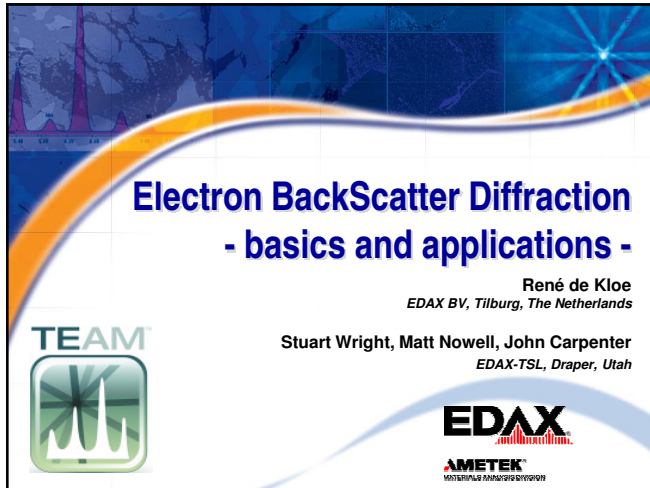
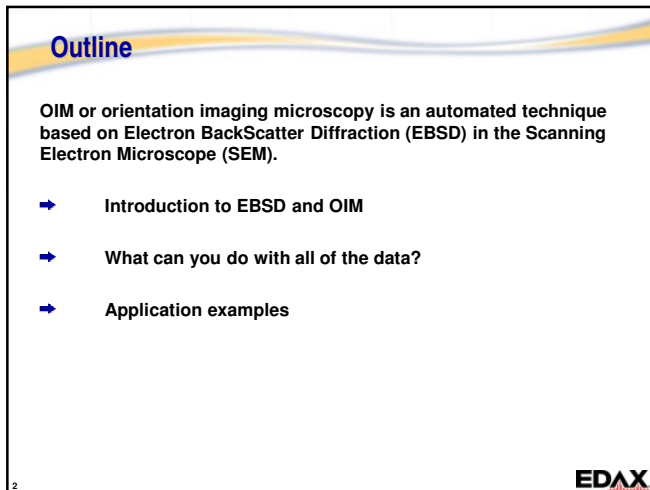
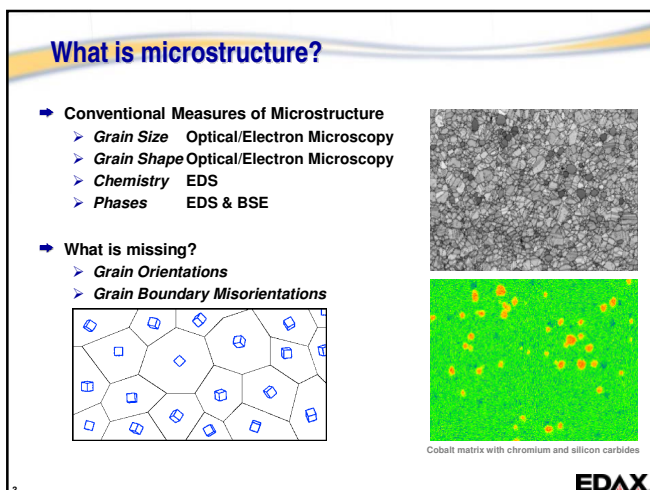


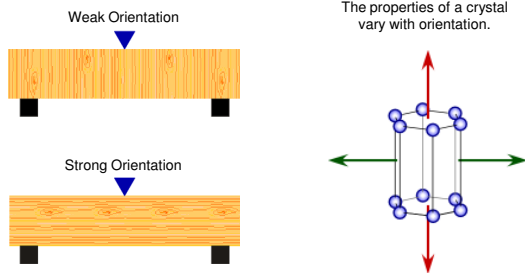






Why do we care about crystallographic orientation?

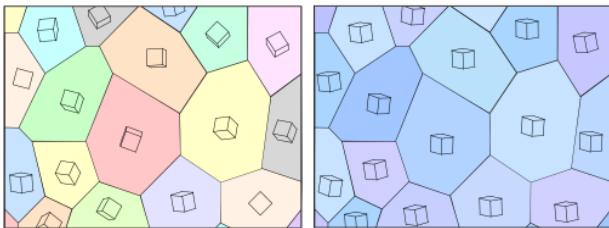
- ➔ Because materials properties are "anisotropic". We all know that the strength of wood varies with the direction of the grain.



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Isn't the anisotropy averaged out in a polycrystal?

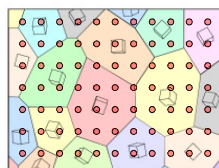
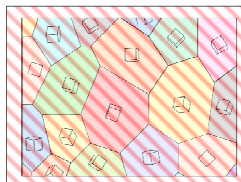
- ➔ Not necessarily. A material will be isotropic if all of the grains have random orientations. But if the grains have similar orientations then the bulk material will exhibit anisotropy similar to the constituent crystals. The distribution of crystal orientations is called **texture**. Most forming processes produce materials with texture.



EDAX

How do you measure texture?

- ➔ "Bulk" techniques
 - X-Ray
 - Neutron Diffraction
- ➔ X-ray gives us the overall texture but doesn't tell us which grains have which orientations...
- ➔ Spatially specific techniques (point-by-point)
 - Synchrotron
 - TEM – Kikuchi Patterns
 - TEM – Spot Patterns
 - Polarized Light
 - Kossel Patterns
 - Channeling Patterns
 - Electron Backscatter Diffraction (EBSD)



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Spatially specific techniques

- ➔ Synchrotron Radiation (H. F. Poulsen & D. Juul-Jensen)
 - Low Spatial Resolution ($5\mu\text{m}$), moderate angular resolution ($1-2^\circ$), Good statistics, 3-d information, difficult mathematical reconstruction, poor availability
- ➔ Polarized Light (R. Heilbronner)
 - Not very quantitative, inexpensive, good accessibility, 2-d, limited materials
- ➔ TEM Diffraction (Kikuchi patterns, Spot patterns, CBED)
 - High spatial resolution, good accuracy, extremely limited area, difficult sample preparation, as of yet - limited automation, some 3-d information, poor statistics
- ➔ Electron Channeling
 - Poor spatial resolution ($5-10\mu\text{m}$), moderate accuracy (0.5°), 2-d, no automation so poor statistics.
- ➔ Kossel X-Ray Diffraction
 - Poor spatial resolution ($10\mu\text{m}$), good accuracy (0.1°), 2-d, limited materials, no automation so poor statistics.
- ➔ Electron Backscatter Diffraction
 - Good spatial resolution ($\sim 20\text{nm}$), good angular resolution ($\sim 0.5^\circ$), reasonable statistics with automation, good availability, 2-d

Values as of 2007

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Historical overview

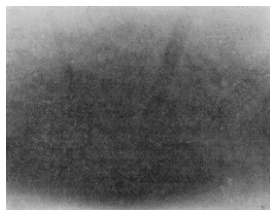
- 1928 – Kikuchi – First reported EBSPs
- 1972 – Venables et. al. – EBSPs in the SEM
- 1982 – Dingley – Computer-assisted indexing
- 1991 – Wright et. al. – Fully automated system
- 1993 – Michael et. al. – Phase ID
- 2000s – TSL – Chemically assisted phase differentiation

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P-patterns

S. Nishikawa, S. Kikuchi (1928) "The Diffraction of Cathode Rays by Calcite", *Proc. Imperial Academy (of Japan)*, 4, 475-477.

A gas discharge beam of 50 keV electrons was directed onto a cleavage face of calcite at a grazing incidence of 6° . Patterns were also obtained from cleavage faces of mica, topaz, zincblende and a natural face of quartz. They were called *P-patterns* or *patterns of the fourth kind*. Later names included *reflection Kikuchi patterns* or *backscatter Kikuchi patterns*.

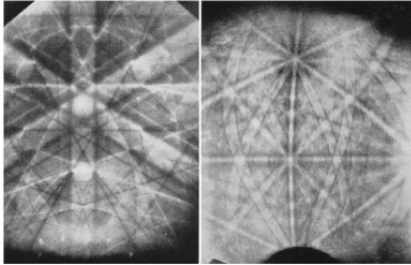


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Wide angle Kikuchi patterns

K. Shinohara (1932), R von Meibohm and E Rupp (1933)

H. Boersch (1937) "About bands in electron diffraction", *Physikalische Zeitschrift*, **38**, 1000-1004



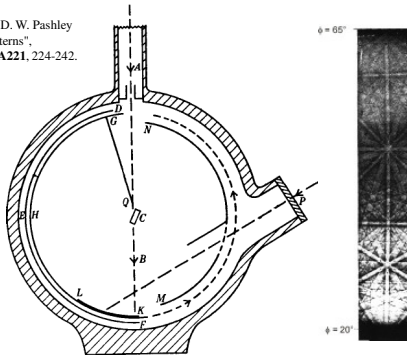
20 kV electrons using cylindrical photographic plates on cleaved, polished and etched surfaces of NaCl, KCl, PbS, CaCO₃, CaF₂, quartz, mica, diamond, Cu and Fe.

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10

High angle Kikuchi patterns

M. N. Alam, M. Blackman, and D. W. Pashley (1954) "High-angle Kikuchi Patterns", *Proc. Royal Society of London*, **A221**, 224-242.



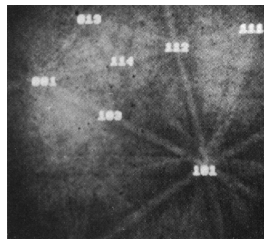
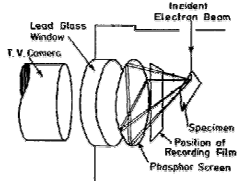
EDAX

11

On-line indexing – zone axes

D.J. Dingley (1984) "Diffraction From Sub-Micron Areas Using Electron Backscattering In A Scanning Electron Microscope", *Scanning Electron Microscopy*, **11**, 569-575.

D. J. Dingley, M. Longdon, J. Wienbren and J. Alderman (1987) "On-line Analysis of Electron Backscatter Diffraction Patterns, Texture Analysis of Polysilicon", *Scanning Electron Microscopy*, **11**, 451-456.



User required to identify the (hkl) 's and the locations of three zone axes in a pattern.

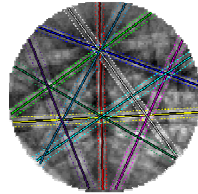
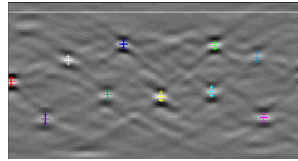
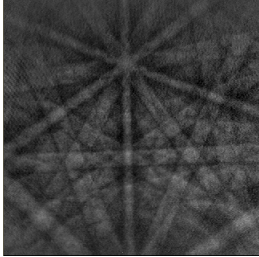
1984 - EBSD in the SEM recorded on film
1987 - Film replaced by TV camera

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12

Hough transform

N. C. Krieger-Lassen, K. Conradsen, and D. Juul-Jensen (1992) "Image Processing Procedures for Analysis of Electron Back Scattering Patterns", *Scanning Microscopy*, 6, 115-121.



