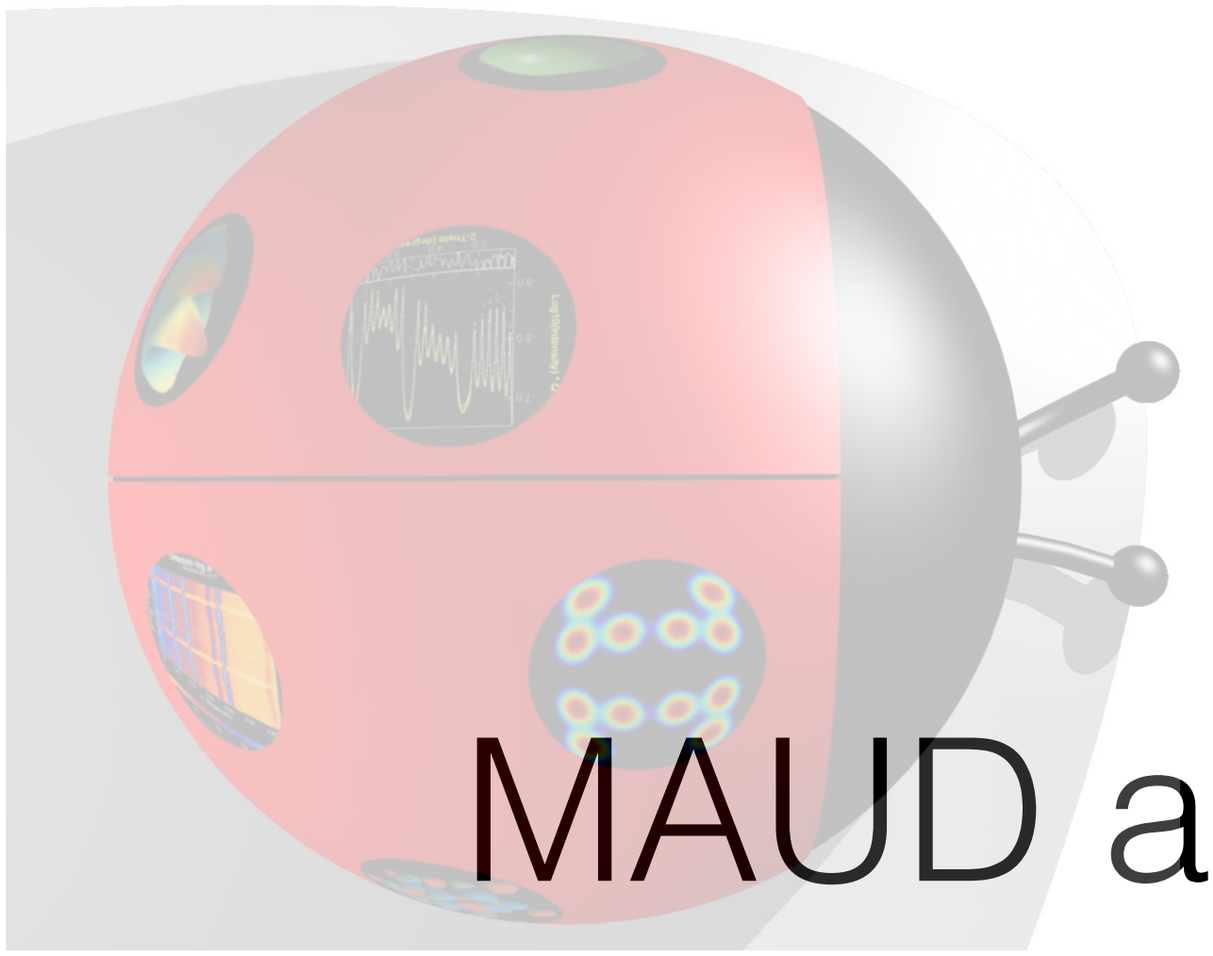


MAUD and MTEX

Luca Lutterotti

UNIVERSITY OF TRENTO - Italy

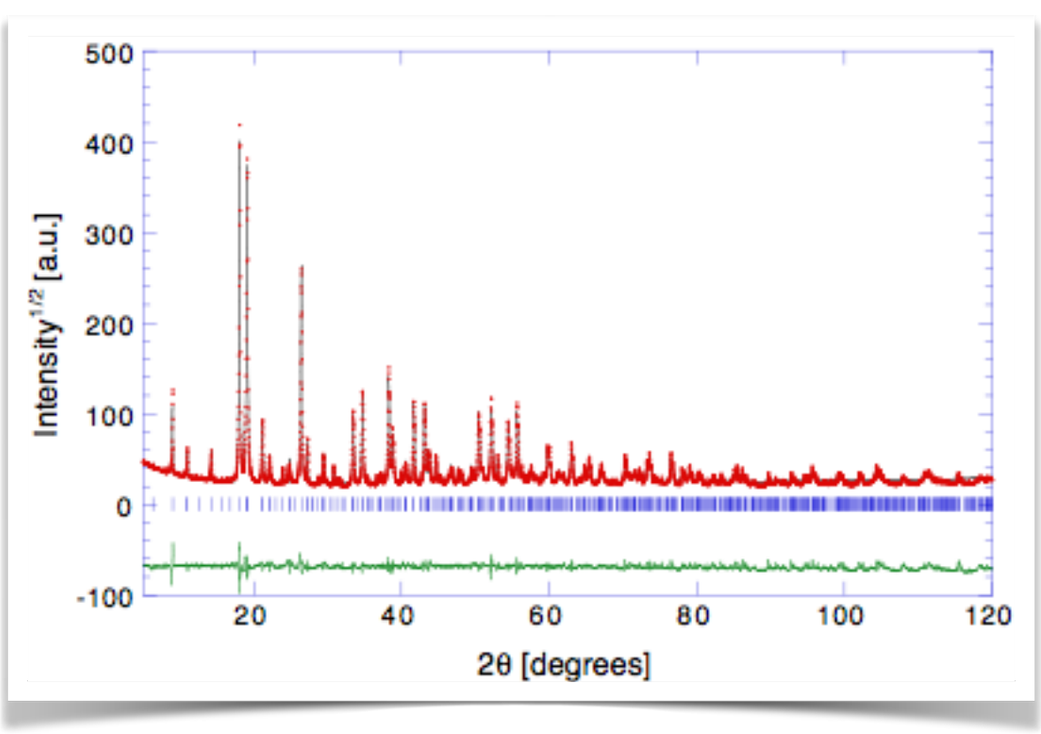
Department of Industrial Engineering

