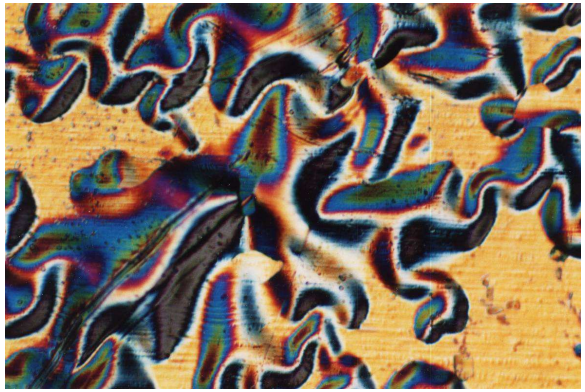
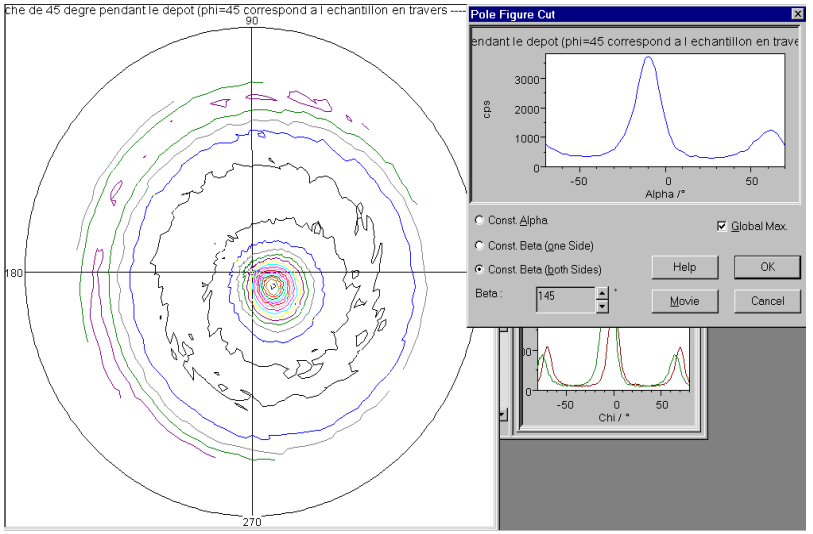
II
Texture analysis

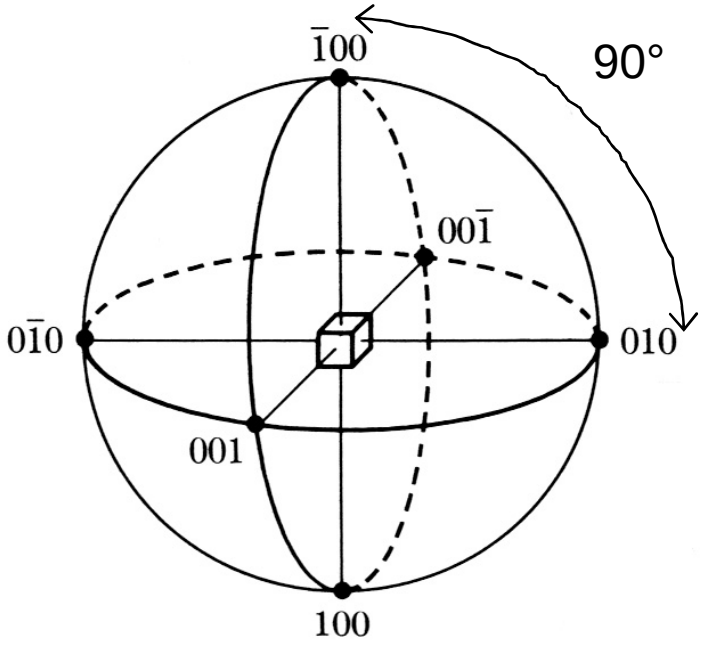
Outlines: -stereographic projection
-crystal orientation
-pole figure





What is a stereographic projection? For illustration, let us consider cubic systems

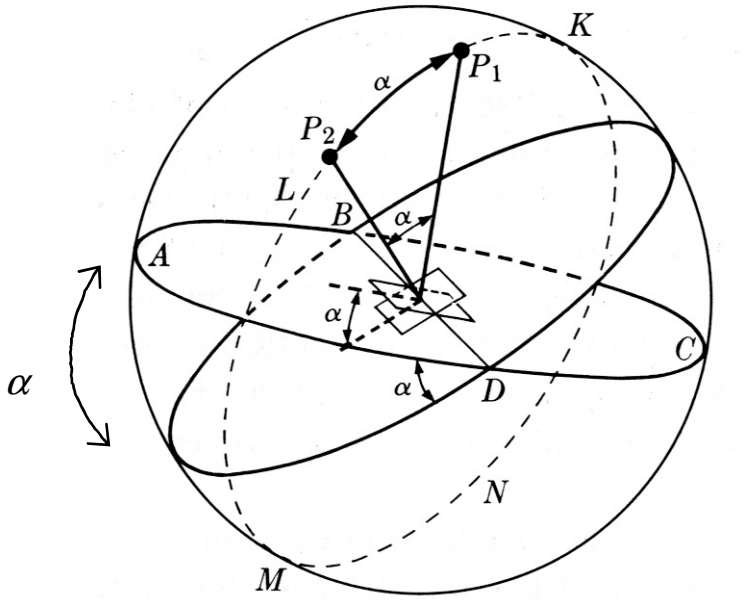
- Using the inclination of the normal to the crystallographic plane. Ex: {100} poles of a cubic crystal



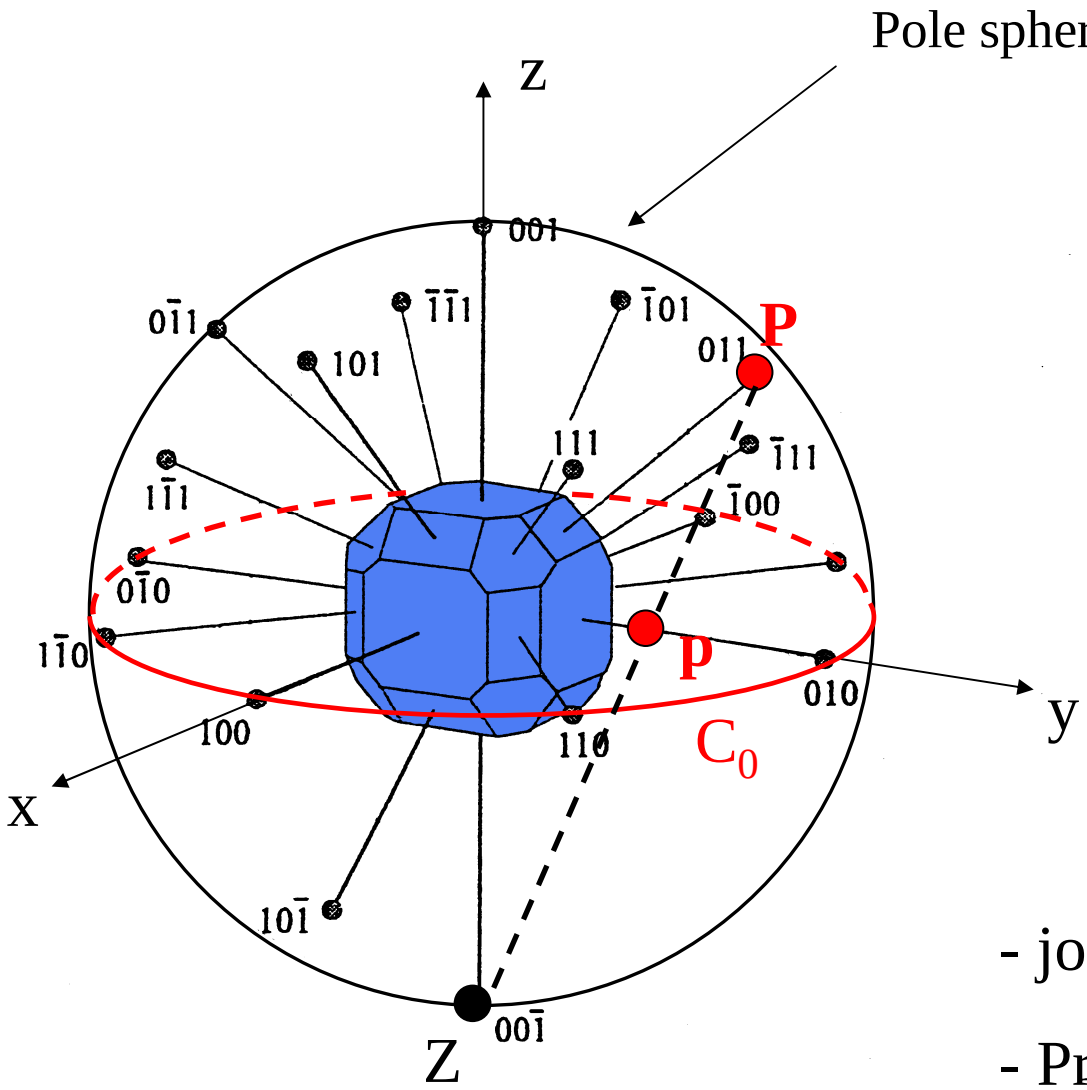
- Angles between two planes:

-Circles ABCD (passing through the center of the sphere)

-Pi normals to the planes



Stereographic Projection : to represent in a plane view complex phenoma which append in the 3D space.

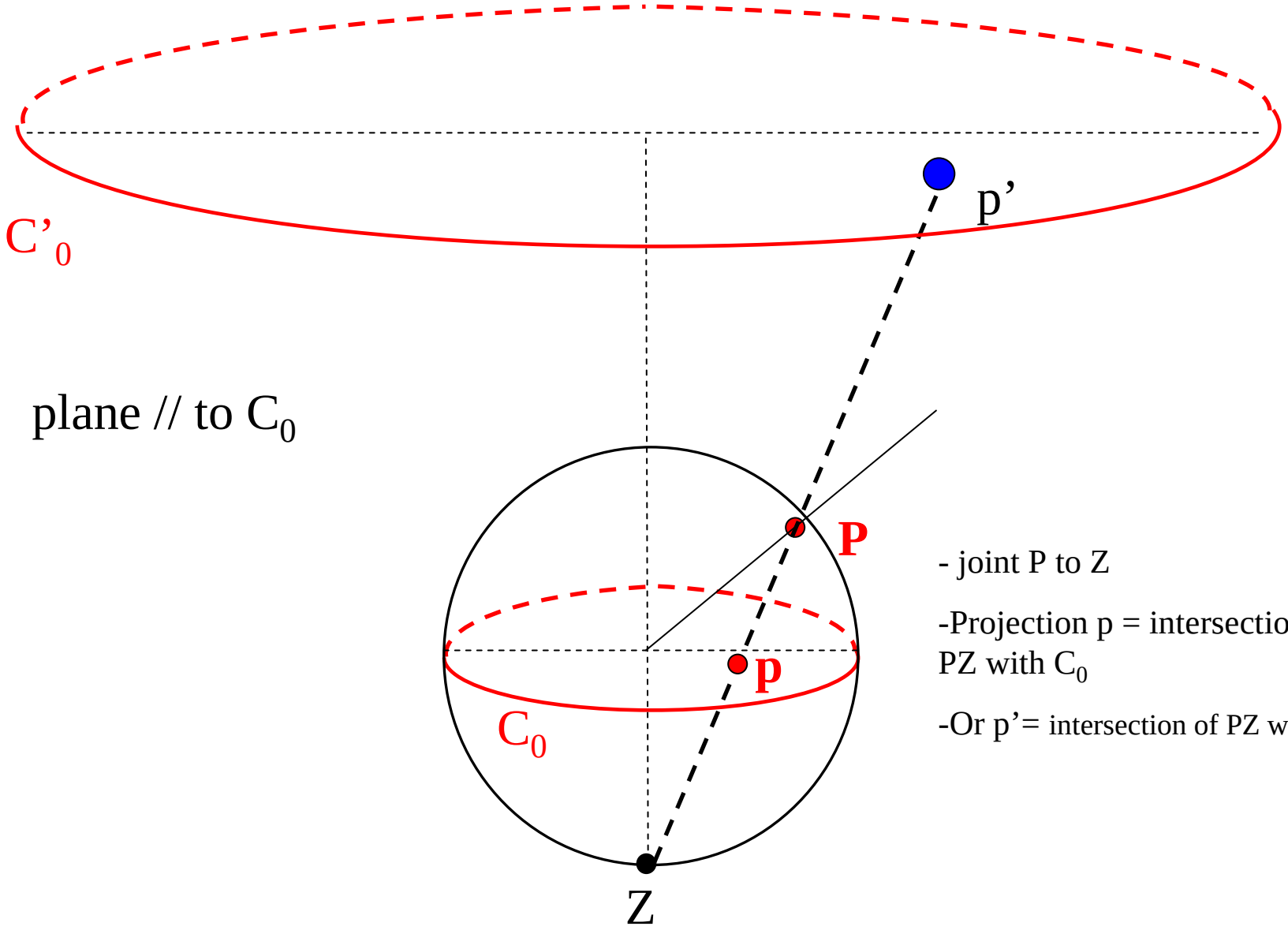


Pole sphere

Proj.

Plane representation in the C_0 plane or in a plane // to C_0

- joint P to Z
- Projection p = intersection of PZ with C_0

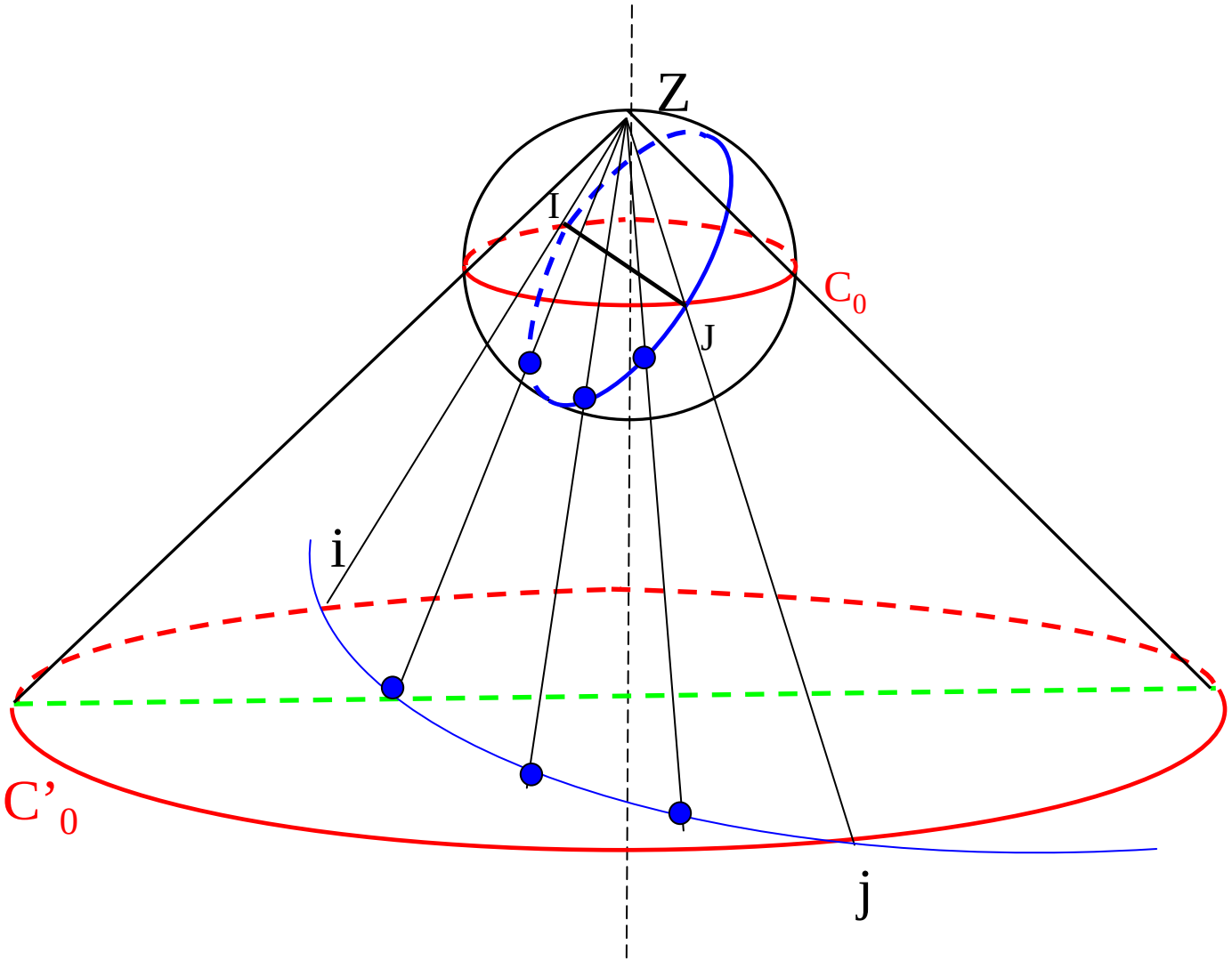


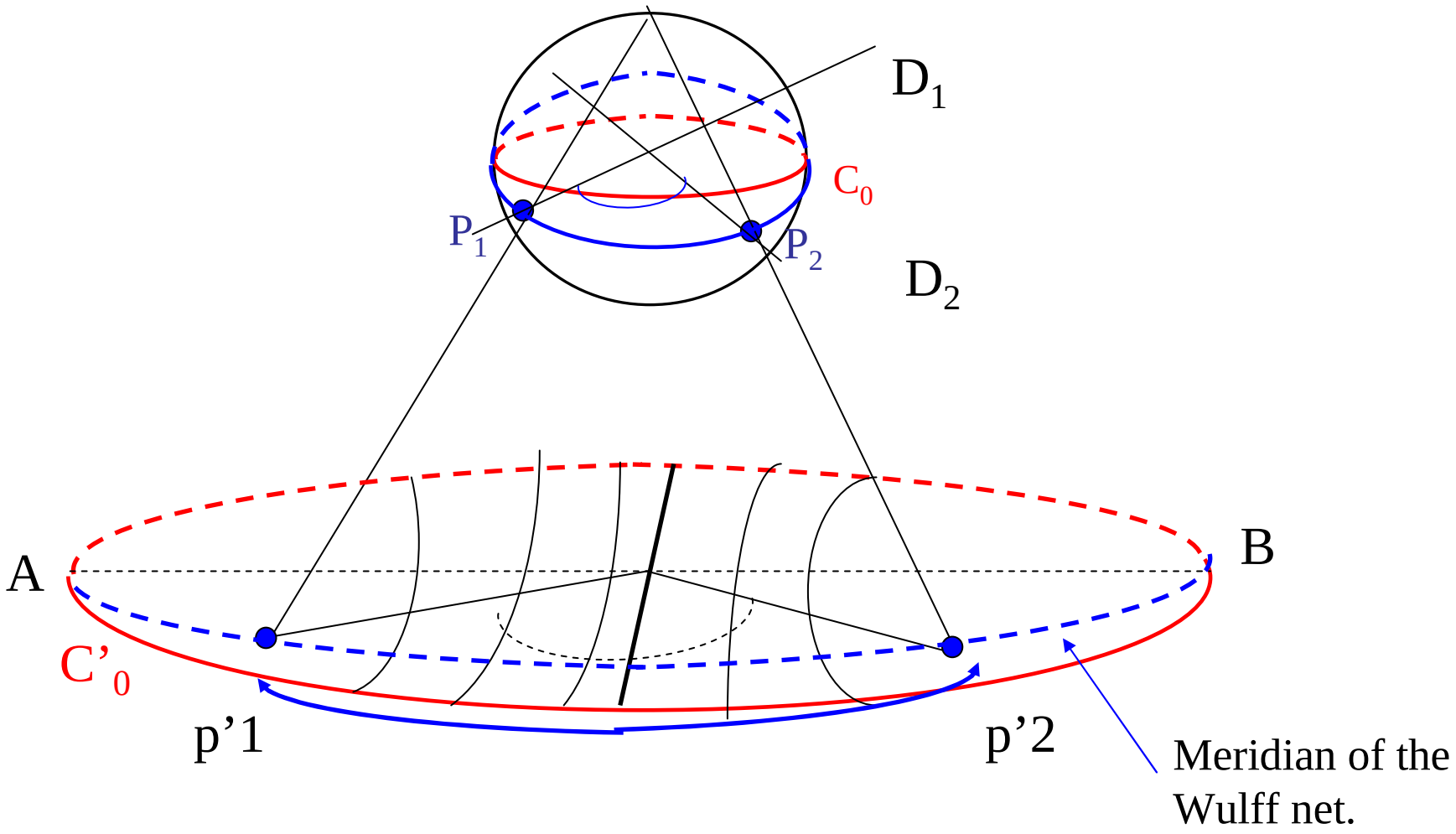
- joint P to Z

-Projection p = intersection of PZ with C_0

-Or p'= intersection of PZ with C'_0

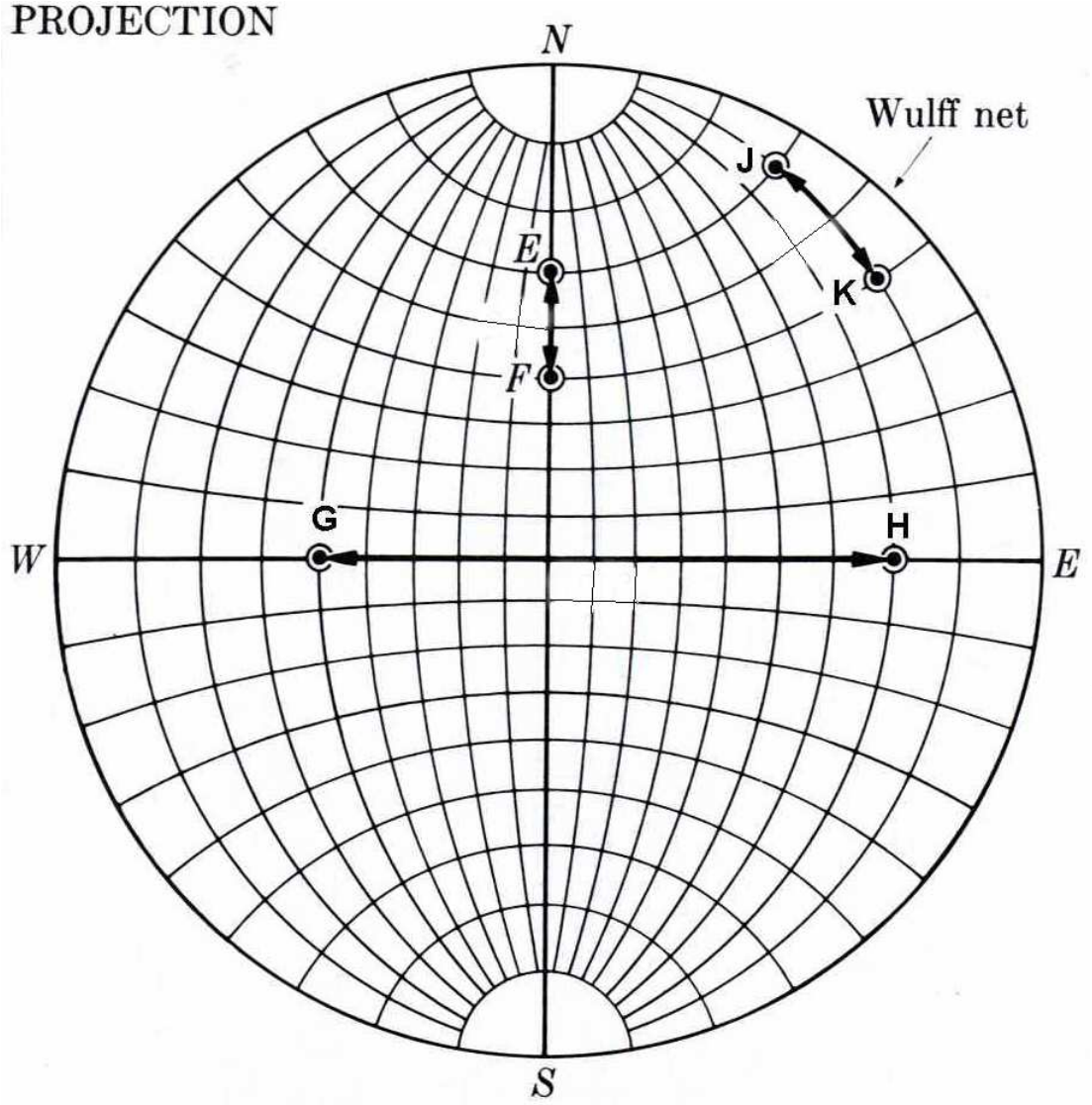
If we consider a given plane (blue circle) and several direction (and pole) in this plane, the projection of this plane is represented by a curve in the plane C'0.

