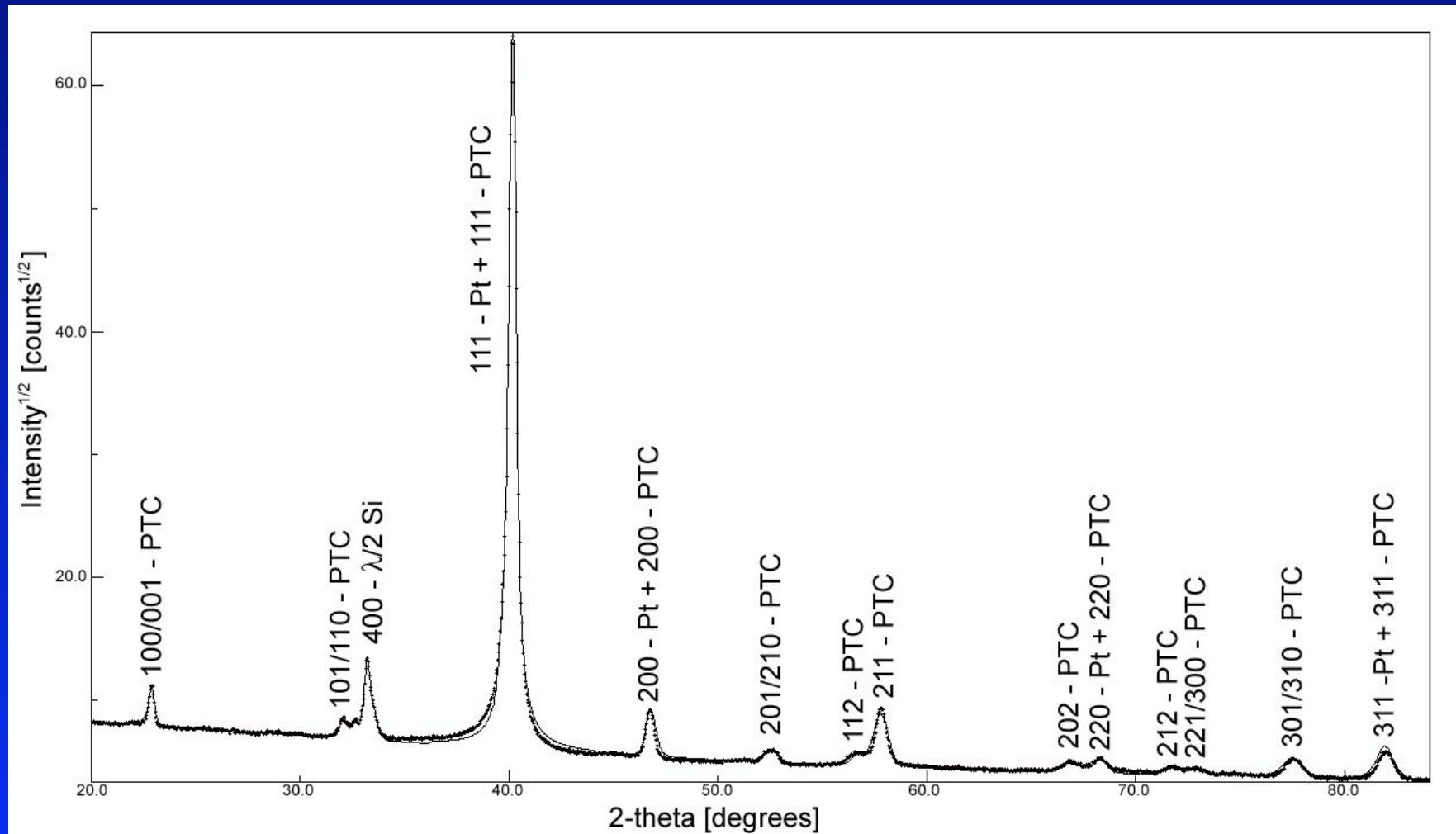
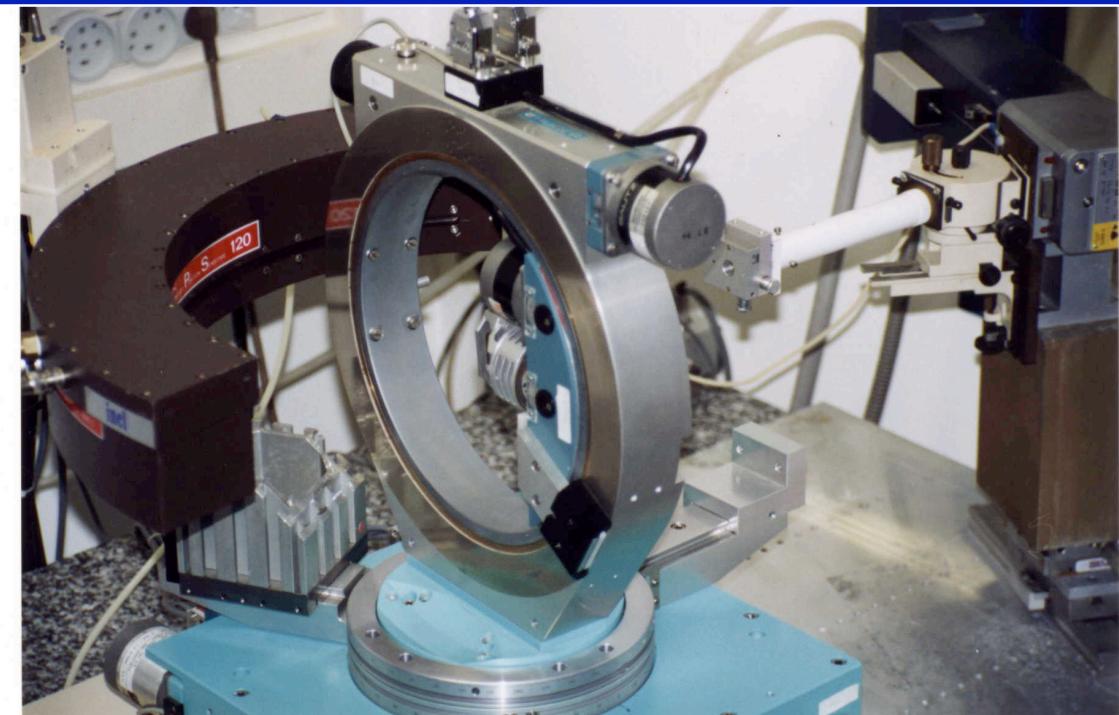


PTC film: the problem



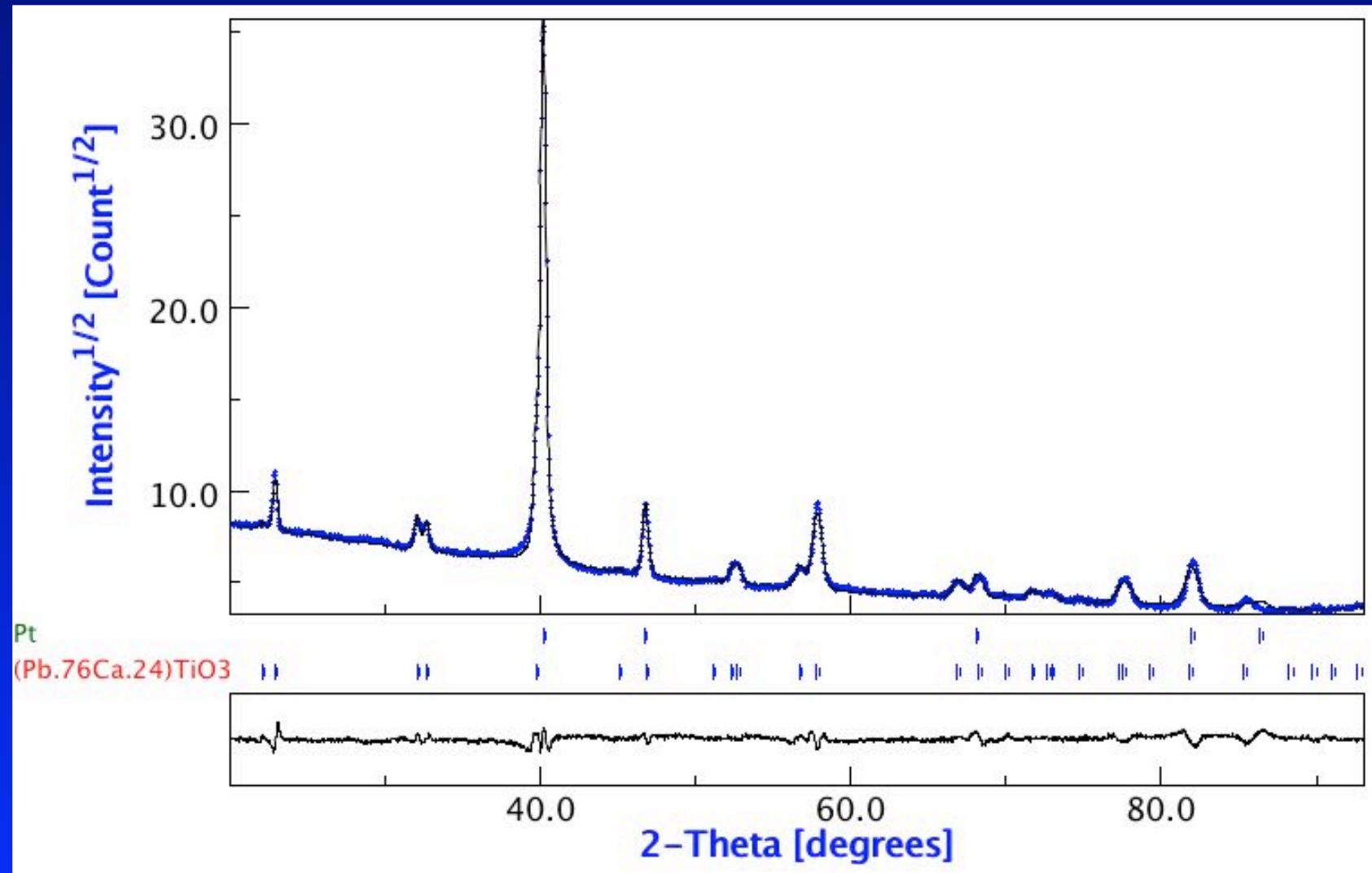
PTC film: experimental

- *Substrate: TiO₂/SiO₂/Si(100)*
- *400 nm of Pb_{0.76}Ca_{0.24}TiO₃ (PTC) film deposited by spin coating of a sol-gel solution (CSIC Madrid).*
- *50 nm of Pt buffer layer.*
- *Instrument: 120 degs curved position sensitive detector on a closed eulerian cradle, graphite primary monochromator (LPEC - Le Mans, France)*
- *Collected full spectra on a 5x5 degs grid in chi and phi. From 0 to 355 in phi and up to 50 deg in chi.*



The LPEC, Le Mans instrument

PTC and Pt phase separation



PTC film: harmonic texture model

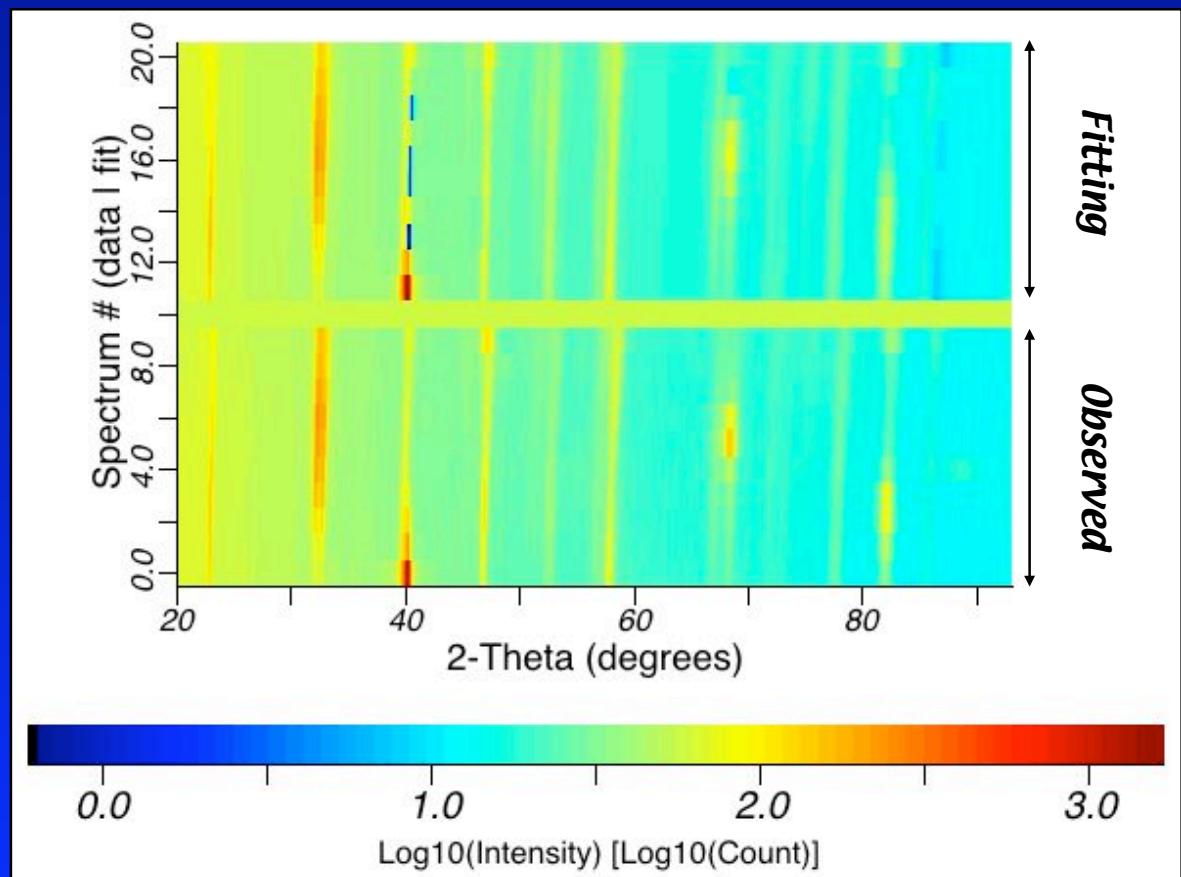
Triclinic sample symmetry: 1245 parameters only for PTC ($L_{max} = 22$)

Increasing sample symmetry to orthorhombic: 181 parameters

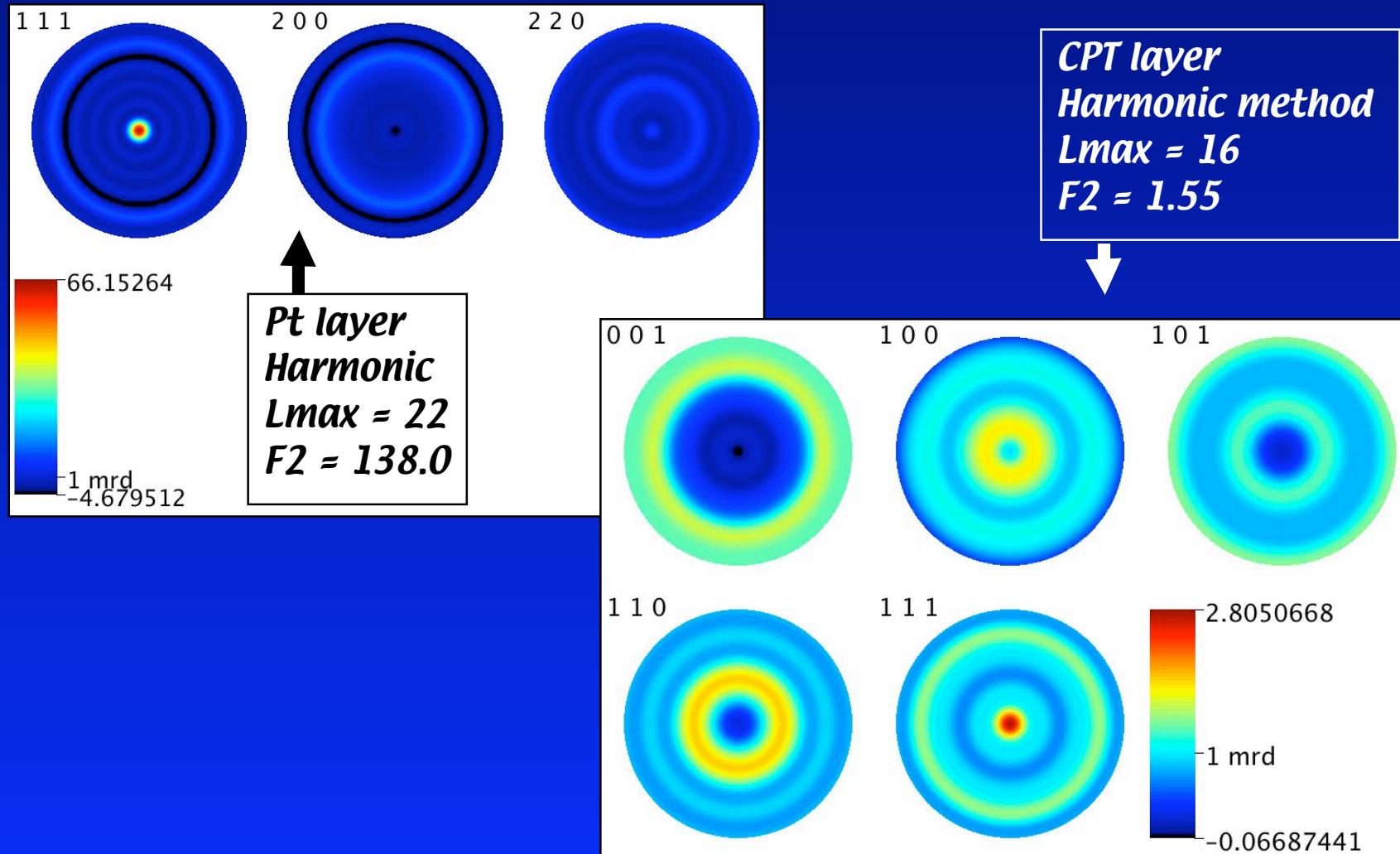
Reducing sample symmetry to fiber and L_{max} to 16: 24 parameters

