Classical Texture Analysis

D. Chateigner
CRISMAT-ENSICAEN; IUT-UCBN
6 bd. M. Juin 14050 Caen

Outline

- Qualitative aspects of crystallographic textures
  - Grains, Crystallites and Crystallographic planes
  - Normal diffraction
- Effects on diffraction diagrams, their limitations
  - θ-2θ scans
  - Asymmetric scans
  - ω-scans (rocking curves)
- Representations of texture: pole figures
  - Pole Sphere
  - Stereographic projection
  - Equal-area projection: Lambert/Schmidt projection
- Pole figures
  - Localisation of crystallographic directions from pole figures
  - Direct and normalised pole figures
  - Normalisation
  - Incompleteness and corrections of pole figures
  - Single texture component
  - Multiple texture components
  - Pole figures and (hkℓ) multiplicity
  - A real example

- Pole figure types
    - Random texture
    - Planar textures
    - Fibre textures
    - Three-dimensional texture
- Pole Figures and Orientation spaces
    - Mathematical expression of diffraction pole figures and ODF
    - From pole figures to the ODF
    - Orientations g and pole figures
    - Euler angle conventions
    - From f(g) to pole figures
    - Deal with ODF in the ☝ space
    - Plotting the ODF
- Inverse pole figures
- ODF refinement
    - Generalised spherical harmonics
    - WIMV
    - Entropy modified WIMV and Entropy maximisation
    - ADC, Vector and component methods
    - ODF coverage
    - Reliability and texture strength estimators
- Why needing Combined analysis

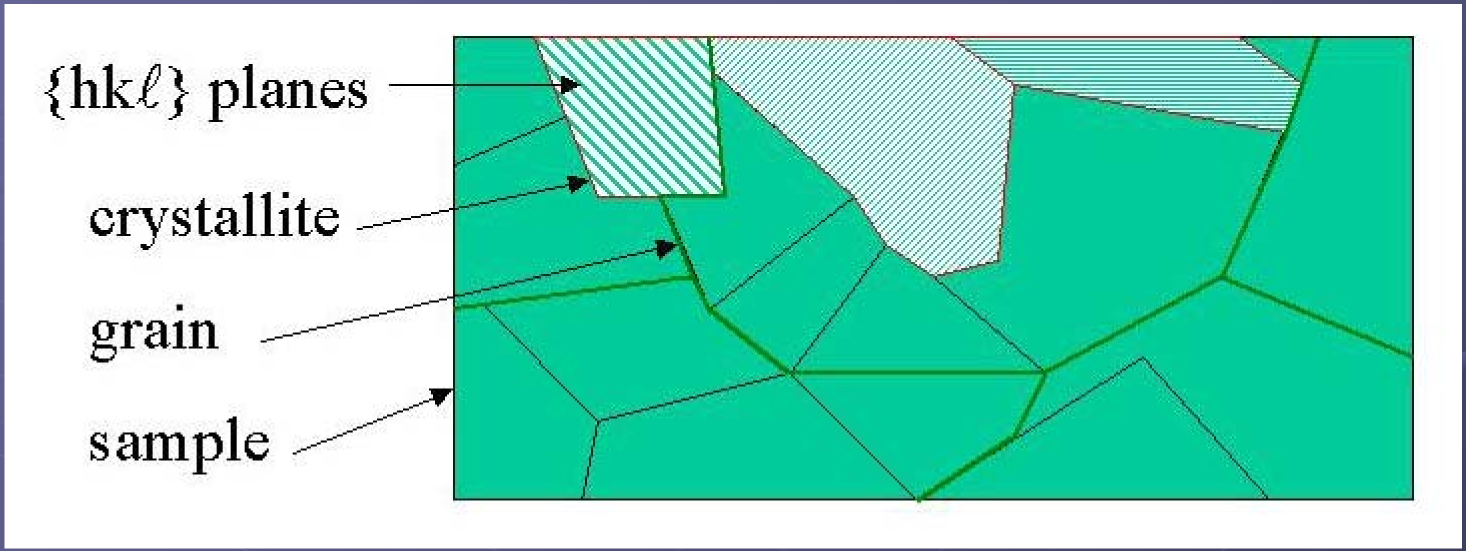
Qualitative aspects of texture

- Polycrystal: aggregate of grains, different phases, sizes, shapes, orientations …

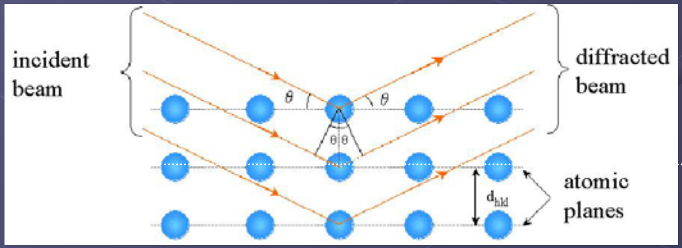
- Diffraction:
  - probes lattice planes: crystallites, not grains
  - x-rays, neutrons or electrons

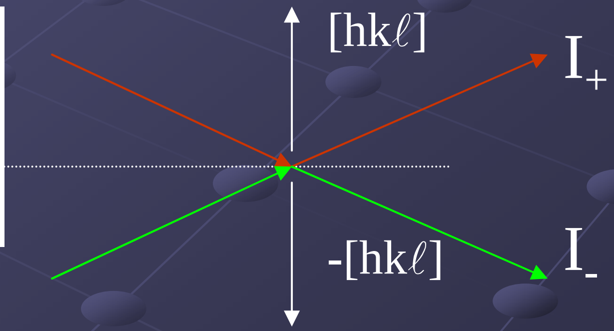
- SEM:
  - grains, not crystallites (coherent, single crystal domains)
  - shape vs crystallographic texture (EBSD)

Grains, crystallites, crystallographic planes



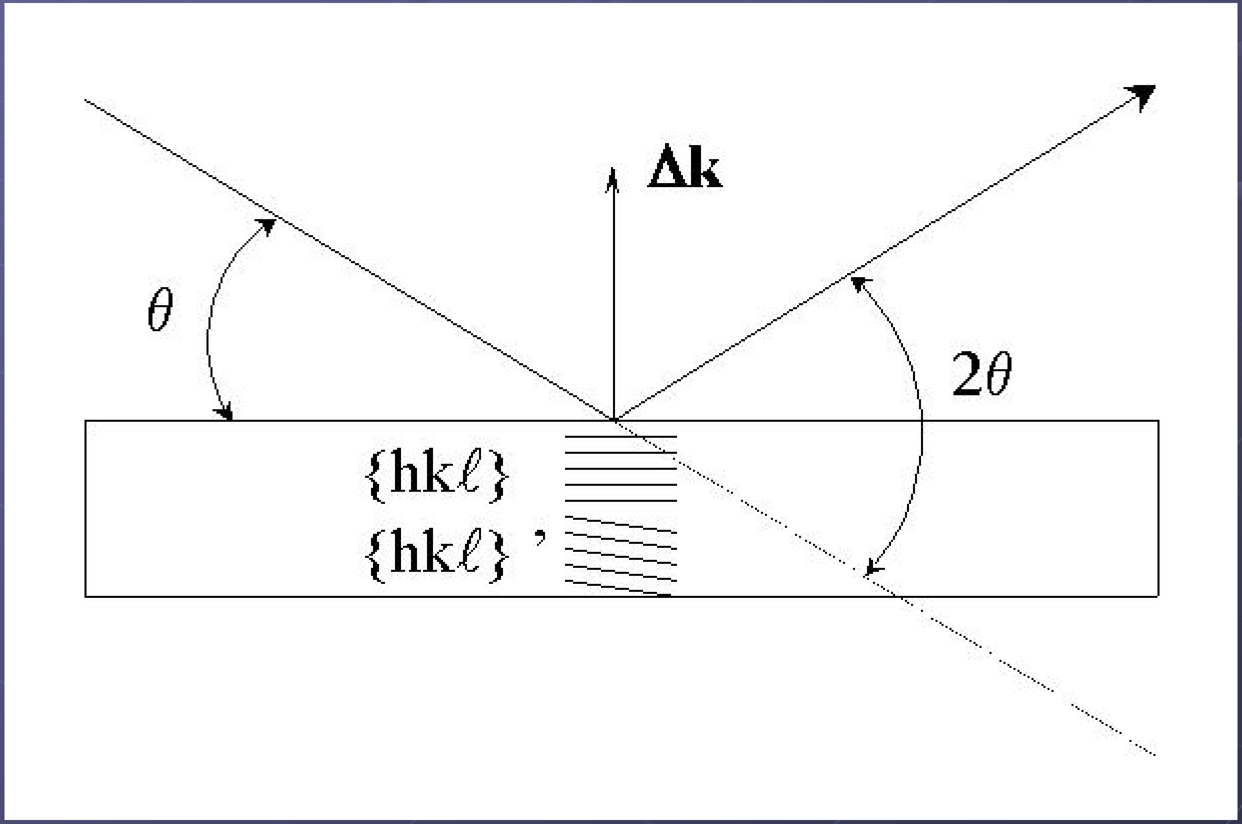
Friedel's law: $I_{hk\ell} = I_{-h-k-\ell}$ using normal diffraction
+ or - directions not distinguished

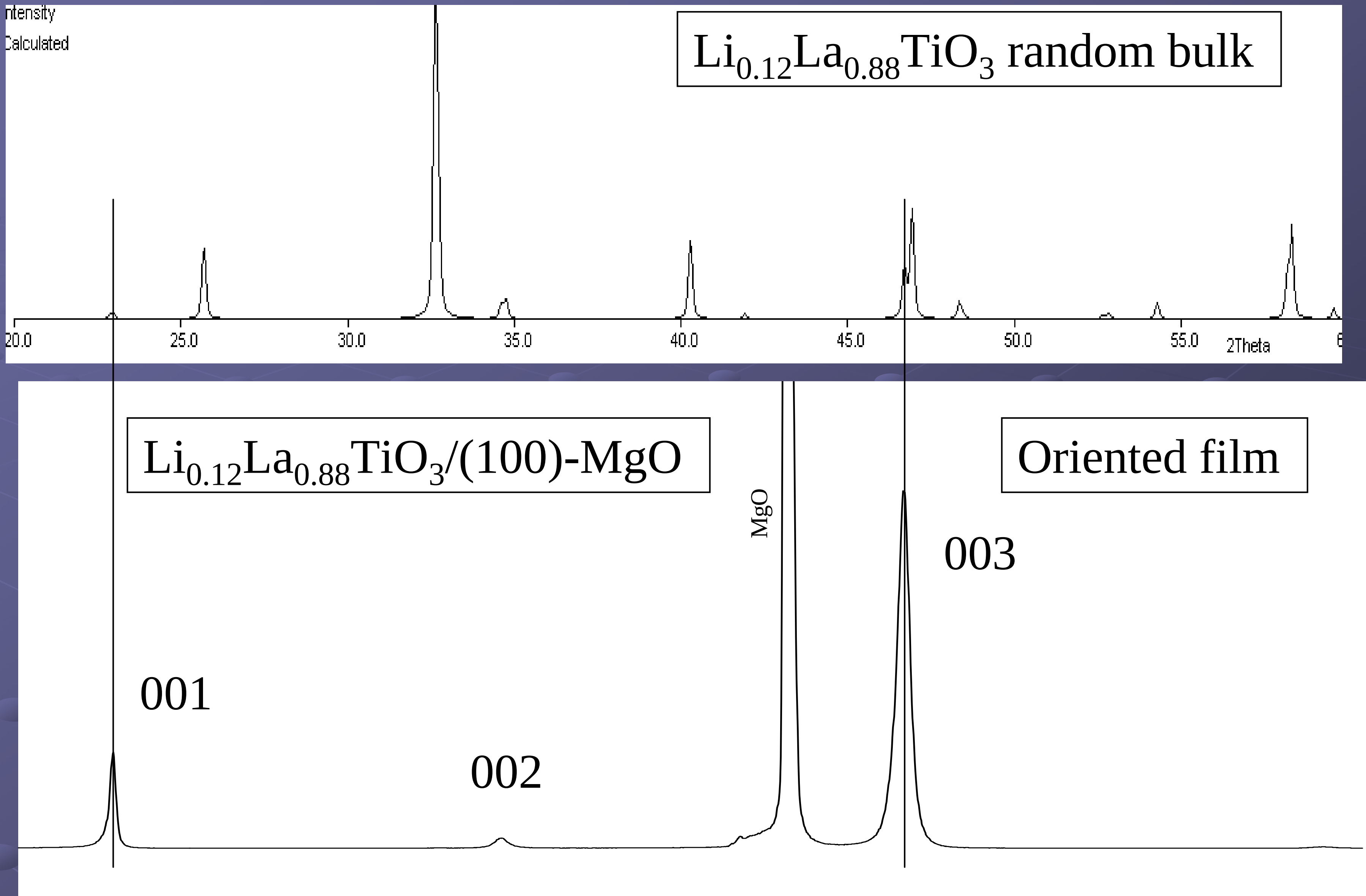




Texture effects on diffraction diagrams

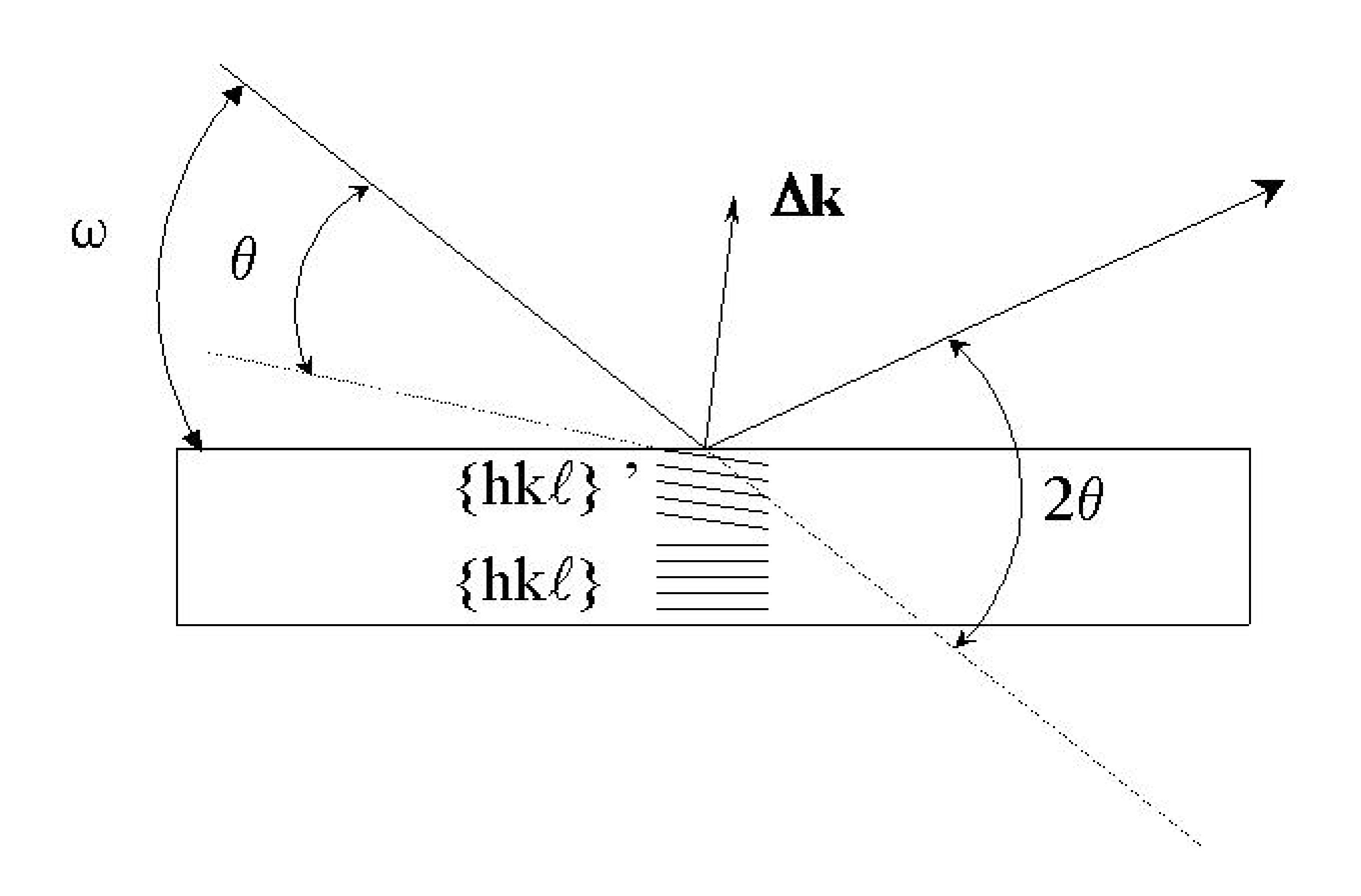
θ-2θ scan: probes only parallel planes



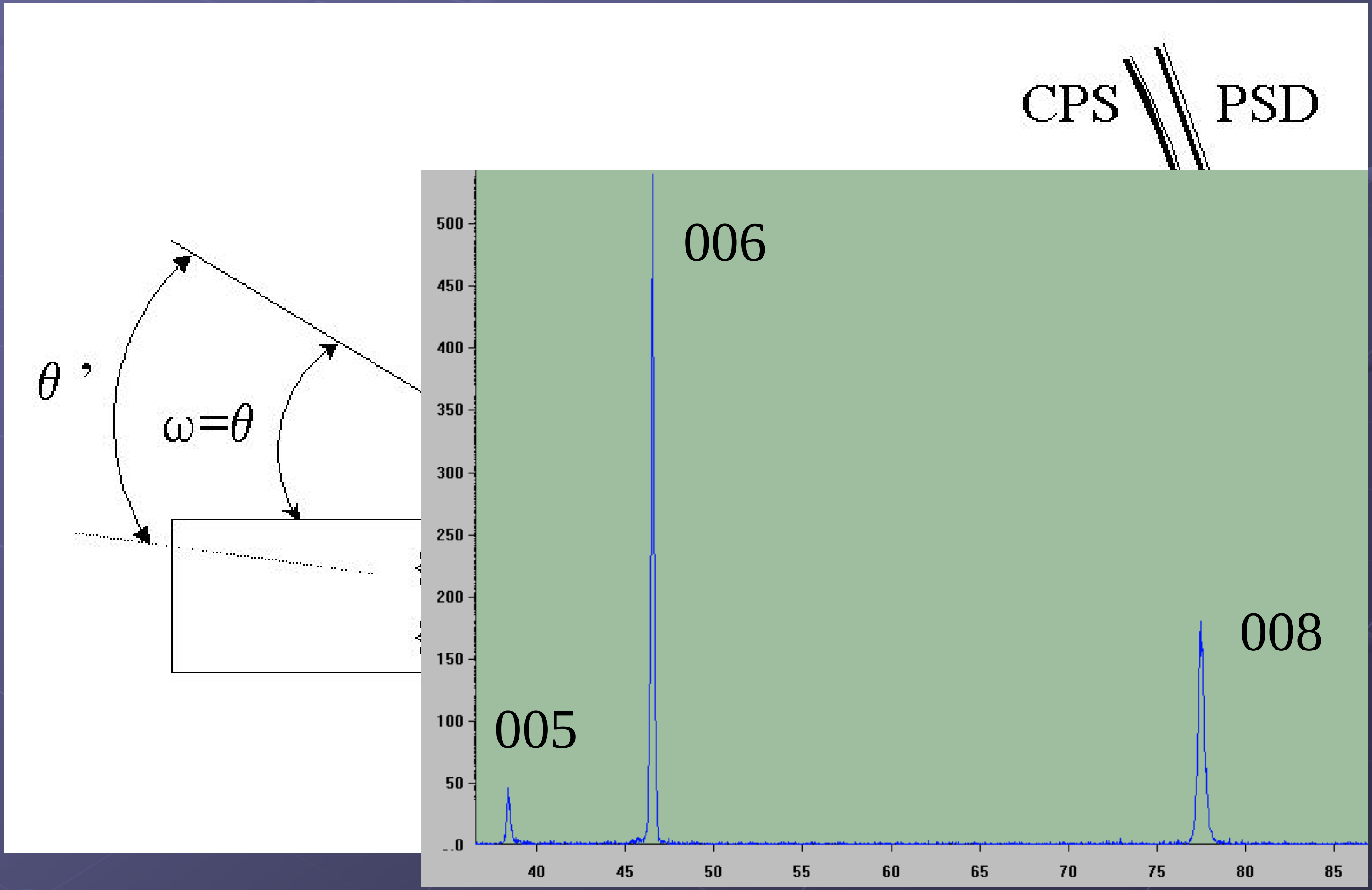


$Li_{0.12}La_{0.88}TiO_3$ random bulk
20.0
25.0
30.0
35.0
40.0
45.0
50.0
55.0
2Theta
$Li_{0.12}La_{0.88}TiO_3$/(100)-MgO
Oriented film
MgO
001
002
003

