Scanning Electron Microscopy (SEM) & Electron Back Scattered Diffraction (EBSD)

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The human eye – your microscope



1. The remanence time of an image on the retina is about 1/24 of a second: this why television screens scanned 25 times per second and in the cinema the film advanced at 24 images per second.

2. The number of grey levels an eye can resolve is about 16 to 20. The human eye can discern 350 000 different shades of colour, but is bad observer of black and white images3. The resolving power of the eye is about 0.125 mm at a distance of 250 mm



Schematic illustration of the resolution of the human eye. The lower portion shows the fovea as a hexagonal array of cones, the top view shows the angular resolution of the eye in terms of the eye–object distance, the angle θ , and the distance between individual object lines.



Schematic illustration of the various length scales, from macroscopic, to microscopic, to nanoscale, to the subatomic. The left hand side of the figure shows the experimental techniques that are used to cover the various length scales. The images in the circles on the right are (from the top down): a quartz crystal (courtesy of D. Wilson); grains in a SrTiO₃ ceramic (courtesy of G. Rohrer); nano-crystalline particles of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ (courtesy of R. Swaminathan); atomic resolution image of BaTiO₃.



Optical and Electronic Microscopes



Magnification $\sim \times 2000$ $\times 50 \sim \times 1,500,000$ $\times 10 \sim \times 1,000,000$

Optical Microscope

Ernest Abbe 1874 (<u>University of Jena</u>) defined the resolution of an optical microscope to be the minimum distance of two structural elements to be imaged (d),

 $d = \lambda / NA = \lambda / n \sin \alpha$

where λ is the wavelength and NA is the numerical aperture of the objective . NA is defined as the sine of the half aperture angle α multiplied by the refractive index of the medium filling the space between the cover glass and the front lens

With α =70° n=1.5 λ =0.5 μ m = 500 nm

The resolution $d = 0.35 \mu m = 350 nm$

N.B. The resolution is same order as wave length λ

Electron microscope

Louis de Broglie in 1924 in his PhD thesis associated the wave length of electrons with the accelerating voltage for which he was awarded the Nobel Prize for Physics in 1929 for this work, which made him the first person to receive a Nobel Prize for a PhD thesis. *Recherches sur la théorie des quanta*, Thesis (Paris).

λ= h / m v

with h = Planck's constant

m = electron's rest mass

v = velocity of the electron

The velocity is related to the kinetic energy of the particule (electron with charge e) accelerated by the voltage V given by :

1/2 mv² = e V

where

 $\lambda = h / (2 m e V)^{1/2} = (1.5/V)^{1/2}$ with λ in nm and V in volts For exemple V = 20 kV λ =0.008 nm V = 100 kV λ =0.004 nm

Compared with optics with λ =500 nm (x 125 000 λ_{optic} à 100 kV)

Today the best electron microscopes have a point to point resolution of about 0.1 nm.

N.B. a resolution of about 100λ ! For optical microscope resolution = λ !!

Average diffraction properties of radiation used for texture measurement by diffraction, with light also included for comparison

	Light	Neutrons	X-rays	Electrons
Wavelength [nm] Energy [eV] Charge [C] Rest mass [g] Penetration depth, absorption length [mm]	400–700 1 0 -	0.05-0.3 10^{-2} 0 1.67×10^{-24} 10-100	$\begin{array}{c} 0.05 - 0.3 \\ 10^4 \\ 0 \\ 0 \\ 0.01 - 0.1 \end{array}$	$\begin{array}{c} 0.001 - 0.01 \\ 10^{5} \\ -1.602 \times 10^{-19} \\ 9.11 \times 10^{-28} \\ 10^{-3} \end{array}$

Wavelength – Voltage



SEM – 10-30 kV

Properties of electrons as a function of voltage, relative to those at 100 kV. Courtesy R. M. Fisher.



Experimental data on penetration in silicon and stainless steel.

A 3 MV HVEM constructed at the C.N.R.S. Laboratories in Toulouse and in operation by 1970. To focus the high-energy electrons, large-diameter lenses were required, and the TEM column became so high that long control rods were needed between the operator and the moving parts (for example, to provide specimen motion). Courtesy of G. Dupouy, personal communication.

Some important dates

- 1924 De Broglie associated the notion of wave length to particles
- 1931 Ruska made first electron microscope
- 1939 Siemens delivered the first commercial transmission electron microscope (TEM)
- 1951 Castaing built the first micro-probe with an x-ray analyser
- 1965 Cambridge Instruments delivered the first commercial scanning electron microscope

Basic SEM column



Basic construction of a SEM.

Characteristic Information: SEM

Topography

The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties

Morphology

The shape and size of the particles making up the object; direct relation between these structures and materials properties

Composition

The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties

Crystallographic Information

How the atoms are arranged in the object; direct relation between these arrangements and material properties

Advantages of Using SEM over OM

Mag	Depth of Field	Resolution
OM: 4x – 1400x	0.5mm	~ 0.2mm
SEM: 10x – 500Kx	30mm	1.5nm

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample.

