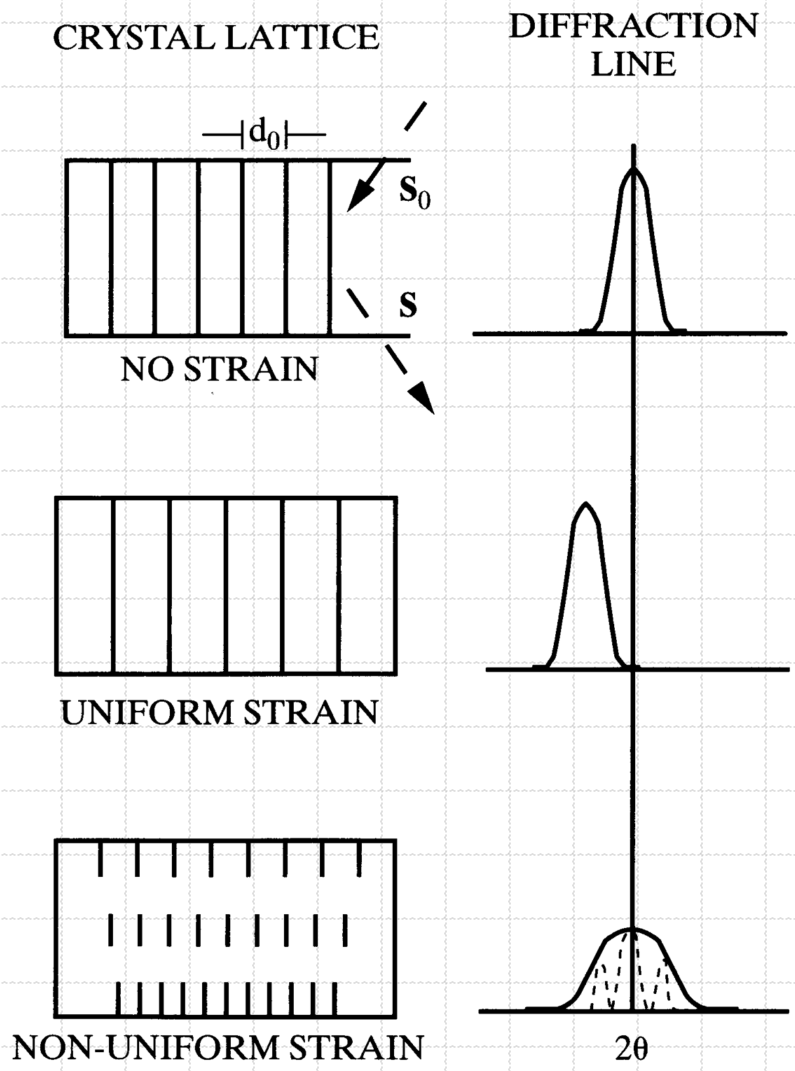
Stress and Texture

Strain

- Two types of stresses:
  - microstresses – vary from one grain to another on a microscopic scale.
  - macrostresses – stress is uniform over large distances.
- Usually:
  - macrostrain is uniform – produces peak shift
  - microstrain is nonuniform – produces peak broadening

$$b = \Delta 2\theta = -2\frac{\Delta d}{d}\tan\theta$$



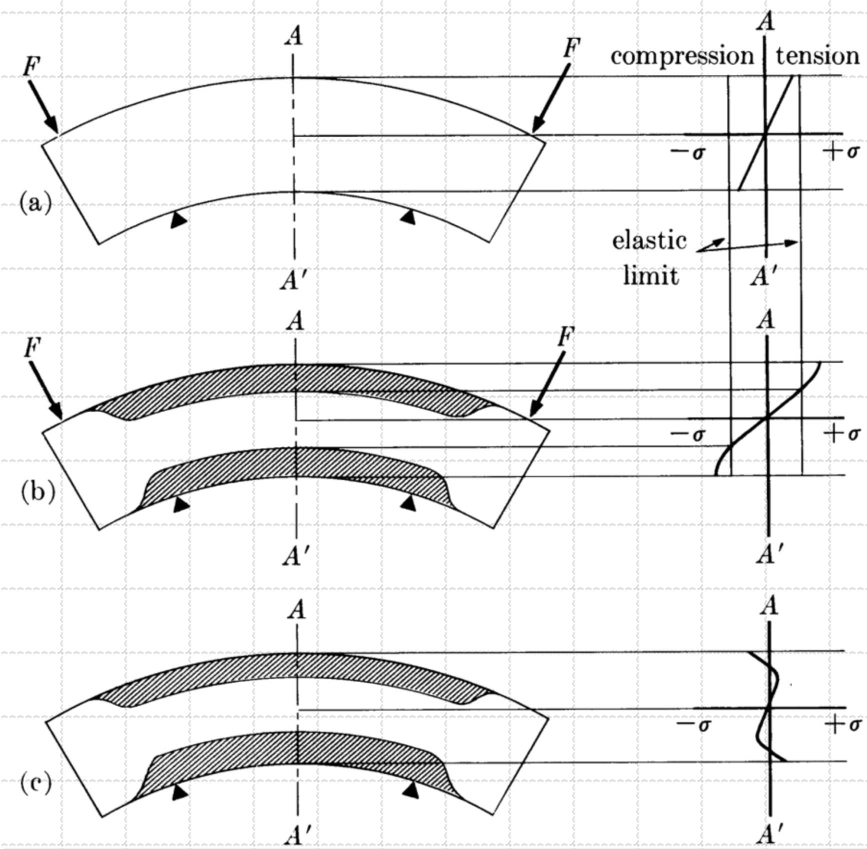
Applied and Residual Stress

- Plastic flow can also set up residual stress.

Loaded below elastic limit

Loaded beyond elastic limit

Unloaded



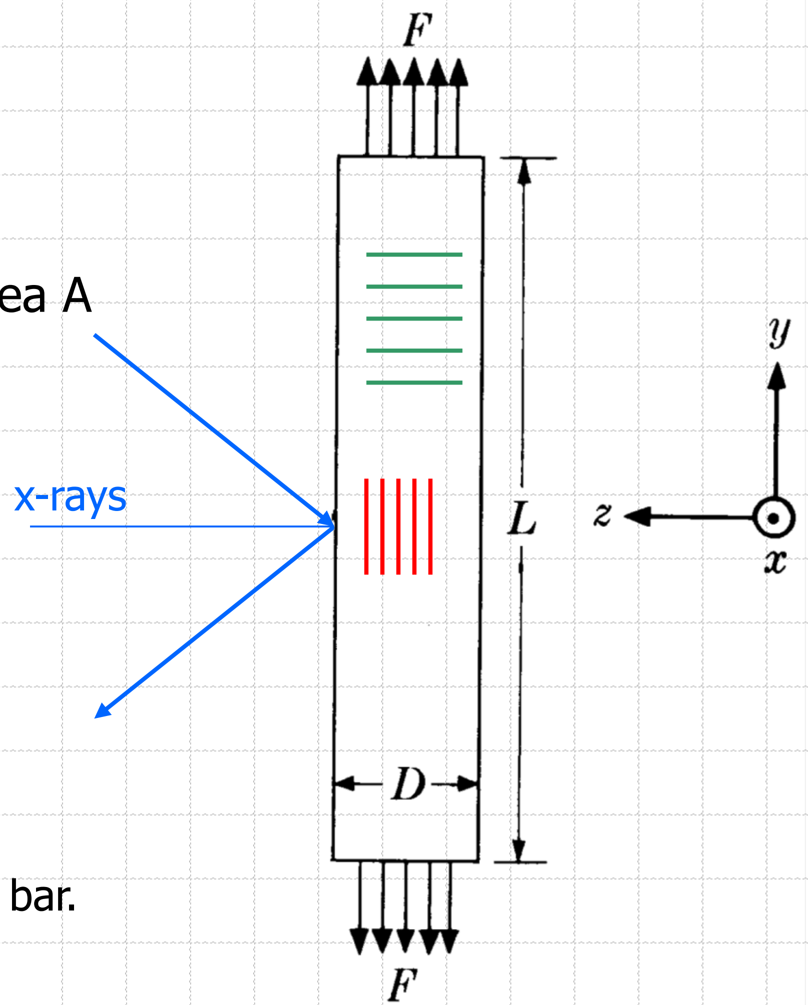
Shaded areas show regions plastically strained

Methods to Measure Residual Stress

- X-ray diffraction.
  - Nondestructive for the measurements near the surface: $t < 2\ \mu m$.
- Neutron diffraction.
  - Can be used to make measurements deeper in the material, but the minimum volume that can be examined is quite large (several mm^3) due to the low intensity of most neutron beams.
- Dissection (mechanical relaxation).
  - Destructive.

General Principles

- Consider a rod of a cross-sectional area A stressed in tension by a force F.



Stress: $$\sigma_y = \frac{F}{A}, \ \sigma_x, \sigma_z = 0$$

Stress σ_y produces strain ε_y:

$$\varepsilon_y = \frac{\Delta L}{L} = \frac{L_f - L_0}{L_0}$$

L_0 and L_f are the original and final lengths of the bar. The strain is related to stress as:

$$\sigma_y = E\varepsilon_y$$

L increases D decreases so:

$$\varepsilon_x = \varepsilon_z = \frac{\Delta D}{D} = \frac{D_f - D_0}{D_0}$$

$$\varepsilon_x = \varepsilon_z = -\nu\varepsilon_y$$ for isotropic material

ν – Poisson’s ratio
usually $0.25 < \nu < 0.45$

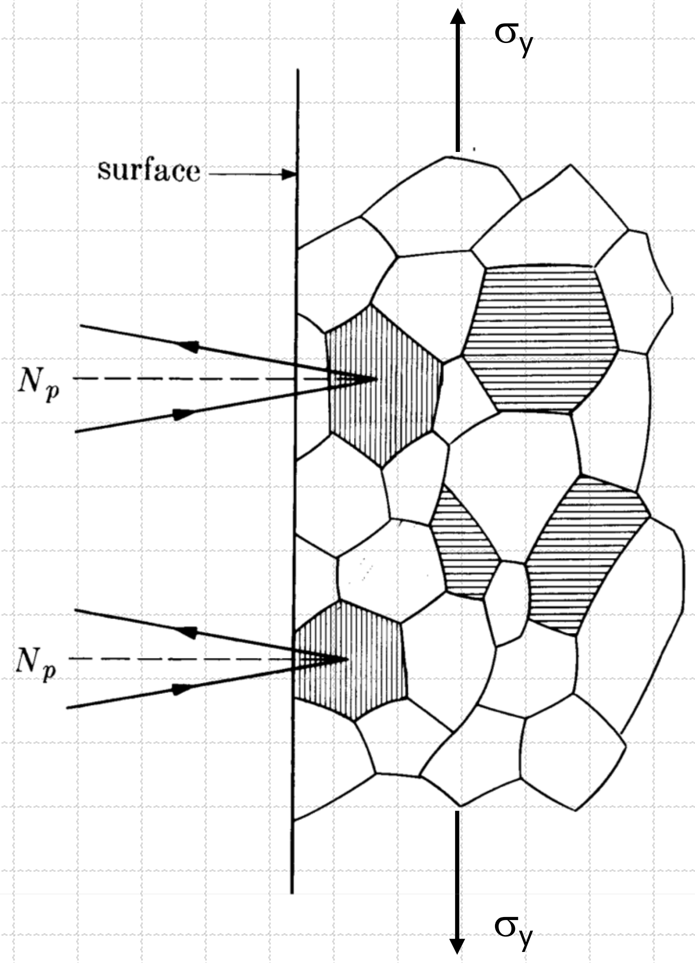
General Principles

- This provides measurement of the strain in the z direction since:

$$\varepsilon_z = \frac{d_n - d_0}{d_0}$$

Then the required stress will be:

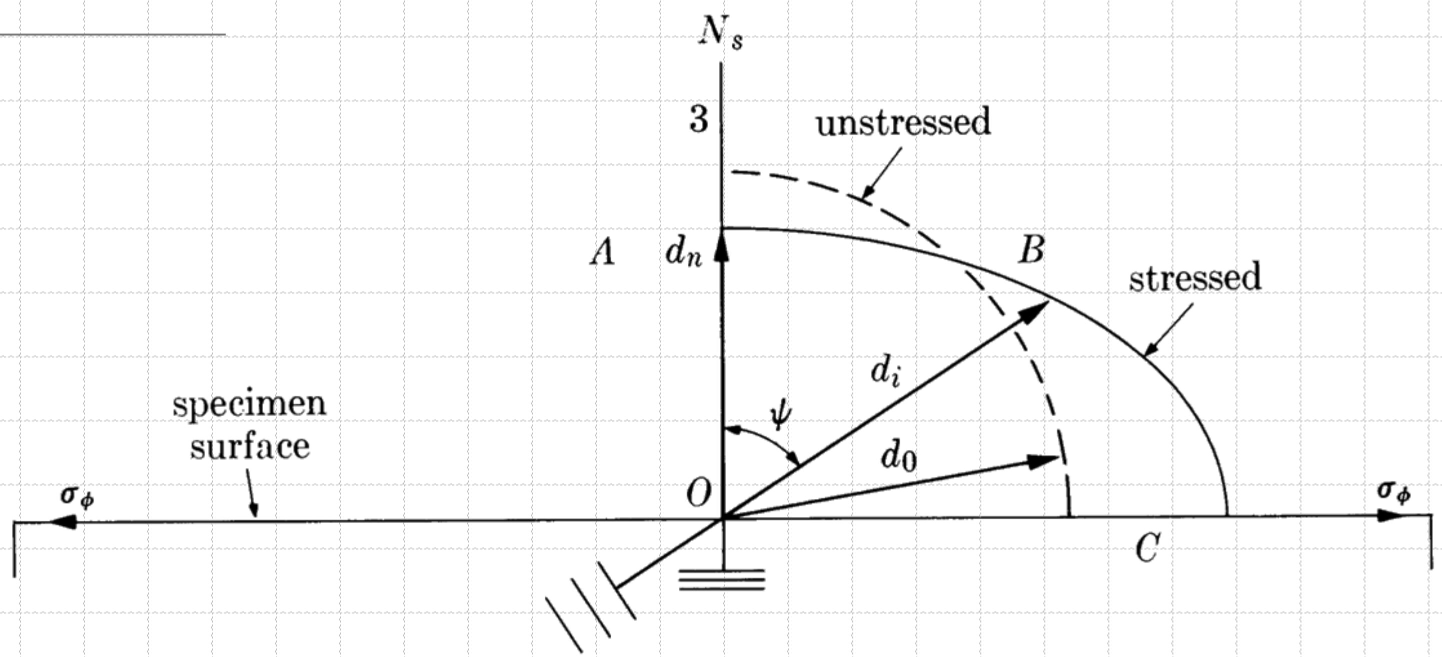
$$\sigma_y = -\frac{E}{\nu}\left(\frac{d_n - d_0}{d_0}\right)$$



Diffraction techniques do not measure stresses in materials directly

- Changes in d-spacing are measured to give strain
- Changes in line width are measured to give microstrain
- The lattice planes of the individual grains in the material act as strain gauges

General Principles



Vector diagram of plane spacings d for a tensile stress σ_ϕ

$$\sigma_y = -\frac{E}{\nu}\left(\frac{d_n - d_0}{d_0}\right)$$

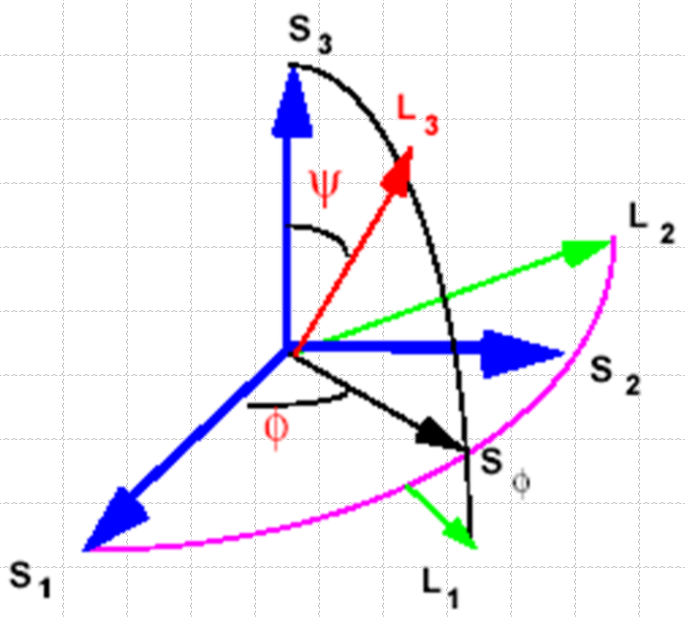
we do not know d_0 !

Elasticity

- In general there are stress components in two or three directions at right angles to one another, forming biaxial or triaxial stress systems.
  - Stresses in a material can be related to the set of three principal stresses σ_1, σ_2 and σ_3.
- To properly describe the results of a diffraction stress measurement we introduce a coordinate systems for the instrument and the sample. These two coordinate systems are related by two rotation angles ψ and ϕ.

$\mathbf{L_i}$ – laboratory coordinate system
$\mathbf{S_i}$ – sample coordinate system

By convention the diffracting planes are normal to L_3



Elasticity

- In an anisotropic elastic material stress tensor σ_{ij} is related to the strain tensor ε_{kl} as:

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$$

where C_{ijkl} is elastic constants matrix.

Similarly:

$$\varepsilon_{ij} = S_{ijkl}\sigma_{kl}$$

where S_{ijkl} is elastic compliance matrix.