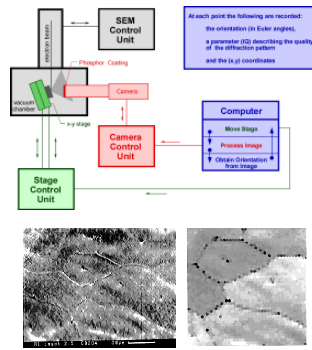
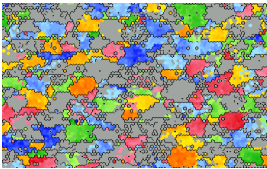
EDAX

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Full automation

S. I. Wright, PhD Thesis, Yale University, 1992.

K. Kunze, S. I. Wright, B. L. Adams, and D. J. Dingley (1993) "Advances in Automatic EBSD Single Orientation Measurements", *Textures Microstructures*, 20, 41-54.

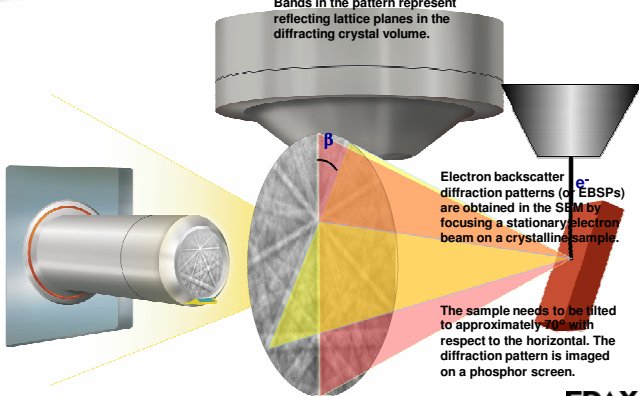


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Introduction to EBSD

Bands in the pattern represent reflecting lattice planes in the diffracting crystal volume.



Electron backscatter diffraction patterns (or EBSDs) are obtained in the SEM by focusing a stationary electron beam on a crystalline sample.

The sample needs to be tilted to approximately 70° with respect to the horizontal. The diffraction pattern is imaged on a phosphor screen.

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Application area

What materials can be analysed using Orientation Imaging Microscopy?

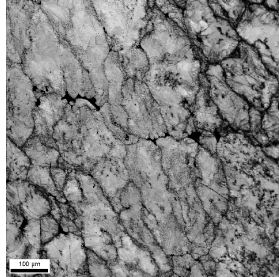


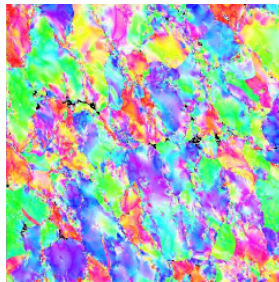
Image Quality map of deformed steel

16

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Application area

If the sample is crystalline and the question is related to a feature of the microstructure, it can be studied using Orientation Imaging Microscopy



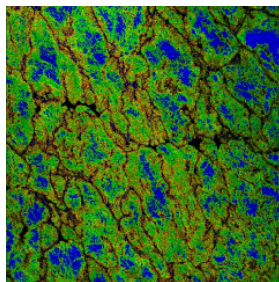
Inverse pole figure map showing crystal directions parallel to the sample normal

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Application area

- ➔ Texture analysis of rolled metals
- ➔ Study of deformation of material
- ➔ Crystal/Grain orientation analysis of metal and ceramics
- ➔ Study of stress corrosion crack
- ➔ Study of anneal and recrystallisation
- ➔ Texture analysis of semiconductor interconnector line
- ➔ Thin film texture analysis
- ➔ Others

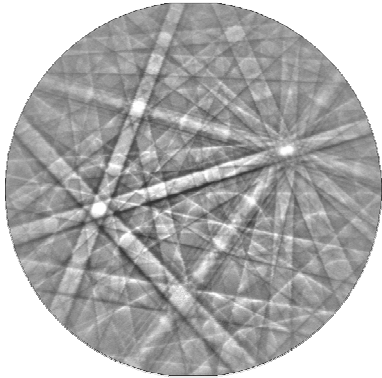


Kernel misorientation map showing local deformation, blue is undeformed

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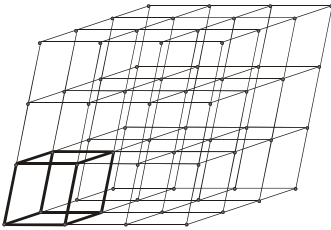
Electron BackScatter Diffraction



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Unit cell – lattice parameters



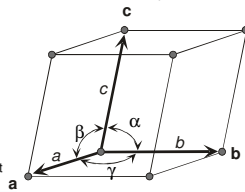
Crystal plane: A set of lattice points that lie in one plane.

Crystal direction: A set of lattice points that lie along a line.

Crystal axes: The reference vectors that define the unit cell.

a, b & c – Vectors defining the unit cell (crystallographic axes)

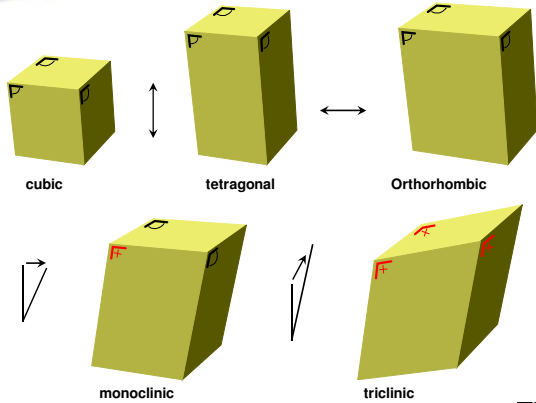
a, b & c are the lengths of these vectors and α , β & γ are the angles between them. Together these form the set of lattice constants or parameters.



20

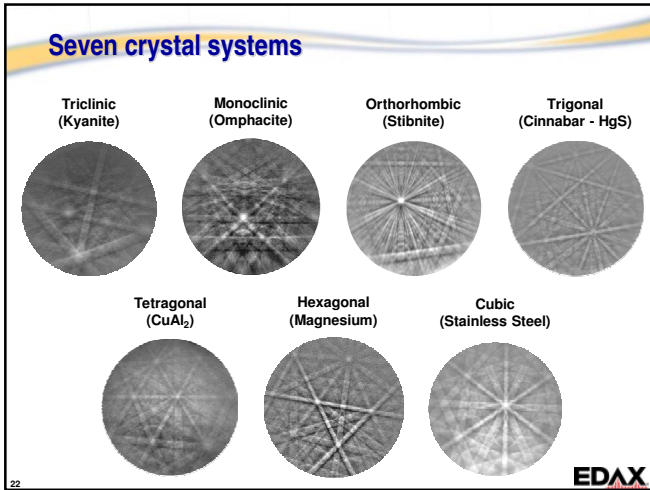
EDAX

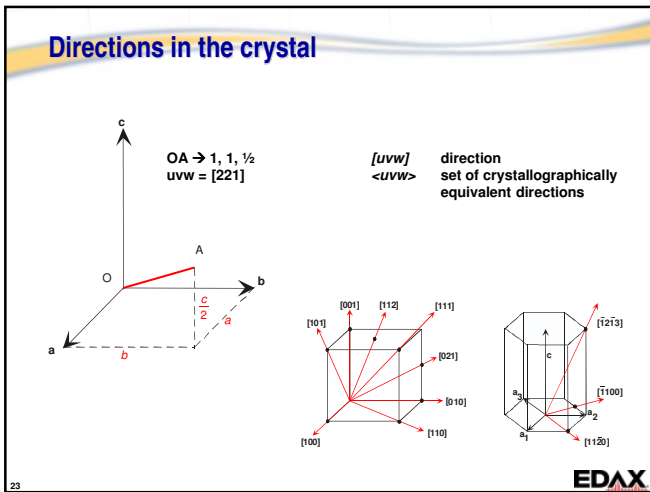
Seven crystal systems

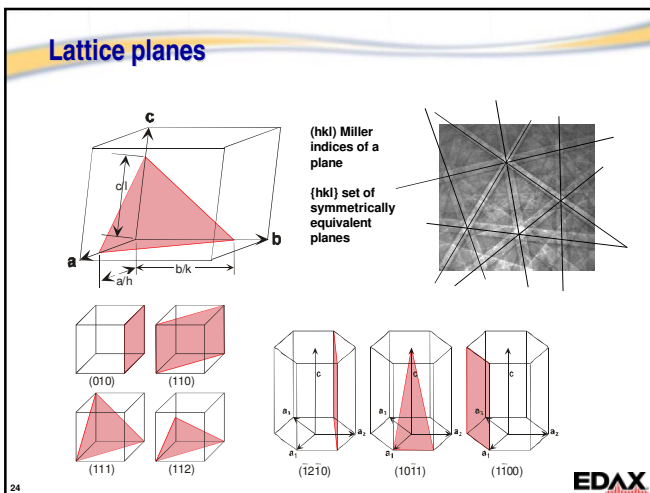


21

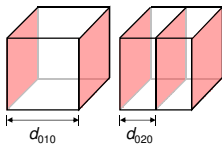
EDAX







Interplanar or d-spacing



$$d_{hkl} = a(h^2 + k^2 + l^2)^{-1/2}$$

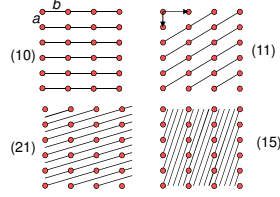
(for cubics)

$$\lambda = 2d_{hkl}\sin\theta$$

$$d_{hkl} = \frac{1}{\sqrt{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}}} = \frac{1}{\sqrt{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}}}$$

$$V = abc[1 - \cos^2\alpha - \cos^2\beta - \cos^2\gamma + 2\cos\alpha\cos\beta\cos\gamma]^{1/2}$$

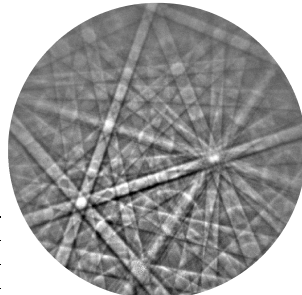
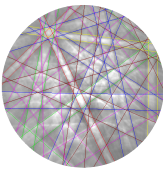
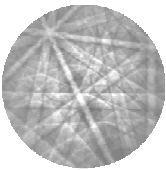
2 dimensional examples



Lines of lowest indices have the greatest spacing and density of lattice points

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Band width is inversely proportional to d-spacing



Color	(hkl)	d-spacing
Red	111	2.31
Yellow	200	2.00
Green	220	1.41
Purple	311	1.21
Pink	331	0.92
Blue	042	0.89

111
222
333

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Zone axes

Zone axis – The line of intersection of two planes is the zone axis of the zone in which the two planes are located

Zone rule

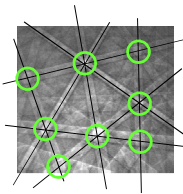
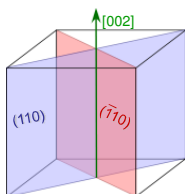
$$u = k_1l_2 - l_1k_2$$

$$v = l_1h_2 - h_1l_2$$

$$w = h_1k_2 - k_1h_2$$

Weiss zone law

$$hu + kv + lw = 0$$



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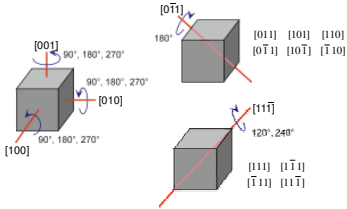
Unit cell – symmetry

Point group symmetry:

The set of rotations (a point group) which when applied to the crystal lattice rotate the lattice into an orientation indistinguishable from the original orientation.