The combination of higher magnification, larger depth of field, greater resolution, compositional and crystallographic information makes the SEM one of the most heavily used instruments in academic/national lab research areas and industry.



SEM : Depth of focus



Relation between the aperture angle of the electron probe and the depth of focus.



Difference of the depth of focus between SEM and optical microscope.





 $\ensuremath{\mathsf{OM}}\xspace$ image of the same field of view.

Depth of Field

The height over which a sample can be clearly focused is called the Depth of Field. The SEM has a large depth of field which produces the images that appear 3-dimensional in nature.

Depth of field is improved by:

- Longer working distance
- Smaller objective apertures
- Lower magnifications

Depth of Field

The angle α is determined by:

 α = r/WD

r = radius of the aperture used, and WD is the workin distance of the aperture from the specimen.

Depth of field in μm with 10-mm working distance

Radius r

Radius /

α

α

D

Magnification	With 100-µm aperture	With 400-µm aperture
100	400	100
1000	40	10
10000	4	1
100000	0.4	0.1

Concept of SEM magnification



Concept of SEM magnification.

Electron beam – specimen interaction



Interaction - TEM



Schematic showing electrons and electromagnetic waves emitted from a specimen as a result of elastic and inelastic scattering of the incident electron waves

Interactions - TEM



Signals excited by the scanning electron beam and their relation to specimen morphol-



Fig. 4.20. Differences in beam broadening in a bulk specimen (a), and a thin film (b). Part **a** shows regions of electron penetration, electron escape, and x-ray emission. For high-energy electrons, dimensions of regions of x-ray emission are typically a few microns, microns for backscattered electrons, tens of Å for secondary electrons. The larger dimensions do not exist for the thin specimen in **b**.



Specimen Interaction Volume

The volume inside the specimen in which interactions occur while interacting with an electron beam. This volume depends on the following factors:

 Atomic number of the material being examined; higher atomic number materials absorb or stop more electrons, smaller interaction volume.

• Accelerating voltage: higher voltages penetrate farther into the sample and generate a larger interaction volume

• Angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the interaction volume.

Secondary (SE) and backscattered electrons (BSE)



Distribution énergétique de l'émission électronique d'une cible.

PEC : pic d'énergie caractéristique (interactions avec les phonons)

Non-conductive specimen



Electric flow in a nonconductive specimen.

Total electron yield – Back-scattered(η) + Secondary (δ) Low voltage microscopy



Total electron yield $(\eta + \delta)$ as a function of primary energy, showing the range $(E_1 \text{ to } E_2)$ over which electrostatic charging of an insulating specimen is not a problem.

$$I_{\text{specimen}} = I_{\text{primary}} - I_{\text{BSE}} - I_{\text{SE}} = I_{\text{primary}} - (I_{\text{BSE}} + I_{\text{SE}}) = I_{p-(\eta+\delta)}$$

 $E_1 = \eta + \delta = 1$ (specimen +ve charged to neutral) $\approx 100-500V$ $E_1 < E_0 < E_2$ no charge Ip $\approx (\eta + \delta)$ Input Beam $E_0 \approx$ Output SE+BSE e.g. $E_1 = 0.1 \text{ kV}$ $E_0 = 1 \text{ kV}$ $E_2 = 1.3 \text{ kV}$ $E_2 = \eta + \delta = 1$ (specimen -ve charged to neutral) $\approx 1-10 \text{ kV}$

No charging at 1.0 kV



(a) 1.0kV Image without charging because the charge equilibrium is obtained. ×3,200



(b) 1.3kV Image with charging

×3,200

Coating



Fig.1 Usage of the coating material according to the granularity (for the SEM observation).

Purpose	Major coating material	
SEM observation (secondary electron image)	Au, Pt, Au-Pd	
Observation of backscattered electron image	С	
Elemental analysis	C, Al, Au	
Magnetic domain or channeling pattern of nonconductive specimens	С	



Schematic dependence of the interaction volume and penetration depth as a function of incident energy E_0 and atomic number Z of the incident (primary) electrons.

Secondary Electrons

These electrons arise due to inelastic collisions between primary electrons (the beam) and loosely bound electrons of the conduction band (more probable) or tightly bound valence electrons. The energy transferred is sufficient to overcome the work function which binds them to the solid and they are ejected.



The interaction is Coulombic in nature and the ejected electrons typically have $\approx 5 - 10 \text{ eV}$. 50 eV is an arbitrary cut-off below which they are said to be <u>secondary</u> <u>electrons</u>.

Detection

Remember, secondary electrons are <u>low energy electrons</u>. We can easily collect them by placing a positive voltage (100 - 300V) on the front of our detector. Since this lets us collect a large number of the secondaries (50 - 100%), we produce a "3D" type of image of the sample with a large depth of field.

The type of detector used is called a scintillator / photomultiplier tube.



Construction of the secondary electron detector.