For Pt layer: fiber texture, $L_{max} = 22 \rightarrow 15$ parameters

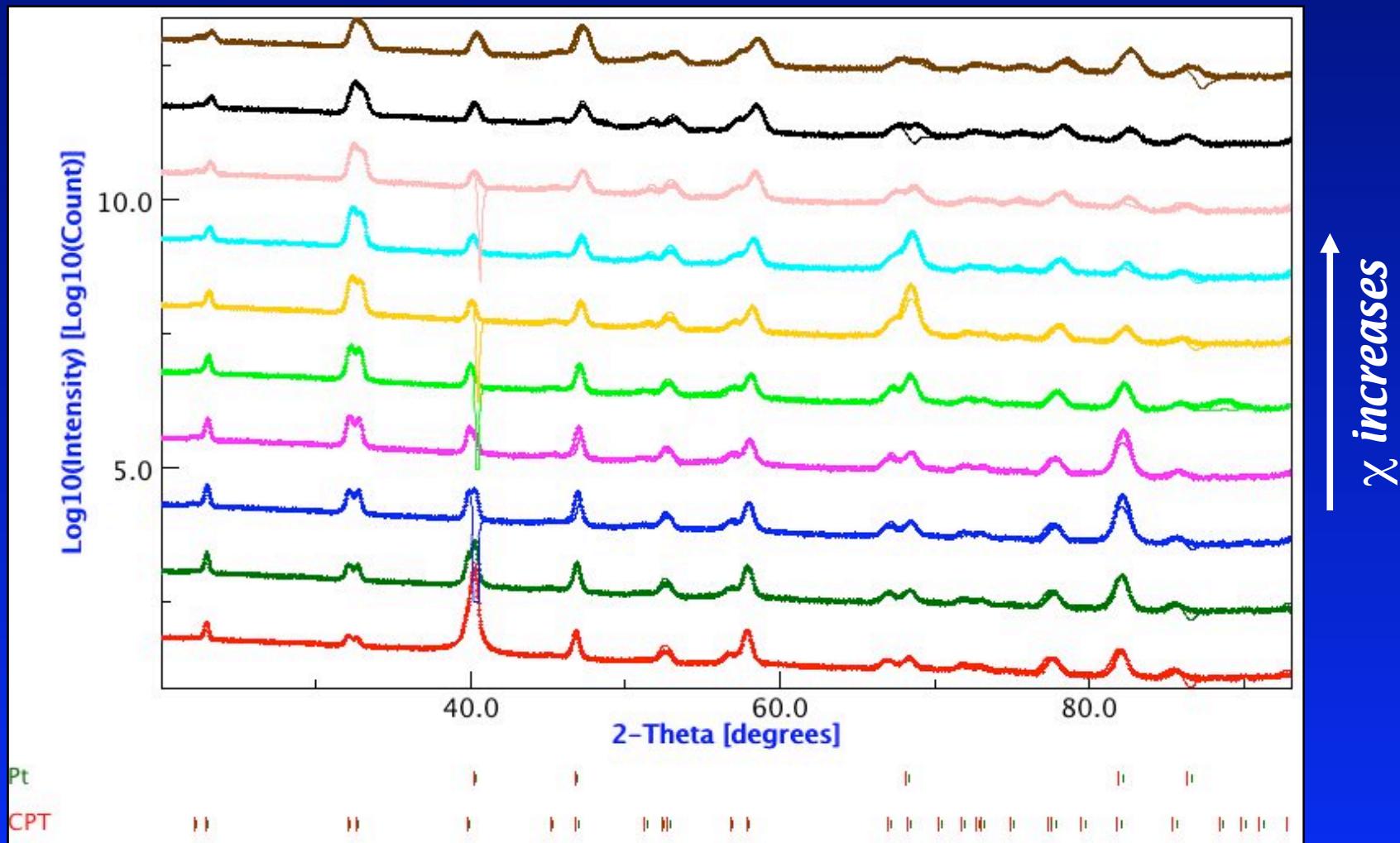
$R_w (\%) = 14.786048$



PTC film: harmonic reconstructed pole figures

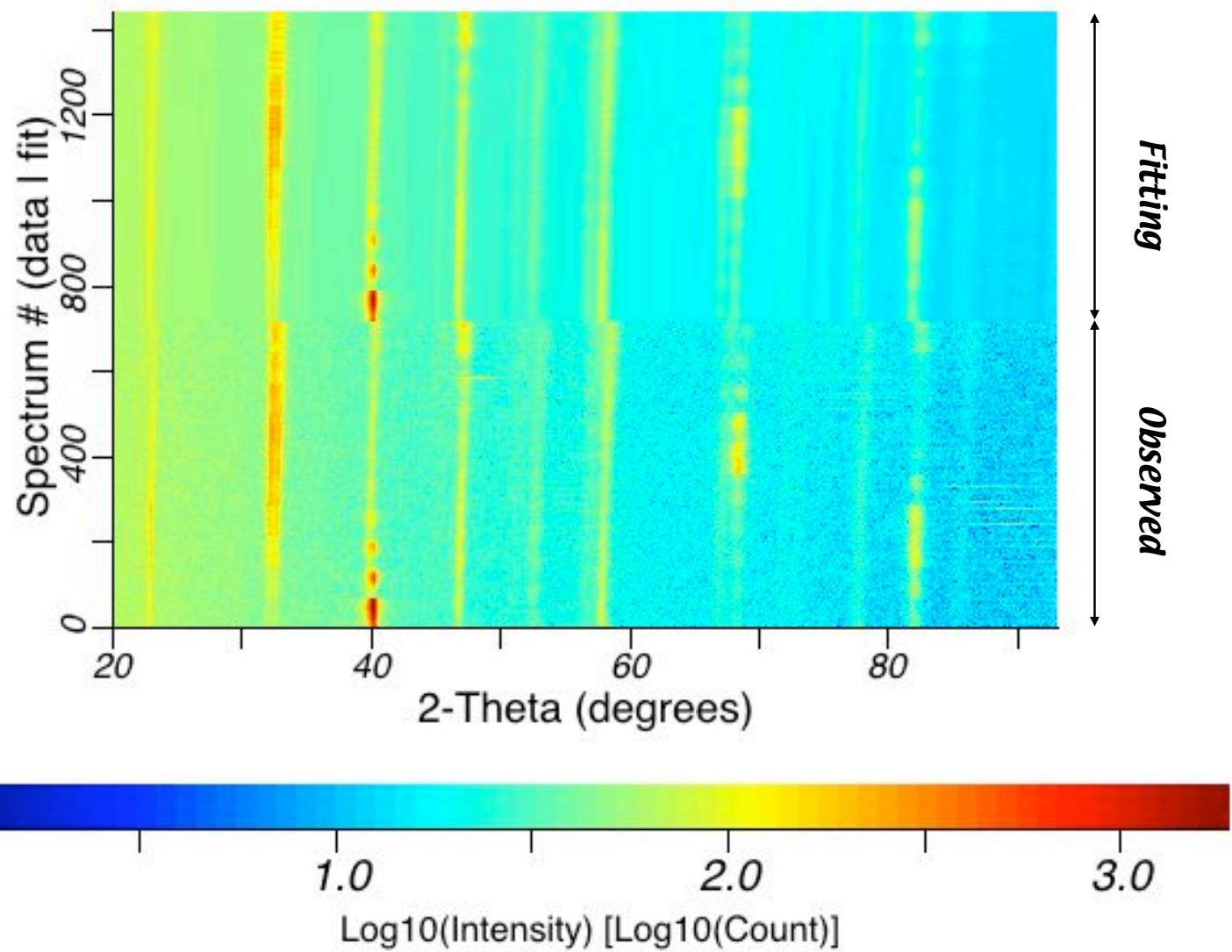


PTC film: harmonic fitting, the problem

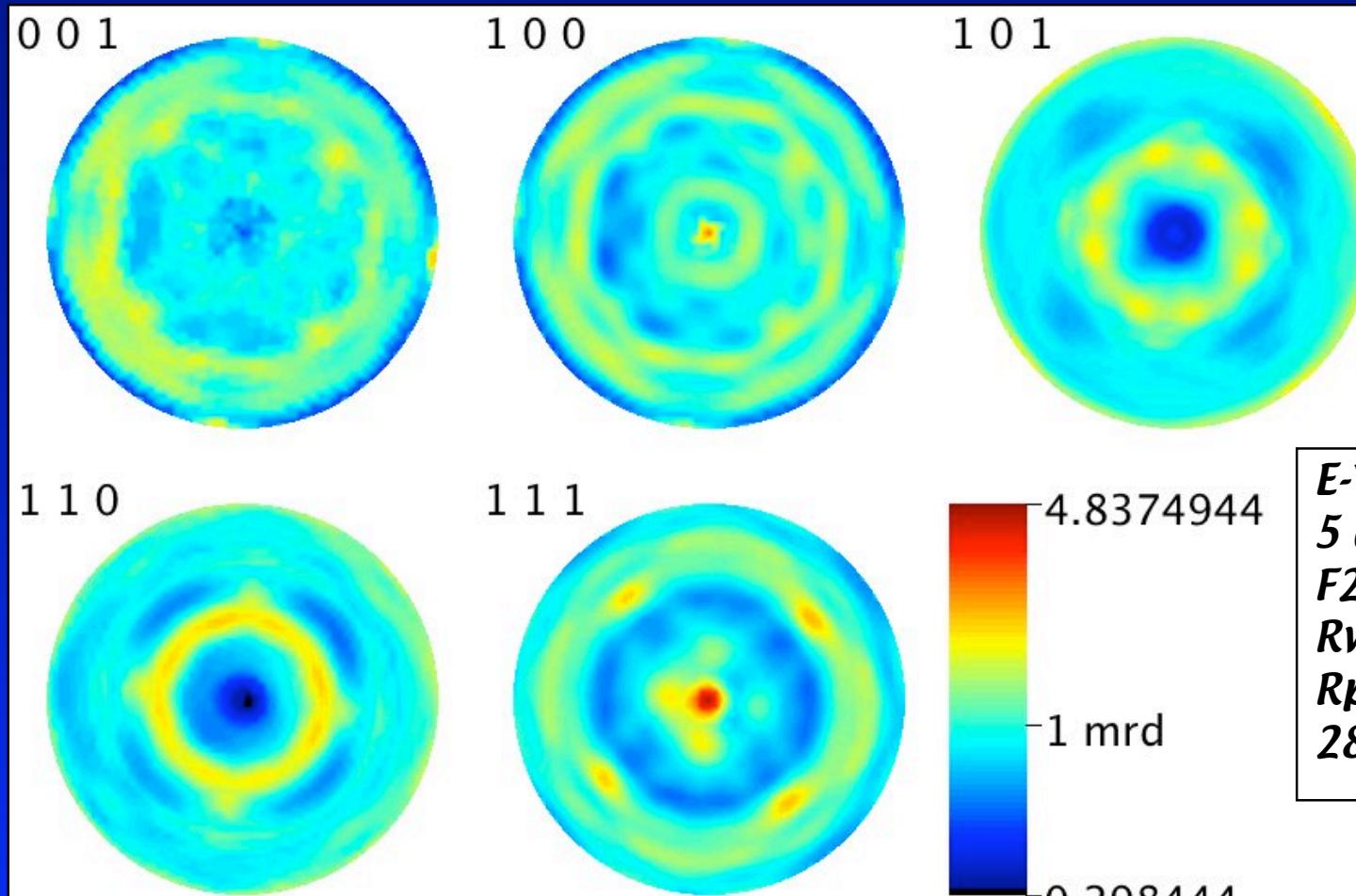


PTC film fitting: E-WIMV

E-WIMV
2 layers
2 phases
 $R_w = 21.7\%$
 $R = 40.0\%$
792 spectra

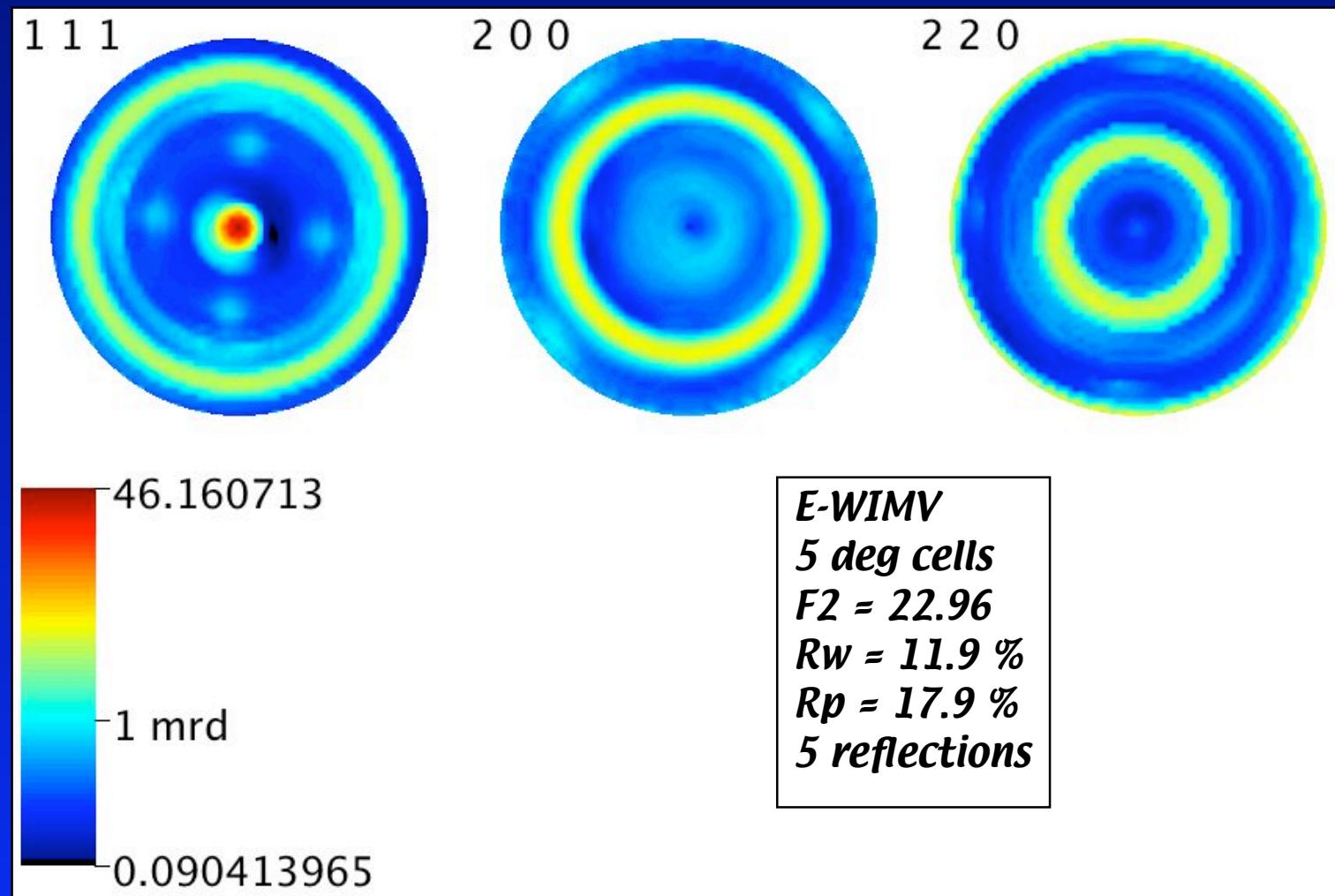


PTC film: PTC reconstructed pole figures



*E-WIMV
5 deg cells
 $F_2 = 1.962$
 $R_w = 74.4 \%$
 $R_p = 24.9 \%$
28 reflections*

Pt buffer layer: reconstructed pole figures



Results on the PTC film

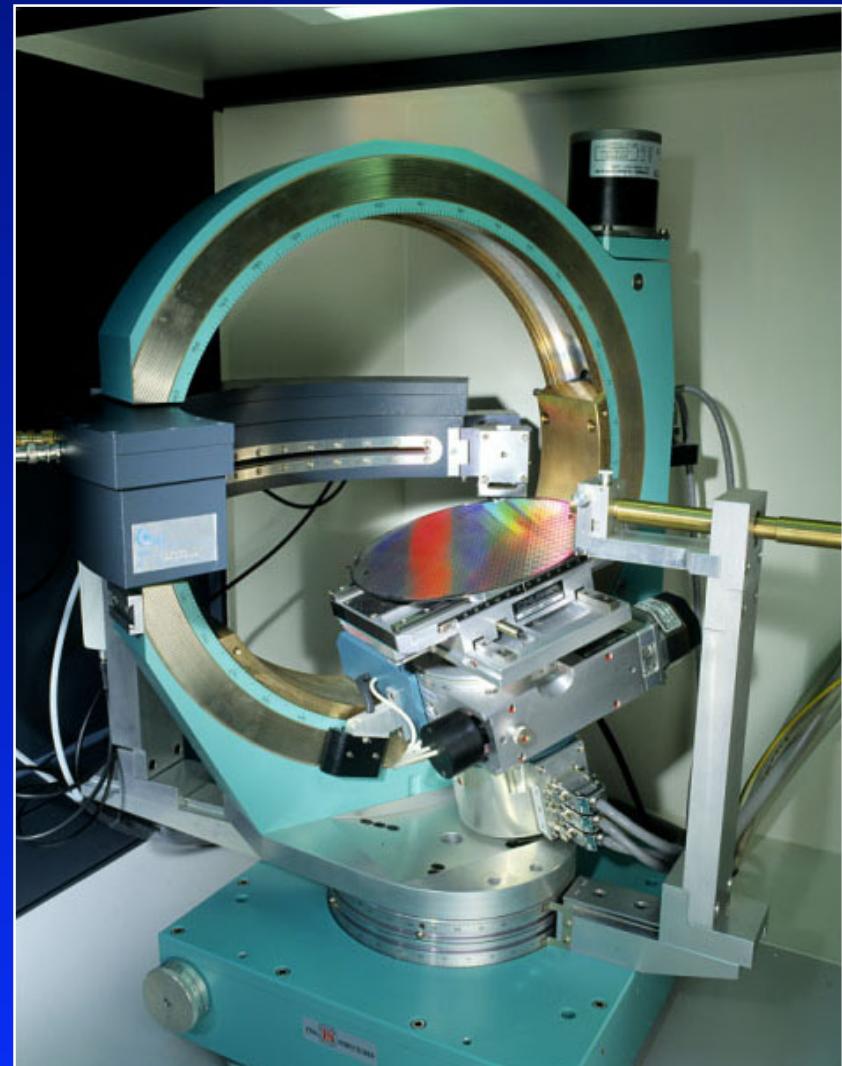
Layer/Phase	Cell parameters (Å)	Cryst. Size (Å)	r.m.s. microstrain	Layer thickness (Å)
Pt	3.955(1)	462(4)	0.0032(1)	458(3)
PTC	a=3.945(1) c=4.080(1)	390(7)	0.0067(1)	4080(1)

