

Imperfections – deformation and microstructures in polycrystals

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3- Orientations in a polycrystal

How to handle and represent orientations in a polycrystal

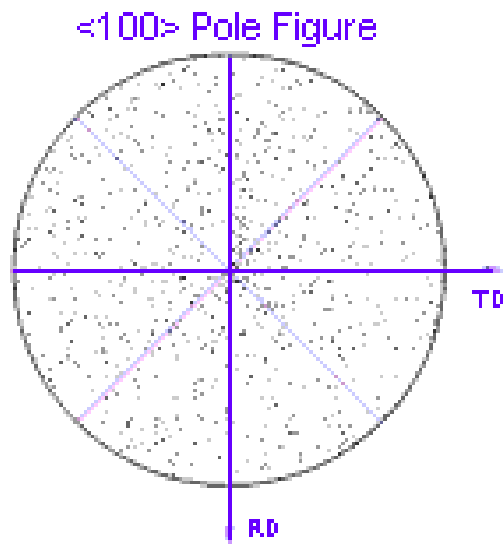
- Pole figures:
 - Introduction,
 - X-ray diffraction measurement,
 - EBSD measurement.
- Mathematical representation:
 - Orientation distribution function,
 - Connection with pole figures,
 - Units.
- Graphical representation:
 - Pole figures,
 - Inverse pole figures.

3- Orientations in a polycrystal a- Pole figures

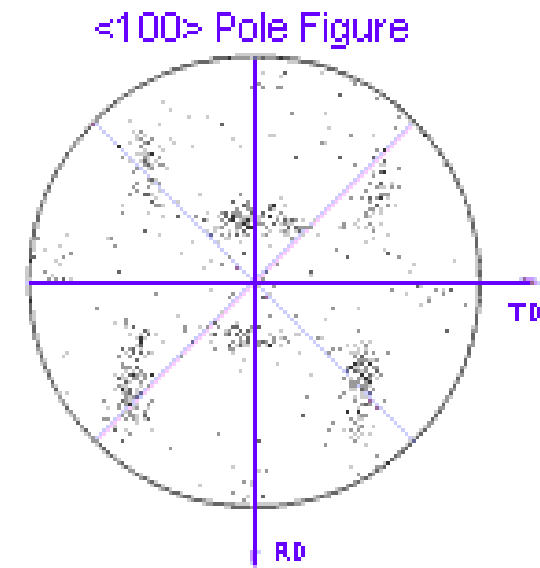
Pole figures

A polycrystal contains thousands of crystallites.

You add all dots from all pole figures for all crystallites in the polycrystal.

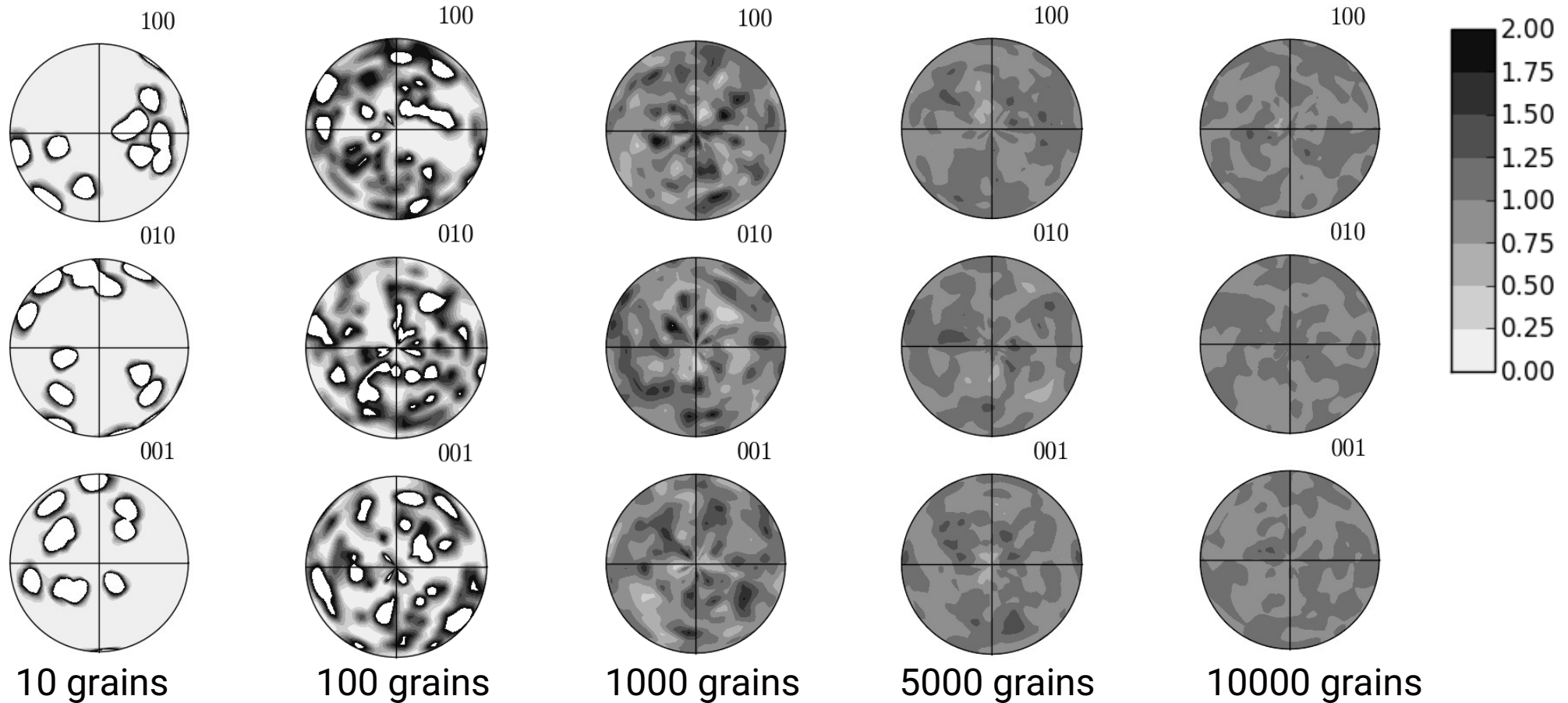


<100> pole figure of a random polycrystal



<100> pole figure of a polycrystal with orientations

You need enough crystallites to extract statistics.



“Random” polycrystal

1000 crystallites minimum for relevant statistics

Texture measurement techniques

X-ray diffraction: surface or bulk measurement (for small samples, less than 1 mm). Measures a projection of the true texture (2 angles instead of 3).

Neutron diffraction: bulk texture (penetration length \sim cm). Measures a projection of the true texture (2 angles instead of 3).

EBSD (Electron BackScattered Diffraction): surface measurement (penetration length \sim nm). Full orientation (3 angles).

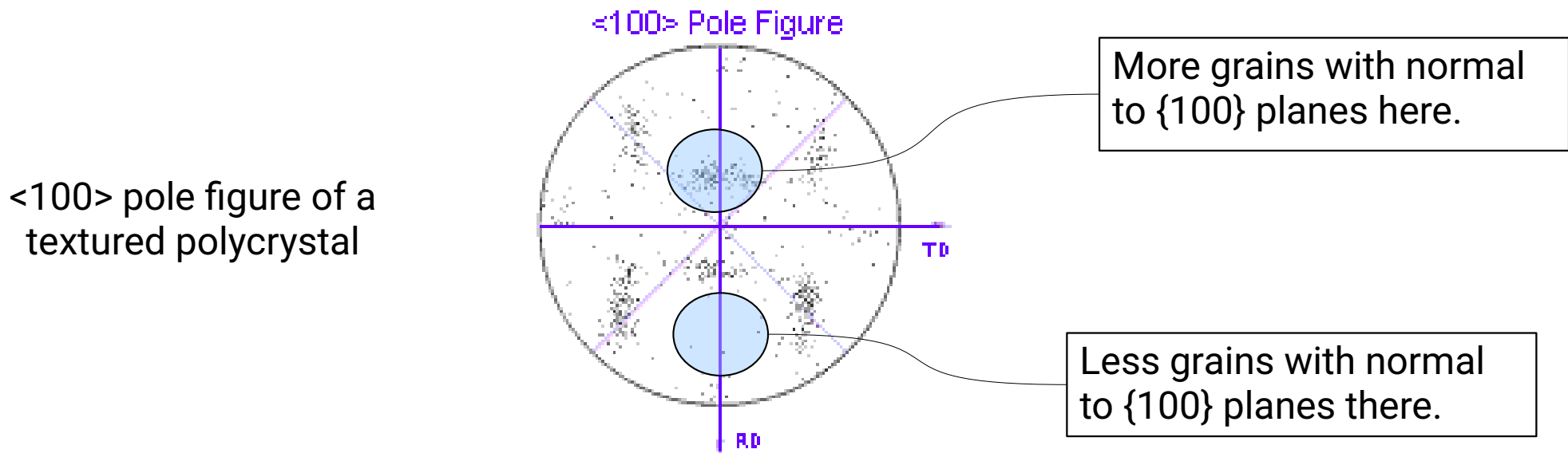
Optical microscopy: limited application (1 angle). Depends on optical properties of the sample.

3- Orientations in a polycrystal ***b-* Powder diffraction measurement**

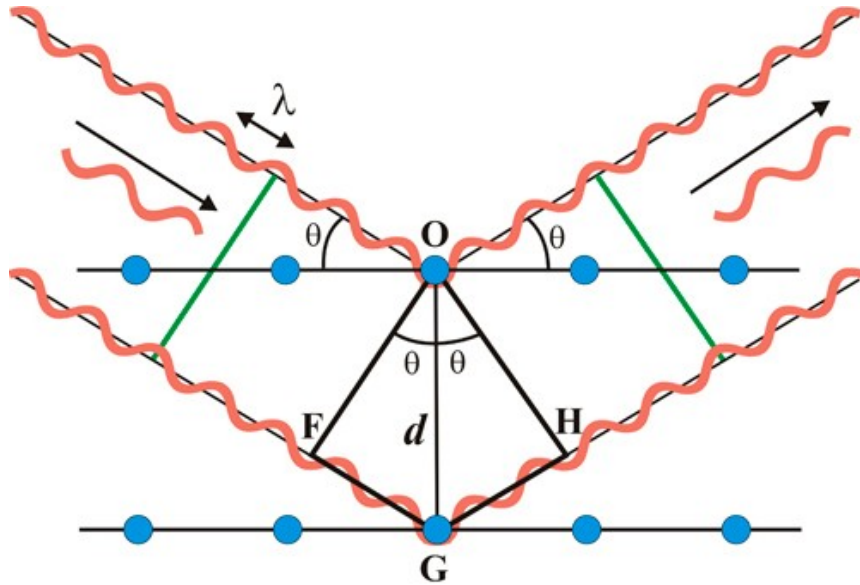
X-ray diffraction measurement

X-ray (and neutron) diffraction directly measure pole figures:

- Variation of diffraction intensity with orientation
- More grains contributing to diffraction → high intensity
- Less grains contributing to diffraction → low intensity
- Pole figures ↔ intensity distribution maps.



