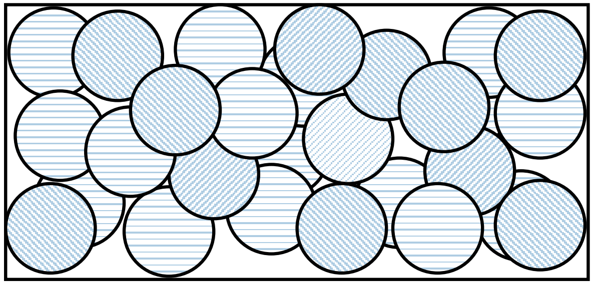
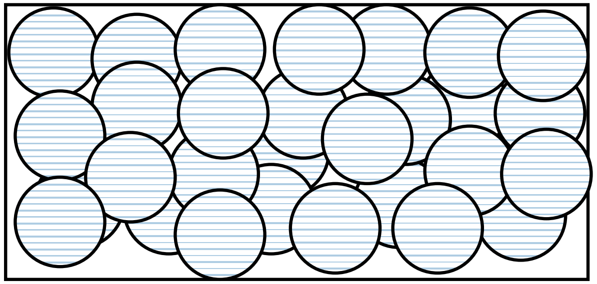
X-ray
Pole figure measurement

Orientations of polycrystalline materials

Polycrystalline materials are composed of millions of crystal grains.



(a) In random orientation



(b) In preferred orientation

When crystals are oriented as shown in Fig (b), a polycrystalline material is said to have *preferred orientation*.

- **Textured material** is a material in which the individual crystallites occupy preferential orientations
- **X-ray Bragg diffraction** is a most appropriate tool for texture analysis; the second important tool is **Electron Backscatter Diffraction** (EBSD)
- The preferred orientation is usually described in terms of **pole figures** (PF) or **inversed pole figures** (iPF) and **orientation distribution function** (ODF)

Texture origin

- Texture **presents** in polycrystalline materials: metals, alloys, inter-metallics, ceramics, composite materials, polymers, semiconductors, nano-crystals, superconductors, minerals
- Texture is **caused** by mechanical deformations (rolling), recrystallization, phase transformations, film synthesis with plasma, laser and ion beams, crystallisation to boundary surfaces, rigid particle rotation, quenching, cold work, annealing, casting or other fluid-to-solid transitions
- Texture **influences** the physical properties of materials: plasticity, elasticity, strength, cleavability, electrical conductivity, thermal extension and conductivity magnetization, corrosion resistance, optical properties, strength, chemical reactivity, stress corrosion cracking resistance, weldability, deformation behavior, resistance to radiation damage, magnetic susceptibility
- Pronounced textures **occur** in thin films, which are intensively used in modern technological devices and to a large extent rely on polycrystalline thin films with thicknesses in the nanometer and micrometer ranges. This is applied in all microelectronic and most optoelectronic systems or sensorics and superconducting layers, protection coatings, internal microchip wiring

Texture and pole figures

Nonoriented sample：brick, concrete, (powder)

Oriented sample：aluminium foil,
iron plate
plastic bottle

Single crystal：silicon wafer, diamond, quartz

Pole figures

- The projection of all plane's normal for the plane (hkl) from all the crystallites irradiated in the sample onto the sphere makes a map on sphere
- Each plane normal intercepting the sphere represents a point on the sphere. These points in return represent the Poles for the planes (hkl) in the crystallites. The number of points per unit area of the sphere represents the pole density I_{hkl} (α, β); where α *and* β are Eulerian angles on sphere
- The I_{hkl} (α, β) map is named an intensity Pole Figure
- There are different types of projections used in texture analysis:
  - Orthographic
  - Stereographic
  - Equal area
  - *etc.*

Pole figures

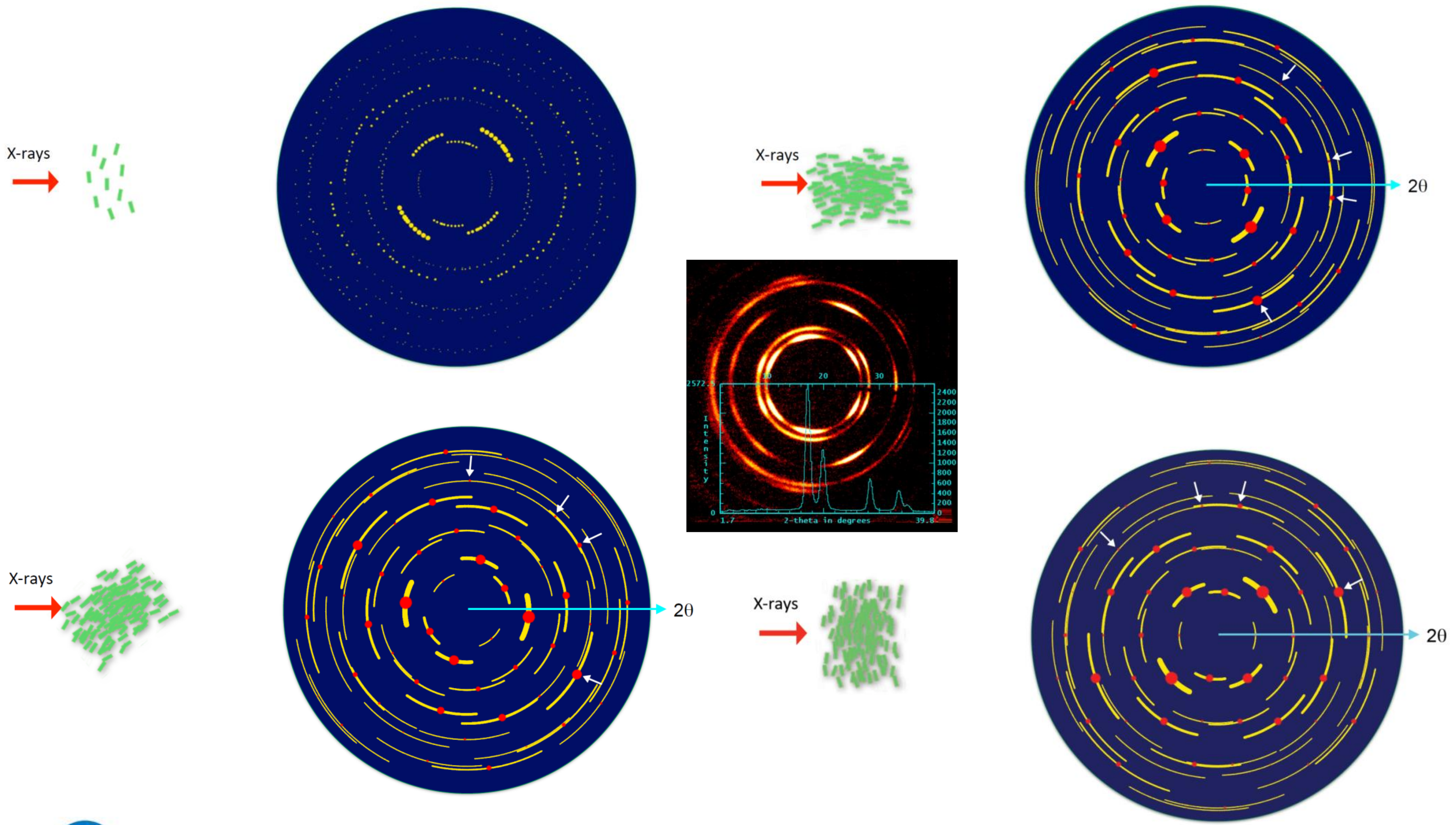
- A **pole figure** is scanned by measuring the diffraction intensity of a given reflection with constant 2θ at a large number of different angular orientations of a sample
- A contour map of the intensity is plotted as a function of the **angular orientation** of the sample
- The intensity of a given reflection is proportional to the **number of *hkl*-planes** in reflecting condition
- The pole figure gives the **probability** of finding a given *(hkl)* plane normal as a function of the sample orientation
- If the crystallites in the sample have **random orientation** the contour map will have uniform intensity contours
- The **intensity** on pole figure must be corrected for:
    - absorption
    - defocusing
    - background
    - variable diffracting volume (for grazing angles)

PF measurement

Absorption correction is necessary in texture evaluation because of variation of measurement angles changes the penetration depth and volume of the sample contributing to measured X-ray intensity (**gauge volume**).

The density and location of the measurement points in PF plays important role in reliability of results and optimization of measurement time. Poorly gridded measurements deliver incomplete or wrong texture evaluation, whereas too dense measurement grid is very time consuming.

