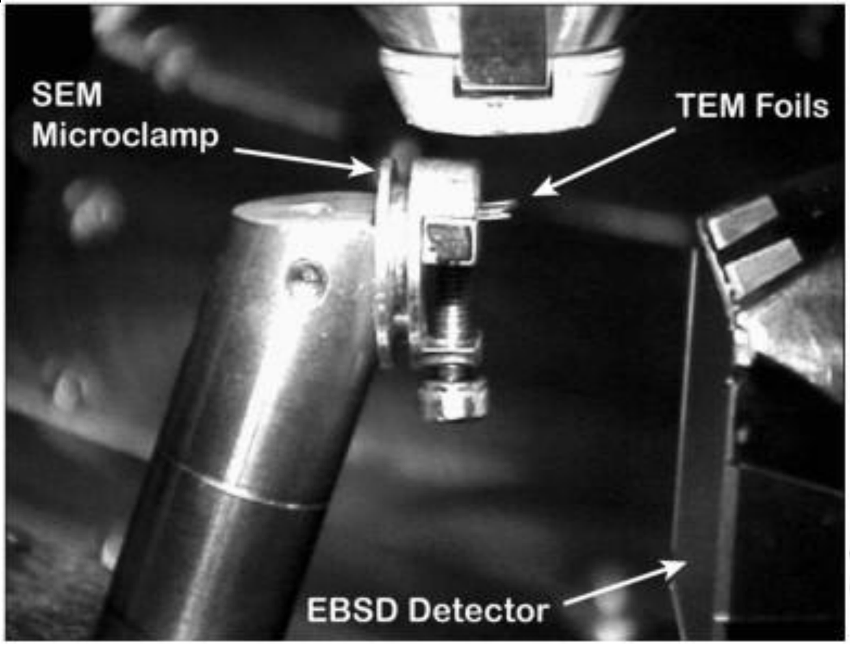
- The sample surface does not accumulate dust or other particles
- The sample surface is not scratched or subjected to further deformation
- The sample does not acquire an oxide layer (on some materials this may be impossible, such as Magnesium, and a quick repolish will be needed before further analyses)
- 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.