For isotropic compound:

$$\varepsilon_{ij} = \frac{1+\nu}{E}\sigma_{ij} - \delta_{ij}\frac{\nu}{E}\sigma_{kk}$$

where δ_{ij} is Kroenecker's delta, "kk" indicates the summation $\sigma_{11} + \sigma_{22} + \sigma_{33}$

Elasticity

- Or we can write it as:

$$\varepsilon_{11} = \frac{1}{E}\left[\sigma_{11} - \nu\left(\sigma_{22} + \sigma_{33}\right)\right],$$

$$\varepsilon_{22} = \frac{1}{E}\left[\sigma_{22} - \nu\left(\sigma_{11} + \sigma_{33}\right)\right],$$

$$\varepsilon_{33} = \frac{1}{E}\left[\sigma_{33} - \nu\left(\sigma_{11} + \sigma_{22}\right)\right],$$

$$\varepsilon_{23} = \frac{1}{2\mu}\sigma_{23},$$

$$\varepsilon_{31} = \frac{1}{2\mu}\sigma_{31},$$

$$\varepsilon_{12} = \frac{1}{2\mu}\sigma_{12}$$

where $\mu = \dfrac{E}{2\left(1+\nu\right)}$

shear modulus

Elasticity

- Lets relate ε_{mn} in one coordinate system to that in another system through transformation matrix:

$$\varepsilon^{L}_{mn} = M^{SL}_{mi} M^{SL}_{nj} \varepsilon^{S}_{ij}$$

The transformation matrix is:

$$M^{SL} = \begin{bmatrix} \cos\phi\cos\psi & -\sin\phi & \cos\phi\sin\psi \\ \sin\phi\cos\psi & \cos\phi & \sin\phi\sin\psi \\ -\sin\psi & 0 & \cos\psi \end{bmatrix}$$

so that we find

$$\begin{aligned} \left(\varepsilon^{L}_{33}\right)_{\phi\psi} &= M^{SL}_{3i} M^{SL}_{3j} \varepsilon^{S}_{ij} \\ &= \varepsilon^{S}_{11} \cos^2\phi \sin^2\psi + \varepsilon^{S}_{12} \sin 2\phi \sin^2\psi \\ &\quad + \varepsilon^{S}_{22} \sin^2\phi \sin^2\psi + \varepsilon^{S}_{33} \cos^2\psi \\ &\quad + \varepsilon^{S}_{13} \cos\phi \sin 2\psi + \varepsilon^{S}_{23} \sin\phi \sin 2\psi \end{aligned}$$

Elasticity

- In terms of stresses:

$$
\begin{aligned}
\left(\varepsilon_{33}^{L}\right)_{\phi\psi} &= \frac{1+\nu}{E}\left\{\sigma_{11}^{S}\cos^{2}\phi+\sigma_{12}^{S}\sin 2\phi+\sigma_{22}^{S}\sin^{2}\phi-\sigma_{33}^{S}\right\}\sin^{2}\psi+\frac{1+\nu}{E}\sigma_{33}^{S} \\
&\quad -\frac{\nu}{E}\left(\sigma_{11}^{S}+\sigma_{22}^{S}+\sigma_{33}^{S}\right)+\frac{1+\nu}{E}\left\{\sigma_{13}^{S}\cos\phi+\sigma_{23}^{S}\sin\phi\right\}\sin 2\psi \\
&= \frac{d_{\phi\psi}-d_{0}}{d_{0}}
\end{aligned}
$$

Biaxial and Triaxial Stress Analysis

- Biaxial stress tensor is in the form:

$$\begin{bmatrix} \sigma_{11} & 0 & 0 \\ 0 & \sigma_{22} & 0 \\ 0 & 0 & 0 \end{bmatrix}$$

since stress normal to a free surface must be zero: $\sigma_{ij}\mathbf{n}_j = 0$

For our tensor lets define:

$$\sigma_\phi^S = \sigma_{11}^S \cos^2\phi + \sigma_{22}^S \sin^2\phi$$

Then the equation for strain becomes:

$$\varepsilon_{\psi\phi}^L = \frac{d_{\phi\psi} - d_0}{d_0} = \frac{1+\nu}{E}\sigma_\phi^S \sin^2\psi - \frac{\nu}{E}\left(\sigma_{11}^S + \sigma_{22}^S\right)$$

The $\sin^2\psi$ Method

- Stress σ_{33} is zero, but strain ε_{33} is not zero. It has finite value given by the Poisson contractions due to σ_{11} and σ_{22}:

$$\varepsilon_{33}^{S} = -\nu\left(\varepsilon_{11}^{S} + \varepsilon_{22}^{S}\right) = -\frac{\nu}{E}\left(\sigma_{11}^{S} + \sigma_{22}^{S}\right)$$

Then strain equation can be written as:

$$\varepsilon_{\phi\psi}^{L} - \varepsilon_{33}^{S} = \frac{(1+\nu)\sigma_{\phi}^{S}}{E}\sin^2\psi$$

$$\varepsilon_{\phi\psi}^{L} - \varepsilon_{33}^{S} = \frac{d_{\phi\psi} - d_0}{d_0} - \frac{d_n - d_0}{d_0}$$

$$= \frac{d_{\phi\psi} - d_n}{d_0} = \frac{(1+\nu)\sigma_{\phi}^{S}}{E}\sin^2\psi$$

The $\sin^2\psi$ Method

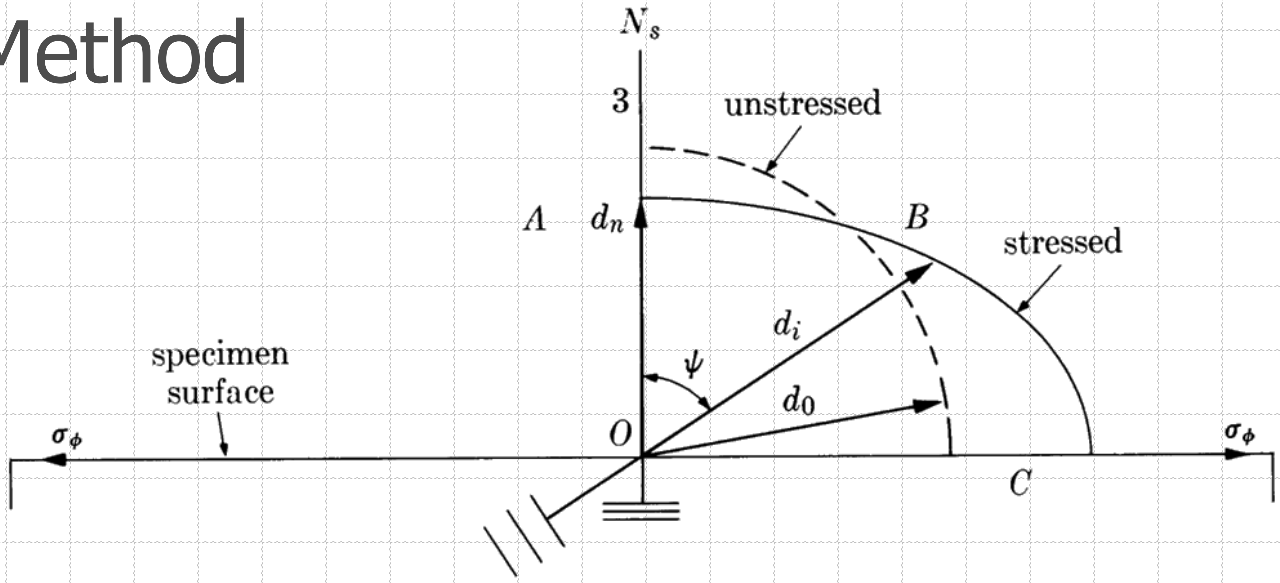
- We make ingenious approximation (by Glocker *et al*. in 1936):
  - d_n, d_i and d_0 are very nearly equal to one another,
  - $(d_i - d_n)$ is small compared to d_0,
  - unknown d_0 is replaced by d_i or d_n with negligible error.

$$\boxed{\frac{d_{\phi\psi} - d_n}{d_n} = \frac{(1+\nu)\sigma_\phi}{E}\sin^2\psi}$$

$$\sigma_\phi = \frac{E}{(1+\nu)\sin^2\psi}\left(\frac{d_{\phi\psi} - d_n}{d_n}\right)$$

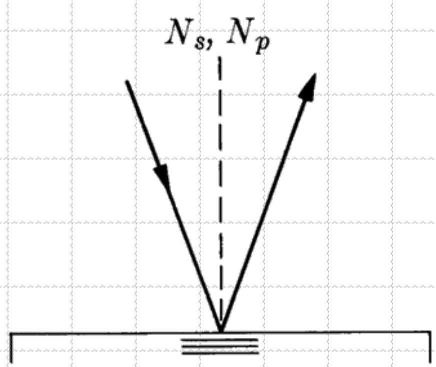
- The stress in a surface can be determined by measuring the *d*-spacing as a function of the angle ψ between the surface normal and the diffracting plane normal
- Measurements are made in the back-reflection regime ($2\theta \rightarrow 180°$) to obtain maximum accuracy

The sin$^2\psi$ Method

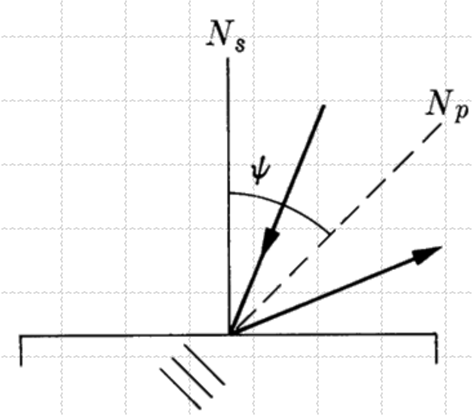


Vector diagram of plane spacings d for a tensile stress σ_ϕ

$$\frac{d_{\phi\psi}-d_n}{d_n}=\frac{(1+\nu)\sigma_\phi}{E}\sin^2\psi$$



Measurement of d_n



Measurement of d_i

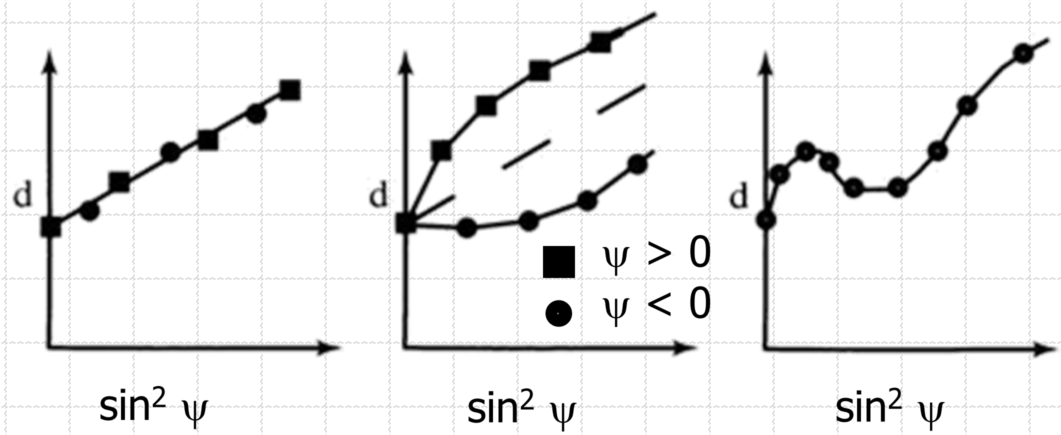
The sin$^2\psi$ Method

- Lets assume that stresses in zx plane are equal. This is referred to as an equal-biaxial stress state. We can write sample frame stress as:

$$\begin{bmatrix} \sigma & 0 & 0 \\ 0 & \sigma & 0 \\ 0 & 0 & 0 \end{bmatrix}$$

No stress dependence on ϕ

$$\frac{d_\psi - d_n}{d_n} = \frac{(1+\nu)\sigma}{E}\sin^2\psi$$



Biaxial or uniaxial stress | Triaxial stresses present | Texture present

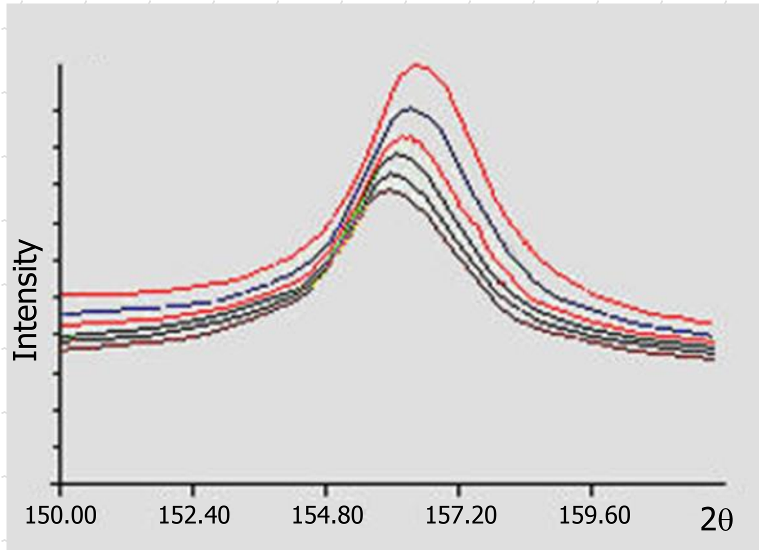
The $\sin^2\psi$ Method

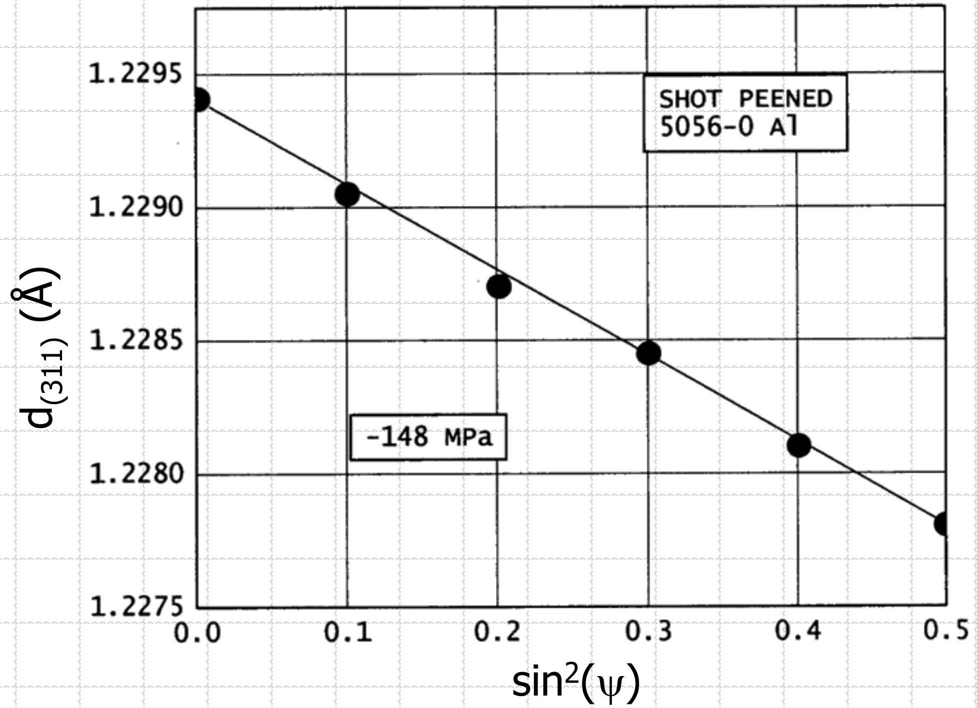
$$d_\psi = \frac{(1+\nu)\sigma}{E} d_n \sin^2\psi + d_n$$

Slope of the plot is:

$$\frac{\partial d_\psi}{\partial \sin^2\psi} = \frac{(1+\nu)}{E}\sigma d_n$$

Generally ν and E are well-known constants

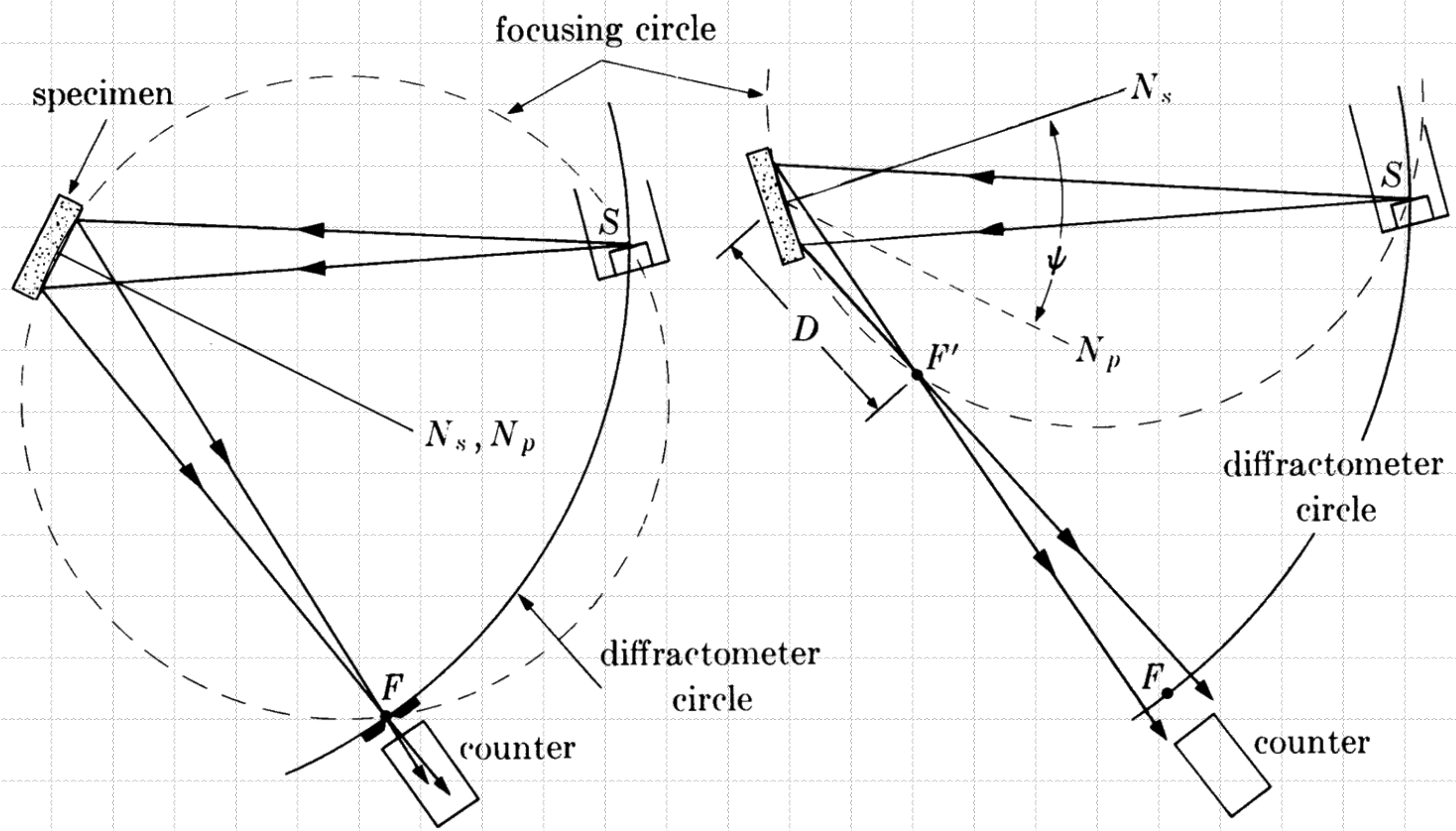




Linear dependence of d (311) upon $\sin^2\psi$ for shot peened 5056-0 aluminum.