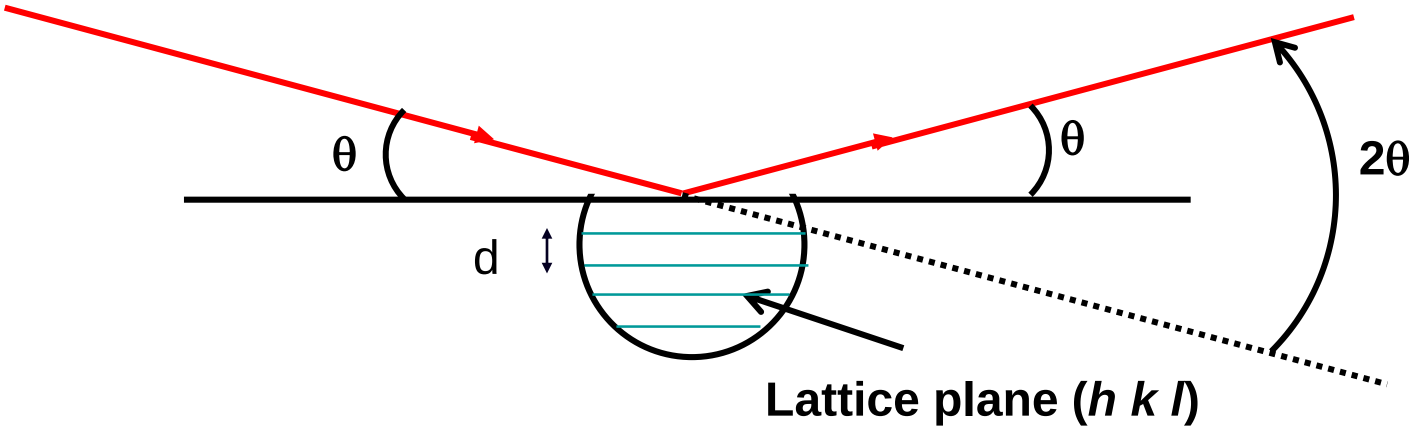
Texture & X-ray diffraction



X-ray diffracted by crystal

- The conditions for a reflected (diffracted) beam are given by the relation

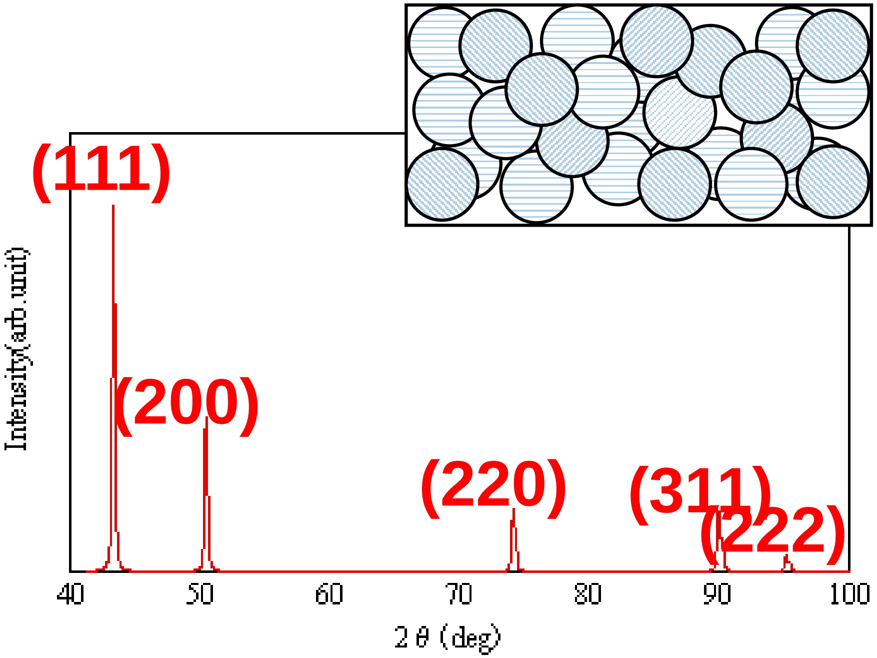
$$2d\sin\theta = n\lambda$$ ***Bragg's equation***



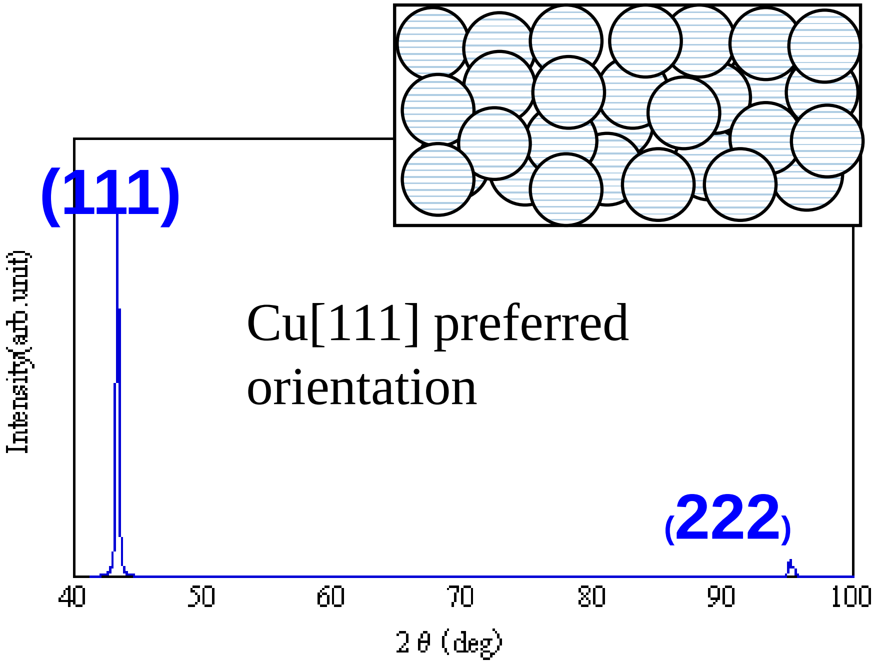
d : Interplanar spacing λ : X-ray wavelength

