Crystallographic Texture of Materials

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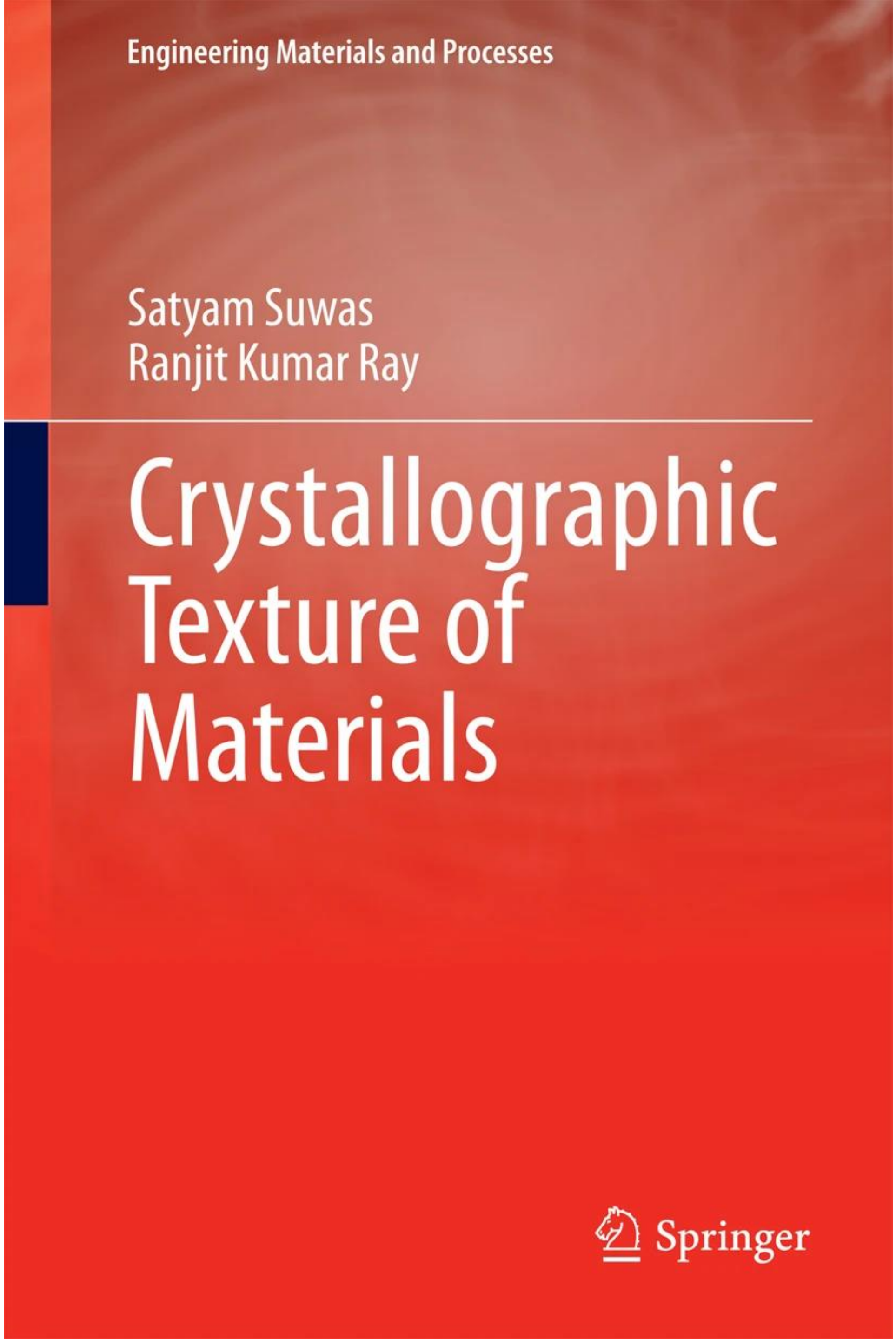


Engineering Materials and Processes

Satyam Suwas
Ranjit Kumar Ray

Crystallographic Texture of Materials

Springer

- A basic text book for beginners

- Provides a comprehensive and invaluable overview of the basics of crystallographic textures and their industrial applications.

Other reference books:

1\. **Introduction to Texture Analysis: Macrotexture, Microtexture, and Orientation Mapping (3rd Edition)**

by Olaf Engler, Stefan Zaefferer, Valerie Randle

2\. **Texture and Anisotropy: Preferred Orientations in Polycrystals and their Effect on Materials Properties**

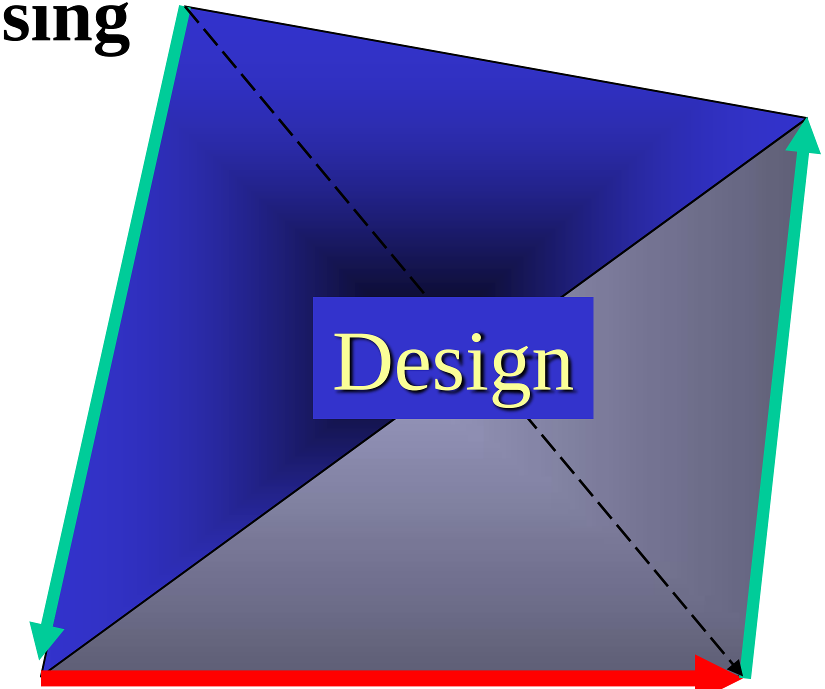
by U. F. Kocks, C. N. Tomé, H. -R. Wenk

Course objectives

- **To learn about the arrangement of crystals in a polycrystalline material**
- **To learn how to represent polycrystal diffraction data in stereographic projection**
- **To learn the principles of texture measurements by X-ray, neutron and electron diffraction**
- **To learn three dimensional texture representation**

Microstructure-Properties Relationship

Material tetrahedron: a representation of different stages of materials engineering leading to product design



Processing

Performance

Design

Microstructure (Morphology+Orientation)

Properties

Source: Ashby and Chowdhary

Understanding materials' tetrahedron

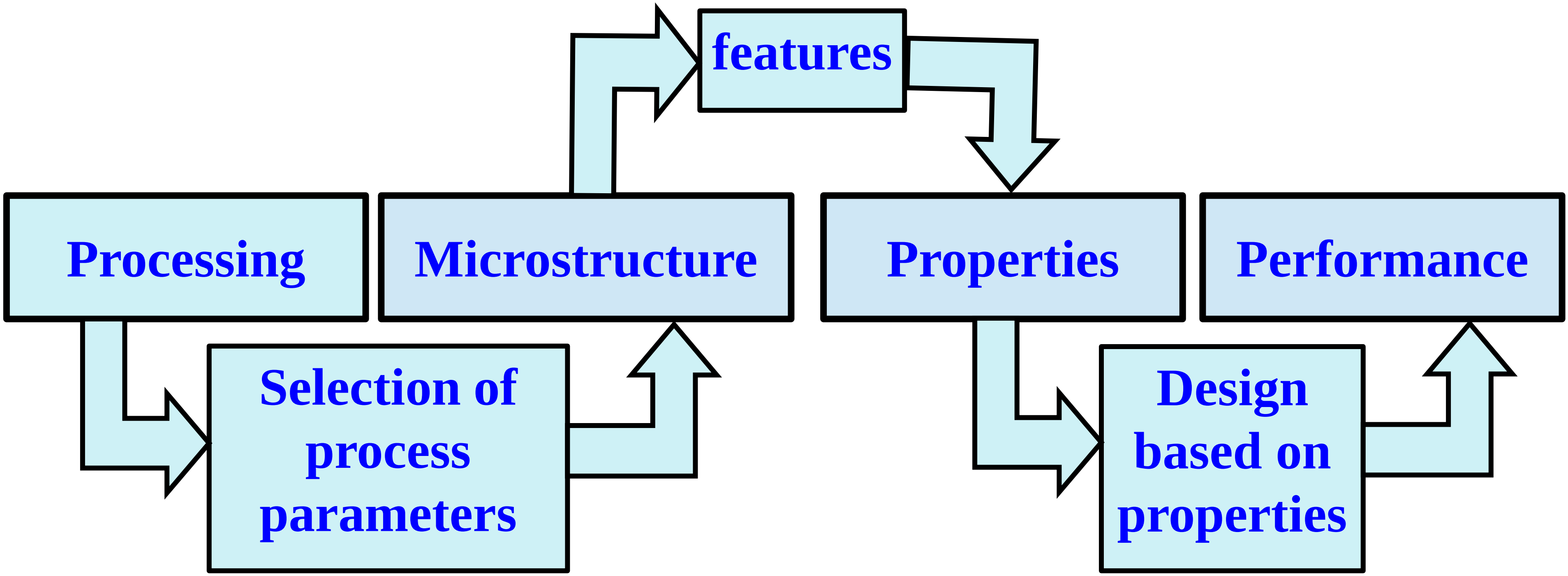
- Processing of Materials determines the microstructure

↓

- Microstructure controls the properties of the material

↓

- Properties decide the performance of a material for an application



features

Processing

Microstructure

Properties

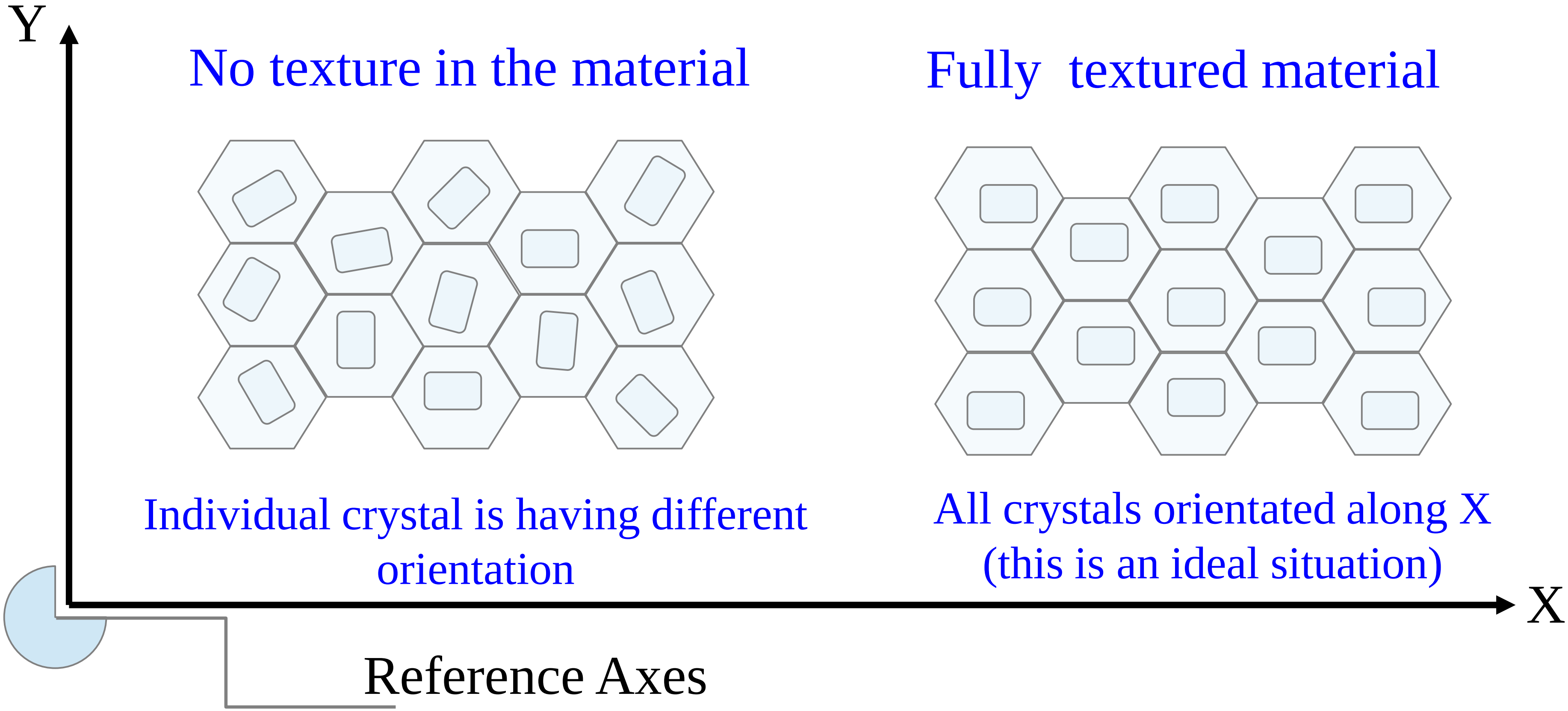
Performance

Selection of process parameters

Design based on properties

Microstructure: The most important parameter

Some possible arrangements of crystallites in polycrystalline materials : a two dimensional view



No texture in the material

Individual crystal is having different orientation

Fully textured material

All crystals orientated along X (this is an ideal situation)

What is a crystallite?

- **Limited volume of material in which periodicity of crystal lattice is present.**
- **Each of these crystallites has *a specific orientation* of the crystal lattic**

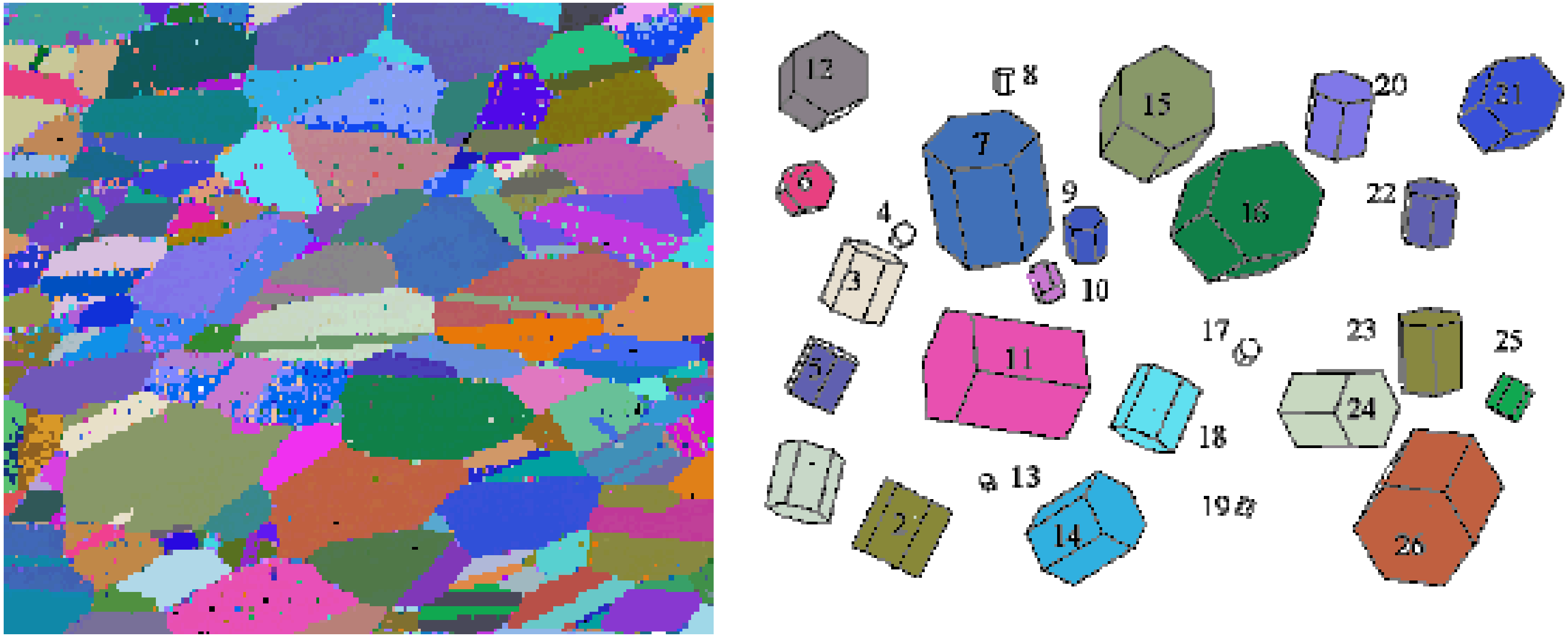
What are the other information that can be derived from the microstructure given in the previous slide?

Grain Size: ***What is the size of each hexagon?***

Grain Boundary fraction: ***What is total boundary length associated with the hexagons?***

Grain size distribution: ***How the hexagons are distributed in the given area, whether then size of the hexagons are following any size distribution etc?***

Grain orientation: ***How are the hexagons orientated with the assumed reference frame?***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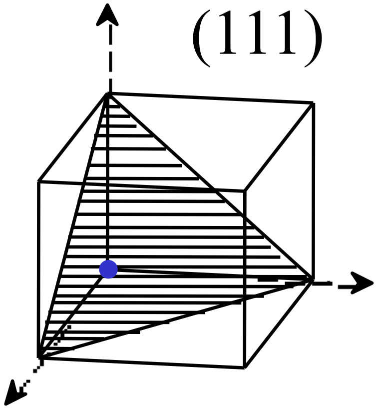
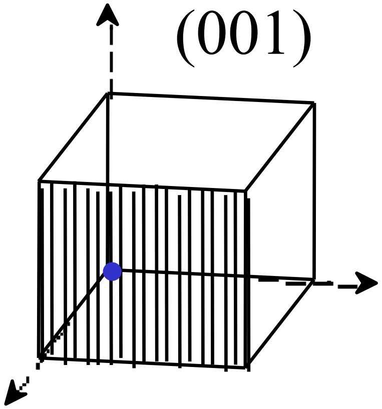
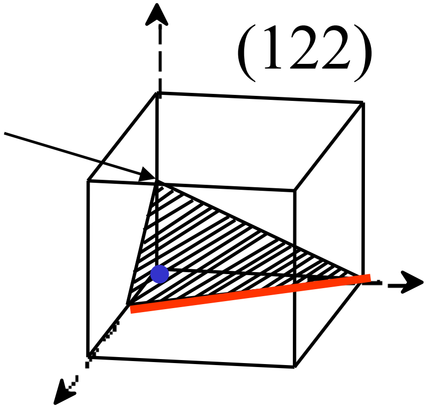
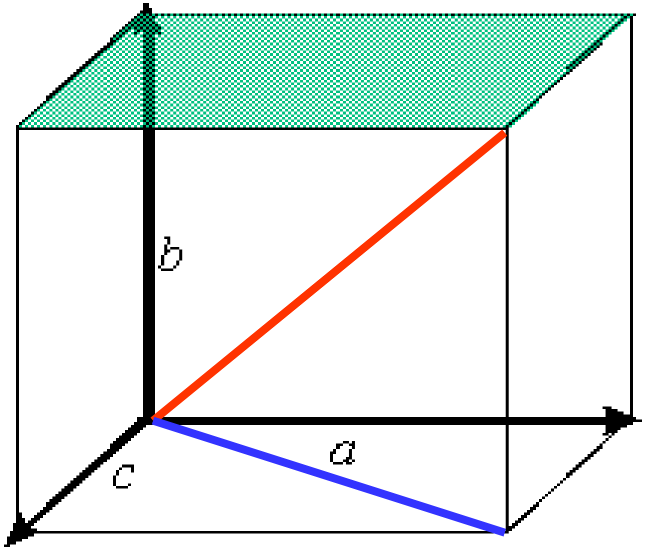
Examples of other crystallographic planes


(111)


(001)


(122)

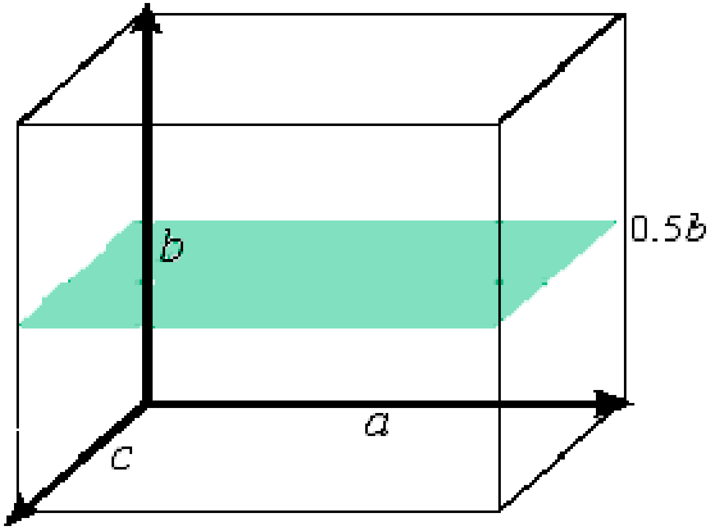
1.



- Intercepts are ∞,1,∞
- Miller indices of the plane are:

$$\left(\frac{1}{\infty}\,\frac{1}{1}\,\frac{1}{\infty}\right) = (010)$$

2.

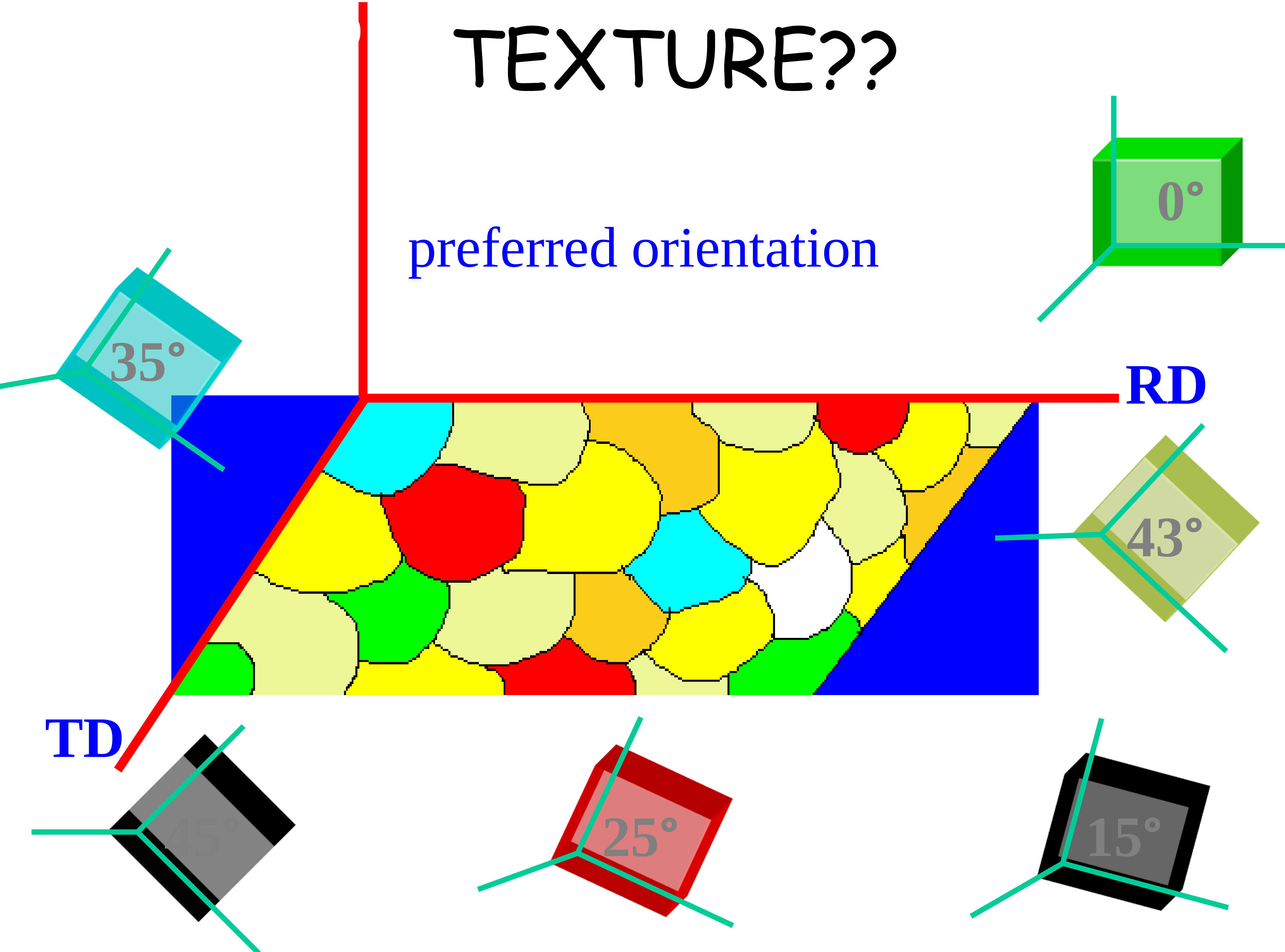


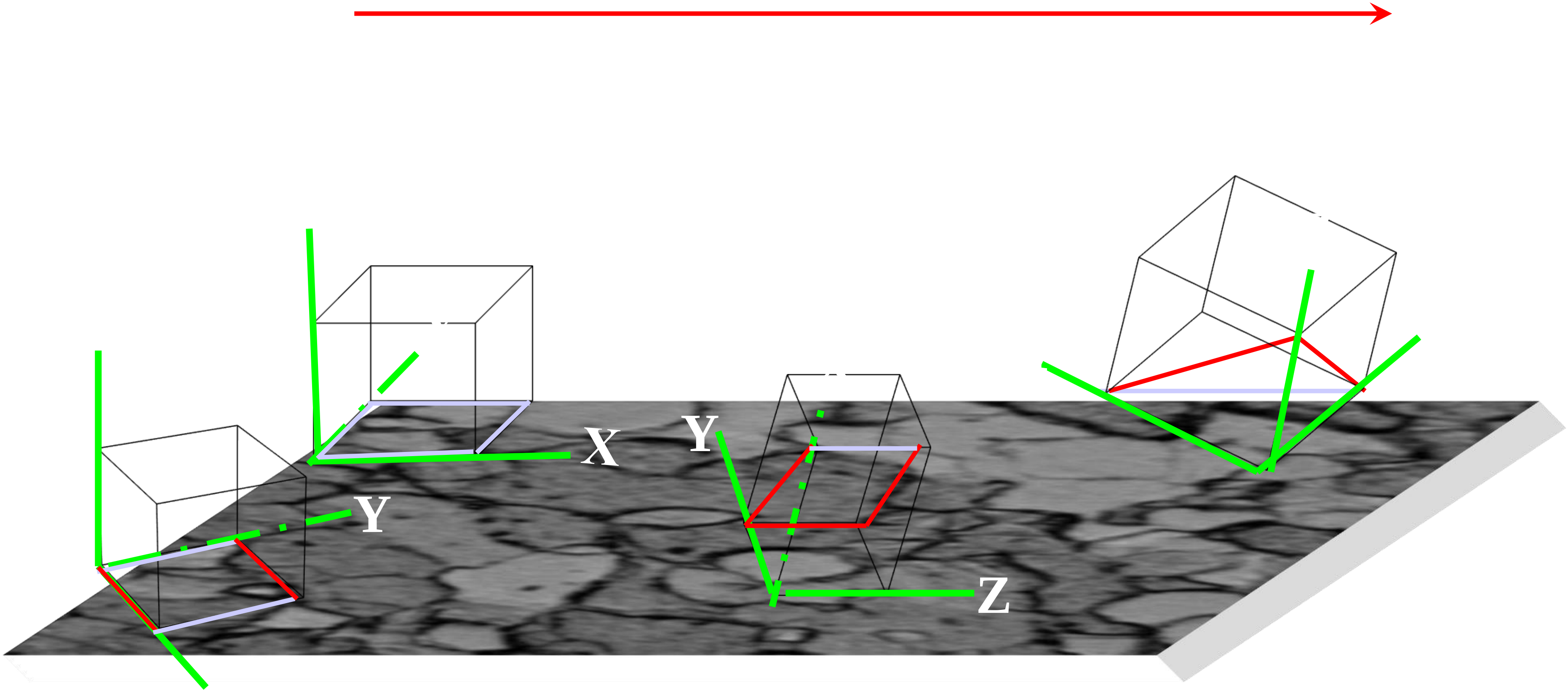
- Intercepts are∞,1/2,∞
- Miller indices of the plane are:

$$\left(\frac{1}{\infty}\,\frac{1}{1/2}\,\frac{1}{\infty}\right) = (020)$$

TEXTURE??

preferred orientation



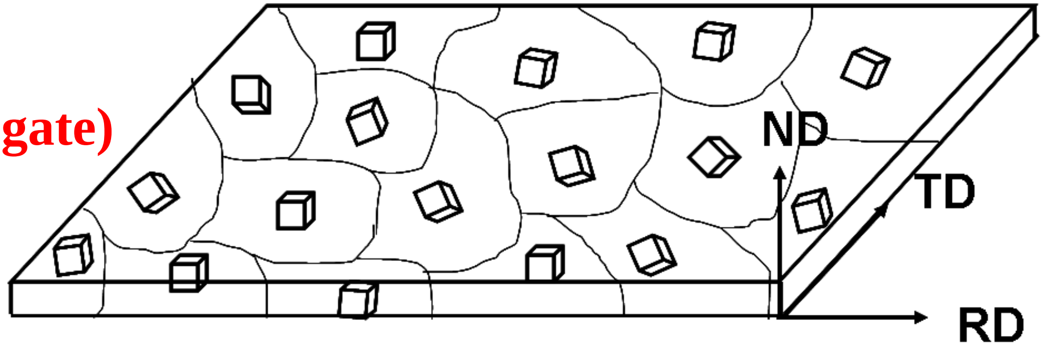

X
Y
Y
Z

- ***Texture deals with grain orientations (Crystallite orientations) in a polycrystalline materials.***

Polycrystalline material is constituted from a large number of small crystallites (limited volume of material in which periodicity of crystal lattice is present). Each of these crystallites has *a specific orientation* of the crystal lattice.

A texture-less sheet

(Configuration like a powder aggregate)



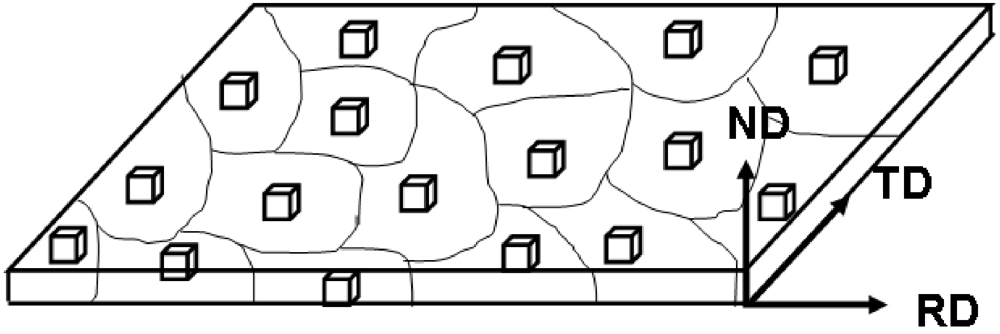
A fully textured sheet

The cube texture

(001)|| ND (Sheet Normal Direction)

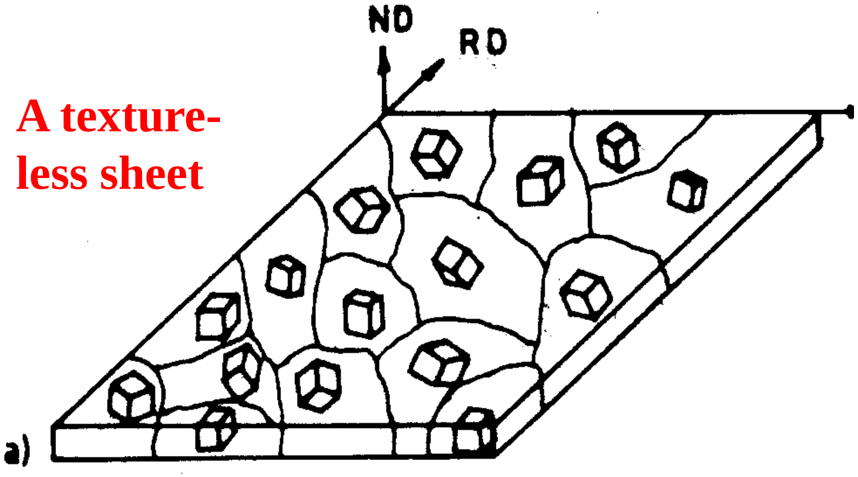
[100]|| RD (Sheet Rolling Direction)

(Configuration like a single crystal)

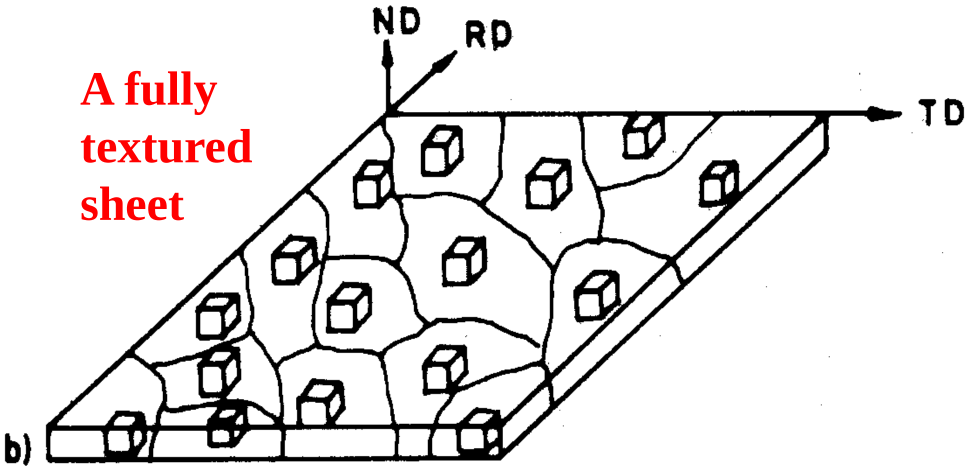


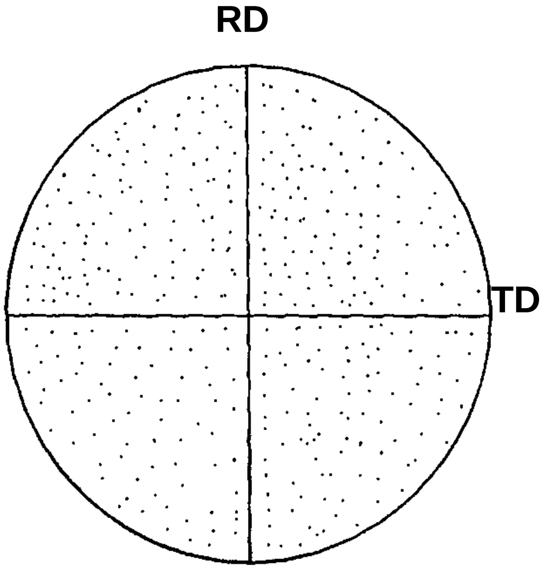
- Some possible arrangements of crystallites in polycrystalline materials !!

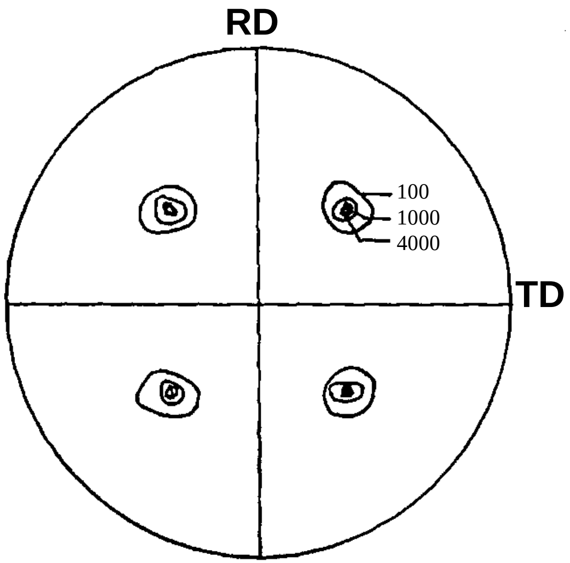
A texture-less sheet

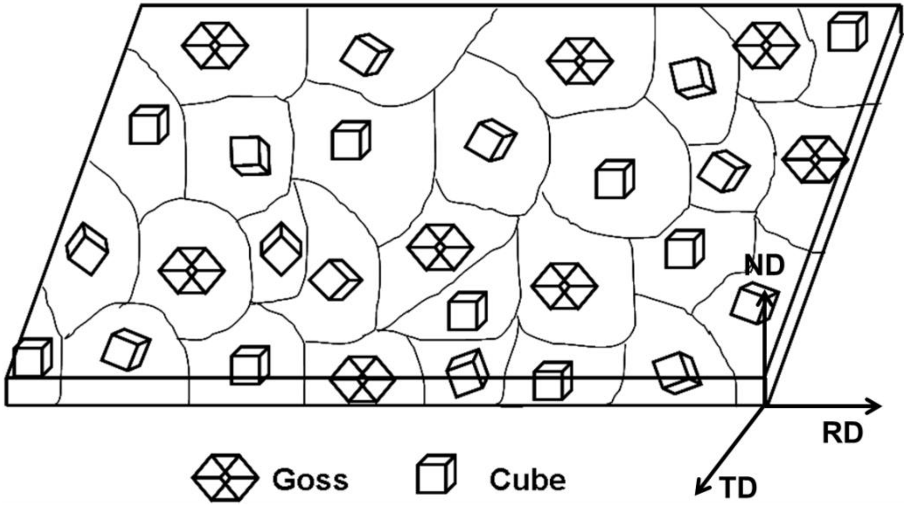


A fully textured sheet









<u>The real situation</u>:

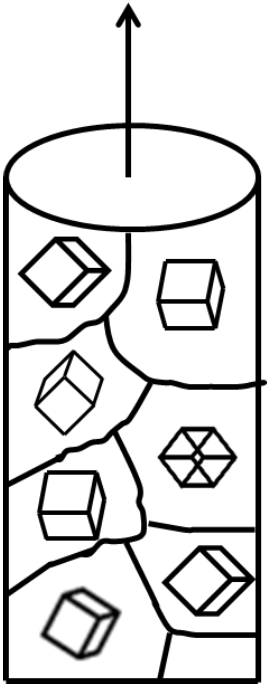
Many grains have common orientation, but they are spatially apart.

How to *Describe Textures?*

For uniaxial deformation or other processes, texture is expressed in terms of miller indices of directions [uvw] aligned along the specimen axis, also called ***Fibre texture***.

Look at the crystal configurations in the wires:

All crystals are randomly oriented

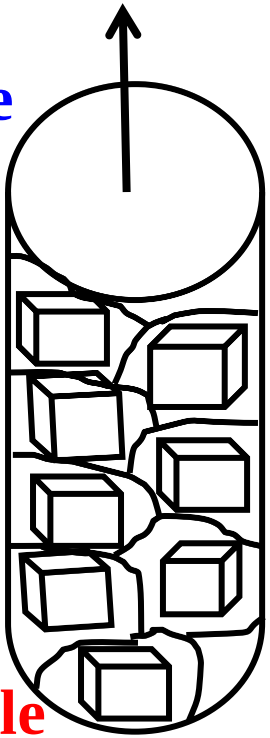


All crystals are oriented with their [001] || WD

-like a single crystal

–an ideal situation

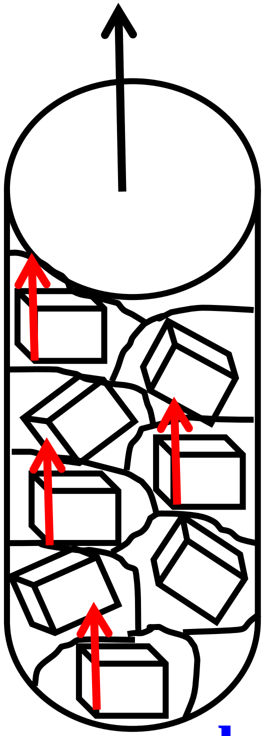
–not practicable



Texture is expressed as <001>, the direction parallel to wire axis

Most crystals are oriented with their [001] || WD

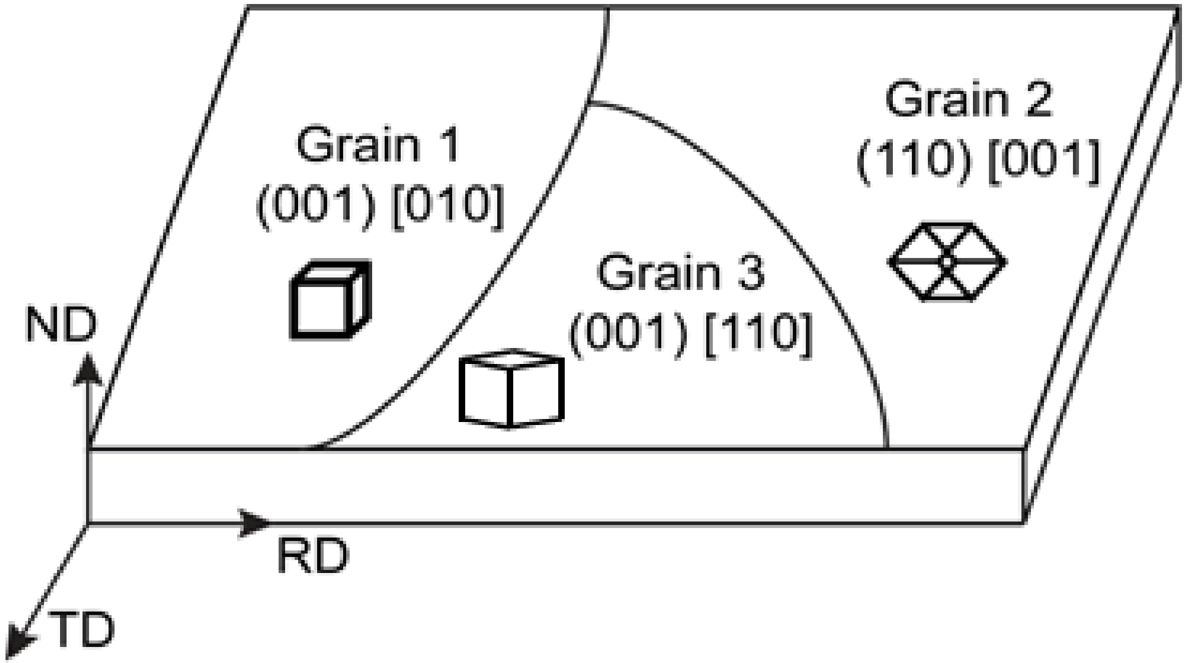
-a real situation



Texture is expressed as <001>, the direction parallel to wire axis

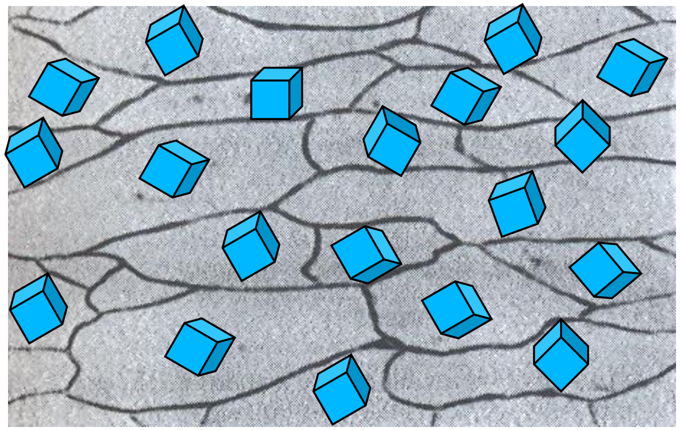
For biaxial deformation, like rolling, a combination of miller indices of sheet plane and the miller indices of the directions parallel to the longitudinal axes, say, (hkl) [uvw]

- **For the subsequent processes also , for example annealing, texture is described in terms of frame of reference of the prior deformation history. That means the texture of annealed sheet will be represented as (hkl)[uvw]**

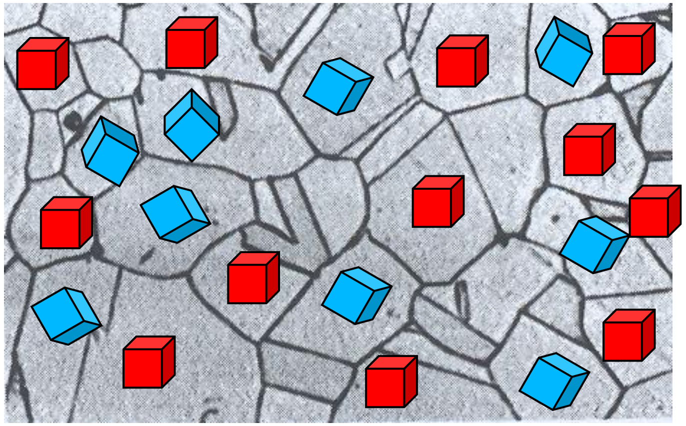


How texture is related to microstructure?

- ***Crystallographic texture is different from the patterns observed in optical or scanning electron micrographs***



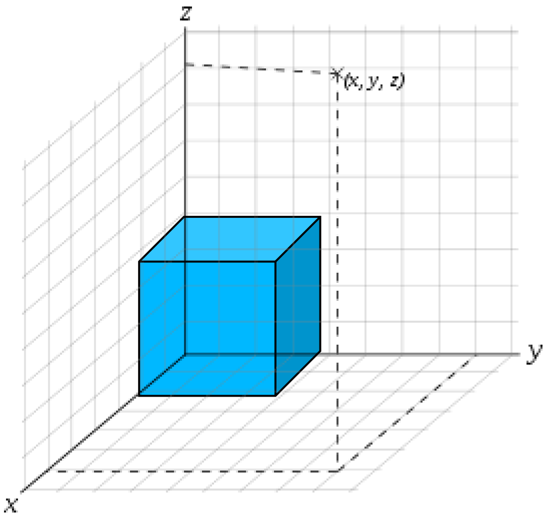
In this micrograph, most of the grains are depicted to have randomly oriented grains, as obtained by cross rolling with intermediate annealing. The morphology of grains appears to be, however, aligned.



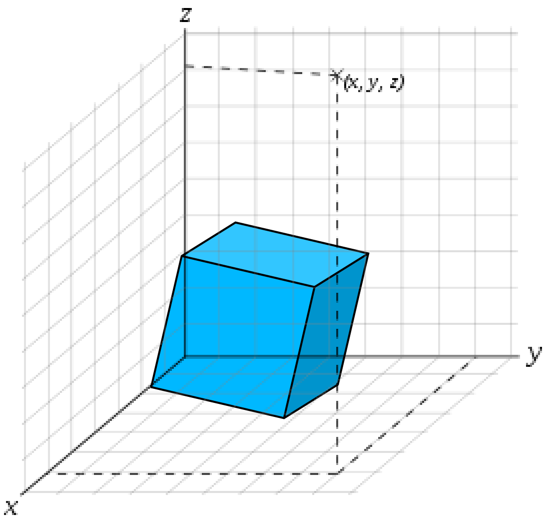
In this micrograph, large number of the red grains are depicted to have cube orientation, as obtained after annealing. The morphology of grains appears to be equiaxed.