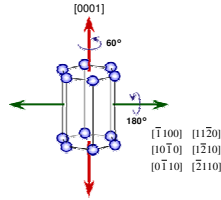
Cubic symmetry:

3 four-fold axes, 6 two-fold axes, 4 three-fold axes (+ the identity). $3 \times 3 + 6 \times 1 + 4 \times 2 + 1 = 24$ symmetry elements



Hexagonal symmetry:

1 six-fold axis + 6 two-fold axes (+ the identity). $1 \times 5 + 6 \times 1 + 1 = 12$ symmetry elements



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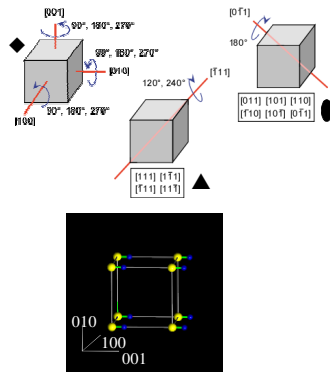
32 point groups

Each crystal system possesses a number of **point symmetry elements** characteristic of the system. ("7 crystal systems")

- Rotational symmetry (Point group)
 - monad, $\bullet$ diad, $\blacktriangle$ triad, $\blacklozenge$ tetrad, $\bullet$ hexad
 - 2π , π , $2\pi/3$, $\pi/2$, $\pi/3$
- Reflection symmetry (Point group)
 - mirror plane
- Center of inversion (Point group)
 - mirror plane with a rotation

Atomic groups at the lattice points reduce that symmetry or introduce new symmetry.

The total number of possible combinations is 32, called the 32 point groups.

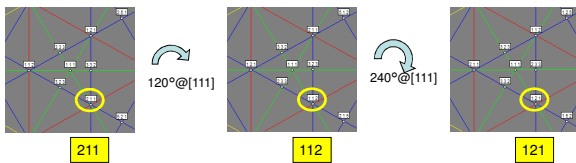


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Symmetry

Point groups (32)

- Rotations, reflections & inversions
- This symmetry is apparent in the patterns



Space groups (230)

- Point group + translations, screw axes & glide planes
- This controls which bands appear in the patterns

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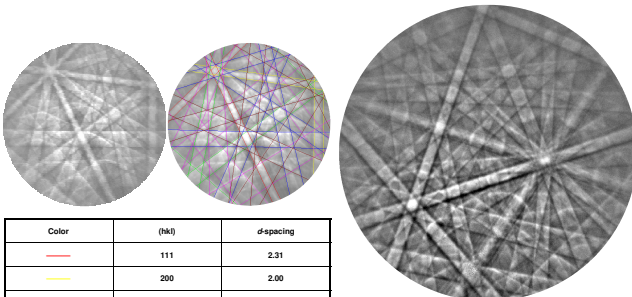
Crystallographic Space Group

- Point Groups** Symmetry operations which leave the crystal in a position indistinguishable from the position prior to the operation. Such operations (e.g. rotations, reflections and inversions) have the property that at least one point of the object was not moved by the operation.
- Translations** A crystal may be regarded as an infinite lattice; a combination of atoms that are repeated over and over throughout three-dimensional space. Lattice translations satisfy the definition of symmetry operations, since the crystal is indistinguishable after such translations.
- Screw Axes** The operation that characterizes a screw axis, denoted by $n\pi$, is a rotation of $2\pi/n$ radians followed by a translation of π/n in the direction of the axis.
- Glide Planes** The combination of the motions of reflections and translation gives a glide plane. The operation consists of reflection in a plane followed by translation. For example, if the glide is parallel to the a axis, then the symbol for the glide plane is a and the operation is reflection in the plane and translation by $a/2$.

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Band width is inversely proportional to d-spacing

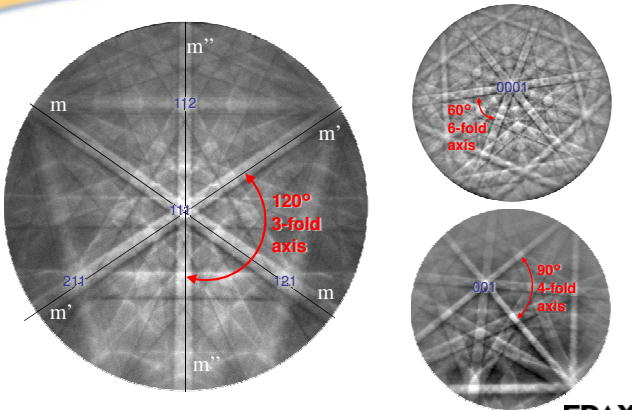


111
222
333

32

EDAX

Symmetry seen in EBSD patterns

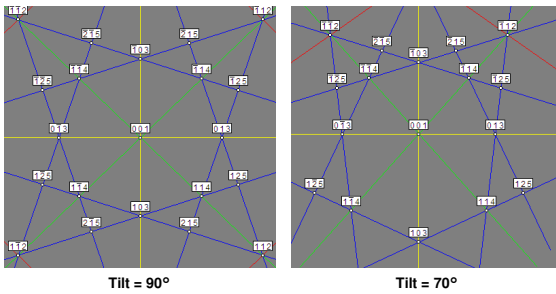


33

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Symmetry in EBSD patterns

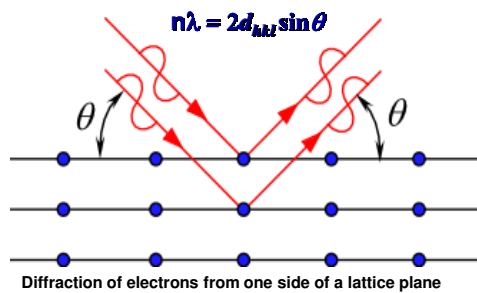
However, the symmetry is not always obvious because of the projection.



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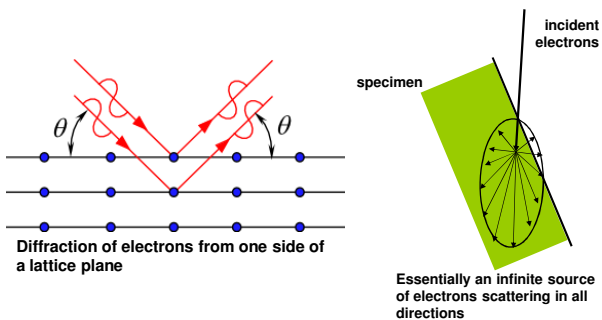
Where do the bands come from: Bragg's law



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



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Bragg's law: $n\lambda = 2d_{hkl}\sin\theta$

Diffraction of electrons occurs from both sides of a lattice plane

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Bragg's law: $n\lambda = 2d_{hkl}\sin\theta$

Phosphor screen

Trace of diffracting plane

Diffracting lattice plane

Cones of diffracted electrons

EBSD band edges

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EBSD

As the beam is moved from grain to grain the electron backscatter diffraction pattern (EBSP) will change due to the change in orientation of the crystal lattice in the diffracting volume.

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EBSD patterns

The configuration of the bands in EBSD patterns are typical for the different crystal structures and allow determination of

- crystal orientation
- phase

Al₈Fe₂Si (hexagonal)

Al₁₃Fe₄ (orthorhombic)

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EBSD – two main application areas

Phase Identification

Orientation Imaging Microscopy (OIM)

EDAX

Phase identification

EDS Spectrum

Database

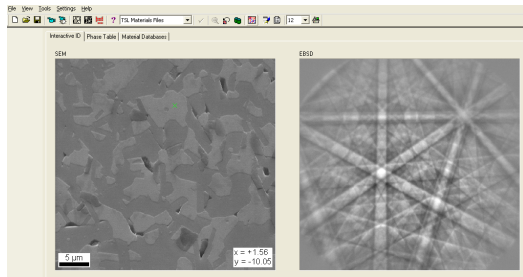
EBSD

Candidate Phases

Phase	Crystal Structure	a	b	c
Fe ₂ TiO ₄	Cubic	a=11.297		
Fe-Ti-O	Cubic	a=11.31		
Fe ₄ (TiO ₄) ₃	Tetragonal	a=9.3, c=9.5		
FeTiO ₃	Trigonal	a=5.0884, c=14.093		
Fe ₂ TiO ₄	Cubic	a=8.5352		
Fe ₃ Ti ₃ O ₁₀	Orthorhombic	a=7.789, b=10.008, c=9.74162		
FeTiO ₃	Orthorhombic	a=5.026, b=5.174, c=7.245		
Fe ₂ Ti ₃ O ₉	Hexagonal	a=2.8667, b=4.5985		

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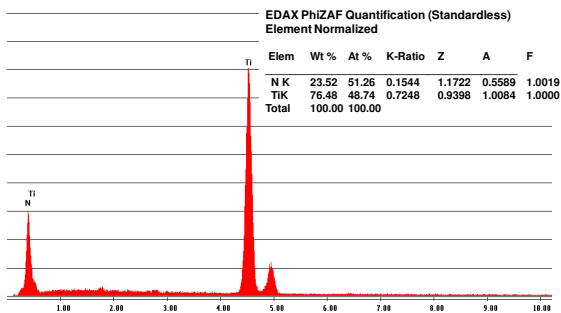
Phase identification – Delphi 3



An analysis location is selected in the SEM image, and an EBSD pattern collected from that position.

