Introduction to EBSD

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EBSD

EBSD = Electron Back Scatter Diffraction which allows:

- Crystallographic Orientations
- Misorientations
- Texture measurement
- Grain size and boundary types
- Phases

To be characterised and quantified on a macro to sub-micron or nano-meter scale

What does EBSD Contribute to Materials Analysis?

- **EDS/WDS** - **Chemical analysis**
  - i.e. **What the sample is made from**
- **EBSD** - **Microstructural analysis**
  - Polycrystalline materials
    - Grain Structure - size/distribution, ASTM number etc.
    - Grain Boundary Characteristics
    - Macro & Micro-Crystallographic Texture
    - Phase Discrimination and distribution
    - Deformation
  - Single Crystal materials/deposited layers
    - Crystal orientation
    - Epitaxy between layers
  - i.e. **How the sample is put together and what condition it is in**

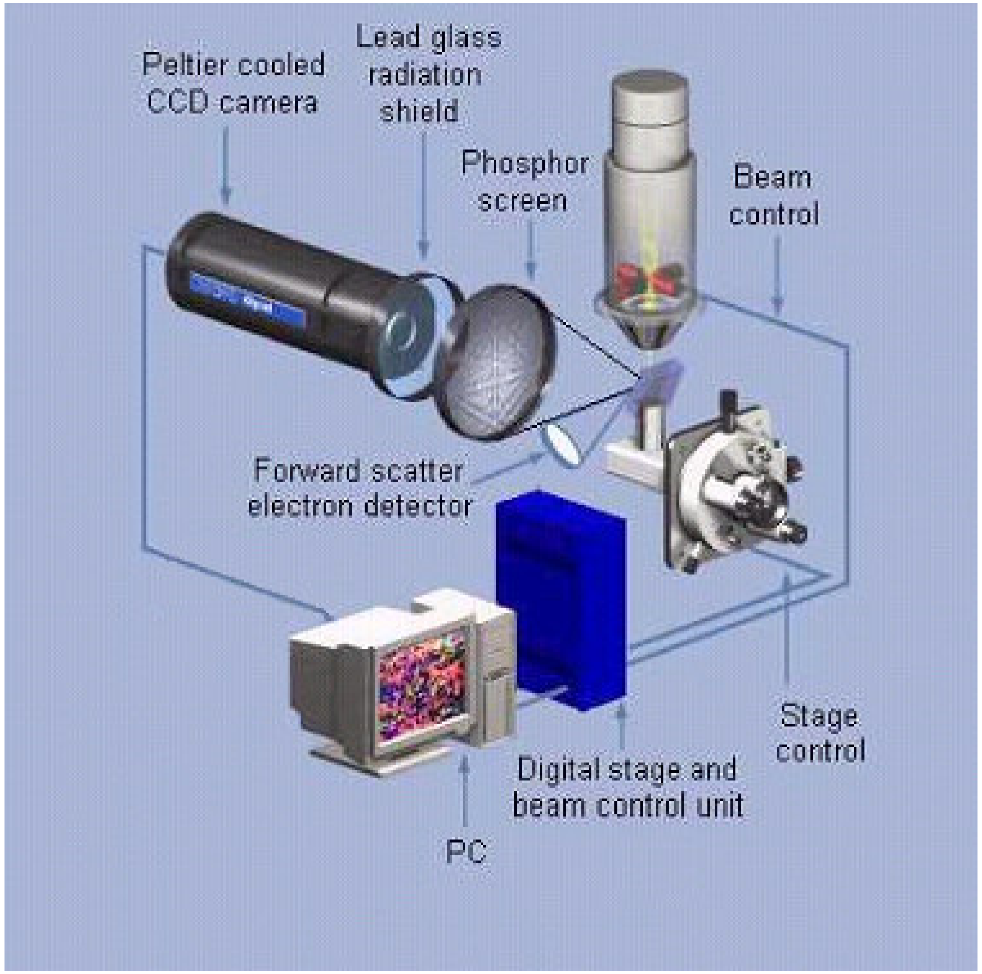
Typical Applications

- Metals
  - Metal production i.e. sheet metal, castings, forgings - automotive, aerospace, power generation and distribution, petrochemical and chemical plant, nuclear i.e. extreme duty materials - high strength, high temperature and corrosive environments. Electronics.
  - Phase identification and discrimination
  - Texture analysis, grain boundary characterisation
  - Deformation
- Geological
  - Phase identification and discrimination
  - Orientation & texture analysis
- Ceramics
  - Phase identification and discrimination
  - Orientation & texture analysis

Typical Areas of Investigation

- Production and process control
  - Cost savings - reduce heat treatment times, optimise heat treatment and phase transformation, investigate grain size, grain boundary and texture evolution
  - Improved product i.e. surface finish, forming or joining characteristics, process optimisation & control
  - Control physical properties - improve or achieve specific properties i.e. high corrosion resistance, fatigue or cracking (stress corrosion cracking) resistance.
- Component life-time prediction
  - Characterise microstructure - identify problem phases or microstructural feature
- Failure Analysis
  - Investigate micrstructural features associated with failure and propose failure mechanism

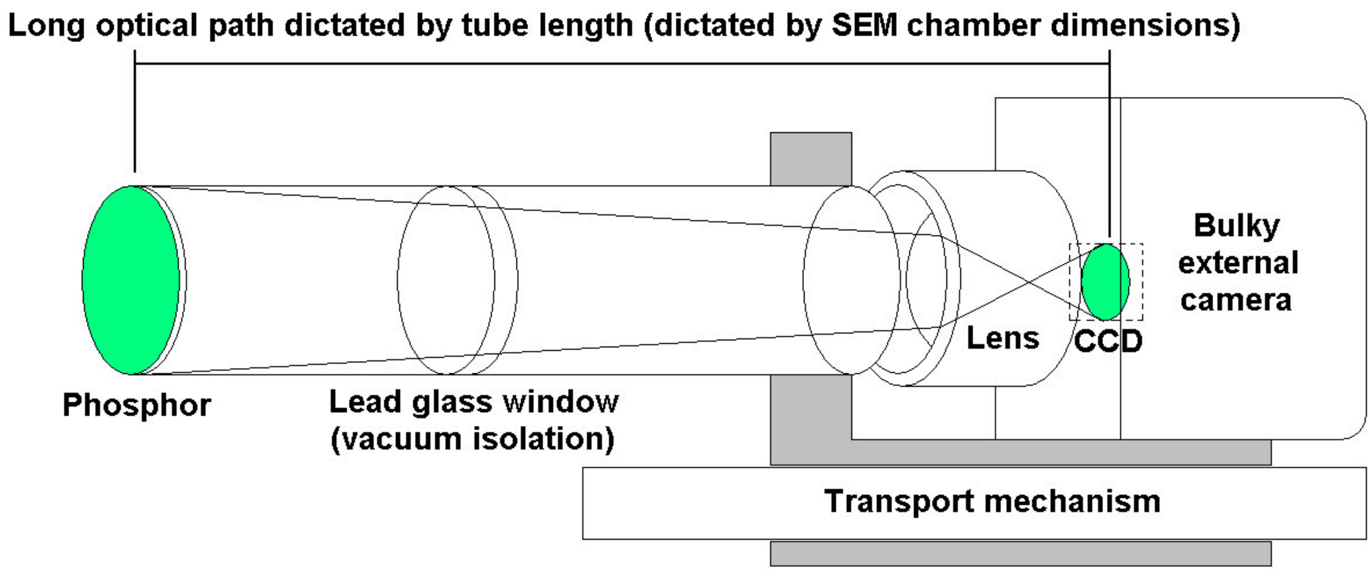
Schematic Layout of EBSD System



- Schematic layout of components
- PC and Imaging hardware common for INCA Energy and Wave
- Market leading EBSD, EDS & WDS all from one vendor

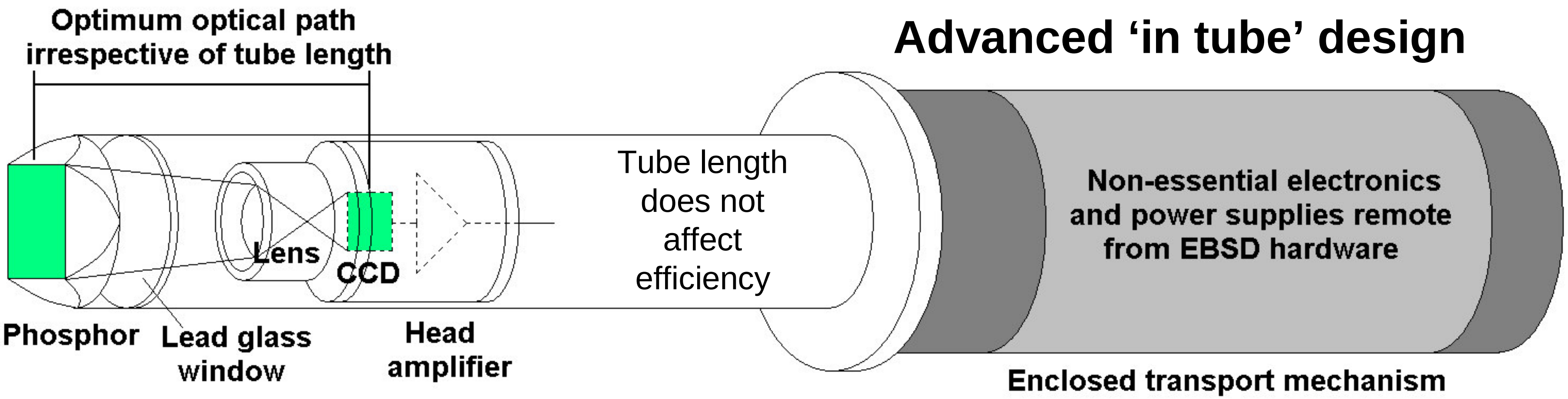
‘Nordlys’EBSD Detector

Comparison of conventional and advanced EBSD hardware design

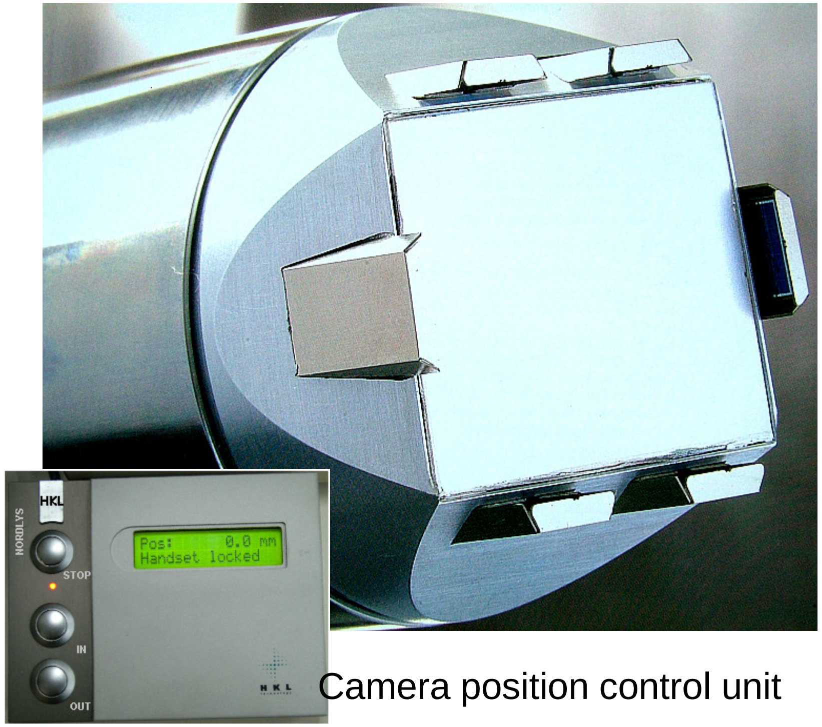


Conventional 'open bracket' design

Advanced 'in tube' design

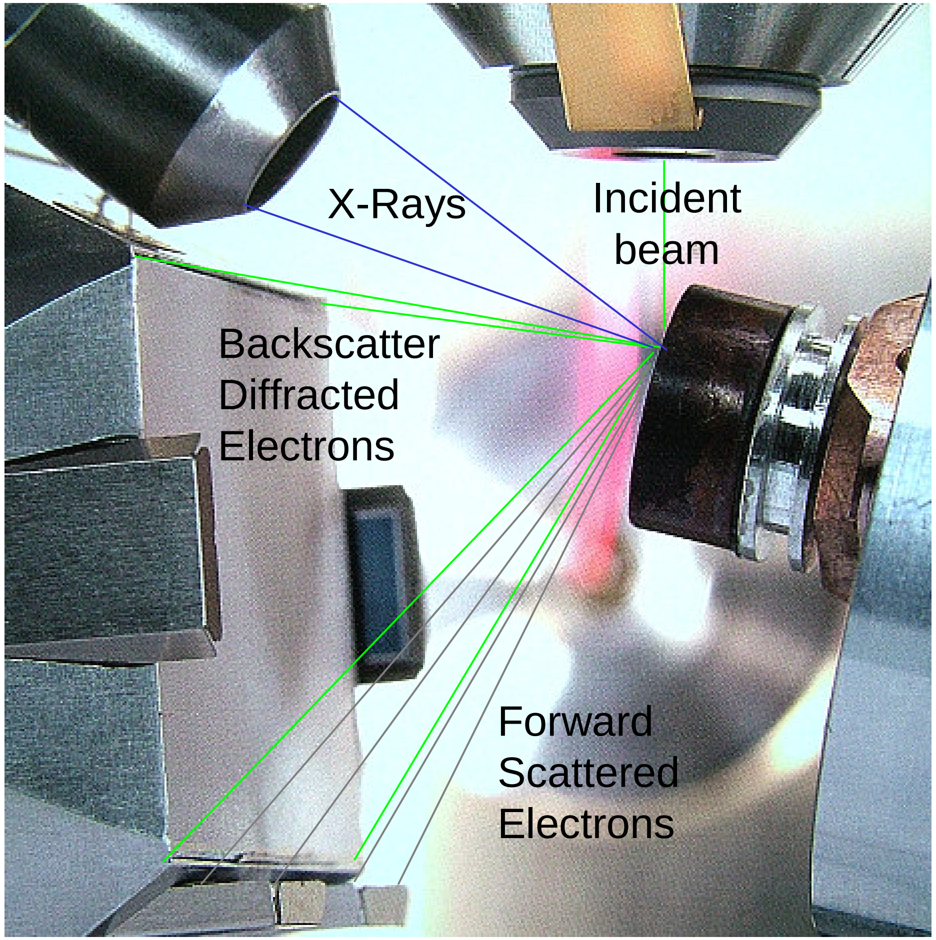


EBSD hardware - configurable collection geometry

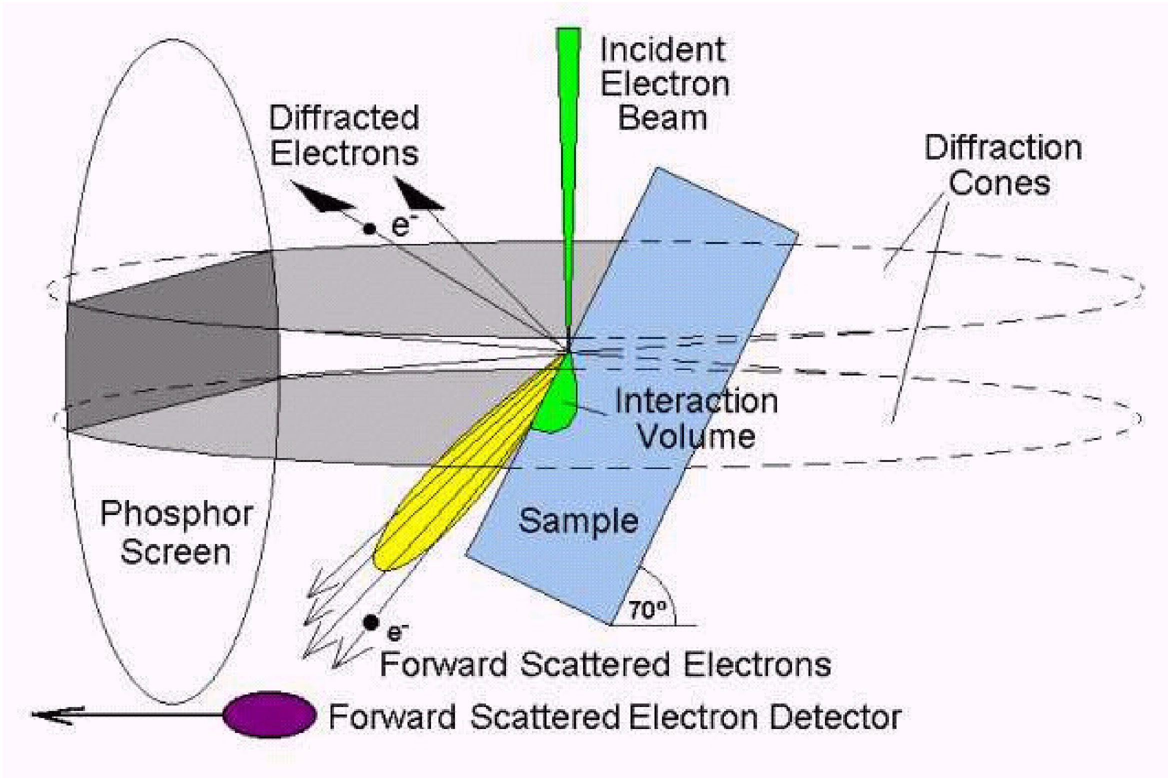


Camera position control unit

- Revolutionary high efficiency, fast detector with rectangular phosphor and multiple FSE detectors
- Motorized insertion/retraction with no constraints on working geometry
- Can be optimised for short WD or to avoid occluding other detectors

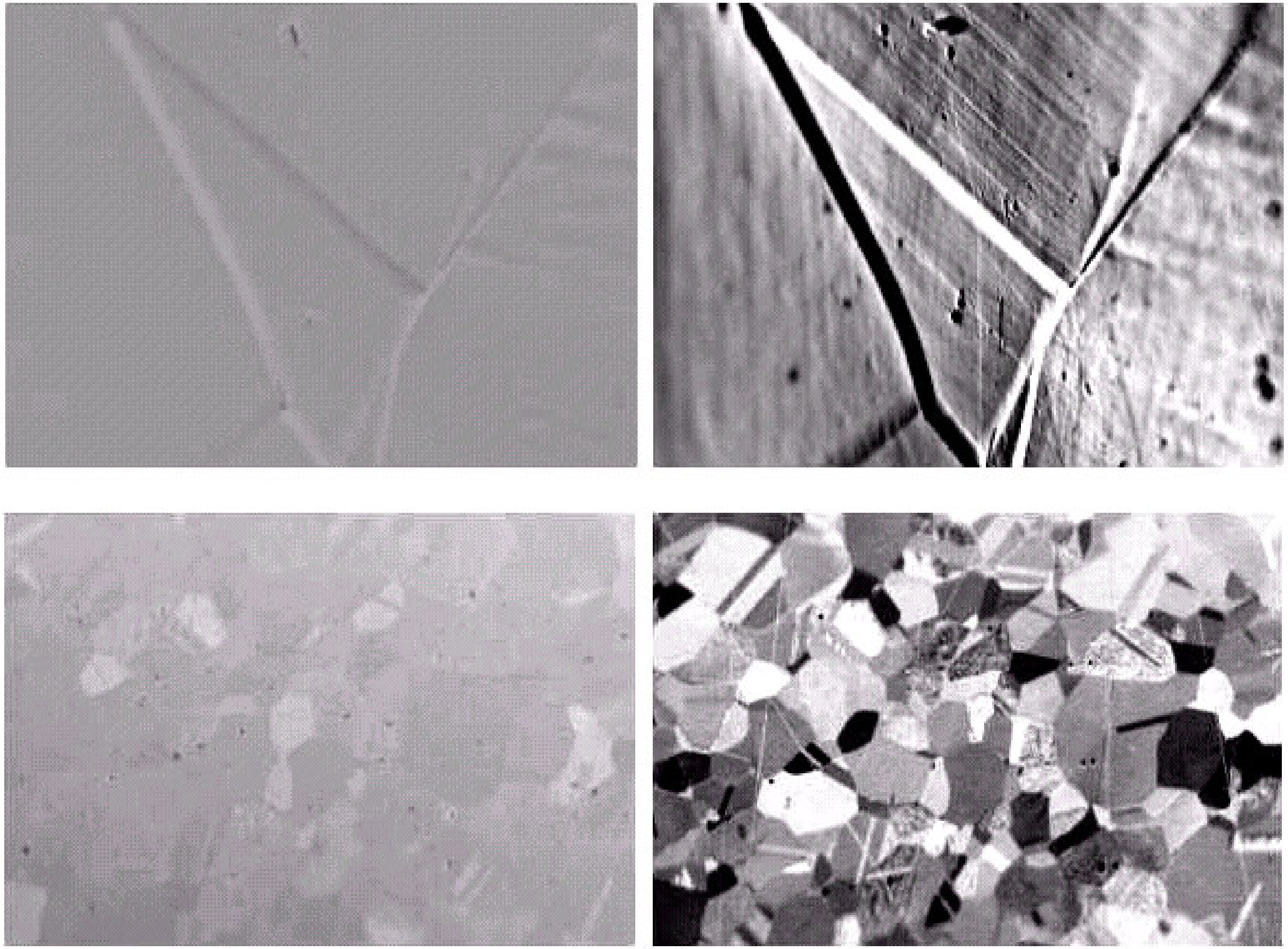


Forward Scattered Electron (FSE) Imaging



- FSE greatly enhances diffraction contrast in imaging
- Grains and grain boundaries are clearly revealed

FSE Imaging using a single detector

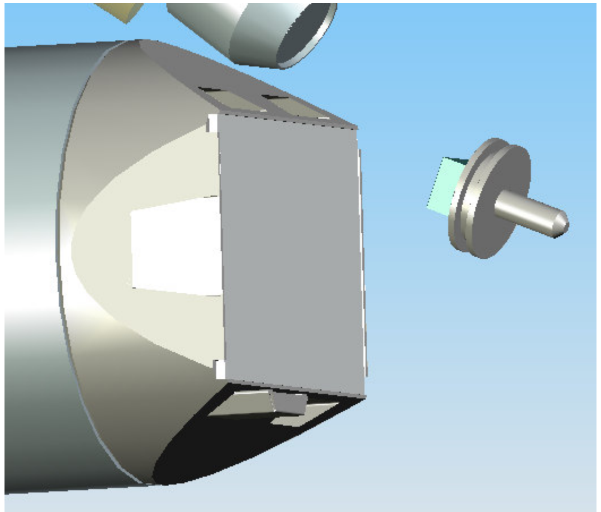


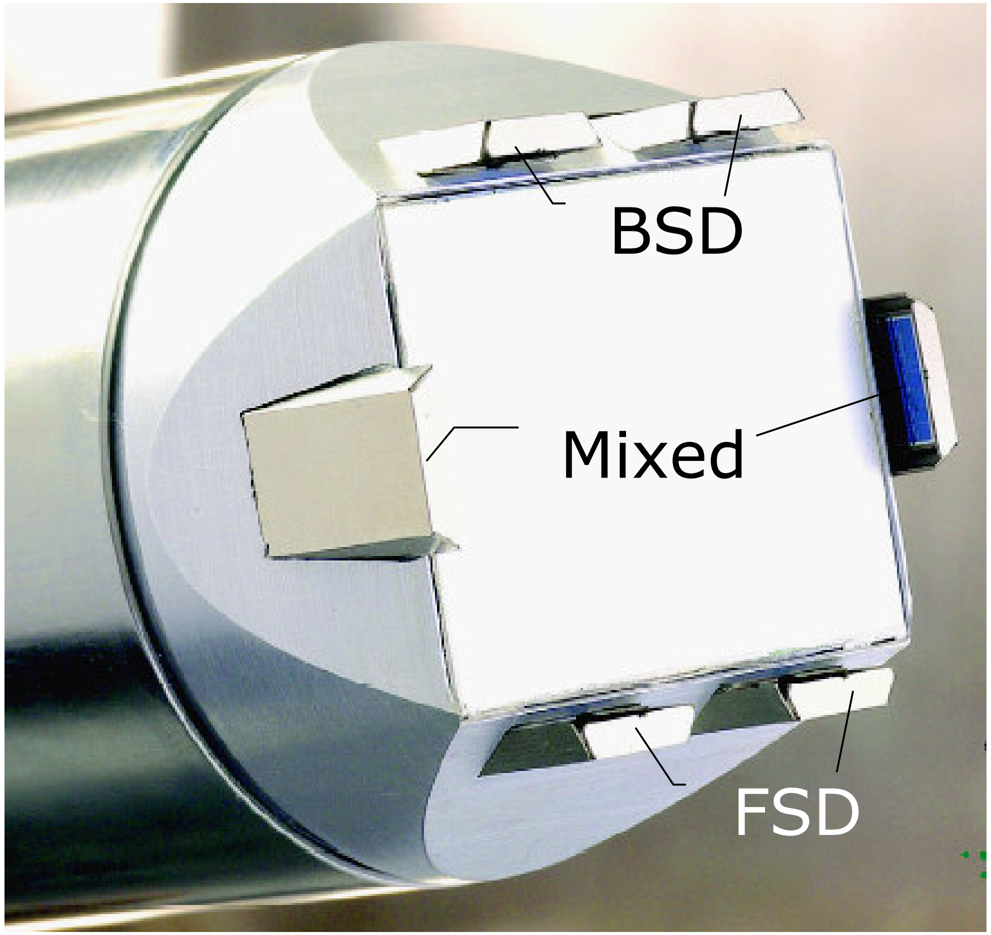
- Nickel
- Austenitic Stainless Steel

Secondary Electron Image　　Forward Scattered Electron Image

Forescatter Detector Option

- For orientation, phase contrast imaging at high tilt
- Two, four or six diodes – may be retrofitted or moved by user
- BSE, FSE or mixed mode detection