θ : Bragg angle n : 1,2,3･･･

XRD pattern of crystalline materials

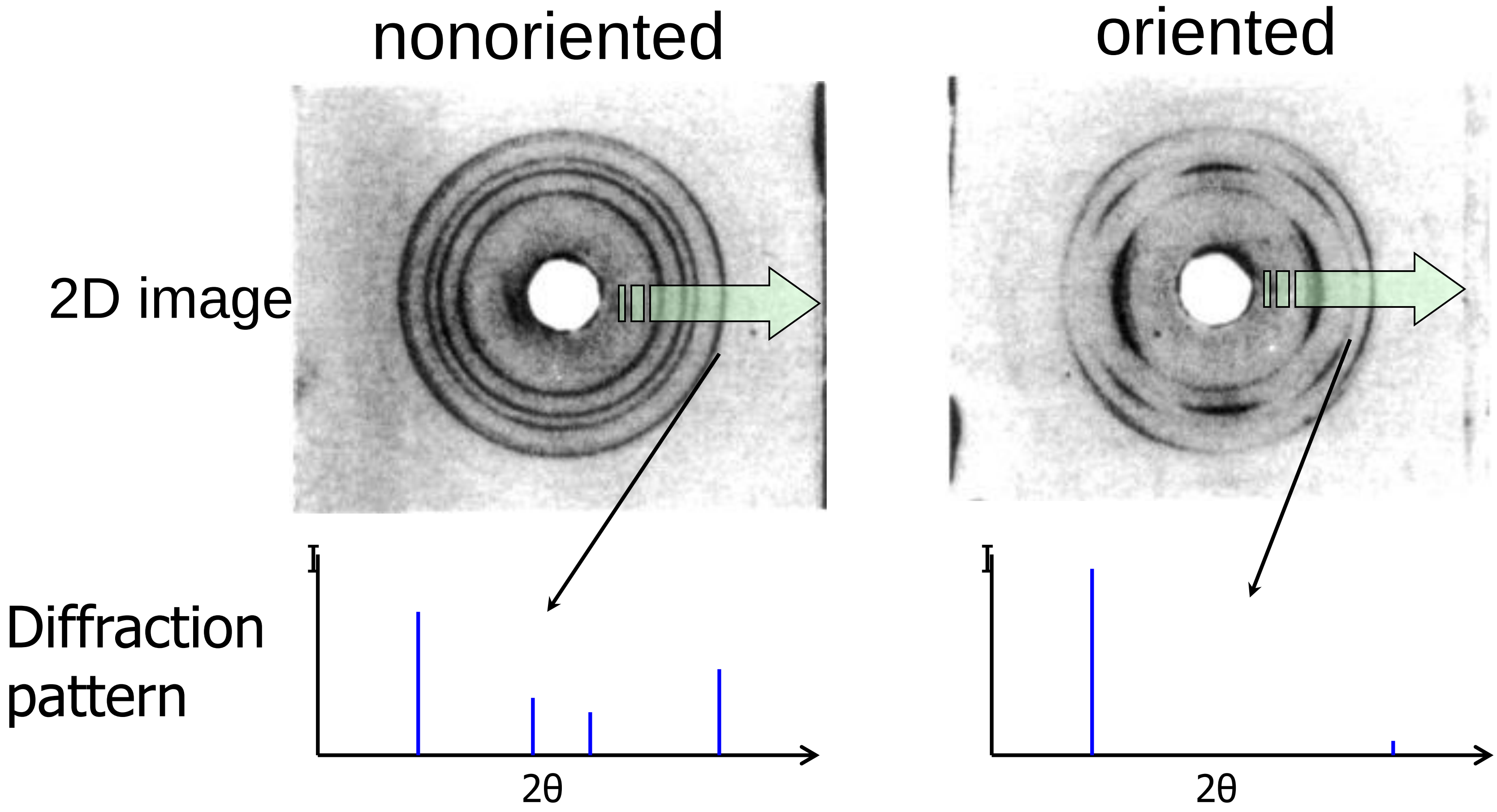


(a) Random orientation



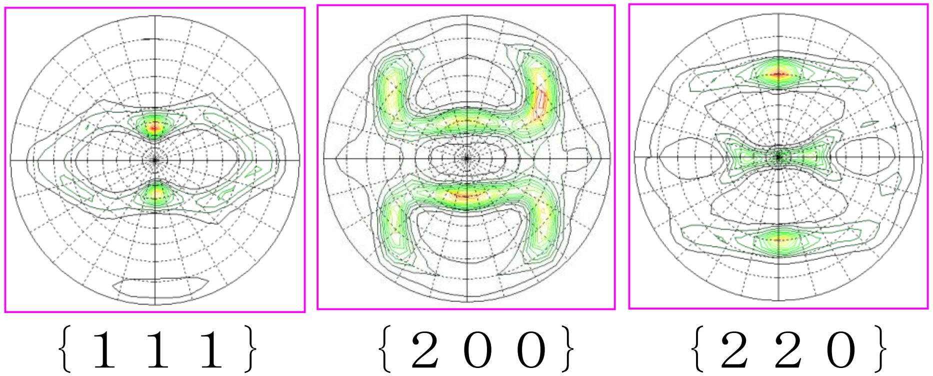
(b) Preferred orientation

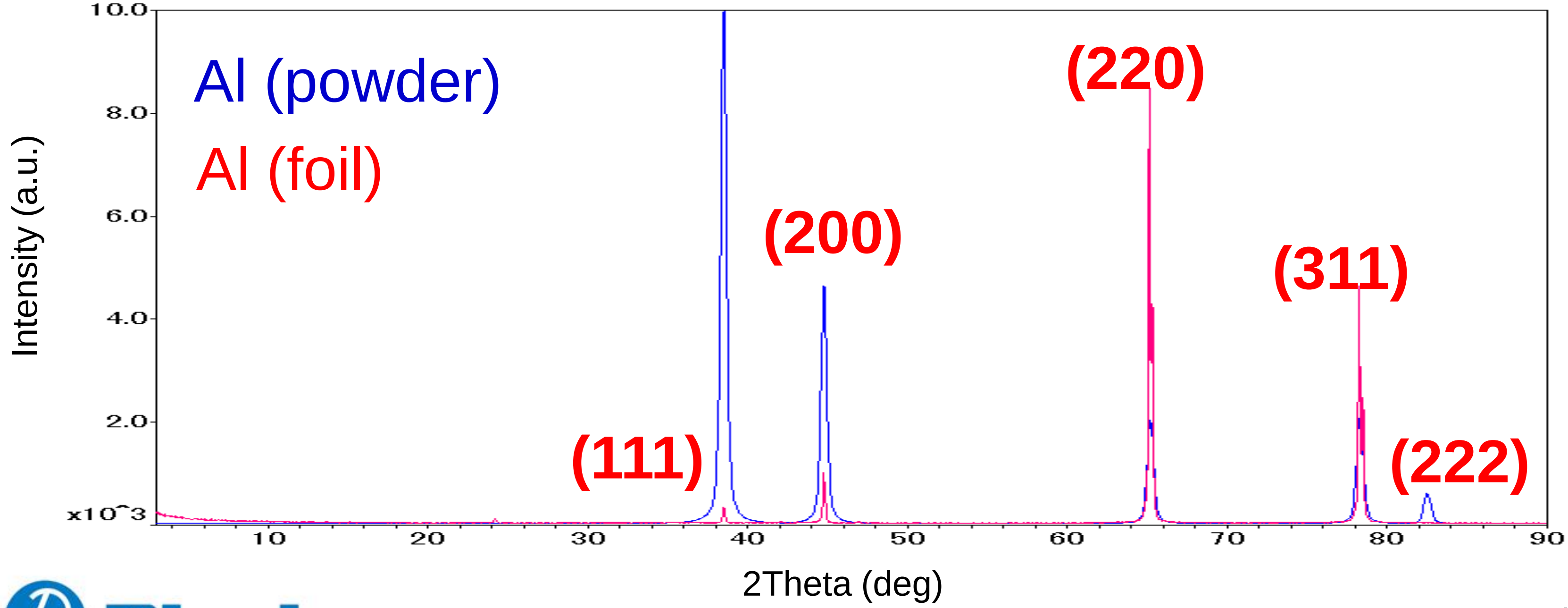
2D image and 2θ-I diffraction pattern



Texture and pole figures

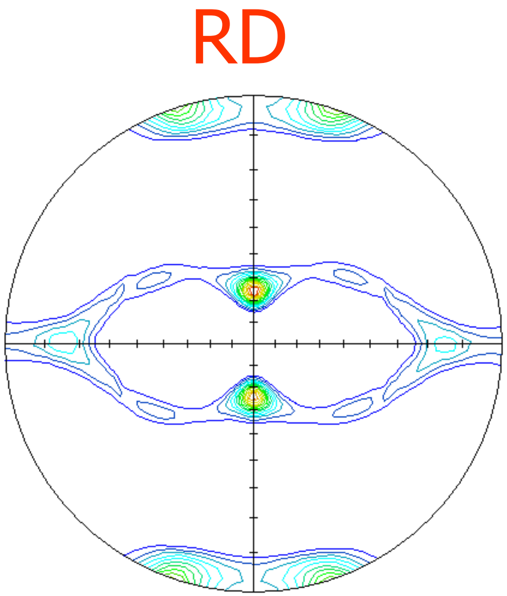
Pole figures

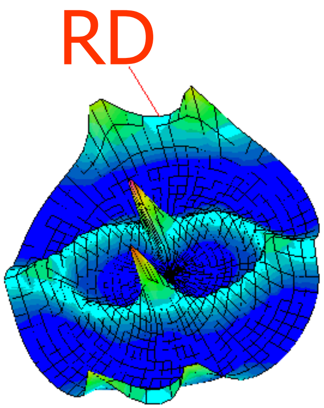


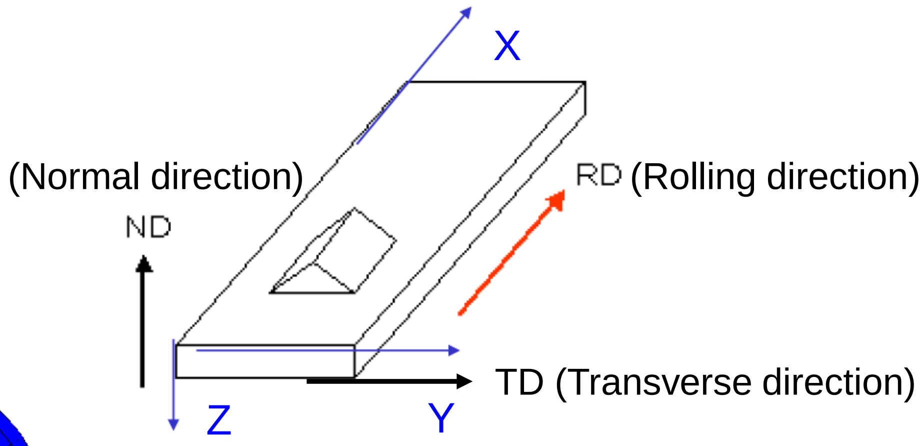


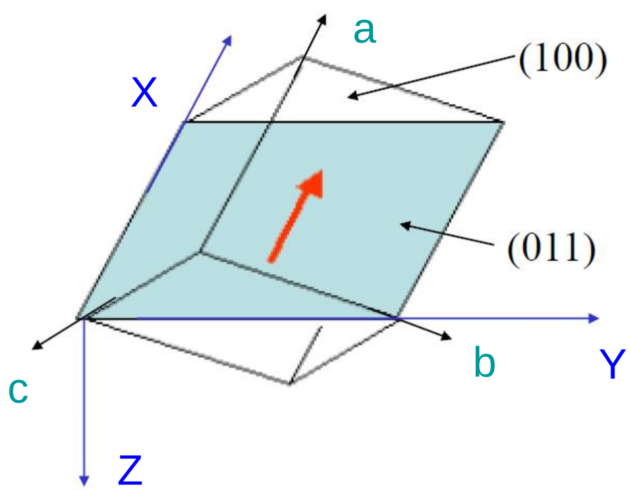
What is the purpose of texture?

Which plane is faced to Rolling direction and Normal direction?