Diffractometer Method

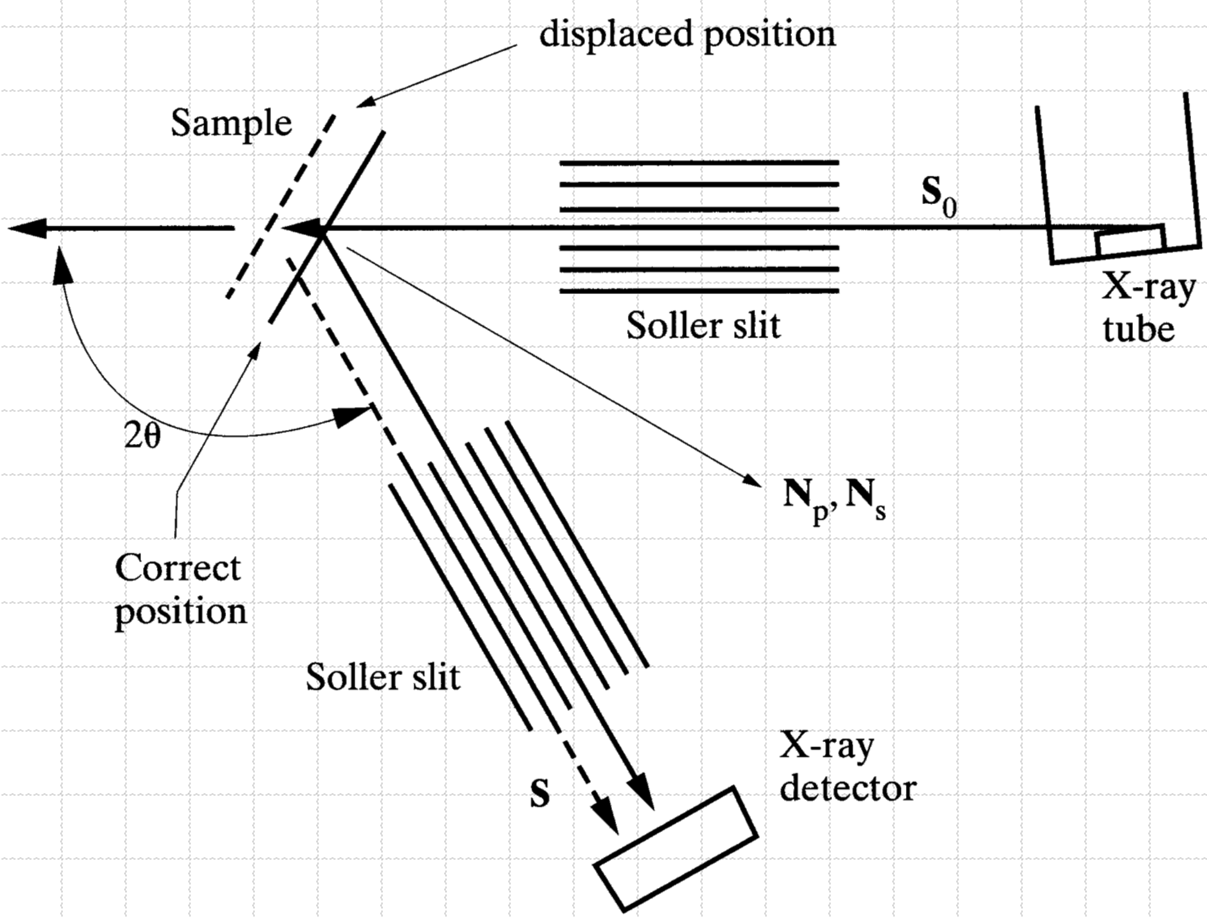
$$\frac{D}{R} = \frac{\sin(\theta - \psi)}{\sin(\theta - \psi)}$$



(a) $\psi = 0$ (b) $\psi = \psi$

Diffractometer Method

- The effect of sample or ψ-axis displacement can be minimized if a parallel beam geometry is used instead of focused beam geometry.



Measurement of Line Position

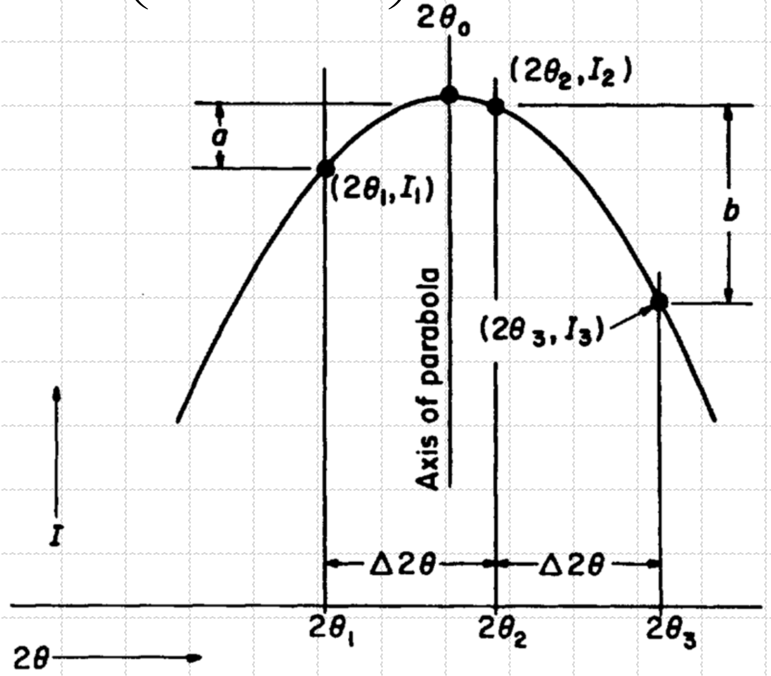
- Sample must remain on the diffractometer axis as ψ is changed (even if the sample is large)
- Radial motion of the detector to achieve focussing must not change the measured 2θ
- L-P factor may vary significantly across a (broad) peak $\Rightarrow \quad LPA = \left(\dfrac{1+\cos^2 2\theta}{\sin^2\theta}\right)(1-\tan\psi\cot\theta)$
- Absorption will vary when $\Psi \neq 0$
- Measurement of peak position often requires fitting the peak with a parabola:

$$2\theta_0 = 2\theta_1 + \frac{\Delta 2\theta}{2}\left(\frac{3a+b}{a+b}\right)$$

$$a = I_2 - I_1$$

$$b = I_2 - I_3$$

$$\Delta 2\theta = 2\theta_2 - 2\theta_1 = 2\theta_3 - 2\theta_2$$



Measurements of Stress in Thin Films

- Thin films are usually textured. No difficulty with moderate degree of preferred orientation.

- Sharp texture has the following effects:
  - Diffraction line strong at $\psi = 0$ and absent at $\psi = 45^o$.
  - If material anisotropic E will depend on direction in the specimen. Oscillations of d vs $\sin^2\psi$.

Measurements of Stress in Thin Films

- In thin films we have a biaxial stress, so:

$$\frac{d_{\phi\psi} - d_n}{d_n} = \frac{(1+\nu)\sigma_\phi}{E}\sin^2\psi$$

If we have equal-biaxial stress then it is even simpler:

$$\frac{a_\psi - a_n}{a_n} = \frac{d_{hkl}^{\psi} - d_{hkl}^{n}}{d_{hkl}^{n}} = \frac{(1+\nu)\sigma}{E}\sin^2\psi$$

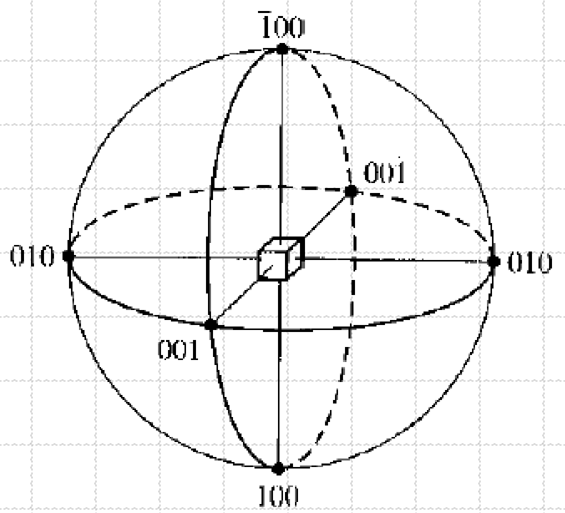
We can calculate ψ for any unit cell and any orientation. For cubic:

$$\psi = a\cos\left(\frac{h_\perp h + k_\perp k + l_\perp l}{\sqrt{(h_\perp^2 + k_\perp^2 + l_\perp^2)(h^2 + k^2 + l^2)}}\right)$$

Symbols $h_\perp$, $k_\perp$ and $l_\perp$ define (hkl) - oriented film

Texture Analysis

- The determination of the lattice preferred orientation of the crystallites in a polycrystalline aggregate is referred to as texture analysis.
- The term texture is used as a broad synonym for preferred crystallographic orientation in a polycrystalline material, normally a single phase.
- The preferred orientation is usually described in terms of pole figures.



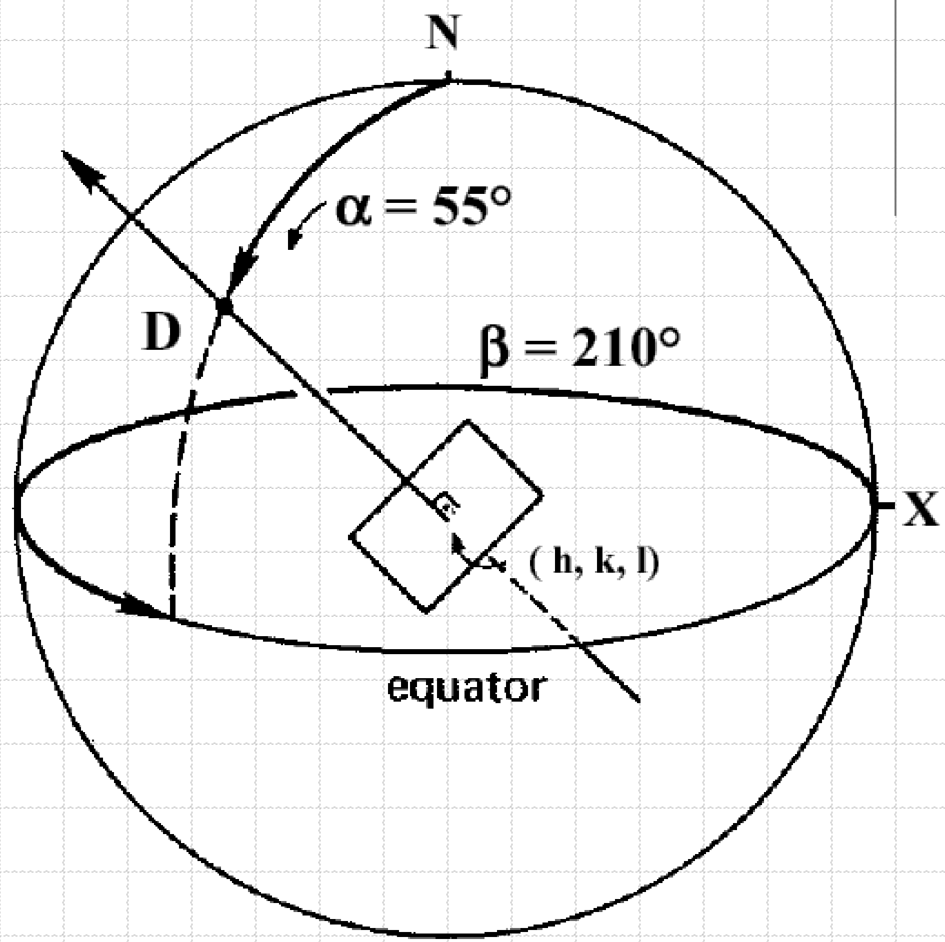
{100} poles of a cubic crystal

The Pole Figures

- Let us consider the plane (h k l) in a given crystallite in a sample. The direction of the plane normal is projected onto the sphere around the crystallite.

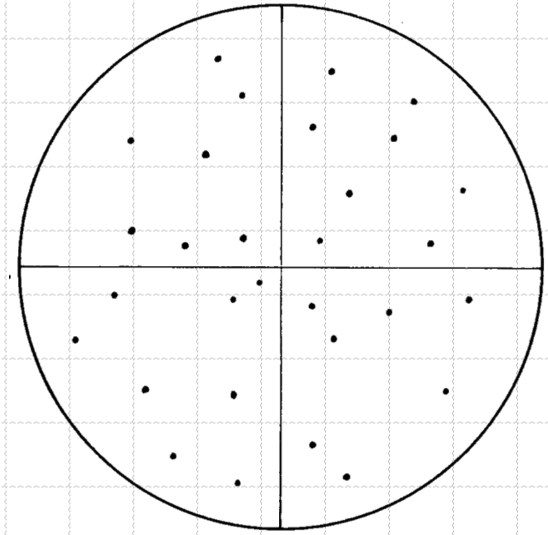
- The point where the plane normal intersects the sphere is defined by two angles: pole distance α and an azimuth β.

- The azimuth angle is measured counter clock wise from the point X.

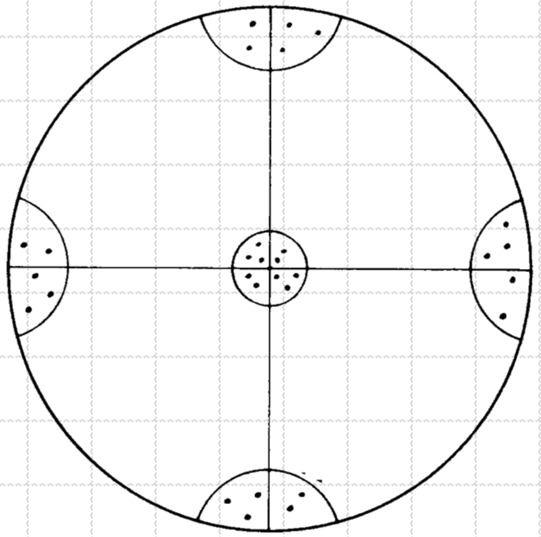


The Pole Figures

- Let us now assume that we project the plane normals for the plane (h k l) from all the crystallites irradiated in the sample onto the sphere.
- Each plane normal intercepting the sphere represents a point on the sphere. These points in return represent the Poles for the planes (h k l) in the crystallites. The number of points per unit area of the sphere represents the pole density.