PTC crystal structure

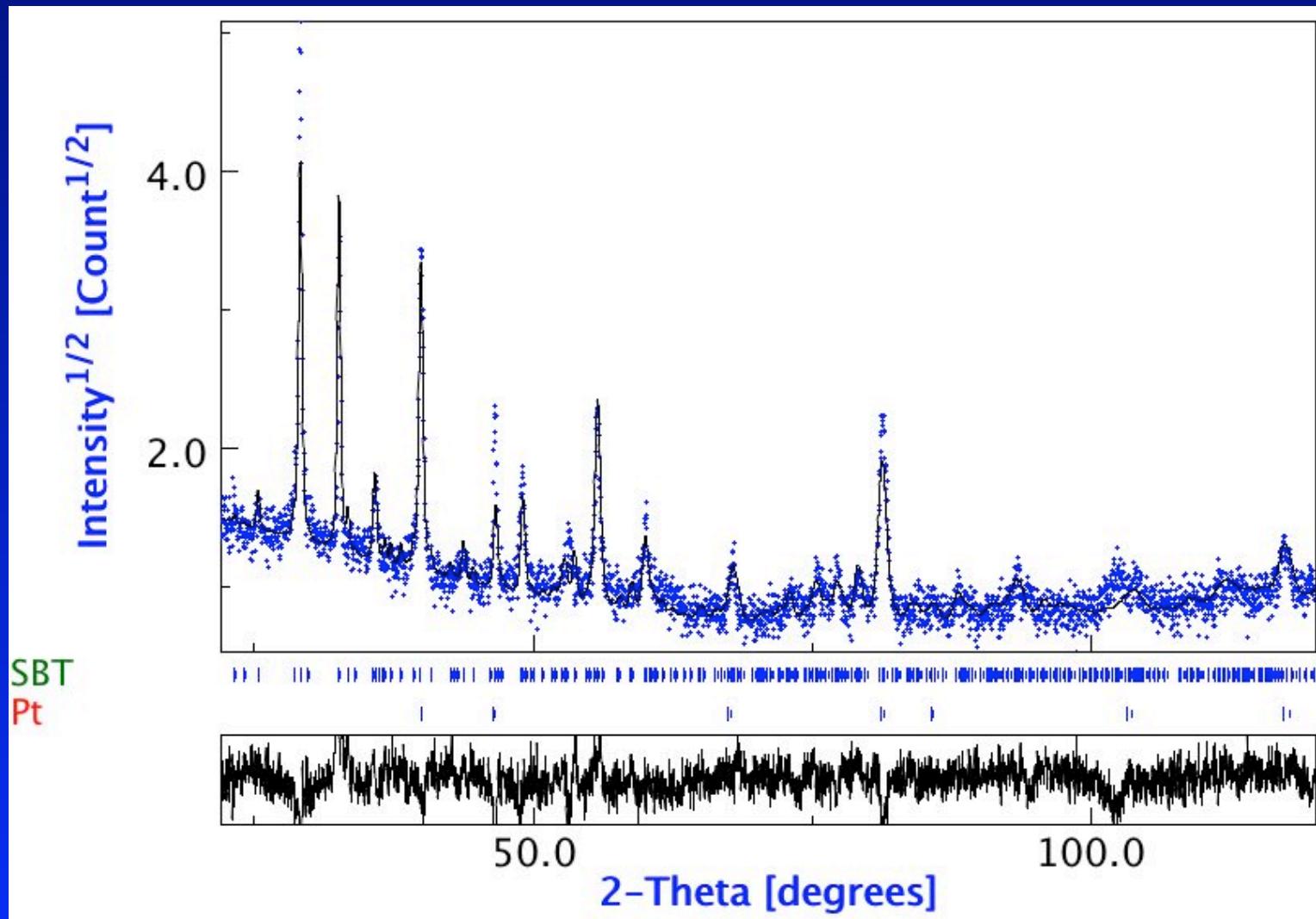
<i>Atom</i>	<i>Occupancy</i>	<i>x</i>	<i>y</i>	<i>z</i>
<i>Pb</i>	0.76	0.0	0.0	0.0
<i>Ca</i>	0.24	0.0	0.0	0.0
<i>Ti</i>	1.0	0.5	0.5	0.477(2)
<i>O1</i>	1.0	0.5	0.5	0.060(2)
<i>O2</i>	1.0	0.0	0.5	0.631(1)

SBT film

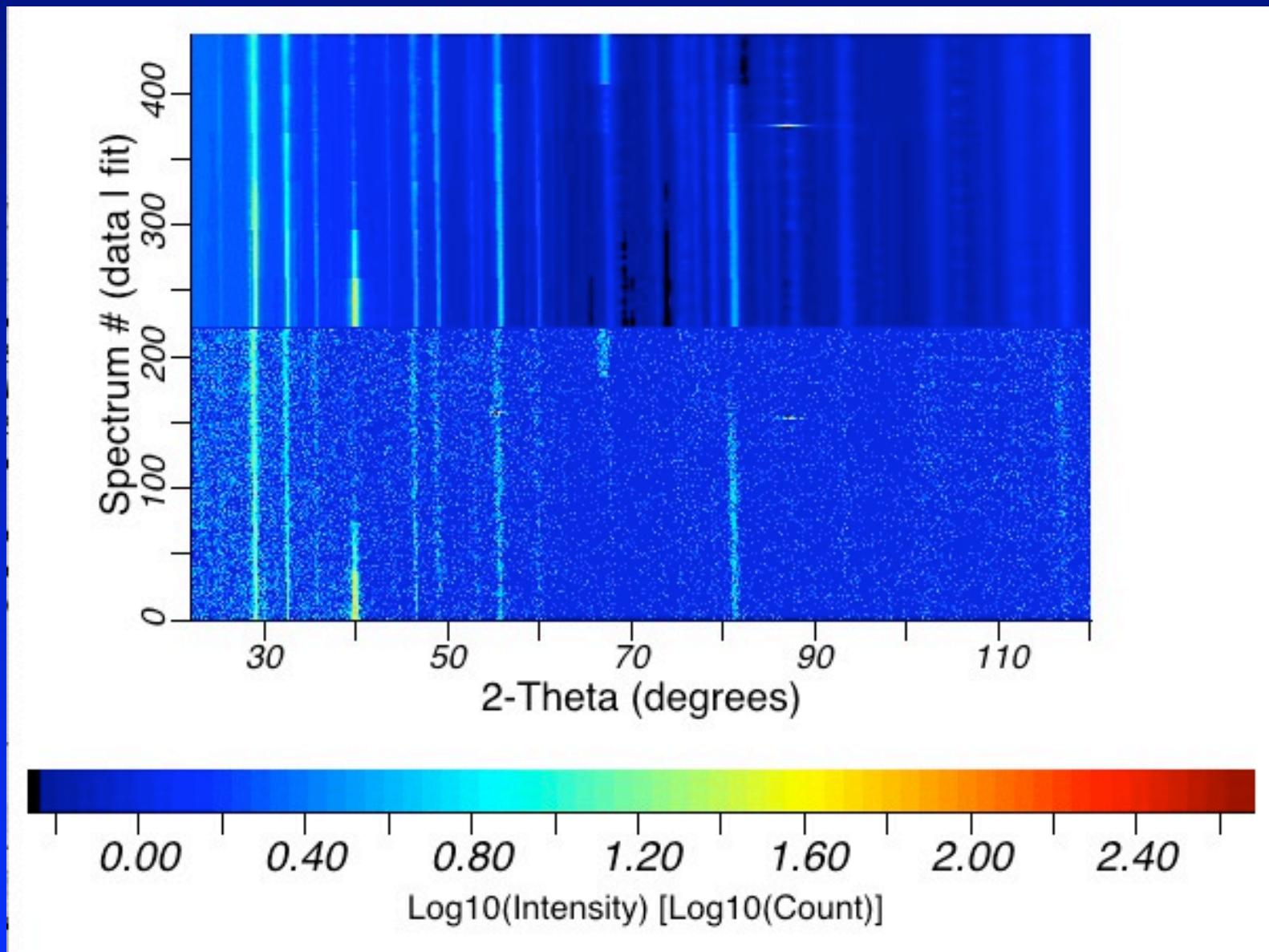
- *Si Wafer + 50 nm Pt buffer layer*
- *~ 300 nm of $(Sr_{0.82}Bi_{0.12})Bi_2Ta_2O_9$*
 - *Orthorhombic A21am:-cba*
- *Spectra collection on the ESQUI diffractometer (right)*
- *120 degs position sensitive detector on an eulerian cradle; multilayer as a primary beam monochromator*
- *Spectra collected in chi from 0 to 45 degrees in step of 5 deg for chi and 0 to 180 in steps of 5 deg for phi*
- *Structure refined*



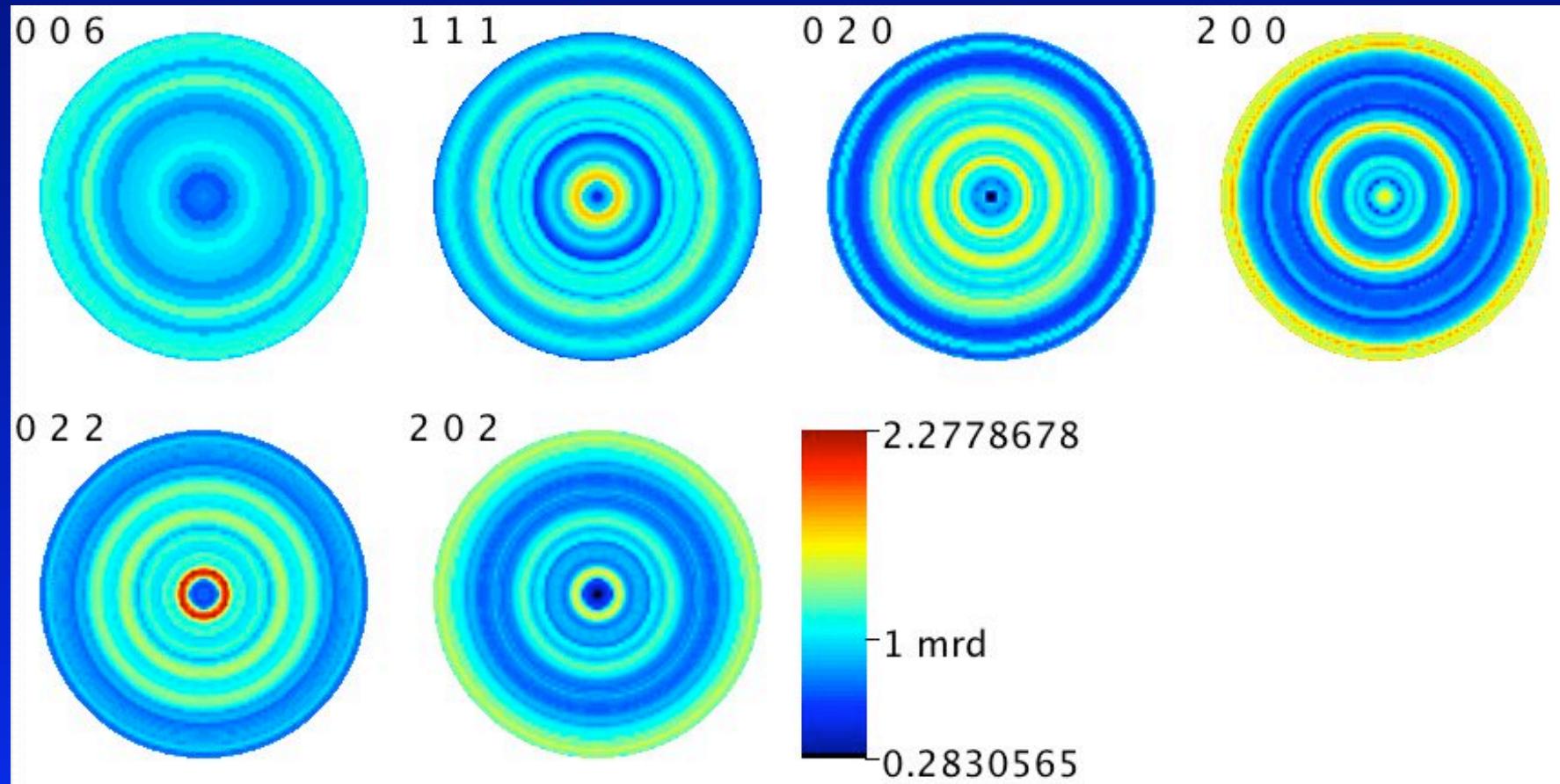
Rietveld Texture refinement



SBT thin film Rietveld fit



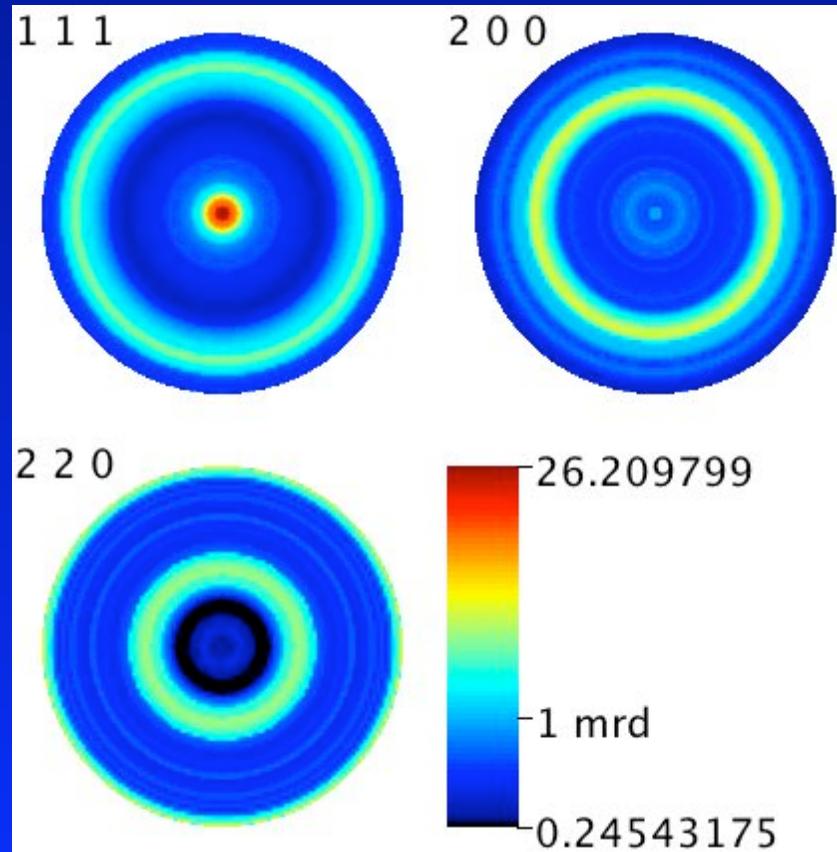
SBT pole figures reconstructed



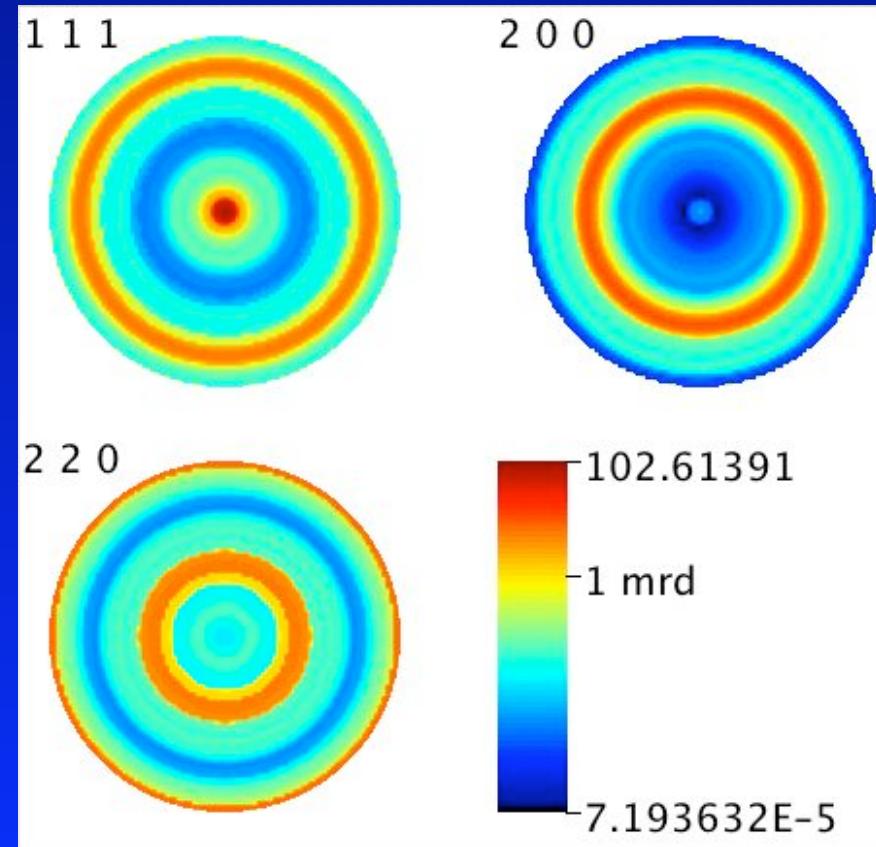
Pt texture too sharp for WIMV

Special texture methodology for Rietveld developed: Entropy based WIMV using tube projections. Interpolation of pole figures avoided.

