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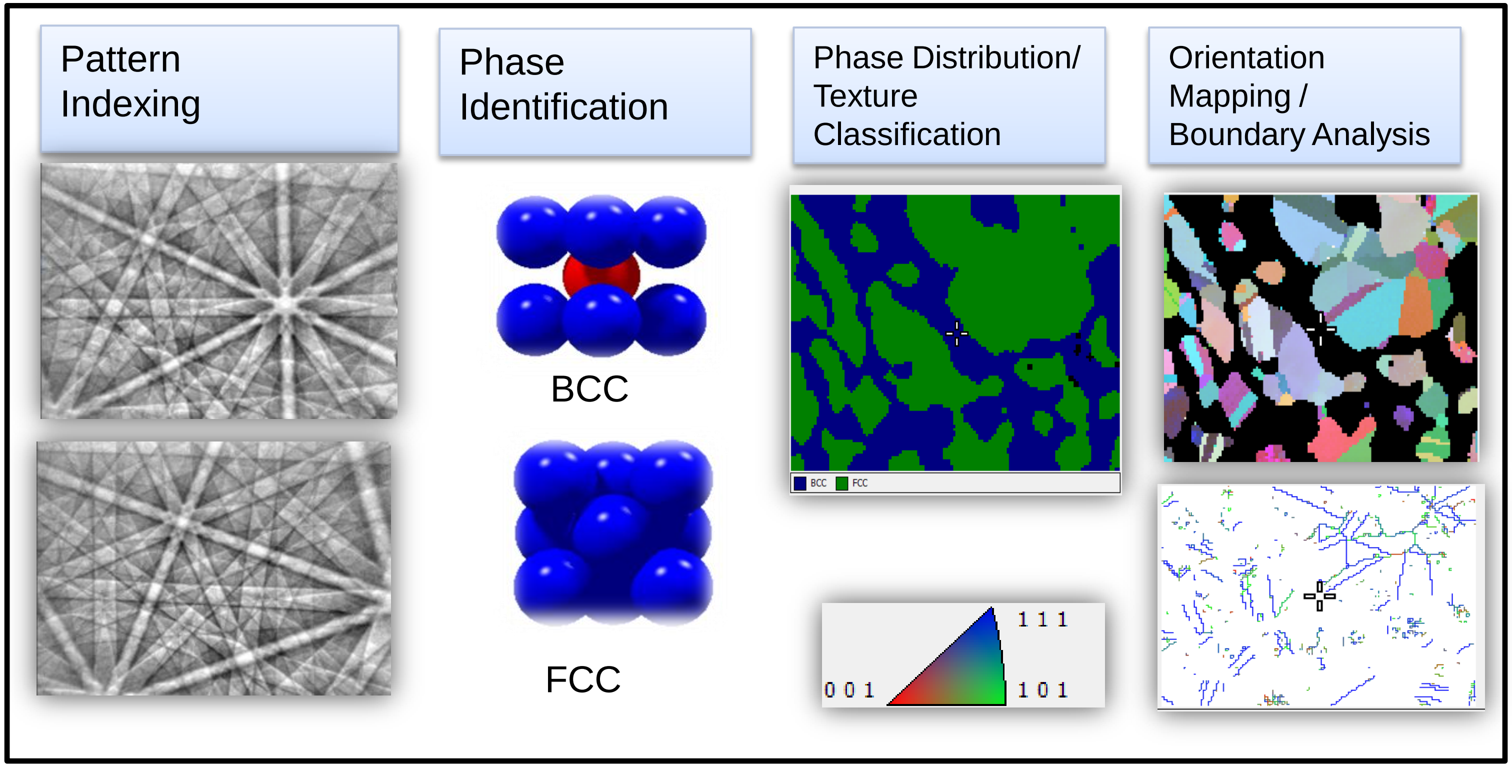
# MAGCIS – A Novel Method for EBSD Surface Preparation

# Webinar Outline

- Introduction
- Review of EBSD preparation techniques
- Introducing MAGCIS for EBSD
- Application Examples

# EBSD Introduction

- EBSD is used in a wide number of disciplines
- Traditionally used in metallurgical applications, now many new areas
- Vast amount of information from a single dataset



# EBSD Comparisons

EBSD offers many advantages over other structural characterisation techniques

vs TEM

- Analysis of bulk samples
- Statistically more reliable
- Simplified preparation routines

vs XRD

- Greater spatial resolution
- Relation between microstructure and texture

vs Optical Microscopy

- Improved resolution and higher magnification
- More comprehensive information, readily combined with other techniques

# EBSD Operating Parameters

Microscope Parameters:

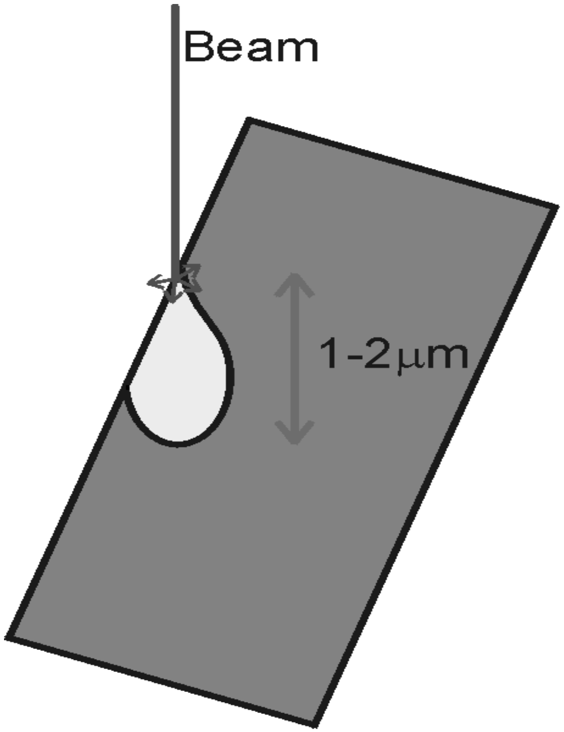
- Working Distance - <5 mm - >40 mm
- Probe current - <2 nA
- Accelerating voltage <5 kV  - >30 kV

EBSD System Parameters:

- Spatial resolution - <20 nm
- Angular resolution - <0.5°
- Indexing speed - >500 pts/sec
- Symmetry – Triclinic and above

# EBSD Sample Preparation

- Good surface preparation is essential
  - Diffraction pattern signal originates from the top few nm of the surface
  - Damage-free crystal lattice
  - Residual deformation
  - High tilt angle can result in shadowing
- Choose the most appropriate technique for your material
- Many preparation techniques actually impair EBSP pattern quality



# Common EBSD Sample Preparation Techniques

- A range of sample preparation techniques are commonly used in EBSD

Sectioning → Mounting → Grinding →

**Mechanical Polishing**

Finishing to colloidal silica

Flexible in applications (Geological, ceramics, soft…)

Time consuming

**Ion Polishing**

Ionized inert gas accelerated towards sample

Excellent spatial resolution

Can deform crystal Lattice

**Chemical Etching**

Removal by chemical action

Removes deformation caused by surface energy of deformed lattice

Can induce high surface topography

**Electro Polishing**

Removal of surface material by electrolytic action

Bright finish at surface

Difficult to reproduce

# Introducting MAGCIS

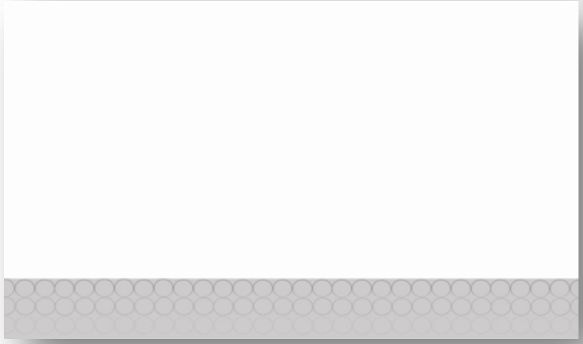
- Monatomic And Gas Cluster Ion Source (MAGCIS)
- Originally developed for XPS market
- Single source for monatomic and cluster beams