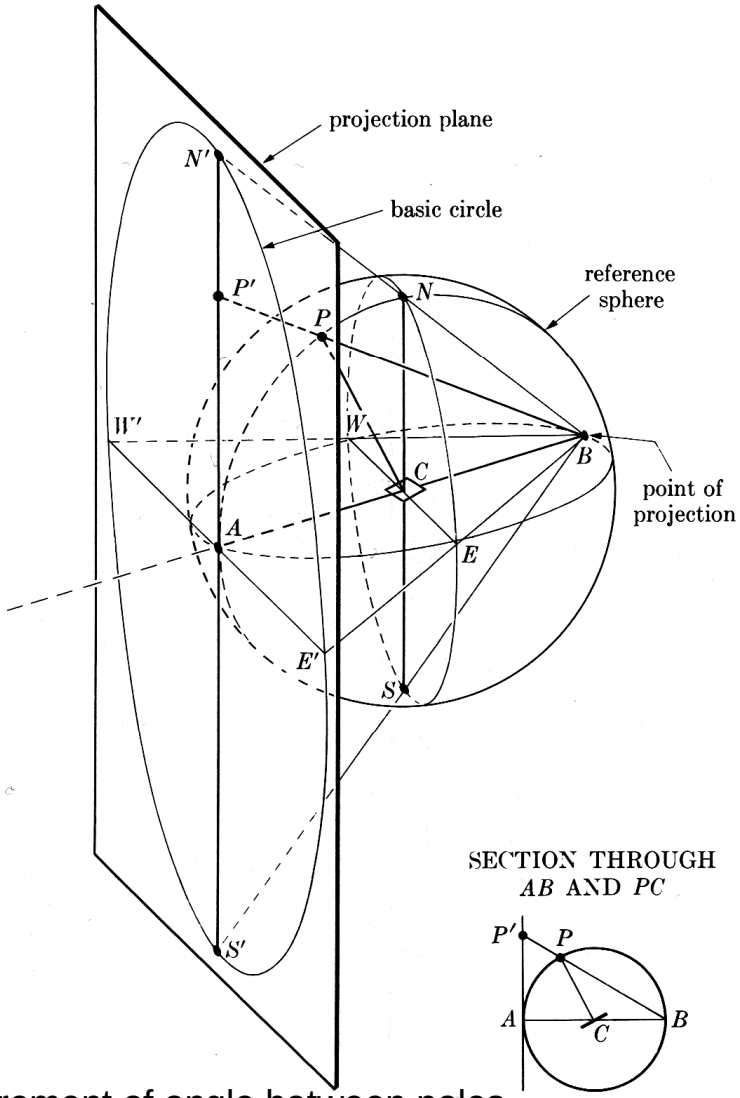




2) The 2 poles directions D1 and D2 belong to the same meridian

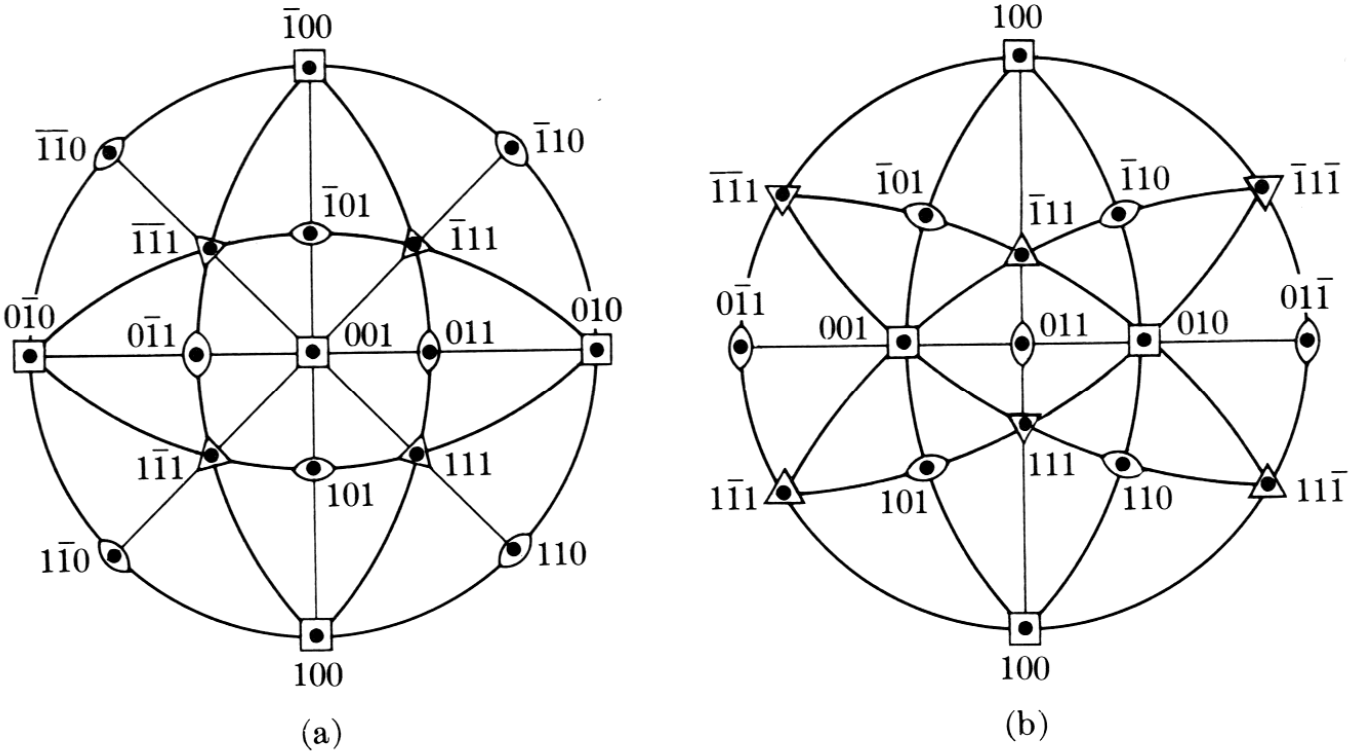
The Stereographic Projection





Stereographic projection superimposed on Wulff net for measurement of angle between poles. The Wulff net is graduated at 10° intervals for illustrat ive purposes. *(angles on the stereo. Proj. ?)*

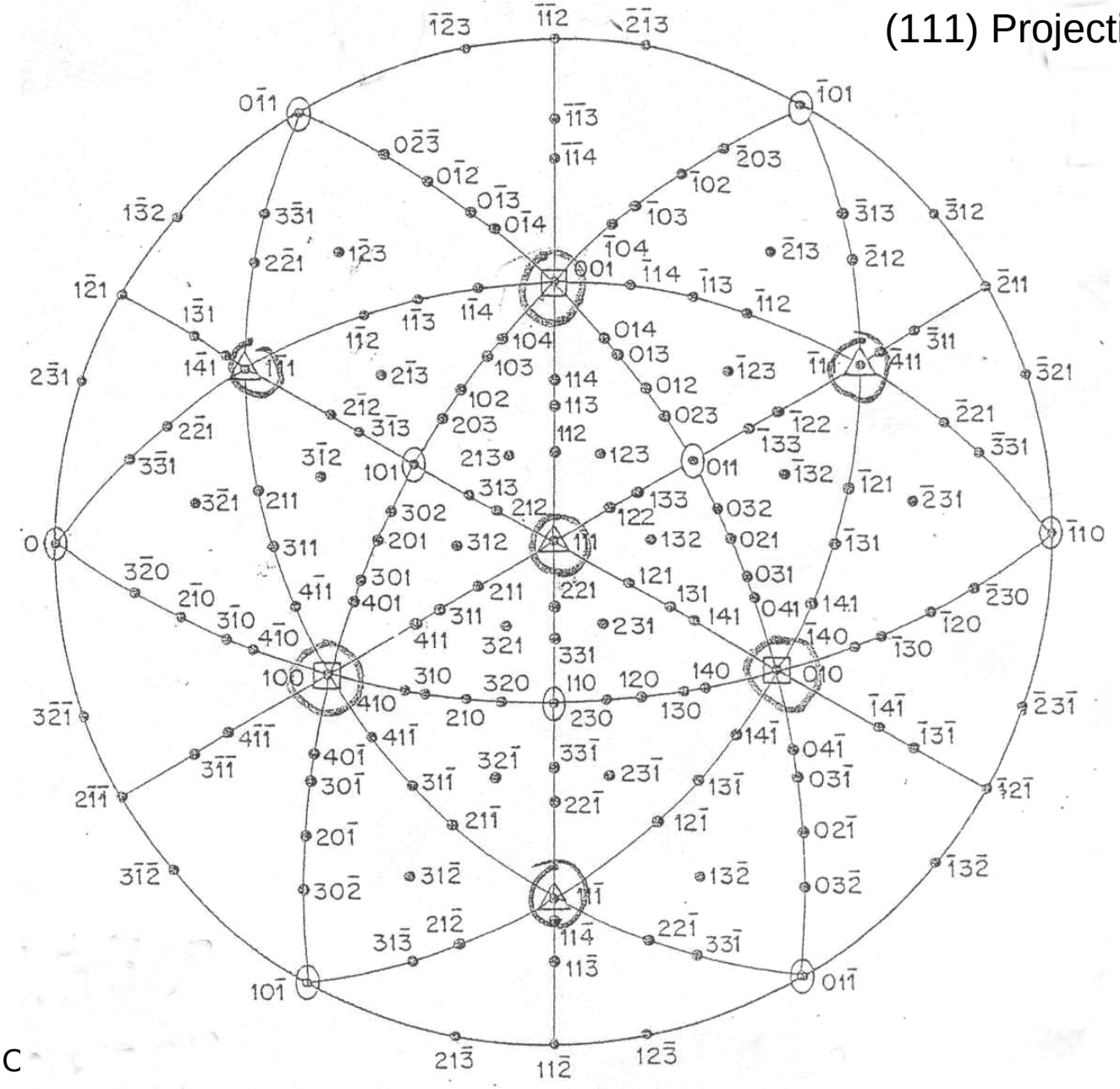
(001) and (011) Projection of cubic crystals



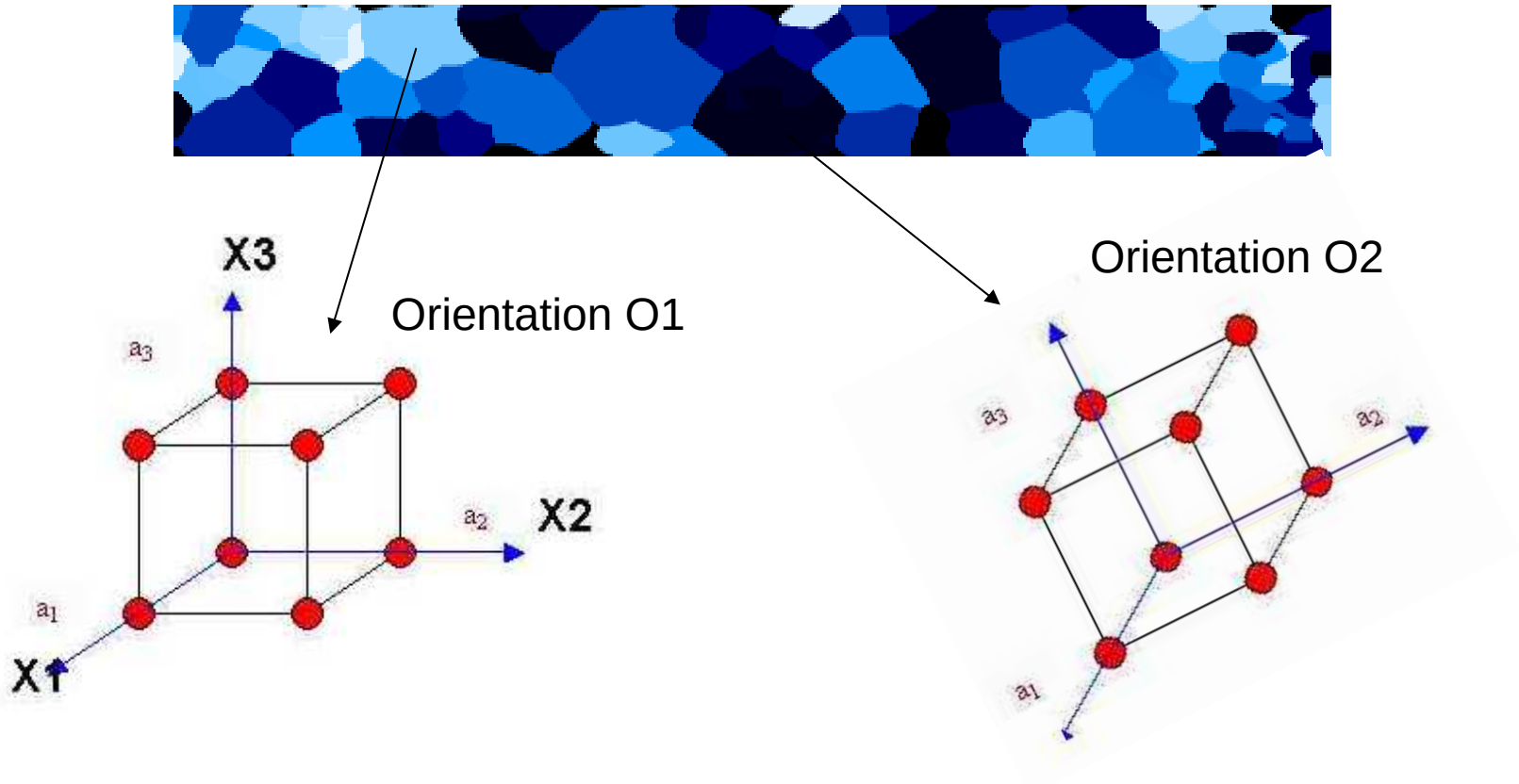
The angle between any two direction $[h_1k_1l_1]$ and $[h_2k_2l_2]$ can be calculated from:

$$\cos\phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{\left(h_1^2 + k_1^2 + l_1^2\right)\left(h_2^2 + k_2^2 + l_2^2\right)}}$$

(111) Projection of cubic crystals



- Solid materials (including thin films), when processed and synthesized develop crystallographic domains with a range of orientation and size distributions and morphology.

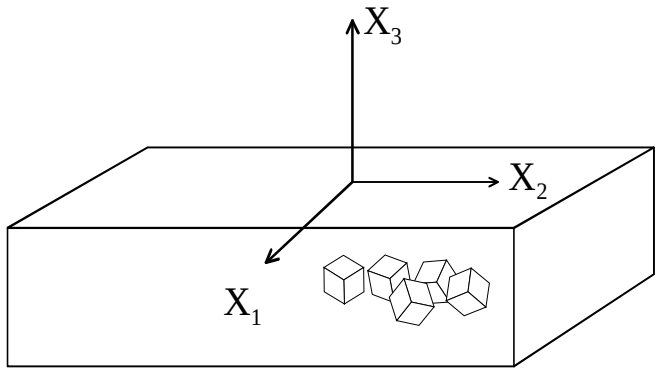


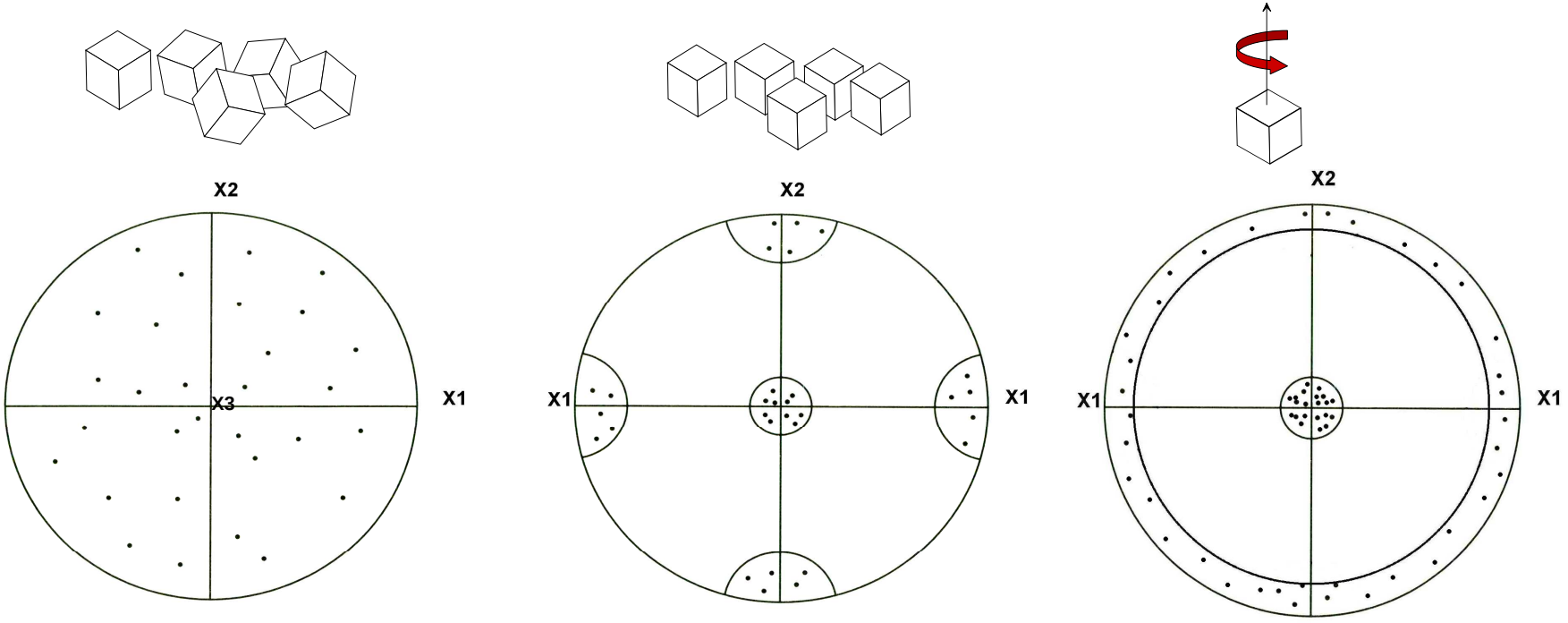
•***Crystallographic texture*** is defined as the preferred alignment of the crystallographic orientations in a polycrystalline medium.

•Textures or preferential orientations can developed during grain growth, heat treatment, plastic deformation,…

- A stereographic projection, with a specified orientation relative to the specimen shows the variation of pole density with pole orientation for a selected set of crystal planes. You just have to keep in mind that the reflected intensity is directly proportional to the diffracting volume.

- Example: [100] pole figure for cubic material





(a) No texture (« isotropic texture »)

(b) textured≡single crystal

(c) Fiber texture

•How to perform a pole figure ?

●Choose appropriate receiving slit. The larger are the slit the smaller are the intensity corrections. But this reduces angular resolution, both for 2theta and for texture. For each application a compromise must be chosen (which is different for relatively smooth textures (e.g. deformed metals) or for very sharp textures (e.g. epitaxial films).

● The ideal specimen should have a flat surface and a roughly circular shape (with a diameter larger than 2 cm)

Perform theta-2theta scan over a Bragg peak to determine the true value of 2theta

Check for different value of chi (e.g. 40°) with a th eta-2theta scan if the value of theta is the same (otherwise the sample height is probably incorrect or you have strong residual stress)

Once the appropriate 2theta value chosen, perform the (phi, chi) scans. A pole figure is scanned by measuring the diffracted intensity at different (phi, chi) settings.

- Measurement of Pole figure:

  -at a fixed 2θ angle (crystal orientation), a complete Phi scan (spins the specimen about its normal) can be preformed at a each specific tilt angle (Psi).

  -Taking several Phi scans between 0 and 85° can be p lotted in 2D, the so-called *Pole Figure.* The latter represents the orientation distribution of one plane of the crystal lattice. For example, a psi step of 5° and a phi step of 5° can be used.

- How to perform a pole figure ?

- Defocusing

  Due to the decreasing inclination of the specimen surface to the x-ray beam, the beam covers a larger area at high angles chi. As inclination (Ψ) of the sample increases, the peaks in the diffraction pattern are broadened. Thus the measured intensities can be corrected by measuring a defocusing scan on a texture free sample (preferably of the same composition). If such a sample does not exist, defocusing corrections can be estimated from the stored data.

- Background Corrections

  -The intensities derived from a pole figure must be background corrected.

  -Background measurements can be performed on the same sample that the pole figure was made on.

  -A powdered sample of the same material can also be used for this purpose.

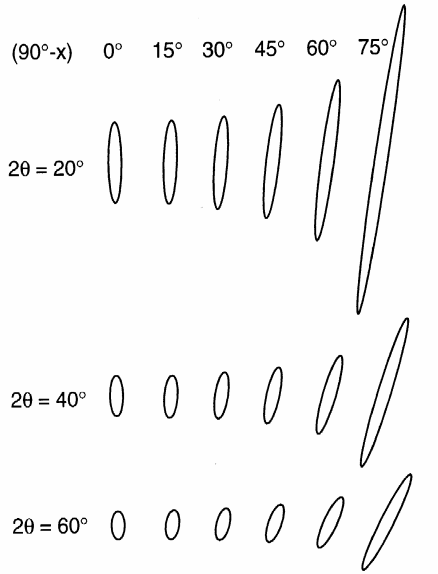
  N.B.:-It is important to verify periodically that the measured intensities have not changed significantly on a standard sample. Changes can indicate misalignement of the goniometer, malfunctionning of electronics or deterioration of detector or x-ray tube.

- 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

- Random texture or isotropic texture [=uniform dispersion of orientations] means *same* intensity in all directions.
- 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.

- 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. Moreover, the absorption correction has to be perform in the case of thin films.**



Change in shape and orientation of the irradiated spot on the sample surface for different sample inclinations as a function of tilt angle and Bragg angle (Bunge).

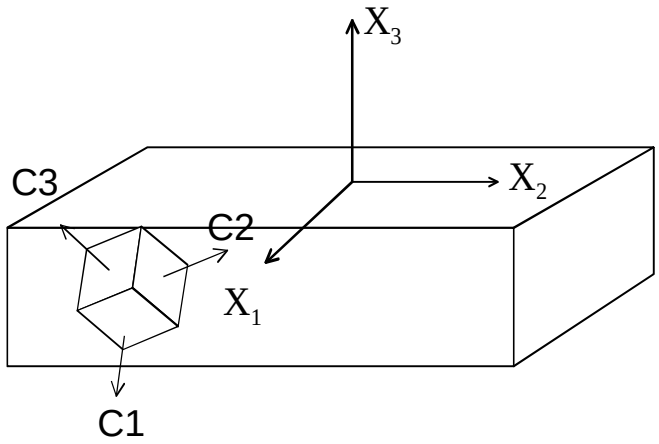
Coordinate systems and Coordinate transformation

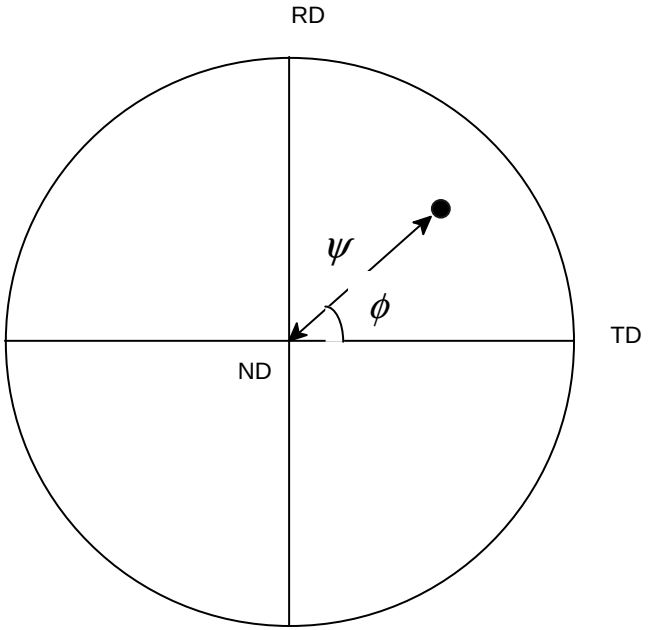
The aim of the pole figure is to give some information on the specimen's microstructure, i.e. the arrangement of grain orientation of the polycrystalline thin film. For this, we use some special coordinate systems (only right-handed cartesian coordinate systems are used); the more used are:

- **Crystallite coordinate system C** (unity vector Ci): the axes are parallel to the symmetry axes of the considered crystal, i.e. the corresponding crystal lattice.

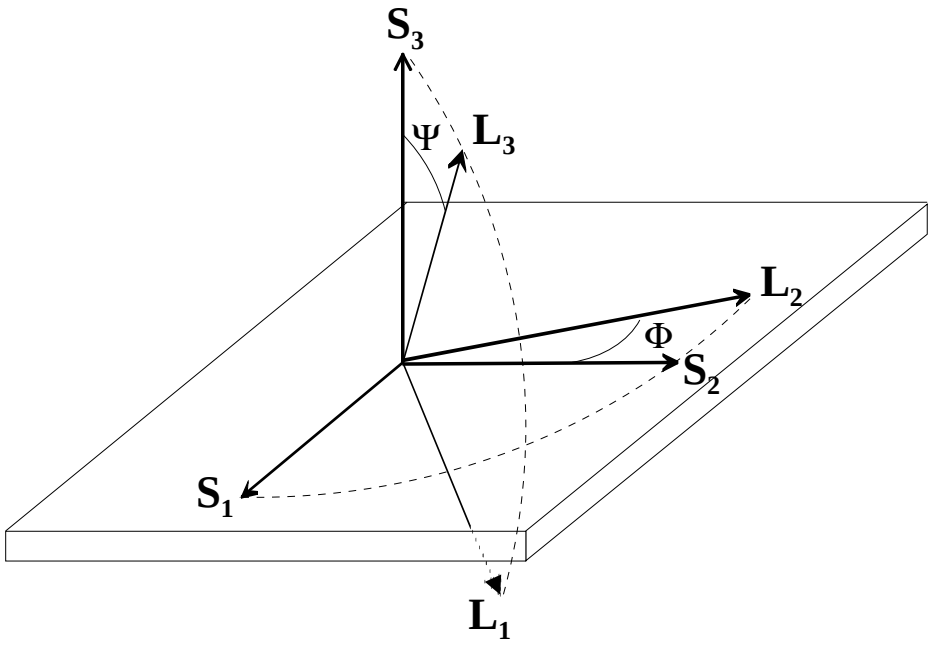
- **Specimen coordinate system S** (unity vector Si): the 3-axis is the specimen's normal (ND), the 1- and 2- axis correlate with the symmetry directions in the surface (as for example the rolling direction (RD) and the transverse direction (TD) in the case of rolled specimens.

- **Laboratory coordinate system L** (unity vector Li): the laboratory system is connected with the direction of measurement. If the measurement is performed in the direction m, the L3 axis is parallel to m. The 2-axis lies parallel to the specimen's surface. Obviously, the direction of the vector L1 is fixed too as the product L2ΛL3.