EDAX

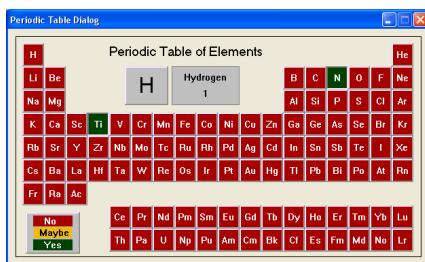
Example 1



An EDS spectrum is also collected. Here Ti and N were identified as being present. Quant analysis indicates 51% N and 49% Ti

EDAX

Example 1

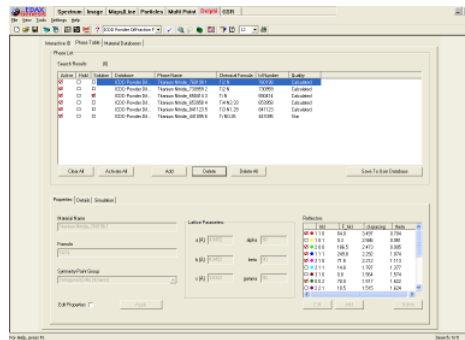


The periodic table is used to specify which elements to search for in the different databases. A Yes/No/Maybe search for each element is possible.

Here Ti and N are identified.

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Example 1

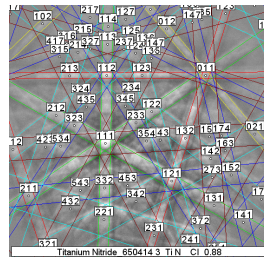


After duplication filtering, 6 candidates with this elemental chemistry are found. The results are shown on the phase table page.

EDAX

Example 1

Reflectors	hkl	F _{hkl}	d-spacing	theta
111	111	1.775	2.460	1.000
200	200	333.0	2.130	1.195
220	220	76.7	1.506	1.634
311	311	14.8	1.284	1.916
331	331	4.8	0.977	2.519
420	420	9.9	0.953	2.594
422	422	8.1	0.870	2.831
511	511	4.4	0.820	3.003

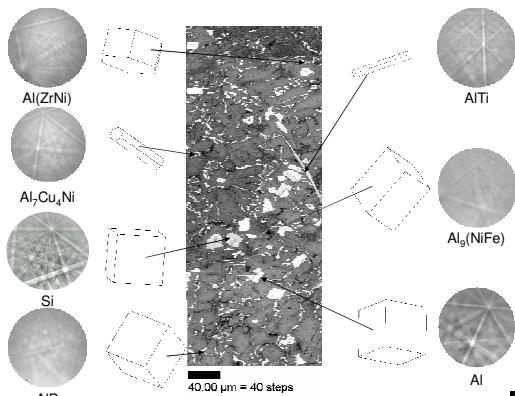


The phase with the best match is selected.

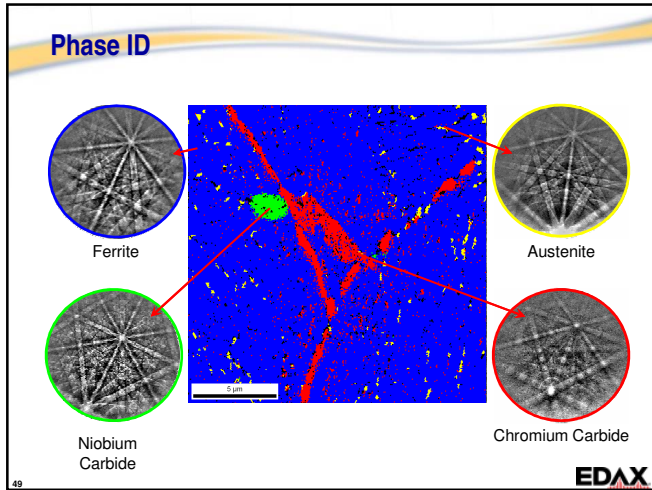
Phase	Votes	F _{hkl}	Q	Rank	Fact
Titanium Nitride_650414.3	82	0.59	0.881	3.00	
Titanium Nitride_653958.4	5	1.17	0.012	1.02	
Titanium Nitride_730959.2	30	2.42	0.060	0.56	
Titanium Nitride_441095.6	4	2.05	0.000	0.45	
Titanium Nitride_750198.1	4	3.51	0.000	0.34	
Titanium Nitride_841123.5	4	5.56	0.000	0.21	

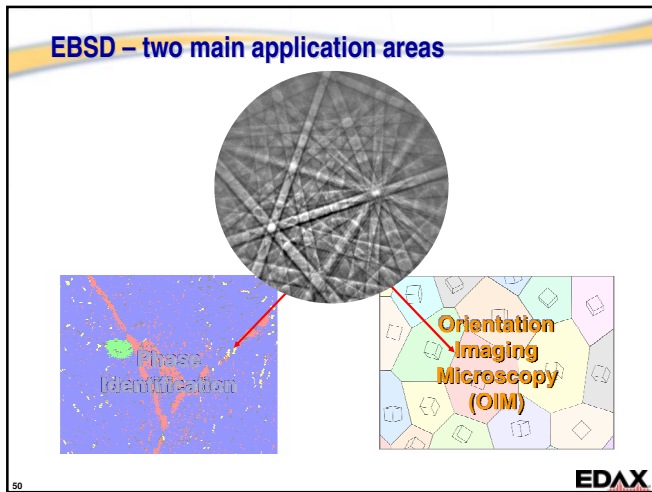
EDAX

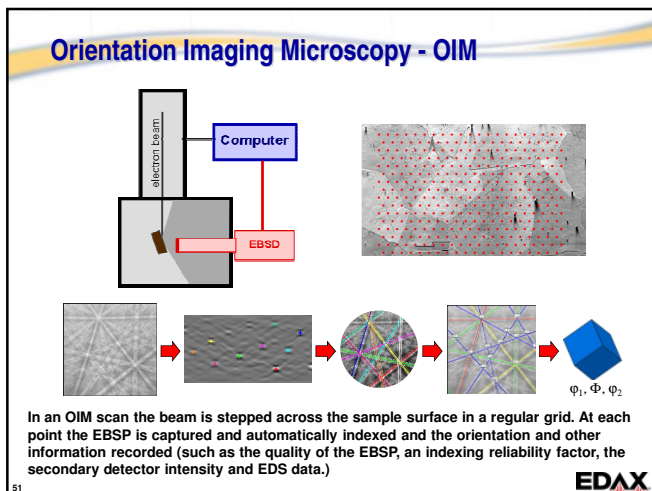
Phase ID



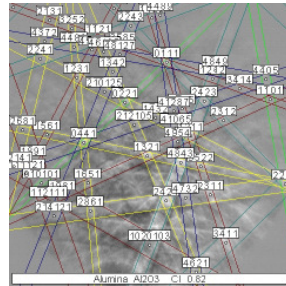
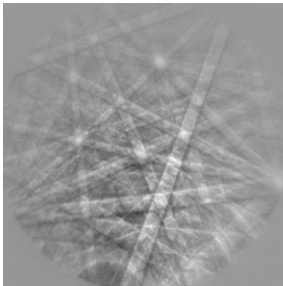
EDAX







EBSD pattern indexing



→ How do we get from here.....to here

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EDAX

First we need to find the bands → Hough Transform



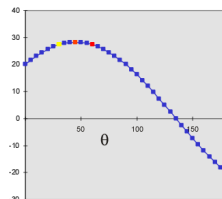
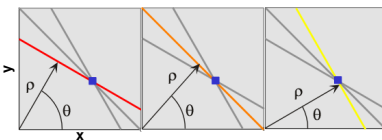
53

EDAX

Hough transform

A given pixel in an image could belong to an infinite set of lines. A line can be parameterized by the Hough parameters ρ and θ . Where θ describes the angle of the line and ρ represents the perpendicular distance of the line from the origin. The relationship between the lines passing through a pixel at a coordinate in the image of x, y can be expressed as: $\rho = x \cos \theta + y \sin \theta$. This means a point in image space transforms to a sinusoidal curve in Hough space.

$$\rho_i = x \cos \theta_i + y \sin \theta_i$$

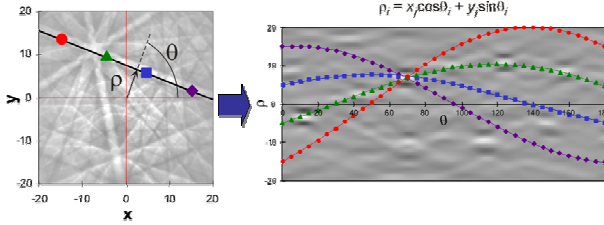


54

EDAX

Hough transform

Consider 4 pixels along a line. For each pixel in the line, all possible ρ values are calculated for θ 's ranging in values from 0 to 180 degrees using the equation: $\rho = x \cos \theta + y \sin \theta$. This produces 4 sinusoidal curves. These curves intersect at a point at a ρ, θ coordinate corresponding to the angle of the line (θ) and its position relative to the origin (ρ). Thus, a line in image space transforms to a point in Hough Space.



55

EDAX

Hough transform

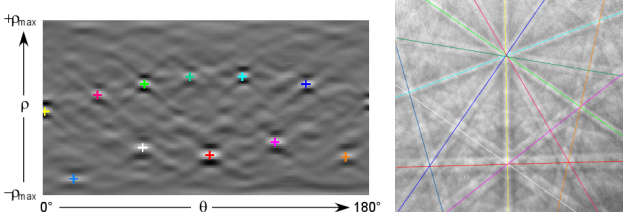
An entire image can be transformed into Hough Space by building an accumulator array $H(\rho, \theta)$ where, for each pixel in the image, all possible ρ values are calculated for θ 's ranging in values from 0 to 180 degrees via the equation $\rho = x \cos \theta + y \sin \theta$. The intensity value of the pixel at x, y is then added to the bin in the array at each corresponding ρ, θ .



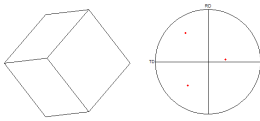
56

EDAX

Hough transform



Orientation solutions can be shown in many different ways, e.g. as crystal wire frame drawing or in a pole figure

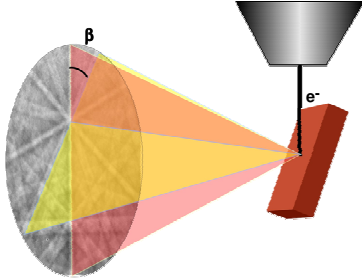


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EDAX

Pattern indexing: interplanar angle

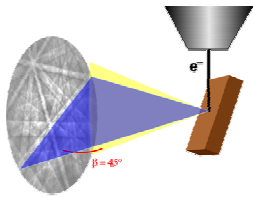
- If the (hkl) indices of the bands in the pattern can be identified then the corresponding orientation can be calculated. By comparing the angle between two bands with an interplanar angle look-up table, the (hkl) pair associated with the two bands can be identified.



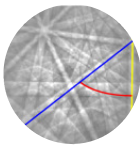
58

EDAX

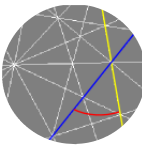
Indexing: bands – interplanar angles



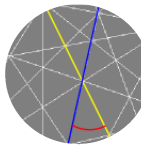
The angles cannot be measured directly from a pattern because the angles are distorted from the projection.