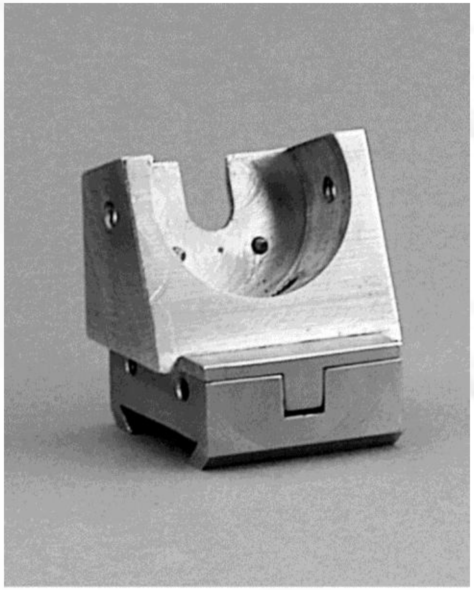
Sample Storage - conclusions

- 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

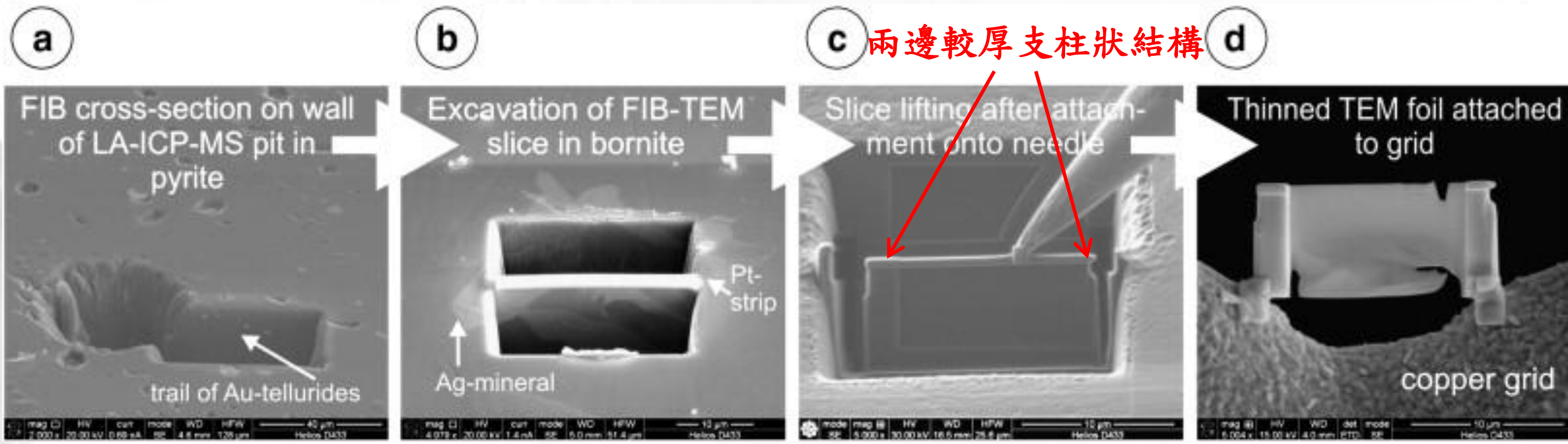




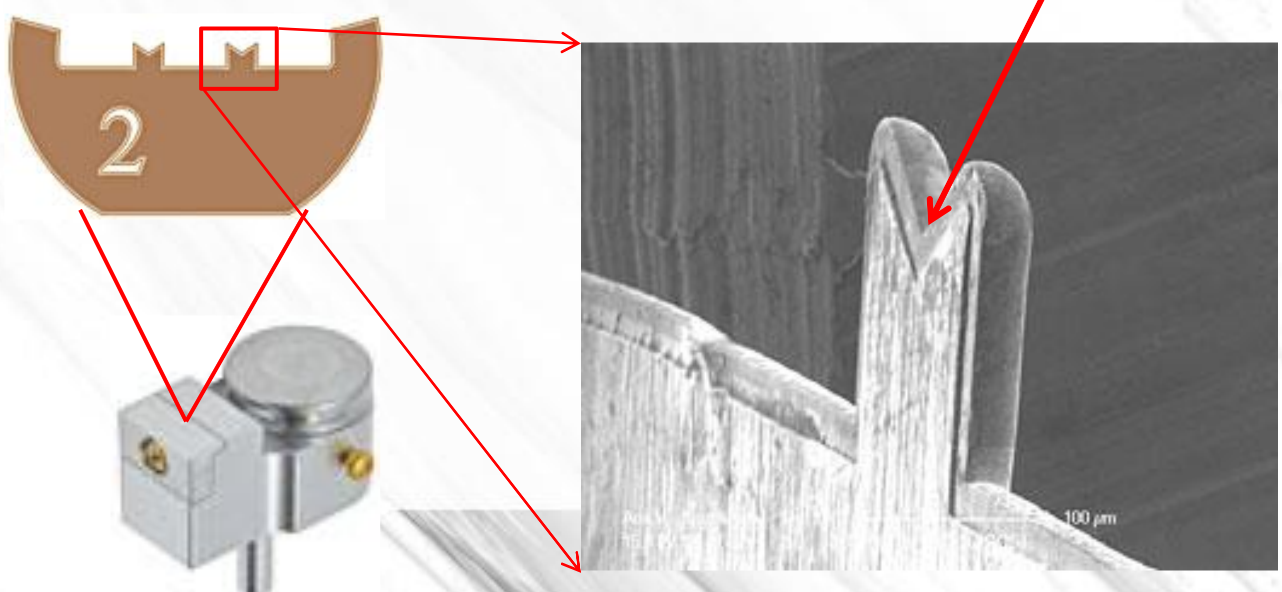

SEM
Microclamp
TEM Foils
EBSD Detector



For HR-TEM (Aberration Corrected (S)TEM) & EBSD Sample: Need Micromanipulator. For all your In situ studies.