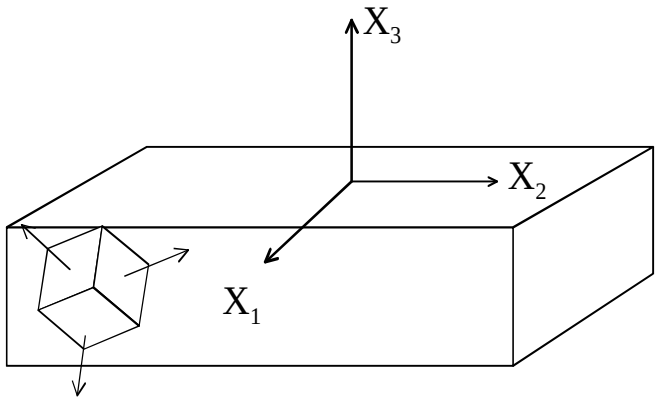
Specimen coordinate system on a stereographic projection (often used for rolled specimens)



Orientation of the laboratory system relative to the specimen system

Coordinate transformation

- Using the specimen system and the crystallite system :



- we can describe the orientation of a crystallite within a polycrystalline medium by specification of the rotation *r*, which transforms the laboratory frame X_L into the crystal system X_C. This may be written symbolically as

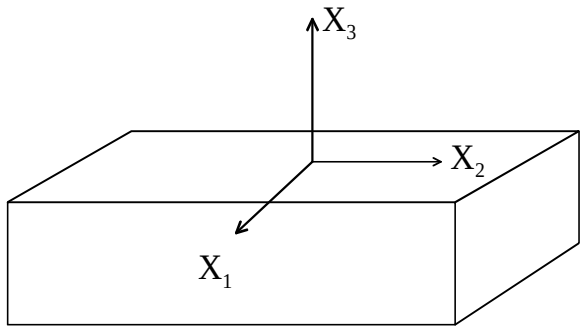
$$X_C = r.X_L$$

- A pole figure is a graphical representation of the ***angular distribution function*** of a chosen crystal direction ***g*** with respect to the Sample Coordinate System *S*.
- It means that the equatorial plane used in the spherical projection is a plane of the Sample Coordinate System
  - In the case of a sheet, it is the sheet plane,
  - for a wire - the plane orthogonal to the wire axis.

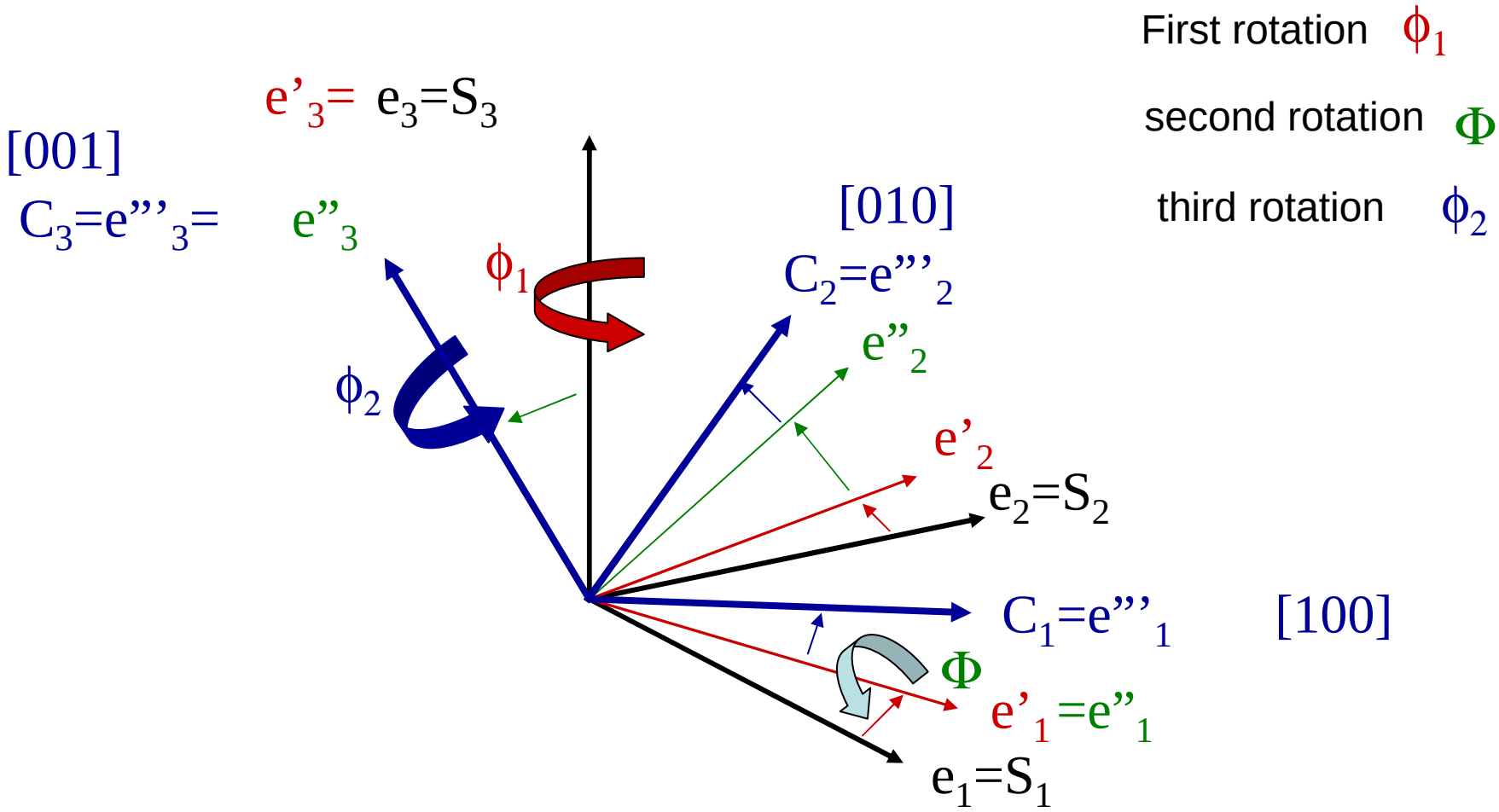
●The orientation of a crystallite in a polycrystalline material can be described in respect to the specimen system and is given by the transformation matrix P that connects the crystal C and the specimen system S. The matrix elements Pij are the cosines between the vectors Sj and Ci.

● It is often useful to express the orientation between the specimen system and the crystal system by the 3 Euler angles (ϕ1,ϕ ,ϕ2) or by the Miller indices {hkl}<uvw>. {hkl} represents the lattice plane that lies parallel to the specimen's surface and <uvw> the lattice direction parallel to the specimen's 1 axis. (In the litterature, different angle notations are used, for a complete review see the book of Bunge).

● The specimen system can be aligned to the crystal system by applying the following consecutive rotations of Euler angles (see animated next slide).



To the definition of the Euler angles



First rotation f1 around S3 transforms S1 and S2 axis in the new axes X1' and X2'. The second rotation f around X1' axis has to align the S3 axis to the C3 axis and transforms X2' to X2''. The third rotation f2 around C3 aligns X1' to C1 as well as X2'' to C2.

●The orientation distribution function (ODF) describes the relative frequency of the different crystal orientations in the polycrystal, that is the fraction of crystallites of the samples that possess the orientation g+dg .

It will be noted as $f(g)$, g stands for the orientation expressed by the Euler angles: f ($\phi1$,ϕ ,$\phi2$). It is normalized:

$$\frac{1}{8\pi^2}\int f(g)dg = 1 \quad \text{or} \quad \frac{1}{8\pi^2}\iiint f(\varphi_1,\Phi,\varphi_2)\sin\Phi d\varphi_1 d\Phi d\varphi_2 = 1$$

● Random distribution means that $f(g)=cst=1$. The so-called texture index F2 is equal to 1 for the isotropic texture case. F2>>1 denotes a strong texture:

$$F_2 = \frac{1}{8\pi^2}\int [f(g)]^2 dg$$

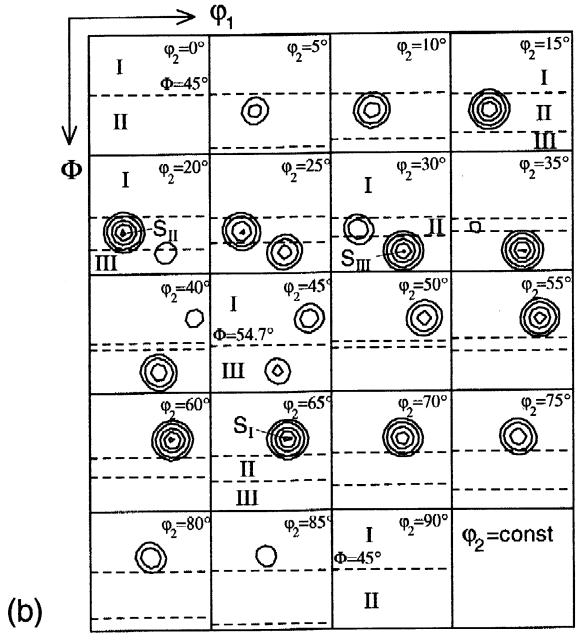
● Usually the ODF is graphically plotted as contour lines in the Euler space ($\phi1$,ϕ ,$\phi2$) for $\phi1$=constant or $\phi2$=constant.

● In textured materials some orientations are highly occupied with crystals whereas other orientations are less represented. Besides, the ODF, the orientations of the mainly present crystallite groups can serve to describe the texture state of a material, using the {hkl}<uvw> notation.

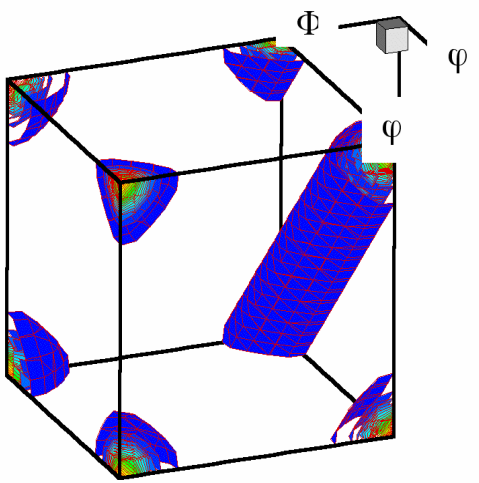
● Crystallite group g means the ensemble of all those crystallites have the same orientation g. Although spatially separated, for calculations of the average elastic behavior the can be treated as one crystal. We will see that for stress analysis method in the next chapter.

Representation of ODF:

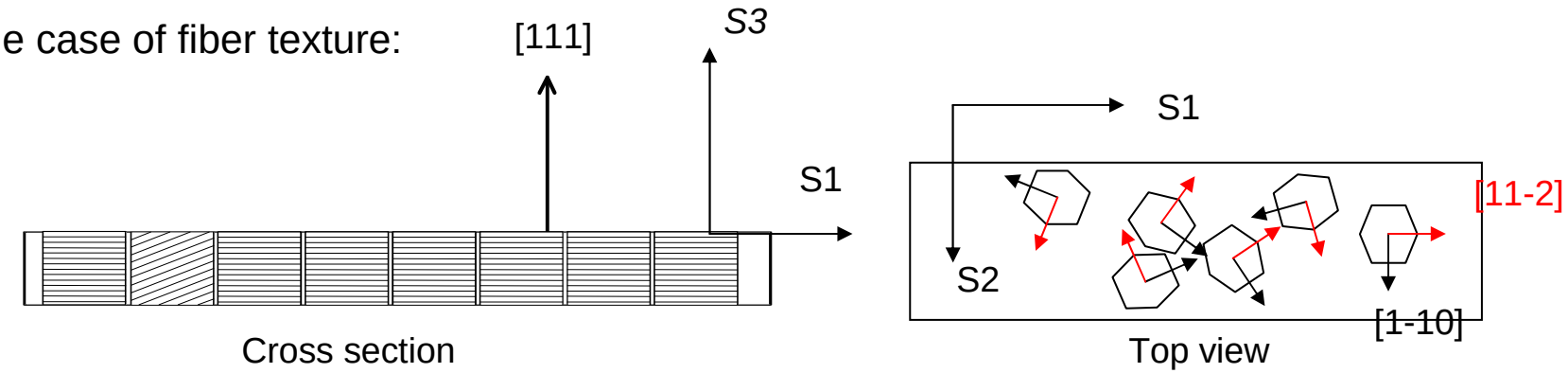
The ODF is graphically plotted as contour lines in the Euler space (as for example here for $\phi 2$=constant). The orientations belonging to different combinations of angles may be physically identical (due to the symmetry of the crystal lattice. It is therefore only necessary to consider the ODF in a part of the total range of angles of the Euler space.



Complete view of ODF in the Euler space:



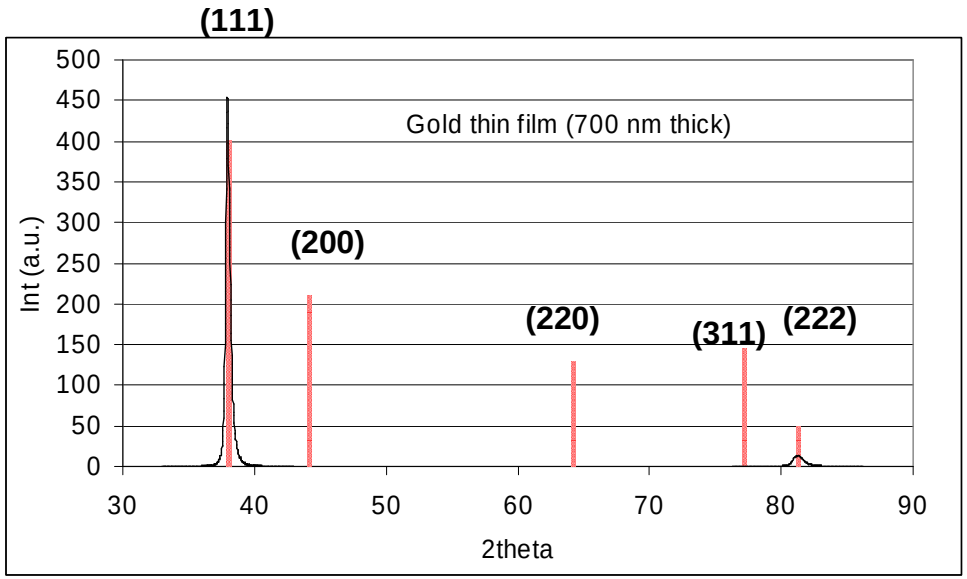
The case of fiber texture:

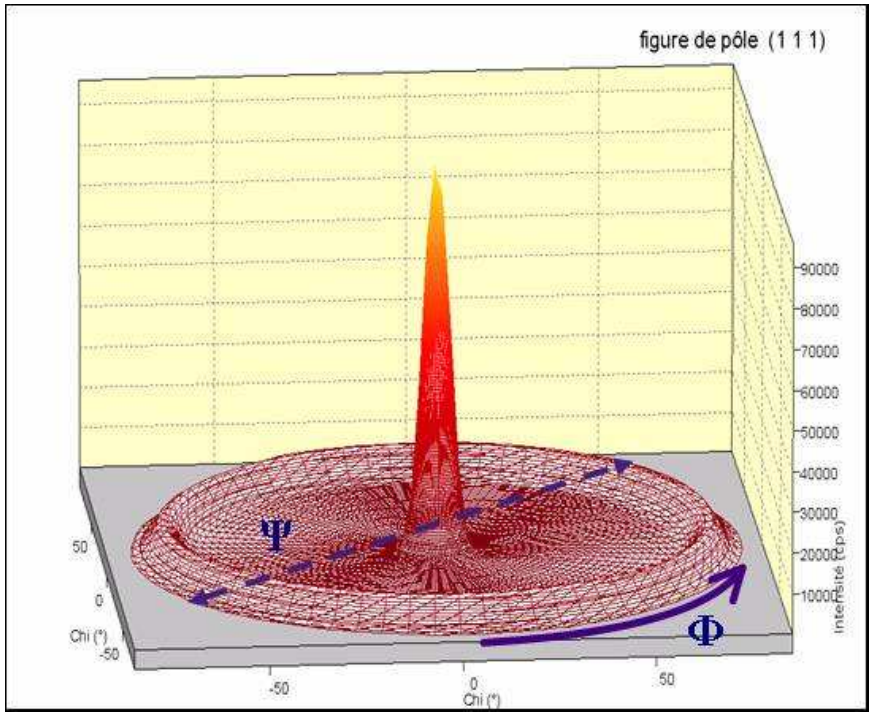


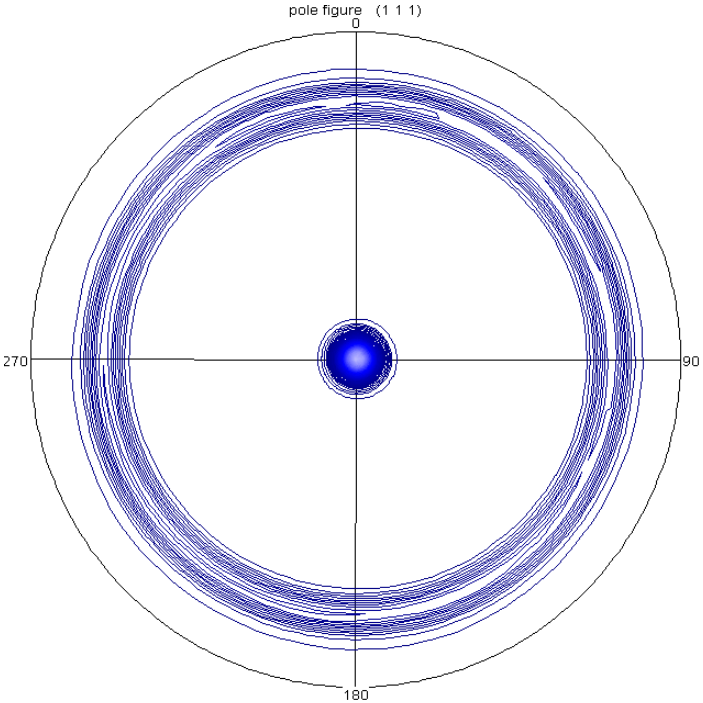
Consider a thin film (700 nm thick) with a columnar microstructure in which the (111) planes are parallel to the surface, but randomly distributed in the plane of the surface → this is the (111) fiber texture

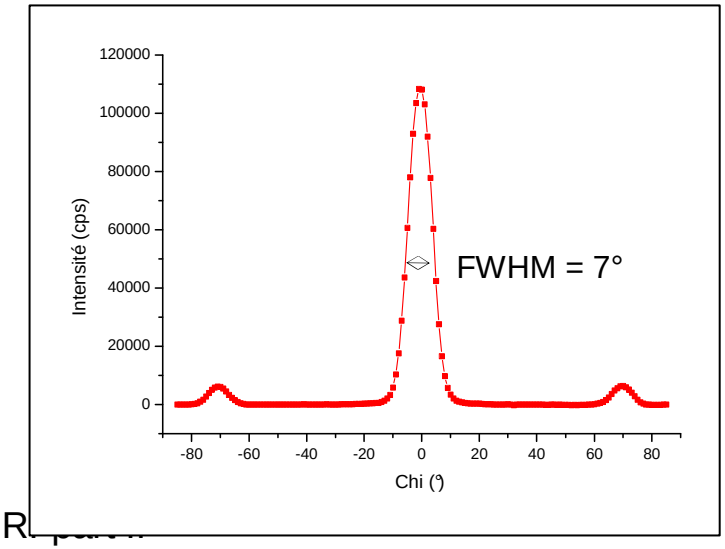
If we perform on common theta/2theta measurement on this specimen, we will obtain only diffracted intensities for (hhh) planes.
Indeed, in a theta/2theta scan, only the planes parallel to the surface can scatter.

N.B.: As you can see, the relative intensities of (111) and (222) peaks do not correspond to the one expected, why ?