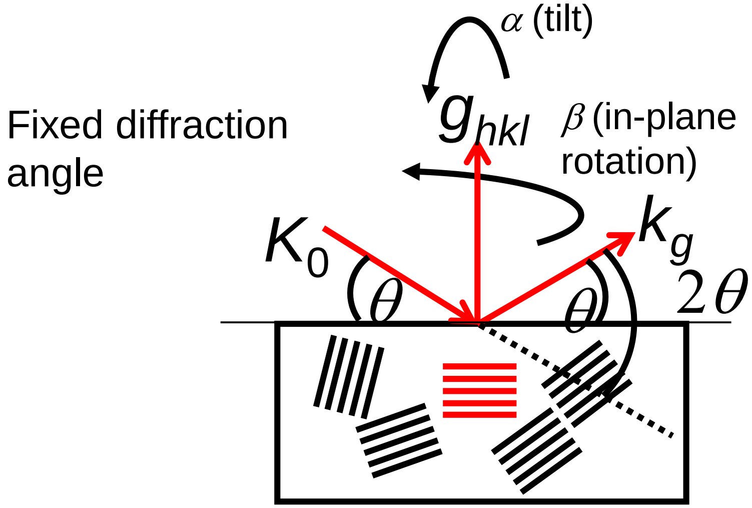




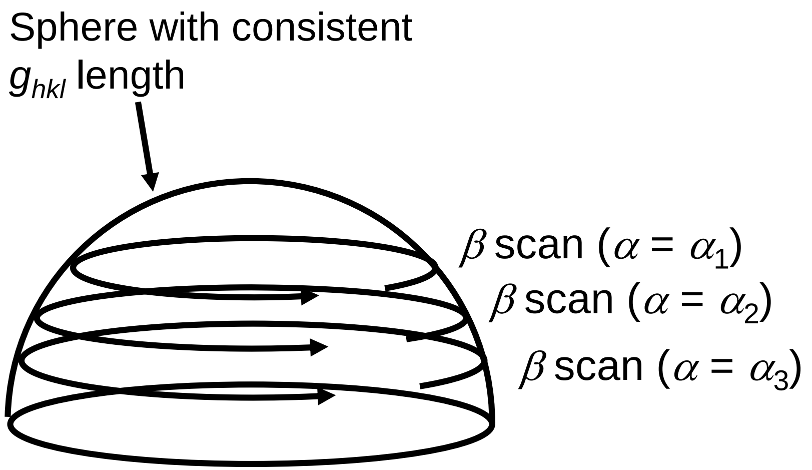


Pole figure measurements

- PF measured with at least 4-axis goniometer: θ, 2θ, φ, χ (or α, β)
- 2 axes (θ and 2θ) are used to set Bragg angle θ_B, which determines the Miller indices of Pole Figure, they remain fixed during the measurements of PF (hkl)
- Third axis χ (or α) tilts specimen plane (latitude angle in the PF, or distance from north pole)
- Fourth axis φ (or β) spins the specimen about its normal (longitude angle in the PF)
- For texture calculation, at least 2 PFs required and 3 are preferable



(a) Scan directions of α and β preferred orientation axes

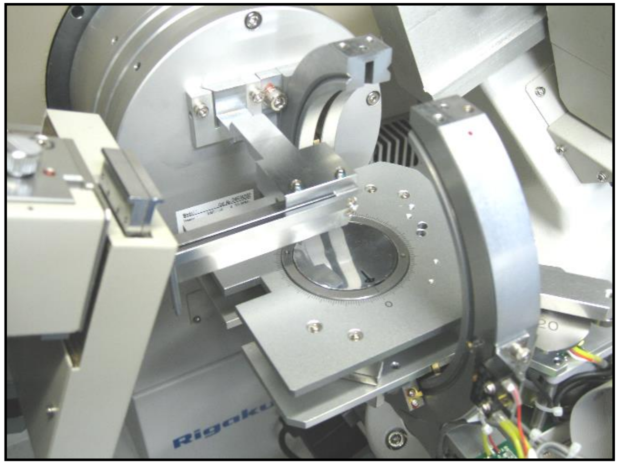


(b) Pole figure measurement method

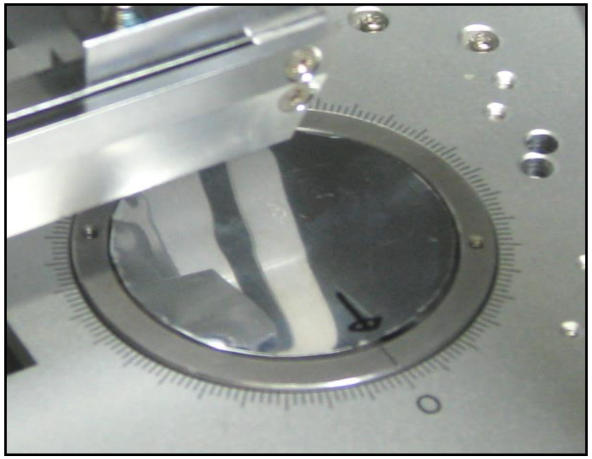
Multipurpose attachment (4 axises)