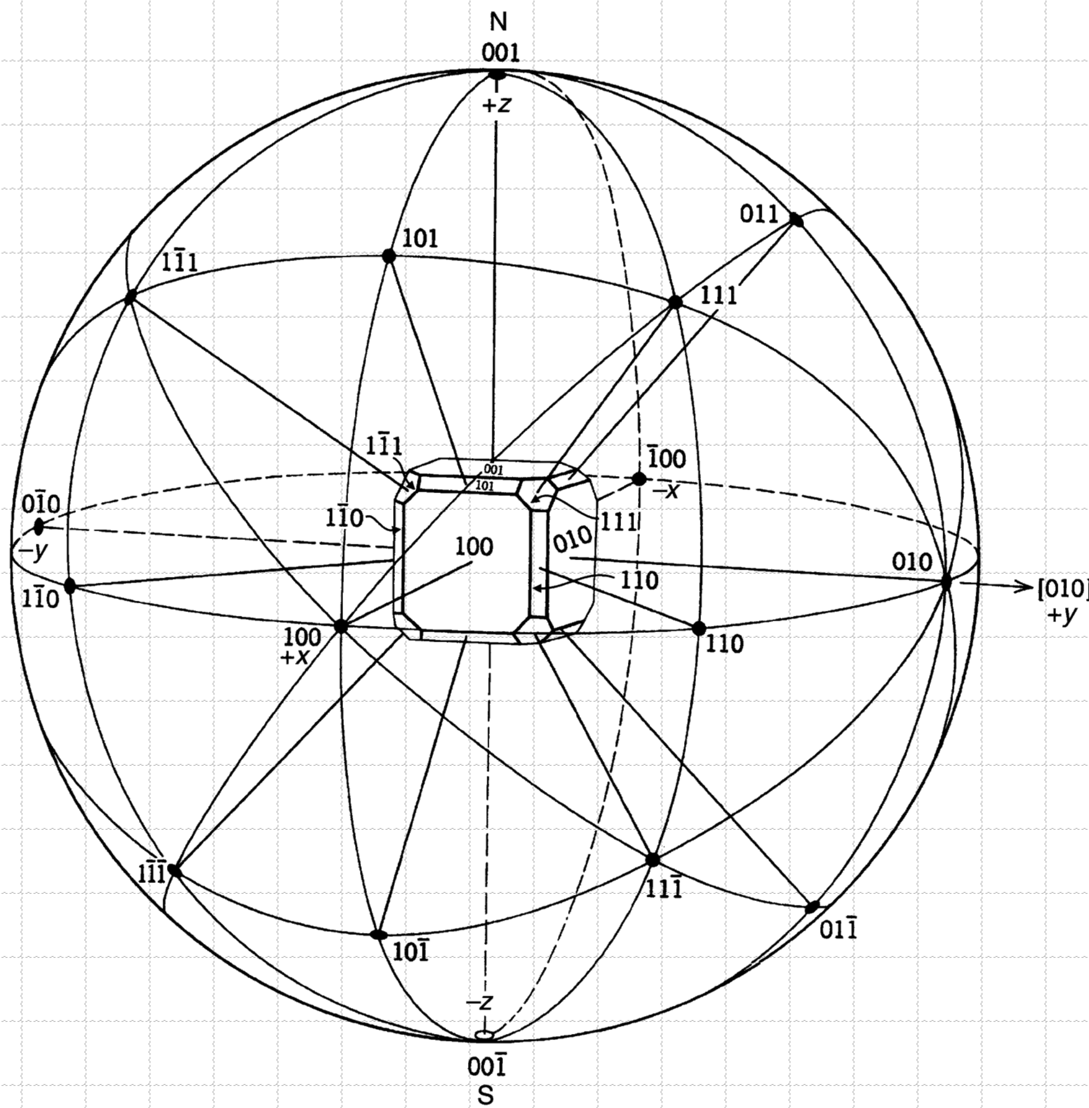


Random orientation

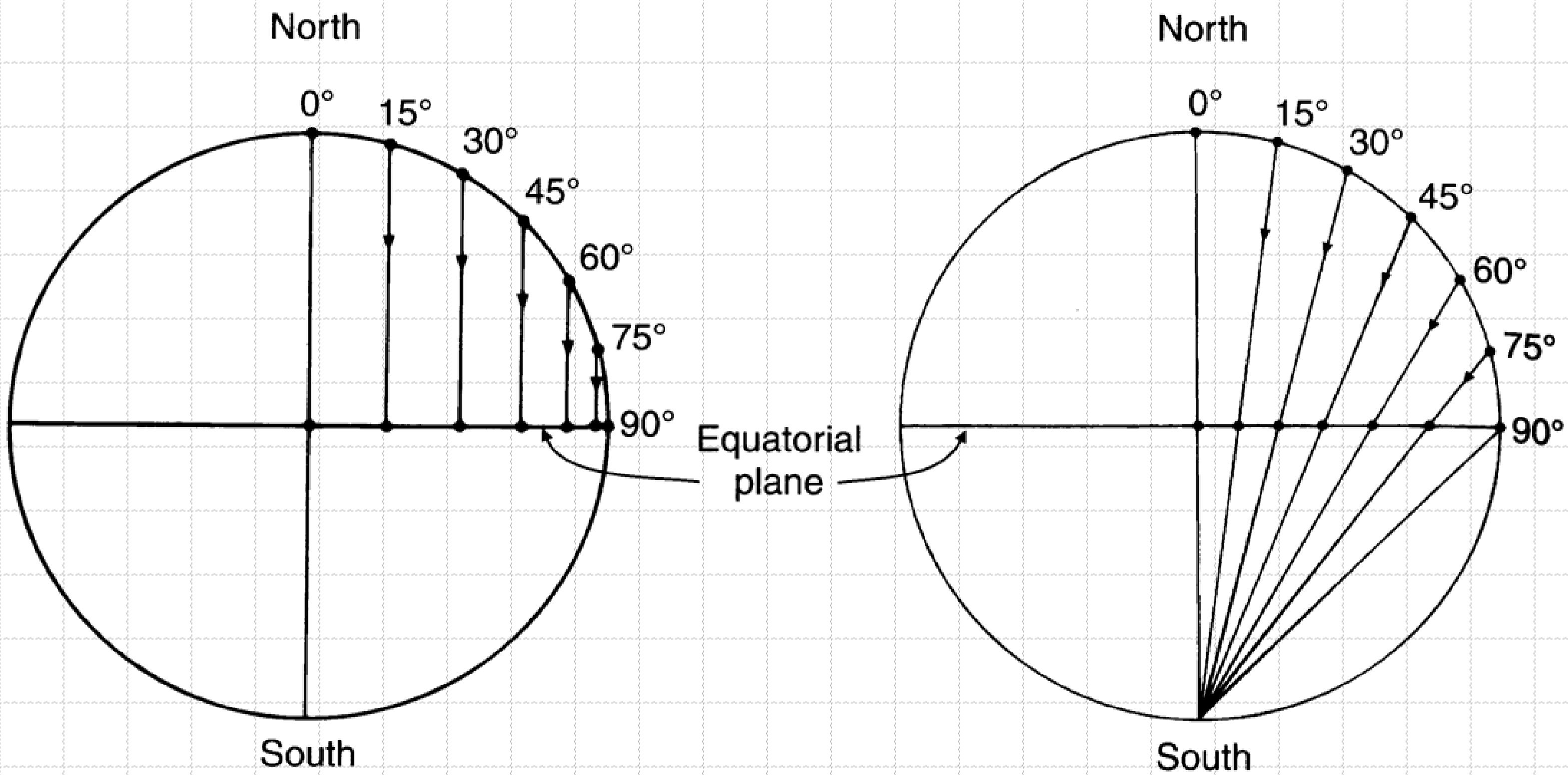


Preferred orientation

The Pole Figures



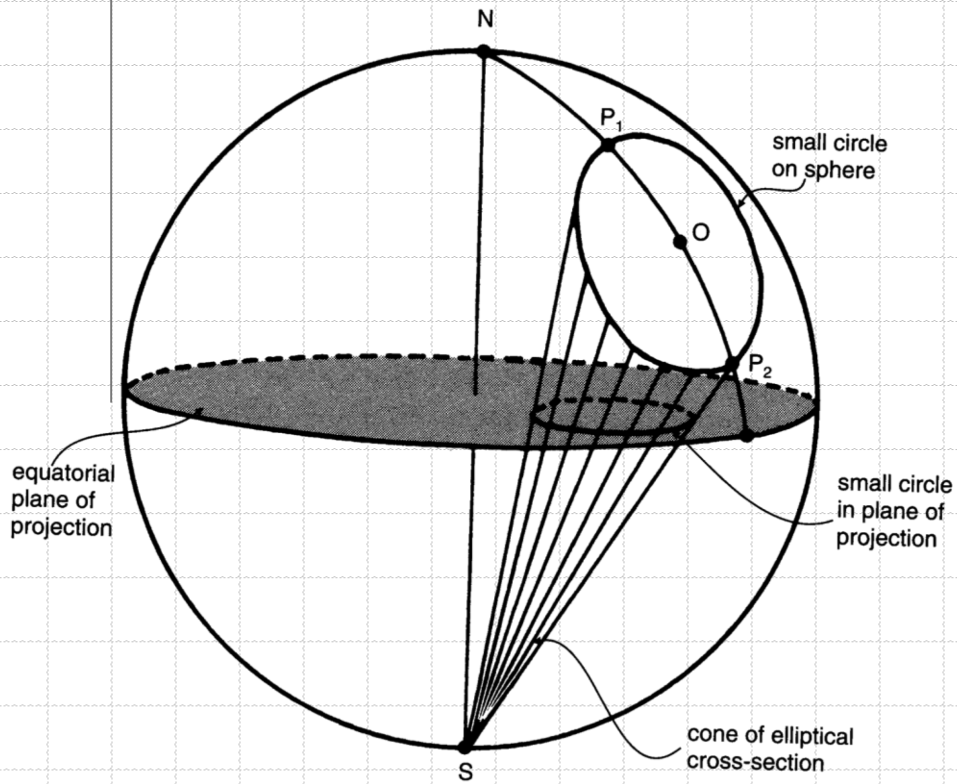
The Stereographic Projection

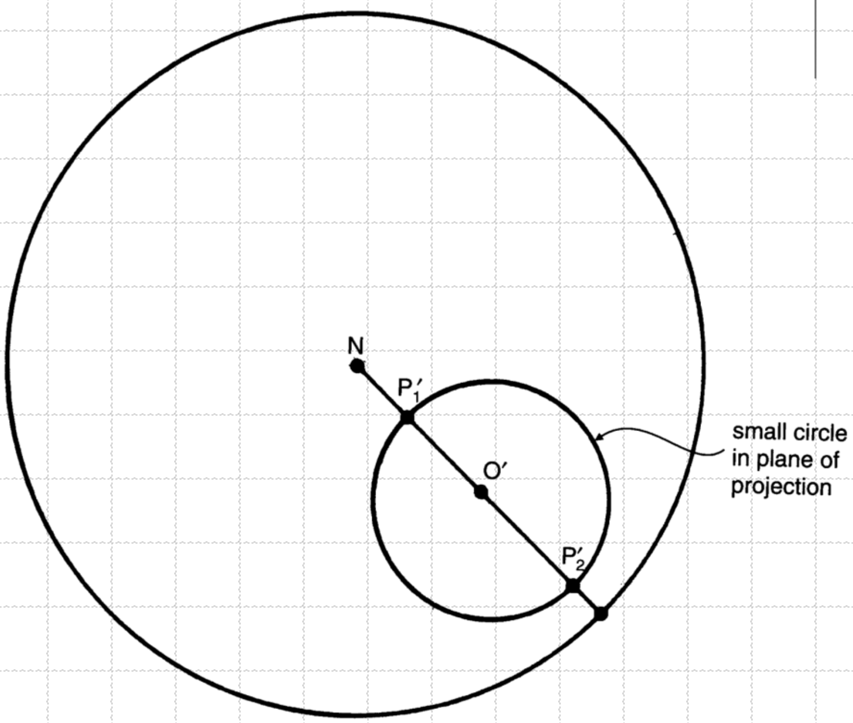


As we look down to the earth

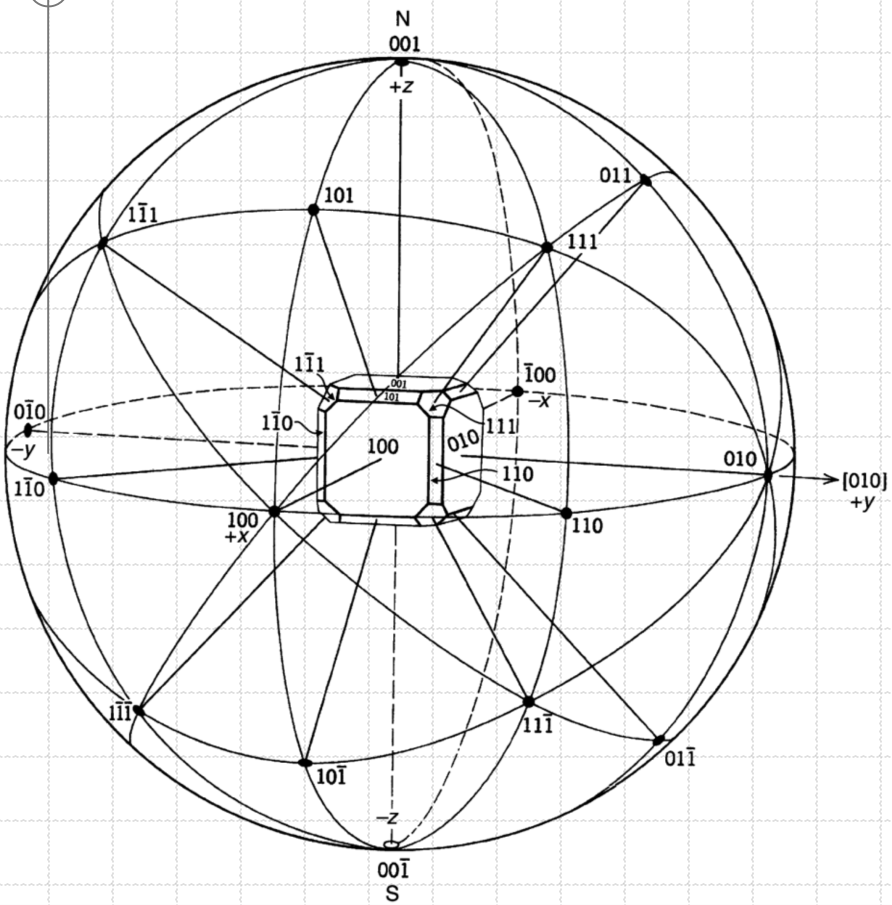
The stereographic projection

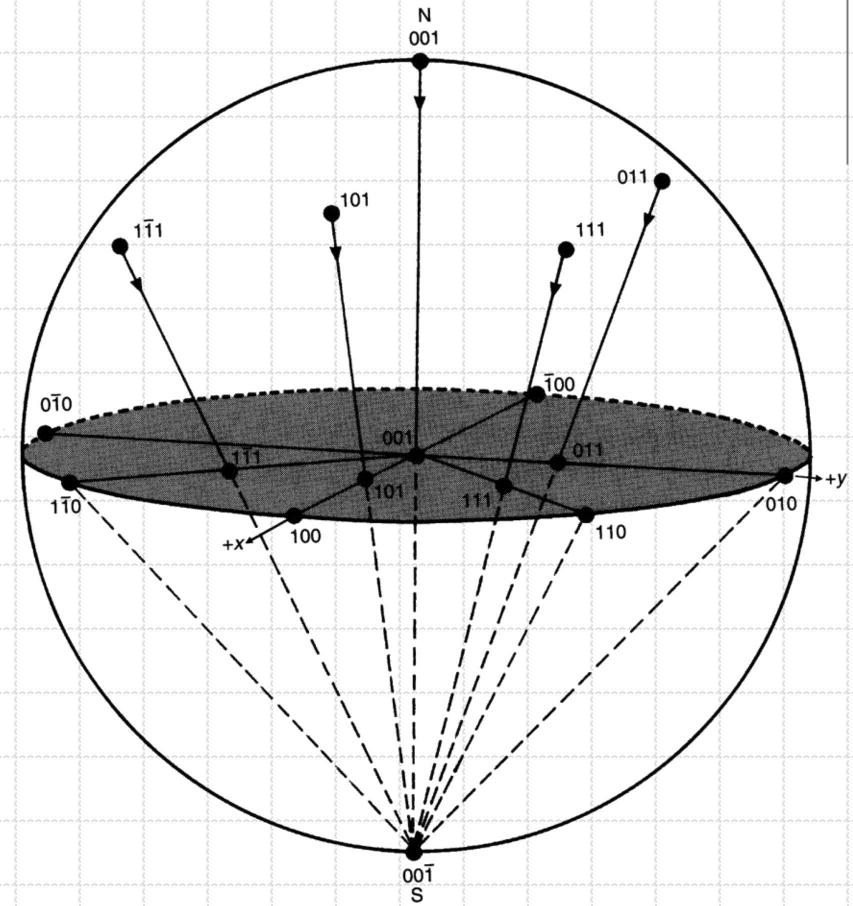
The Stereographic Projection



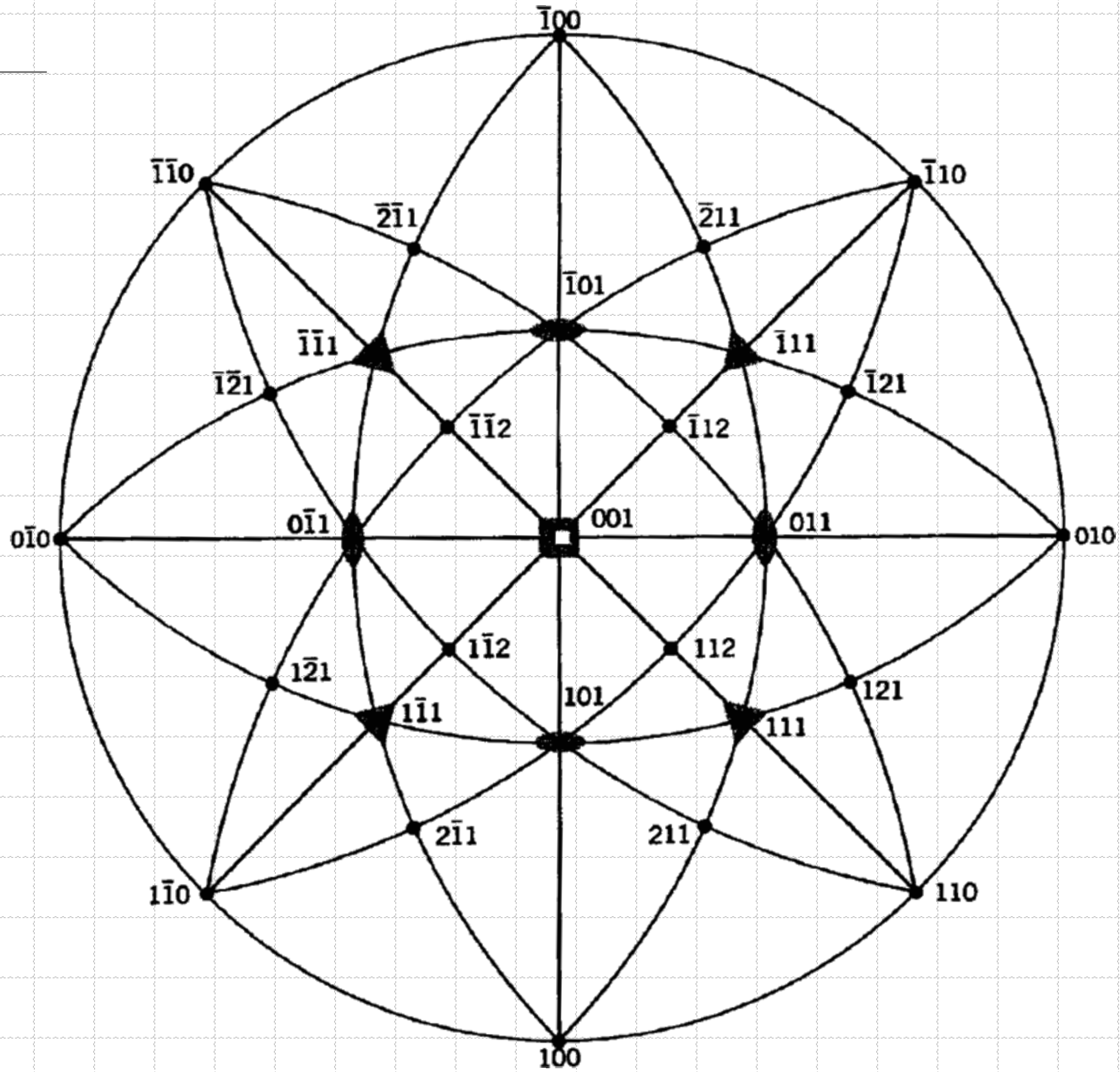


The Pole Figures





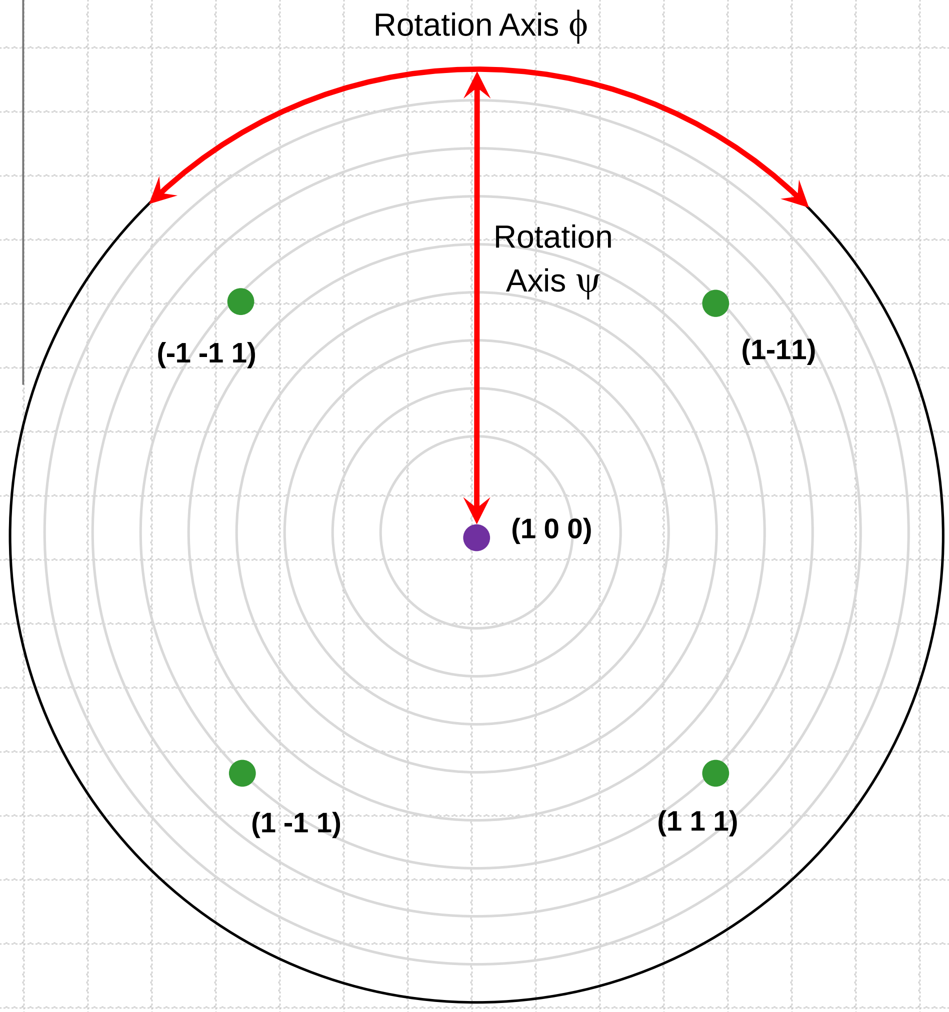
The Pole Figures

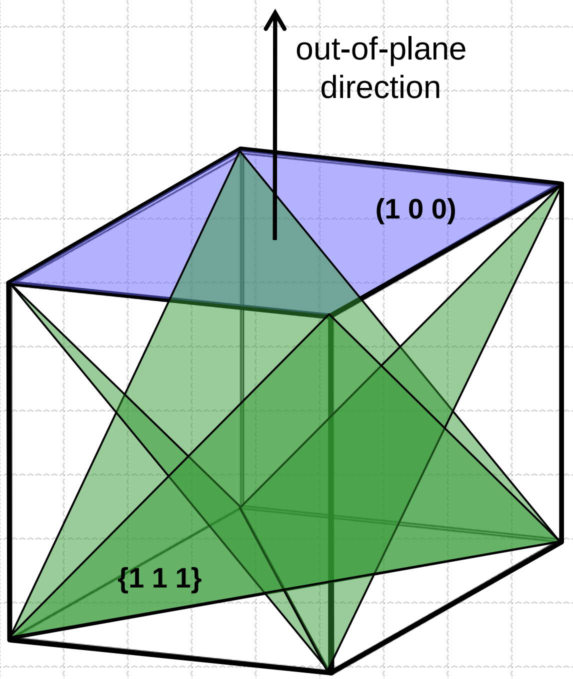


The Pole Figures

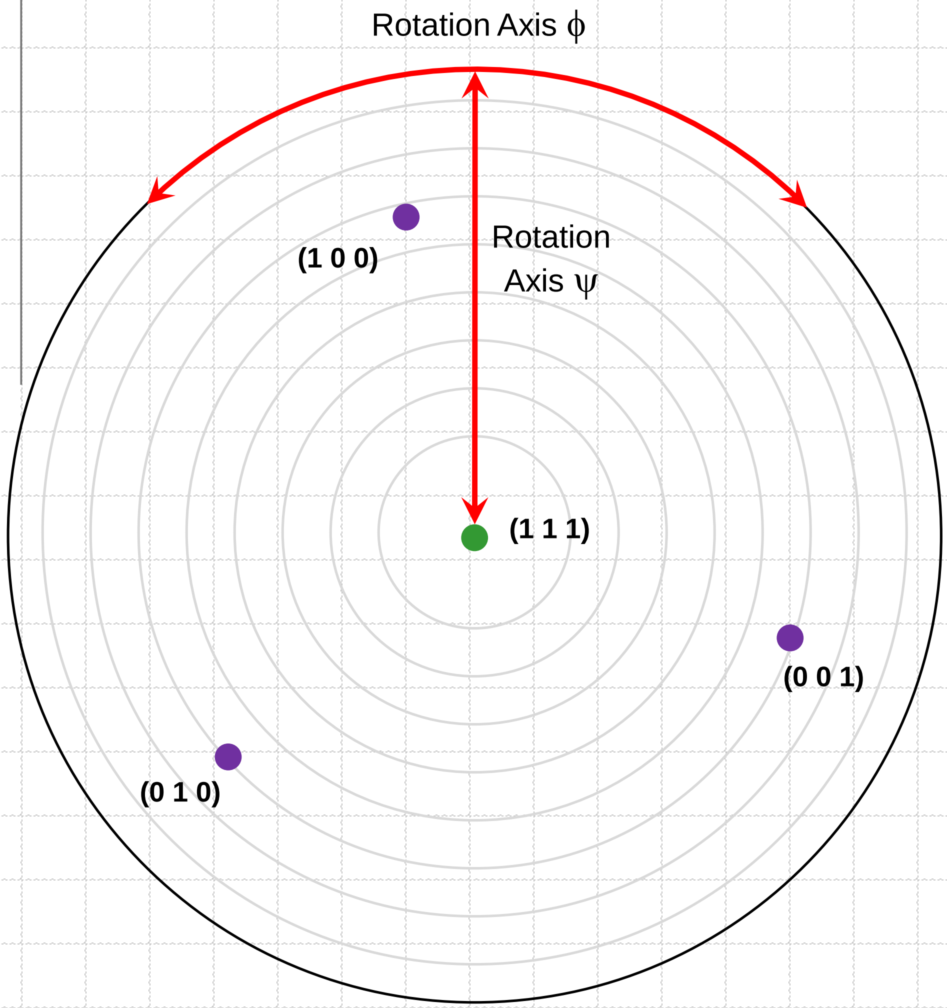
- We now project the sphere with its pole density onto a plane. This projection is called a pole figure.
  - A pole figure is scanned by measuring the diffraction intensity of a given reflection with constant 2θ at a large number of different angular orientations of a sample.
  - A contour map of the intensity is then plotted as a function of the angular orientation of the specimen.