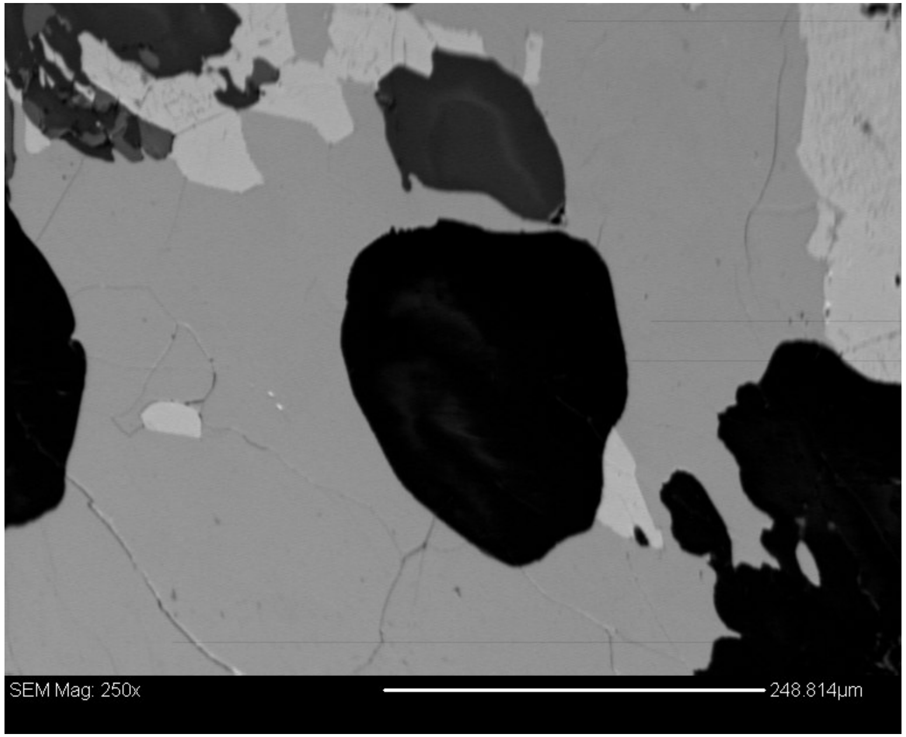




Multiple Forescatter Detector Option

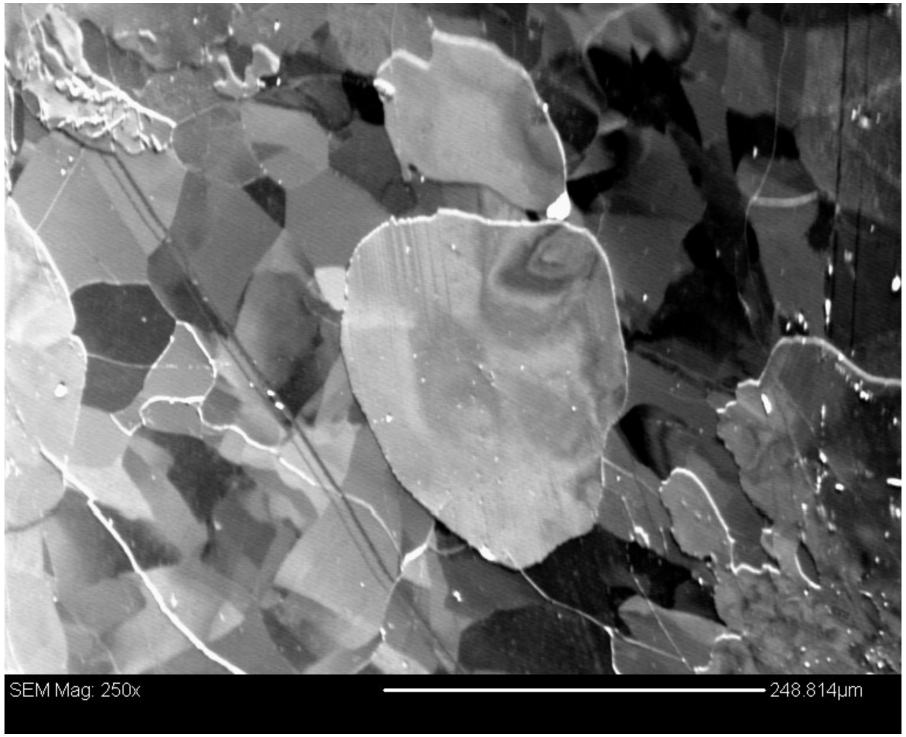
Diode orientation and position optimal for high tilt EBSD conditions

Forescatter Detector: Orientation Contrast & Phase Contrast



BSE Image – Density (phase) contrast

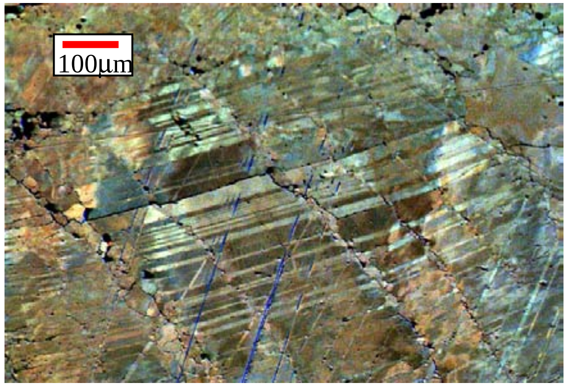
Multiphase rock (gabbro)



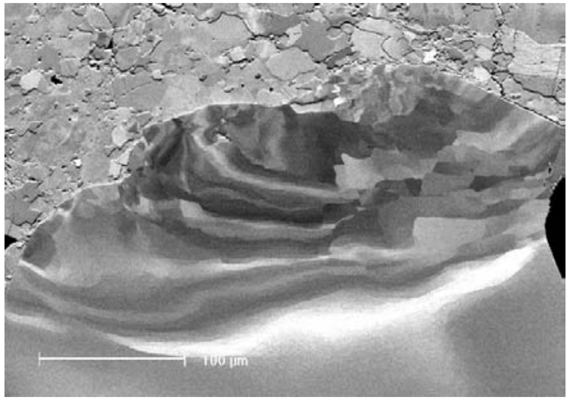
OC (FSD) Image – Channeling contrast

Forescatter gallery

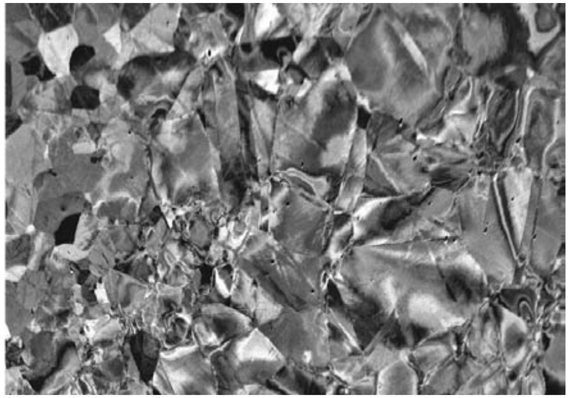
Calcite (Dave Prior, Liverpool)



Welded superalloy (NPL)



Zircon grain

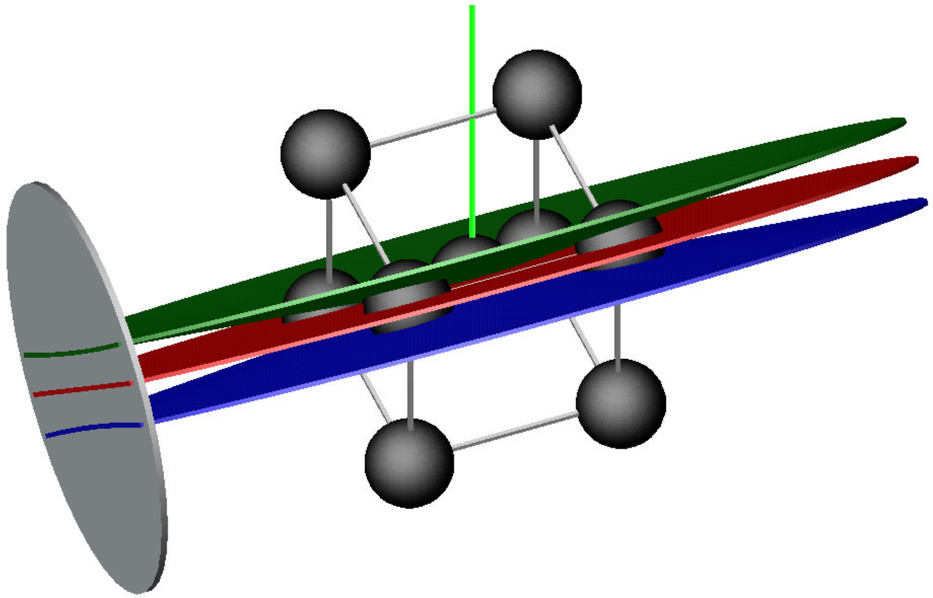


Deformed superalloy

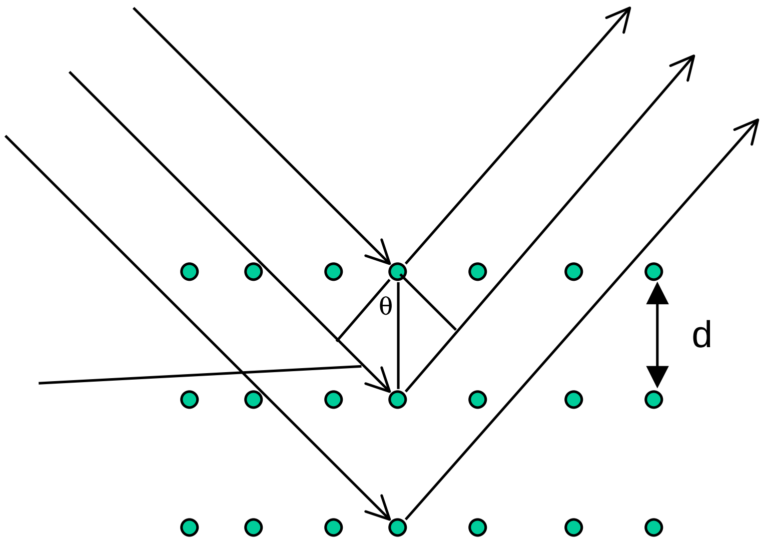
Plagioclase

EBSP Formation

- The electron beam strikes the specimen
- Scattering produces backscatter electrons in all directions, most intensely downward & outward
- Electrons that travel along a crystallographic plane trace generate Kikuchi bands whose widths are dictated by the Bragg Law and specimen-to-phosphor screen distance.
- The electrons hit the imaging phosphor and produce light
- The light is detected by a CCD/SIT camera and converted to an image
- Which is indexed...



Diffraction



Extra path length
= 2d sinθ

$\lambda = 12.26 * E^{-1/2}$

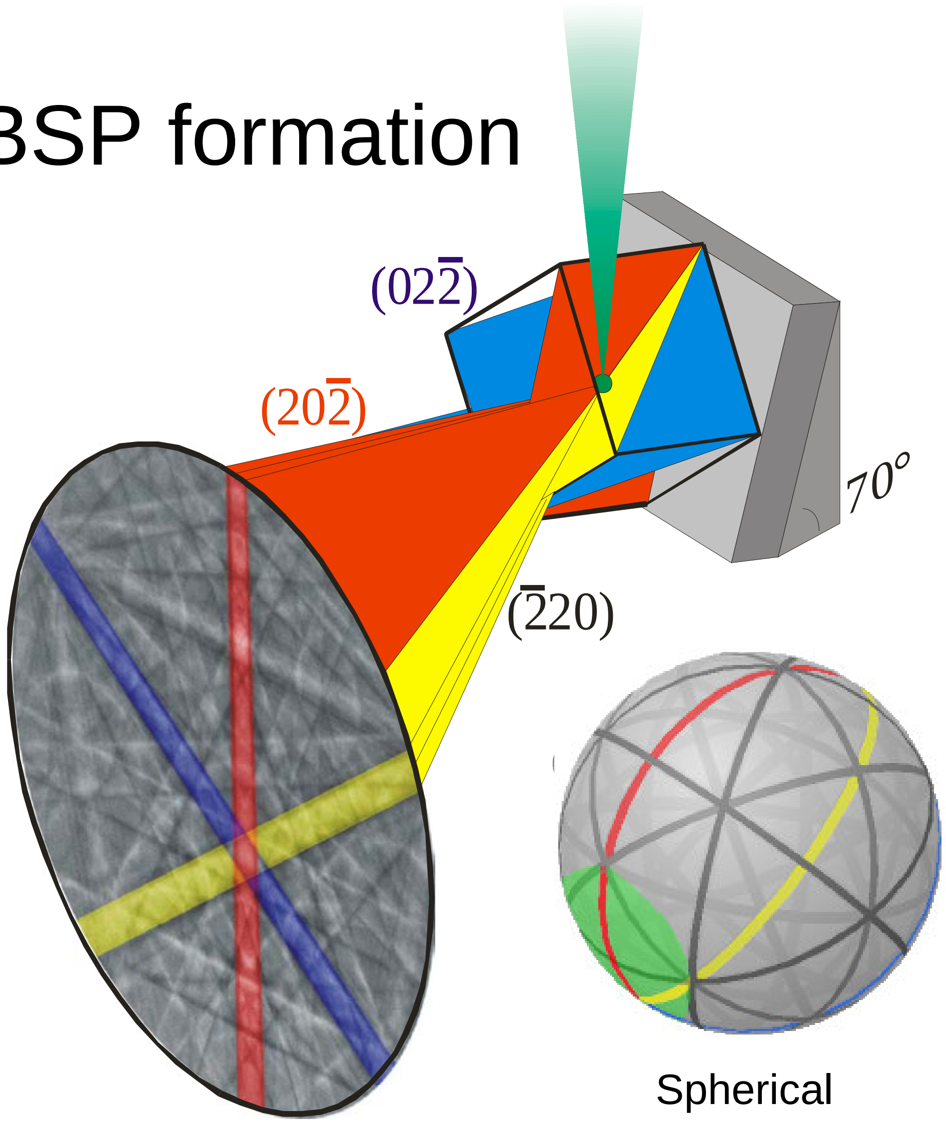
E = incident beam energy in volts

For 20 kV and a plane spacing of 0.2 nm, the Bragg angle, θ,is about 1.25°.

Constructive interference occurs when the extra path length is an integral number of wavelengths.

Bragg's Law $n\lambda=2d \sin\theta$

EBSP formation

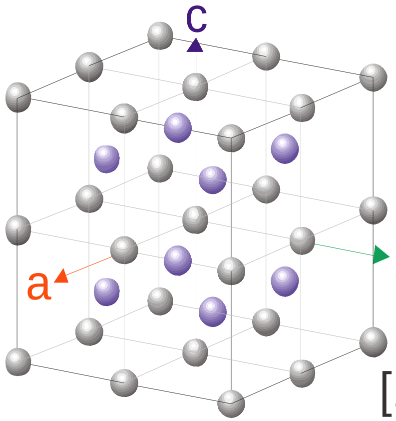


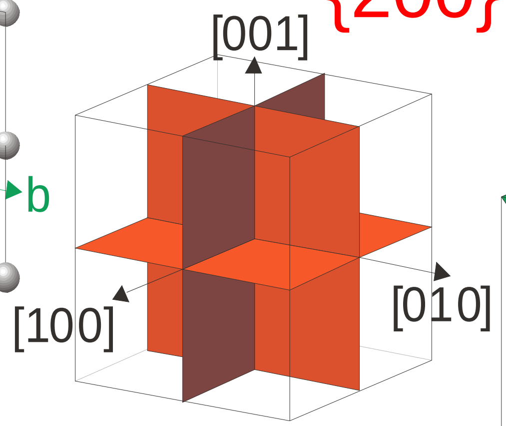
Phosphor screen

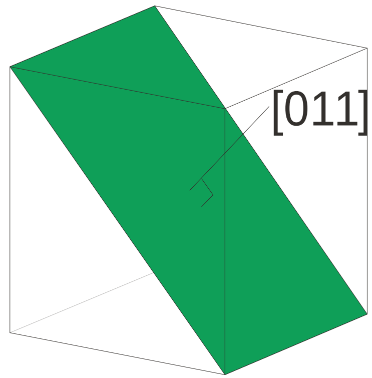
Spherical Kikuchi map

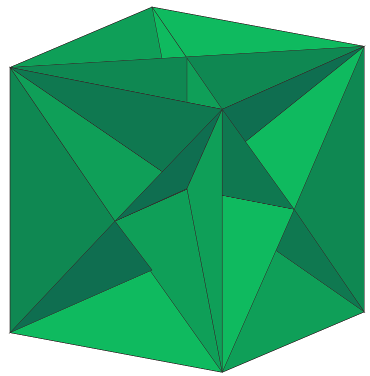
EBSP formation

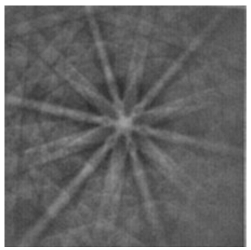
Body centred cubic Iron

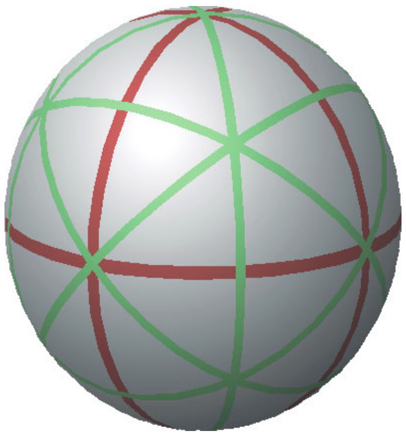


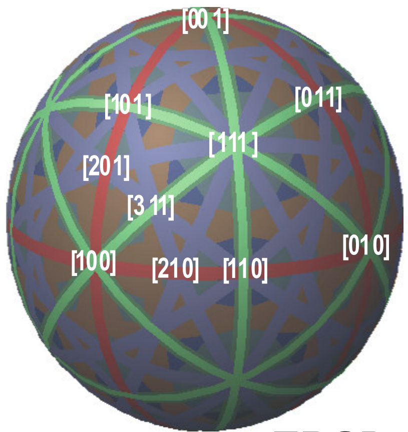






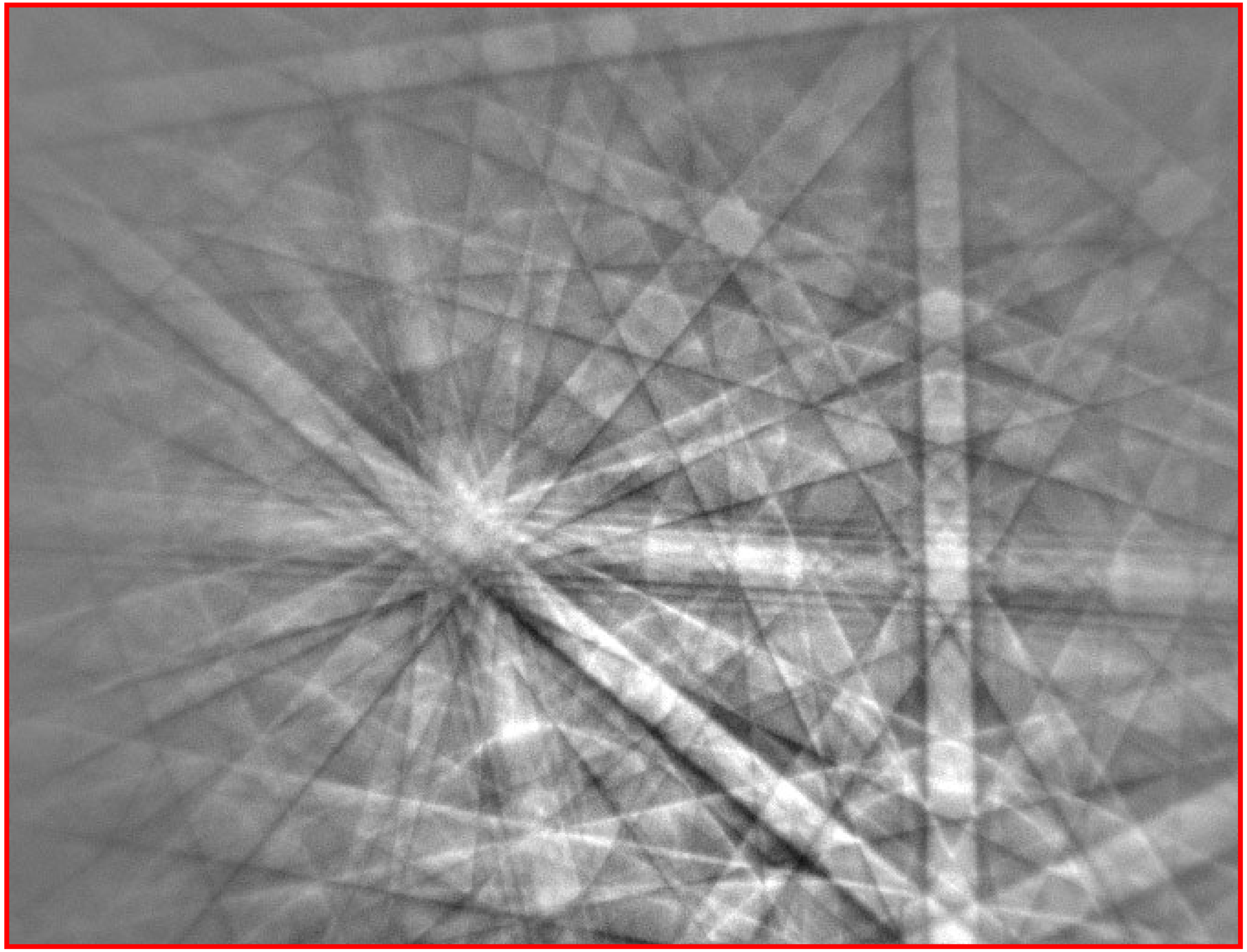






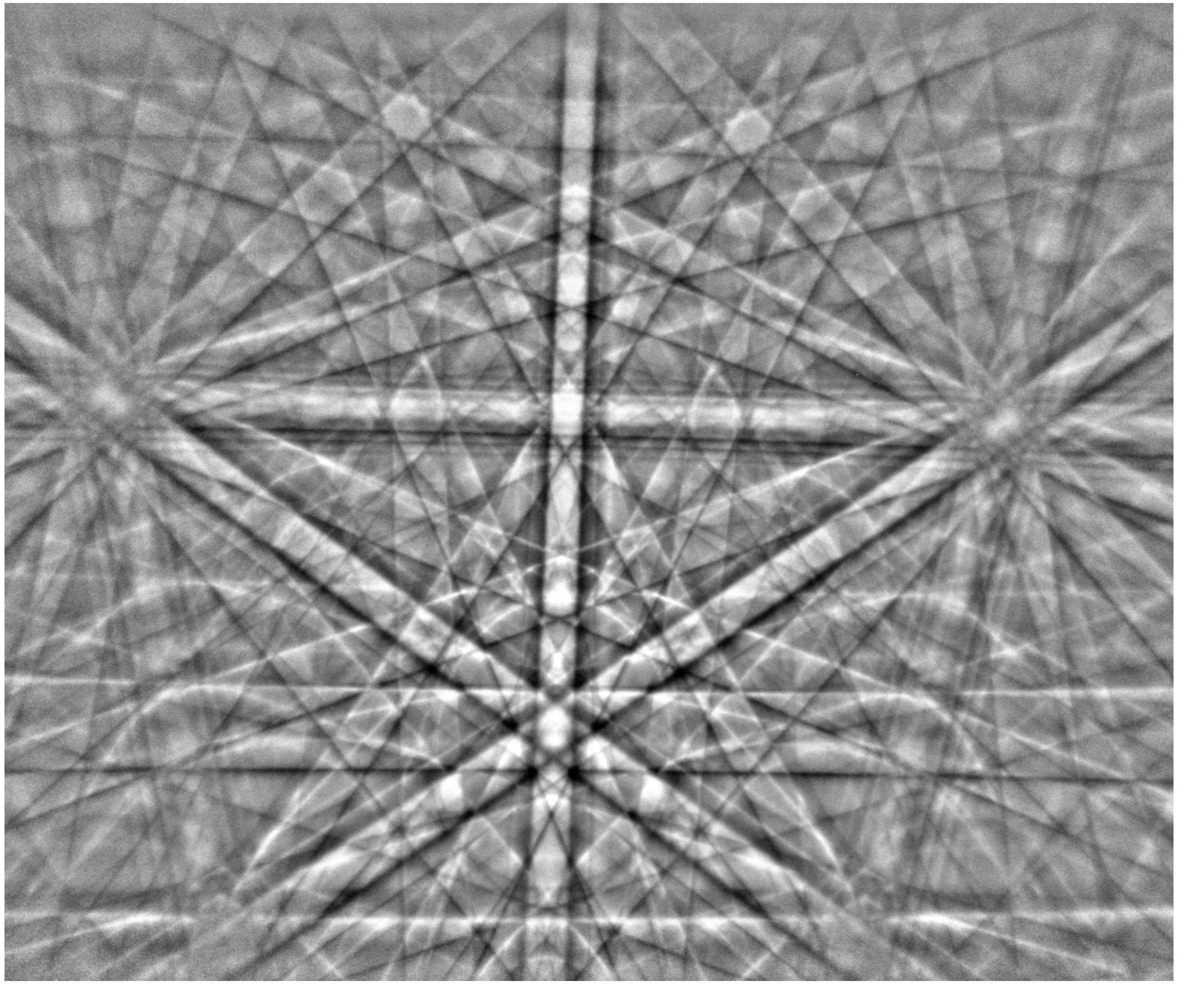
See the new dedicated EBSD website: http://www.EBSD.com

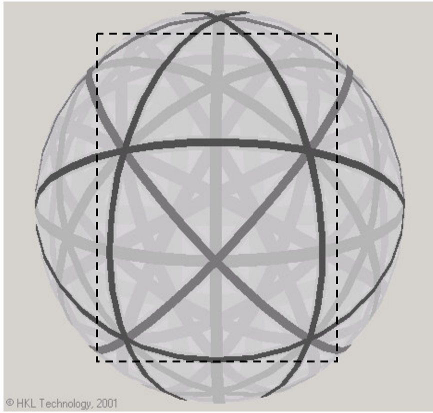
What does an EBSP look like?