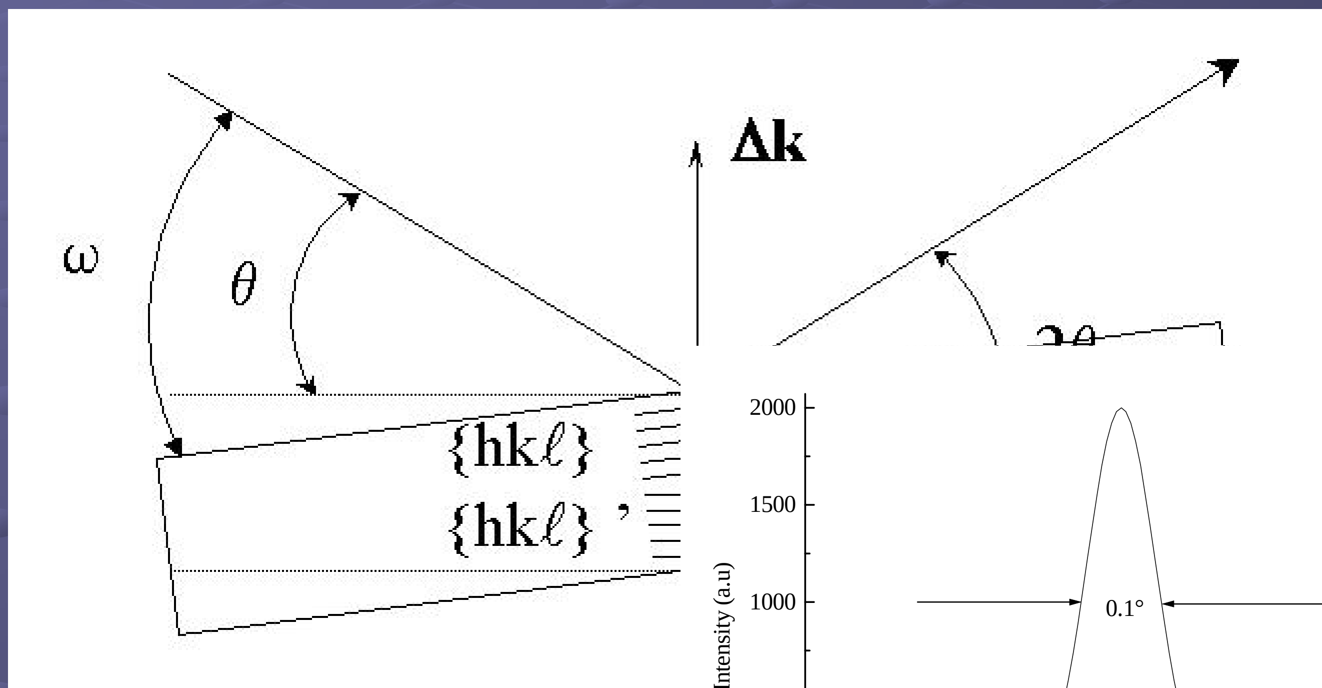
asymmetric scan: probes only inclined planes

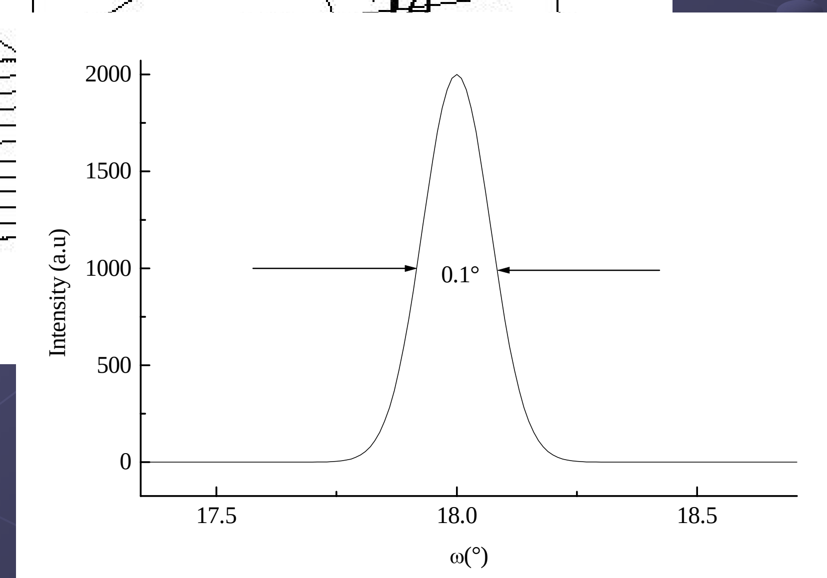


mixed scan: probes specific planes for specific orientations



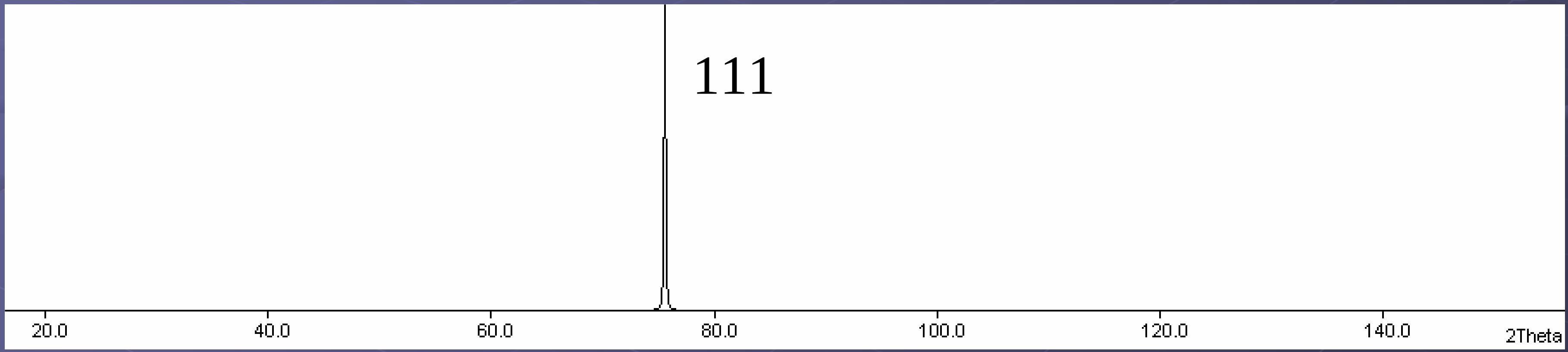
ω scan: probes orientation of only one plane type (fixed θ), only for small ω-θ





limitations: available θ (or other) range

diamond (Fd3m), 2.52 Å neutrons, up to 2θ = 150°

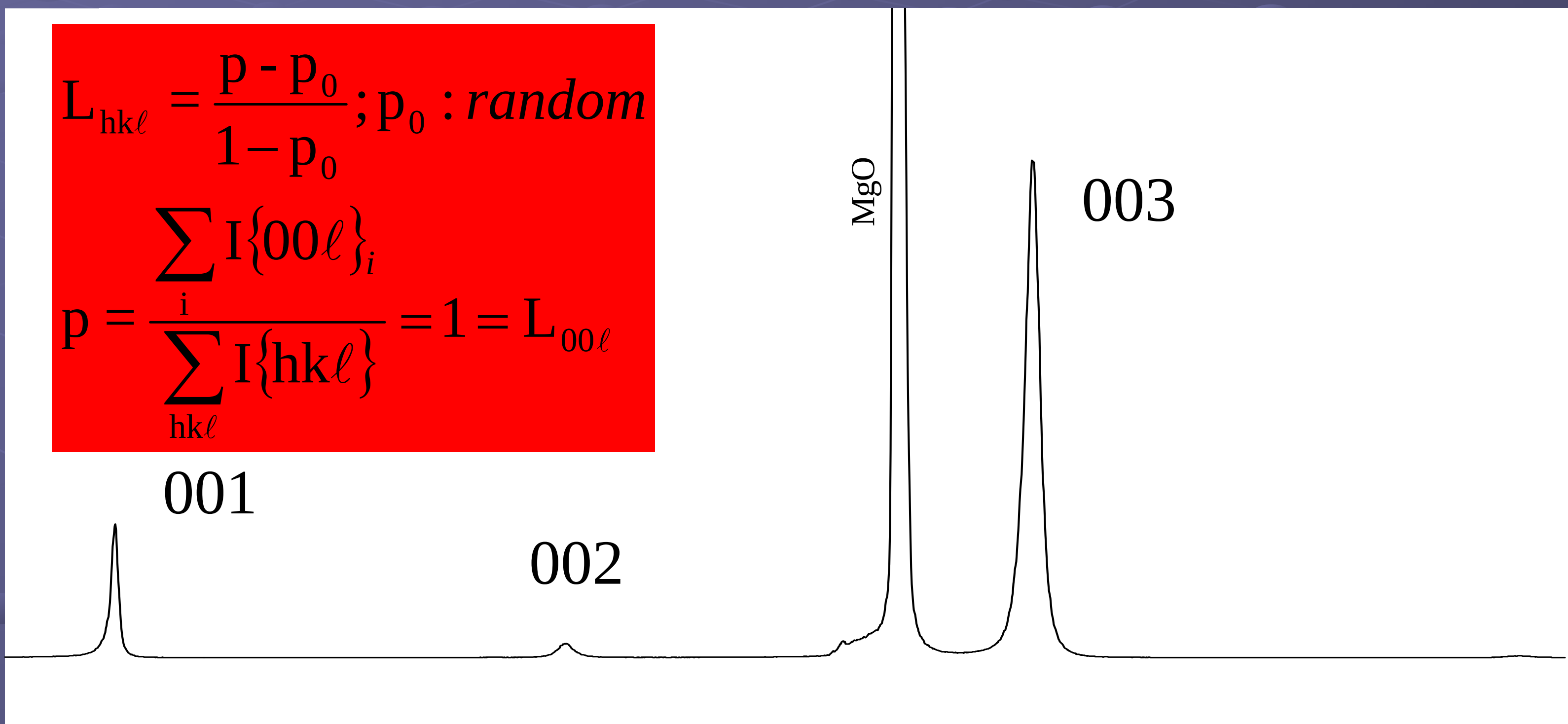


limitations: 2 texture components

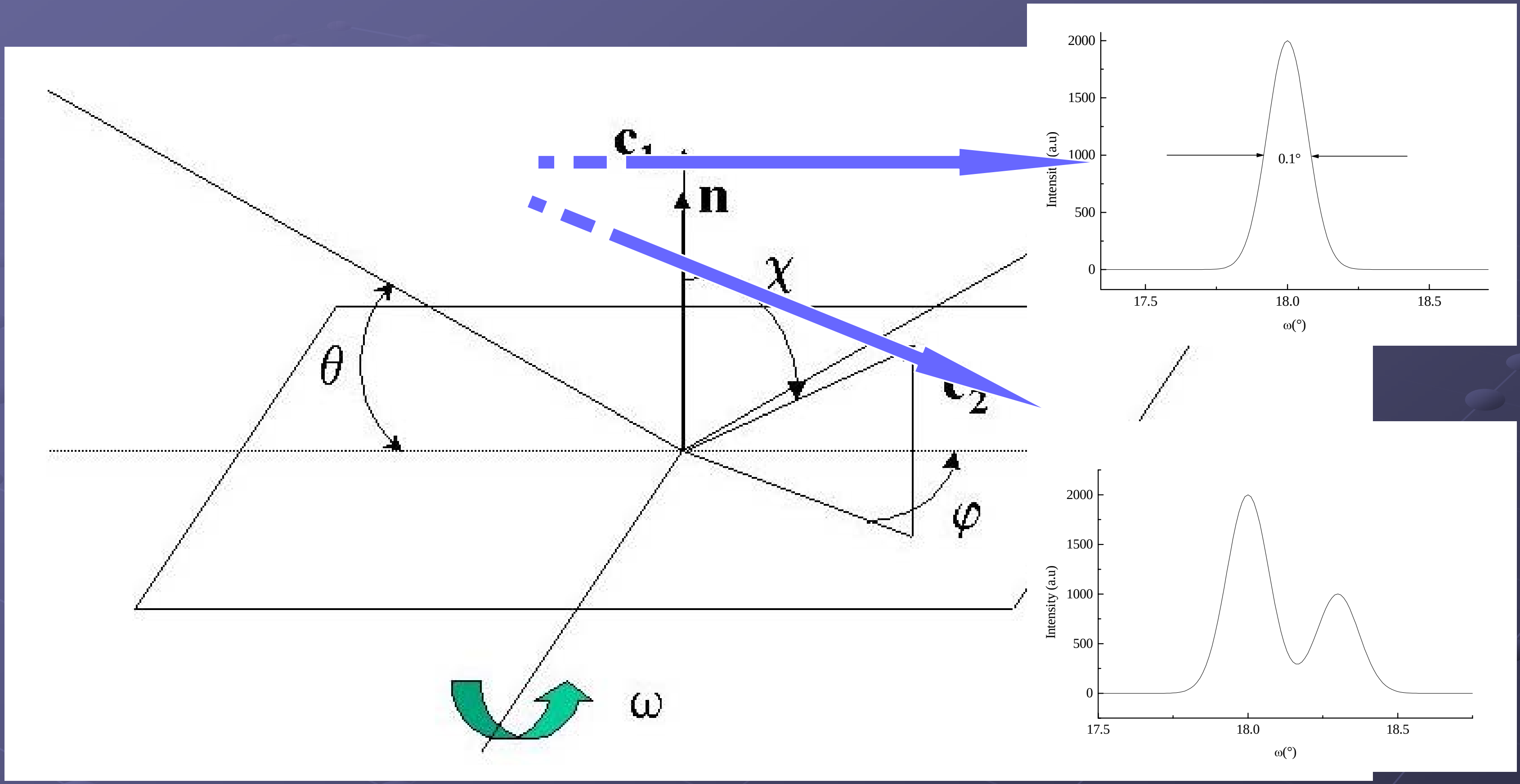
same c-axes direction, but not same a

$$L_{hk\ell} = \frac{p - p_0}{1 - p_0}; \; p_0 : random$$

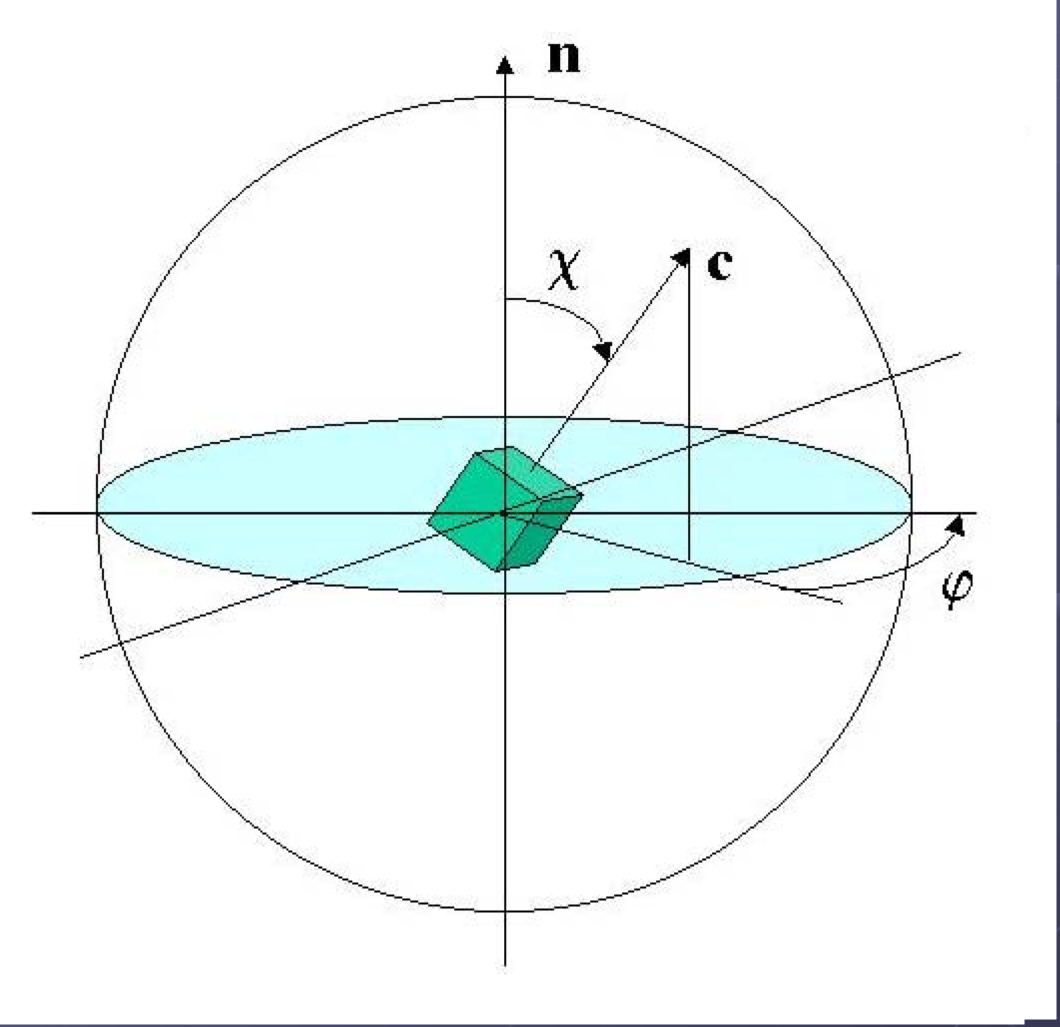
$$p = \frac{\sum_i I\{00\ell\}_i}{\sum_{hk\ell} I\{hk\ell\}} = 1 = L_{00\ell}$$