- **Elongated or flattened grains do not imply a certain texture, or even the presence of texture at all**
- **Presence of equiaxed grains does not imply a random orientation.**

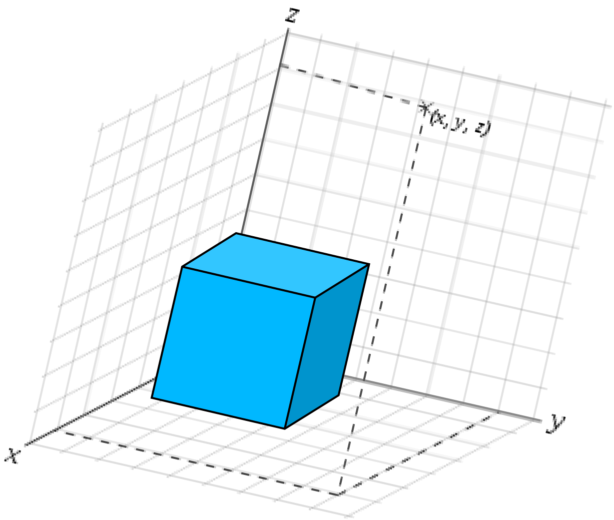
Reference system is the most important parameter to define crystallographic texture



Configuration A



Configuration B



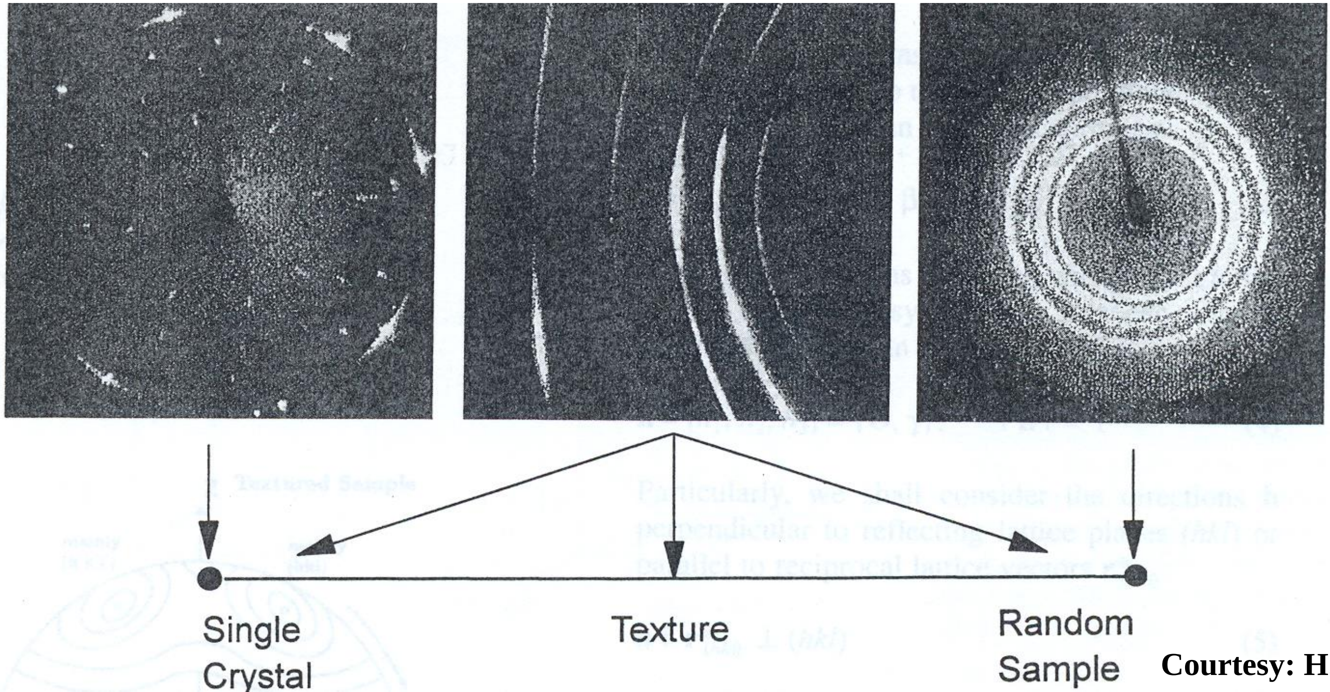
Configuration C

- **Configurations B and C represent different orientations**
- **Configurations A and C represent the same orientation**

Diffraction patterns from textured polycrystalline material

- **Diffraction patterns from a single crystal is in the form of isolated spots, while for a randomly oriented polycrystalline material, the diffraction pattern is in the form of concentric rings (Debye-Scherrer rings).**
- **Textured polycrystalline materials are somewhat in-between. Here the diffraction pattern consists of Debye-Scherrer rings, but the intensity distribution is non-unform along the circumference of the rings.**

Diffraction pattern from the crystals of materials



Courtesy: H.-J. Bunge

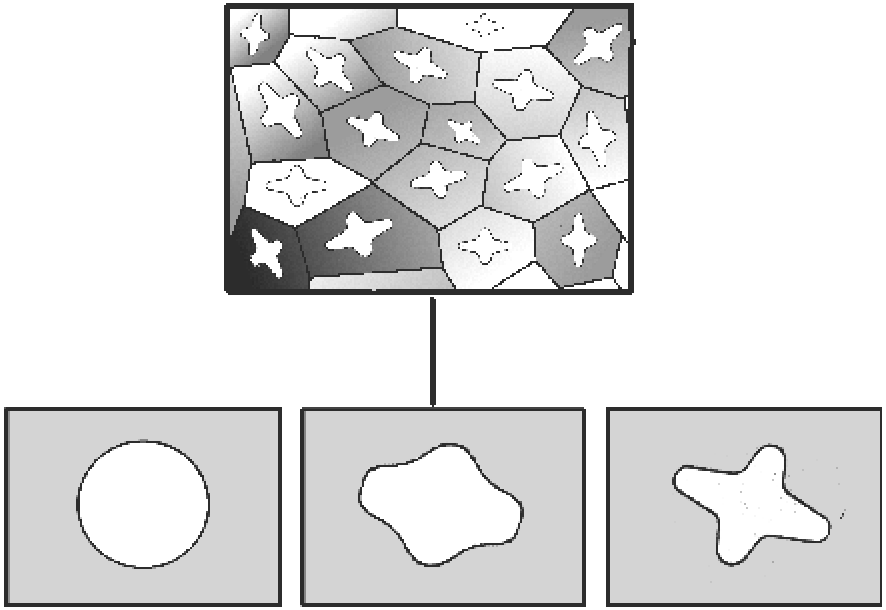
Texture Formation processes

Texture develops or changes due to:

- **(i) Crystallisation/ solidification**
  - ***(from a non-crystalline / liquid state)***
- **(ii) Plastic deformation**
  - ***(by slip and twinning)***
- **(iii) Annealing**
  - ***(from the same phase)***
- **(iv) Phase transformation**
  - ***(from a different phase)***

Why textures?

Texture influences the following properties:



- Elastic modulus
- Yield strength
- Tensile ductility and strength
- Formability
- Fatigue strength
- Fracture toughness
- Stress corrosion cracking
- Electric and Magnetic properties

..........and many other properties.

Major fields of application

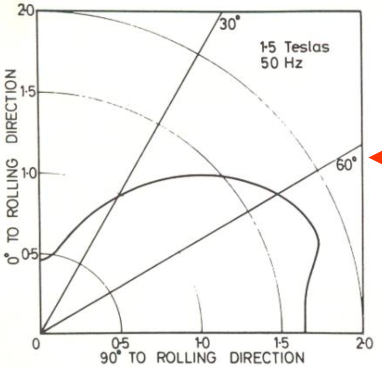
A. Conventional

- Aluminium processing
- Steel processing
  - LC steels
  - Electrical steels (transformer

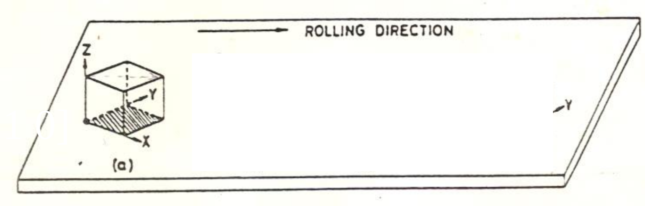
- Titanium alloy processing
- Zirconium base nuclear grade alloys
- ....

A. Modern

- High T_c superconductors
- Thin films for semiconducting and magnetic devices
- Bulk magnetic materials
- Structural Ceramics
- Polymers



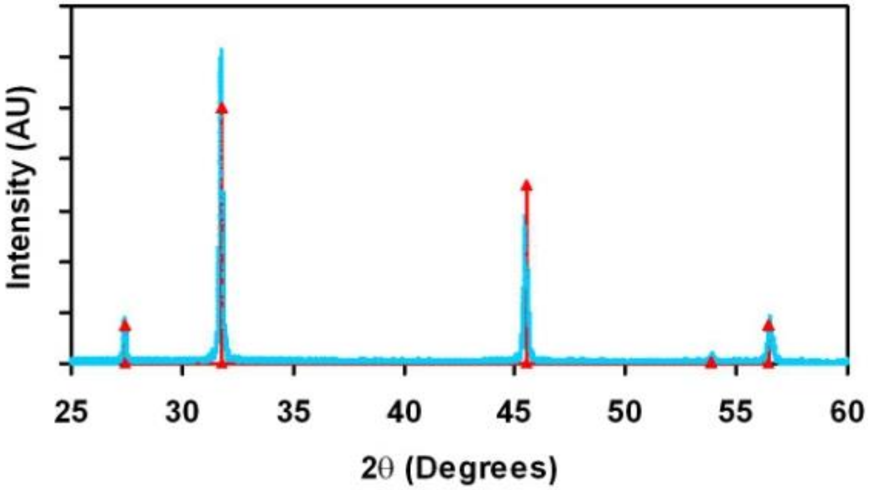
Variation of magnetic power loss with angle around the sheet for for Goss textured silicon iron

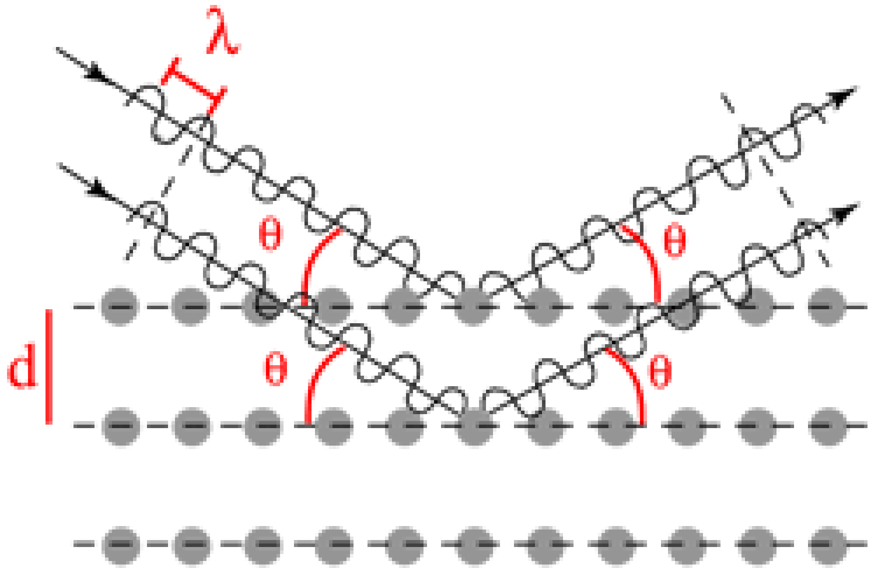


Fundamental Principles of Diffraction

Bragg's law

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





For parallel planes of atoms, with a space d_{hkl} between the planes, constructive interference only occurs when Bragg's law is satisfied.

First, the plane normal must be parallel to the diffraction vector

Plane normal: the direction perpendicular to a plane of atoms

Diffraction vector: the vector that bisects the angle between the incident and diffracted beam

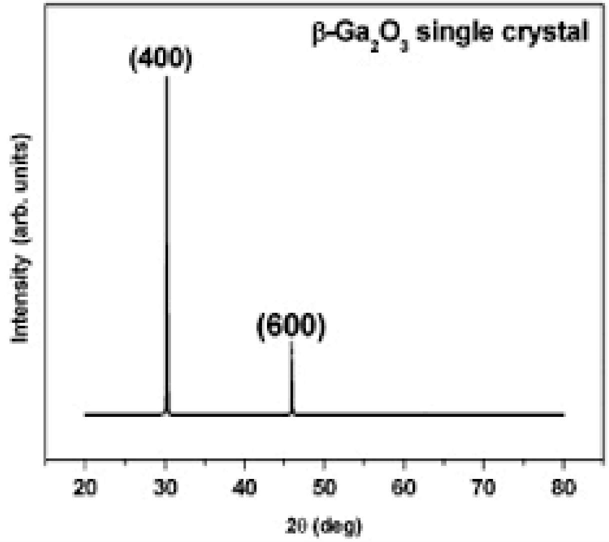
X-ray wavelengths l are:

Cu $K_{\alpha1}$=1.540598 Å and Cu $K_{\alpha2}$=1.544426 Å:: Or Cu $K_{\alpha(avg)}$=1.54278 Å

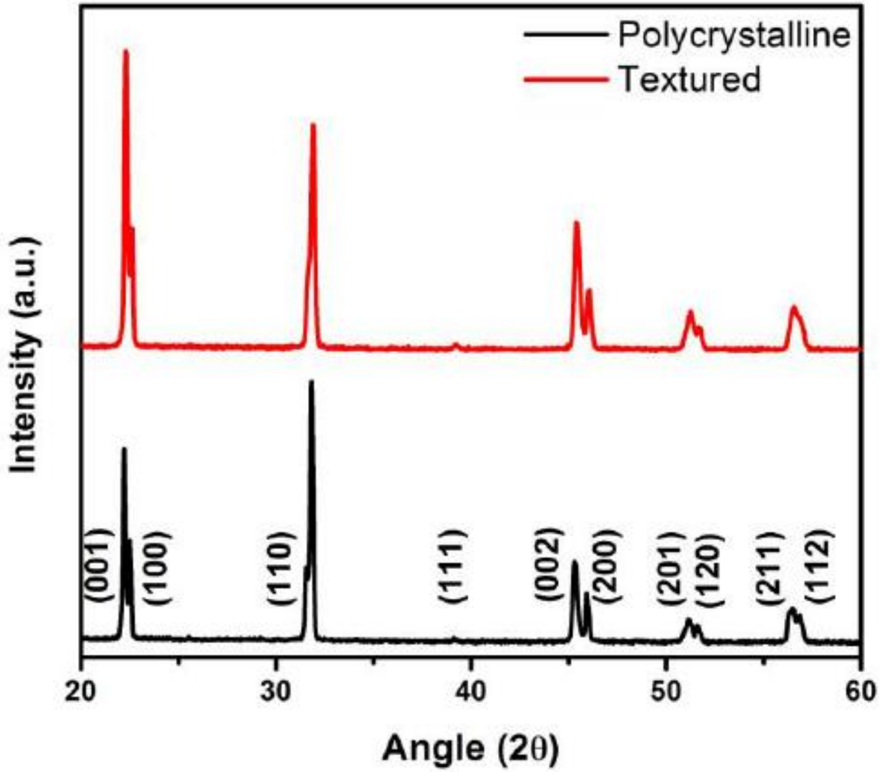
d_{hkl} is dependent on the lattice parameter (atomic/ionic radii) and the crystal structure

$I_{hkl}=I_opCL_P[F_{hkl}]^2$ determines the intensity of the peak

- **X-ray diffraction patterns provide a lot of information about materials**
  - **Single crystalline materials (e.g. turbine blades)**

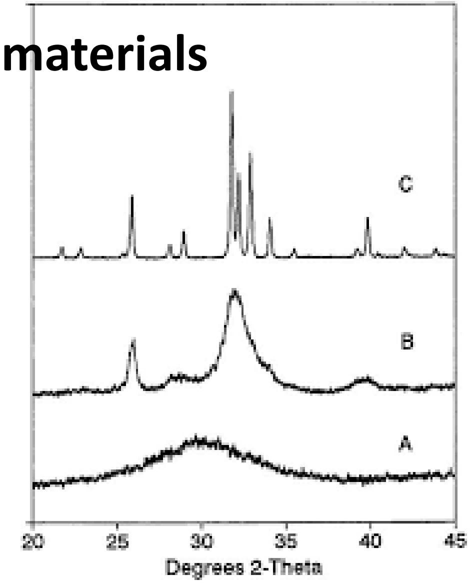


  - **Polycrystalline materials (e.g. Powders)**
  - **Textured polycrystalline materials (e.g. sheets , wires , rods etc.)**

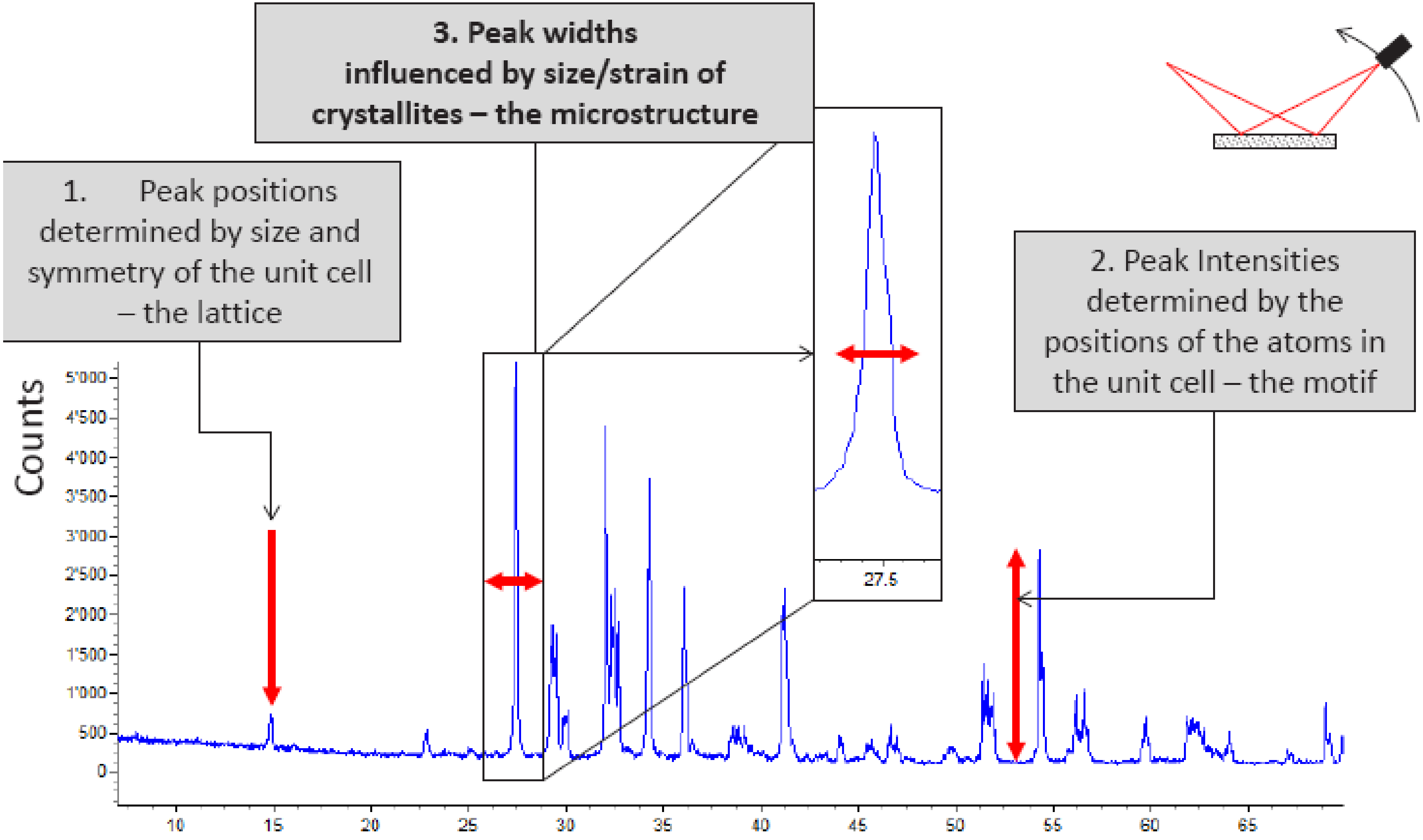


  - **Amorphous materials**

(e.g. glasses)



Inside the powder diffraction pattern

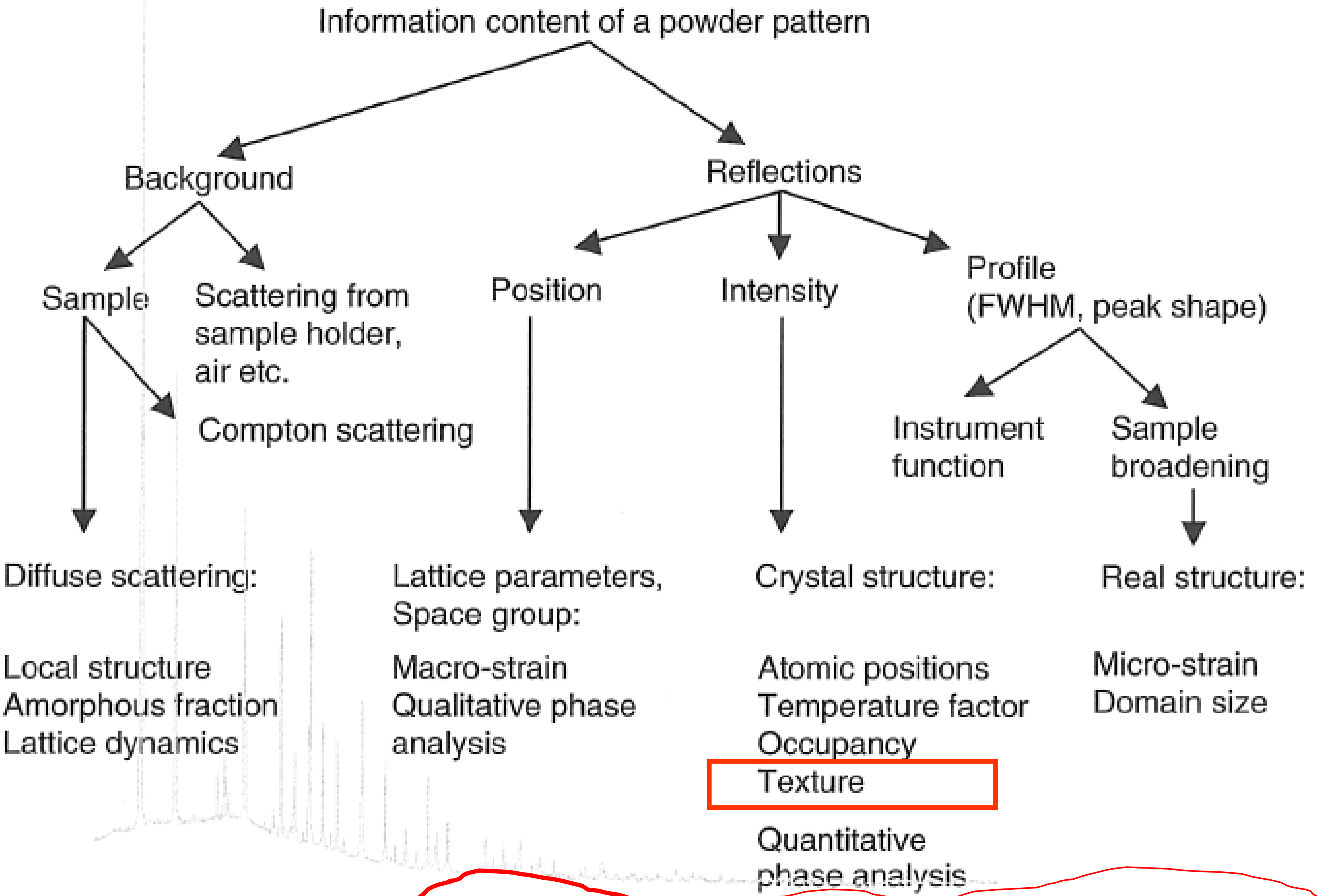


3. Peak widths influenced by size/strain of crystallites – the microstructure

1. Peak positions determined by size and symmetry of the unit cell – the lattice

2. Peak Intensities determined by the positions of the atoms in the unit cell – the motif

Information content of a powder pattern



Background

Sample

Scattering from sample holder, air etc.

Compton scattering

Diffuse scattering:

Local structure
Amorphous fraction
Lattice dynamics

Reflections

Position

Lattice parameters, Space group:

Macro-strain
Qualitative phase analysis

Intensity

Crystal structure:

Atomic positions
Temperature factor
Occupancy
Texture

Quantitative phase analysis

Profile (FWHM, peak shape)

Instrument function

Sample broadening

Real structure:

Micro-strain
Domain size

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

$$\mathbf{F}(\mathbf{h}) = \sum_{j=1}^{n} g^{j} t^{j}(s) f^{j}(s) \exp(2\pi i \mathbf{h} \cdot \mathbf{x}^{j})$$

Crystal orientation

Texture

Graphical description of texture

Pole figure

A stereographic projection with a specified orientation relative to the specimen

Shows variation of pole density with pole orientation for a selected set of crystal planes

Stereographic projection

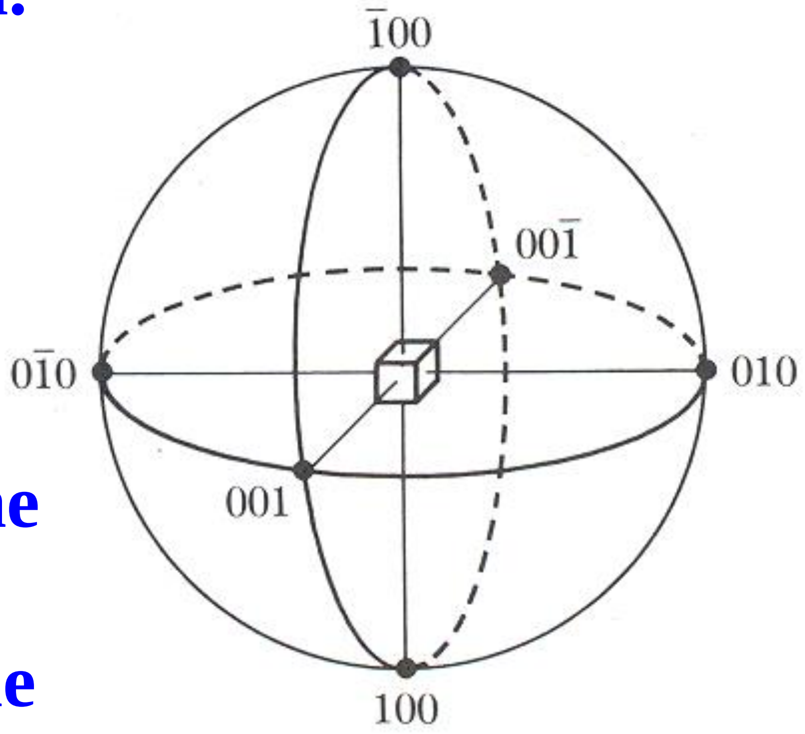
- **mapping of crystallographic planes and directions in a convenient and straightforward manner.**
- **two dimensional drawing of three dimensional data.**
- **planes are plotted as great circle lines, and directions are plotted as points.**
- **sometimes, planes are also indicated by the normal to them, that is, by a point.**

What do we learn further **:**

- **geometrical correspondence between crystallographic planes and directions with their stereographic projections.**
- **important crystallographic directions that lie in a particular plane of a crystal.**

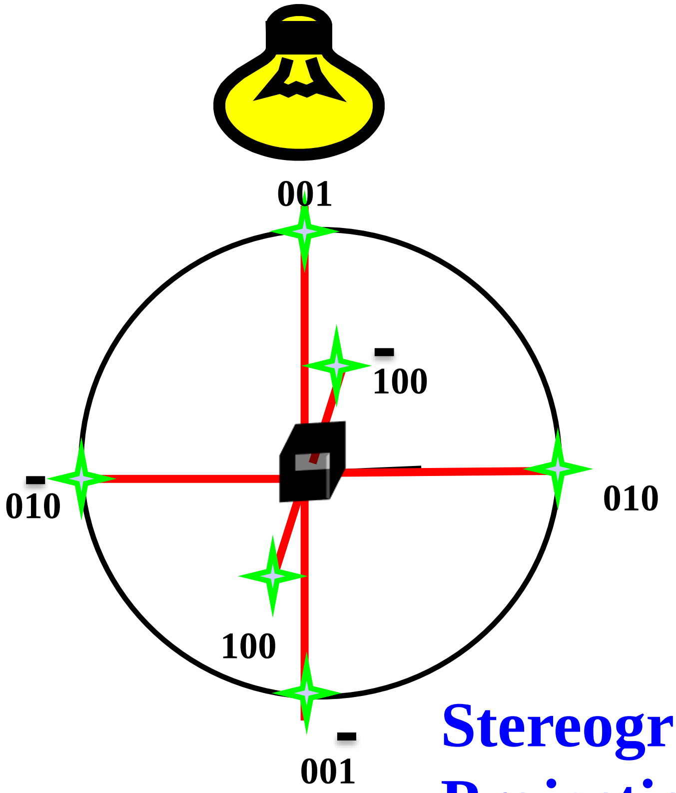
What is a pole?

- Let us consider a small unit cell.
- Construct a big sphere, called the Reference Sphere, around the point unit cell taken as the centre.
- Next draw perpendiculars to the six cube faces of the unit cell and extend these till they cut the reference sphere.



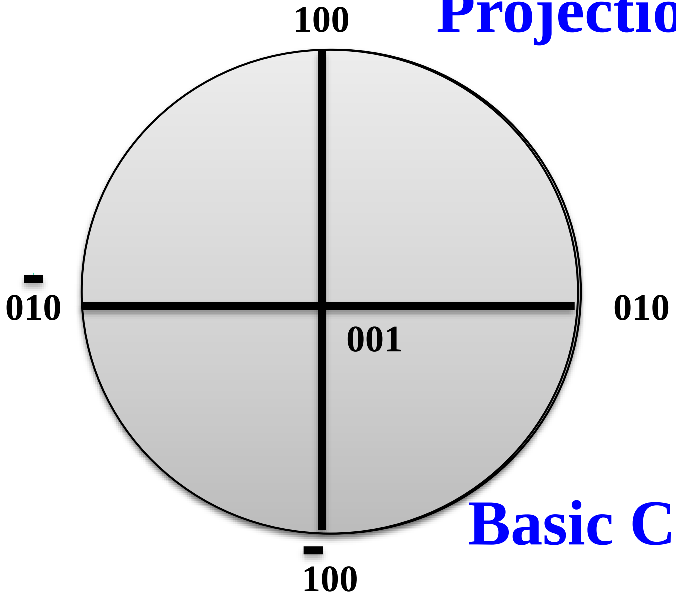
- These points of intersection of the plane normals with the reference sphere are known as the poles of the respective cube planes, (100), ($\bar{1}$00), (010), (0$\bar{1}$0), (001) and (00$\bar{1}$).

- Place a source of light, say at the 001 pole position and then allow the light rays passing through the poles 100, $\bar{1}00$, 010 and $0\bar{1}0$ to fall on a piece of paper put perpendicular to the 001- $00\bar{1}$ axis.

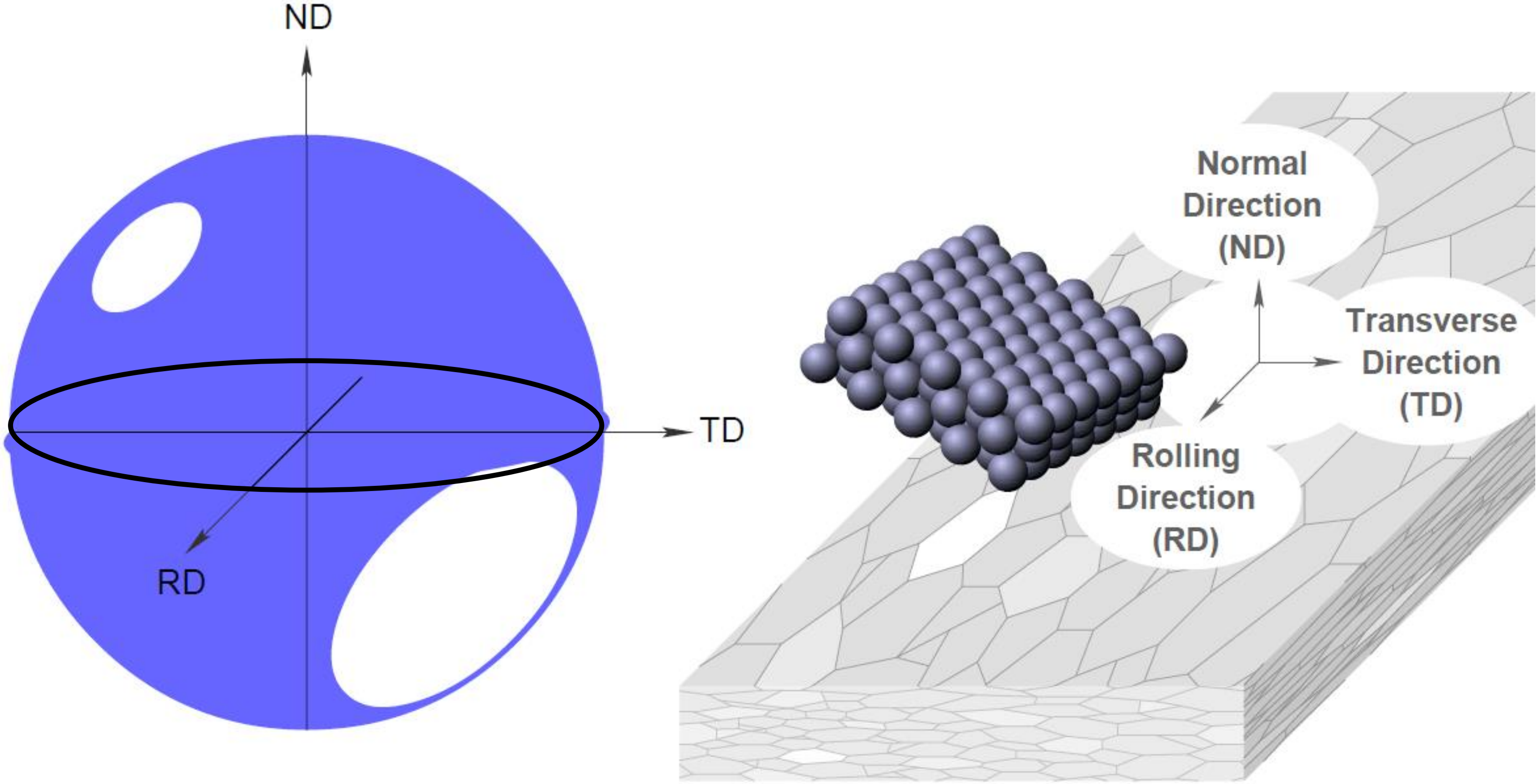


Stereographic Projection

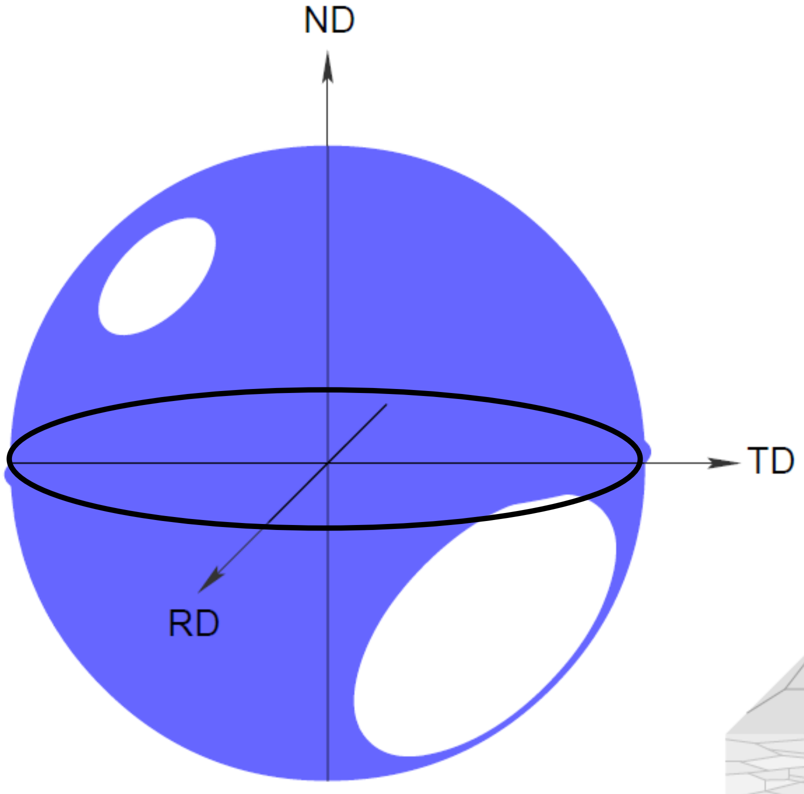
- The lower half of the reference sphere will be projected as a circle, known as the Basic Circle on the piece of paper which is the projection plane, parallel to the (001) plane of the unit cell.

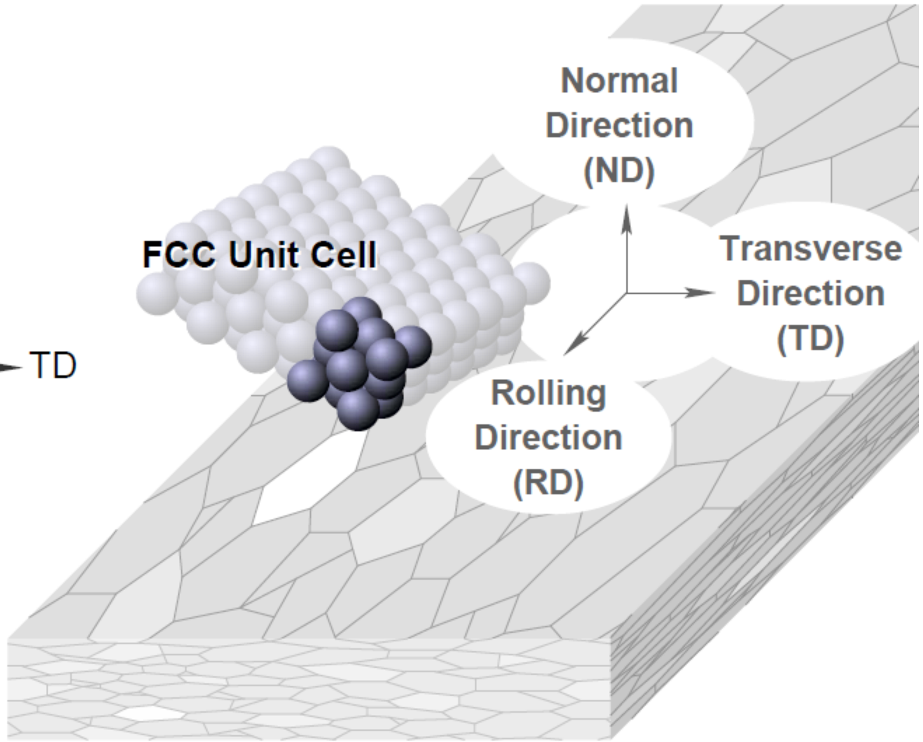


Basic Circle

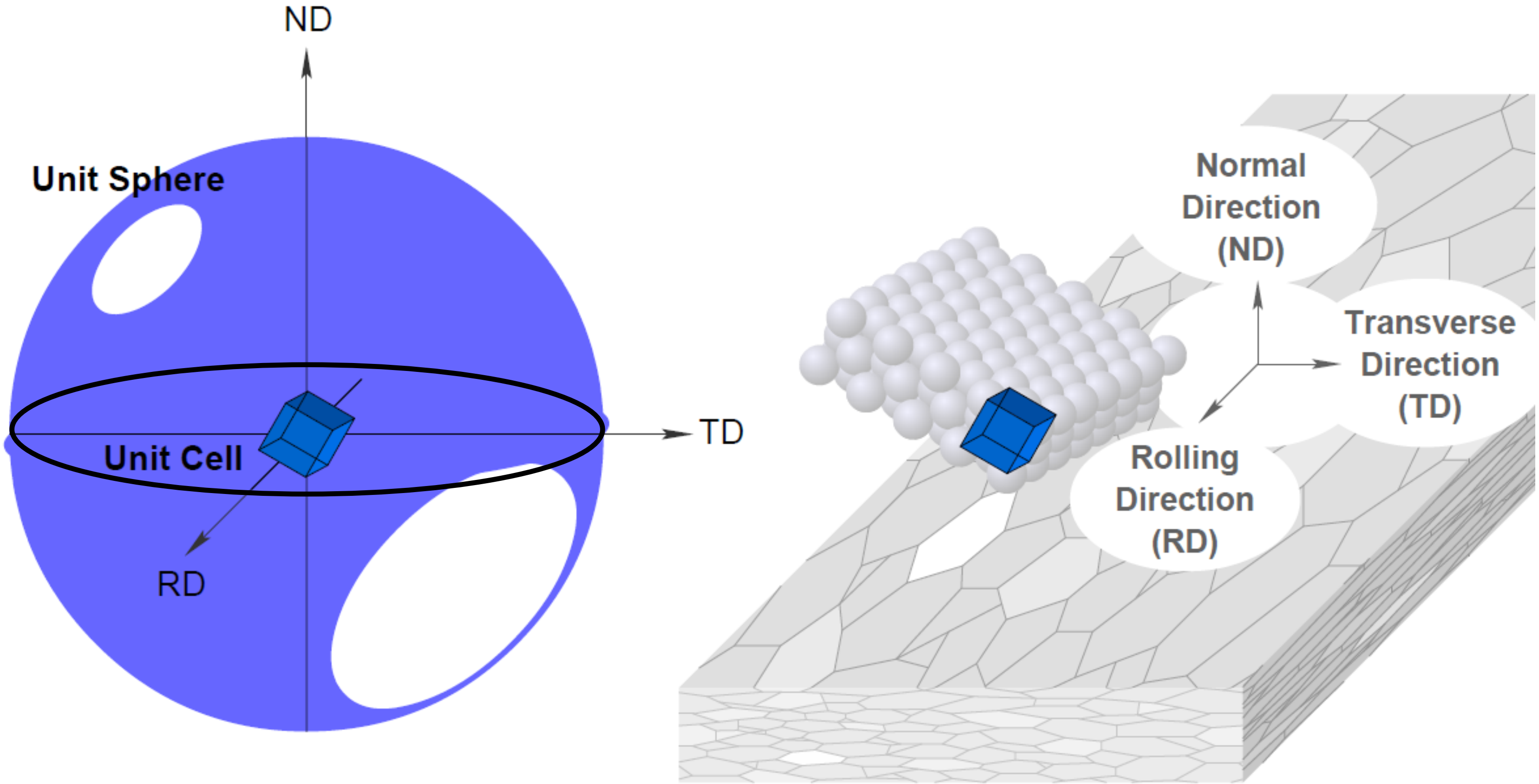


A section of the grain's atomic structure is shown. Note the FCC (face-centred cubic) crystal structure of aluminium.

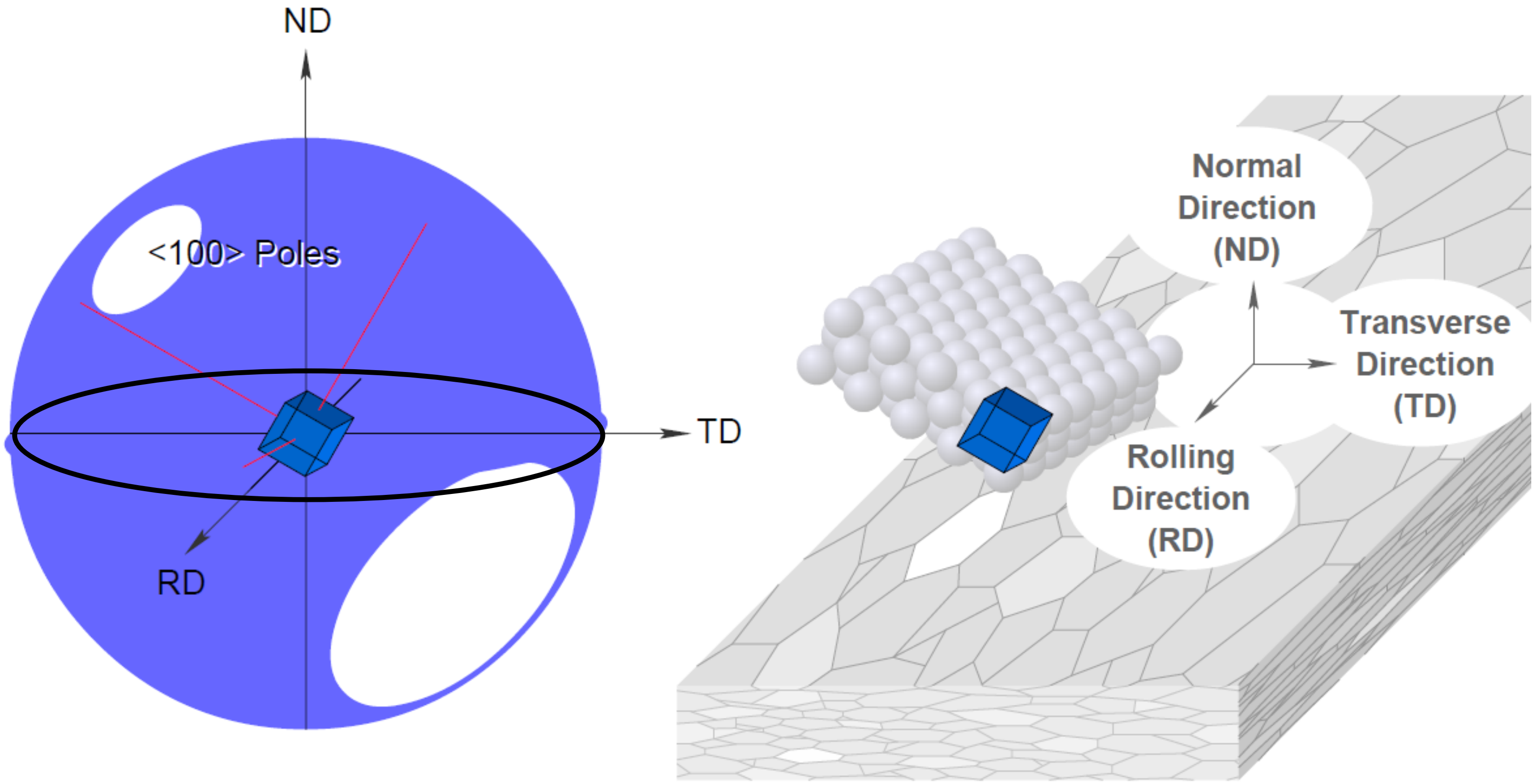




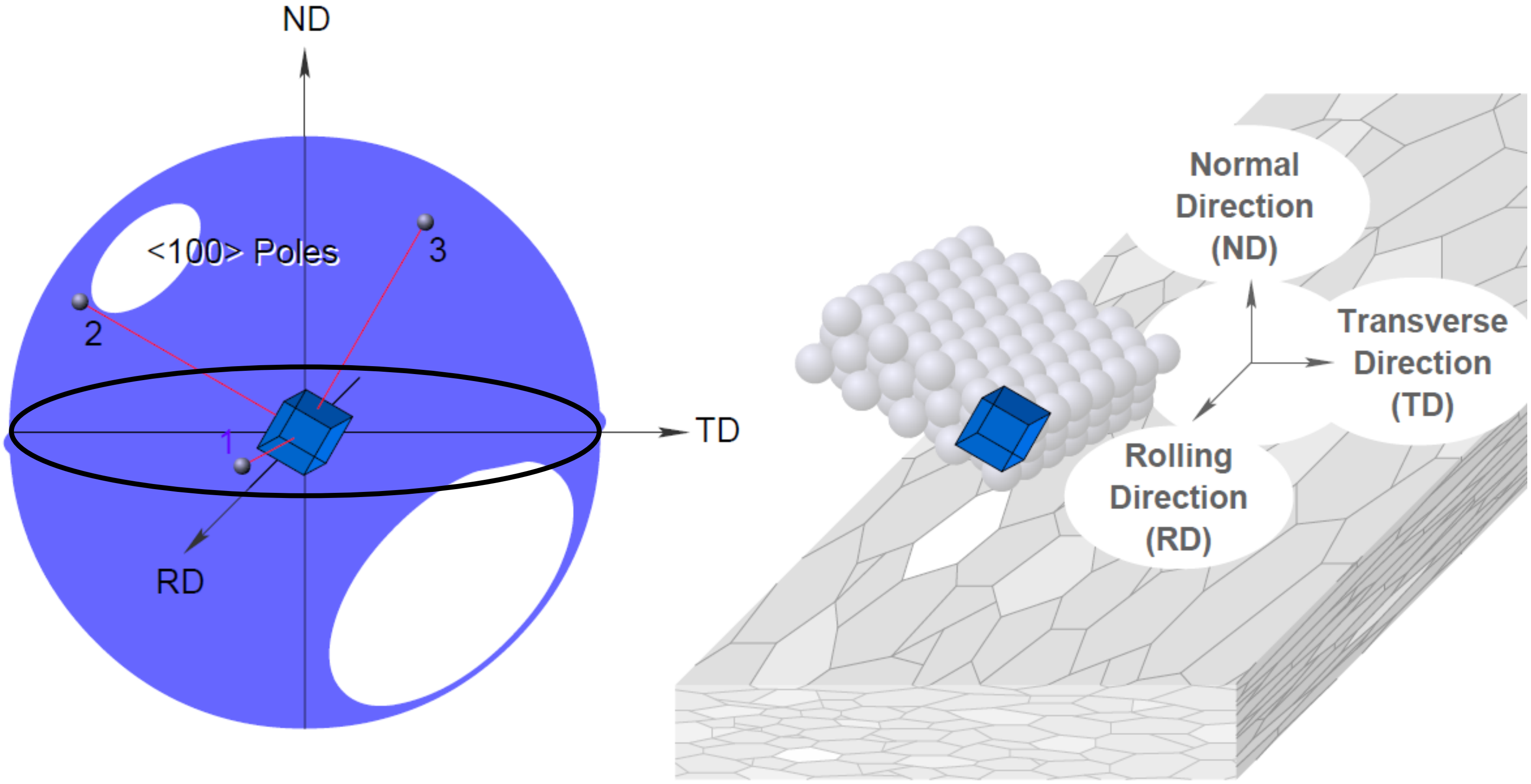
A single unit cell of the FCC crystal structure is highlighted.