Reflection method arrangement

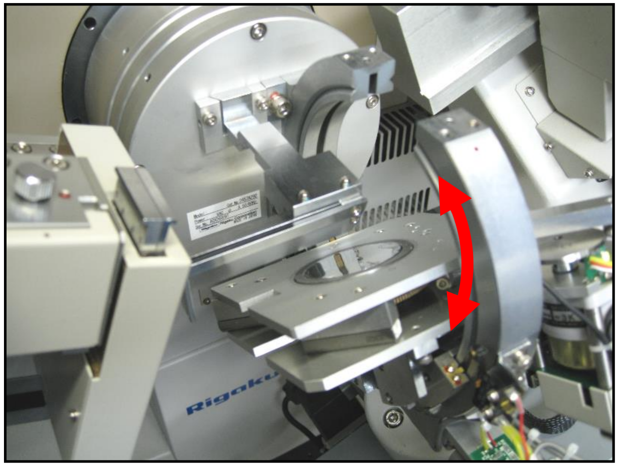
α:0deg



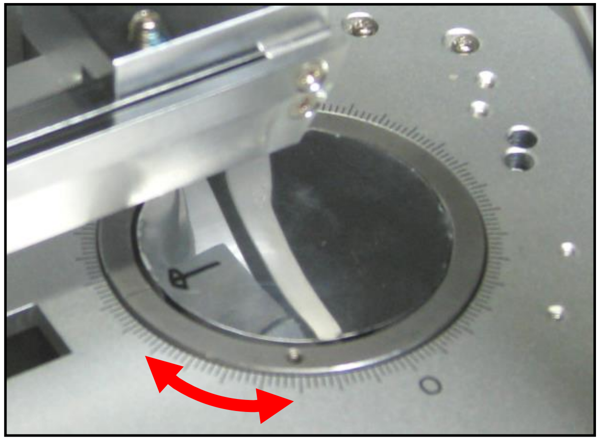
β:0deg



α:55deg

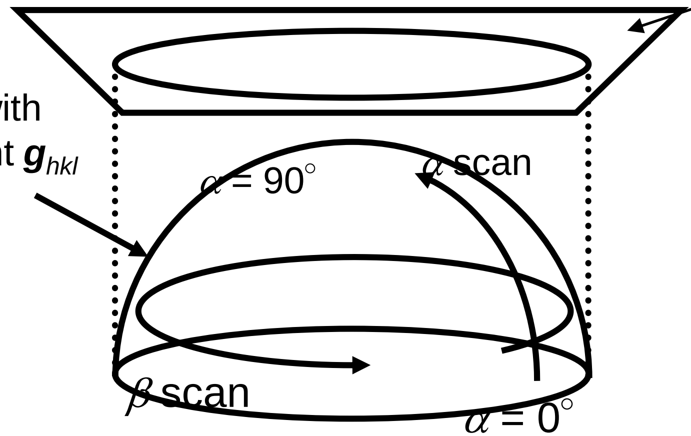


β:90deg

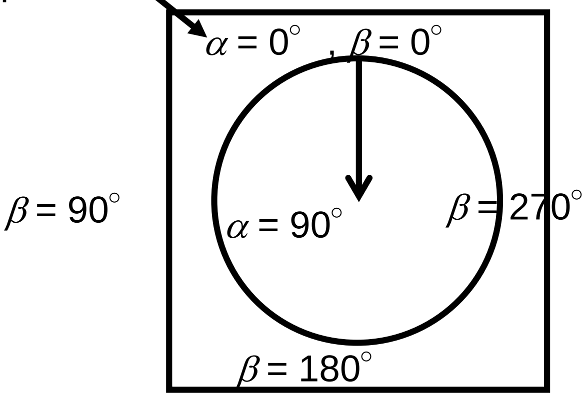


Pole figure

The measurement result is expressed by a pole figure

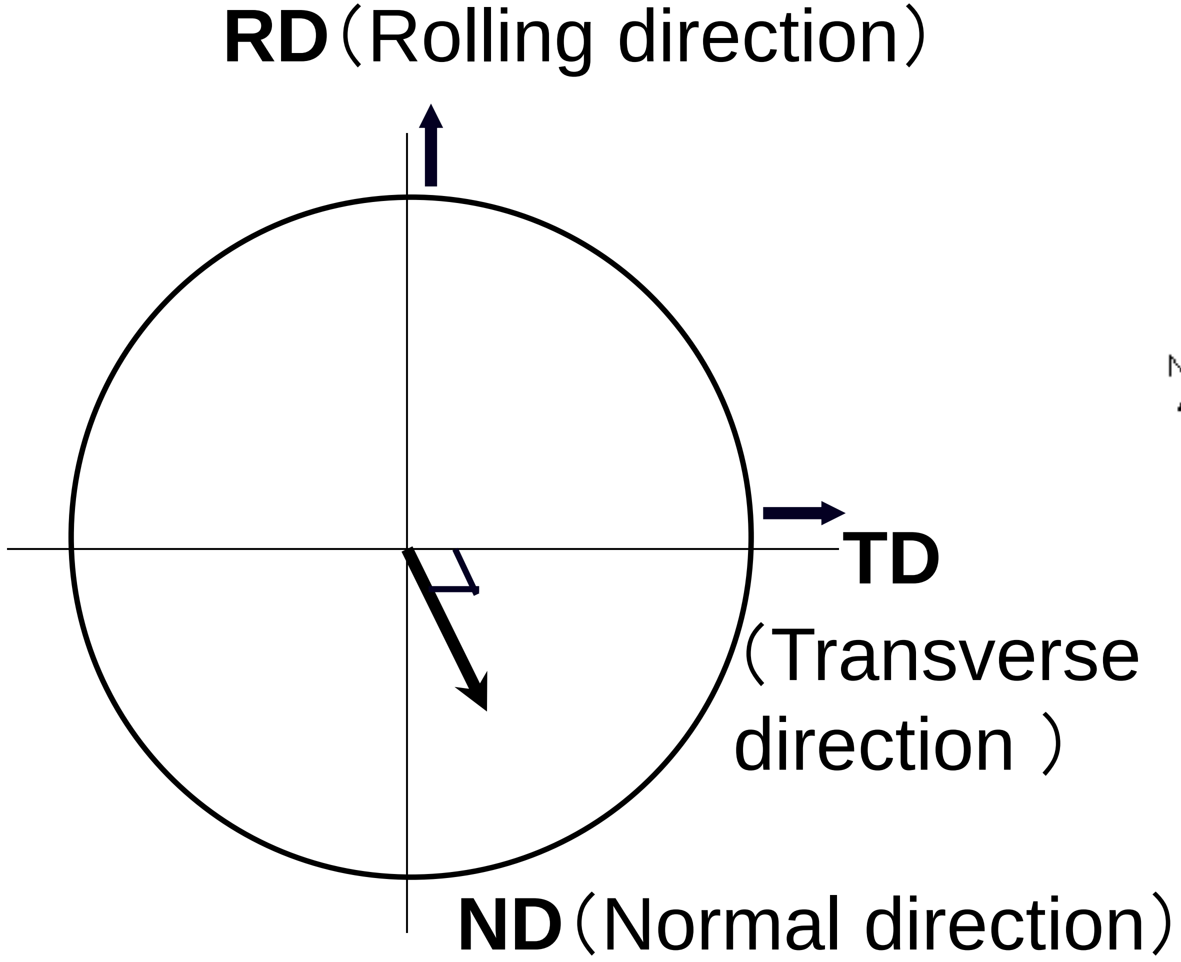


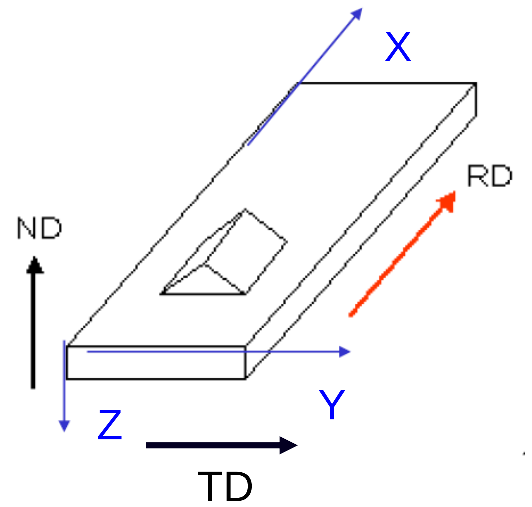
Pole figure measurement (reciprocal space)



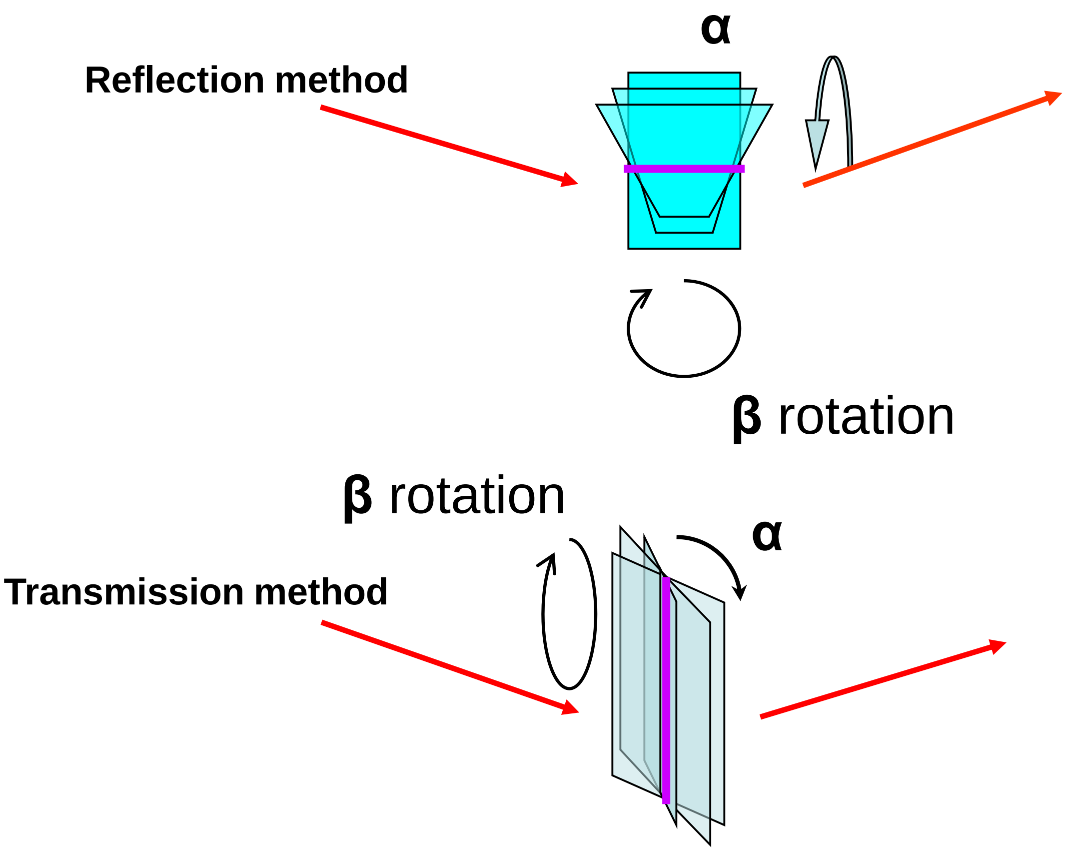
Pole figure

Pole figure


RD（Rolling direction）
TD
（Transverse
direction ）
ND（Normal direction）


X
RD
ND
Z
Y
TD

Sample behavior in Reflection method and Transmission method



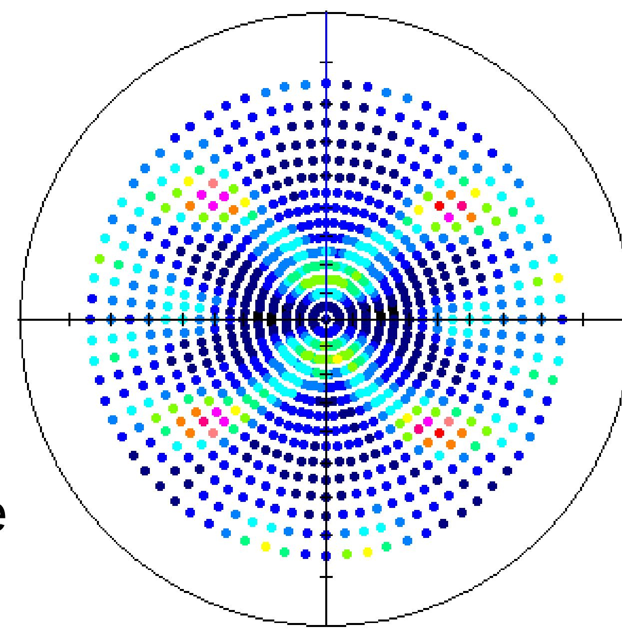
Beta rotation at each alpha angle

Alpha: 5 deg. step

(ex.15→20・・・85→90 deg.)

