Mesures Physiques

IUT

Combined Analysis: texture, structure, microstructure, phase, stress, reflectivity (multiphase bulks and thin films, x-rays and neutron diffraction): some case studies

Daniel Chateigner
CRISMAT-ENSICAEN (Caen-France)

Bi2223 Superconductors

PCT & PMN-PT Ferroelectrics

Irradiated FAp ceramics

CRISMAT

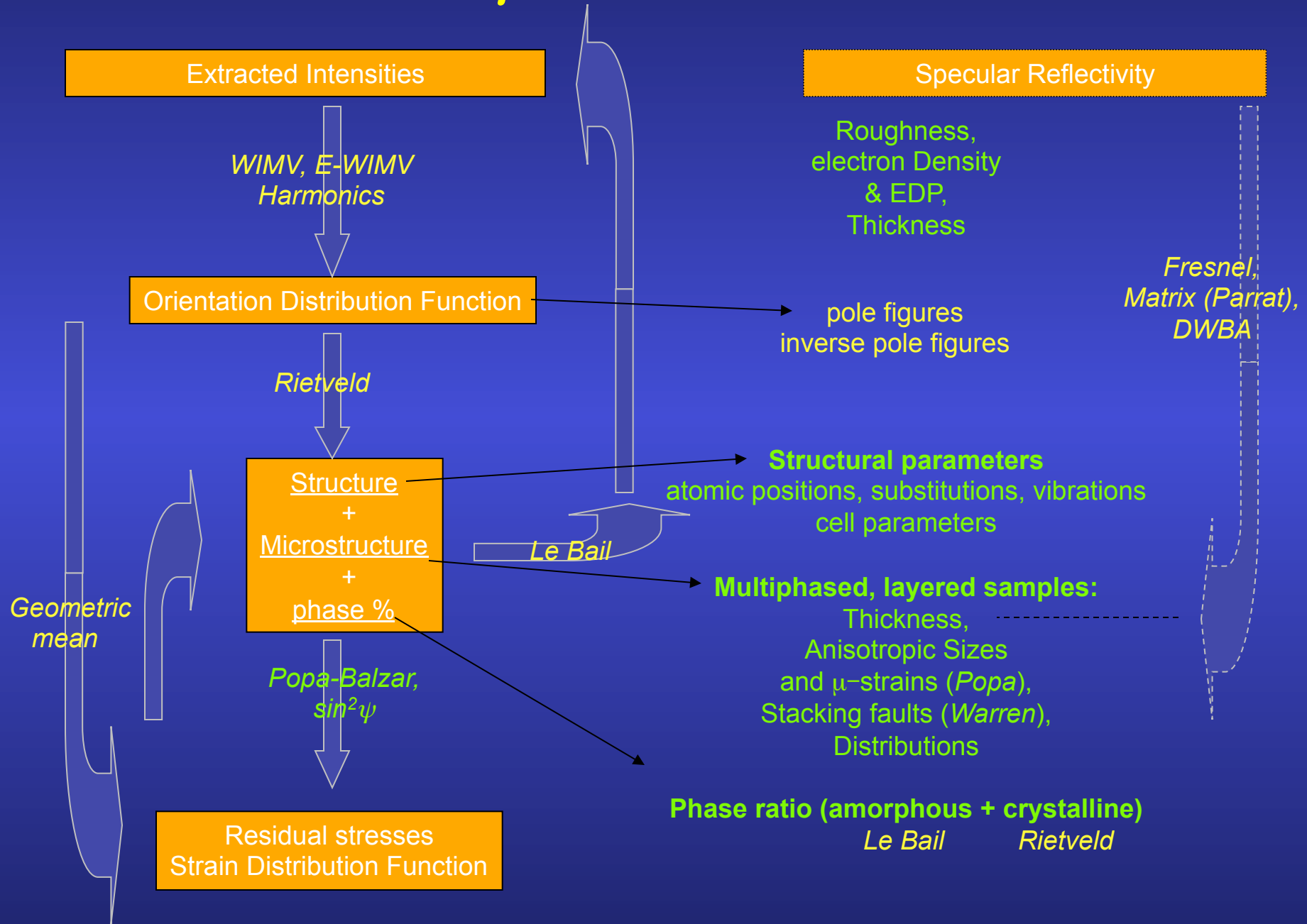
$Ca_3Co_4O_9$ Thermoelectrics

nano-Si thin films

ENSICAEN

IMN Jean Rouxel 2006

Implemented codes

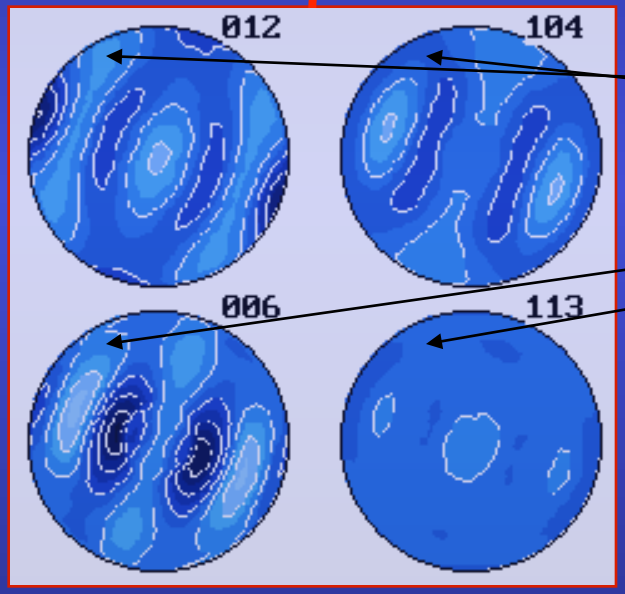


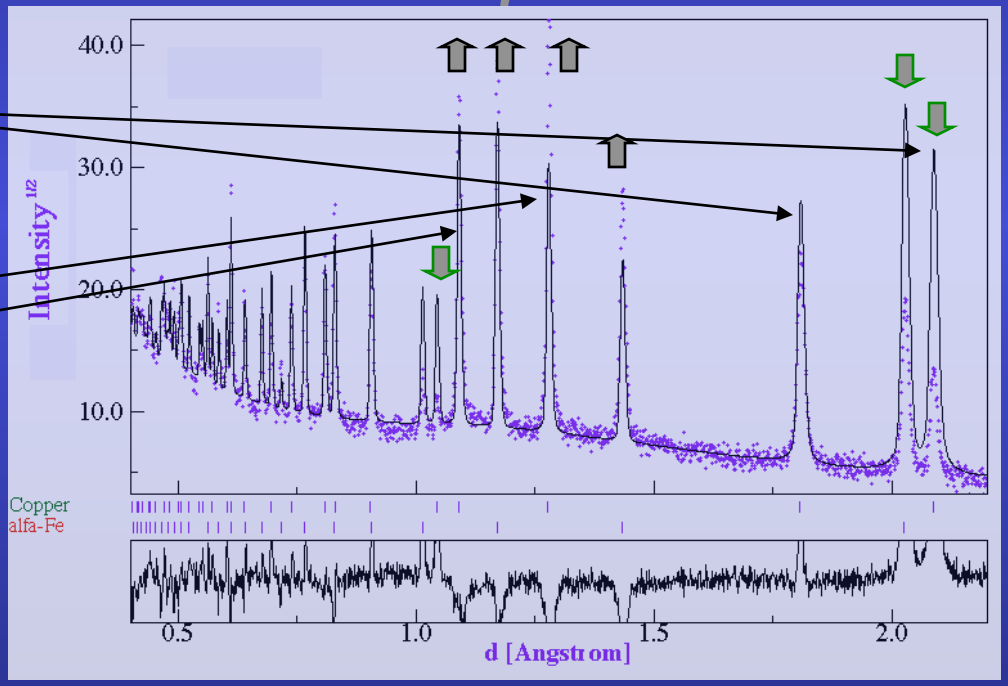
Texture from Spectra

Orientation Distribution Function (ODF)

From pole figures

From spectra





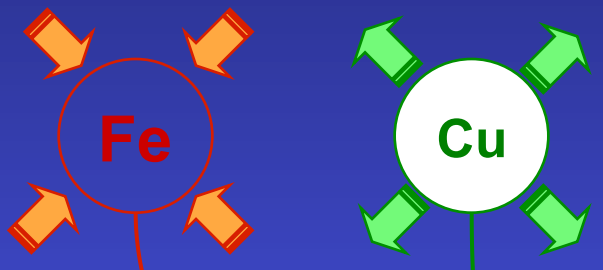
Residual Stresses and Rietveld

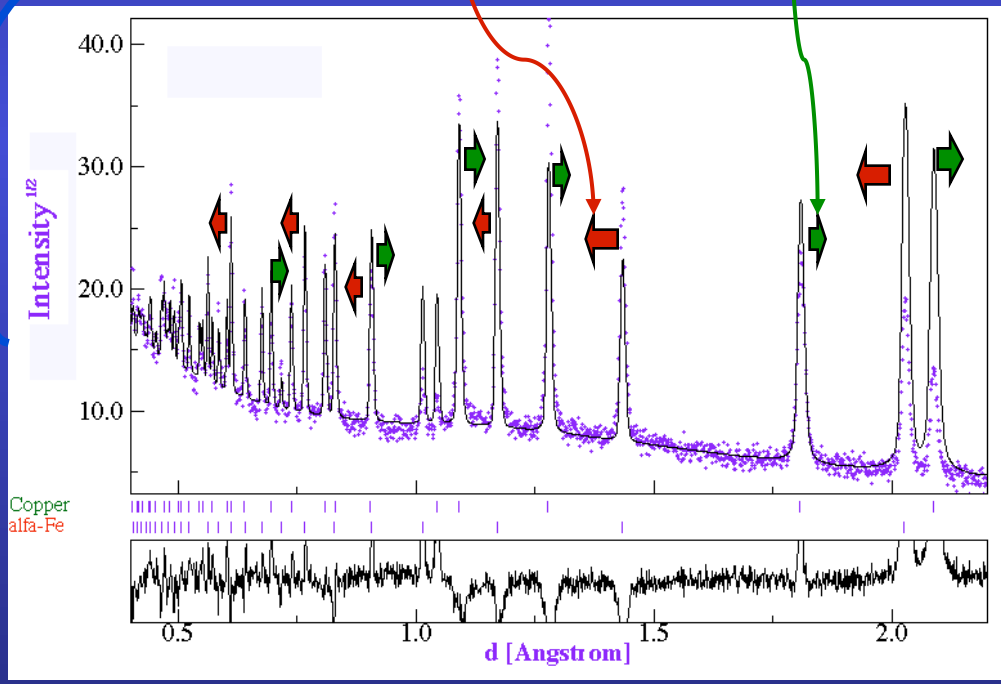
- Macro elastic strain tensor (I kind)
- Crystal anisotropic strains (II kind)

C

Macro and micro stresses

Applied macro stresses





Textured samples: Reuss, Voigt, Hill, Bulk geometric mean approaches

How it works (Combined)

$$I_i^{calc}(\chi,\phi) = \sum_{n=1}^{Nphases} S_n \sum_k L_k \left|F_{k;n}\right|^2 S\left(2\theta_i - 2\theta_{k;n}\right) P_{k;n}(\chi,\phi) A + bkg_i$$

Texture

$$P_k(\chi,\phi) = \int_{\varphi} f(g,\varphi)\, d\varphi$$

- from Generalized Spherical Harmonics:

$$P_k(\chi,\phi) = \sum_{l=0}^{\infty} \frac{1}{2l+1} \sum_{n=-l}^{l} k_l^n(\chi,\phi) \sum_{m=-l}^{l} C_l^{mn} k_n^{*m}\left(\Theta_k \phi_k\right)$$

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

- from the WIMV (left) iterative process or entropy maximisation (right):

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

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

Layering

$$C_{\chi}^{\text{top film}} = g_1\left(1-\exp\left(-\mu T g_2/\cos\chi\right)\right)/\left(1-\exp\left(-2\mu T/\sin\omega\cos\chi\right)\right)$$

$$C_{\chi}^{\text{cov. layer}} = C_{\chi}^{\text{top film}}\left(\exp\left(-g_2\sum\mu_i' T_i'/\cos\chi\right)\right)/\left(\exp\left(-2\sum\mu_i' T_i'/\sin\omega\cos\chi\right)\right)$$

Popa anisotropic shapes & microstrains

$$\langle R_{\mathbf{h}}\rangle = R_0 + R_1 P_2^{\,0}(x) + R_2 P_2^{\,1}(x)\cos\varphi + R_3 P_2^{\,1}(x)\sin\varphi + R_4 P_2^{\,2}(x)\cos 2\varphi + R_5 P_2^{\,2}(x)\sin 2\varphi +$$

$$\langle \varepsilon_{\mathbf{h}}^{\,2}\rangle E_{\mathbf{h}}^{\,4} = E_1 h^4 + E_2 k^4 + E_3 \ell^4 + 2E_4 h^2 k^2 + 2E_5 \ell^2 k^2 + 2E_6 h^2 \ell^2 + 4E_7 h^3 k + 4E_8 h^3 \ell + 4E_9 k^3 h + 4E_{10} k^3 \ell + 4E_{11} \ell^3 h + 4E_{12} \ell^3 k + 4E_{13} h^2 k \ell + 4E_{14} k^2 h \ell + 4E_{15} \ell^2 k h$$

Roughness and/or microabsorption

$$R^{rough}(q_z) = R(q_z)\exp(-q_{z,0}q_{z,1}\sigma^2)$$ Low-angles (reflectivity)

$$S_R = 1 - p\exp(-q) + p\exp\left(\frac{-q}{\sin\theta}\right)$$ high-angle (Suortti)

Specular reflectivity: **q**=(0,0,z)

- Fresnel:

$$R(\mathbf{q}) = \left| \frac{q_z - \sqrt{q_z^2 - q_c^2 + \frac{32i\pi^2\beta}{\lambda^2}}}{q_z + \sqrt{q_z^2 - q_c^2 + \frac{32i\pi^2\beta}{\lambda^2}}} \right|^2 \delta q_x \delta q$$

- matrix:

$$R^{flat} = \frac{r_{0,1}^2 + r_{1,2}^2 + 2r_{0,1}r_{1,2}\cos 2k_{Z,1}h}{1 + r_{0,1}^2 r_{1,2}^2 + 2r_{0,1}r_{1,2}\cos 2k_{Z,1}h}$$

- Born approximation:

$$R(q_z) = r.r^* = R_F(q_z)\left| \frac{1}{\rho_s} \int_{-\infty}^{+\infty} \frac{d\rho(z)}{dz} e^{iq_z z}\, dz \right|^2$$

Phase

$$W_\Phi = \frac{S_\Phi Z_\Phi M_\Phi V_\Phi}{\sum_{i=1}^{N_\Phi} S_i Z_i M_i V_i}$$

Strain-Stress

$$\boldsymbol{\varepsilon}(\mathbf{X}) = \boldsymbol{\varepsilon}^{\mathbf{I}} + \boldsymbol{\varepsilon}^{\mathbf{II}}(\mathbf{X}) + \boldsymbol{\varepsilon}^{\mathbf{III}}(\mathbf{X})$$

$$\begin{aligned}\langle \varepsilon_h(\mathbf{y}) \rangle_{V_d} &= \frac{1}{V_d}\int_{V_d}(\varepsilon_{33}^{I} + \varepsilon_{33}^{II} + \varepsilon_{33}^{III})dV \\ &= (\varepsilon_{11}^{I}\cos^2\phi + \varepsilon_{12}^{I}\sin 2\phi + \varepsilon_{22}^{I}\sin^2\phi - \varepsilon_{33}^{I})\sin^2\psi + \varepsilon_{33}^{I} + \\ &\quad (\varepsilon_{13}^{I}\cos\phi + \varepsilon_{23}^{I}\sin\phi)\sin 2\psi + \frac{1}{V_d}\int_{V_d}(\varepsilon_{33}^{IIe} + \varepsilon_{33}^{IIti} + \varepsilon_{33}^{IIpi})dV \\ &= \frac{\langle d(hkl,\phi,\psi)\rangle_{V_d} - d_0(hkl)}{d_0(hkl)}\end{aligned}$$

Isotropic samples:
Tri-, bi-, uni-axial stress states

Textured samples:
Tri-, bi-, uni- stress states
+ ODF + SDF + model

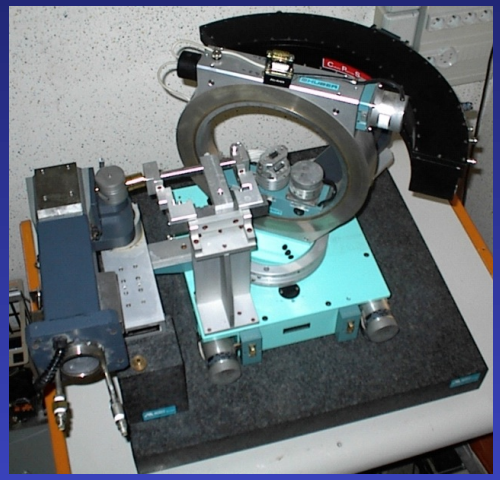
$$\langle E(\mathbf{g}) \rangle_{V_d} = \frac{1}{V_d}\int_{V_d} E^{SC}(g) f(g) dg \quad \Rightarrow \quad C^{M}_{ijkl} \neq \left(S^{M}_{ijkl}\right)^{-1}$$

$$= \left(\prod_{V_d} E^{SC}(g) f(g) dg\right)^{\frac{1}{V_d}} \quad \Rightarrow \quad C^{M}_{ijkl} = \left(S^{M}_{ijkl}\right)^{-1}$$