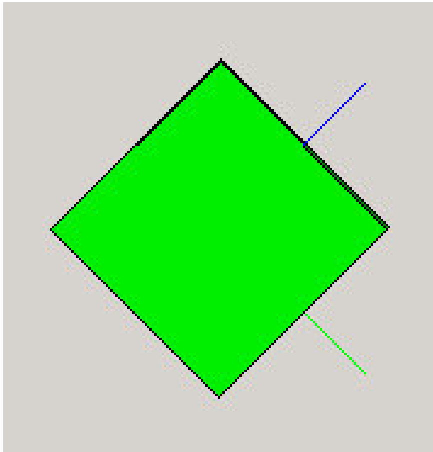


Silicon at 20kV

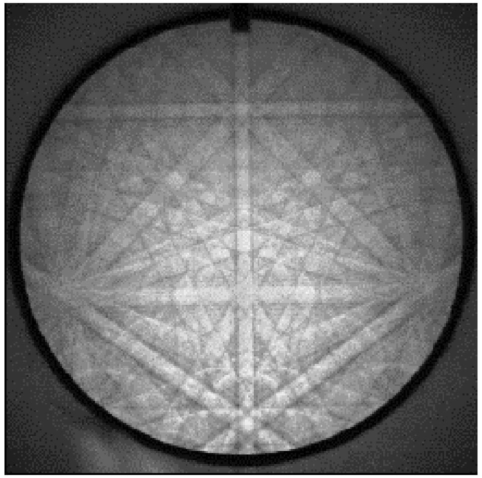
Hi-res EBSP - Si 20kV



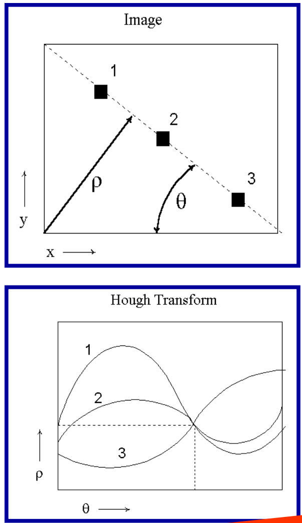


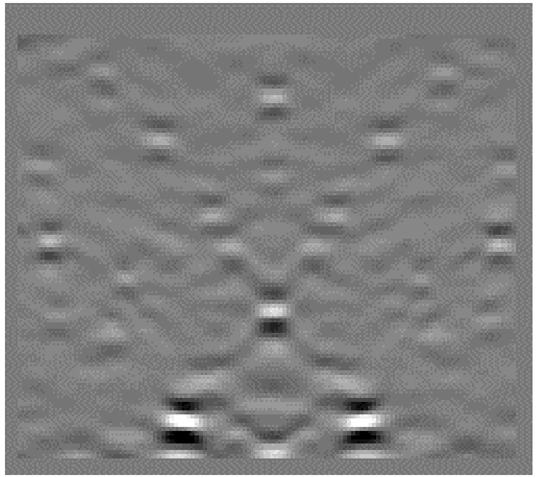


From EBSP to Orientation

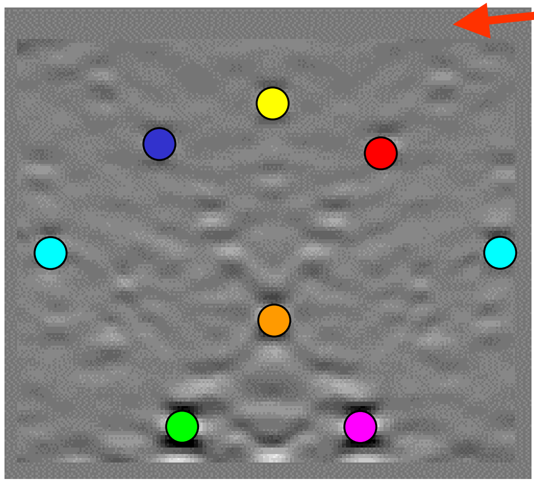


EBSP

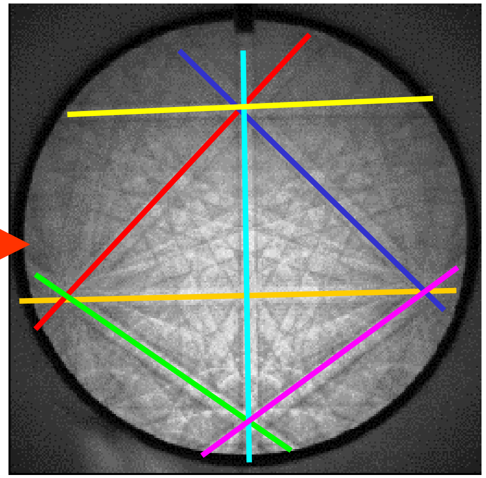




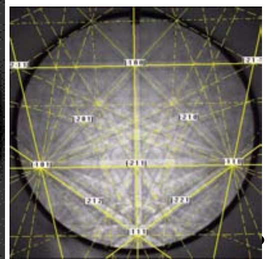
Hough 'space'



Hough Peaks found



EBSP Bands detected



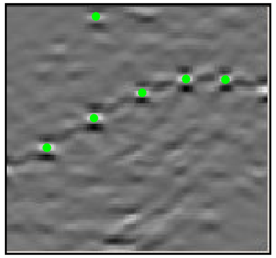
'solve'

- 'Matching' algorithm compares detected bands with those calculated from the crystal structure for the phase(s) of interest
- EBSP 'solved' or 'indexed' to find orientation of crystal under the beam

Indexing: Phase discrimination & orientation determination (simplified)

Band Detection

- List of angular relationships between detected bands
- Band positions on phosphor screen
- Other information also extracted (e.g. band widths)
- Highest intensity bands most likely to be detected



Reflector Calculation

For each potential phase, list of allowed reflections, diffraction band widths and intensity hierarchy generated from kinematic electron diffraction theory or reflector list is loaded. Also, for reflections above an intensity cutoff, interband angles are calculated.

h k l	d [Å]	Intensity	visible (if not cropped)
1 -1 -1	3.1356	100.0	+
1 1 -1	3.1356	100.0	+
1 1 1	3.1356	100.0	
1 -1 1	3.1356	100.0	
2 -2 0	1.9201	65.5	+
2 0 -2	1.9201	65.5	+
0 2 -2	1.9201	65.5	+
2 2 0	1.9201	65.5	+
0 2 2	1.9201	65.5	+
2 0 2	1.9201	65.5	
0 0 4	1.3578	24.7	+
4 0 0	1.3578	24.7	
0 4 0	1.3578	24.7	+
1 -1 -3	1.6375	21.1	+
1 3 -1	1.6375	21.1	+
1 1 3	1.6375	21.1	
1 -3 -1	1.6375	21.1	+
1 3 1	1.6375	21.1	+
1 -3 1	1.6375	21.1	+

Match?

Yes

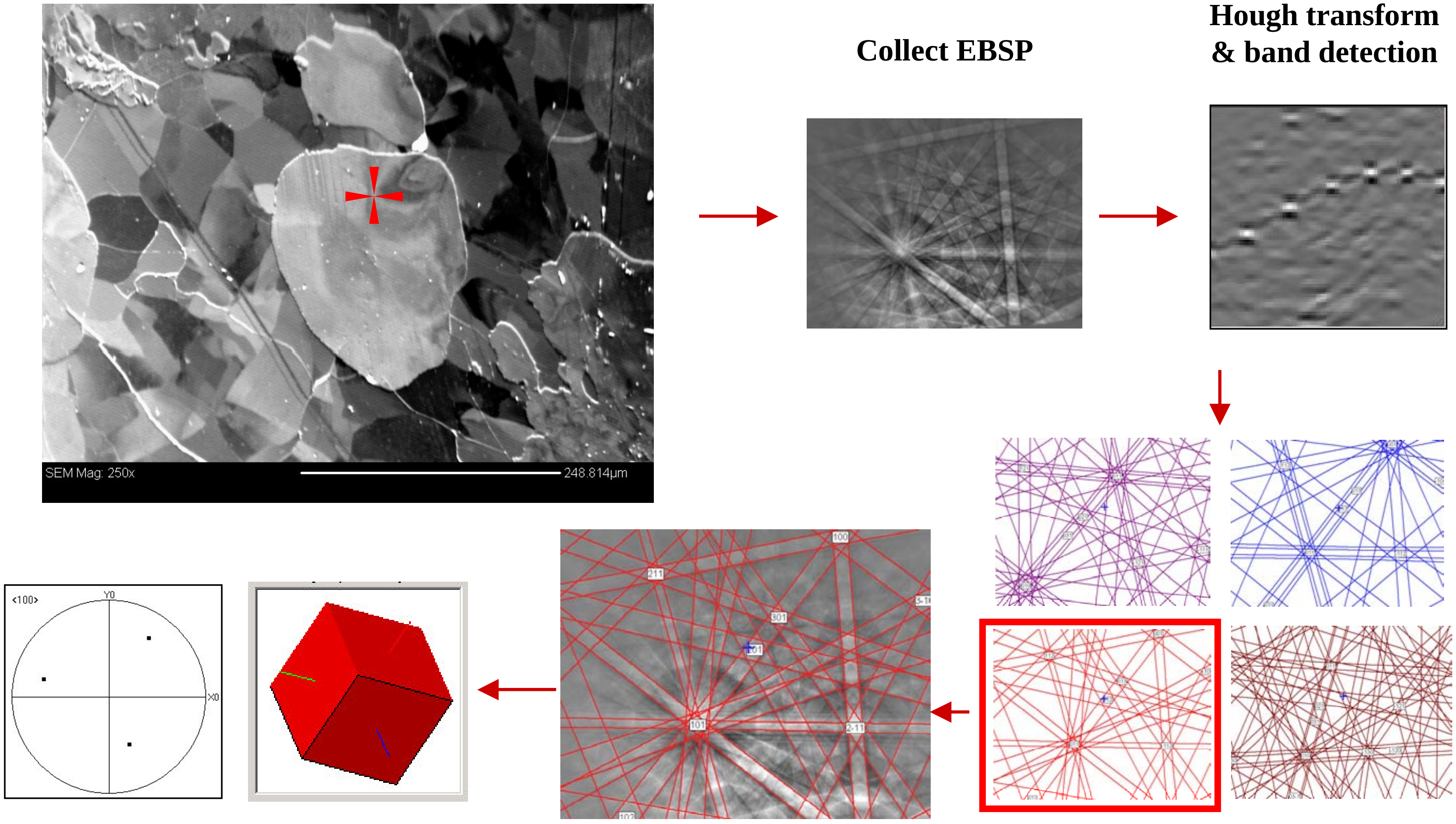
Phase discriminated
Orientation determined

Indexing cycle

Position beam

Collect EBSP

Hough transform & band detection



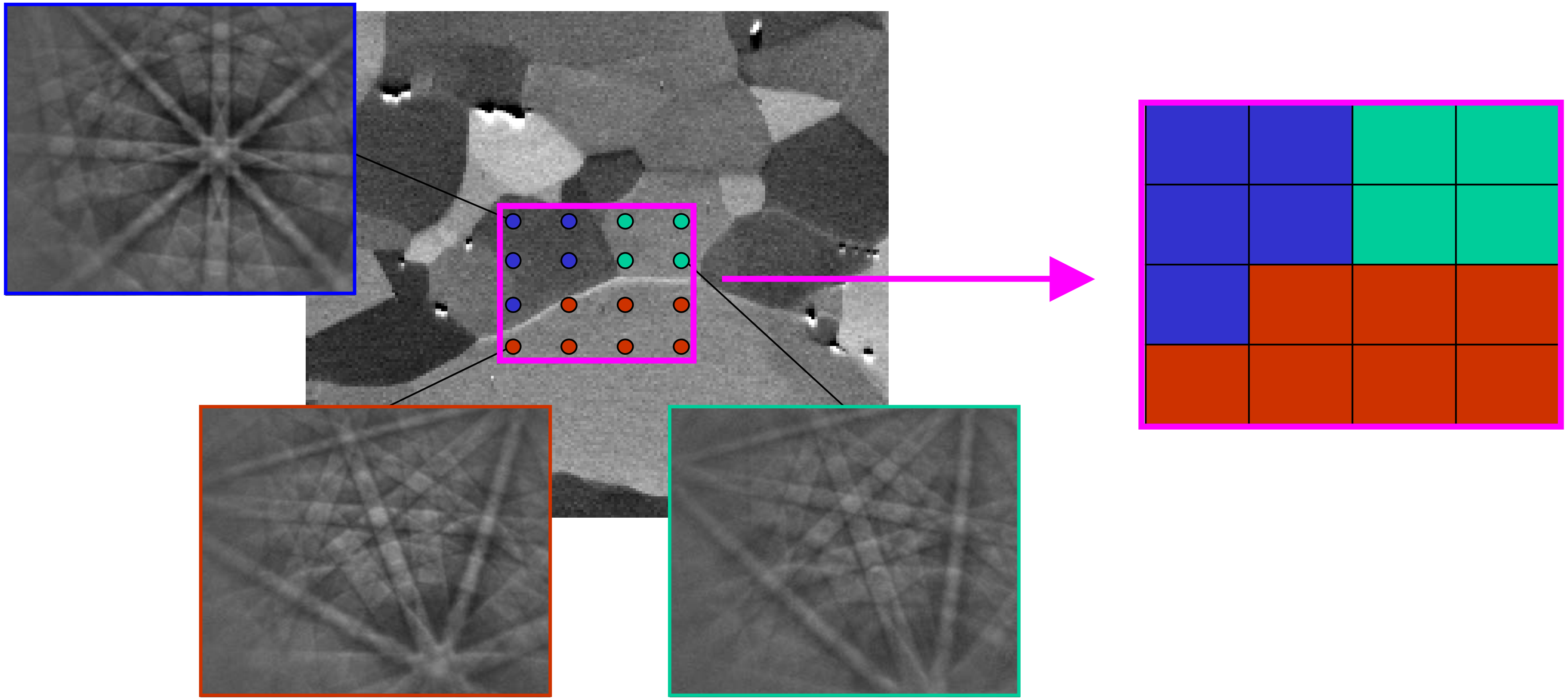
Phase and orientation

Verify match: Indexed EBSP

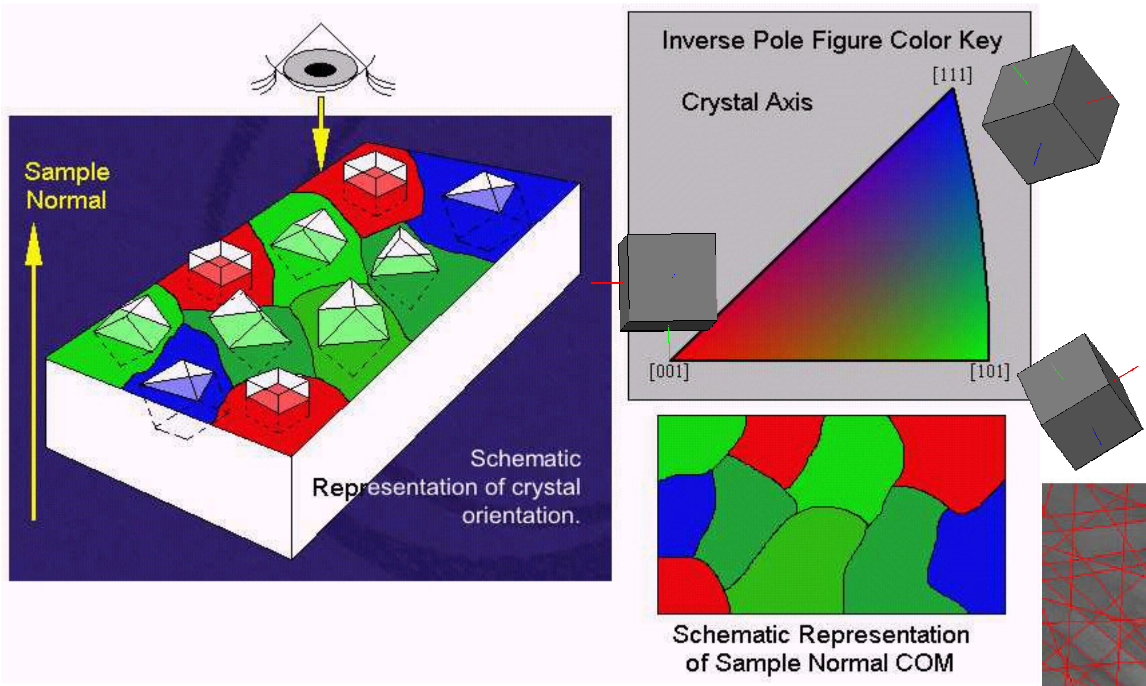
Match to phase & orientation

See the new dedicated EBSD website: http://www.EBSD.com

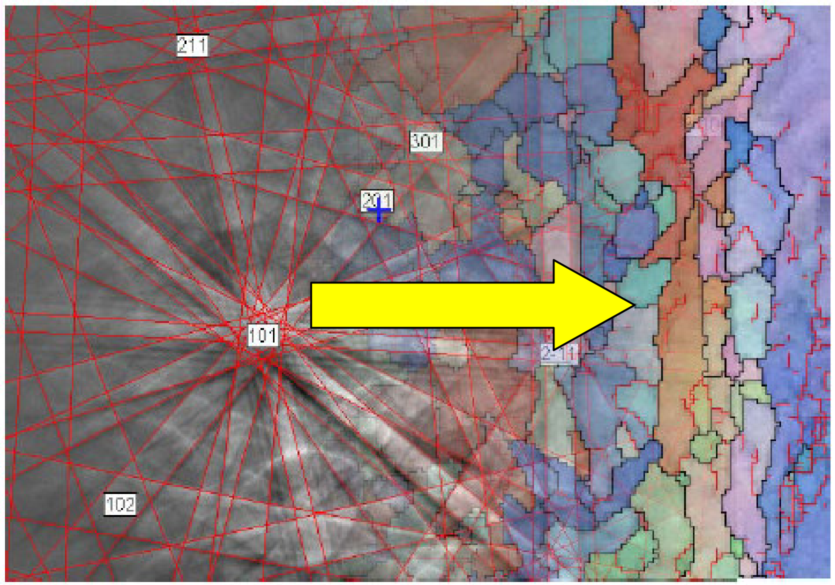
Mapping



From EBSP to Crystal Orientation Map



- COM with Inverse Pole key for cubic material
- Red = 100
- Green = 110
- Blue = 111 planes parallel to the surface



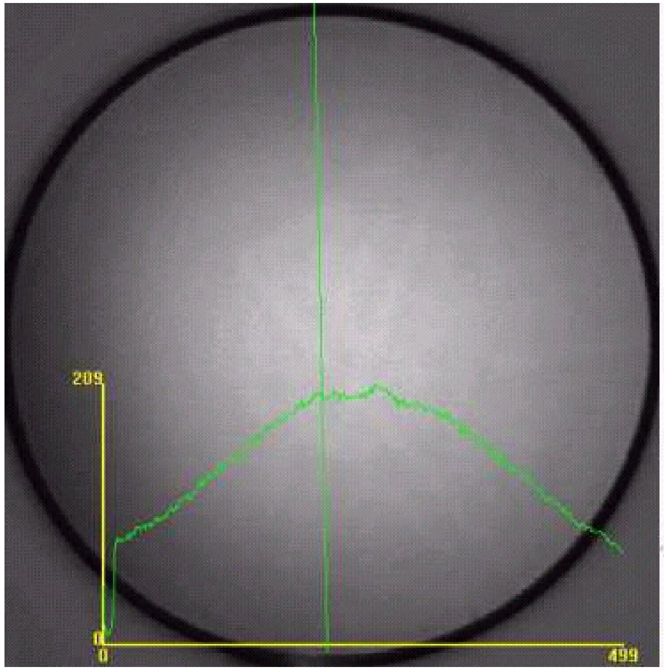
- Orientation obtained at every pixel
- Colour derived from inverse Pole Figure color key or Euler angles
- Hough transform for every pixel stored for post acquisition reprocessing

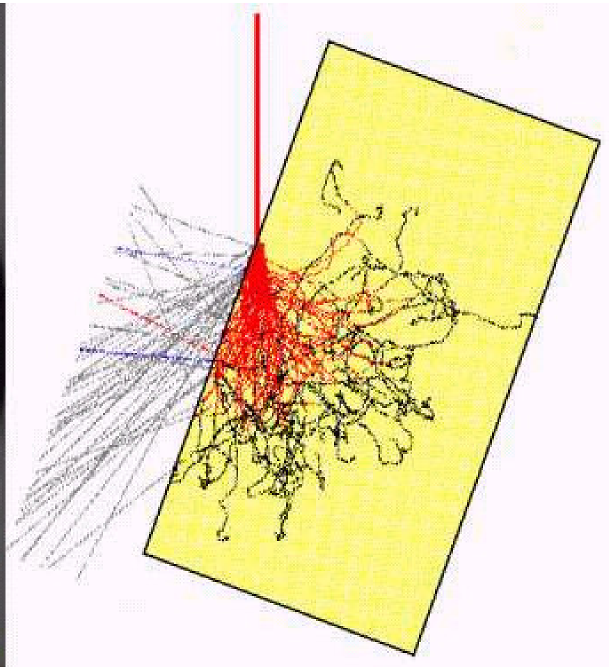
Factors affecting EBSP Quality

Electron Back Scattered Patterns (EBSP’s)
vary greatly...

EBSP ‘Background’

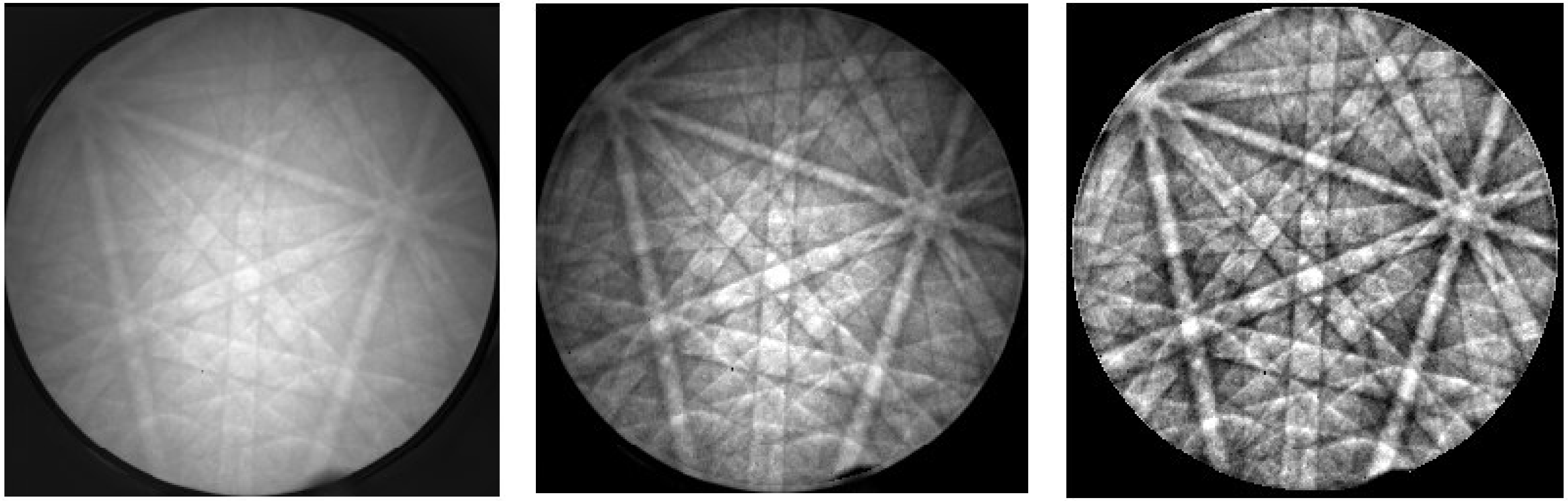
- Only a small proportion of the electrons arriving at the phosphor screen are diffracted.
- Therefore the pattern is superimposed on a ‘background’





- Background removal is required to enhance pattern contrast

Enhancing EBSP Contrast - Background Removal



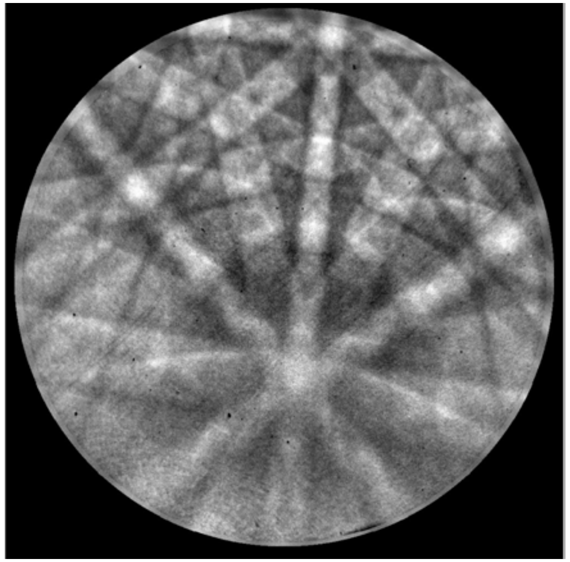
Fe 20kV raw EBSP

70% Background subtracted

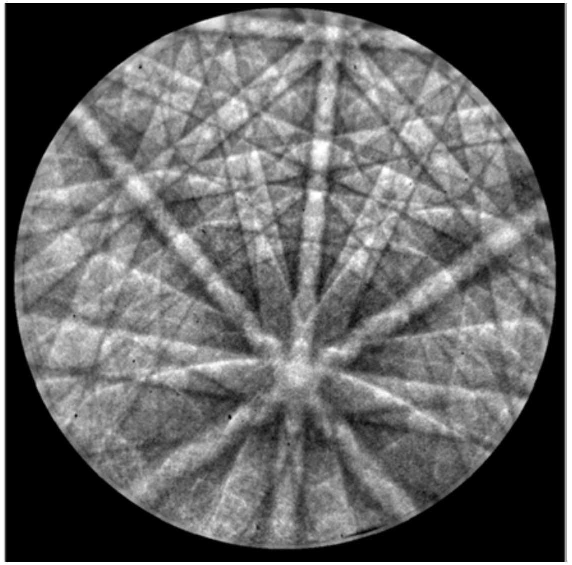
Background Divided

- EBSP contrast enhanced by background subtraction or division
- Particularly useful in lighter atomic number materials or when pattern quality (contrast) is low