limitations: 2 texture components, one inclined



Representations of texture: pole figures

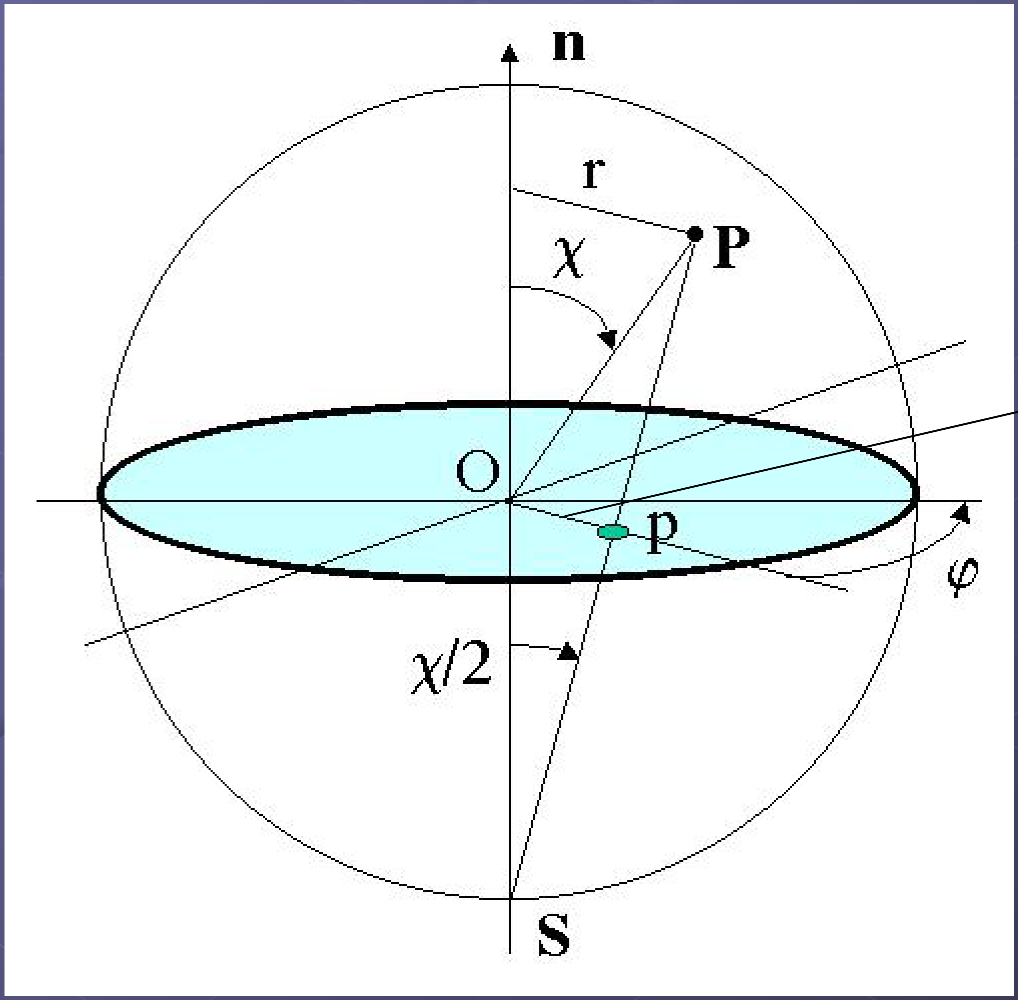


One crystallite oriented in the <u>Pole sphere:</u>

- location of all [hkℓ] ∈ unit sphere
- $dS = \sin\chi \, d\chi \, d\varphi$
- (χ,φ): angles in the <u>diffractometer space</u> ♦

Hard to visualise: needs <u>pole figures</u>

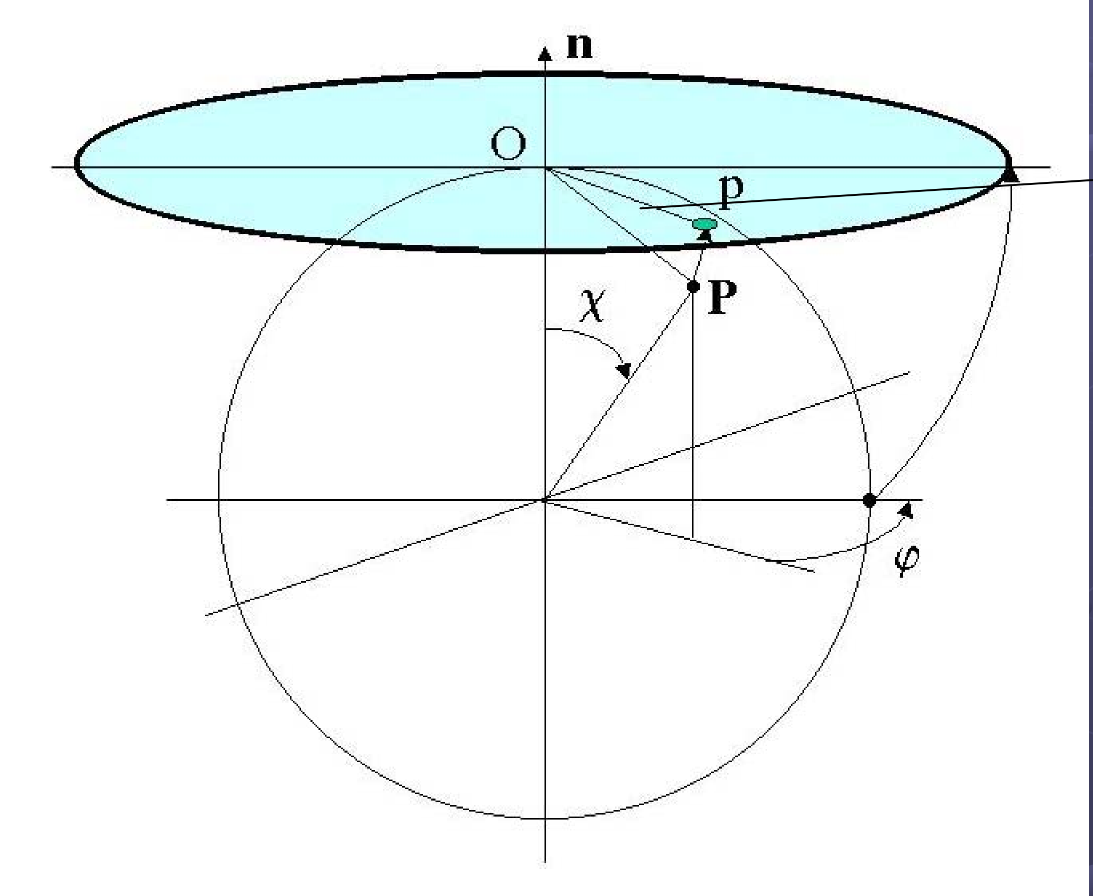
Stereographic projections: equal angle



Poles: p(r',φ):

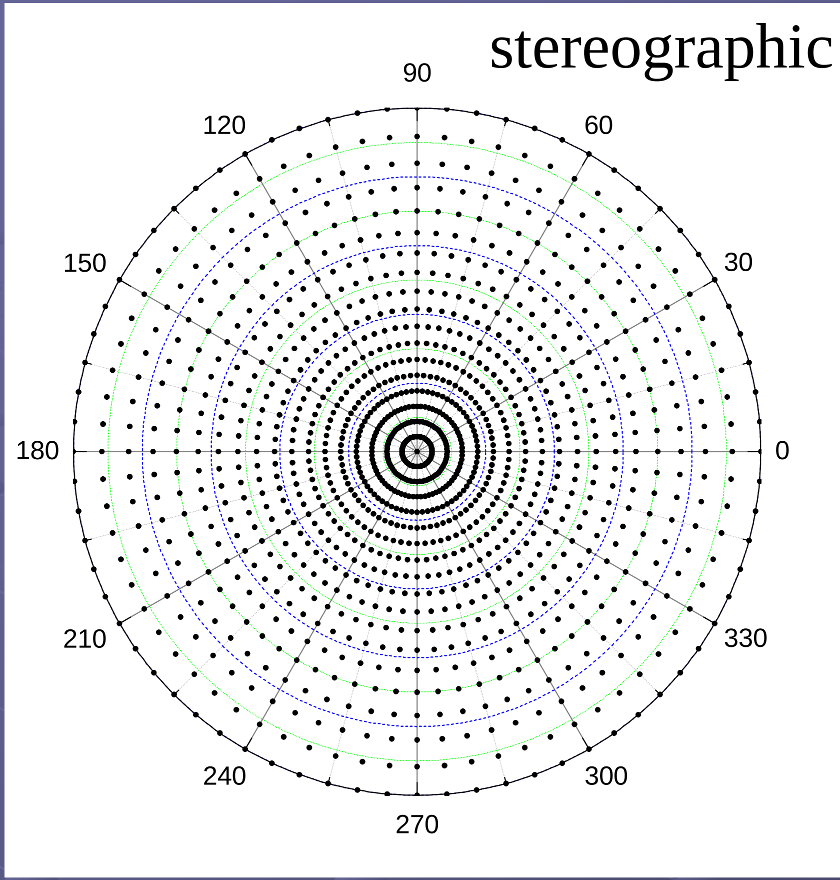
r’ = R tan(χ/2)

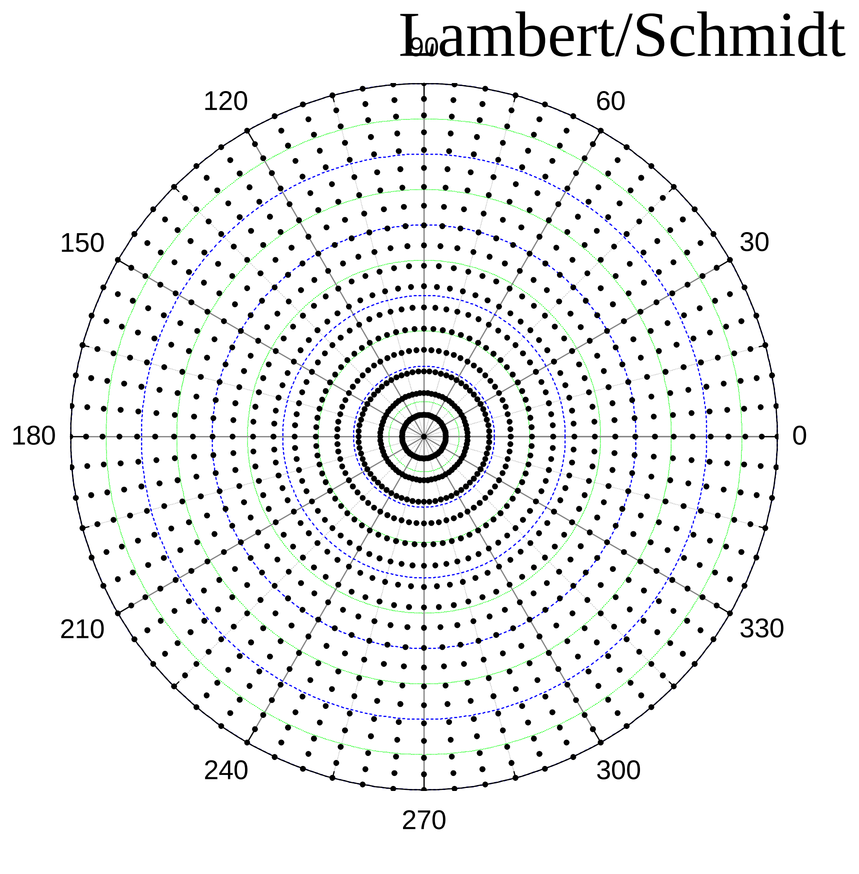
Lambert projections (equal area)



Poles: p(r',φ):

r' = 2R sin(χ/2)



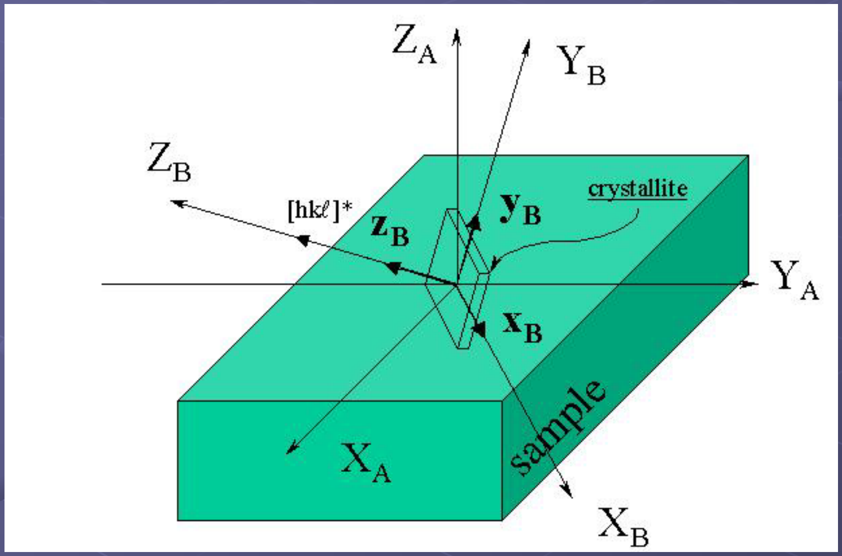


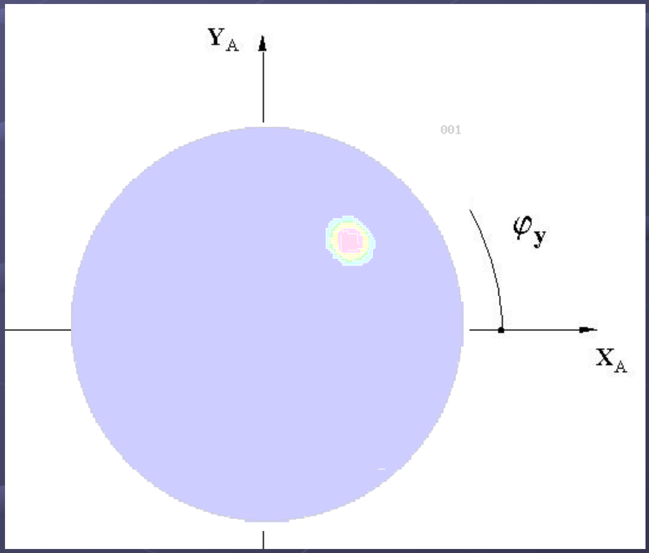
5° x 5° grid: 1368 points

Pole figures

{hkℓ}-Pole figure: location of distribution densities, for the {hkℓ} diffracting plane, defined in the crystallite frame K_B, relative to the sample frame K_A.

Pole figures space: ✡, with **y** = (ϑ_y,φ_y) = [hkℓ]*

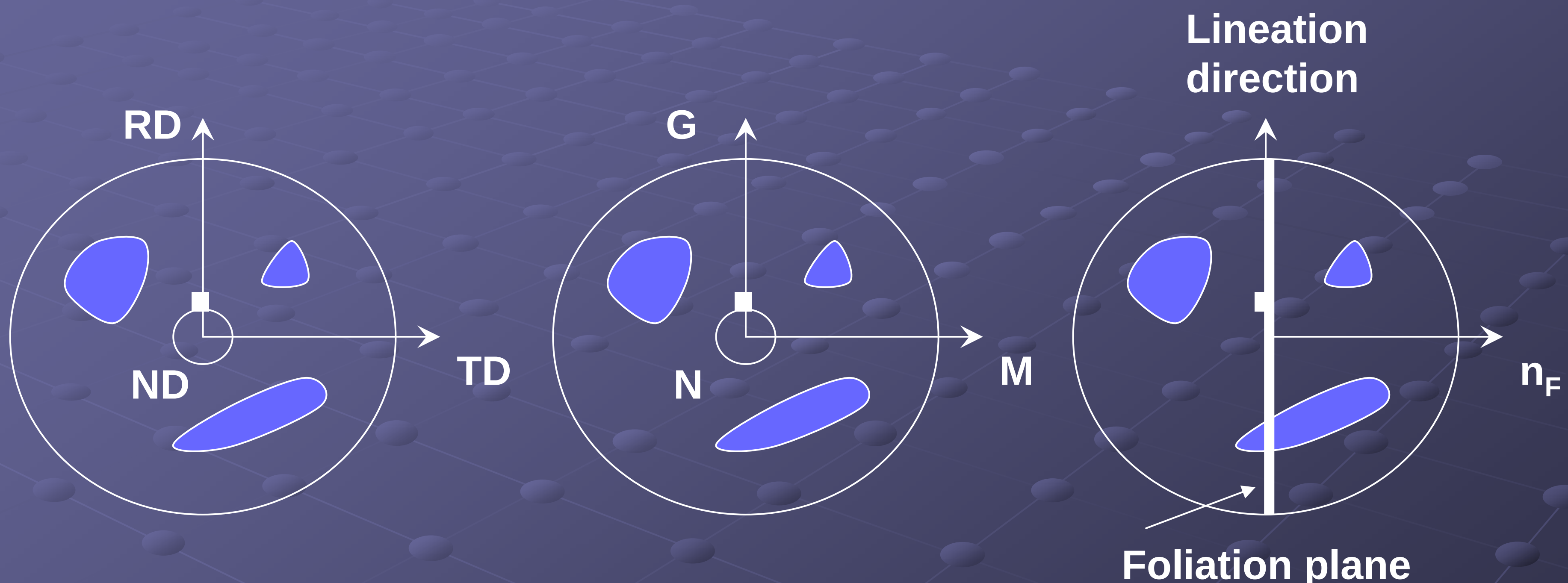




Direct Pole Figure: built on diffracted intensities $I_h(\mathbf{y})$, **h** = <hkℓ>*
Normalised Pole Figure: built on distribution densities $P_h(\mathbf{y})$

Density unit: the "multiple of a random distribution", or "m.r.d."