SE : Secondary Electrons



Probability of escape of secondary electrons generated at a depth Z below the sample surface (Koshikawa and Shimizu 1974).

Secondary Electronns : SE Topography



Fig. 4.21. The escape probability of a secondary electron depends on the surface topography.


Collection of secondary electrons from a three-dimensional specimen by a detector with a positively biassed grid.

SE – strong topographic contrast



Backscattered Electrons

Backscattered electrons (BSE) arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher Z, more BSE emitted.



As the name implies, elastic scattering results in little (< 1 eV) or no change in energy of the scattered electrons, although there is a change in momentum (p). Since p = mv and the mass of the electron doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0 to 180°.

BSE Detection

Since BSE have high energies, they can't be pulled in like secondaries. If you placed a potential on a grid to attract them, you would also attract the incident beam!!

The most common detector used is called a surface barrier detector. It sits above the sample, below the objective lens. BSE which strike it are detected.



Back-scattered Electrons : BSE 1000 Å (1µm) depth



Transmission coefficient η_T and backscatter coefficient η as a function of film thickness for a copper target, $E_0 = 10$ keV. Solid lines represent experimental results (Cosslett, 1966); points represent individual Monte Carlo calculations (Newbury and Yakowitz, 1976).

BSE versus SE: Z atomic number



Comparison of backscattered electron coefficients and secondary-electron coefficients as a function of atomic number (Wittry, 1966; Heinrich, 1966).

BSE – effect of kV



Variation of the backscatter coefficient as a function of atomic number at $E_0 = 10$ keV and $E_0 = 49$ keV. Data of Heinrich (1966a).

BSE – effect of Tilt



Backscatter coefficient of iron as a function of tilt. Beam energy 30 keV. Monte Carlo calculations and experimental measurements. Newbury *et al.* (1973).

Mean Z for Minerals

Mean Z	Mineral	Mean Z	Mineral		
6.0	Graphite	16.4	Brookite		
7.7	Borax	16.4	Rutile		
9.4	Magnesite	16.5	Perovskite		
10.0	Zoisite	16.5	Siderite		
10.0	Spodumene	16.9	Ferrosilite		
10.2	Clinochlore	17.2	Tephroite		
10.3	Serpentine	18.6	Malachite		
10.4	Humite	18.7	Fayalite		
10.4	Kaolinite	19.0	Ilmenite		
10.4	Periclase	19.9	Chromite		
10.5	Analcite	20.0	Ulvospinel		
10.6	Forsterite	20.6	Haematite		
10.6	Glaucophane	20.7	Pyrite		
10.6	Spinel	21.0	Magnetite		
10.6	Topaz	22.2	Pyrrhotite		
10.7	Albite	22.9	Pentlandite		
10.7	Andalusite	23.5	Chalcopyrite		
10.7	Corundum	23.7	Celestine		
10.7	Enstatite	24.2	Apophyllite		
10.7	Jadeite	24.2	Xenotime		
10.7	Kyanite	24.6	Columbite		
10.7	Pyrope	24.6	Willemite		
10.7	Sillimanite	24.8	Zircon	13.6	Wollast
10.8	Quartz	25.3	Bornite	13.8	Montec
11.1	Lepidolite	25.4	Sphalerite	14.1	Apatite
11.1	Mullite	25.6	Strontianite	14.2	Arfved
11.1	Muscovite	26.4	Chalcocite	14.7	Fluorit
11.1	Sodalite	26.7	Cuprite	14.7	Titanite
11.9	Anorthite	26.8	Benitoite	15.0	Riebecl
11.9	Orthoclase	27.2	Celsian	15.2	Spessar
12.1	Leucite	27.3	Arsenopyrite	15.3	Hercyn
12.4	Aragonite	27.6	Cobaltite	15.3	Uvarov
12.4	Calcite	29.0	Copper	15.6	Almano
12.4	Gypsum	31.7	Baddeleyite	15.8	Andrad
12.9	Grossular	32.5	Tetrahedrite	15.9	Rhodo
13.4	Anhydrite	37.3	Barite		

Table 4.2. Minerals in order of mean atomic number

Vollastonite	38.7	Monazite
Montecellite	41.1	Cassiterite
Apatite	41.1	Stibnite
Arfvedsonite	41.3	Witherite
Fluorite	43.0	Argentite
Fitanite	59.4	Anglesite
Riebeckite	65.3	Cerussite
Spessartine	65.4	Tantalite
Hercynite	70.5	Bismuthinite
Jvarovite	73.2	Galena
Almandine	78.0	Platinum
Andradite	79.0	Gold
Rhodochrosite	82.0	Uraninite

Argile - électrons rétrodifusés - contraste Z



(3)

FeS₂

Figure 3.49. An SEM image formed from the backscattered electrons whose intensity depends on the average atomic number of the specimen being bombarded. The specimen shown here is a polished rock slice of shale. The brightest grains are pyrite, FeS₂, the grey grains are ferromagnesian silicates such as mica and pyroxene, and the darkest contrast grains are quartz SiO₂. The numbered labels refer to crystals whose X-ray spectra are shown in Figure 3.50.