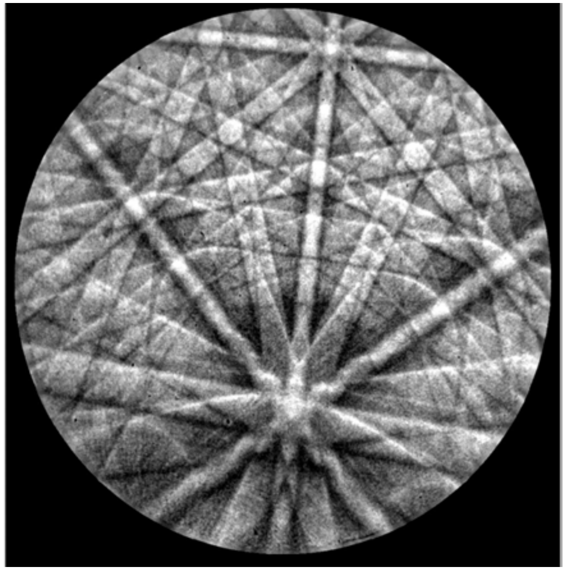
Accelerating Voltage - Effect of kV on Pattern Quality



Nickel 10kV



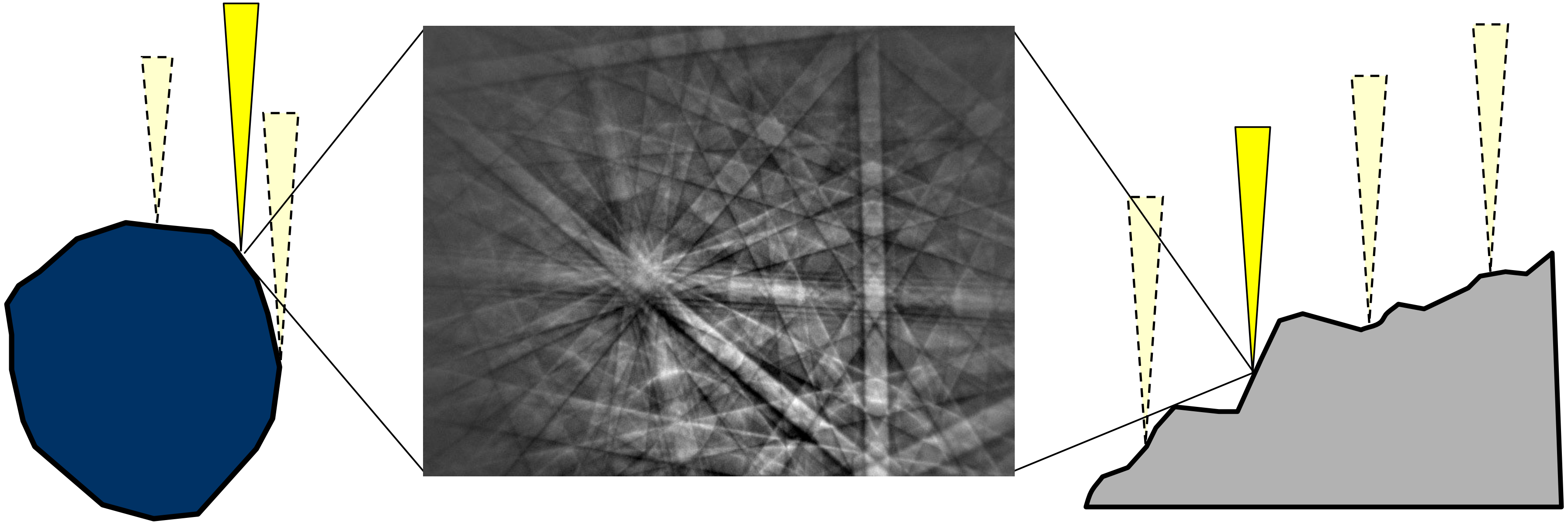
Nickel 20kV



Nickel 30kV

- Bands are broader and less sharp at low kV
- Band detection can be influenced by Hough parameters

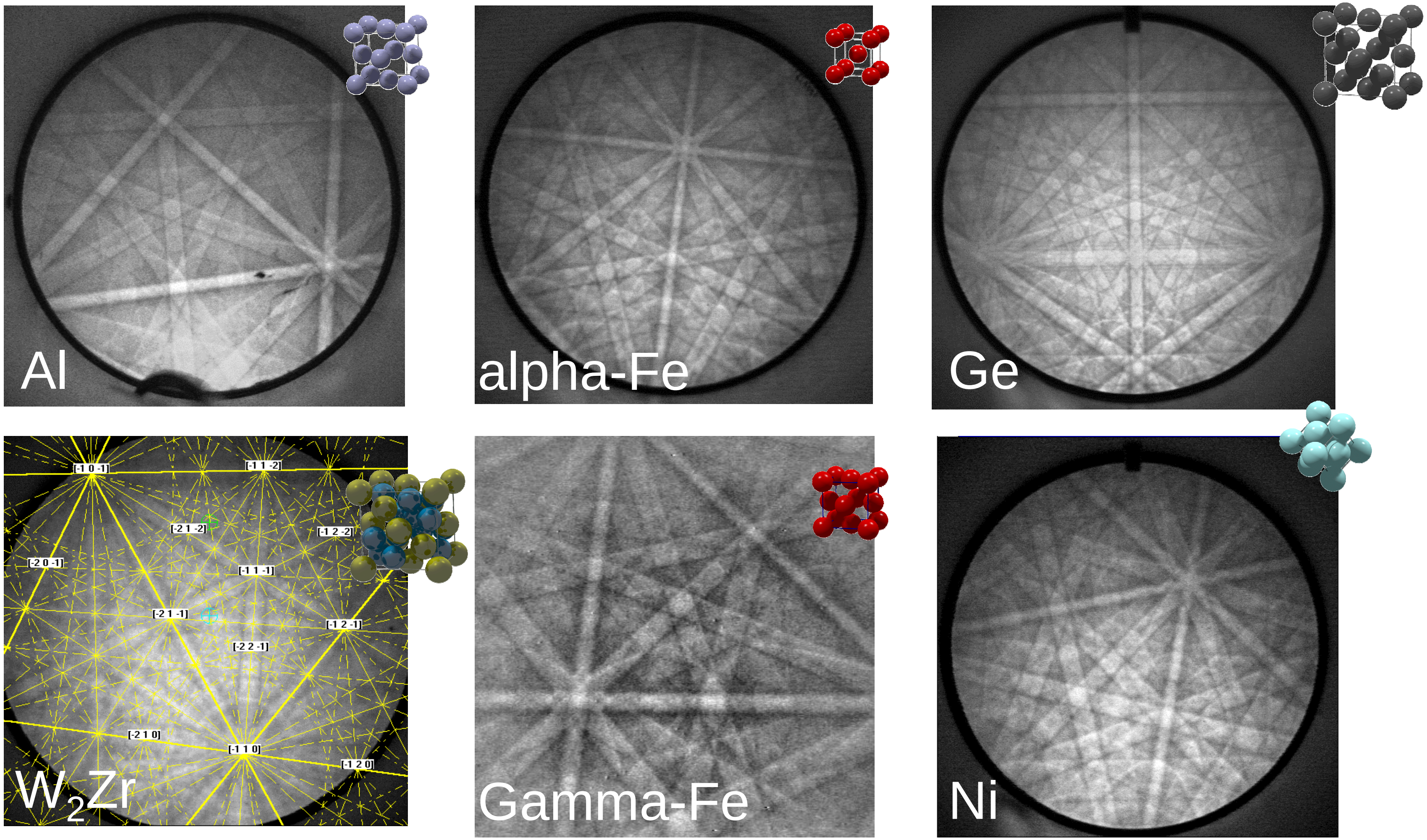
Sampling: Non-planar surfaces



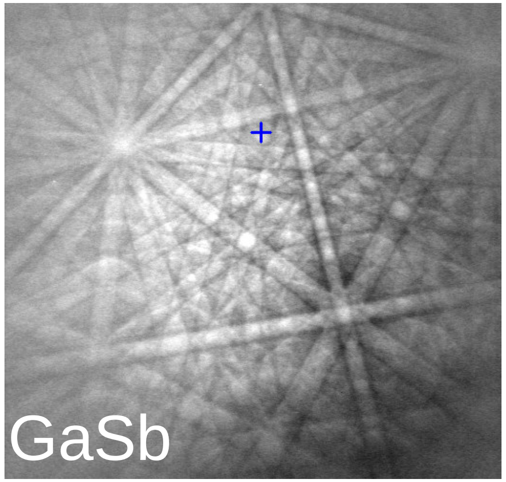
Sampling selection on fracture surface or free sediment

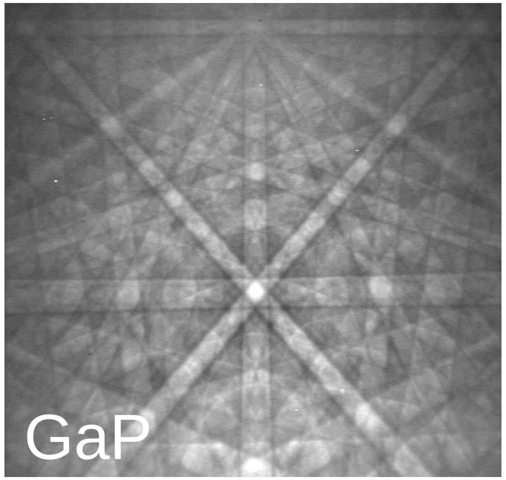
- Operator must search for locations where local topography & damage level allow clear patterns to reach detector
- FIB-SEM allows precision selection of areas for sectioning & mapping

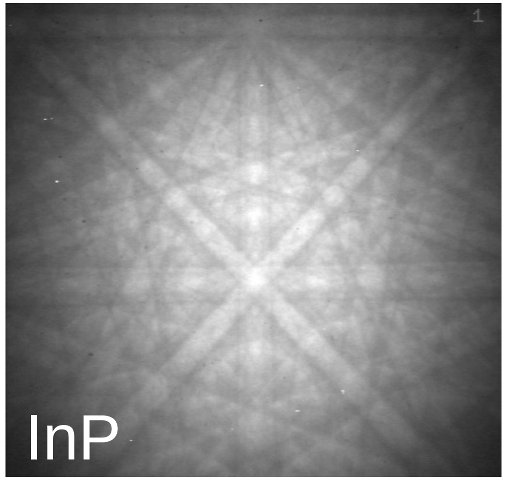
Examples of EBSPs

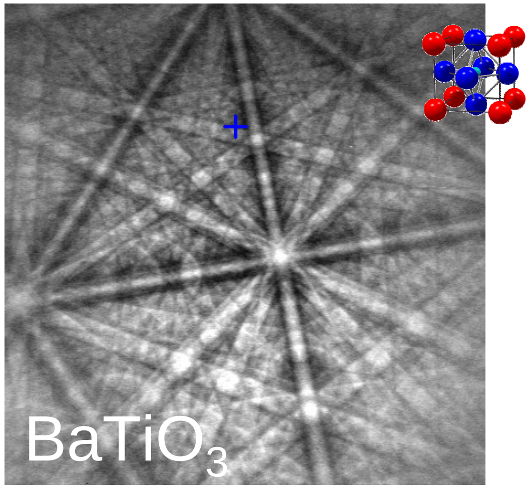


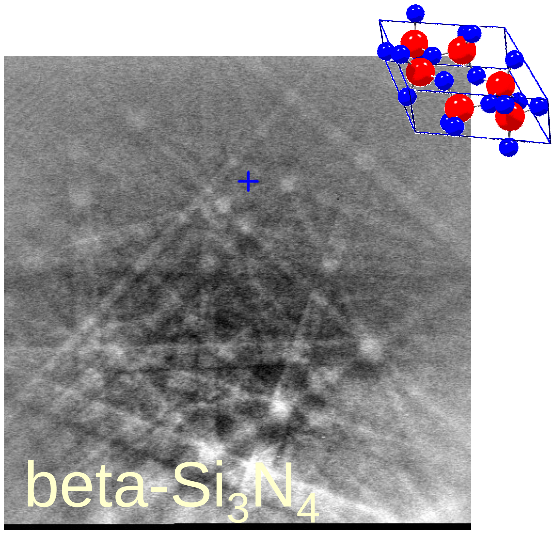
Examples of EBSPs

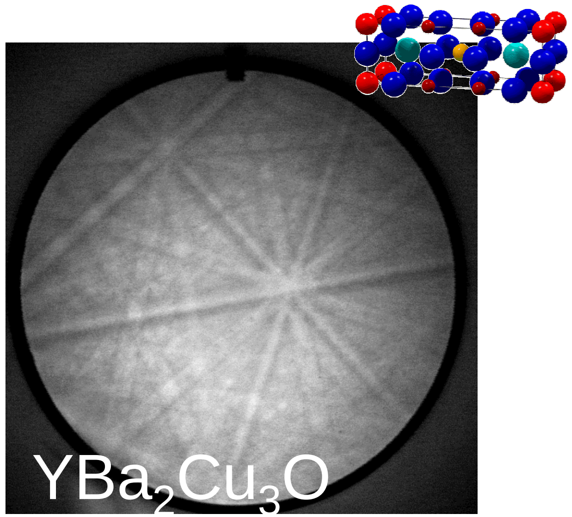




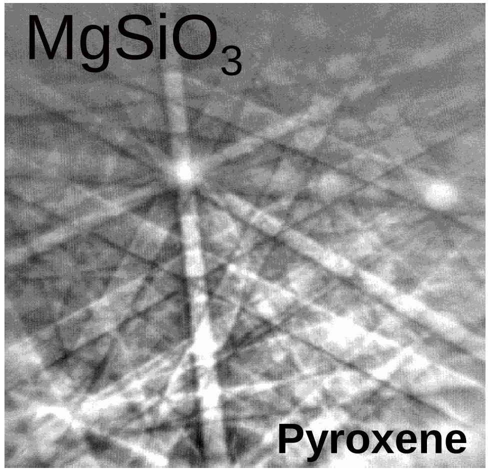


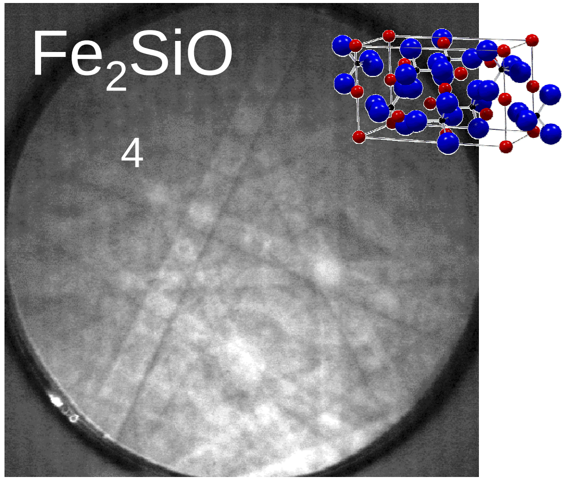


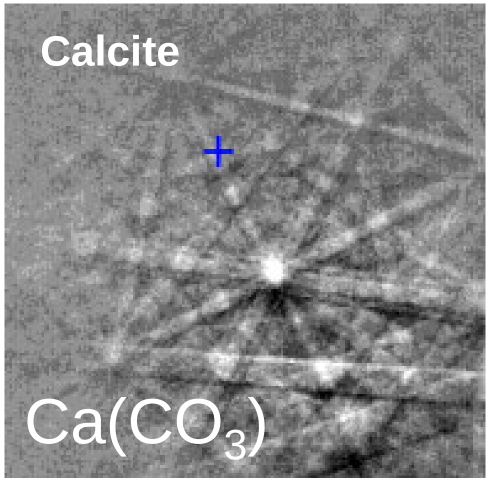


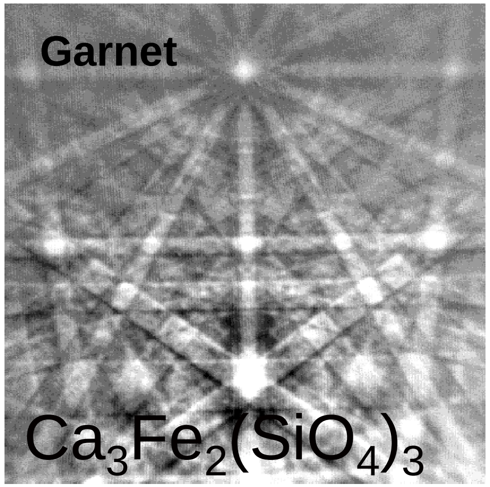


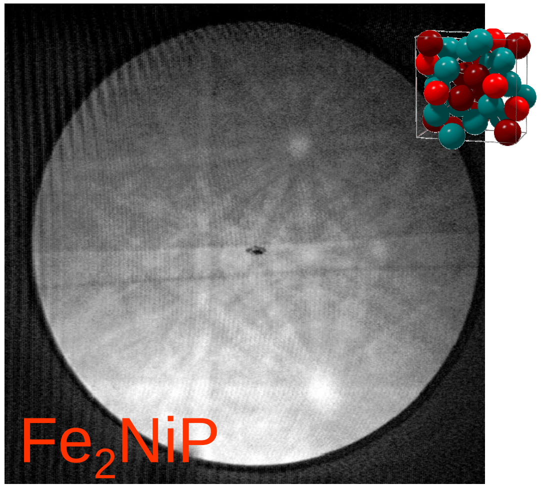
Examples of EBSPs





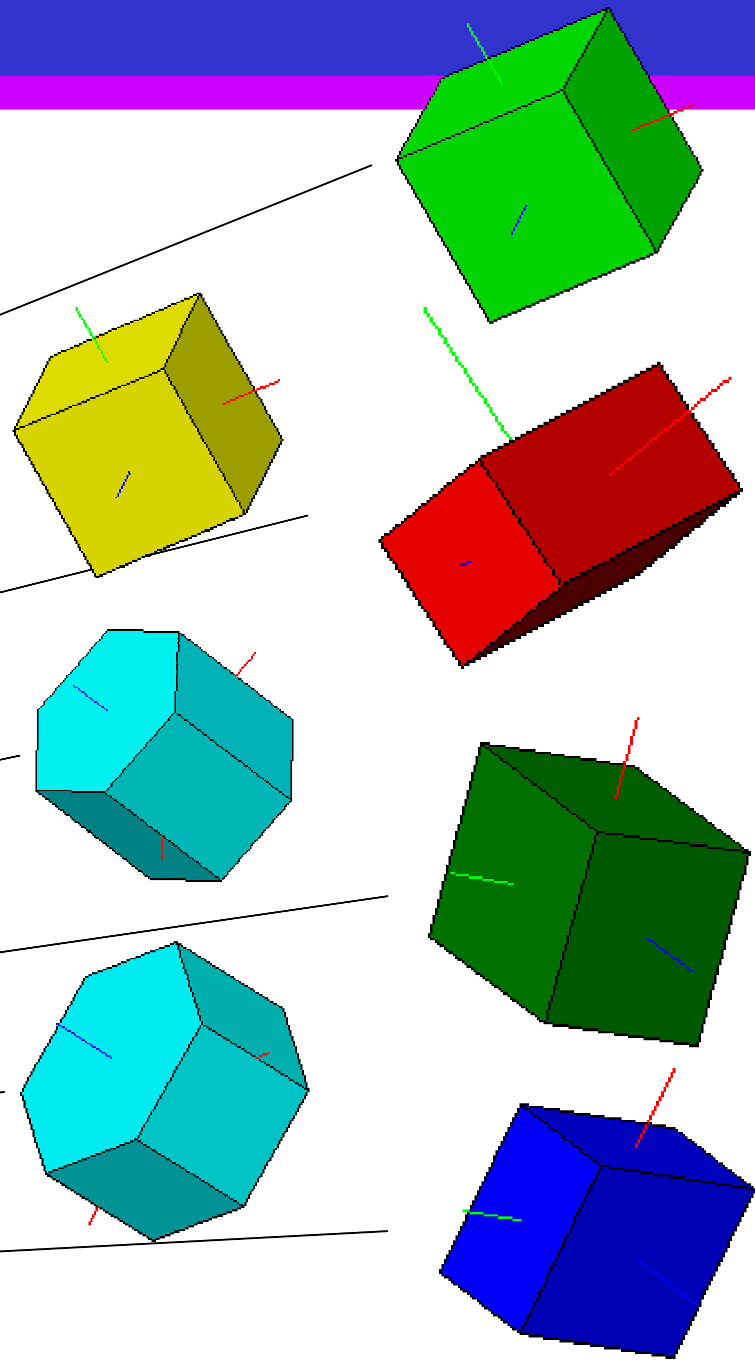




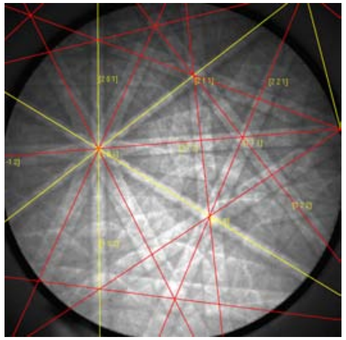


Seven crystal systems

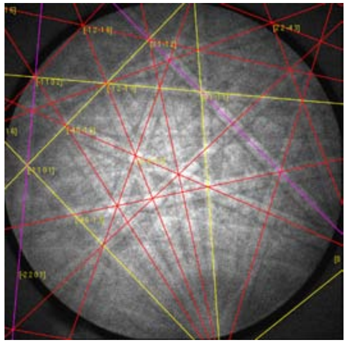
- Cubic (Al, Fe)
- Tetragonal (Lead Titanate)
- Orthorhombic (YBaCuO)
- Hexagonal (SiC, Titanium)
- Monoclinic (Zirconia)
- Trigonal (Quartz)
- Triclinic (Kyanite)



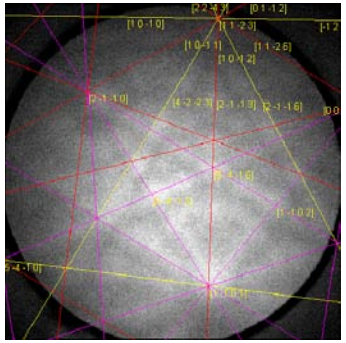
7 Crystal Symmetry Systems



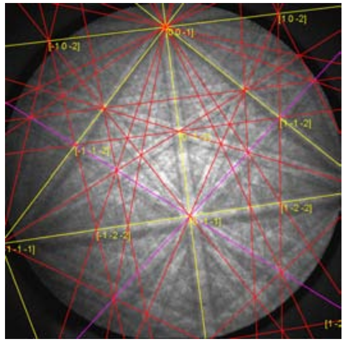
Cubic



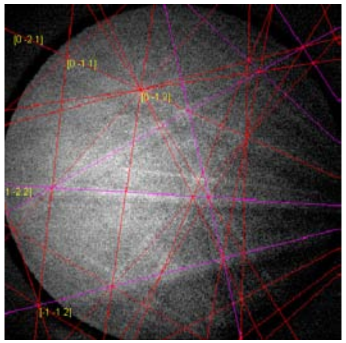
Hexagonal



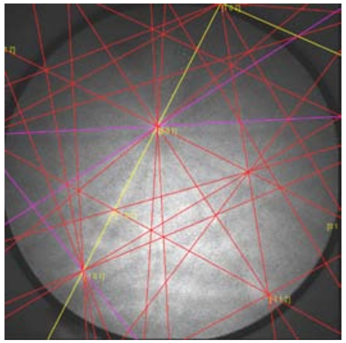
Trigonal



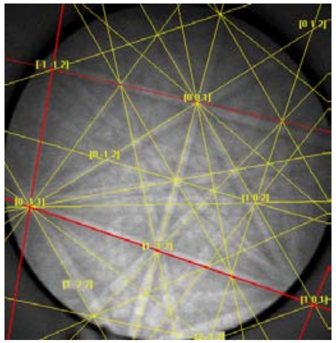
Tetragonal



Orthorhombic



Triclinic



Monoclinic

- Examples of all seven crystal systems solved

Summary of Factors Affecting Pattern Quality

Acquisition conditions affect the EBSP:

- Band width varies with kV
- Band contrast varies with kV
- Band sharpness varies with kV
- Camera Sensitivity (‘binning’ level/resolution)
- Background removal
- Integration time
- Beam current (spot size) & current density
- Vacuum level
- Microscope resolution/aberrations

Sample conditions affect the EBSP:

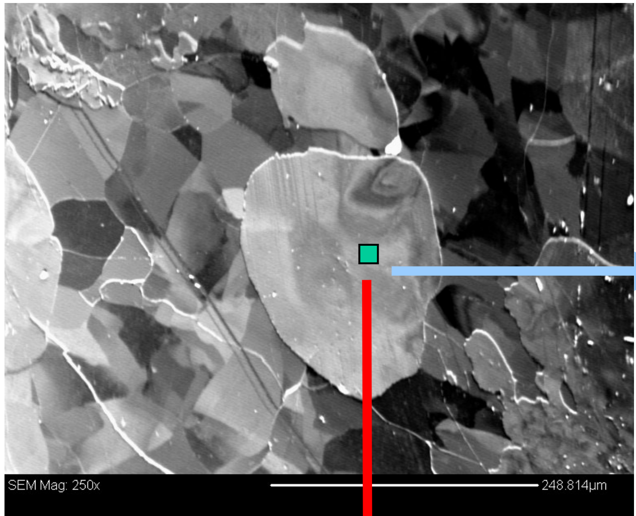
- Atomic number
- Grain size
- Preparation damage/residues
  - surface contamination e.g. carbon from SEM
- Surface oxidation or reaction products
- Surface morphology
- Surface coatings
- Amorphised surface - ion milling

The Hough Transform can be configured to optimize band detection for a wide range of operating and sample conditions...