Usual pole figure frames K_A



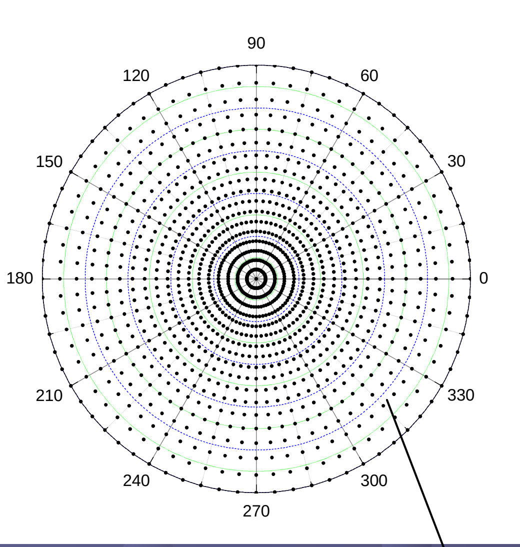
metallurgy

malacology

geophysics

Thin films: substrate directions … X_A, Y_A, Z_A

Normalisation



$I_{\mathbf{h}}(\vartheta_y, \varphi_y)$

$$I_{\mathbf{h}}^{\text{total}} = \int_{\varphi_y=0}^{2\pi} \int_{\vartheta_y=0}^{\pi/2} I_{\mathbf{h}}(\vartheta_y, \varphi_y) \sin\vartheta_y \, d\vartheta_y d\varphi_y$$

$$I_{\mathbf{h}}^{\text{random}} = I_{\mathbf{h}}^{\text{total}} / \int_{\varphi_y=0}^{2\pi} \int_{\vartheta_y=0}^{\pi/2} \sin\vartheta_y \, d\vartheta_y d\varphi_y$$

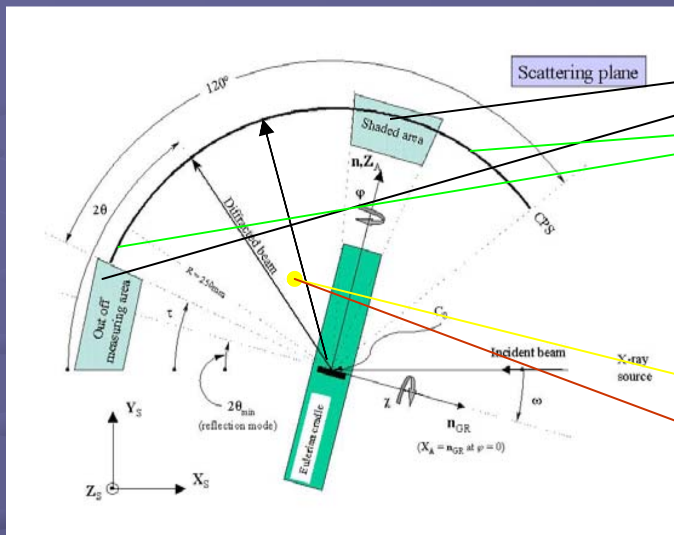
$$P_{\mathbf{h}}(\mathbf{y}) = \frac{I_{\mathbf{h}}(\mathbf{y})}{I_{\mathbf{h}}^{\text{random}}}$$

- Only valid for complete pole figures:

neutrons in symmetric geometry

- Needs a refinement strategy to get I^{random} for all **h**'s

Incompleteness and corrections of pole figures

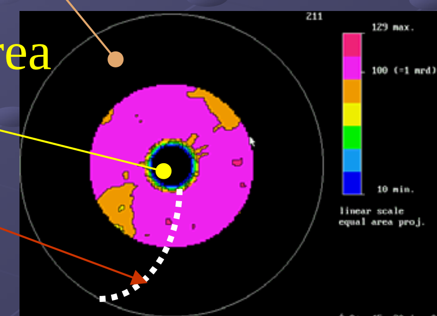


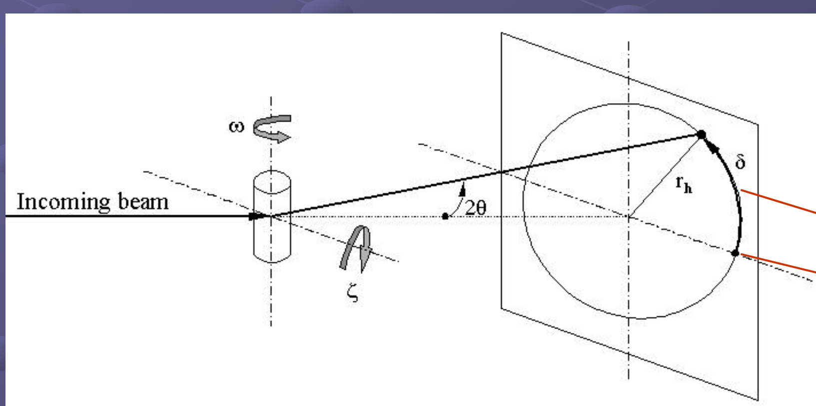
Missing Bragg peaks

Absorption + volume

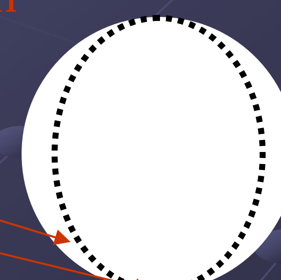
Defocusing (x-rays)

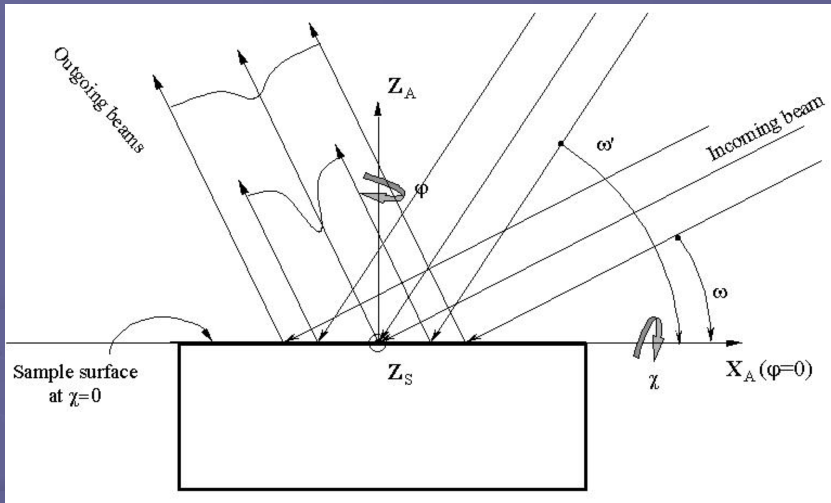
Blind area

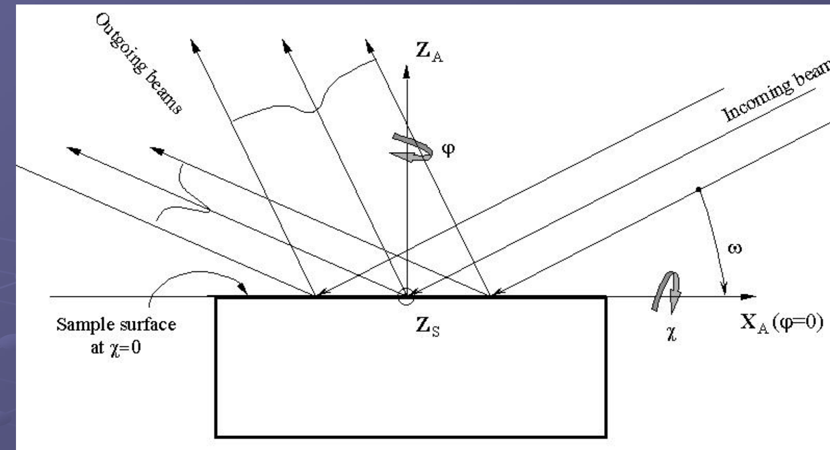




Localisation



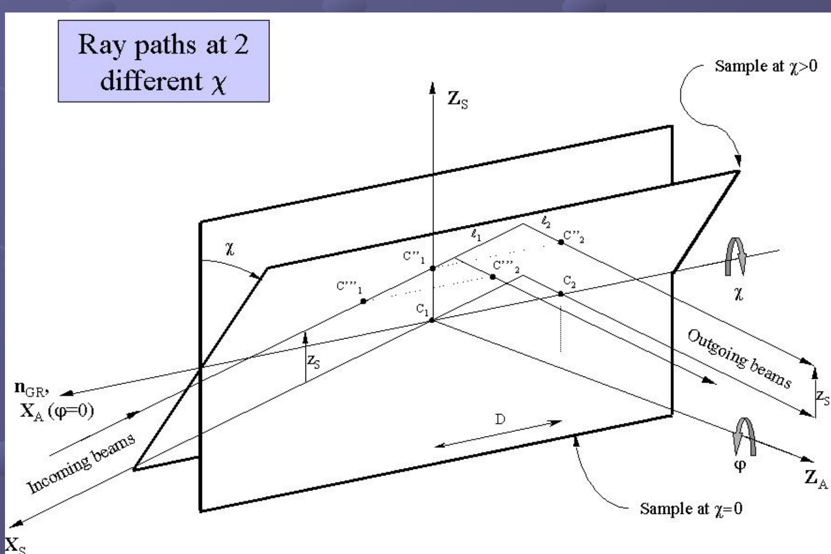


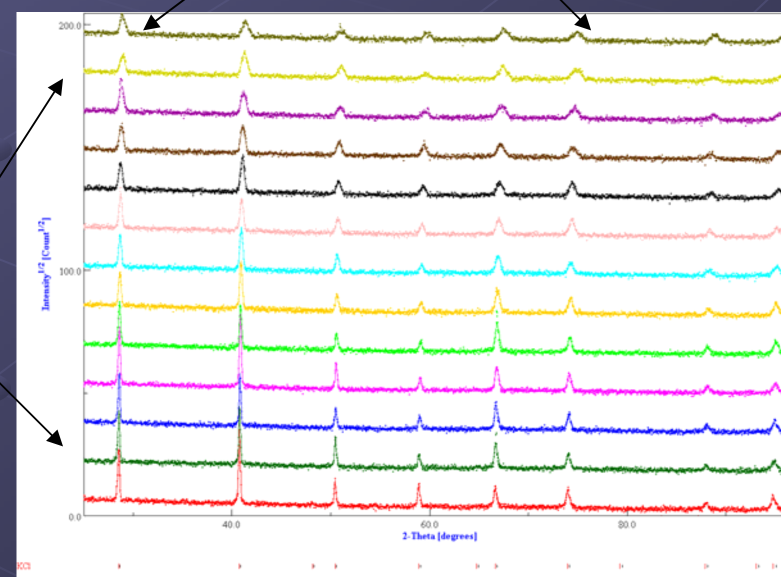


ω-defocusing

2θ-defocusing

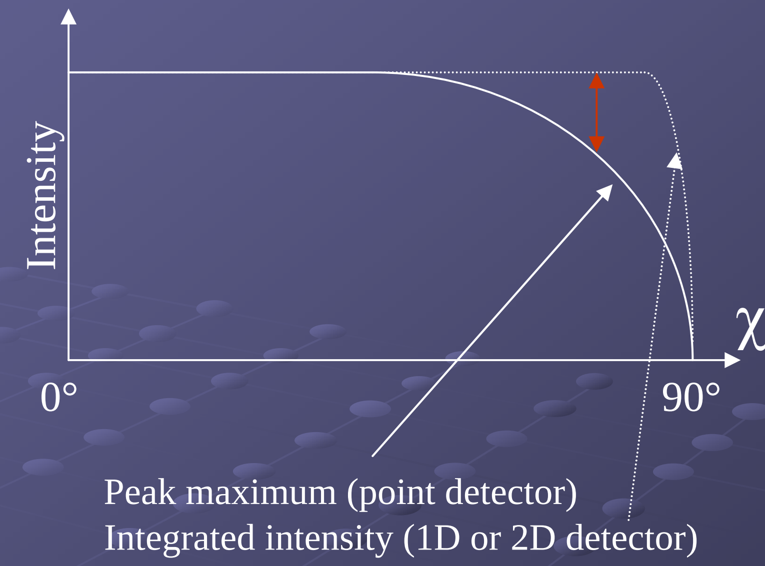
χ-defocusing





Defocusing corrections:

- Calibration on a random powder

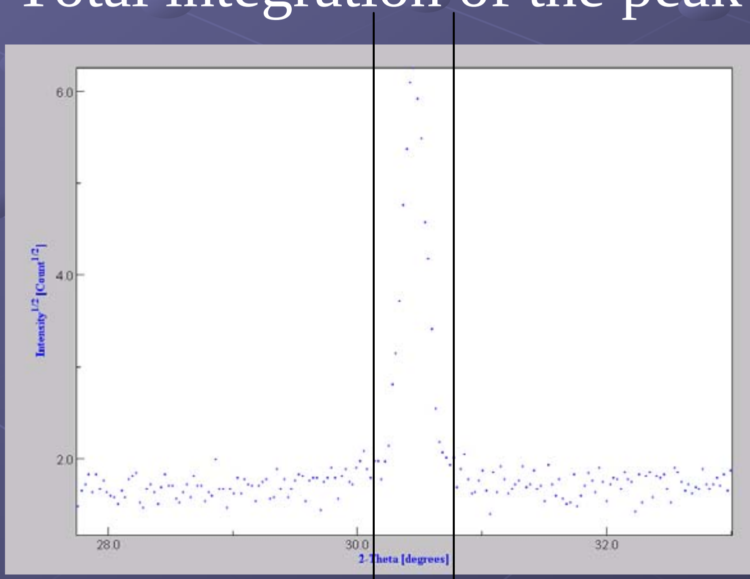


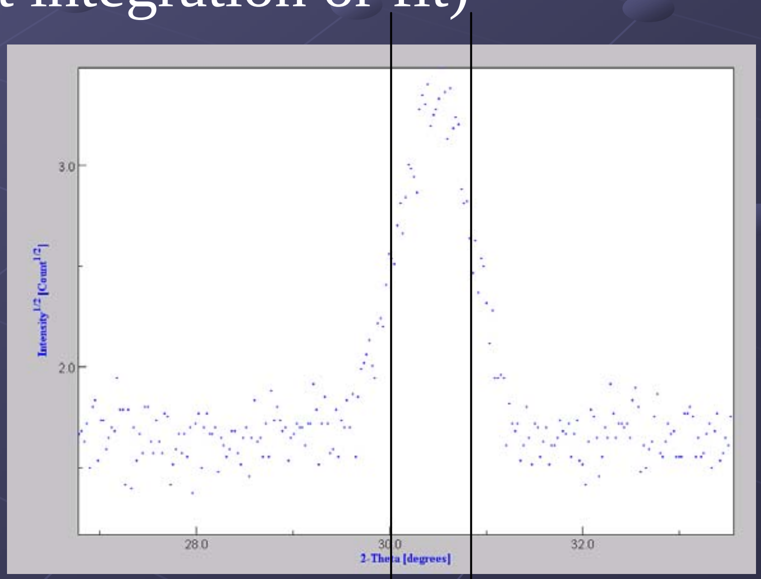
$$I^{cor}_{\chi,\omega,\theta} = I^{meas}_{\chi,\omega,\theta}\frac{I^{rand}_{0,\omega,\theta}}{I^{rand}_{\chi,\omega,\theta}}$$

Net intensities (point detector)

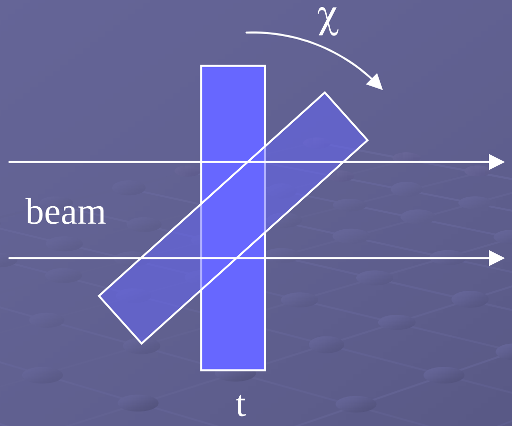
$$= \left[I^{meas}_{\chi,\omega,\theta} - I^{bkg}_{0,\omega,\theta}\frac{I^{bkg}_{\chi,\omega,\theta}}{I^{bkg}_{0,\omega,\theta}}\right]\frac{I^{rand}_{0,\omega,\theta} - I^{bkg}_{0,\omega,\theta}}{I^{rand}_{\chi,\omega,\theta} - I^{bkg}_{0,\omega,\theta}}$$

- Total integration of the peak (direct integration or fit)

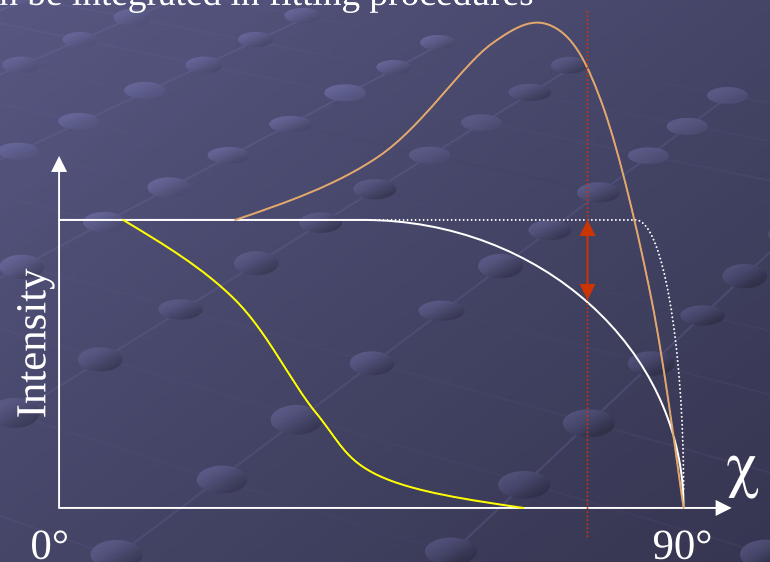




Absorption/Volume corrections:



Specific to each instrumental geometry
Sample dependent (films, multilayers …)
Modifies the defocusing curves
Can be integrated in fitting procedures