  - The intensity of a given reflection is proportional to the number of hkl planes in reflecting condition.
  - Hence, the pole figure gives the probability of finding a given (h k l) plane normal as a function of the specimen orientation.
  - If the crystallites in the sample have random orientation the contour map will have uniform intensity contours.

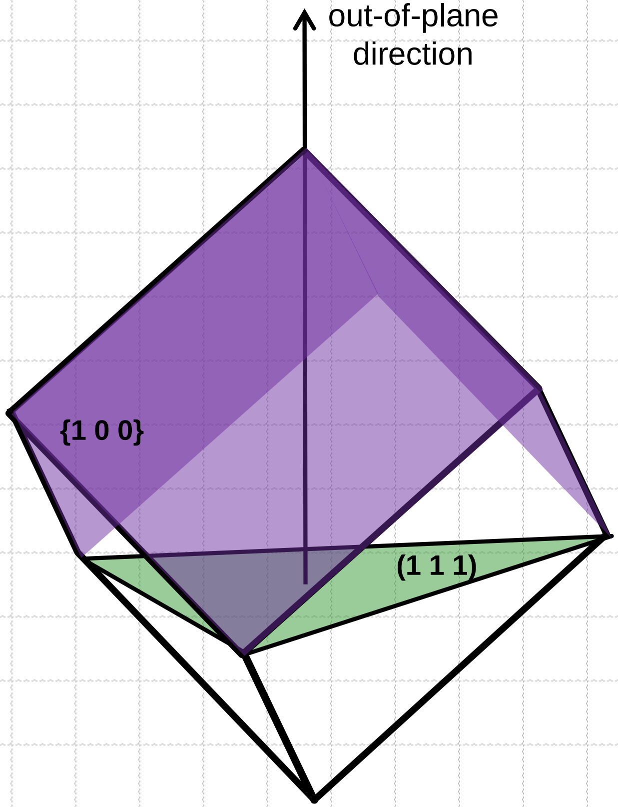
The Pole Figures





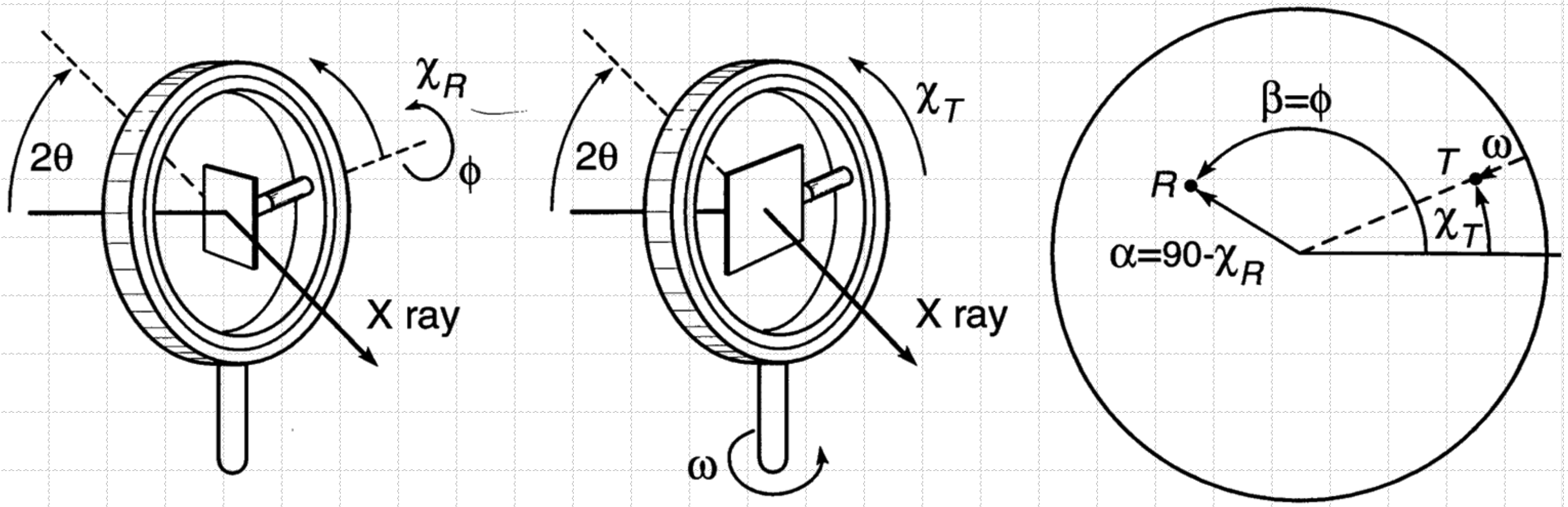
The Pole Figures





Texture Measurements

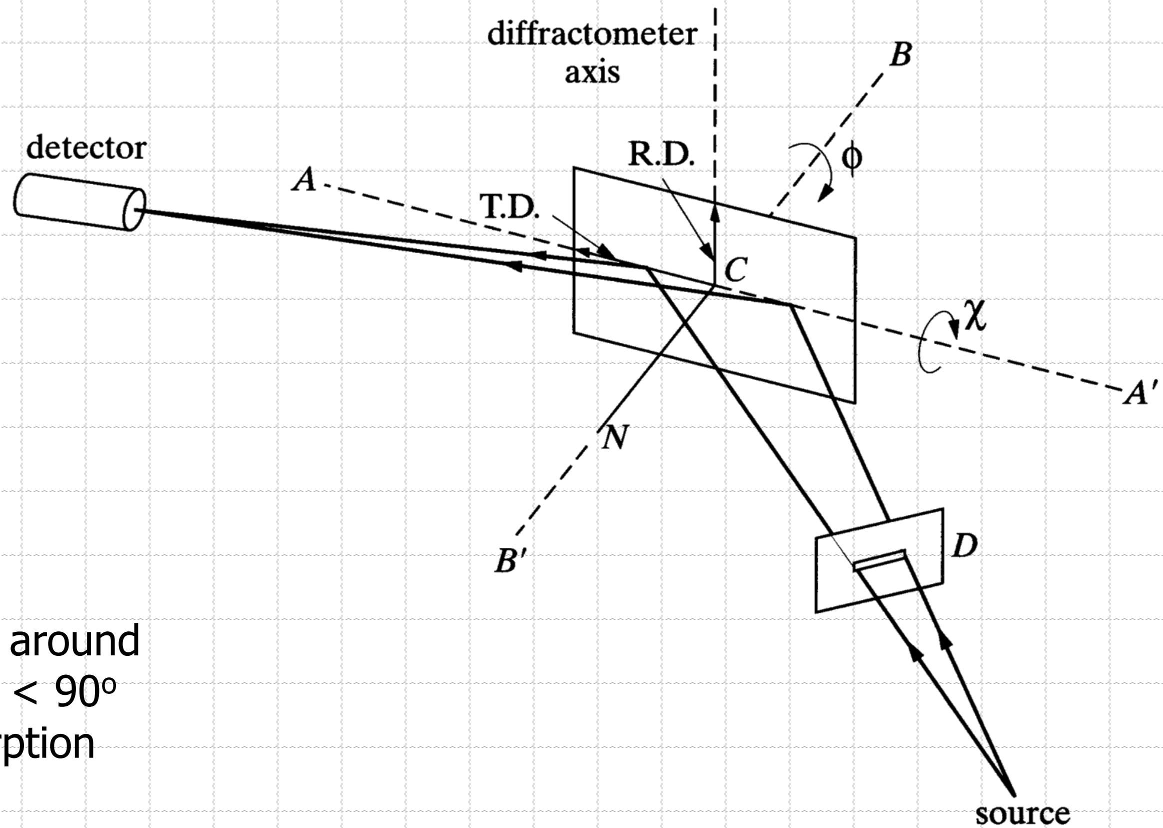
- Requires special sample holder which allows rotation of the specimen in its own plane about an axis normal to its surface, ϕ, and about a horizontal axis, χ.



Reflection Transmission

Schulz Reflection Method

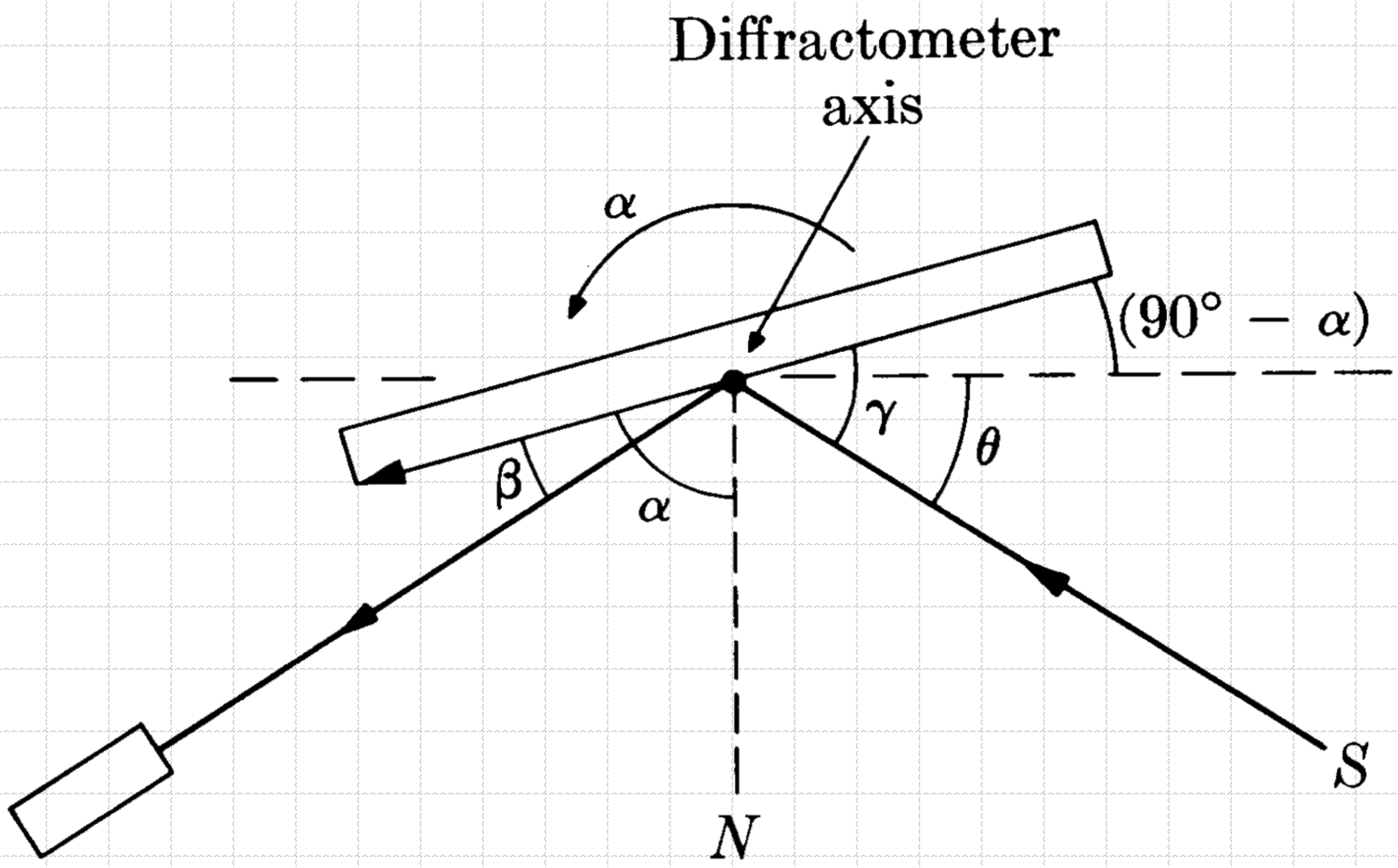
- In the Bragg-Brentano geometry a divergent x-ray beam is focused on the detector.
- This no longer applies when the sample is tilted about χ.