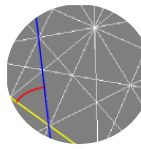
$\beta_M = 53^\circ$



$\beta_M = 48^\circ$



$\beta_M = 39^\circ$



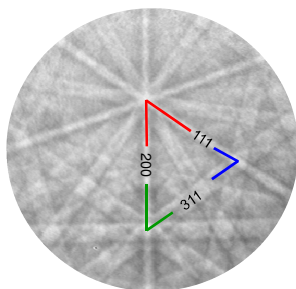
$\beta_M = 47^\circ$

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EDAX

Indexing: bands – triplet solutions

A set of orientations is obtained from a triplet of bands by comparing the interplanar angles against a look-up table.



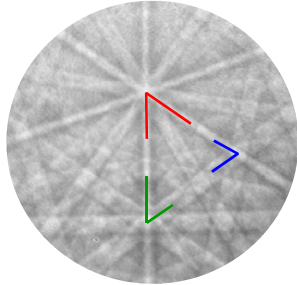
Angle	(hkl) ₁	(hkl) ₂
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

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Pattern indexing: triplet solutions

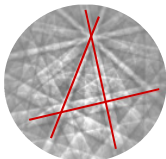
- Determine a crystal orientation for each possible combination of three bands.
- The limited accuracy of the band detection requires the use of a tolerance so that each triplet could represent multiple orientations.



Angle	(hkl) ₁	(hkl) ₂
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
57.7	311	211
59.5	111	311
60.0	220	202
65.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

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Pattern indexing: triplet solutions

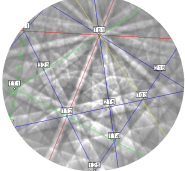
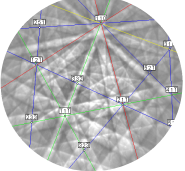
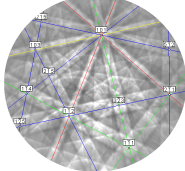


For a set of three bands, compare the interplanar angles against the LUT and determine all possible indexing solutions.

Solution 1

Solution 2

Solution 3



Pattern indexing: Confidence Index

The number of votes for the best and second best orientation solutions is used to calculate the Confidence Index (patented).

The Confidence Index gives an indication if the indexing solution is correct.

$$CI = \frac{V_1 - V_2}{V_{\text{Ideal}}}$$

$$CI = \frac{9 - 4}{10} = 0.5$$

A correct solution typically has $CI > 0.1$

Voting Table

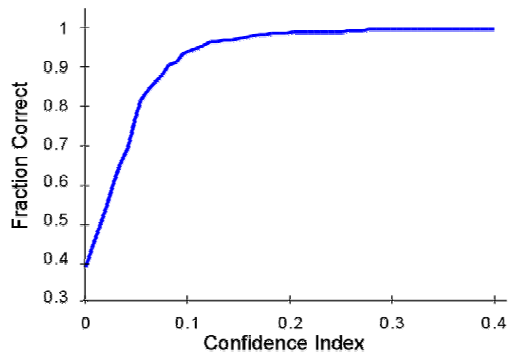
		Indexing Solutions										
		1	2	3	4	5	6	7	8	9	10	11
Band Triplets		x	x	x								
					x							
					x							
		x	x	x	x							
					x							
					x							
					x							
					x							
					x							
					x							
					x							
	10	2	4	9	2	1	1	1	1	1	1	1

Reflectors: 111, 200, 220, 311

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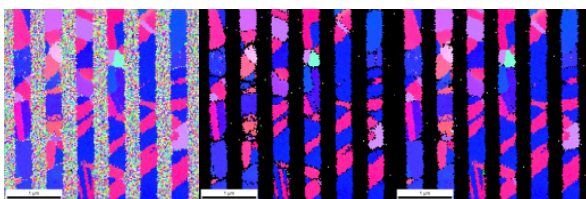
Indexing: bands – confidence index



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Indexing: bands – confidence index



All Data

$Cf > 0.3$

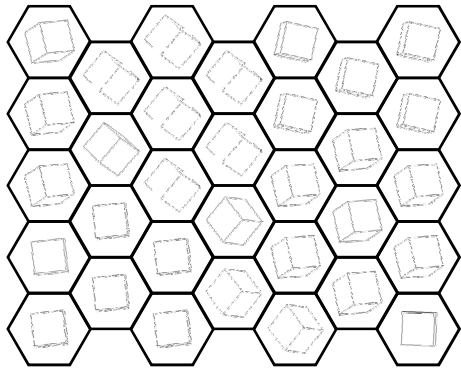
$Cf > 0.3$ After CIS

The Confidence Index Standardization (CIS) feature recovers the low confidence data points that have the correct orientation measurements. This example shows the results from Cu IC lines, where the area between the lines is filled with amorphous SiO_2 . In the all data image, the SiO_2 area is noisy. In the $Cf > 0.3$ area, this area is black, indicating a low confidence as expected. However, some of the grain boundary areas are also black. These are recovered using the CIS feature.