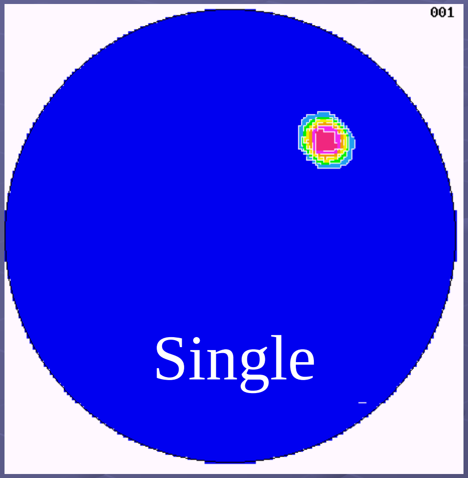
Top film

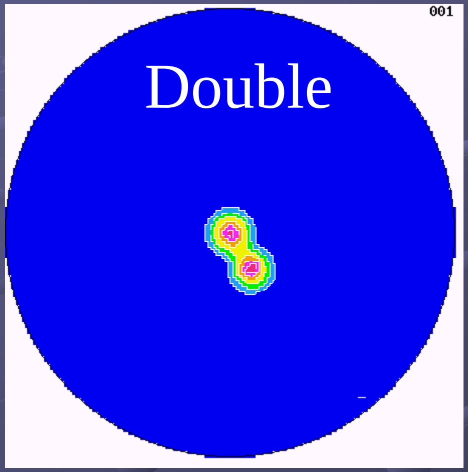
$$\mathrm{I}(0) = \mathrm{I}(\chi)\frac{\left(1-\exp\left(-2\mu T/\sin\theta_i\right)\right)}{\left(1-\exp\left(-2\mu T/\sin\theta_i\cos\chi\right)\right)}$$

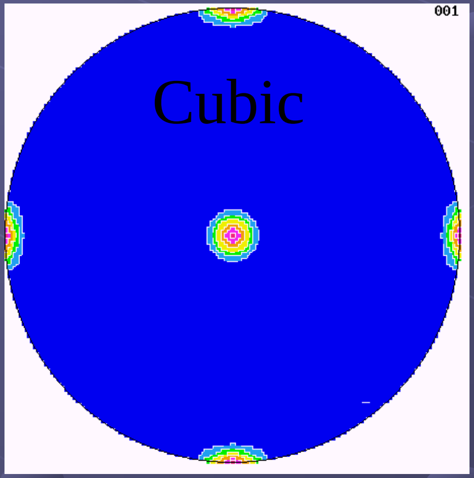
Covered layer

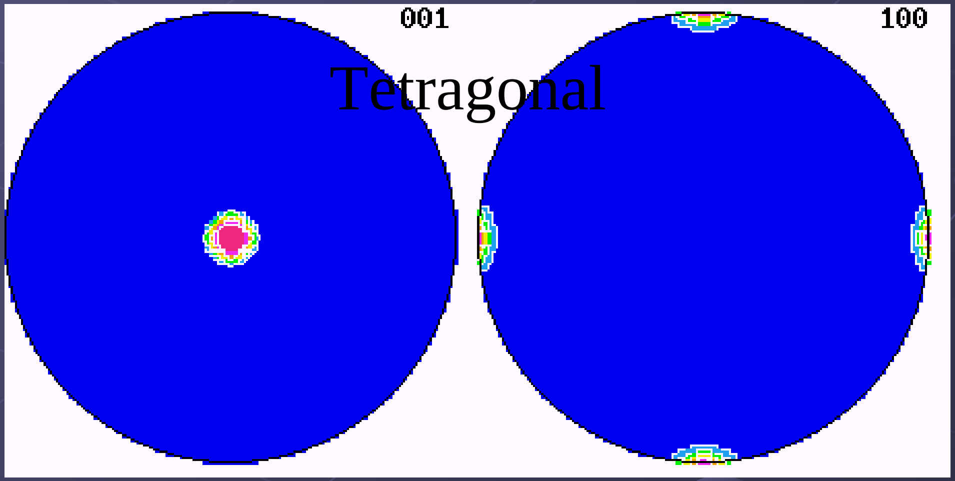
$$\mathrm{I}(0) = \mathrm{I}(\chi)\frac{\left(1-\exp\left(-2\mu T/\sin\theta_i\right)\right)\exp\left(\frac{-2\sum_j \mu_j T_j}{\sin\theta_i}\right)}{\left(1-\exp\left(-2\mu T/\sin\theta_i\cos\chi\right)\right)\exp\left(\frac{-2\sum_j \mu_j T_j}{\sin\theta_i\cos\chi}\right)}$$

Single or multiple texture components, multiplicity

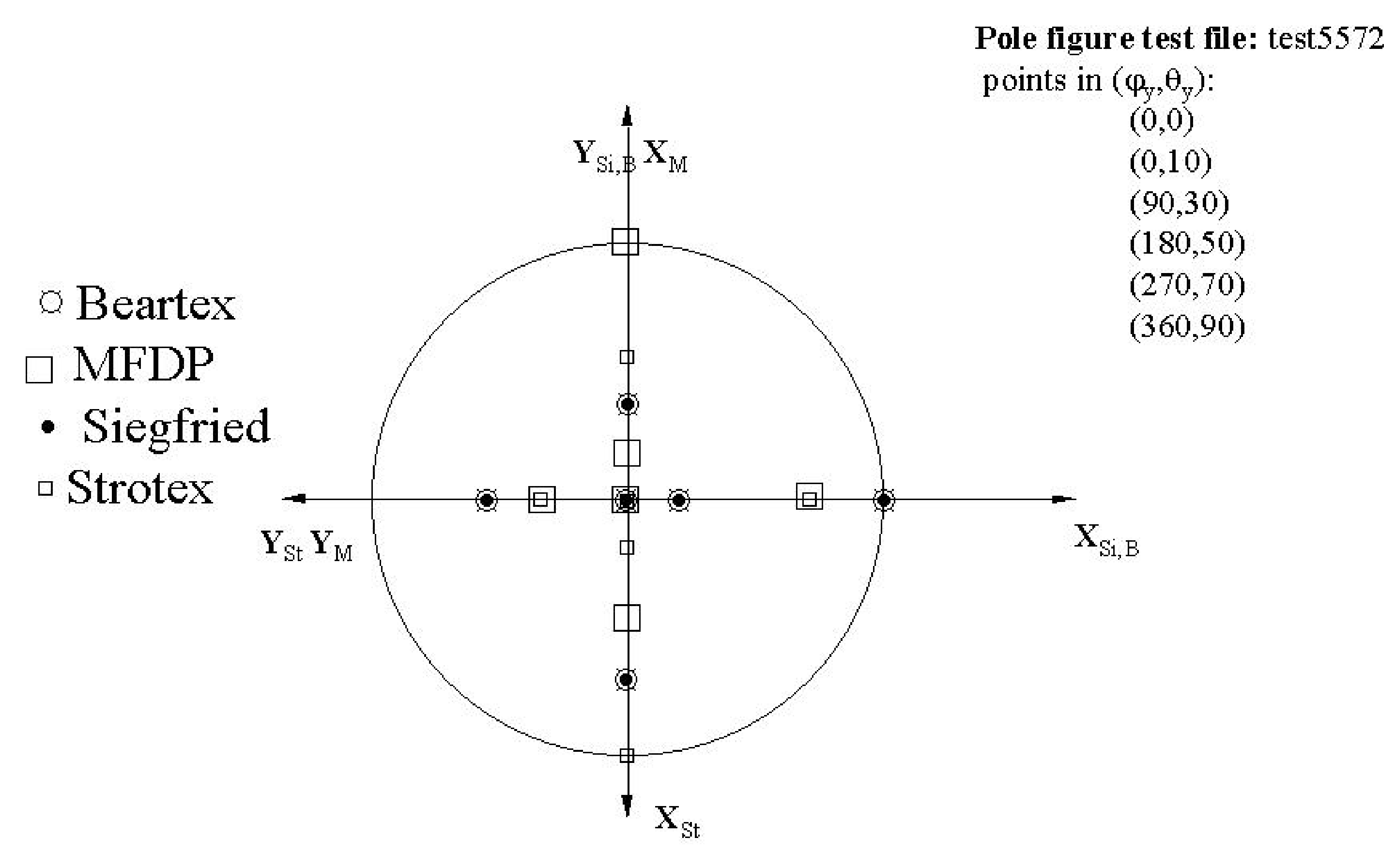






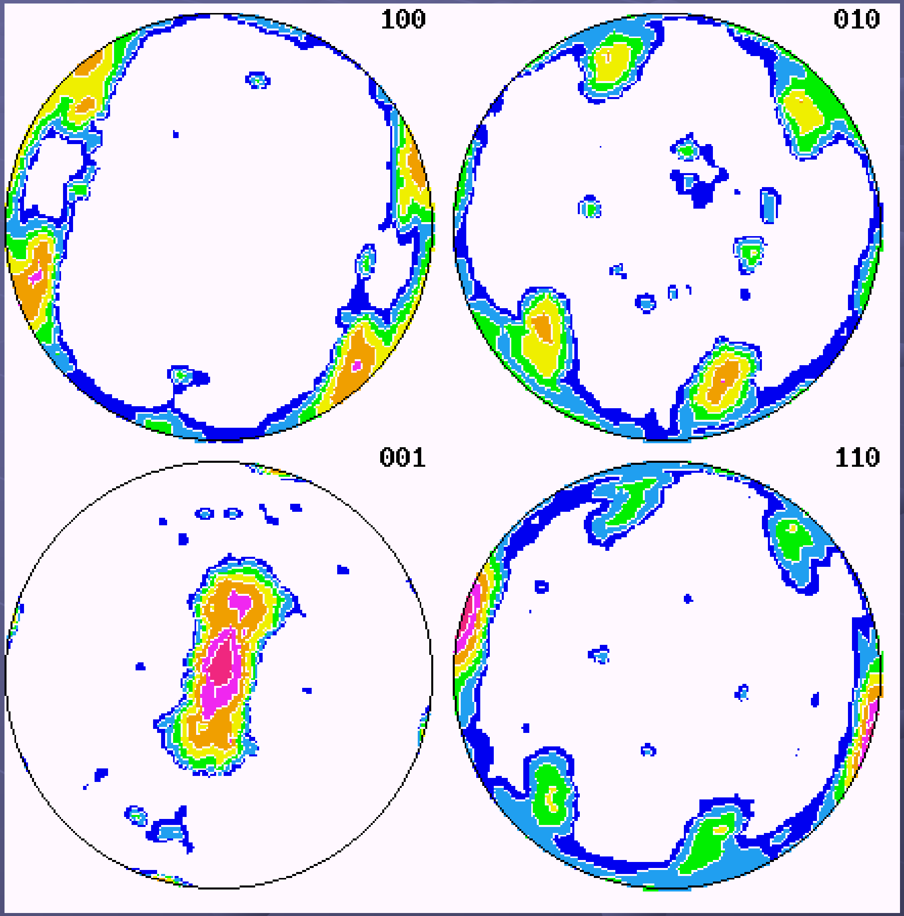


Program convention !



Pole figure plot programs correspondences

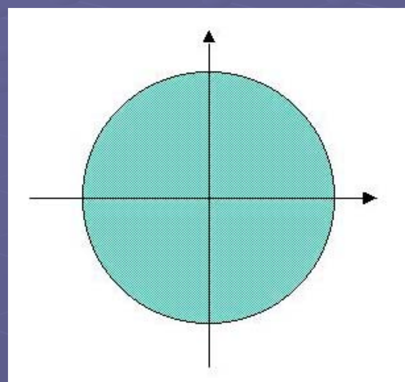
A real example



Cypraea testudinaria

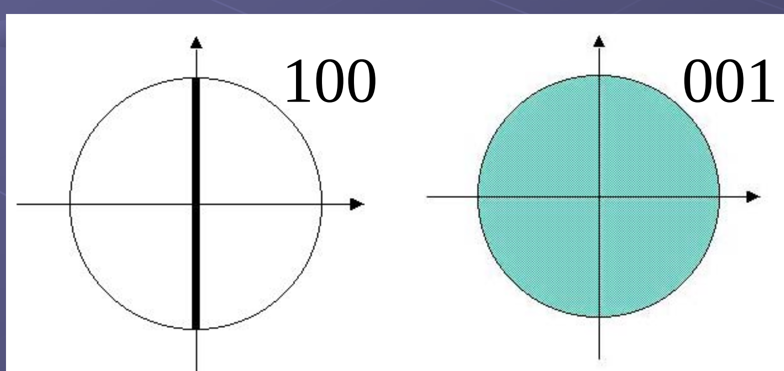
Outer aragonite layer
Pnma space group

Texture types



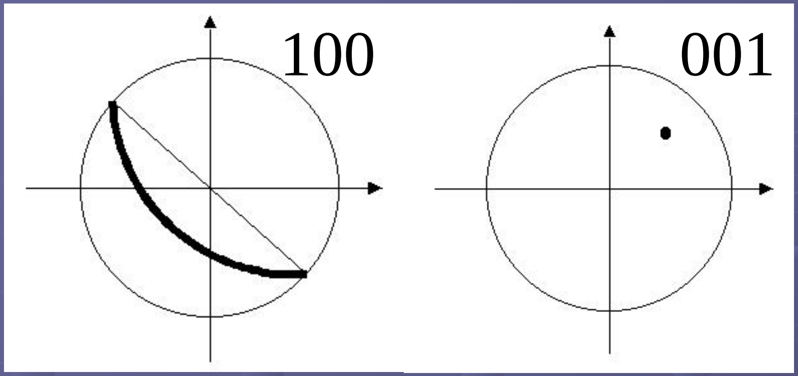
Random texture

3 degree of freedom
All $P_h(\mathbf{y})$ homogeneous
1 m.r.d. density whatever $\mathbf{y}$



Planar texture

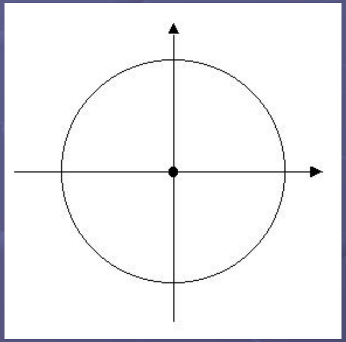
2 degree of freedom
1 [hkℓ] at random in a plane



Fibre texture

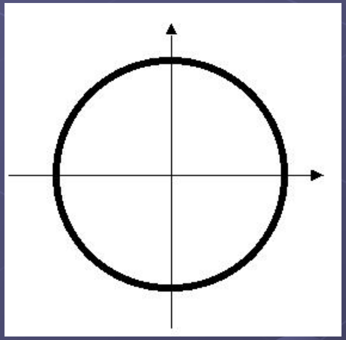
1 degree of freedom

1 [hkℓ] along 1 **y** direction



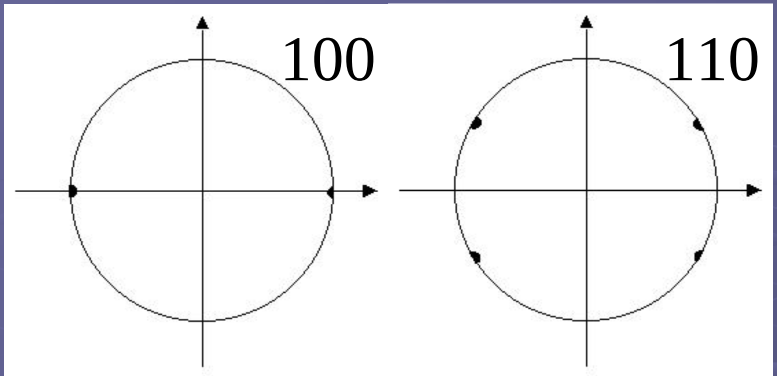
Cyclic-Fibre texture

c // Z_A



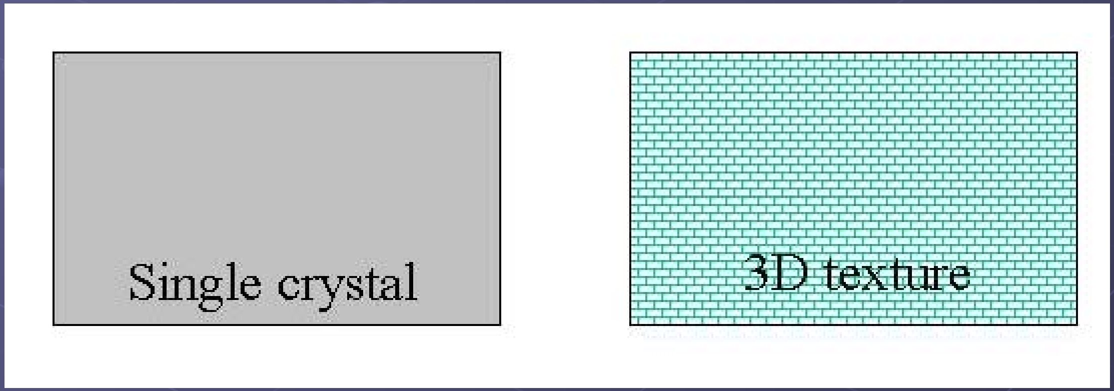
Cyclic-Planar texture

(**a**,**b**) // (X_A,Y_A)



Single crystal-like texture

0 degree of freedom
2 [hkℓ]'s along 2 **y** directions



Single-crystal and perfect 3D orientation not distinguished

Pole figure and Orientation spaces

Pole figure expression:

$$\frac{dV(\mathbf{y})}{V} = \frac{1}{4\pi} P_{\mathbf{h}}(\mathbf{y})\, \mathbf{dy}$$

$\mathbf{dy} = \sin\vartheta_y \, d\vartheta_y \, d\varphi_y$

$$\int_{\varphi_y=0}^{2\pi} \int_{\vartheta_y=0}^{\pi/2} P_{\mathbf{h}}(\vartheta_y, \varphi_y) \sin\vartheta_y \, d\vartheta_y d\varphi_y = 4\pi$$