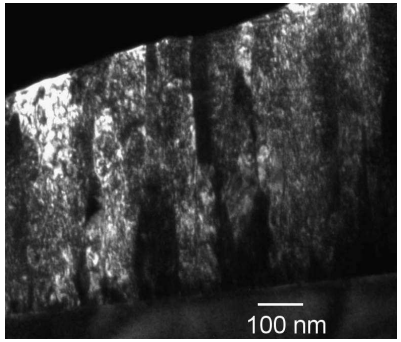


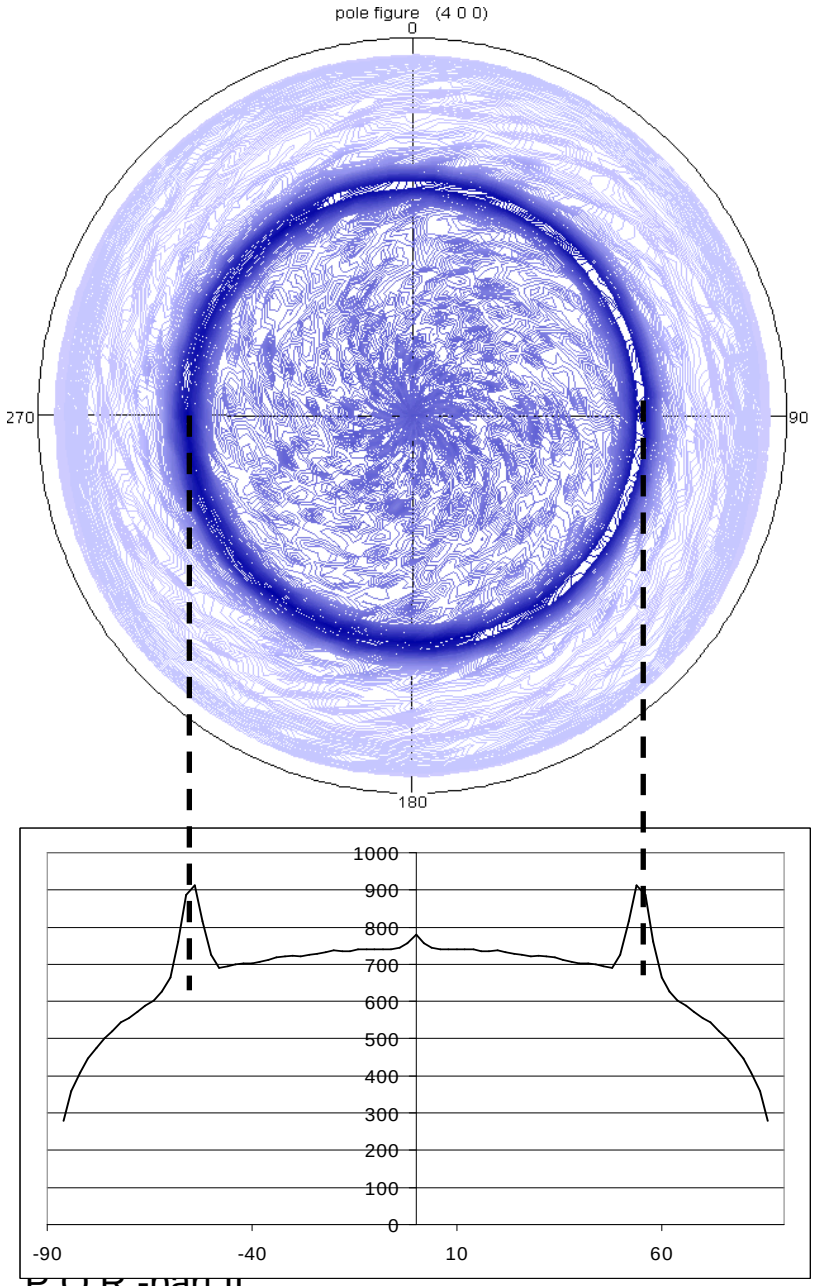


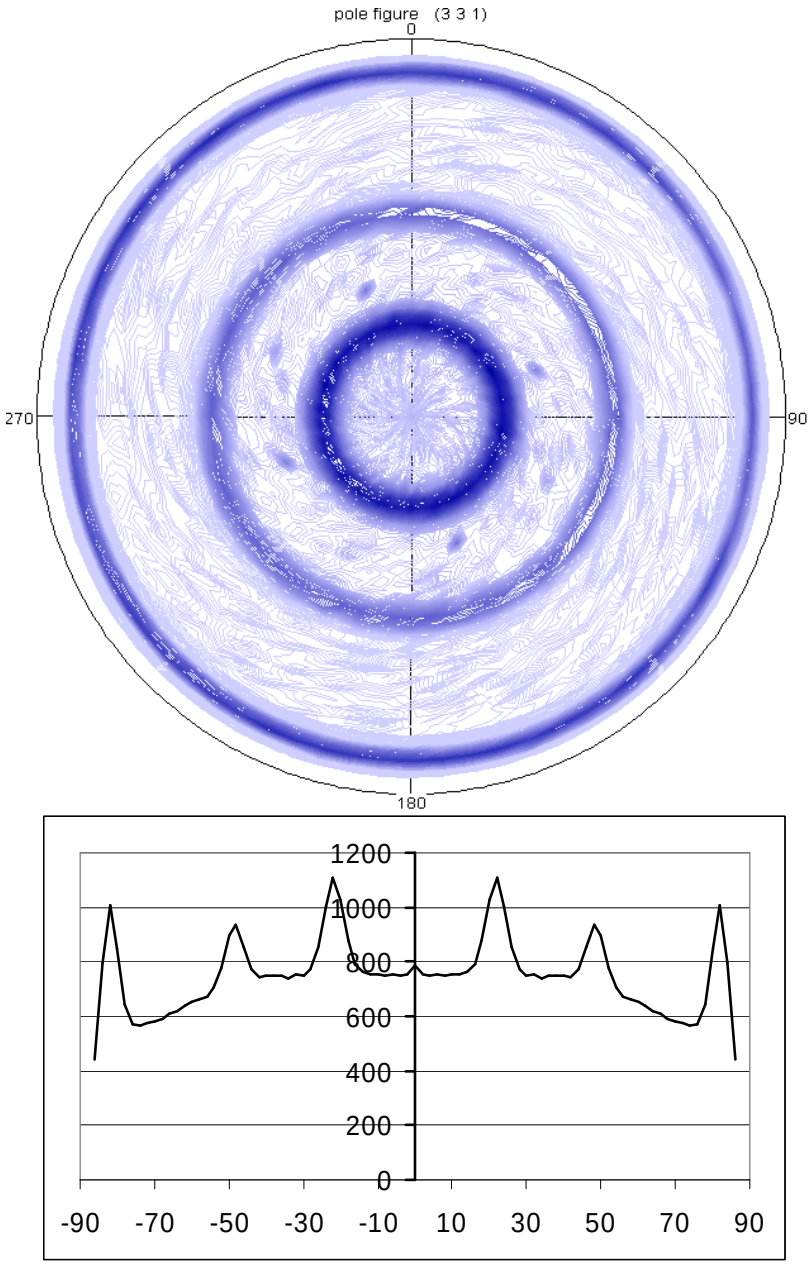




Different view of the {111} pole figure on a gold thin film, TEM micrograph







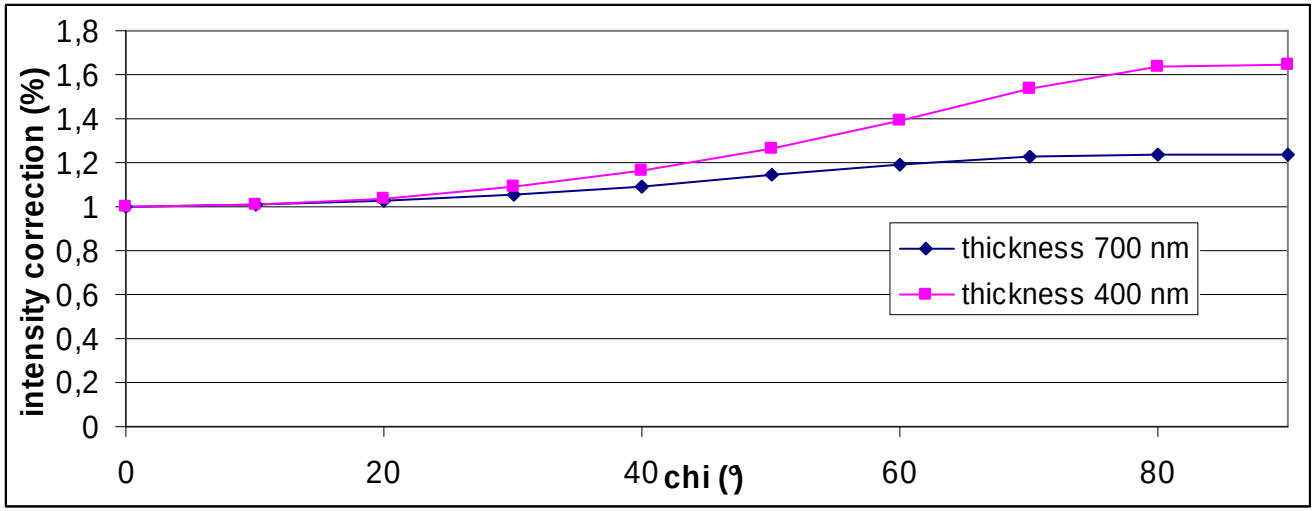
P.O.R.-part II

chi

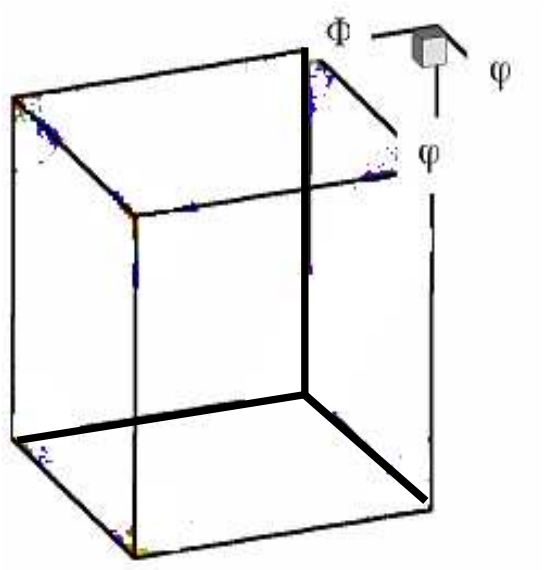
As already written, for thin films the effect of absorption and volume on the effective intensity has to be taken into account. This effect as a function of the inclinaison angle and sample thickness is expressed by the factor:

$$F(\chi,\theta,t) = 1 - \exp\left(-\frac{2\mu t}{\sin\theta.\cos\chi}\right)$$

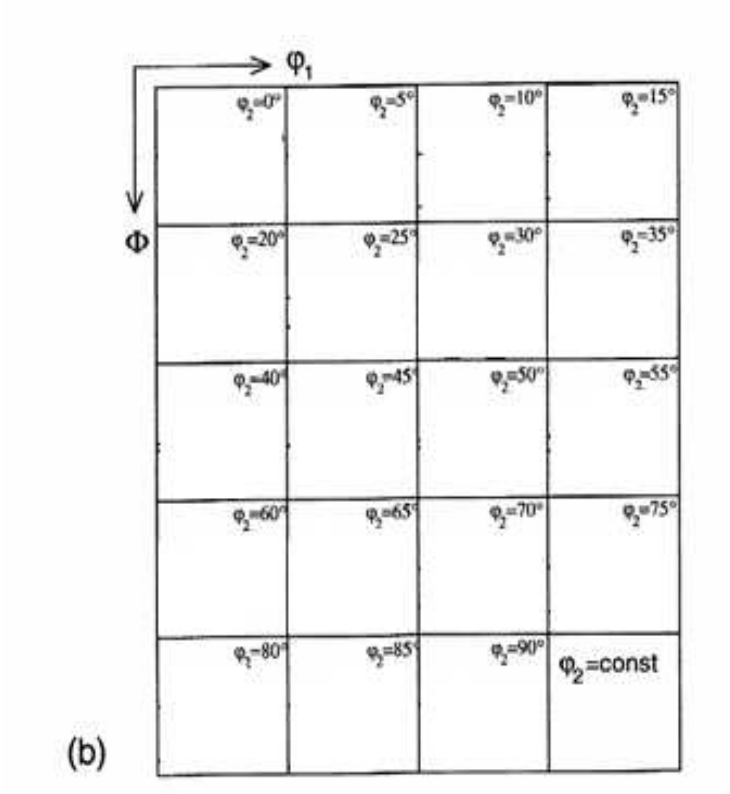
Au	mass absorption coefficient in cm²/g	208
	density in g/cm3	19.3



The case of fiber texture:



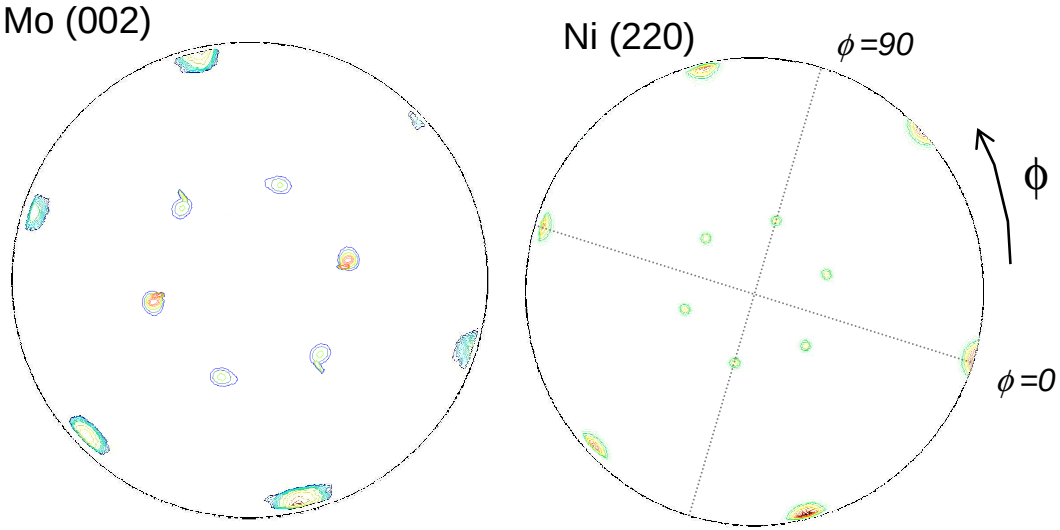
Complete view of ODF in the Euler space:



Representation of ODF:

Epitaxy :

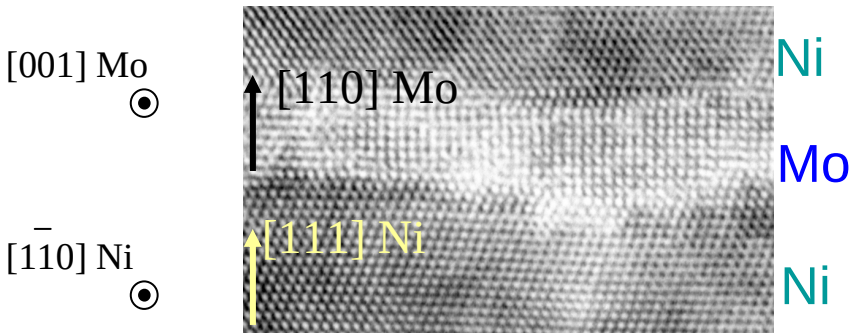
Poles figures



Epitaxial Relations:

$[\bar{1}\bar{1}2]$ (111) Ni // $[1\bar{1}0]$ (110) Mo

$[\bar{1}10]$ (111) Ni // [001] (110) Mo



TEMHR

III
Anisotropic Elasticity

Objective

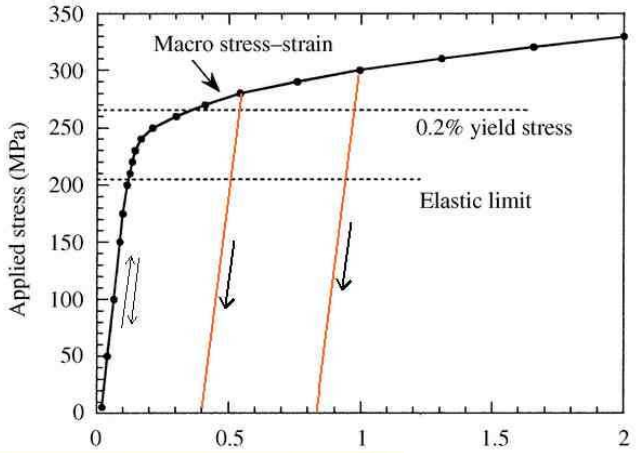
●The objective of this lecture is to provide a mathematical framework for the description of properties, especially when they vary with direction.

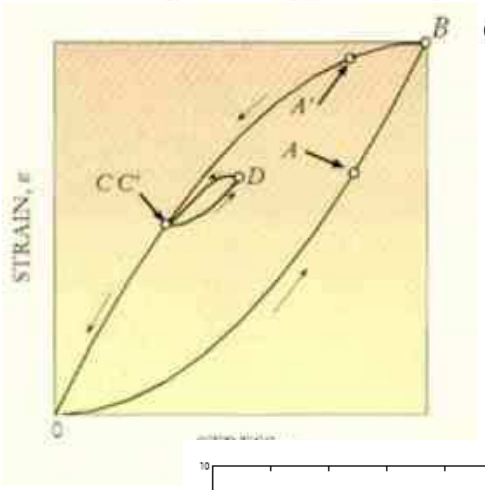
● A basic property that occurs in almost applications is *elasticity*. Although elastic response is *linear* for a lot of practical purposes, it is often *anisotropic* (composites, textured polycrystals etc.).

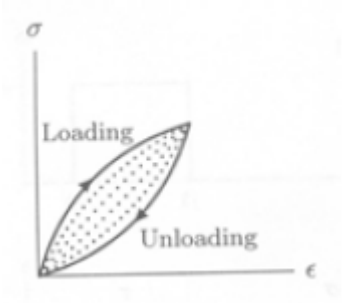
Example of non-linear properties:

● An example of non-linear mechanical properties is plasticity, i.e. the irreversible deformation of solids.

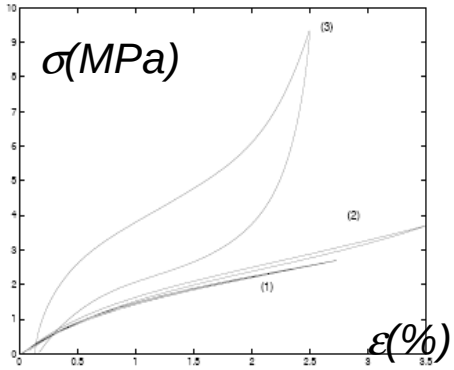
● Rocks display unique elastic behavior. They are extremely nonlinear, being hysteretic, possessing discrete memory, and having slow dynamics [a long term memory of strain]. Although some of these types of nonlinearities may exist in, for example, powdered metals, it is rocks where these characteristics were first observed. The class of materials includes rock, damaged solids, and, compressed powdered metals. Further, nonlinear behavior plays a central role in developing new methods with which to characterize material properties, for instance, interrogating the elastic microstructure of rock, determining if a material is damaged, or monitoring progressive damage. Nonlinear attributes of rock have important consequences on processes in the earth such as earthquake strong ground motion, reservoir subsidence, seismic wave propagation and attenuation, stress fatigue damage, hydraulic fracturing, etc.







● Stress-strain curves for (1) unfilled, (2) lightly filled and (3) highly filled rubber in tensile deformations.



Linear properties

● Certain properties, such as elasticity, are assumed to be perfectly linear. This means that we can write in the case of elasticity that the strain is proportional to the stress

$$\boldsymbol{\sigma} = C\,\boldsymbol{\varepsilon}$$

where C is the stiffness tensor

For uniaxial tension test or one dimensional case, *C ≡ Young's modulus (or elasticity modulus) E.*

● For isotropic materials, the Hooke's law can be used:

$$\varepsilon_{ij} = \left(\frac{1+\nu}{E}\right)\cdot\sigma_{ij} - \frac{\nu}{E}\cdot\delta_{ij}\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)$$

where ν is the Poisson's ratio and δij : kronecker's symbol

$$\sigma_{ij} = 2\mu\cdot\varepsilon_{ij} + \lambda\cdot\delta_{ij}\left(\varepsilon_{11}+\varepsilon_{22}+\varepsilon_{33}\right)$$

where λ,μ are the Lamé moduli

Elasticity

BUT:

- *Elasticity*: this property requires tensors to describe it fully.

- Even in cubic metals, a crystal is quite anisotropic. We will speak of local anisotropy. For example, the [111] direction in many cubic metals is stiffer than the [100] direction.

- In cubic materials, 3 numbers or coefficients or moduli are required to fully describe elastic properties.

- isotropic materials only require 2, as for example ν and E or λ and μ (see Hooke's law on the preceding slide). They are also bulk modulus B, shear modulus G=μ.

Elasticity anisotropy

● First we restate the linear elastic relations for the properties *Compliance*, written *S or* (s_{ij}), and *Stiffness*, written *C or* (c_{ij}) , which connect stress, $\boldsymbol{\sigma}$, and strain, $\boldsymbol{\varepsilon}$. We write it first in vector-tensor notation with *":"* signifying *inner product* (i.e. add up terms that have a common suffix or index in them):

$$\boldsymbol{\sigma} = C:\boldsymbol{\varepsilon}$$
$$\boldsymbol{\varepsilon} = S:\boldsymbol{\sigma}$$

In *component* form (with suffices),

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

● The definitions of the stress and strain tensors mean that they are both symmetric (second rank) tensors. Therefore we can see that

$$\varepsilon_{23} = S_{2311}\sigma_{11}$$
$$\varepsilon_{32} = S_{3211}\sigma_{11} = \varepsilon_{23}$$

which means that,

$$S_{2311} = S_{3211}$$

and in general,

$$S_{ijkl} = S_{jikl}$$

We will see later on that this reduces considerably the number of different coefficients needed.

Stiffness in sample coordinate

● Consider how to express the elastic properties of a single crystal in the sample coordinates. In this case we need to rotate the (4^{th} rank) tensor from crystal coordinates **to** sample coordinates using the orientation (matrix):

$$c_{ijkl}' = a_{im}\, a_{jn}\, a_{ko}\, a_{lp} c_{mnop}$$

● Note how the transformation matrix appears four times because we are transforming a 4^{th} rank tensor.

Young's modulus from compliance

● Young's modulus as a function of direction can be obtained from the compliance tensor as $E=1/s'_{1111}$. Using compliances and a stress boundary condition (only $\sigma_{11}\neq 0$) is most straightforward. To obtain s'_{1111}, we simply apply the same transformation rule,

$$s'_{ijkl} = a_{im}\, a_{jn}\, a_{ko}\, a_{lp} s_{mnop}$$

Matrix notation

● It is useful to re-express the three quantities involved in a simpler format. The stress and strain tensors are vectorized, i.e. converted into a 1x6 notation and the elastic tensors are reduced to 6x6 matrices.
For stress tensor:

$$\begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix} <-> \begin{pmatrix} \sigma_1 & \sigma_6 & \sigma_5 \\ \sigma_6 & \sigma_2 & \sigma_4 \\ \sigma_5 & \sigma_4 & \sigma_3 \end{pmatrix} <-> (\sigma_1, \sigma_2, \sigma_3, \sigma_4, \sigma_5, \sigma_6)$$

For strain tensor:

$$\begin{pmatrix} \varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\ \varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33} \end{pmatrix} <-> \begin{pmatrix} \varepsilon_1 & \frac{1}{2}\varepsilon_6 & \frac{1}{2}\varepsilon_5 \\ \frac{1}{2}\varepsilon_6 & \varepsilon_2 & \frac{1}{2}\varepsilon_4 \\ \frac{1}{2}\varepsilon_5 & \frac{1}{2}\varepsilon_4 & \varepsilon_3 \end{pmatrix} <-> (\varepsilon_1, \varepsilon_2, \varepsilon_3, \varepsilon_4, \varepsilon_5, \varepsilon_6)$$

The particular definition of shear strain used in the reduced notation happens to correspond to that used in mechanical engineering such that ε_4 is the change in angle between direction 2 and direction 3 due to deformation.

Work conjugacy

• The more important consideration is that the reason for the factors of two is so that work conjugacy is maintained.

$$dW = \sigma : d\varepsilon = \sigma_{ij} : d\varepsilon_{ij} = \sigma_k \bullet d\varepsilon_k$$

Also we can combine the expressions $\boldsymbol{\sigma} = C:\boldsymbol{\varepsilon}$ and $\boldsymbol{\varepsilon} = S:\boldsymbol{\sigma}$ to give:

$\boldsymbol{\sigma} = C:S:\boldsymbol{\sigma}$, which shows: $I = C:S$, or, $C = S^{-1}$

Conversions of stiffness tensor:

• Lastly we need a way to convert the tensor coefficients of stiffness and compliance to the matrix coefficients. For stiffness, it is very simple because one substitutes values according to the following table, such that ${}^{matrix}C_{11} = {}^{tensor}C_{1111}$ for example.

Tensor	11	22	33	23	32	13	31	12	21
Matrix	1	2	3	4	4	5	5	6	6

Stiffness matrix

$$C = (cij) = \begin{bmatrix} c_{11} & c_{12} & c_{13} & c_{14} & c_{15} & c_{16} \\ c_{21} & c_{22} & c_{23} & c_{24} & c_{25} & c_{26} \\ c_{31} & c_{32} & c_{33} & c_{34} & c_{35} & c_{36} \\ c_{41} & c_{42} & c_{43} & c_{44} & c_{45} & c_{46} \\ c_{51} & c_{52} & c_{53} & c_{54} & c_{55} & c_{56} \\ c_{61} & c_{62} & c_{63} & c_{64} & c_{65} & c_{66} \end{bmatrix}$$

Conversions of compliance's tensor

- For compliance some factors of two are required and so the rule becomes:

$$S_{mn} = p.S_{ijkl}$$

$$p = 1 \quad \text{for } (m \leq 3 \quad \text{and} \quad n \leq 3)$$

$$p = 2 \quad \text{for } (m \leq 3 \quad \text{and} \quad n > 3) \text{ or vice versa}$$

$$p = 4 \quad \text{for } (m > 3 \quad \text{and} \quad n > 3)$$

Relationships between elastic coefficients

- Some additional useful relations between coefficients for cubic materials are as follows. Symmetrical relationships exist for compliances in terms of stiffnesses.

$$C_{11} = (S_{11}+S_{12})/\{(S_{11}-S_{12})(S_{11}+2S_{12})\}$$

$$C_{12} = -S_{12}/\{(S_{11}-S_{12})(S_{11}+2S_{12})\}$$

$$C_{44} = 1/S_{44}.$$

- The relationships for S in terms of C are symmetrical to those for stiffnesses in terms of compliances (a simple exercise in algebra!).

$$S_{11} = (C_{11}+C_{12})/\{(C_{11}-C_{12})(C_{11}+2C_{12})\}$$

$$S_{12} = -C_{12}/\{(C_{11}-C_{12})(C_{11}+2C_{12})\}$$

$$S_{44} = 1/C_{44}.$$

Effect of symmetry on stiffness matrix

- Why do we need to look at the effect of symmetry? For a cubic material, only 3 *independent* coefficients are needed as opposed to the 81 coefficients in a 4th rank tensor. The reason for this is the symmetry of the material.
- What does symmetry mean? Fundamentally, if you pick up a crystal, rotate [mirror] it and put it back down, then a symmetry operation [rotation, mirror] is such that *you cannot tell that anything happened*.
- From a mathematical point of view, this means that the property (its coefficients) *does not change*. For example, if the symmetry operator changes the sign of a coefficient, then it must be equal to zero.

Example of the effect of symmetry on the stiffness matrix :

Let's apply a 90° rotation about the crystal-z axis (axis 3),

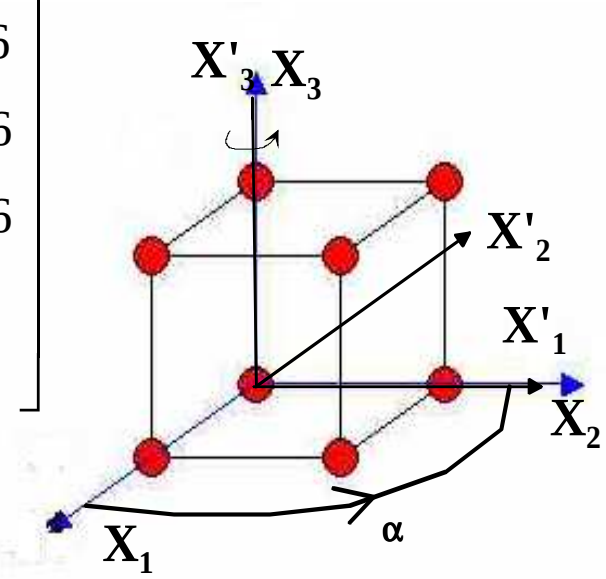
$C'_{ijkl} = a_{im}a_{jn}a_{ko}a_{lp}C_{mnop}$

And, because of the symmetry:

$\boldsymbol{C'} = \boldsymbol{C}$

$$(a_{ij}) = \begin{pmatrix} 0 & -1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

$$C' = \begin{bmatrix} C_{22} & C_{21} & C_{23} & C_{25} & -C_{24} & -C_{26} \\ C_{21} & C_{11} & C_{13} & C_{15} & -C_{14} & -C_{16} \\ C_{23} & C_{13} & C_{33} & C_{35} & -C_{34} & -C_{36} \\ C_{25} & C_{15} & C_{35} & C_{55} & -C_{54} & -C_{56} \\ -C_{24} & -C_{14} & -C_{34} & -C_{54} & C_{44} & C_{46} \\ -C_{26} & -C_{16} & -C_{36} & -C_{56} & C_{46} & C_{66} \end{bmatrix}$$



C is the tensor in the reference frame associated to the cubic lattice
C' is the tensor in the reference frame obtained with a 90° rotation

Using $C' = C$ (because of the symmetry) , we can equate coefficients and find that:

$C_{11}=C_{22}$, $C_{13}=C_{23,}$ $C_{44}=C_{35}$, $C_{16}=-C_{26,}$

and moreover

$C_{14}=C_{25} = -C_{14} = 0$

Thus we have also:

$C_{14}=C_{15} = C_{24} = C_{25} = C_{34} = C_{35} = C_{36} = C_{45} = C_{46} = C_{56} = 0.$

Finally:

$$C' = \begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & C_{16} \\ C_{12} & C_{11} & C_{13} & 0 & 0 & -C_{16} \\ C_{13} & C_{13} & C_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & C_{46} \\ C_{16} & -C_{16} & 0 & 0 & C_{46} & C_{66} \end{bmatrix}$$

Thus by repeated applications of the symmetry operators, one can demonstrate (for cubic crystal symmetry) that one can reduce the 81 coefficients down to only 3 independent quantities.

$$C = \begin{bmatrix} c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & c_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & c_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & c_{44} \end{bmatrix}$$

In the reference frame associated to the crystallite system *C*

Thus, the elasticity of a cubic material is completely characterized by three elastic coefficients such as the 3 stiffnesses (or 3 compliances).

→These become two in the case of isotropy (because $c_{44}=1/2(c_{11}-c_{12})$).

Cubic crystals : anisotropy factor or Zener's factor

• If one applies the symmetry elements of the cubic system, it turns out that only three independent coefficients remain: C_{11}, C_{12} and C_{44}, (similar set for compliance). From these three, a useful combination of the first two is

$$C' = (C_{11} - C_{12})/2$$

• $C' = (C_{11} - C_{12})/2$ turns out to be the stiffness associated with a shear in a <110> direction on a plane. In certain martensitic transformations, this modulus can approach zero which corresponds to a structural instability.