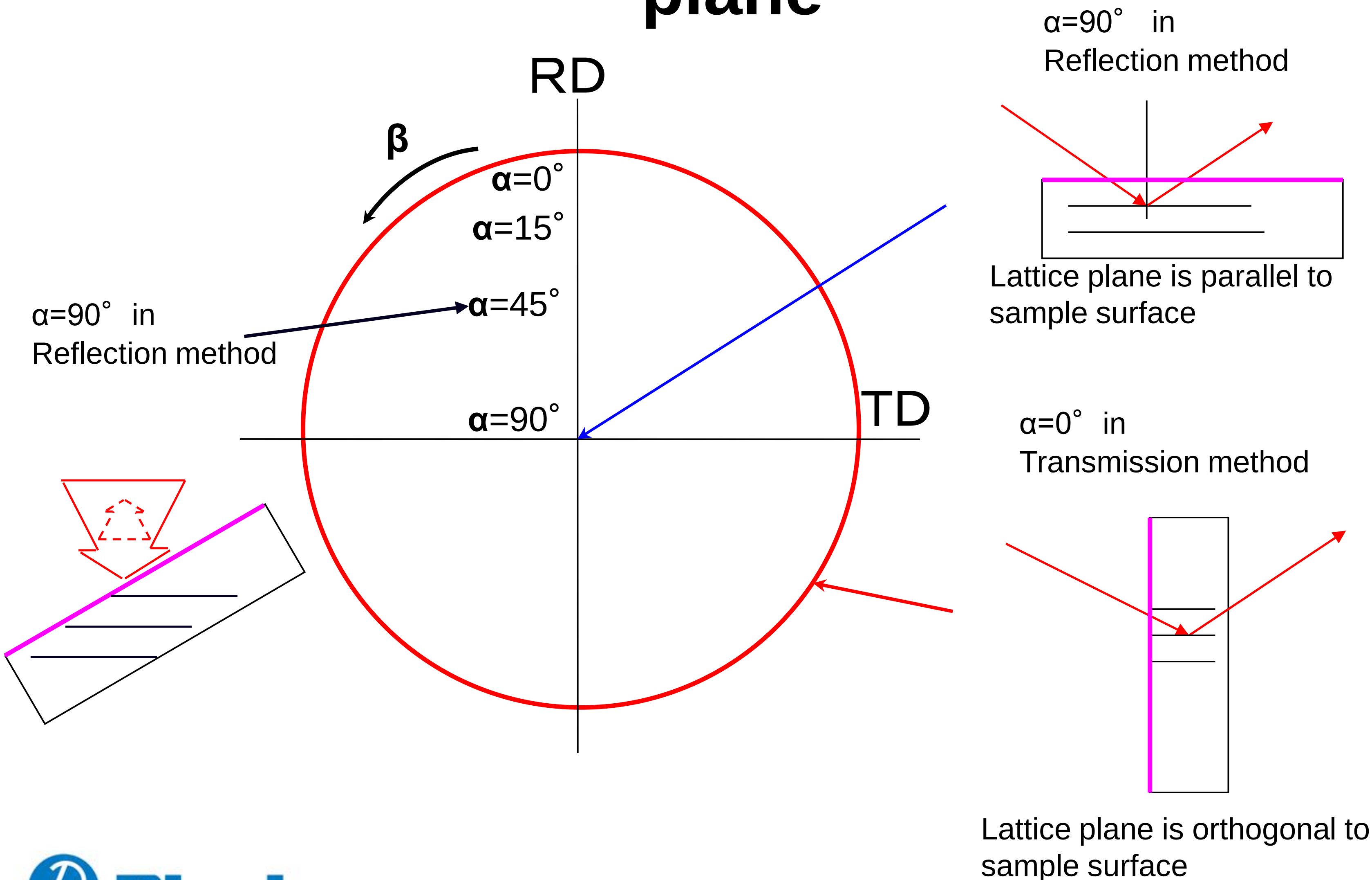
Beta: 5 deg. step

(ex.0→5・・・355→360 deg./α angle)



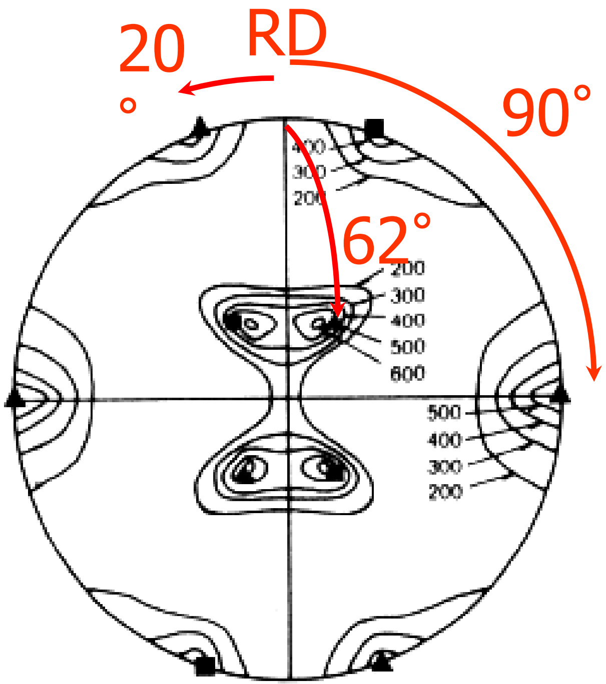
Sample is moved centering around purple line

Relations of sample surface and lattice plane

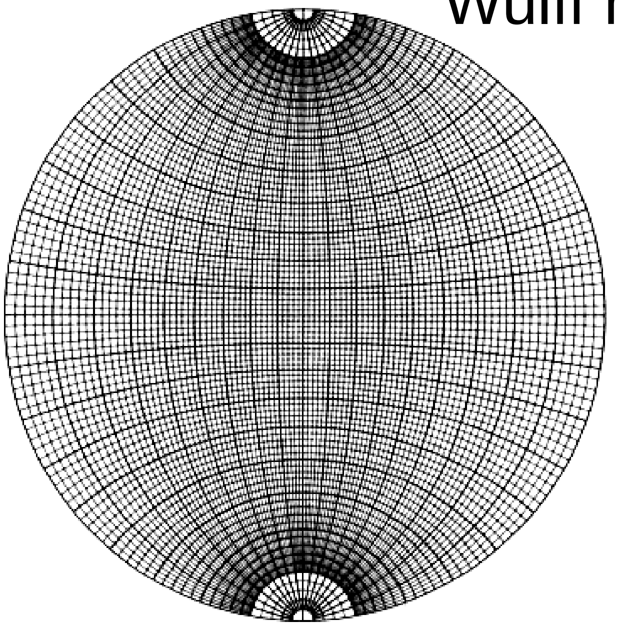


Orientation analysis: step1

Pole figure of (111) in rolled Cu-Zn(70-30)

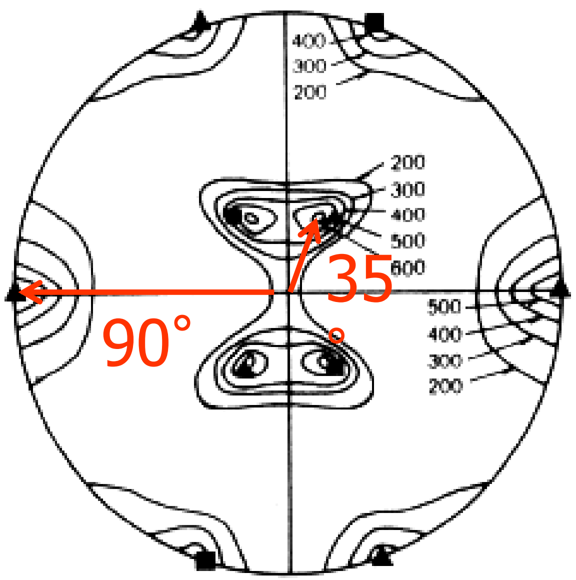


Wulff net.

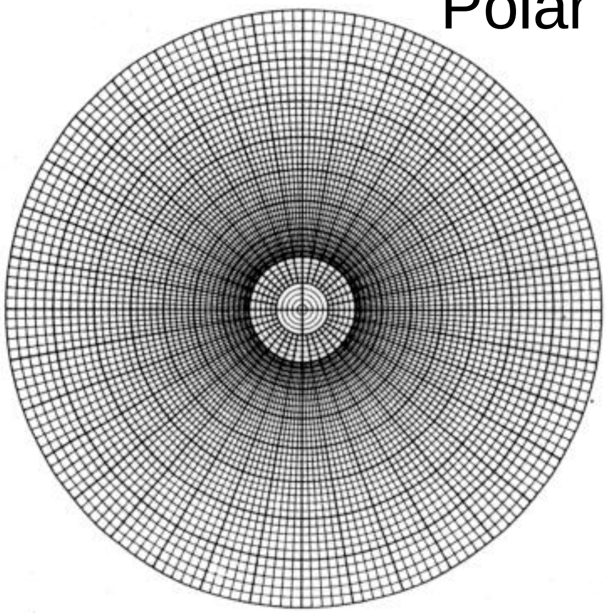


The angle between RD and each pole is estimated with Wulff net.

Orientation analysis: step2



Polar net



The angle between ND and each pole is estimated with Polar net.

Orientation analysis: step3

Angle between plane in $(h_1k_1l_1)$ and $(h_2k_2l_2)$ of cubic crystal

$(h_1k_1l_1)$	$(h_2k_2l_2)$				
	100	110 (ND)	**111**	210	211
100	0 90				
110	45 90	0 60 90			
111	54.7	35.3 90	0 70.5 109.5		
210	26.6 63.4 90	18.4 50.8 71.5	39.2 75.0	0 36.9 53.1	
211 (RD)	35.3 65.9	30 54.7 73.2 90	19.5 61.9 90	24.1 43.1 56.8	0 33.6 48.2

Orientation analysis: step4

Angle between plane of cubic

(111) (110) =35.5, 90° → ND

(111) (211) =19.5, 61.9, 90° → RD

Rotate standard (110) projection of cubic crystal 35degree clockwise.

RD is $[1\bar{1}2]$

This texture is $(110)[1\bar{1}2]$.