WIMV

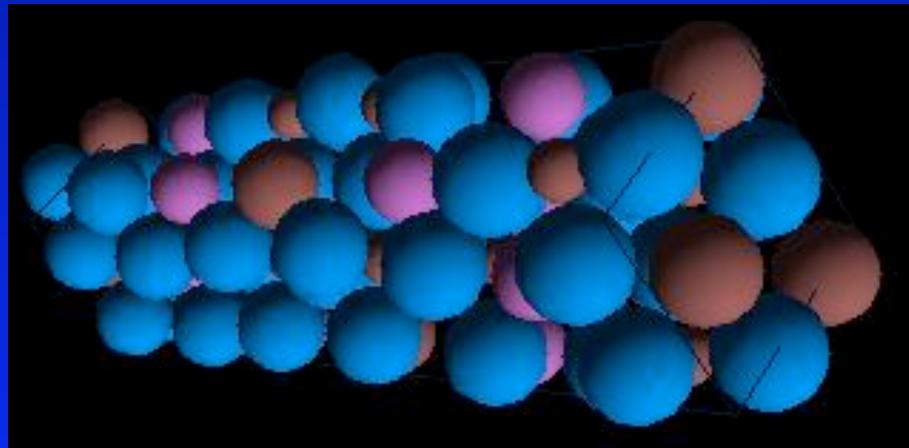


E-WIMV



SBT film microstructure and crystal structure

	<i>Cell parameters (Å)</i>	<i>Cryst. Size (Å)</i>	<i>Microstrain</i>	<i>Layer thickness (Å)</i>
SBT	$a \approx b = 5.5473(2)$ $c = 25.316(2)$	565(5)	0.0037(3)	3579(72)
Pt	3.9411(1)	317(4)	0.0029(2)	557(15)



Space group: A21am:-cba

14 atomic position parameters refined

Extremely sharp Al film (ST microelectronics)

Aluminum film

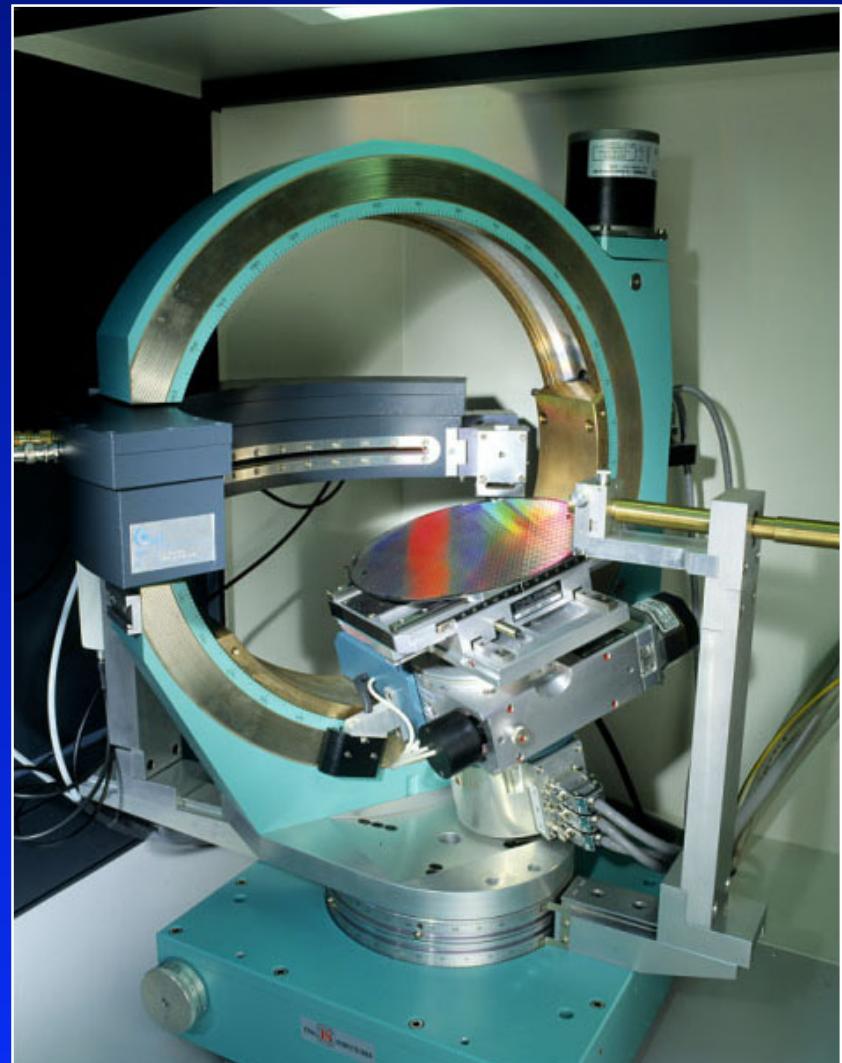
Si wafer substrate

*Spectra collection on the ESQUI
diffractometer (right)*

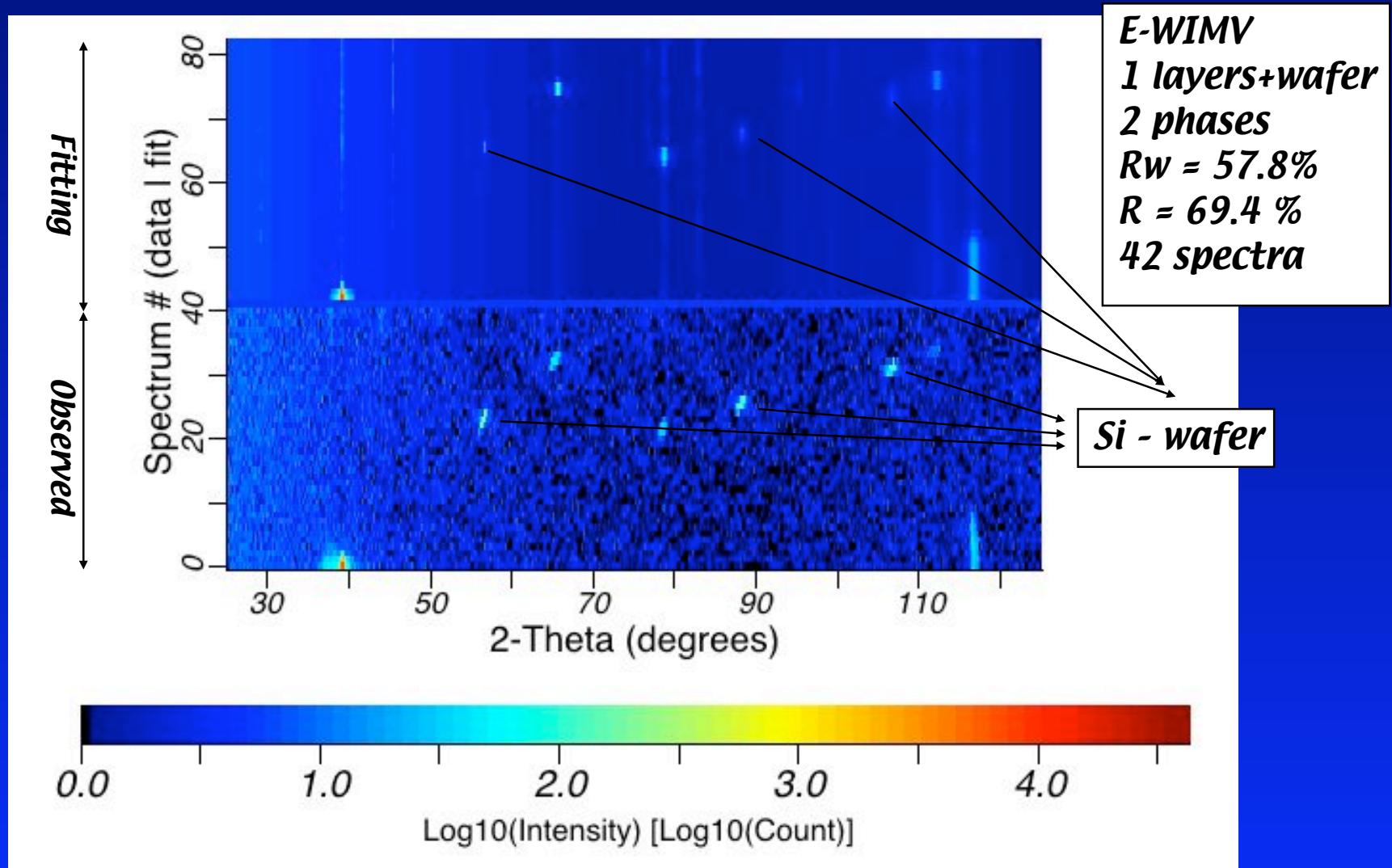
*120 degs position sensitive detector on
an eulerian cradle; multilayer as a
primary beam monochromator*

*Spectra collected in chi from 0 to 45
degrees in step of 1 deg turning
continuously the phi motor (fiber
texture)*

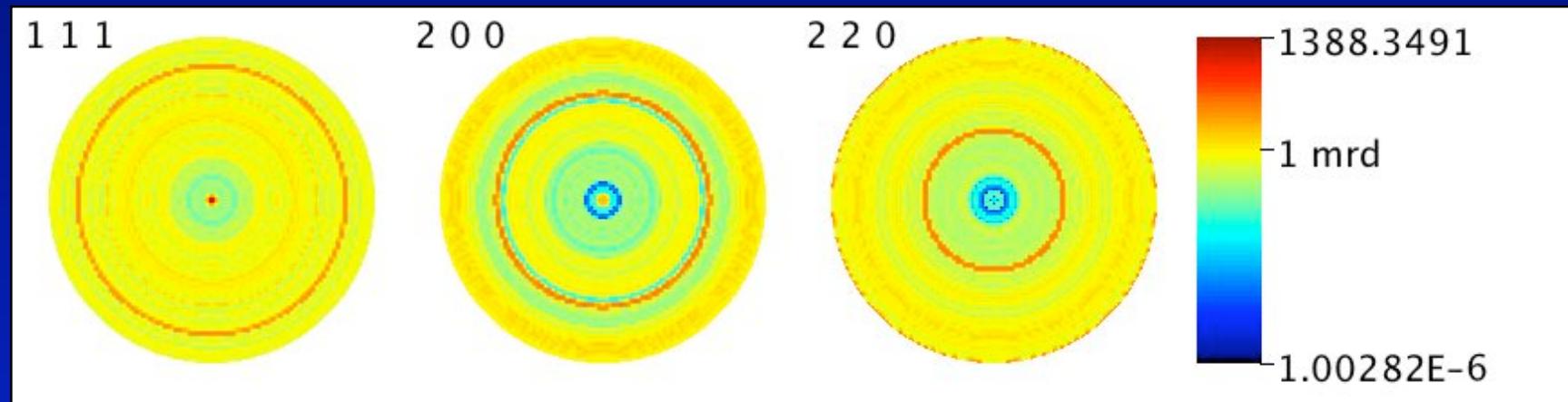
*E-WIMV used only; too sharp texture
even for WIMV*



Al film: fitting the spectra



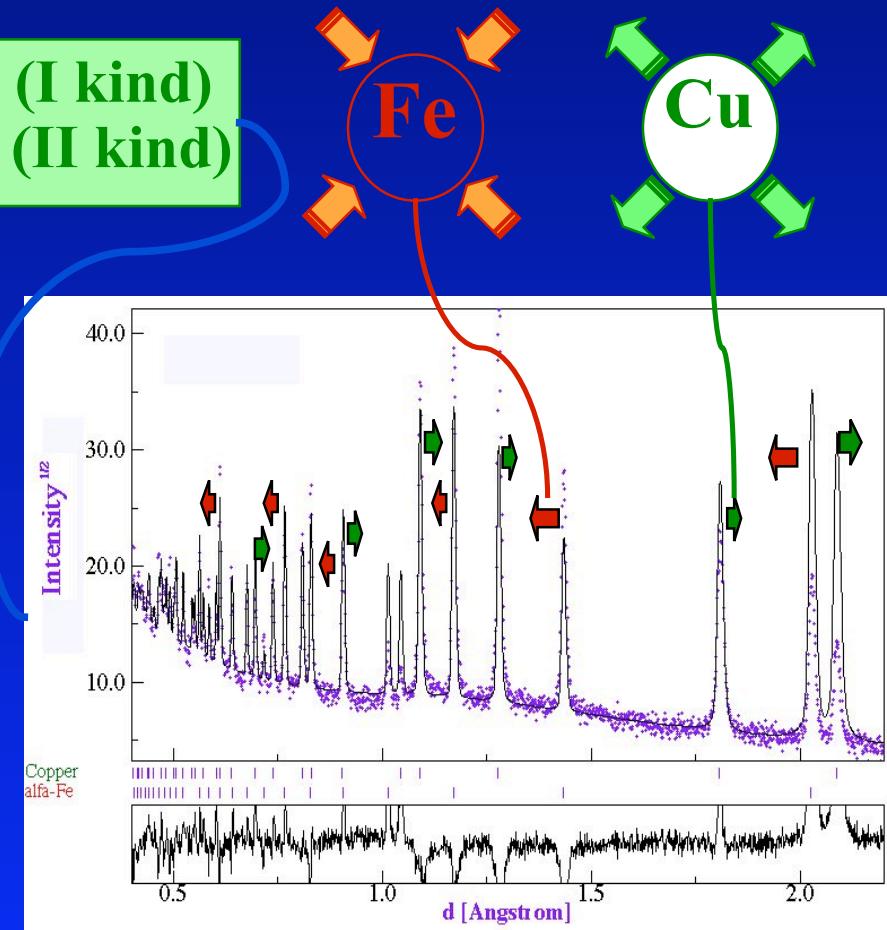
Al film: Al reconstructed pole figures



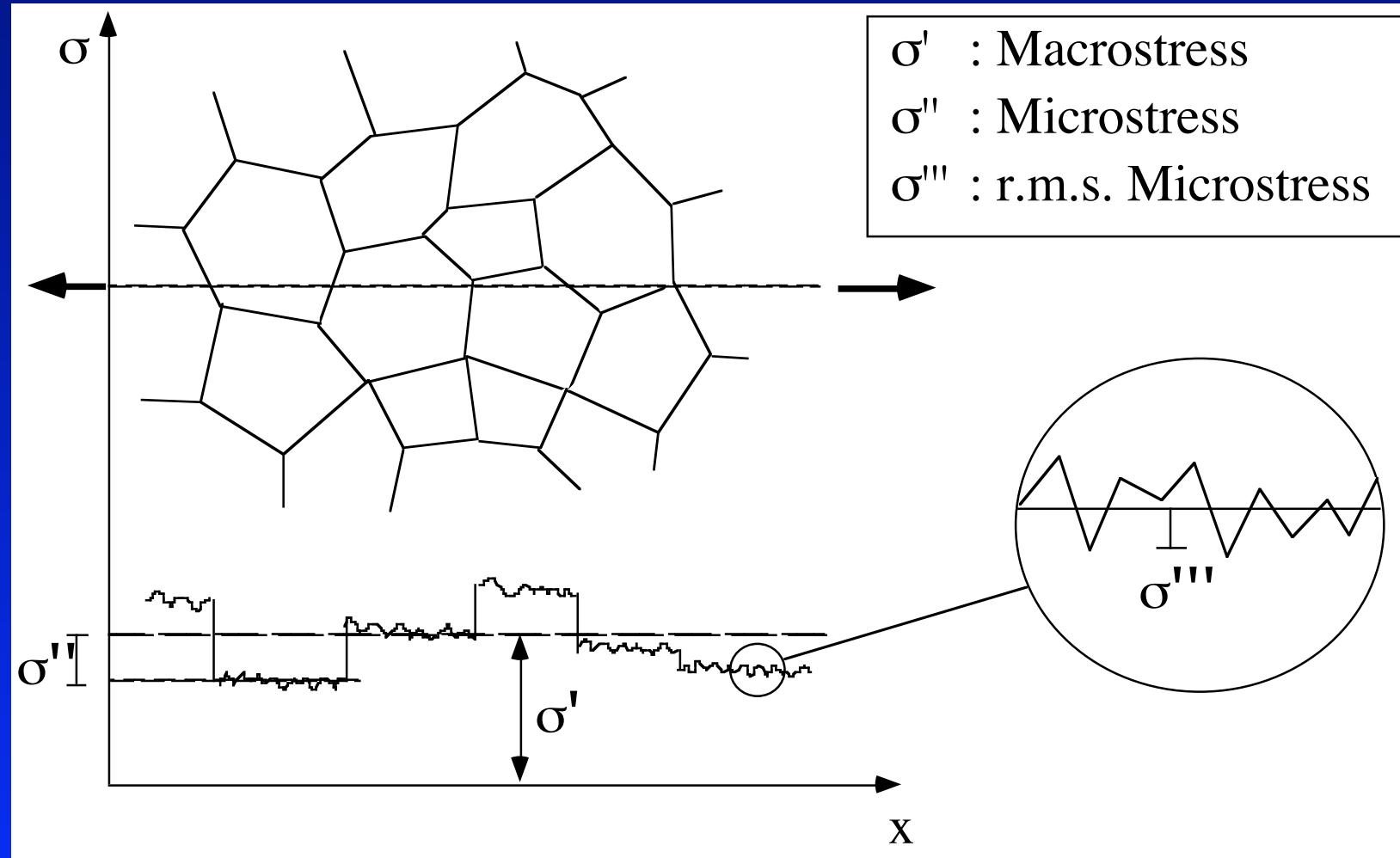
*E-WIMV
1 deg cells
 $F2 = 1100.9$
 $Rw = 15.4 \%$
 $Rp = 19.5 \%$
8 reflections*