• Zener proposed a measure of elastic anisotropy based on the ratio A=C_{44}/C'. This turns out to be a useful criterion for identifying materials that are elastically anisotropic.

$$A = \frac{2c_{44}}{c_{11} - c_{12}} = \frac{2(s_{11} - s_{12})}{s_{44}}$$

Rotation of compliance's matrix

• Given an orientation a_{ij}, we transform the compliance tensor, using cubic point group symmetry, and find that:

$$s'_{11} = s_{11}\left(a_{11}^4 + a_{12}^4 + a_{13}^4\right) + 2s_{12}\left(a_{12}^2 a_{13}^2 + a_{11}^2 a_{12}^2 + a_{11}^2 a_{13}^2\right) + s_{44}\left(a_{12}^2 a_{13}^2 + a_{11}^2 a_{12}^2 + a_{11}^2 a_{13}^2\right)$$

• This can be further simplified with the aid of the standard relations between the direction cosines, $a_{ik}a_{jk} = 1$ for $i=j$; $a_{ik}a_{jk} = 0$ for $i \neq j$, ($a_{ik}a_{jk} = \delta_{ij}$) to read as follows.

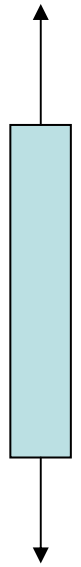
$$s'_{11} = s_{11} - 2s_0\left(a_1^2 a_2^2 + a_2^2 a_3^2 + a_3^2 a_1^2\right) \qquad \text{with} \qquad s_0 = s_{11} - s_{12} - \tfrac{1}{2}\, s_{44}$$

• These relation is also written as a function of Γ (orientation parameter)

$$s'_{11} = s_{11} - 2\left(s_{11} - s_{12} - \frac{1}{2}s_{44}\right)\Gamma \qquad \text{with} \qquad \Gamma = \frac{h^2k^2 + h^2l^2 + k^2l^2}{\left(h^2 + k^2 + l^2\right)^2}$$

For an uniaxial tensile test:

$$\begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \\ \varepsilon_4 \\ \varepsilon_5 \\ \varepsilon_6 \end{pmatrix} = \begin{bmatrix} s_{11} & s_{12} & s_{13} & s_{14} & s_{15} & s_{16} \\ s_{21} & s_{22} & s_{23} & s_{24} & s_{25} & s_{26} \\ s_{31} & s_{32} & s_{33} & s_{34} & s_{35} & s_{36} \\ s_{41} & s_{42} & s_{43} & s_{44} & s_{45} & s_{46} \\ s_{51} & s_{52} & s_{53} & s_{54} & s_{55} & s_{56} \\ s_{61} & s_{62} & s_{63} & s_{64} & s_{65} & s_{66} \end{bmatrix} \cdot \begin{pmatrix} \sigma_1 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{pmatrix}$$



$$\varepsilon_1 = s'_{11} . \sigma_1$$

By definition, the Young's modulus in any direction is given by the reciprocal of the compliance, $E = 1/S'_{11}$

Anisotropy for cubic materials

● Thus the second term on the expression of s'_{11} is zero for <100> directions and, for $s_0>0$, maximum in <111> directions (conversely a minimum for $s_0<0$).

The following table shows that most cubic metals have positive values of Zener's coefficient so that <100> is soft and <111> is hard, with some exceptions like Nb, Mo and NaCl.

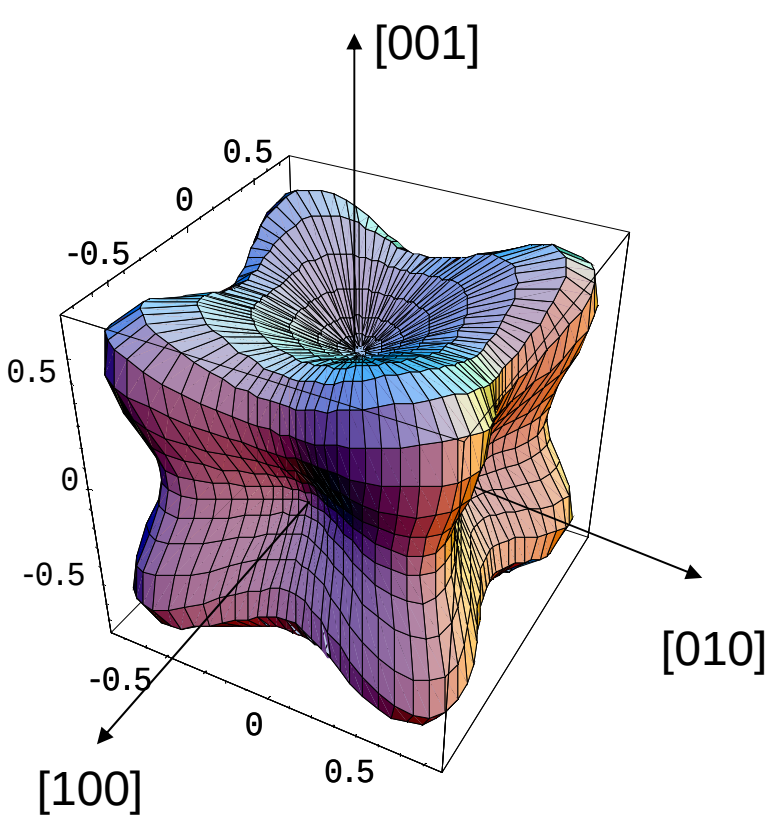
	material	E<100> (GPa)	E<111> (GPa)	E<111>/ E<100>	A
bcc	**Nb**	152	82	0,54	0,51
bcc	**Nb**	145	83	0,57	0,55
	NaCl	44	33	0,75	0,70
bcc	**Mo**	384	287	0,75	0,71
bcc	**W**	407	411	1,01	1,01
	Fe3O4	214	243	1,14	1,16
fcc	**Al**	63	76	1,20	1,22
	MgO	251	355	1,42	1,55
bcc	**Ta**	146	217	1,49	1,56
diamond	**Si**	130	188	1,44	1,56
fcc	**Ni**	129	305	2,36	2,67
bcc	**Fe**	119	283	2,38	2,70
fcc	**Au**	43	117	2,71	2,85
fcc	**Ag**	43	121	2,78	3,04
fcc	**Cu**	66	192	2,92	3,28

A<1

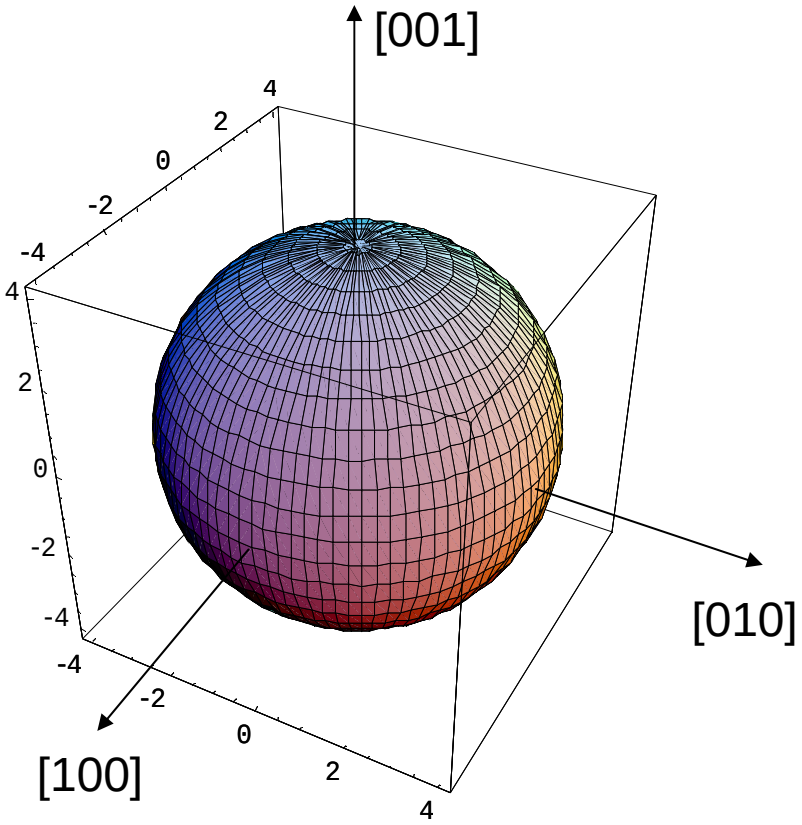
Locally perfectly isotropic

A>1

Au : A=3
Locally anisotropic material



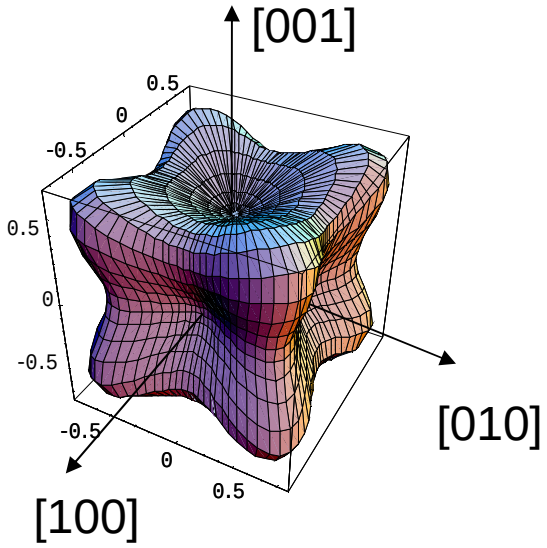
W : A=1
Locally isotropic material

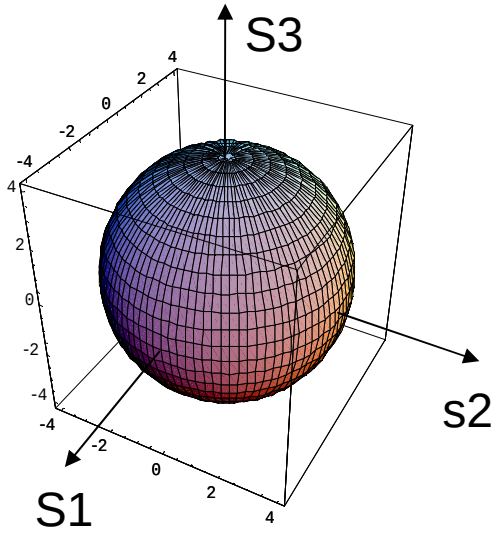


Young's modulus can be represented by means of a spherical polar diagram, that is, with a surface generated by a vector whose length is proportional to the value of Young's modulus in the direction pointed by the vector itself.

What about the macroscopic elasticity ?

-if you have a random distribution of orientations of grains or crystallites, you have to average over all orientations.





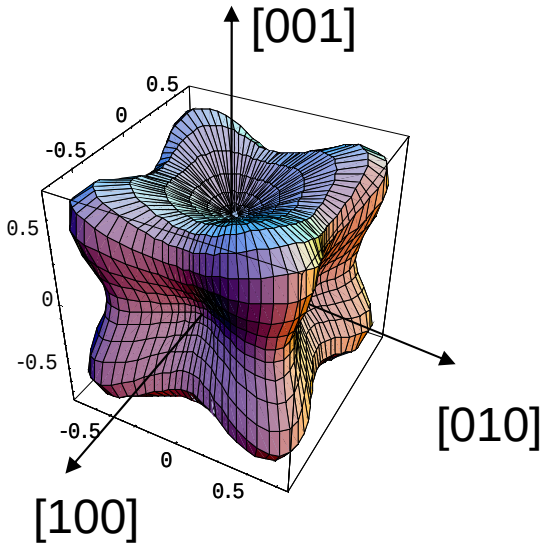
Crystallite reference frame

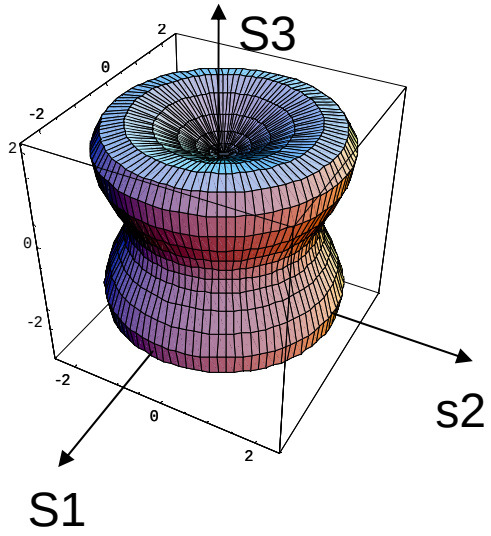
Specimen reference frame

Locally anisotropic crystallite  macroscopically isotropic material

« Quasi-isotropic » materials

-if you have a non random of orientations of grains or crystallites. Let us consider the simple case of {100} fiber texture (i.e. (100) planes // surface and random distribution of crystallites in the plane), you have to average over all orientations in the plane:





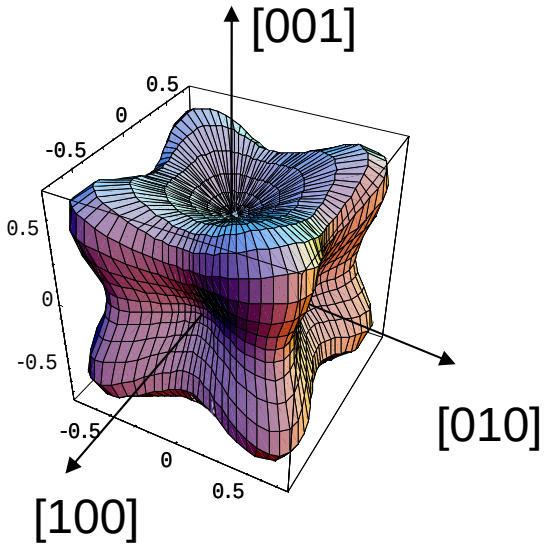
Crystallite reference frame

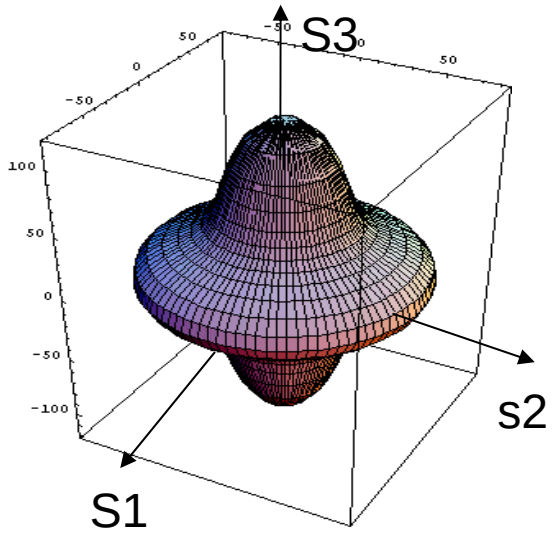
Specimen reference frame

Locally anisotropic crystallite  macroscopically anisotropic material

But in this particular case, transversal isotropy

-if you have a non random of orientations of grains or crystallites. Let us consider the simple case of {111} fiber texture (i.e. (111) planes // surface and random distribution of crystallites in the plane), you have to average over all orientations in the plane:





Crystallite reference frame

Specimen reference frame

Locally anisotropic crystallite → macroscopically anisotropic material

But in this particular case, transversal isotropy

Summary

We have covered the following topics:

-Linear properties

-Examples of properties

-Tensors, vectors, scalars.

-Elasticity, as example as of higher order property, also as example as how to apply (crystal) symmetry.

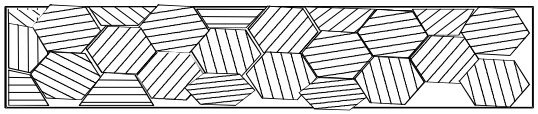
The basic relations we have described are necessary for the stress analysis using X- and neutron rays, for the interpretation of the measured data and for the calculation of averaged elastic properties and X-ray elastic constants (XEC) or diffraction elastic constants (DEC).

The basic relations we have described are necessary for the stress analysis using X- and neutron rays, for the interpretation of the measured data and for the calculation of averaged elastic properties and X-ray elastic constants (XEC) or diffraction elastic constants (DEC).

Homogeneisation

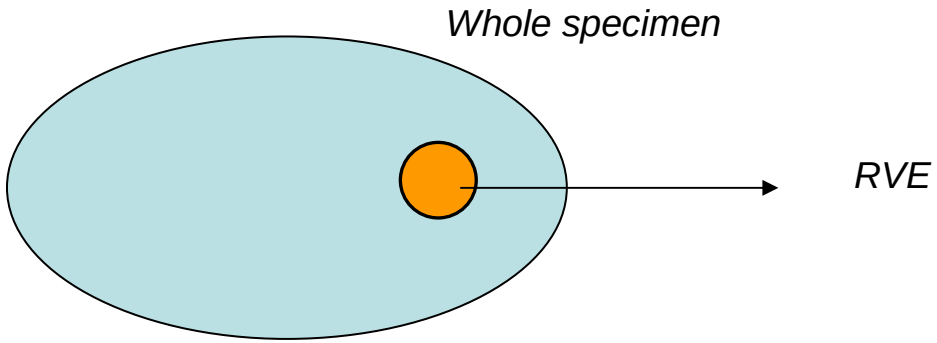
• Stimuli and responses of interest are, in general, not scalar quantities but tensors. Elasticity is considered as linear property but some of the properties of interest, such as the plastic properties of a material, are far from linear at the scale of a polycrystal. Nonetheless, they can be treated as linear at a suitably local scale and then an averaging technique can be used to obtain the response of the polycrystal. The local or microscopic response is generally well understood but the validity of the averaging techniques is still controversial in many cases.

• *There are many problems in which a non-homogeneous response to a homogeneous stimulus is of critical importance. Stress-corrosion cracking, for example, is a wildly non-linear, non-homogeneous response to an approximately uniform stimulus which depends on the mechanical and electro-chemical properties of the material*.



→ A polycrystal is composed of grains which are elastically anisotropic, and is therefore strongly heterogeneous from the mechanical point of view. It can therefore be considered as a composite. What are the effective properties of the polycrystal or the composite ?

- The question of the effective properties of a composite implicitely assumes that the problem contains 2 scales which are well separated:
  - The microscopic scale is small enough for the heterogeneities to be smeared out.
  - The effective properties at the macroscopic scale of the composite are determined from geometrical and material data available from the study of a representative volume element. The geometrical and/or material properties of random composites are only partially known through statistical informations, e.g. correlation functions of various orders.

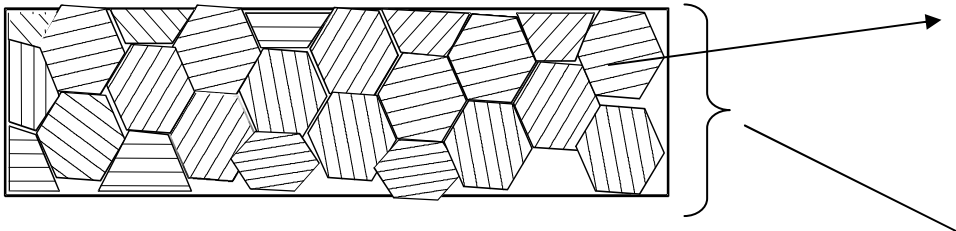


● In order to describe the properties of a material, it is useful to define a *representative volume element* (RVE) that is large enough to be statistically representative of that region (but small enough that one can subdivide a body).

● For example, consider a polycrystal: how many grains must be included in order for the element to be representative of that point in the material?

Homogeneisation

●A polycrystal is composed of grains which are elastically anisotropic, and is therefore strongly heterogeneous from the mechanical point of view:



Crystallite = single crystal → well known properties

What about polycrystal properties ?

In a polycrytalline material, the strains differ between grains and all grains interact with each other due to the incompatibility of these different strains. As a consequence, the average stresses in each grain also differ.

To determine the average stress in each grain, several assumption can be used

- from the most complicated ones (but realistic ones) : the self-consistent methods

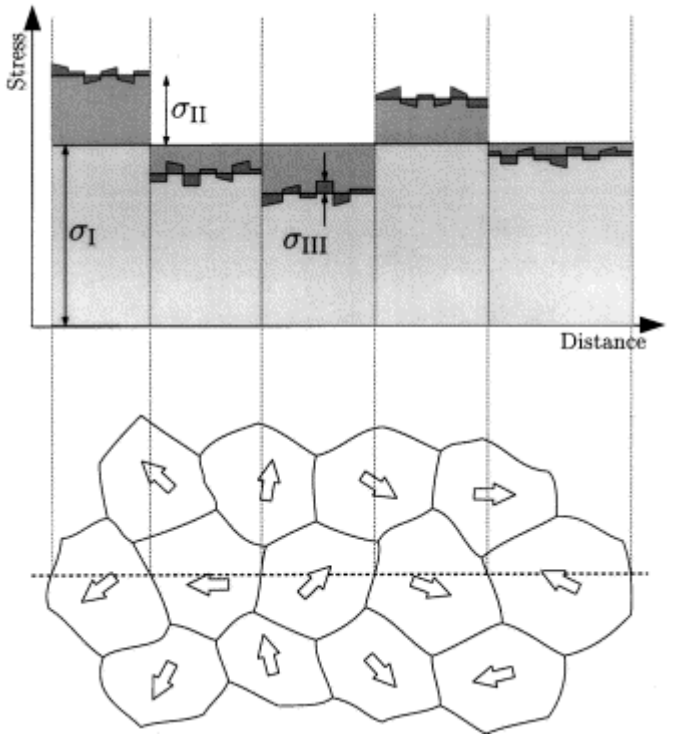
-to the simplest ones (as Voigt → homogeneous strain or Reuss→ homogeneous stress).

→ A grain interaction model has to be used….

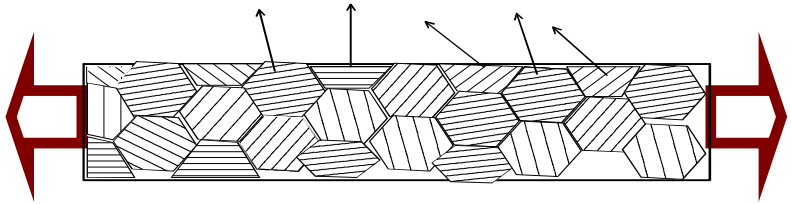
IV
X-ray strain measurements– Stress analysis:

For x-ray stress analysis or neutron stress analysis, the diffraction data are information at the microscopic level. Moreover, the diffraction information is only coming from a small part of the polycrystalline specimen. We have information on the diffracting volume alone.

How to obtain macroscopic stress σ_I (macroscopic residual stress or applied stress) from microscopic strain measurements (diffraction data) is the main question in XSA ?



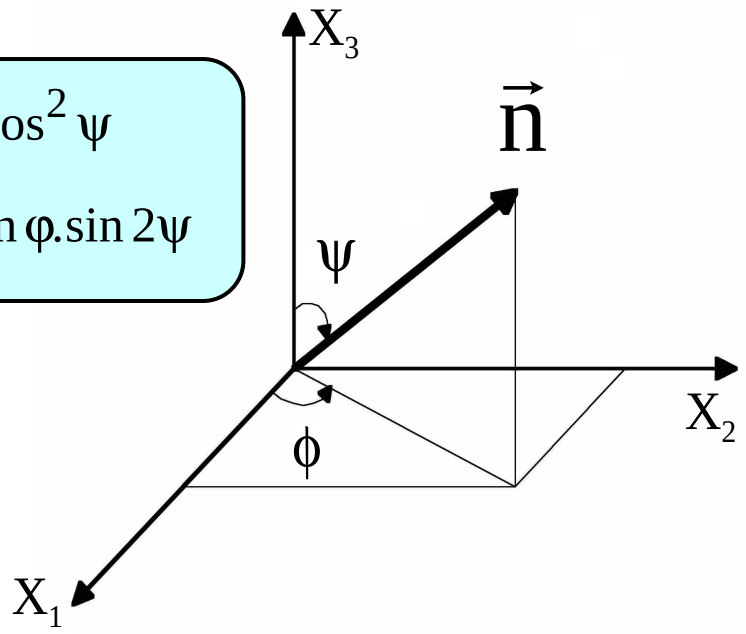
The principle is quite easy to understand:



●Fundamental equation of x-ray strain determination:

The strain in the direction of the vector n (azimuth ϕ and polar angle ψ):

$$\varepsilon_{\varphi\psi} = \varepsilon_{11}\cos^2\varphi.\sin^2\psi + \varepsilon_{22}\sin^2\varphi.\sin^2\psi + \varepsilon_{33}\cos^2\psi + \varepsilon_{12}\sin 2\varphi.\sin^2\psi + \varepsilon_{13}\cos\varphi.\sin 2\psi + \varepsilon_{23}\sin\varphi.\sin 2\psi$$



The strain in the direction n can be related to the lattice spacing:

$$\varepsilon_{\vec{n}} = \varepsilon_{\varphi\psi} = \ln\left(\frac{d_{\varphi\psi}}{d_0}\right) \approx \frac{d_{\varphi\psi} - d_0}{d_0}$$

where d_0 is the strain free lattice spacing or stress free lattice spacing.