<u>Monatomic mode</u>

- Comparable to standard ion polishing
- 500eV-4keV with high beam currents

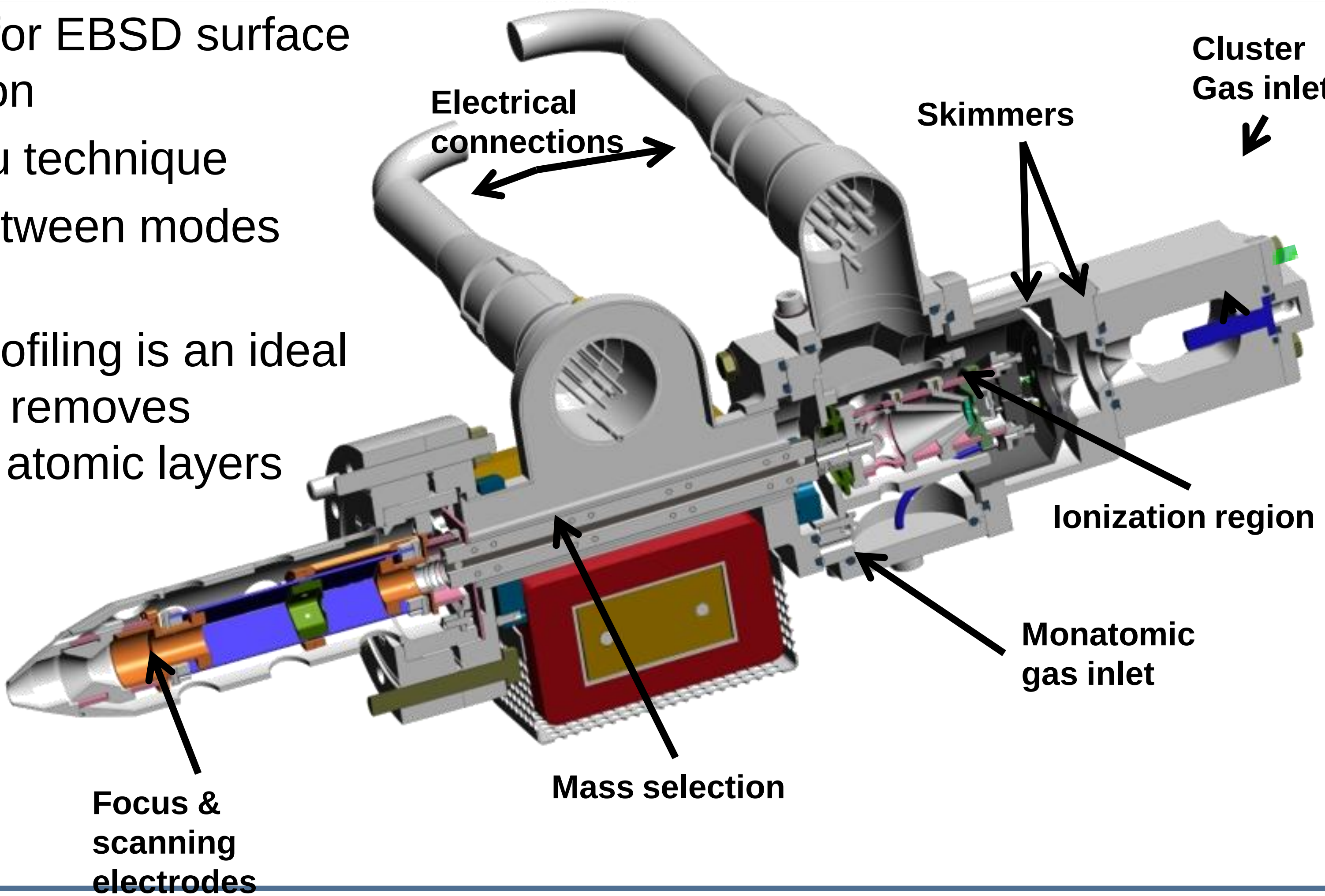
<u>Cluster mode</u>

- Variable cluster sizes (0<n<2000 atoms)
- Energy/atom: 1eV upwards
- Extra low beam energies possible (1000eV & 2000eV modes)

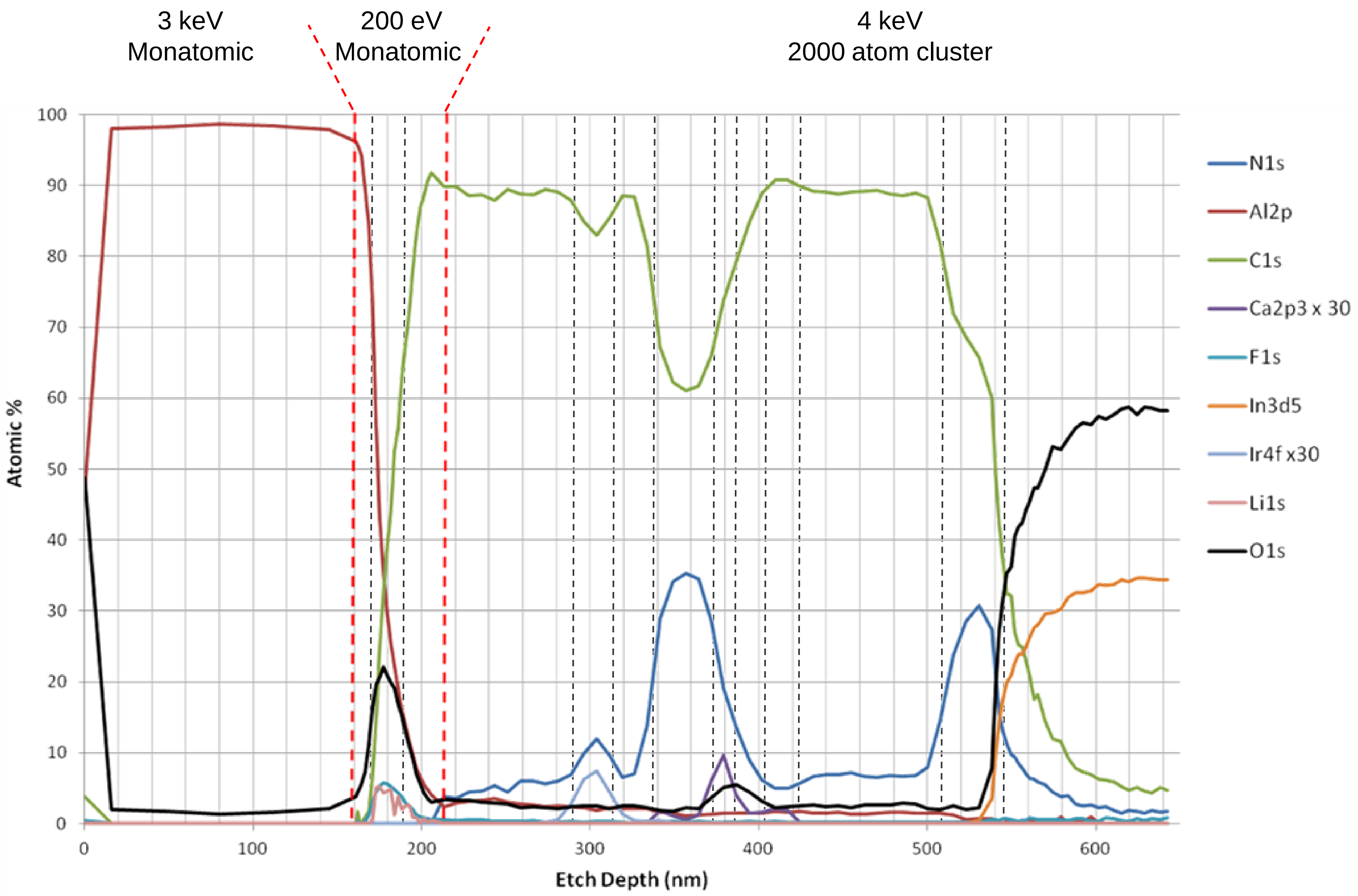


# MAGCIS Advantages

- MAGCIS offers excellent potential for EBSD surface preparation
- Full in-situ technique
- Switch between modes instantly
- Cluster profiling is an ideal final step, removes individual atomic layers