PHN 1611 - 220 km 1600[]C



Kimberlite nodules From South Africa

Geoff Lloyd (Leeds,UK)



Accelerating Voltage (kV) : depth



Four Key Parameters for SEM



Unités de pression

	Pa	bar	mbar	torr	mmHg	atm
Pa	1	10 ⁻⁵	10 ⁻²	7,5 10 ⁻³	7,5 10 ⁻³	9,869 10 ⁻⁶
bar	10 ⁵	1	10 ³	750	750	0,9869
mbar	10 ²	10 ⁻³	1	0,75	0,75	9 , 869 10 ⁻⁴
torr	133,3224	1,333 10 ⁻³	1,33322	1	1	1,3158 10 ⁻³
mmHg	133,3	1,333 10 ⁻³	1,33322	1	1	1,3158 10 ⁻³
atm	1,013 10 ⁵	1,013	1013	760	760	1

Conversions entre les principales unités de pression

Pour résumer, rappelons-nous que le millibar vaut un hectoPa, qu'une atmosphère correspond à 1013 millibars ou à 760 mmHg, et qu'un torr est équivalent à un mmHg.

Pressure variable (PV), low vacuum (LV) or Environmental SEM



Schematic diagram illustrating the principle of the LVSEM for observation of nonconductive specimens.

- (a) Decreasing the vacuum to from 10⁻⁴ to 15 Pa increases the number of residual gas molecules in the chamber.
- (b) The electron beam ionizes residual gas molecules in vacuum to create a number of positive (+ve) ions.
- (c) The positive ions neutralizes the excess electron negative (-ve) charge on the specimen surface.

Environmental Scanning Electron Microscope

Le microscope environnement est un microscope présentant l'avantage de pouvoir travailler en trois modes qui sont :

- Le mode balayage classique (CSEM) sous vide secondaire (10-7 à 10– 4 Torr) pour des échantillons conducteurs et non déshydratés.
- Le mode Low Vacuum (LV-CMEM), la chambre est en vide partiel de 0.1 à 2 Torr pour des échantillons non métallisés.
- Le mode environnemental (E-SEM), sous vide partiel de 0.1 à 50 Torr en présence d'un gaz tel que la vapeur d'eau, qui permet d'étudier des échantillons fragiles, hydratés et non métallisés.



CrystalProbe = LV-CSEM http://www.gm.univ-montp2.fr/PERSO/mainprice/CrystalProbe.html

Phase Diagram for water at SEM pressures and temperatures



Hydrocarbon particule in diesel



Diagramme d'équilibre Pression Température de l'eau



Elements of SEM : specimen stage



Construction of the specimen stage.

Tilting Stage



Non-Eucentric Stage

- a) Beam position on specimen is changed when stage is tilted (black to white dot)
- b) The focus position (WD) is changed for a for tilted specimen when moved in the X-direction

Eucentric Stage

- a) Beam position on specimen is NOT changed when stage is tilted
- b) The focus position (WD) is NOT changed for a for tilted specimen when moved in the X-direction

EBSD = Electron Back Scattered Diffaction



Why should we be interested in EBSD ?

- Can work with tradition petrographic thin sections (combine with studies using probes...)
- Measure the orientation of transparent and opaque minerals of any symmetry
- Measure orientations of rocks with complex mineralogies (real rocks !)
- Measure orientations and misorientation of crystals (e.g.twins, subgrains..) in the microstructure at submicron to cm scale

Diffraction Pattern	Microscope	Spatial	Angular	Specimens
Spot	TEM	0.5 µm	1°	thin films
Kikuchi	TEM	0.5 µm	0.1°	thick films
Kikuchi	STEM	10 nm	0.2°	thick films
ECP	SEM	1-10 μm	0.1°	bulk samples
EBSD	SEM	1-2 μm	1°	bulk samples
EBSD	FEG-SEM	100 nm	1°	bulk samples

Table: Spatial and angular resolution of orientation measurements



A little history



- First electron backsacttered diffraction by Kikuchi in 1928 on a thin mica crystal.
- Later in 1954 Alum et al. published patterns of lead sulfide.
- First SEM based work by Coates in 1967.
- First SEM/EBSD by Venables in 1972.
- First EBSD on polycrystalline materials by Dingley in 1981.
- 1985 first computer controlled system... 21 years ago



Forescatter detector

Effect of tilt angle on EBSD patterns







55°

Background correction





scan

х-у





Raw pattern recorded in spot mode

Background pattern recorded in scanning mode Background corrected pattern



Example of EBSP image processing. (a) A raw EBSD pattern from tungsten carbide. (b) The EBSD pattern background signal. (c) The corrected EBSD pattern produced by dividing the raw pattern by the background and then adjusting the brightness and contrast of the resulting image to fit the available gray-level range. (d) Pattern after single frame averaging. (e) Pattern after frame averaging over three frames. (f) Pattern after frame averaging over 20 frames. (Courtesy of K. Mingard and A. Day.)