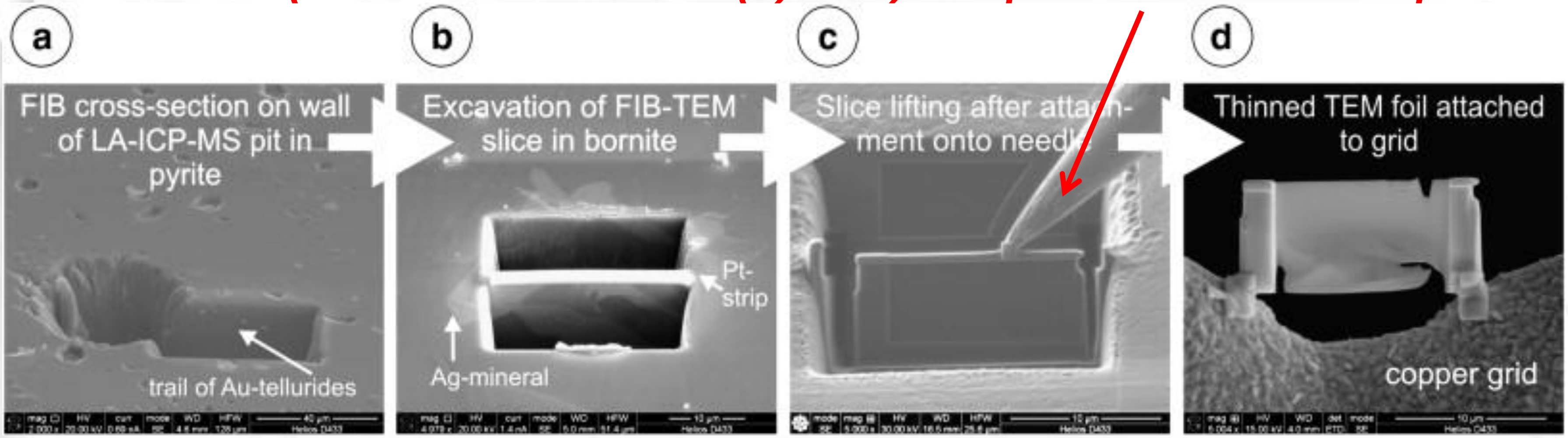


Samples can be further thinned on the TEM grid. Ideal for applications such as EELS or EBSD as there is no carbon film support.