Bragg's law

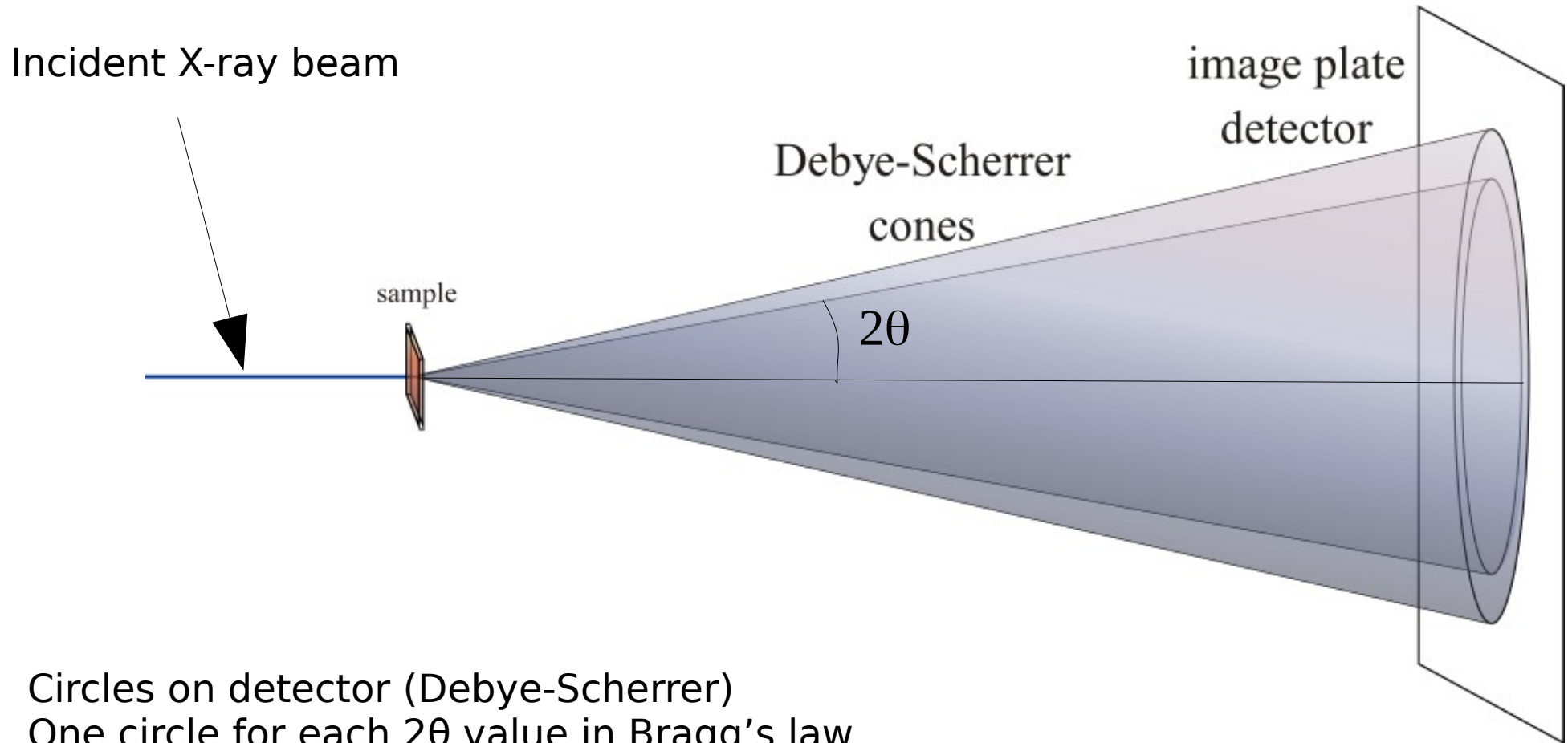


- Bragg's law:
Diffraction at fixed θ
 $n \lambda = 2d \sin \theta$
with
- n : integer,
 - λ : wavelength
 - d : interplanar spacing
 - 2θ : diffraction angle

For a polycrystalline sample with a fixed incoming wavelength λ :

- diffraction peak at fixed 2θ values
- each diffraction peak corresponds to family of planes and a single value of n
- hkl Laue indices of the Bragg reflection indicate the corresponding planes and diffraction order (i.e. 200 can be $n=2$ for $\{100\}$ planes, or $n=1$ for $\{200\}$ planes. It depends).

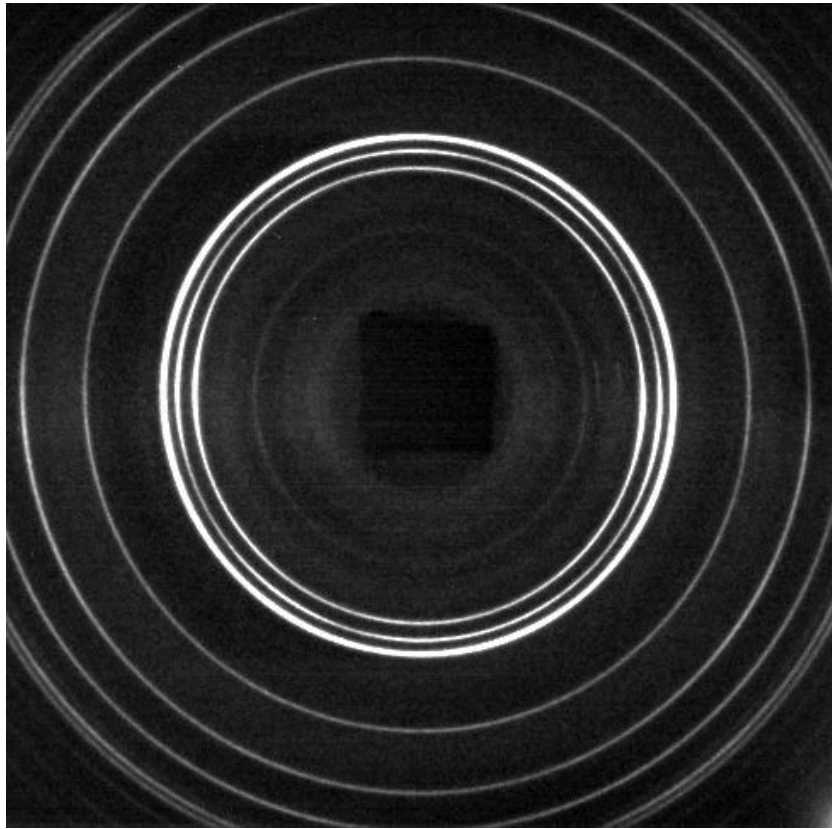
Geometry example



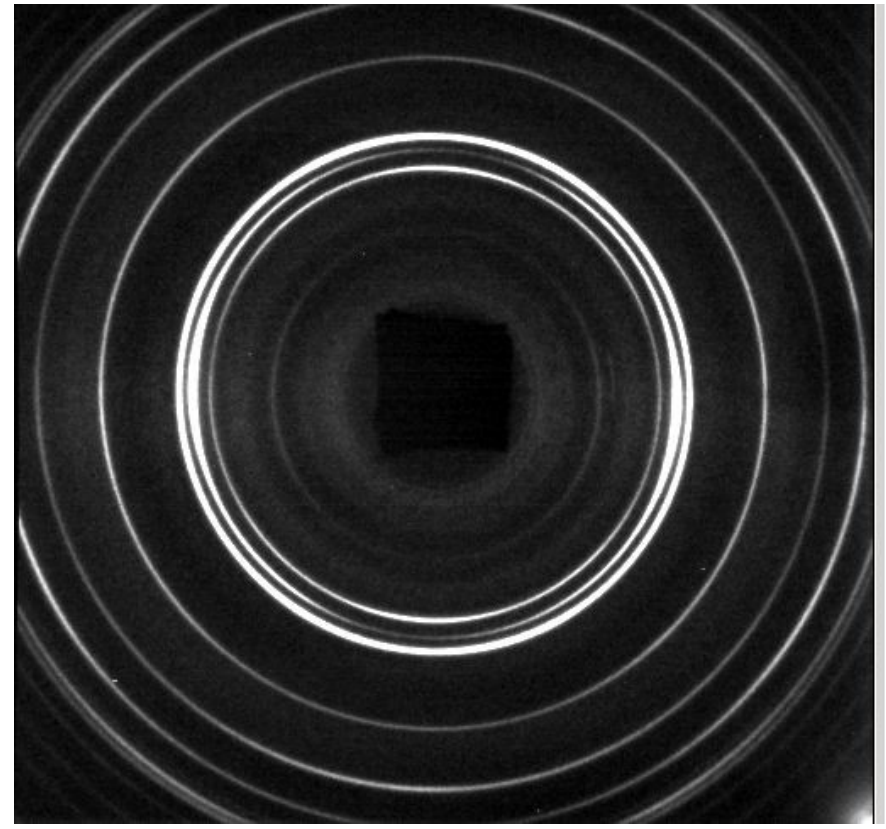
Circles on detector (Debye-Scherrer)
One circle for each 2θ value in Bragg's law
Typical Laue indices of diffraction peaks: 100, 110, 111...