EDAX

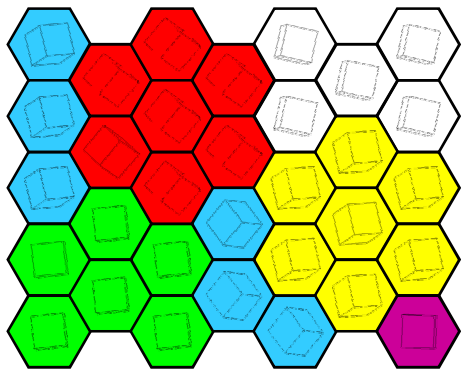
86

Grouping measurements as grains



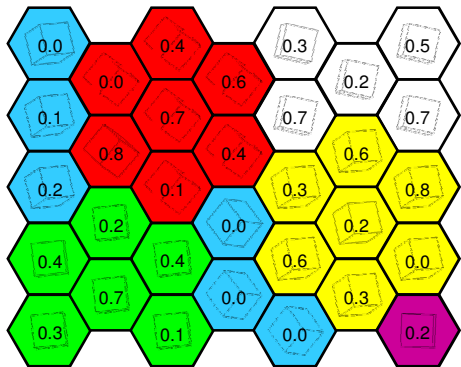
EDAX

Grouping measurements as grains

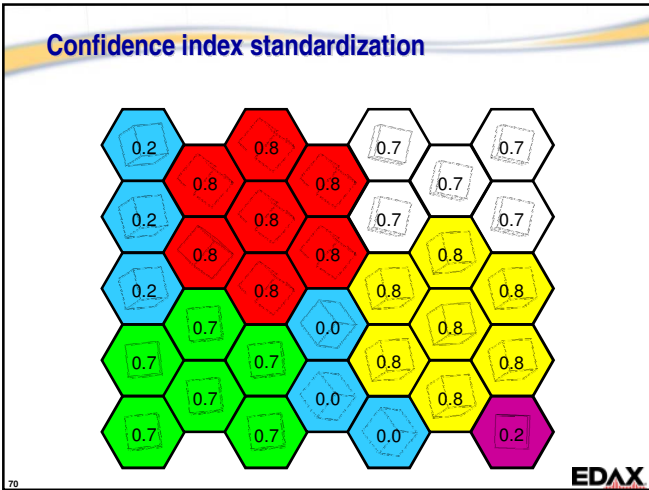


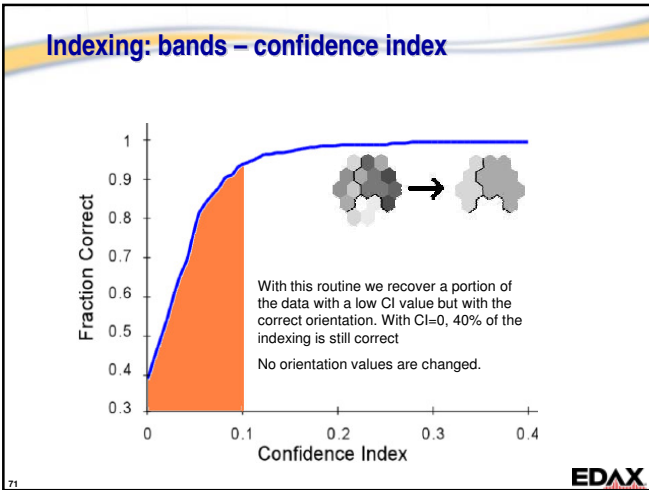
EDAX

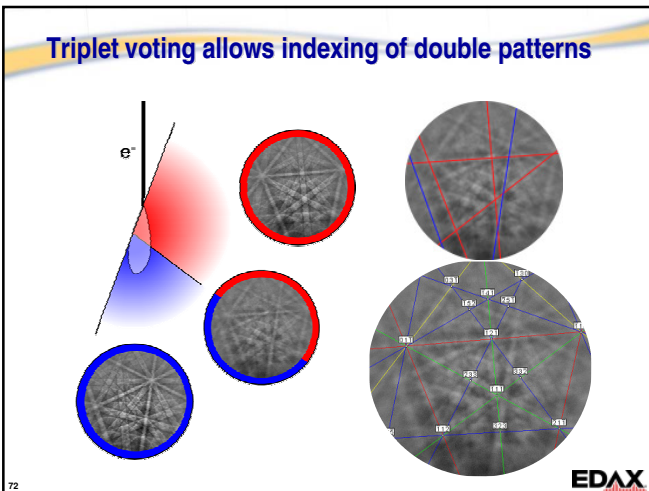
Initial confidence index distribution

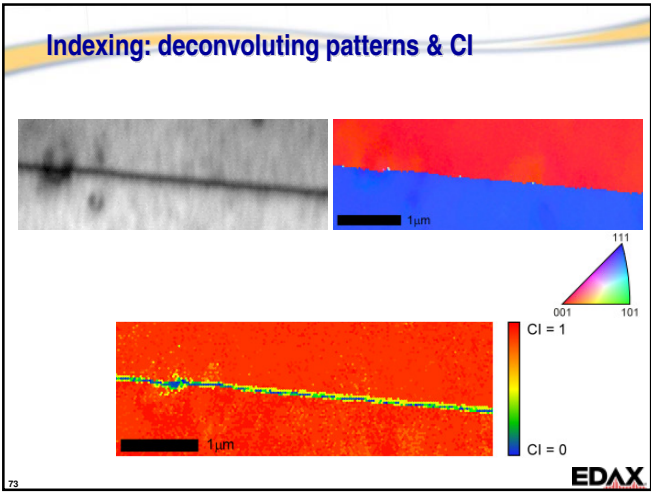


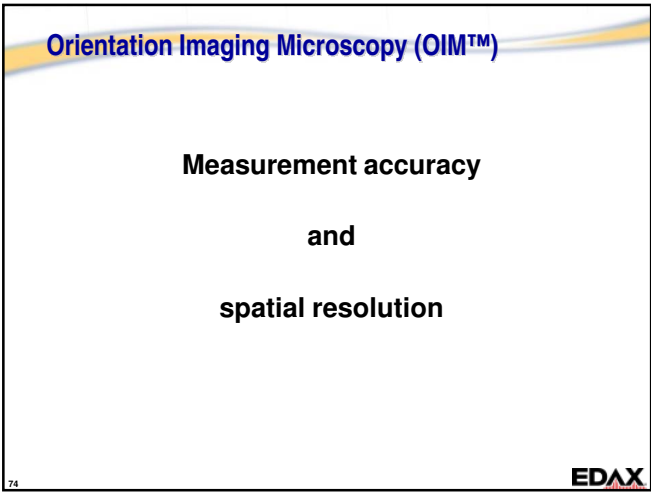
EDAX

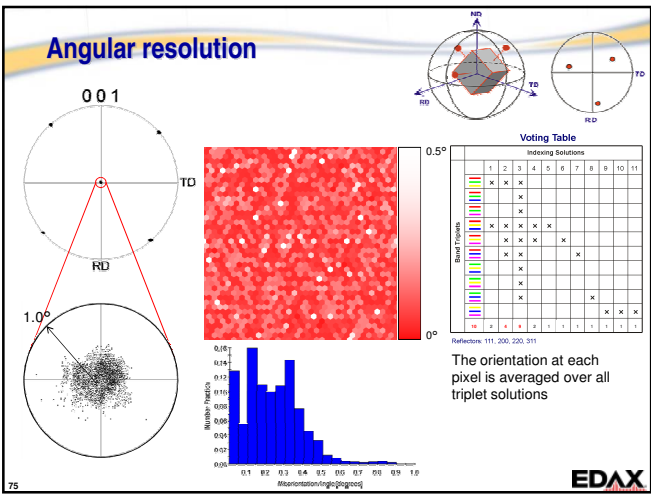






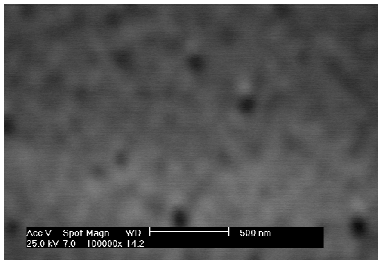




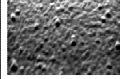


Resolution: spatial accuracy

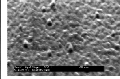
SEM imaging resolution



XL30 feg spot 2



XL30 feg spot 4



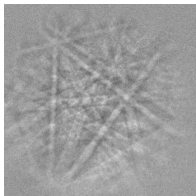
XL30 feg spot 7

Pt film: imaging resolution is limited by probe diameter.

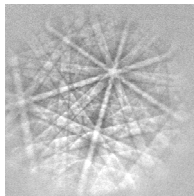
EDAX

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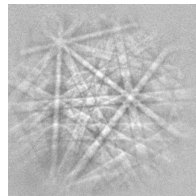
EBSD spatial resolution



Overlapping 1 and 2



Grain 1



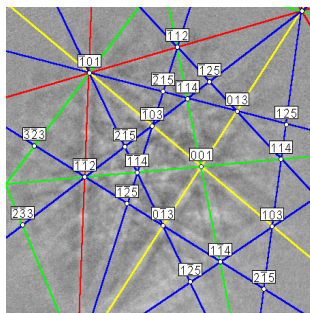
Grain 2

One of the overlapping patterns is brighter than the other

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EBSD spatial resolution → pattern deconvolution

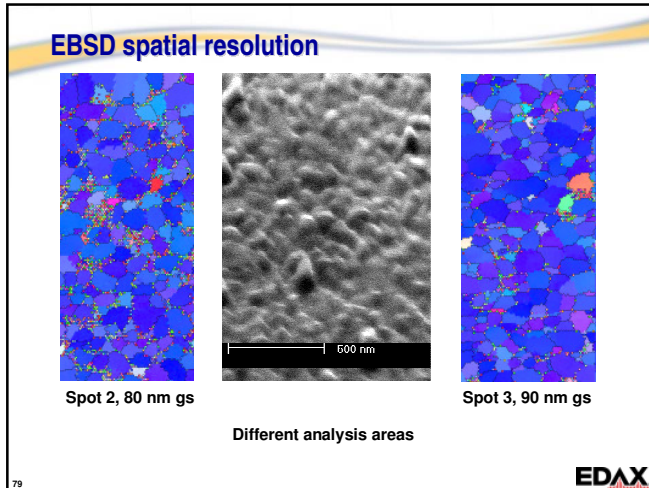


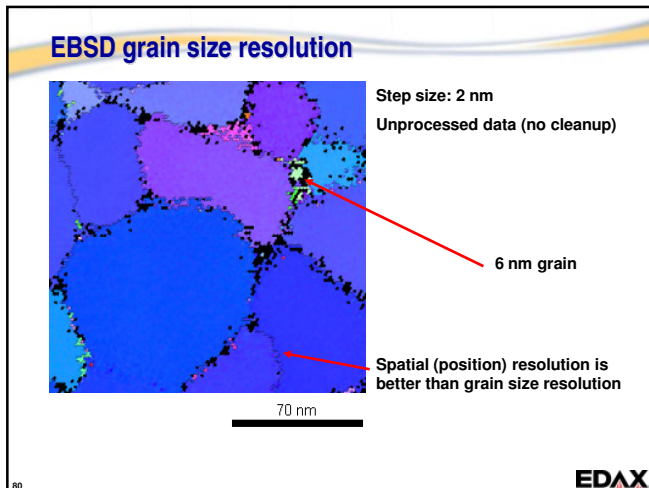
Orientation solution is determined for the strongest of the two patterns.

→ Actual spatial resolution of EBSD is determined by the beam diameter in combination with the triplet voting indexing routines.

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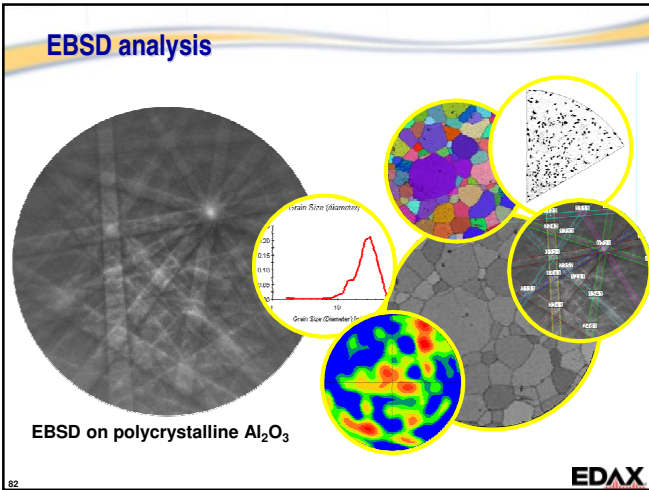


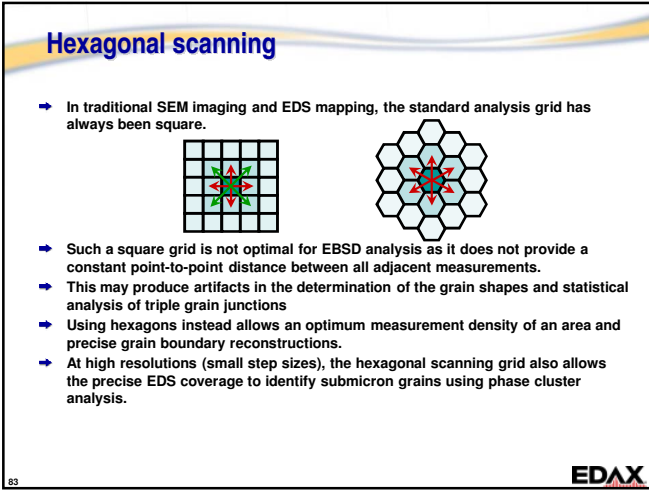
Outline

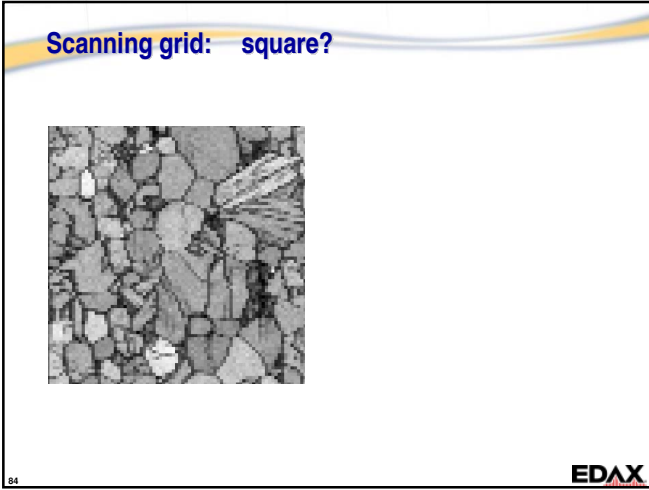
OIM or orientation imaging microscopy is an automated technique based on Electron BackScatter Diffraction (EBSD) in the Scanning Electron Microscope (SEM).

- ➔ Introduction to EBSD and OIM
- ➔ What can you do with all of the data?
- ➔ Application examples

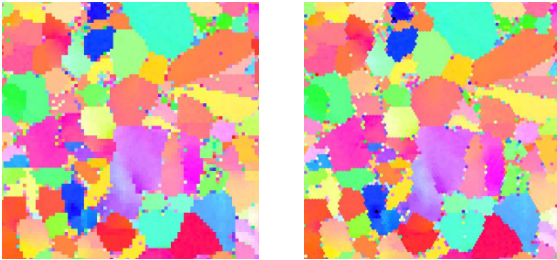
EDAX







Scanning grid: square or hexagonal?



Grain shapes are better resolved on a hexagonal grid
Centre-to-centre distances are the same in all directions

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Analysis Tools

Orientation Imaging Microscopy:
Imaging and Quantification of Crystal Related Features

1. Grain size, orientation, and shape
2. Crystal directions and orientations
3. Boundary type and misorientation distribution
4. Plastic deformation
5. Texture (PF, IPF, ODF, MDF)
6. Spatial distribution

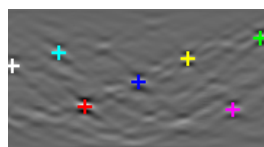
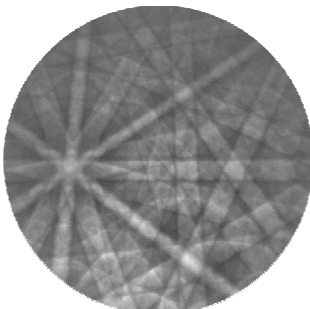
All measurements are linked to their positions

→ This allows advanced interactive analysis of the distribution of microstructural features

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Analysis tools: image quality (IQ)



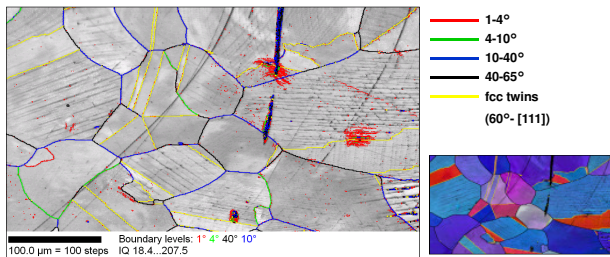
Color	Theta	Rho	Height	d-Spacing
+	53	33	8101	1.74
+	174	80	5021	1.68
+	124	66	4356	1.6
+	90	50	4557	1.31
+	155	31	3894	1.37
+	35	70	3520	1.36
+	3	61	2875	1.17

Image quality value is proportional to the average brightness of the diffracted bands in an EBSD pattern (intensity of the detected Hough peaks).