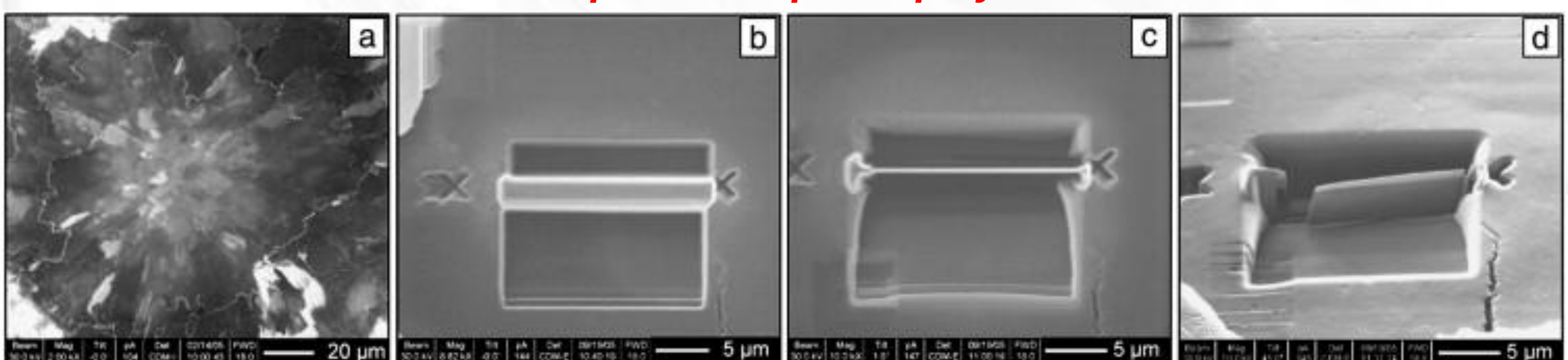


TEM Sample Preparation – Demo result
Need Micromanipulator and Low kV Final Polishing

For HR-TEM (Aberration Corrected (S)TEM) Sample: Need Micromanipulator

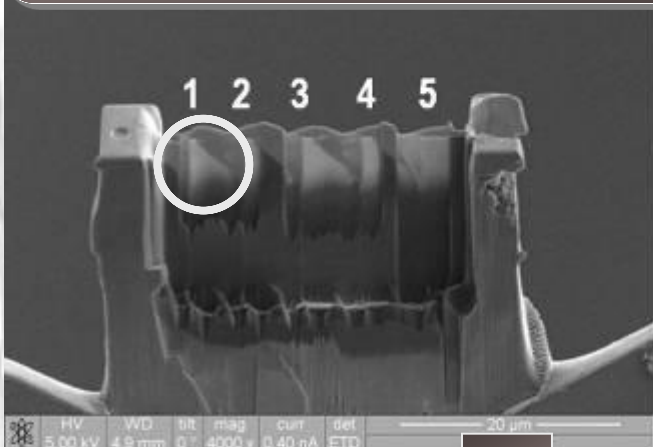


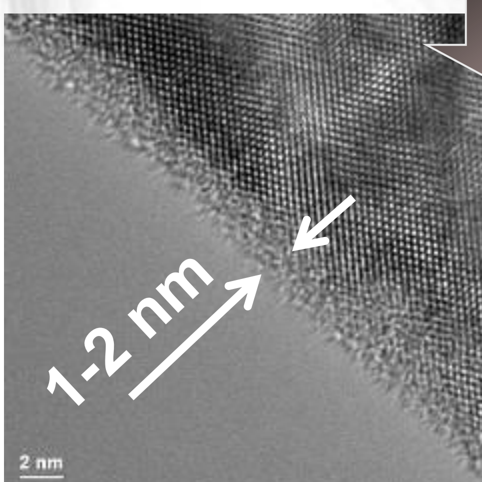
For Conventional TEM Sample: Need pick-up system



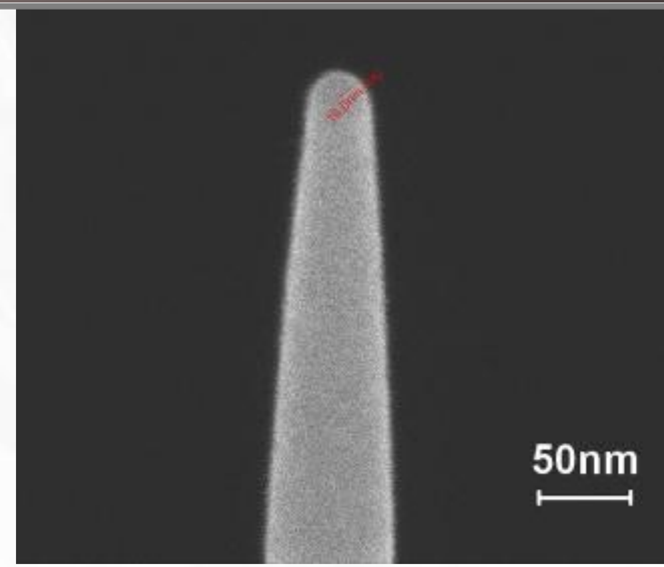
Thinner Sample Prep. With Ultimate Quality