# Elemental Profile



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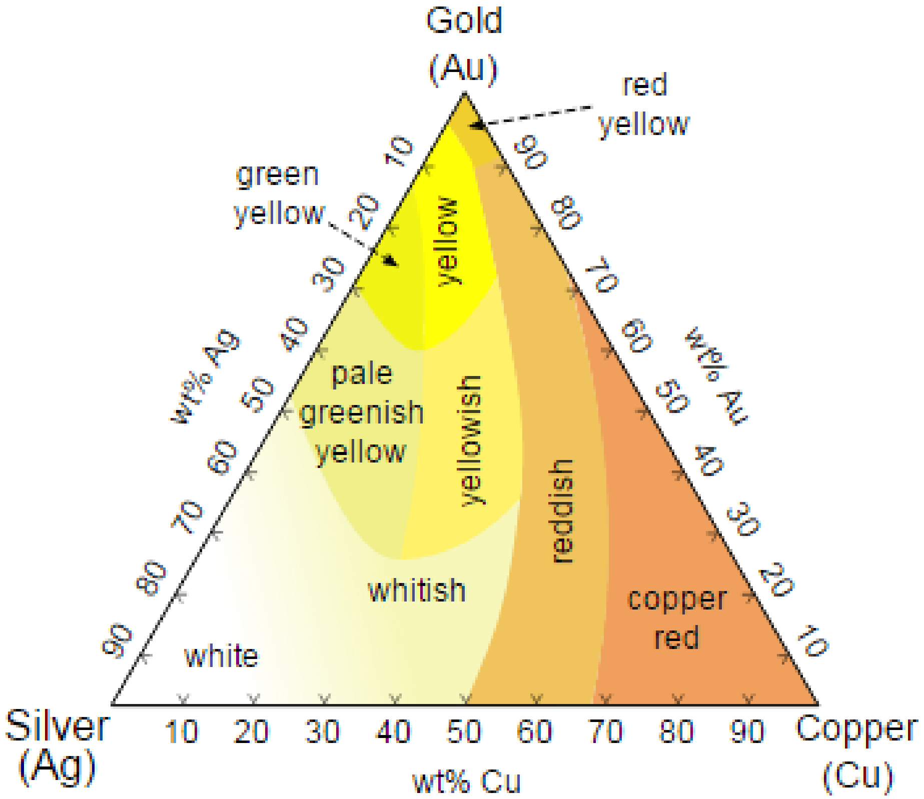
# Application 1: Au Alloy

- ‘Red Gold’ is a gold and copper alloy widely used in industry.
- The microstructure of the material is related to the alloy composition and production conditions
- Gold alloys are usually soft making them difficult to prepare for EBSD analysis

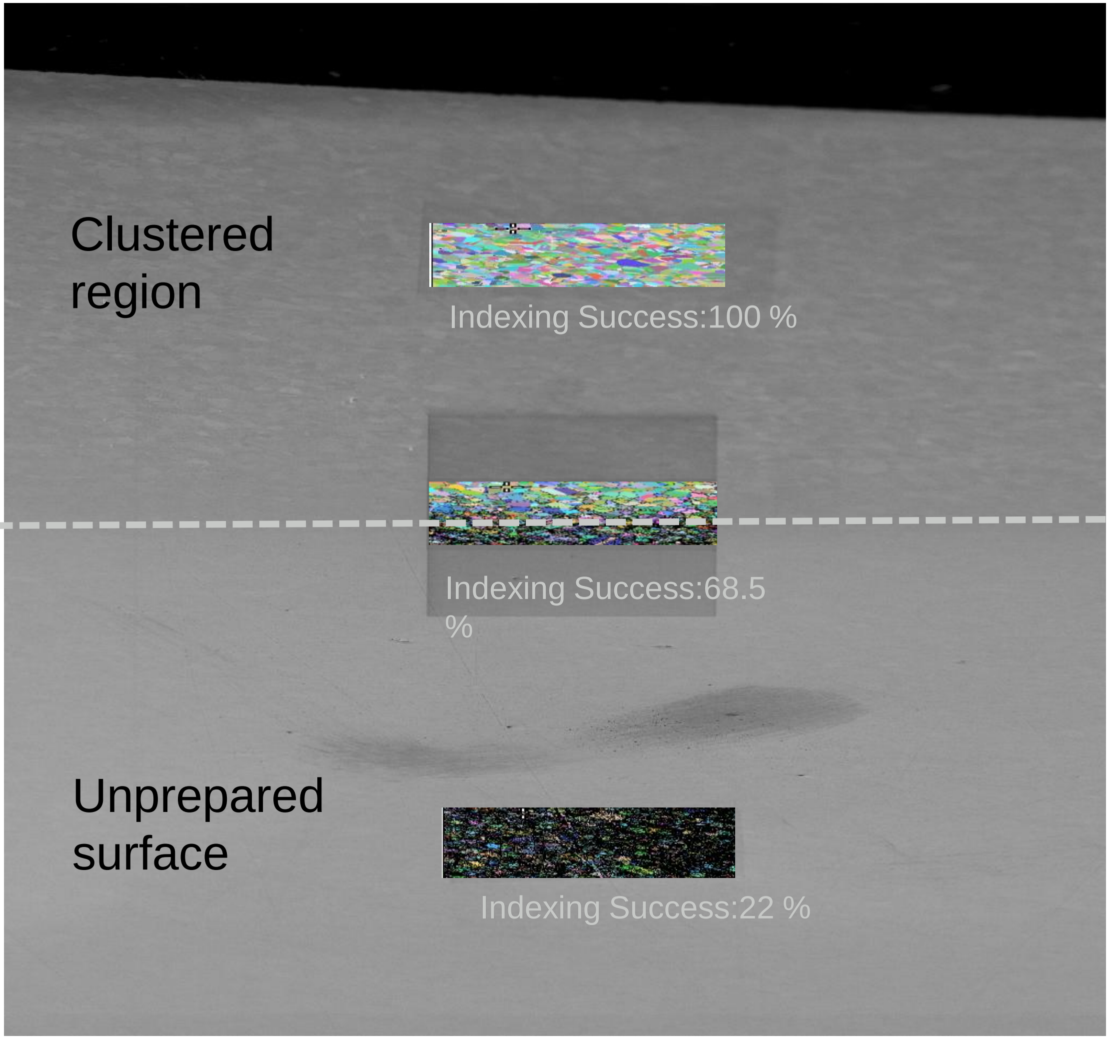


# Application 1: Sample Preparation

- Sample: cold treated Au:Cu alloy – FCC structure –
- Polished using colloidal Si suspension
- Initially polished with, following a standard recipe for gold, this yielded a very low overall indexing quality <10%
- Prepared using cluster ion source on, 20 minutes at 3 kV Monoatomic, 2 minutes cluster profile as a final step
- Milling area 1 x 2 mm

# Application 1: Sample Overview

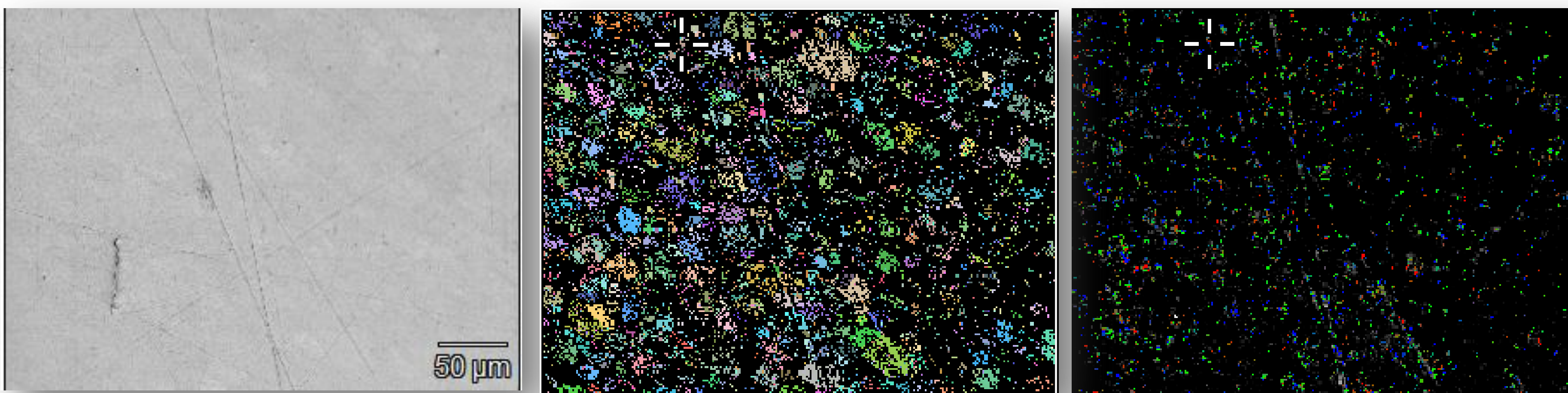
- Differences between milled and unmillled areas.
- 3 regions selected from sample, milled, unprepared and transition
- EBSD data acquired under same conditions for each region



# Application 1: Unprepared Region

- Regions prepared using traditional polishing techniques have low quality Kikuchi patterns
- Deformation over several levels
- Longer exposure times required using this approach

| | |
|---|---|
| Image Resolution: | 1024 by 768 |
| Image Pixel Size: | 0.33 µm |
| Acc. Voltage: | 20.0 kV |
| Magnification: | 400 |
| Exposure Time: | 9.0 ms |
| Indexing Success: | 22 % |