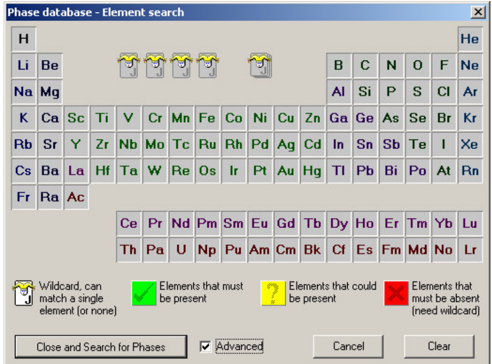
Basic Modes of Operation

1. Point Analysis
2. Mapping

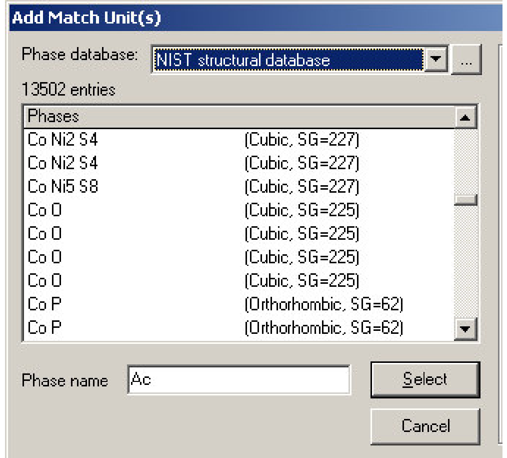
Phase Discrimination

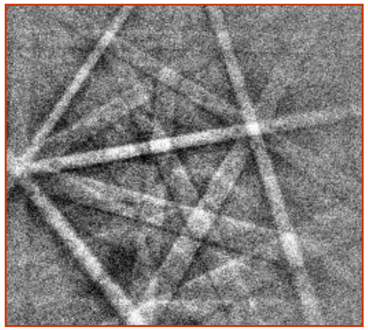


Acquire Chemistry

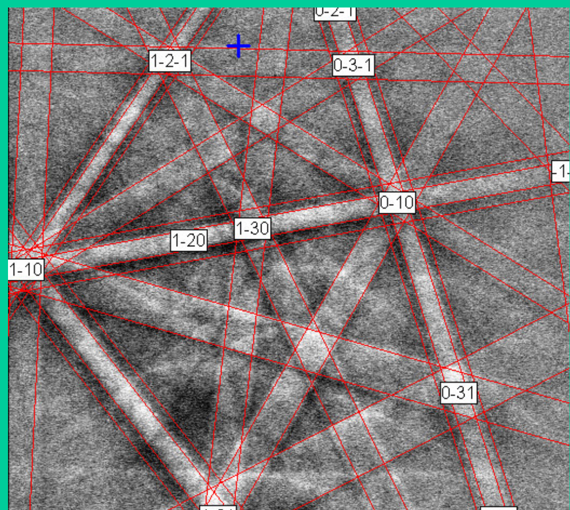


Search Structural Database





Acquire EBSP



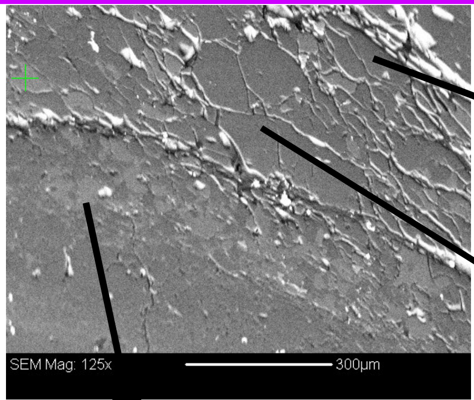
Index...

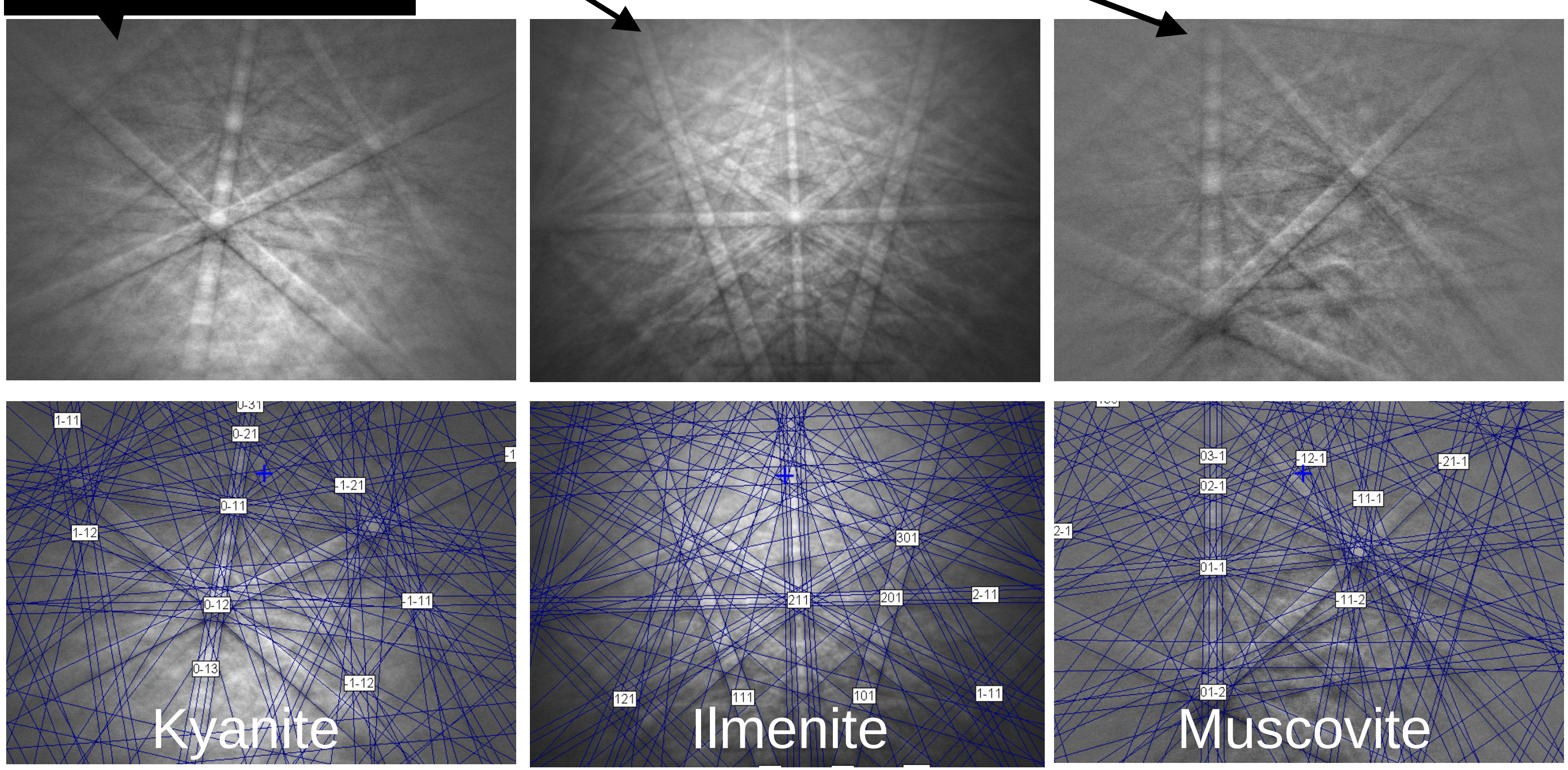
Solutions:

Phase	MAD	Bands
Aluminium	0.0521	6
Al As2 Cs3	1.9693	6
Al As2 Cs3	1.9697	6
Al As2 Cs3	1.9829	6

Phase Identified

Phase Discrimination by EBSD



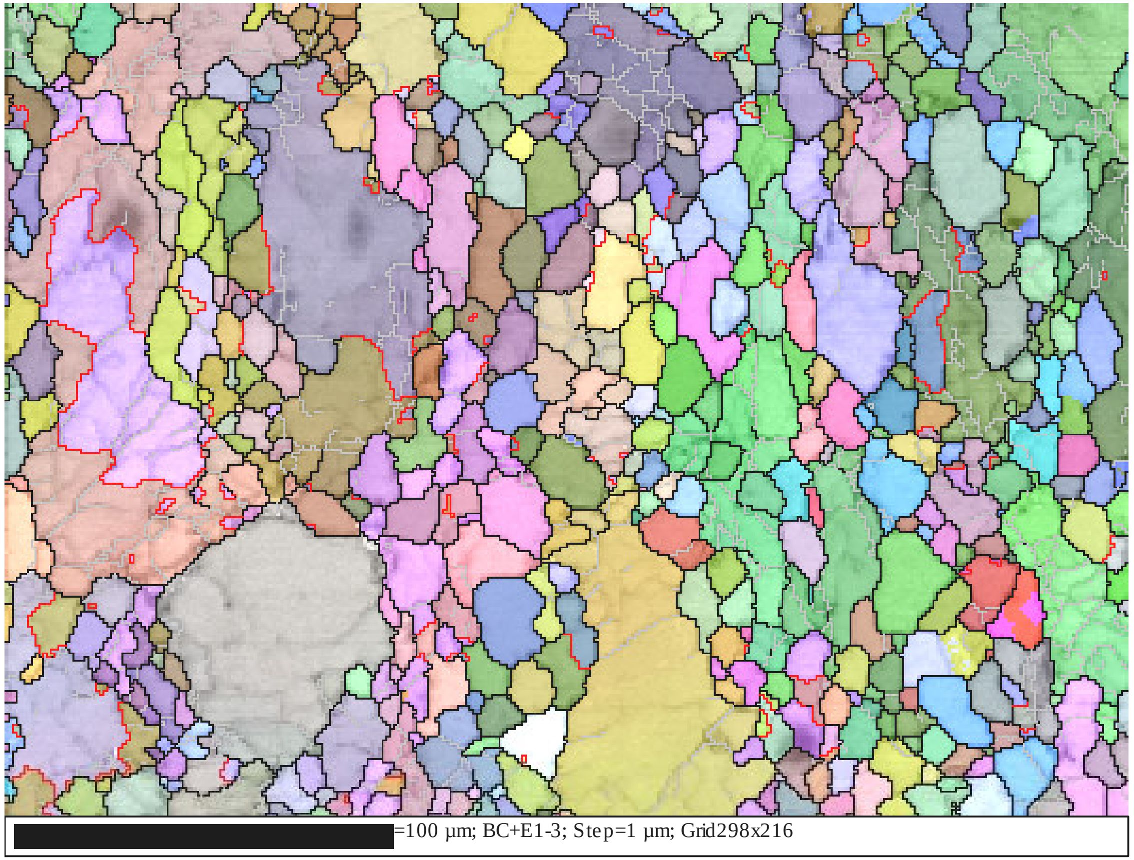


Mapping

General Examples

Visualisation of data - general microstructure

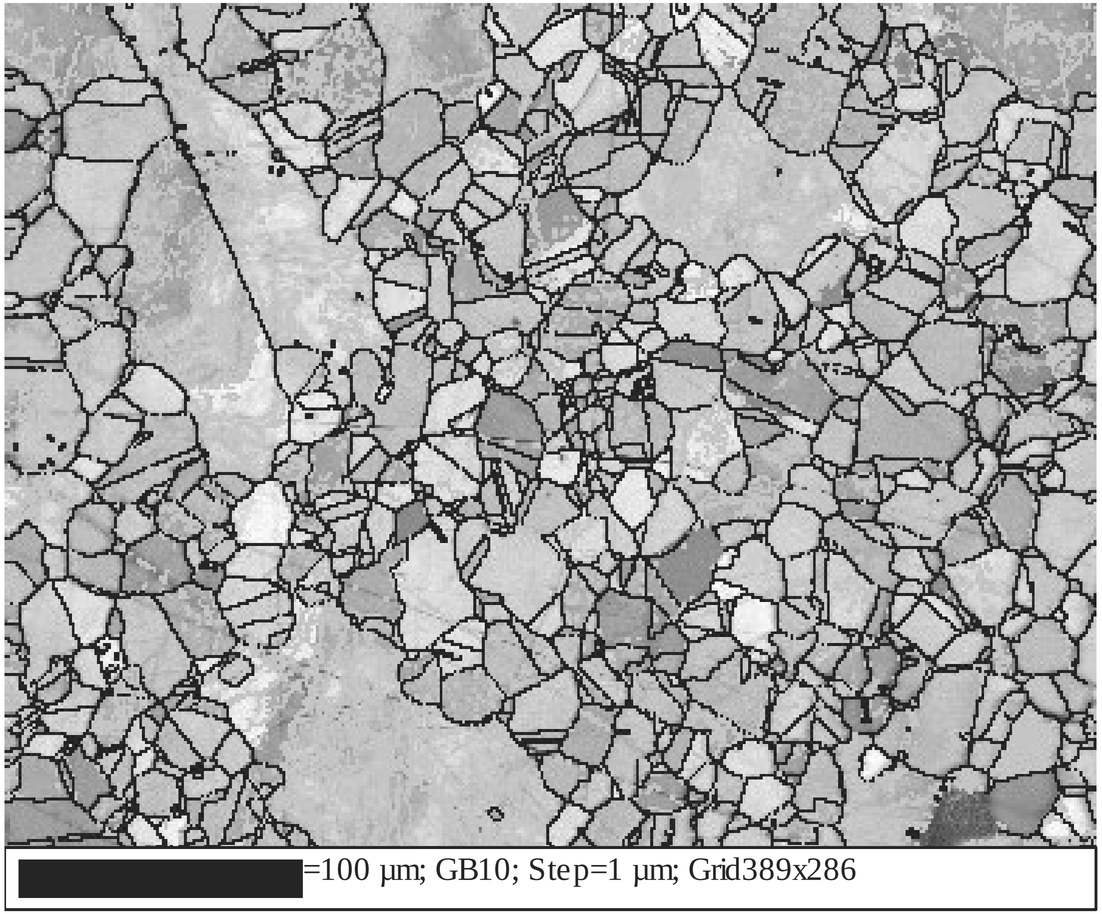
Quartzite



Orientation map + pattern quality + grain boundaries

Map Display Options

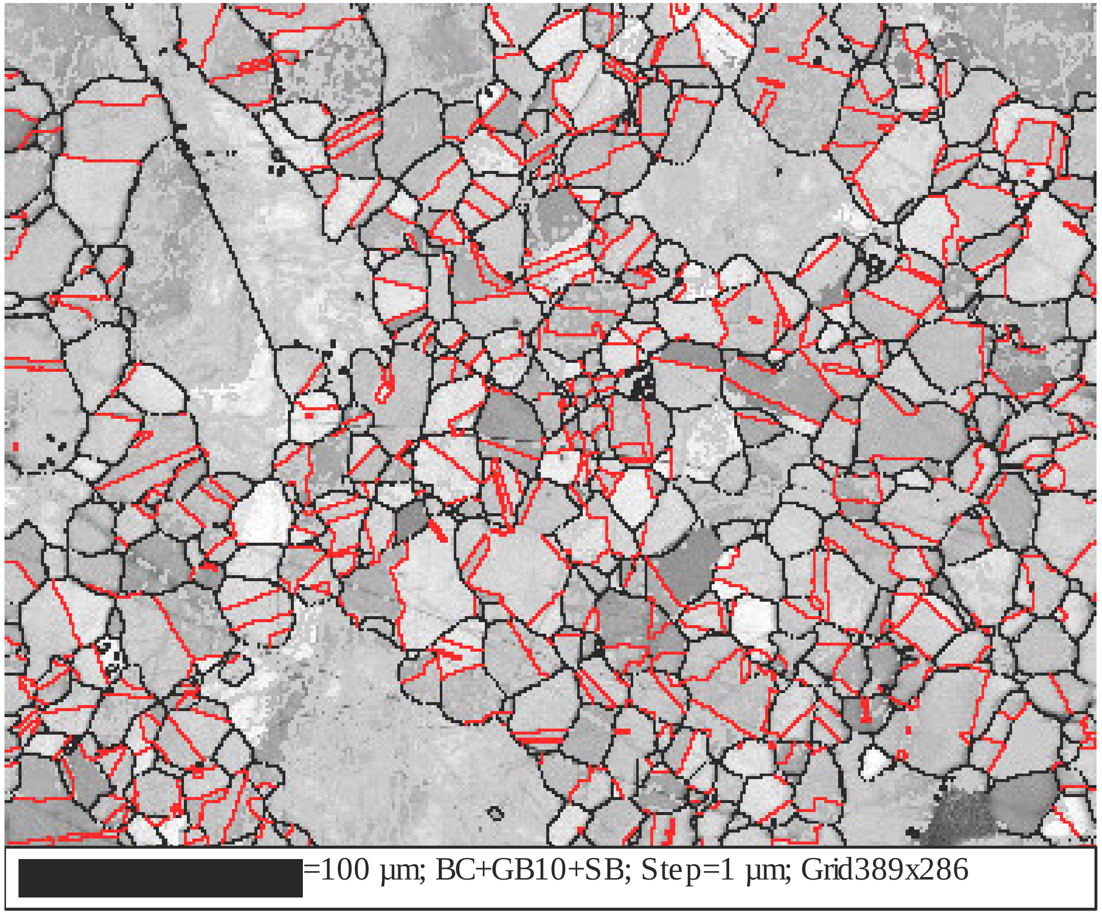
Ni Superalloy



Band Contrast + Grain Boundaries (high angle – general)