Advantage: rotation in around χ in the range $40^o < \chi < 90^o$ does not require absorption correction.

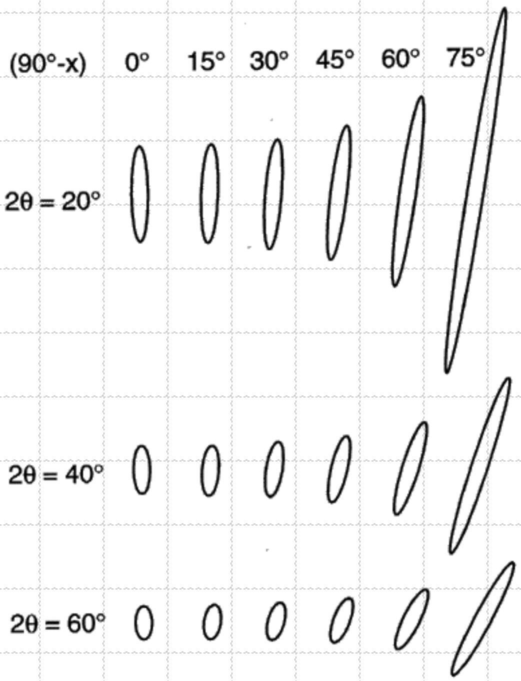
Field and Merchant Reflection Method

- The method is designed for a parallel incident beam.

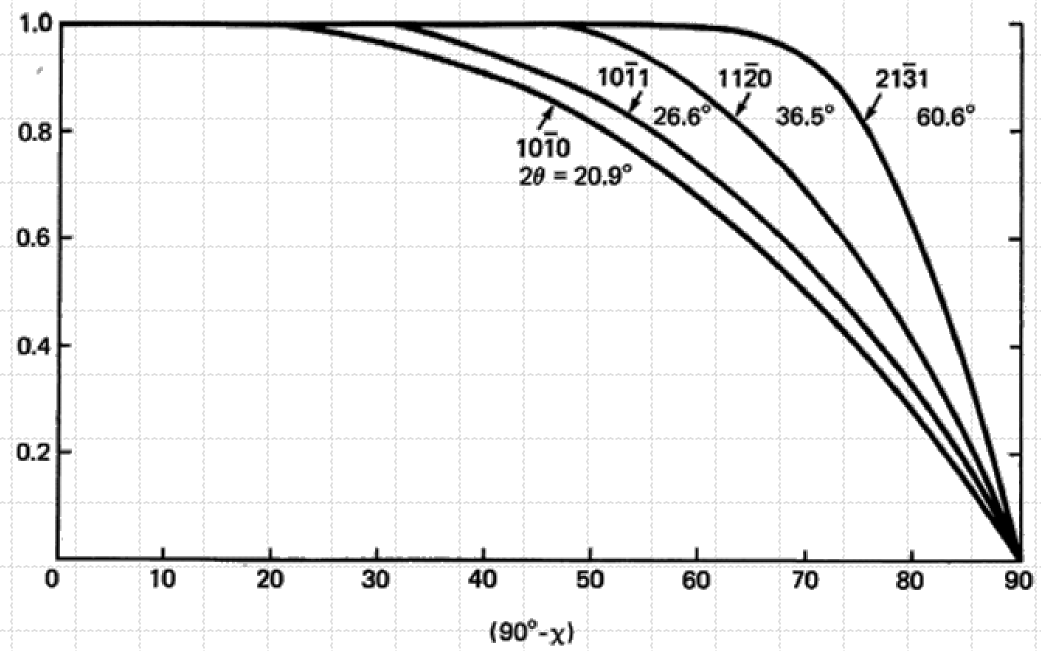


Defocusing Correction

- As sample is tilted in χ, the beam spreads out on a surface.
- At high χ values not all the beam enters the detector.
- Need for defocusing correction.



Change in shape and orientation of the irradiated spot on a sample surface for different sample inclinations as a function of tilt angle α and Bragg angle 2θ. The incident beam is cylindrical.



Intensity correction for x-ray pole figure determination in reflection geometry. Selected reflections for quartz.

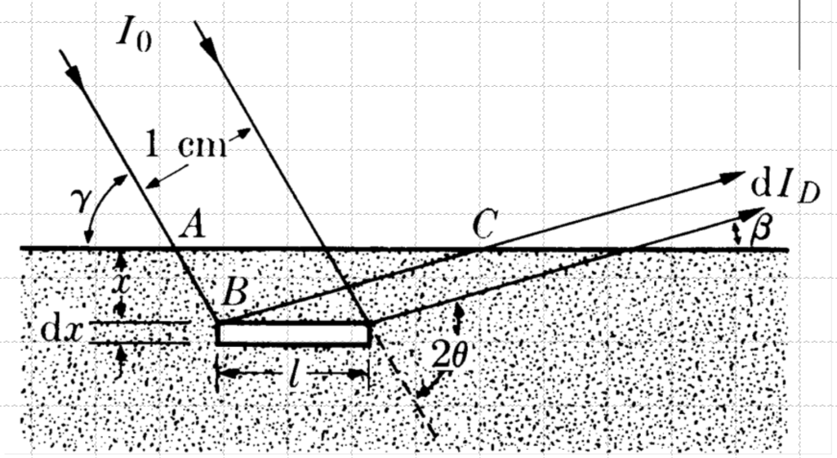
U.F. Kocks, C.N. Tomé, and H.-R. Wenk, "Texture and Anisotropy" 2000.

Absorption Correction

- Diffracted intensities must be corrected for change in absorption due to change in α.

$$dI_D = \frac{I_0 ab}{\sin\gamma} e^{-\mu x(1/\sin\gamma + 1/\sin\beta)} dx$$

a – volume fraction of a specimen containing particles having correct orientation for reflection of the incident beam.
b – fraction of the incident energy which is diffracted by unit volume



substitute

$$\gamma = \theta + (90^o - \alpha),\ \beta = \theta - (90^o - \alpha)$$

Integrate 0 < x < ∞

$$I_D = \frac{I_0 ab}{\mu\{1 - [\cos(\alpha-\theta)/\cos(\alpha+\theta)]\}}$$

We interested in the intensity at angle α, relative to intensity at α = 90°:

$$S = \frac{I_D(\alpha = \alpha)}{I_D(\alpha = 90^o)} = 1 - \cot\alpha\cot\theta$$

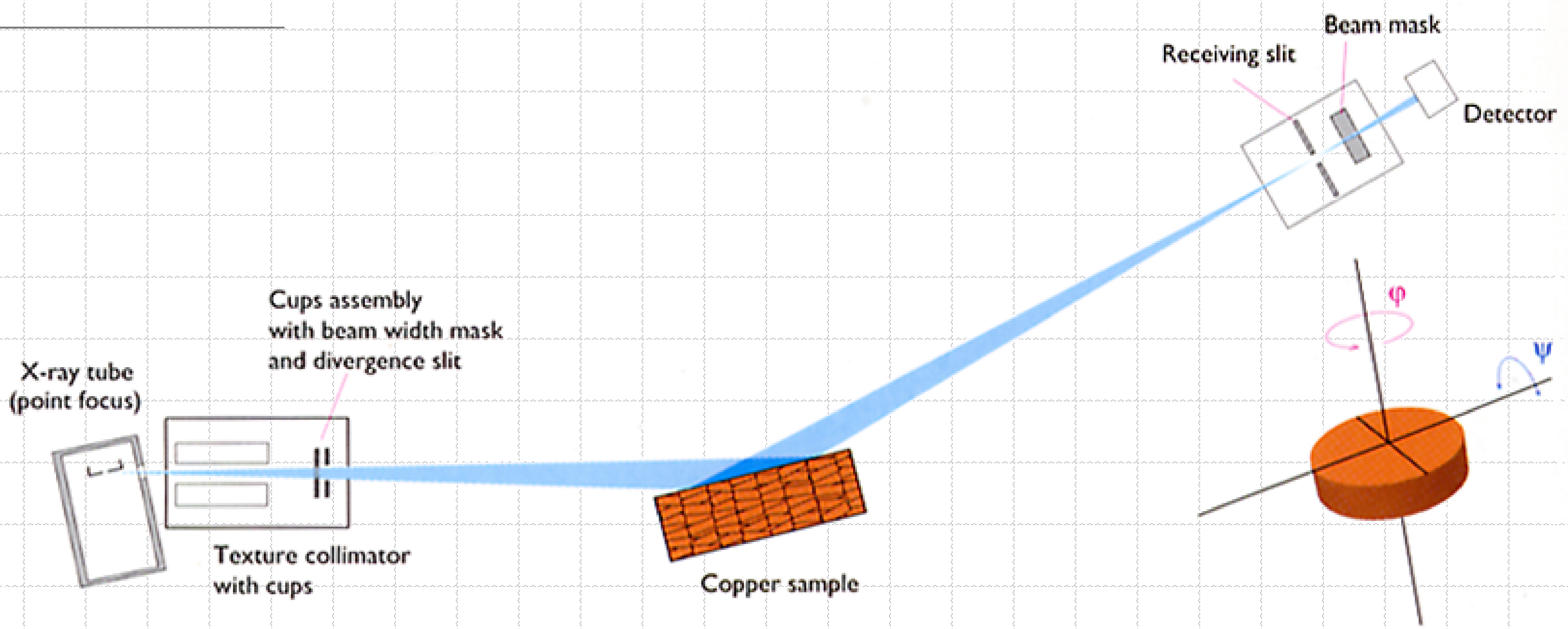
Pole Figure Measurement

- Pole figure diffractometer consists of a four-axis single-crystal diffractometer.

Rotation axes:

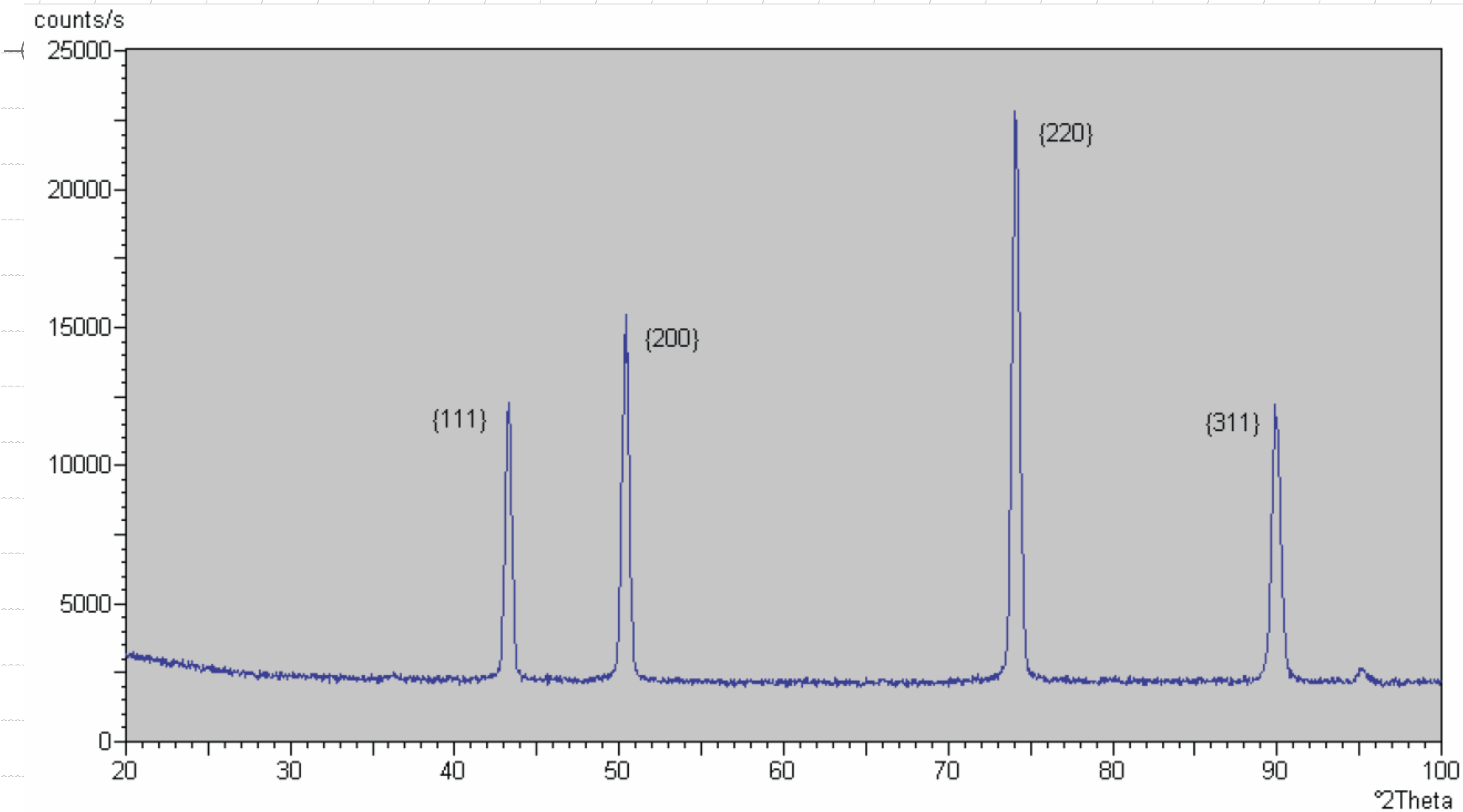
θ, ω, χ, and ϕ

Example: Rolled Copper



- Texture measurements were performed on Cu disk Ø = 22 mm, t = 0.8 mm.
- Four pole figures (111), (200), (220) and (311) were collected using Schulz reflection method.
- Background intensities were measured next to diffraction peaks with offset $2\theta = \pm 4^o$.
- Defocusing effects were corrected using two methods:
  - measured texture free sample
  - calculated (FWHM of the peaks at $\psi = 0^o$ is required – obtained from θ-2θ scan.
- X'Pert Texture program was used for quantitative analysis.

Example: Rolled Copper

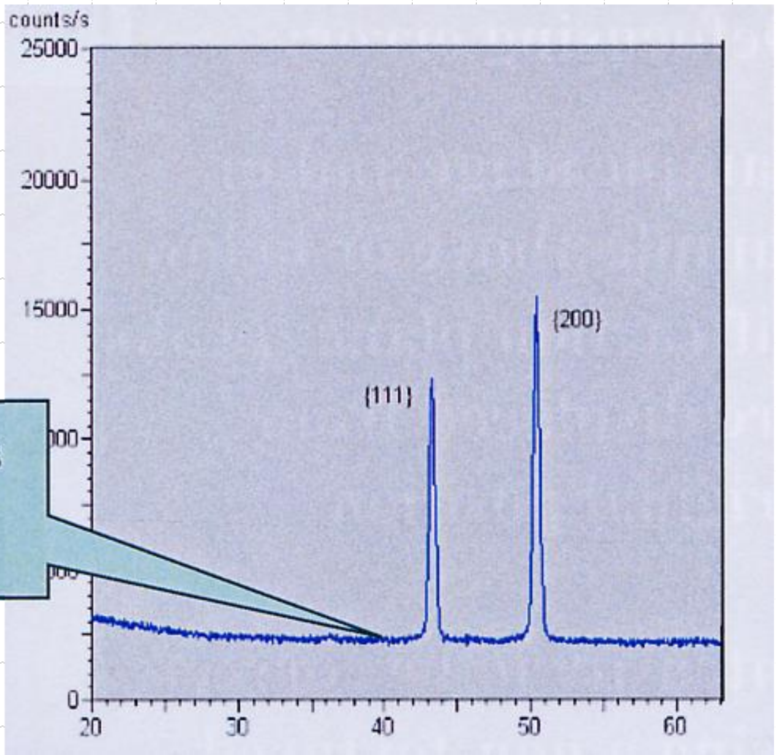


- Measure θ-2θ scan in order to determine the reflections used for the pole figure measurements.
- Use FWHM of the peaks to calculate the defocusing curve.

Example: Rolled Copper

Background Correction

- Pole figure intensities include background.
- Correction for background radiation is performed by measuring the intensity vs. ψ-tilt next to the diffraction peak.

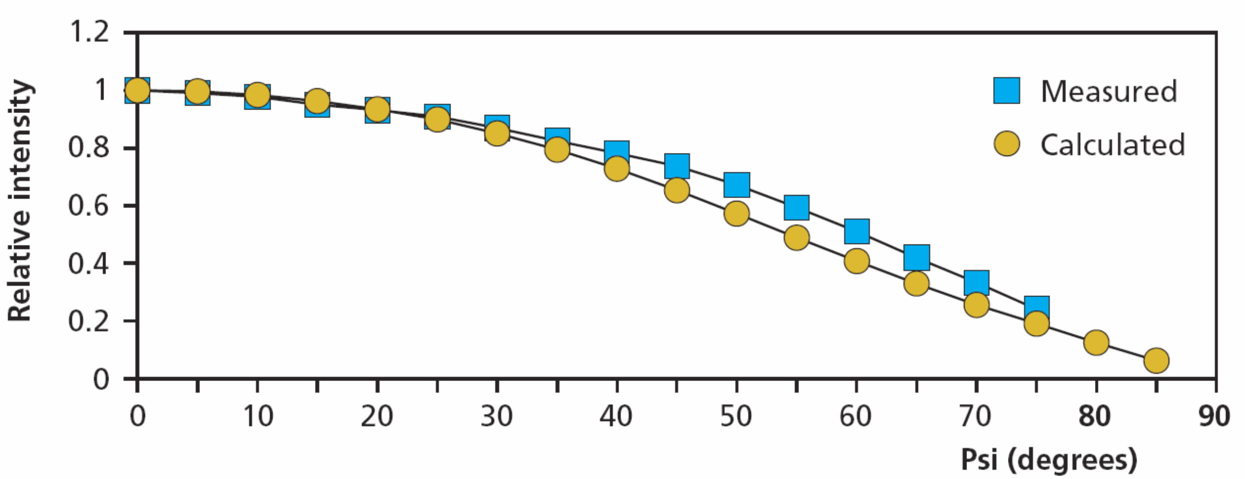


Background correction measurements beside the peaks

Example: Rolled Copper

Corrections

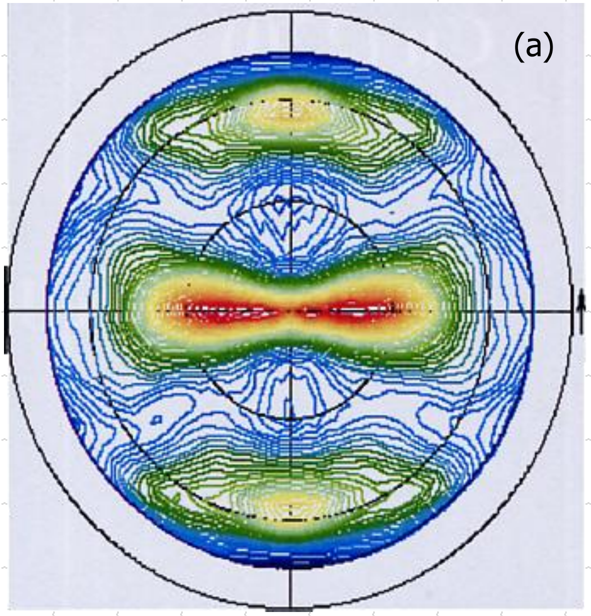
- Experimental pole figures are corrected for background intensities.
- Either experimental or theoretical defocusing correction curve is applied.