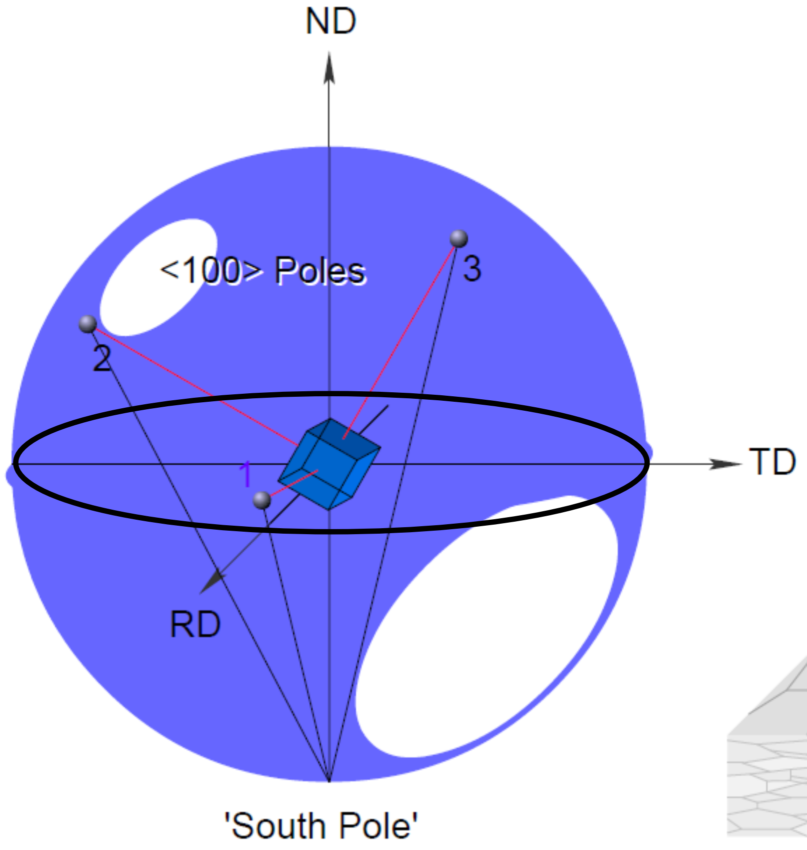
Let's transpose the unit cell in its identical orientation to the centre of the unit sphere.
The unit sphere has the same axes (RD, TD, ND) as the cold-rolled plate.

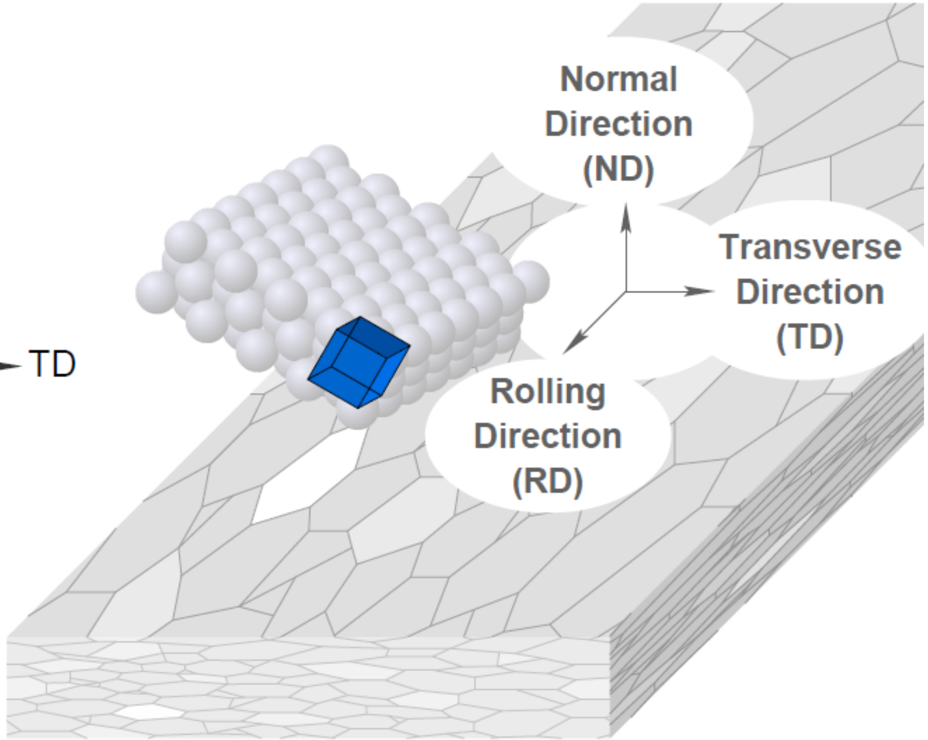


The normal vectors from each face of the unit cube are drawn to intersect the surface of the unit sphere. These are termed the <100> poles. Only those on the 'northern' hemisphere are required for this construction.

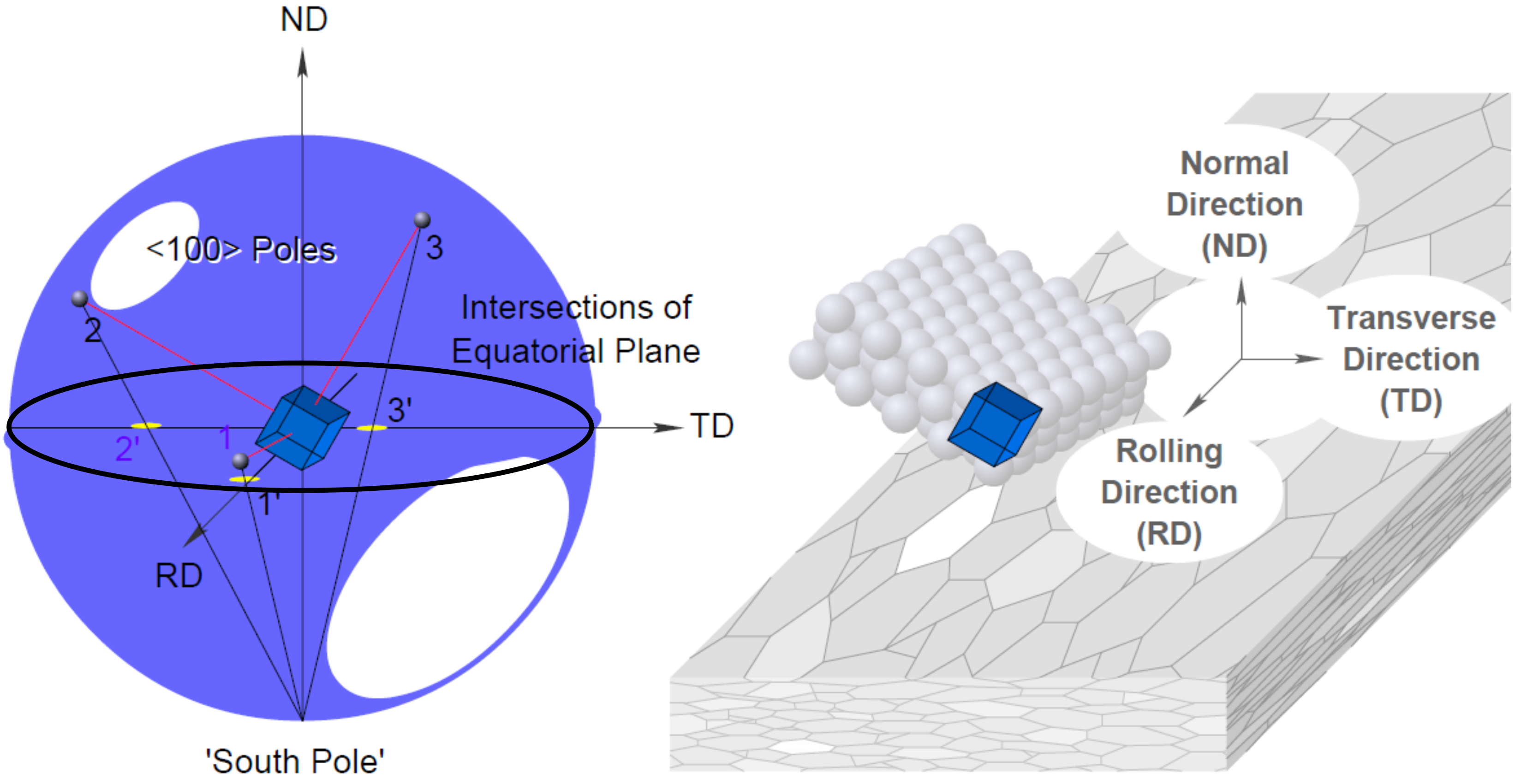


The points of of intersection of the <100> poles with the unit sphere are labelled 1, 2 and 3.

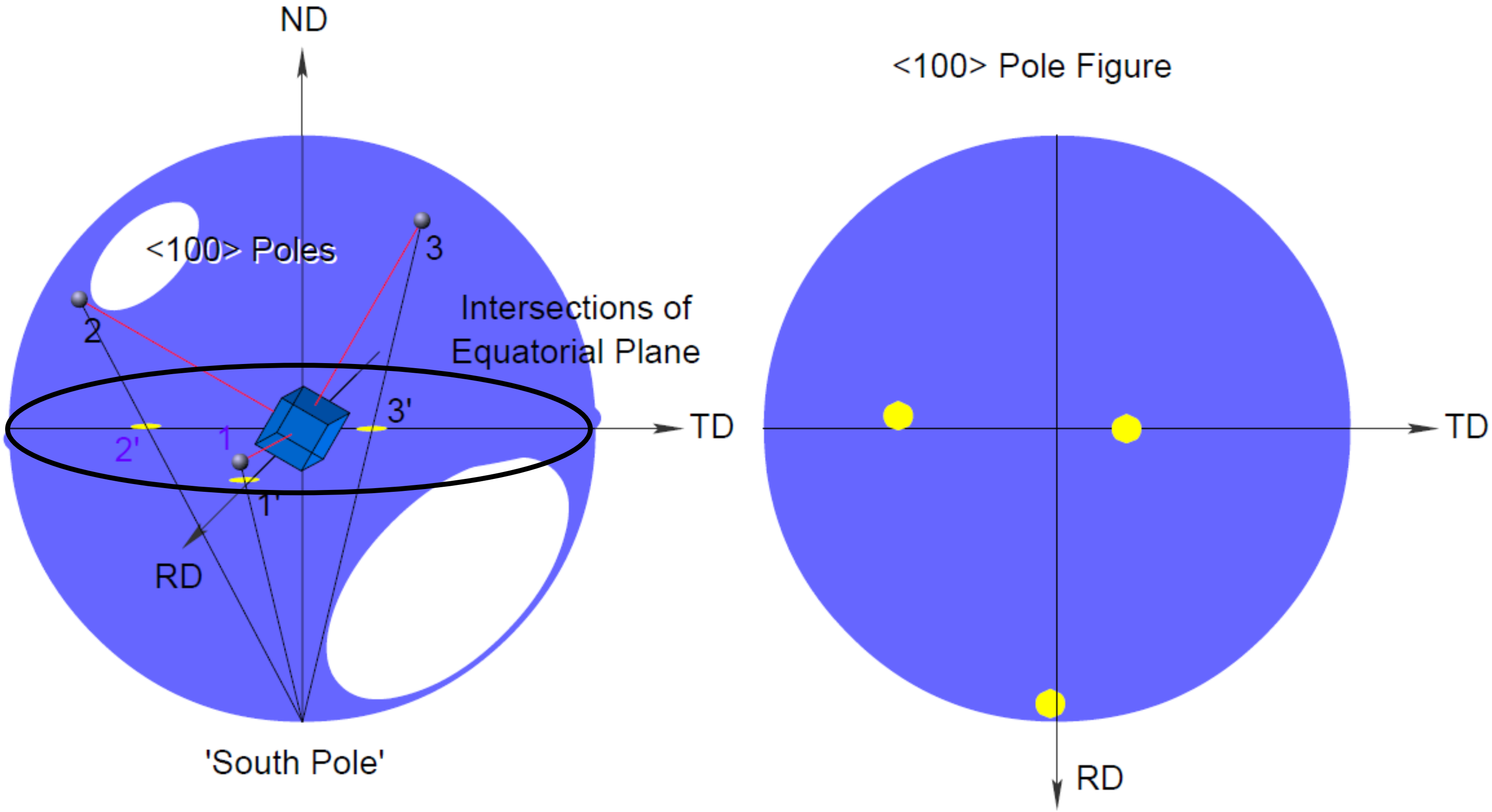




Now we connect the points 1, 2 and 3 to the south pole.

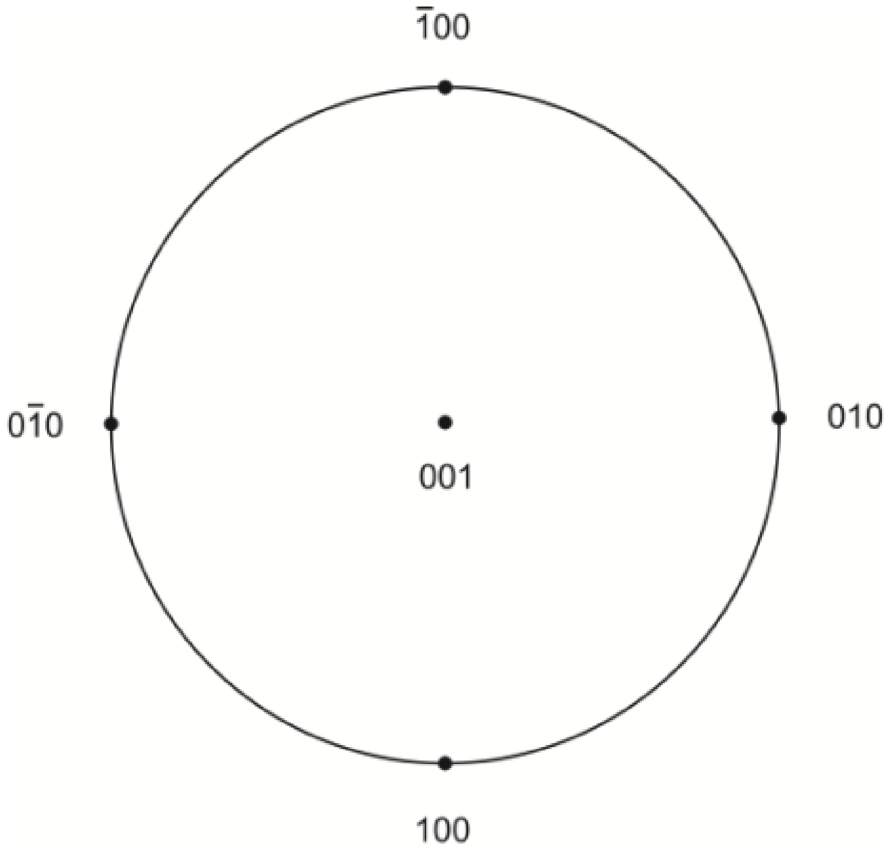


Points 1', 2' and 3' are drawn where these connecting lines intersect the equatorial plane. These are the 100 poles and uniquely represent the orientation of the unit cell in 3D space on a 2D plane.



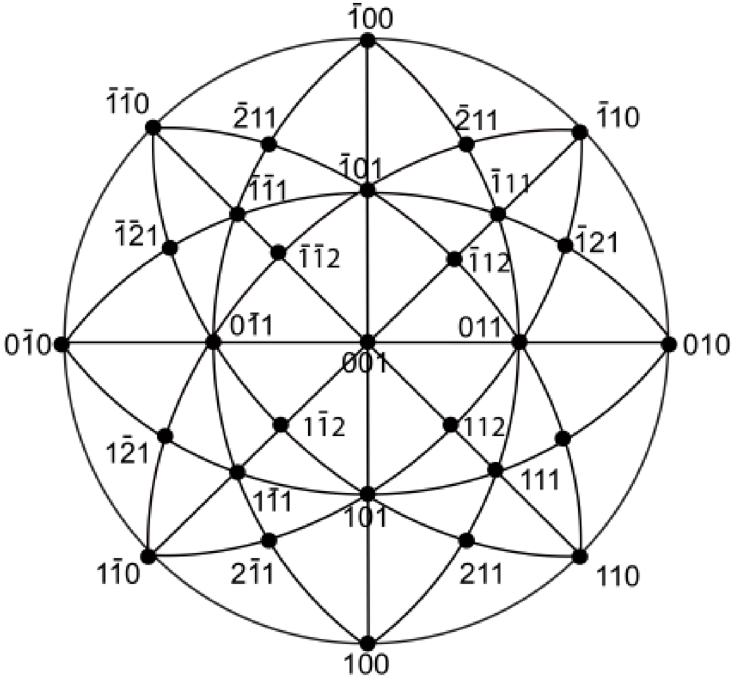
Here the equatorial plane is viewed from above to form a stereographic projection.

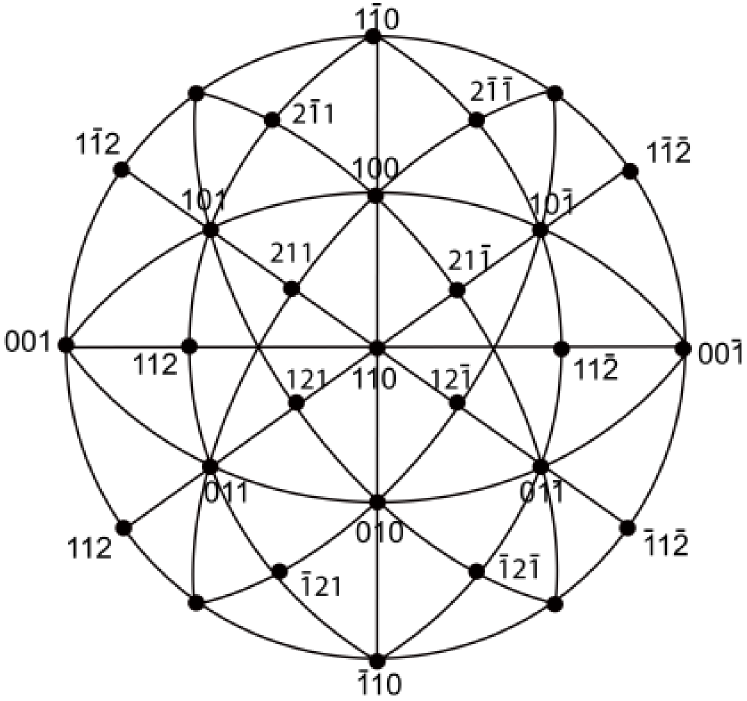
- **The pole 001 will be at the centre of the basic circle, while the poles 100, $\bar{1}00$, 010 and $0\bar{1}0$ will appear on the periphery of the basic circle.**

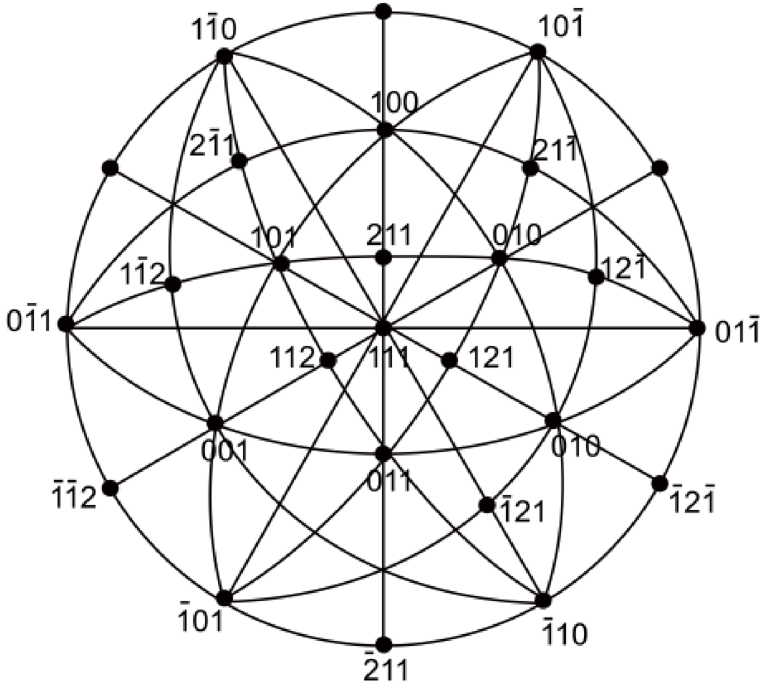


- **The basic circle, along with the poles of different {100} type planes is a stereographic projection of the three dimensional unit cell, placed at the centre of the reference sphere.**
- **Since the plane of the projection is parallel to the (001) plane, the projection is known as the (001) stereographic projection of the cubic unit cell.**

- **In a similar manner, the poles of the {100}, {110} and {111} planes can also be plotted with, say {100} and {111}, as the projection planes and the resulting stereographic projections (or stereograms)**





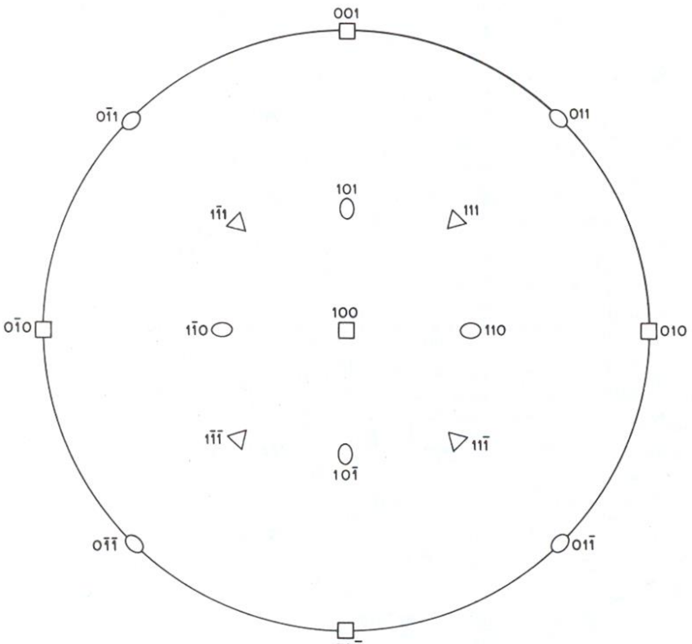


Pole figure

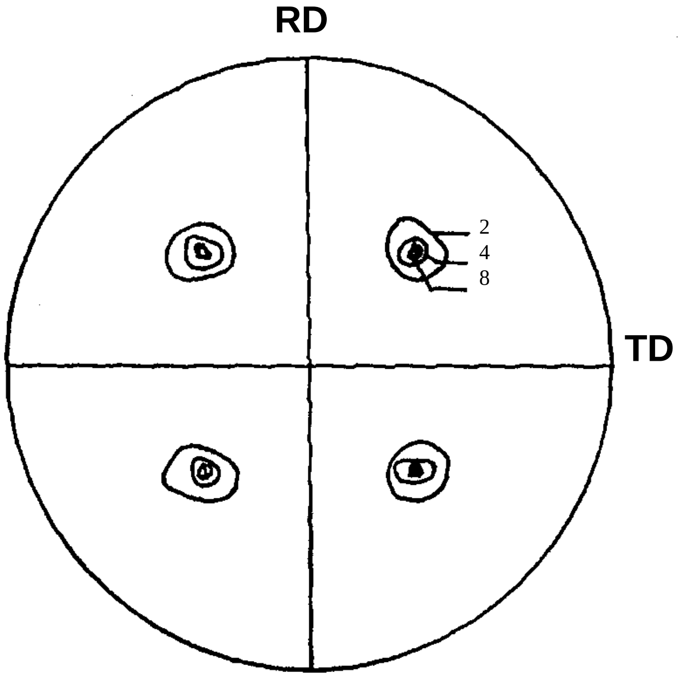
- **Orientation of crystallites can be represented through pole figures, which are modified form of stereographic projection.**

Stereogram: **A representation of 3D unit cell in 2D plane**

Pole figure: **Stereogram of polycrystal with sample frame of reference embedded**



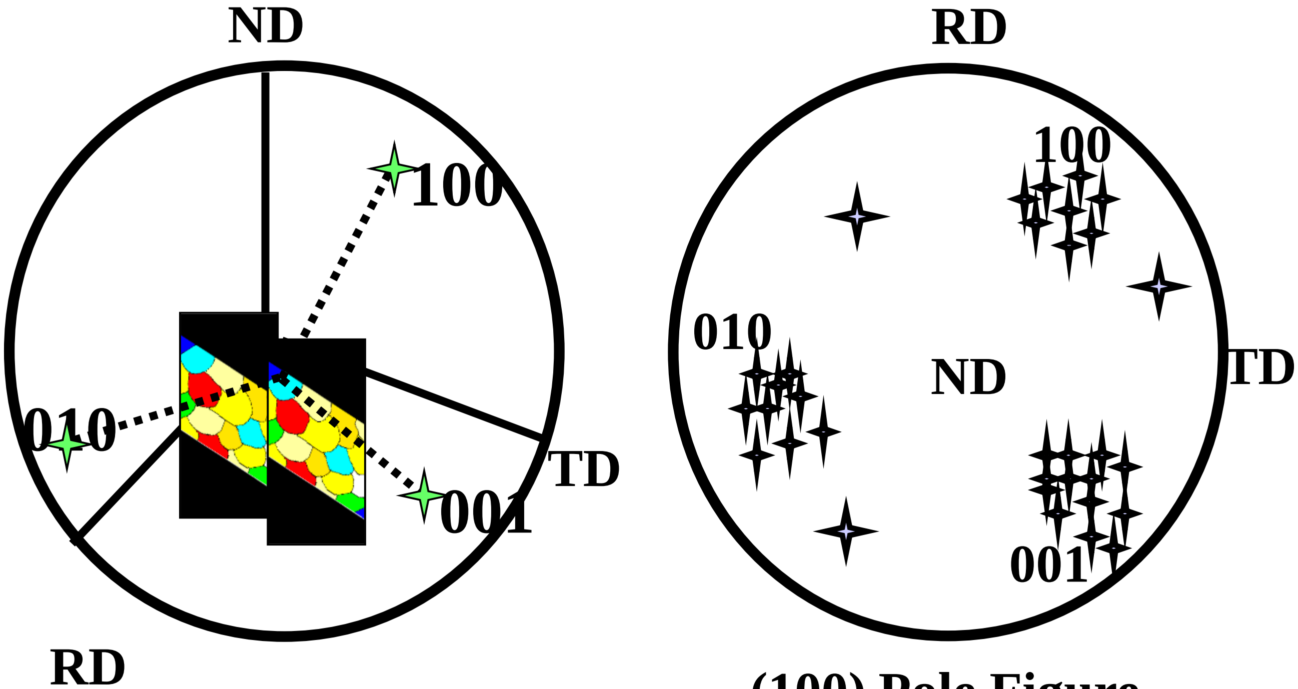
Stereogram



Pole figure

Pole figure

- Grains those are oriented along (001) in the predefined reference will show intensity around the (100) poles



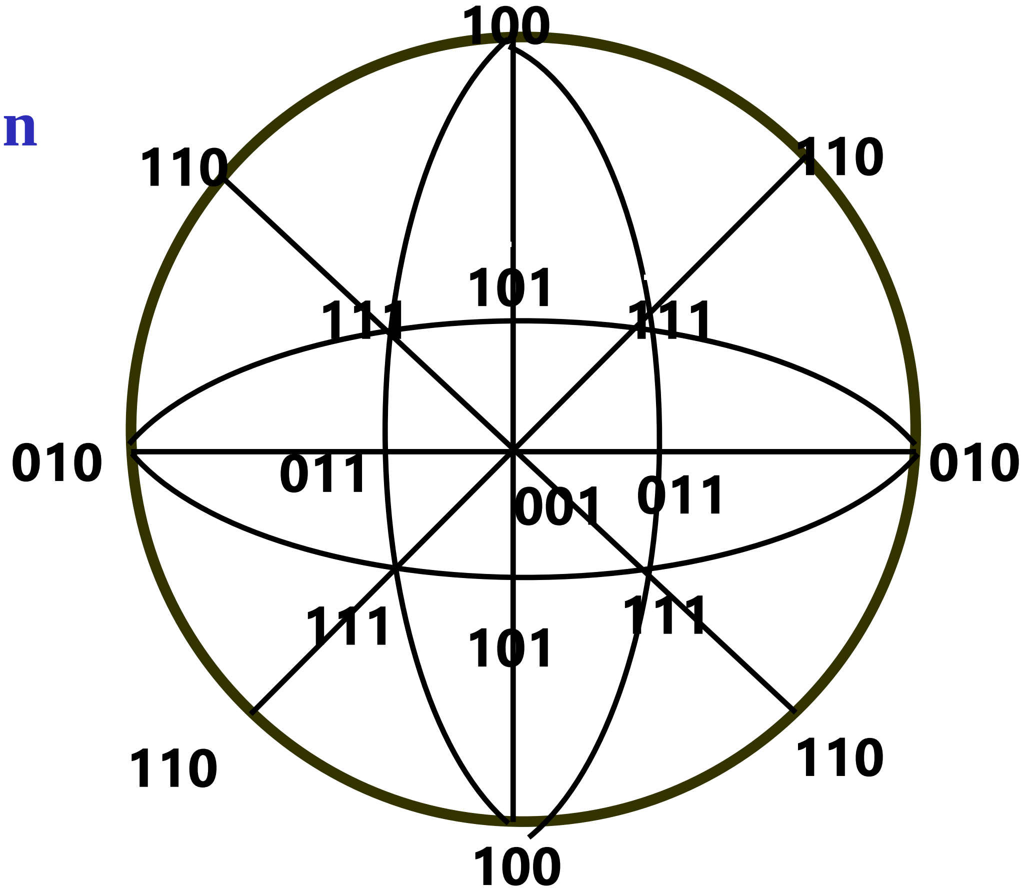
(100) Pole Figure

Schematic representation of the orientation distribution in (100) pole figure

- Pole figures essentially represent the orientation spread around the predefined pole or reference

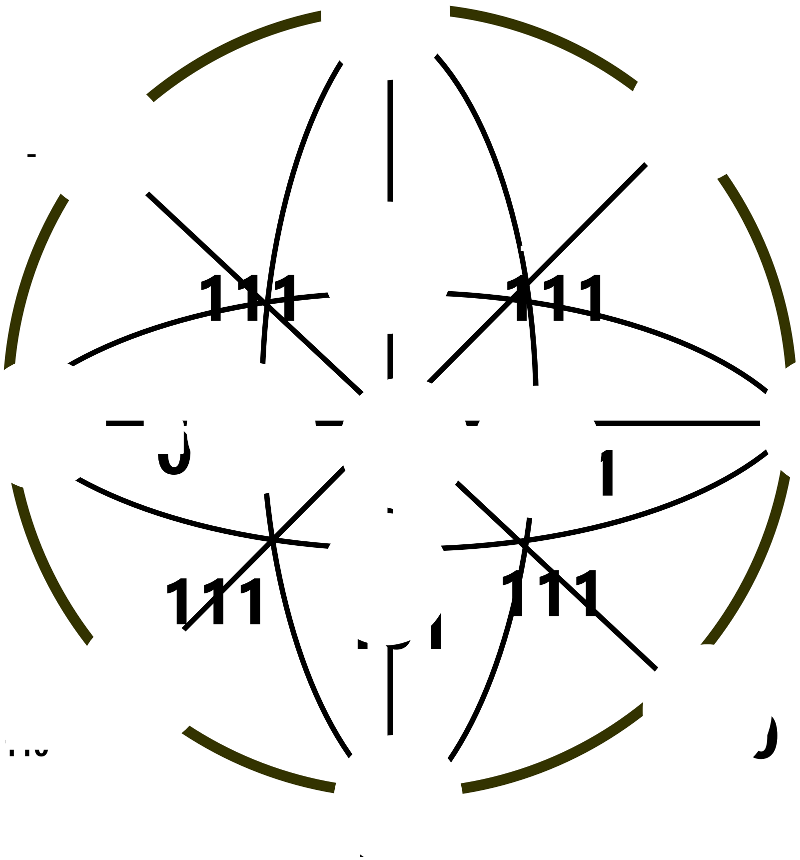
Orientations representation in a pole figure: Cubic Case

- (001) Standard stereographic projection of a cubic crystal
- (001) Pole lies at the center of stereogram



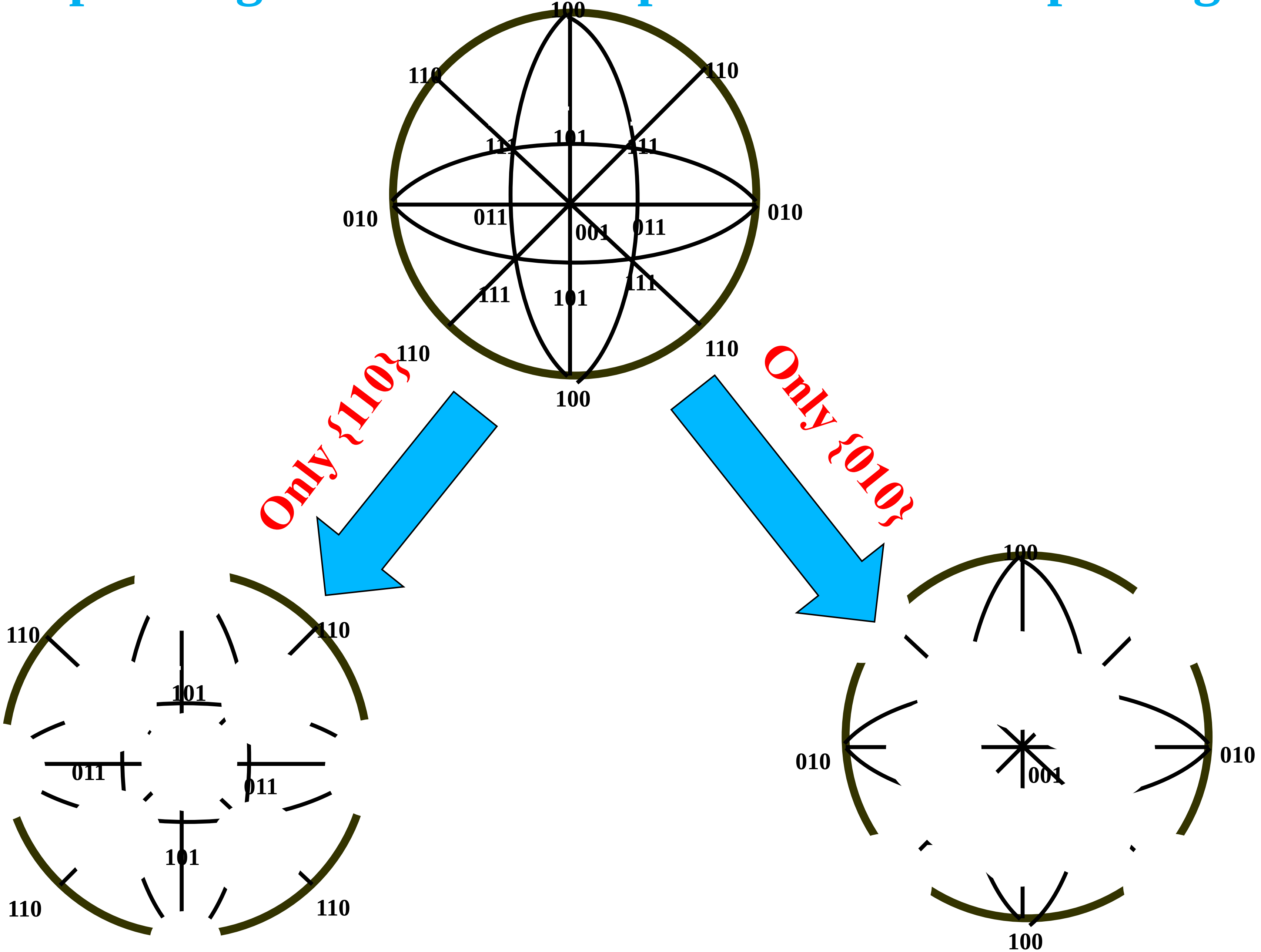
Orientations representation in a pole figure: A Cubic Case

If only {111}
orientations are present
in the sample

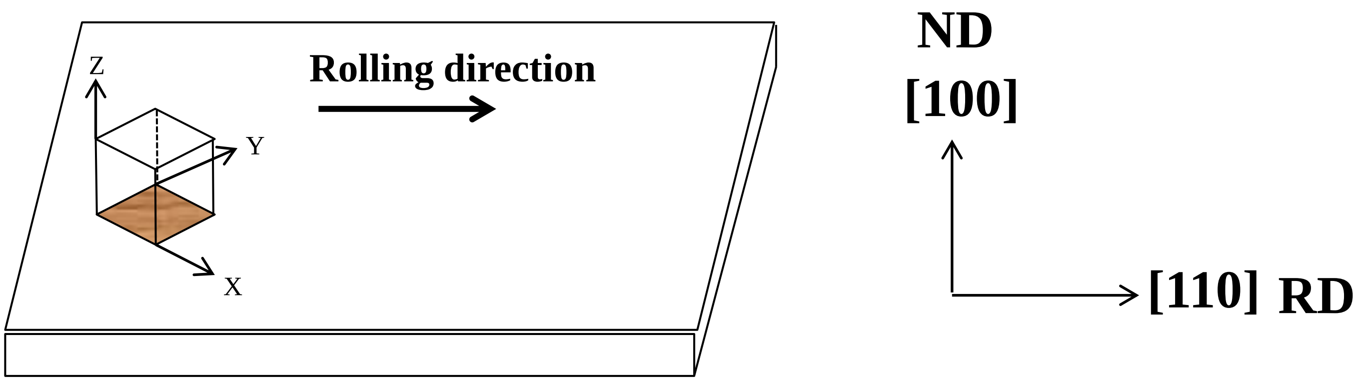


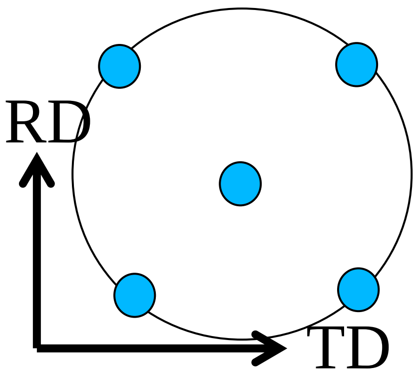
All the orientations appear
around {111} in the stereogram

Corresponding Orientations representation in a pole figure

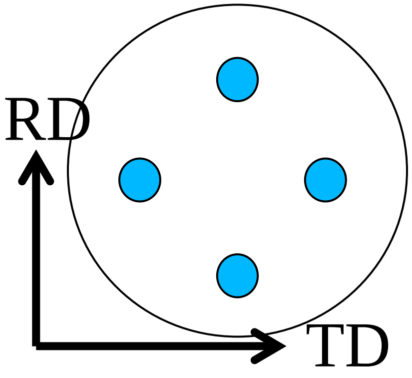


(100)[110] Orientation representation in sample frame of reference

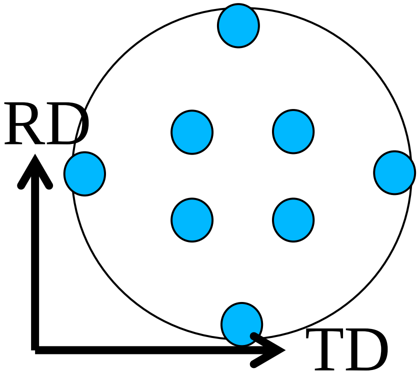




(100) Pole figure



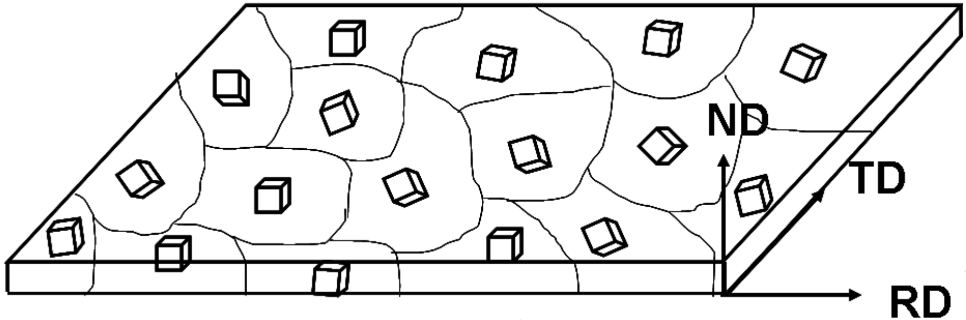
(111) Pole figure

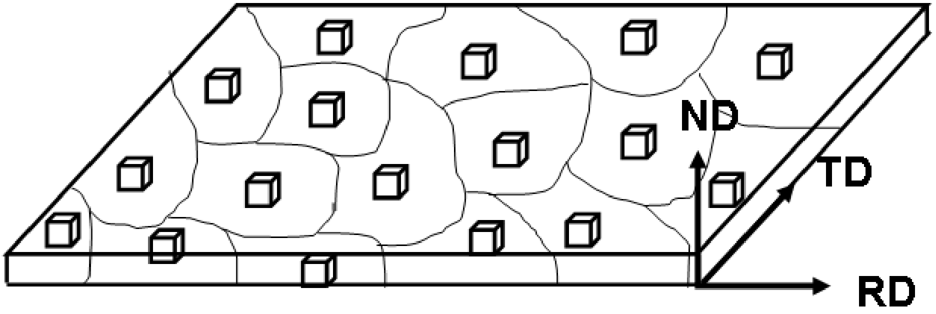


(110) Pole figure

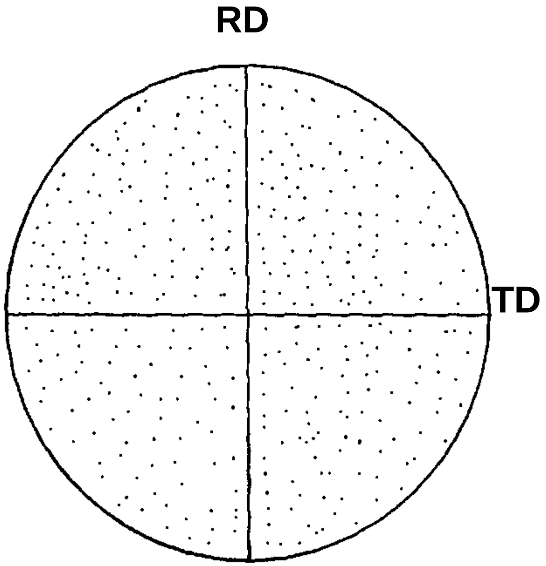
Crystal orientation represented in the RD, TD and ND of rolling co-ordinate

Representation of orientation in polycrystalline material

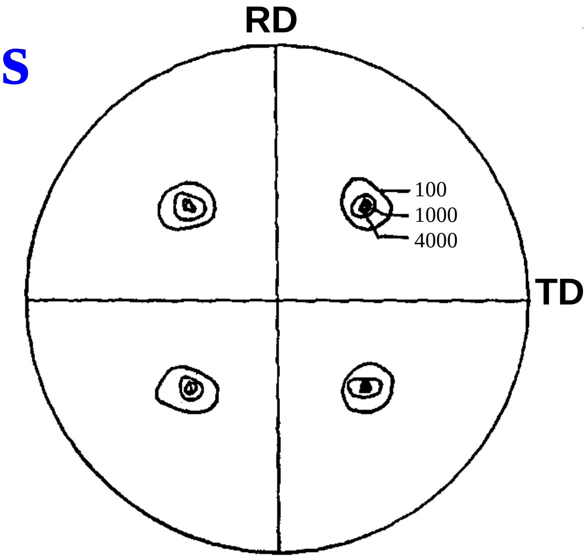




Pole figures corresponding to above crystal configurations



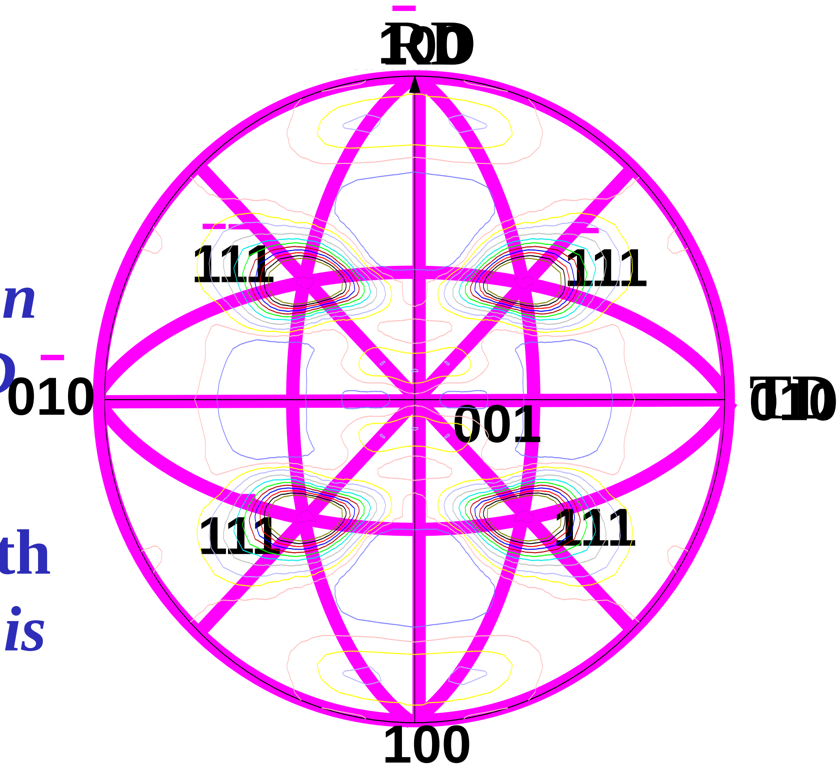
(111) Or any other Pole figure



(111) Pole figure

How to read the orientation from the pole figures?