Map Display Options

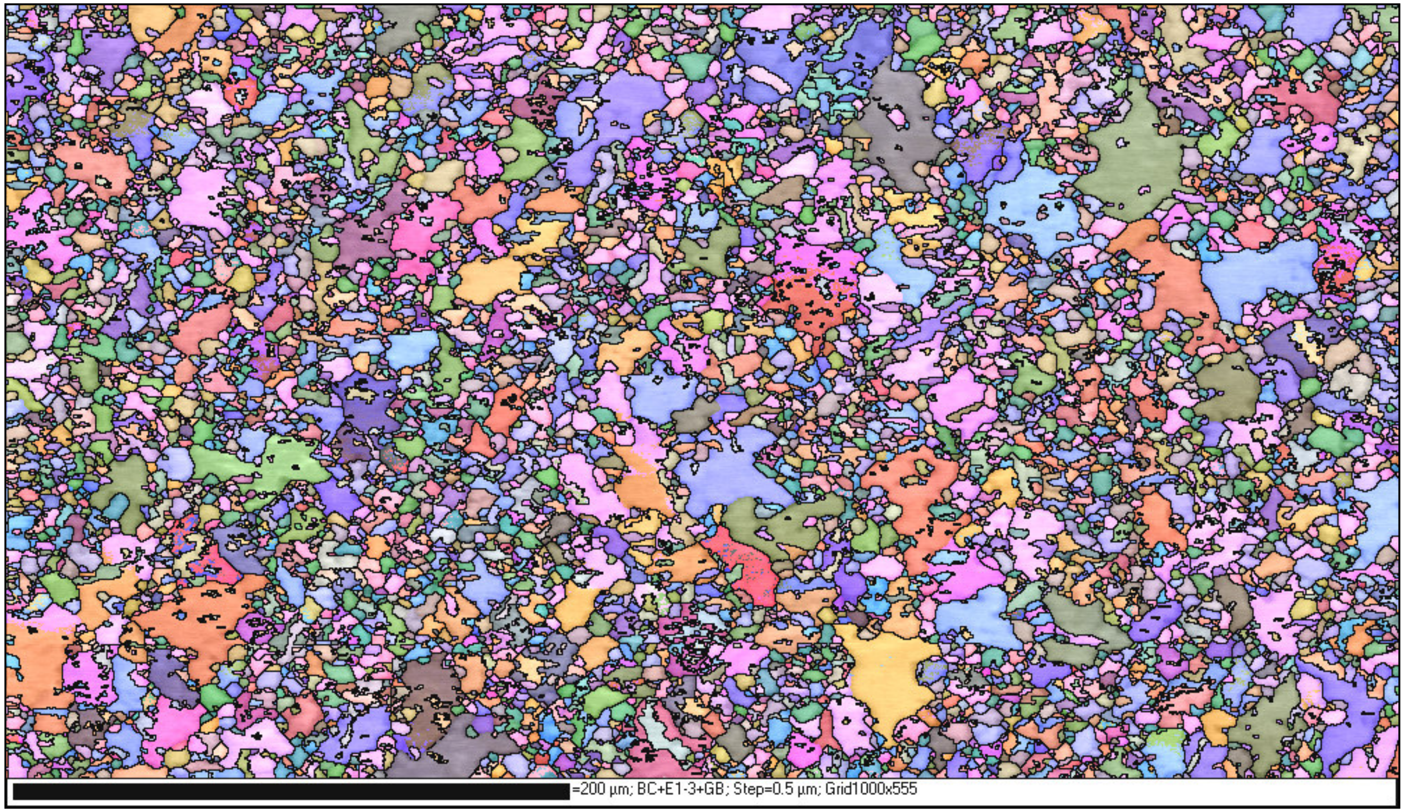
Ni Superalloy

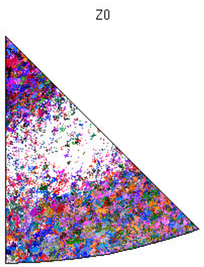


Band Contrast + Grain Boundaries + Twins/CSLs

Map Display Options

Cu thin film

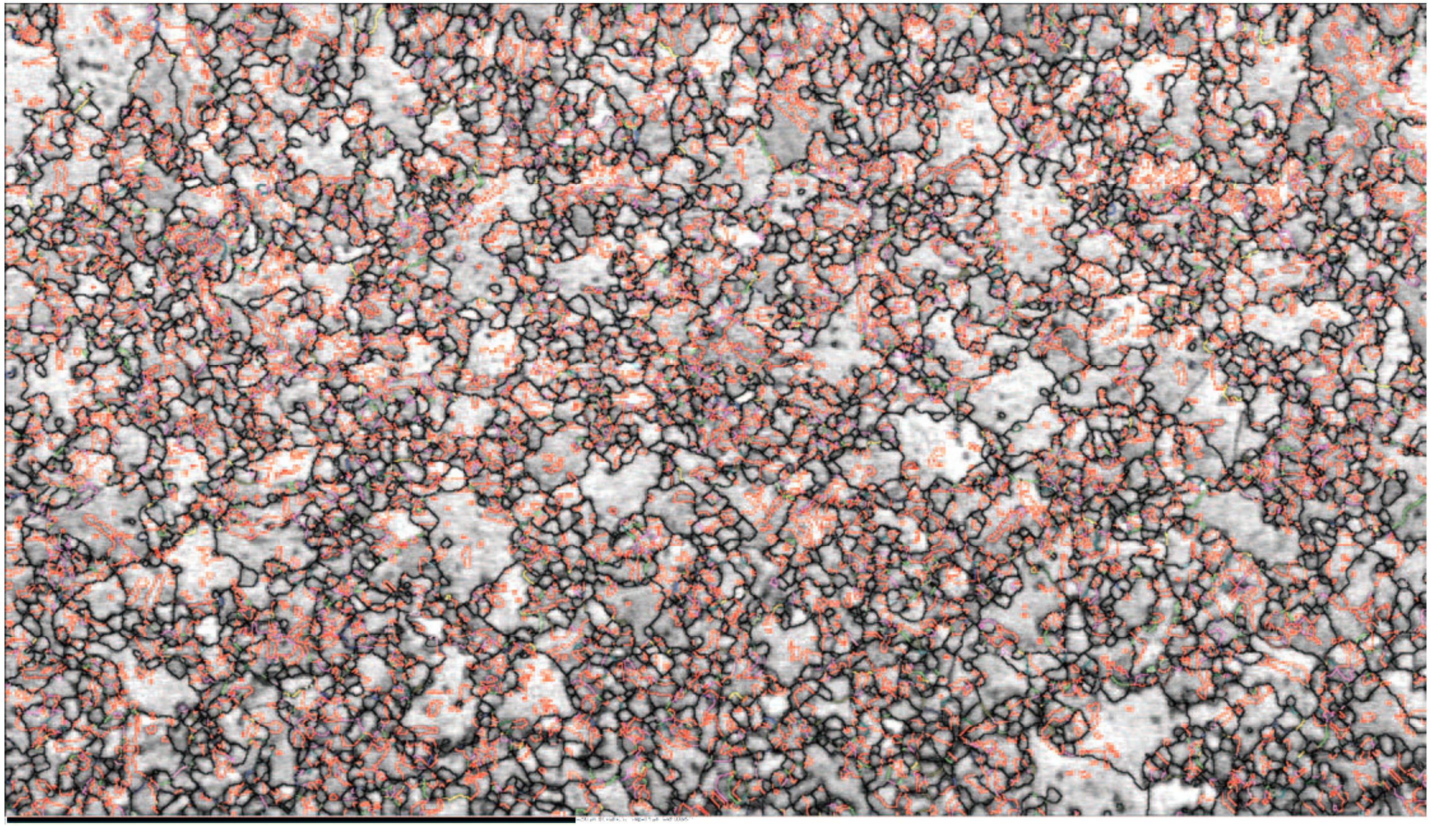




All Euler orientation + pattern quality map

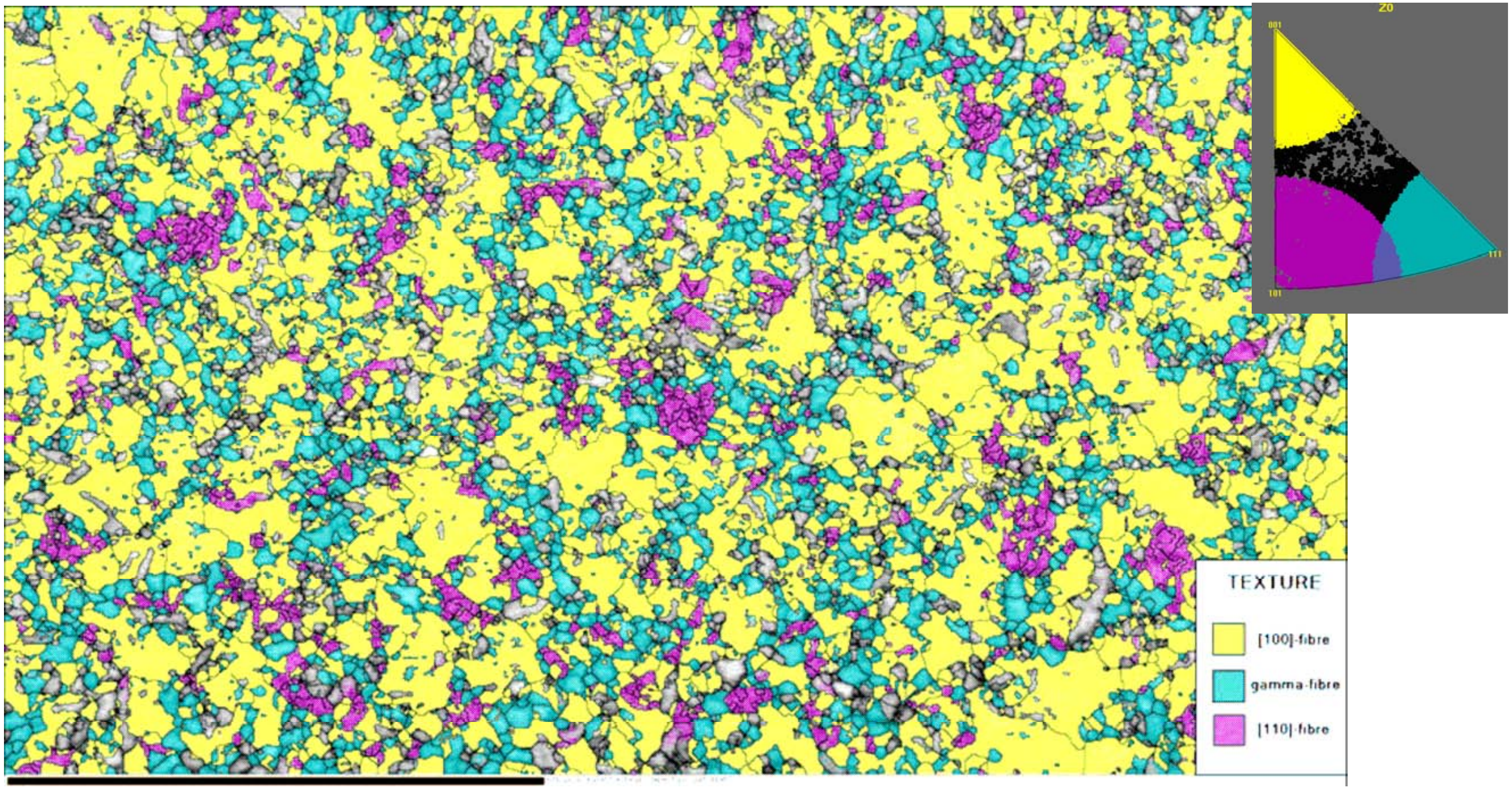
Map Display Options

Cu thin film



Band contrast + GB map

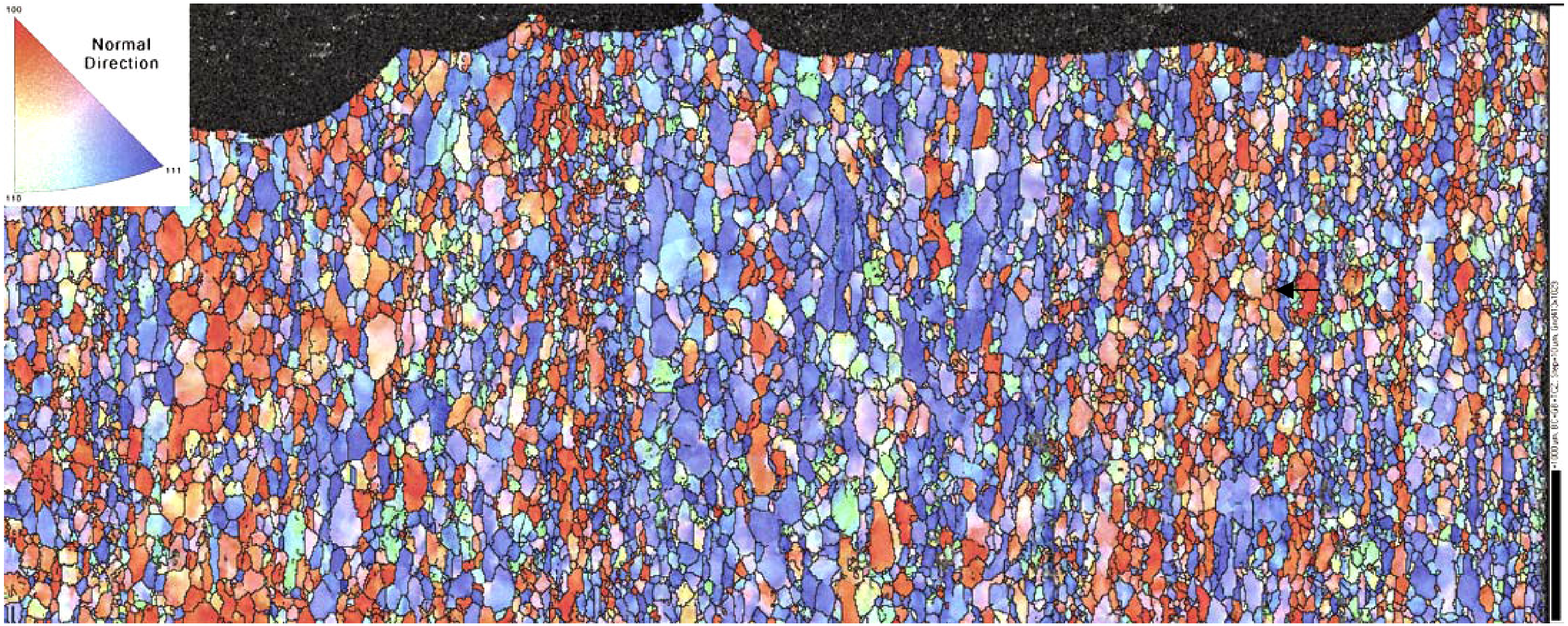
Map Display Options



Grouped orientation map

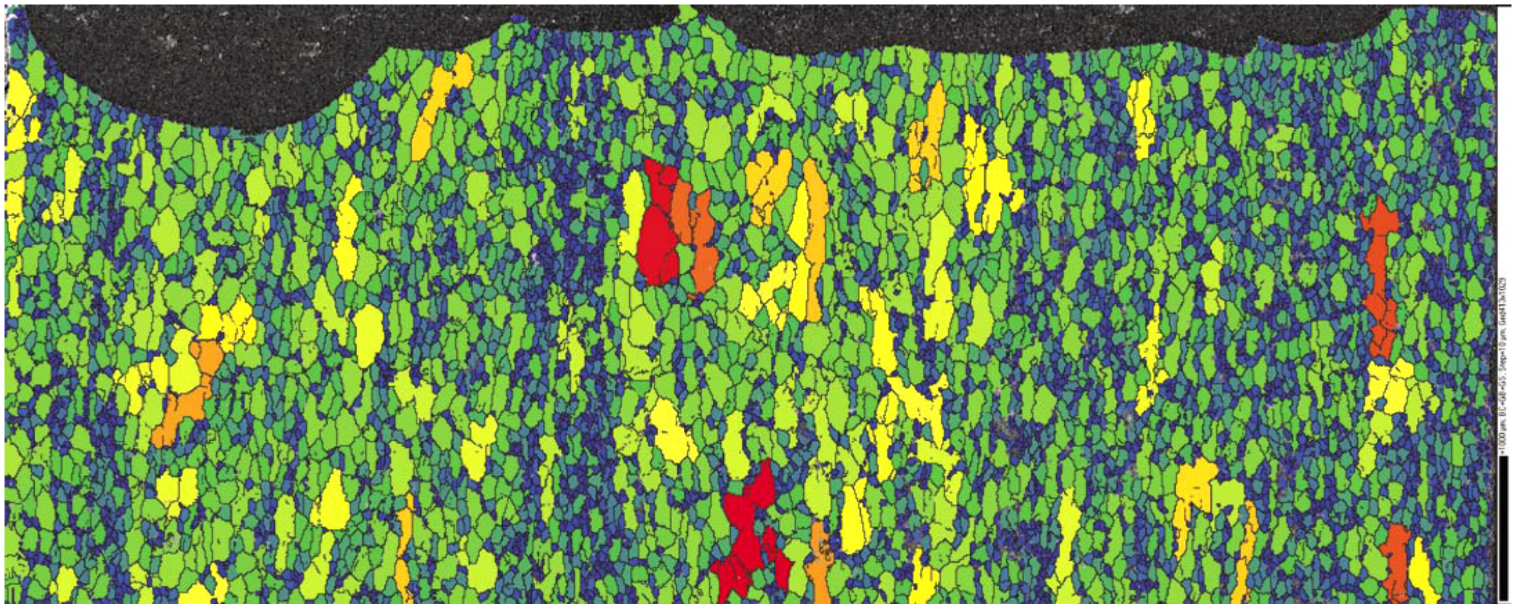
Map Display Options

Ta sputter target

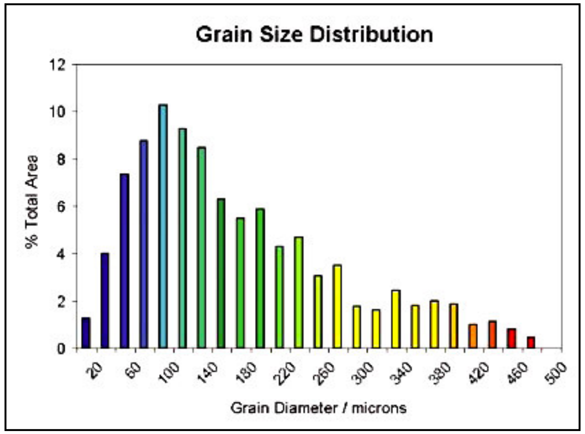


IPF-based orientation + pattern quality map

Map Display Options



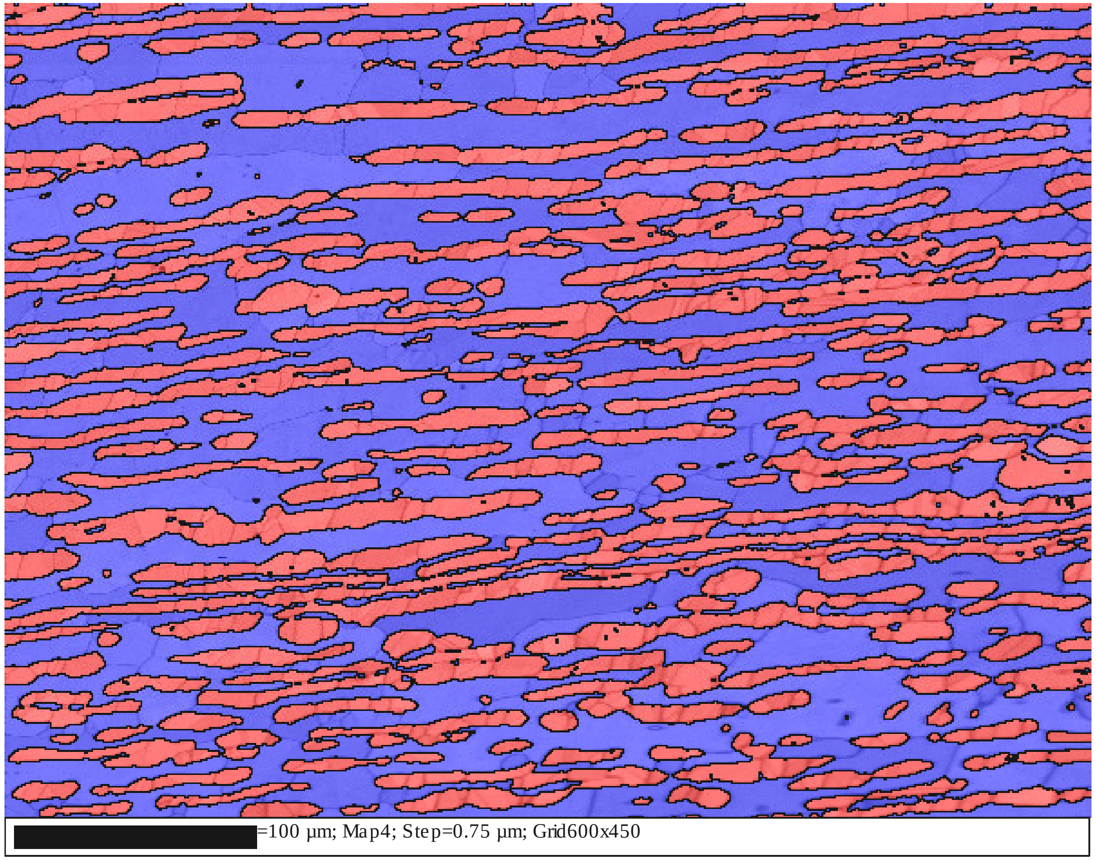
Map with grain size coloring scheme

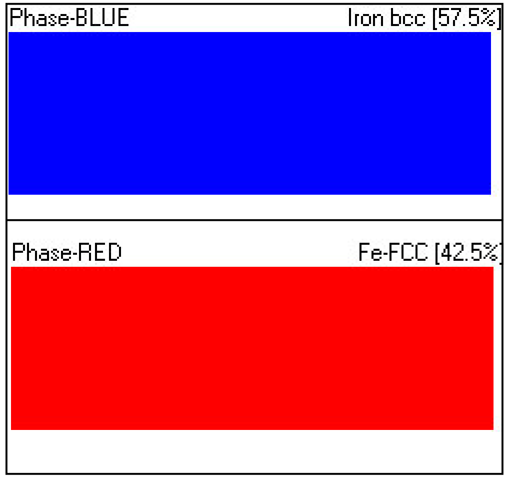


Grain size distribution

Phase Area Fraction & Distribution

Duplex Steel



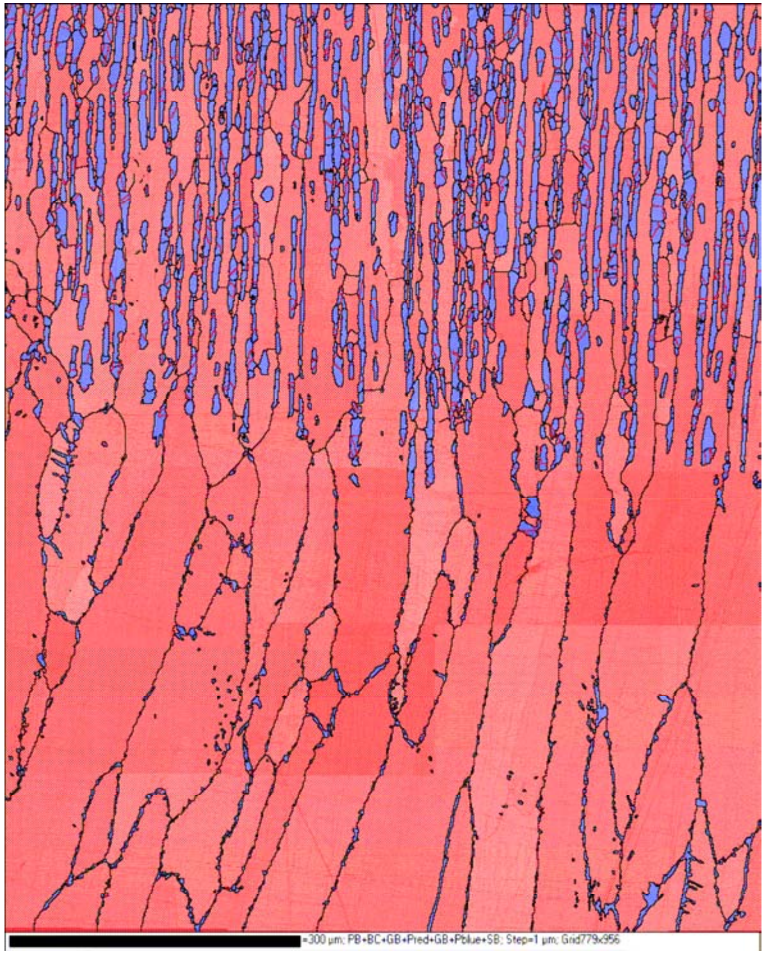


Statistics

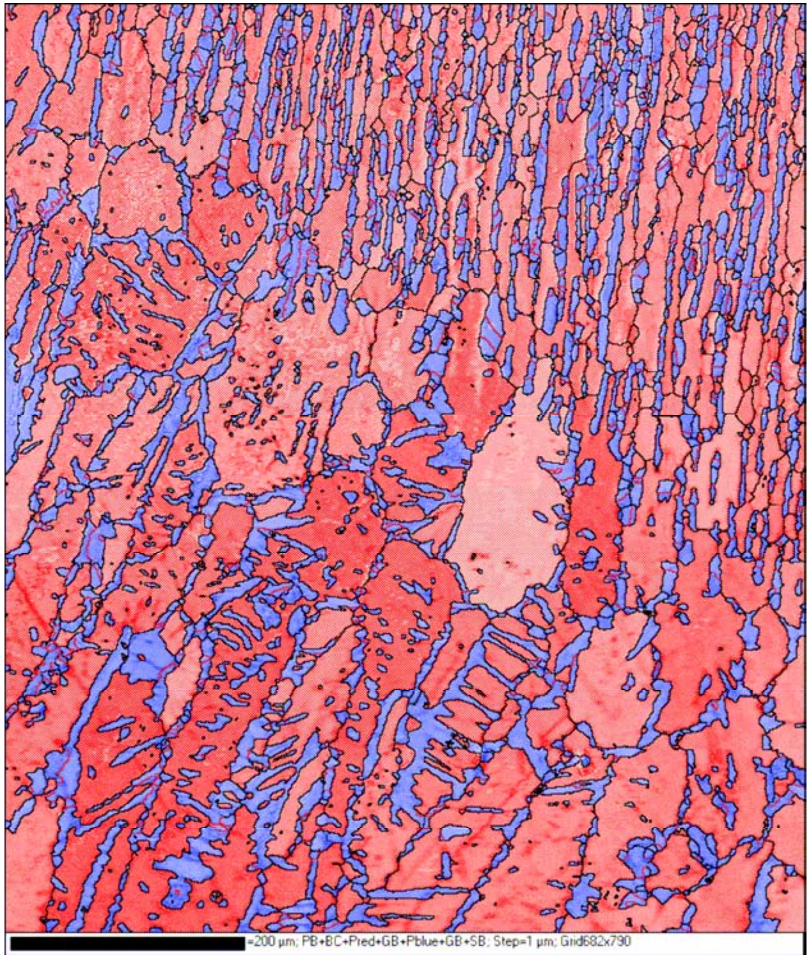
Phase	%	mean BC	mean BS	mean MAD
Zero solutions	0	n/a	n/a	n/a
Fe-FCC	42.48	167.6	86.33	0.5486
Iron bcc	57.52	162.3	90.17	0.5348
Total	100	164.6	88.54	0.5407

Applications Example: Phase distribution & texture in laser welded duplex steel

Non-preheated

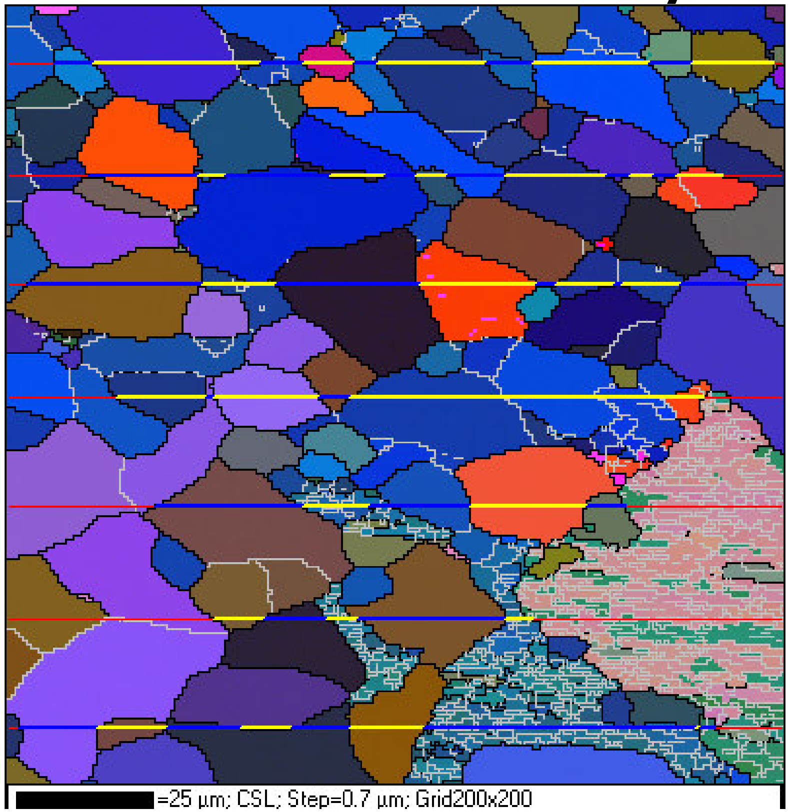


Preheated

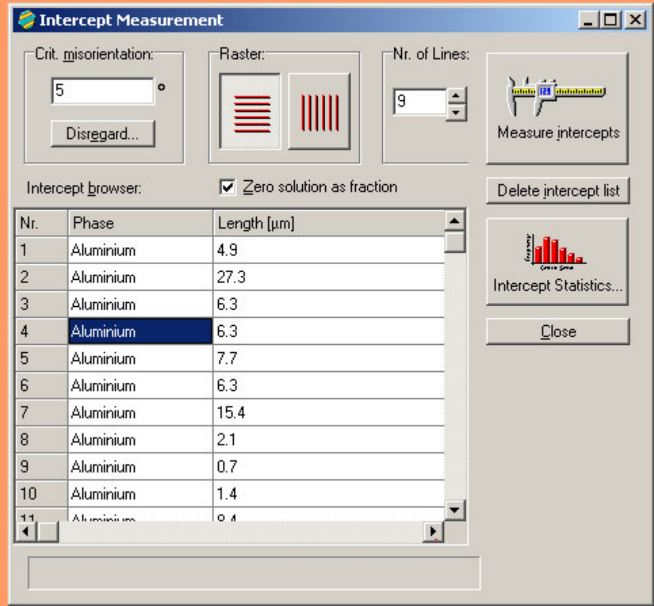


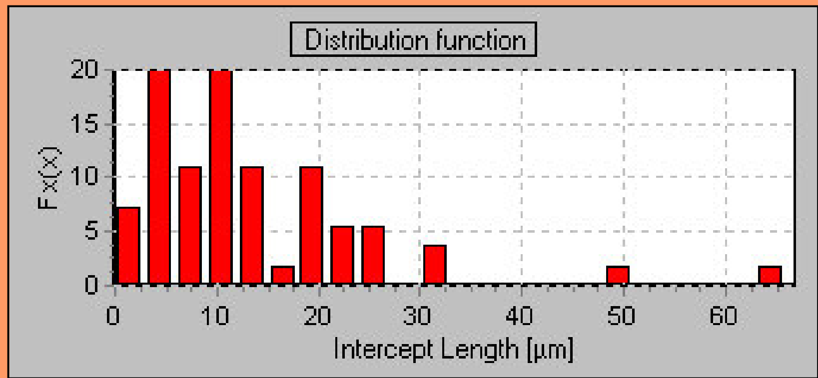
Phase maps (+ GBs): red = ferrite, blue = austenite

Grain Size Analysis: Line Intercept Method



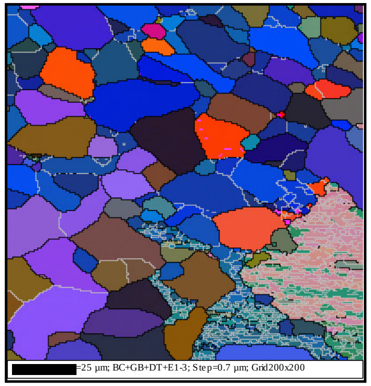
Unlike optical line-intercept GS analysis, twin boundaries and low-angle grain boundaries are positively identified and filtered from the data (if desired); tricky GB etch techniques not necessary; weaknesses of relying on reflected light orientation contrast are avoided



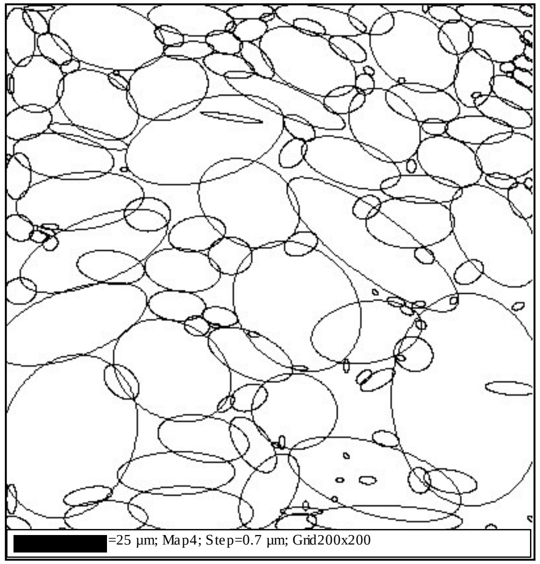


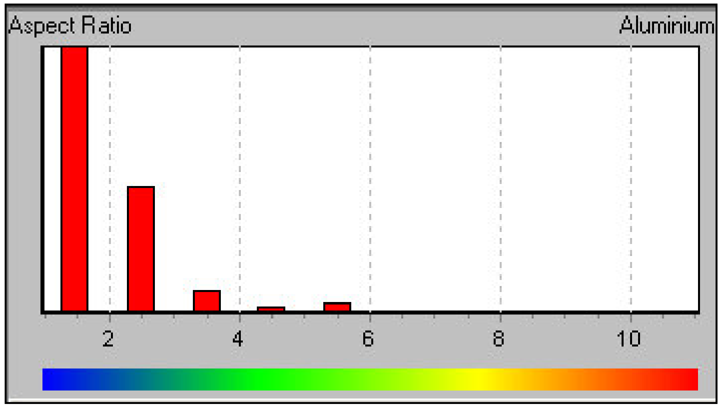
Data available as text and in graphical format