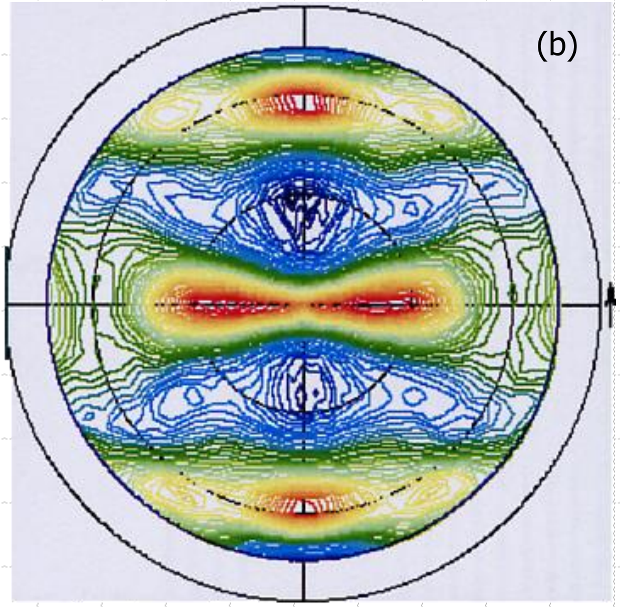


Measured and calculated defocusing corrections for Cu(220) pole figure

Example: Rolled Copper

Corrections



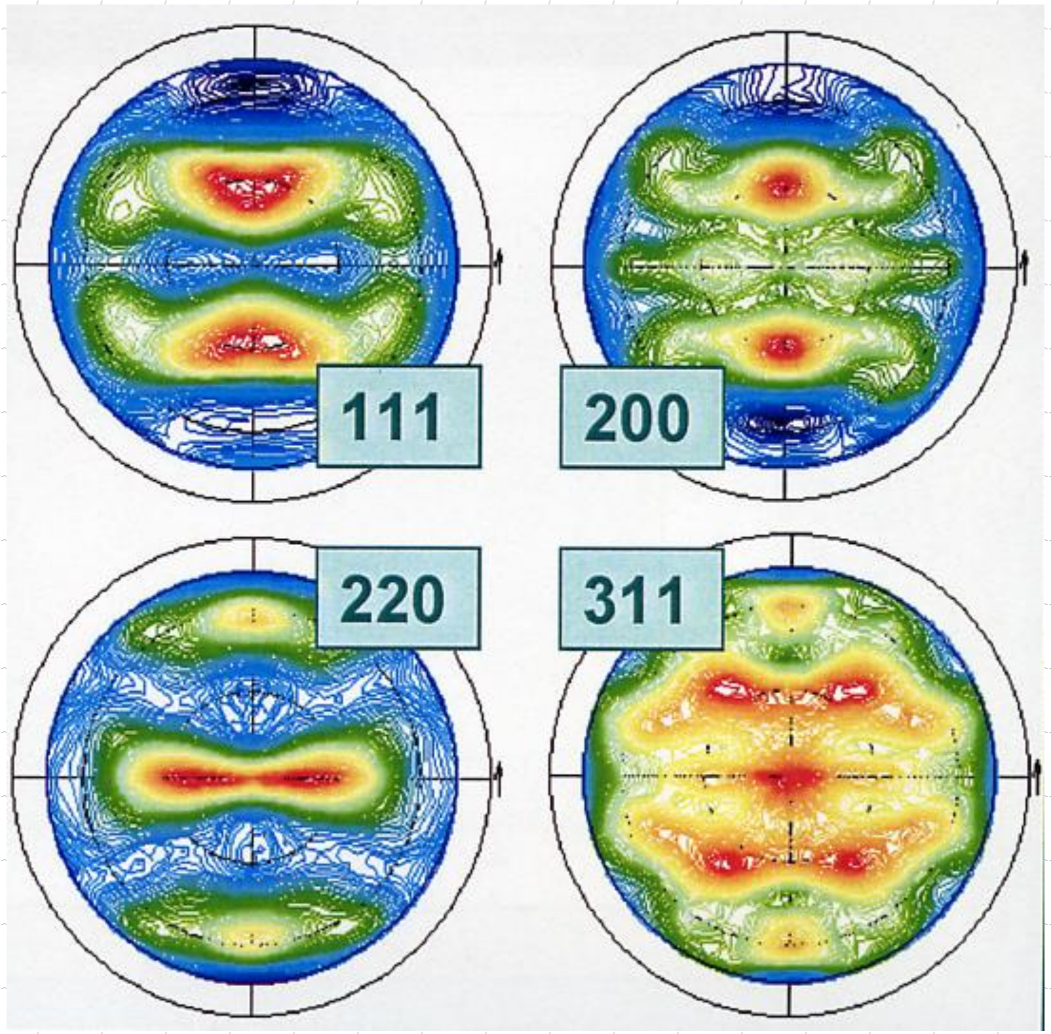


- 2D representation of Cu(220) pole figure as (a) measured and (b) corrected.
- The most noticeable effect is at higher ψ-tilt angles.

Example: Rolled Copper

Pole Figure Measurements

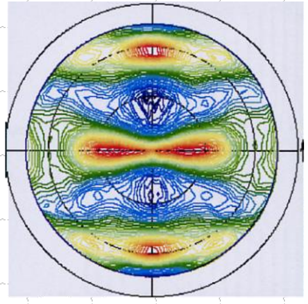
- Four pole figures have been measured.
- Symmetry of rolling process is obtained from the pole figures:
  - pole figure is symmetrical around $\phi = 0^o$ and $\phi = 90^o$.
- The symmetry is called orthorhombic sample symmetry.

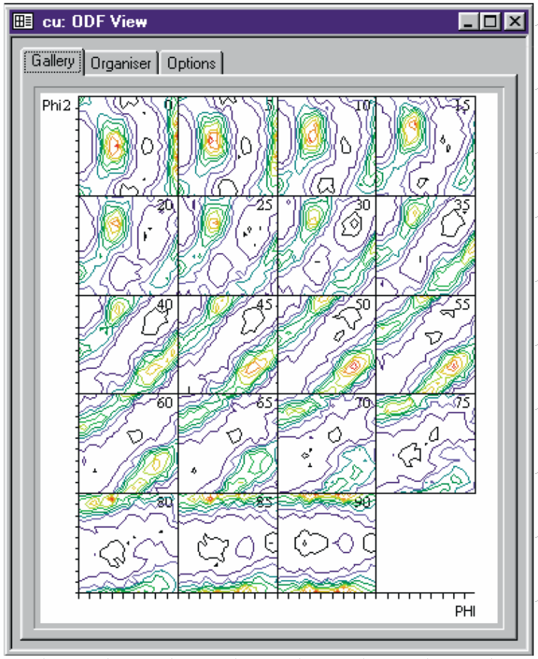


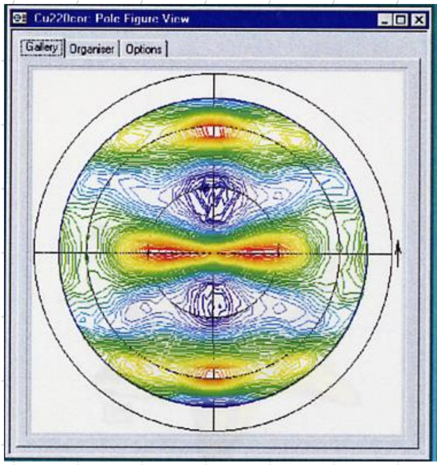
Example: Rolled Copper

Orientation Distribution Function Calculation

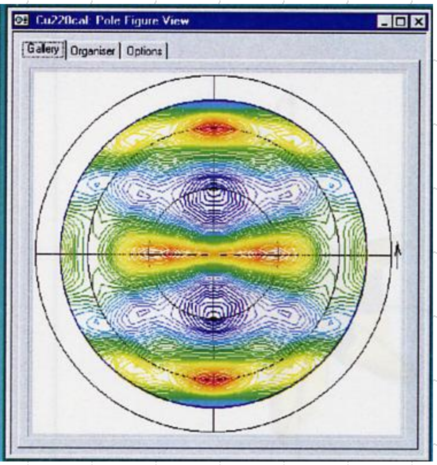
- X'Pert Texture calculates ODF
- When ODF is available X'Pert Texture can calculate pole figures and inverse pole figures for any set of (hkl).