Informations: strains

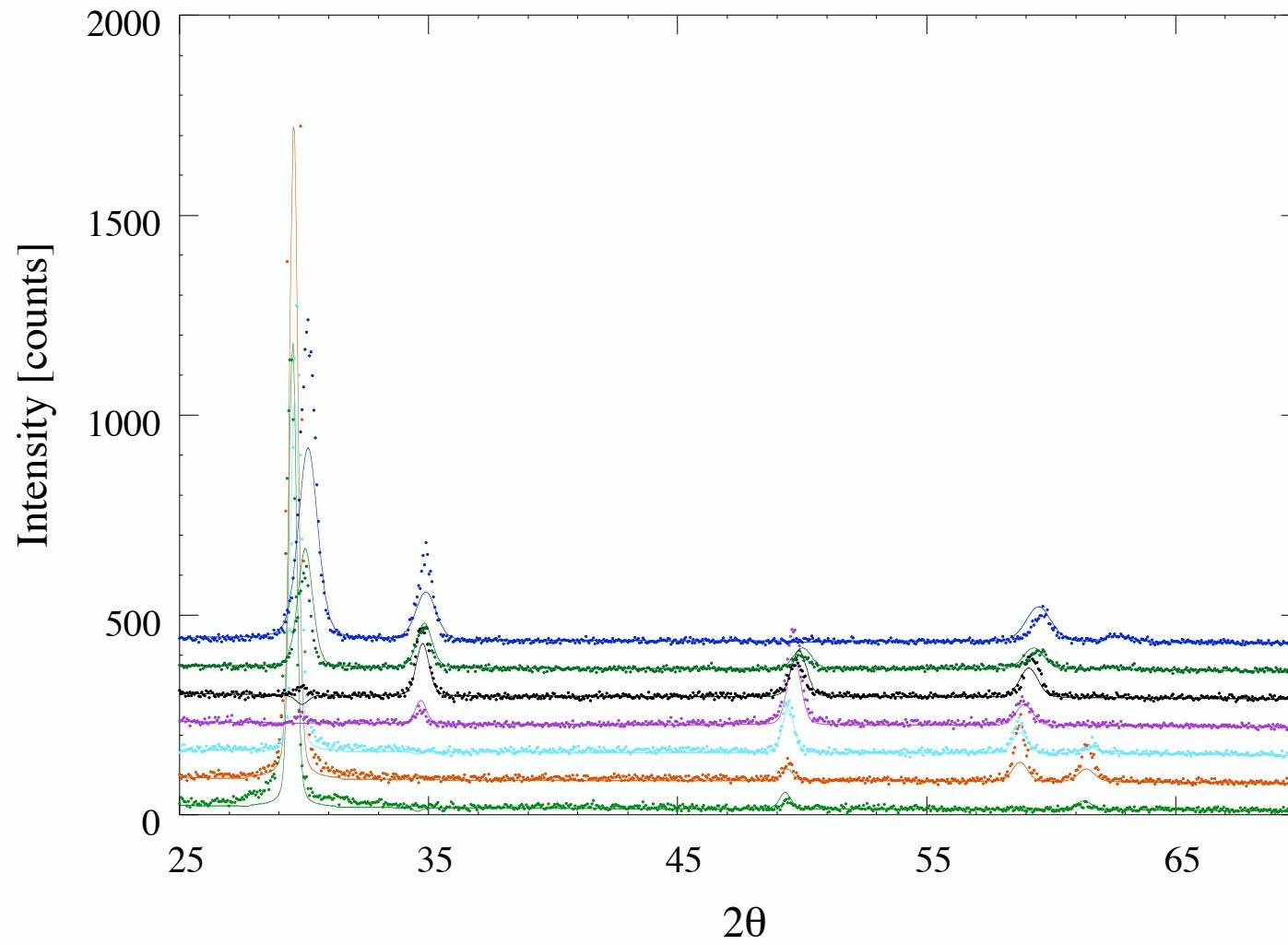
- Macro elastic strain tensor (I kind)
- Crystal anisotropic strains (II kind)



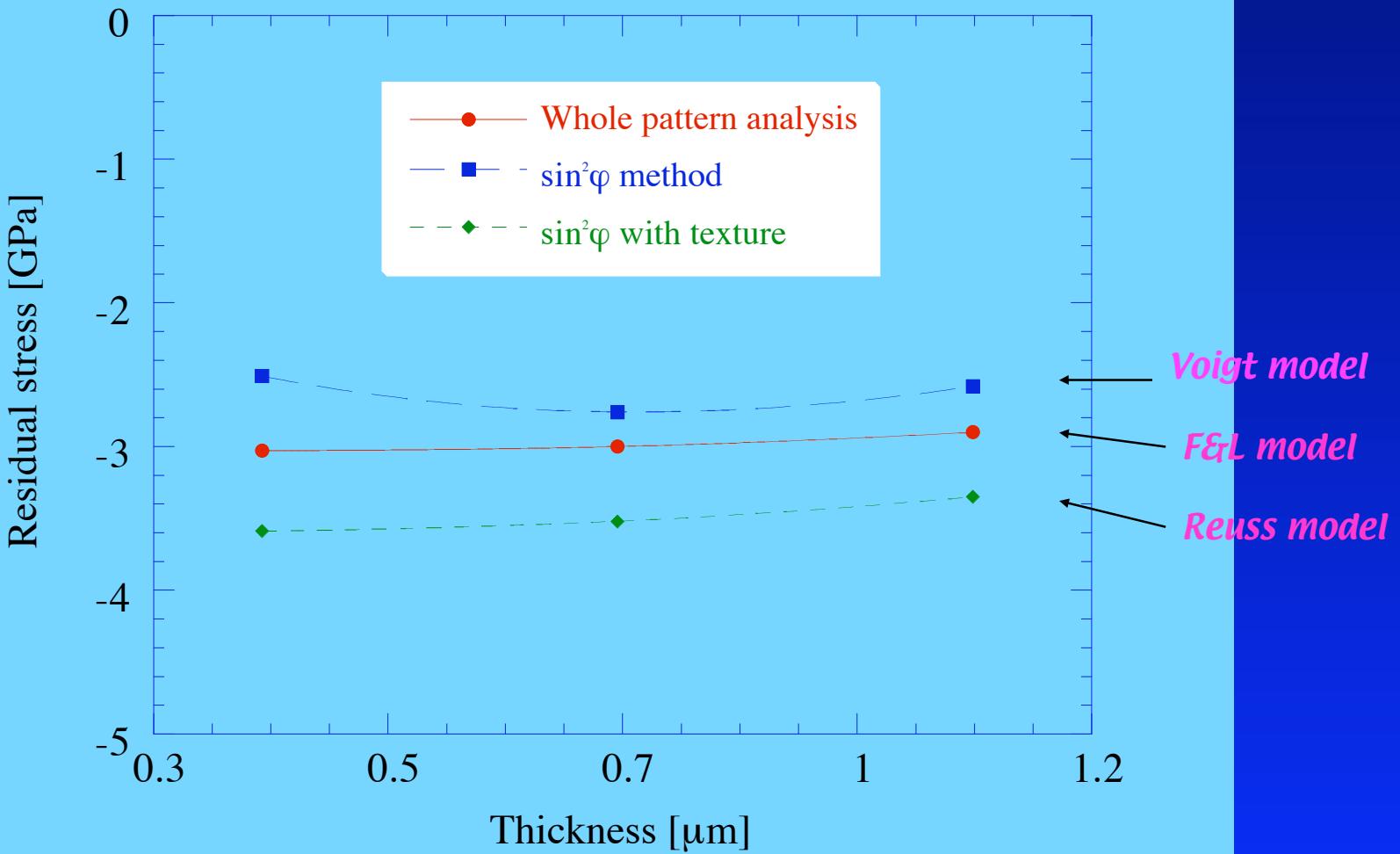
Residual Stress/Strain definition



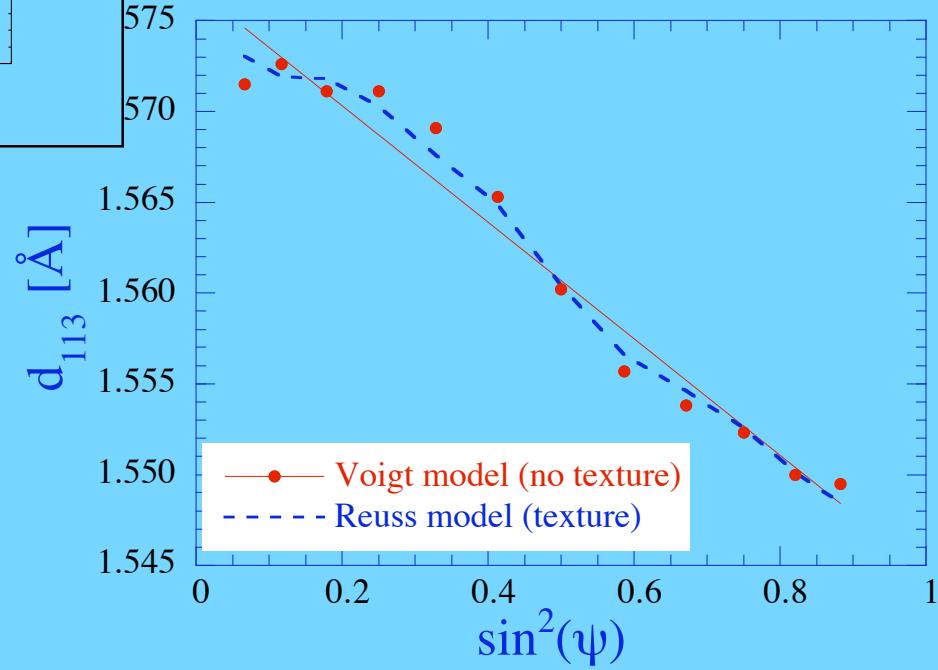
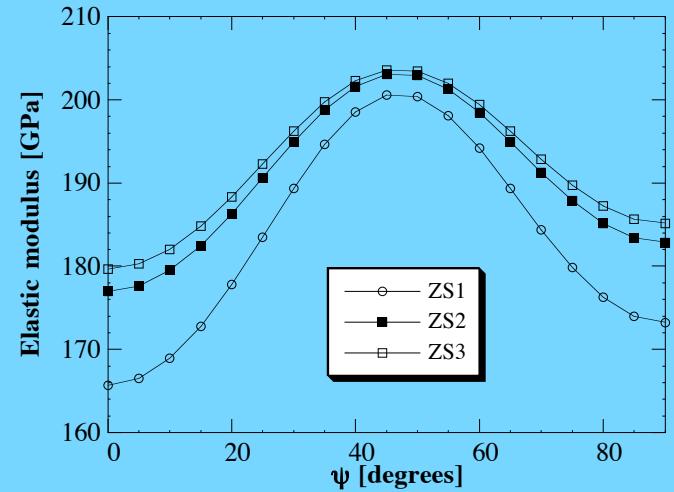
ZrO₂ thin films



Macro residual stress on the ZrO₂ serie

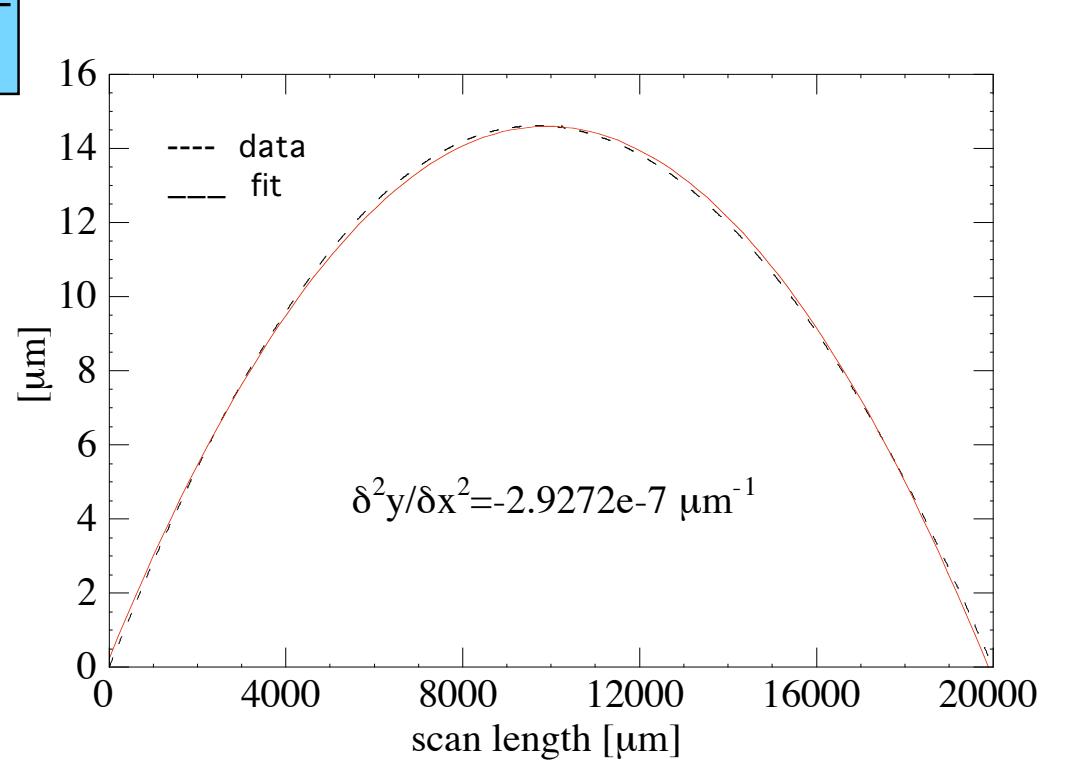


Traditional methods for the ZrO₂ films



Measuring the stress also by the curvature

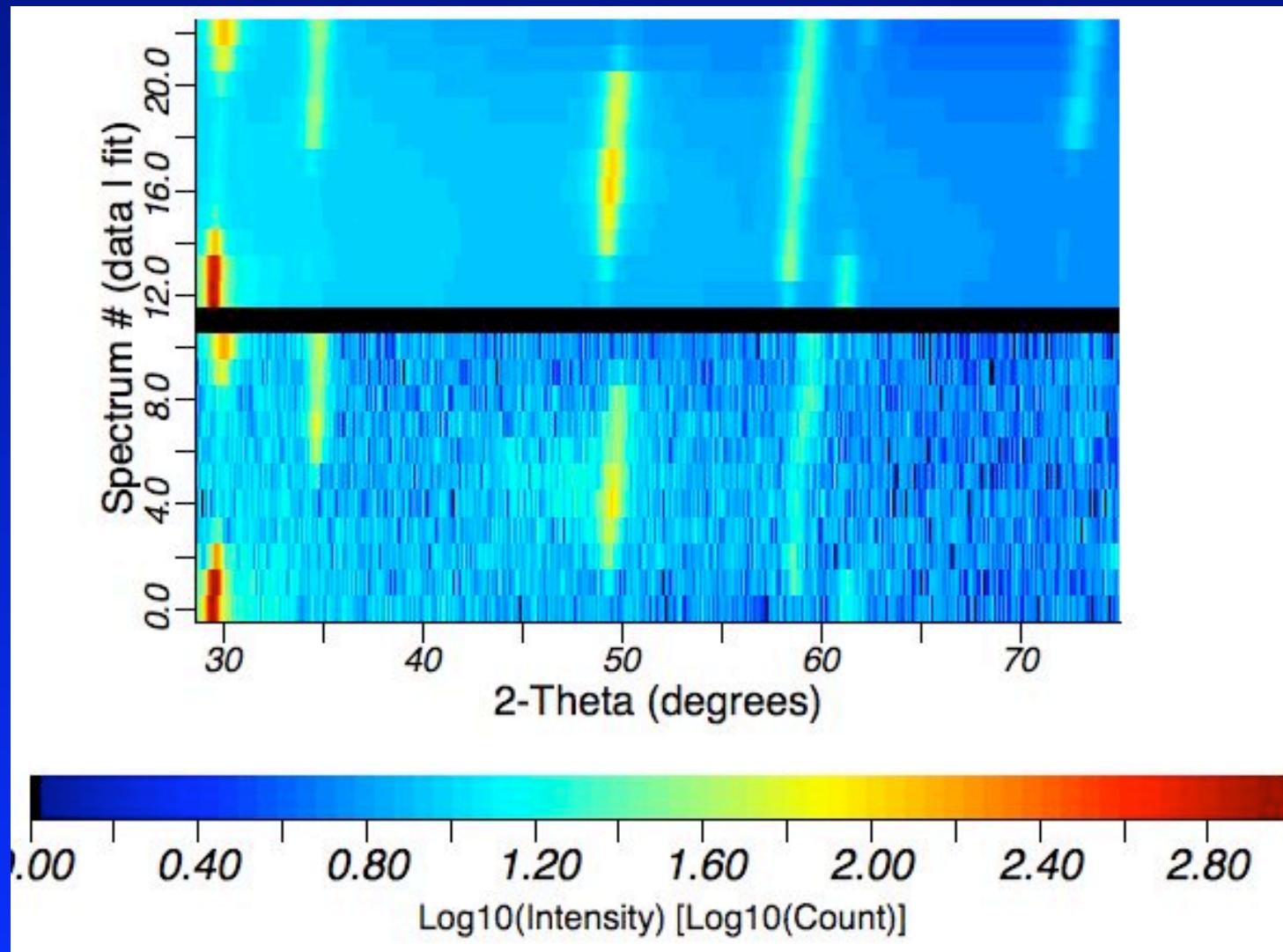
$$\sigma^* = K \cdot \frac{d_s^2}{d_f} \cdot \frac{a_{11}}{2}$$