FEI pioneers and leads the race in characterizing and delivering solutions for ultimate quality sample prep



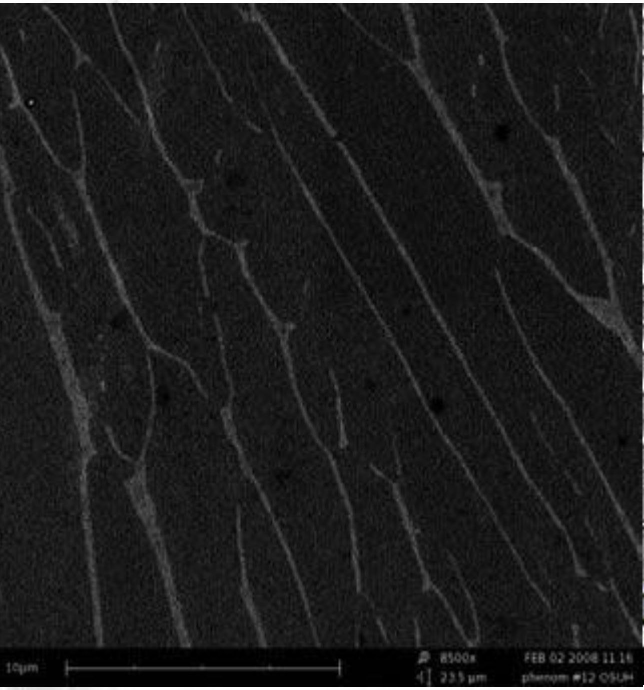


The thinnest samples can be achieved, like this atom probe tip with a final radius of 16 nm

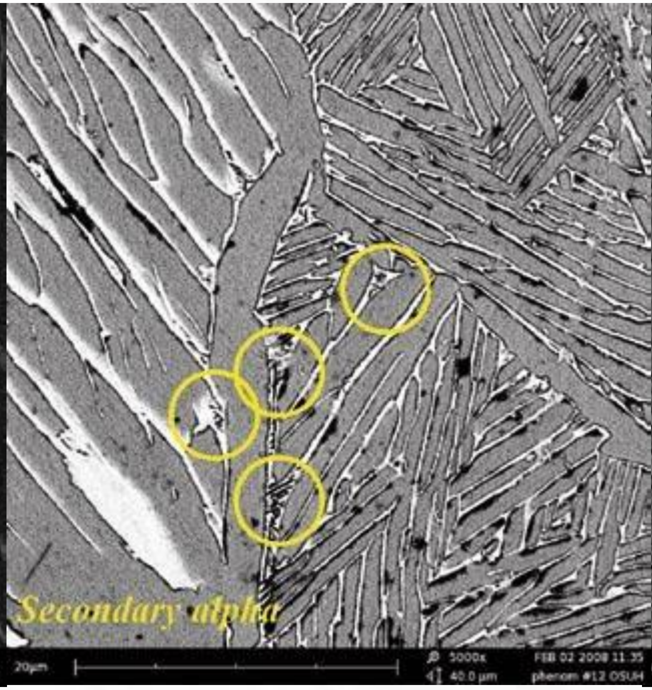


Very low kV FIB allows a 40 nm thick sample to keep over 90% of damage-free material, required for getting the best images from your HR corrected TEM

a.



b.