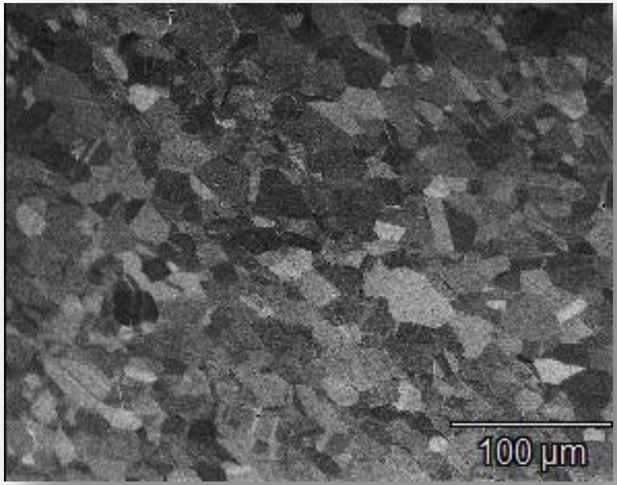


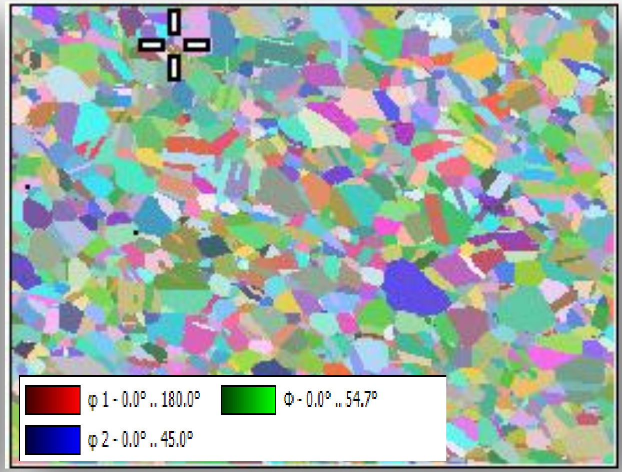

***Left** SEI image of mechanically polished surfaces. **Centre** Orientation image map showing a low overall indexing sucess **Right** Grain boundary map, which does not return any meaningful information*

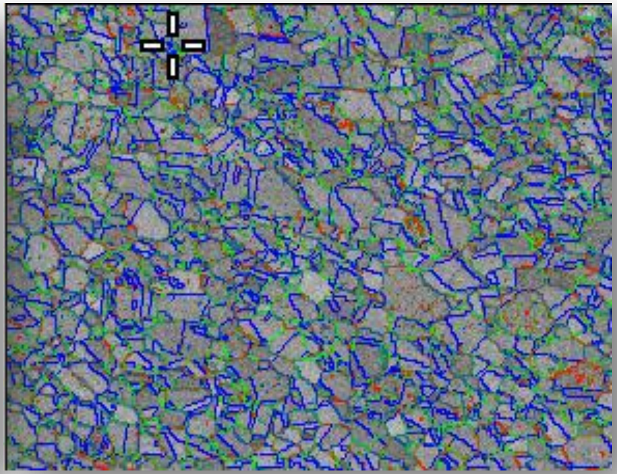
# Application 1: MAGCIS Cluster Region

- Regions prepared using MAGCIS cluster polishing have high quality Kikuchi patterns
- Indexing success of 100%Enables rapid analysis of large areas

| | |
|---|---|
| Image Resolution: | 1024 by 768 |
| Image Pixel Size: | 0.38 µm |
| Acc. Voltage: | 20.0 kV |
| Magnification: | 400x |
| Exposure Time: | 9.0 ms |
| Indexing Success: | **100.0 %** |





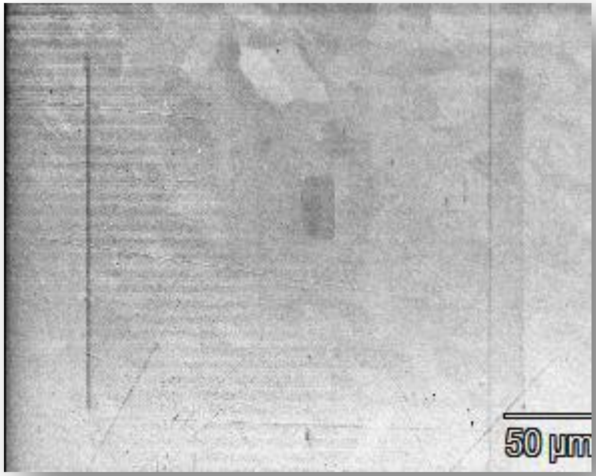


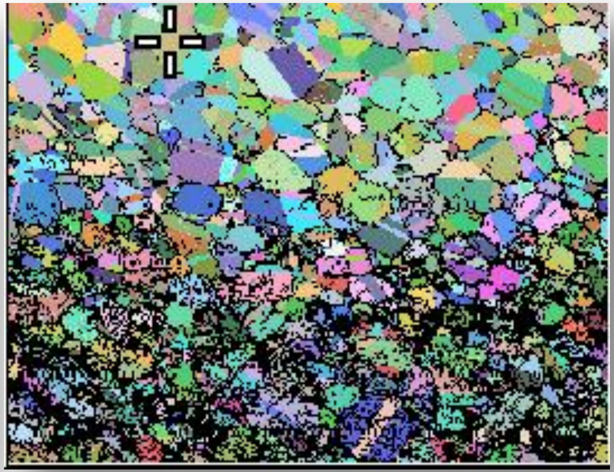
***Left*** *SEI image of MAGCIS polished surfaces.* ***Centre*** *Orientation image map showing a high overall indexing success* ***Right*** *Grain boundary angle map, showing a preferred microtexture*

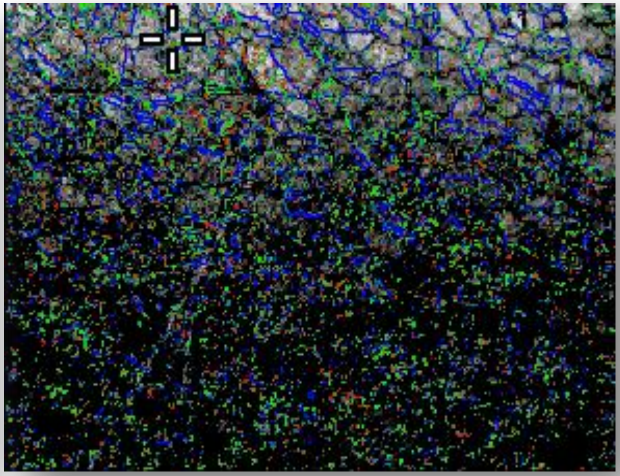
# Application 1: Transition Region

- EBSD analysis of a transition region
- Partial indexing success close to interface
- Large grains successfully indexed

| | |
|---|---|
| Image Resolution: | 1024 by 768 |
| Image Pixel Size: | 0.33 µm |
| Acc. Voltage: | 20.0 kV |
| Magnification: | 405 |
| Working Dist: | 14.9 mm |
| Exposure Time: | 9.0 ms |
| Indexing Success: | 68.5 % |