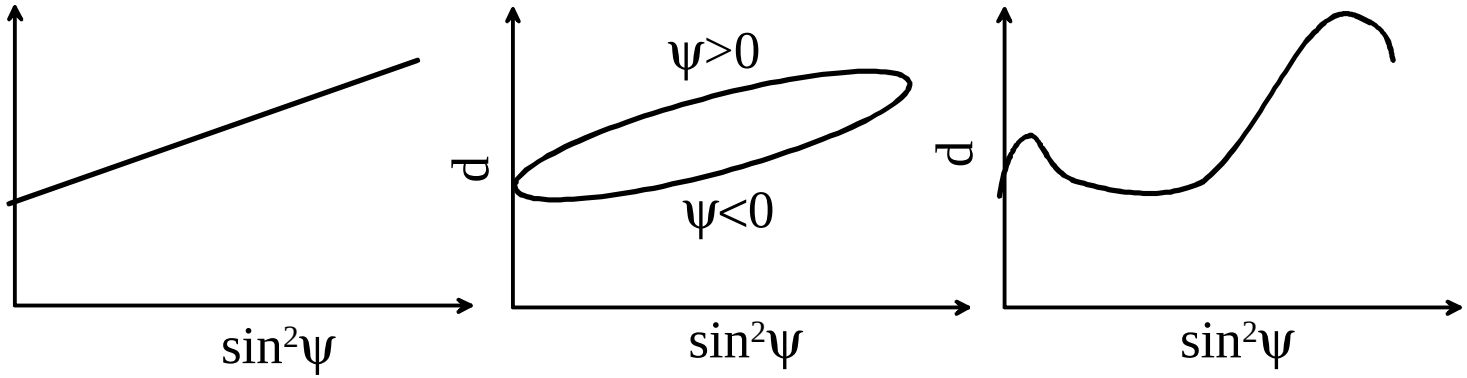
Conventional and Rational definition of strain

If we can measure d0 and $d_{\phi\psi}$, the fundamental equation of x-ray strain determination has 6 unknowns: ε11, ε22, ε33, ε12, ε13, ε23.
Thus, theoritically it can be solved if $d_{\phi\psi}$ is measured along six independents directions. In practice, more points are measured to improve accuracy.

$$\varepsilon_{\varphi\psi} = \ln\left(\frac{d_{\varphi\psi}}{d_0}\right) = \varepsilon_{11}\cos^2\varphi.\sin^2\psi + \varepsilon_{22}\sin^2\varphi.\sin^2\psi + \varepsilon_{33}\cos^2\psi$$

$$+ \varepsilon_{12}\sin 2\varphi.\sin^2\psi + \varepsilon_{13}\cos\varphi.\sin 2\psi + \varepsilon_{23}\sin\varphi.\sin 2\psi$$

In polycrystalline material, where it is possible to obtain a diffracted intensity for a lot of psi angles, different d or $d_{\phi\psi}$, vs sin²y behavior are observed.



isotropic texture and no shear stress
→ Straight lines

isotropic texture and shear stress
→ Ψ splitting

Texture material
→ Oscillatory behavior

●Stress analysis:

Let us consider an locally isotropic material → $$\varepsilon_{ij} = \left(\frac{1+\nu}{E}\right)\cdot\sigma_{ij} - \frac{\nu}{E}\cdot\delta_{ij}\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)$$

The fund.eq. becomes:

$$\rightarrow \begin{cases} \varepsilon_{\varphi\psi} = \dfrac{1+\nu}{E}\left\{\sigma_{11}.\cos^2\varphi + \sigma_{12}.\sin 2\varphi + \sigma_{22}.\sin^2\varphi - \sigma_{33}\right\}\sin^2\psi \\ \quad + \dfrac{1+\nu}{E}\sigma_{33} - \dfrac{\nu}{E}\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right) \\ \quad + \dfrac{1+\nu}{E}\left\{\sigma_{13}.\cos\varphi + \sigma_{23}.\sin\varphi\right\}\sin 2\psi \end{cases}$$

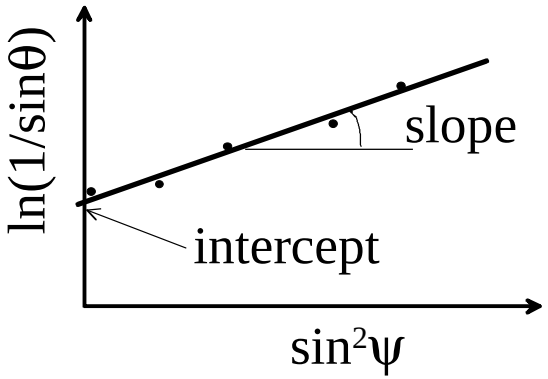
In the case of thin films (thickness < a few µm) deposited on a substrate by PVD or CVD, a few hypothesis may be used (and are often encountered in practice):

$\sigma_{33}=0$ / no shear stresses / equi biaxial stress state

This above equation becomes: $$\varepsilon_{\varphi\psi} = \ln\left(\frac{d_{\varphi\psi}}{d_0}\right) = \frac{1+\nu}{E}\sigma_{11}\sin^2\psi - 2\frac{\nu}{E}\sigma_{11}$$

Experimentally, the measurement of reflected intensity is performed for a few different tilt angles (different ψ angles), and the following equation can be plotted:

$$\ln\left(\frac{1}{\sin\theta_{\varphi\psi}}\right)=\frac{1+\nu}{E}\sigma_{11}\sin^2\psi-2\frac{\nu}{E}\sigma_{11}+\ln\left(\frac{1}{\sin\theta_0}\right)$$



If the elastic constants of the material are known:

$$\text{slope}=\frac{1+\nu}{E}\sigma_{11} \quad \Rightarrow \quad \textit{stress}$$

But, there are two unknowns in the above equation: the stress σ_{11} AND the position of Bragg peak for the stress free specimen θ_0, i.e. the stress or strain free lattice parameter a_0.

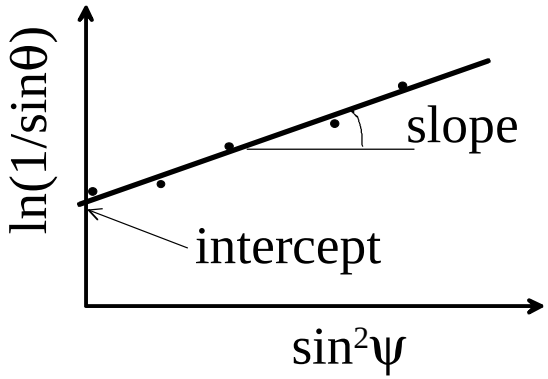
→*Several possibilities are encountered to solve this problem:*

-the simplest one is to estimate first the slope, and to obtain the stress

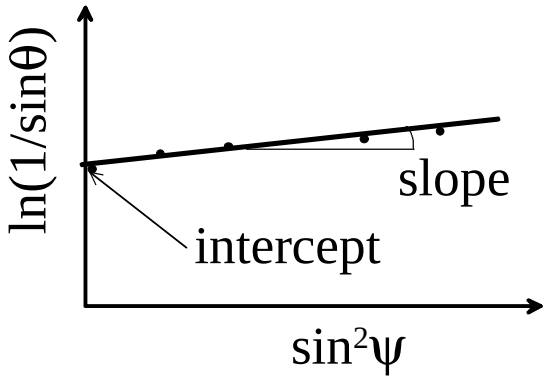
-then from the origin ordinate, you calculate the strain(or stress) free lattice parameter

If we removed one of the three preceding hypotheses we have done on the stress state, the experimental procedure is the same but the measurements have to be performed in other directions (i.e. several phi angles).

Example: the stress are no more equi biaxial, the measurements are performed for two phi angles



ϕ=0°



ϕ=90°

If $\sigma 11 \neq \sigma 22$, the slope for f=0°will be different from the slope fo r f=90°

If now there are also shear stresses, the measurements have to be performed for negative and positive psi angles and also several phi angles.

●Stress analysis:

Let us consider a locally non isotropic material → (but macroscopically isotropic)

Locally false

$$\varepsilon_{ij} = \left(\frac{1+\nu}{E}\right)\cdot\sigma_{ij} - \frac{\nu}{E}\cdot\delta_{ij}\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)$$

Macroscopically true

The fund.eq. was written as:

$$\varepsilon_{\varphi\psi} = \frac{1+\nu}{E}\left\{\sigma_{11}.\cos^2\varphi + \sigma_{12}.\sin 2\varphi + \sigma_{22}.\sin^2\varphi - \sigma_{33}\right\}\sin^2\psi$$

$$+\frac{1+\nu}{E}\sigma_{33} - \frac{\nu}{E}\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)$$

$$+\frac{1+\nu}{E}\left\{\sigma_{13}.\cos\varphi + \sigma_{23}.\sin\varphi\right\}\sin 2\psi$$

By analogy, X-ray elastic constants have been introduced:

$$\frac{1+\nu}{E} \leftrightarrow \frac{1}{2}S_2^{hkl} \qquad \left\langle \frac{1}{2}S_2^{hkl}\right\rangle^{hkl} = \frac{1+\nu}{E}$$

Where obviously

$$-\frac{\nu}{E} \leftrightarrow S_1^{hkl} \qquad \left\langle S_1^{hkl}\right\rangle^{hkl} = -\frac{\nu}{E}$$

(for relations of XEC see for example : Hauk book)

Voigt:

$$S_1^{hkl} = -\frac{3}{6}\frac{c_{11}+4c_{12}-2c_{44}}{(c_{11}-c_{12}+3c_{44})(c_{11}+2c_{12})} \qquad \frac{1}{2}S_2^{hkl} = \frac{5}{2c_{11}-2c_{12}+6c_{44}}$$

Reuss:

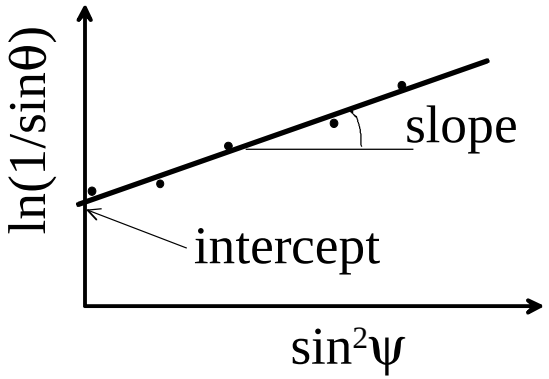
$$S_1^{hkl} = s_{12} + s_0\Gamma \qquad \frac{1}{2}S_2^{hkl} = s_{11} - s_{12} - 3s_0\Gamma$$

with

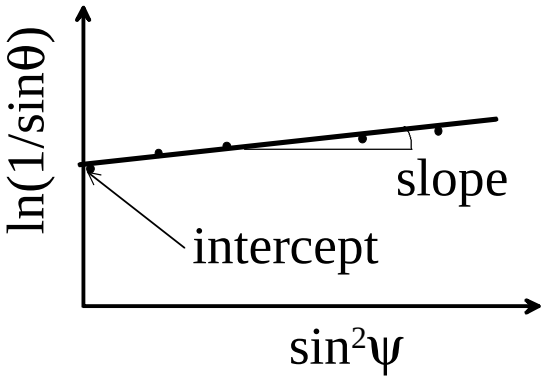
$$s_0 = s_{11} - s_{12} - \frac{1}{2}s_{44} \qquad \Gamma = \frac{h^2k^2 + h^2l^2 + k^2l^2}{\left(h^2 + k^2 + l^2\right)^2}$$

Γ=orientation parameter

Indeed as we have already seen, the Young's modulus is direction dependent. Thus, the x-ray strain measurements will differ from a plane family to another:



Measurements on {h00}



Measurements on {hhh}

For the same stress state, the slope will be larger for a softer family. For example, consider a polycrystalline material which is elastically anisotropic (A=3), the slope of {h00} family will be larger than the slope of {hhh} family.

- The determination of X-ray elastic constants (XEC):

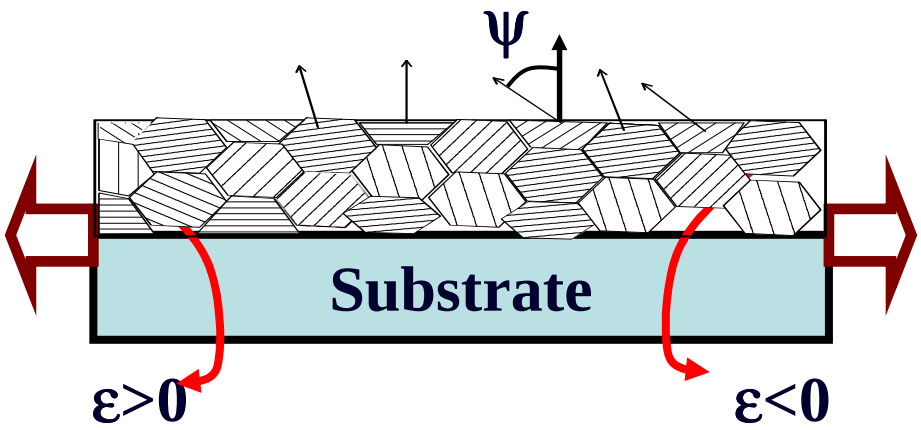
Several models of grain interaction may be used. The two simplest are:

-Voigt →isodeformation
-Reuss → iso-stress

The most realistic models are:

-Hill-Neerfeld → average of the 2 above models (experimentally this model is quite good but it has no physical justification)

-Kröner-Eshelby models→ the more realistic (but mathematically a little bit more complex)…



●Experimental errors:

Phase identification and particularly stress analysis rely on peak positions. Thus, we have to know something about the instrumental errors !!
The instrumental errors may be classified into 2 groups:

→ errors dependent on beam optics,
→ errors dependent on alignment.

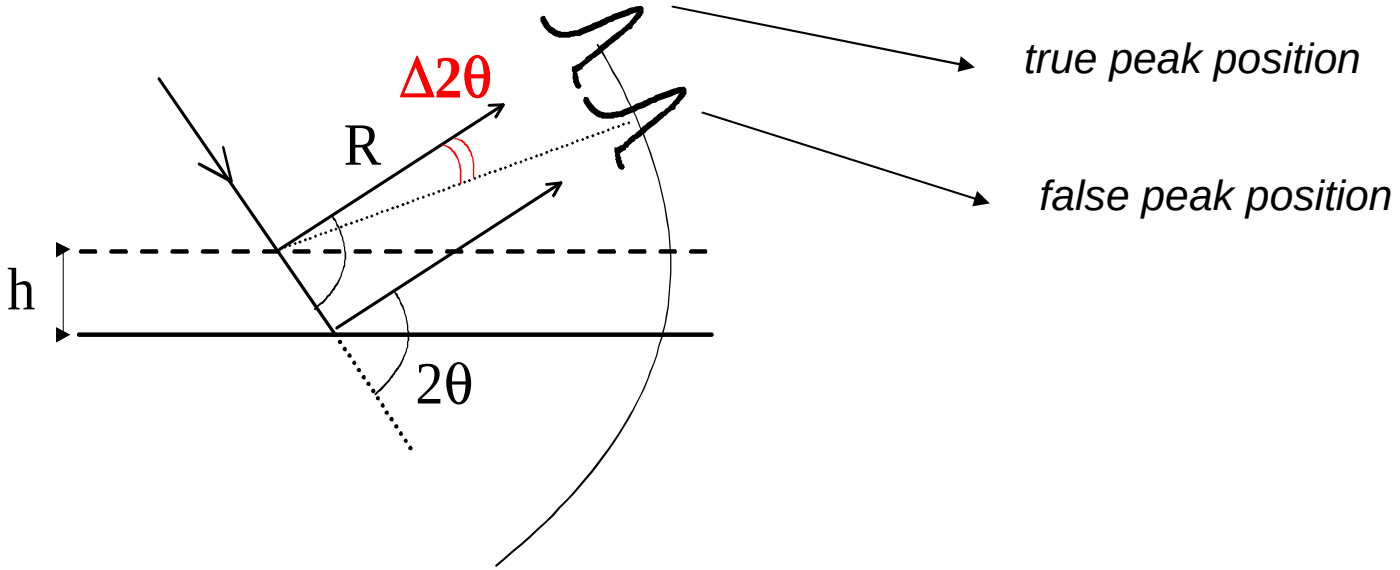
All these errors can be checked with standard specimens, i.e. specimens for which you perfectly know the lattice parameter, the texture, the residual stress state.

For details of influence of each parameters, you should refered to the books cited in the first slide.

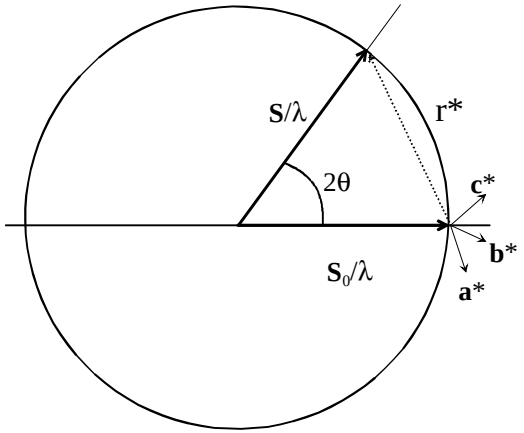
If we consider that we have no problem with optics, let us interest to errors which originate from instrumental misalignment: sample displacement from the center of the goniometer, effect of psi axis not corresponding to the 2theta axis.

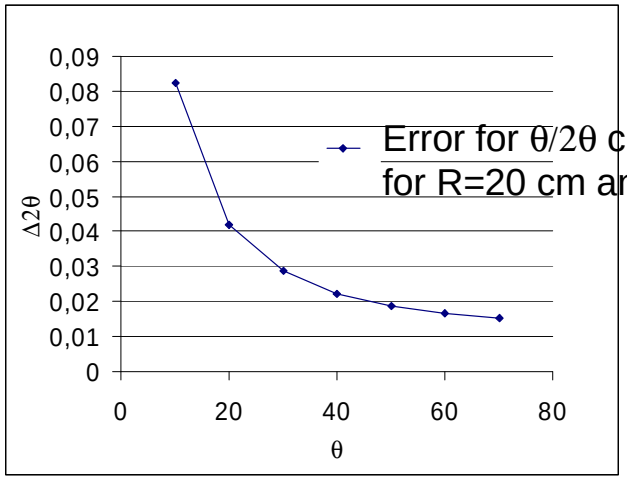
The first thing to do is to put the specimen on the center of the goniometer !

Instrumental errors



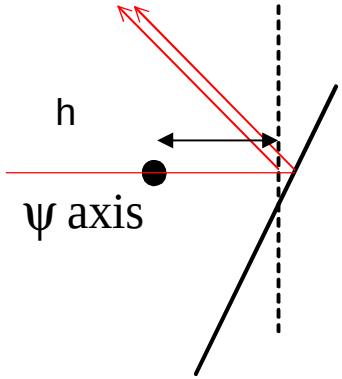
The error due to the sample displacement depend on 2theta angles:



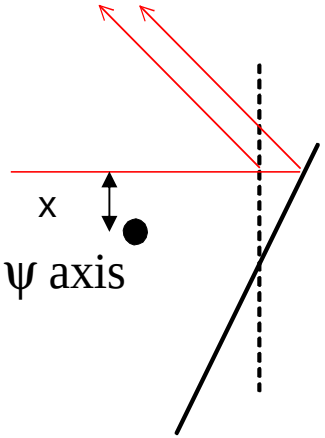


Moreover, if the specimen is displaced from the center of the goniometer, the error in 2theta angles will depend on the psi angles.

Displacement in the psi geometry:



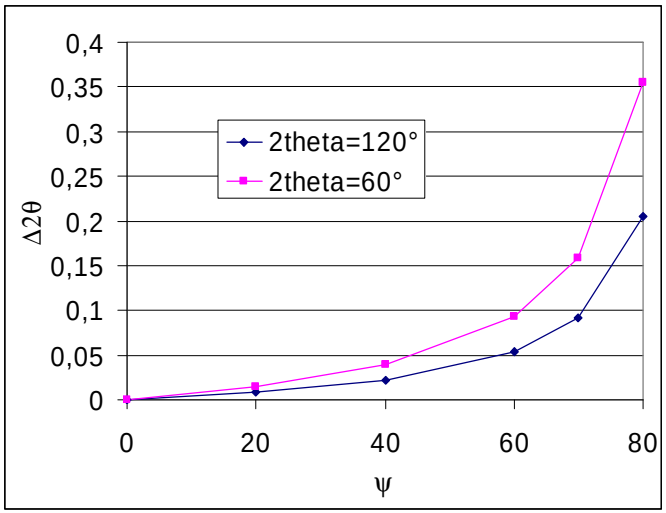
Simple case (only error displacement of the specimen)



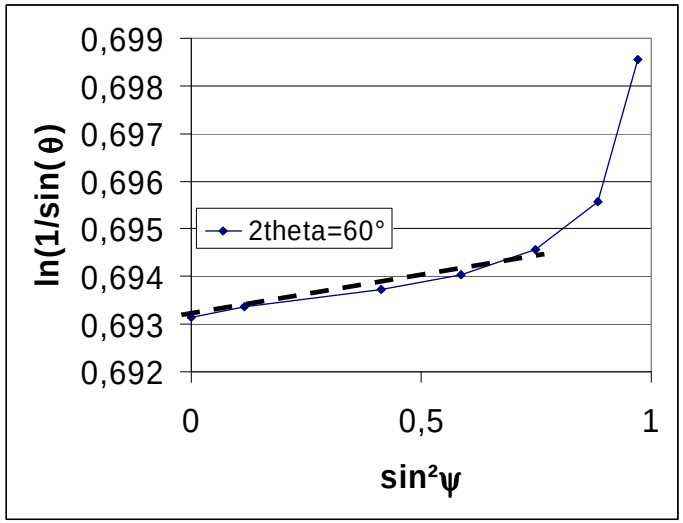
general case-error displacement of the specimen and x-ray beam not on the psi axis

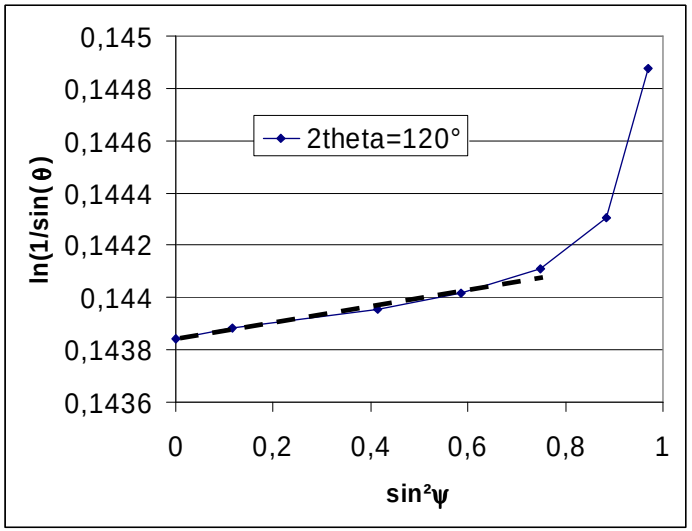
The peak shift between ψ=0 and ψ is given by:

$$\Delta 2\theta = \frac{2}{R}.\cos\theta.\left(h.\left(\frac{1}{\cos\psi} - 1 \right) + x.\tan\psi \right)$$



If you perform x-ray stress analysis (XSA):





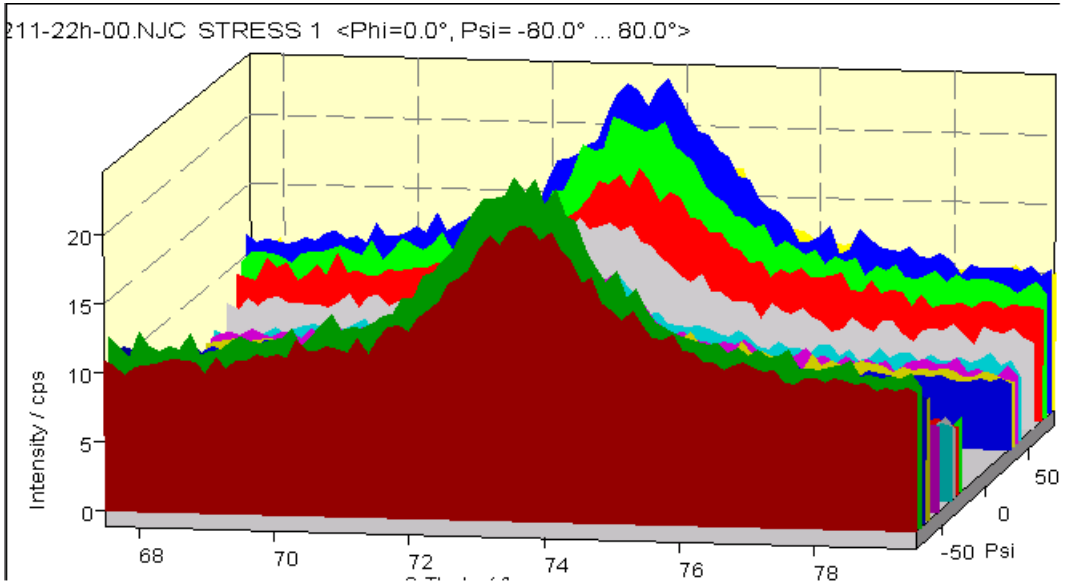
For 2theta=60° slope=17.5x10 $^{-4}$ For 2theta=120° slope=3.37x10 $^{-4}$

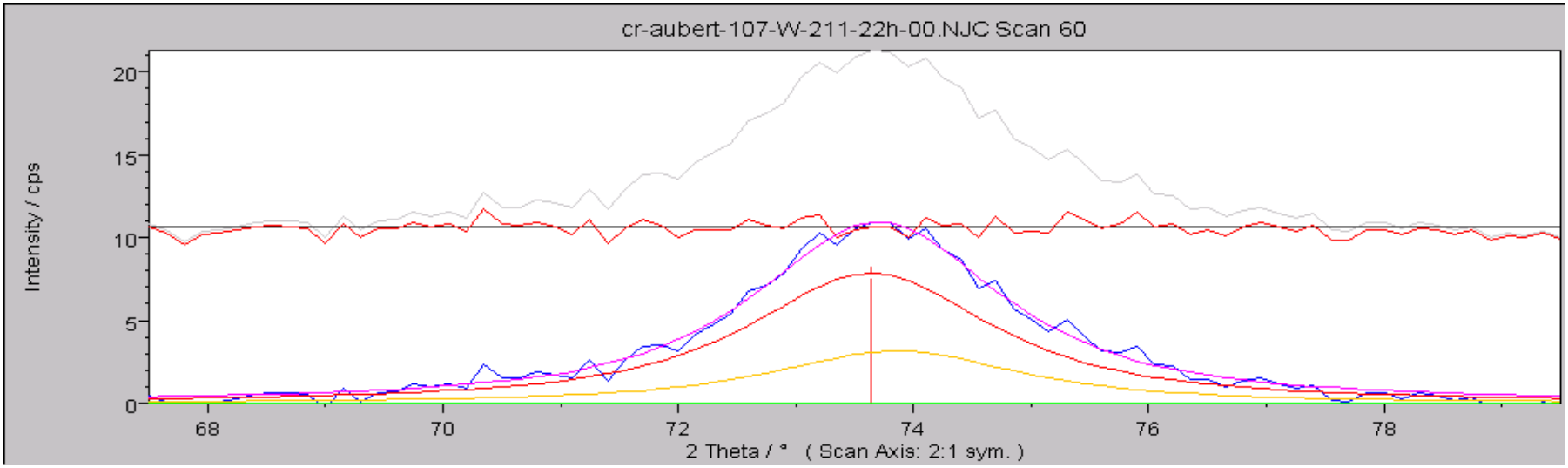
This means that for an position error of 50 µm (x and h), we will obtain stress in the sample. And the stress values are 5 times higher at 2theta=60° than 120°!!