Grain Shape Analysis

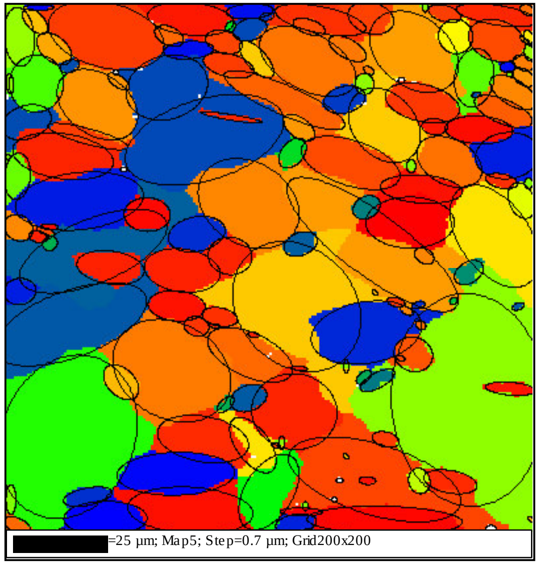


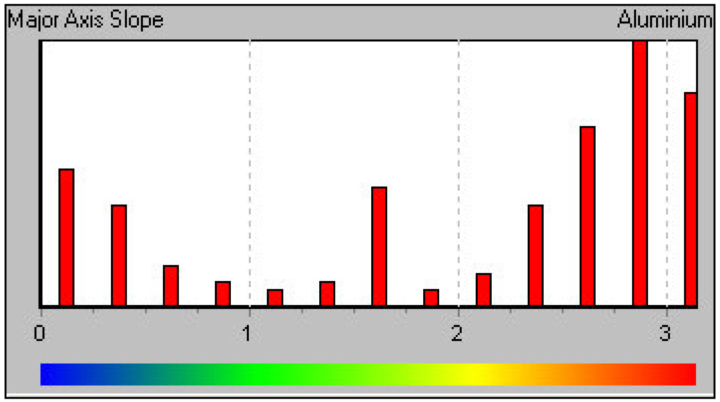
Aspect Ratios



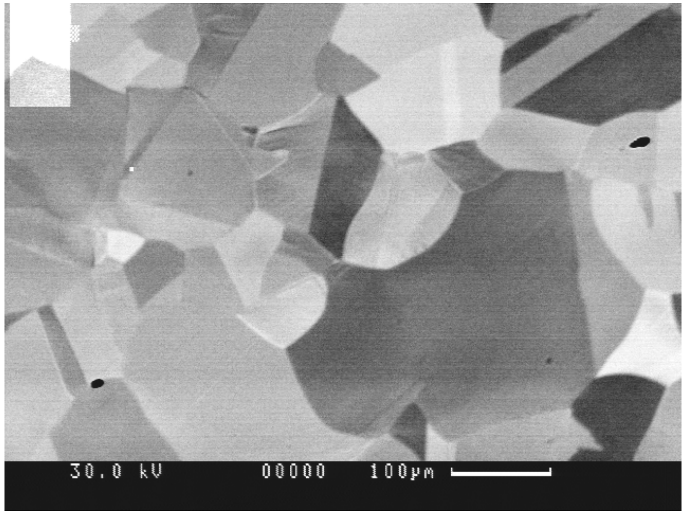


Major Axis Orientation

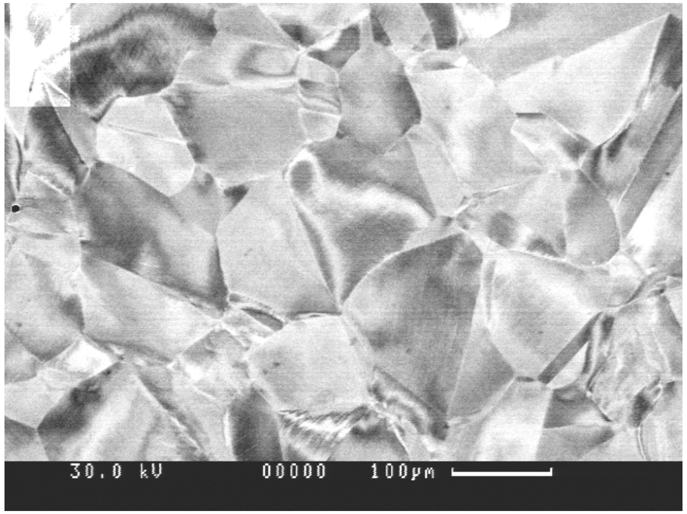




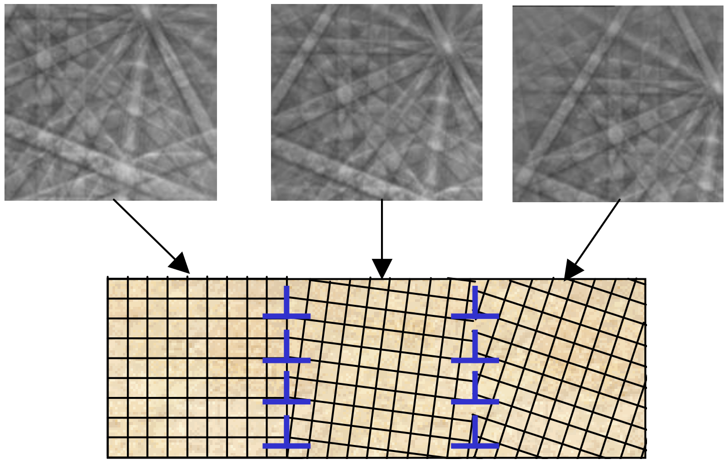
Analysis of strain



Recrystallized



Deformed

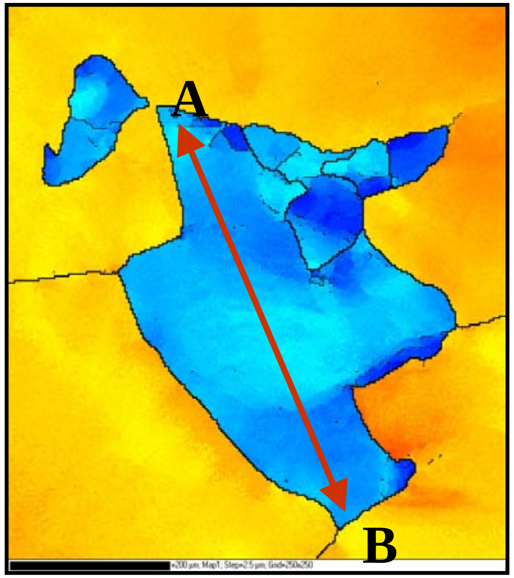


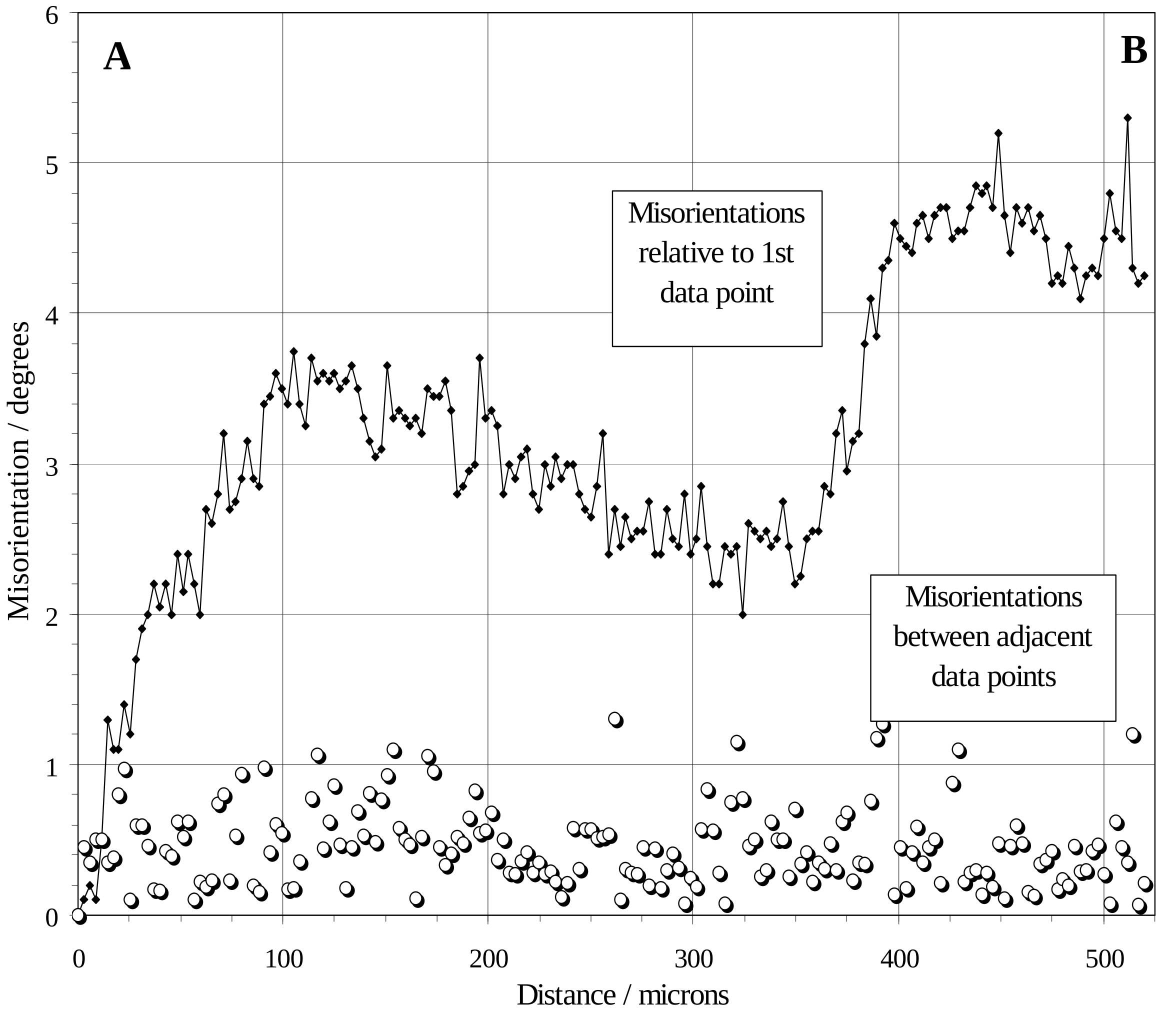
Plastic strain manifested at least in part as ordered dislocation arrays can cause semi-continuous lattice rotation within a grain. This "intragranular misorientation" is measurable. Random or disordered dislocations only result in pattern quality degradation

Images courtesy L.N. Brewer, M.A. Othon (GE GRC)

Analysis of Strain - Intra Grain Misorientation

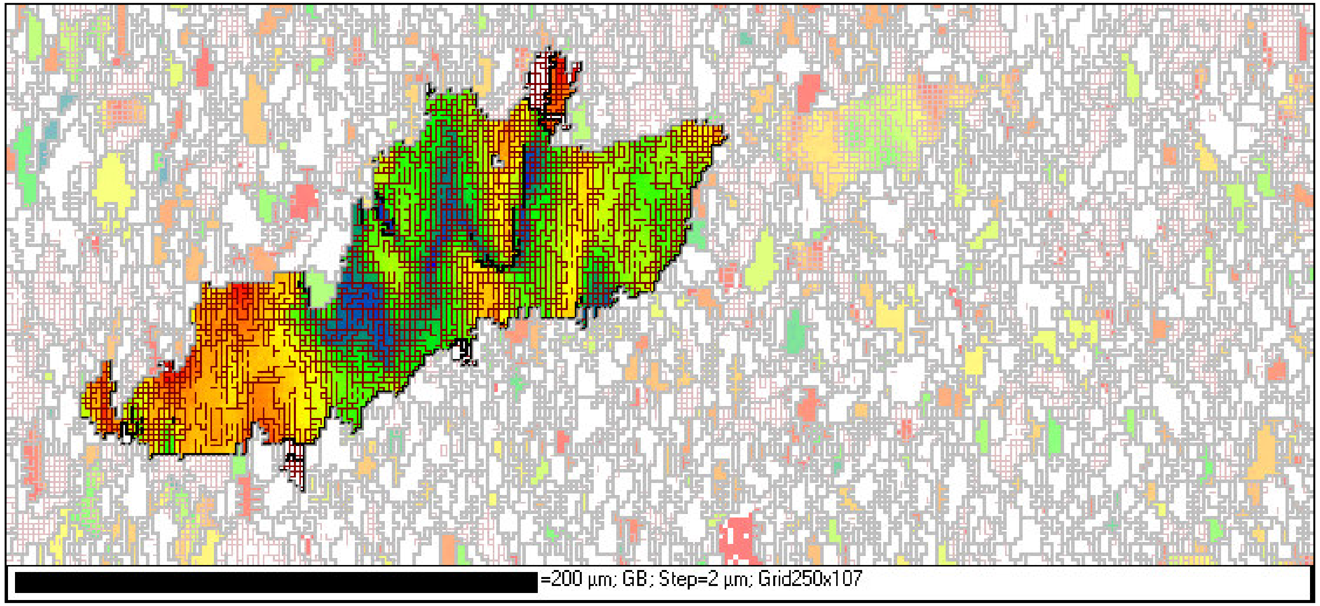
Halite

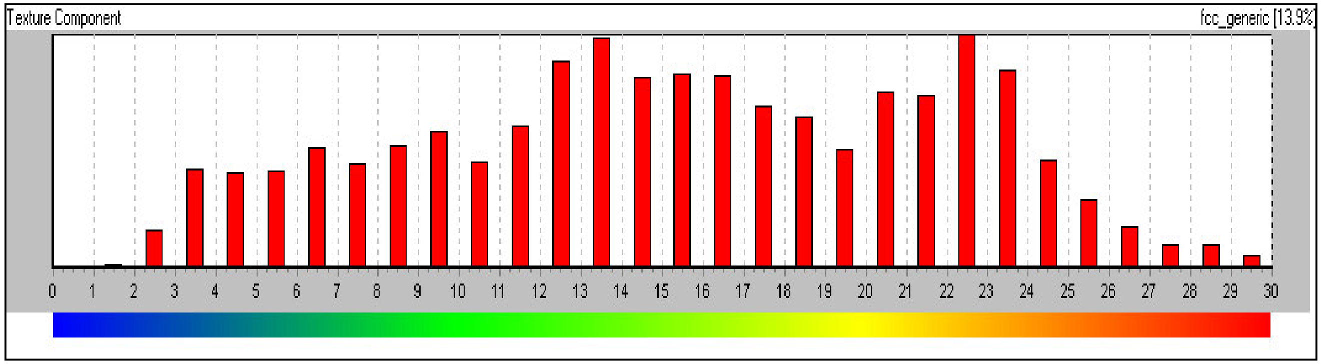




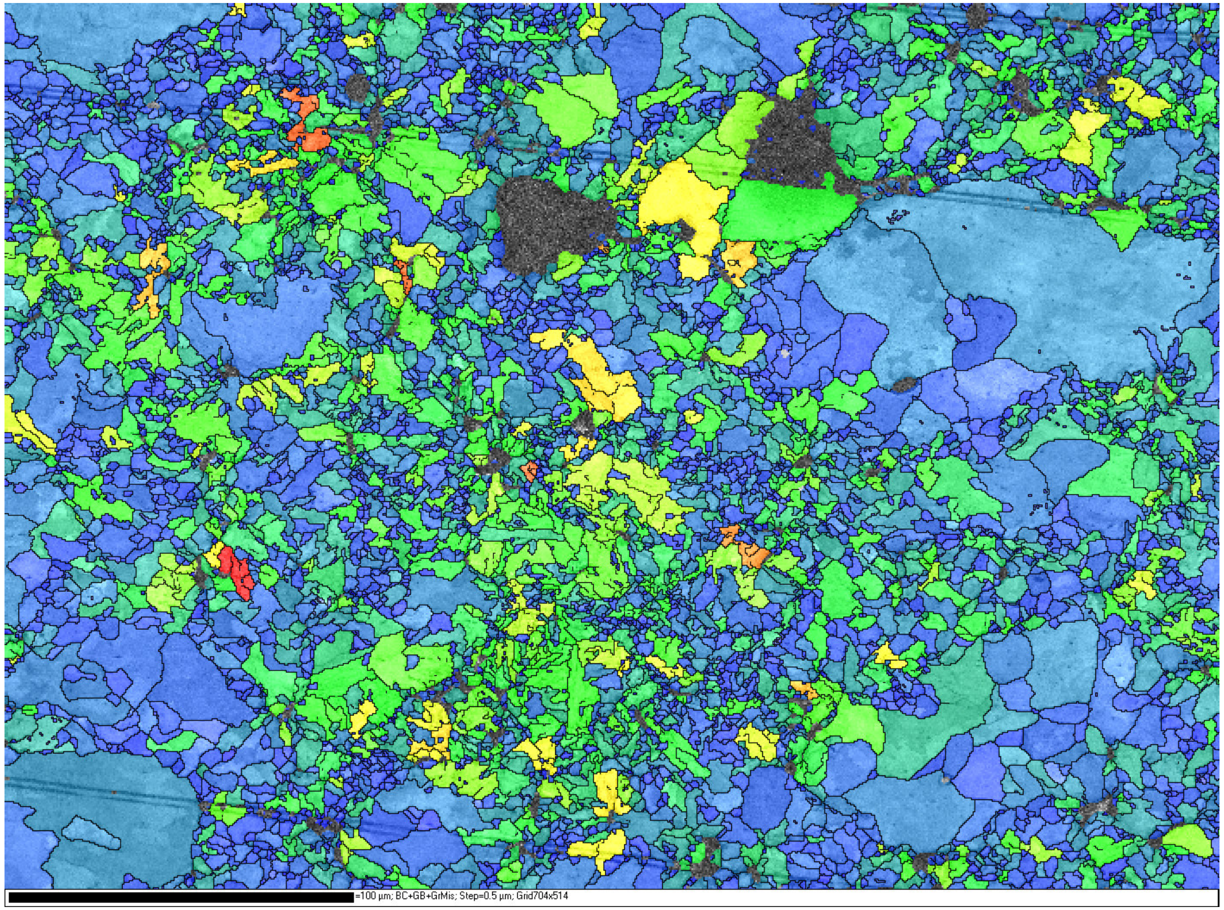
Analysis of strain

Deformation-induced misorientation within large grain in Ni-base superalloy





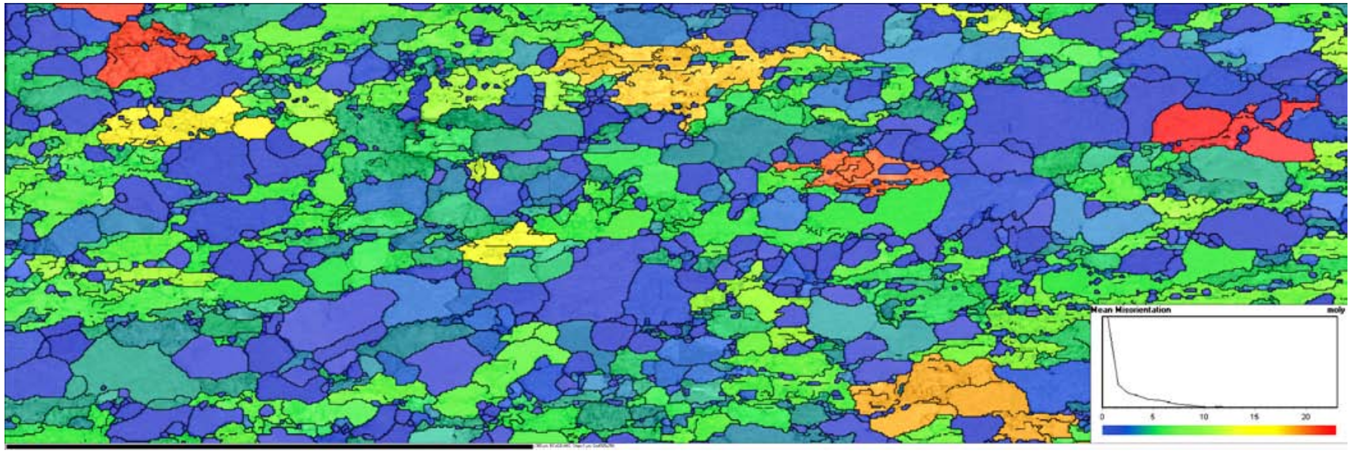
Analysis of strain



copper

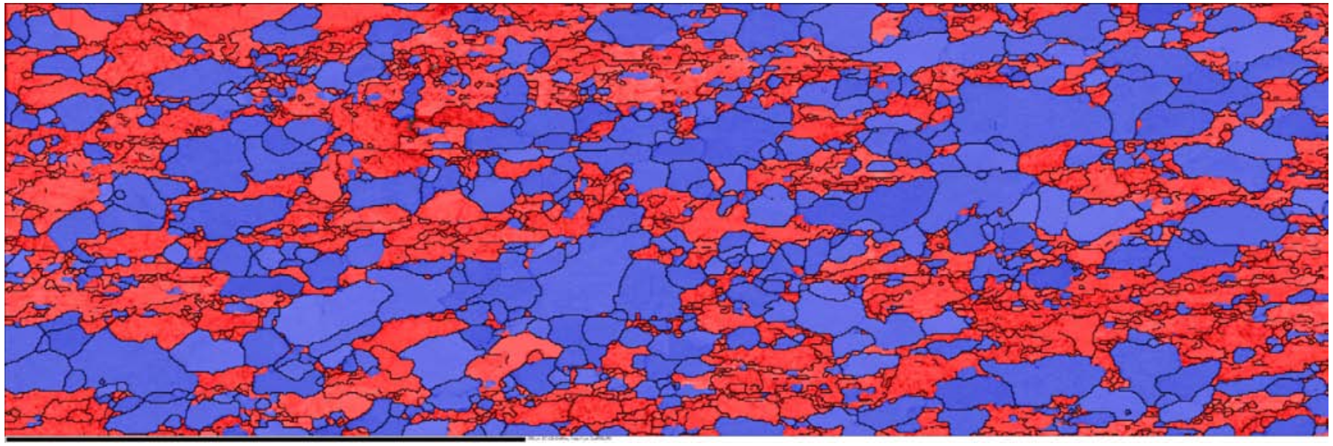
- Isolate grains by strain

Segmenting data by strain state - Recrystallisation



Strain (grain average misorientation) distribution map

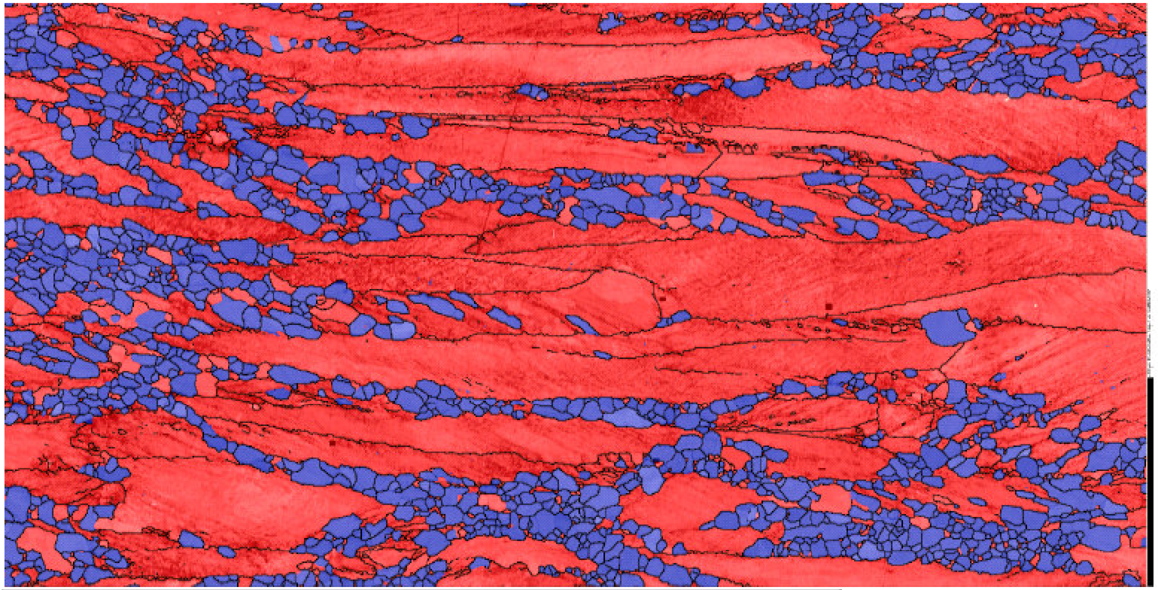
Rolled Mo Sheet



Strain (grain average misorientation) classification map

Red = deformed
Blue = Recrystallized

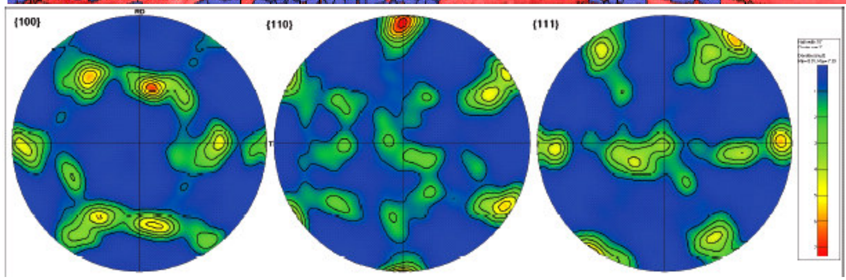
Segmenting data by strain state - Recrystallisation