- If the (hkl) pole figure has region of high intensity at the centre, choose the (hkl) standard projection
- Coincide the centre of (any) stereogram with ND position of the (hkl) pole figure *(e.g. the (001) position of the stereogram is coincided with ND of the (111) pole figure in the figure)*
- Note the pole (uvw) coinciding with RD of the pole figure. *(e.g. (100) pole is coincident with RD in the pole figure given here.*
- The texture component will be (hkl)[uvw]. *(e.g. the texture component is (001)[100])*



The pole density locations coincides with (111) poles of the (001) stereogram

- **If the measured (hkl) pole figure does not have a region of high intensity at the centre then exclude the (hkl) projection and take any other projection to index the pole figure**

- **Rotate the projection in such a manner that the regions of high intensity in the measured pole figure coincide with the (hkl) poles of the projection**

- **Note the pole (hkl) on the projection coinciding the ND position of the measured pole figure and the pole (uvw) coinciding the RD position of the pole figure**

- **The texture component will be (hkl)[uvw]**

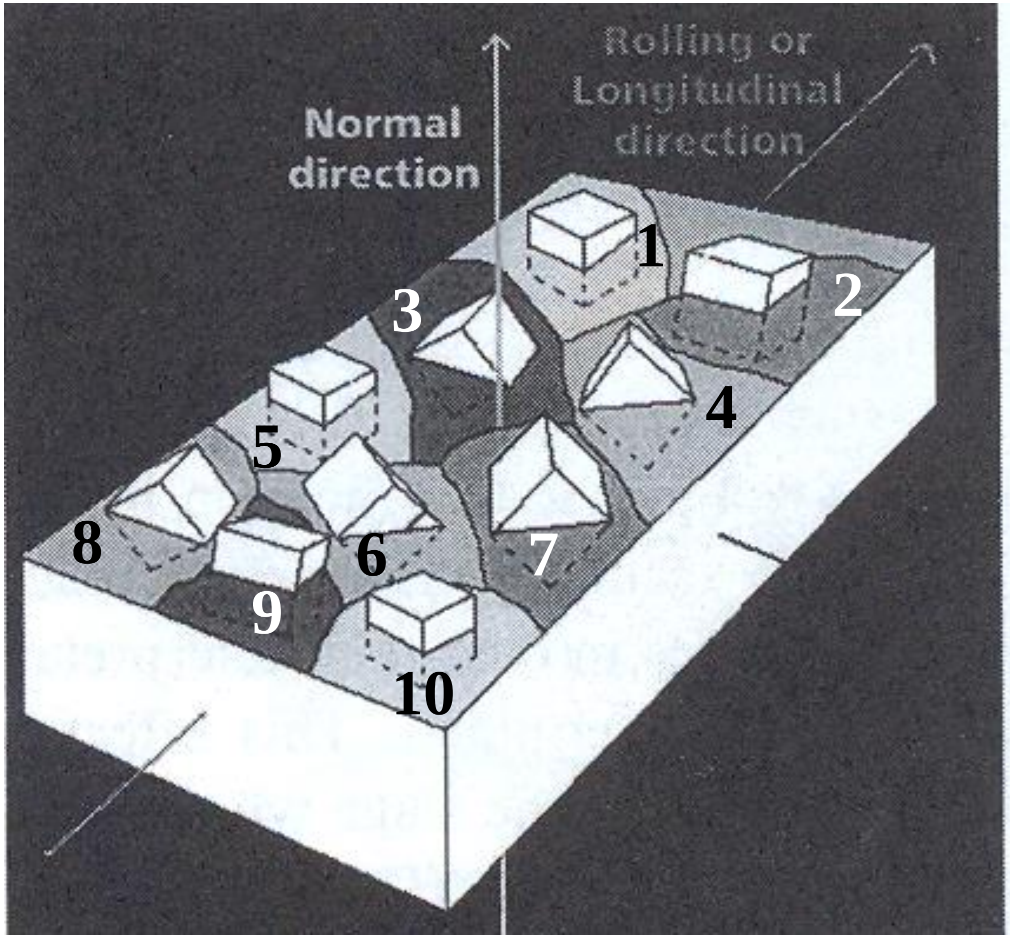
Bulk texture vs. Micro-texture

- **Bulk texture,** also known as macro-texture, provides information about the density and volume fraction of any given orientation that is prevalent in the microstructure and that constitutes the texture. But it does not furnish any detail about the spatial location of those orientations in the microstructure.
- **Bulk texture** is generally represented by pole figures and orientation distribution functions. Bulk textures are generally measured by X-Ray or neutron diffraction.

- **Microtexture** is the microstructure based orientation determination. Its advantage over bulk texture is it provides information on orientations of individual grains and its neighbors.
- **Microtexture** is generally represented by orientation image maps, as obtained by Electron Back-scattered Diffraction (EBSD) technique.

Bulk texture

See the schematic of crystal unit cells embedded in a polycrystalline sample with different orientations, as shown below:

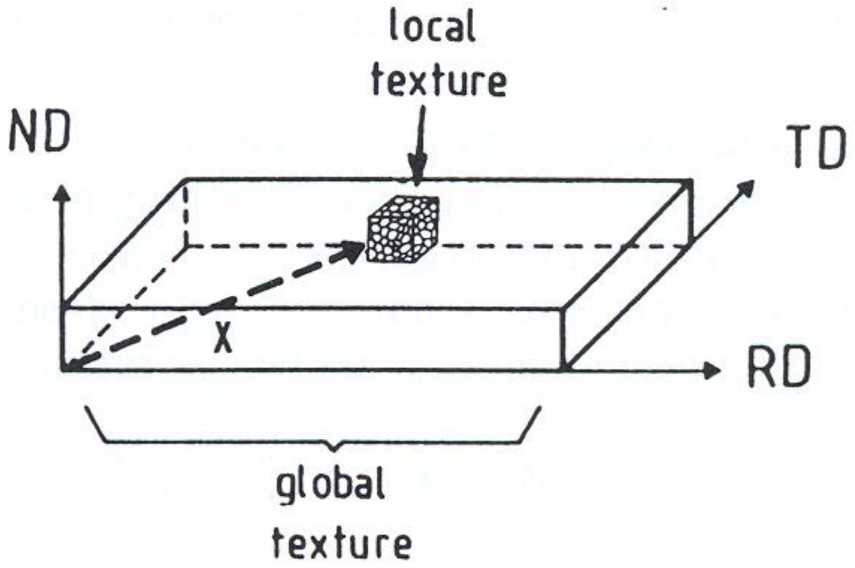


- **Grains 1 and 10 have similar orientation**
- **Grains 2 and 9 have similar orientation**
- **Grains 3 and 8 have similar orientation**

These pair of grains have common orientation but all such grains are spatially apart.

Micro-texture

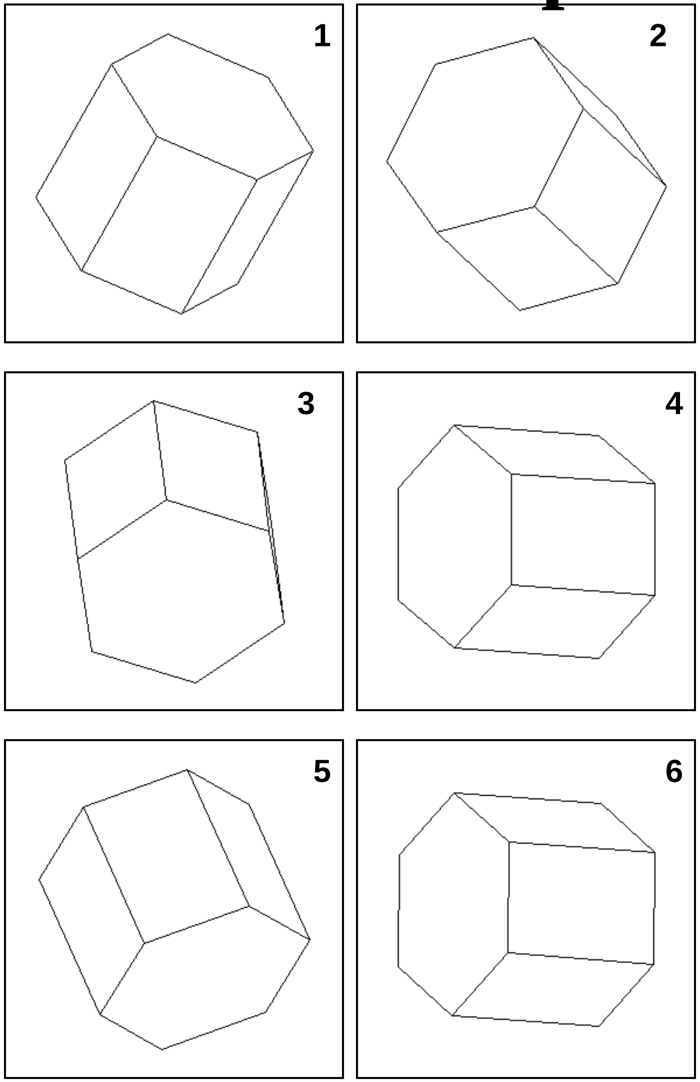
The difference between bulk and local texture

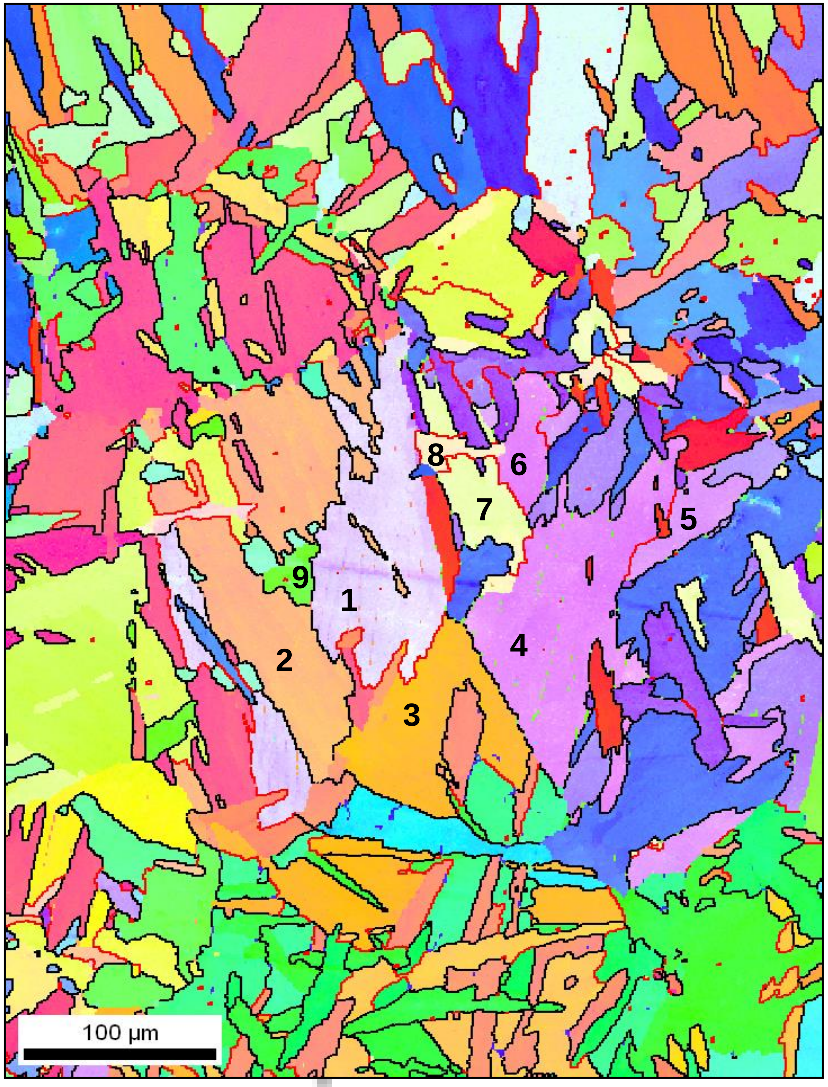


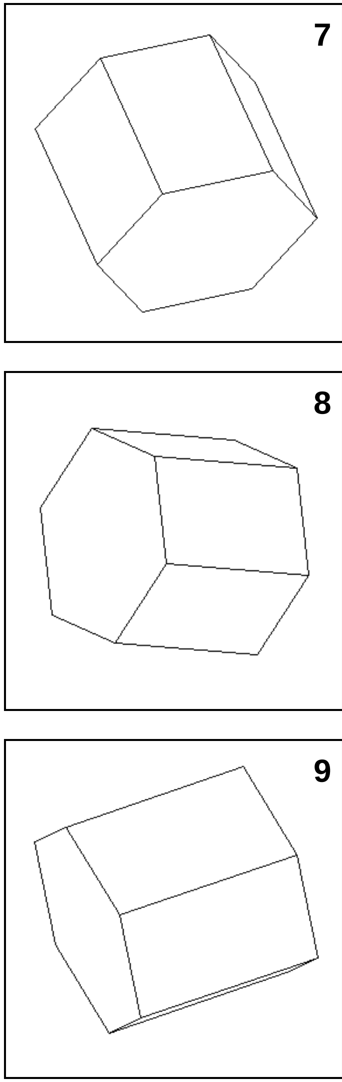
(Courtesy: H.-J. Bunge)

The figure on the next slide shows the orientation image map obtained by EBSD. Individual colors of grains corresponds to different orientations. Orientation of individual unit cell is shown at the bottom of the figure along with the two types (low and high angle) of grain boundaries.

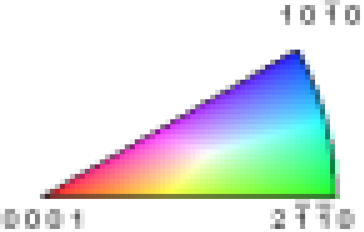
Depiction of Micro-texture







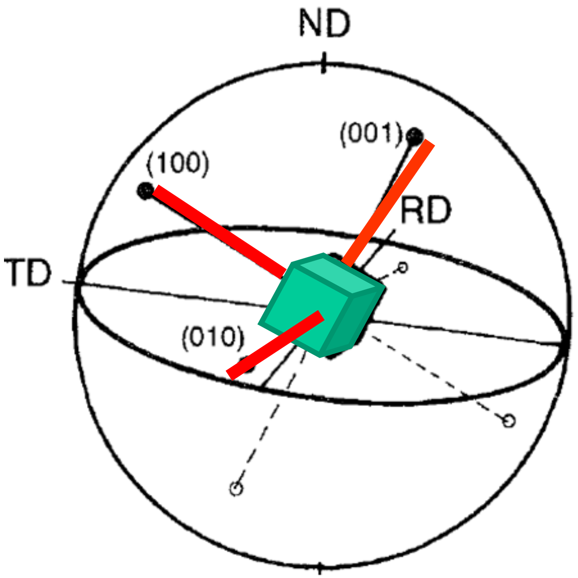
ND Inverse pole figure



Boundaries: Rotation Angle

	Min	Max	Fraction	Number	Length
■	55°	75°	0.423	31638	2.16 cm
🟥	85°	95°	0.173	6085	6.07 mm

X-ray Pole Figures



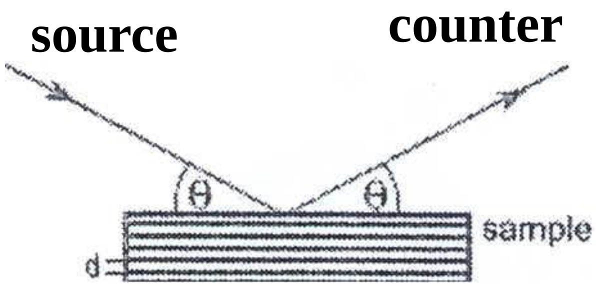
Pole figures are the representation of crystal orientation with respect to sample frame of reference i.e ND, RD, TD.

In the fig on the left it should be imagined that the sample is kept in the center of a reference sphere. Every sample inherently contains three orthogonal axes within itself, namely ND, RD, TD.

In the above fig., the cube at the center is a unit cell embedded in the sample with a certain orientation. The unit cell itself has a distinct orthogonal reference axes. In the case of cubic it is (100), (010), (001).

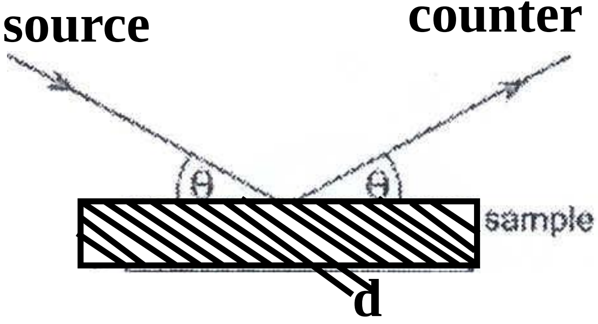
The intersection of the reference sphere and the plane normal is defined as the pole. The pole figure is the stereographic projection of the poles used to represent the orientation of an object in space.

Bragg reflection occurs only when the diffracting planes are parallel to the sample surface, as shown in (a).



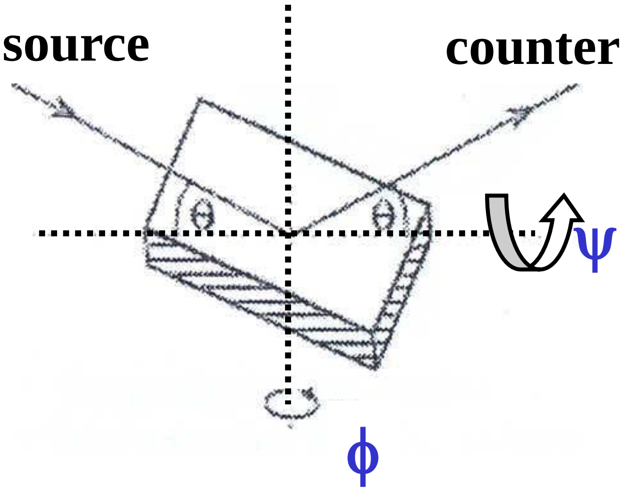
(a) lattice plane (h k l)

reflection



(b) lattice plane (h k l)

No reflection



(c) Rotation of the sample to bring all the crystal planes in diffraction condition

(Figure inspired by: “Introduction to texture and orientation mapping” by V. Randle and O. Engler)

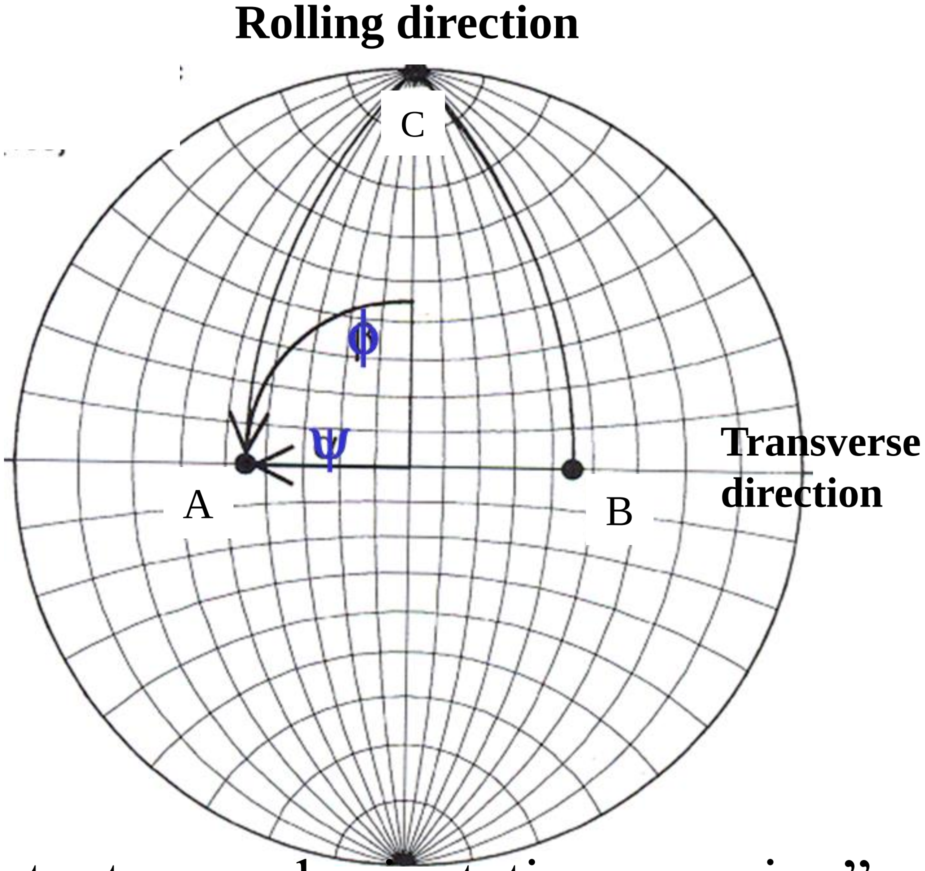
How to determine the orientation of a crystal if its diffracting planes are not parallel to the surface, as shown in (b)?

Rotate the crystal along two normal axes (φ, ψ) so that the planes come under diffraction condition, as shown in (c). Every orientation in space has an unique rotation angles.

Pole figures

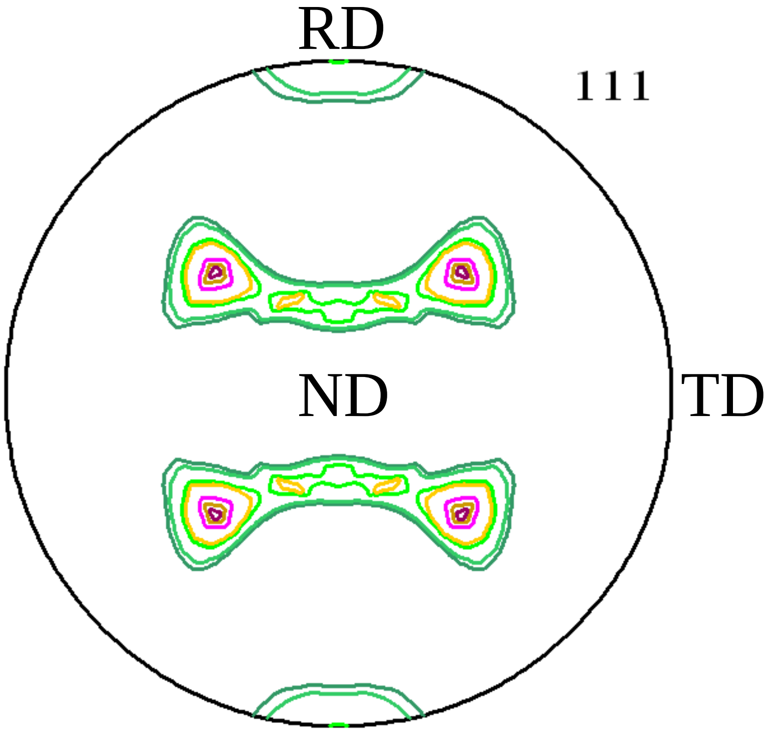
As we have learnt earlier that the pole figures are the stereographic projections of poles of various orientations. To get information from various orientations in a polycrystalline aggregate, the sample have to be rotated/tilted in all possible ways to get the diffraction.

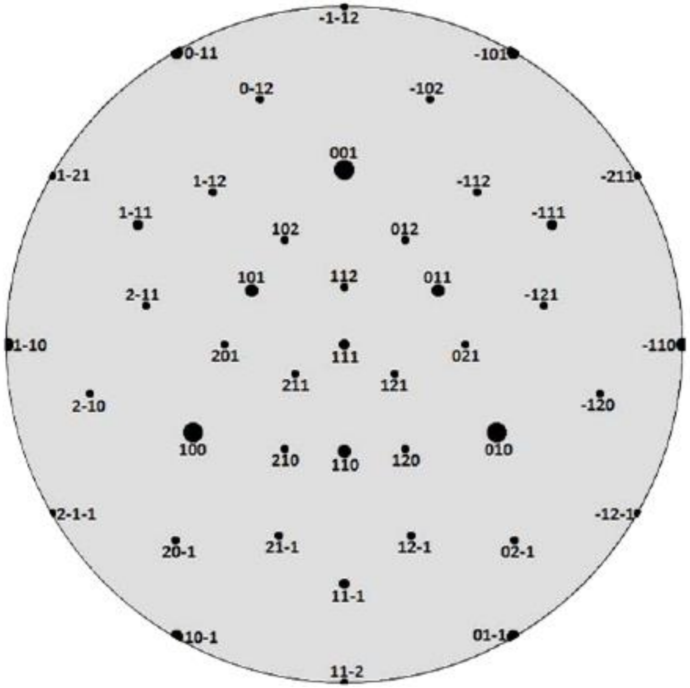
e.g. If we need to get reflection from {110}<001> orientation from 200 planes, it is required to tilt the sample along ψ followed by a rotation along φ. Similarly, all the orientations in a given pole figure can be represented by two angles (φ, ψ).



(Figure inspired by: “Introduction to texture and orientation mapping” by V. Randle and O. Engler)

How to correlate pole figures with stereographic projection?





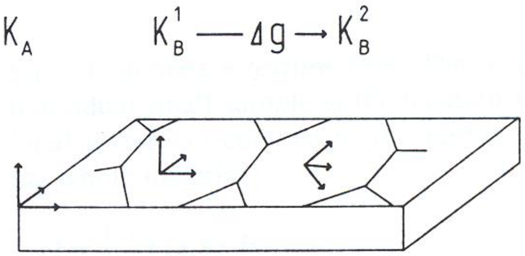
The figure on the left is the 111 pole figure of an annealed aluminum sample. The figure on the right is the standard 111 stereogram for cubic crystal symmetry. Any orientation X in a pole figure can be found as follows: Superimpose the standard stereogram on the experimental pole figure and match the 111 poles of stereogram with that of pole figures.

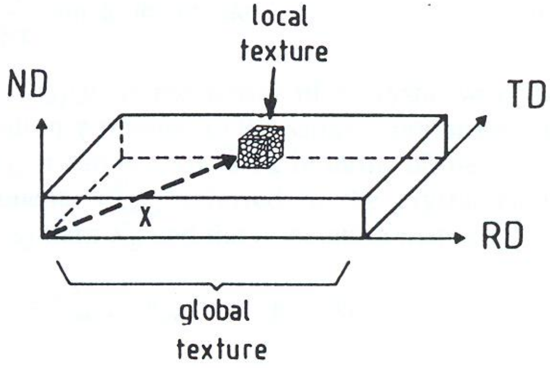
Definition of Micro-texture

Texture may be different in different volume elements of a material. In that case, one has to distinguish:

micro or local textures $f(g, r) = f(\phi_1, \varphi, \phi_2, x, y, z)$

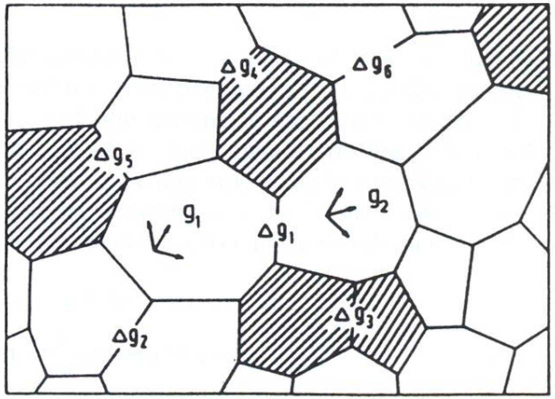
from the global texture of the whole material.



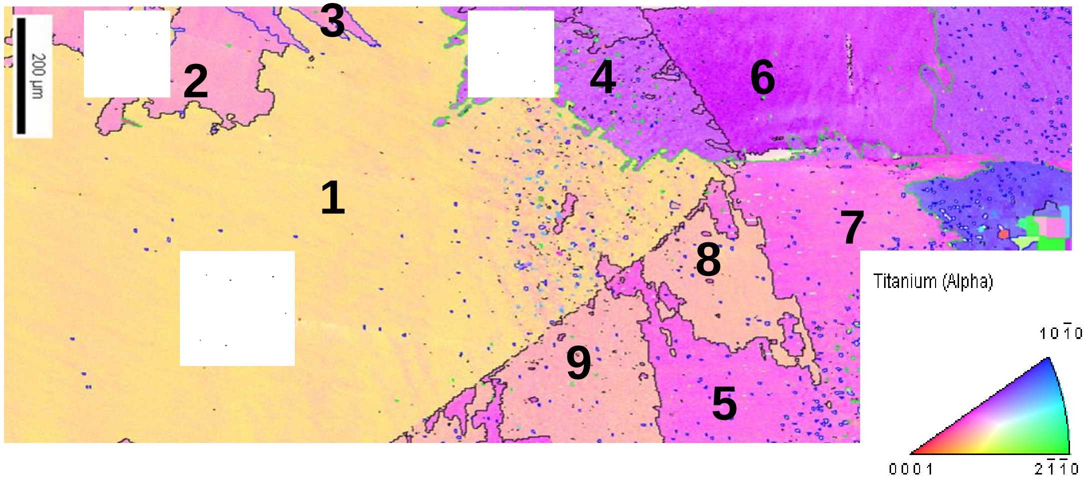


K_A= sample coordinate
K_B= crystal coordinate
g = orientation of the crystallite with respect to K_A

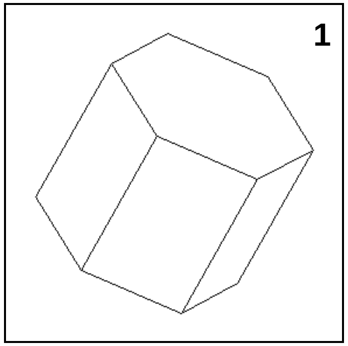
K_B= crystal coordinate for crystal B^1
K_B= crystal coordinate for crystal B^2
Δg = mis-orientation of the crystallite B^1 with respect to B^2

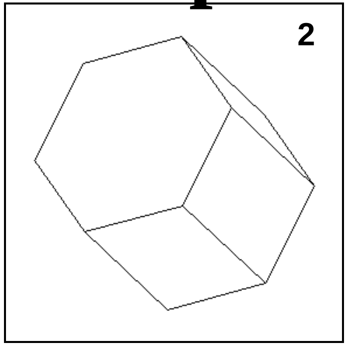


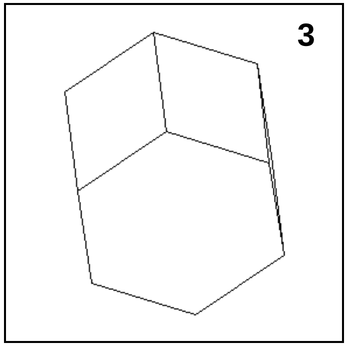
Orientation and misorientation of grains of different phases (different crystal structure) (Courtesy: H.-J. Bunge)

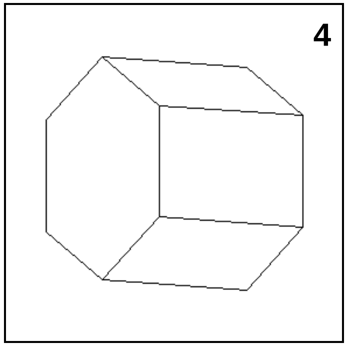


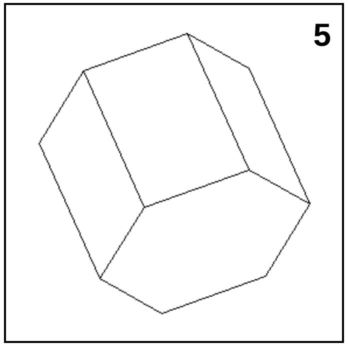
Depiction of Micro-texture

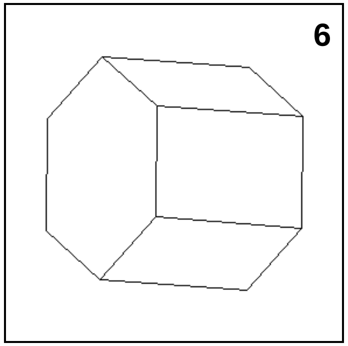


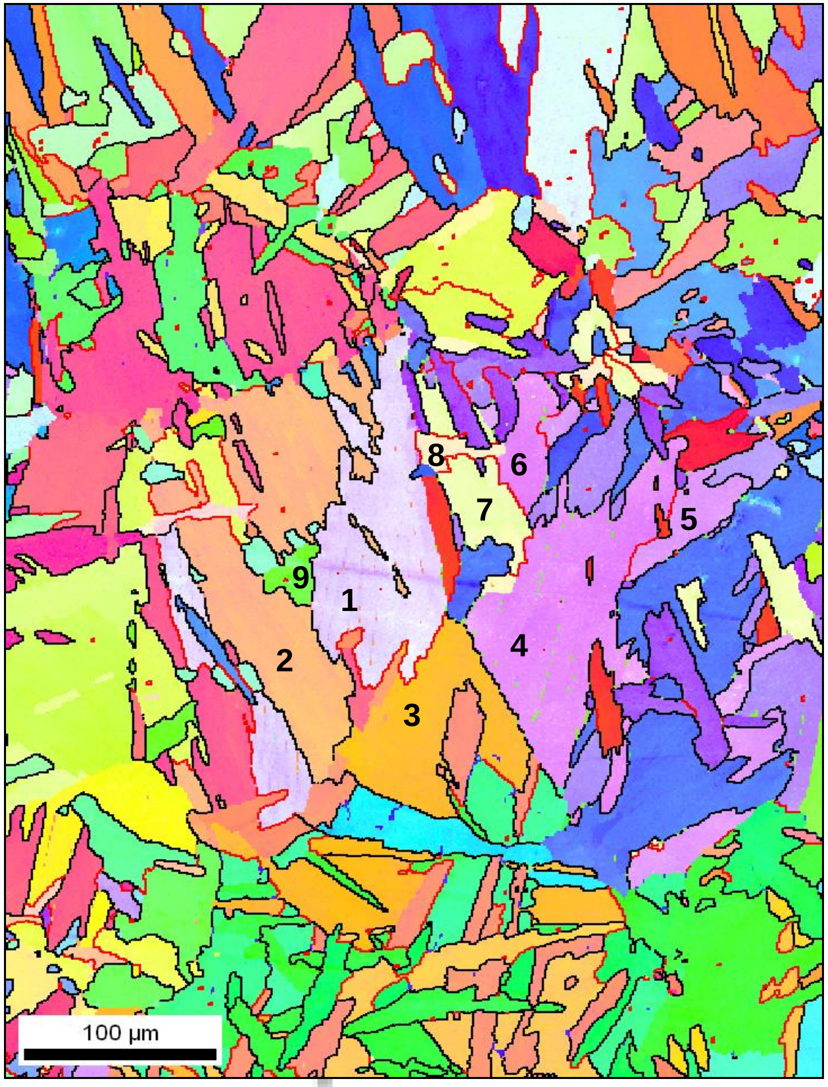


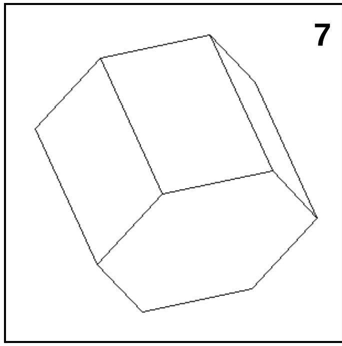


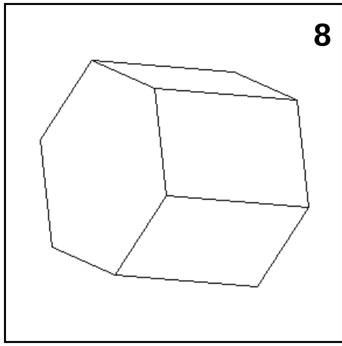


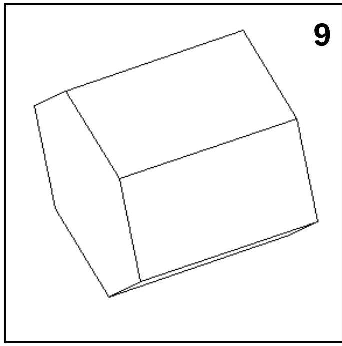




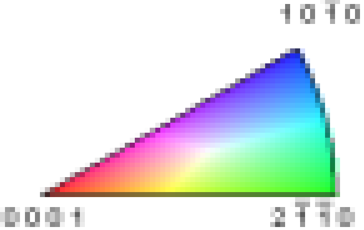








ND Inverse pole figure



Boundaries: Rotation Angle

	Min	Max	Fraction	Number	Length
■	55°	75°	0.423	21838	2.16 cm
🟥	85°	95°	0.173	6085	6.07 mm

Texture Measurement

- **X-ray diffraction method** **-most common method**
- **Neutron diffraction method** **-used, where X-ray methods fail**
- **Electron diffraction methods** **-SEM (EBSD) and TEM (OIM)**
- **Synchrotron X-ray method** **-very specialised, quite powerful**
- **Ultrasonic methods** **-non-destructive method**

How to Measure Texture

- X-ray diffraction; pole figures; measures *average* texture at a surface (*µms* penetration); projection (*2 angles*).

- Neutron diffraction; type of data depends on neutron source; measures *average* texture in bulk (*cms* penetration in most materials) ; projection (*2 angles*).

- Electron [back scatter] diffraction; easiest [to automate] in scanning electron microscopy (SEM); *local* surface texture (*nms* penetration in most materials); complete orientation (*3 angles*).

- Optical microscopy: optical activity (plane of polarization); limited information (*one angle*).

Virtually all techniques for texture analysis are based upon diffraction of radiation by the crystal lattice: X-ray, electrons, neutrons.

	light	neutrons	X-rays	electrons
wavelength [nm]	400-700	0.05-0.3	0.05-0.3	0.001-0.01
energy [eV]	1	10^{-2}	10^{4}	10^{5}
charge[C]	0	0	0	-1.602×10^{-19}
rest mass [g]	0	1.67×10^{-24}	0	9.11×10^{-28}
penetration depth, absorption length [mm]	-	10-100	0.01-0.1	10^{-3}

Particle beams (electrons, neutrons): de Broglie relation: $\lambda = h/mv$

or, $\lambda = h/\sqrt{2mE_{kin}}$

- **Interaction of radiation with lattice atoms and penetration depth / absorption length ultimately decides whether a particular radiation will be giving information about microtexture or macrotexture.**

•electrons	**nuclei + shell electrons**	**microtexture**
•X-ray	**shell electrons**	**macrotexture**
•neutrons	**nuclei**	**macrotexture**

All diffraction methods use

- A monochromatic beam of radiation with the Bragg condition fixed for a single set of reflecting planes – which is maintained throughout the determination of a particular pole figure.

↓

- The normal K of the diffracting planes (hkl) remains fixed in space and the specimen is rotated through a wide range of angles

↓

- Many different orientations in the sample are brought into coincidence with the diffracting normal

- Total diffracted intensity *(at any instant)*
  $\propto$ volume of the specimen in which the (hkl) planes are so-oriented

- **All diffraction methods use a monochromatic beam of radiation with the Bragg condition fixed**
- **For a single set of reflecting planes – maintained throughout the determination of a particular pole figure - the normal K of the diffracting planes (hkl) remains fixed in space and the specimen is rotated through a wide range of angles. This brings many different orientations in the sample into coincidence with the diffracting normal**

Total diffracted intensity *(at any instant)*

$\propto$ volume of the specimen in which the (hkl) planes are so-oriented

X-ray Pole Figures

- **X-ray pole figures are the most common source of texture information; easiest to measure.**

- **Pole figure represents variation in diffracted intensity with respect to direction in the specimen.**

- **Pole figures are represented by mapping *of crystal directions w.r.t. sample reference frame.***

Pole figures: Historical perspective

1924: Wever (Berlin) generated 1st pole figure from evaluating the inhomogeneous intensity distribution along the Debye-Scherrer rings.

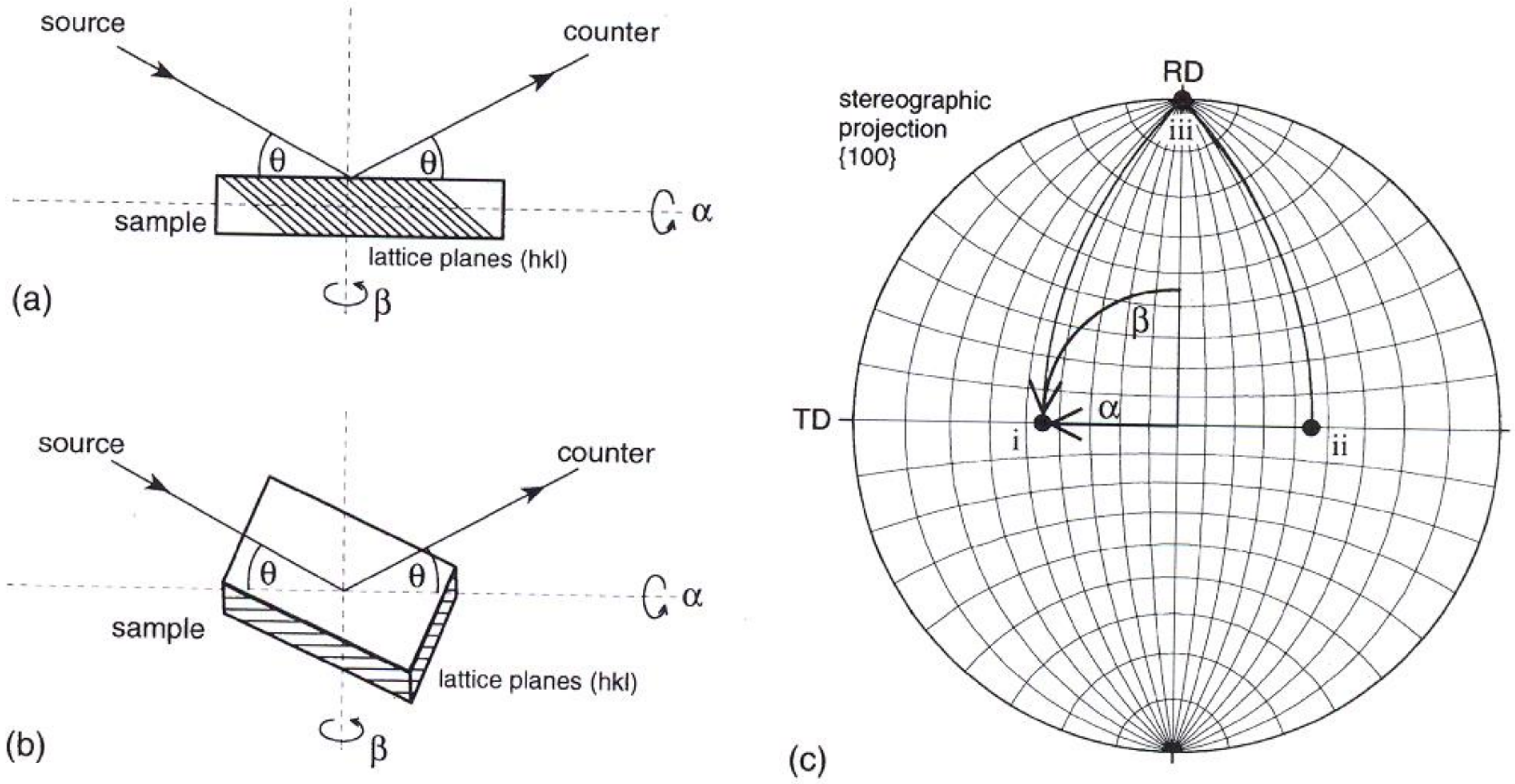
1948: Introduction of texture goniometer and Geiger counters (Decker *et al.*, Norton).

1949: Schulz initiated modern quantitative X-ray texture analysis.

Since 1960s: Pole figure determination by neutron diffraction.

Today, Automated computer-controlled systems are available and the corresponding measuring techniques and the necessary correction methods are well-established.

Principle of pole figure measurement



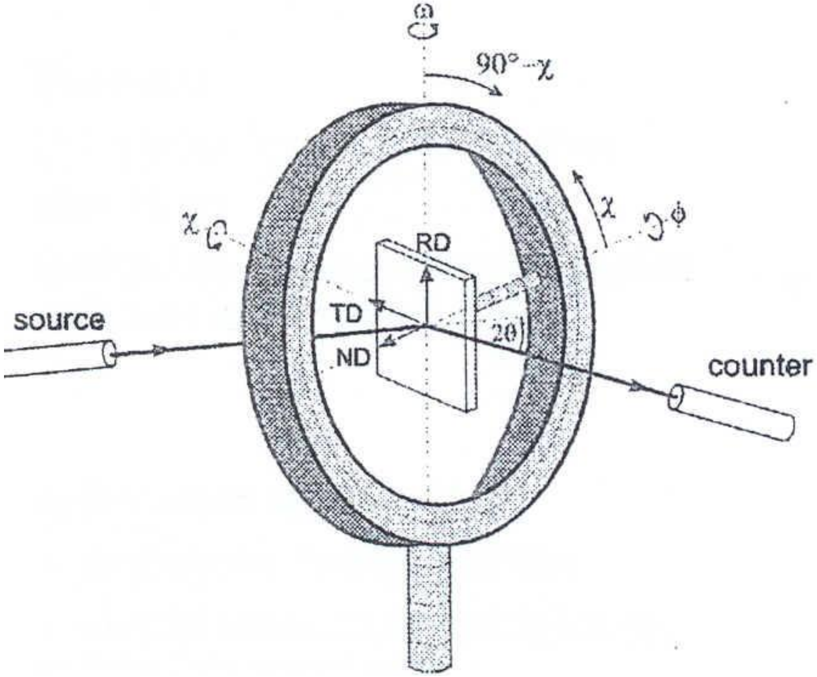
Sketch to illustrate the effect of sample rotation of the arrangement of the lattice planes. (a) untilted position ($\alpha = \beta = 0°$); (b) sample tilted such that the lattice planes are in Bragg condition ($\alpha > 0°$, $\beta > 0°$); (c) reflection peaks of a Goss-$\{110\} < 001 >$ oriented crystal in the stereographic projection at (i) $\alpha = 35°$, $\beta = 90°$, (ii) $\alpha = 35°$, $\beta = 270°$, (iii) $\alpha = 90°$, $\beta = 0°$.