Reuss, Voigt, Hill

Geometric mean, *VPSC*

Minimum experimental requirements



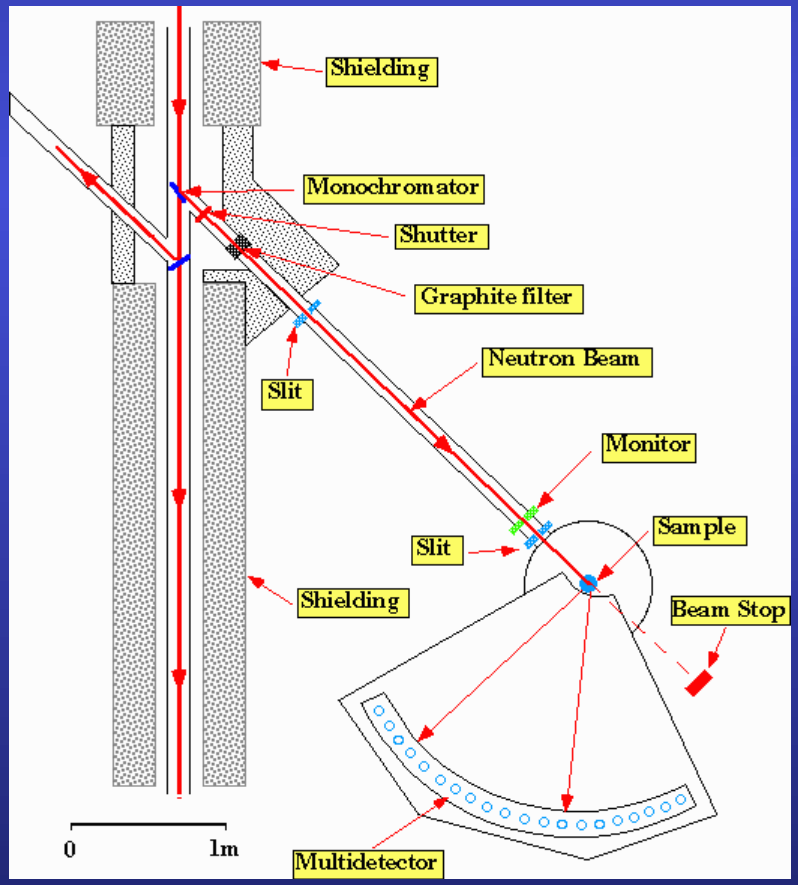
1D or 2D Detector + 4-circle diffractometer
(X-rays and neutrons)
CRISMAT, ILL

+

~1000 experiments (2θ diagrams)
in as many sample orientations

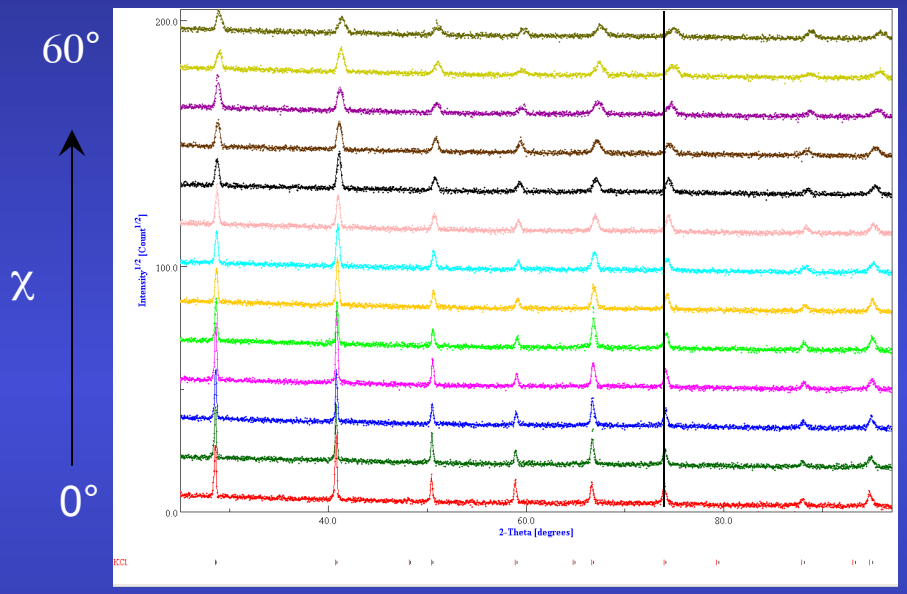
+

Instrument calibration
(peaks widths and shapes,
misalignments, defocusing …)

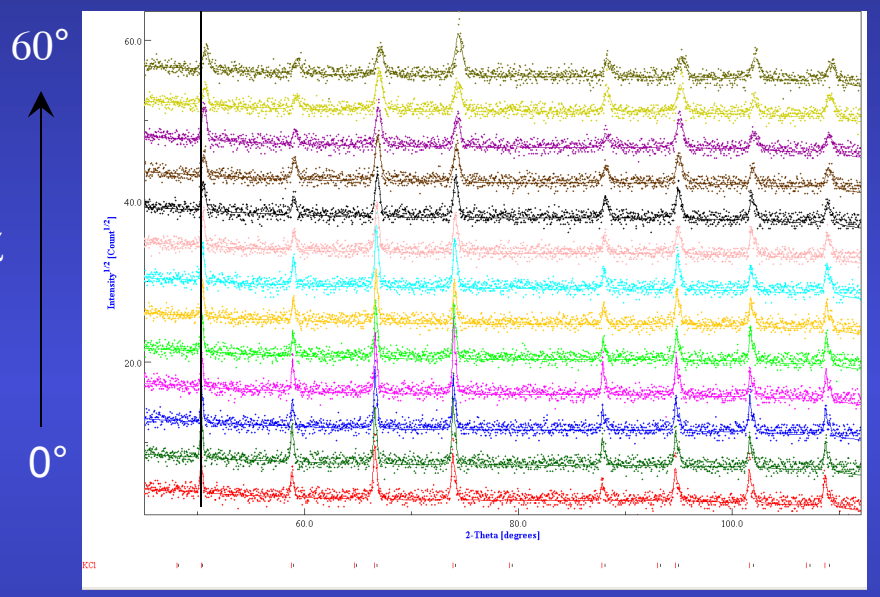


Calibration

ω = 20°



ω = 40°

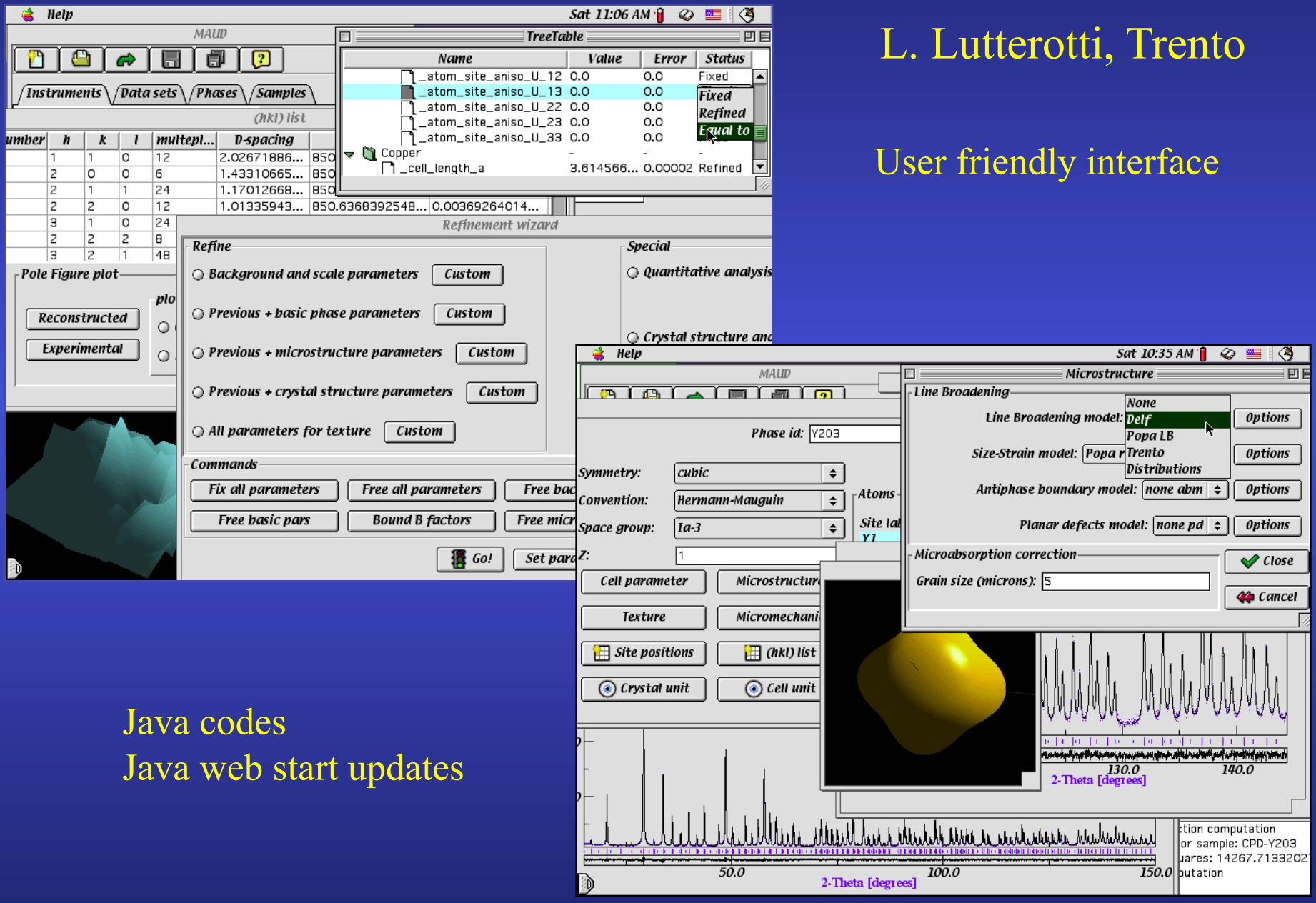


KCl, LaB_6 …

→

FWHM (ω, χ, 2θ …)
2θ shift
gaussianity
asymmetry
misalignments ...