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Analysis tools: boundary maps



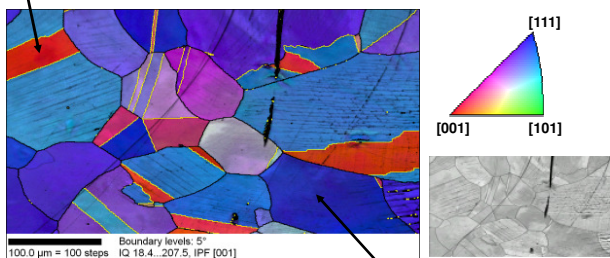
OIM Image Quality Map with superimposed boundary misorientations

IQ is a function of crystal quality (lattice distortion), camera detection parameters (brightness and contrast), SEM settings, specimen surface condition, and local crystal orientation.

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Inverse pole figure maps



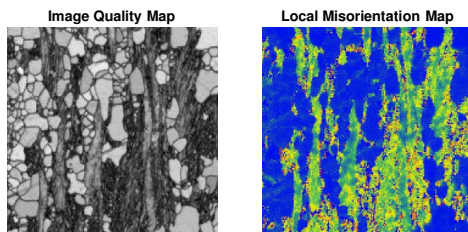
OIM Inverse Pole Figure Map

Colour coding indicates crystal direction parallel to a reference direction (here // sample normal)

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Local misorientation maps



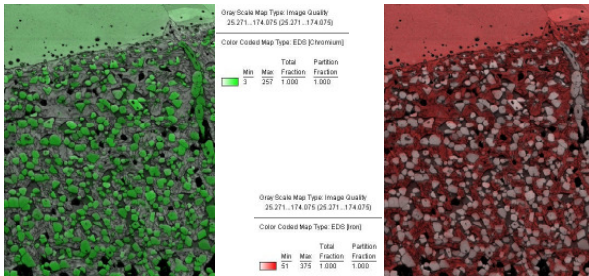
EBSD pattern from recrystallised material
→ sharp

EBSD pattern from deformed material
→ blurred

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Maps: colour & gray scale

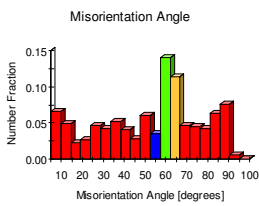


Combined IQ and EDS maps showing the distribution of Fe and Cr in an oxidised reaction zone on steel

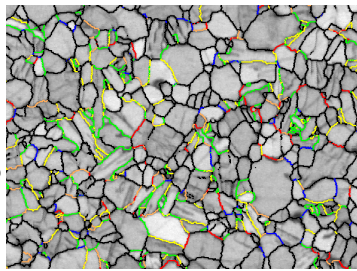
EDAX

OIM interactive analysis

Misorientation analysis



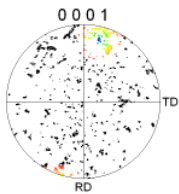
Interactive analysis:
all maps, charts, and plots are linked, allowing fast determination of spatial distributions



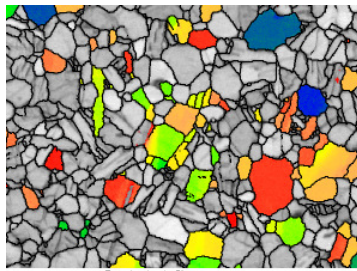
EDAX

OIM interactive analysis

Pole figures



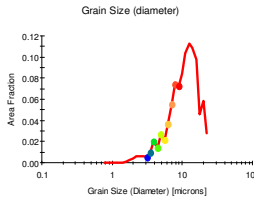
Interactive analysis:
all maps, charts, and plots are linked, allowing fast determination of spatial distributions



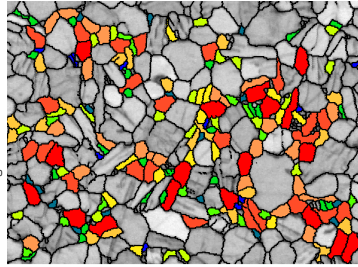
EDAX

OIM interactive analysis

Grain size analysis



Interactive analysis:
all maps, charts, and plots are
linked, allowing fast
determination of spatial
distributions

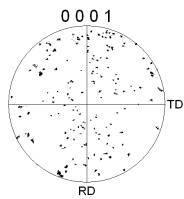


40.00 µm = 80 steps

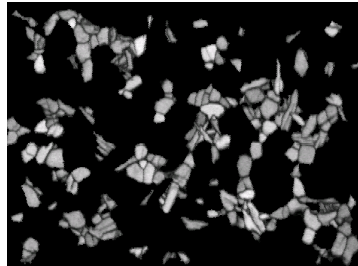
EDAX

OIM interactive analysis

Grain size analysis



highlighted points can be
extracted and analysed
separately,
e.g. in pole figures



40.00 µm = 80 steps

EDAX

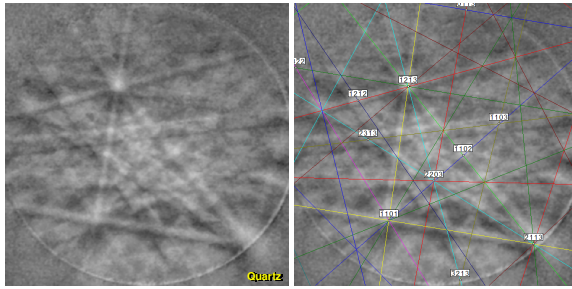
Outline

OIM or orientation imaging microscopy is an automated technique based on Electron BackScatter Diffraction (EBSD) in the Scanning Electron Microscope (SEM).

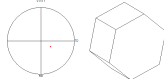
- ➔ Introduction to EBSD and OIM
- ➔ What can you do with all of the data?
- ➔ Application examples

EDAX

Single phase example

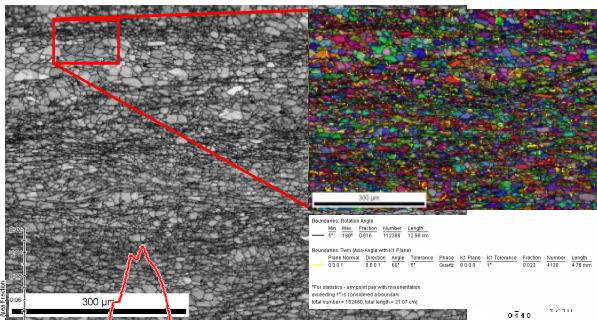


- ➔ Collect a pattern
- ➔ Match it against the given crystal structure to determine the orientation



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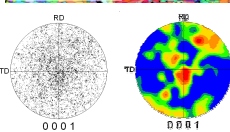
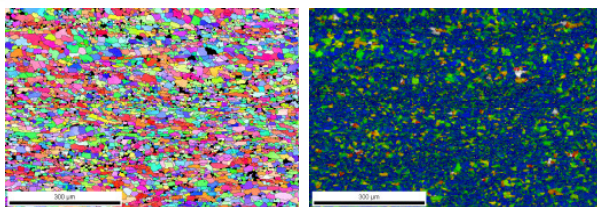
Single phase deformed quartz



In this example, hundreds of grains were measured to quantify the grain size distribution together with grain boundary statistics.

EDAX

Single phase deformed quartz



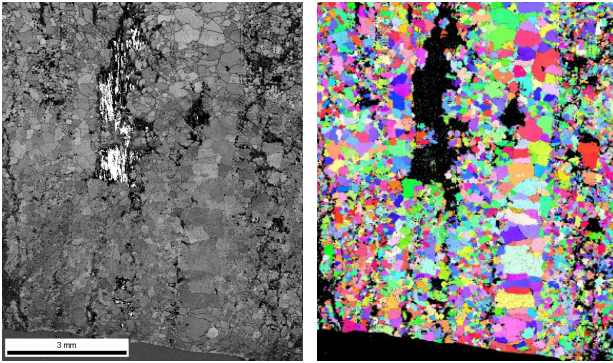
Color Coded Map Type: Grain Reference Orientation Deviation

Min	Max	Total Fraction	Partition Fraction
0	10	0.998	0.998

OIM can be used to examine crystallographic preferred orientations (CPO) or texture, grain to grain properties (misorientation relationships, plastic deformation, grain shape), and seismic propagation rates (tensor properties) among other things.

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Large area quartz scan

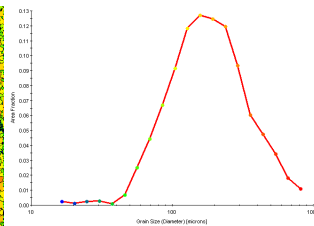
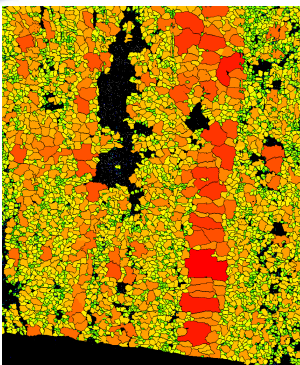


OIM data can be collected from the nanometer scale to the millimeter scale.

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Large area quartz scan

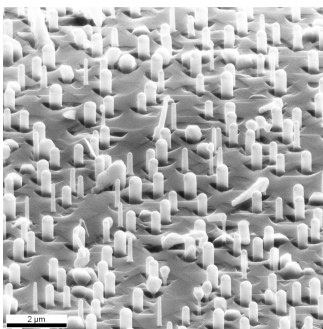


Grain size distribution

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Application – silicon nanowires



- ➔ Silicon nanowires grown on Si single crystal substrate
- ➔ All nanowires should have a gold "cap" of Au.

Questions:

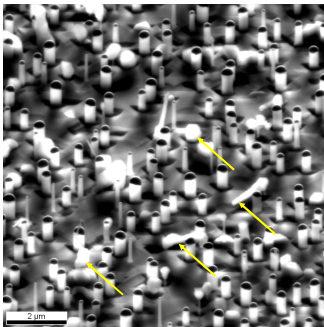
1. Do all wires have a gold "cap" ?
2. Is there a special orientation relation between the wires and the gold ?
3. Are Si nanowires epitaxial on the substrate ?