Texture Goniometer

Goniometer for texture analysis:

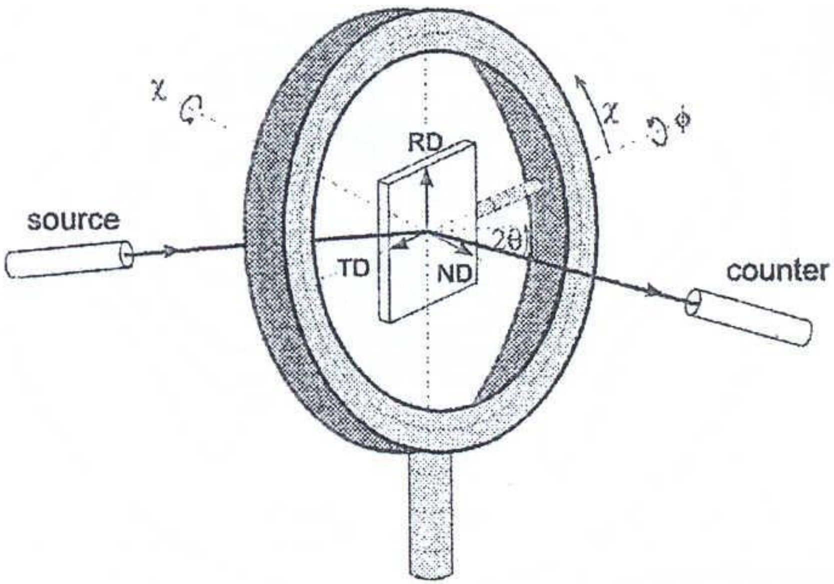
4-circle spectrometer

4 angles: θ,ϕ, ξ, ω ⟺ pole figure angles θ,α, β



Reflection geometry

Thick samples (X-ray)

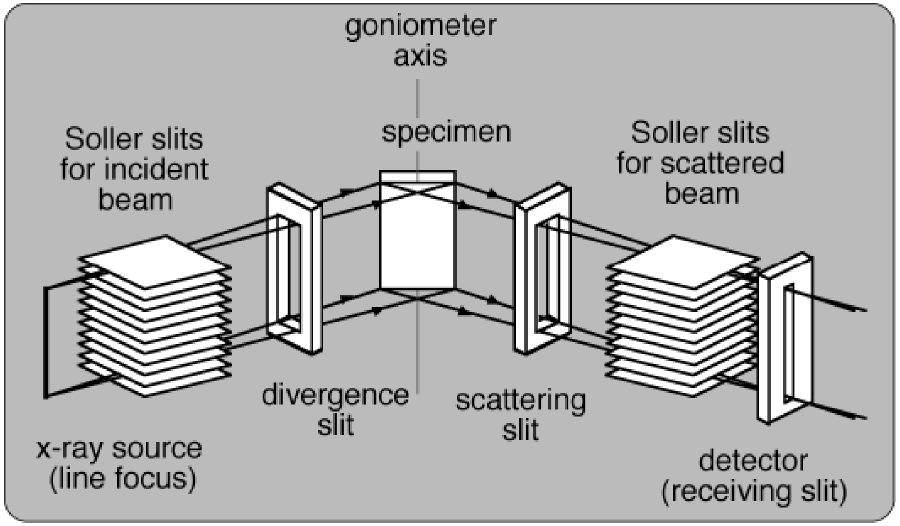


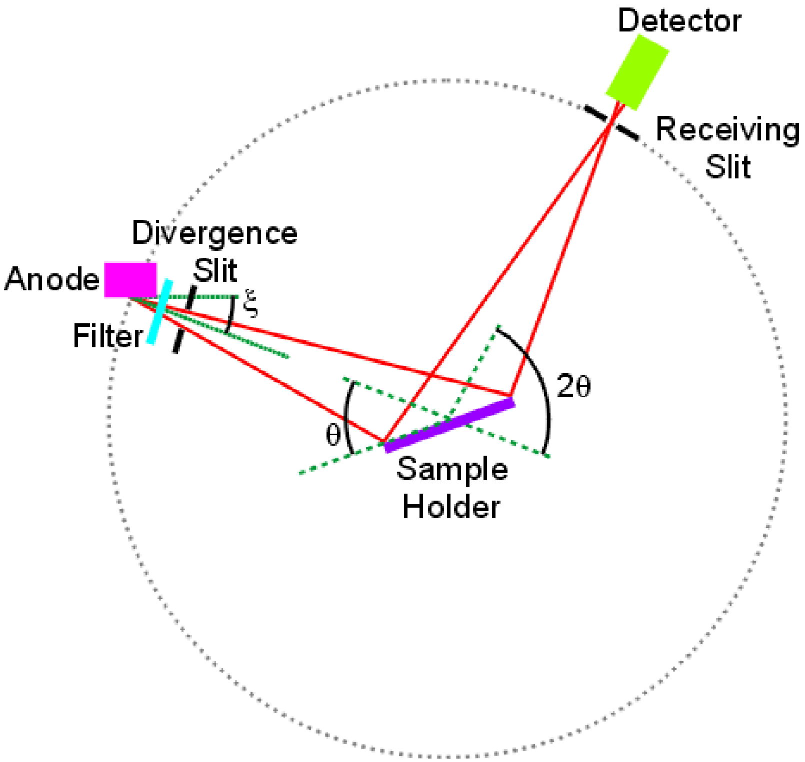
Transmission geometry

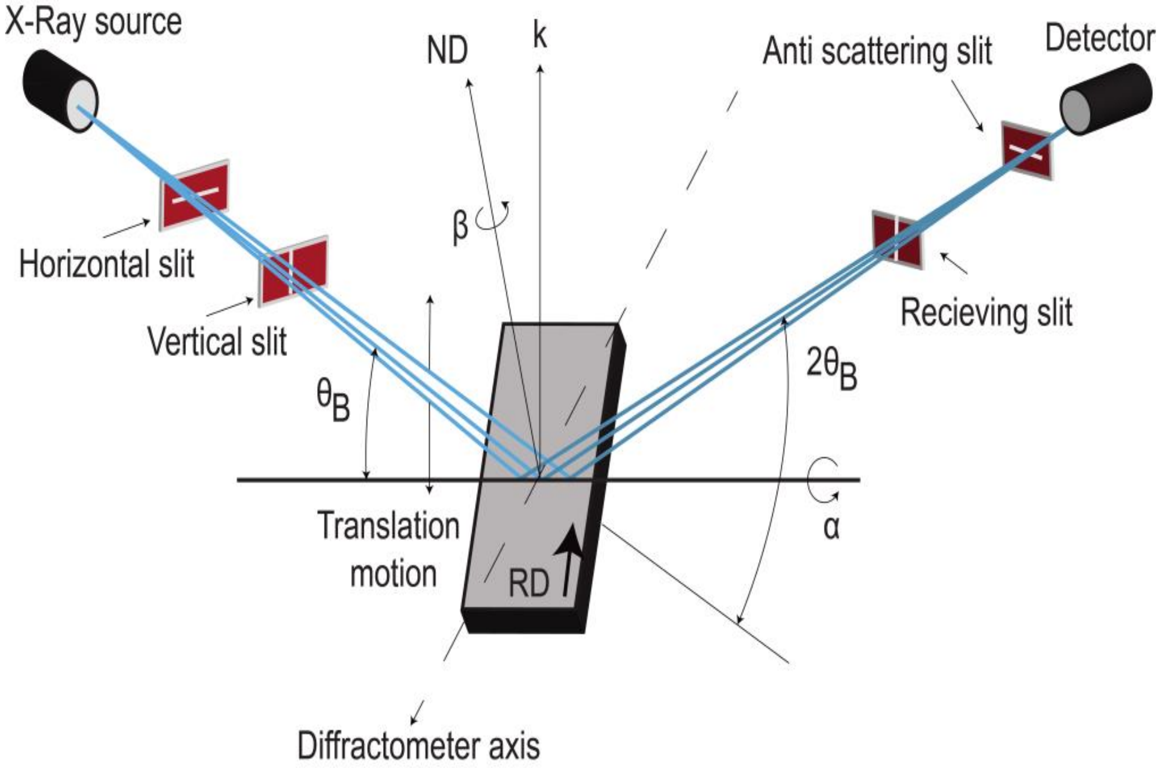
Thin samples (X-rays, neutrons)

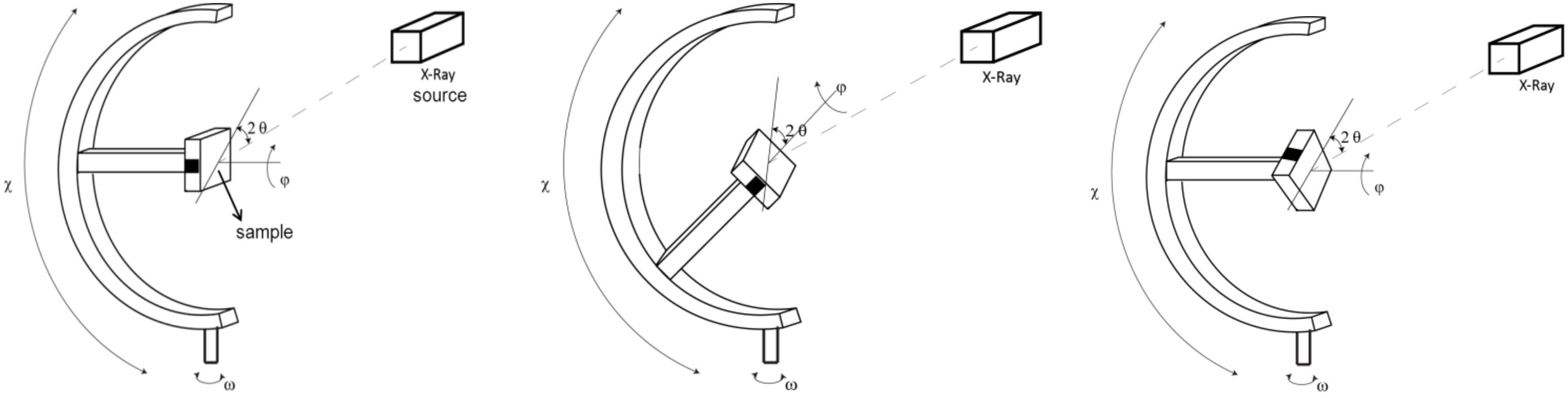
Bragg-Brentano Geometry

- Eulerian cradle is set in such a way that *the ϕ circle coincides with the bisector of the angle between incident and diffracted beam*, and any direction on the pole figure can be brought parallel to this direction by the two rotations 90-ϕ and ψ corresponding to the pole figure coordinates α and β.





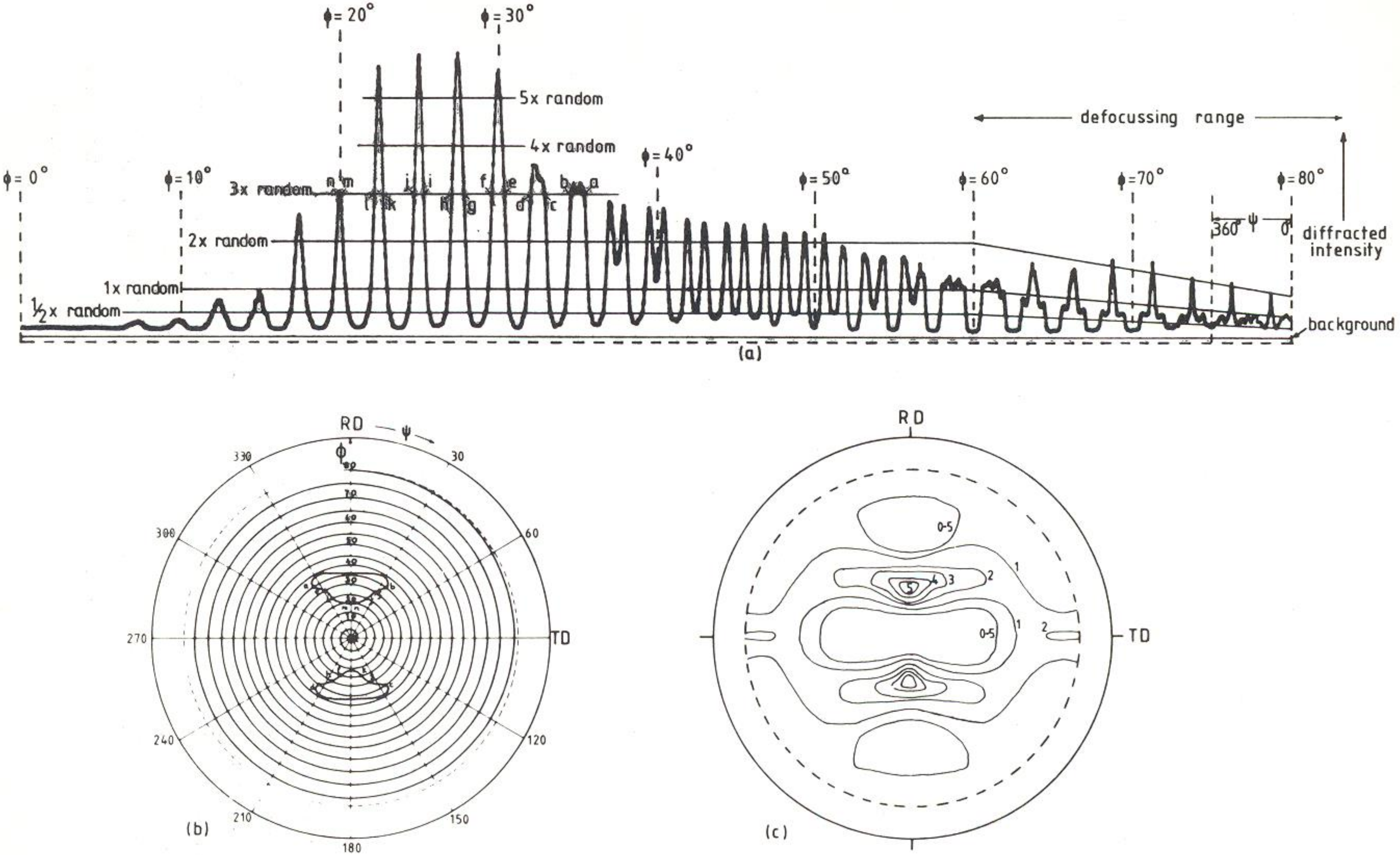

X-Ray source
ND
k
Anti scattering slit
Detector
Horizontal slit
β
Vertical slit
Recieving slit
θB
2θB
Translation motion
α
RD
Diffractometer axis

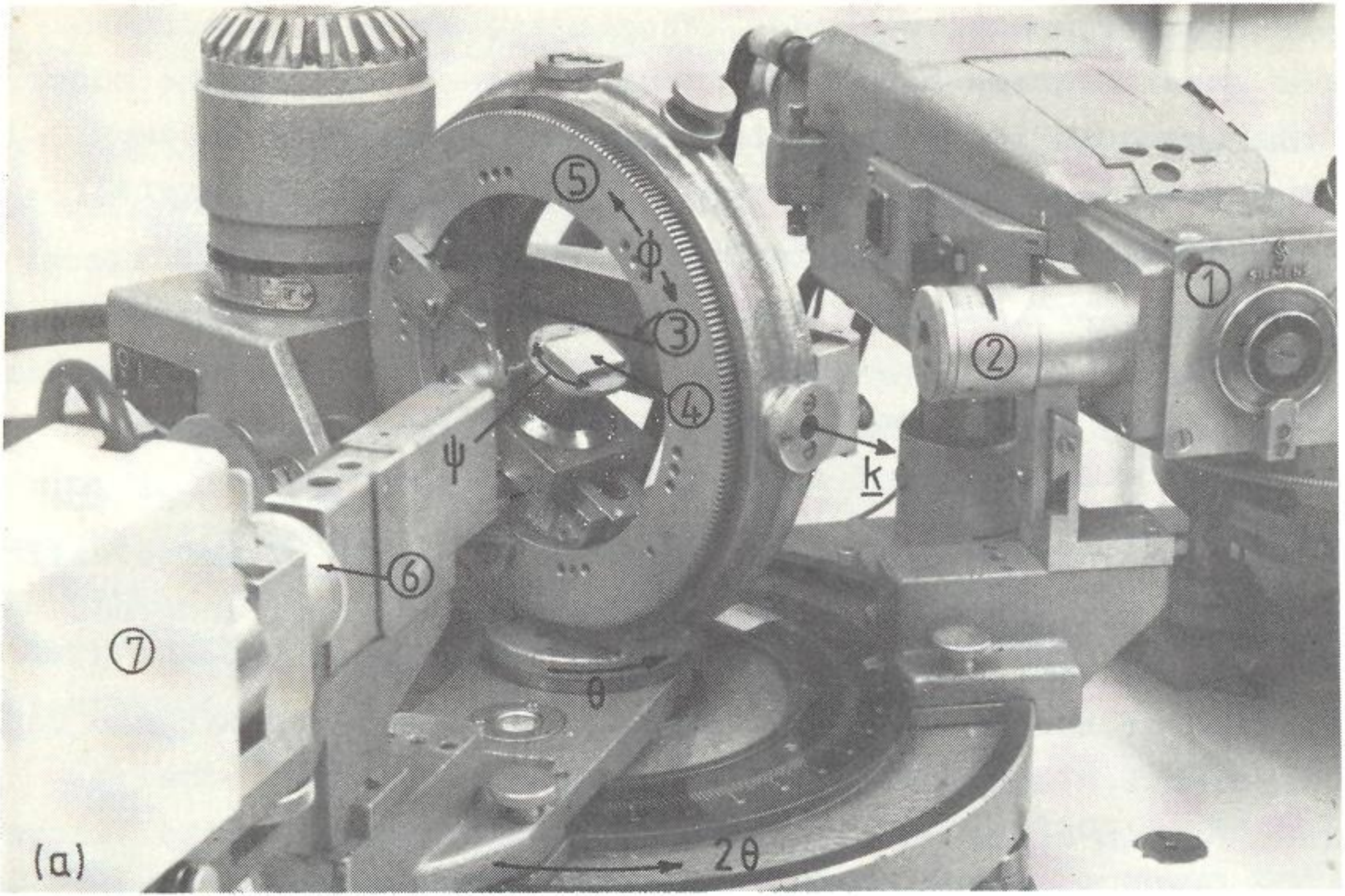

X-Ray source
2 θ
φ
χ
sample
ω
X-Ray
φ
2 θ
χ
ω
X-Ray
2 θ
φ
χ
ω

Schultz reflection method

- **PF measured with 5-axis goniometer.**
- **2 axes used to set Bragg angle (choose a specific crystallographic plane with θ/2θ).**
- **Third axis tilts specimen plane w.r.t. the focusing plane.**
  □rotation about an axis parallel to the sheet surface (angle ψ)
- **Fourth axis spins the specimen about its normal.**
  □rotation about an orthogonal axis through angle ϕ.
- **Fifth axis oscillates the Specimen under the beam.**
  □simple translation – to and fro – improves the statistical averaging of the texture measurement by increasing the number of grains that are sampled
- **Deviations of relative intensities in a θ/2θ scan from powder file indicate texture.**

□X-ray beam must not be transmitted through the specimen ⇨ ***thickness>~0.2 mm***


ϕ = 0°
ϕ = 10°
ϕ = 20°
ϕ = 30°
ϕ = 40°
ϕ = 50°
ϕ = 60°
ϕ = 70°
ϕ = 80°
5x random
4x random
3x random
2x random
1x random
½x random
n m
l k
j i
h g
f e
d
c
b a
defocussing range
360°
ψ
0°
diffracted intensity
background
(a)
RD
ψ
ϕ
330
30
300
60
270
TD
240
120
210
150
180
90
70
60
50
40
30
20
10
(b)
RD
TD
0-5
1
2
3
4
5
0-5
1
2
(c)

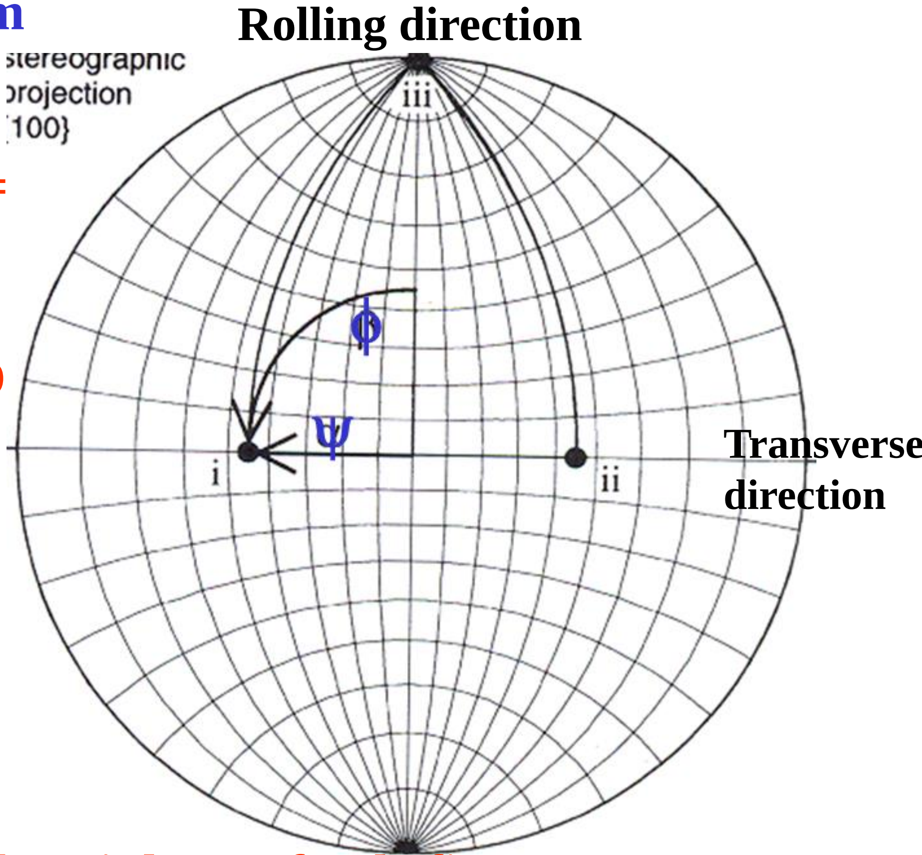


1. Source; 2. Divergence slit; 3. Narrow horizontal slit;
4. Specimen; 5. Major circle of the goniometer; 6. Receiving slit;
7. Counter.

- 1st situation: Specimen is arranged with RD pointing vertically -sheet normal bisects angle between incident and diffracted beams

➡ ND coincides with diffracting vector K

In this case, measured intensity comes from the (hkl) planes || sheet plane



- 2nd situation: Rotate the specimen by $\psi = 90^{o}$ (along Euler Cradle)
  - RD || K

➡ measured intensity corresponds to RD

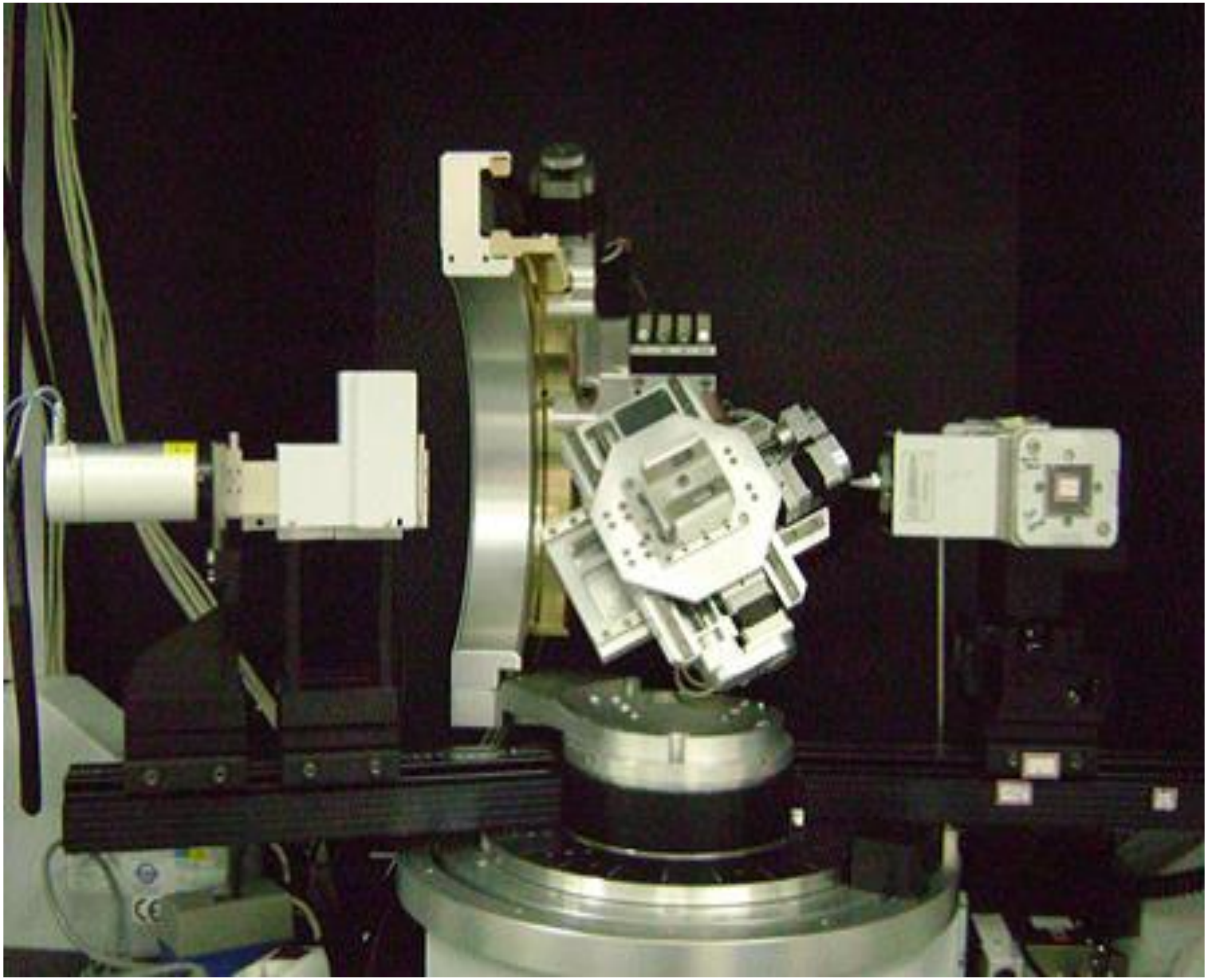
This type of rotation gives radial scan of the pole figure.

- 3rd situation: Rotate specimen by $\phi = 90^{o}$ (about axis perpendicular to specimen surface)
  - TD || K

➡ diffracted intesity is sampled around periphery of pole figure

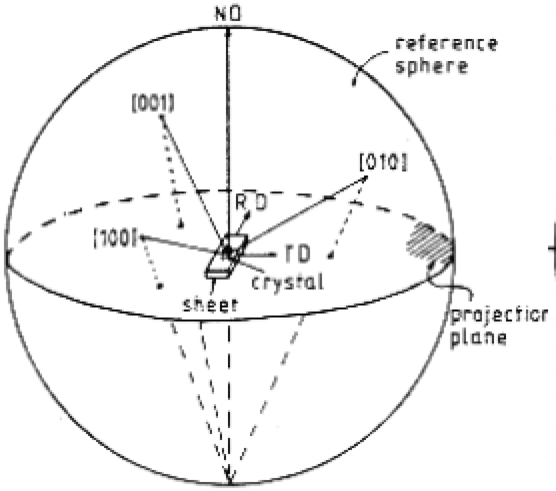
- A fast rotation through ψ + a slow rotation through ϕ = diffracted intensity along the spiral trace

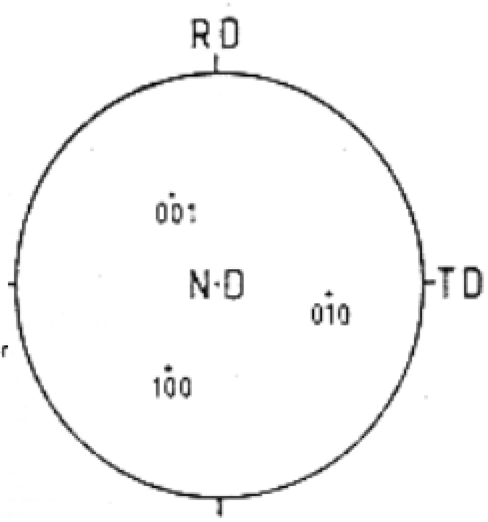
➡ total intensity to the counter

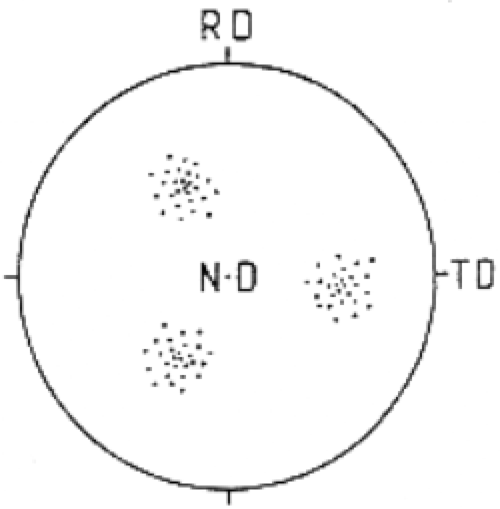


Texture goniometer at IISc Bangalore

- If the goniometer is set for {100} reflections, then all directions in the sample that are parallel to <100> directions will exhibit diffraction.







Practical Aspects

- Typical to measure three PFs for the 3 lowest values of Miller indices.

- ***Why?***

– A single PF does not uniquely determine orientation(s), texture components because only the plane normal is measured, but not directions in the plane (2 out of 3 parameters).

– Multiple PFs required for calculation of Orientation Distribution.

Limitation of Schultz method

- Defocussing error
  - Narrow divergence slit – affects total count rate and statistial significance of the measurement
  - Wide receiving slit- reduces peak to background ratio
  - Larger Bragg angle by changing to a lower wavelength- severe reduction in count rate
- Difficulties in preparing random sample

Defocussing

- **The combination of the θ-2θ setting and the tilt of the specimen face out of the focusing plane spreads out the beam on the specimen surface.**

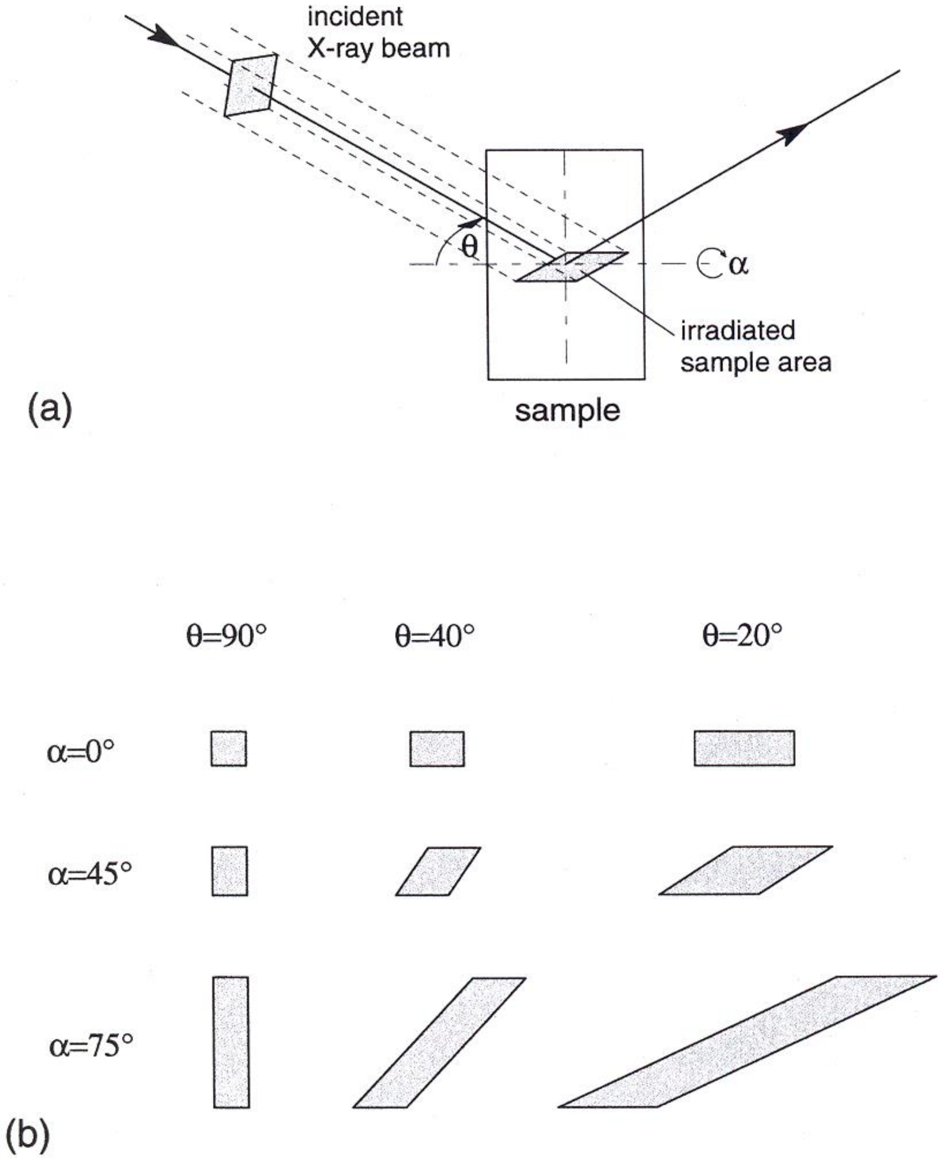
- **Above a certain spread, not all the diffracted beam enters the detector.**

- **Therefore, at large tilt angles, the intensity decreases *for purely geometrical reasons*.**

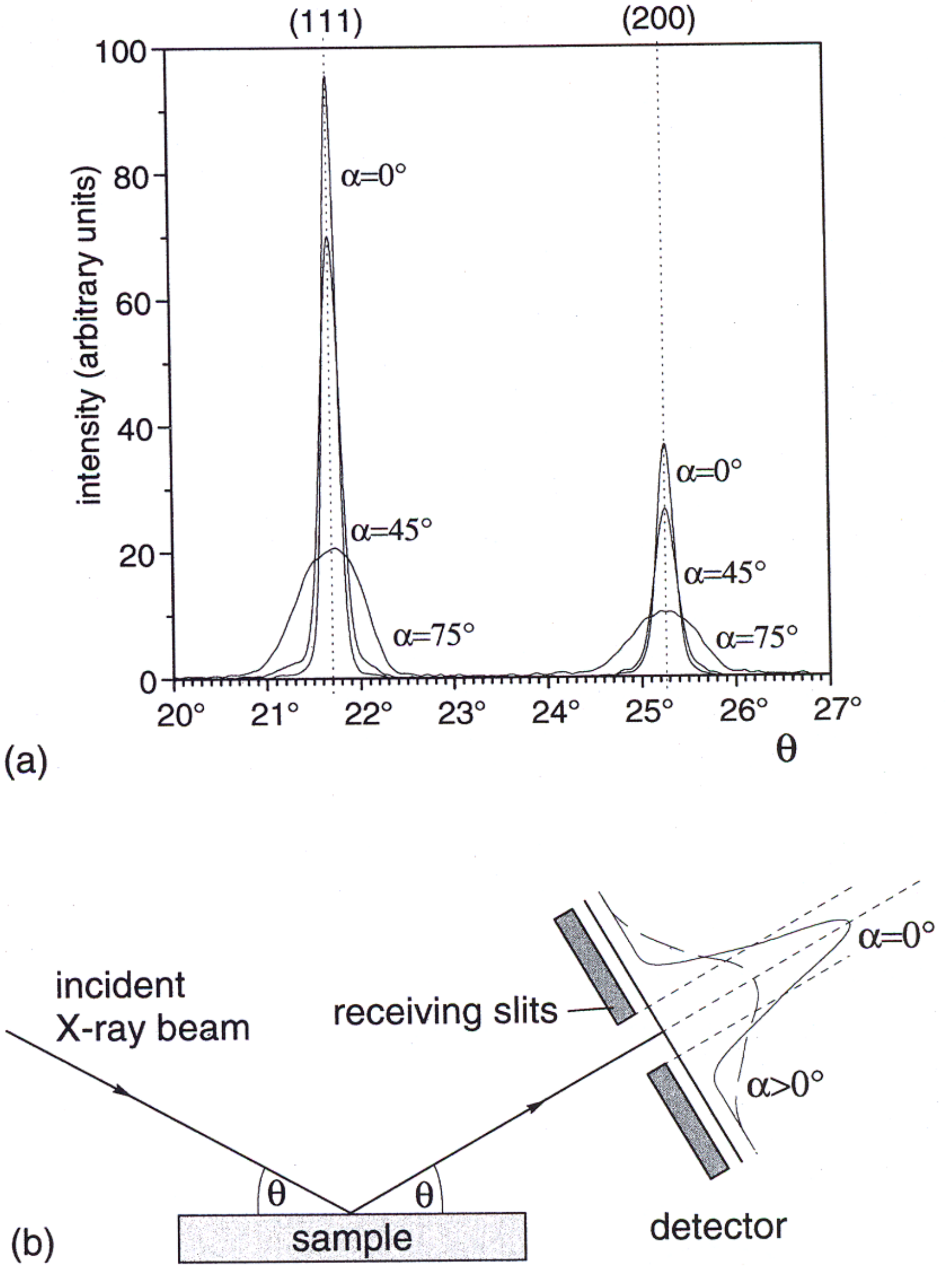
- **This loss of intensity must be compensated for, using the *defocussing correction*.**

Defocusing Correction

- **Defocusing correction more important with decreasing *2θ* and narrower receiving slit.**



(a) Sketch to visualise the distortion of the irradiated spot on the sample surface; (b) Shape of the irradiated spot in dependence on tilting angle α and Bragg angle θ for a square-shaped incident beam.



(a) Peak broadening with increasing tilting angle α (copper powder sample, Cu$K\alpha$ radiatic
(b) Sketch to visualise the defocusing error.

Corrections to Measured Data

- **Random texture [=uniform dispersion of orientations] means *same* intensity in all directions.**
- **Background count must be subtracted.**

$$I_{corr} = I_{meas}(\alpha,\beta) - BG(\alpha)$$

- **X-ray beam becomes defocused at large tilt angles (>~60°); measured intensity from random sample *decreases* towards edge of PF.**
- **Defocusing correction required to *increase* the intensity towards the edge of the PF.**

$$I_{corr} = \underline{I_{meas}(\alpha,\beta) - BG(\alpha)}$$

$$U(\alpha)$$

- **Absorption**

$$I(t)/I\propto = 1- \exp(-2\mu t / \sin\theta \cos\alpha)$$

- **Couting Statistics**

Pole Figure Nomalization

Diffraction: intensities in the form of counts (or counts per second;cps)

Pole figure: multiples of a random sample (MRD)

$$I_{norm}(\alpha, \beta) = (1/N).\ I_{corr}(\alpha, \beta)$$

- **Normalization is the operation that ensures that “random” is equivalent to an intensity of one.**

- **This is achieved by integrating the un-normalized intensity, $f(\theta,\psi)$, over the full area of the pole figure, and dividing each value by the result, taking account of the solid area.**

Thus, the normalized intensity, *f*(θ,ψ), must satisfy the following equation, where the 2π accounts for the area of a hemisphere:

$$\frac{1}{2\pi}\int f(\Theta,\psi)\sin\Theta\, d\Theta\, d\psi = 1$$

- **Integral over entire pole figure: 1/intensity of a random sample: 1**
- **pole figure regions >1: more poles than in a random sample .**
- **pole figure regions <1: less poles than in a random sample.**

incomplete pole figures (pseudo-normalisation):

$$N' = \frac{\sum_{\alpha_{\min}}^{\alpha_{\max}}\sum_{\beta=0}^{360} I_{corr}(\alpha,\beta)\cdot\left(\cos\left(\alpha-\frac{\Delta\alpha}{2}\right)-\cos\left(\alpha+\frac{\Delta\alpha}{2}\right)\right)\cdot\Delta\beta}{2\pi\cdot\left(\cos\left(\alpha_{\min}-\frac{\Delta\alpha}{2}\right)-\cos\left(\alpha_{\max}+\frac{\Delta\alpha}{2}\right)\right)}$$

(or ODF-analysis)

Three-Dimensional Texture Analysis

- **Since orientation of a crystallite (which is three dimensional) in a three-dimensional body, minimum three (3) parameters are needed to describe the orientation (of a crystal relative to the embedding body or its environment).**

- **So far discussed method of texture representation is by pole figure method, which involves description of the loacation of a pole using two angles.**

- **Is this description enough?**

- **Description of textures by pole figures is incomplete, as it refers only to the statistical distribution of a single direction.**

- Description of textures by pole figures is incomplete, as it refers only to the statistical distribution of a single direction.

- See the figure. Here the orientation of the (0001) plane has been depicted on a reference direction with respect to an external frame of reference.

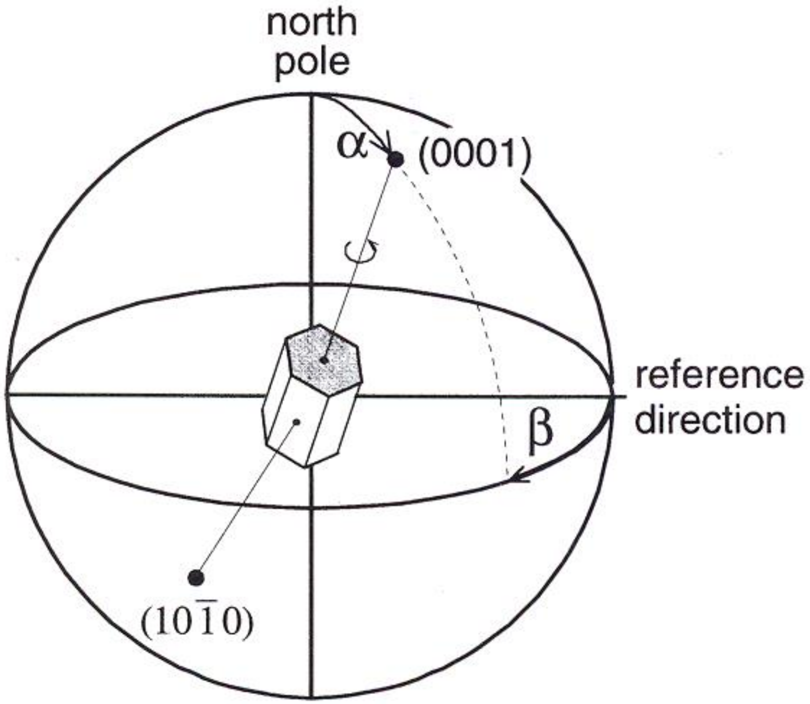


Figure 2.5 Orientation of the basal plane (0001) in a hexagonal crystal. The position of the (0001) pole on the unit sphere with regard to an external reference frame is described by the two angles α and β. However, since the crystal can still rotate about the (0001) pole, for an unequivocal definition of the orientation of the crystal more information, here the position of the $(10\bar{1}0)$ pole, is required.

- There is no way of using it to obtain the complete orientation of individual grains or volume elements.

- **There is another limitation also. Two poles that belong to completely different crystallites can be as closely spaced as they become almost indistinguishable in the pole figure.**

- **All these indicate the limitation of projection based techniques of orientation representation.**

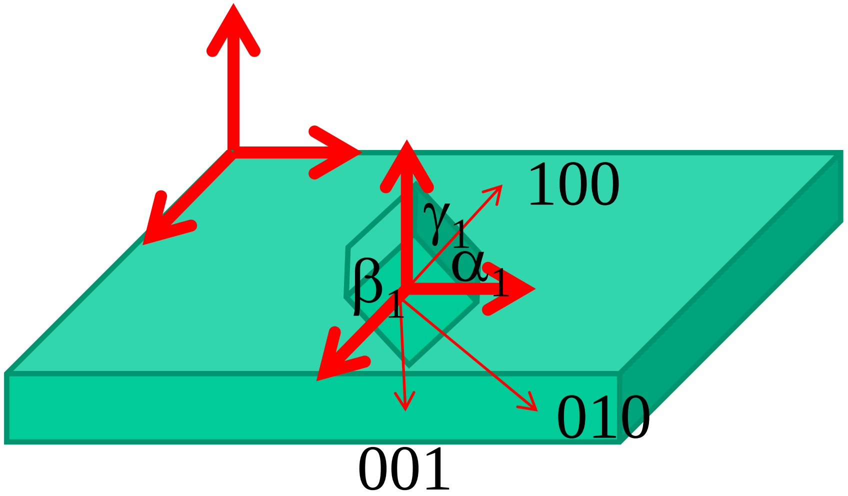
- **It is, therefore, worthwhile to look for direct description of orientation**

Direct Description of orientation

To specify an orientation:

- **The first step is to set up coordinate systems of the specimen and the crystal**
- **The second step is to find a relation between them.**
- **Expressing these relations require:**
  - **both the system should (preferably) be Cartesian and right handed**

Relation between the crystal and specimen coordinate systems



- **The most important step to describe the crystal orientation is to define the reference systems or coordinate systems for the crystallite and the physical body (specimen)**

- **We need two co-ordinate systems simultaneously**
  - **One required to define whole specimen (Specimen co-ordinate system ‘S’)**
  - **Other required to relate crystal (Crystal co-ordinate system ‘C’)**

Specimen Coordinate System

- For specimen coordinate system, the axes are chosen according to important surfaces or directions associated with deformation geometry or external form or shape of the specimen
- **One of the most common reference systems that relates to the geometry of the rolled samples, hence direction associated with the external geometry is defined by:**

Direction normal to rolling plane (ND)

Rolling Direction (RD)

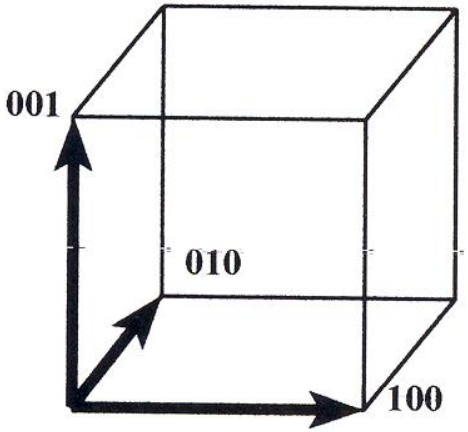
Transverse Direction (TD)

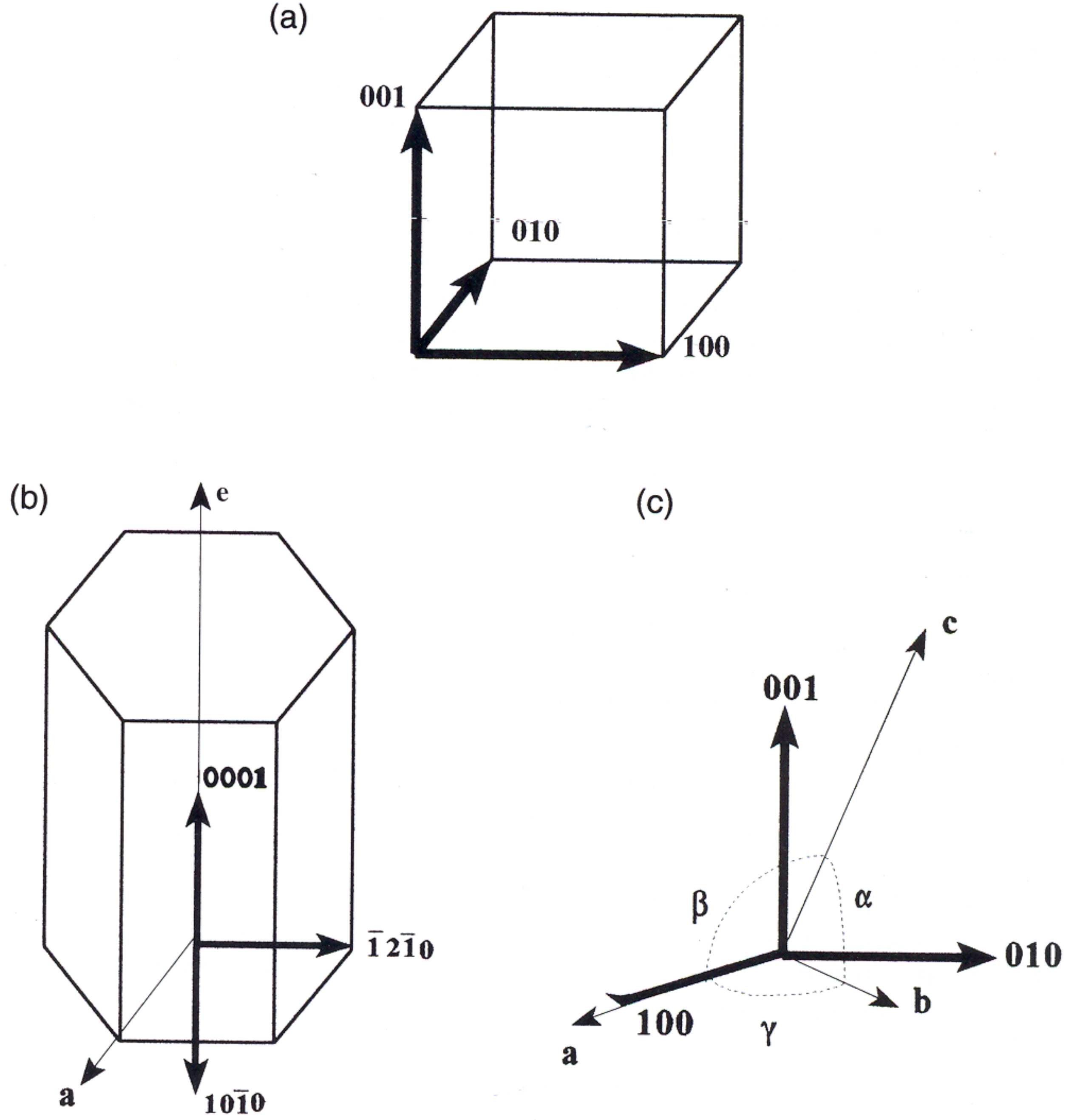
- The choice of specimen frame of reference is dependent on geometry of deformation also.
- Tensile specimens: Uniaxial symmetry, hence one axis is necessary to specify in the coordinate system other two can be chosen arbitrarily.
- In the natural rocks the plane of foliation and a line of lineation within that plane often makes a natural choice for the sample axis
- Sometimes the sample axes are chosen arbitrarily
- Sample symmetry of annealed specimens is defined in terms of prior deformation geometry

Crystal Coordinate System

- **In principle, choices of directions are arbitrary. However, it is convenient to adapt with crystal symmetry. For example, for orthogonal symmetry (cubic, tetragonal, orthorhombic) the axes [100], [010] and [001] form an orthogonal frame and adopted as crystal coordinate system**

- **For convenience, we describe for orthogonal symmetry: [100], [010] and [001] form an orthogonal frame - adopted as crystal coordinate system**





1 Orthonormalised crystal coordinate systems for (a) cubic; (b) hexagonal and (c) general (triclinic) symmetries.

For hexagonal symmetry, an orthogonal frame needs to be associated with the crystal axes:

Two choices

$$X_1 = [10\bar{1}0], \quad Y_1 = [\bar{1}2\bar{1}0], \quad Z = [0001]$$

$$X_2 = [2\bar{1}\bar{1}0], \quad Y_2 = [01\bar{1}0], \quad Z = [0001]$$

Crystal axes are to be made orthonormal - normalised to be all in same length

This is done by pre-multiplying a zone axis by matrix L,

$$\mathrm{L} = \begin{pmatrix} l_{11} & l_{12} & l_{13} \\ l_{21} & l_{22} & l_{23} \\ l_{31} & l_{32} & l_{33} \end{pmatrix}$$

where,

$$l_{11} = a$$
$$l_{12} = b\cos\gamma$$
$$l_{13} = c\cos\beta$$
$$l_{21} = 0$$
$$l_{22} = b\sin\gamma$$
$$l_{23} = c(\cos\alpha - \cos\beta\cos\gamma)/\sin\gamma$$
$$l_{31} = 0$$
$$l_{32} = 0$$
$$l_{33} = c(1 + 2\cos\alpha\cos\beta\cos\gamma - \cos^2\alpha - \cos^2\beta - \cos^2\gamma)^{1/2}/\sin\gamma$$

(Young and Litton, 1972, J. Appl. Phys. 43, 1408)

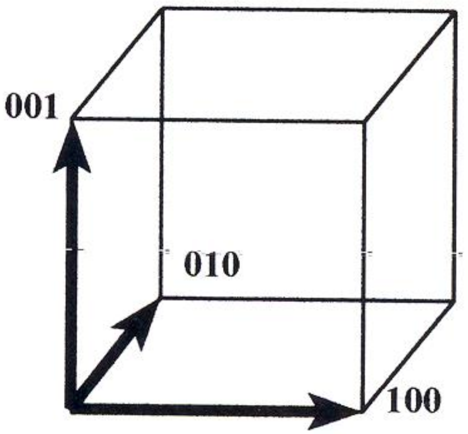
where, a, b, c are lattice parameters and α, β, γ are interzonal angles