Methodology implementation



L. Lutterotti, Trento

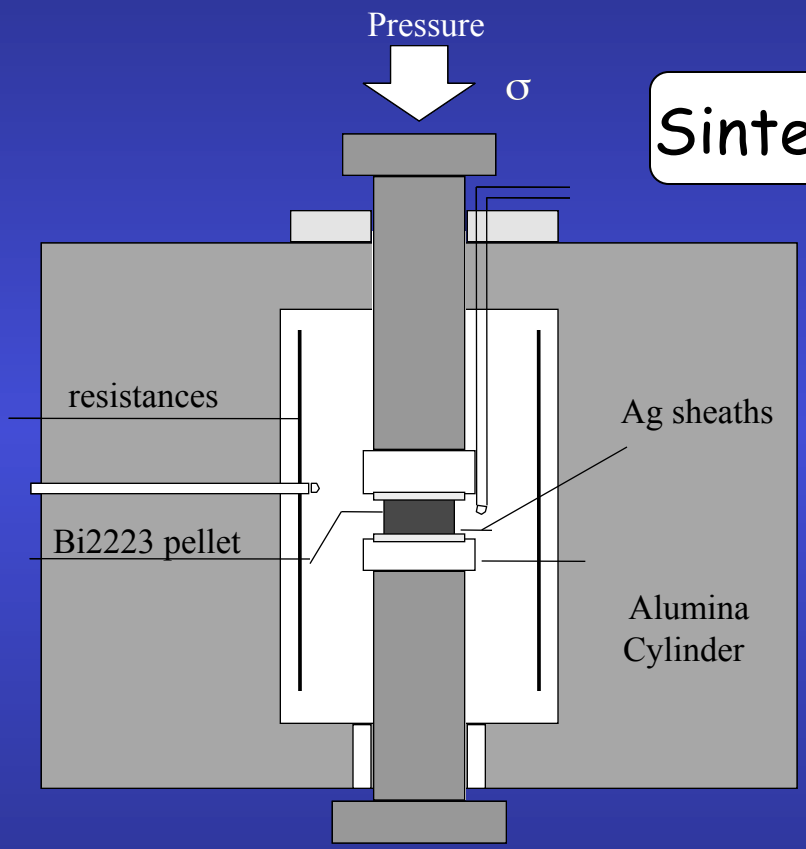
User friendly interface

Java codes

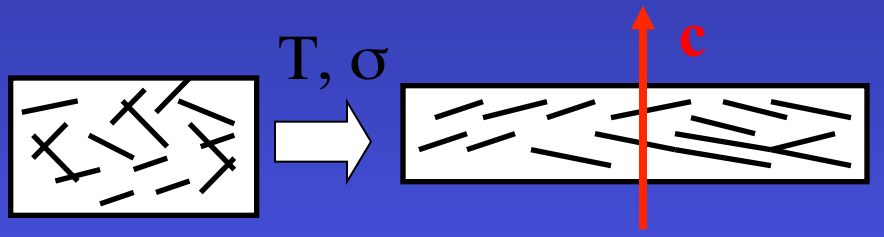
Java web start updates

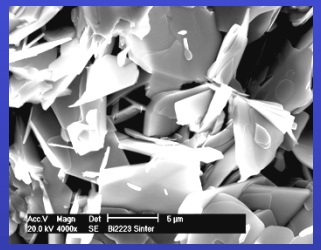
Bi2223 compounds

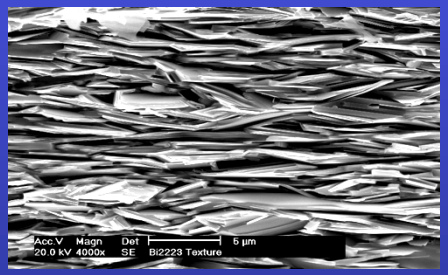
E. Guilmeau, CRISMAT



Sinter-Forging

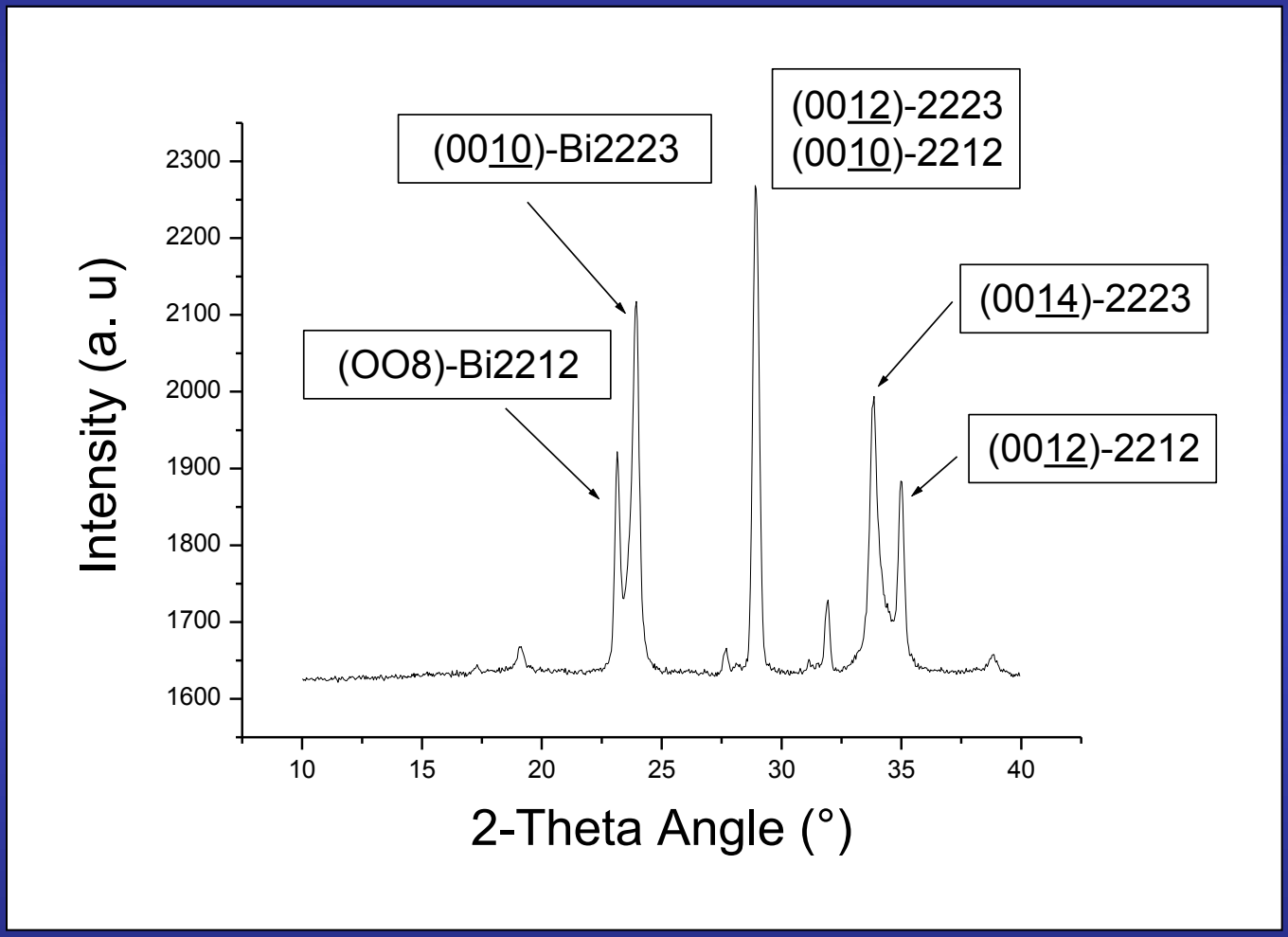




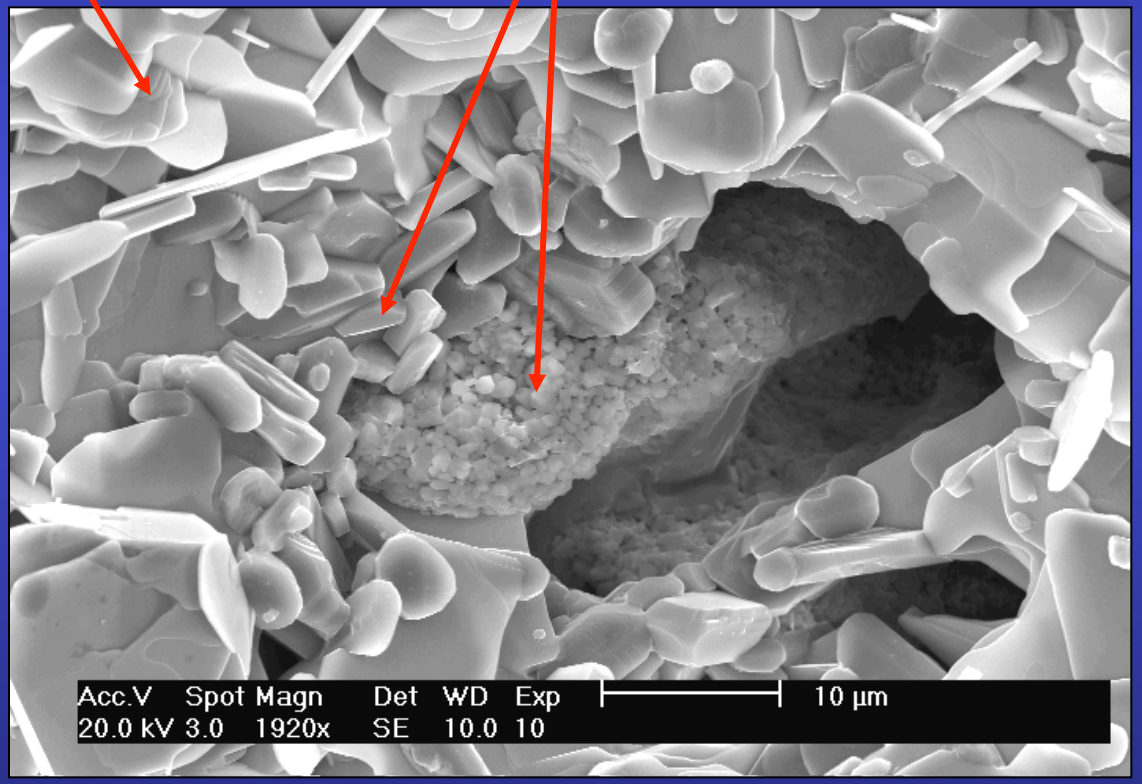


Grain alignment ⇒ ↗ Jc

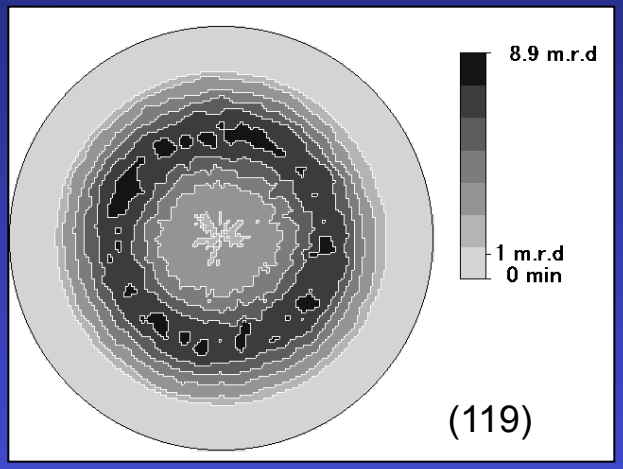
(00ℓ) Texture

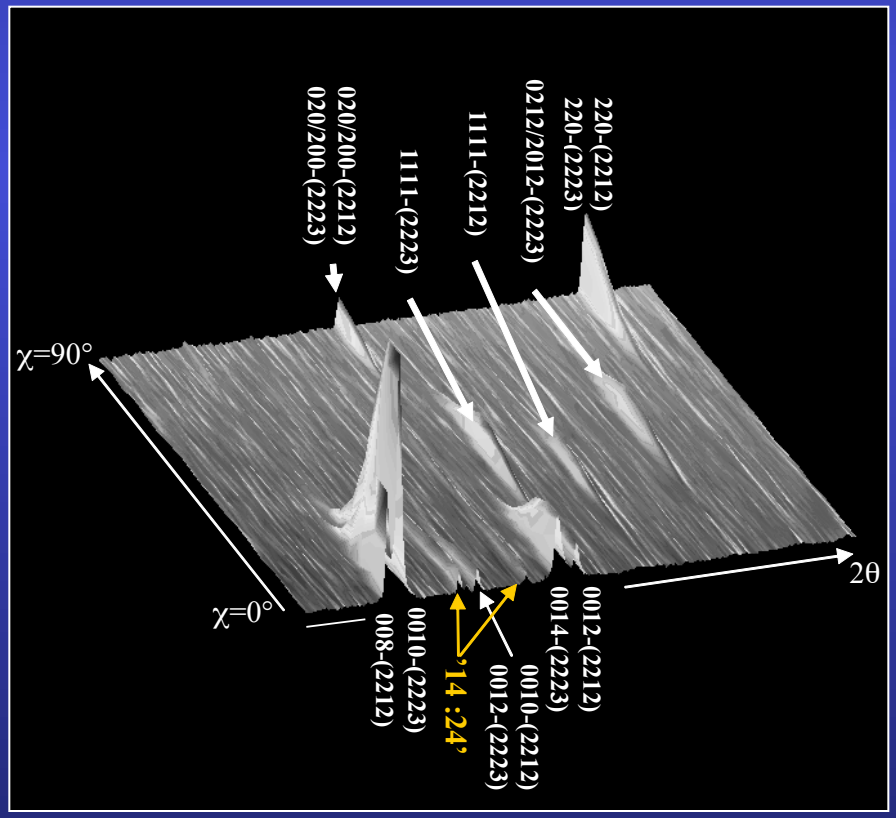


Bi2212 + Secondary phases → Bi2223

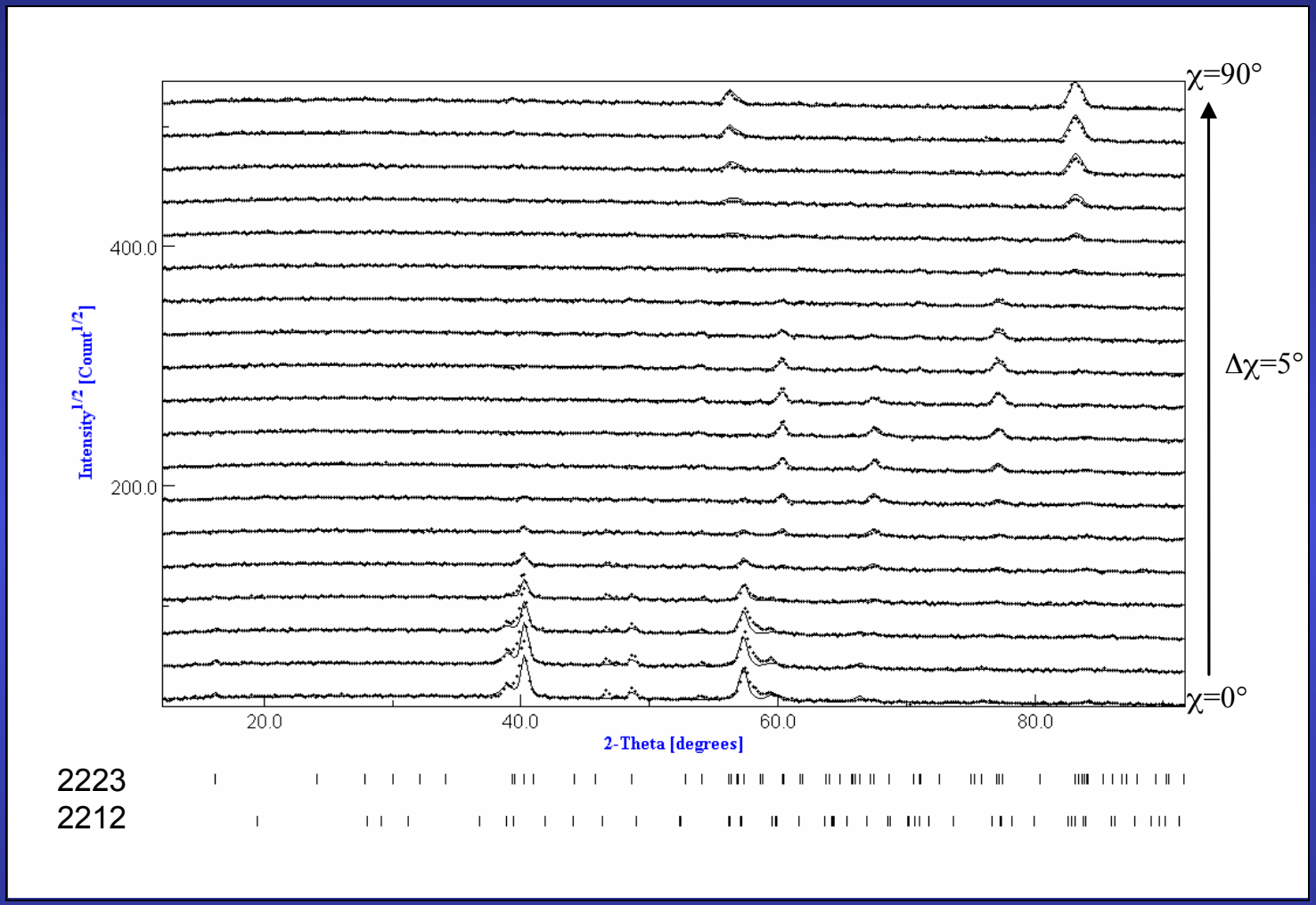


Combined Analysis

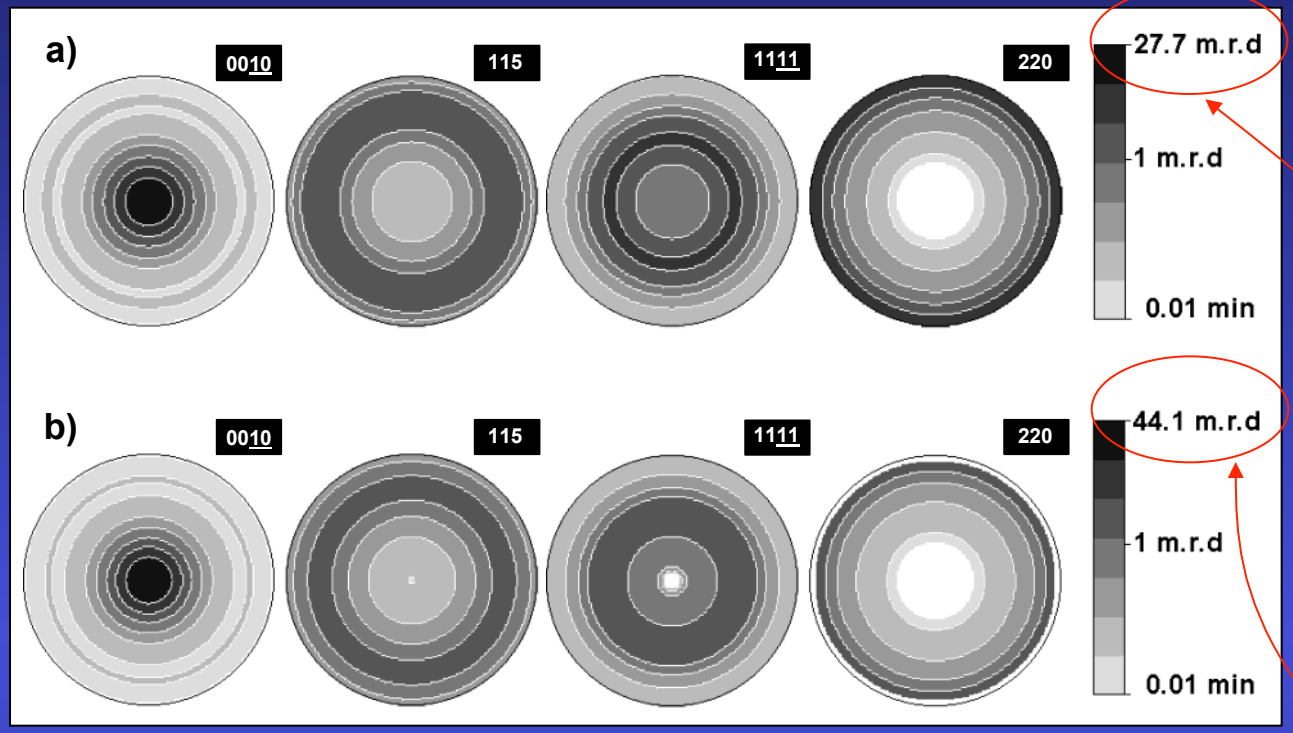




-Neutrons
-Sample: ~70 mm^3
-2θ patterns for χ=0° to 90°
-No φ rotation (fibre texture).



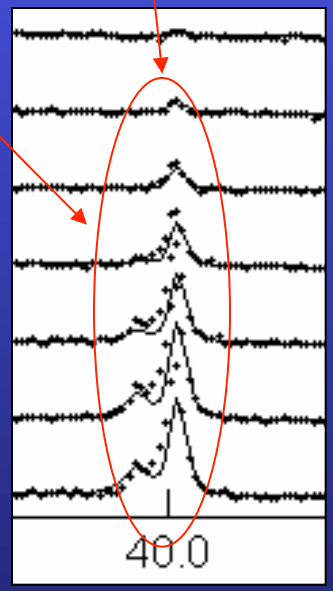
Rw=9.12
RP=16.24

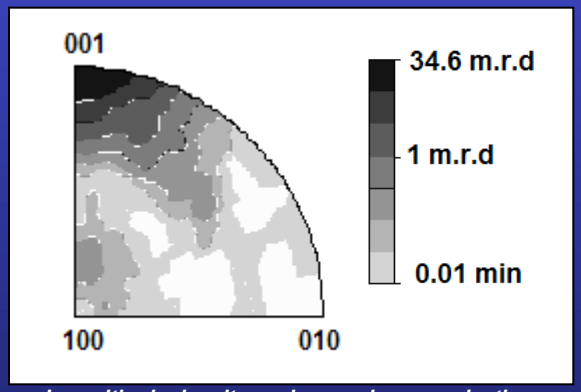


Logarithmic density scale, equal area projection

Recalculated (WIMV)

Extracted (Le Bail)





Logarithmic density scale, equal area projection

Stacking faults and/or intergrowth on the c-axis →New periodicities and peaks characterized with intermediate c parameters.

However, no algorithm is included to solve intergrowths in the combined approach.