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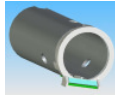
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Application – nanowires – gold cap ?



- ➔ In the SE image of the tilted specimen, the gold caps are almost invisible.
- ➔ Imaging the sample with a FSD integrated with the EBSD detector produces high contrast images due to the higher atomic number of gold caps.
- ➔ Anomalous nanowires do not have a gold cap (arrowed).

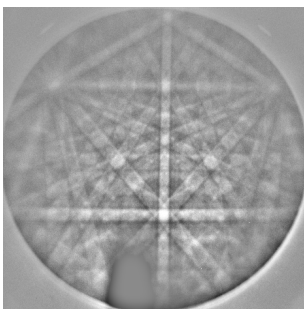


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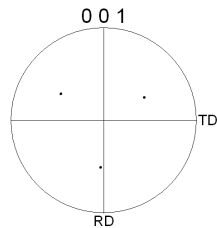
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Application – nanowires – special gold orientation ?



Reference pattern of the silicon substrate.

Dark area is caused by shadowing due to nanowires.



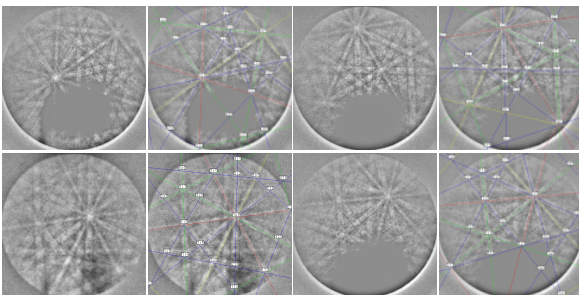
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Application – nanowires – special gold orientation ?

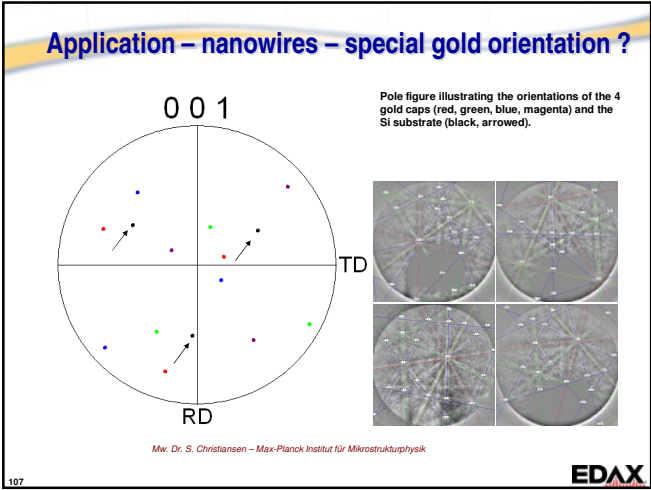
Individual patterns could be obtained from the gold caps.

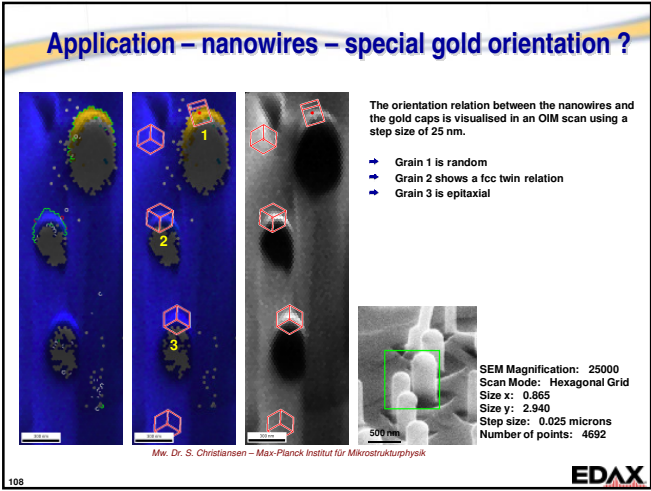


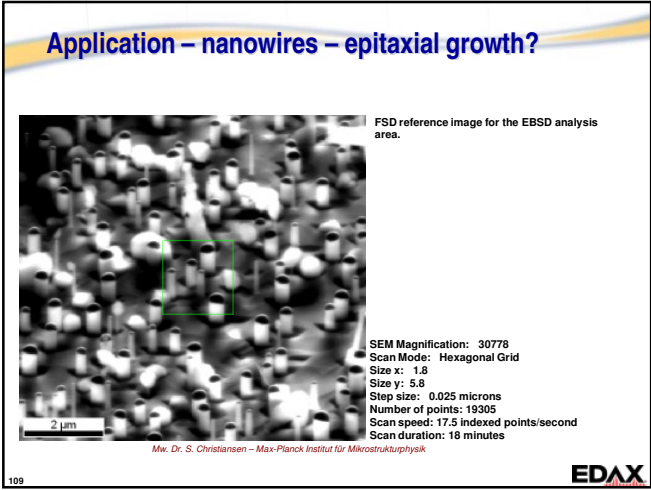
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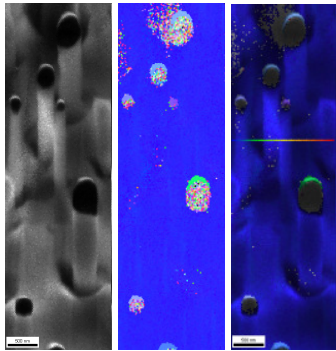
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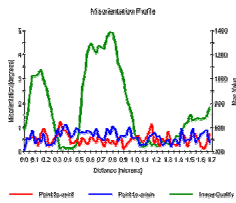


Application – nanowires – epitaxial growth?



IQ and IPF maps as measured (left).
IPF on IQ map with orientation profile.

In the chart, the location of the nanowires is visible in the variation of the IQ values (green line)
There is no orientation change in the nanowires with respect to the substrate.



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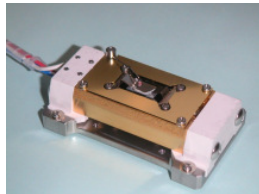
Application – nanowires – summary

- ➔ Do all wires have a gold "cap" ?
 - No, there are anomalous wires without gold
- ➔ Is there a special orientation relation between the wires and the gold ?
 - Yes
- ➔ Are Si nanowires epitaxial on the substrate ?
 - Yes

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Application – heating experiment, setup



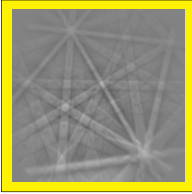
- ➔ Heating stage is small enough to operate in the 70° tilt position for EBSD
- ➔ Capable of temperatures up to ~700° C
- ➔ Sample size: 10mm x 5mm x 2mm

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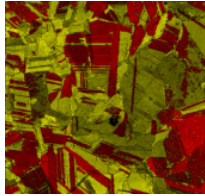
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Application – heating experiment, phase

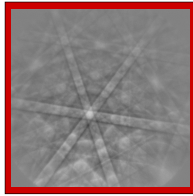
Beta: FCC Phase



Cobalt
Transition Temperature = 422°C



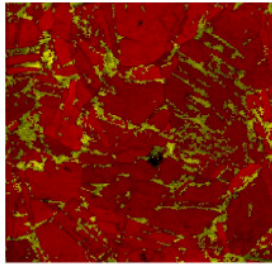
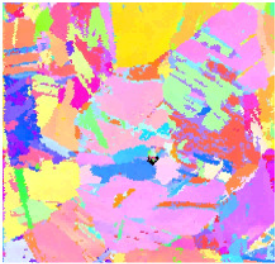
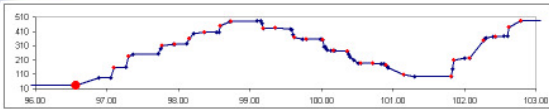
Alpha: HCP Phase



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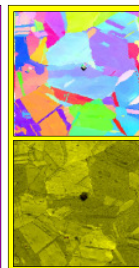
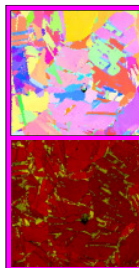
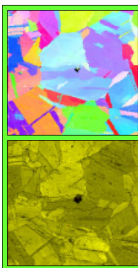
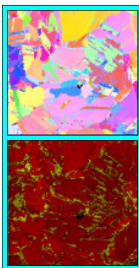
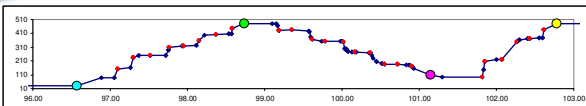
Application – phase transformation, cobalt



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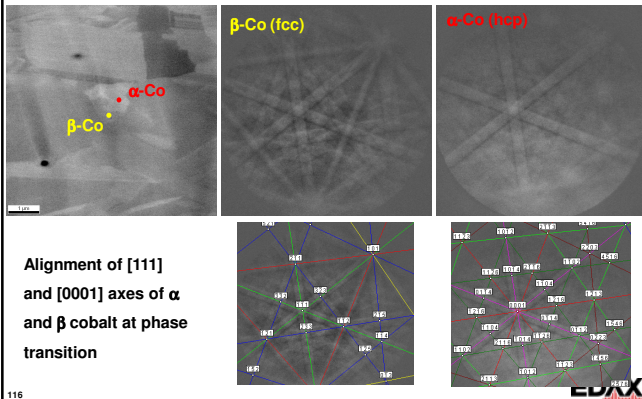
Application – phase transformation, reversability



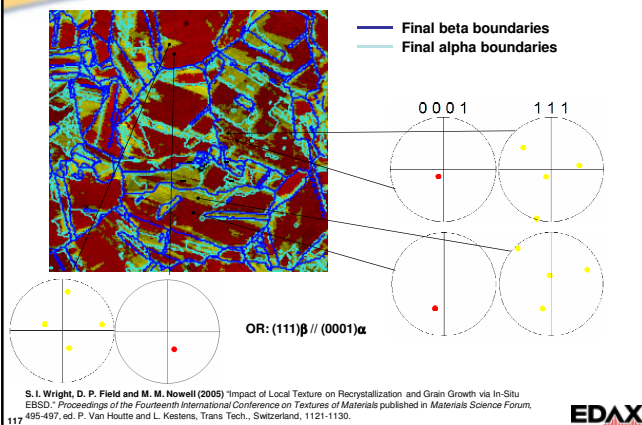
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Application – phase transformation, misorientations



Application – phase transformation, misorientations



Conclusions

OIM EBSD analysis is a powerful tool for investigating many aspects of the microstructure of polycrystalline materials of all crystal systems including:

- grain properties: shape, size, orientation, internal deformation, Schmid factors, ...
- grain boundaries: misorientation distributions, twin boundaries, CSL boundaries, phase boundaries, ...
- texture analysis: pole figures, inverse pole figures, ODF plots, ...
- simultaneous EDS analysis
- all plots, maps, and charts are interactively linked for easy correlation between features