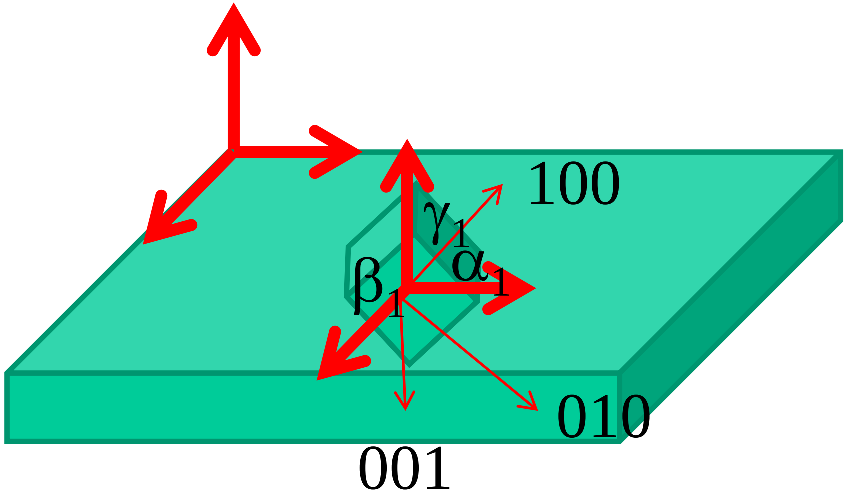
For hexagonal crystals,

$\alpha = 90°$
$\beta = 90°$
$\gamma = 120°$

$$L = \begin{pmatrix} a & -a/2 & 0 \\ 0 & (a\sqrt{3})/2 & 0 \\ 0 & 0 & c \end{pmatrix}$$

- **The following figure depicts the relationship between the specimen coordinate system RD, TD, ND of a rolled material and the crystal coordinate system [100], [010] , [001].**





Orientation thus can also be defined as "the position of crystal coordinates with reference to the specimen coordinates"

$$C_C = g.\ C_s$$

Crystal coordinate system

Specimen coordinate system

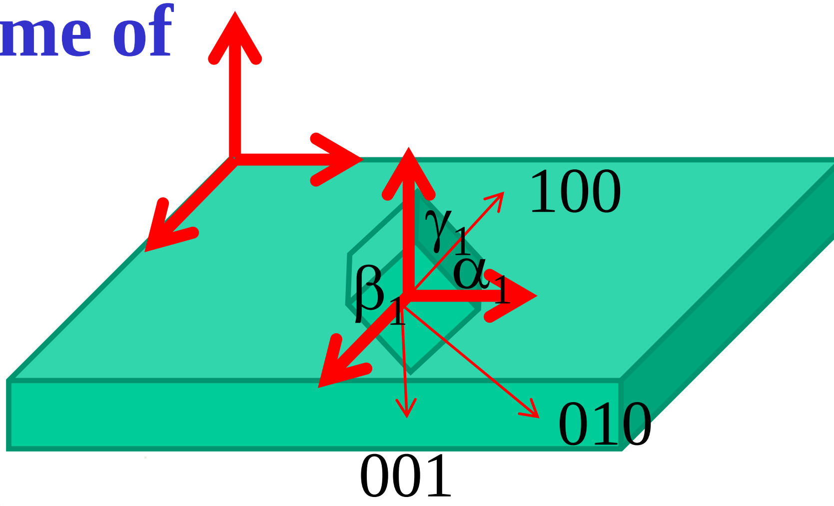
Rotation or orientation matrix: Embodies the rotation of specimen coordinates onto Crystal coordinate system

1st row of matrix: cosines of the angle between 1st crystal axis with each of the 3 specimen axes

2nd row of matrix: cosines of the angle between 2nd crystal axis with each of the 3 specimen axes

3rd row of matrix: cosines of the angle between 3rd crystal axis with each of the 3 specimen axes

Orientation is defined as the position of crystal coordinates with reference to the specimen frame of reference,



$$C_C = g \cdot C_S$$

Crystal coordinate system

Specimen coordinate system

Rotation or orientation matrix

embodies the rotation of specimen coordinates onto Crystal coordinate system

- **Orientation matrix thus allows a crystal direction to be expressed in terms of the specimen direction and vice versa**

- **Rotation or orientation matrix (g) is given by**

$$g = \begin{pmatrix} \cos\alpha_1 & \cos\beta_1 & \cos\gamma_1 \\ \cos\alpha_2 & \cos\beta_2 & \cos\gamma_2 \\ \cos\alpha_3 & \cos\beta_3 & \cos\gamma_3 \end{pmatrix} = \begin{pmatrix} g_{11} & g_{12} & g_{13} \\ g_{21} & g_{22} & g_{23} \\ g_{31} & g_{32} & g_{33} \end{pmatrix}$$

- **Orientation matrix, however, overdetermines the orientation, the most well established method of expressing is as Euler angles resides in Euler space**

- **Only 3 variables are needed to specify an orientation**

- **So we have to find an alternate way**

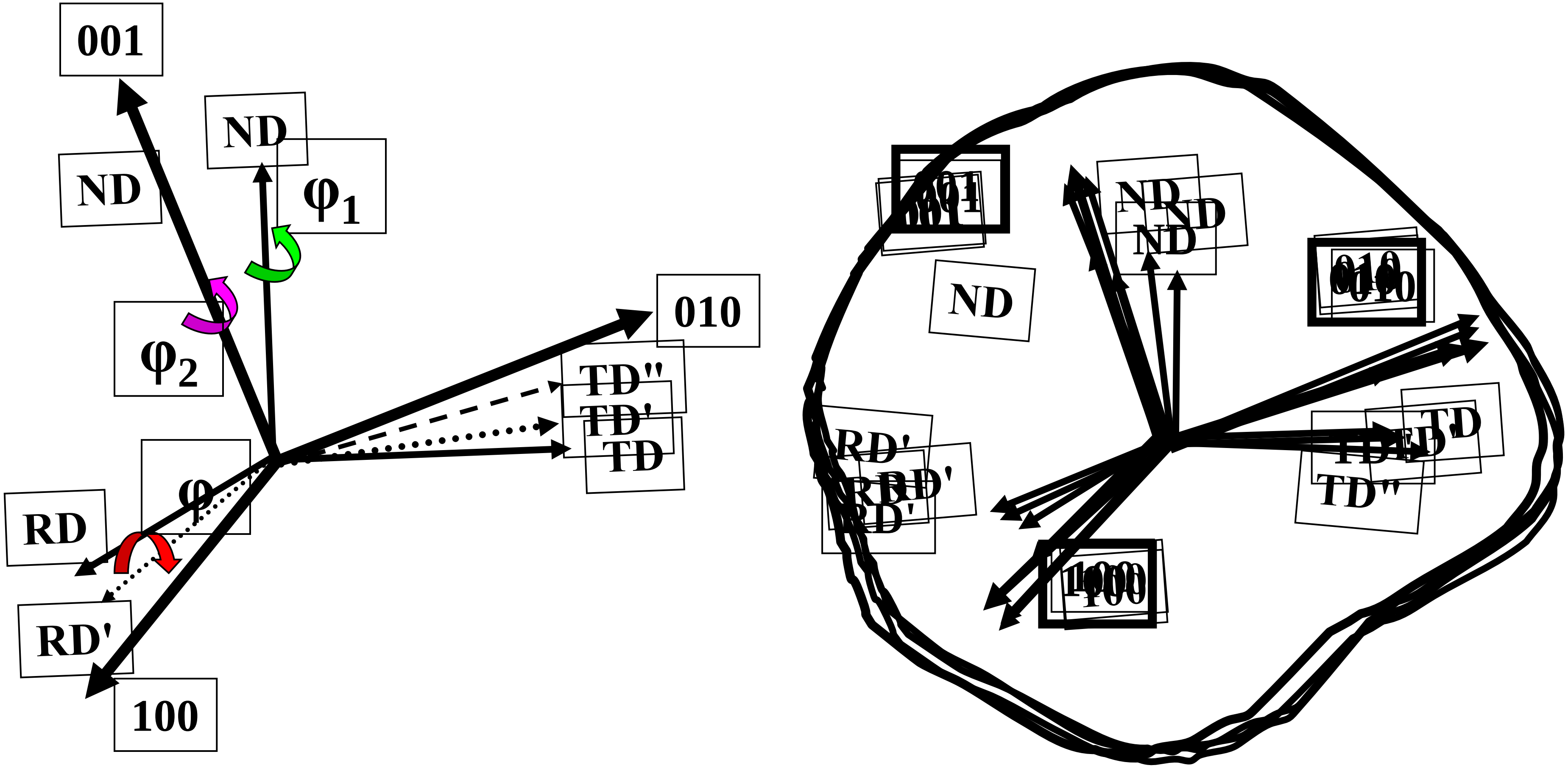
The Euler Angle Representation

- **Euler angle representation is the most convenient way to describe an orientation**
- **Euler angles are the three rotations which, when performed in the correct sequence,transform the specimen coordinate onto the crystal coordinate system.**
- **There are three established ways of doing this:**
  - ***(1) Bunge Method***
  - ***(2) Roe Method***
  - ***(3) Williams Method***

- The most common way to obtain Euler angles to orientation is due to Bunge. The sequence of rotation is as follows:
  - First rotation ϕ_1 about the normal direction ND, transforming TD into TD’ and RD into RD’
  - Second rotation Φ about RD’ transforming ND into ND’([001] and TD’ into TD”
  - A third rotation ϕ_2 about ND’ such that RD’ transforms into [100] and TD” into [010]
- How it is done, is depicted in the next slide

Euler Angles determination: Bunge Method

- First rotation ϕ_1 about the normal direction ND, transforming TD into TD’ and RD into RD’
- Second rotation Φ about RD’ transforming ND into ND’([001] and TD’ into TD”
- A third rotation ϕ_2 about ND’ such that RD’ transforms into [100] and TD” into [010]



- **The three rotations described in the previous slide can be expressed mathematically as**

$$g_{\varphi_1} = \begin{pmatrix} \cos\varphi_1 & \sin\varphi_1 & 0 \\ -\sin\varphi_1 & \cos\varphi_1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

$$g_{\Phi} = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\Phi & \sin\Phi \\ 0 & -\sin\Phi & \cos\Phi \end{pmatrix}$$

$$g_{\varphi_2} = \begin{pmatrix} \cos\varphi_2 & \sin\varphi_2 & 0 \\ -\sin\varphi_2 & \cos\varphi_2 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

- **The orientation matrix can, therefore, be described as**

$$g = g_{\varphi_2} \cdot g_{\Phi} \cdot g_{\varphi_1}$$

Recall: The orientation matrix

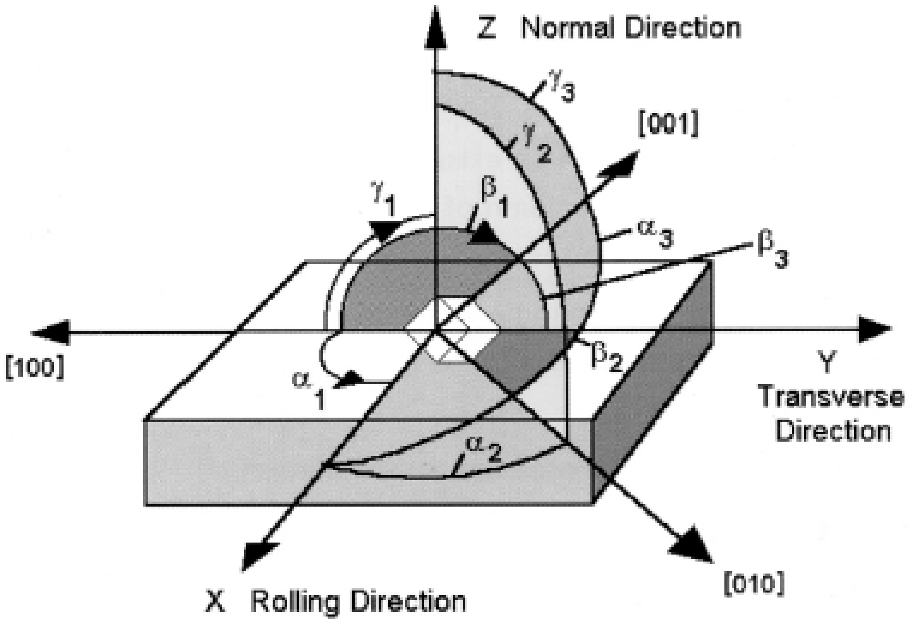
$$g = \begin{pmatrix} \cos\alpha_1 & \cos\beta_1 & \cos\gamma_1 \\ \cos\alpha_2 & \cos\beta_2 & \cos\gamma_2 \\ \cos\alpha_3 & \cos\beta_3 & \cos\gamma_3 \end{pmatrix} = \begin{pmatrix} g_{11} & g_{12} & g_{13} \\ g_{21} & g_{22} & g_{23} \\ g_{31} & g_{32} & g_{33} \end{pmatrix}$$

A comparison with the orientation matrix derived in terms of Euler angles

$$g = g_{\varphi_2} \cdot g_\Phi \cdot g_{\varphi_1}$$

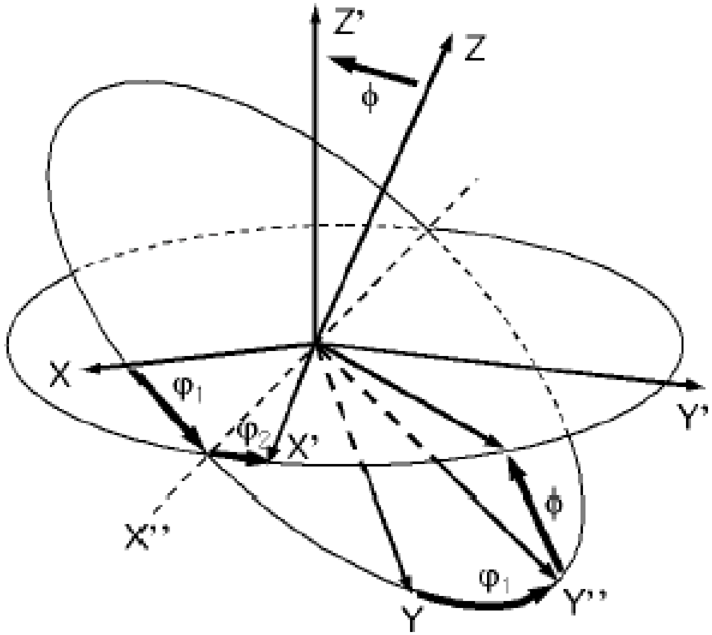
The elements of the matrix in terms of the Euler angles are

$$g_{11} = \cos\varphi_1 \cos\varphi_2 - \sin\varphi_1 \sin\varphi_2 \cos\Phi$$
$$g_{12} = \sin\varphi_1 \cos\varphi_2 + \cos\varphi_1 \sin\varphi_2 \cos\Phi$$
$$g_{13} = \sin\varphi_2 \sin\Phi$$
$$g_{21} = -\cos\varphi_1 \sin\varphi_2 - \sin\varphi_1 \cos\varphi_2 \cos\Phi$$
$$g_{22} = -\sin\varphi_1 \sin\varphi_2 + \cos\varphi_1 \cos\varphi_2 \cos\Phi$$
$$g_{23} = \cos\varphi_2 \sin\Phi$$
$$g_{31} = \sin\varphi_1 \sin\Phi$$
$$g_{32} = -\cos\varphi_1 \sin\Phi$$
$$g_{33} = \cos\Phi$$



$$g = \begin{bmatrix} \cos\alpha_1 & \cos\beta_1 & \cos\gamma_1 \\ \cos\alpha_2 & \cos\beta_2 & \cos\gamma_2 \\ \cos\alpha_3 & \cos\beta_3 & \cos\gamma_3 \end{bmatrix} = \begin{bmatrix} u & r & h \\ v & s & k \\ w & t & l \end{bmatrix}$$

Directional Cosine



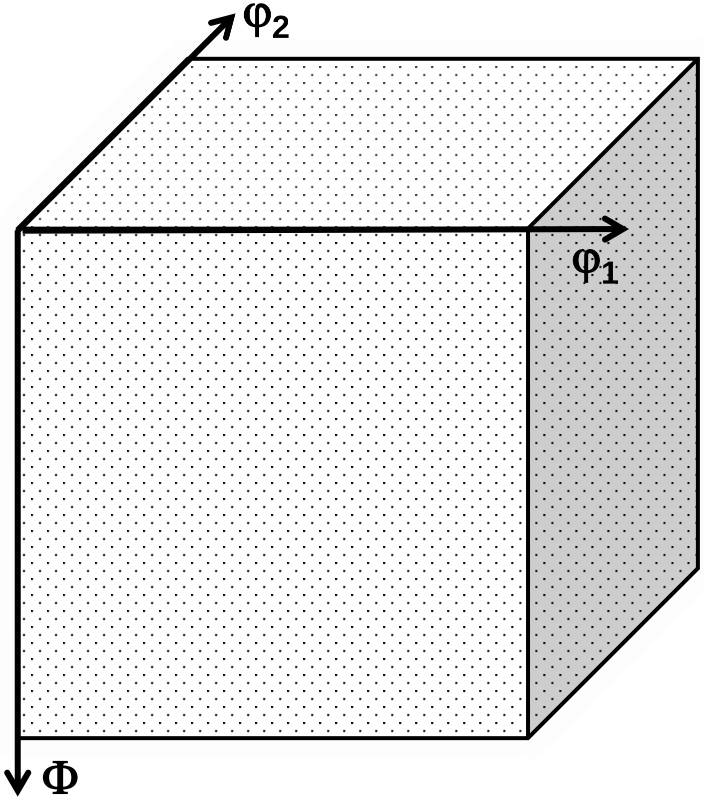
$$\phi = \arccos\frac{l}{\sqrt{h^2+k^2+l^2}}$$

$$\varphi_2 = \arccos\frac{k}{\sqrt{h^2+k^2}} = \arcsin\frac{h}{\sqrt{h^2+k^2}}$$

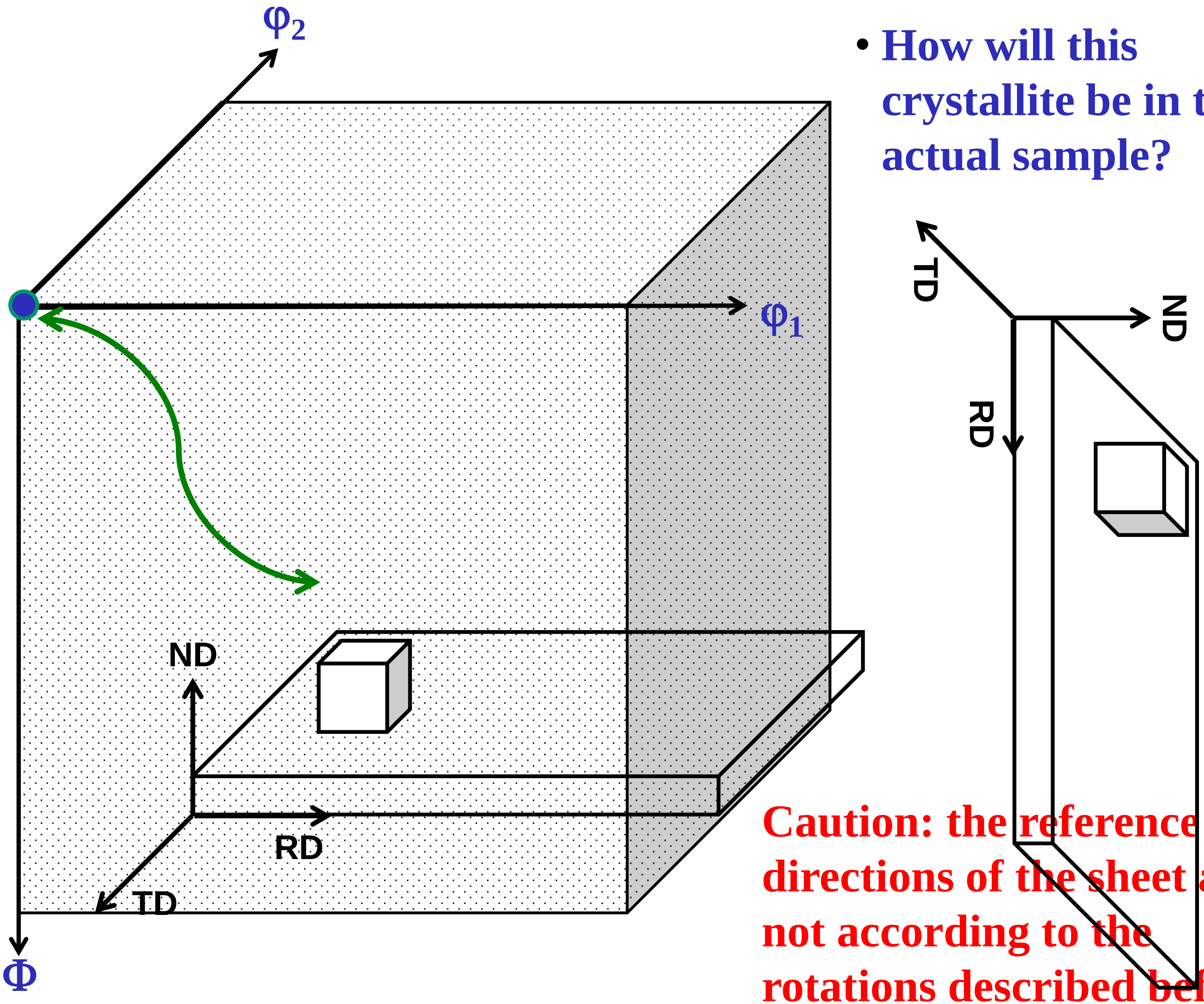
$$\varphi_1 = \arcsin\left[\frac{w}{\sqrt{u^2+v^2+w^2}} \cdot \sqrt{\frac{h^2+k^2+l^2}{h^2+k^2}}\right]$$

Euler angle

- With the above treatment it is clear that the orientation of the crystallites as defined in terms of specimen frame of reference can be expressed in terms of the three Euler angles $(\varphi_1, \Phi, \varphi_2)$ in a very handy manner.
- Extending this, it is also obvious from the preceding treatments that each crystallite orientation (hkl)[uvw] can be uniquely described by a particular value of $(\varphi_1, \Phi, \varphi_2)$.
- It is, therefore, worthwhile to construct a new coordinate system whose axes are φ_1, Φ and φ_2. Each point in this space will represent a particular crystallite with specific orientation (hkl)[uvw].
- Let's see how it is done in the next slide.

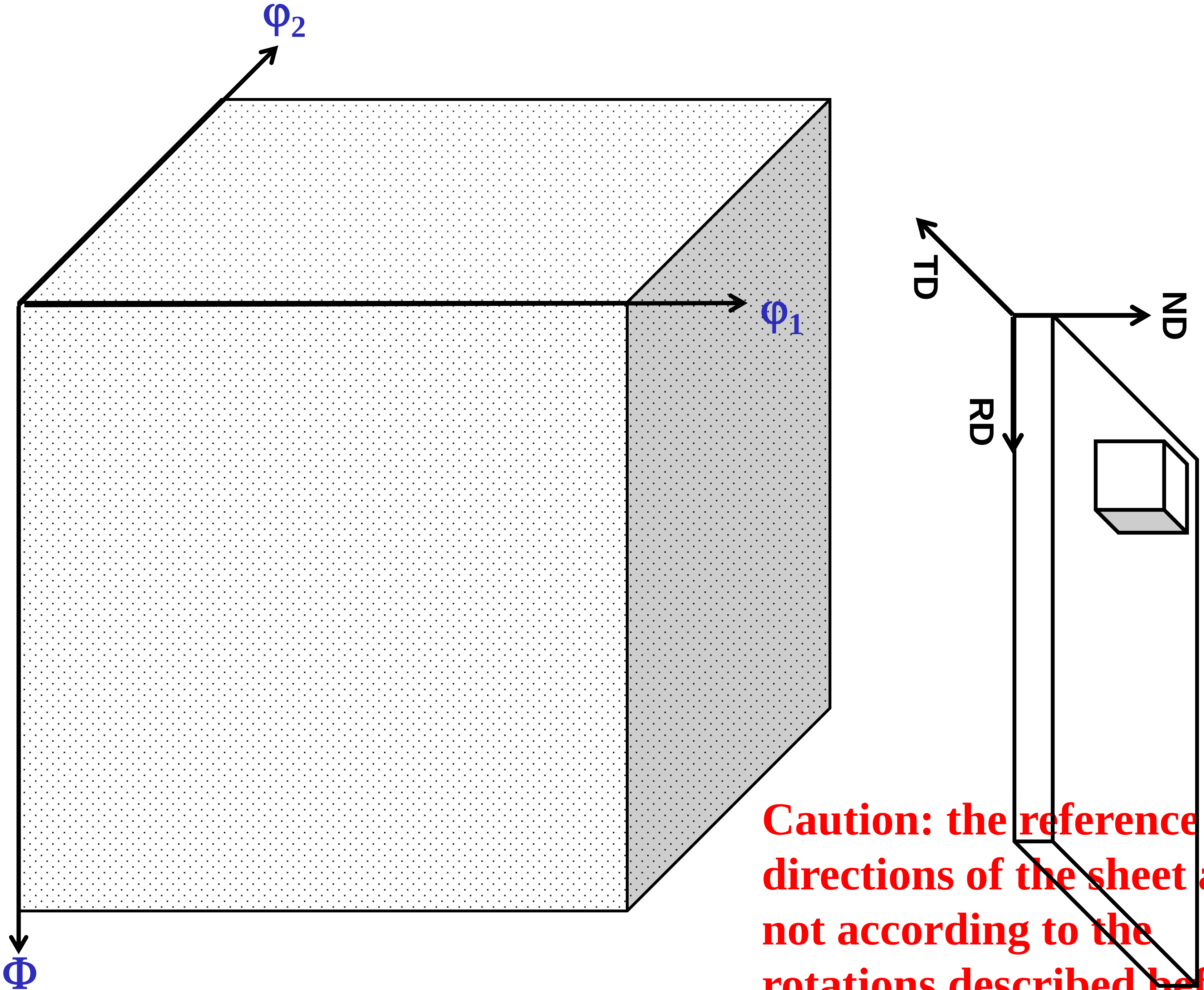


- Each dot in the Euler space depicted here represent an orientation (hkl)[uvw]
- Let us consider the point right at the corner (exaggerated blue dot) – the value of φ_1, Φ, φ_2 for the crystallite here is (0, 0, 0).
- This implies that no rotation is required for making the crystal and specimen frames of reference coincident.



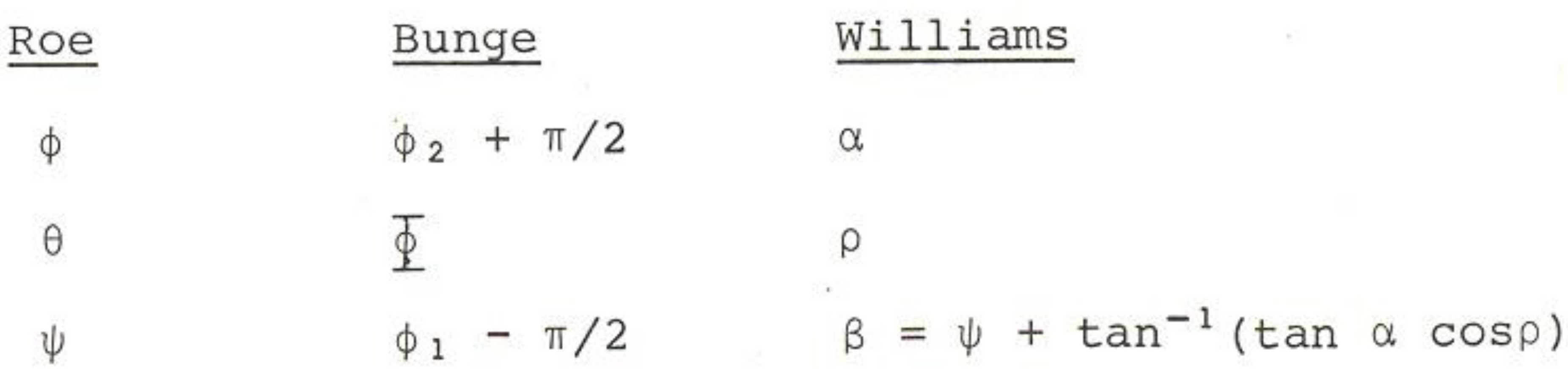
- How will this crystallite be in the actual sample?

Caution: the reference directions of the sheet are not according to the rotations described before.



Caution: the reference directions of the sheet are not according to the rotations described before.

Similarly, the Euler angles proposed by Roe and Williams can also be obtained by following their way of rotation that makes the specimen and the crystal frames coincident. However, these angles are related. The relationship amongst them is as follows:



Roe	Bunge	Williams
ϕ	$\phi_2 + \pi/2$	α
θ	Φ	ρ
ψ	$\phi_1 - \pi/2$	$\beta = \psi + \tan^{-1}(\tan\alpha\cos\rho)$

Mathematical Description of Texture

- **Texture of a single phase polycrystalline material is defined by *orientation distribution function (ODF)*.**
- **ODF describes the volume fraction of crystallites having the orientation of the crystal axis with reference to a fixed coordinate system (RD, TD, ND)**

by volume $\frac{dV(g)}{V} = f(g)dg; \quad g = g(\phi_1, \varphi, \phi_2)$

by number $\frac{dN(g)}{N} = f(g)dg; \quad dg = \frac{1}{8\pi^2} \sin \varphi \, d\varphi \, d\phi_1 \, d\phi_2$

- **Parameters of Orientation "g",**

$g = [g_{ij}] \qquad = \{\phi_1, \varphi, \phi_2\} \qquad = \{hkl\}<uvw>$

Orientation matrix   Euler angle   Crystal direction

Assumption:

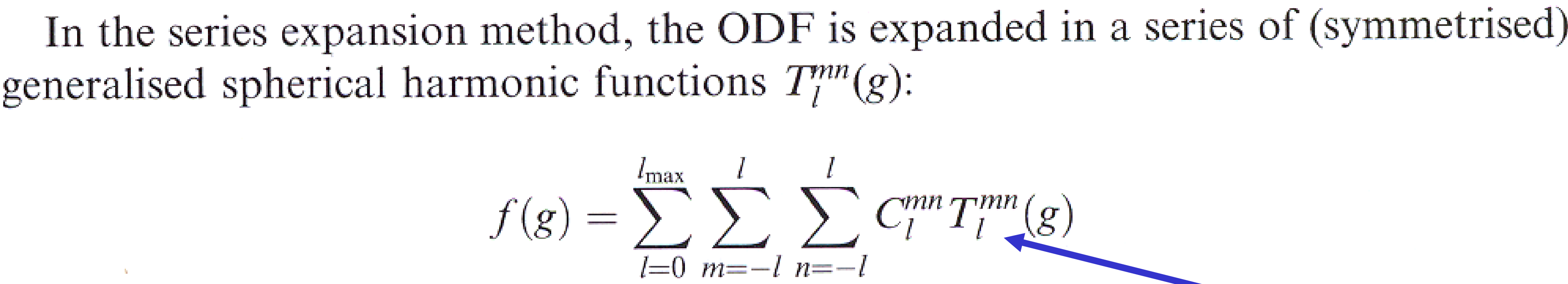
- **Both the measured pole figure and the resulting ODF can be fitted by a series expansion with suitable mathematical functions**

↓

Spherical Harmonic functions

↓

Harmonic method, or Series expansion method



In the series expansion method, the ODF is expanded in a series of (symmetrised) generalised spherical harmonic functions $T_l^{mn}(g)$:

$$f(g) = \sum_{l=0}^{l_{\max}} \sum_{m=-l}^{l} \sum_{n=-l}^{l} C_l^{mn} T_l^{mn}(g)$$

Spherical Harmonic functions

↓

can be calculated for all the orientations `g´- usually stored in libraries

Therefore, ODF f(g) can be completely described by the series expansion coefficients `c´

$$f(g) = \sum_{l=0}^{l_{\max}} \sum_{m=-l}^{l} \sum_{n=-l}^{l} C_l^{mn} T_l^{mn}(g)$$

- **ODF f(g) can be completely described by the series expansion coefficients \`C_l^{mn}´.**
- **The goal is to obtain the coefficients \`C_l^{mn}´.**

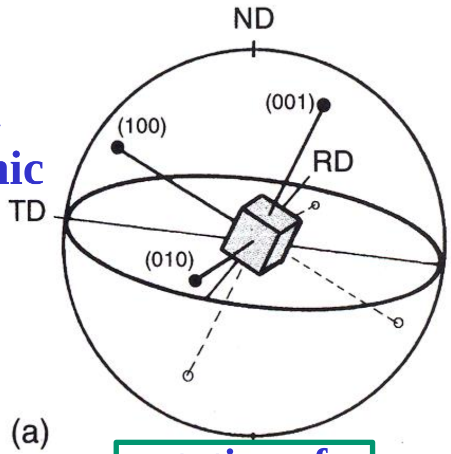
How to do this?

- **The experimental texture is measured in the form of pole figures**
- **The information from pole figure must be transformed to compute the ODF.**
- **The procedure of doing so is called “pole figure inversion”. In this procedure, the pole figure is also expressed in terms of spherical harmonic function whose value is known for all possible orientations on the pole figure and finally the coefficients are calculated. This is done as follows:**

How can we express pole figures mathematically?

Let us recall the previously learnt description of a pole figure.

(100) Poles of a cubic crystal in the stereographic projection

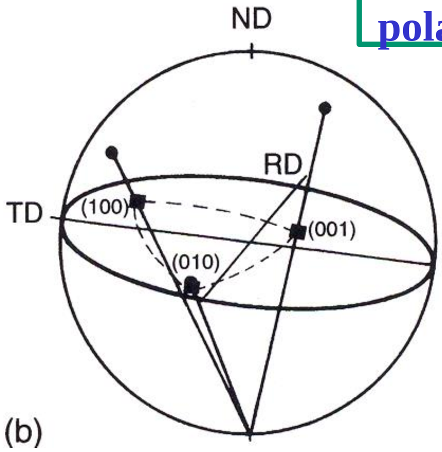


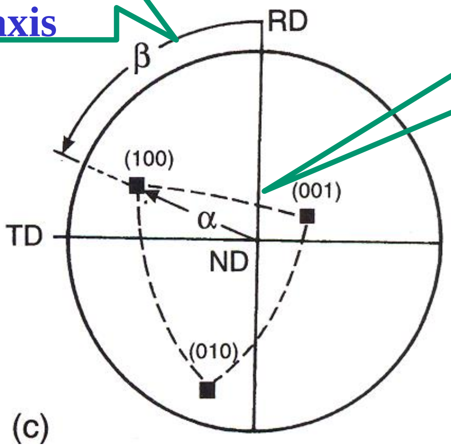
In a pole figure, the position of a given pole on the sphere is characterised in terms of 2 angles:

1. **The rotation of the pole around the polar axis**
2. **The azimuth of the pole**

rotation of the pole around the polar axis

azimuth of the pole

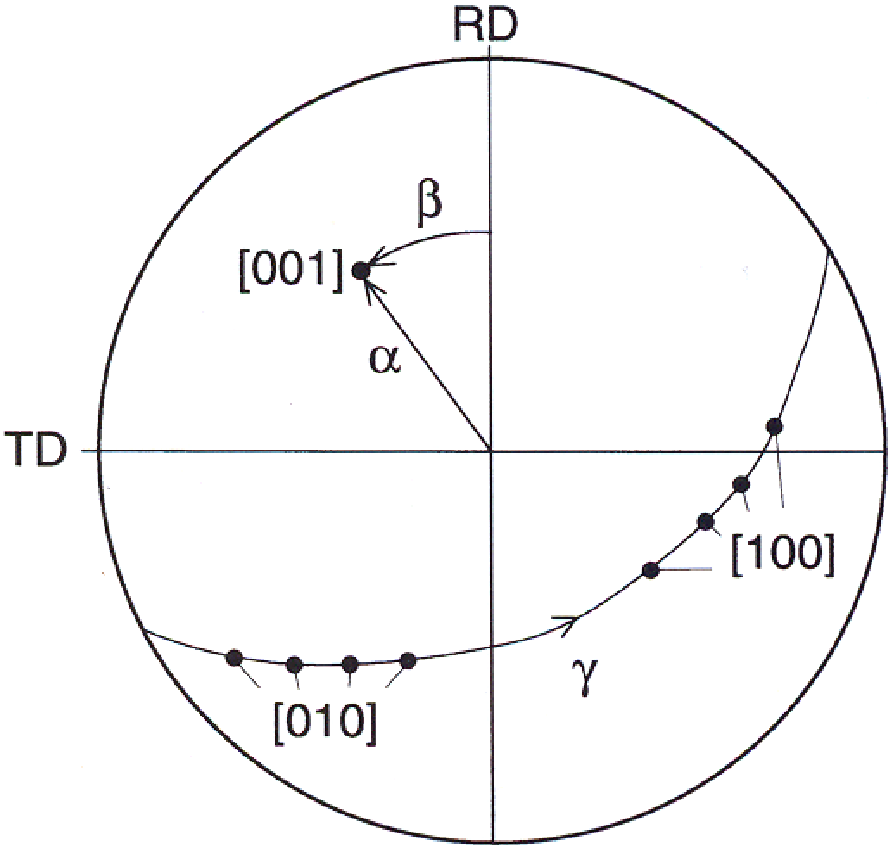




For rolled sheet geometry, the orientation of a cubic crystal is defined as
α= 0° for ND
β= 0° for RD

A pole which is defined by a direction y, e.g. (α, β) in a given 2 D pole figure $P_h(y)$ corresponds to a region in the 3 D ODF which contains all possible rotations with angle γ about this direction y in the pole figure.

That is, $$P_h(y) = \frac{1}{2\pi}\cdot\int_0^{2\pi} f(g)\,d\gamma \quad \text{where} \quad y = \{\alpha, \beta\}; \quad g = \{\varphi_1, \Phi, \varphi_2\}$$



Representation of the fundamental equation of pole figure inversion in a (100) pole figure.

The pole figures can also be expanded in a series expansion. As the pole figure is characterised by the two angles α and β, an expansion in (symmetrised) spherical harmonic functions $K_l^n(y)$ is sufficient:

$$P_h(y) = \sum_{l=0}^{l_{\max}} \sum_{n=-l}^{l} F_l^n(h) K_l^n(y)$$

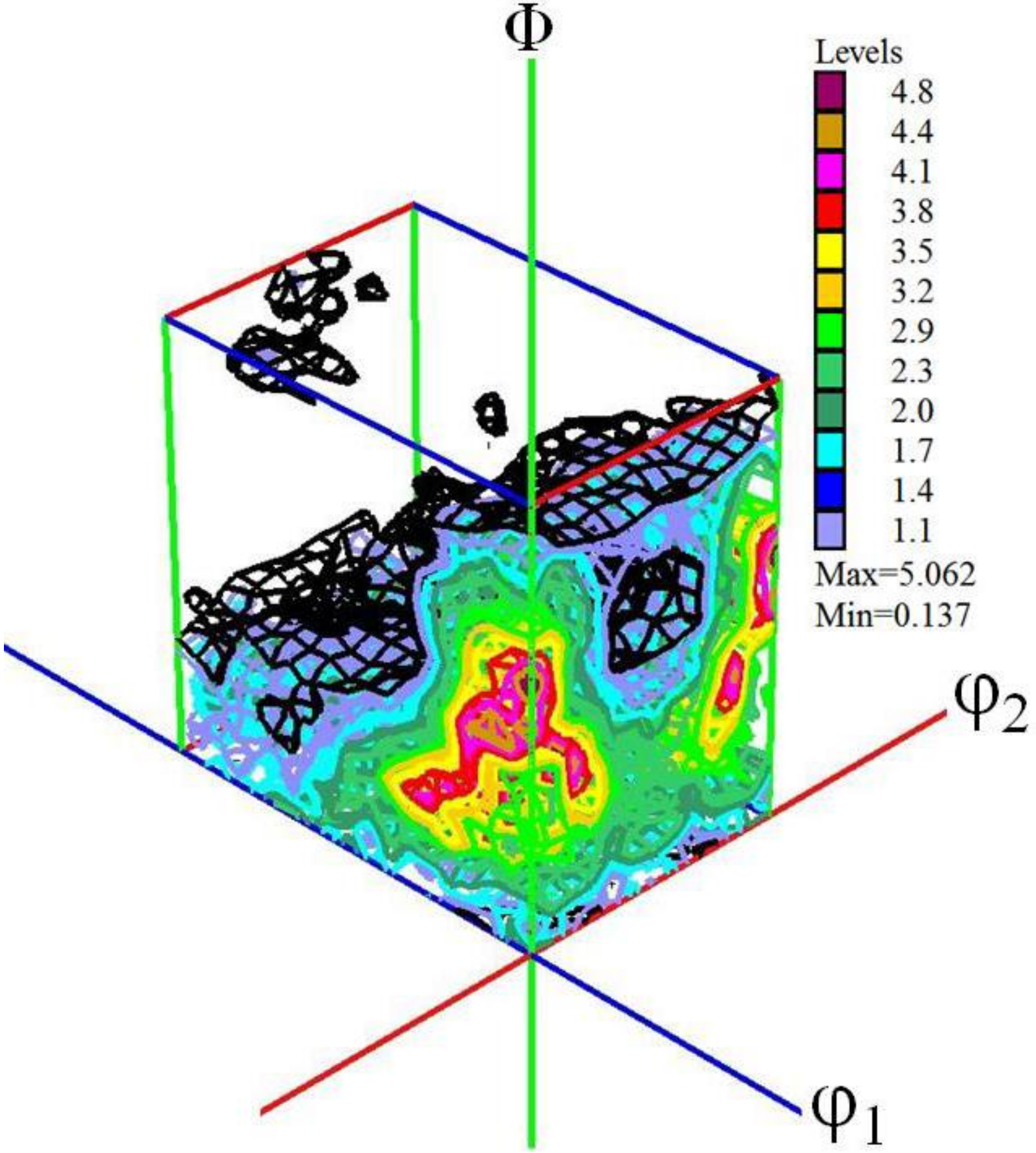
where F^n_l are series expansion coefficients, and $K^n_l(y)$ are the spherical harmonic function

The two-dimensional series expansion coefficients F_l^n are related to the C_l^{mn}-coefficients through:

$$F_l^n(h) = \frac{4\pi}{2l+1} \cdot \sum_{m=-l}^{l} C_l^{mn} K_l^{*m}(h)$$

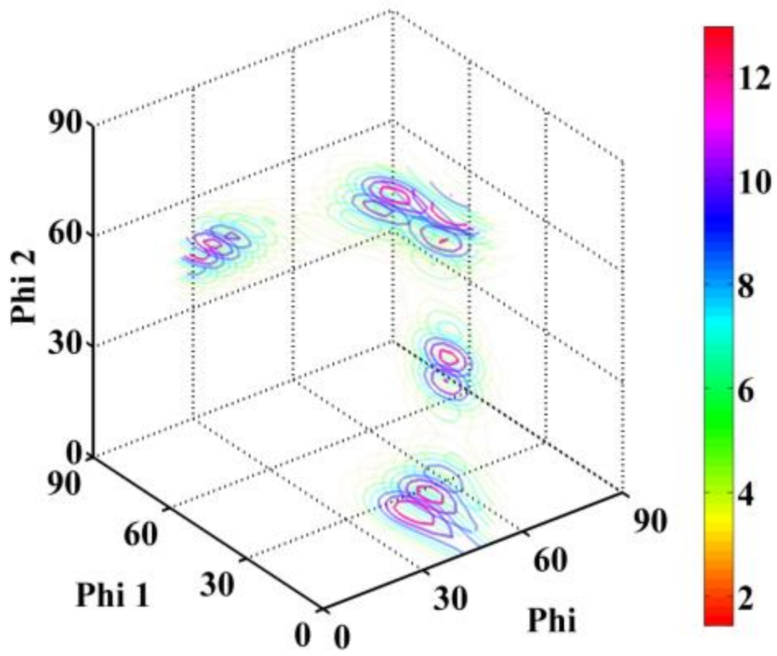
- Usually several pole figures P_h are required to calculate the ODF.
- Lower the symmetry of crystal structure, higher number of pole figures are required.
  - For cubic crystal structure, 3-4 pole figures are desirable
  - For hexagonal crystal structure, 6 pole figures are required
  - For orthorhombic crystal structure , 7 pole figures are a must.
- The size and shape of the Euler space depends on the combination of specimen symmetry and crystal symmetry.

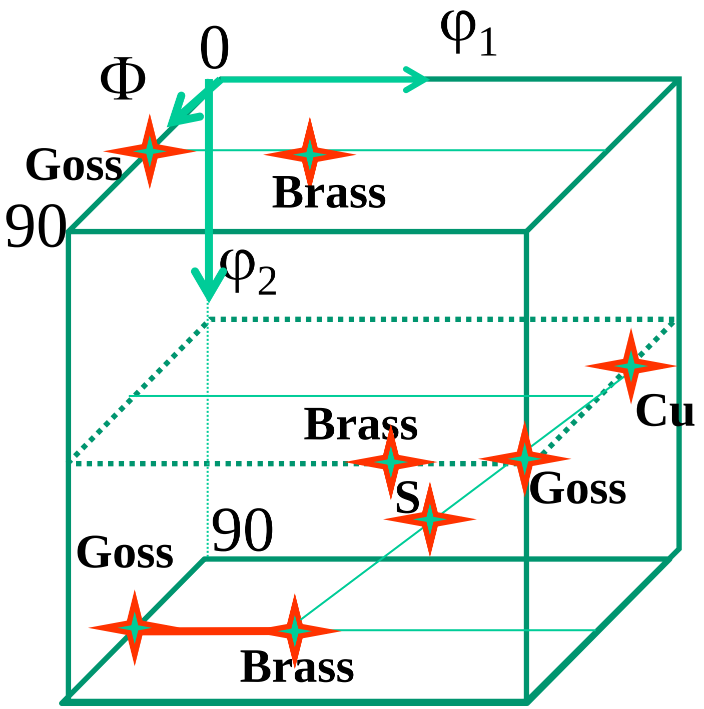
A typical three dimensional orientation distribution function plot appears like follows:



This Euler space is for a hexagonal metal. Therefore, it is not a symmetrical. The φ2 axis ranges from 0-60 degree while the other two axes have their range 0-90 degree. In a cubic material, all the three axes are 0-90 degrees, therefore the Euler space is a perfect cube.