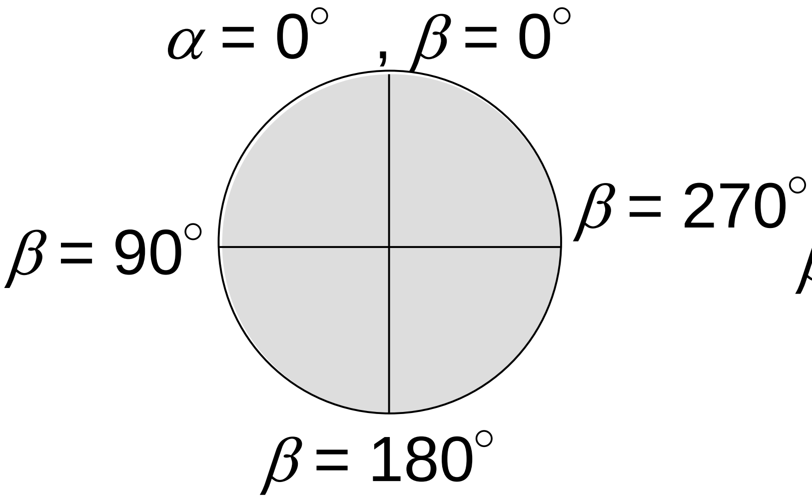
(hkl)[*uvw*]=h**u*+k**v*+l**w*=0

ND RD

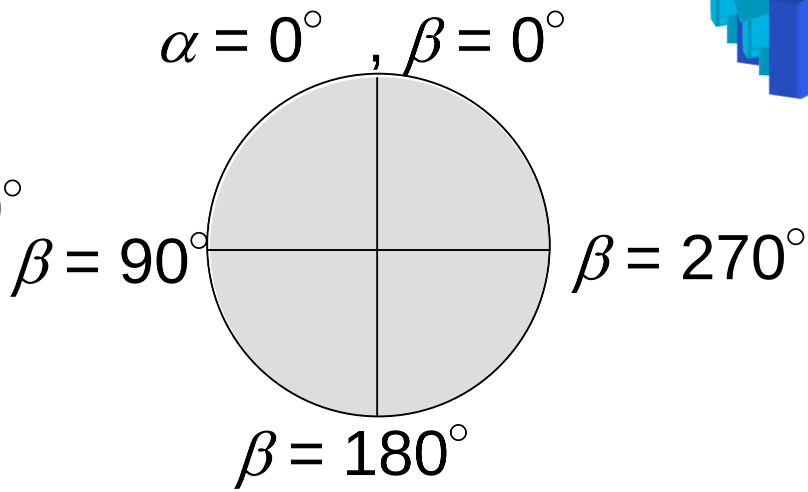
Examples of pole figures

Random orientation crystals.

Diffraction intensity does not change with the sample orientation, and a flat intensity distribution is obtained



(111) plane pole figure

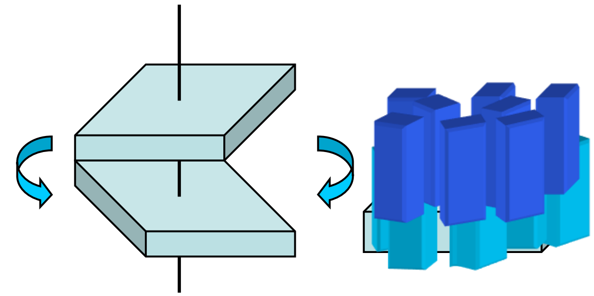


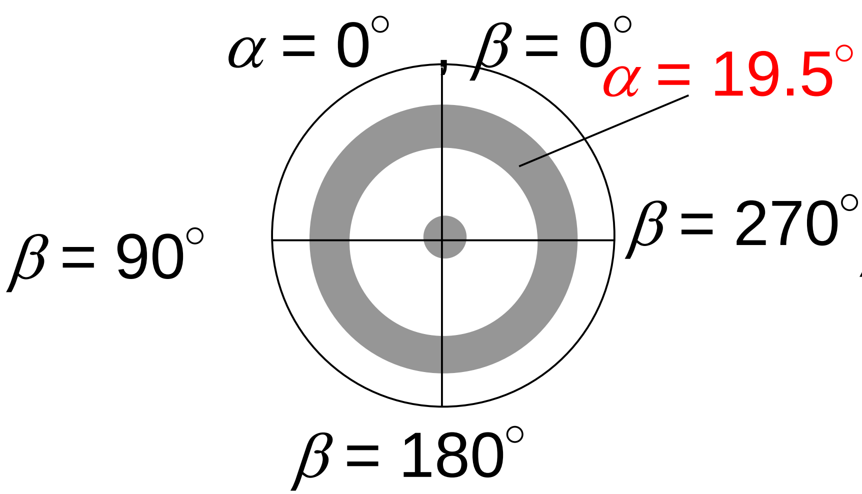
(220) plane pole figure

Examples of pole figures

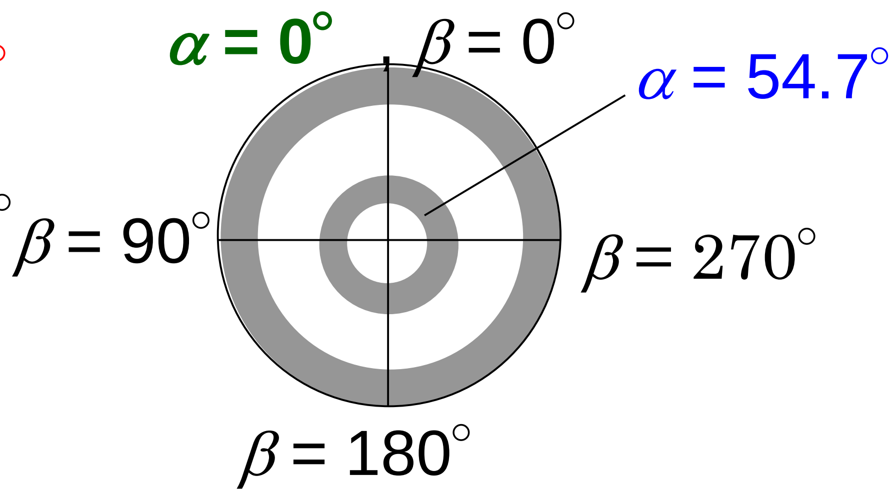
(111) plane shows *fiber texture* aligned parallel to the sample surface

$(h_1k_1l_1)$	$(h_2k_2l_2)$		
	100	110	111
111	54.7	**35.3** **90**	0 **70.5** 109.5





(111) plane pole figure

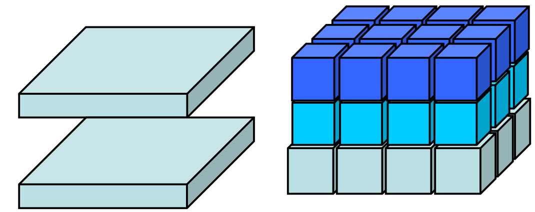


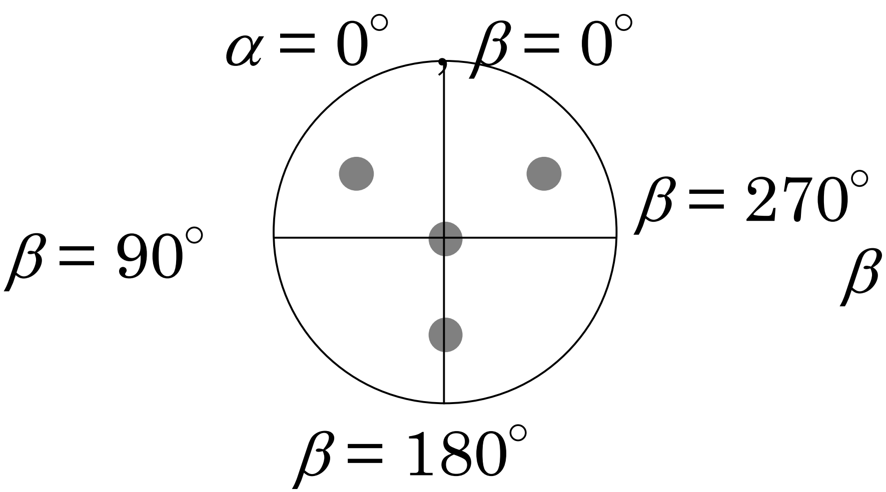
(220) plane pole figure

Examples of pole figures

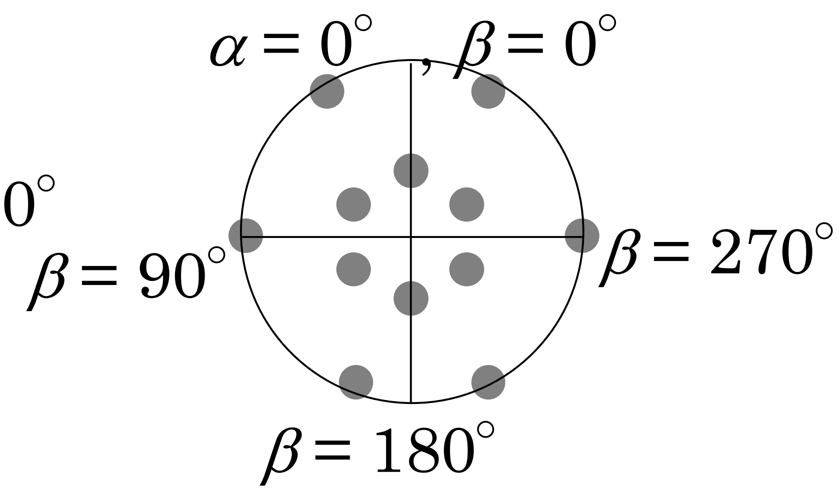
(111) plane consists of *single crystals* aligned parallel to the sample surface.