Image Wikipedia

X-ray diffraction example



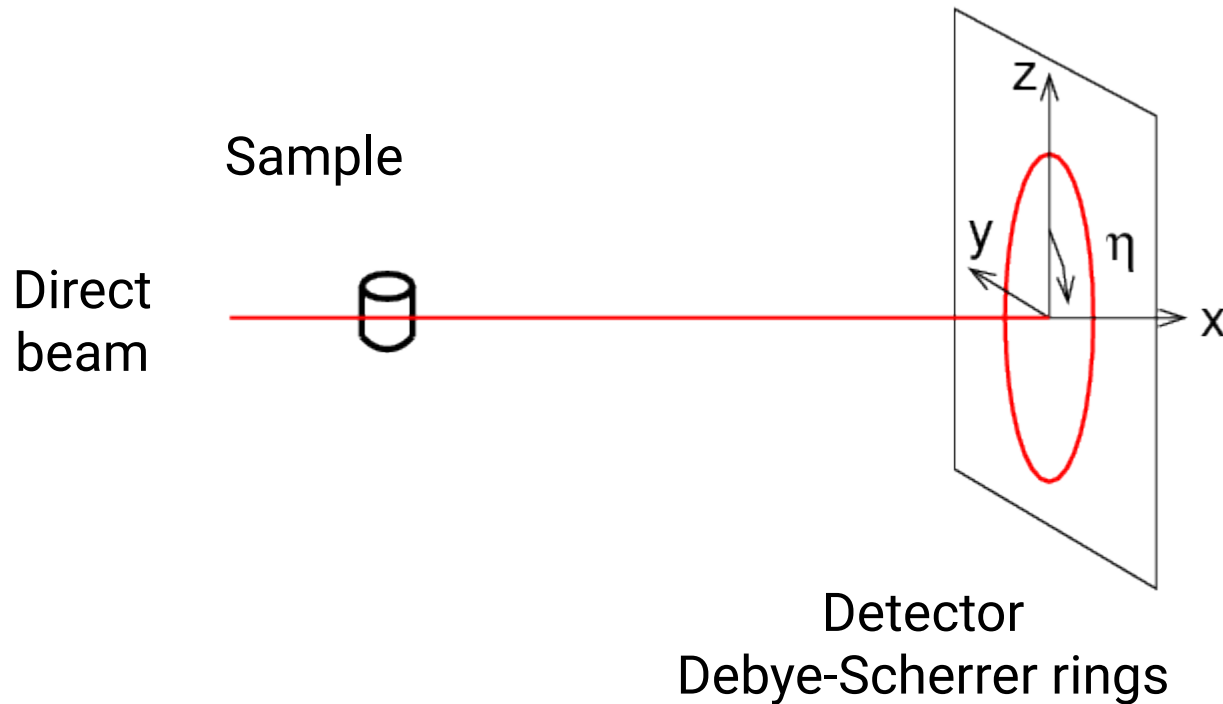
A (nearly) random polycrystal



A textured polycrystal

Sample: ϵ -Fe at high pressure
Axial compression experiment

Sample pole figure measurement

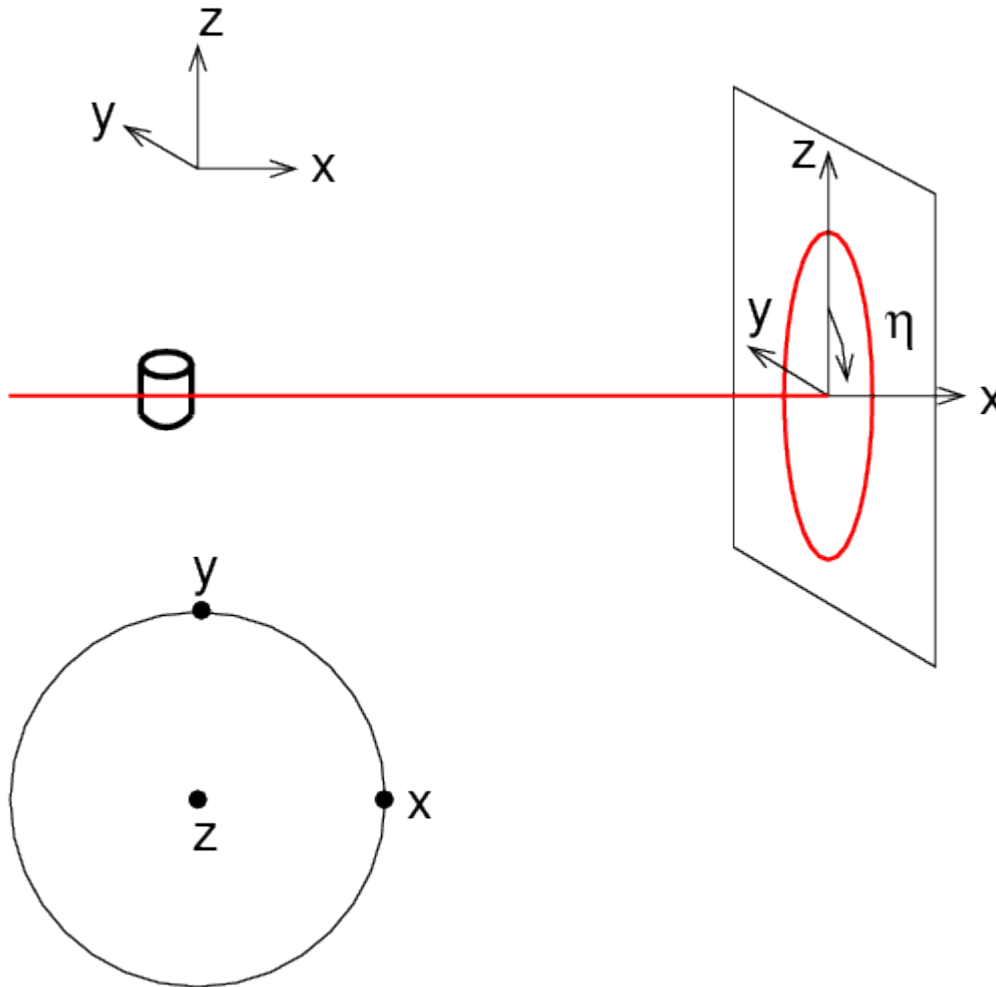


In this geometry, the laboratory reference frame can be defined with

- x // beam
- z // vertical
- y to form a Cartesian frame

2θ : diffraction angle
 η : azimuth angle on detector

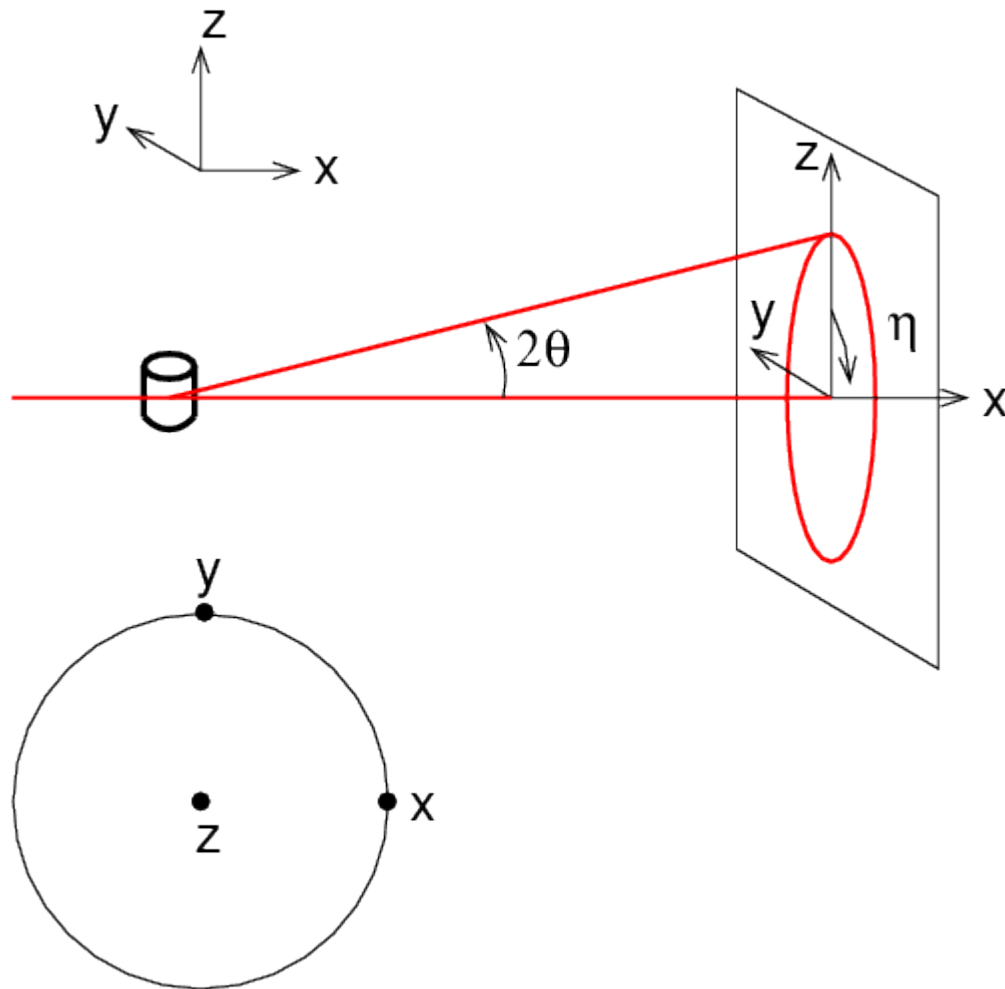
Sample pole figure measurement



Let's plot a pole figure for the Debye-Scherrer ring, in the laboratory reference frame.

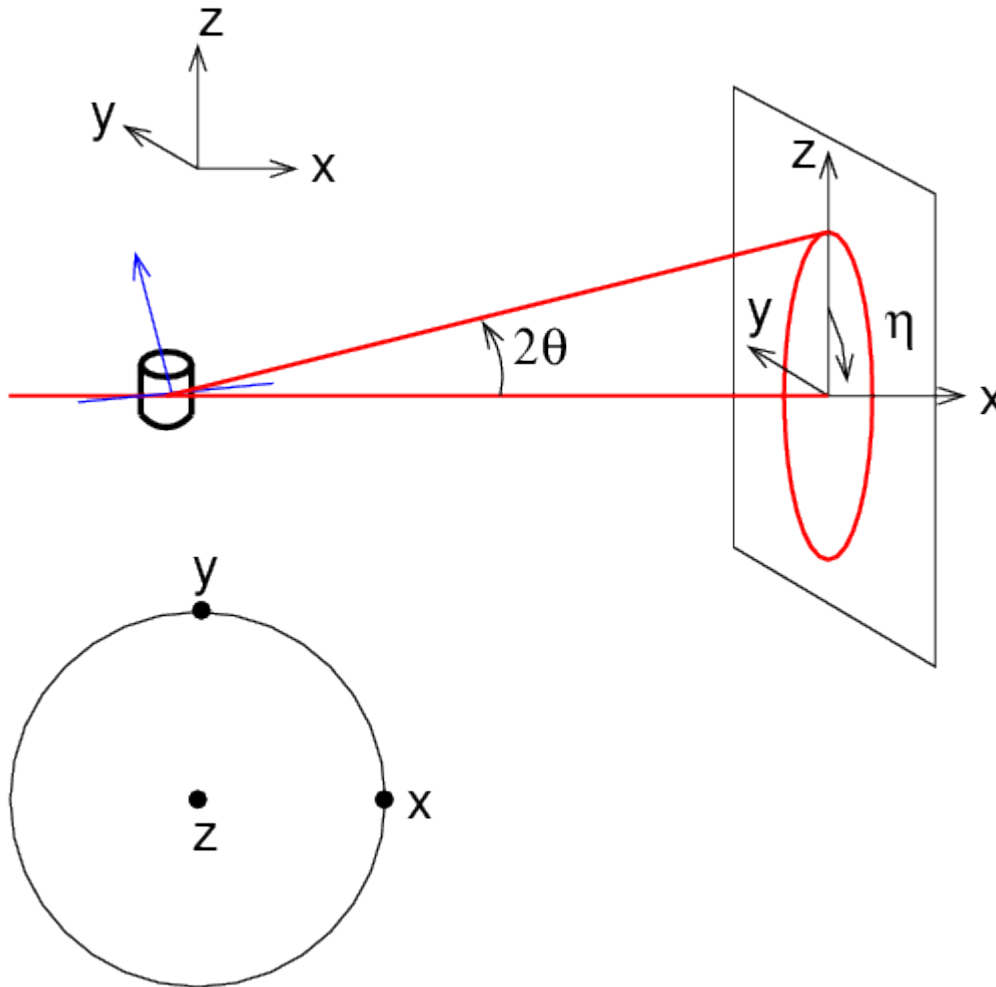
For each orientation, diffraction intensity is proportional to the total crystallite volume in reflection condition.

Sample pole figure measurement



Grains contributing to diffraction at $\eta=0^\circ$

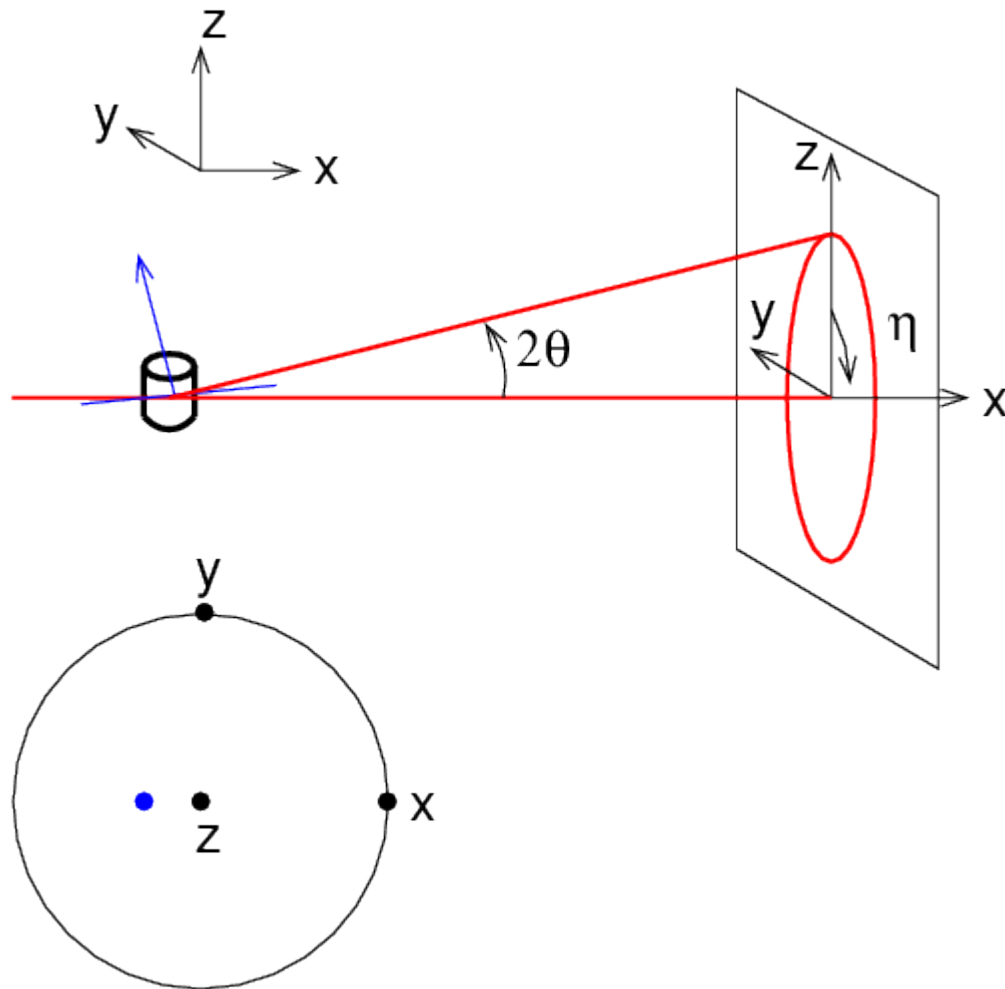
Sample pole figure measurement



Grains contributing to diffraction at $\eta=0^\circ$

Normal to diffracting planes (i.e. diffraction vector)