Effect of the sinter-forging treatment on the texture development, crystal growth, transport properties

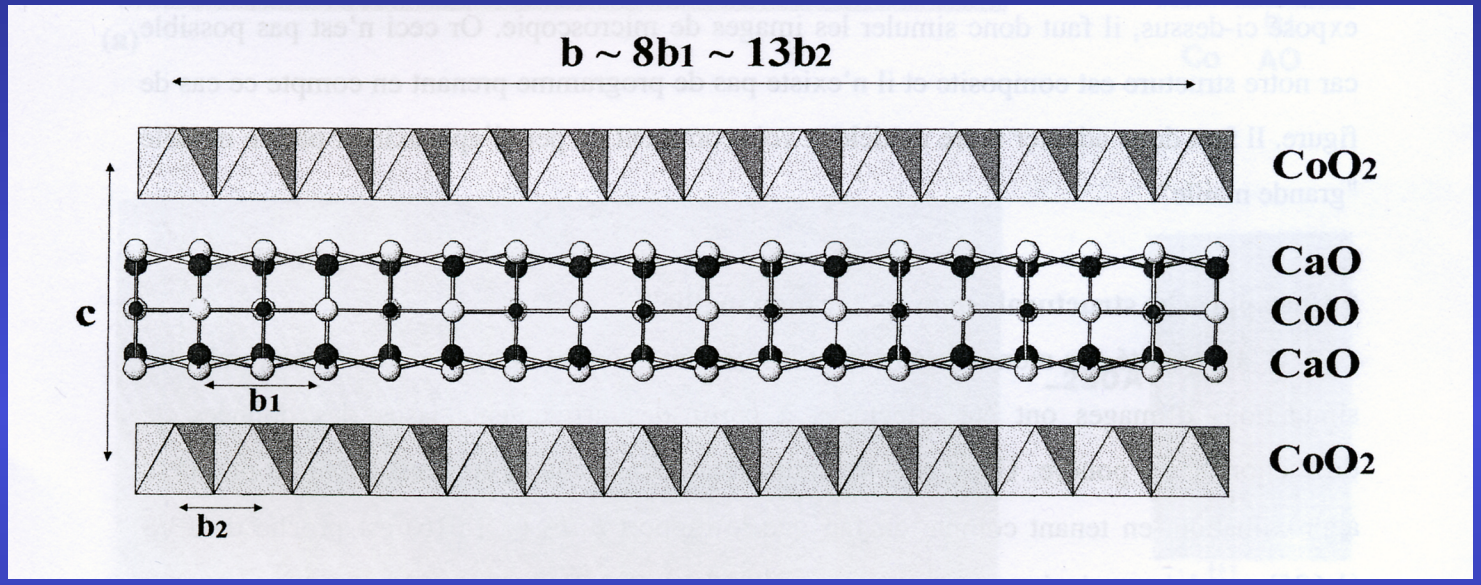
Sinter-forging dwell time (h)	Orientation Distribution Max (m.r.d.)		% Bi2223	Cell parameters (Å)		Crystallite size Bi2223 (nm)	Rb (%)	Rw (%)	Rexp (%)	RP0 (%)	RP1 (%)	J_c (A/cm²)
	Bi2212	Bi2223		Bi2223	Bi2212							
20	21.8	20.7	59.9±1.3	a=5.419(3) b=5.391(3) c=37.168(3)	a=5.414(3) b=5.393(3) c=30.800(3)	205±7	7.56	11.1	4.55	17.74	10.56	12500
50	24.1	24.4	72.9±2.9	a=5.419(3) b=5.408(3) c=37.192(3)	a=5.416(3) b=5.396(3) c=30.806(3)	273±10	7.54	11.37	4.58	17.05	11.04	15000
100	31.5	25.2	84.4±4.6	a=5.410(3) b=5.405(3) c=37.144(3)	a=5.412(3) b=5.403(3) c=30.752(3)	303±10	5.4	8.04	3.69	13.54	9.31	19000
150	65.4	27.2	87.0±4.1	a=5.417(3) b=5.403(3) c=37.199(3)	a=5.413(3) b=5.407(3) c=30.792(3)	383±13	6.13	9.12	4.8	16.24	12.25	20000

E. Guilmeau et al., JAC

$Ca_3Co_4O_9$ thermoelectrics

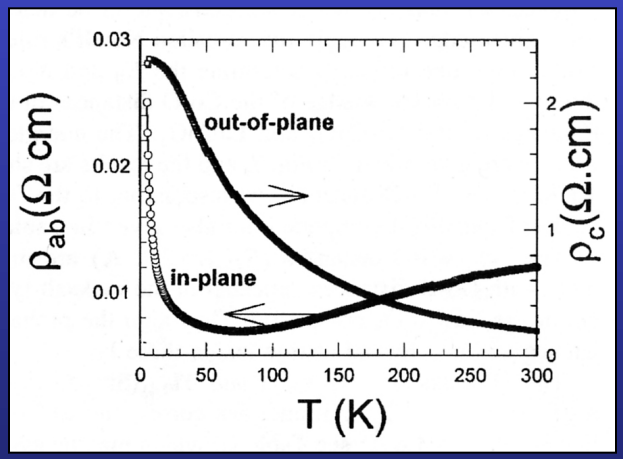
M. Prevel, CRISMAT

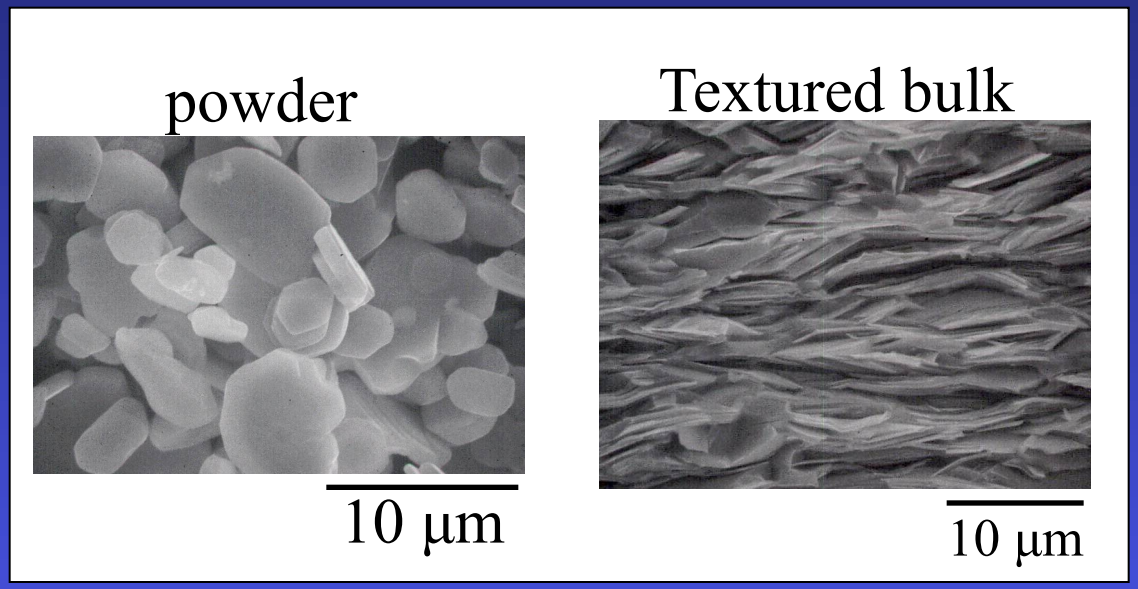
$Ca_3Co_4O_9$: Misfit lamellar and modulated Structure, with high thermopower



Two monoclinic sub-systems:
S1 with a ~ 4.8Å, b_1 ~ 4.5Å, c ~ 10.8Å et β ~ 98° (NaCl-type)
S2 with a ~ 4.8Å, b_2 ~ 2.8Å, c ~ 10.8Å et β ~ 98° (CdI_2-type)

$\Gamma=\sigma_{ab}/\sigma_c$~10 ⇒ Texture

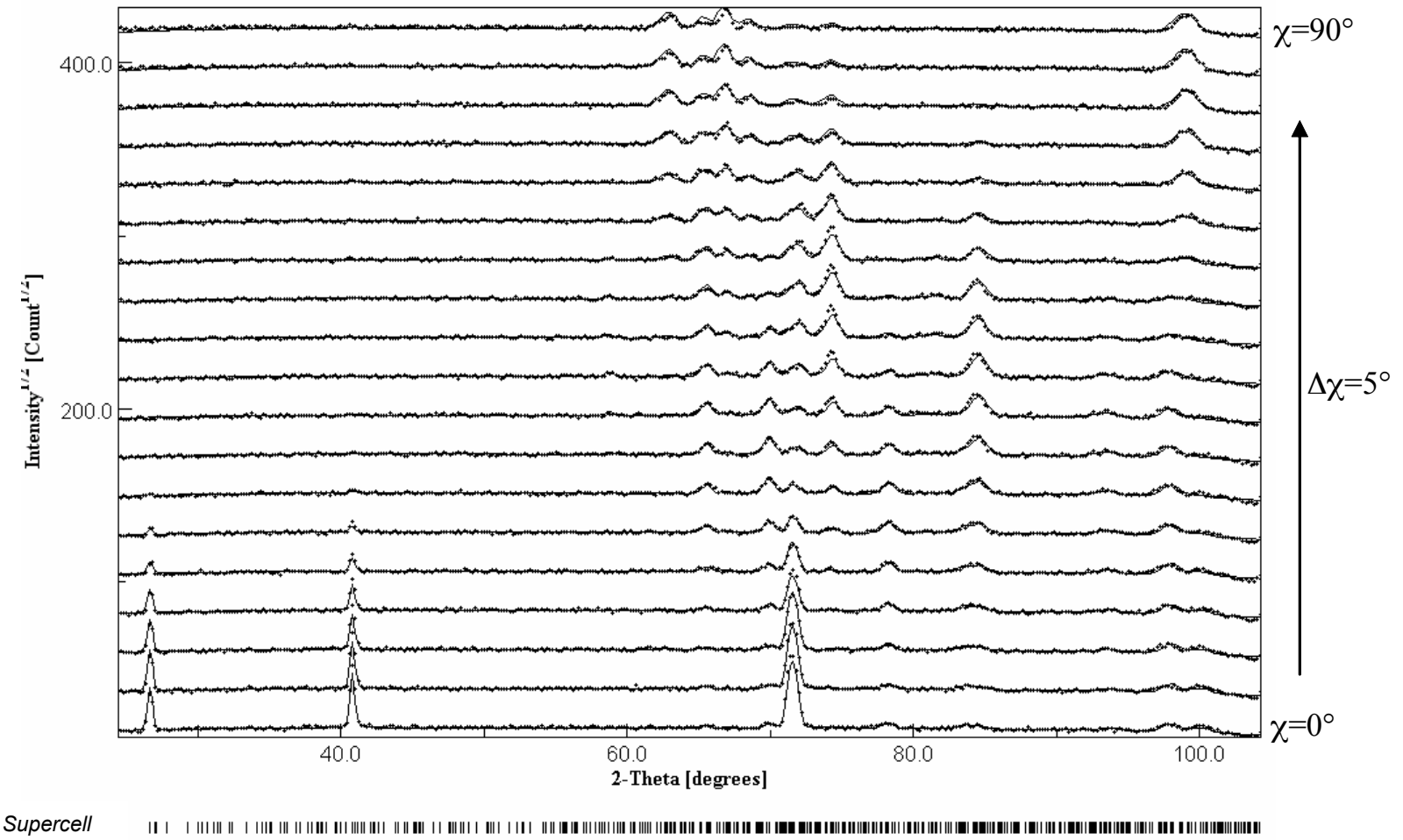




Magnetic alignment and Templated Growth method

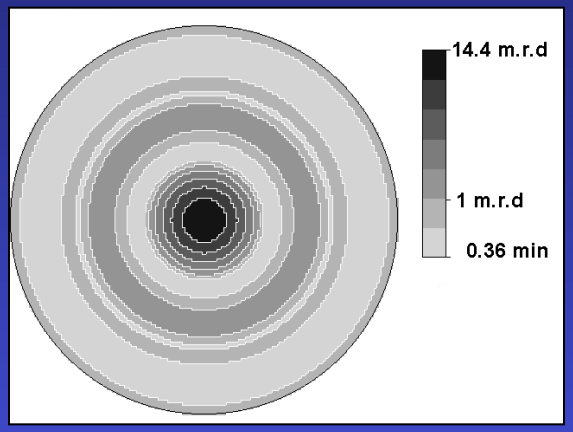
Analysis:

- neutrons
- 3D Supercell: a=4.8309Å, b~8b1~13b2~36.4902Å, c=10.8353Å, β=98.13°
174 atoms/cell
-Sample : 0.6 cm^3

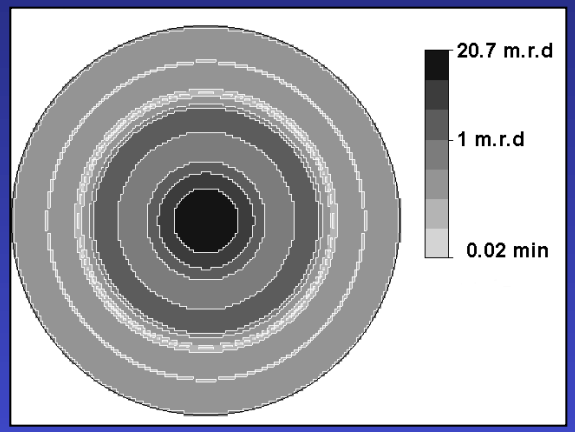

χ=90°
Δχ=5°
χ=0°
400.0
200.0
Intensity^1/2 [Count^1/2]
40.0
60.0
80.0
100.0
2-Theta [degrees]
Supercell

RP=19.7%, Rw=11.9%

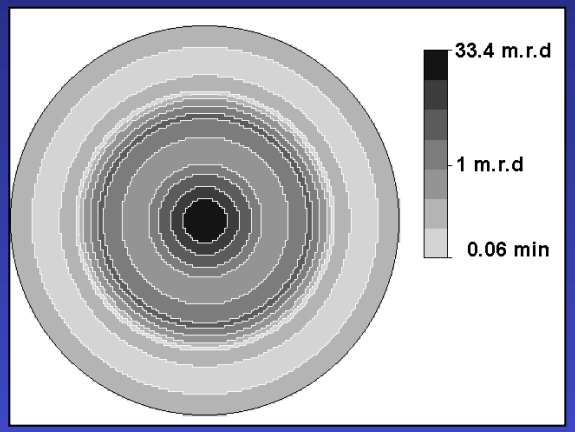
9.8 MPa for 2 h

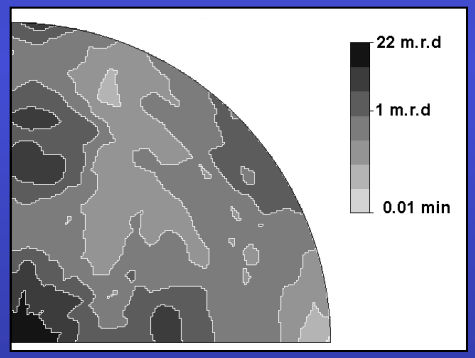


19.6 MPa for 6 h

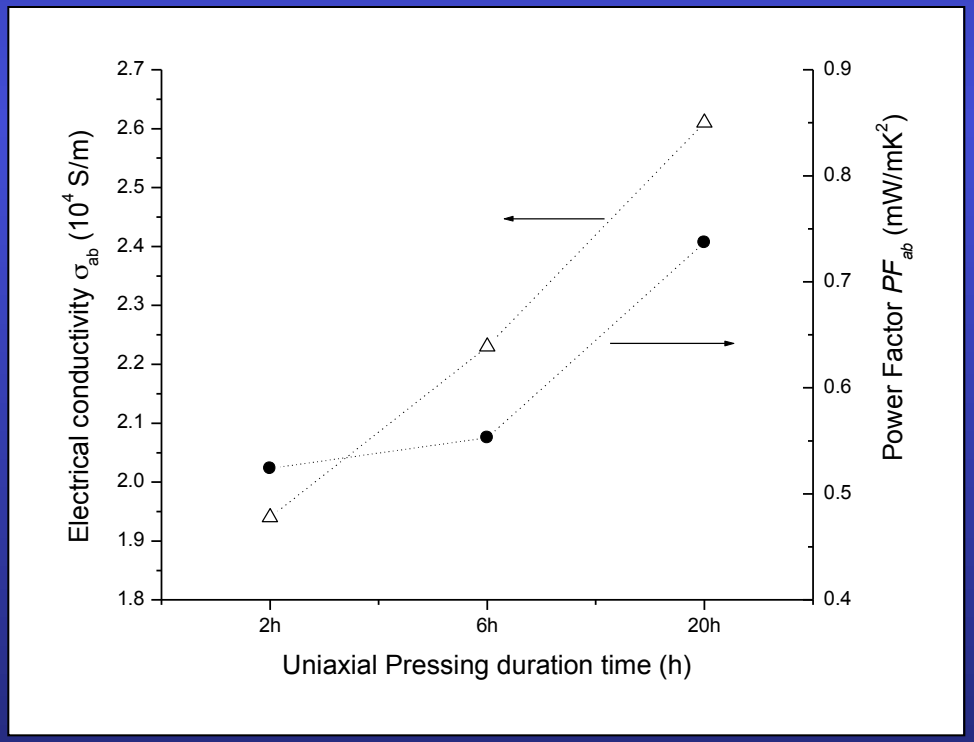


19.6 MPa for 20 h



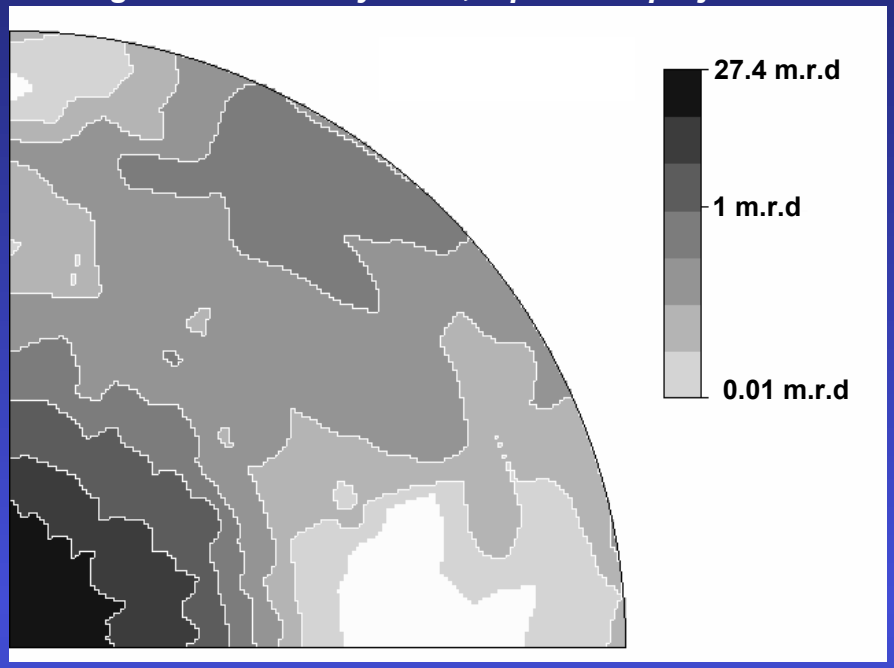


Templated Growth Method



Magnetic Alignment

Logarithmic density scale, equal area projection



- magnetic alignment really efficient to obtain strong textures
- combined analysis of modulated structures possible