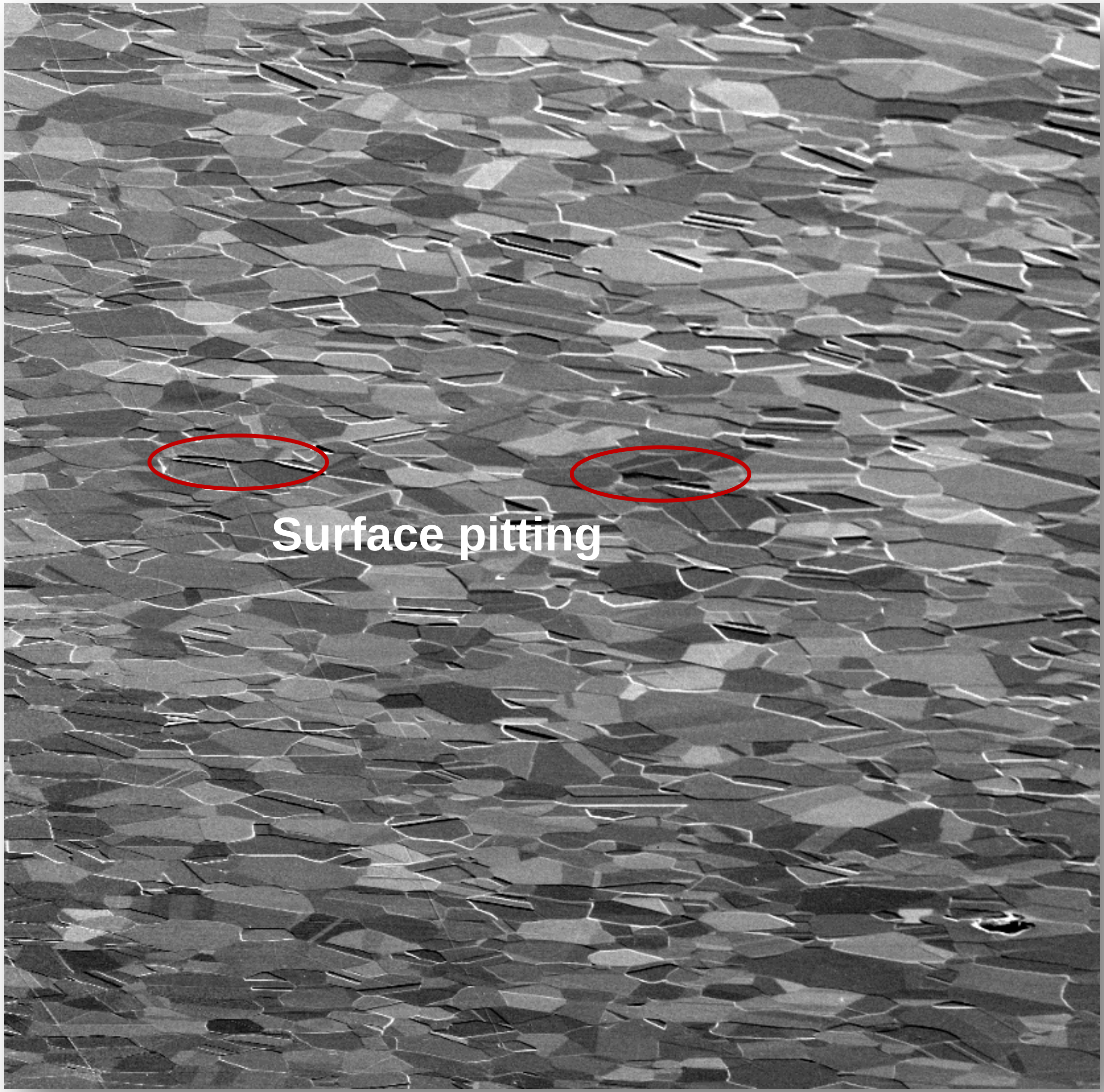





***Left*** *SEI image of transition region between MAGCIS and non-MAGCIS regions .* ***Centre*** *Orientation image map showing a low overall indexing success* ***Right*** *Grain boundary map, which does not return any meaningful information*

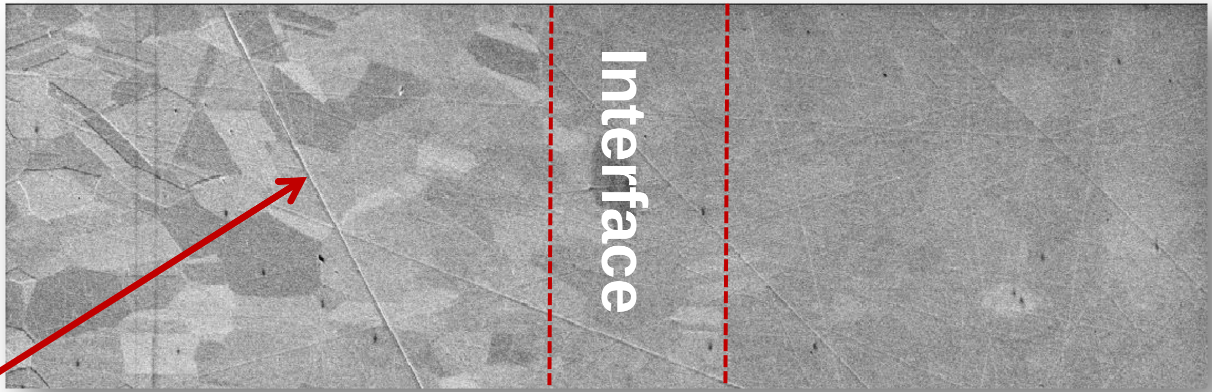
# Application 1: Surface Topography

- Induced topography is related to beam energy
- 3 kV milling for 30 minutes results in some preferential etching of individual grains
- Less apparent at lower beam energy
- Results in shadowing and reduced EBSP quality

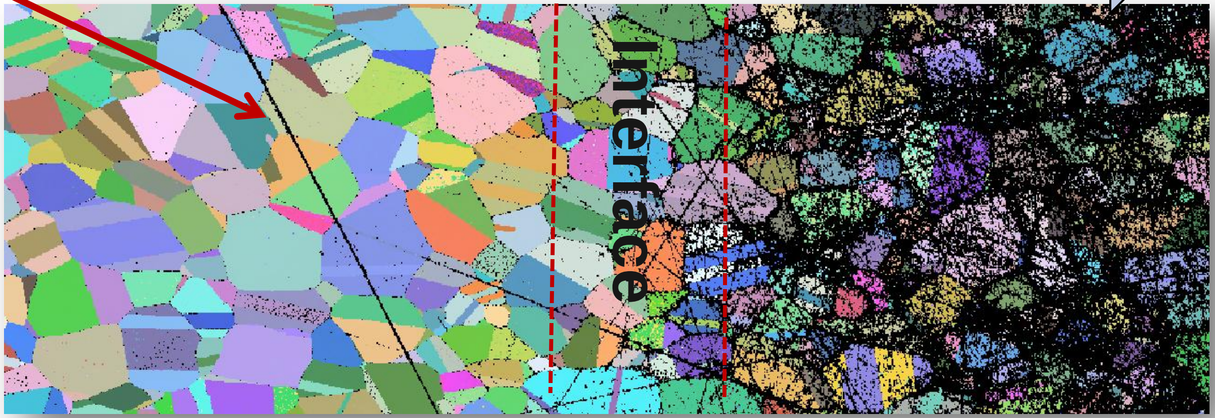


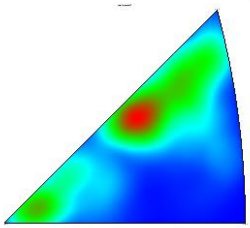
# Application 1: Large Area Overview

- Lower kV (<2) MAGCIS induces smaller topographical effect
- Individual artefacts will remain
- Variable milling




Large feature remains





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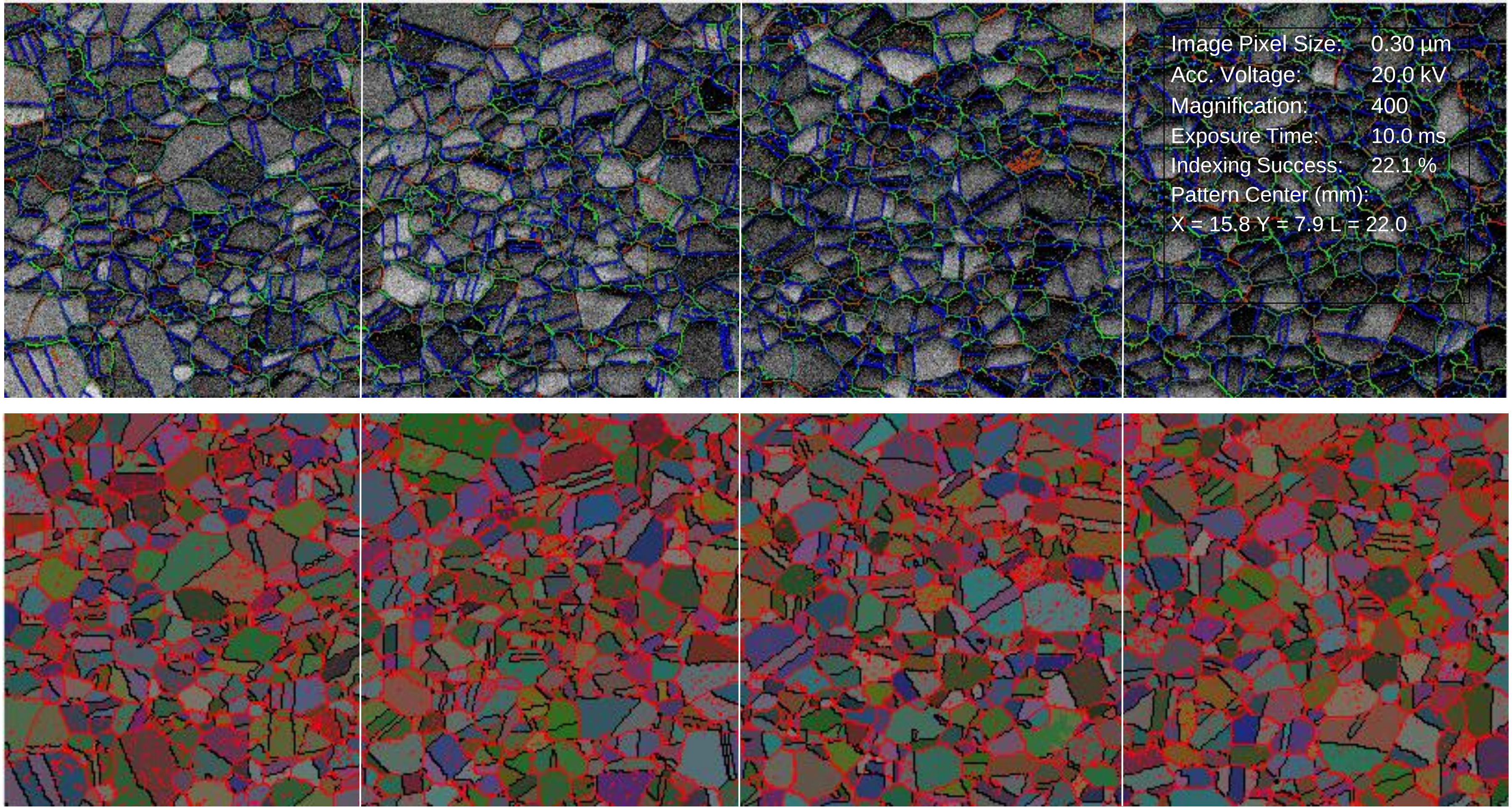
# Application 1: Large Area Mapping