Sample pole figure measurement

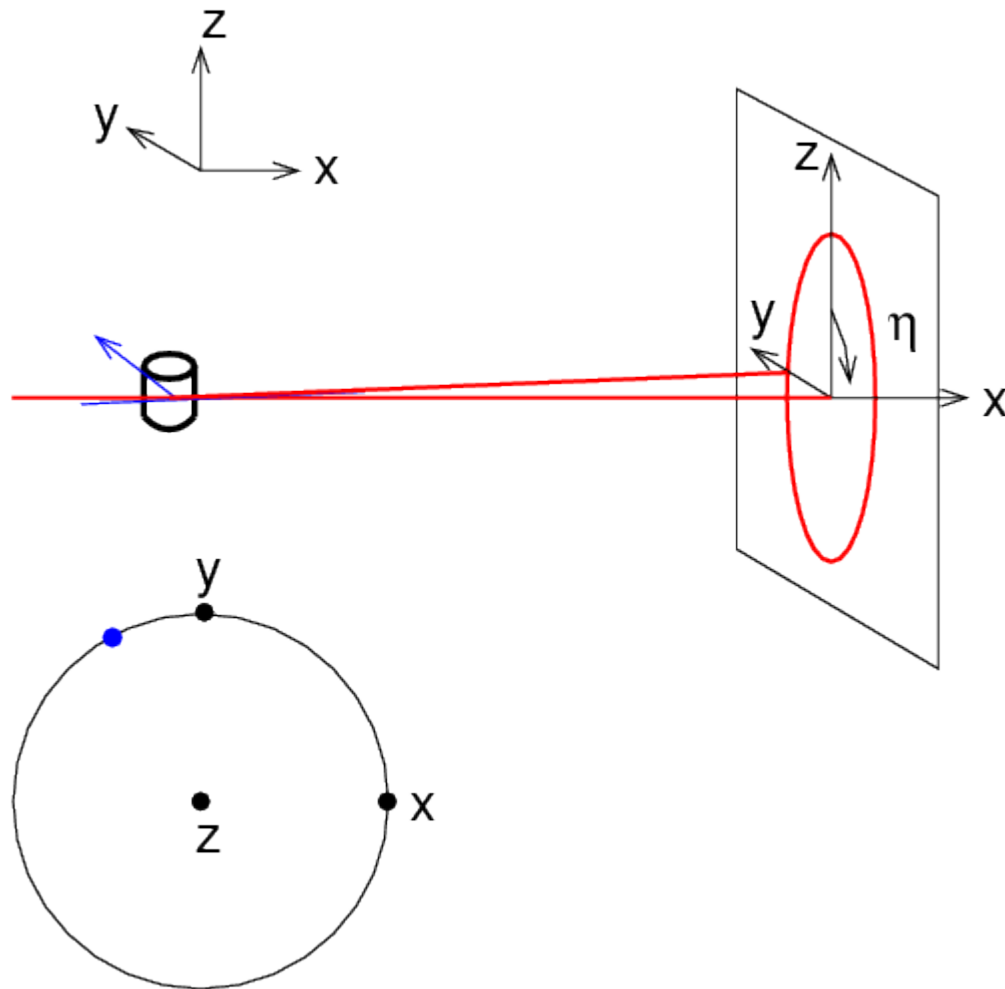


Grains contributing to diffraction at $\eta=0^\circ$

Normal to diffracting planes (i.e. diffraction vector)

Projection in the pole figure

Sample pole figure measurement

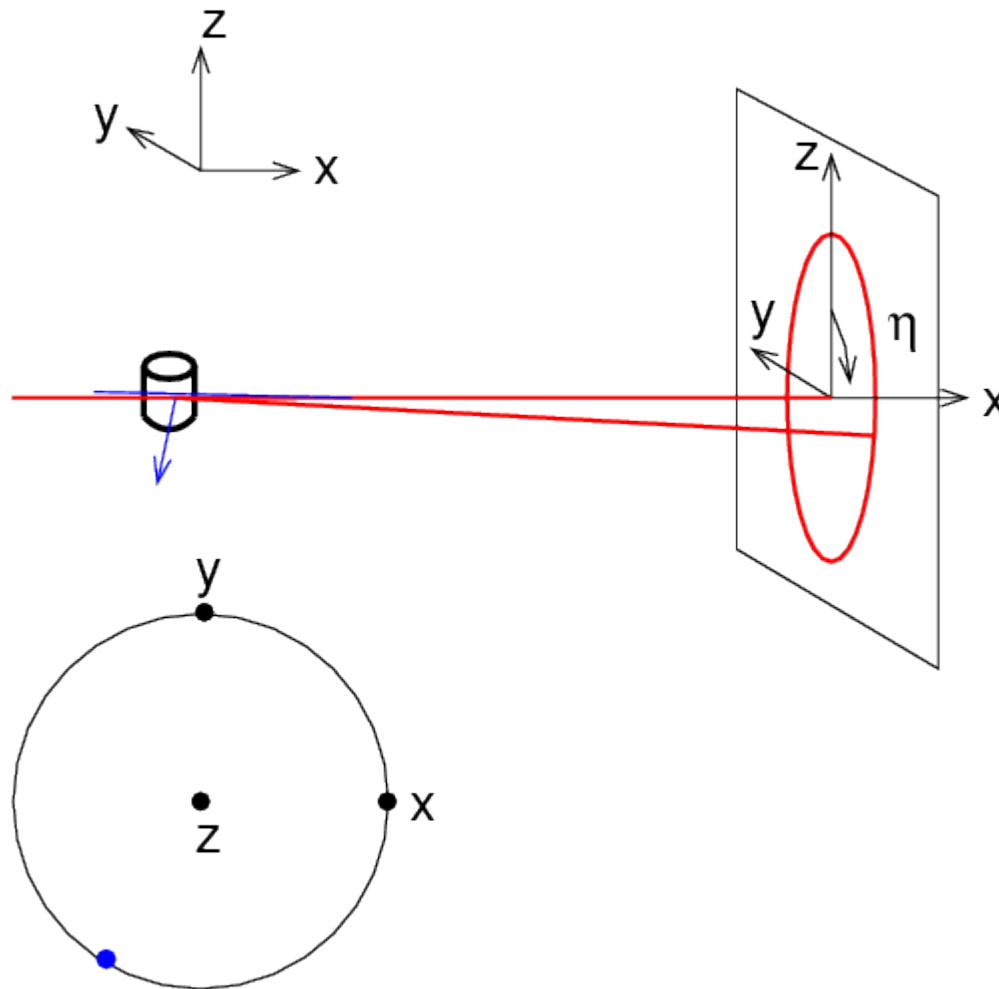


Grains contributing to diffraction at $\eta=270^\circ$

Normal to diffracting planes (i.e. diffraction vector)

Projection in the pole figure

Sample pole figure measurement

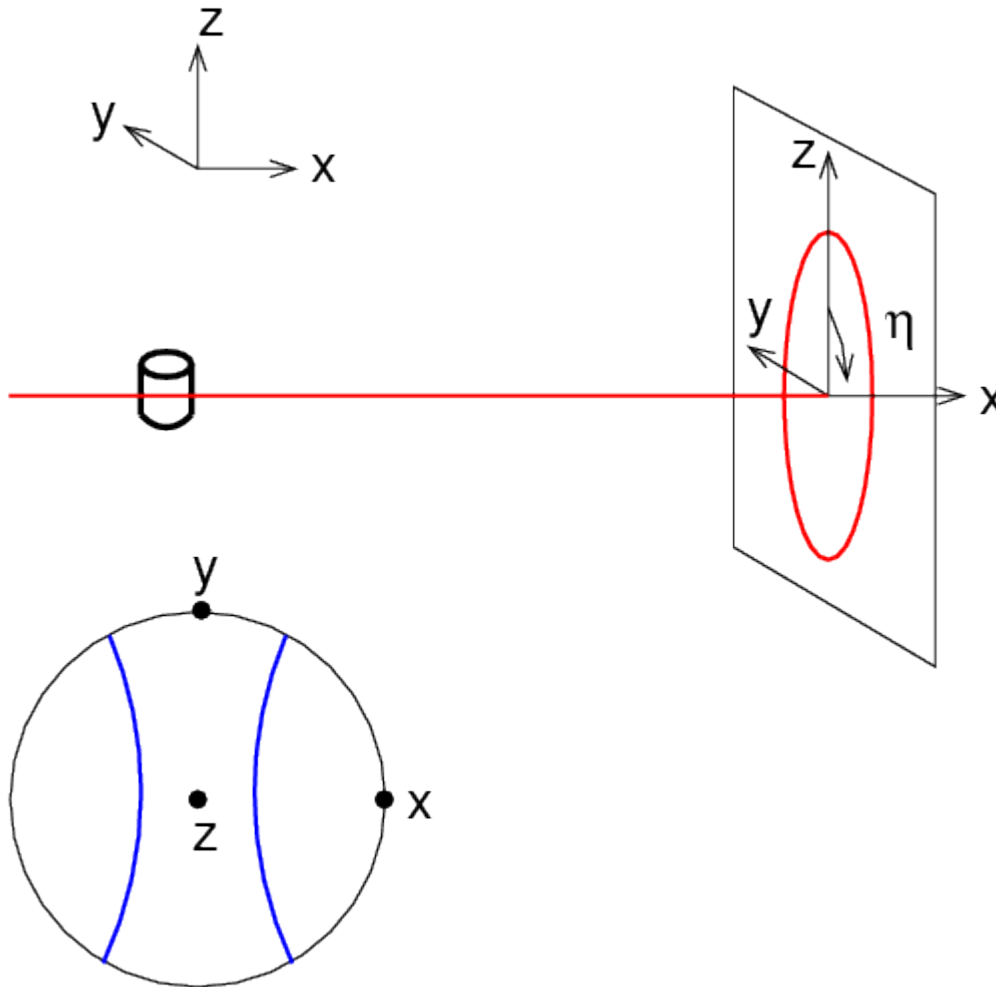


Grains contributing to diffraction at $\eta=90^\circ$

Normal to diffracting planes (i.e. diffraction vector)

Projection in the pole figure

Sample pole figure measurement



Complete coverage for the Debye-Scherrer ring

For each position in the pole figure, diffraction intensity is reported with a color code

The full figure is obtained from

- Known sample symmetries (if any)
- Rotating the sample and adding additional measurements

This was a simplified example.

1 pole figure for each diffraction peak (1 figure for 100, 1 figure for 110, 1 figure for 111...)

If no know sample symmetry, one needs at least 2 full pole figures to build a full texture for a cubic material. Additional figures are required for other symmetries.

There are many experimental implementations of the technique. They are very much sample and experiment dependent.

Corrections on diffraction intensities may be necessary.

Have a look at technical descriptions in books and experimental facilities.

Example will be shown in *Advanced Materials Characterization* course.

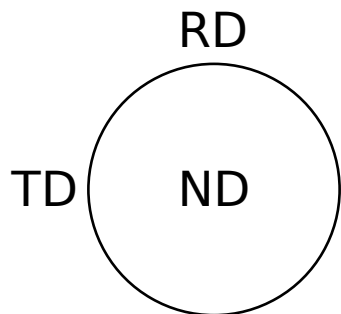
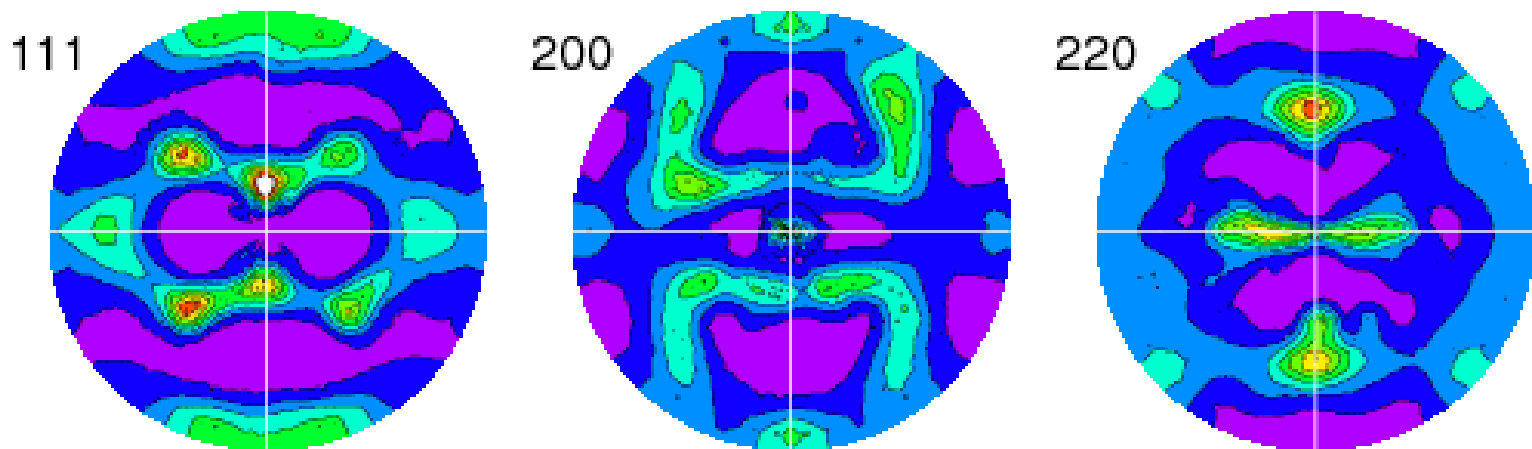
Pole figure example