→ You have to pay carefull attention to the installation of the specimen on the goniometer…

How to perform experimentaly stress analysis ?

-choose a Bragg peak, and perform theta/2theta scans for a set of psi angles





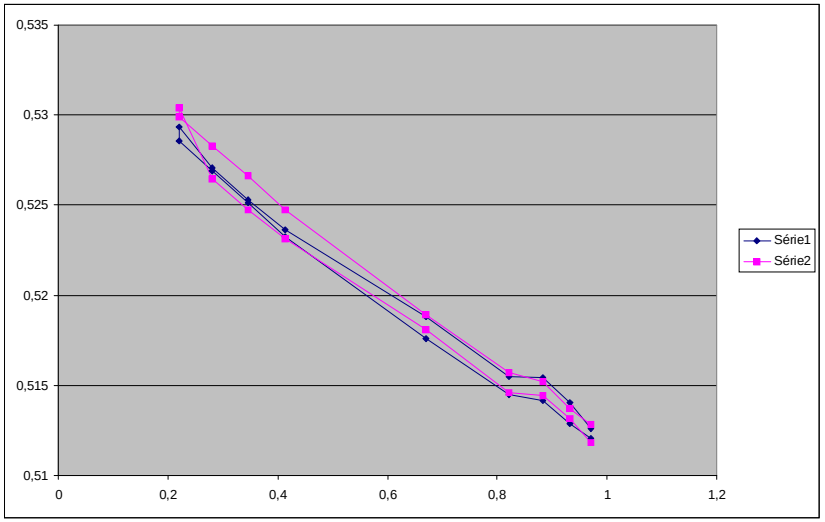
How to perform experimentaly stress analysis ?

W-211-4h-00,NJC

2θ	Center of Gravity	FWHM	Inetto int	Inetto integral
73,5534	73,5535	1,7871	15,07	35,33
72,7901	72,7906	1,7096	9,43	20,42
72,6599	72,6606	1,8469	8,82	21,39
72,7355	72,7363	1,572	9,86	21,6
72,8686	72,8679	1,6398	9,28	22,63
72,9105	72,9116	1,7464	6,45	15,94
73,2034	73,2022	1,3791	5,55	10,92
73,3743	73,3741	1,7634	9,37	22,67
73,4746	73,4734	1,8174	14,02	34,52
73,5482	73,5487	1,8099	15,83	36,18

SIN(Ψ²)	LN(1/SINθ)	avec correction
0,88302222	0,51299811	0,514019322
0,28081443	0,52197194	0,52335973
0,22040355	0,52351517	0,5245805
0,28081443	0,52261865	0,523574504
0,3454915	0,52104327	0,5220631
0,41317591	0,52054813	0,521539969
0,67101007	0,51709743	0,517889843
0,8213938	0,51509252	0,515583494
0,88302222	0,51391877	0,51442639
0,9330127	0,51305882	0,513522535

sans correction	avec correction
pente	pente
-0,014317445	-0,015117299
ordonnées origine	ordonnées origine
0,526393269	0,527721417
coeff. Déterm.	coeff. Déterm.
0,991350926	0,996777678
sigma (Gpa)	sigma (Gpa)
-4,6	-4,9
a0 (Angstrom)	a0 (Angstrom)
3,17413	3,17724

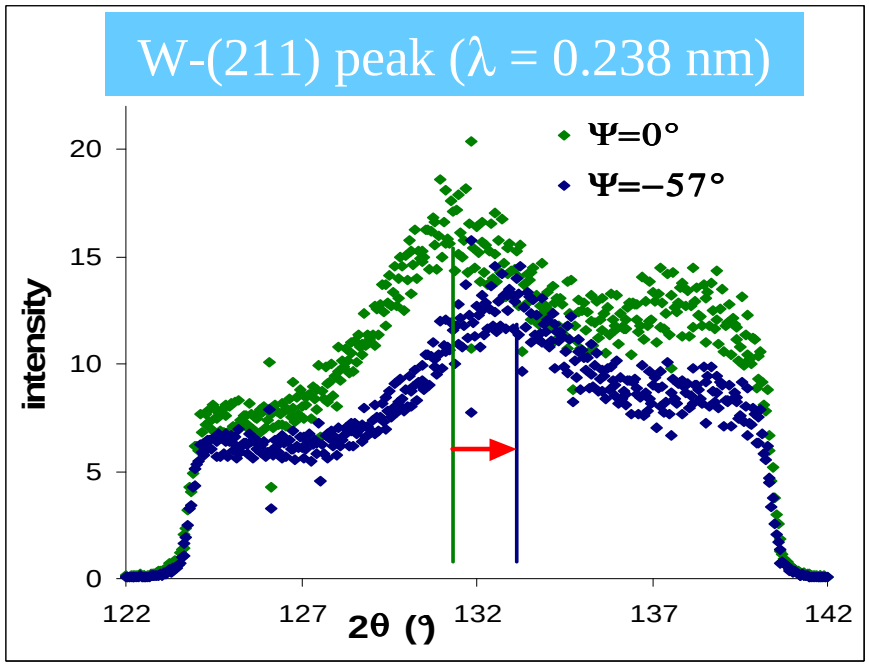


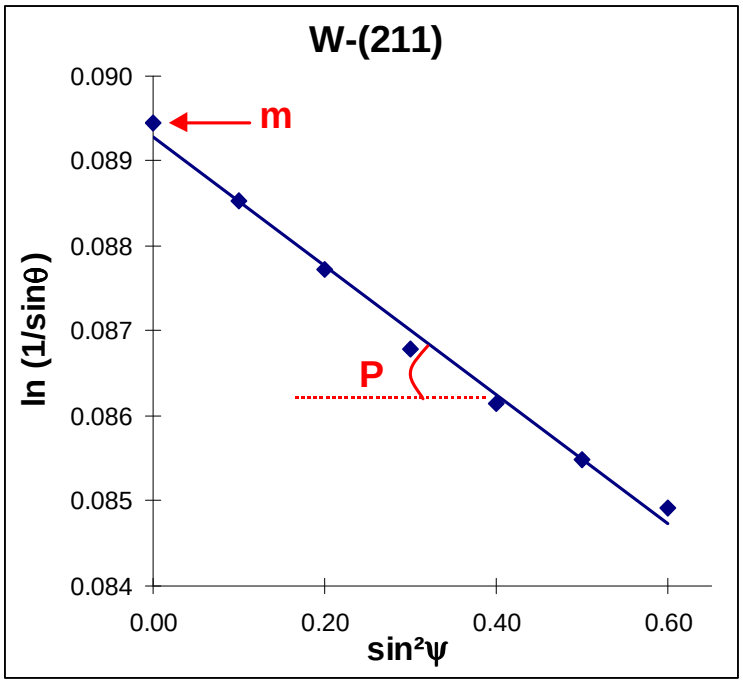
Ln(1/sinθ)

Sin²ψ

W/WC multilayer
Total thickness 200 nm

Example of XSA:





200 nm tungsten film on polyimide:

≈ 2° shift of the peak position

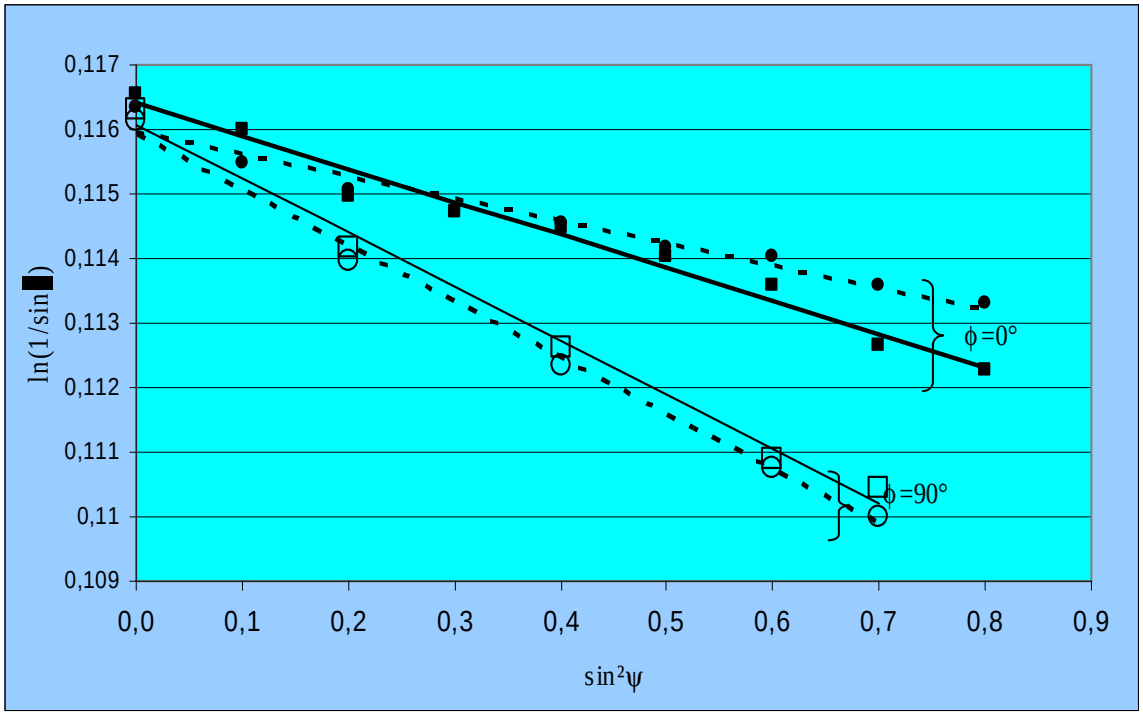
$$\ln\left(\frac{1}{\sin\theta}\right) = \frac{1+\nu}{E}\sigma\sin^2\psi - 2\frac{\nu}{E}\sigma + \ln\left(\frac{1}{\sin\theta_0}\right)$$

P→ σ = -2.3 GPa **m→ $a_0 > a_{bulk}$**

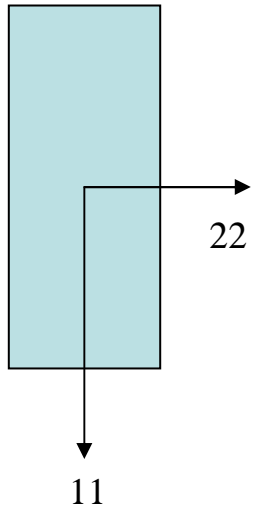
Example of XSA:

Stainless steel thin film deposited by PVD onto kapton substrate

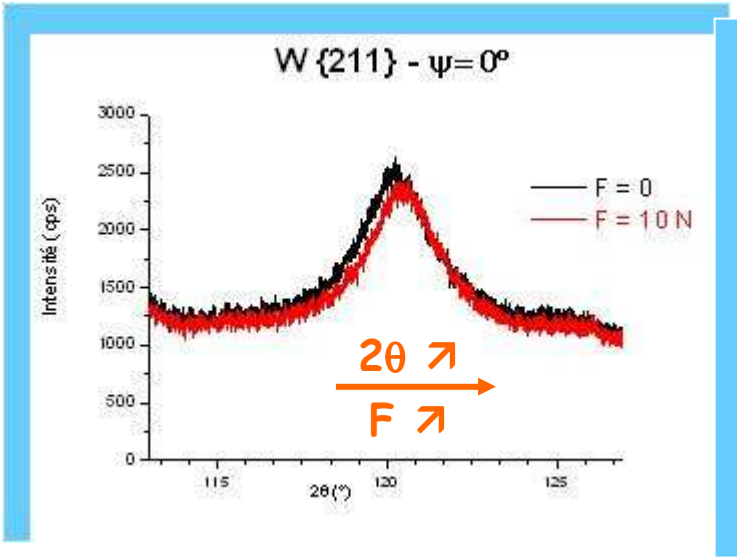
Polycrystalline non textured and 340 nm thick

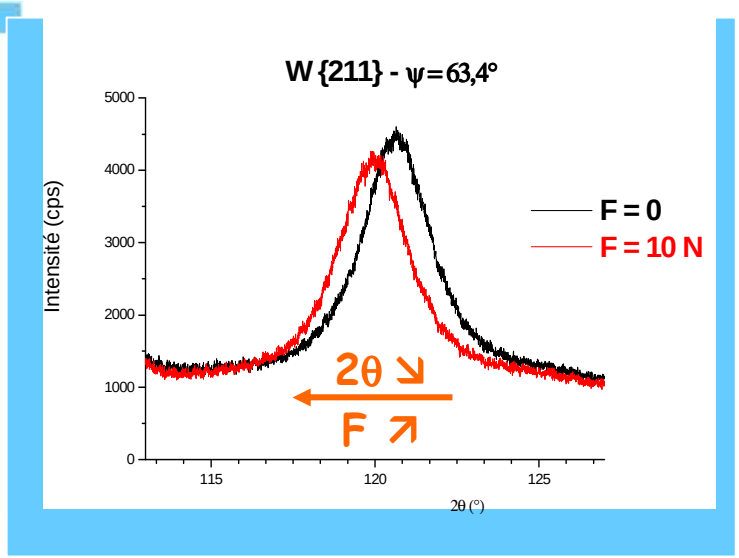


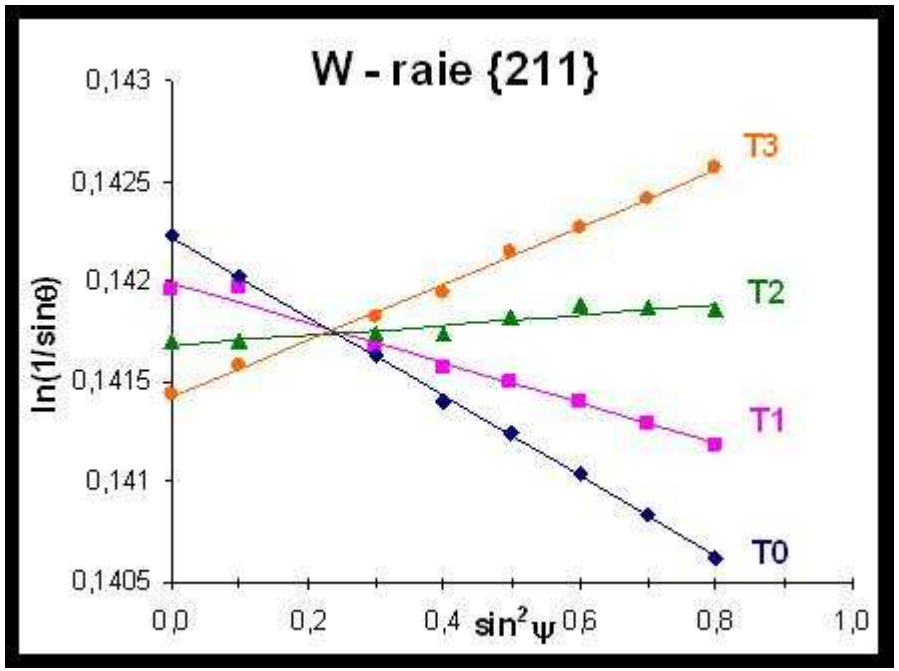
Top view



Example of XSA: **Polycrystalline isotropic (D ~ 10 nm)**







X-ray strain analysis

- isotropic or quasiisotropic thin film (tungsten):

$$\varepsilon_{0,\Psi} = \left(\frac{1+\nu_f}{E_f}\right)_{\mathbf{hkl}} \cdot \sigma_{11}^f \cdot \sin^2\Psi - \left(\frac{\nu_f}{E_f}\right)_{\mathbf{hkl}} \cdot \left(\sigma_{11}^f + \sigma_{22}^f\right)$$

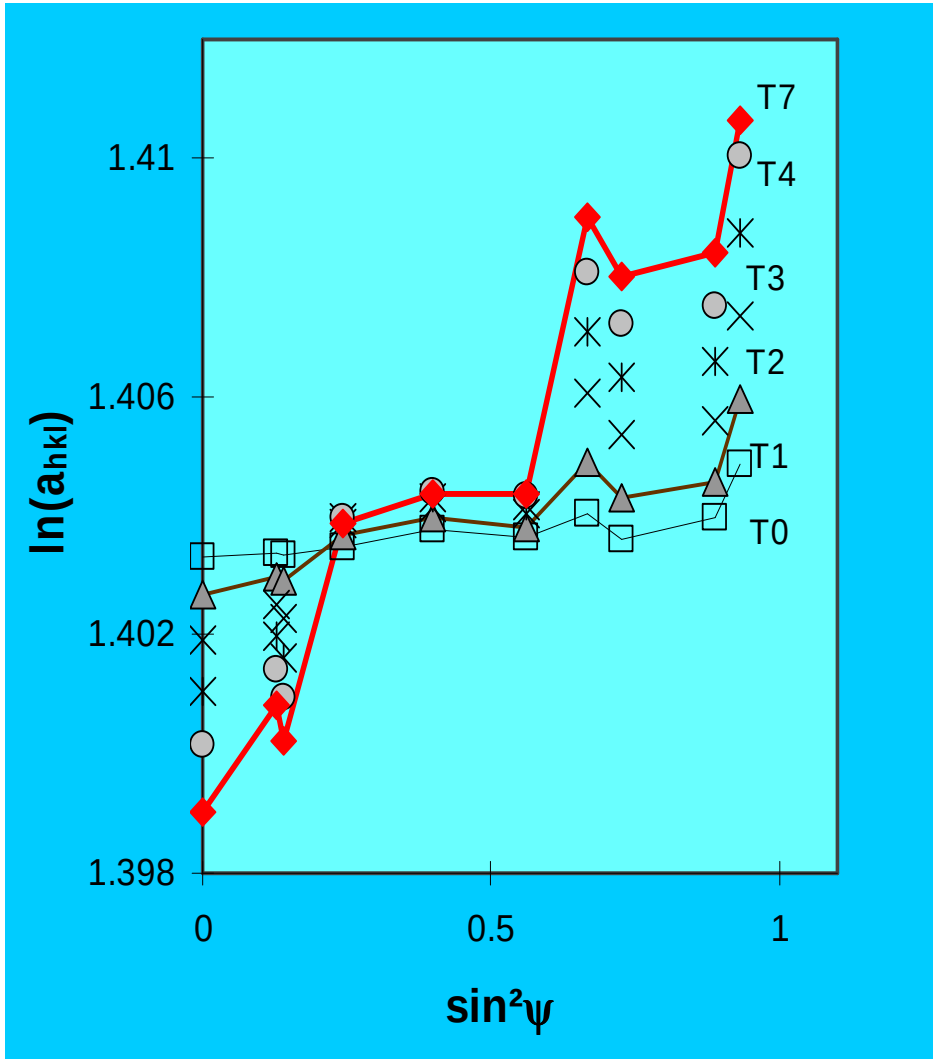
(classical $\sin^2\psi$ method)

- (111) textured anisotropic thin film (Gold) :

$$\varepsilon_{\Phi\Psi}^{33} = \left(\sigma_{22}^f - \sigma_{11}^f\right)\left[\frac{2s_{11}-2s_{12}-s_{44}}{6\sqrt{2}}\sin(\Phi+2\Phi_\sigma)\sin 2\Psi + \frac{-s_{11}+s_{12}-s_{44}}{6}\cos 2(\Phi-\Phi_\sigma)\sin^2\Psi\right]$$
$$+\left(\sigma_{22}^f + \sigma_{11}^f\right)\left[\frac{2s_{11}+4s_{12}-s_{44}}{6} + \frac{s_{44}}{4}\sin^2\Psi\right]$$

(Crystallite-group method)

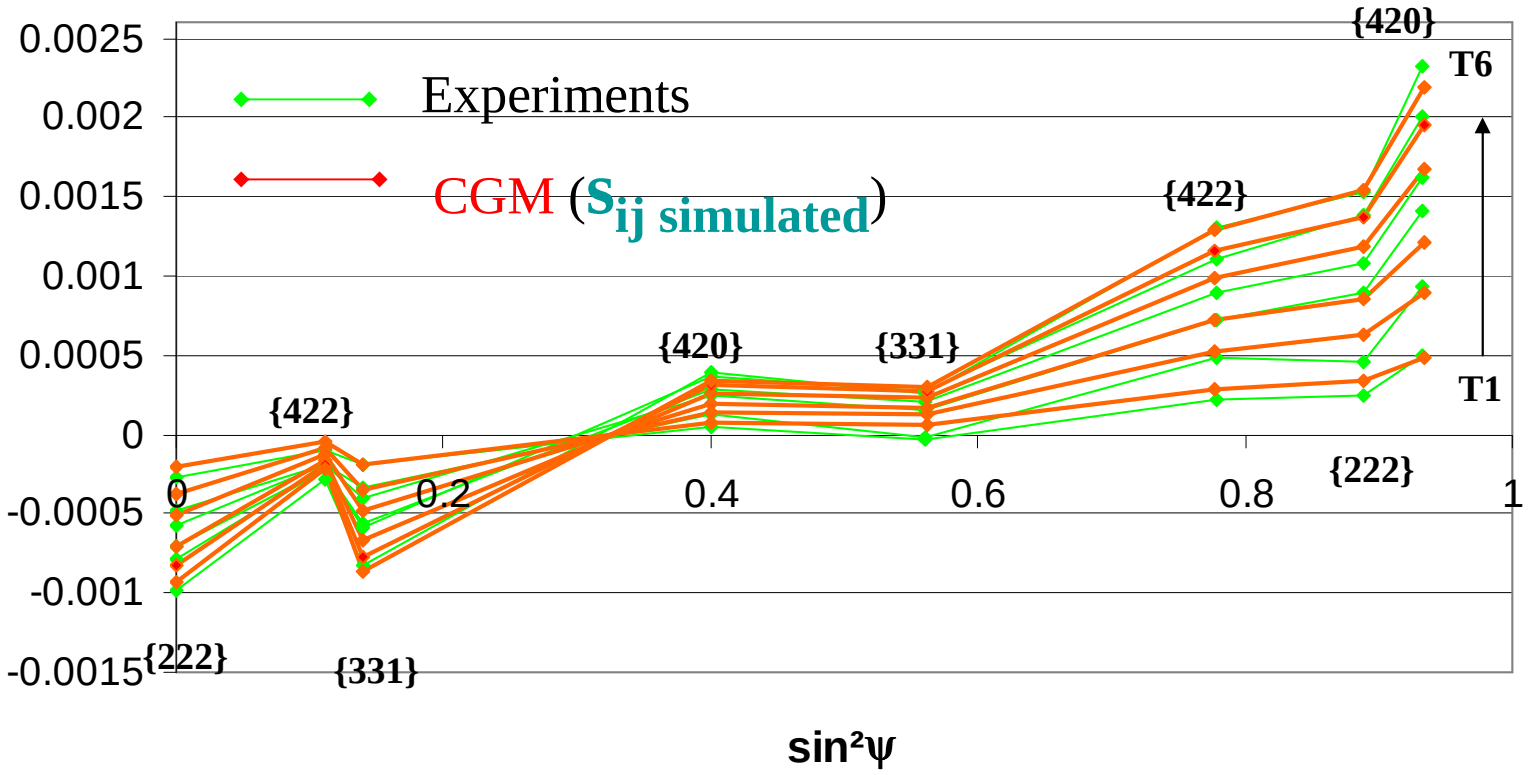
Gold thin film deposited on kapton substrate



Average slope

Applied load

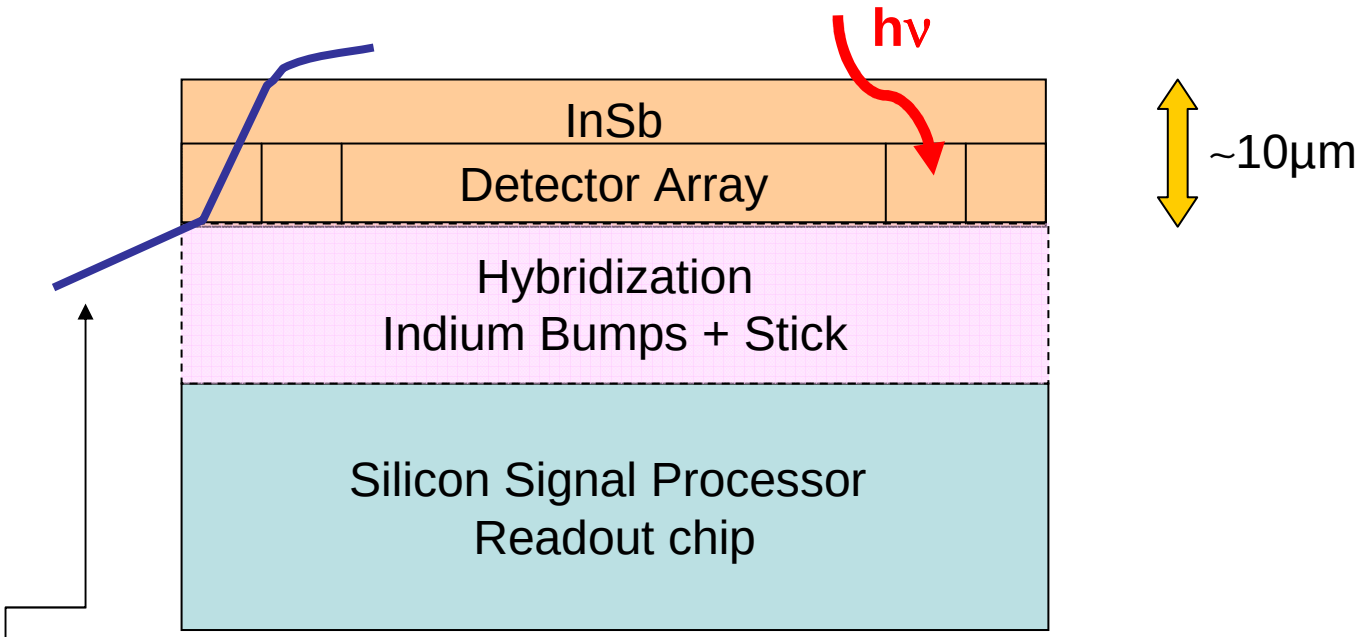
Gold thin film deposited on kapton substrate