- High resolution mapping shows twinning present within many grains
- No image enhancement required

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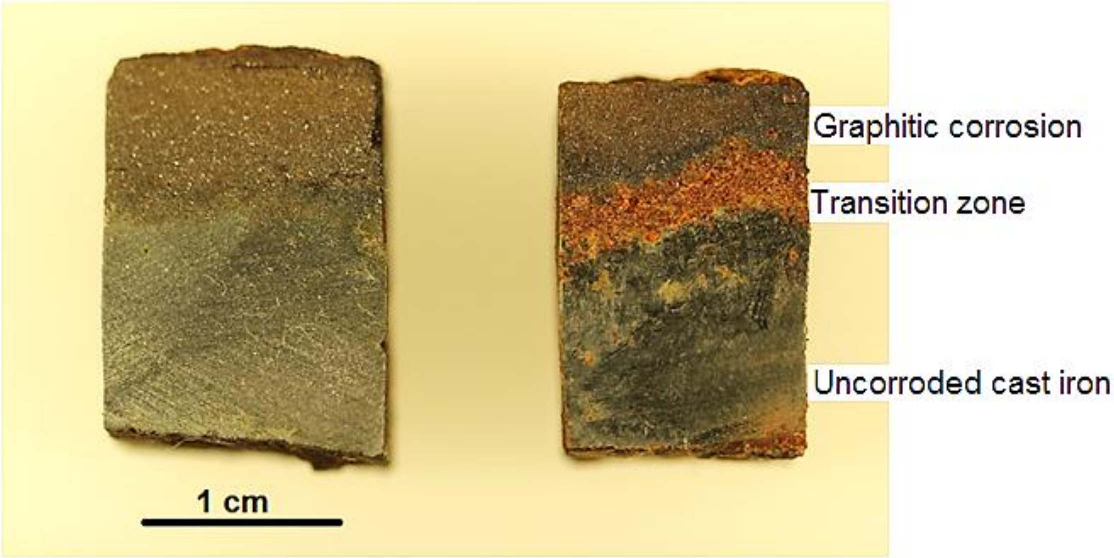
# Application 1: Summary

- MAGCIS is an excellent tool for the preparation of soft materials such as gold alloys
- Large areas can be prepared for analysis very quickly, with little structural damage to the material
- Successfully indexed a fine small grain gold alloy

# Application 2: Background

- Cast irons are ferrous irons consisting of 2-4% carbon, 1% Silicon and ~1% Phosphorous
- Distribution of regions can be seen using optical microscopy
- Multiple hard and soft phases, a challenge for traditional sample polishing

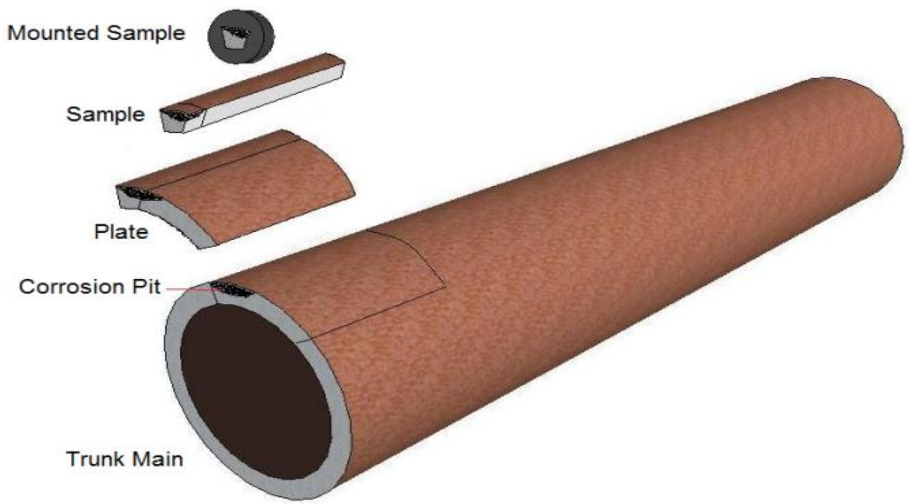


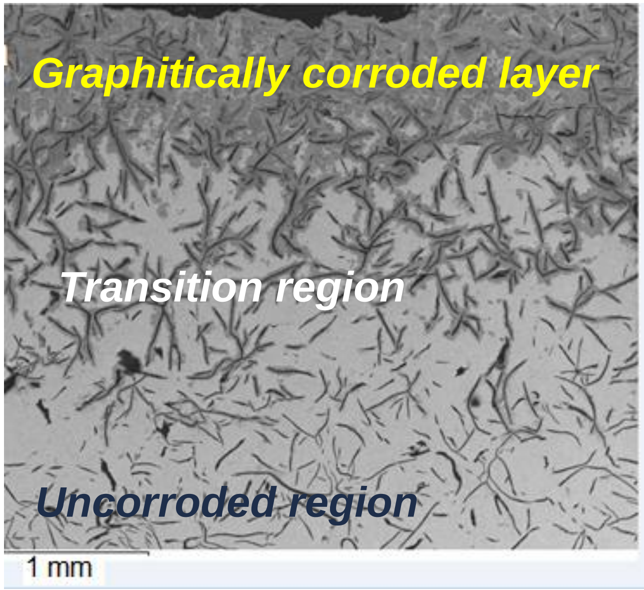

*Optical microscopy image of corrosion regions.*

*R. Logan University of Surrey, UK*

# Application 2: Sample Preparation

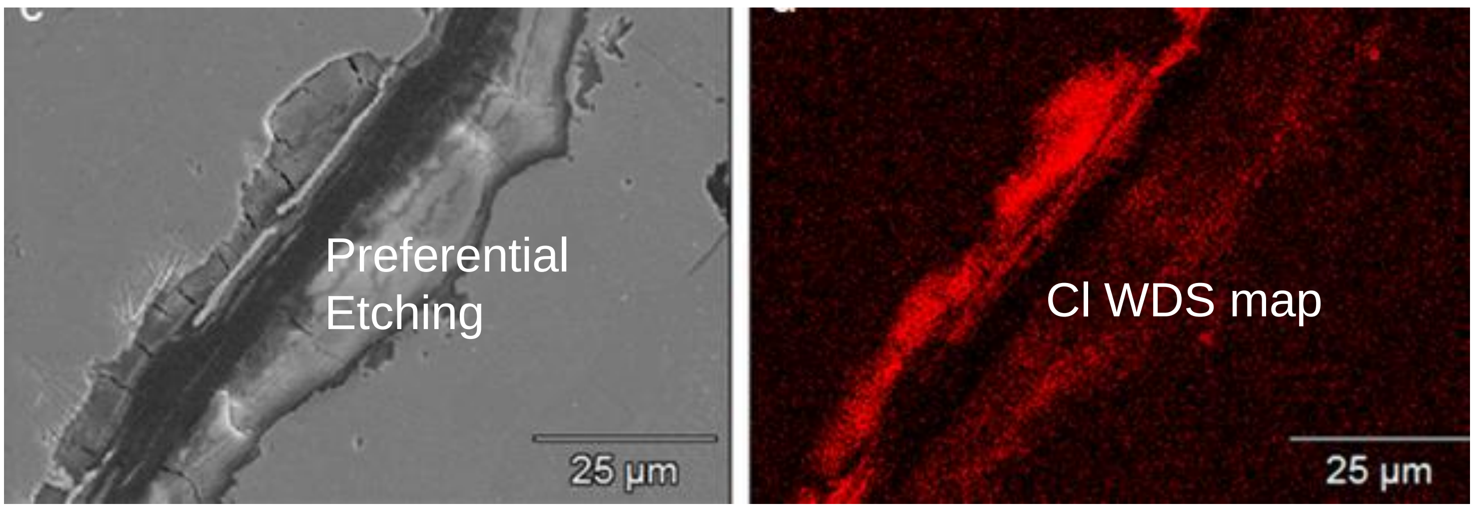
- Sample mounted in Bakelite relative to the 'normal' axis
- Mechanical polish down to colloidal silica used for SEM, EDS and WDS analysis