Filename root: demo_ful



Stereographic

Multiples of Random



Copper rolling texture
Image A. Rollett
Practice: describe this texture

3- Orientations in a polycrystal c- EBSD measurement

EBSD : Electron BackScattered Diffraction

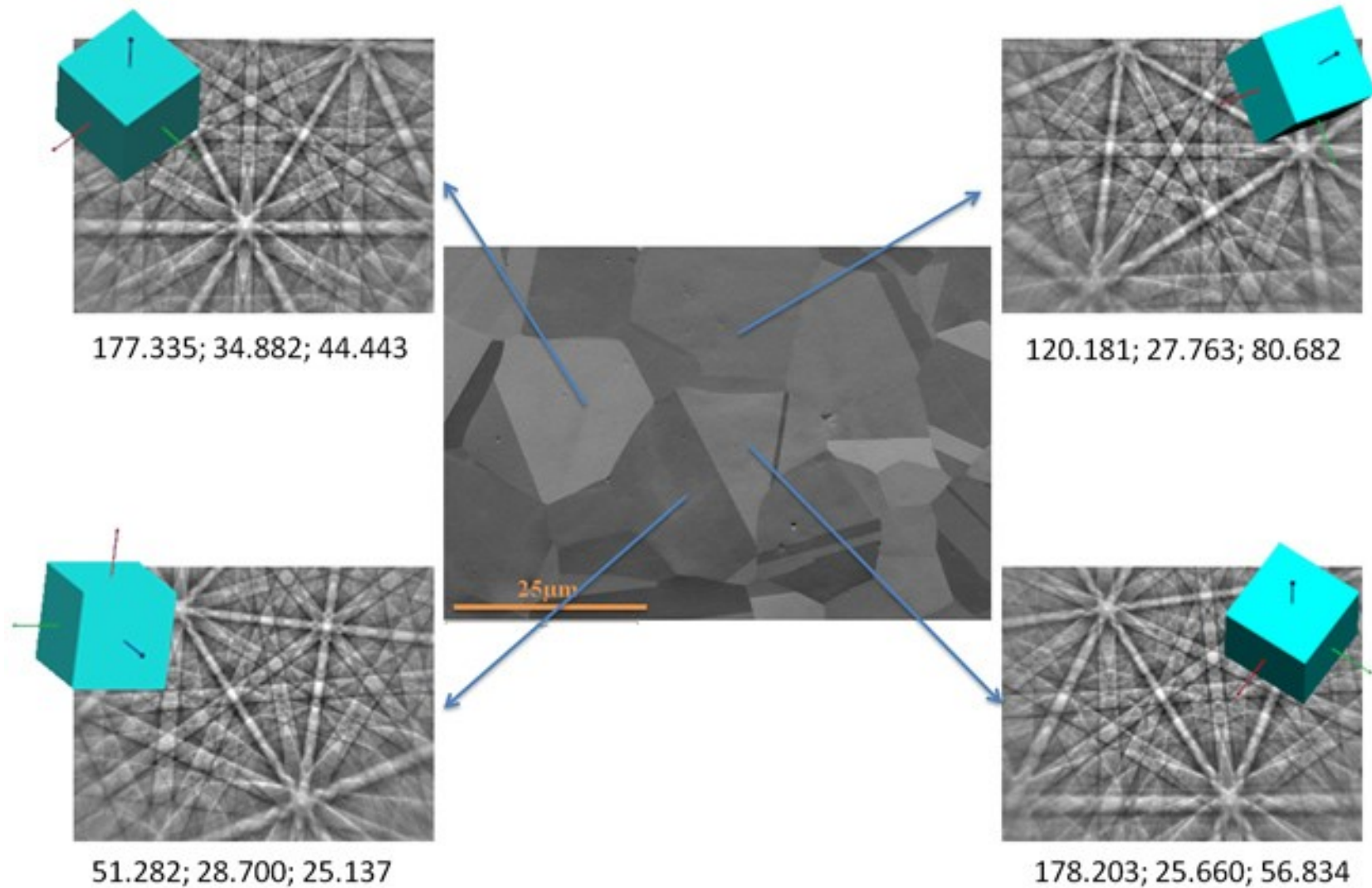
Scanning electron microscopy technique

Surface analysis

Principles

- Moves across a sample, step by step,
- At each pixel, reconstruct the crystallite orientation,
- Reconstruct grains based on contiguous regions with similar orientation,
- At each pixel, one can identify the orientation matrix of the diffracting crystallite.

EBSD: principes



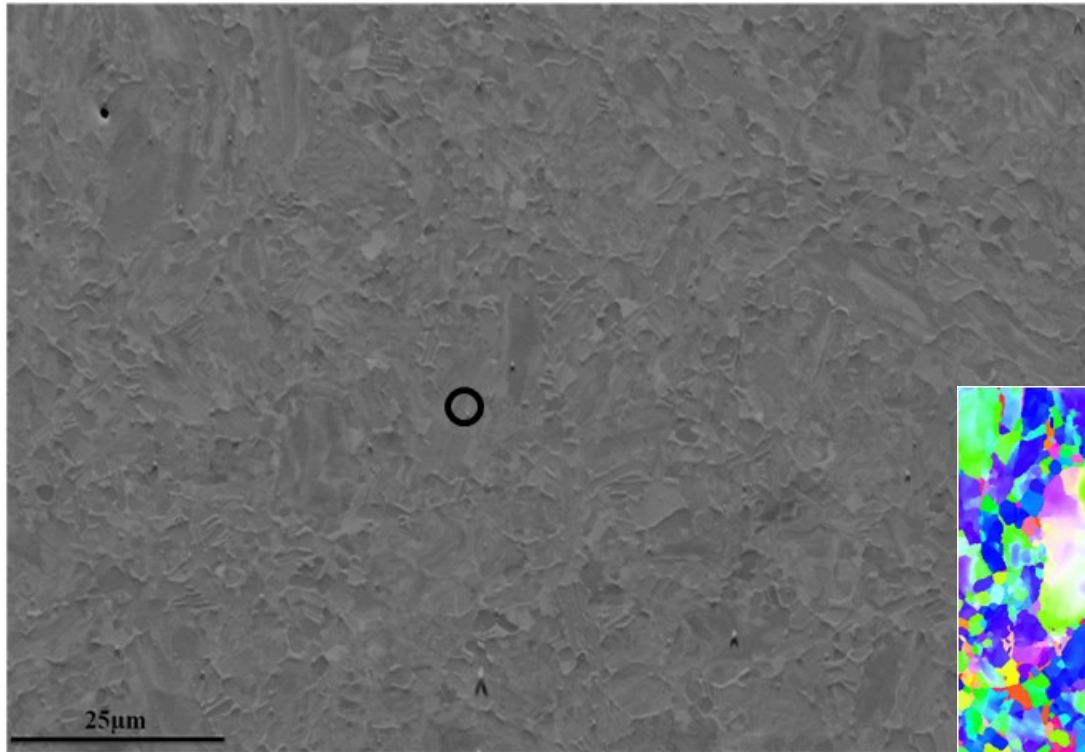
Images Oxford Instruments

SEM / EBSD system at ULille



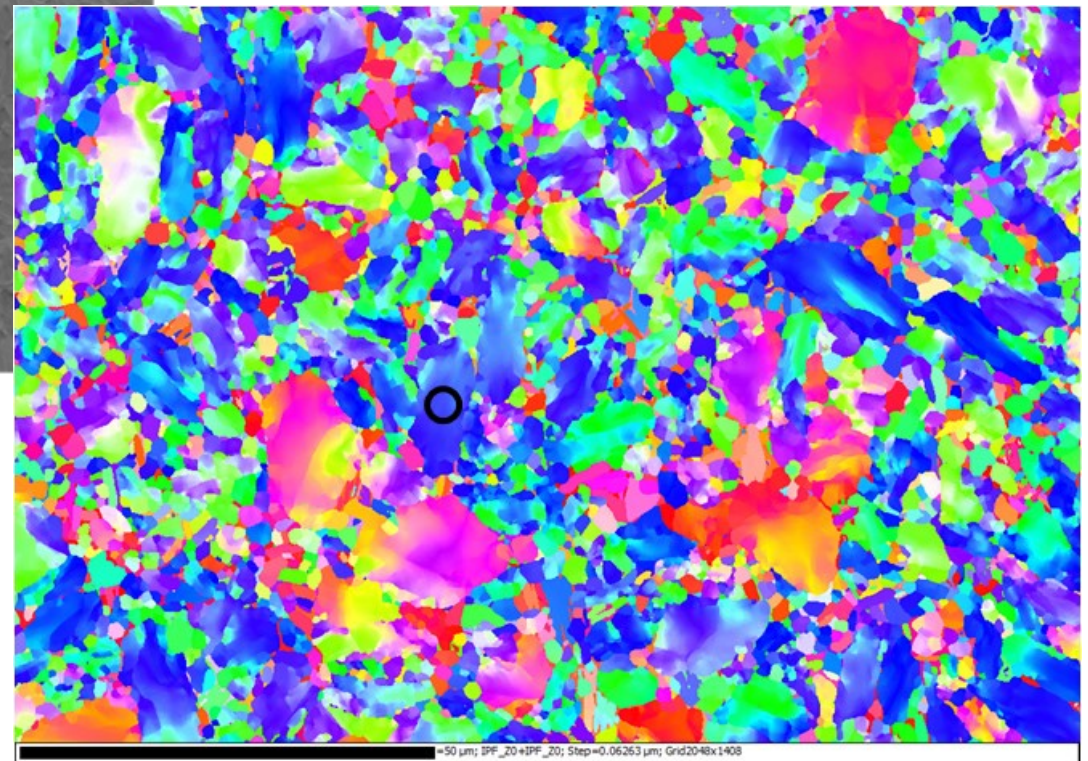
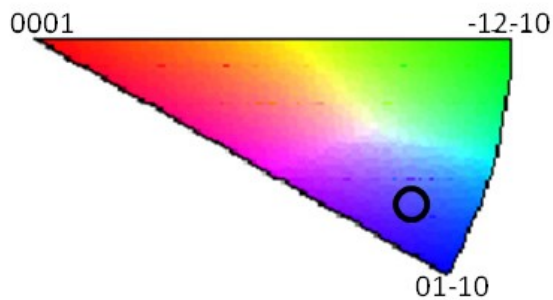
Images Plateforme de Microscopie Electronique de Lille

EBSD: illustration



SEM image of a titanium alloys

EBSD map of the same alloy



Images Oxford Instruments

3- Orientations in a polycrystal ***d*- Mathematical representation**

Orientation Distribution Function (ODF)

- Statistical approach to orientations in a polycrystal.
- OD – Orientation Distribution.
- Generation of an ODF, Orientation Distribution Function, f .
- f depends on the 3 Euler angles φ_1 , ϕ and φ_2 : $f(\varphi_1, \phi, \varphi_2)$.
- $f(g)$ where g is a rotation in 3D-space.
- Probability approach: f is a probability density.
- Units : MRD (Multiples of a Random Distribution).
- Random polycrystal: the ODF is 1, whatever the combination of Euler angles.
- Oriented polycrystal:
 - some orientations with $f(g) > 1$ (more probable than average),
 - other orientations with $f(g) < 1$ (less probable than average).

Why defining an ODF?

- Allows for a mathematical representation of texture.
- Polycrystal properties depend on the orientations of the crystallites.
- Pole figures are projections of the ODF. They can be used to visualize texture but information is missing.
- The ODF can be used to finely study a sample texture (identify a sub-space with no crystallite, for instance).
- The ODF can be used to calculate the properties of the polycrystal.

Probability density ???