- Euler space for Cubic materials with the main orientations depicted



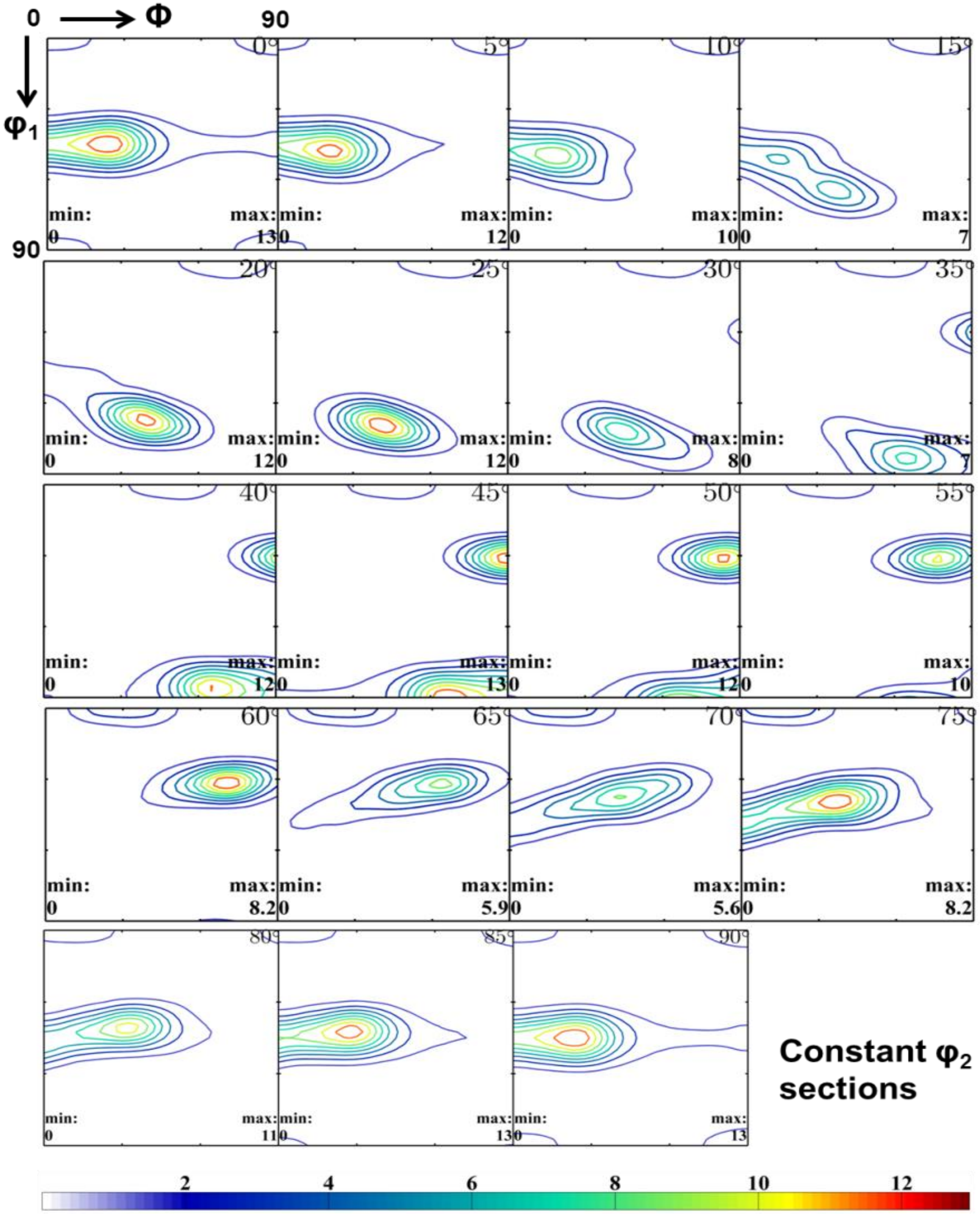


Courtesy: R. Madhavan

- It is, however, not convenient to read orientations, that is the orientation coordinates {φ_1, Φ, φ_2}, therefore the Euler space is sliced at certain angular intervals along one of the axes and the slices are spread on the sheet

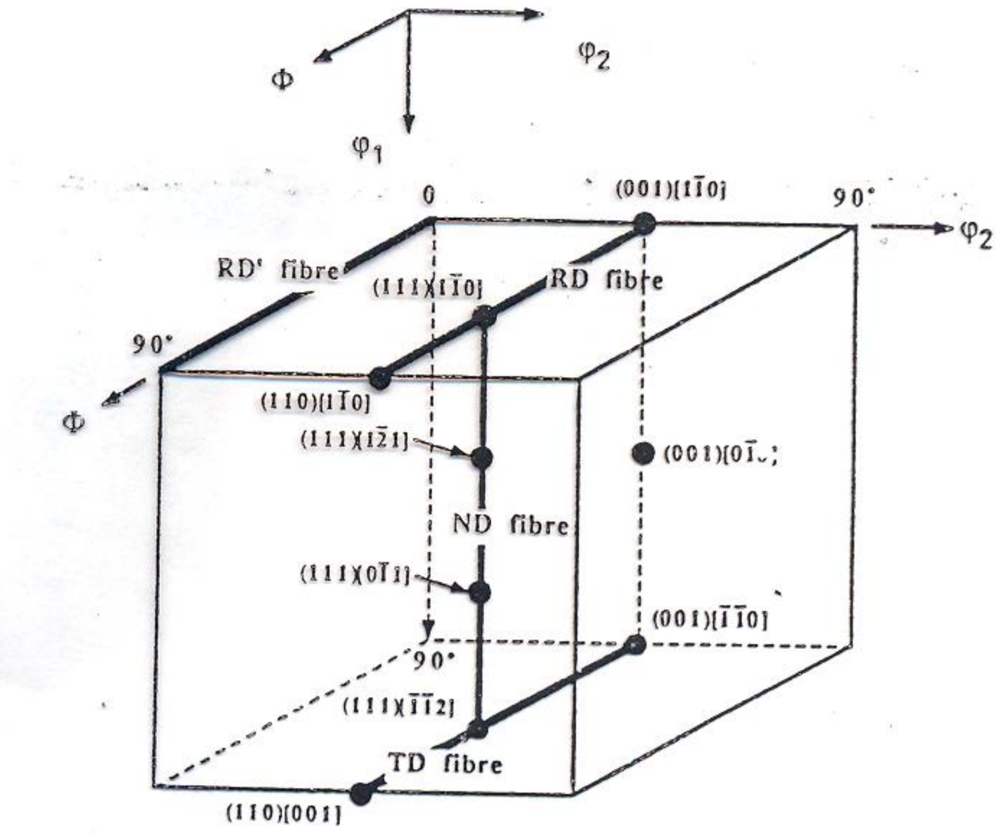
- The spread cuts look like this figure →

- By identifying the maxima locations in the cut-sections, one can obtain {φ_1, Φ, φ_2}, from which the texture component (hkl)[uvw] can be computed.



Constant φ_2 sections

Courtesy: R. Madhavan

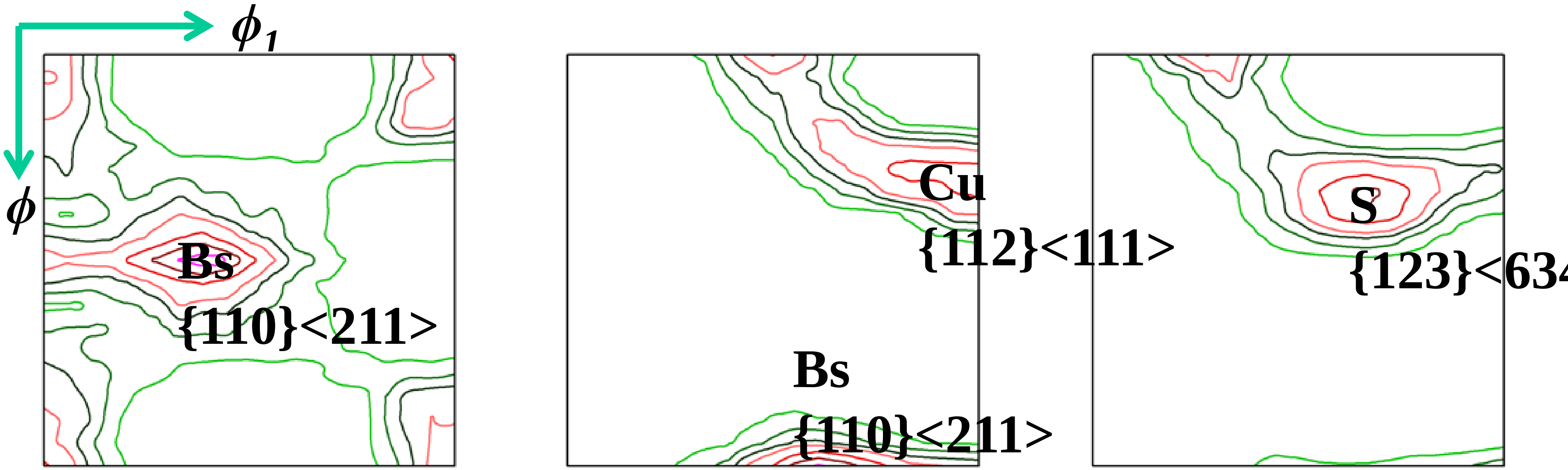


3 Three dimensional view of Euler space with locations of some important ideal orientations and fibres (Bunge notation)

http://aluminium.matter.org.uk/content/html/eng/

Representation of 3D ODF in 2D

Section (at some angular interval) the 3D euler space along ϕ_2 or any of ϕ_1, ϕ , ϕ_2

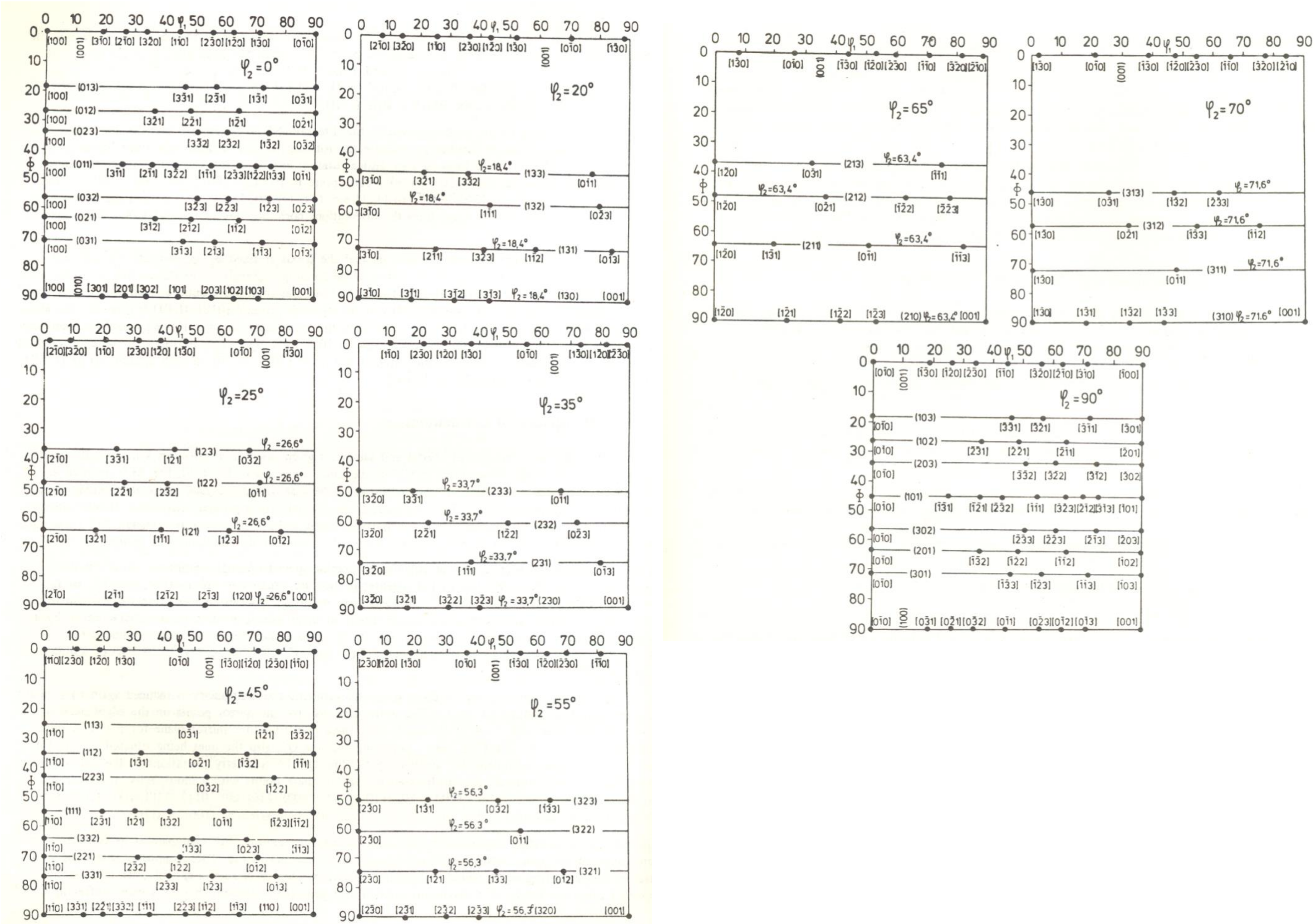


$\Phi_2=0^{\circ}$ $\Phi_2=45^{\circ}$ $\Phi_2=65^{\circ}$

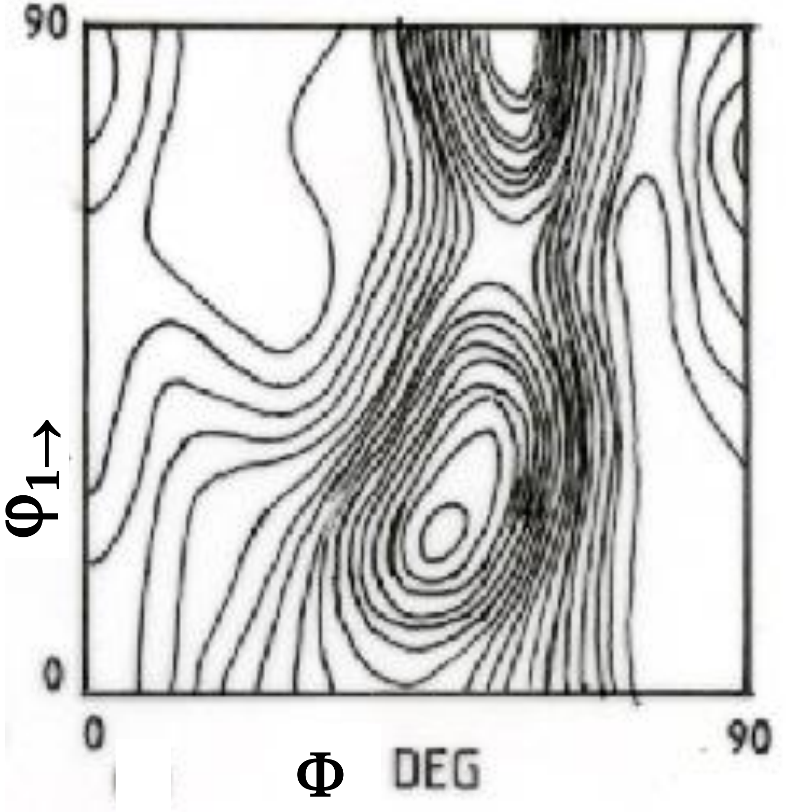
In the representation the Euler space is sectioned along ϕ_2 and most relevant sections are displayed

- **Standard charts are available with (hkl)[uvw] computed for various ({φ_1, Φ, φ_2}**

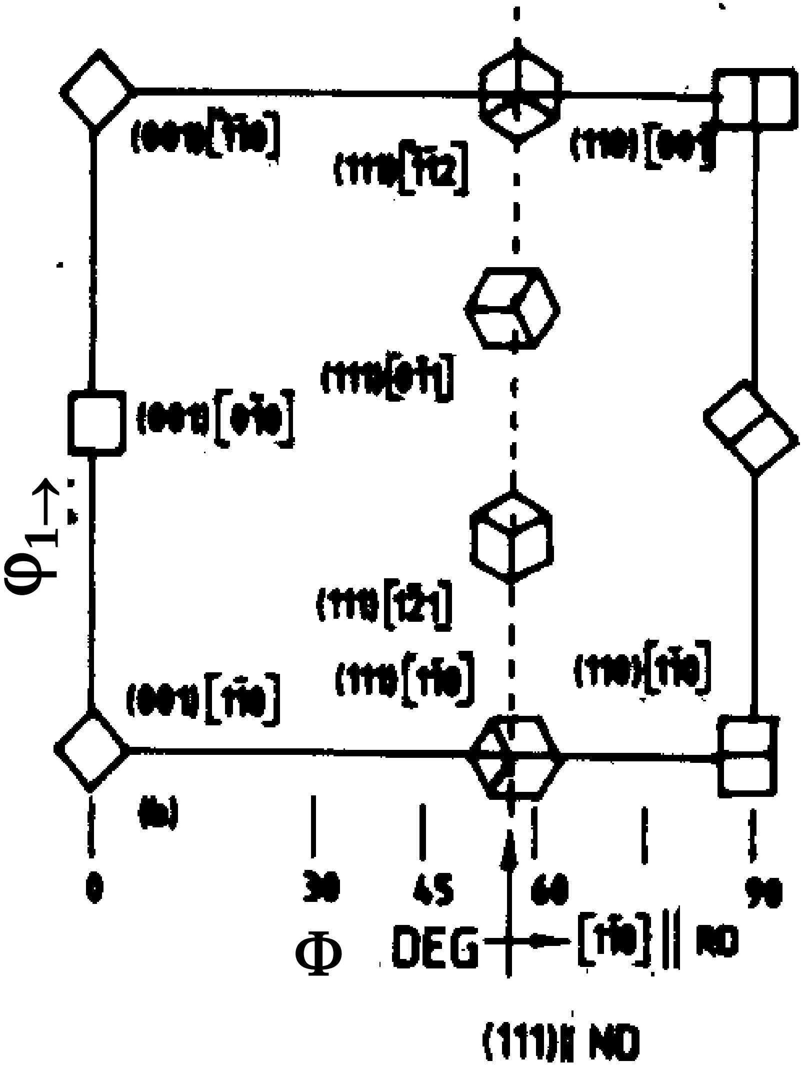
- **By matching the position of maxima with the corresponding location in the chart, it is possible to identify the texture component (hkl)[uvw]**


φ2 = 0°
φ2 = 20°
φ2 = 25°
φ2 = 35°
φ2 = 45°
φ2 = 55°
φ2 = 65°
φ2 = 70°
φ2 = 90°
φ1
Φ
0 10 20 30 40 50 60 70 80 90
(013)
(012)
(023)
(011)
(032)
(021)
(031)
(133)
(132)
(131)
(130)
φ2 = 18,4°
(123)
(122)
(121)
(120)
φ2 = 26,6°
(233)
(232)
(231)
(230)
φ2 = 33,7°
(113)
(112)
(223)
(111)
(332)
(221)
(331)
(110)
(323)
(322)
(321)
(320)
φ2 = 56,3°
(213)
(212)
(211)
(210)
φ2 = 63,4°
(313)
(312)
(311)
(310)
φ2 = 71,6°
(103)
(102)
(203)
(101)
(302)
(201)
(301)
[100]
[010]
[001]

- By this way, it is easier to visualize the crystallite orientation as follows.



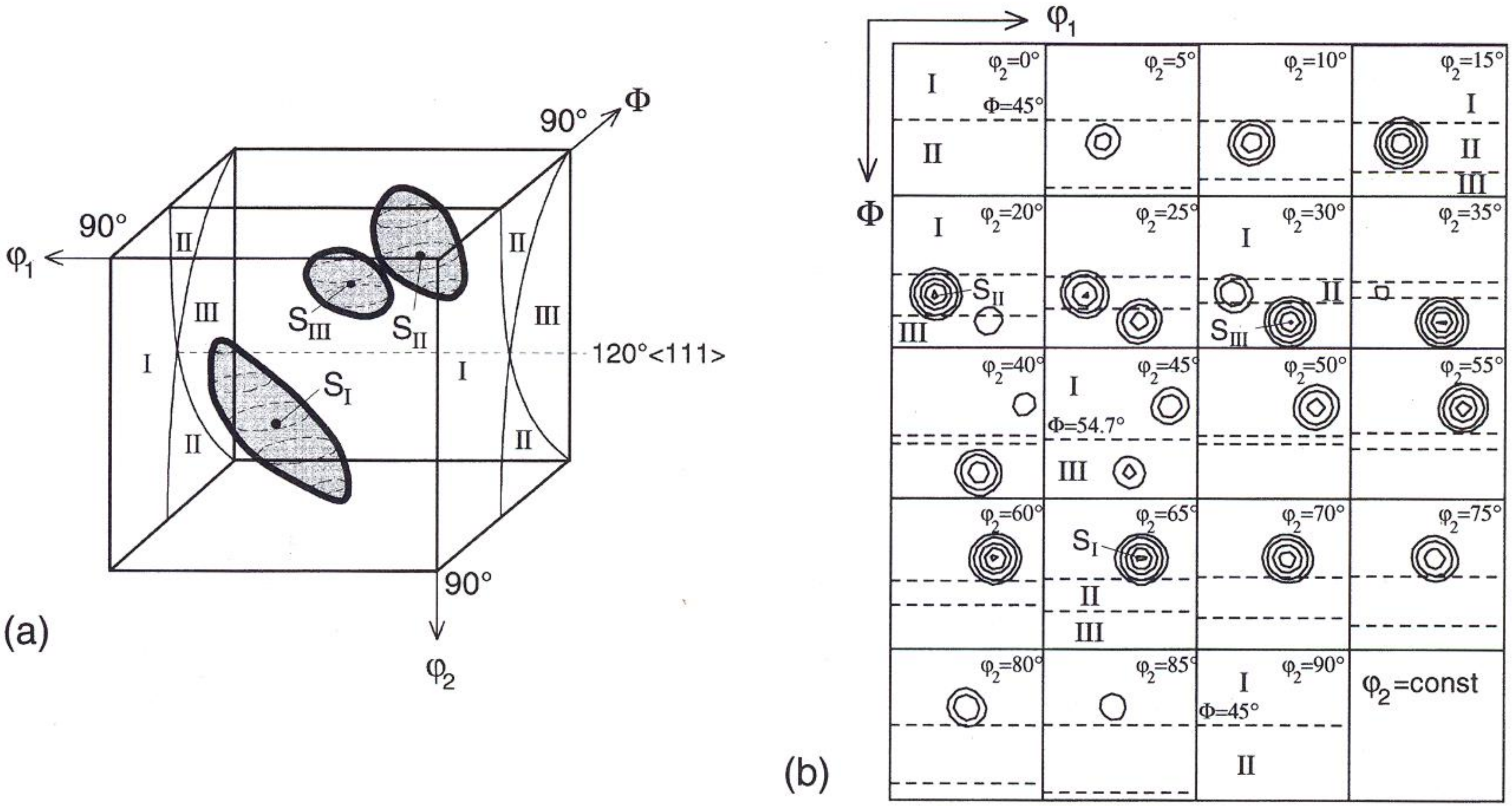
The ODF section $\varphi_2 = 45°$



Courtesy: R.K. Ray

The orientation of crystallites represented in the given ODF section

Some important points in ODF representation



Spatial arrangement of the three components of the orientation (123)[63$\bar{4}$] with isotropic scatter of 10° in the reduced Euler angle space showing strong distortions at small angles Φ. (b) Same texture as in (a) plotted as iso-intensity lines in φ_2-sections ($\Delta\varphi_2 = 5°$) through the Euler space.

Measurement of microtextures

- **Micro-texture measurements are based on individual grain technique**
- **Primary output in all the techniques are: diffraction patterns from each sampled volume**
- **Diffraction pattern from each sampled volume contains *complete crystallographic information along with the orientation* of the respective sampled volume**
- **The sampled volume is *an individual crystallite wherein the orientation can be taken to be uniform***

Following techniques can be used for the determination of microtexture

- Selected area channeling (SAC) in SEM
- Electron back-scatter diffraction (EBSD) in SEM
- Microdiffraction or convergent beam electron diffraction (CBED) in the TEM

Most frequently micro-texture analysis is done with EBSD

Selected Area Diffraction, SAD

coherent, elastic scatter of electrons at crystal lattice ⇒ SAD point diagrams

- SEM (20kV) $\lambda = 0.0086\text{nm}$
- TEM (200kV) $\lambda = 0.0025\text{nm}$

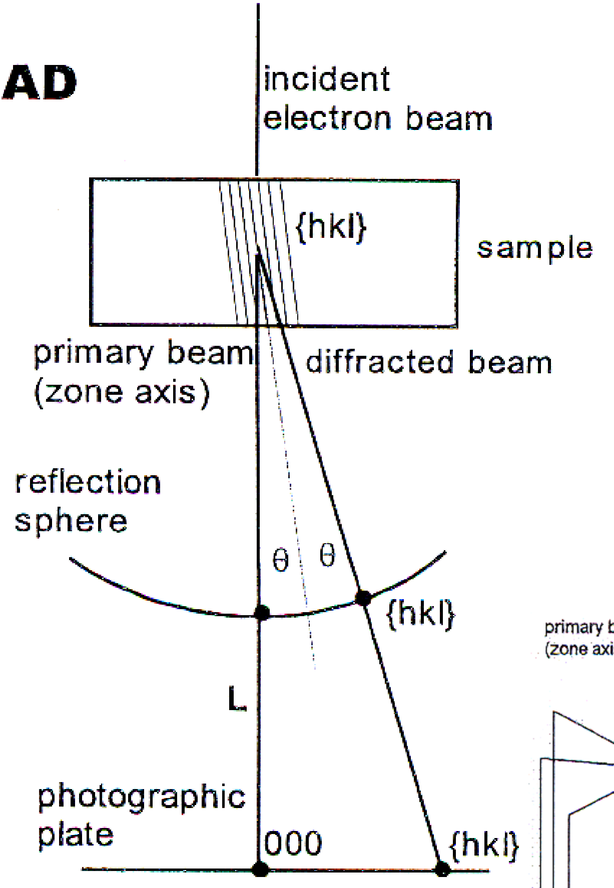
⇒ $\theta < 2°$

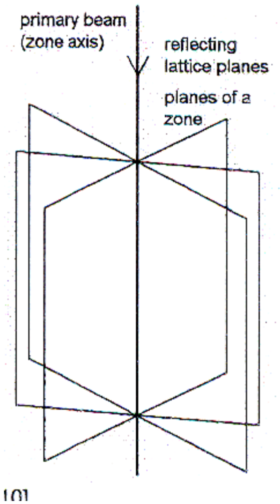
⇒ $\sin\theta \approx \theta$

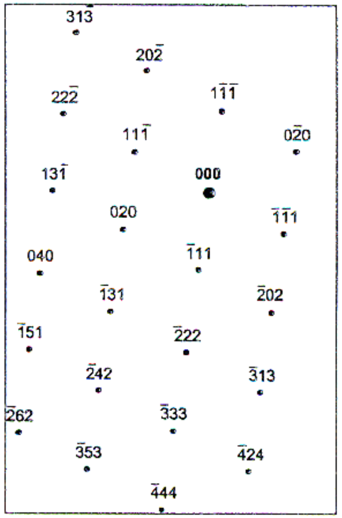
$$n\lambda = 2d\sin\theta = 2d\theta = 2dR_{hkl}/2L$$

(with $n=1$) $\lambda L = R_{hkl}d$ or $R_{hkl} = \lambda L/d$

λL: camera constant (magnification)







2 steps in the determination of crystallographic orientation:

1. **Pattern is identified by crystallographic indices of poles and bands/lines in the pattern**
2. **Relative position of poles on bands/lines with respect to external frame of reference**

Origin of Kikuchi pattern

Kikuchi diffraction pattern can arise due to following electron diffraction techniques

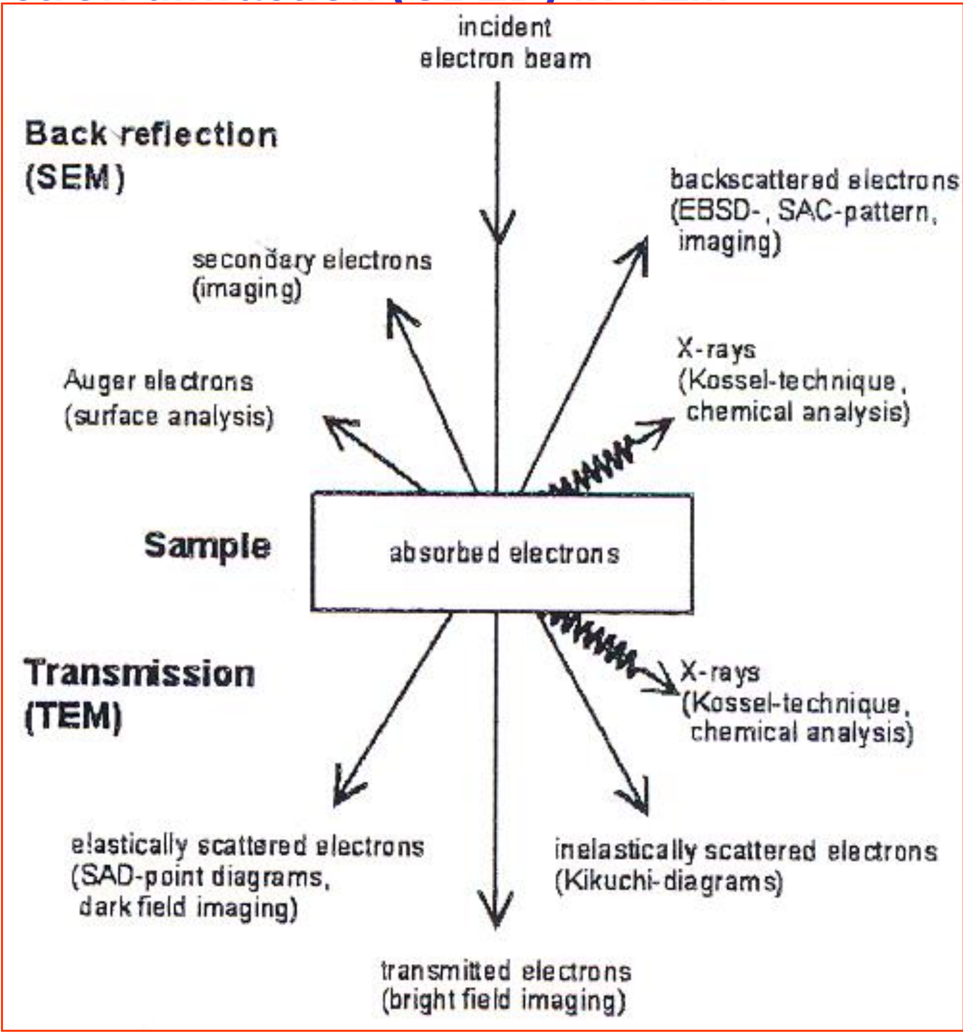
- Selected area channelling (SAC) in SEM
- Electron back-scatter diffraction (EBSD) in the SEM
- Microdiffraction or convergent beam electron diffraction (CBED) in TEM

- Kikuchi diffraction occurs both in SEM and TEM (easier to understand),

Electrons

- only radiation with electrical charge
- strong interaction with nuclei and shell electrons
- vacuum, electron microscope
- minimum penetration depth, maximum spatial resolution (0.1µm and 1nm for SEM and TEM; resp.)
- *wave length*

$$\lambda = \frac{h}{\sqrt{2 \cdot m \cdot e \cdot U}} = \frac{h}{\sqrt{2 \cdot m_0 \cdot e \cdot U \cdot \left(1 + e \cdot U / (2 m_0 c^2)\right)}}$$



Interaction of electrons with sample in TEM

History of EBSD Development

- **1928: Observation of Kikuchi lines in TEM by Kikuchi**
- **1954: Back reflected Kikuchi lines obtained in TEM by Alam.**
- **1969-1979:3 diffraction technique in SEM**
  - **SACP : 10mm in practical resolution**
    [Joy et al. at Oxford Univ.]
  - **Kossel diffraction : 20 mm practical resolution**
    [Dingley et al. at Bristol Univ.]
  - **EBSD : Less than 1mm practical resolution**
    [Venables et al. at Sussex Univ.]
- **1982-1984:Computer assisted indexing of EBSD, subtraction of background value**
- **1990: Fully automated indexing of EBSD patterns by Hough transform [Yale, Clausthal, RisØ]**
- **2000: *-Chemically assisted phase identification***
  - ***-High Resolution* EBSD**
  - ***-In-situ* Experiment EBSD**
  - ***-High current* EBSD**

Steps in the determination of crystallographic orientation

1. Acquire a diffraction pattern

2. Pattern is indexed by identifying crystallographic indices of poles and bands/lines in the pattern

3. Relative position of poles on bands/lines with respect to external frame of reference

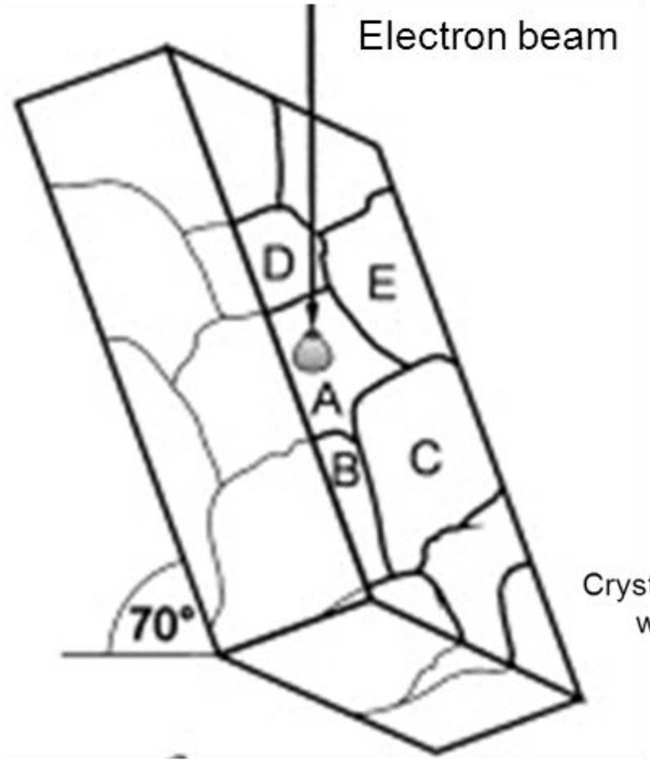
Origin of Kikuchi pattern

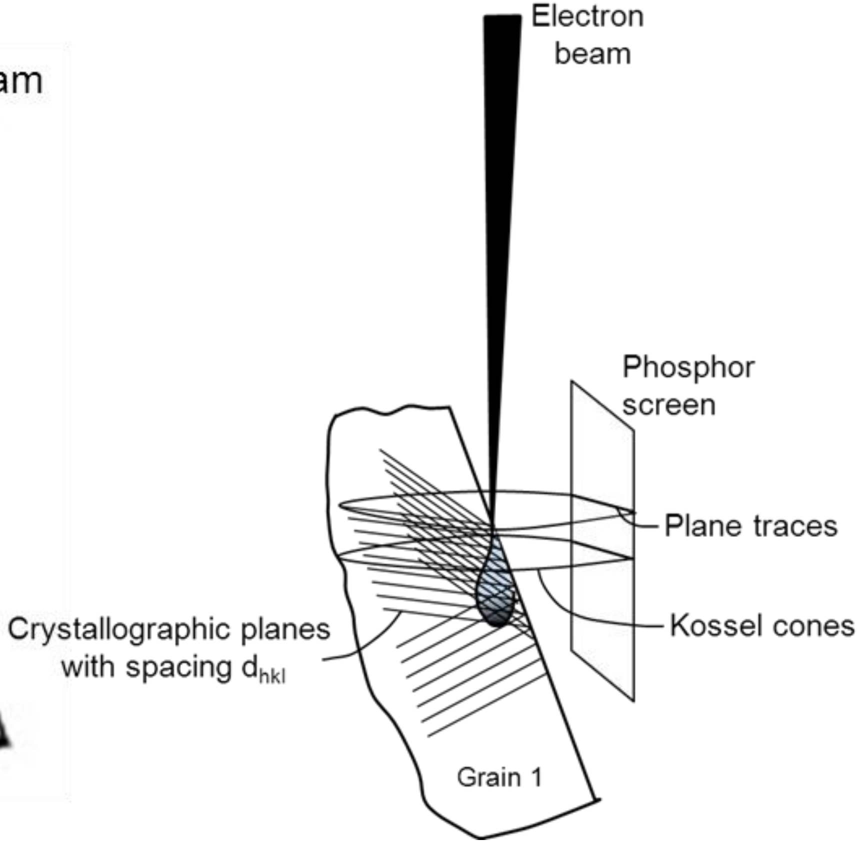
- **When an electron enters a crystalline solid, it is diffusely scattered in all directions.**

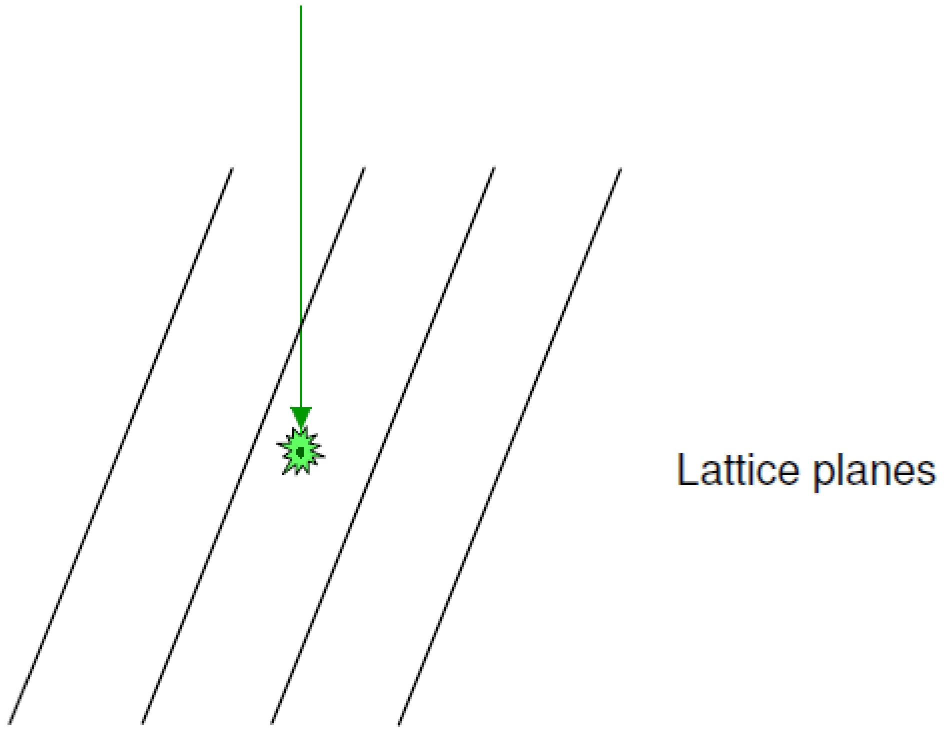
- **Kikuchi lines form by inelastic scattering of electrons, giving Bragg diffraction at source S on lattice planes hkl**

- **There must be some electrons arriving at Bragg angle at every set of lattice planes, and these electrons can undergo elastic scattering to give a strong, reinforced beam.**

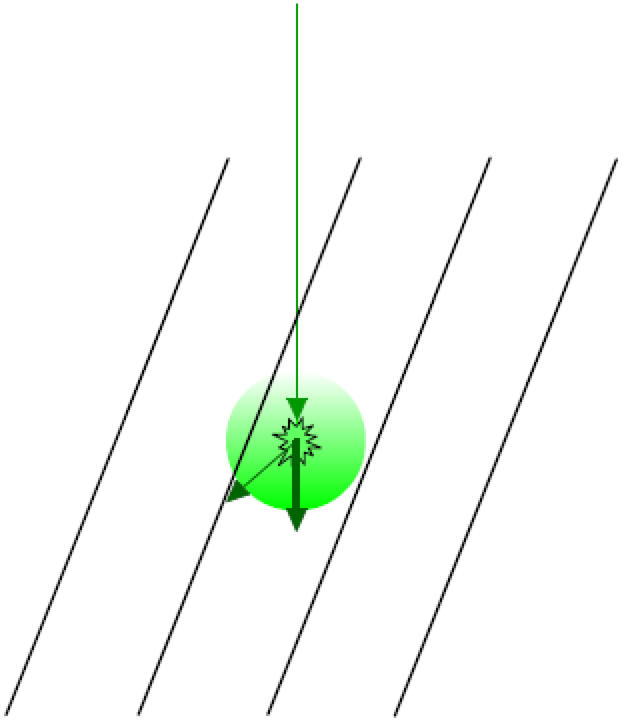
- **Since diffraction of electrons occurs in all directions, the locus of diffracted radiation is the surface of a cone which extends about the normal of the reflecting atomic planes with half apex angle 90-θ.**



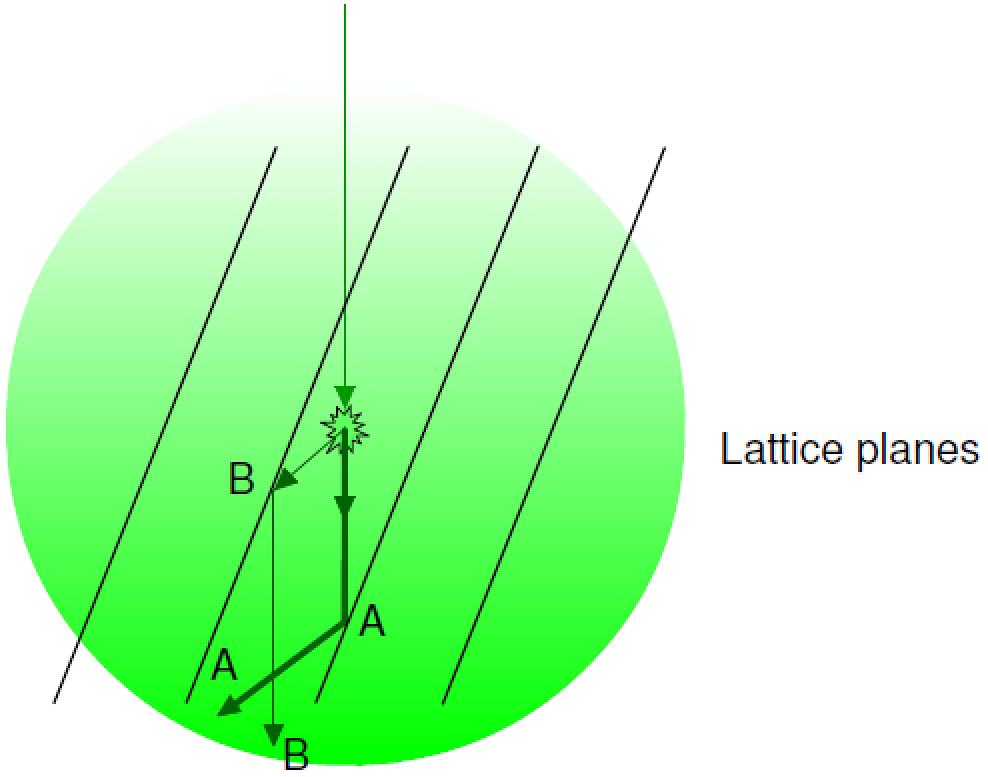




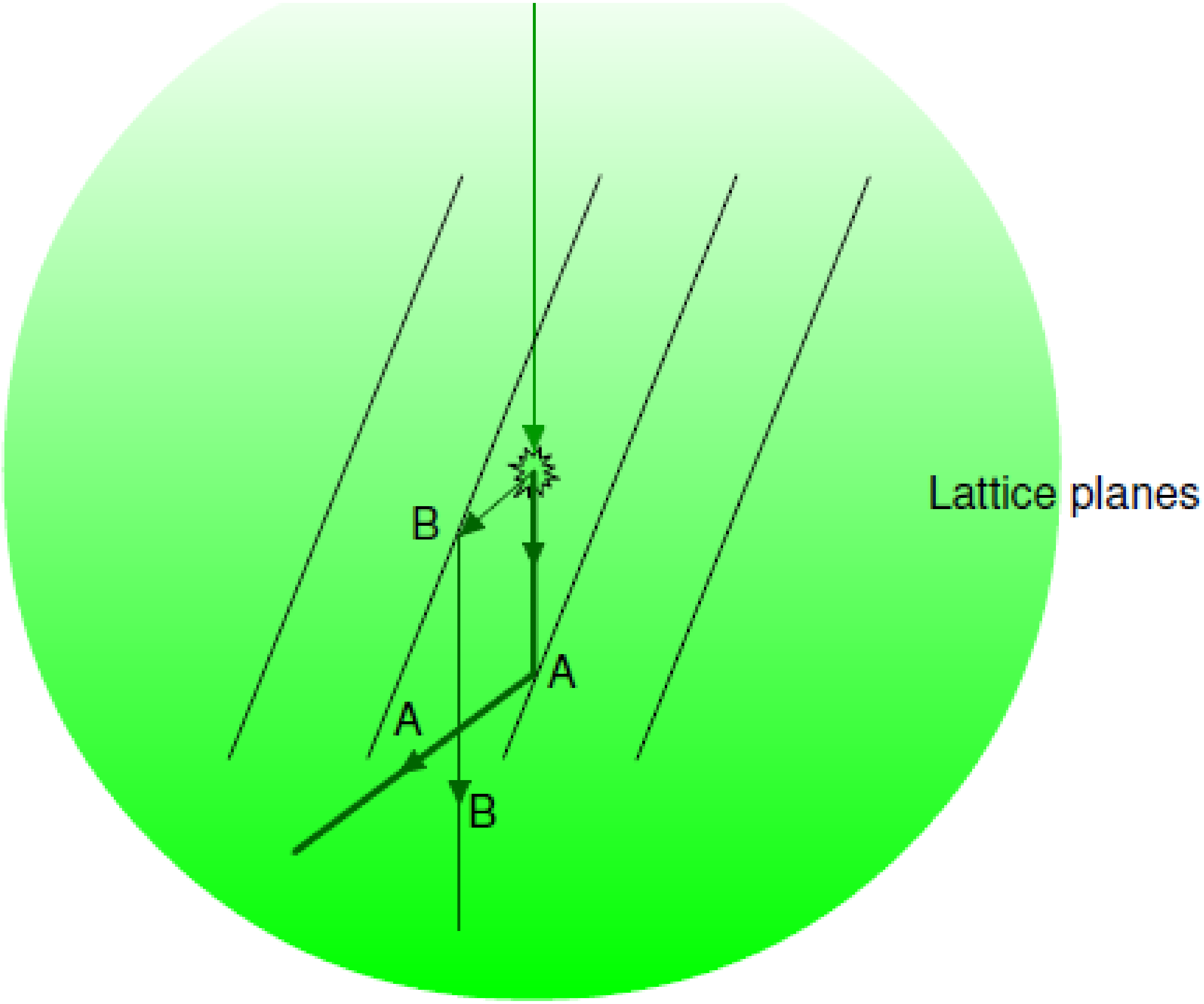
Electron is inelastically scattered by an atom in the sample.



Inelastic collision acts as a new centre for wavelets. The intensity of these wavelets is not uniform. The highest intensity is found in the direction the original beam was following, and intensity decreases continuously as the angle from this direction increases.

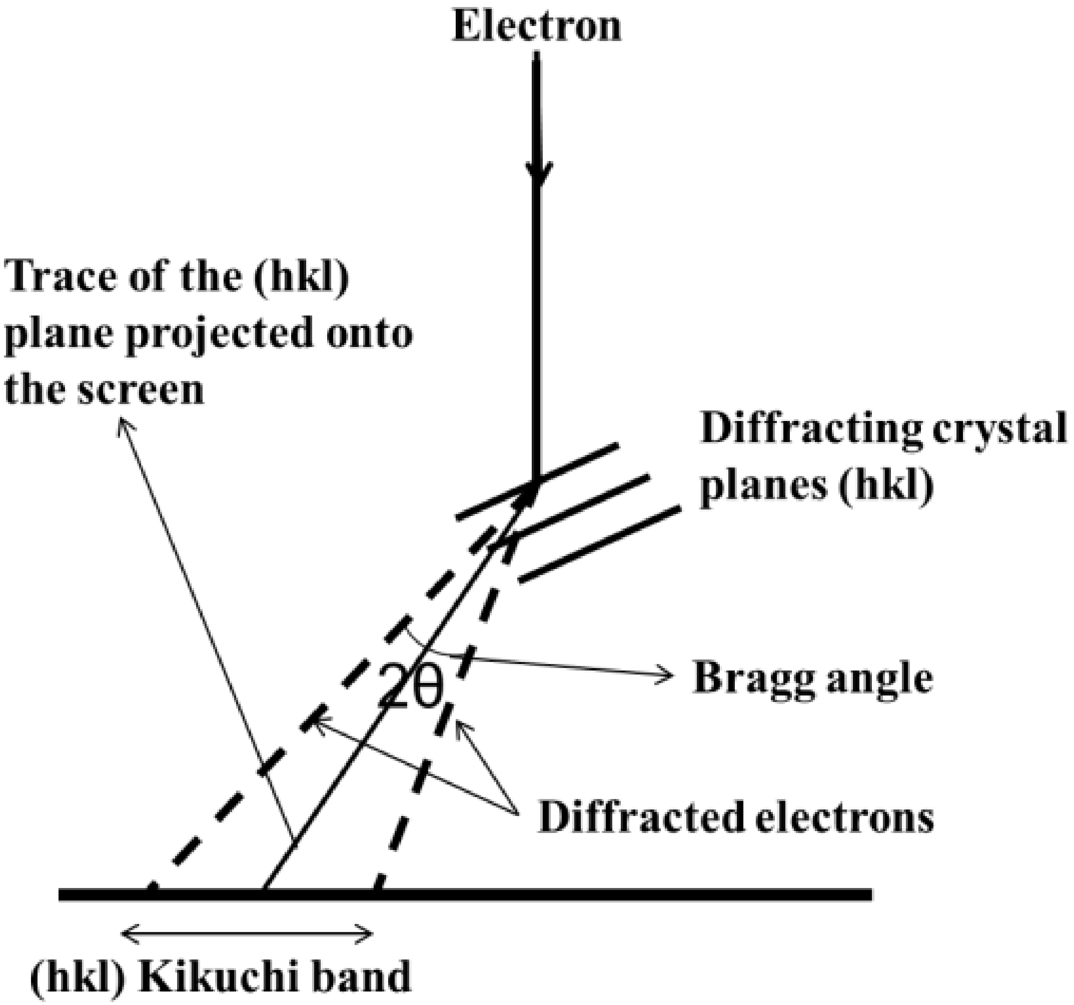


Some of the inelastically scattered waves are at the right Bragg angle for some of the crystal planes. The total amplitude of waves following path A is higher than the total amplitude of waves following path B.