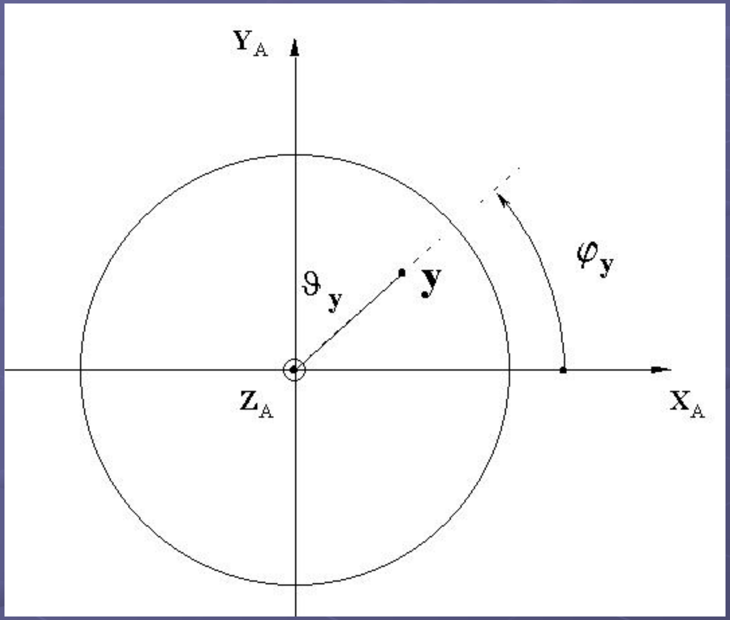
Orientation Distribution Function f(g):

$$\frac{dV(g)}{V} = \frac{1}{8\pi^2} f(g)\, dg$$

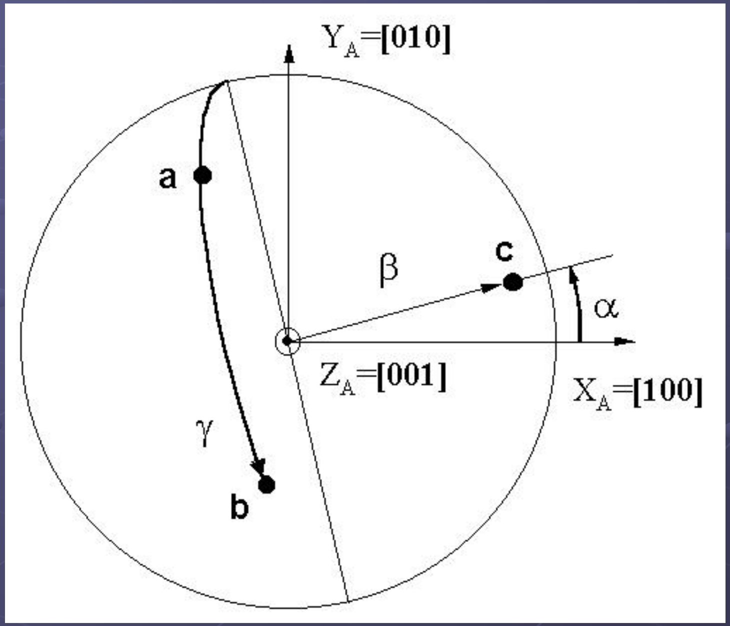
$dg = \sin(\beta) d\beta d\alpha d\gamma$

$$\int_{\alpha=0}^{2\pi} \int_{\beta=0}^{\pi/2} \int_{\gamma=0}^{2\pi} f(g)\, dg = 8\pi^2$$

From Pole figures to the ODF



Pole figure: one direction fixed in K_A



Orientation: two directions fixed in K_A

Fundamental Equation of QTA

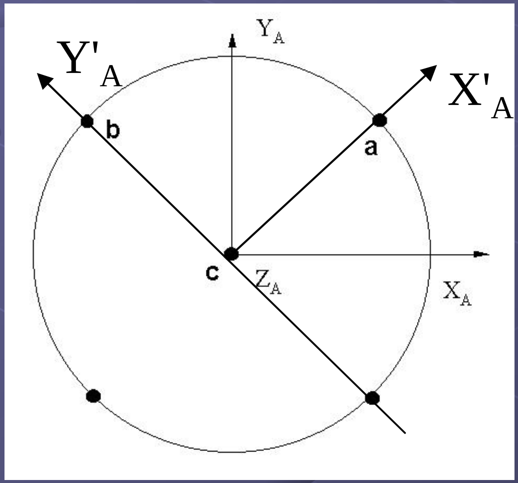
$$P_{\mathbf{h}}(\mathbf{y}) = \frac{1}{2\pi} \int_{\mathbf{h}//\mathbf{y}} f(g)\, d\tilde{\varphi}$$

Needs several pole figures to construct the f(g)

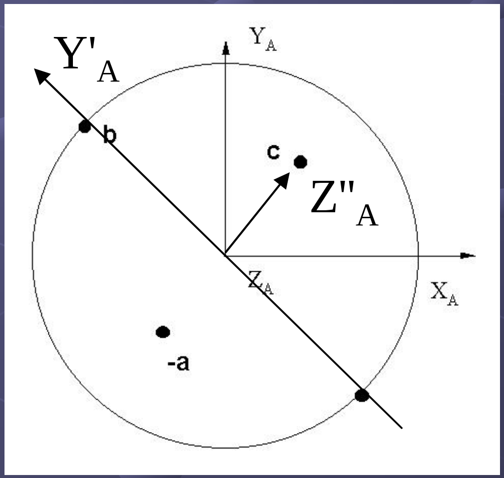
Pole figures from g

- Rotation of K_A about the axis Z_A through the angle α:
  $[K_A \mapsto K'_A]$; associated rotation $g_1 = \{\alpha,0,0\}$
- Rotation of K'_A about the axis Y'_A through the angle β:
  $[K'_A \mapsto K''_A]$; associated rotation $g_2 = \{0,\beta,0\}$
- Rotation of K''_A about the axis Z''_A through the angle γ:
  $[K''_A \mapsto K'''_A // K_B]$; associated rotation $g_3 = \{0,0,\gamma\}$

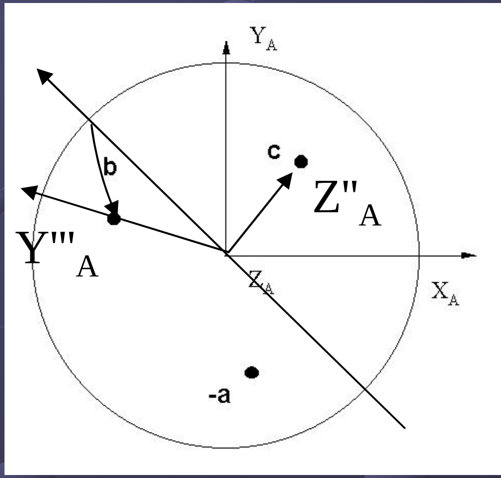
finally: $g = g_1\, g_2\, g_3 = \{\alpha,0,0\}\,\{0,\beta,0\}\,\{0,0,\gamma\} = \{\alpha,\beta,\gamma\}$



$g_1 = \{45,0,0\}$



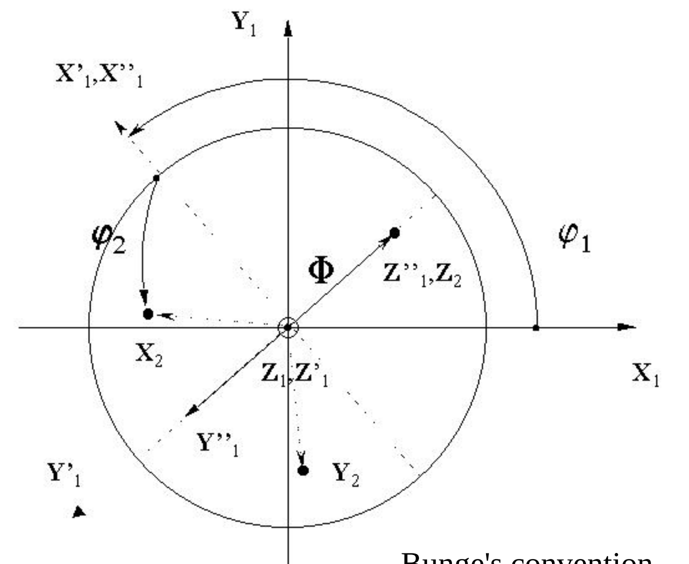
$g_2 = \{45,45,0\}$



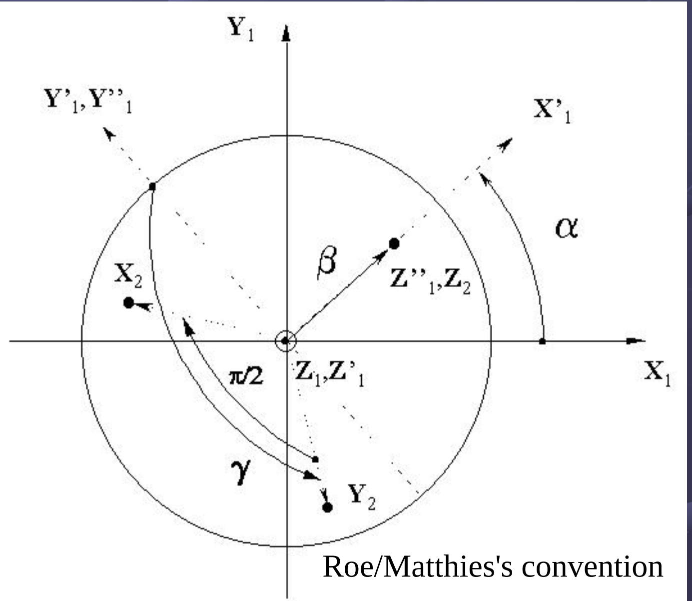
$g_3 = \{45,55,45\}$

Euler angles conventions

Matthies	Roe	Bunge	Canova	Kocks
α	Ψ	$\varphi_1 = \alpha + \pi/2$	$\omega = \pi/2 - \alpha$	Ψ
β	Θ	Φ	Θ	Θ
γ	Φ	$\varphi_2 = \gamma + 3\pi/2$	$\phi = 3\pi/2 - \gamma$	$\Phi = \pi - \gamma$

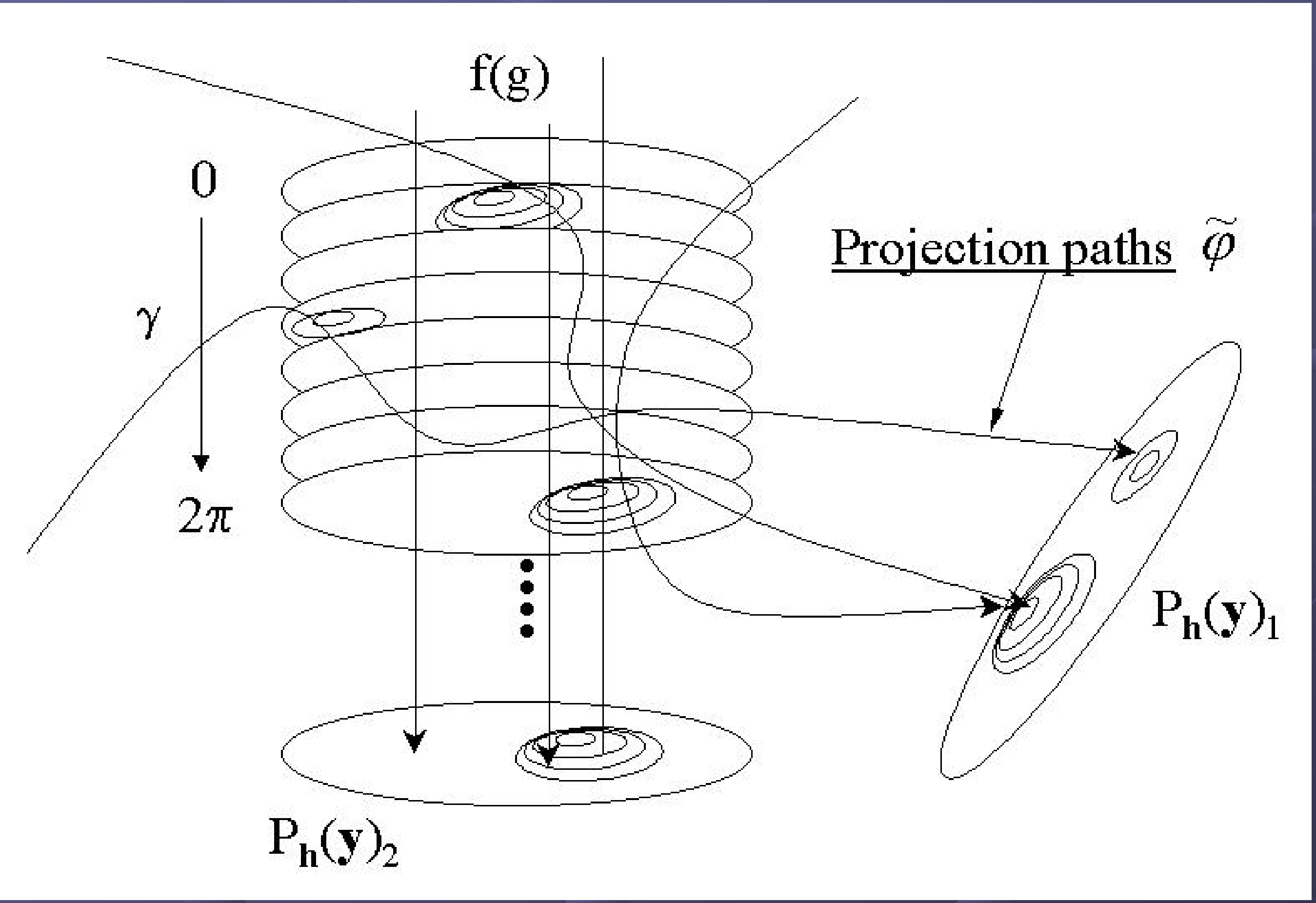


Bunge's convention

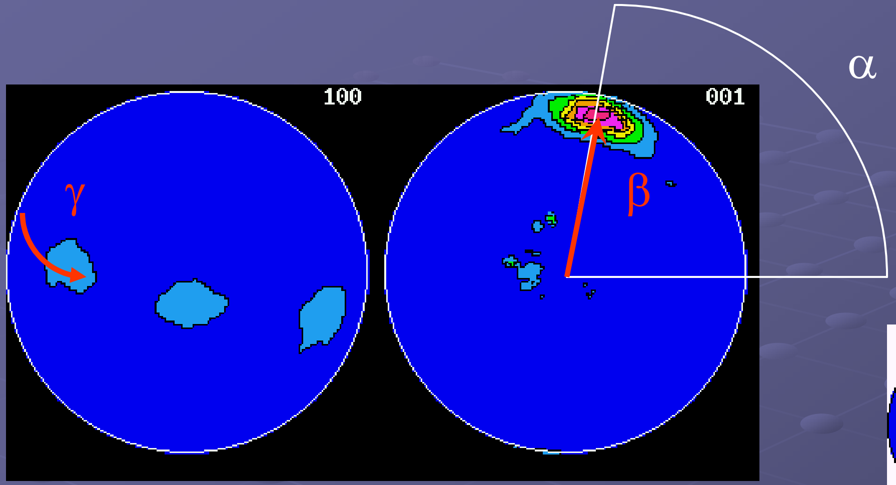


Roe/Matthies's convention

From f(g) to the pole figures

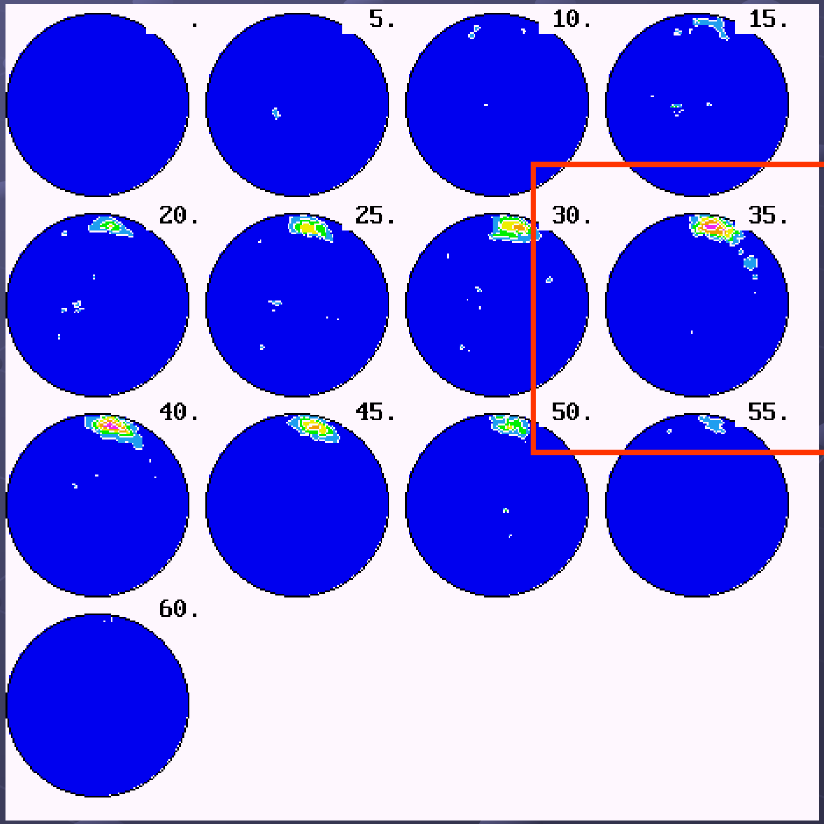


Deal with components in the ODF space



Pole figures

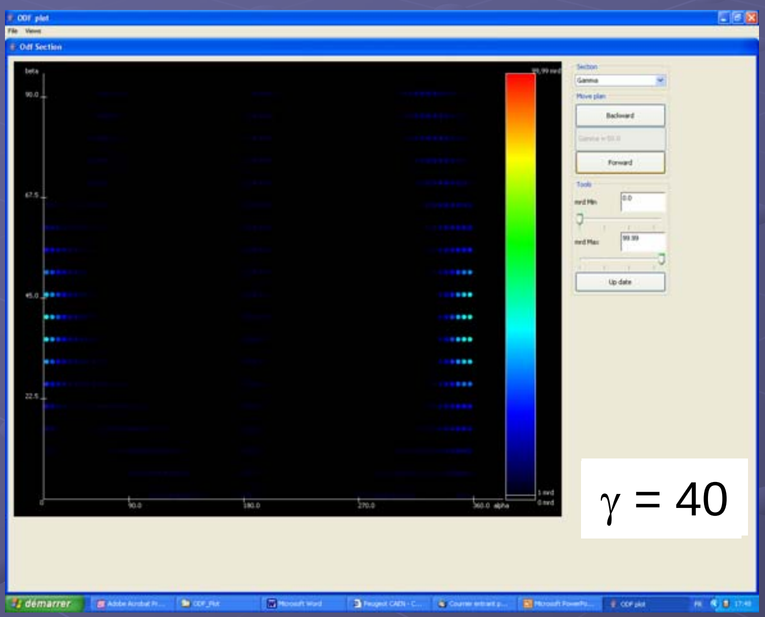
ODF γ-sections

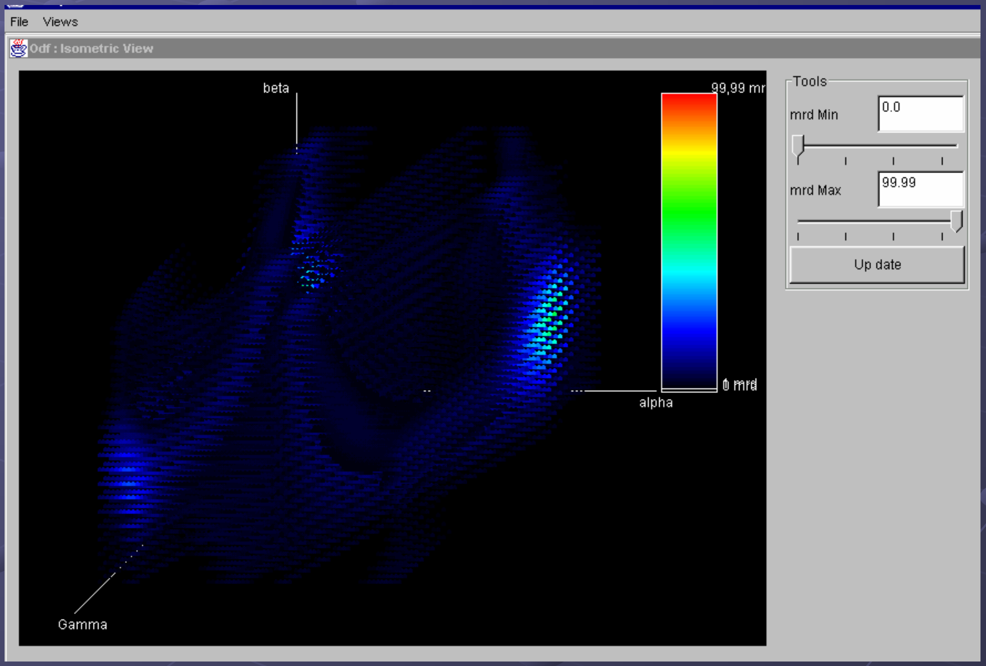


Component:
(Hexagonal system)
g = {85,80,35}

Plotting f(g)