If f is a probability density over $[-\infty; +\infty]$

Then

$$f(x) \geq 0, \forall x$$

(a probability can be null but not below zero)

It is normalized to 1:

$$\int_{-\infty}^{+\infty} f(x) dx = 1$$

And finally

$$\mathcal{P}(a \leq x \leq b) = \int_a^b f(x) dx$$

Tricks with orientation space: 2D

Take an orientation space in 2D

Stereographic representation, in 2D

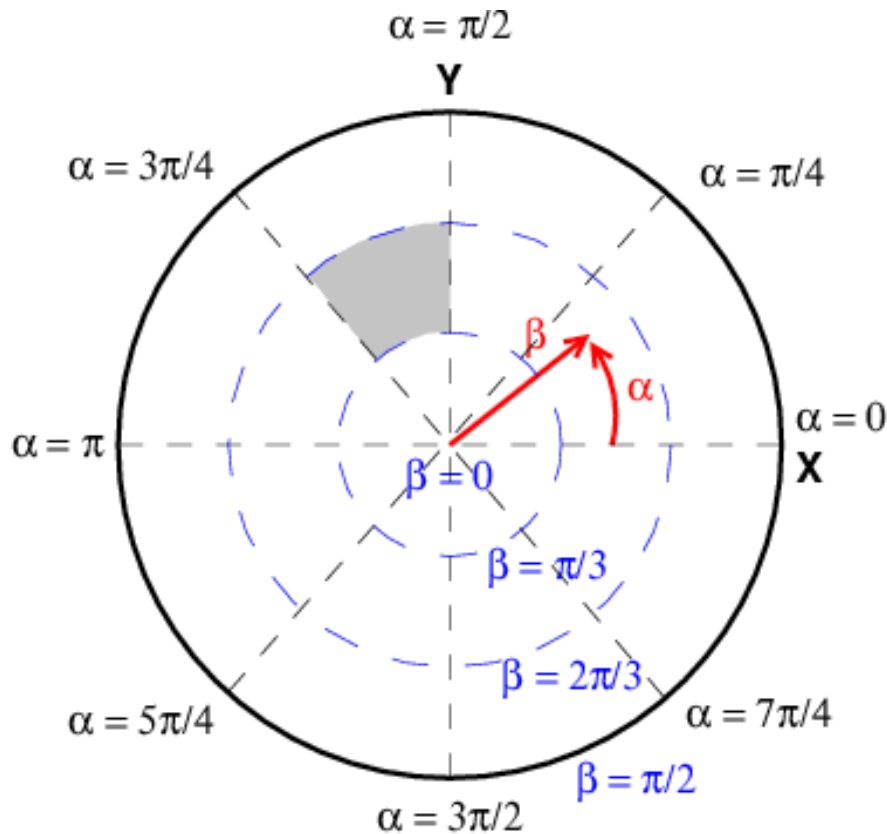
Rotation defined by 2 successive rotations: α (around z), β (around y)

$$0 \leq \beta \leq \pi/2$$

$$0 \leq \alpha \leq 2\pi$$

Size of integration domain:
 $\sin \beta \, d\beta \, d\alpha$

$$\int_{\alpha=0}^{\alpha=2\pi} \int_{\beta=0}^{\beta=\pi/2} \sin \beta \, d\alpha \, d\beta = 2\pi$$



Euler angles domains

$$0 \leq \varphi_1 \leq 2\pi ; 0 \leq \phi \leq \pi ; 0 \leq \varphi_2 \leq 2\pi$$

Volume element

$$dg = 1/8\pi^2 \sin \phi d\varphi_1 d\phi d\varphi_2$$

The ODF is normalized such that its integral over the whole orientation space is 1.

The $1/8\pi^2$ prefactor is the size of the full orientation domain, in radians.

In degrees, one gets $360^\circ \cdot 2 \cdot 360^\circ = 259200^\circ$.

$$\frac{1}{8\pi^2} \int \int \int f(\varphi_1, \phi, \varphi_2) \sin \phi d\varphi_1 d\phi d\varphi_2 = 1$$

Example of a random ODF

Notice the small number of points near $\phi = 0^\circ$

On a powder diffraction image, the intensity would be constant (due to the $\sin \phi$ factor).

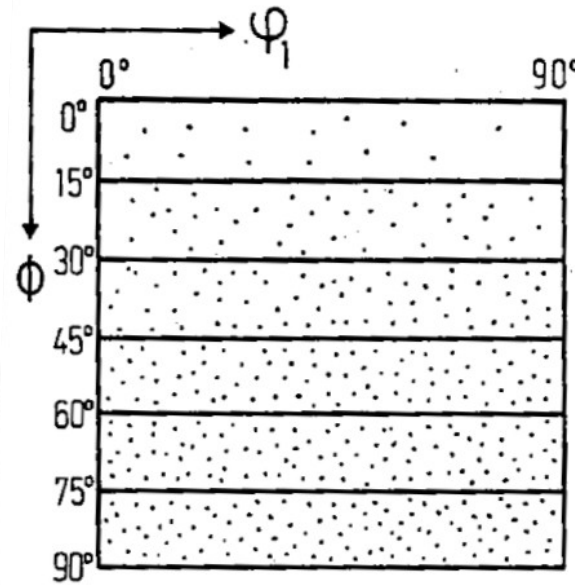


Figure 2.25 On the definition of the invariant measures by a random distribution in the EULER space $\varphi_1\Phi\varphi_2$

Projection of a random ODF in the (ϕ, φ_1) plane.

Bunge 1982

Harmonic or direct representation

- The ODF can be described using **harmonic** or **direct** representations.
- Harmonic method: the ODF is a continuous function, represented with spherical harmonics.
- Direct method: the orientation space is cut into boxes ($5^\circ \times 5^\circ \times 5^\circ$, for instance) with a coefficient for each box.
- Harmonic method
 - Pros: efficient for weak and simple textures (few coefficients), automatic smoothing of experimental data.
 - Cons: truncation errors, not fitted for sharp textures.
- Direct method
 - Pros: works for any type of texture, even with a low number of grains.
 - Cons: many coefficients (186624 for boxes of $5^\circ \times 5^\circ \times 5^\circ$)

Pole figure vs. ODF

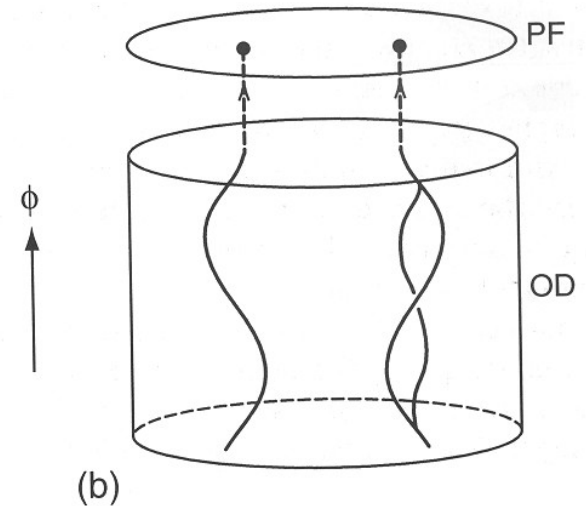
A pole figure is a projection of the information inside an ODF.

Several points of an ODF contribute to a single point of a pole figure.

Intensity at a point in a pole figure = integral of all corresponding ODF values.

The integration path in Euler space is complex.

Other representations (Rodrigues space, for instance) exist to make this easier.



(b) Illustration from book Kocks, Tomé, Wenk, 1998

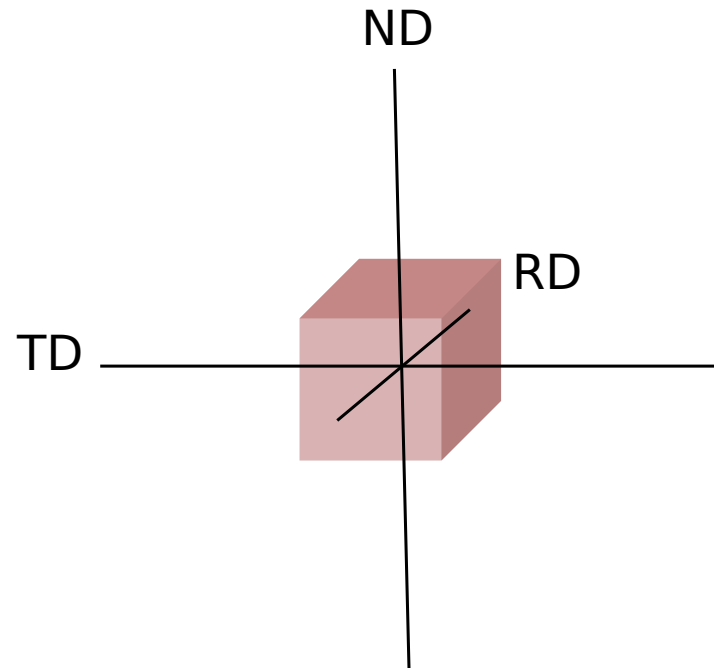
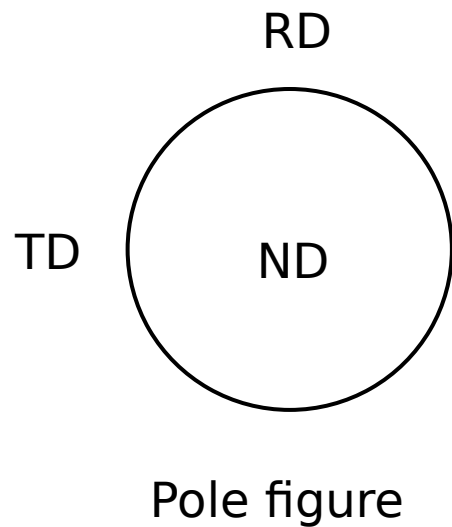
Intensity for pole hkl in direction y

$$P_{hkl}(y) = \frac{1}{2\pi} \int_{hkl \parallel y} f(g) dg ; \quad y = \{\alpha, \beta\}$$

Integral over the ODF for all orientations for which $y \parallel g_{hkl}$

100 pole for a cubic crystal. Which are the Euler angle domains contribution to the center of a pole figure?

100 pole for a cubic crystal. Which are the Euler angle domains contribution to the center of a pole figure?



Normal to (001) // ND : $\phi = 0^\circ$, any φ_1 and φ_2

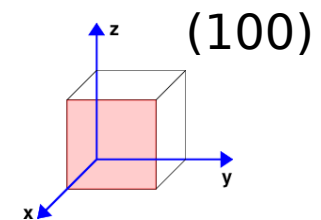
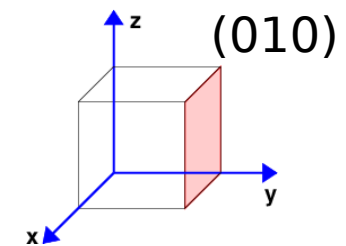
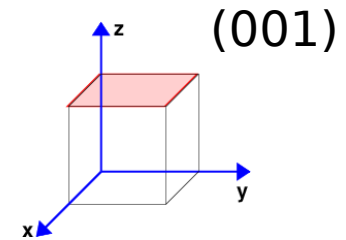
Normal to {001} // ND :

(001) : $\phi = 0^\circ$

(010) : $\varphi_1 = 0^\circ - \phi = 90^\circ - 0^\circ \leq \varphi_2 \leq 360^\circ$

(100) : $\varphi_1 = 90^\circ - \phi = -90^\circ - 0^\circ \leq \varphi_2 \leq 360^\circ$

+ more angle combinations, (00-1), (0-10), (-100)



Planes contributing to 100 pole

Units (ODF and pole figures)

ODF coefficients or pole figures expressed in *Multiples of a Random Distribution* (mrd)

Random polycrystal

- $f(g) = 1$ mrd, for all orientations g

Oriented polycrystal

- $f(g) > 1$ for some orientations, $f(g) < 1$ for other orientations,
- The integral of f over orientation space is 1,
- $f(g) = 2 \rightarrow$ this orientation is twice more likely to occur than in a random polycrystal,
- $f(g) = 0.5 \rightarrow$ this orientation is twice less likely to occur than in a random polycrystal,

Single crystal

- $f(g) = \infty$ at the crystal orientation
- $f(g) = 0$ elsewhere

Same for pole figures

“strength” of an orientation → *texture index*, F_2 :

$$\begin{aligned} F_2 &= \frac{1}{8\pi^2} \int \int \int [f(\varphi_1, \phi, \varphi_2)]^2 \sin \phi \, d\varphi_1 \, d\phi \, d\varphi_2 \\ &= \frac{1}{8\pi^2} \int_G [f(g)]^2 \, dg \end{aligned}$$

- For a random polycrystal: $F_2 = 1$
- For a polycrystal with preferred orientations (texture): $F_2 > 1$
- For a single crystal: $F_2 = +\infty$.