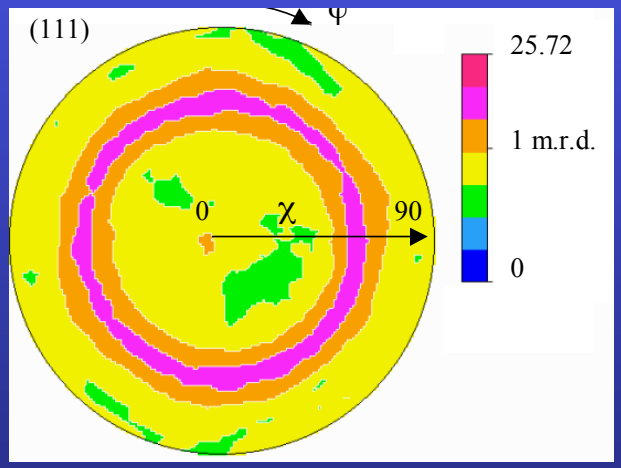
Ferroelectric PCT films

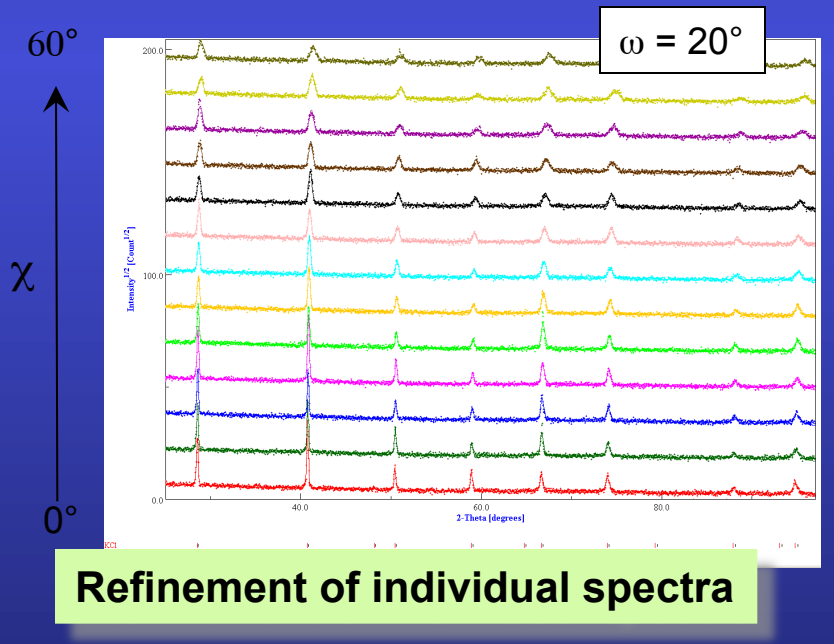
J. Ricote, DMF-Madrid

thin films:

$(Ca_{0.24}Pb_{0.76})TiO_3$ sol-gel synthesised solutions deposited by spin coating on a substrate of $Pt/TiO_2/Si$, with and without a treatment at 650ºC for 30 min.

All films are crystallised at 700ºC for 50 s by Rapid Thermal Processing (RTP; 30ºC/s). A series is also recrystallised at 650ºC for 1 to 3 h.





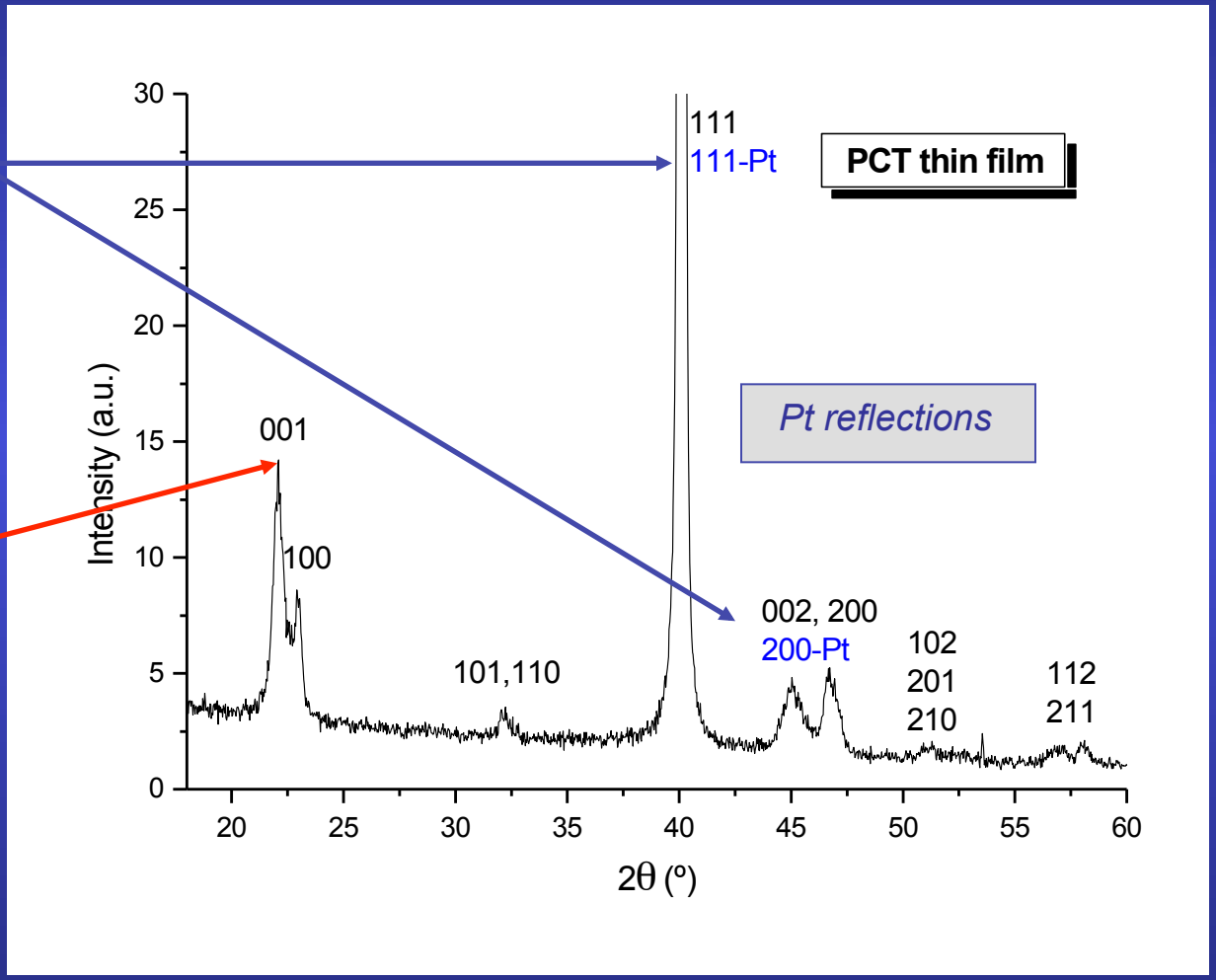
Refinement of individual spectra

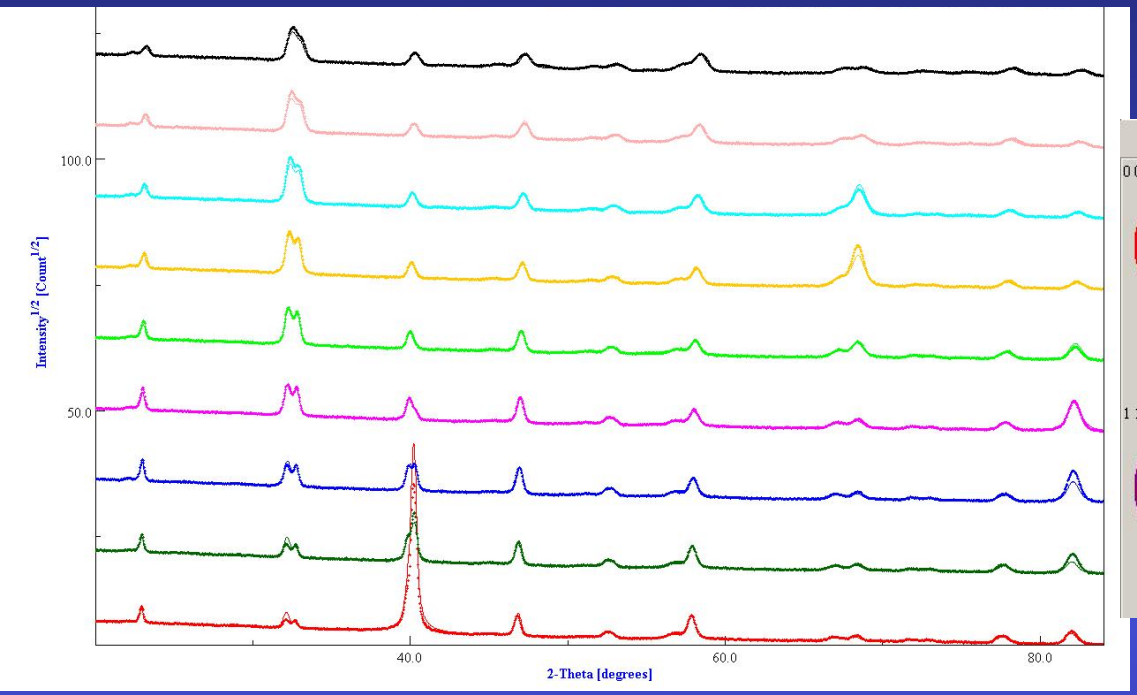
Limitations of the simple Quantitative Texture Analysis

Structural parameters are difficult to obtain due to:

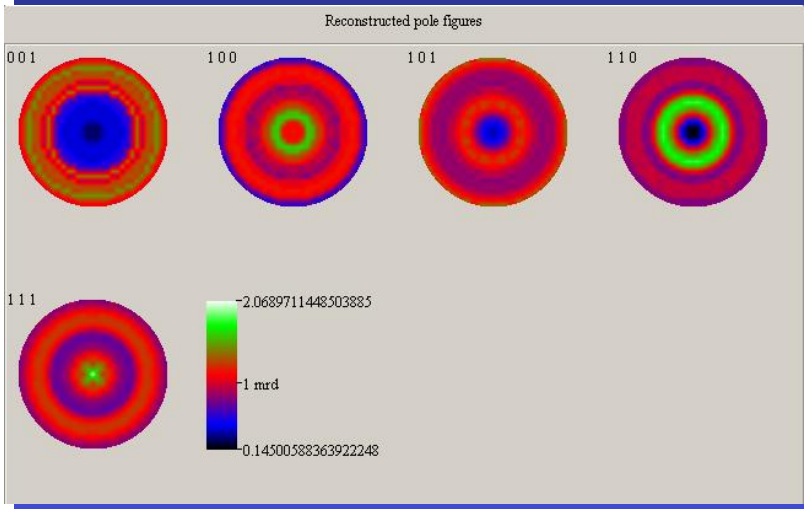
Substrate influence:
overlapping of reflections from the film and the substrate

TEXTURE effects:
peaks that do not appear at low χ angles



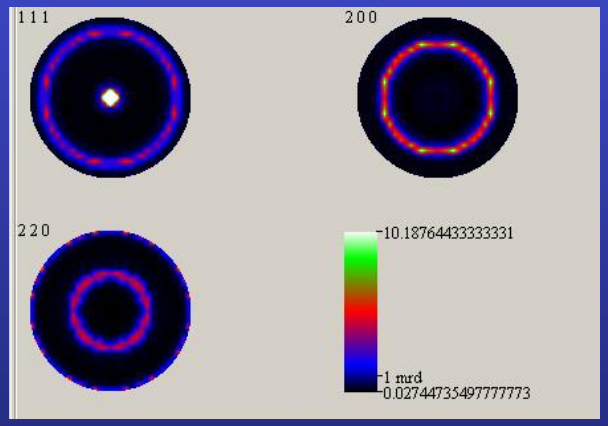


PCT



a = 3.9156(1) Å
c = 4.0497(3) Å
T = 2525(13) Å
t_{iso} = 390(7) Å
ε = 0.0067(1) rms

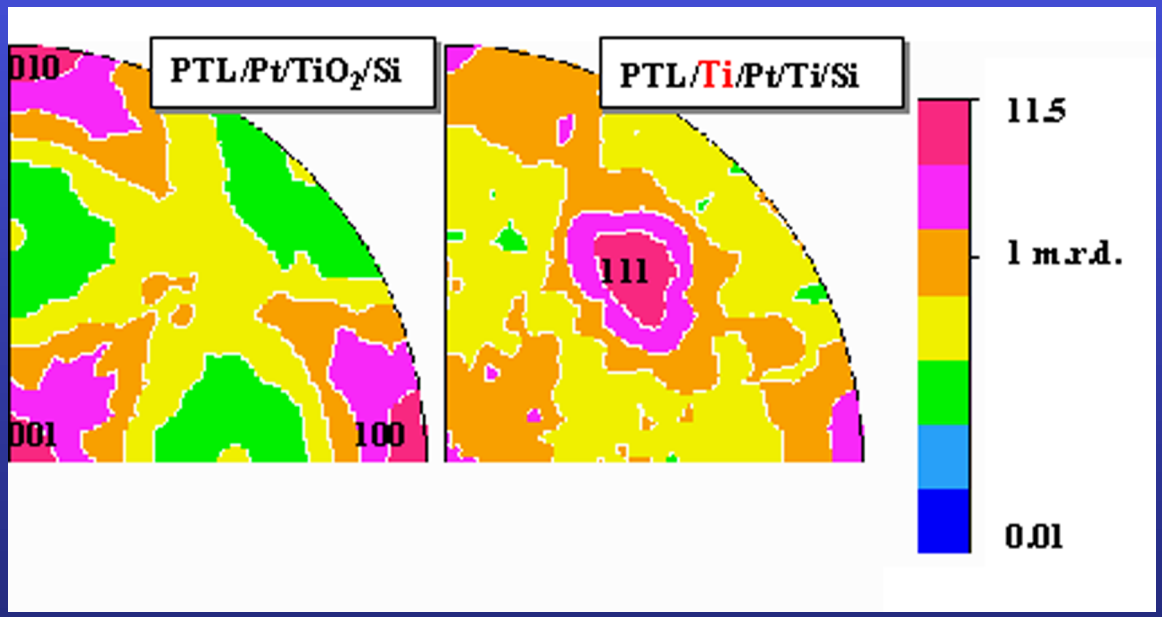
Pt



a = 3.9108(1) Å
T = 457(3) Å
t_{iso} = 458(3) Å
ε' = 0.0032(1) rms

R_W = 13%; R_B = 12%; R_{exp} = 22%.(Rietveld)
R_W = 5%; R_B = 6% (E-WIMV)