A straight line is defined by the ρ and θ

In Hough space a straight line corresponds to a point

$$x \cos \theta + y \sin \theta = \rho$$

Hough Space





(a) original image

(b) Hough-space

Figure 6.8 Schematic representation of the Hough transform. (a) Two bands in the original image; (b) bands-from (a) in the Hough space.

Diffraction Pattern



Hough Transform



Detected Bands



Indexed EBSP



Crystallographic Orientation







Tilt 70° 15 kV

Geometry of a backscattered Kikuchi bands






Working Distance & Pattern Centre



Si [100] wafer calibration of camera length and PC

Calibrating the Phase ID Geometry

In order to accurately analyze the Kikuchi pattern, the identification geometry is first calibrated. Using a pattern from a standard Si [100] wafer specimen, the software automatically performs the calibration. It finds the exact location of the pattern center on the Kikuchi pattern and the diffraction camera length. The pattern center is the (x, y) location on the pattern that traces back to the impact position on the specimen of the electron beam. The camera length is the distance from the impact position on the specimen to the pattern center. This image *must* be used to accurately index all patterns acquired using this analysis geometry.



Pattern Loaded

A flat field corrected pattern is loaded for phase analysis.

Analyzing the Pattern

Once the software is calibrated for the analysis geometry, a Kikuchi pattern is analyzed. The extracted parameters are used in a search of the ICDD phase database. Most of the tedious work and all of the mathematics have been automated so that the optimum phase selection is done in a timely manner.

The software performs a Hough transform, which extracts the dominant Kikuchi bands from image. The widths of the bands and their relative angles are calculated.



Si [100] wafer calibration

Pattern Simulation

If requested, a simulation of the Kikuchi pattern for the selected crystal can be overlaid on the pattern. The complete line pattern and zone index markers are overlaid on the Kikuchi pattern. Zone text markers are automatically provided and planar text markers can be shown, if desired.



Pattern Center (PC) and Working Distance (WD)

A) Near top of screen



electron beam

B) Middle of screen









Parameters required for EBSD orientations measurements: pattern source point on the specimen, SP, pattern centre on the recording screen, PC, specimen-to-screen distance L (or Z_{SSD}), and three sets of orthogonal axes, xyz (screen/pattern axes), $X_s Y_s Z_s$ (specimen axes) and $X_m Y_m Z_m$ (microscope axes).



An EBSD pattern of (001) silicon with the specimen normal direction labeled A and the pattern center labeled B.

Typical commerical

nanmatry



Oxford Instruments (UK)





Fig. 1. (a) schematic diagram showing the geometry of the specimen and scintillator configuration used for EBSD. (b) an EBSD pattern from Si obtained by exposing an em film plate in the SEM specimen chamber. The circle marked on this pattern indicates the reduced capture angle ($\sim 14^{\circ}$) used in this work.

The geometry of an EBSD system



Kinematic intensities - good approximation for EBSD ?



FIGURE 2. (a) EBSP patterns from calcite (CaCO₃), with a $\{10\overline{1}4\}$ cleavage surface inclined by 70° toward the phosphor screen. (b) Calculated pattern for calcite in the same orientation. In (b), Kikuchi bands are shown as a pair of straight lines whose color (depth) is proportional to the kinematical-diffraction amplitude of the corresponding reflections.

Calcite {1 0 -1 4} cleavage

Kogure 2002 AM





EBSD Maps - Beam or Stage scanning



EBSD Maps - Combined Beam and Stage scanning



to select beam scan "square"



EBSD System : UMR Tectonophysique - Université Montpellier 2

Tilt (70°) corrected images



No correction



Corrected



Home-made Tilt correction electronics

Géosciences - Université Montpellier II EBSD system



Specimen Preparation

1) Polished thin sections or blocks.

2) The mechanical surface damage can be removed by:

- a)Chemical-mechanical (SYTON) polish.
- b)Etching.
- c)Electro-polishing.
- d)lon beam milling.

3) Natural fracture or growth surfaces.

4) Uncoated or very thin carbon or Pt coat.

Typical sample preparation for EBSD

Impregnate sample with resin to avoid holes & surface topography

A) Lap with 220 grit silicon carbide until flat.

B) Lap with 500 grit silicon carbide for 5 mins.

C) Polish with 9 micron diamond for 15 mins.

D) Polish with 3 micron diamond for 15 mins.

E) Polish with 1 micron diamond for 15 mins.

F) Syton polish for 1 - 20 hours.

The charging problem....

A) Low energy (0.1-3 kV) electron beam (only negative charge)

B) Ion beam (postive or negative charges)

C) Ultraviolet light

D) Low vacuum SEM (need NEW SEM !)