3- Orientations in a polycrystal *d*- Graphical representation

Not always straightforward!

- The ODF is a continuous function in a space with 3 Euler angles
- The orientation space is not Cartesian. Orientations between $\phi = 0^\circ$ and 5° hold less weight than orientations between $\phi = 85^\circ$ and 90° .

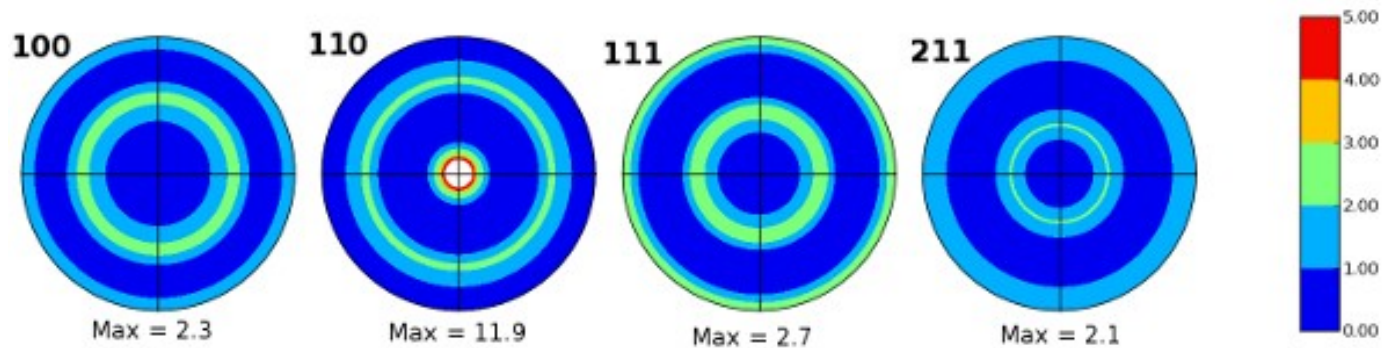
Typical representations

- Sections: slices through the ODF,
- Pole figures,
- Inverse pole figures.

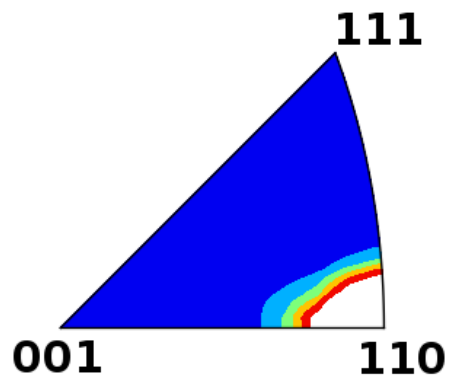
Pole figures / inverse pole figures

Pole figure: polycrystal grain orientations shown in the sample reference frame

Simulation of compression for a fcc metal.

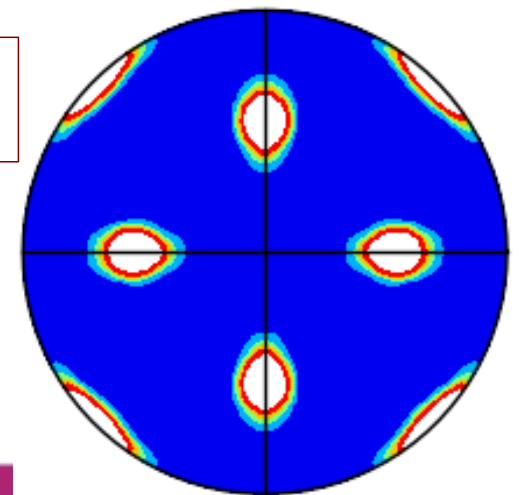


Inverse pole figure: projection of sample direction in the crystal reference frame



Minimum inverse pole figure

Full inverse pole figure



Stereographic projection

Heavily used in crystallography and electron microscopy.

Projection of crystal directions or normal to planes in the crystal reference frame.

Practice: build a stereographic projection for a cubic crystal and show the following planes :
 $\{100\}$, $\{110\}$, $\{111\}$...

Stereographic projection

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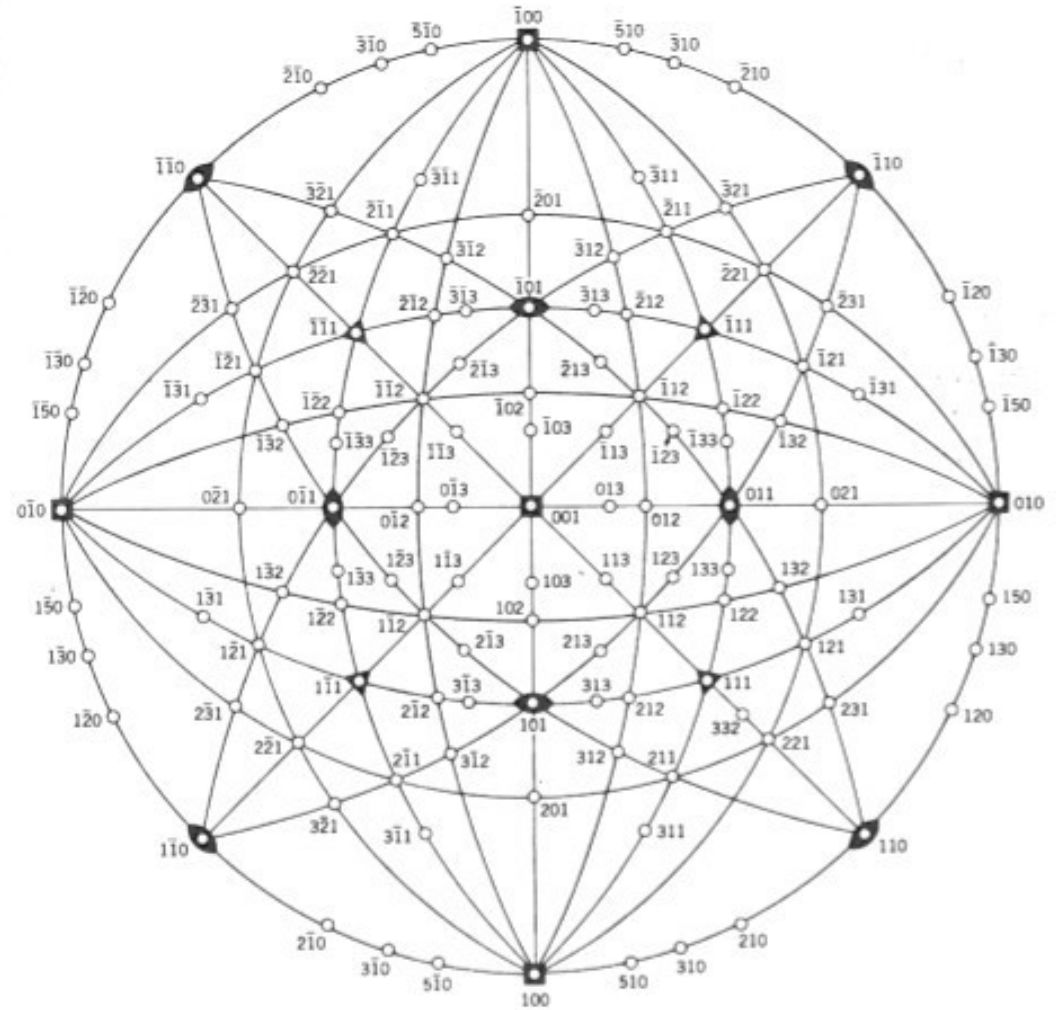


Fig. 2-9 Standard (001) stereographic projection of poles and zone circles for cubic crystals. (After E. A. Wood, "Crystal Orientation Manual," Columbia, New York, 1963.)

Inverse pole figure

Shows the density of orientations for a sample direction, projected in the crystal reference frame.

Very useful for samples with a simple geometry or symmetries: compression, tension, etc

For instance: inverse pole figure the compression direction

- Plot the probability that a given pole is parallel to the compression direction

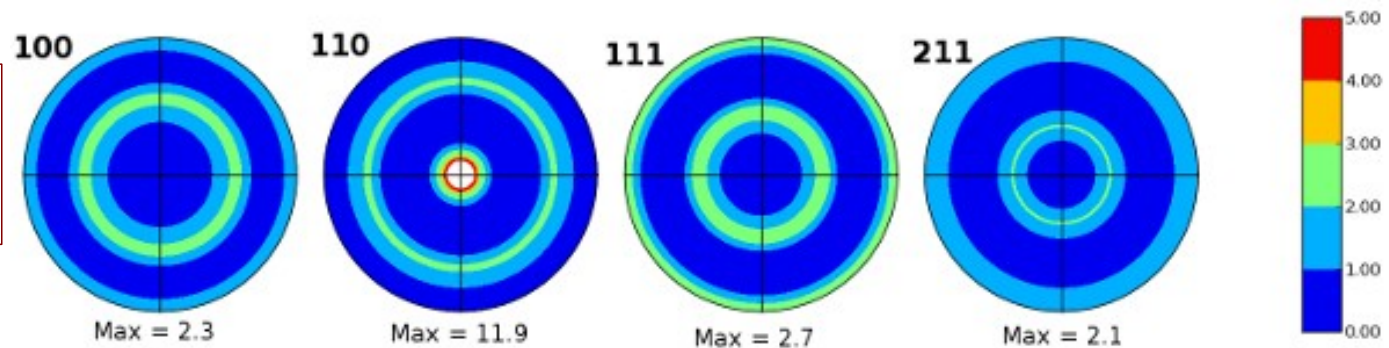
Less useful for complex geometries (rolling, etc).

Information projected in the crystal reference frame, using the stereographic projection (there are others, they look very similar).

Practice 1

Pole figure: polycrystal grain orientations shown in the sample reference frame. Compression direction is at the center.

Simulation of
compression
for a fcc metal

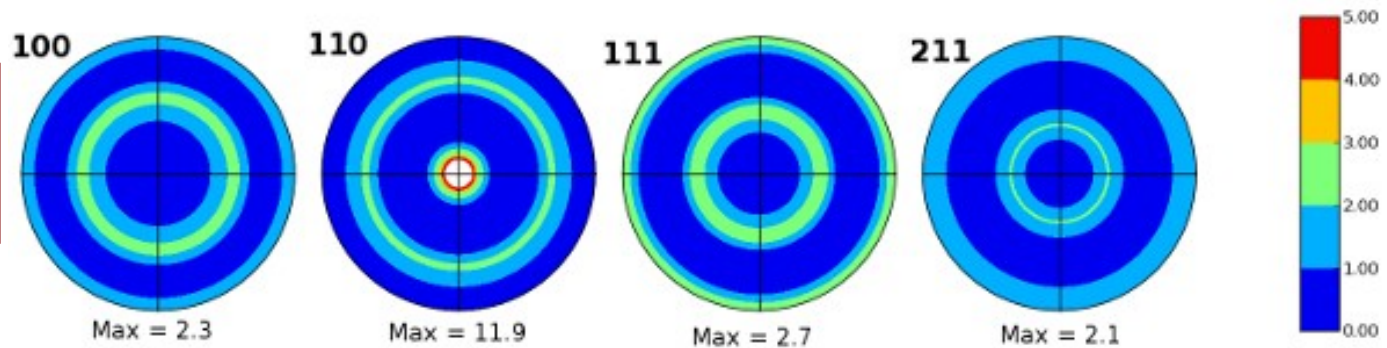


Practice: plot the inverse pole figure of the compression direction

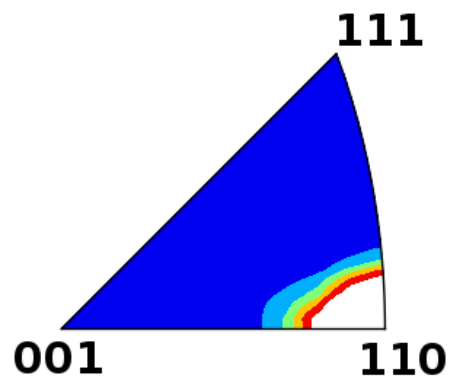
Practice 1

Pole figure: polycrystal grain orientations shown in the sample reference frame. Compression direction is at the center.