Atom	Occupancy	x	y	z
Pb	0.76	0.0	0.0	0.0
Ca	0.24	0.0	0.0	0.0
Ti	1.0	0.5	0.5	0.477(2)
O1	1.0	0.5	0.5	0.060(2)
O2	1.0	0.0	0.5	0.631(1)



Structural parameters

Pt layer

	a (Å)	thickness (nm)	R factors (%)
non-treated substrate			
Pt	3.9108(1)	45.7(3)	R_W=13, R_B=12, R_{exp}=22
annealed substrate			
Pt	3.9100(4)	46.4(3)	R_W=8, R_B=14, R_{exp}=21
Pt (Recryst. 1h)	3.9114(2)	47.8(3)	R_W=9, R_B=20, R_{exp}=21
Pt (Recryst. 2h)	3.9068(1)	46.9(3)	R_W=9, R_B=14, R_{exp}=22
Pt (Recryst. 3h)	3.9141(4)	47.5(9)	R_W=27, R_B=12, R_{exp}=21

Annealing of the substrate does not introduce significant variations on the structure of the Pt layer

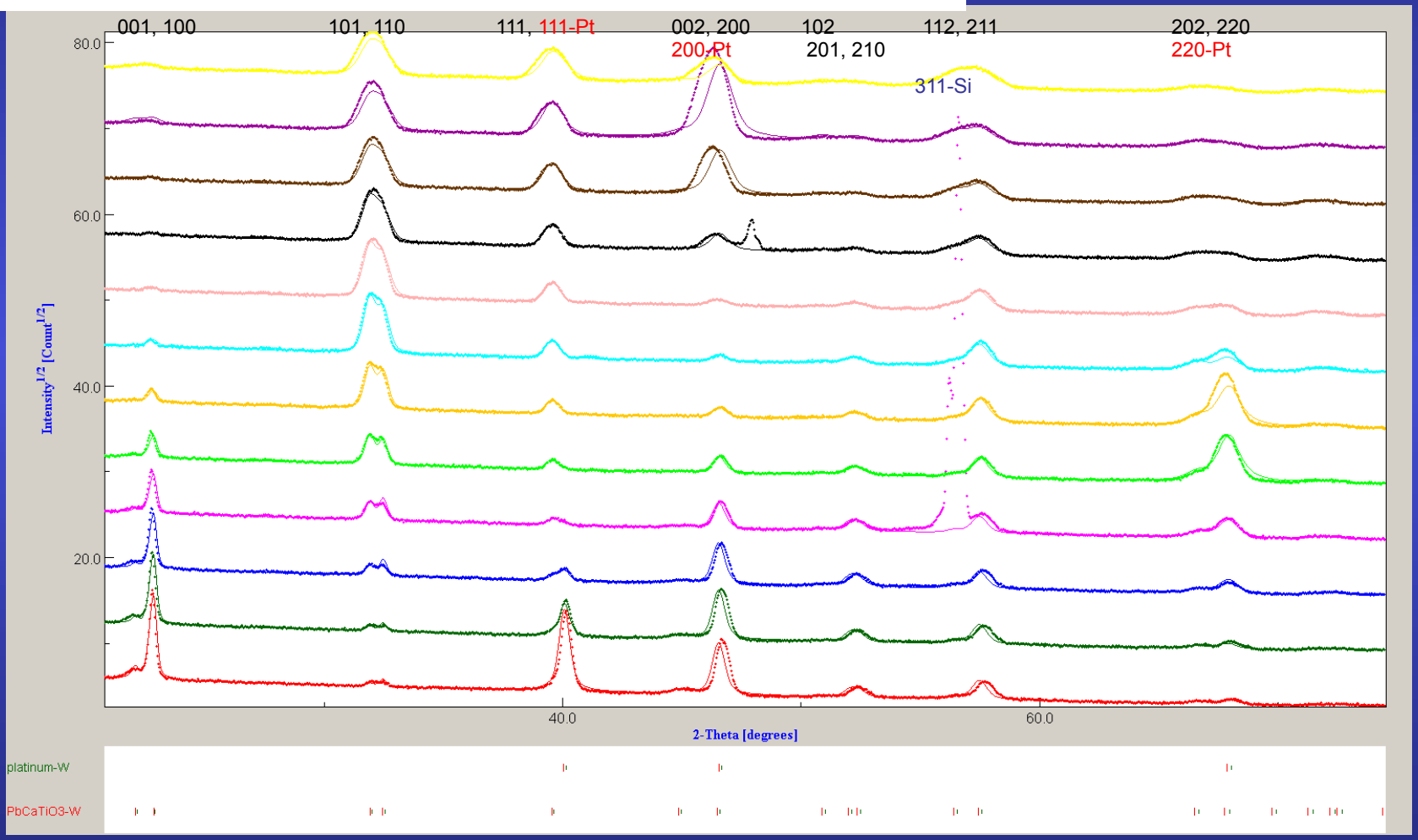
PTC film

	a (Å)	c (Å)	thickness (nm)
on non-treated substrate			
PCT	3.9156(1)	4.0497(6)	272.5(13)
on annealed substrate			
PCT	3.8920(6)	4.0187(8)	279.0(9)
PCT (Recryst. 1h)	3.8929(2)	4.0230(4)	266.1(11)
PCT (Recryst. 2h)	3.8982(2)	4.0227(4)	258.4(9)
PCT (Recryst. 3h)	3.9001(4)	4.0228(11)	253.6(29)

Recrystallisation reduces the stress on the film, and, increases the lattice parameters

Structural, microstructural and texture quantitative characterisation of ferroelectric thin films by the combined method

Analysis of the X-ray diffraction diagrams of a PCT film on $Pt/TiO_2/Si$



R_W = 13%; R_B = 12%; R_{exp} = 22%.(Rietveld)
R_W = 5%; R_B = 6% (E-WIMV)

Substrate influence on Residual Stress and Texture

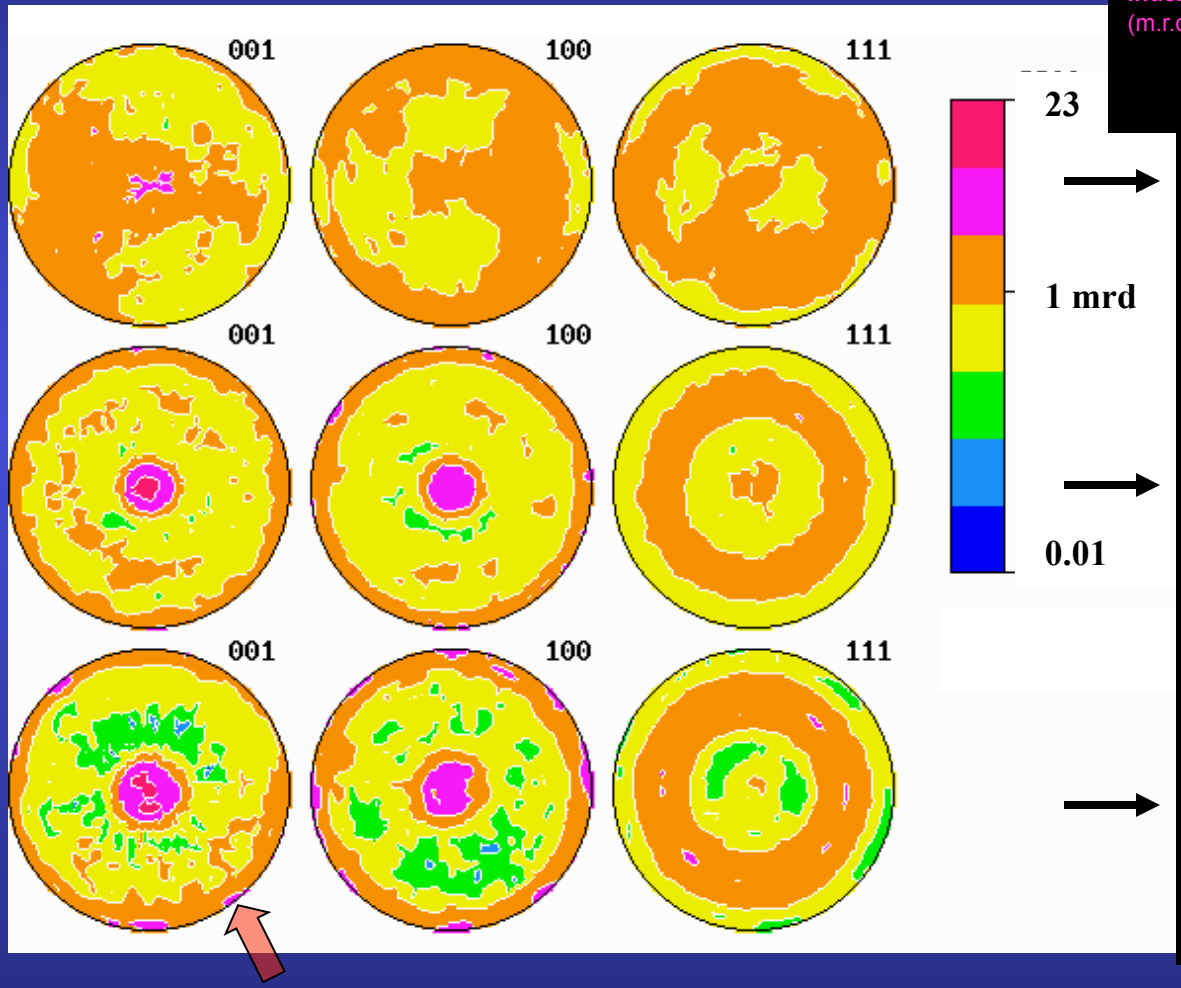
Tensile stress

PCT on $Pt/TiO_2/(100)Si$

PCT on $Pt/(100)MgO$

PCT on $Pt/(100)SrTiO_3$

Compressive stress



Texture Index (m.r.d.^{2})	Pyroelectric Coefficient (10^{-8}C cm^{-2} K^{-1})
2.1	0.3
5.1	1.5
7.9	1.1

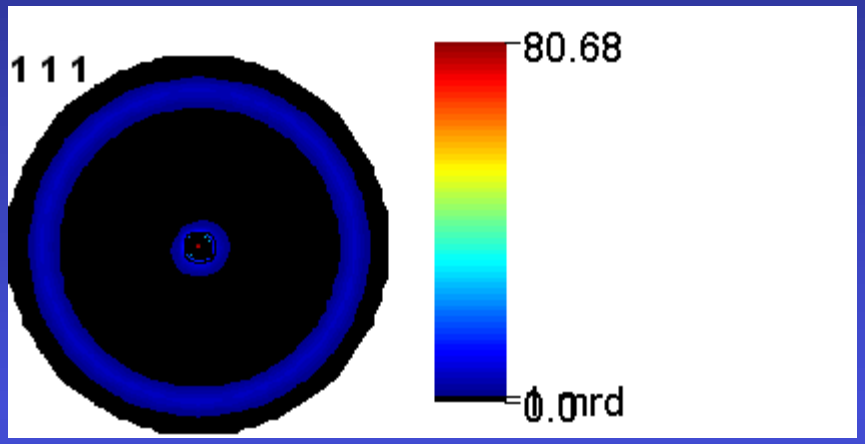
Enhancement of <001> texture

Compliance coefficients [10^{-3} GPa^{-1}]	$PbTiO_3$ single crystal (data set A)	Film random orientation	PCT-Si <001> contrib≈17%	PLT <001> contrib.≈49%	PCT-Mg <001> contrib.≈68%
s_{11}	6.5	10.1	10.5	10.0	9.7
s_{22}	6.5	10.0	10.5	10.0	9.7
s_{33}	33.3	9.8	9.0	10.3	11.3
s_{44}	14.5	13.2	12.8	12.9	13.1
s_{55}	14.5	13.2	12.8	13.0	13.1
s_{66}	9.6	13.4	14.0	13.5	12.7
s_{12}	-0.35	-3.3	-3.5	-3.2	-3.0
s_{21}	-0.35	-3.3	-3.5	-3.2	-3.0
s_{13}	-7.1	-3.2	-3.1	-3.4	-3.6
s_{31}	-7.1	-3.2	-3.1	-3.4	-3.6
s_{23}	-7.1	-3.2	-3.1	-3.4	-3.6
s_{32}	-7.1	-3.2	-3.1	-3.4	-3.6
s_{33}/s_{11}	5.1	0.97	0.86	1.03	1.16
s_{13}/s_{12}	20.3	0.97	0.89	1.06	1.20

Geometric mean average + biaxial stress state

Ferroelectric PMN-PT films

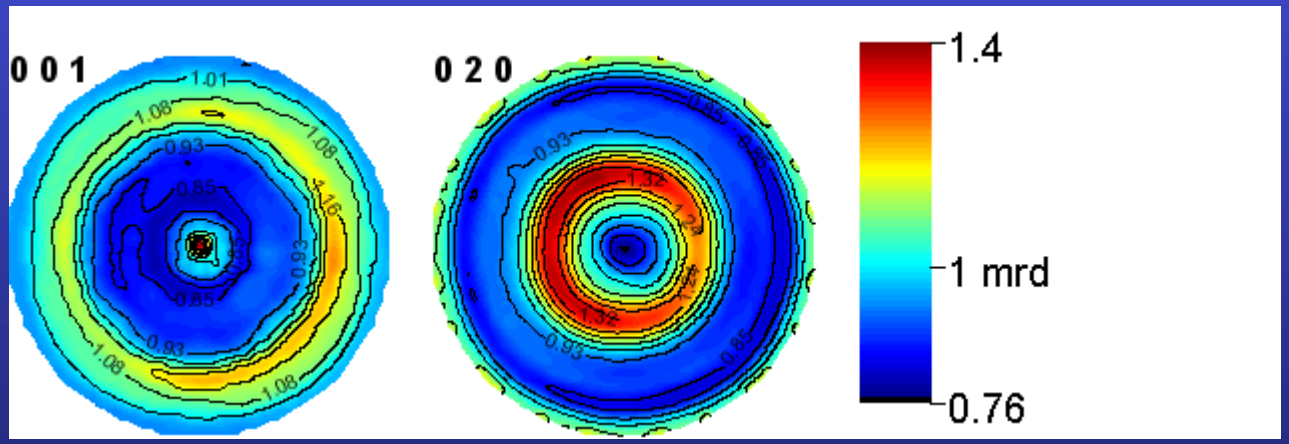
J. Ricote, DMF-Madrid



Pt

a = 3.91172(1) Å
T = 583(5) Å
t_{iso} = 960(1) Å
ε = 0.0032(1) rms
σ_{11}= 0.639(1) GPa
σ_{22}= 0.651(1) GPa
σ_{12}= -0.009(1) GPa

$Pb_{0.7}(Mg_{1/3}Nb_{2/3})O_3$-$Pb_{0.3}TiO_3$ /$Ti0_2$/Pt/Si-(100)



a = 5.67858(9) Å
b = 5.69038(9) Å
c = 3.99558(4) Å
β = 90.392(1) Å
T = 1322(9) Å
t_{iso} = 1338(2) Å
ε = 0.0067(1) rms

Si nanocrystalline thin films

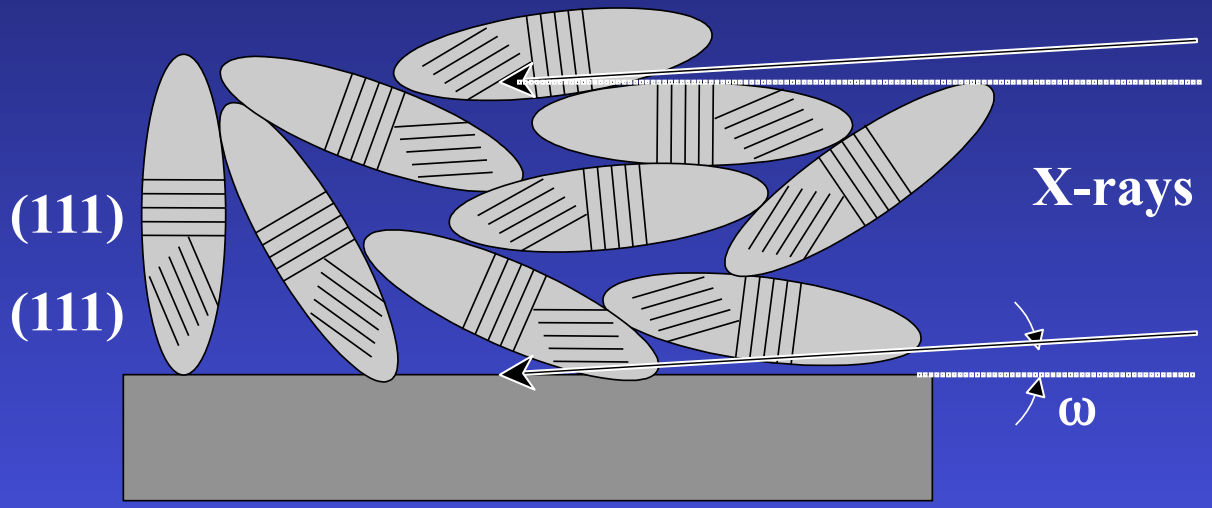
M. Morales, SIFCOM-Caen

Silicon thin films deposition by reactive magnetron sputtering:

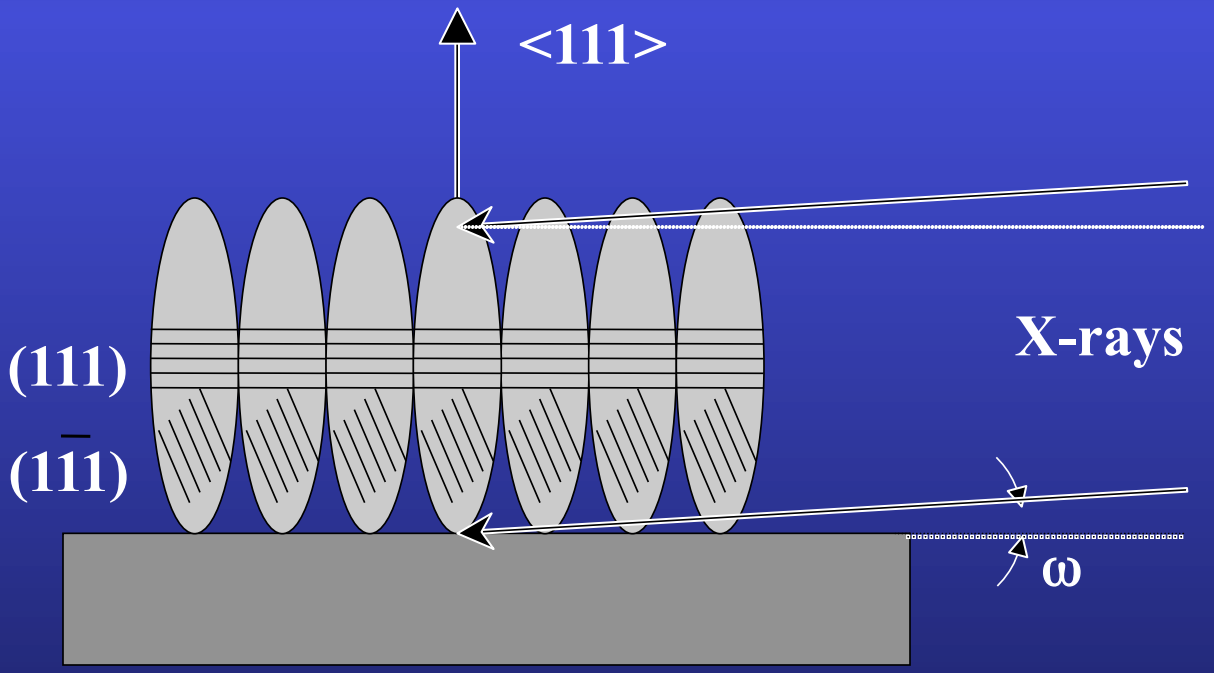
- power density 2W/cm^2
- total pressure: $p_{total} = 10^{-1}$ Torr
- plasma mixture: H_2 / Ar, $pH_2 / p_{total} = 80$ %
- temperature: 200°C
- substrates: amorphous SiO_2 (a-SiO_2)
  (100)-Si single-crystals
- target-substrate distance (d)
  - a-SiO_2 substrates: d = 4, 6, 7, 8, 10, 12 cm
    films A, B, C, D, E, F
  - (100)-Si: d = 6, 12 cm
    films G, H

Aim: quantum confinement, photoluminescence properties

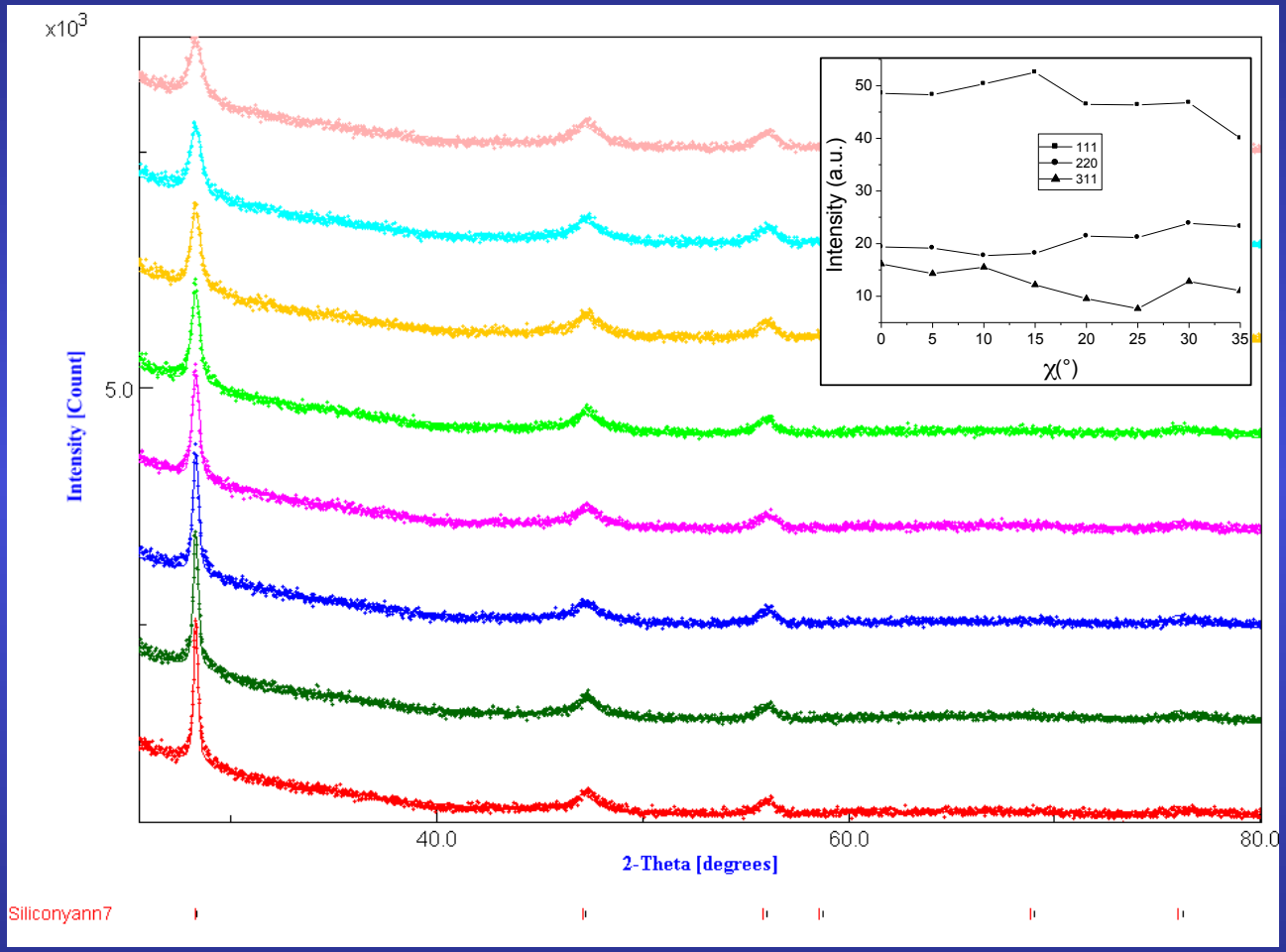
Isotropic



Textured



Typical refinement



χ = 35°

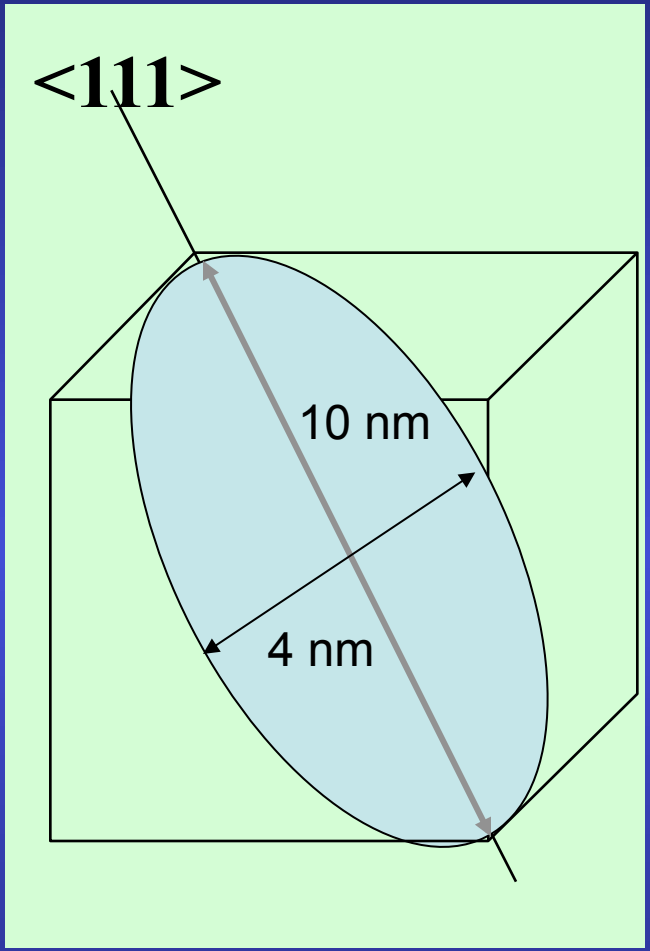
χ = 0°

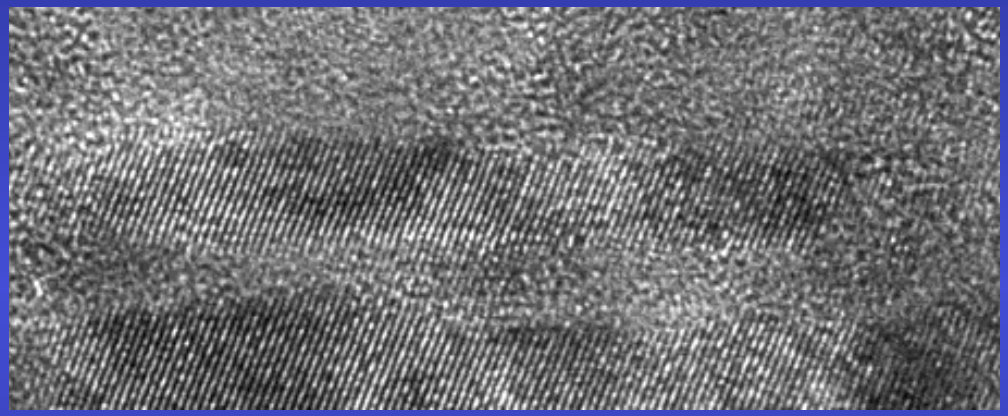
broad, anisotropic diffracted lines, textured samples

Refinement Results

Sample	d (cm)	a (Å)	RX thickness (nm)	Anisotropic sizes (Å)			Texture parameters			Reliability factors (%)			
				<111>	<220>	<311>	Maximum (m.r.d.)	minimum (m.r.d.)	Texture index F^2 (m.r.d^2)	RP_0	R_w	R_B	R_{exp}
A	4	5.4466 (3)	---	94	20	27	1.95	0.4	1.12	1.72	4.0	3.7	3.5
B	6	5.4439 (2)	711 (50)	101	20	22	1.39	0.79	1.01	0.71	4.9	4.3	4.2
C	7	5.4346 (4)	519 (60)	99	40	52	1.72	0.66	1.05	0.78	4.3	4.0	3.9
D	8	5.4461 (2)	1447 (66)	100	22	33	1.57	0.63	1.04	0.90	5.5	4.6	4.5
E	10	5.4462 (2)	1360 (80)	98	20	25	1.22	0.82	1.01	0.56	5.0	3.9	4.0
F	12	5.4452 (3)	1110 (57)	85	22	26	1.59	0.45	1.05	1.08	4.2	3.5	3.7
G	6	5.4387 (3)	1307 (50)	89	22	28	1.84	0.71	1.01	1.57	5.2	4.7	4.2
H	12	5.4434 (2)	1214 (18)	88	22	24	2.77	0.50	1.12	2.97	5.0	4.5	4.3

Mean anisotropic shape



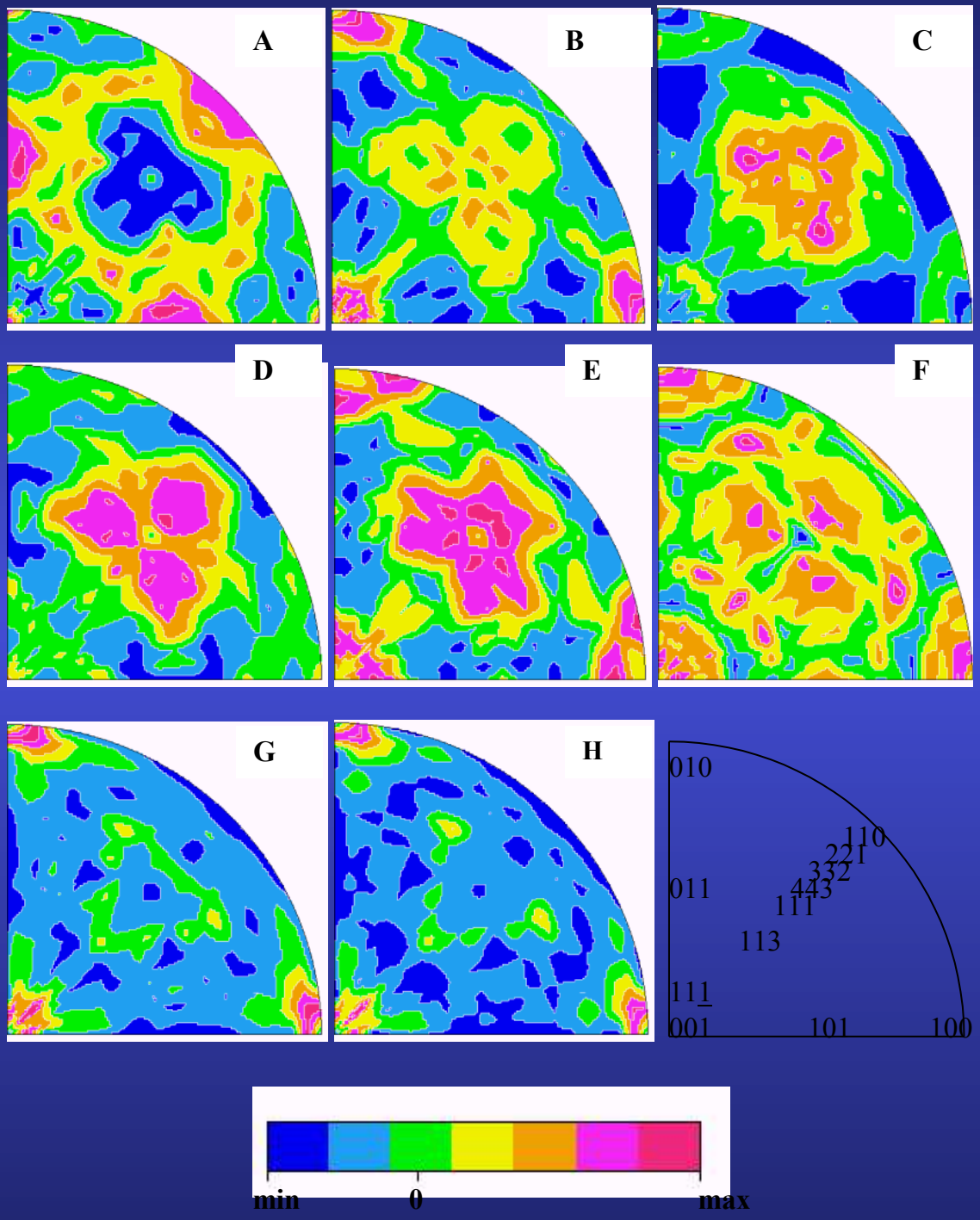


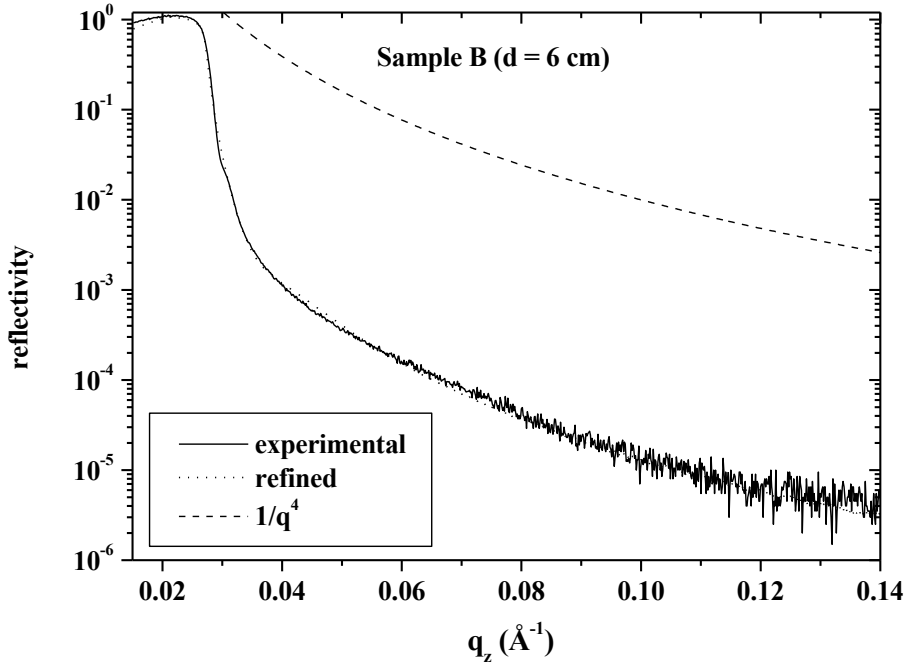
Schematic of the mean crystallite shape for Sample D represented in a cubic cell, as refined using the Popa approach and exhibiting a strong elongation along <111>, and TEM image