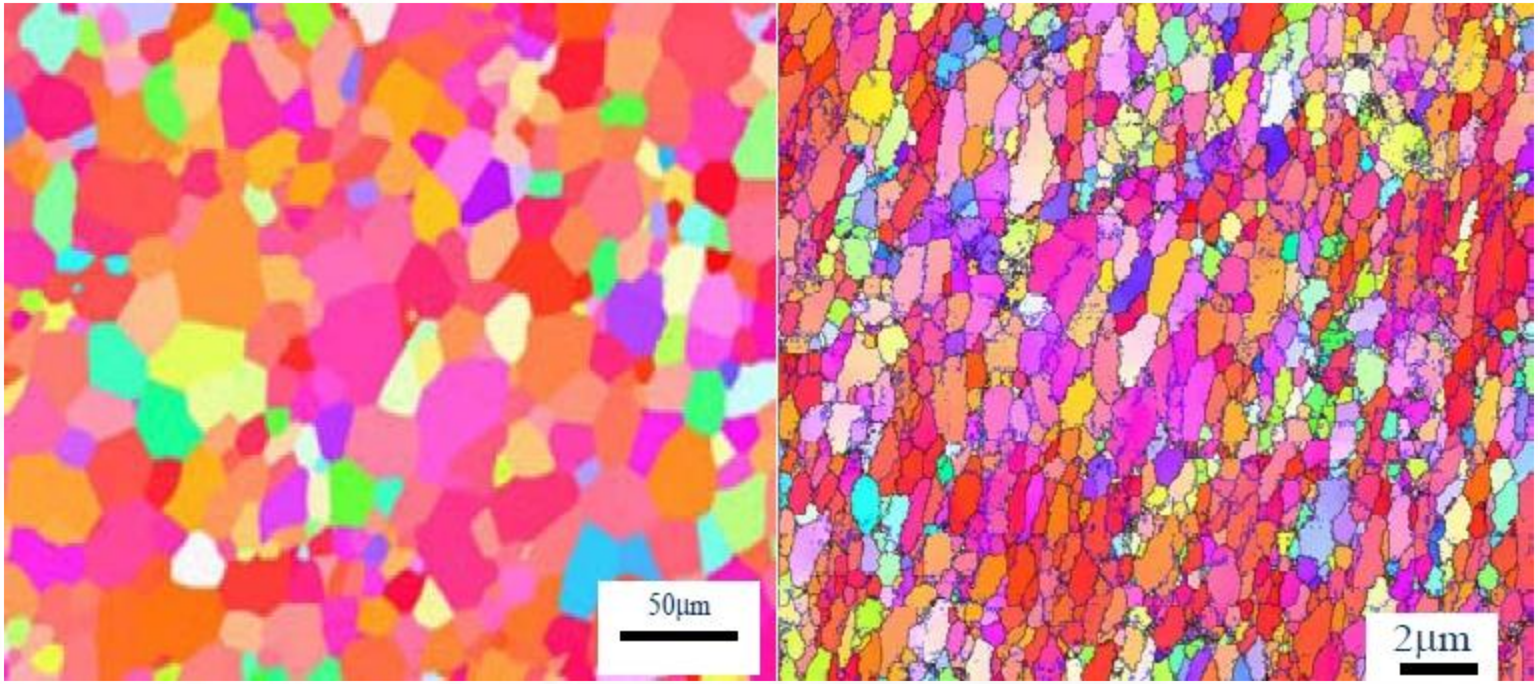
c.

Application for Titanium Alloy and Cast Aluminum Alloy:

a. Can show the α-laths and the β-laths between the laths of β-processed Ti-6-4. The average thickness of these laths is only about 50~100nm.