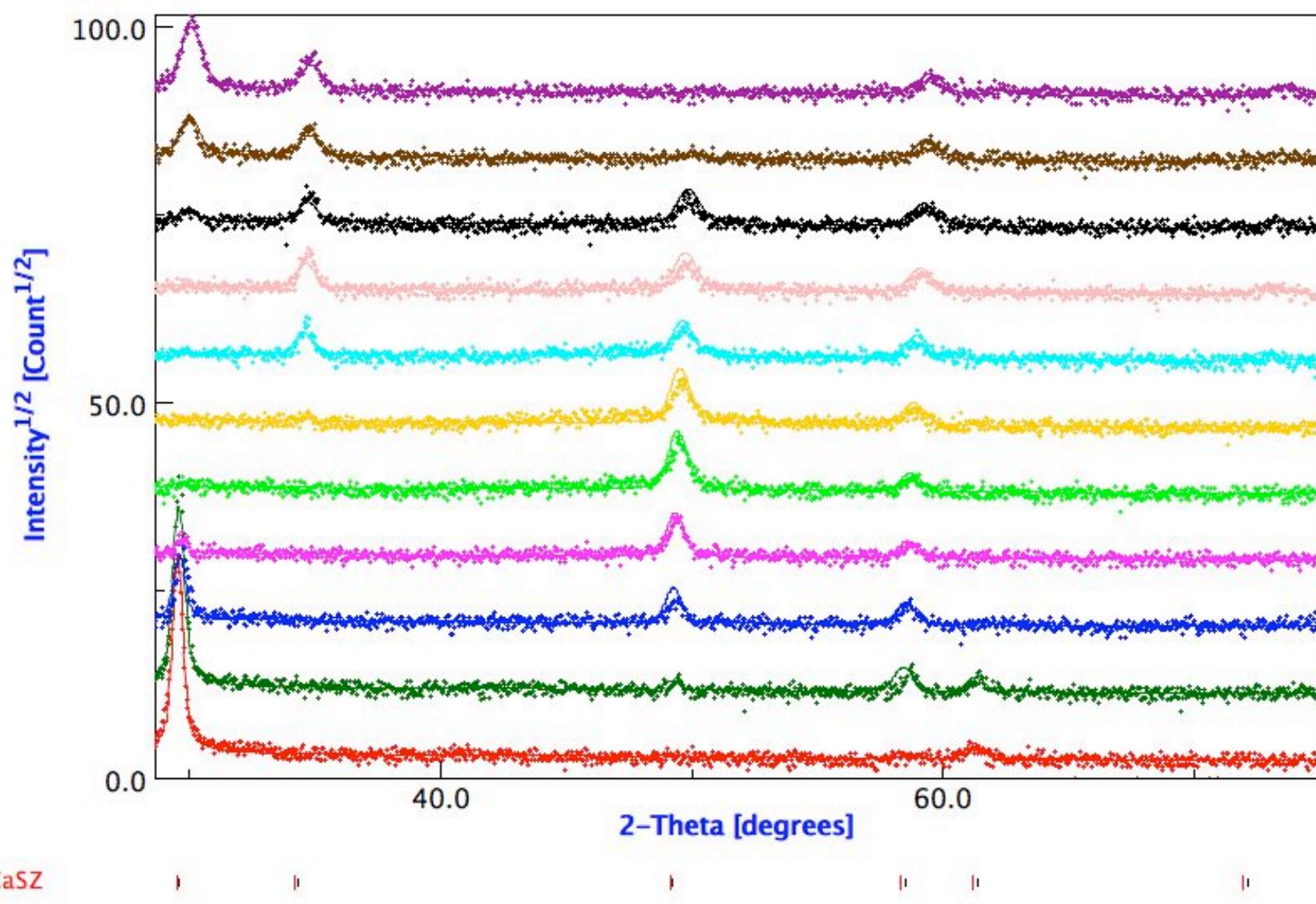
Comparison of results

Residual Stresses/Texture analysis

- *Voigt model + WIMV*



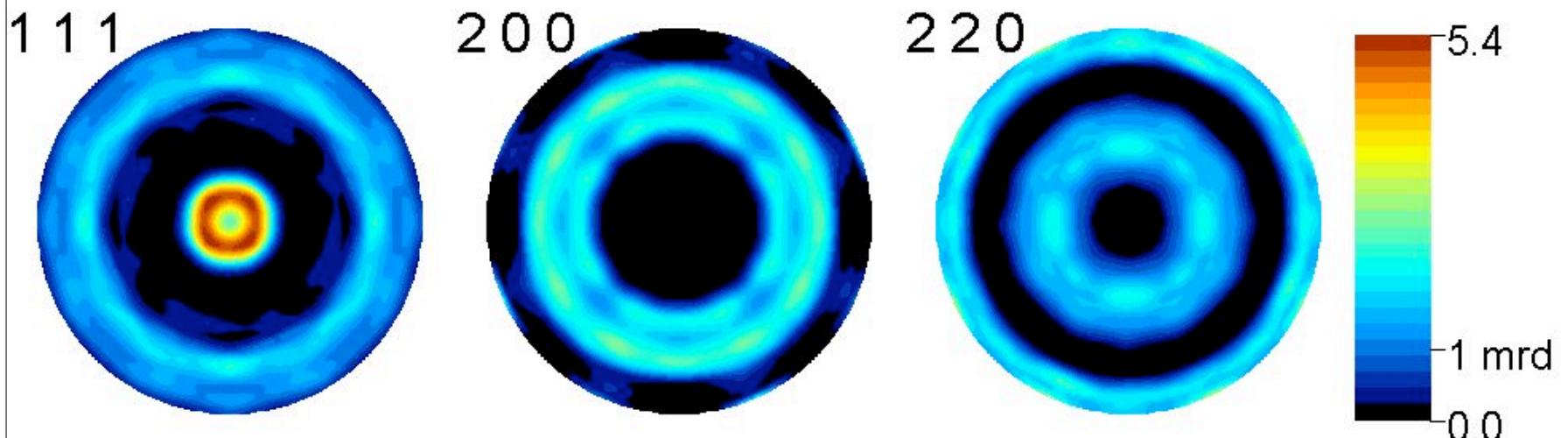
Multi-plot for the zirconia film (WIMV)



ZrO₂ film: results

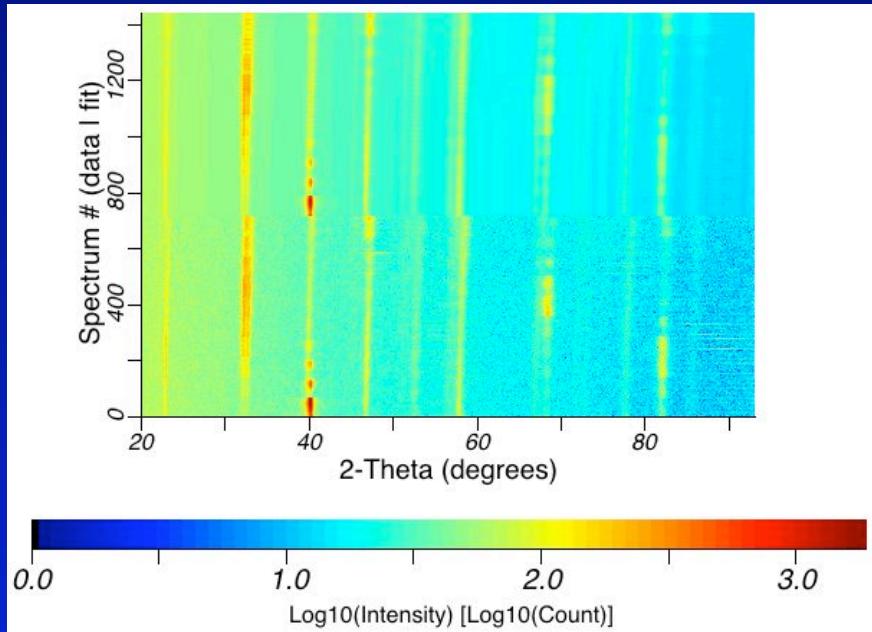
*Very high in plane residual stresses
(compression):
Reuss model: 3.6 GPa
Bulk Path GEO: 3.47(5) GPa
Curvature method: > 10 Gpa !?
Thickness: 320 Nanometer*

Reconstructed pole figures

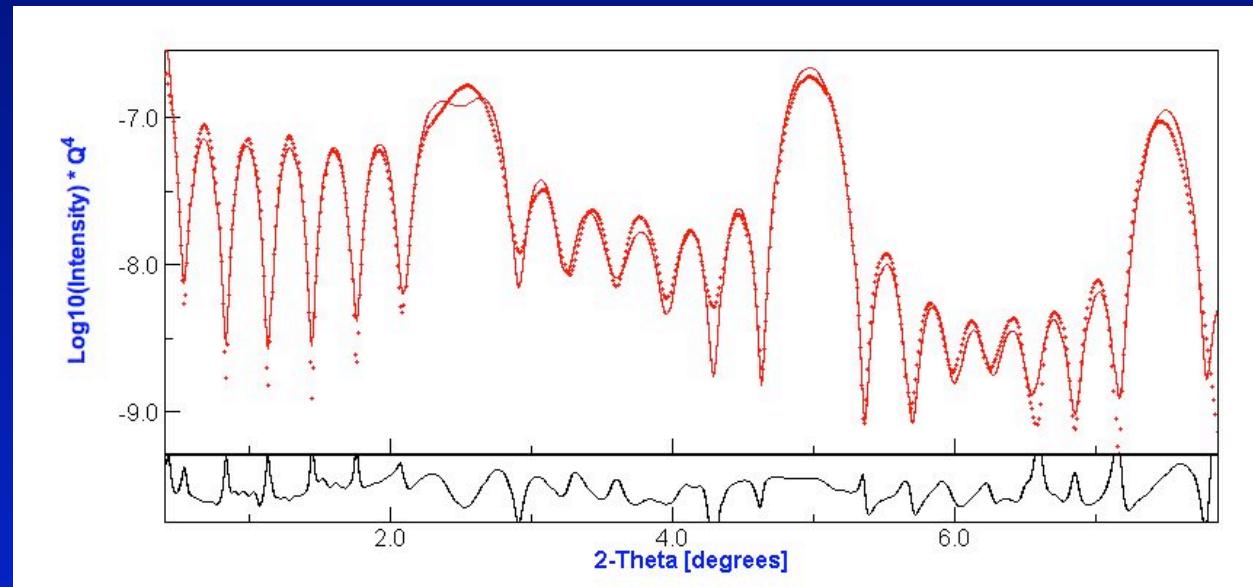


Experimental errors

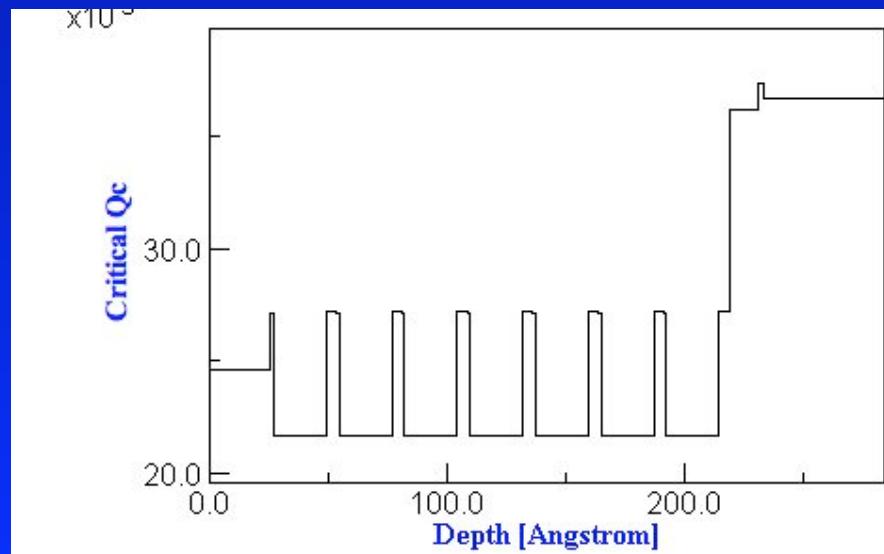
- *Example: the CPT film shows big shift of the peaks increasing χ .*
- *The shift is not smaller at low 2theta angle.*
- *In the fitting was perfectly reproduced by a beam 0.59 mm higher than the goniometer center.*
- *Using the Rietveld method peak shifts from low angle positions are also used normally -> good sample positioning required, perfect alignment of the instrument also.*



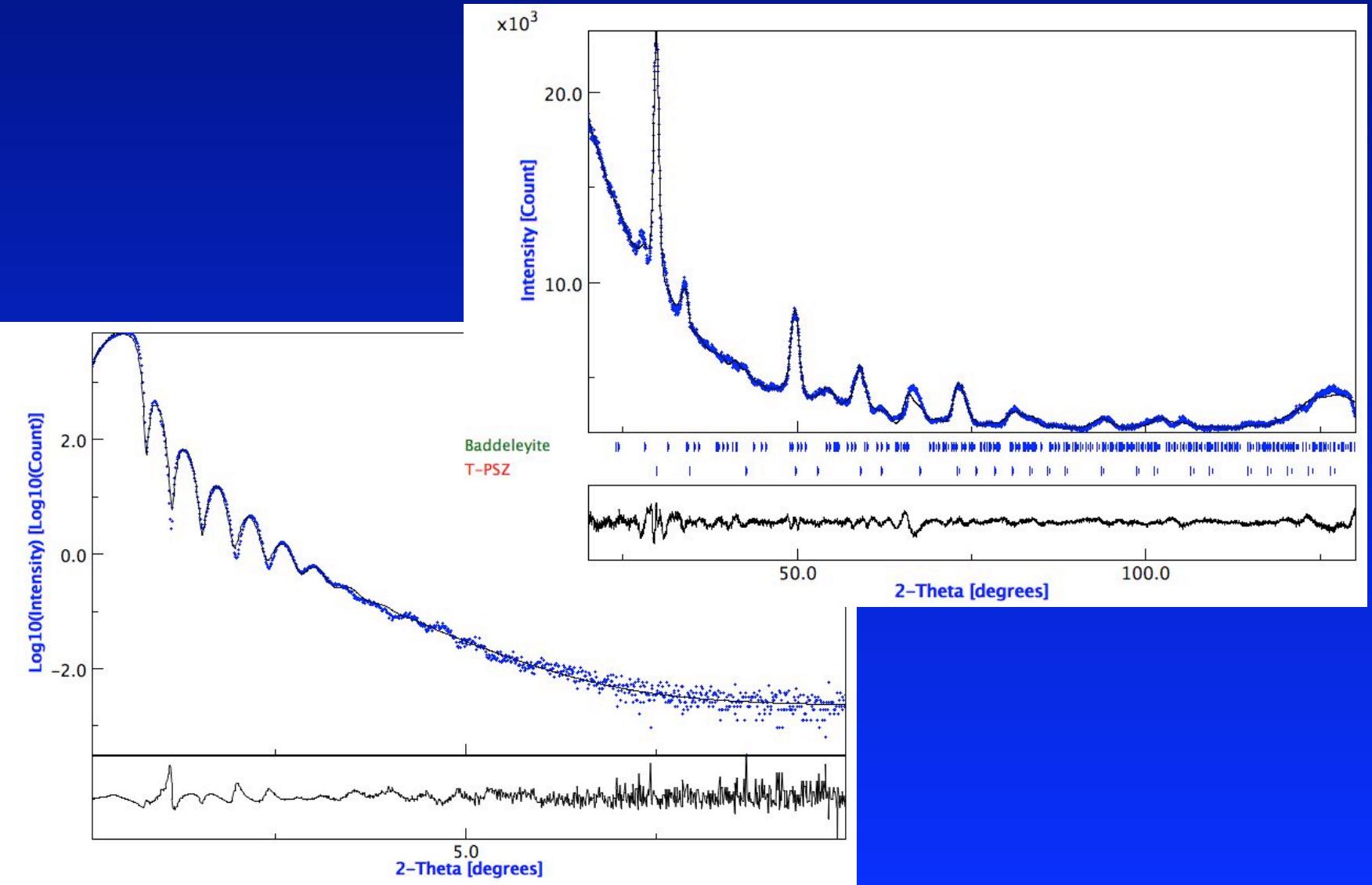
Reflectivity for multilayer analysis



- *Langmuir-Blodgett film*
- *24 layers sequence*
- *Matrix method used for the analysis in Maud*
- *Film and data collected by A. Gibaud (LPEC, Le Mans)*