$(h_1k_1l_1)$	$(h_2k_2l_2)$		
	100	110	111
111	54.7	**35.3** **90**	0 **70.5** 109.5





(111) plane pole figure



(220) plane pole figure

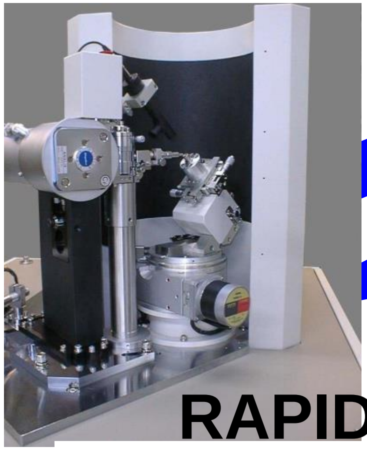
Systems

SmartLab

Automated XRD system

D/MAX 2000/PC

Standard XRD system



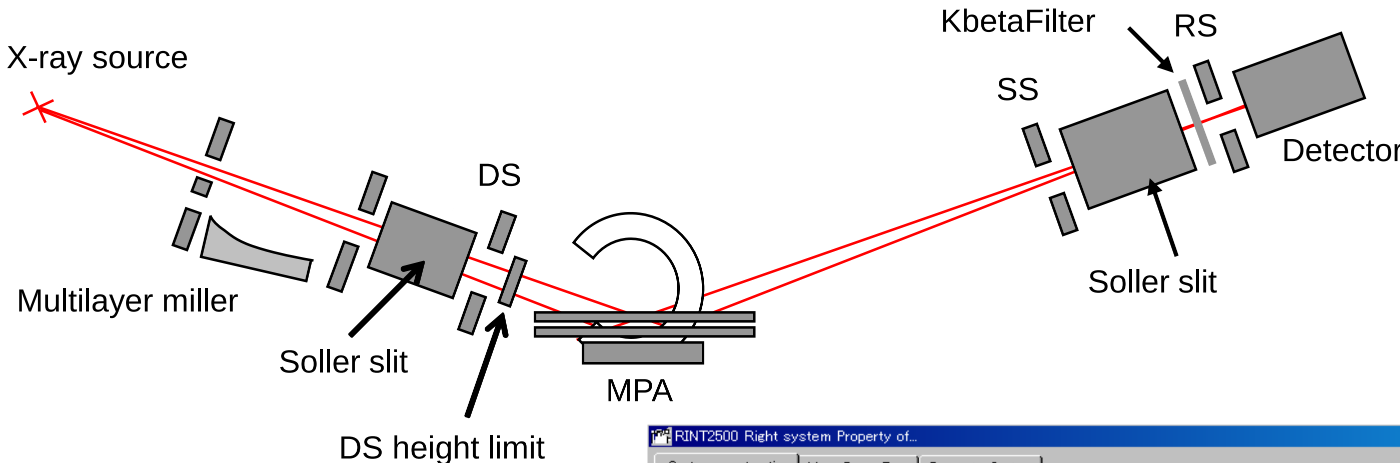
RAPID II

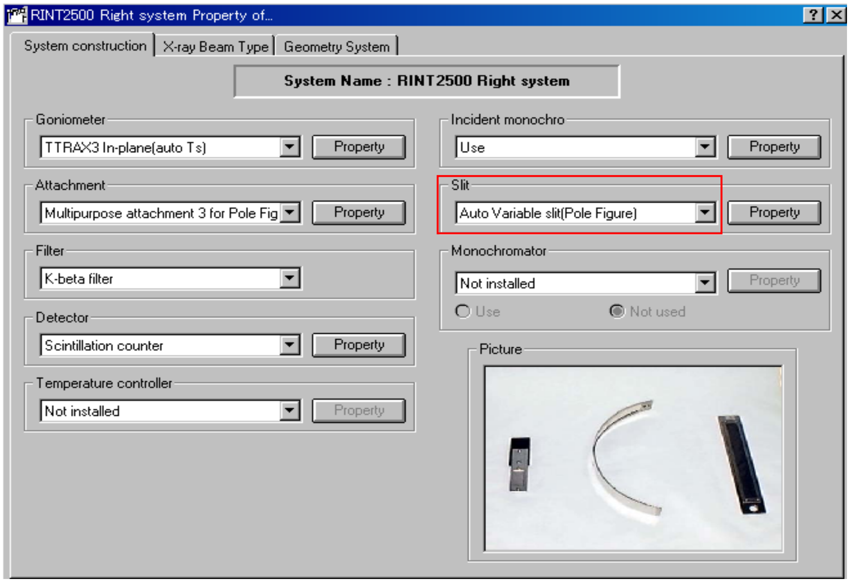
Microdiffraction XRD

UltimaⅣ

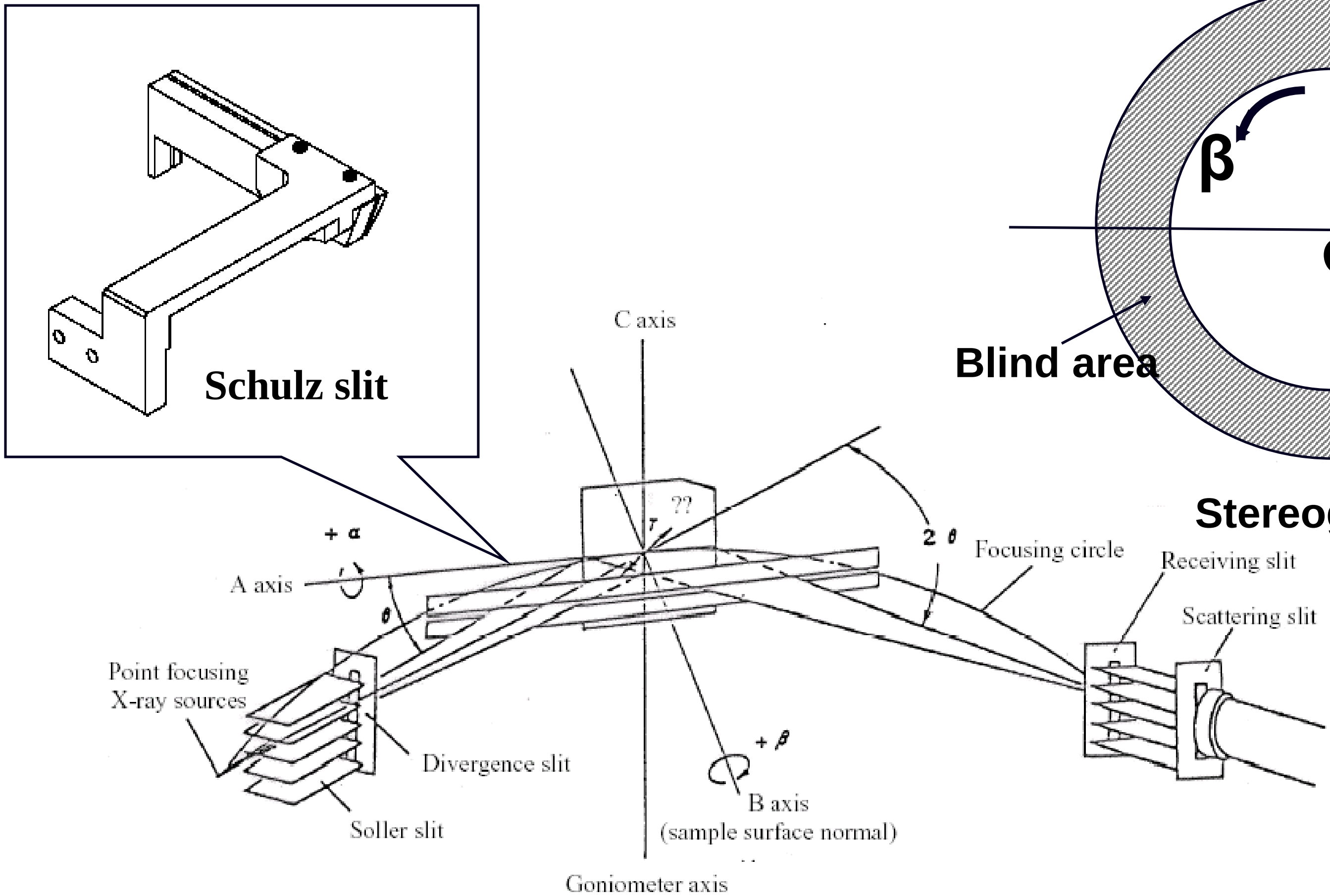
Multipurpose high-performance XRD

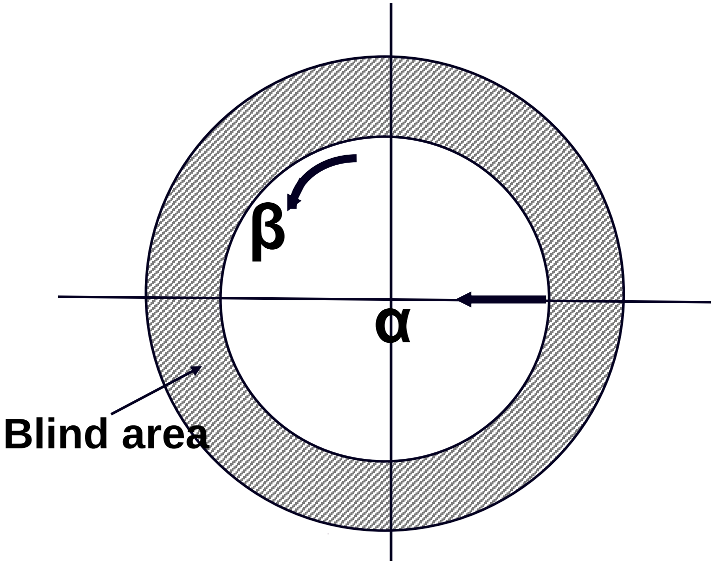
Optical system of pole figure





Schulz reflection method





Stereographic projection

Supplementary information

Measuring the spread (width) of a cross-section of a pole figure intensity distribution pattern enables us to analyze the degree of preferred orientation and the mosaic spread.

The degree orientation(%)

= (Spread of diffraction intensity)/360 × 100

The mosaic spread(°)

= (Spread of diffraction intensity)