# WDS Characterisation

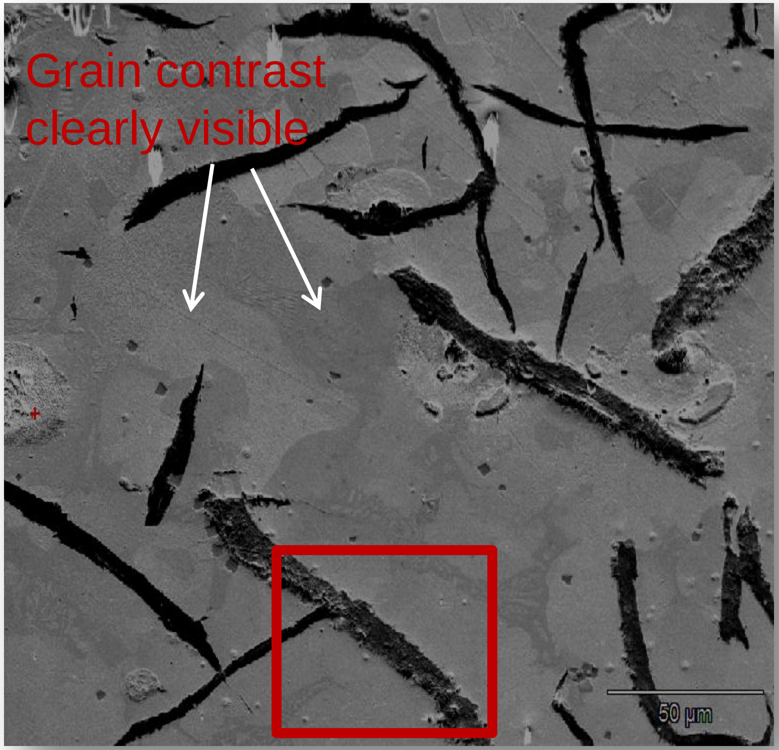
- WDS suggests trace Chlorine present in graphitic corrosion region
- No chloride detectable in uncorroded regions, therefore soil electrolyte concentration affects rate of corrosion
- Surfaces prepared in this way are too uneven to give complimentary EBSD structural information. No EBSPs observed

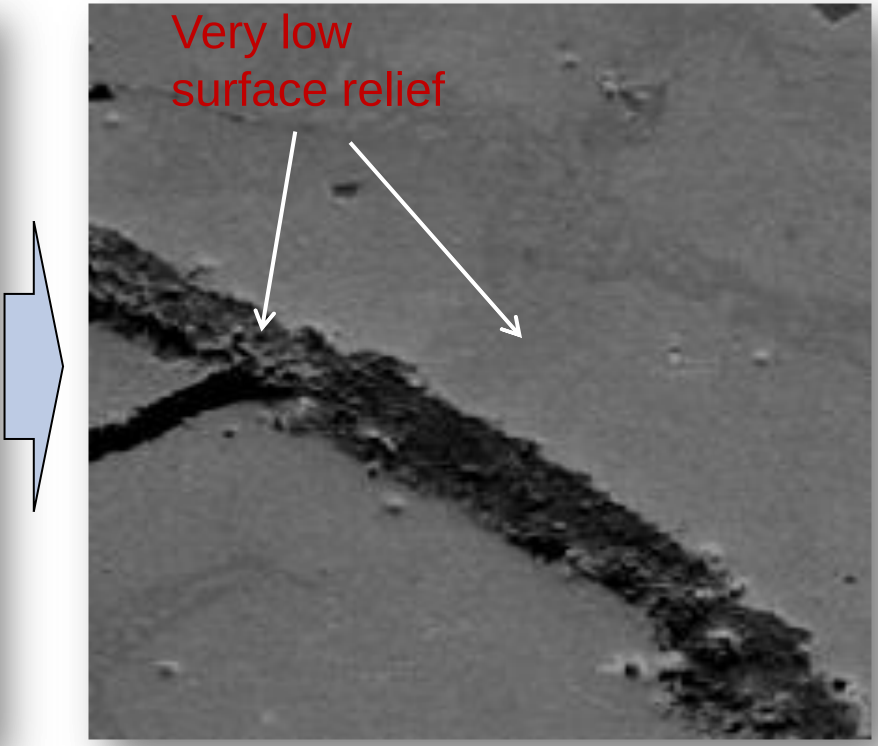



***left** SEM showing preferential etching, **right** WDS scan showing trace >1%*

# SEM Analysis of MAGCIS Sample Milling

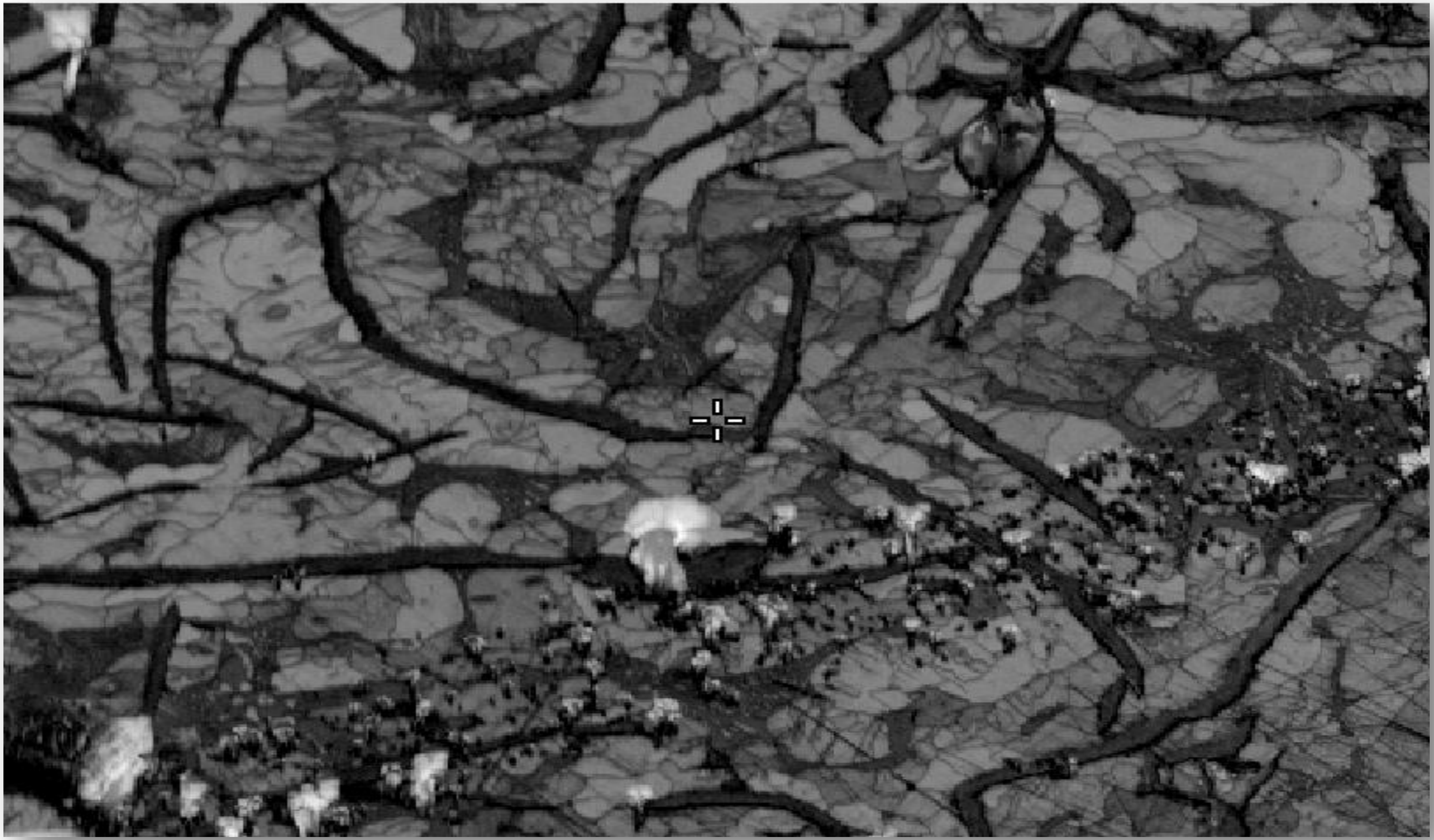
- Samples etched using MAGCIS for 30 minutes at 3 kV monatomic, followed by 10 minutes at 1 kV cluster
- Low topographical differences between iron and carbon phases