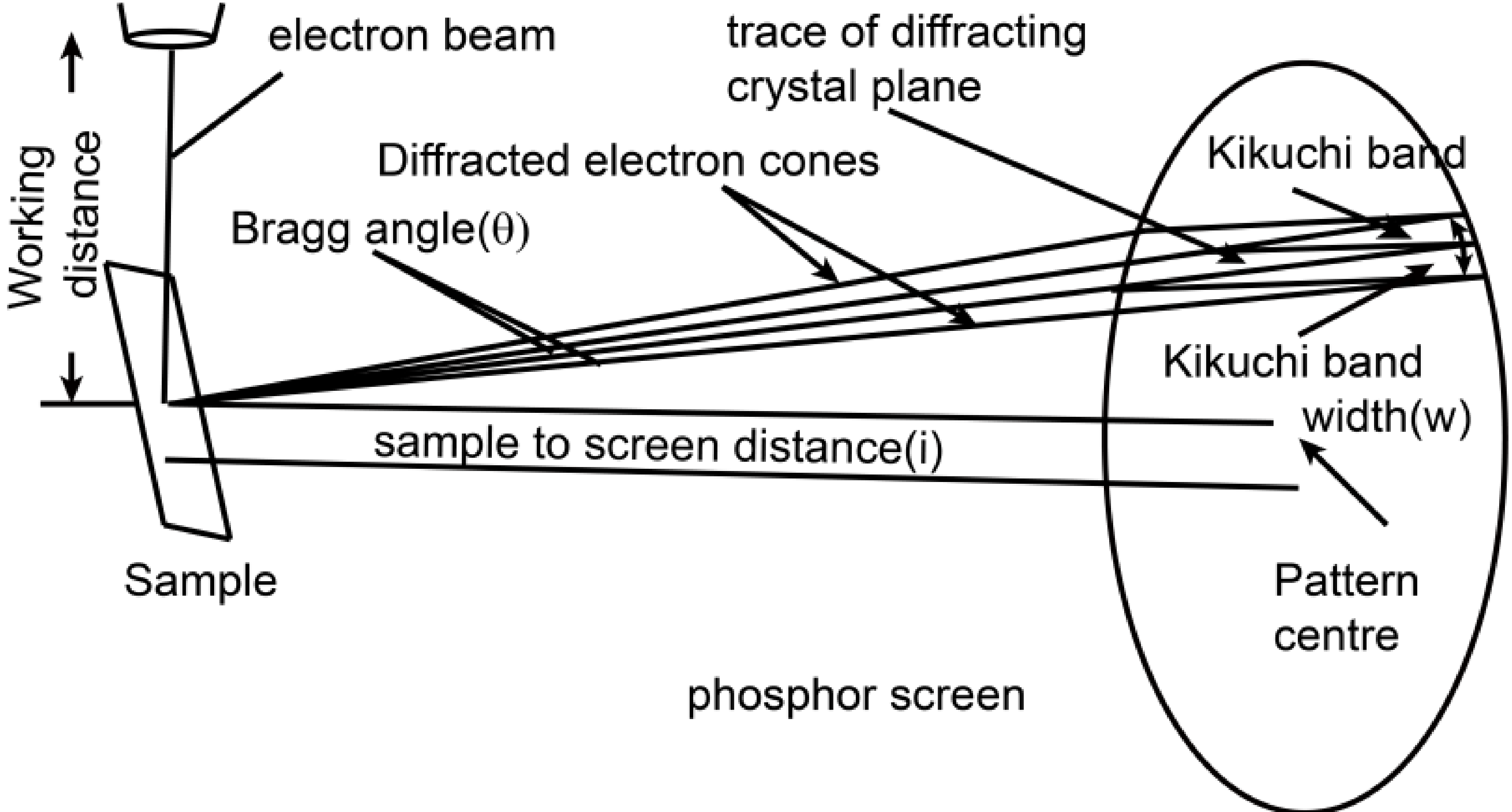
Because they originated from a different angle to that which they ended up at, the Bragg-reflected secondary waves are of different intensity than the background of secondary waves around them - they should be noticeable on the background of your diffraction pattern. But what as…?

- **The source of electron scattering can be considered to be between lattice planes–two cones of radiation result from each family of planes**



- **Bragg angle ~ 0.5 deg ⇒ apex angle of the cone 180 deg**

  ⇒ cones are almost flat



Interpretation of Kikuchi patterns

Interpretation of Kikuchi patterns involves two steps:

- **Determination of crystallographic indices of the Kikuchi bands or corresponding lattice plane**

- **Determination of relative position of bands or poles with reference to external frame of reference**

Micro-texture measurement in SEM-EBSD

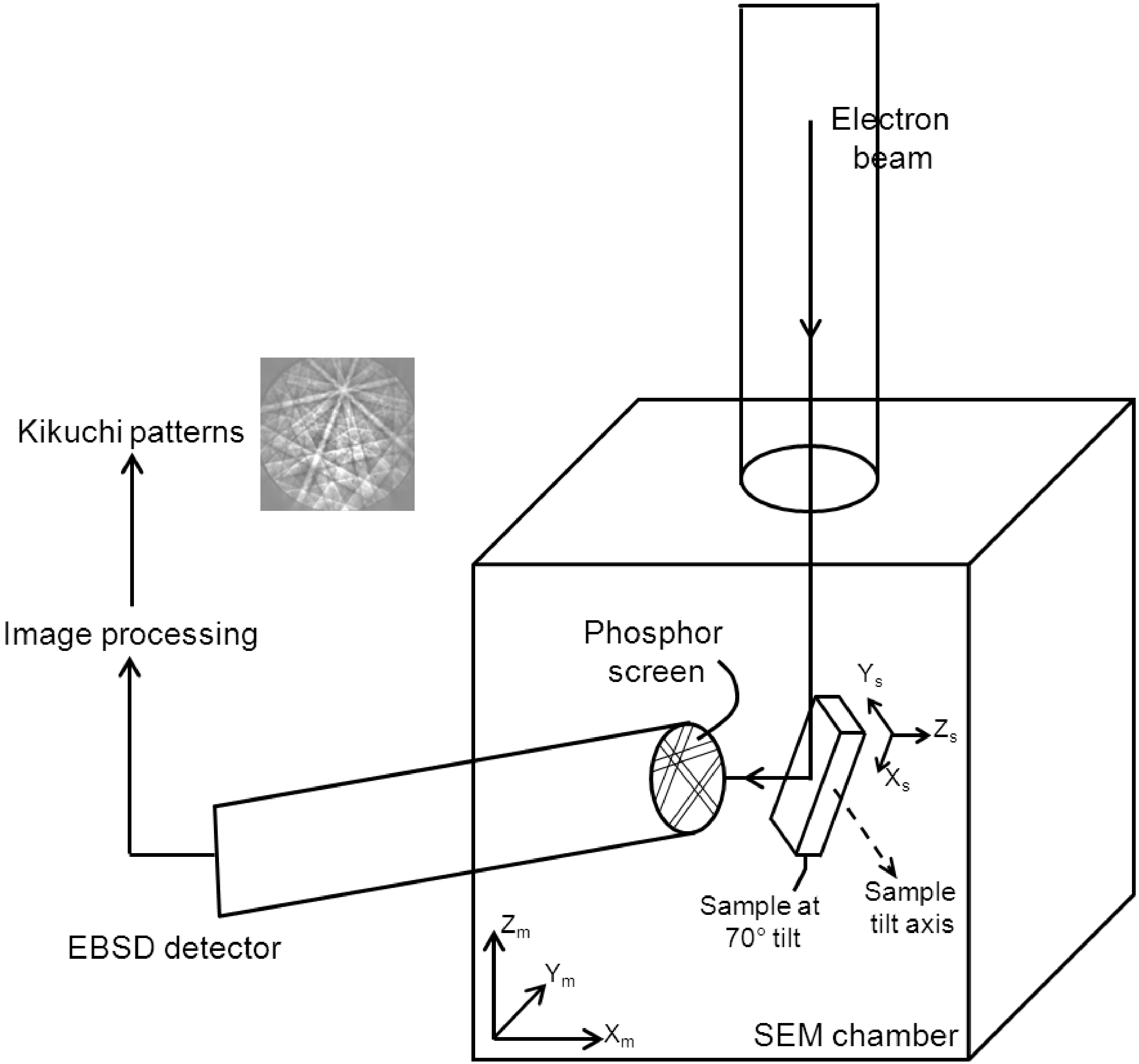
Principle of Pattern formation in EBSD

For EBSD, a beam of electrons is directed at a point of interest on a tilted crystalline sample in SEM

The following steps need to be followed to get the Kikuchi pattern:

- **Tilt the specimen to get optimum backscattered efficiency**
- **Stop the scanning of surface in SEM to make stationary electron beam**
- **Placement of a recording medium to record the diffracted backscattered electron in SEM**

Schematic of an SEM-EBSD set up



Electron beam

Kikuchi patterns

Image processing

Phosphor screen

Y_s

Z_s

X_s

EBSD detector

Sample at 70° tilt

Sample tilt axis

Z_m

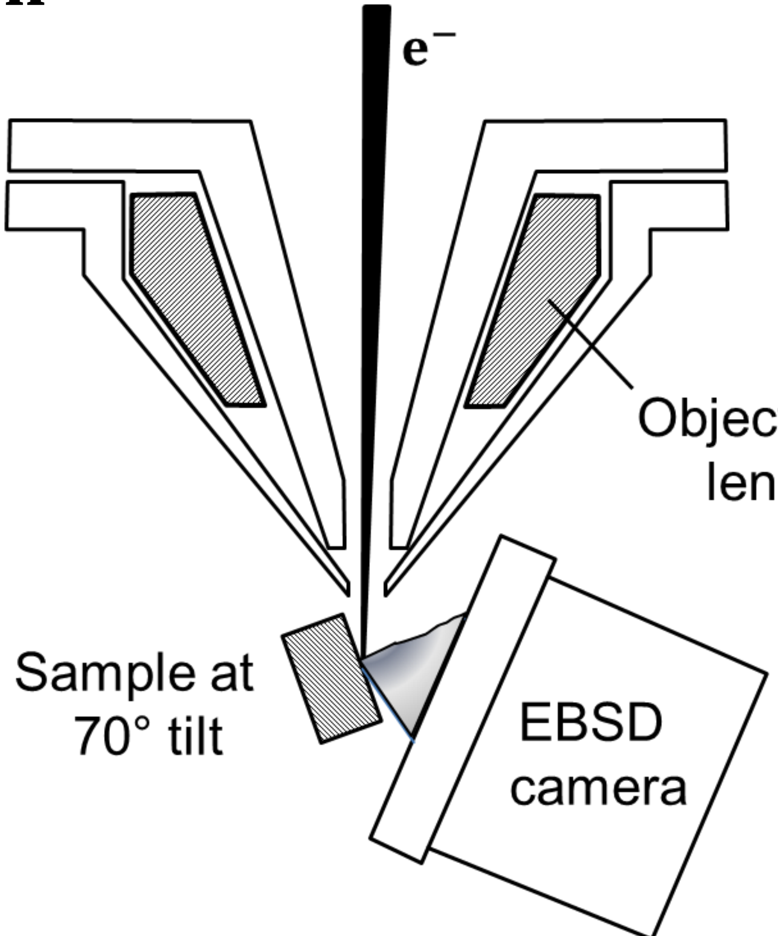
Y_m

X_m

SEM chamber

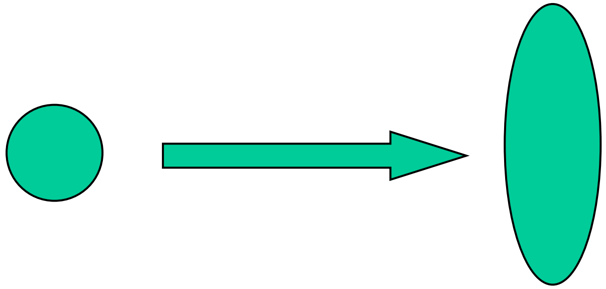
Tilting of sample in SEM for EBSD: It is Critical

It reduces the path length of backscattered electrons escaping from sample after diffraction thus enhance the data resolution

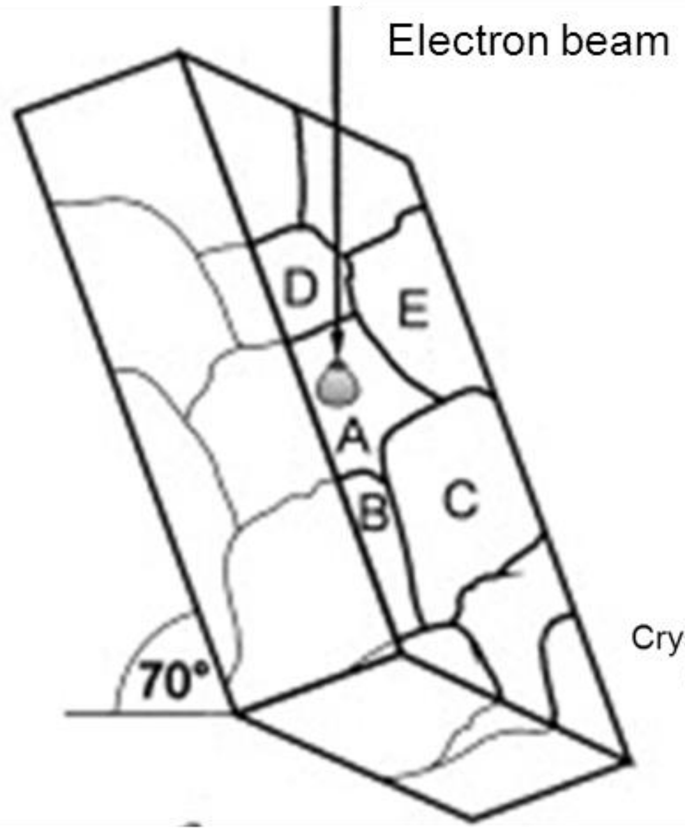


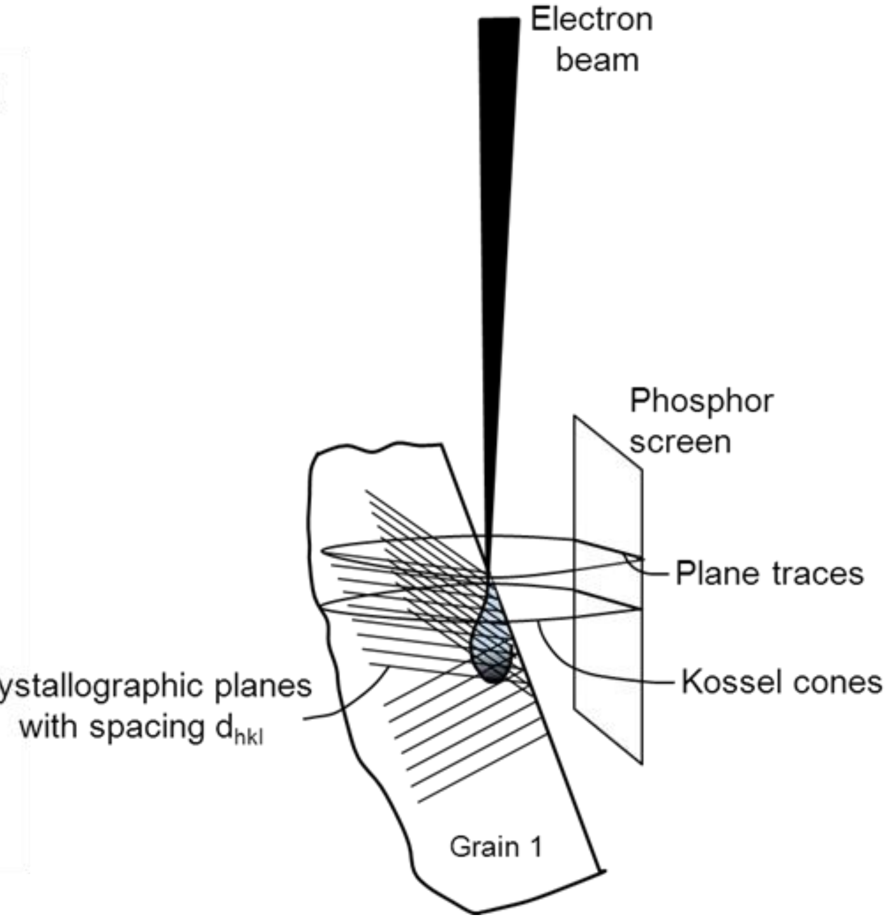
- The electron/sample interaction volume modifies from pear shape to shallow depth volume

- The beam circular sizes starts distorting to elliptical with the tilting of the sample thus reduces the resolution

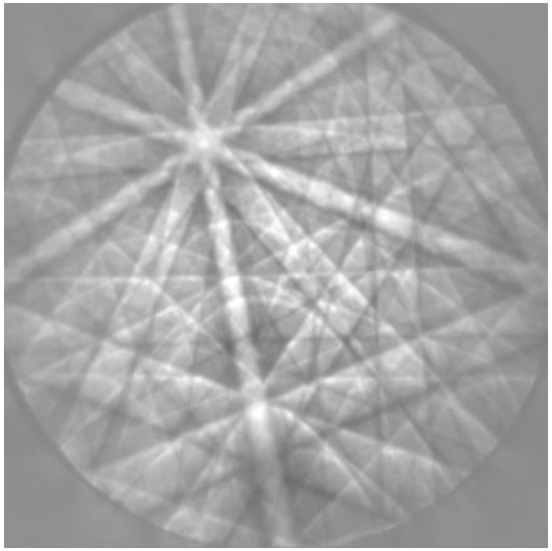


- Incident beam size : Tilting Increase ⇒ Bad resolution
  Optimum = 70º to the horizontal (Experimental optimization of efficiency and resolution





- **Because the diffraction pattern is bound to the crystal structure of the sample, as the crystal orientation changes the resultant diffraction pattern also changes**



- Intersections of the Kikuchi bands correspond to the intersection of zone axes in the crystal. These points can be labeled by the crystal direction for the zone axis

- The width w of the Kikuchi bands close to the pattern center is given by

$$w \simeq 2l\theta \simeq (nl\lambda/d)$$

where, l is the distance from the sample to the screen. Hence, planes with wide d-spacings give thinner Kikuchi bands than narrow planes

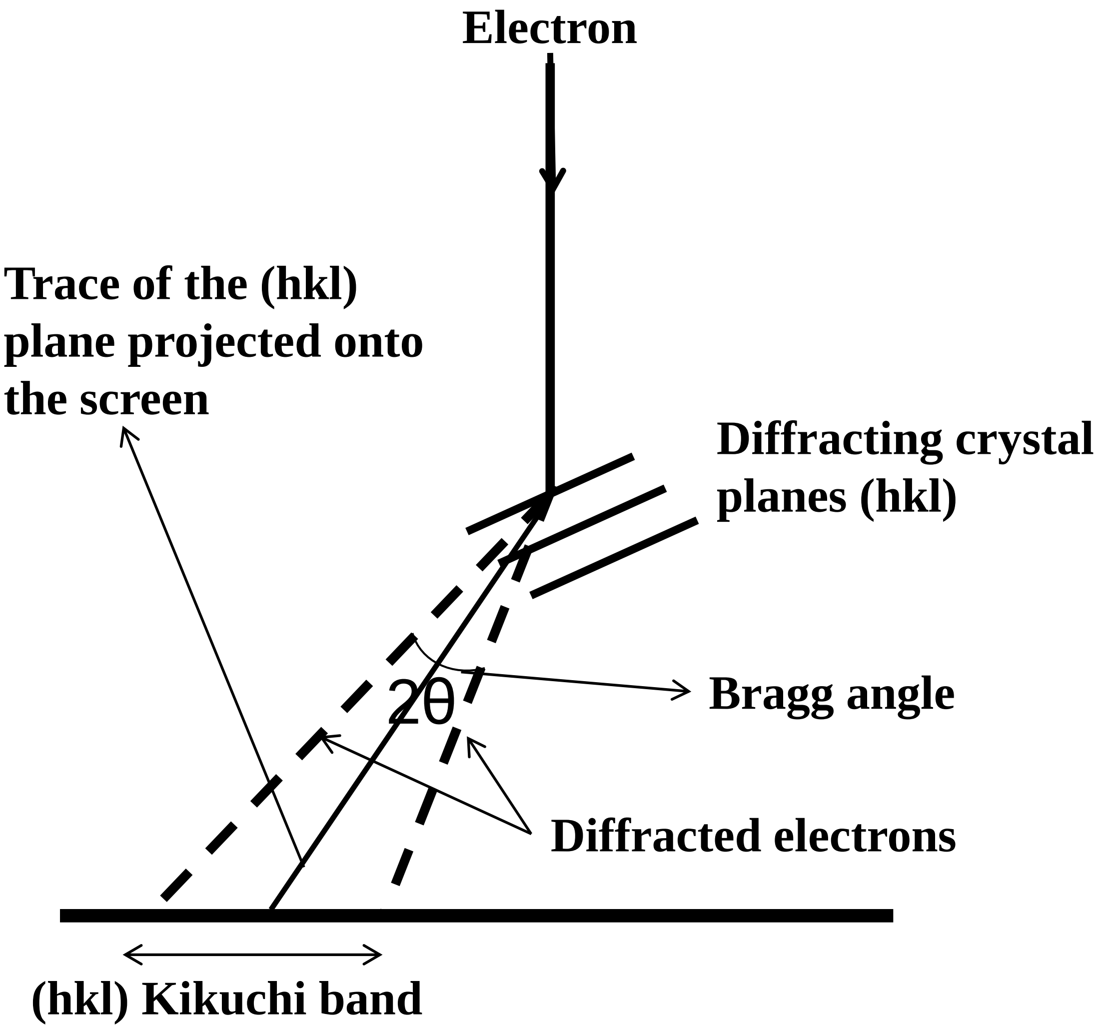
- Therefore, positions of the Kikuchi bands can be used to calculate the orientation of the diffracting crystal

Interpretation of EBSD pattern

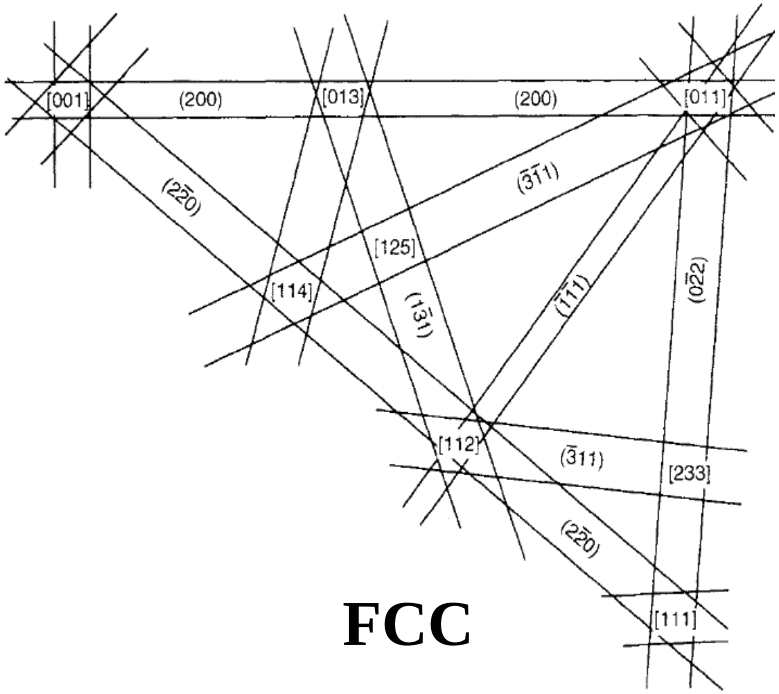
- **The centre lines of the Kikuchi bands correspond to the projection of the diffracting planes on the phosphor screen.**

- **Each Kikuchi band can be indexed by Miller indices of the diffracting crystal plane which formed it.**

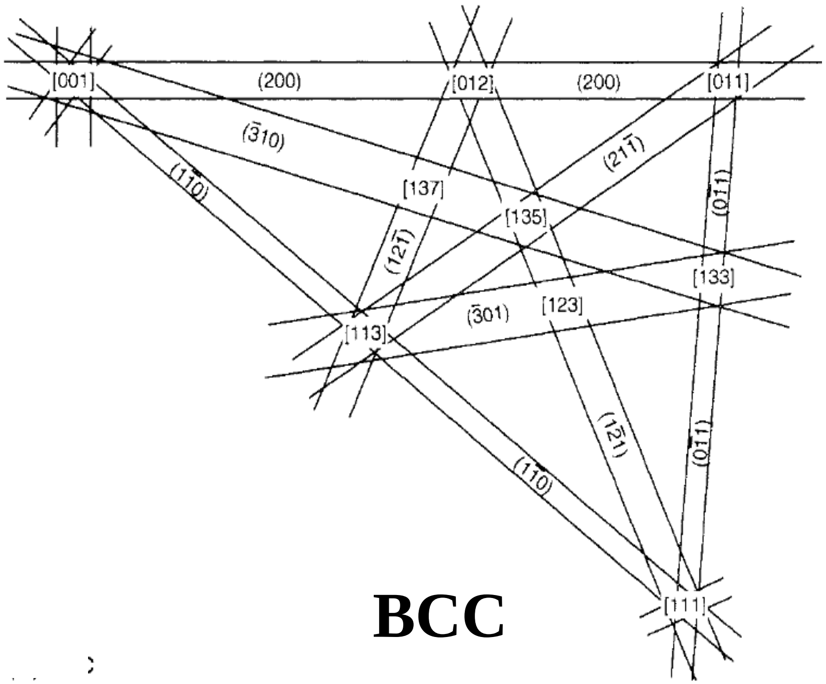
- **Each point on the phosphor screen corresponds to the intersection of a crystal direction with the screen.**



Representation of Kikuchi patterns (one stereographic triangle)



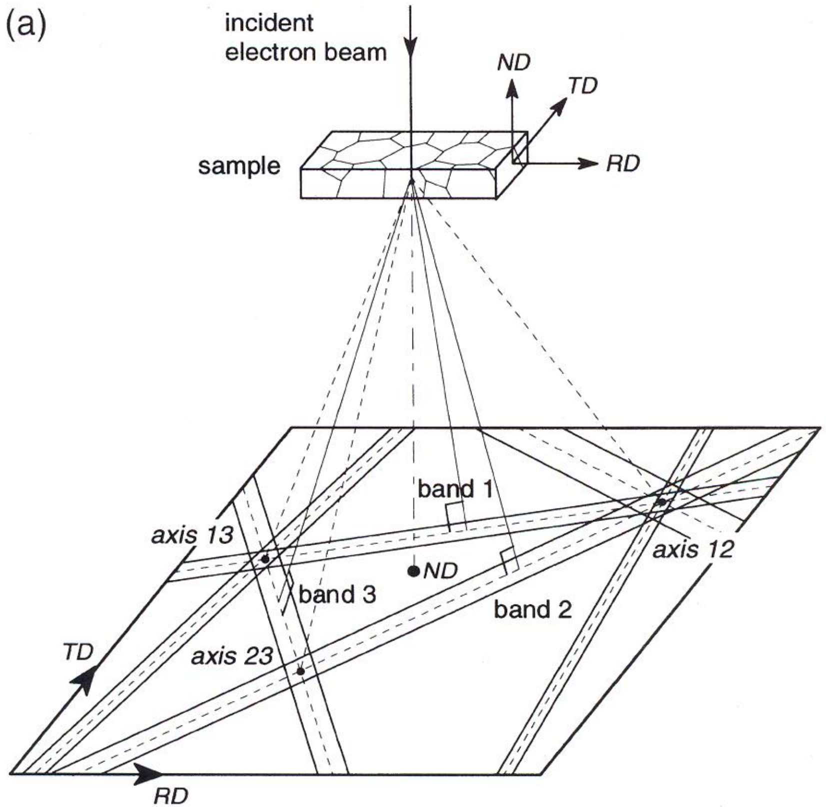
FCC



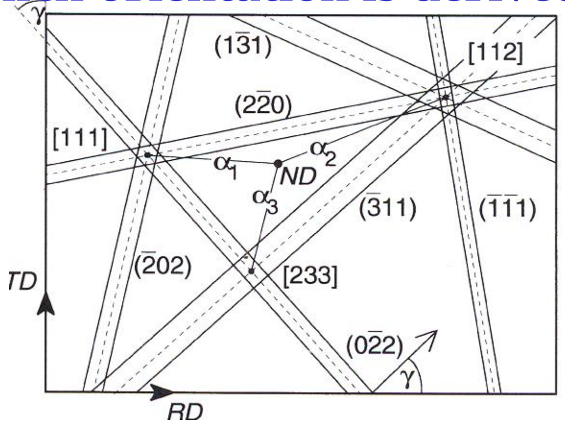
BCC

How to evaluate a Kikuchi pattern

Correlation between specimen frame and Kikuchi pattern



Indexed Kikuchi pattern from which orientation is derived



Evaluation of Kikuchi-Patterns

1. Indexing

bands (hkl) ⇔ zone axes (uvw)

(*uvw*) = (h_1,k_1,l_1) x (h_2,k_2,l_2) and vice versa

(non-cubic structures: multiplication with (inverse) crystal-matrix)

normalisation with known zone axes or band width 2θ

2. Determination of beam normal (BN) *hkl*

q_i are the crystallographic indices for the bands

known: zones *or* zone axes

angles between q^i and BN *hkl* α^i $q^i = [q_1^i, q_2^i, q_3^i]$

The orientation of the sample normal in terms of its crystallographic indices (hkl) →

$$\begin{pmatrix} q_1^i \\ q_2^i \\ q_3^i \end{pmatrix} \cdot \begin{pmatrix} h \\ k \\ l \end{pmatrix} = \cos\alpha_i \cdot \sqrt{(q_1^i)^2 + (q_2^i)^2 + (q_3^i)^2}$$

i =1, 2, 3: 3 equations with 3 unknown ⇒ *hkl*

Here i= 1,.......,n , where n is the number of identified bands or poles, α_i is the angle between each band and the beam norma

<u>example</u>: α_1=8.5°, α_2=11.6°, α_3=7.2° ⇒ (*hkl*) = (0.486, 0.541, 0.686) ≈ (567)

The root term in the RHS of the equation normalises the indices of the bands or axes
(e.g. (022) or [112]) to unity.

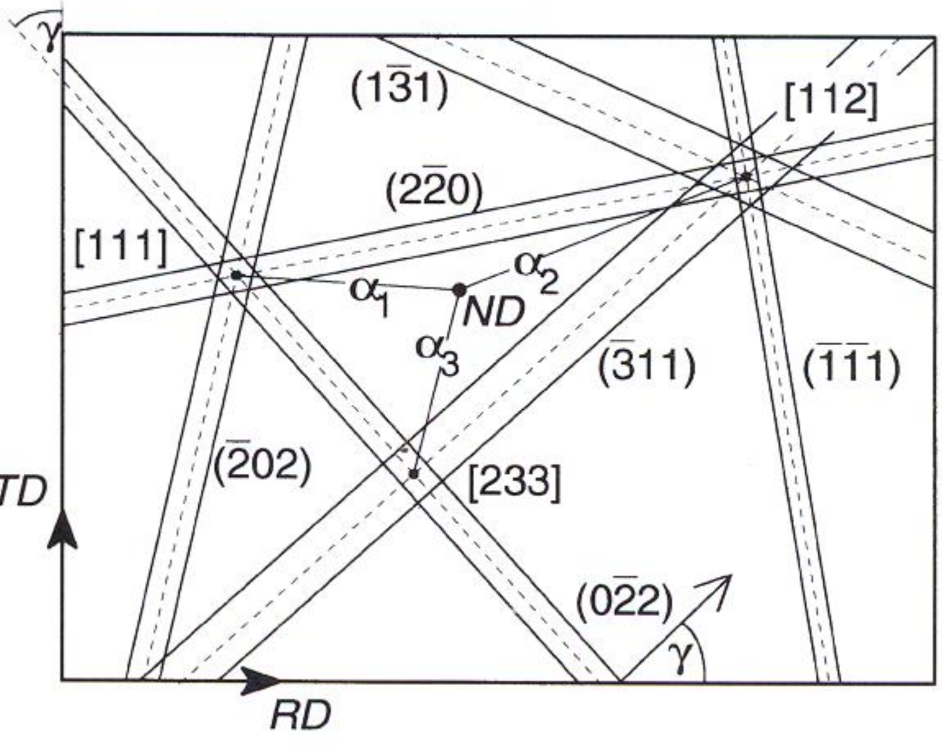
Hence the resulting vector (hkl) is directly normalised to unity.

3 equations are necessary to determine (hkl)
3 bands or poles are to be indexed.

How to determine the direction?

If the direction of the reference axis in the specimen surface—the edge of the photographic plate or the rolling direction—is known in the final Kikuchi pattern,
→ its crystallographic indices [*uvw*] can be determined by tilting the sample around this axis and determining the new beam normal $(h'k'l')$.

Then, $[uvw] = (hkl) \times (h'k'l')$.



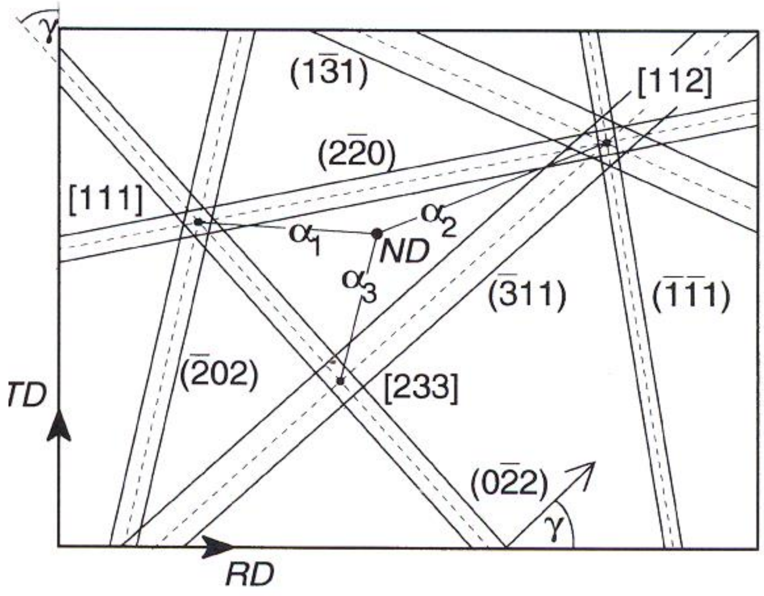
Define an intermediate pattern frame with:

- ***Z* axis || beam normal *BN***
- ***Y* axis || any of the (indexed) Kikuchi bands with the normal *hkl*, which means *Y* is given by *BN* × *hkl*.**

- **Thus, the rotation matrix R_{CP} from the crystal frame *C* to the intermediate pattern frame *P***

$$R_{CP} = \left((BN \times hkl) \times BN \quad BN \times hkl \quad BN \right)$$

- **Pattern frame *P* contains the information of the indexed beam normal *BN***
- **Sample frame *S differs from P*, by a rotation of angle γ around *BN* = *z*,**
  γ = angle between the Kikuchi band and the sample direction *Y*
  = angle between the *normal* to that Kikuchi band and sample direction *X*

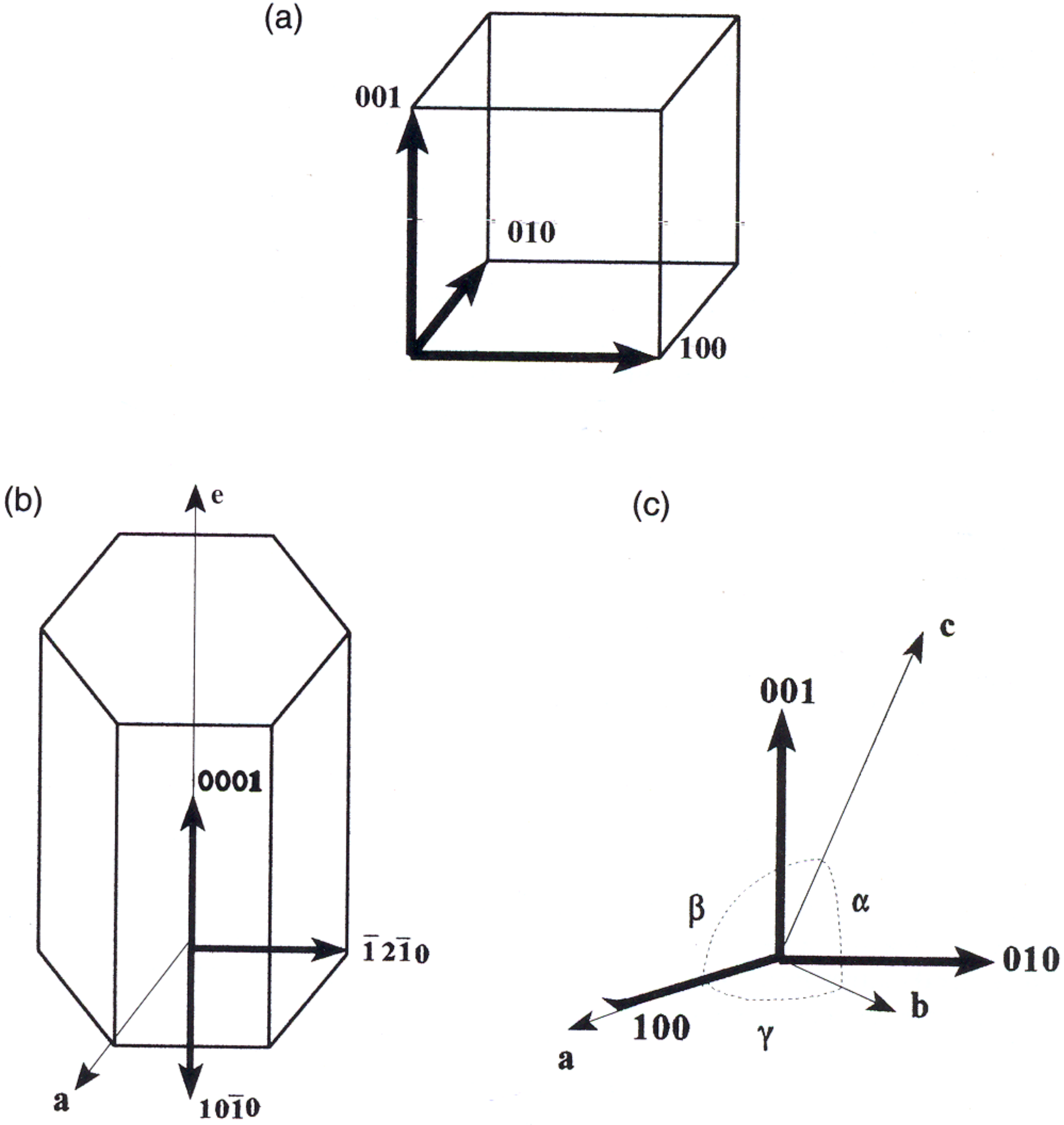


- **Matrix R_{PS} for a rotation by angle γ about *BN* = *z* is**

$$R_{PS} = \begin{pmatrix} \cos\gamma & -\sin\gamma & 0 \\ \sin\gamma & \cos\gamma & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

Combination of the two rotation matrices R_{CP} and R_{PS} yields the rotation R_{CS} between the crystal and the sample frame, that is, the desired orientation matrix g:

$$\boxed{g = R_{CS} = R_{PS} \cdot R_{CP}^{T}}$$



1 Orthonormalised crystal coordinate systems for (a) cubic; (b) hexagonal and (c) general (triclinic) symmetries.

For hexagonal symmetry, an orthogonal frame needs to be associated with the crystal axes:

Two choices

$$X_1 = [10\bar{1}0], \quad Y_1 = [\bar{1}2\bar{1}0], \quad Z = [0001]$$

$$X_2 = [2\bar{1}\bar{1}0], \quad Y_2 = [01\bar{1}0], \quad Z = [0001]$$

Crystal axes are to be made orthonormal - normalised to be all in same length

This is done by pre-multiplying a zone axis by matrix L,

$$L = \begin{pmatrix} l_{11} & l_{12} & l_{13} \\ l_{21} & l_{22} & l_{23} \\ l_{31} & l_{32} & l_{33} \end{pmatrix}$$

where,

$$l_{11} = a$$
$$l_{12} = b\cos\gamma$$
$$l_{13} = c\cos\beta$$
$$l_{21} = 0$$
$$l_{22} = b\sin\gamma$$
$$l_{23} = c(\cos\alpha - \cos\beta\cos\gamma)/\sin\gamma$$
$$l_{31} = 0$$
$$l_{32} = 0$$
$$l_{33} = c(1 + 2\cos\alpha\cos\beta\cos\gamma - \cos^2\alpha - \cos^2\beta - \cos^2\gamma)^{1/2}/\sin\gamma$$

(Young and Litton, 1972, J. Appl. Phys. 43, 1408)

where, a, b, c are lattice parameters and α, β, γ are interzonal angles

For hexagonal crystals,

$\alpha = 90°$
$\beta = 90°$
$\gamma = 120°$

$$L = \begin{pmatrix} a & -a/2 & 0 \\ 0 & (a\sqrt{3})/2 & 0 \\ 0 & 0 & c \end{pmatrix}$$

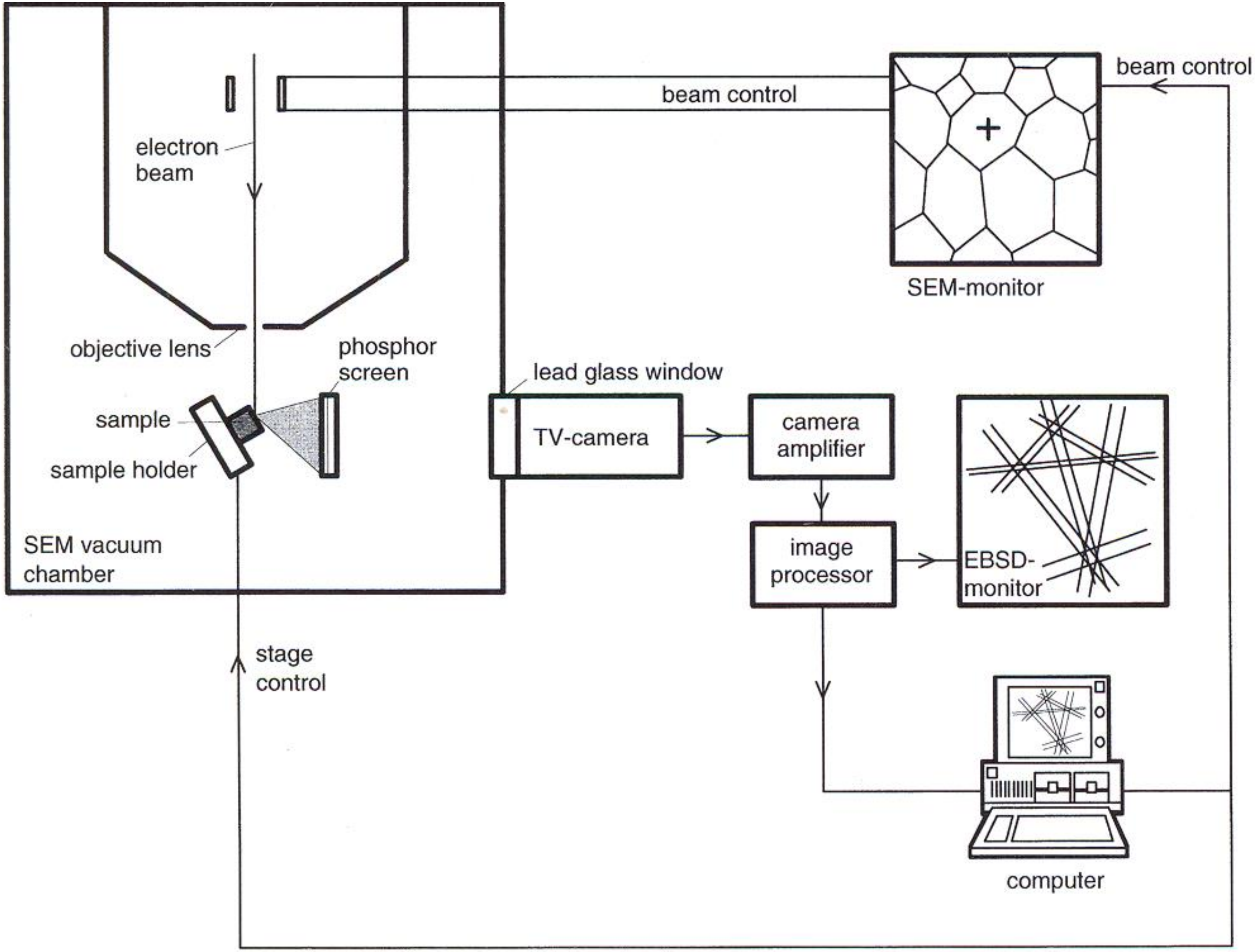


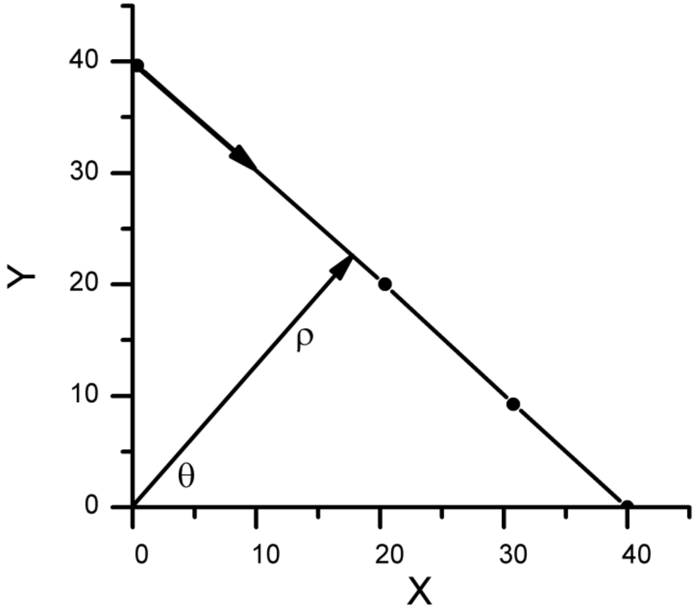
Figure 7.7 Components of an EBSD system.

Automated indexing and orientation measurement

- **Crystal orientation is calculated from the Kikuchi band positions by the computer processing the digitized diffraction pattern collected by the CCD camera.**

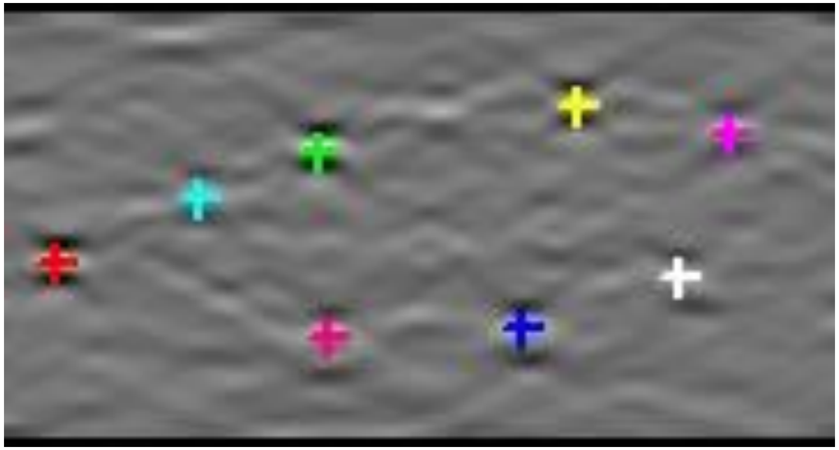
- **The Kikuchi band positions are found using the Hough transform. The transform between the coordinates (x,y) of the diffraction pattern and the coordinates (ρ, θ) of Hough space is given by**

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



- A straight line is characterized by
  (i) the perpendicular distance from the origin (ρ), and
  (ii) the angle made with the x-axis (θ)

- Therefore a line is represented by a single point (ρ, θ) in Hough space. Kikuchi bands transform to bright regions in Hough space which can be detected and used to calculate the original positions of bands.



- **Using the system calibration, the angles between the planes producing the detected Kikuchi bands can be calculated.**

- **These are compared with a list of inter-planar angles for the analyzed crystal structure to allocate Miller indices to each plane.**