Simulation of compression for a fcc metal

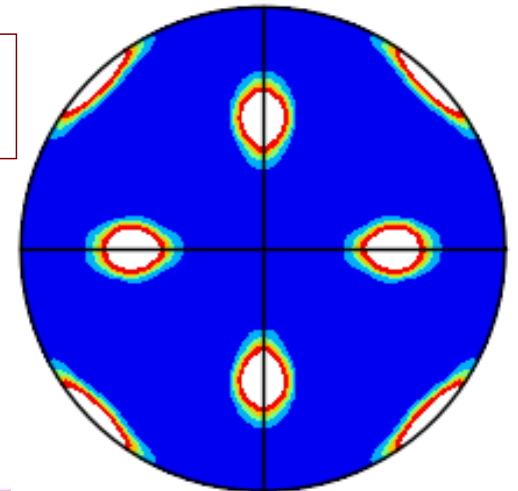


Practice: plot the inverse pole figure of the compression direction



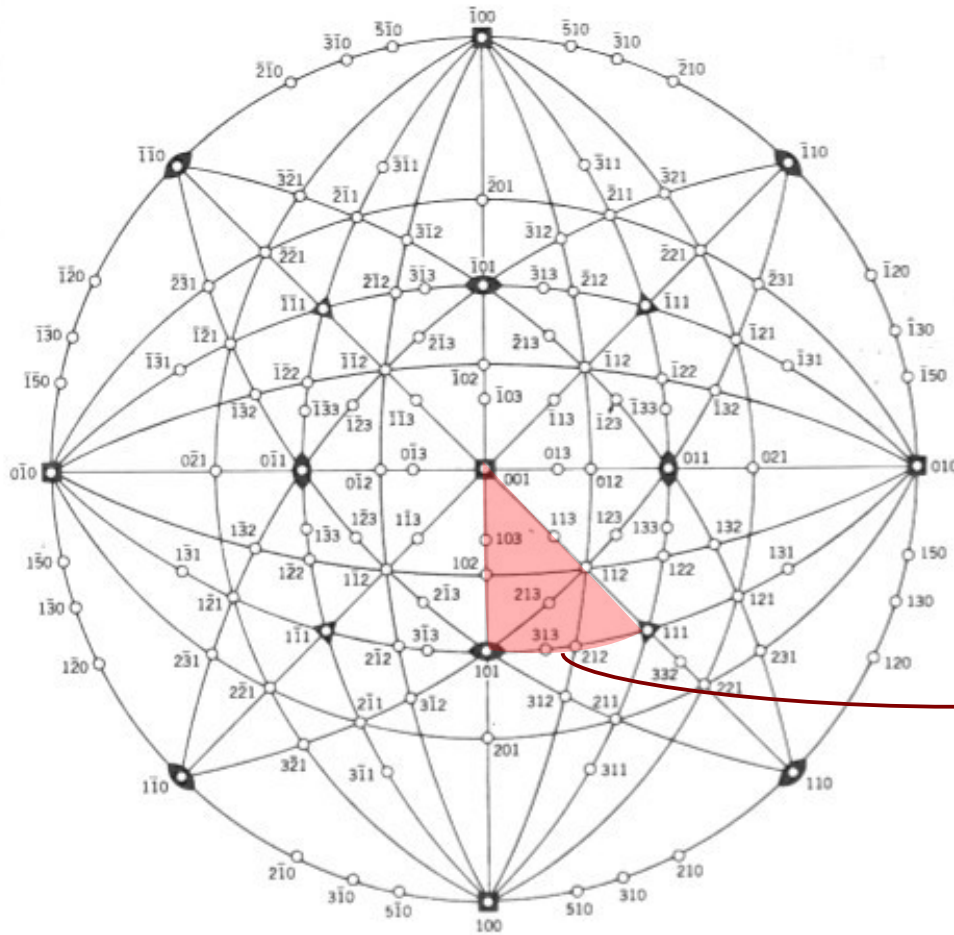
Minimal inverse pole figure

Full inverse pole figure



Projection for a cubic crystal

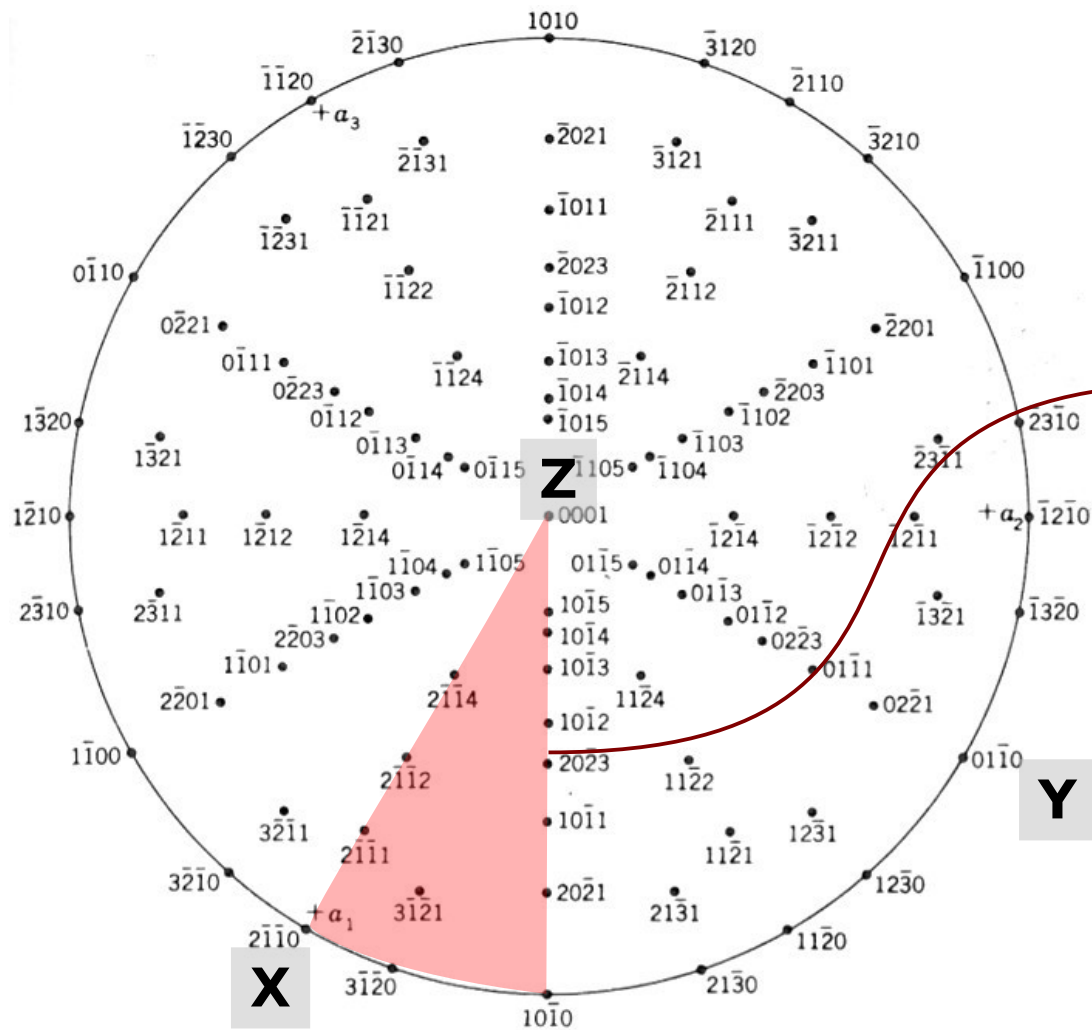
Stereographic projection of a cubic crystal



Cubic sector, sufficient for representing all information in an inverse pole figure.

Fig. 2-9 Standard (001) stereographic projection of poles and zone circles for cubic crystals. (After E. A. Wood, "Crystal Orientation Manual," Columbia, New York, 1963.)

Projection for hexagonal symmetry



Stereographic projection for Zinc
(hexagonal-closed-packed)

“hexagonal” sector: sufficient to represent the full IPF of an hexagonal crystal

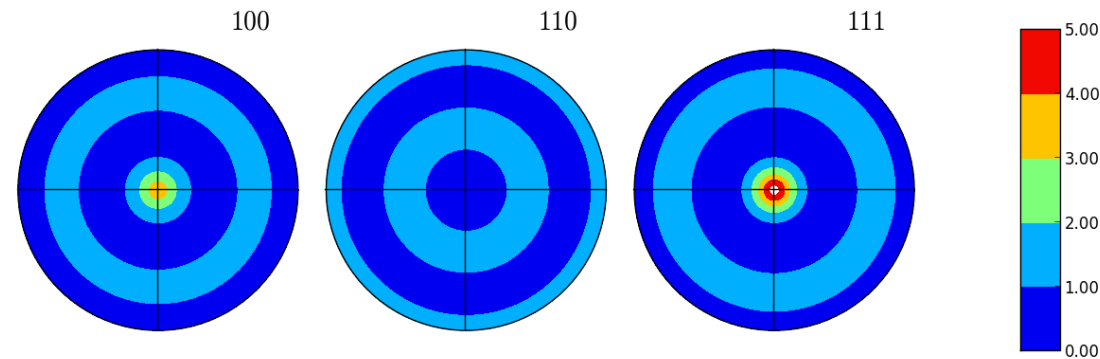
Z // [0001]
X // [2-1-10]
Y // [01-10]

Fig. 2-10 Standard (0001) projection for zinc (hexagonal, $c/a = 1.86$).

Barret & Massalski, *Structure of Metals*,
Permagon (1980)

Pole figure: polycrystal grain orientations shown in the sample reference frame. Extension direction is at the center.

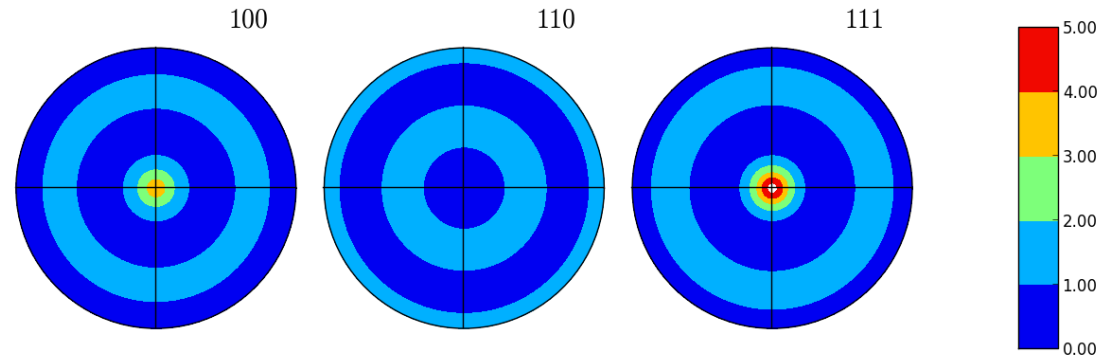
Simulation of extension for an fcc metal



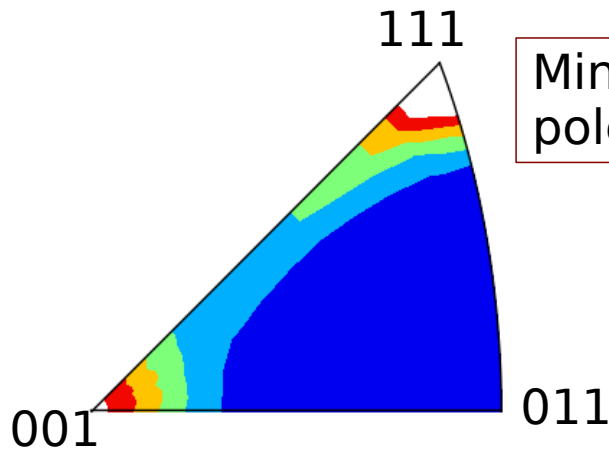
Practice: plot the inverse pole figure of the extension direction

Pole figure: polycrystal grain orientations shown in the sample reference frame. Extension direction is at the center.

Simulation of extension for an fcc metal



Practice: plot the inverse pole figure of the extension direction



Minimal inverse pole figure

Full inverse pole figure