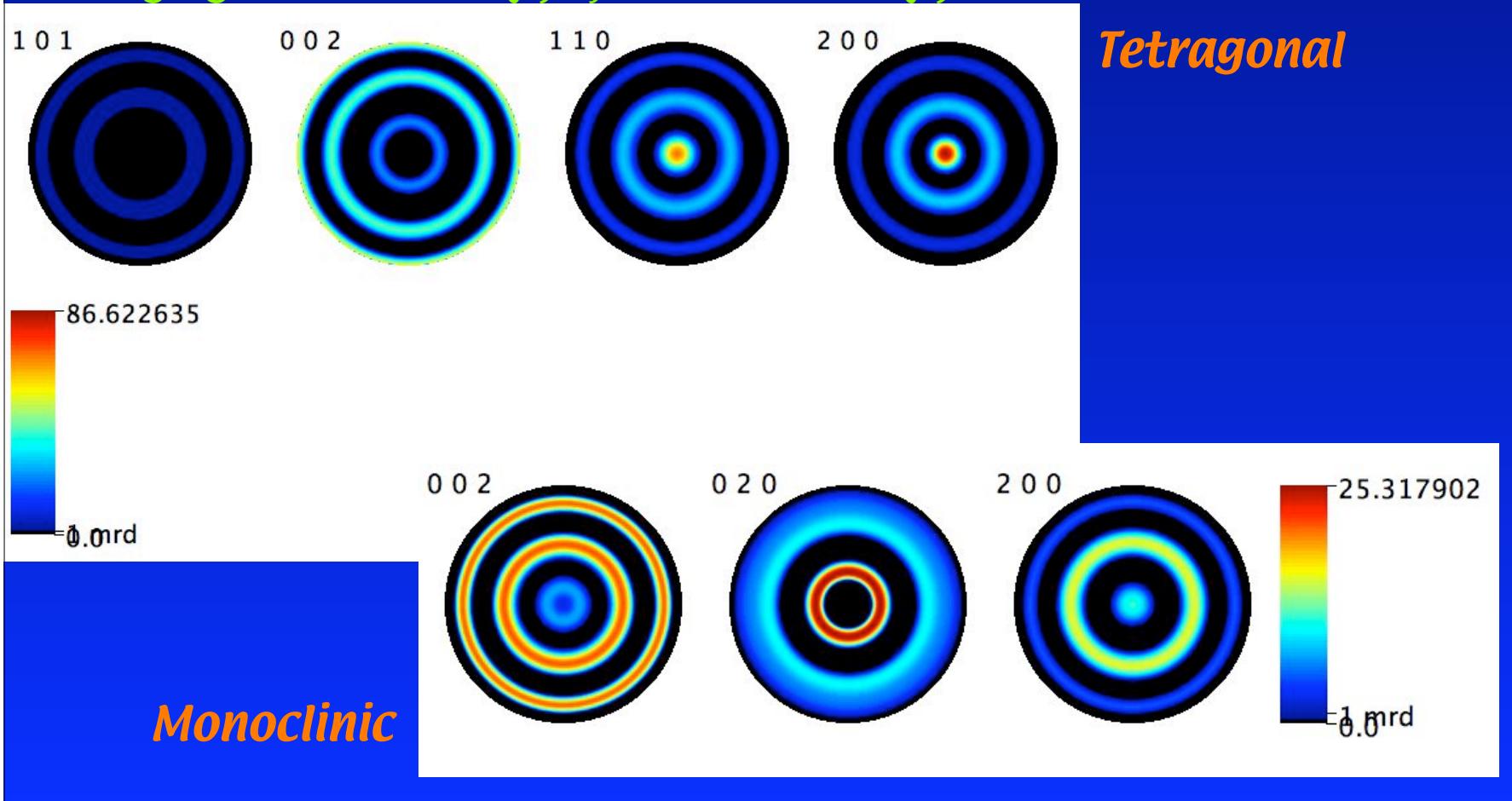


ZrO₂ results - 1



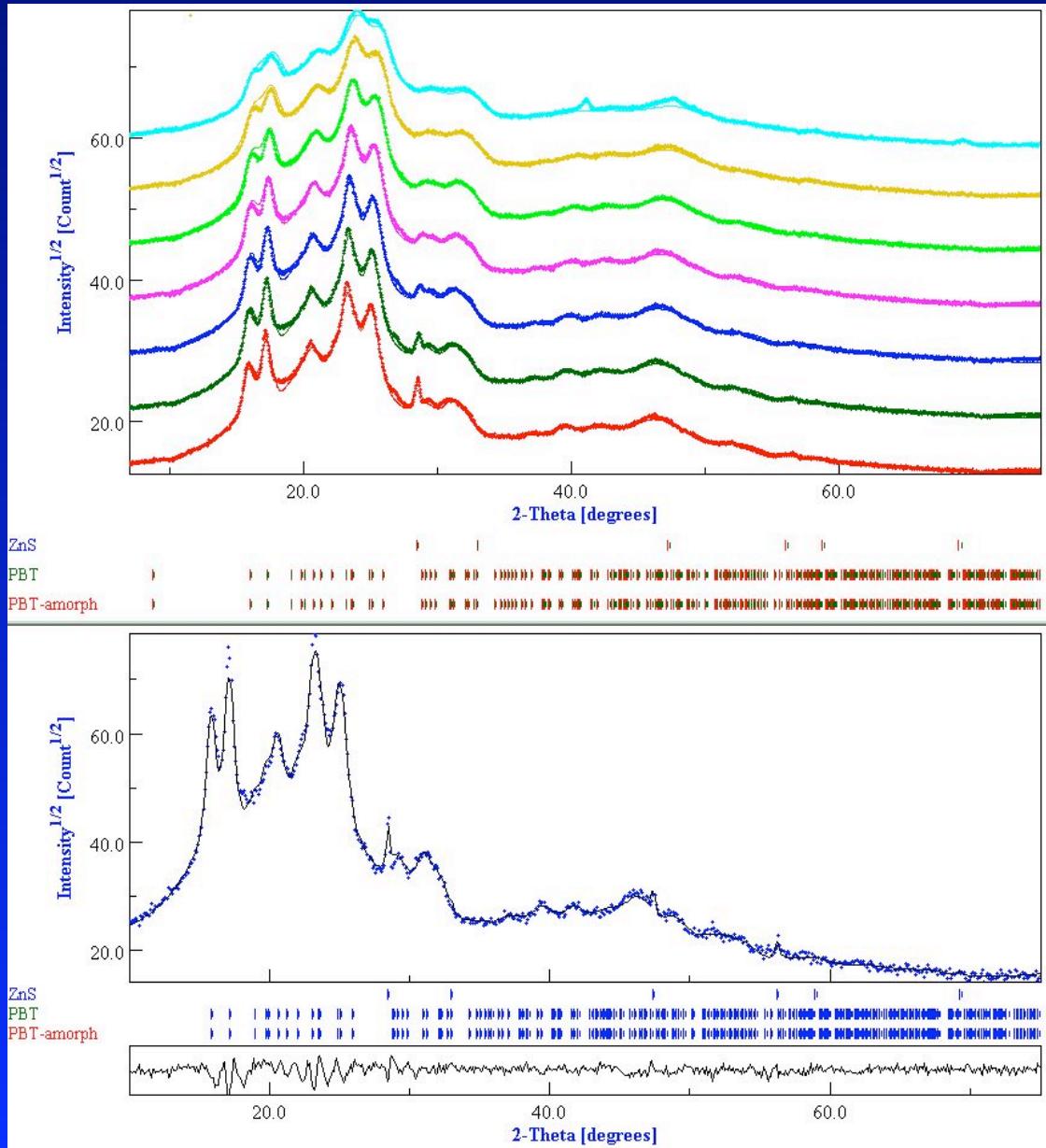
ZrO₂ results - 2

- *ZrO₂ thickness: 196(1) Angstrom*
- *Baddeleyite: <D> = 28(1) Å, <ε²>^{1/2} = 0.0027(1), volume fraction = 0.530*
- *Tetragonal: <D> = 130(7) Å, <ε²>^{1/2} = 0.017(1)*

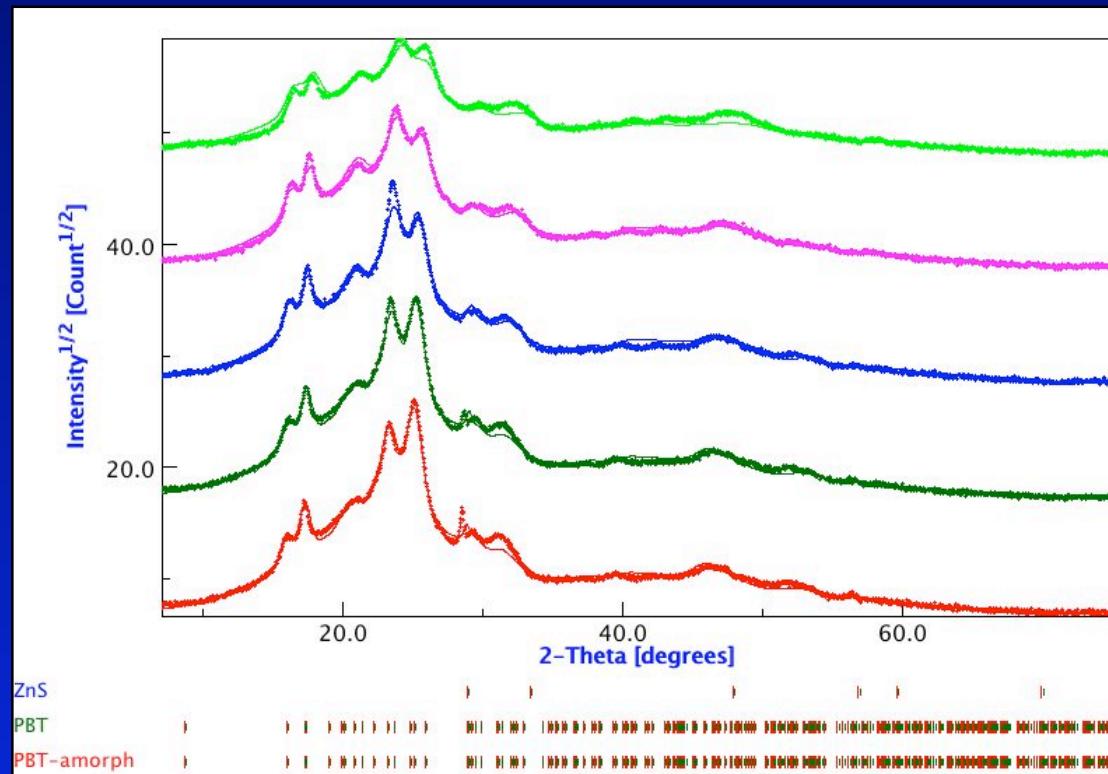


PBT polymer (thick film): texture analysis

- Polybutilentereftalate: triclinic
- Only partially crystallized
- Deformed at different rate (uniassial compression)
- Annealed
- Spectra for both deformed and deformed+annealed samples collected in Le Mans (CPS 120 and eulerian cradle)
- Analyzed by Maud assuming amorphous and crystallized phases having:
 - Same structure
 - Same texture

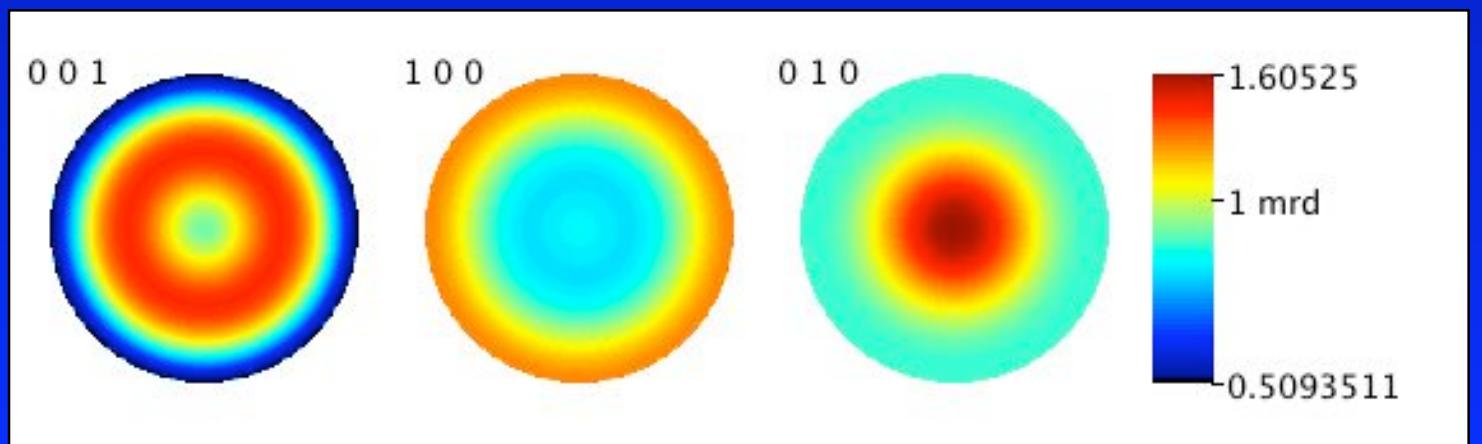


PBT deformed and annealed



Harmonic
 $L_{max} = 4$
 $F^2 = 1.37$
 $R_w = 6.27 \%$
 $R_p = 22.1 \%$
>300 reflections

Reconstructed pole figures



PBT results

- *Deformed samples become textured at higher deformation values*
- *Crystallite sizes decrease with deformation and microstrains (paracrystallinity) increase.*
- *With the annealing crystallite sizes recuperate their original dimension and microstrains decrease. Texture remains and becomes more evident. Residual deformation is not recuperated.*
- *Good fitting requires the use of the texture on the amorphous PBT*

C182 sample

Amorphous carbon film ?

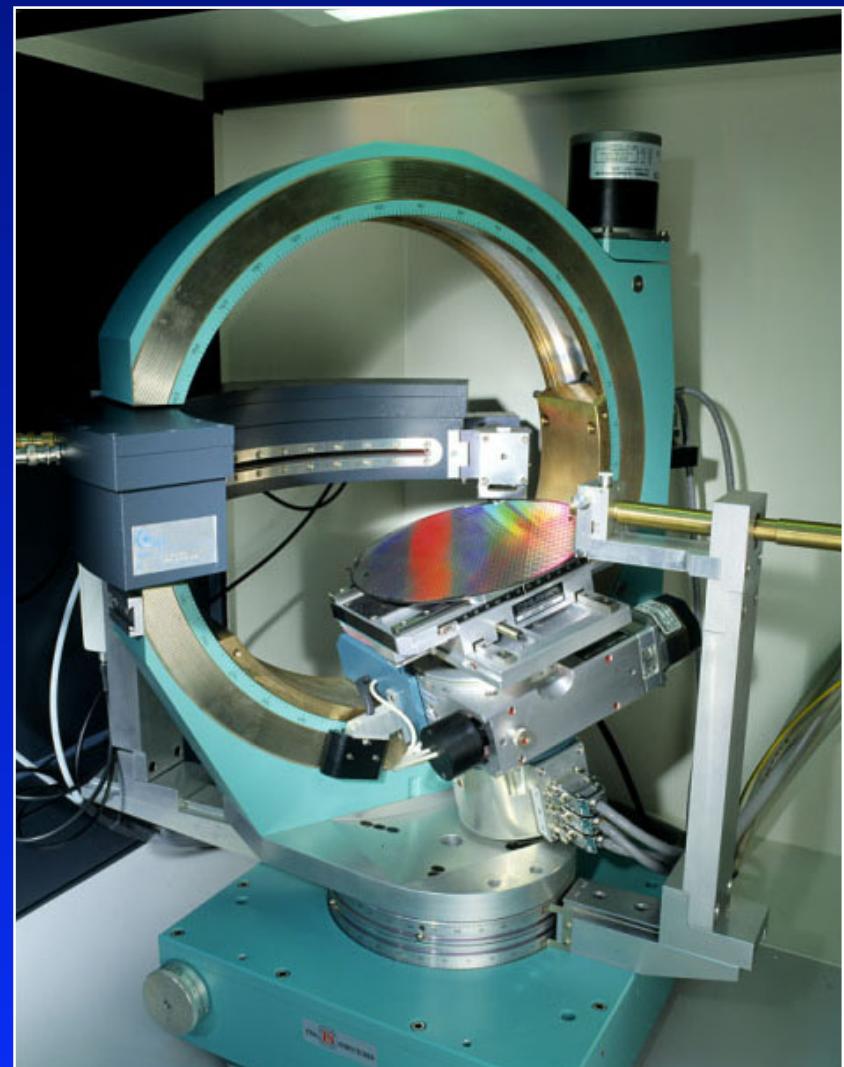
Si wafer substrate

*Spectra and reflectivity collected on
the ITS HR-XRD diffractometer
(right)*

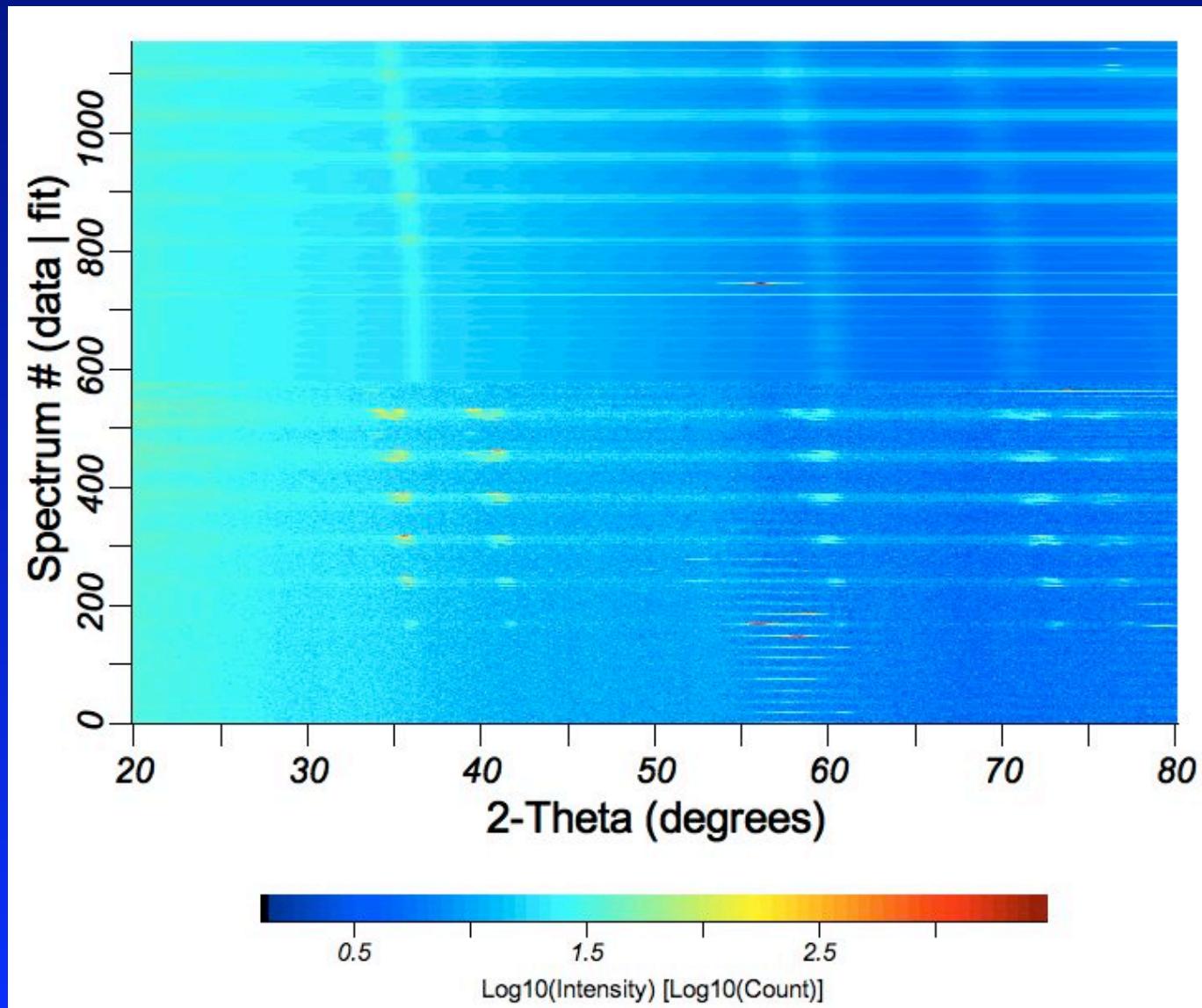
*120 degs position sensitive detector on
an eulerian cradle; multilayer as a
primary beam monochromator*