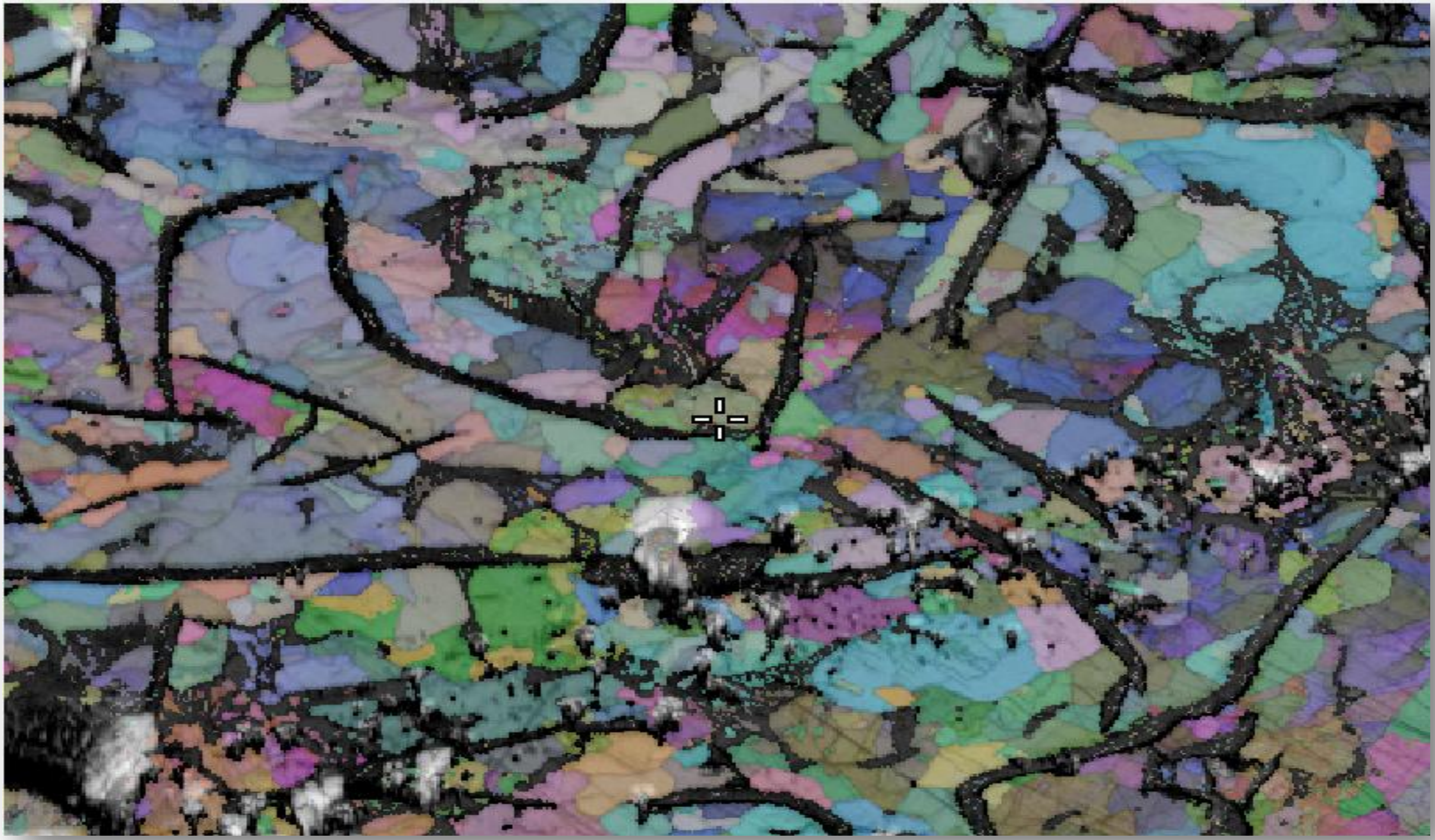
# EBSD Pattern Quality Map

- Clearly shows grain boundaries and microstructure of ferrite phases



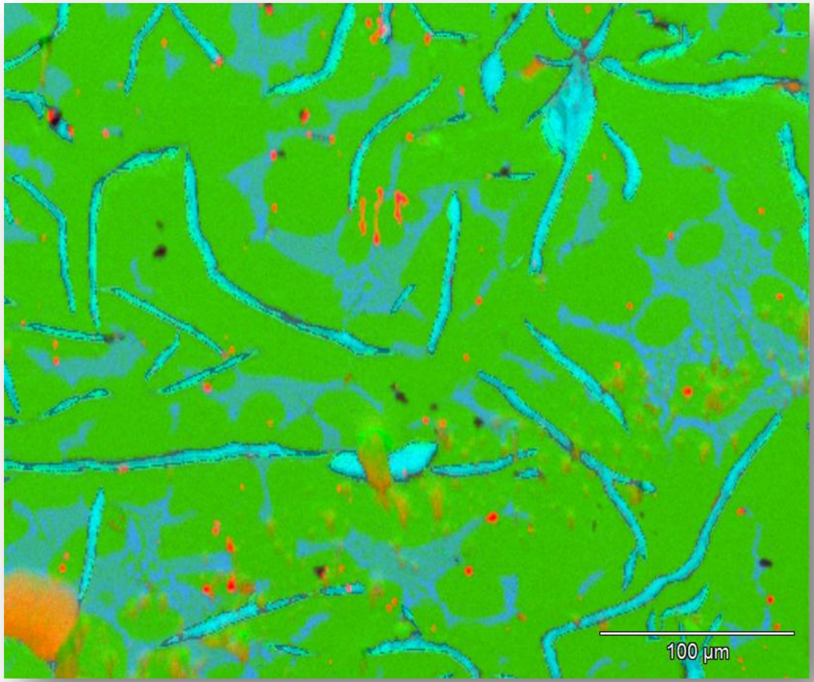
# Euler Map

- Overlaid Euler map for texture classification of Ferrite phases

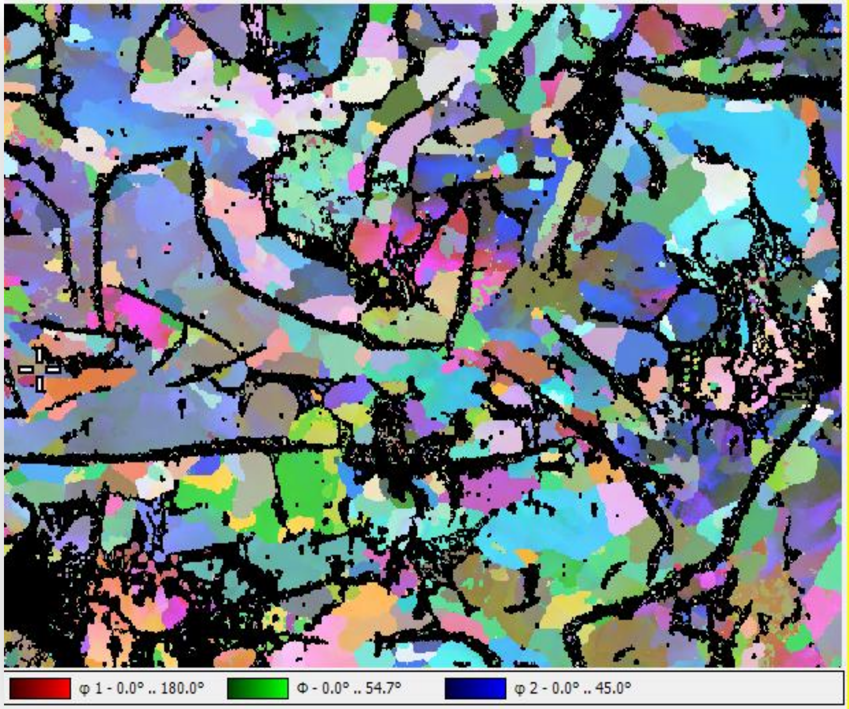


# EDS/EBSD Comparisons

- Concurrent EDS analysis suggest that Ferrite phases and corrosion regions are currently indexed
- Carbon rich phases do not yield EBSPs




Fe C P O



# Summary: MAGCIS in EBSD

- MAGCIS enables the characterisation of complex multiphase materials using EBSD
- Induces reduced mechanical damage when compared to conventional techniques
- An excellent *in-situ* preparation technique with further development opportunities