K-feldspar. 20keV ~15nA Image from David Prior, University of Liverpool, UK



Illustration of the penetration depth of the electron beam in a silicon EBSD specimen. (a) No coating, 40 kV accelerating voltage; (b) no coating, 10 kV accelerating voltage; (c) coating with 5 nm of nickel, 40 kV accelerating voltage; (d) coating with 5 nm nickel, 10 kV accelerating voltage. There is less beam penetration at 10 kV since the underlying silicon pattern is indistinct. (Courtesy of J.R. Michael.)

1. What is EBSD ?

2. Symmetry and EBSD

- 3. Applications
- 4. Future developments

Symmetry for routine indexing of known phases

Diffraction symmetry – Friedel's law



X-ray reflection at both sides of a set of lattice planes (Friedel's law)



Friedel's Law: The intensities of reflections (structure factors) hkl et -h-k-l are equal: $|F(hkl)|^2 = |F(-h-k-l)|^2$

11 Laue Classes or Groups



Fig. 5. Symmetry elements and crystallographers' conventions for the choice of axes in the 11 Laue groups (which are all centrosymmetric). (Note that, elsewhere in this book, *x* and *y* are usually used such that the quadrant with positive *x* and positive *y* is the upper right one.)

		crystal class	no. of	no. of centro-sym. ten	no. of centro-sym. tensor prop's.	
CRYSTAL SYSTEM		Laue rot.	sym. el.	sym. 2 nd -rank sym.	sym. 2 nd -rank sym. 4 th -rank	
triclinic		Ī 1	1	6	21	
monoclinic		2/m 2	2	4	13	
orthorhombic		mmm 222	4	3	9	
	†	4/m 4	4	2	7	
tetragonal		4/mmm 422	8	2	6	
	†	<u>3</u> 3	3	2	7	
trigonal	ŧ		6	2	6	
	†	6/m 6	6	2	5	
hexagonal		6/mmm 622	12	2	5	
	ŧ	m3 23	12	1	3	
cubic		m3m 432	24	1	3	

Table I. Crystal symmetries relevant for orientations

† These groups have lattice planes which have the same spacing but are not symmetrically equivalent; thus they exhibit overlapping peaks in diffraction patterns; an example is (121) and (211) in crystal class 4/m.





FIG. 2–51 Left-handed and right-handed quartz crystals. (After E. S. Dana, A Textbook of Mineralogy, revised by W. E. Ford, 4th ed., Wiley, 1955.)

QUARTZ TWIN LAWS





Fig. 200 Growth twinning (Brazil twinning) of quartz. A. The morphology of a Brazil twin: forms present; prism $\{10\overline{1}0\}$; rhombohedra $\{10\overline{1}1\}$ and $\{01\overline{1}1\}$ and trigonal trapezohedra $\{51\overline{6}1\}$. B. An etched basal section showing plane composition surfaces.



Fig. 202 Dauphiné twinning of quartz.
A. The morphology of a Dauphiné twin.
B. An etched basal section showing irregular composition surfaces.
Brazil twinning shown to right.

EBSD - forward scattering image : Dauphiné Twins





ECP orientation contrast of Dauphiné twins

Mainprice, Lloyd and Casey 1993
Dauphiné Twin related orientations <u>с</u> 73 Z a m 🗆 z z 74 1 r 73 z 73 z 74 a 74 m 74 Υ a 73 a 73 Х m 73 m 74 m 73 00 a 74 a 73 m 73 m 74 a 74 74 z 73 z 73 Twin Numbers

7374

Mainprice, Lloyd and Casey 1993

Quartz : - Indexing Dauphiné Twins







Feldspar forward scattered image Dr. David Prior (Liverpool,UK)

Orientation contrast images are not quantatitve

Two grains of the same orientation
can have different signalsTwo grains of different orientation
can have the same signal



EDS X-ray dectector and EBSD



Phase ID : EDS + EBSD



XRD also identifies the same ICDD Card #4-787 for Aluminum.

EBSD data



Example - BaTi0₃ powder



SEM image of compressed powder sample (70 degree tilt).



EDS spectrum of compressed powder sample.



Pattern of compressed powder sample.

Card #: 05-0626 Entry #: 2 of 101 four Name: Barium Titanium Oxide		C	rystal Identification		
Name: Barium Titanium Oxide	Card #:	05-0626	Entry #: 2	of 101	found
	Name:	Barium Titanium (Dxide		
Mineral:	Mineral:				

Summary of selected crystal: tetragonal Barium Titanium Oxide (BaTiO₃).



Simulation of BaTiO₃ overlaid on pattern.



- 1. What is EBSD ?
- 2. Symmetry and EBSD

3. Applications

4. Future developments

Applications of EBSD

- 1. Orientation of crystals (most common use).
- 2. Measuring elastic strain from diffraction pattern disortion -> elastic stress
- 3. Estimating plastic strain from quality of diffraction pattern.
- 4. Aiding phase identification (e.g. coupled with EDS X-rays).
- 5. Determination of lattice constants.

 Table 2. Comparison of EBSD with conventional methods of quantitative metallography for a typical sample.