→ Several Bragg's families AND → no more linear

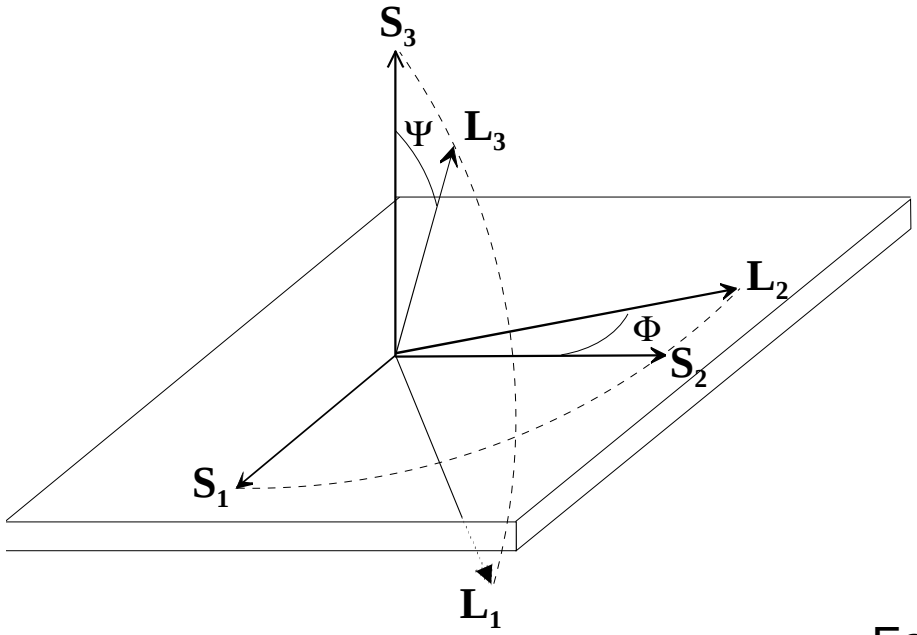
Another example: X-ray diffraction measurement of thermal strain in InSb-based devices

Hybrid Focal Plane Arrays (FPA) Technology
Backside-illuminated InSb IR Detector Arrays
High Fill Factor / High Sensitivity



Working Temperature : Liquid Nitrogen
Problems upon cooling
Example: Cleavage of the thin brittle InSb layer

Some mechanics:



$$\varepsilon_{ij} = s_{ijkl}.\sigma_{kl}$$

$$\overline{\overline{\sigma}} = \begin{pmatrix} \sigma_{11} & 0 & 0 \\ 0 & \sigma_{11} & 0 \\ 0 & 0 & 0 \end{pmatrix}$$

For Silicon:

S_3// [001]

$$\begin{pmatrix} S_{11} & S_{12} & S_{12} & 0 & 0 & 0 \\ S_{12} & S_{11} & S_{12} & 0 & 0 & 0 \\ S_{12} & S_{12} & S_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & S_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & S_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & S_{44} \end{pmatrix}$$

For InSb:

S_3//[111]

$$\begin{pmatrix} s'_{11} & s'_{12} & s'_{13} & 0 & s'_{15} & 0 \\ s'_{12} & s'_{11} & s'_{13} & 0 & -s'_{15} & 0 \\ s'_{13} & s'_{13} & s'_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & s'_{44} & 0 & s'_{46} \\ s'_{15} & -s'_{15} & 0 & 0 & s'_{44} & 0 \\ 0 & 0 & 0 & s'_{46} & 0 & s'_{66} \end{pmatrix}$$

Some mechanics

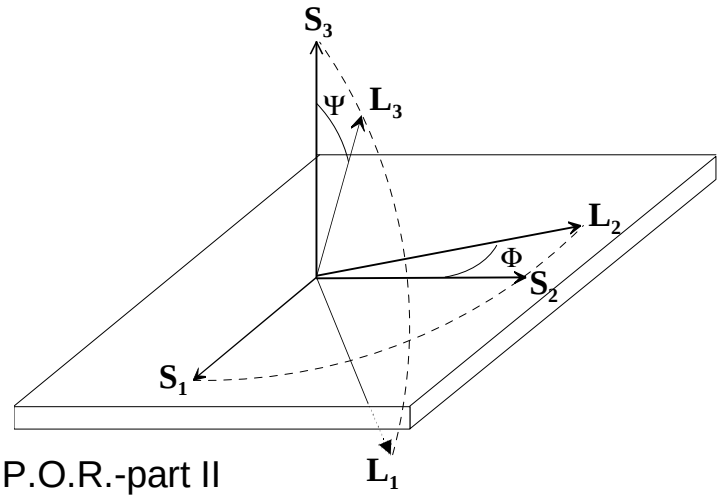
For the specific orientation of the two single crystals, the mismatch of CTE induces a quite simple equibiaxial strain tensor (i.e. which is isotropic in the plane).

- $\bar{\bar{\sigma}} = \begin{pmatrix} \sigma_{11} & 0 & 0 \\ 0 & \sigma_{11} & 0 \\ 0 & 0 & 0 \end{pmatrix}$
- $\varepsilon_{ij} = s_{ijkl}.\sigma_{kl}$

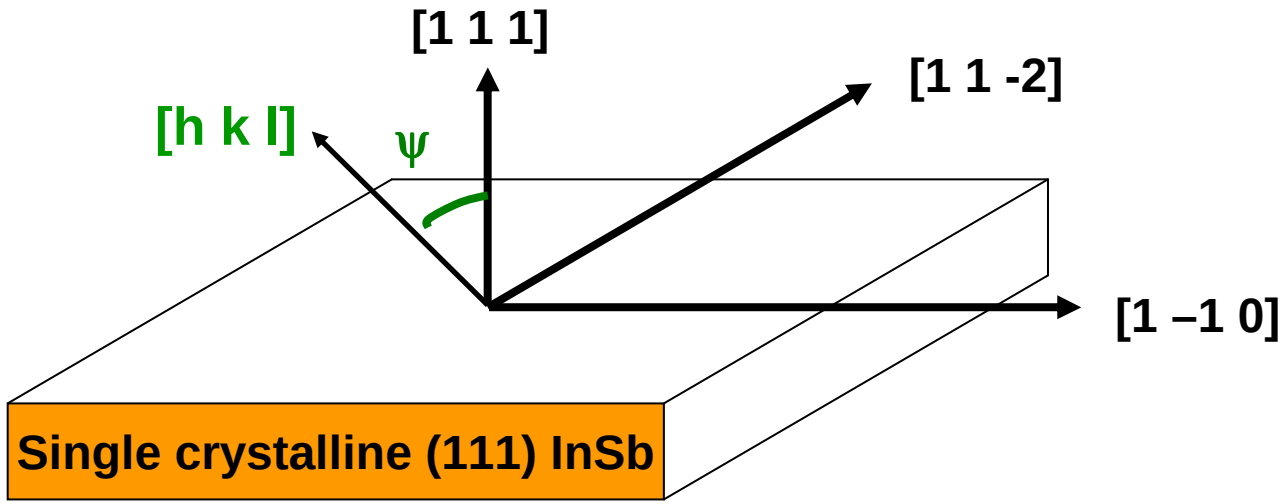
$$\bar{\bar{\varepsilon}} = \begin{pmatrix} \varepsilon_{11} & 0 & 0 \\ 0 & \varepsilon_{11} & 0 \\ 0 & 0 & \varepsilon_{33} \end{pmatrix}$$

$$\begin{cases} \varepsilon_{11} = \varepsilon_{22} = (s'_{11}+s'_{12}).\sigma_{11} \\ \varepsilon_{33} = 2s'_{13}.\sigma_{11} \end{cases}$$

$$\varepsilon_{\varphi\psi} = (\varepsilon_{11} - \varepsilon_{33}).\sin^2\psi + \varepsilon_{33}$$



$$\varepsilon_{InSb/Si} = \frac{l^{100K}_{InSb/Si} - l^{100K}_{InSb}}{l^{100K}_{InSb}}$$



XRD measurement along [hkl] :

lattice spacing (parameter) d_ψ (a_ψ)

Stress free lattice spacing (parameter) d_0 (a_0)

InSb (111) on Si (100) : A simple mechanical problem

Isotropic strain is in the plane of the InSb layer	$\varepsilon_{11}=\varepsilon_{22}$
Equi-biaxial thermal stress tensor	$\sigma_{11}=\sigma_{22}$
No shear stress	$\sigma_{ij}=0$ for i≠j
Free surface	$\sigma_{33}=0$

$$\varepsilon_\psi = \ln\left(\frac{d_\psi}{d_0}\right) = \ln\left(\frac{a_\psi}{a_0}\right)$$

$$\varepsilon_\psi = (\varepsilon_{11} - \varepsilon_{33}).\sin^2\psi + \varepsilon_{33}$$

and

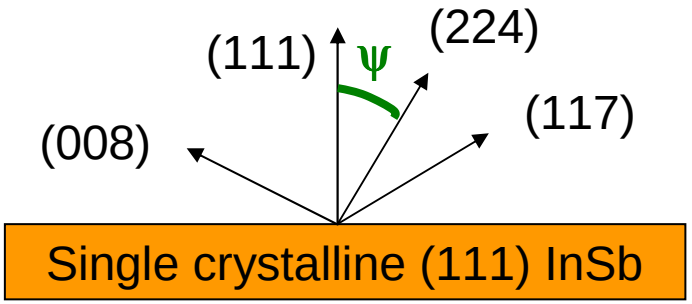
$$\ln\left(a_\psi\right) = (\varepsilon_{11} - \varepsilon_{33}).\sin^2\psi + \varepsilon_{33} + \ln\left(a_0\right)$$

$$\begin{cases} \varepsilon_{11} = \varepsilon_{22} = \left(s'_{11} + s'_{12}\right).\sigma_{11} \\ \varepsilon_{33} = 2s'_{13}.\sigma_{11} \end{cases}$$

$\lambda_{CuK\alpha 1}$ = 0.15406 nm

Probing depth in InSb

1.5 µm to 3 µm

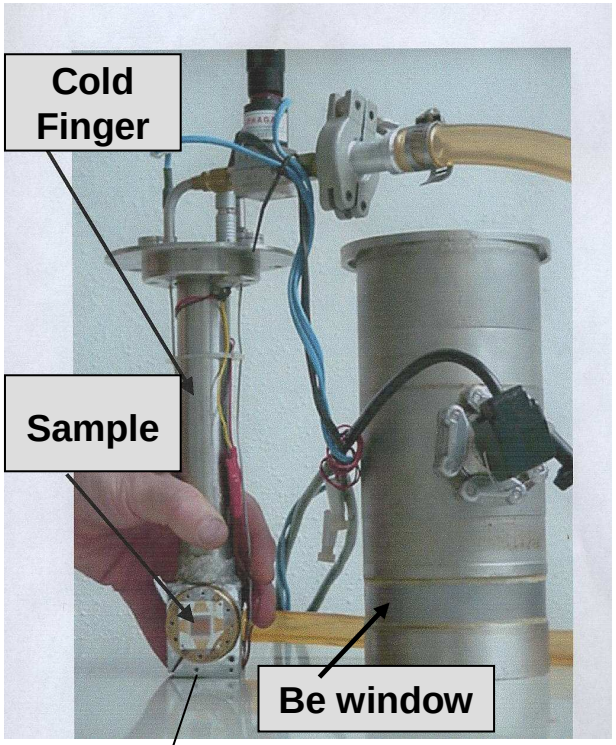


InSb	(333)	(444)	(224)	(551)	(117)	(008)
2θ	76.29	110.89	71.23	116.19	116.19	143.97
ψ	0	0	19.47	-27.21	43.31	54.74
$\sin^2\psi$	0	0	0.11	0.21	0.47	0.67

	Mass absorption coefficient (cm²/g)	density (g/cm3)
Indium (In)	243	7.31
Antimony (Sb)	270	6.697

Experimental Set-up

Home-made Cryostat



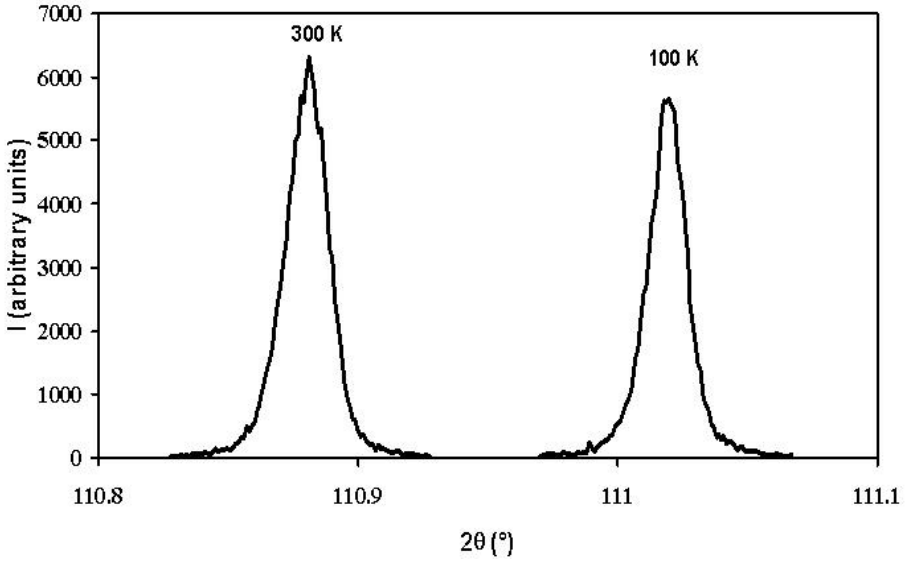
XRD 3000 - SEIFERT

Samples: **InSb** arrays hybridized on **Si (625 µm** or **100 µm)** on **AlN** ($\alpha \# \alpha^{Si}$)

or **Al_2O_3** ($\alpha > \alpha^{Si}$)

Bulk stress free InSb

Lattice Parameter



(444) Diffraction peak

Bulk InSb	300K	100K
a (nm) (measured)	**0.64800** ±0.00005	**0.64745** ±0.00005
a (nm) (theoretical)	**0.64800** (known)	**0.64744** (calculated)

$$l_T = l_{T0}.(1+\alpha\Delta T)$$

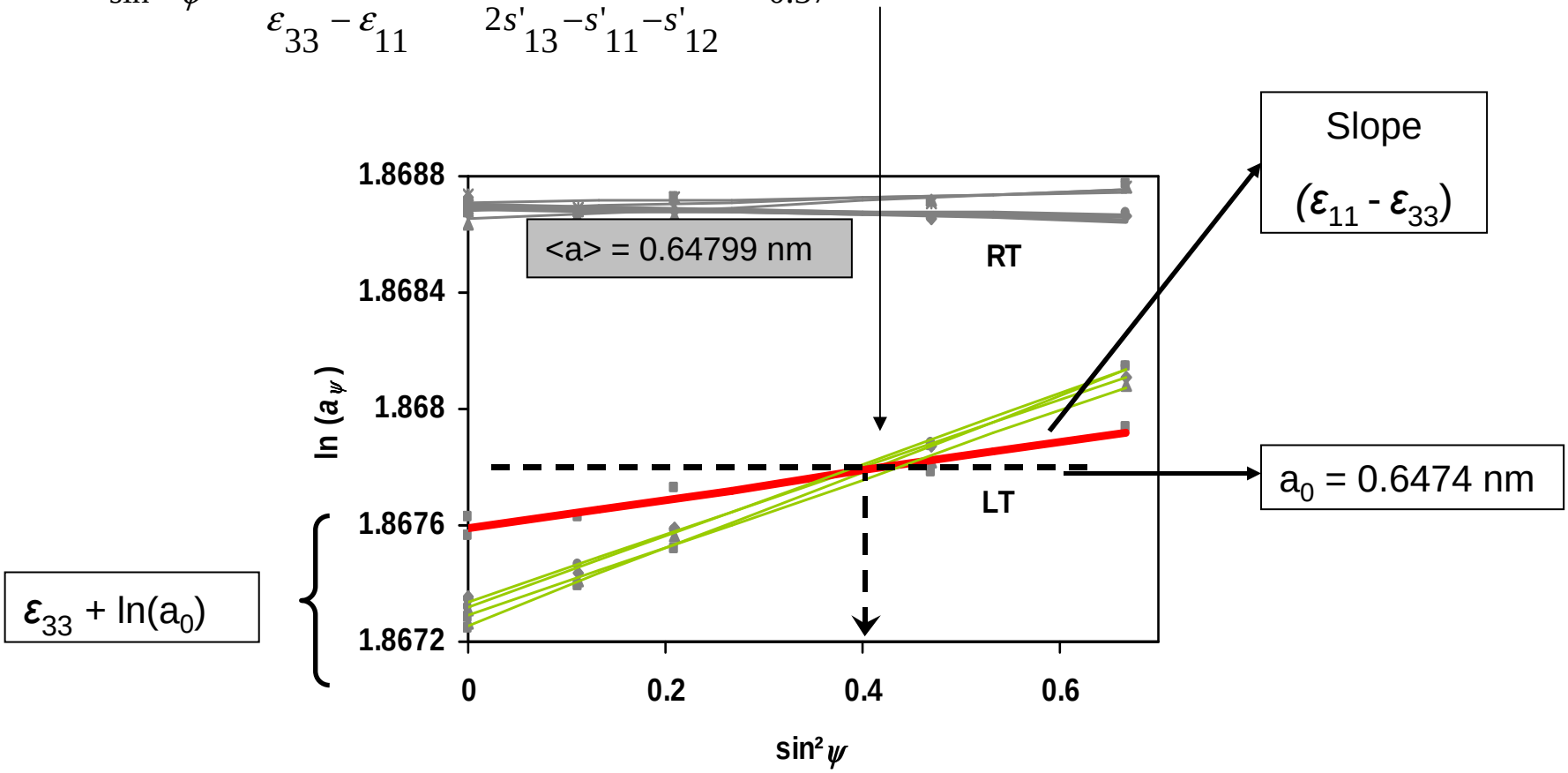
α^{InSb} = 4.35x10^{-6} / K

ΔT = - 200 K

$$\ln\left(a_\psi\right) = (\varepsilon_{11} - \varepsilon_{33}).\sin^2\psi + \varepsilon_{33} + \ln\left(a_0\right)$$

$$\left\{ \begin{array}{l} \varepsilon_{11} = \varepsilon_{22} = \left(s'_{11} + s'_{12}\right).\sigma_{11} \\ \varepsilon_{33} = 2s'_{13}.\sigma_{11} \end{array} \right.$$

$$\sin^2\psi^* = \frac{\varepsilon_{33}}{\varepsilon_{33} - \varepsilon_{11}} = -\frac{2s'_{13}}{2s'_{13} - s'_{11} - s'_{12}} = 0.37$$



RESULTS

InSb (10 µm) at 100 K	ε_{33}	ε_{11}	σ_{11} (MPa)	Estimation (MPa)
4 devices Si (625 µm)	-5×10^{-4}	7×10^{-4}	77±7	62
1 device Si (100 µm)	-2×10^{-4}	3×10^{-4}	29±4	59

CONCLUSIONS

1) Yield Stress of InSb at 100 K (>500 MPa) $> \sigma_{11}$

2) Influence of the underlying substrate (Al_2O_3)
More thicker than Si (100 µm) and $\alpha > \alpha^{Si}$
and \ or: bad quality of the hybridization

3) not any studied device has cleaved upon cooling

cleavage is related to defective cutting of the initial InSb wafer

- XRD is a unique tool for contactless determination of low temperature-induced strain in the thin InSb part of ready-to-work InSb-based infrared photo-detectors.

- The mechanical simplicity of the studied system (InSb (111) on Si (100) in a planar geometry) allowed simplifications in the formal treatment leading to an easy evaluation of the thermal stress.

- This study has ruled out the influence of the difference between CTE of InSb and silicon in problems encountered upon cooling of such devices.

Summary:

-We have seen that the diffraction method of analysing stress consists to measure strains along various directions in the specimen and then use these values to calculate the stresses along any direction through tensor transformation formulae and the appropriate form of Hooke's law.
-The method permits the determination of general triaxial stress state.
-The method require a homogeneous strain distribution within the irradiated volume.

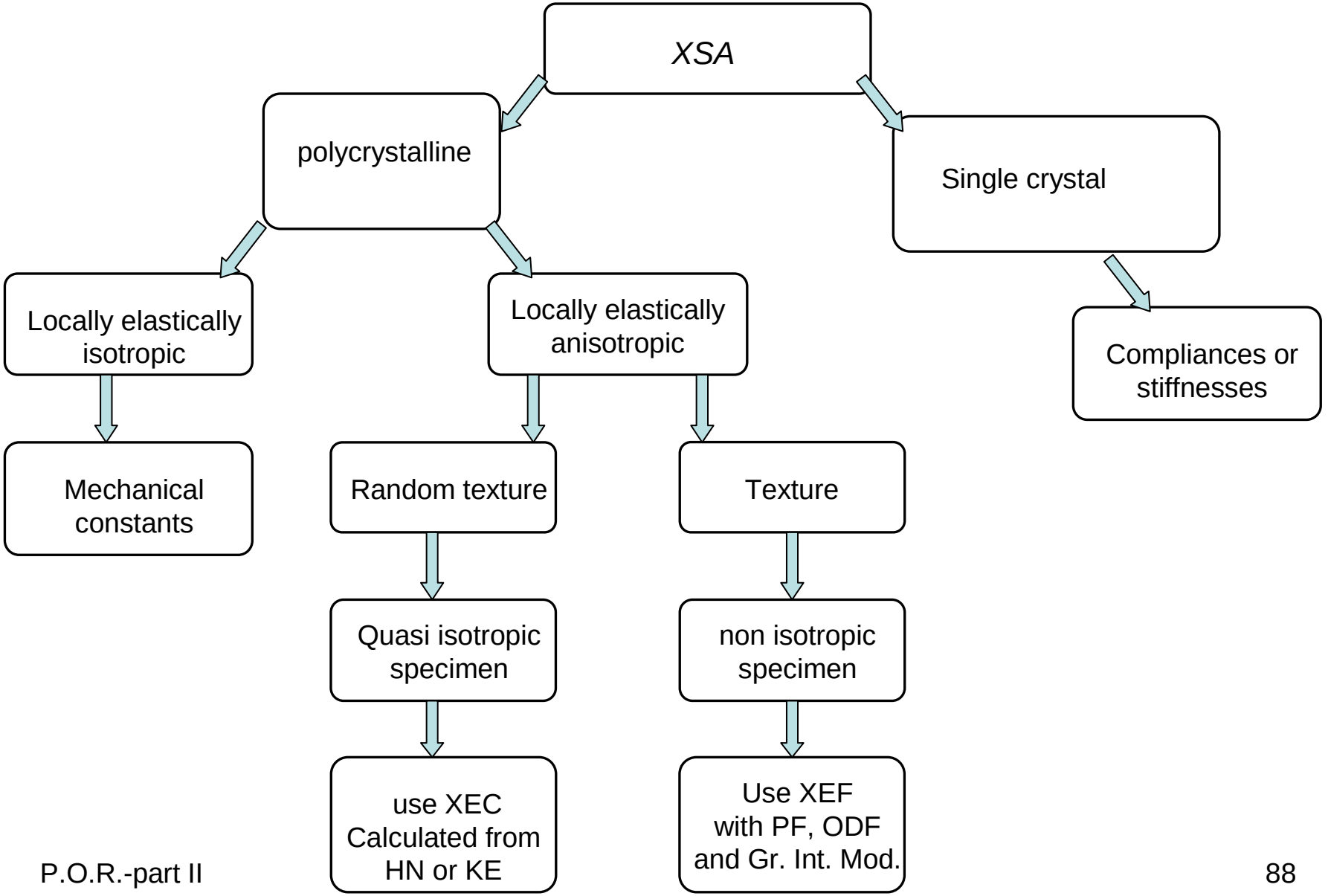
-experimentally determined d vs $\sin^2\psi$ plots indicate that the strain distribution within the irradiated volume is indeed homogeneous.

Two properties of measured data should be used first to check the dimensionality of the strain tensor:

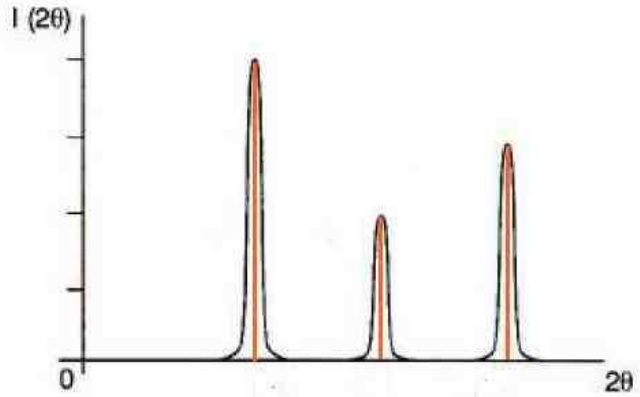
-ψ splitting in d vs $\sin^2\psi$ plots which is characteristic of shear strains.
-curvature in d vs $\sin^2\psi$ plots which indicates strain gradients with depth.

-The method is quite simple and accurate for macroscopically isotropic material (that is locally isotropic or anisotropic materials (quasi-isotropic material)). For macroscopically anisotropic material, i.e. texture material, the mechanical modeling of the material is more complex and sometimes less confident. The stress analysis is still possible but with less accuracy.

If the specimen is ***homogeneous***, i.e no stress gradient, no composition gradient,…



What is the informations in a Bragg peak ?



- **Position**
- **Intensity**
- **Shape → FWHM**

- Phase analysis (peak positions)
- Texture analysis (variation of peak intensity)
- Grain size – microdistortions (peak shape)
- X-ray Strain or Stress analysis (shift of peak position)