*Spectra collected in chi from 0 to 50
degrees in step of 5 deg and phi
from 0 to 360 in step of 5 also*

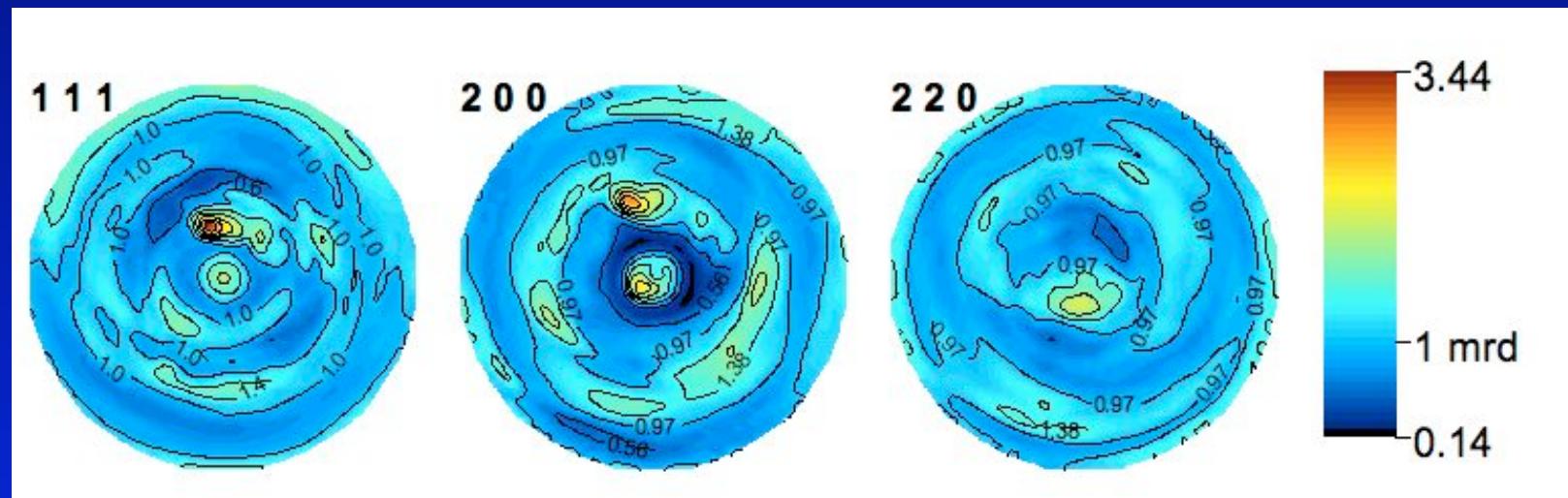
*E-WIMV and standard function for Si
substrate*



C182 Texture



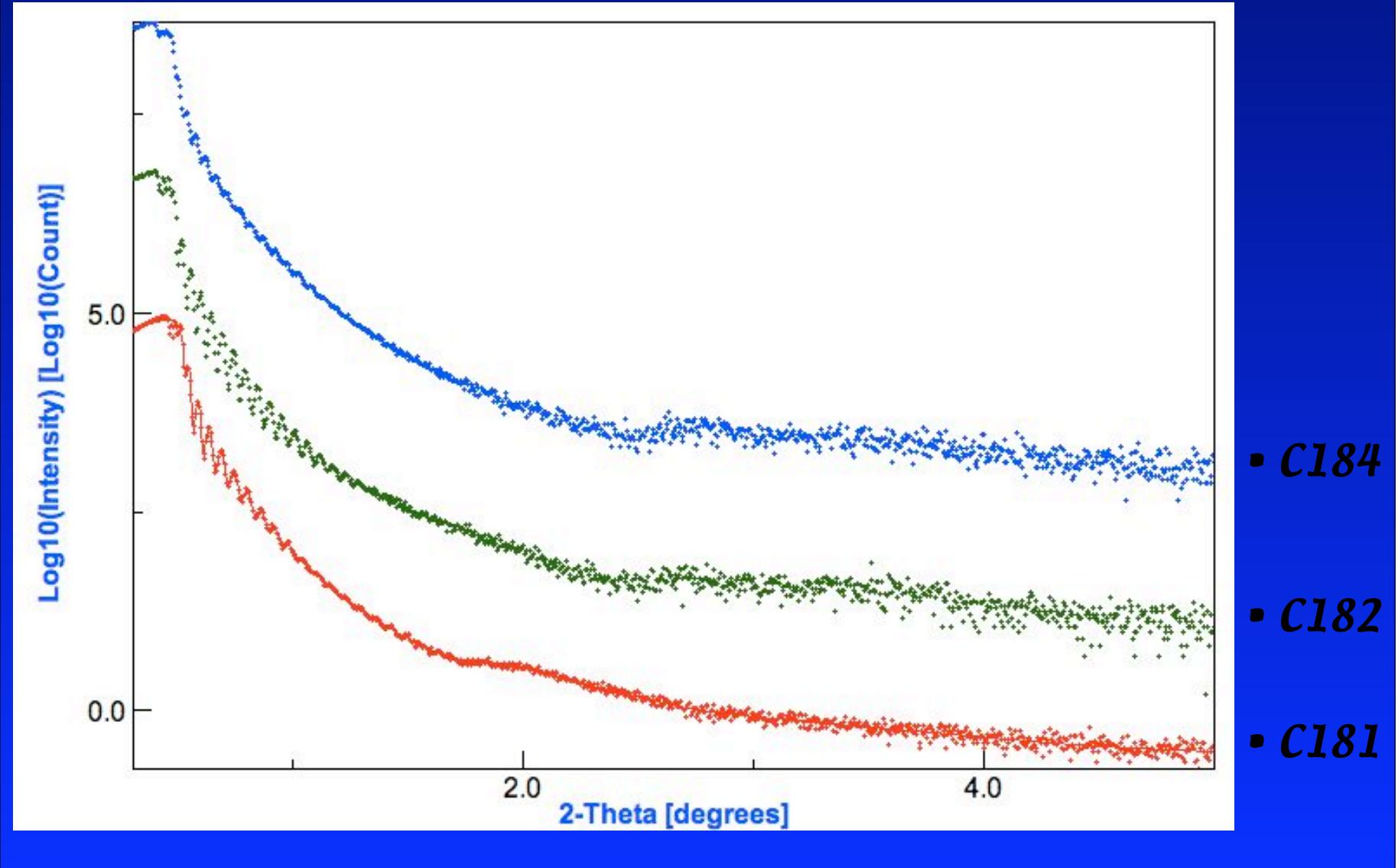
C182 Reconstructed pole figures



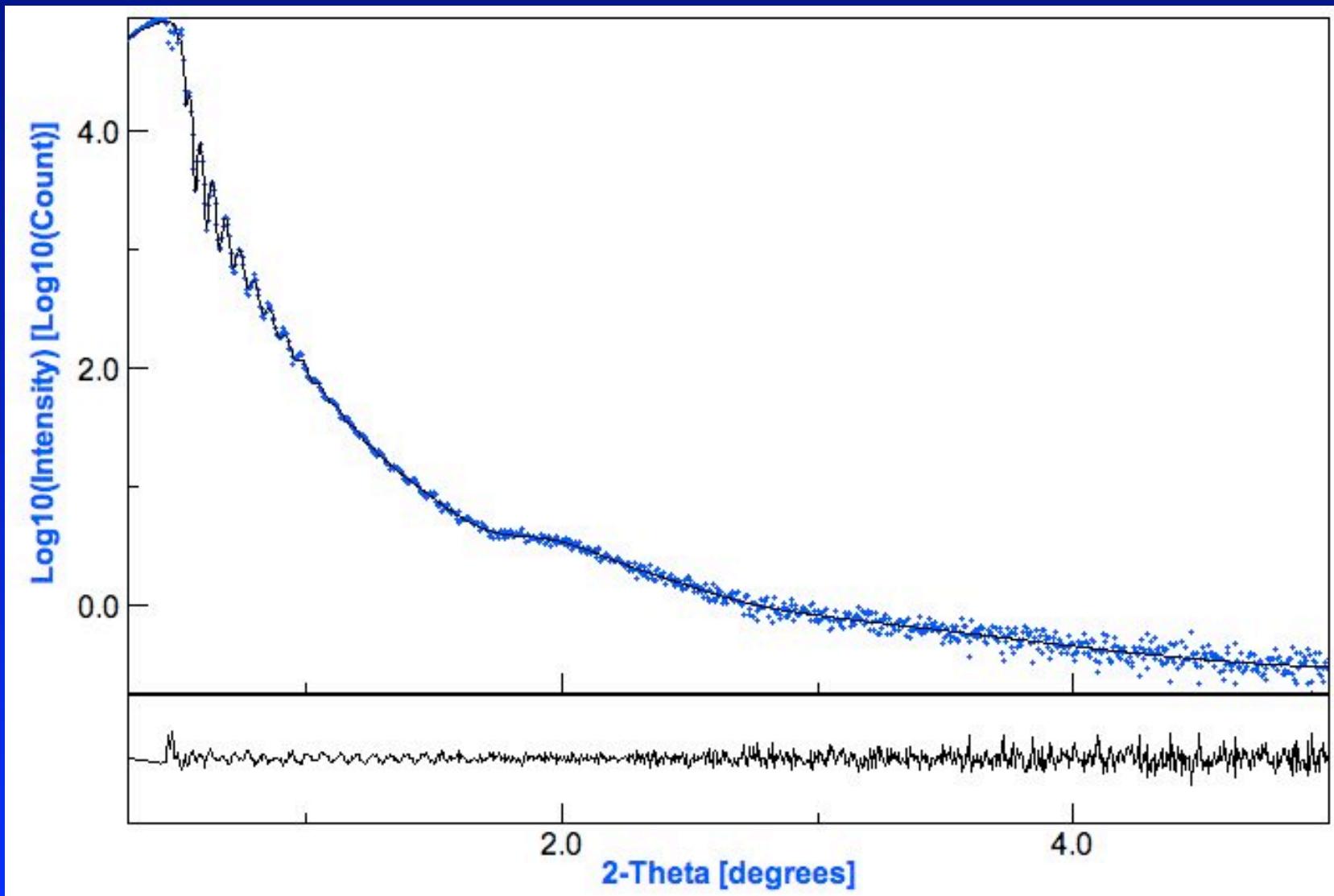
C182 diffraction result

- *Phase: SiC structure ??*
- *Texture as shown before*
- *Strain: very high in plane strain ~0.1 ??*
- *Relevant second kind microstress (not modeled)*
- *Crystallite size ~400 Angstrom, microstrain ~0.1*

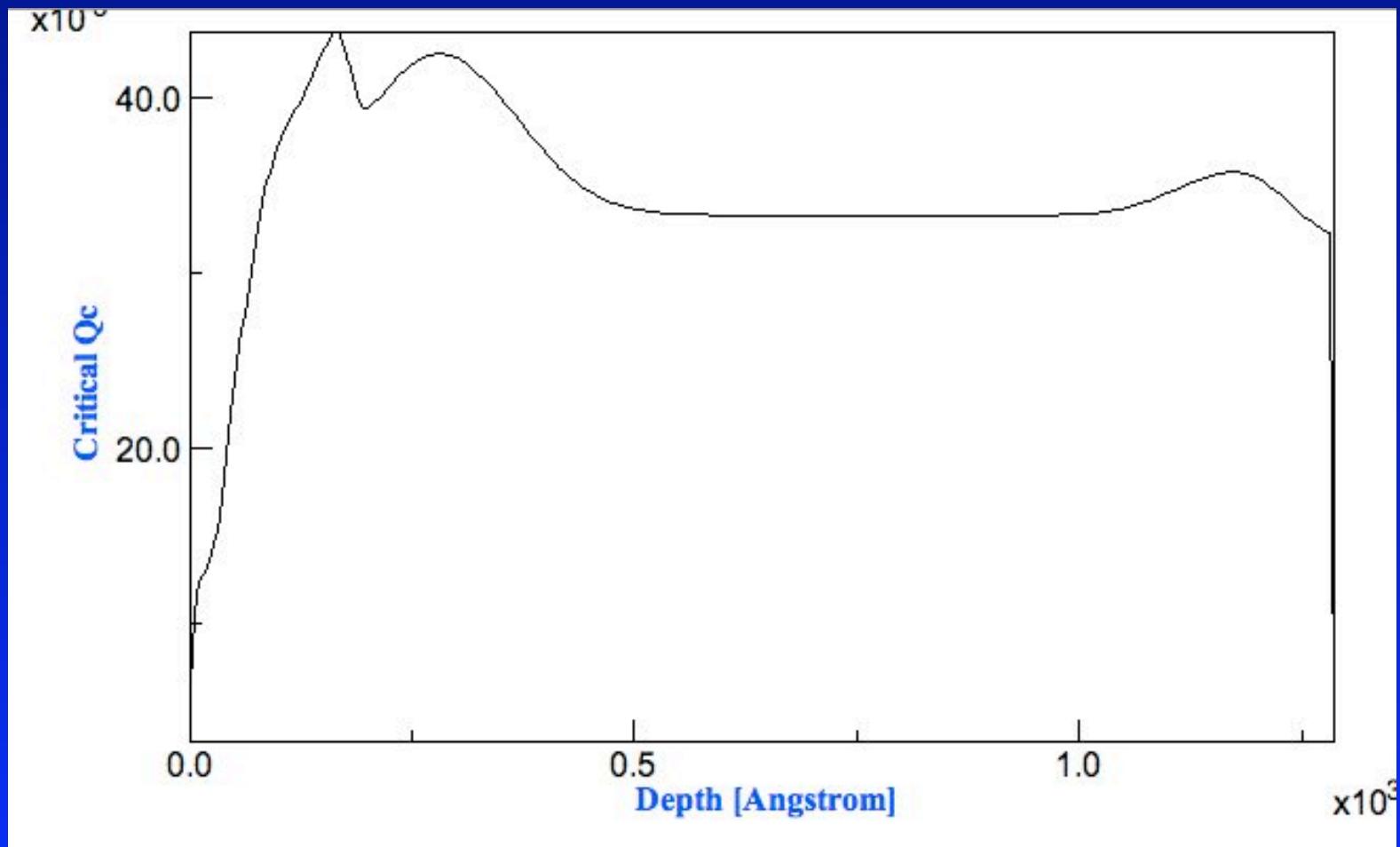
C18x reflectivity



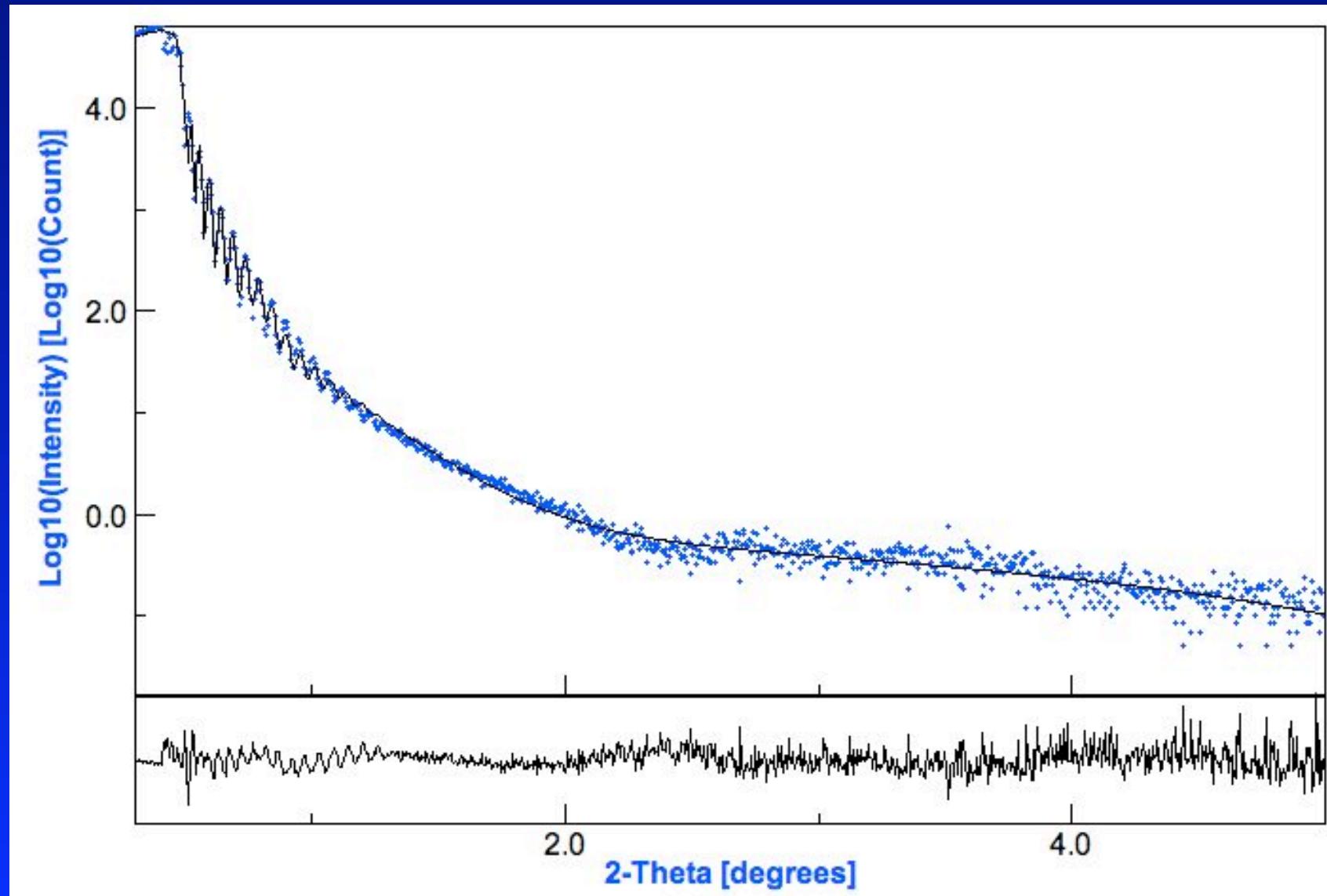
C18I - DCD model



C181 - DCD model



C182 - DCD model



C182 - DCD model