Application	Conventional technique(s)	Approximate EBSD data acquisition time (min)	Advantages of EBSD	Limitations of EBSD
Phase fractions (point count)	Optical SEM	20	Speed, Accuracy, Automation	Phases must diffract
Grain size (linear intercept)	Optical SEM	20	Speed, Accuracy, Automation, Boundary information	Requires undeformed or coarse subgrain stucture
Grain size (image reconstruction)	Optical SEM + image analysis	120	Boundary characterization	Speed Requires undeformed or coarse subgrain structure
Boundary misorientation distributions (linear intercept)	TEM	20	Speed, Automation	Requires undeformed or coarse subgrain structure
Subgrain size (linear intercept)	TEM SEM	20	Speed Automation	Minimum size $\sim 2 \mu m$
Subgrain size (image reconstruction)	TEM SEM	120	Full characterization	Minimum size $\sim 2 \mu m$
Subgrain misorientation (linear intercept) Fraction recrystallized (point count)	TEM Optical SEM	20 20	Speed Automation	Minimum size $\sim 2 \mu m$ Minimum misoriention $\sim 1^{\circ}$ Requires careful calibration Unsuitable for many samples
Fraction recrystallized (linear intercept)	Optical SEM	20	Early stages of recrystallization	Requires coarse subgrain structure
Bulk texture (point count)	X-ray	20	Speed Through thickness measurements possible	Small samples Requires undeformed or coarse subgrain stucture

Bio-materials - Mollusc shell





Poor pattern quality Low indexation rate

 $\{100\}$ and $\{001\}$ pole figures of *Crassostrea gigas*, obtained from EBSD (top row) and x-ray (bottom) analyses. Max = 100 m.r.d. Logarithmic density scale, equal area projections.

Aragonite

Daniel Chateigner HDR 2000

Pattern symmetry analysis : Silicon

Zone axes with 4mm, 3m and 2mm point group symmetries

Identification as m3m



EBSD pattern of Si with symmetry axes indicated.

Some cubic whole pattern symmetries for EBSD point group determination.

Point Group						
	<111>	<100>	<110>	<uu0></uu0>	<uuw></uuw>	<uvw></uvw>
m3m	3m	4mm	2mm	m	m	1
43m	3m	2mm	m	1	m	1
432	3	4	2	1	1	1

AFC Toulouse 2006

Polymorphs - SiC using HOLZ rings



EBSD patterns obtained from two polytypes of SiC. The HOLZ rings analyzed are indicated by arrows. (a) EBSD pattern from 6H polytype, and (b) EBSD pattern from 15R polytype.

6H polymorph (1.858nm) 15R polymorph (1.545nm) UVW = 2 4 1 UVW = 10 5 1J.R. Michael & J.A. Eades (2000) Ultramicoscopy

AFC Toulouse 2006

Polymorphs - Lepidolite

Lithium rich mica



FIGURE 4. Four EBSD patterns acquired from the different terraces in a lepidolite crystal from Sample C. The pattern analysis indicated that $2M_1$ and 3T (or 1Md) regions are embedded in a $2M_2$ matrix.

AFC Toulouse 2006

Kogure & Bunno 2004 AM

Exsolution : Diopside in Wollastonite



FIGURE 4. The relations between the wollastonite matrix and the diopside lamellae. (a) Shapes of the lamellae and the cross section (optical microscopy) and (b) the detailed crystallographic relation (see context).

Wollastonite - CaSiO₃ pyroxenoid

Diopside - CaMgSi₂O₆ pyroxene

Seto et al 2006 AM



Figure 1. Schematic diagram for detection of EBSD patterns.

Quartz Sphere



Indexing: The basis of this is to match a sample EBSP to some knowledge of all possible EBSPs for the crystal structure of interest

ECP « globe » for quartz by Geoff Lloyd (Leeds,UK)

Automatic indexing is now possible





Spatial resolution of EBSD in nickel as a function of accelerating voltage. (Adapted from Drake, A. and Vale, S.H., *Inst. Phys. Conf. Ser.* 147, 137, Inst. Phys. Pub. Inc., Bristol, 1995.)



(a) The effect of probe current on effective resolution for several aluminum specimens. The minima in the plots are caused by the reduced pattern-solving accuracy at low probe currents. (From Humphreys, F. J. et al., *J. Microsc.*, 195, 212, 1999.) (b) Effective EBSD spatial resolution for various metals in tungsten filament and FEG SEMs. (From Humphreys, F.J., *Scripta Mater.*, 51, 771, 2004.) (c) Misorientation measurements between adjacent points on a single-crystal silicon specimen for four different probe currents (in amperes). The highest-probe current provides the most accurate result. (Courtesy of F. J. Humphreys.)