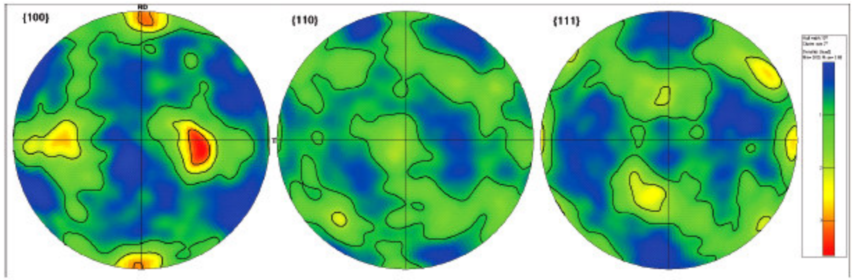
Rolling & heat treatment of Fe-Al binary alloy resulted in a partly deformed, partly recrystallized microstructure

Grains subgrouped by residual strain state:

Red deformed, blue recrystalllized



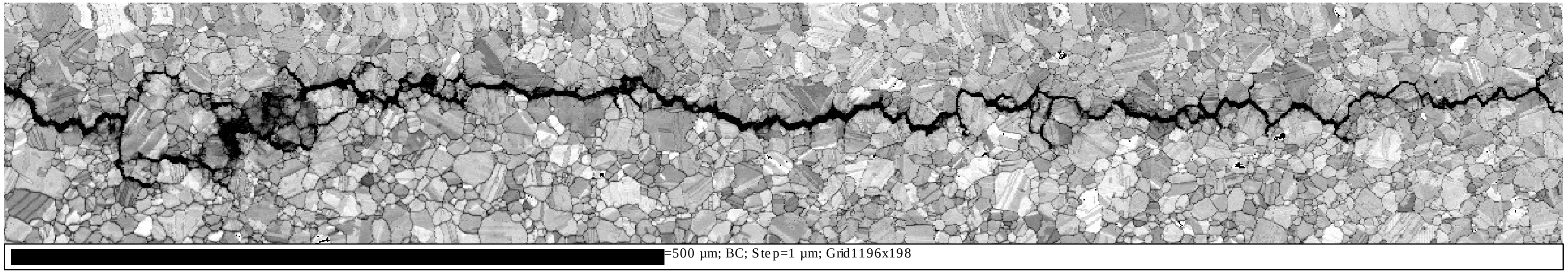
Deformed

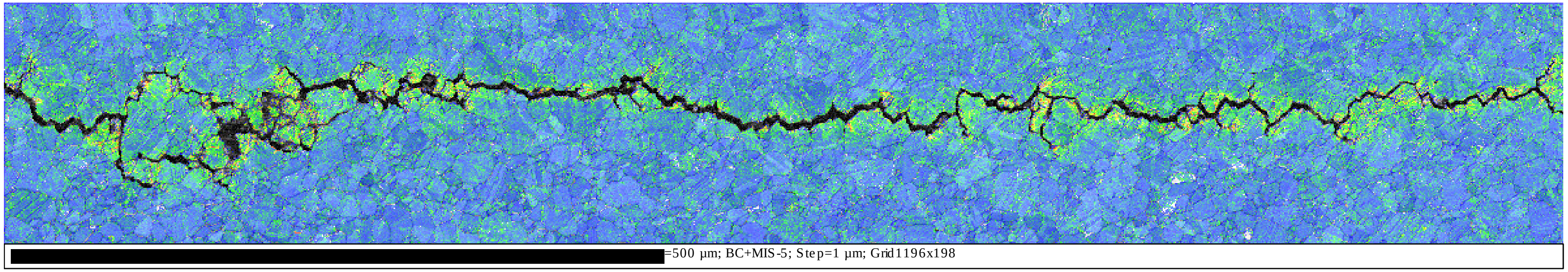


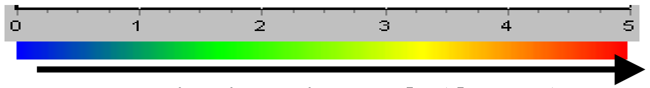
Recrystallized

Texture ‘partitioning’ revealed

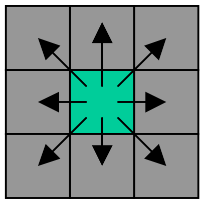
Visualisation of strain







Disorientation angle (degrees)

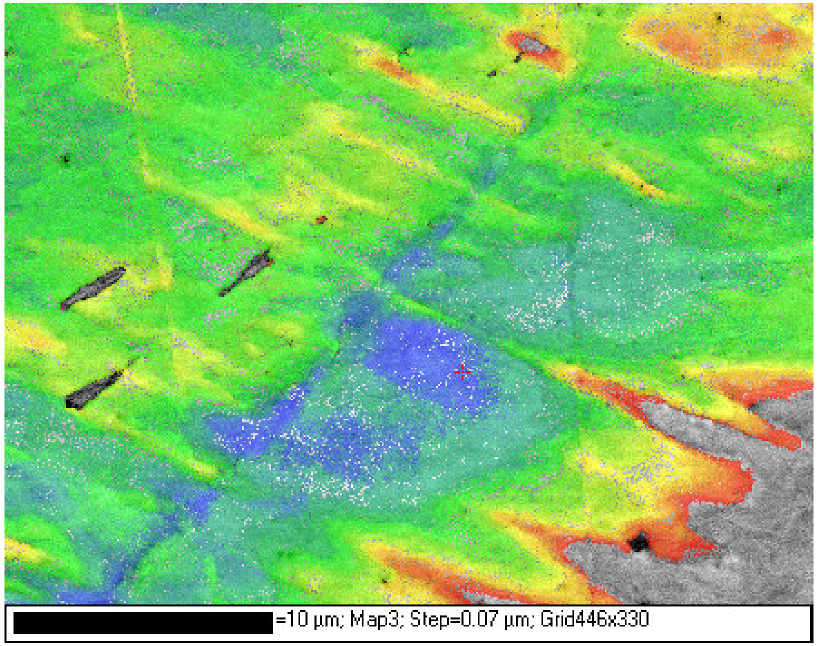


Each orientation is surrounded by 8 nearest neighbors. The disorientation is calculated through all of the 8 nearest neighbors to get an averaged value.

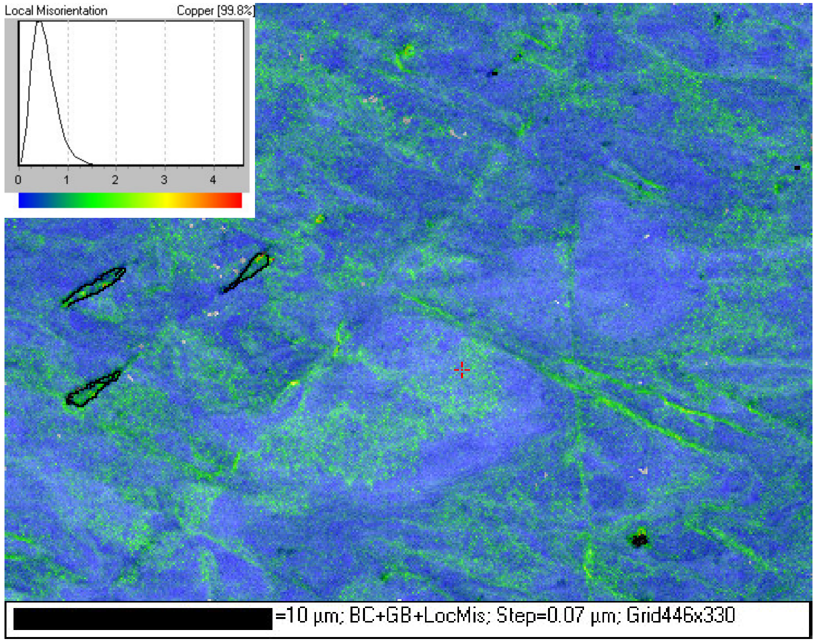
$$g_{ij} = \sum g_i \bullet g_j^{-1}$$

Visualisation of Strain

Cu



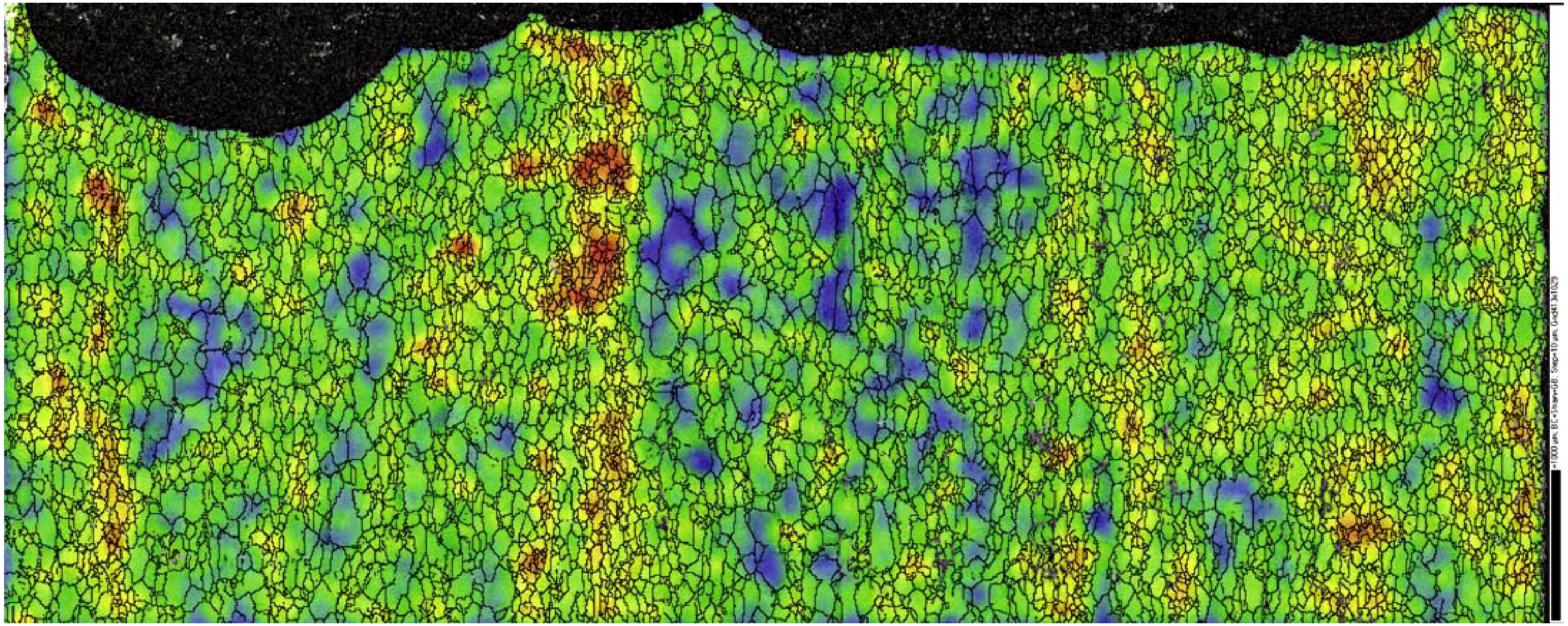
Relative to selected reference point (cumulative rotation)



For each pixel, relative to surrounding pixels (local rotation)

Visualisation of Strain

Ta sputter target



Strain localization + GB map

Texture Analysis

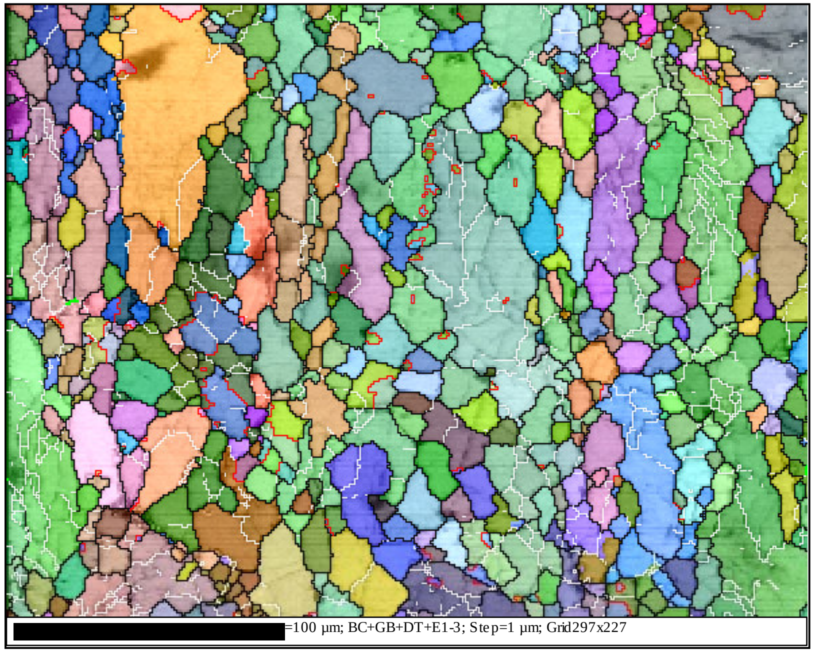
Pole Figures

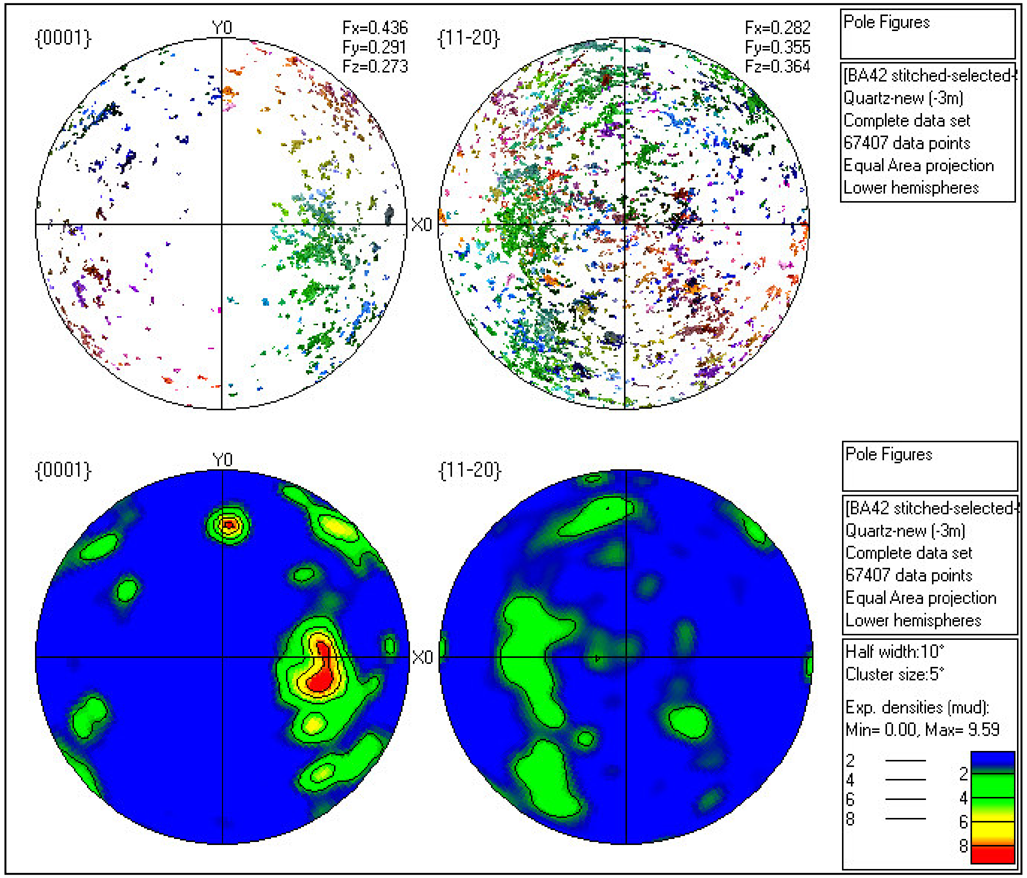
Inverse Pole Figures

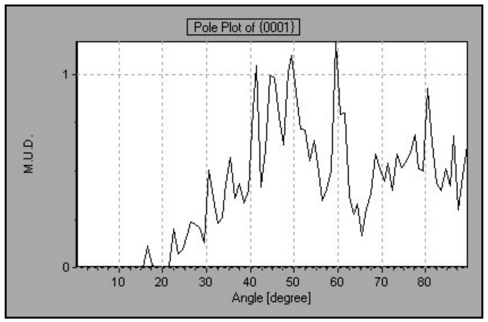
ODF

Texture Analysis: Pole Figures

Quartzite



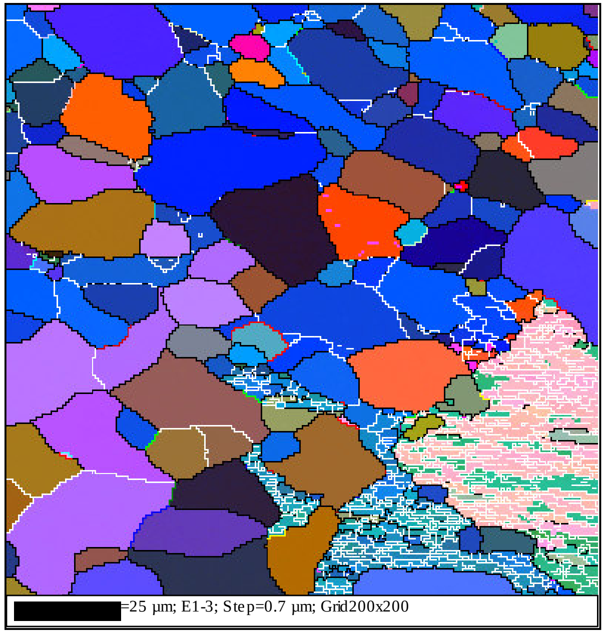


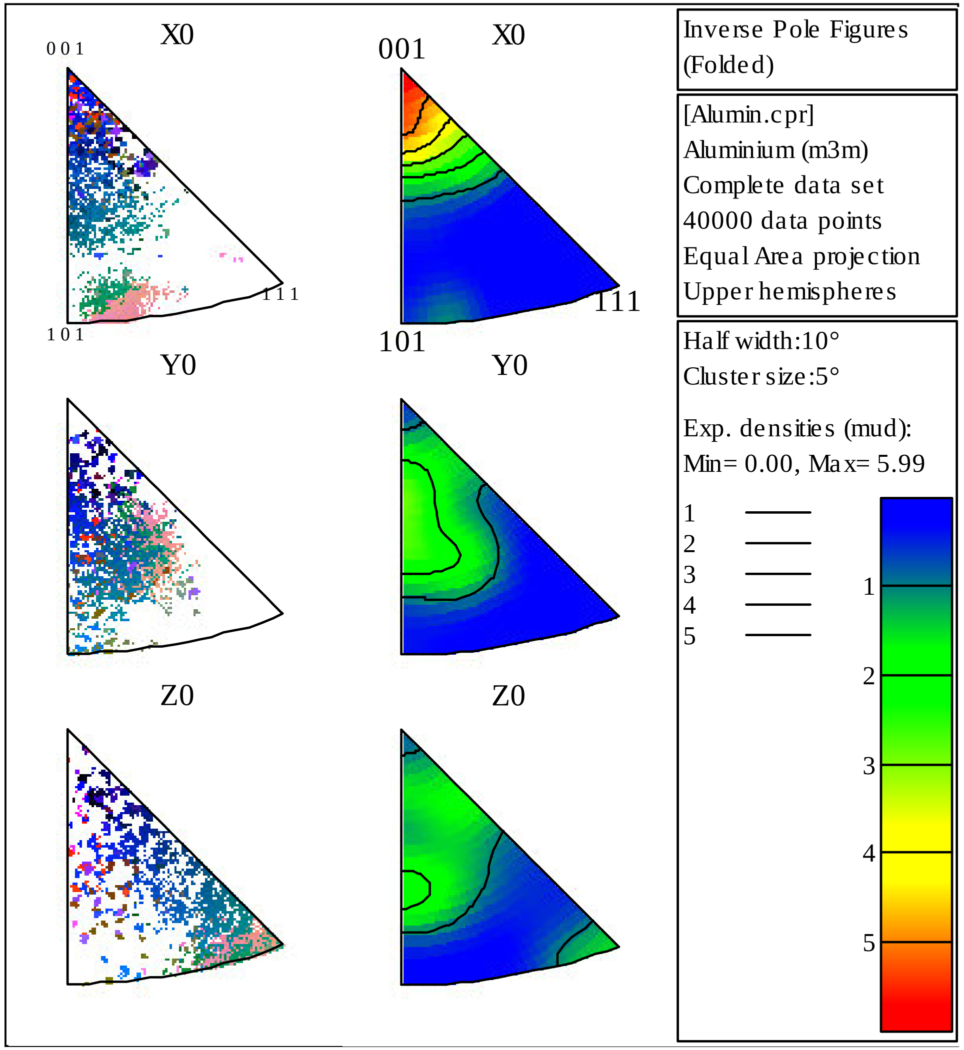


“Pole Plot” for {0001}

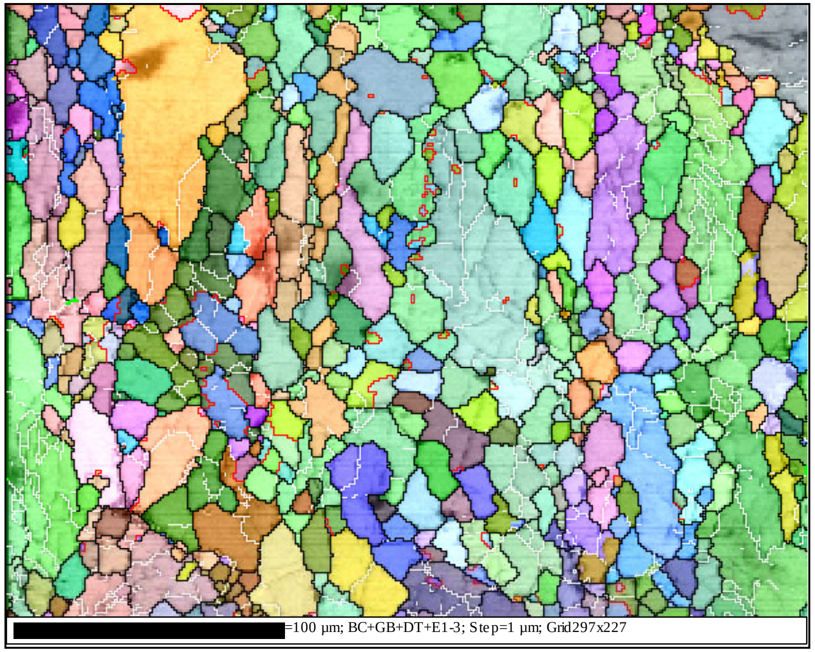
Texture Analysis: Inverse Pole Figures

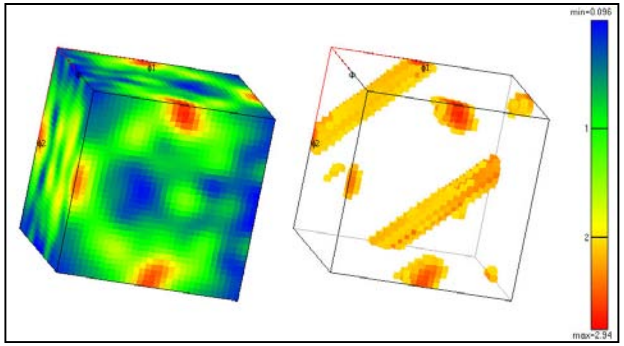
Rolled Al

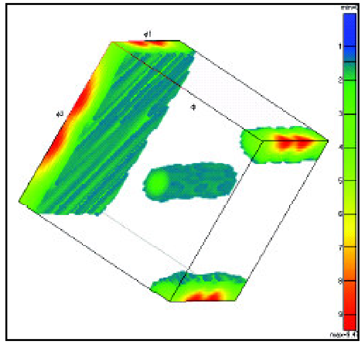


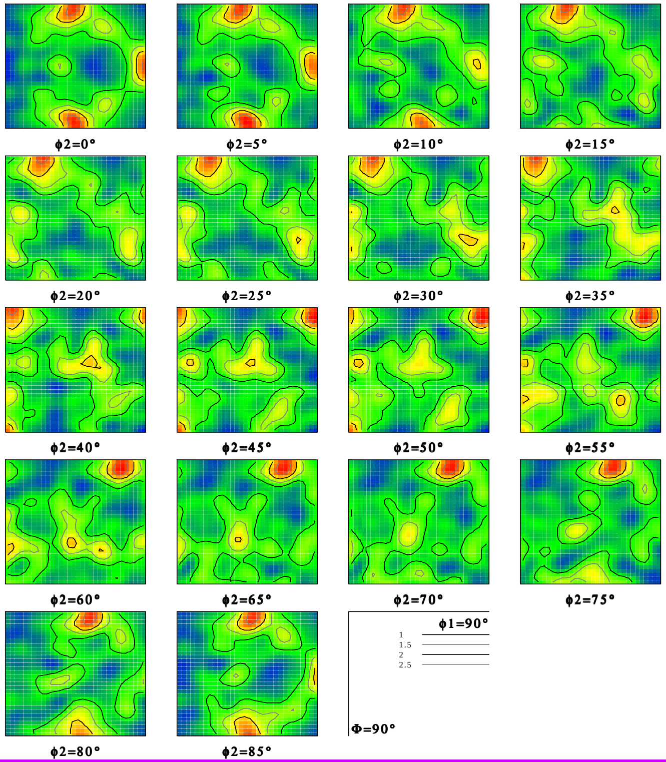


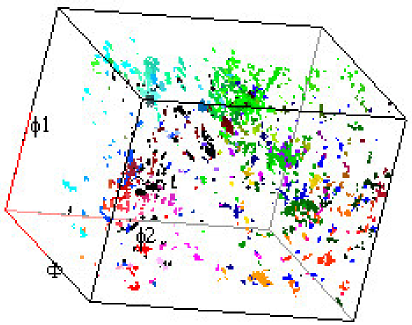
Texture Analysis: Orientation Distribution Functions (ODF)











Thank you