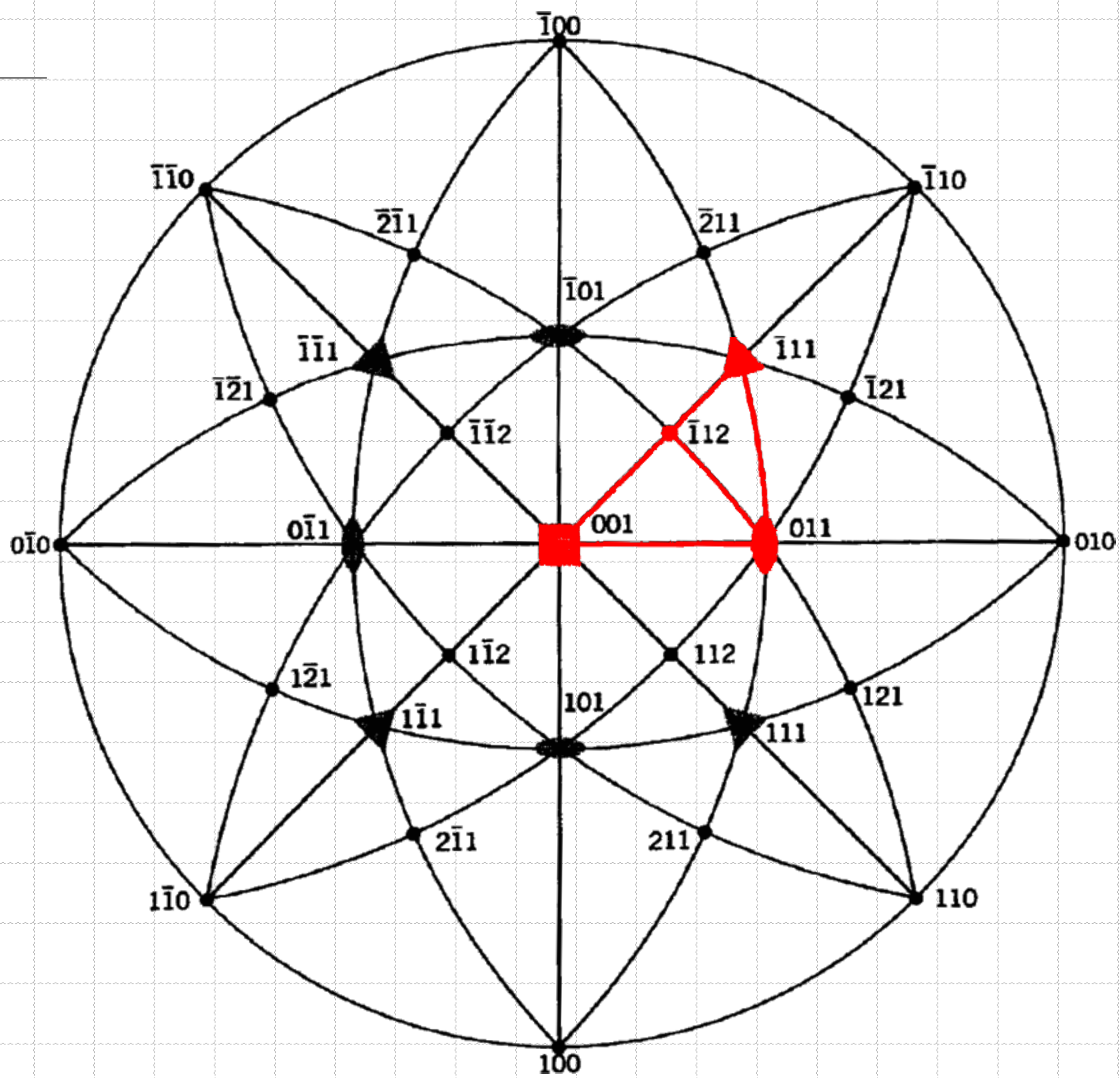


Measured

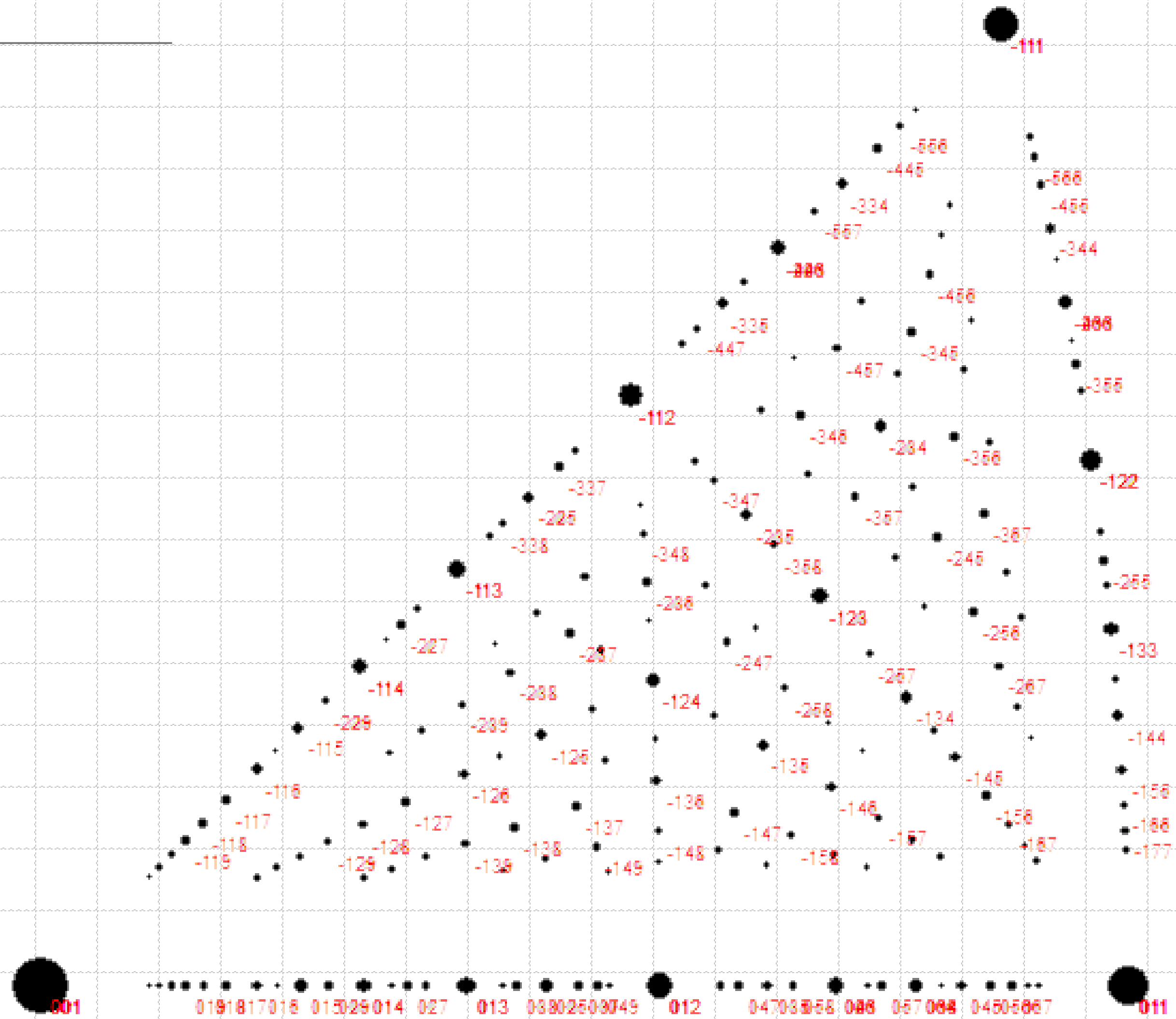


Calculated

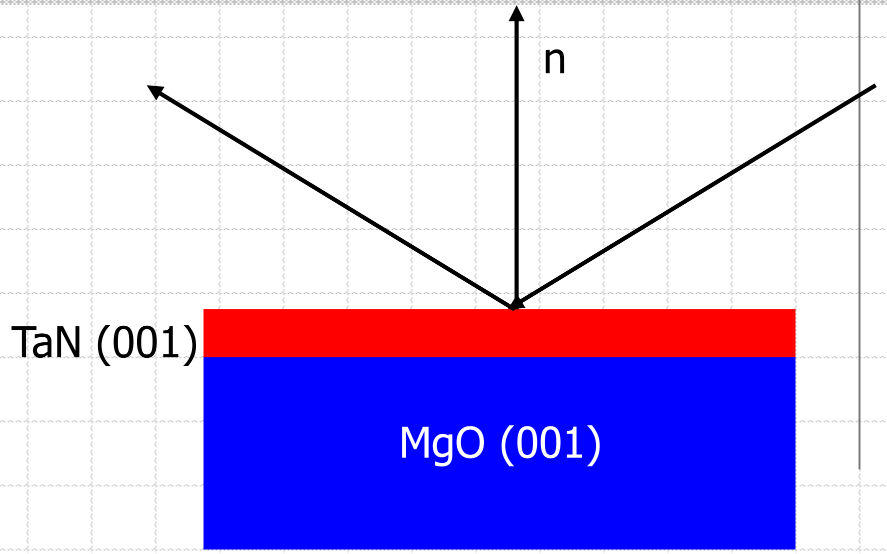
Inverse Pole Figures

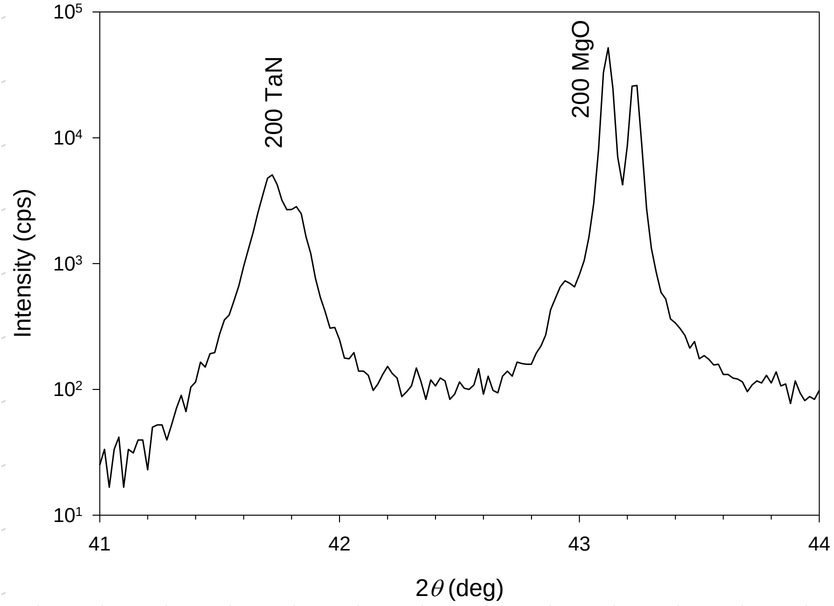


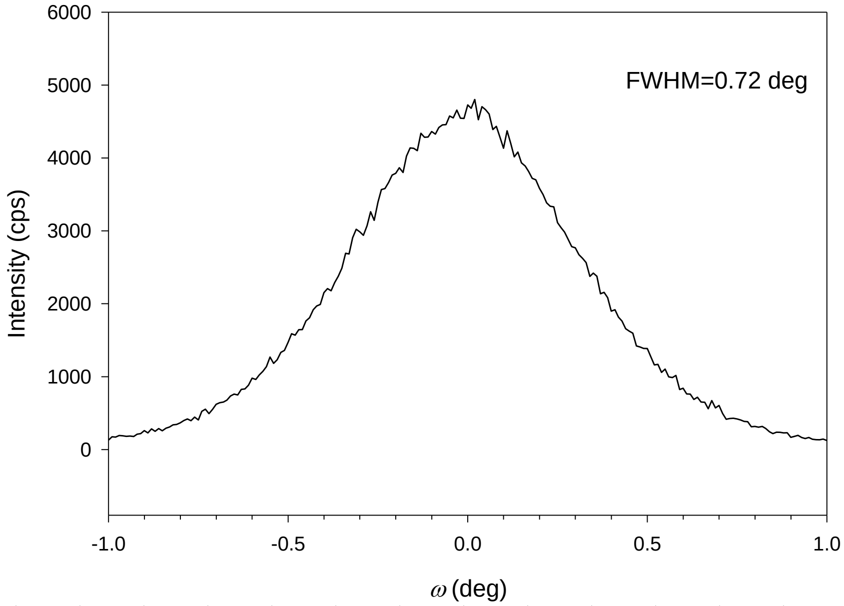
Inverse Pole Figures



TaN Thin Film

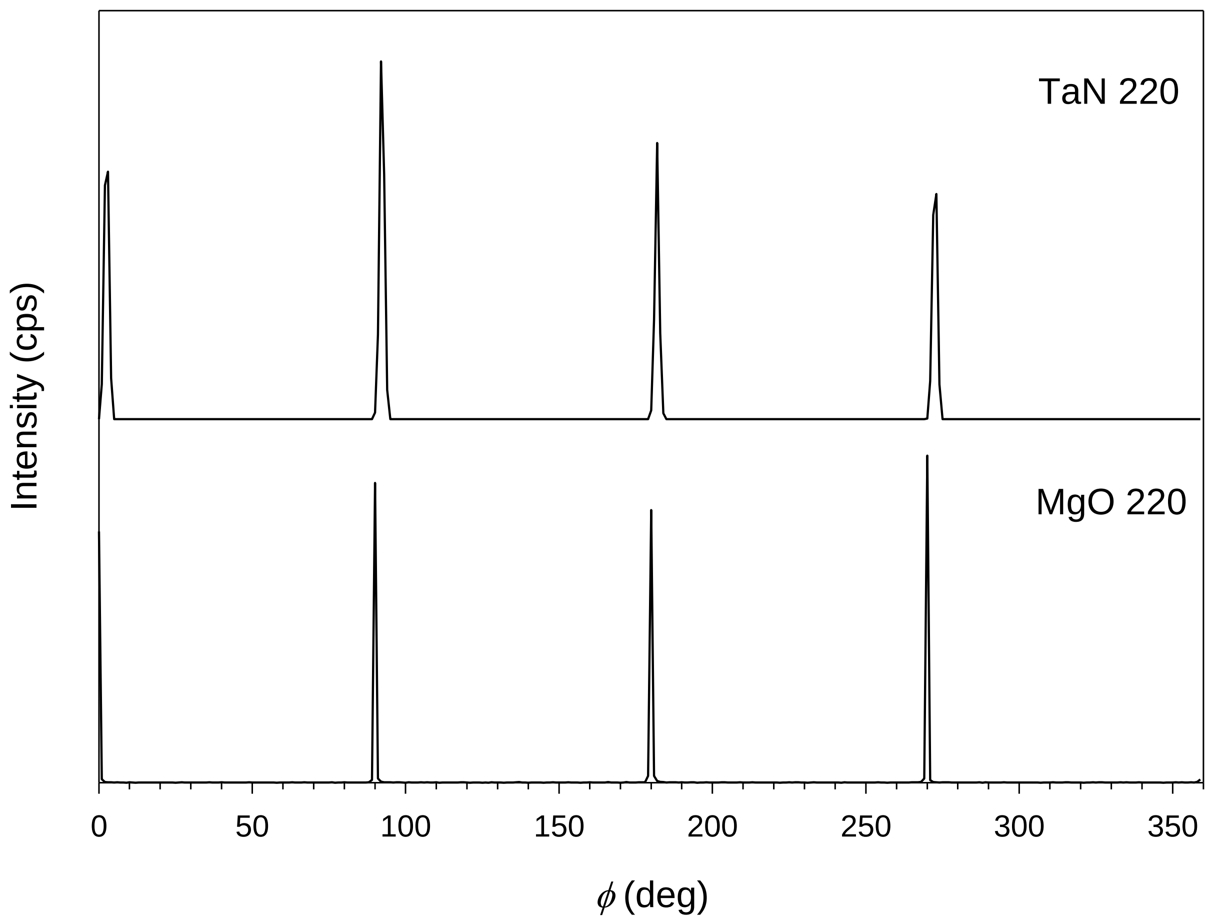






TaN Thin Film

- ϕ-scan of TaN (202) and MgO (202) reflections.



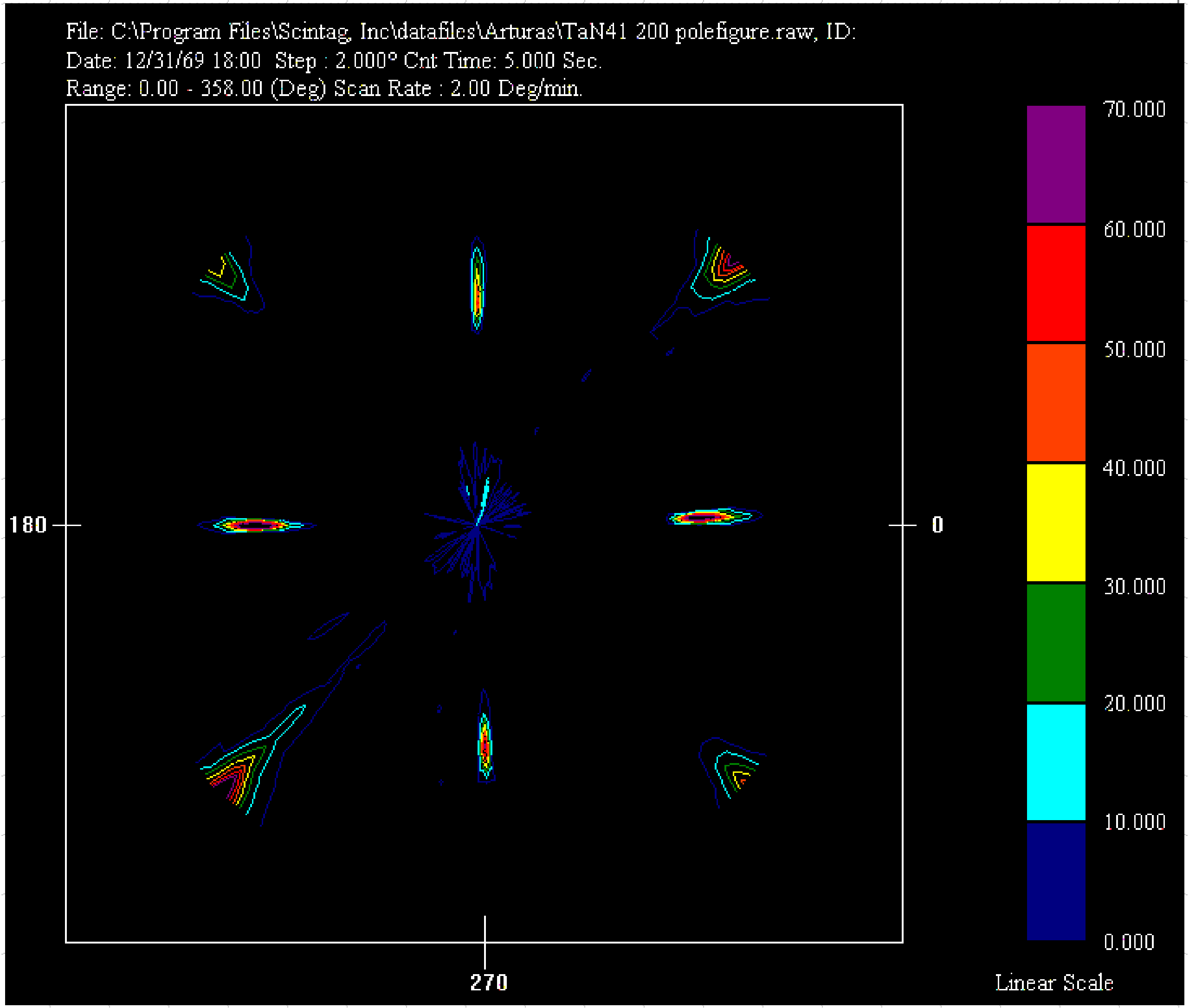
TaN Thin Film

- TEM reveals additional structure.

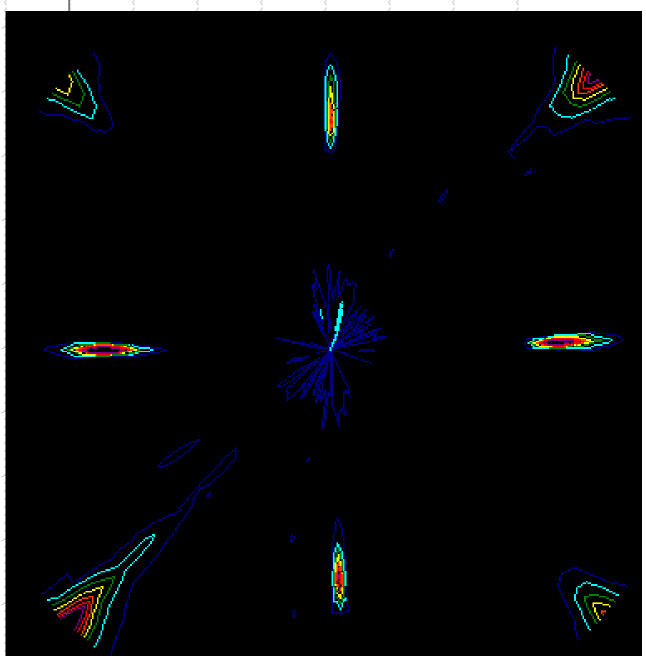
TaN23

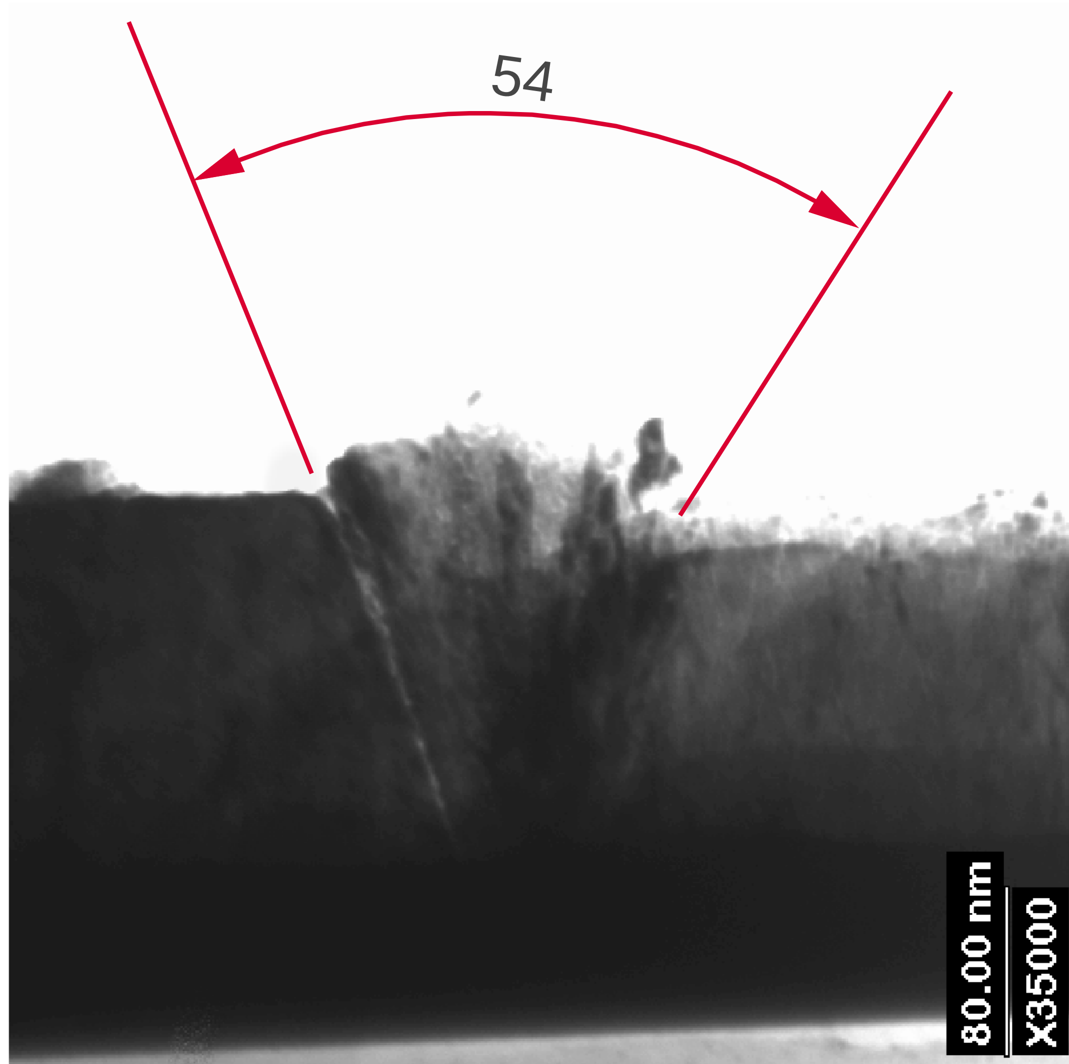
TaN Thin Film

- XRD 002 pole figure of TaN film



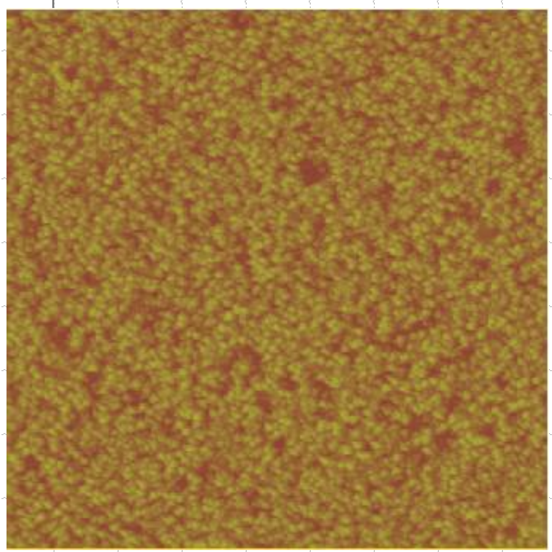
TaN Thin Film



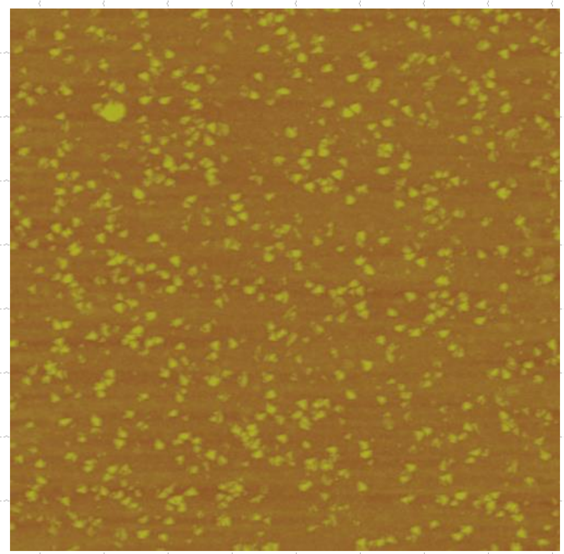


TaN Thin Film

- Atomic Force Microscopy images of TaN films prepared under different N_2 partial pressure



p_N=2 mTorr



p_N=2.5 mTorr

p_N=4 mTorr