A 3D plotting program: ODF plot





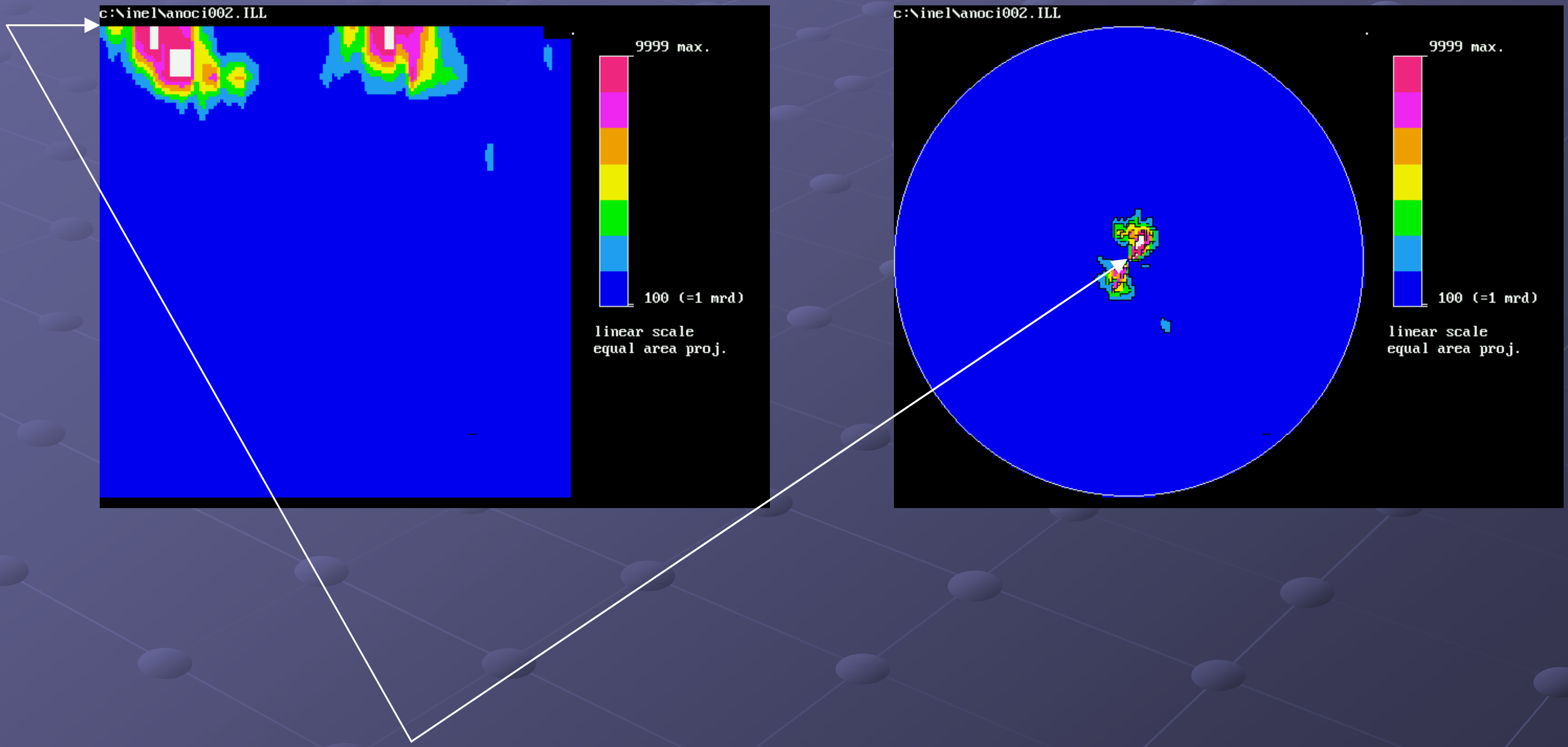
ODF sections (α, β, or γ)

ODF 3D-isometric view

Cartesian or Polar f(g) view

Cartesian

Polar



β = 0: space deformation

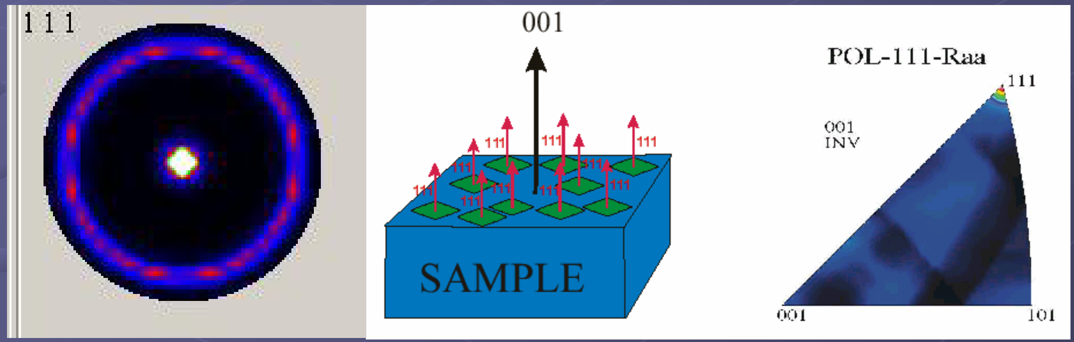
Inverse pole figures

$$P_{\mathbf{h}}(\mathbf{y}) = \frac{1}{2\pi} \int_{\mathbf{h}//\mathbf{y}} f(\mathrm{g})\, \mathrm{d}\tilde{\varphi}$$

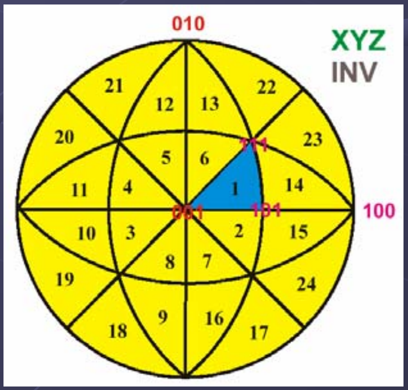
Pole figures

$$R_{\mathbf{y}}(\mathbf{h}) = \frac{1}{2\pi} \int_{\mathbf{y}//\mathbf{h}} f(\mathrm{g})\, \mathrm{d}\tilde{\tilde{\varphi}}$$

Inverse Pole figures



24 equivalent cubic sectors for the Inverse pole figure of a cubic system



ODF refinement

One has to invert:

$$P_{\mathbf{h}}(\mathbf{y}) = \frac{1}{2\pi} \int_{\mathbf{h}//\mathbf{y}} f(g)\, d\tilde{\varphi}$$

- from Generalized Spherical Harmonics (Bunge):

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

$$P_{\mathbf{h}}(\mathbf{y}) = \sum_{l=0}^{\infty} \frac{1}{2l+1} \sum_{n=-l}^{l} k_l^n(\mathbf{y}) \sum_{m=-l}^{l} C_l^{mn} k_n^{*m}(\Theta_{\mathbf{h}} \phi_{\mathbf{h}})$$

Least-squares Refinement procedure

$$\sum_{\mathbf{h}} \sum_{\mathbf{y}} \left[I_{\mathbf{h}}(\mathbf{y}) - N_{\mathbf{h}} P_{\mathbf{h}}(\mathbf{y})\right]^2 \mathbf{dy}$$

But even orders are the only available parts:

$$f^e(g) = \sum_{\lambda=0(2)}^{\infty} \sum_{m,n=-\lambda}^{\lambda} C_\lambda^{mn} T_\lambda^{mn}(g)$$

- from the WIMV iterative process (Williams-Imhof-Matthies-Vinel):

$$f^{n+1}(g) = N_n \frac{f^n(g) f^0(g)}{\left(\prod_{\mathbf{h}=1}^{I} \prod_{m=1}^{M_\mathbf{h}} P_\mathbf{h}^n(\mathbf{y}) \right)^{\frac{1}{IM_\mathbf{h}}}}$$

and

$$f^0(g) = N_0 \left(\prod_{\mathbf{h}=1}^{I} \prod_{m=1}^{M_\mathbf{h}} P_\mathbf{h}^{\exp}(\mathbf{y}) \right)^{\frac{1}{IM_\mathbf{h}}}$$

E-WIMV (Rietveld only):

$$f^{n+1}(g) = f^n(g) \prod_{m=1}^{M_\mathbf{h}} \left(\frac{P_\mathbf{h}(\mathbf{y})}{P_\mathbf{h}^n(\mathbf{y})} \right)^{r_n \frac{w_\mathbf{h}}{M_\mathbf{h}}}$$

with $0 < r_n < 1$, relaxation parameter,
$M_\mathbf{h}$ number of division points of the integral around k,
$w_\mathbf{h}$ reflection weight

- Entropy maximisation (Schaeben):

$$f^{n+1}(g) = f^n(g) \prod_{m=1}^{M_\mathbf{h}} \left(\frac{P_\mathbf{h}(\mathbf{y})}{P_\mathbf{h}^n(\mathbf{y})} \right)^{\frac{r_\mathbf{n}}{M_\mathbf{h}}}$$

- arbitrarily defined cells (ADC, Pawlik):

Very similar to E-WIMV, with integrals along path tubes

- Vector method (Ruer, Baro, Vadon):

I linear equations for J unknown quantities:

$$\mathbf{P_i(h) = [\sigma_{ij}(h)]\ f_j}$$

- Component method (Helming):

$$f(g) = F + \sum_c I^c f^c(g)$$

Gaussian component:

$$f(g, g^c) = f(\tilde{g}) = \frac{2\sqrt{\pi}}{\zeta\left\{1 - \exp\left(-\left(\frac{\zeta}{2}\right)^2\right)\right\}} \exp\left(-\left(\frac{\tilde{g}}{\zeta}\right)^2\right)$$

$$S = \frac{\ln 2}{1 - \cos\left(\frac{\zeta}{2}\right)} \qquad N(S) = \frac{1}{I_0(S) - I_1(S)}$$

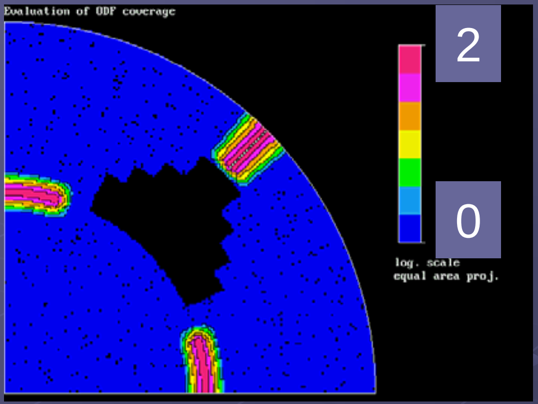
Evaluation of the OD coverage

Say 20 measured (5° x 5°) complete pole figures:

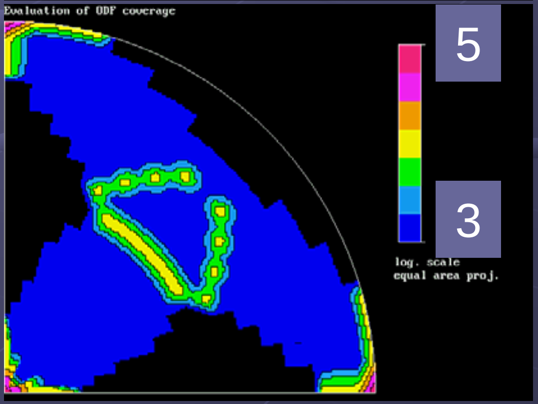
= 20 x 1368 = 27360 experimental points

ODF (5° x 5° x 5°, triclinic): 98496 points to refine

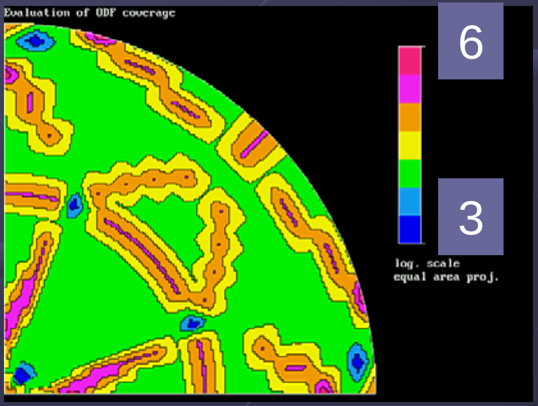
{100} pole figure, measured up to χ = 45°:



{100} + {110}, measured up to χ = 45°:

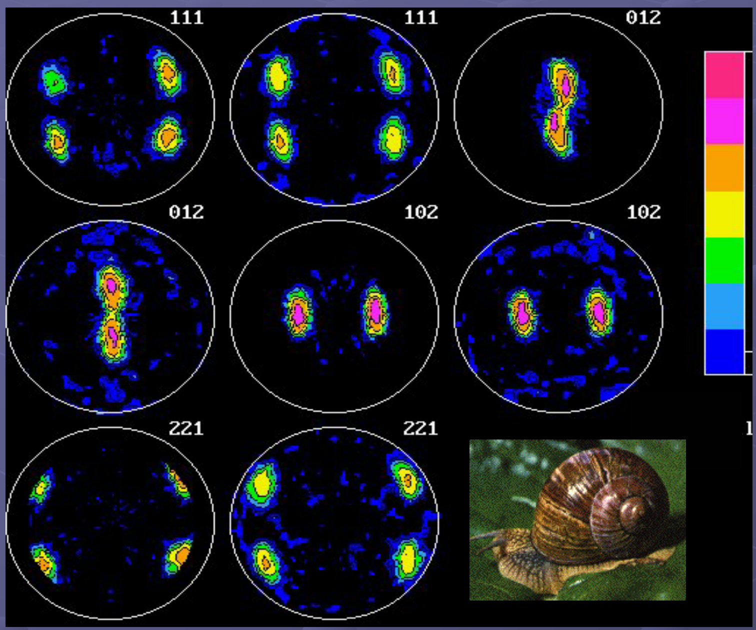


{100} + {110} + {111}, up to χ = 45°:

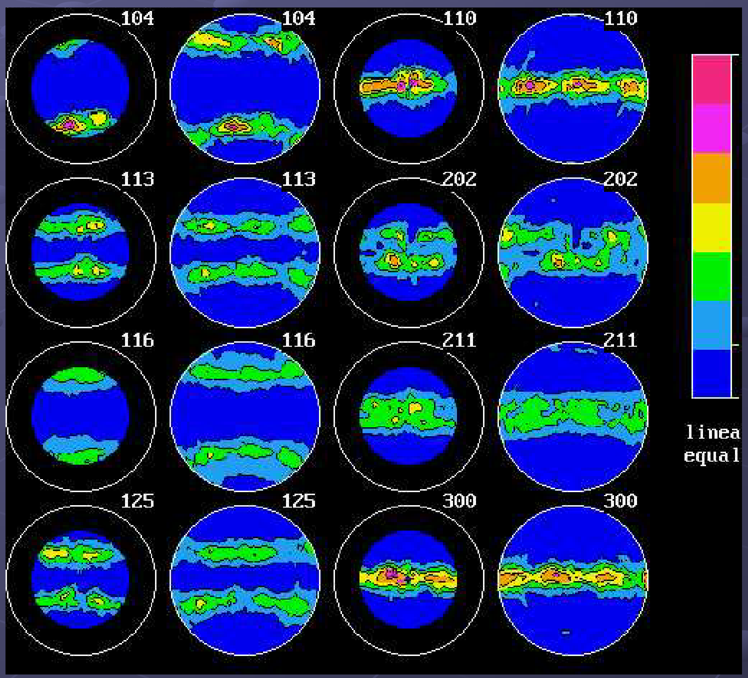


Estimators of Refinement Quality

Visual assessment



Helix pomatia (Burgundy land snail: Outer com. crossed lamellar layer)



Bathymodiolus thermophilus (deep ocean mussel: Outer Prismatic layer)

RP Factors:

Individual pole figures:

$$RP_x(h_i) = \frac{\sum_{j=1}^{J} \left| \tilde{P}^o_{h_i}(y_j) - \tilde{P}^c_{h_i}(y_j) \right|}{\sum_{j=1}^{J} \tilde{P}^o_{h_i}(y_j)} \theta\left(x, \tilde{P}^o_{h_i}(y_j)\right)$$

$$\theta(x,t) = \begin{cases} 1 \text{ for } t > x \\ 0 \text{ for } t \leq x \end{cases}$$

$$x = \varepsilon, 1, 10 \ldots$$

Averaged on all pole figures:

$$\overline{RP}_x = \frac{1}{I} \sum_{i=1}^{I} RP_x(h_i)$$

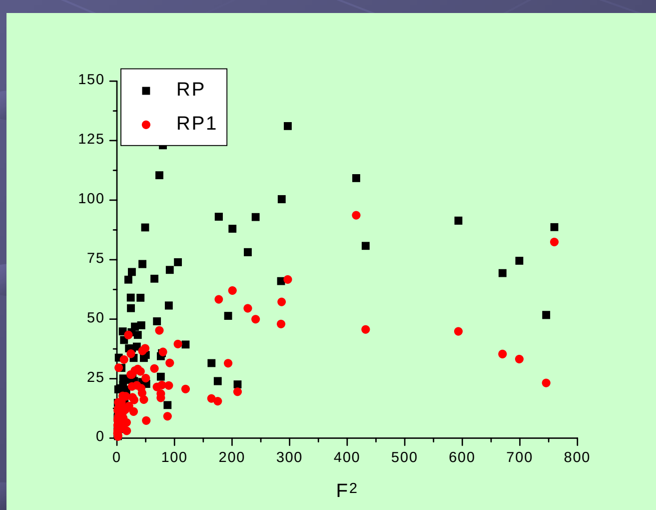
Bragg R-Factors:

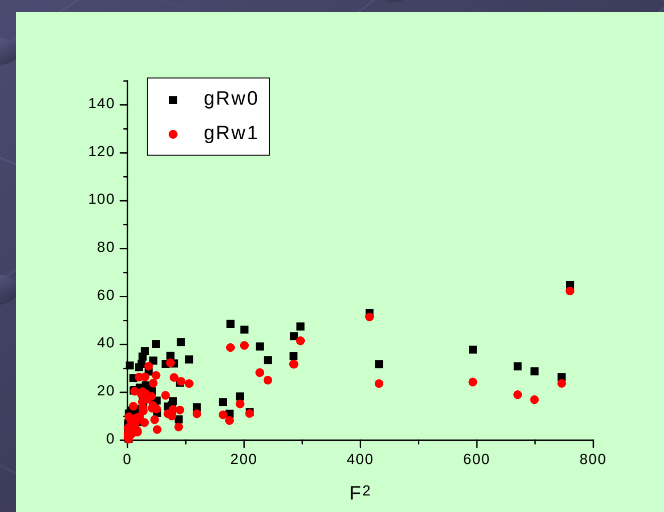
$$RB_x(h_i) = \frac{\sum_{j=1}^{J}\left[\tilde{P}^{o}_{h_i}(y_j) - \tilde{P}^{c}_{h_i}(y_j)\right]^2}{\sum_{j=1}^{J}\tilde{P}^{o\,2}_{h_i}(y_j)}\,\theta\left(x, \tilde{P}^{o}_{h_i}(y_j)\right)$$

Weighted Rw-Factors:

$$w_{ij} = \frac{1}{\sqrt{I^{o}_{h_i}(y_j)}}$$

$$Rw_x(h_i) = \frac{\sum_{j=1}^{J}\left[w^{o}_{ij} I^{o}_{h_i}(y_j) - w^{c}_{ij} I^{c}_{h_i}(y_j)\right]^2}{\sum_{j=1}^{J} w^{o}_{ij} I^{z\,2}_{h_i}(y_j)}\,\theta\left(x, \tilde{P}^{o}_{h_i}(y_j)\right)$$





Texture strength estimators

ODF Texture Index:

$F^2 \in]1,\infty[$
- > 1 m.r.d^2
- $= 1$: powder
- $= \infty$: single crystal

$$F^2(\text{m.r.d.}^2) = \frac{1}{8\pi^2}\sum_i f^2(g_i)\Delta g_i$$

Discrete OD

$$F^2 = 1 + \sum_{\lambda=2}^{L}\left[\frac{1}{2\lambda+1}\right]\sum_{m=-\lambda}^{\lambda}\sum_{n=-\lambda}^{\lambda}\left|C_\lambda^{mn}\right|^2$$

Continuous ODF

Pole figures Texture Index:

$$J_{\mathbf{h}}^{\mathbf{2}} = \frac{1}{4\pi}\sum_{\mathrm{i}}\left[P_{\mathbf{h}}(\mathbf{y}_{\mathrm{i}})\right]^2\Delta\mathbf{y}_{\mathrm{i}}$$

Texture Entropy:

$$S \in [0,-\infty[\qquad \leq 0$$

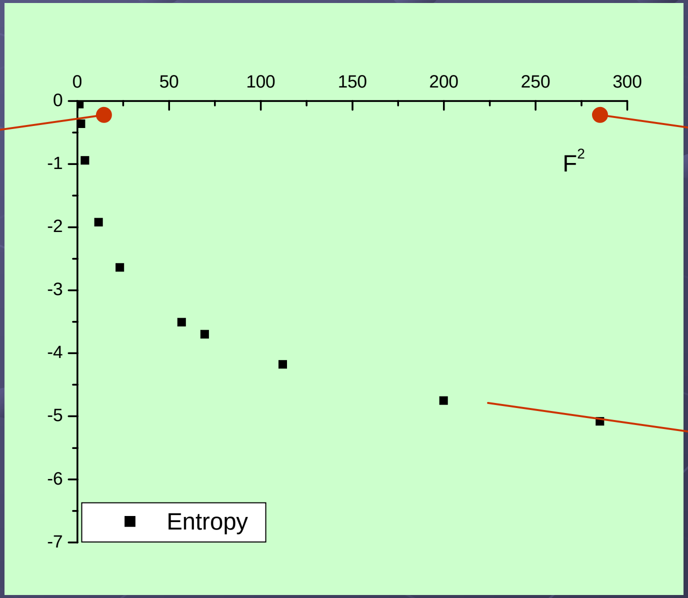
$= 0$: powder

$= -\infty$: single crystal

$$S = \frac{-1}{8\pi^2} \sum_i f(g_i) \ln[f(g_i)] \Delta g_i$$

S - F²:

Fon +
smooth
texture component(s)

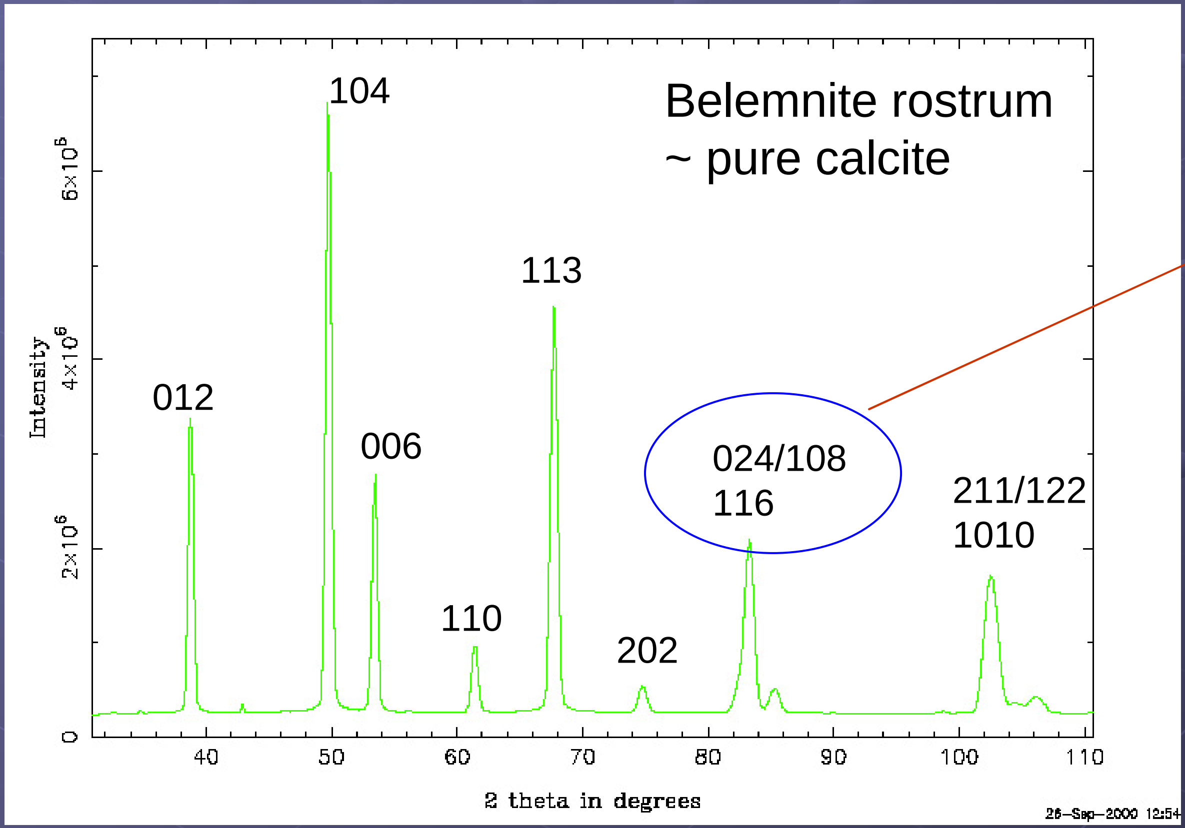


Fon +
Dirac-like
texture component

Lower bound:
Fon = 0

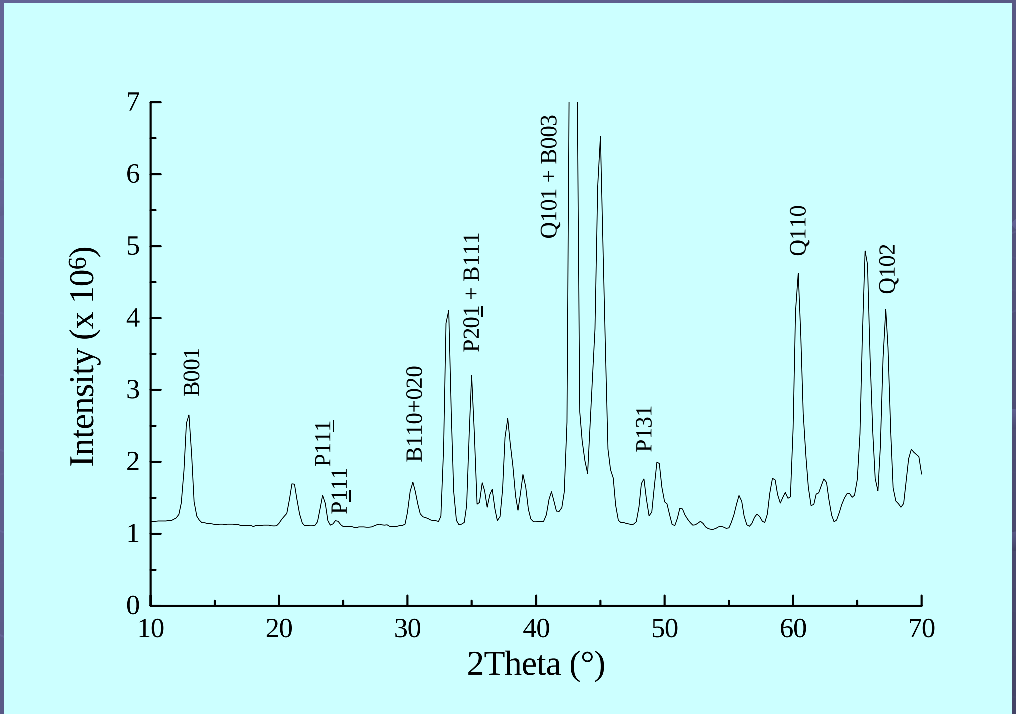
Why needing combined analysis

- Solve the peak-overlap problems (intra- and inter-phases)



Resolved during ODF refinement

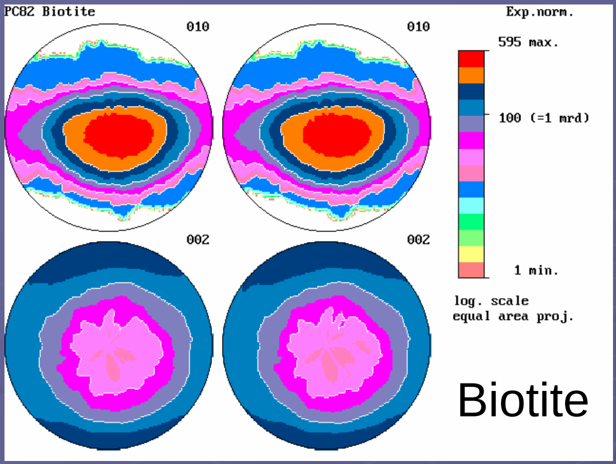
Polyphased Mylonite (Palm Canyon, CA)

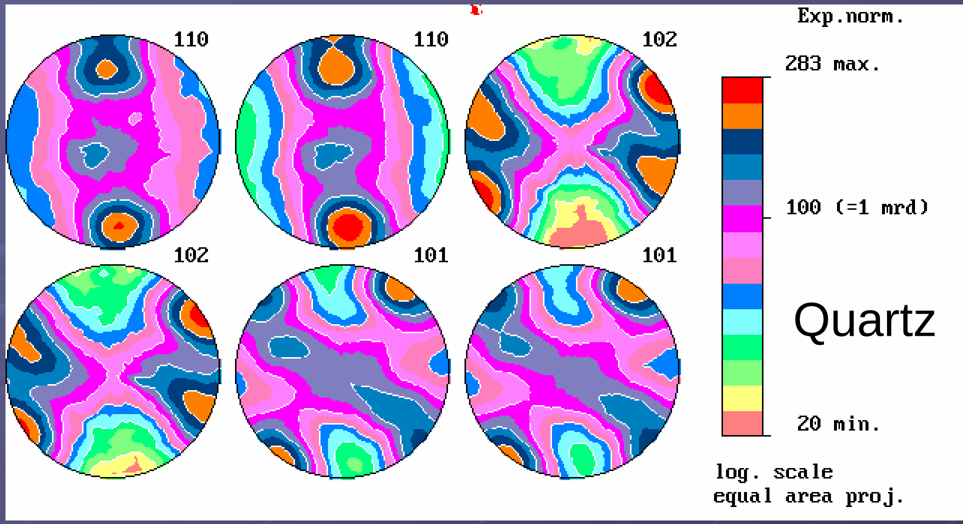


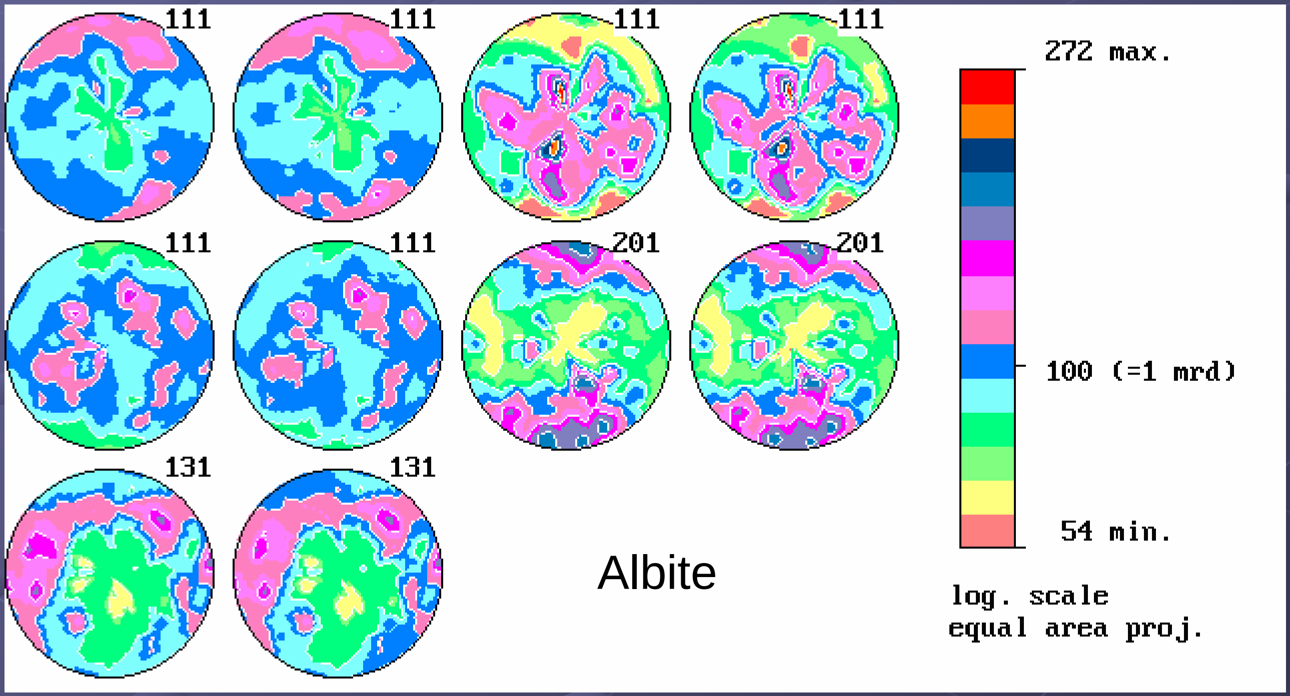
Using 0D detector hardly manageable

PC 82 mylonite	Biotite	Quartz	Albite	Anorthite	K-spar
Composition (weight %)	9.0	24.2	31.7	17.4	14.1
Space group	C2/m	R3	C-1		

Textures & Microstructures **33**, 1999, 35-43

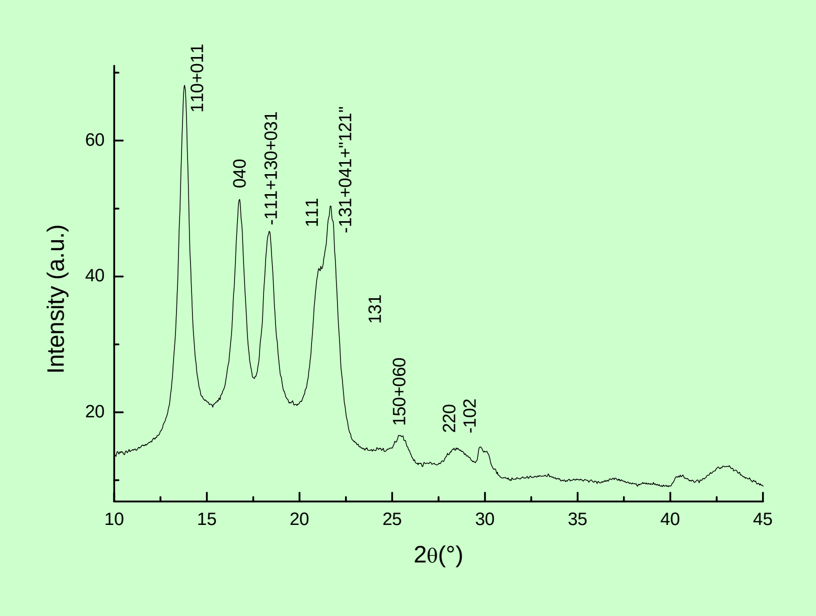

PC82 Biotite
010
010
002
002
Exp.norm.
595 max.
100 (=1 mrd)
1 min.
log. scale
equal area proj.
Biotite


110
110
102
102
101
101
Exp.norm.
283 max.
100 (=1 mrd)
20 min.
Quartz
log. scale
equal area proj.


111
111
-111
-111
111
111
201
-201
131
131
272 max.
100 (=1 mrd)
54 min.
Albite
log. scale
equal area proj.


lineation
foliation

Plasma-treated polypropylene films



Large broadening + overlaps + amorphous phase

- Don't want or can't powderise your sample:
  - . Rare: Ice from deep cores, meteorite rocks ...
  - . Expensive: high-tech materials
  - . Impossible: hard materials, polymers, thin structures ...

- Decreases instrument time:
  - . 5° x 5° grid = 1368 points / pole figure
  - . ODF: needs as much pole figures as possible

- Access to other parameters:
  - . crystal sizes, micro-strains, stacking faults + twins (QMA)
  - . residual strains and stresses (QSA)
  - . Structure determination
  - . Phase proportions (QPA)
  - . Thicknesses, roughnesses (XRR)

- Avoid false minima due to parameter correlation:
  - . phase and texture
  - . Structure and texture
  - . Structure and strains
  - . Thickness and phase
  - …

- Benefit of these correlation to access "true" values
  Textured materials: between powder and single-crystal, angular discrimination

- Easier to practice !