EBSD Mapping

Crystallographic Mapping of Kimberlite xenolith : beam scanning



Raw data on SEM image Raw O

Raw Orientation Map

Extrapolated Map

Crystallographic Mapping of Kimberlite xenolith



Kimberlite Xenolith

[100],[010] & [001] Olivine pole figures







Current level of EBSD mapping



Olivine coloured by orientation Cpx – grey level by band contrast

Cpx – coloured by orientation **Olivine** – grey level by band contrast

9510-16 Low cpx lherzolite

1cm scale bar 50 micron steps

Orientation of a crystal defined by 3 Euler angles



Beware there many different conventions - here the convention of Bunge (1982)





Cartesian Reference Frame I

- Measurement of orientation using Euler angles requires the definition of a right-handed Cartesian (also called orthonormal) system in crystal co-ordinates.
- For cubic, tetragonal and orthorhombic the obvious choice is to use the orthogonal lattice basis vectors a[100], b[010] and c [001] of the crystal axes. However, for most general case of triclinic crystal symmetry where a, b, and c are not orthogonal, there are many possible choices and no general convention.
- The choice of a specific reference frame is often imposed by the EBSD software, but users are often not aware what choice has been made.

Cartesian Reference Frame II

- Here are 3 possible choices, for the tensor Cartesian reference frame for Euler angles (e₁,e₂,e₃);
- a) $\mathbf{e}_3 = \mathbf{c}[001]$, $\mathbf{e}_2 = \mathbf{b}^* \perp (010)$ and hence for a right-handed system $\mathbf{e}_1 = \mathbf{e}_2 \times \mathbf{e}_3$ (e.g. BearTex software)
- b) e₃ = c[001], e₁ = a* ⊥(100) and e₂ = e₃ x e₁ (e.g. HKL Channel software)
- c) $\mathbf{e_3} = \mathbf{c}^* \perp (001)$, $\mathbf{e_1} = \mathbf{a}[100]$ and $\mathbf{e_2} = \mathbf{e_3} \times \mathbf{e_1}$



Reference Frames a), b) and c) for triclinic plagioclase Labradorite An66 (**a**=0.817 nm **b**=1.287 nm **c**=1.420 nm α =93.46° β =116.09° γ =90.51°)

Orientation Distribution Function & J index

Measured ODF: f(g) - series of individual orientations g1,g2,g3...gn







Definition of inverse pole figure & Jipf index



5000 Random orientations

Equal angle pole figure



Equal area pole figure



Rotation axis/angle; [uvw]/θ



Fig. inter-Two penetrating lattices with misorientation and θ Ψ. inclination boundary Shown here is the special case of a tilt boundary (asymmetric); in the general case, the axes of θ and Ψ would not be coincident.

Misorientation

- Relative misorientation between crystal A and crystal B in crystal co-ordinates
- Rotation matrix; $\Delta g_{ij} = g^{A}_{ik} \cdot g^{B}_{jk} = g^{A} \cdot (g^{B})^{t}$
- Rotation axis/angle; [uvw]/θ
- Euler angles; $\phi 1 \Phi \phi 2$










Misorientation

- <u>Uncorrelated</u> misorientation between random pairs A and B in the microstructure
- <u>Correlated</u> (physical) misorientation between neighbouring crystals A and B
- <u>Autocorrelated</u> misorientation between crystal A and every other pair B in the microstructure, similar to uncorrelated

Misorientation Histogram



9510-16 Low cpx lherzolite



Noise at low misorientation



Fig. 8. Schematic diagram showing the relationship between the real and measured misorientation distributions and the orientation noise.



Crystallographic relationships

Misorientation Δ g between lattices : olivine - ringwoodite





Y.Fei & C.M.Bertka (1999)



Hélène Couvy & Patrick Cordier

Bayerisches Geoinstitut, Universitaet Bayreuth, Bayreuth, Allemagne LSPES, Université des sciences et technologies de Lille, Lille, France

CPO of Olivine and Wadsleyite in simple shear



CPO Simulation for Wadsleyite



Oxygen Sublattices of Olivine polymorphs



S 3254 - Wadsleyite 60 min



Wadsleyite CPO inherited from Olivine



TEM Photo of S3253 (olivine) Wadsleyite inclusions in Olivine



Phase transformation and Variant selection





Case for olivine (orthorhombic) to wadselyite (orthorhombic)

g = orientation

 Δg = misorientation olivine to wadselyite

S = point group rotations (4 for olivine, so 4 new crystals)

wt = volume faction weighting

$$g(i...4)^{\text{new}} = \Delta g \cdot S(i...4)^{\text{old}} \cdot g^{\text{old}}$$

$$g(i...4)^{\text{new}} = \Delta g \cdot S(i...4)^{\text{old}} \cdot g^{\text{old}} \cdot wt(i...4)$$

Predicted Misorientation for phase transformation

Predicted misorientation in Wadselyite : 72 degrees rotation about [100] axis.

Found in MisOrientation Distribution Function (MODF)



MODF section at 70 degrees rotation angle

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Future developments

In-situ high temperature EBSD

Crystal Probe



In-situ experiments in SEM

Seward et al., 2000 Scanning, 24, 232-240.