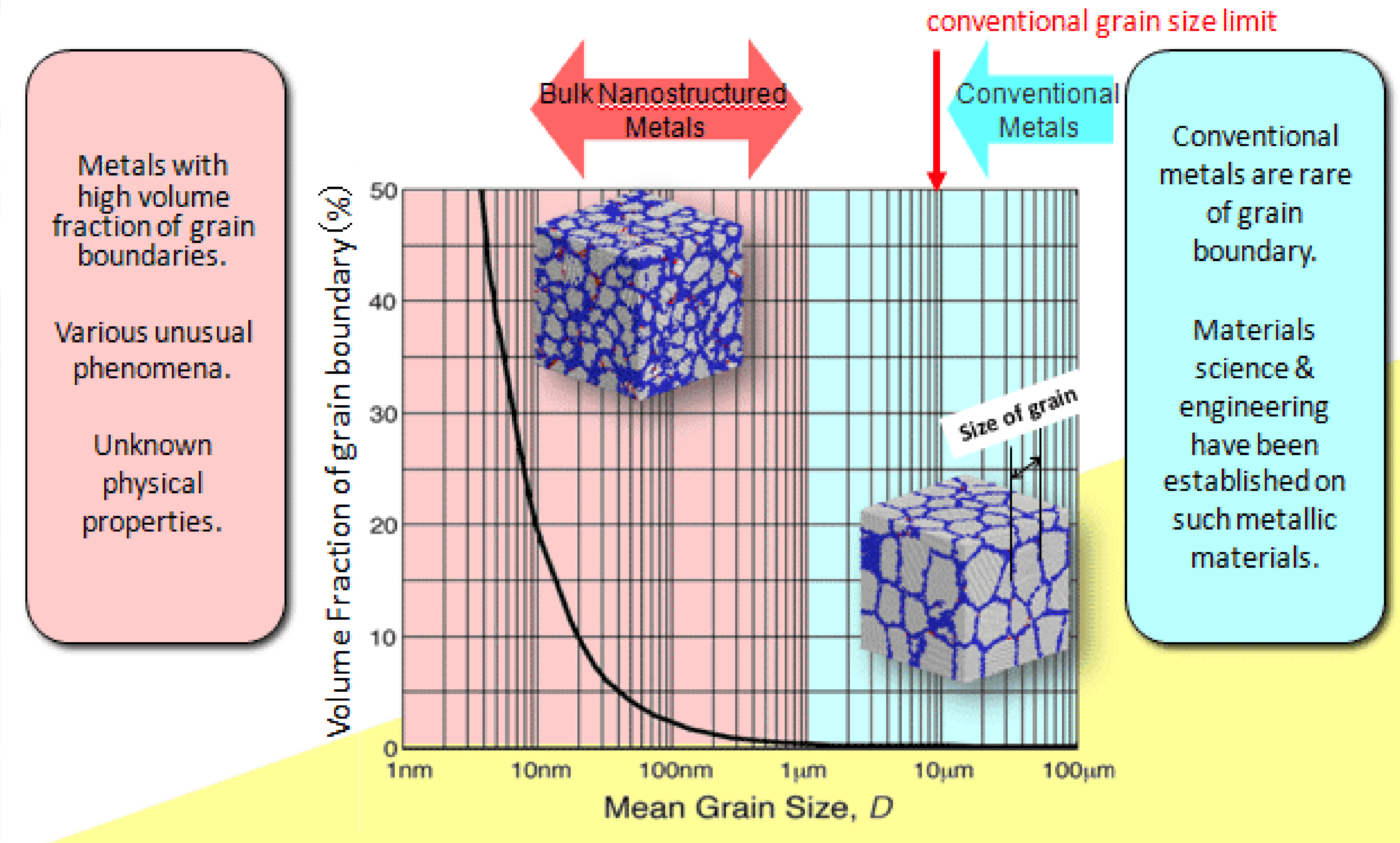
b. Can show the second nucleation of sub-micron size, this characteristic can often strengthen the alloy.

c. Can show the size and distribution of the intermetallic phases and shrinkage porosity.



Application for Titanium Alloy: Safer Titanium for prosthesis and implants.

- **Alloy involve risk, using 100% pure Titanium is one way to avoid this risk.**
- **We severely deform the metal so that its grain structure changes size from a scale of tens of micros to one of tens of nanometres.**
- **For high added-value application.**
- **Processed Titanium’s biocompatibility is better than pure Titanium’s.**



材料科學未來的研究方向 – 更小的晶粒尺寸，讓每個晶體被高密度的晶界牢牢束縛，機械性能超越傳統金屬，使鋁的強度達到傳統鋼材的水準，而且是具柔韌性的高強度，環保且可回收再製特性，顛覆傳統合金概念，簡單的化學組成，不需使用稀土元素，可使材料科學發展進入新的境界。