001 Inverse Pole Figures

a-SiO_2

(100)-Si



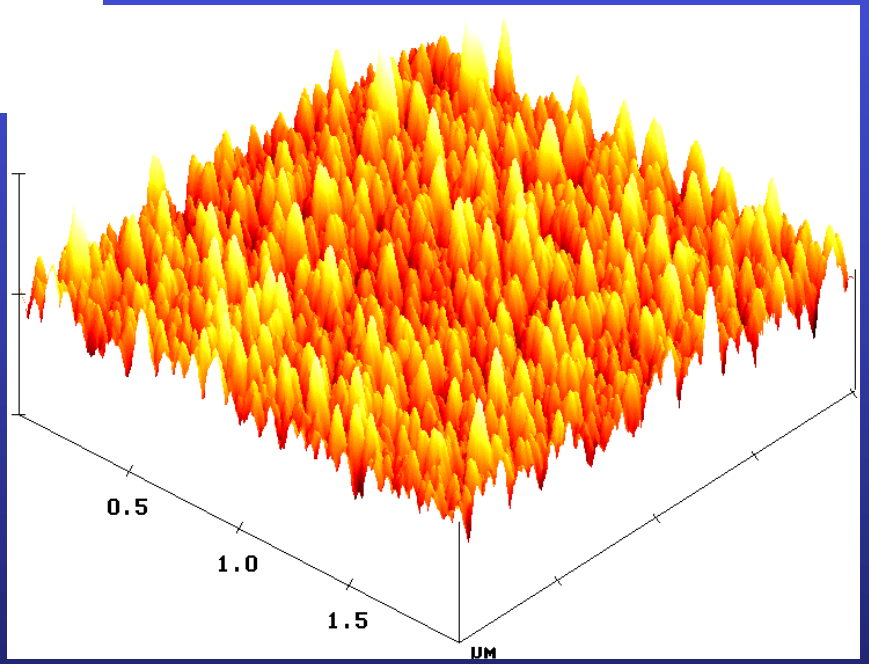


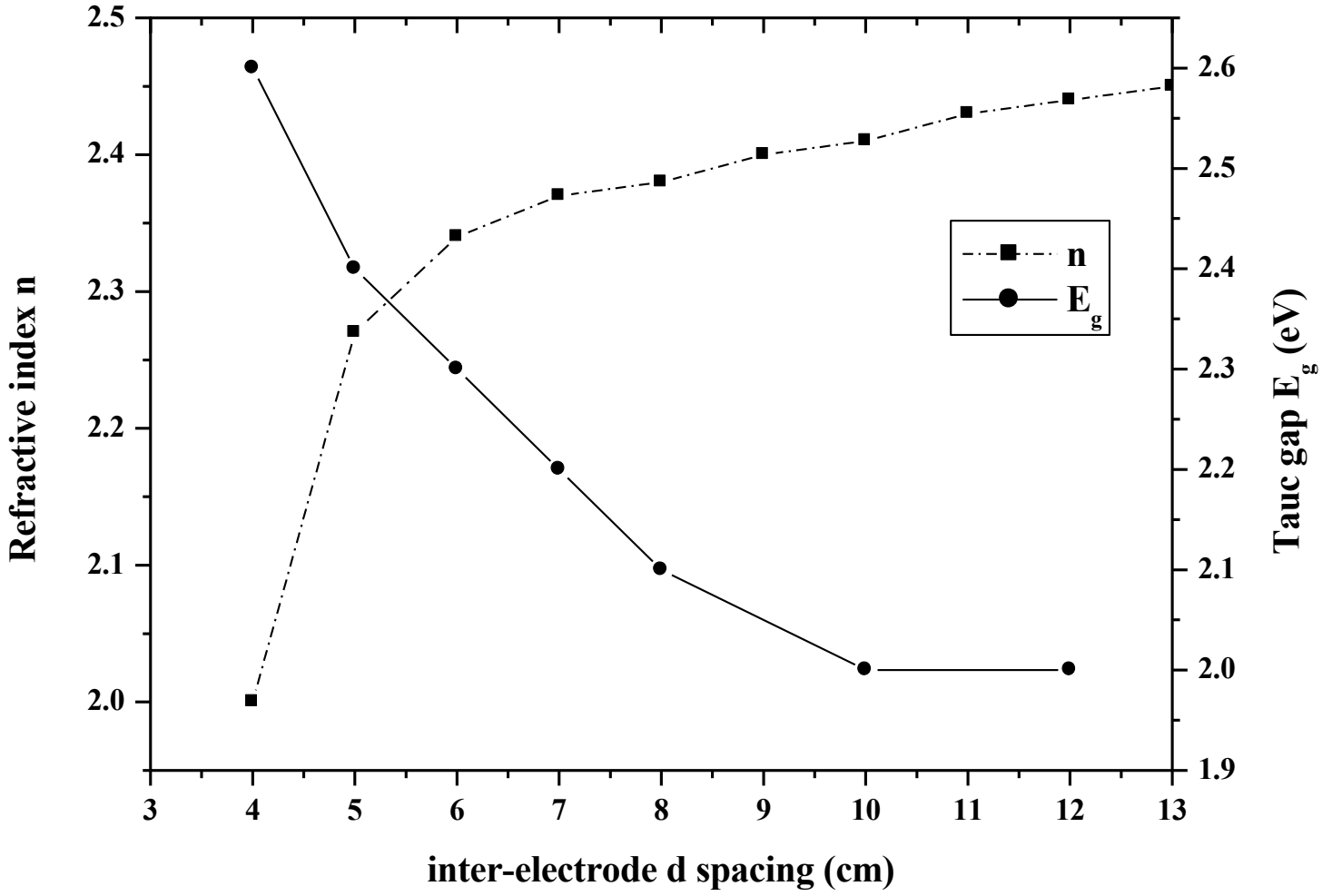
XRR:

Roughness governed

AFM:

homogeneous roughness

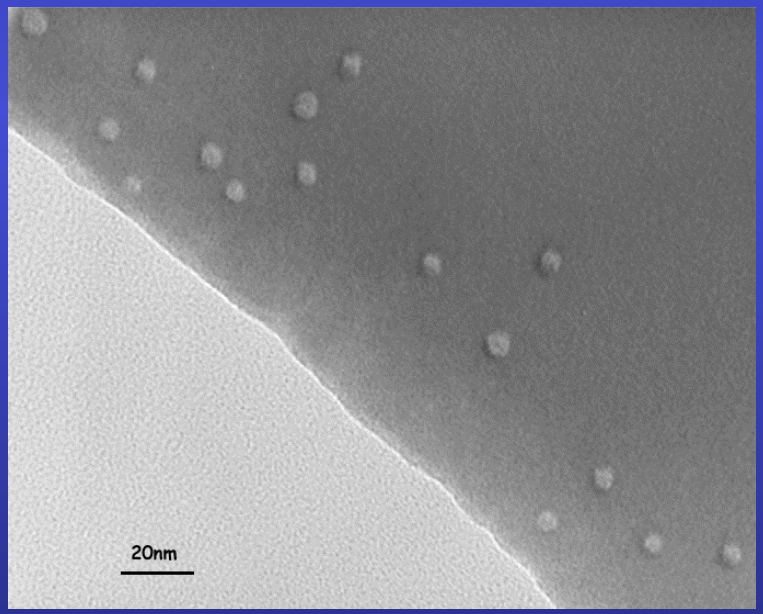



Refractive index n
Tauc gap E_g (eV)
inter-electrode d spacing (cm)
n
E_g
2.5
2.4
2.3
2.2
2.1
2.0
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
3
4
5
6
7
8
9
10
11
12
13

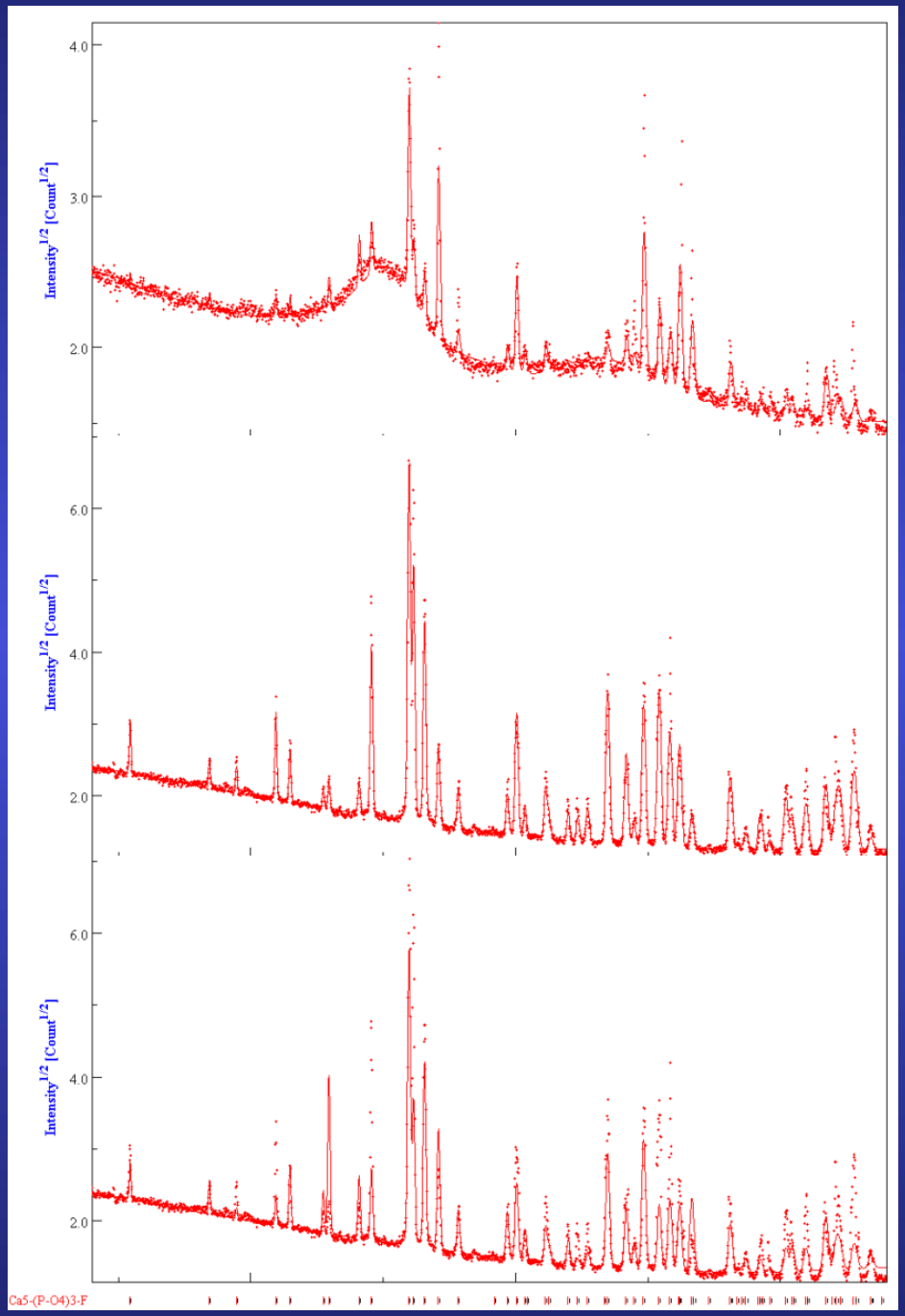
Irradiated FluorApatite (FAp) ceramics

S. Miro, PhD CRISMAT

Self-recrystallisation under irradiation, depending on SiO_4 / PO_4 ratio (FAp / Nd-Britholite) and on irradiating species



TEM of FAp irradiated with 70 MeV, 10^{12} Kr cm^{-2} ions



texture corrected,

10^{13} Kr cm^{-2}

Virgin, with texture correction

Virgin, no texture correction

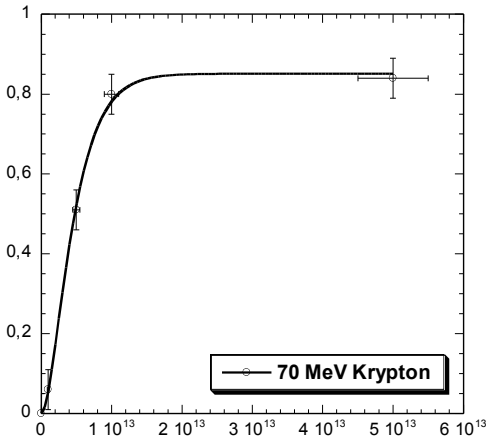
Fluence (ions.cm^{-2})	Vc/V (%)	A (Å)	c (Å)	<t> (nm)	$\Delta a/a_0$ (%)	$\Delta c/c_0$ (%)	R_w (%)	R_B (%)
0	100	9.3365(3)	6,8560(5)	294(22)	-	-	14.6	9.1
Kr								
10^{11}	100	-	-	-	-	-		
10^{12}	100	-	-	-	-	-		
5.10^{12}	49(1)	9.3775(9)	6.8912(8)	294(20)	0.44	0.53	24	15
10^{13}	20(1)	9.4236(5)	6.9105(5)	291(20)	0.94	0.82	9.9	6
5.10^{13}	14(1)	9.3160(4)	6.8402(5)	294(22)	-0.21	-0.22	10.5	5.9
I								
10^{11}	-	-	-	-	-	-		
5.10^{11}	86(2)	9.3603(3)	6.8790(5)	90(10)	0.26	0.35	23.9	15.1
10^{12}	-	-	-	-	-	-		
3.10^{12}	47(2)	9.3645(3)	6.8840(5)	91(6)	0.30	0.42	13.3	9
5.10^{12}	29.2(5)	9.3765(5)	6.8881(6)	77(11)	0.44	0.48	10.4	7.3
10^{13}	13.2(2)	9.3719(4)	6.8857(6)	82(9)	0.38	0.45	6.7	4.9

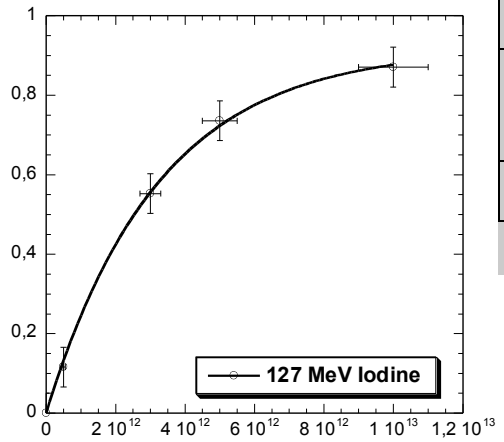
Single impact model associated to crystal size reduction

Cell parameters and volume increase, then relax

Amorphisation / recrystallisation competition: single or double impact

Amorphous/crystalline volume fraction (damaged fraction Fd = Va / V) as determined by x-ray diffraction





B

Fitting parameters	Krypton		Iodine
	Single impact $Fd = B(1- \exp(-A\phi t))$	**Double impact** $Fd = B(1 - (1+ A\phi t) \exp(-A\phi t))$	**Single impact** $Fd = B(1- \exp(-A\phi t))$
$A = \pi R^2$ (cm^2)	$1.85 \pm 0.15\ 10^{-13}$	$4.1 \pm 0.15\ 10^{-13}$	$3.3 \pm 0.15\ 10^{-13}$
Radius R (nm)	2.4 ± 0.2	3.6	3.2
B (Max.damage rate)	0.87	0.85 ± 0.2	0.92 ± 0.2
χ_2	0.013	0.0006	0.0004

Conclusions

a) Texture affects phase ratio and structure determination

b) Microstructure (crystallite size) affects texture (go to a)

c) Stresses shift peaks then affects structure and texture determination

d) Combined analysis may be a solution, unless you can destroy your sample or are not interested in macroscopic anisotropy ...

e) If you think you can destroy it, perhaps think twice

f) more information is always needed: local probes ...

g) www.ecole.ensicaen.fr/~chateign/texture/combined.pdf