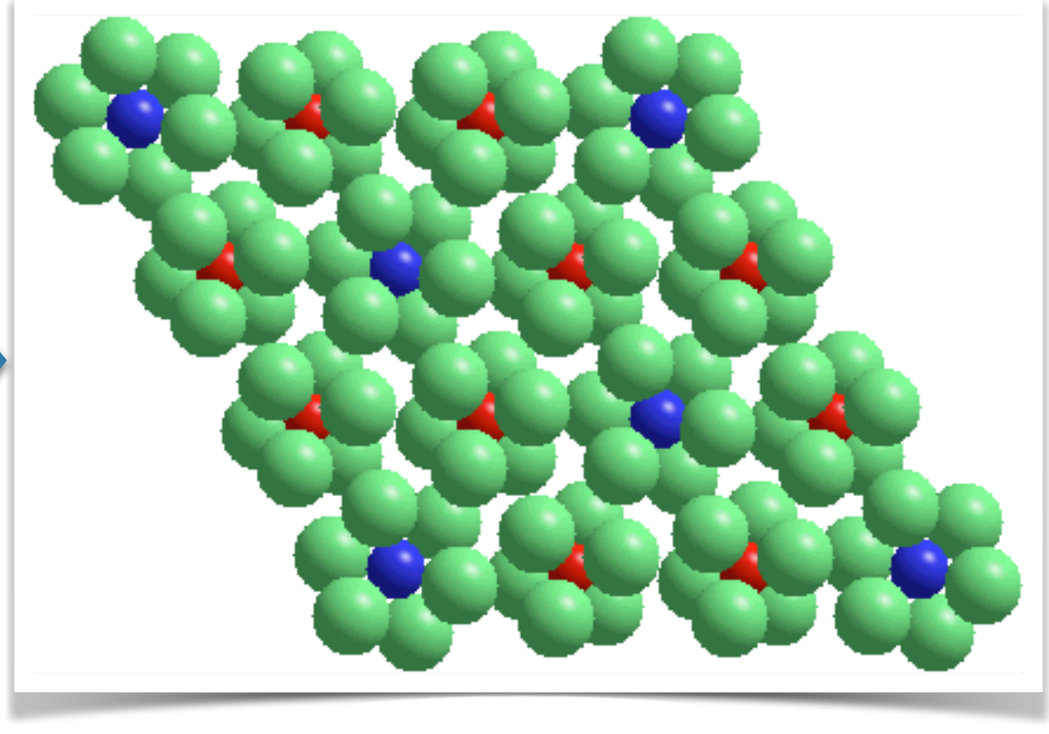
Combined analysis: general principles and theory

Pattern fitting: the Rietveld method

- Least squares minimization of:

$$WSS = \sum_i w_i \left(I_i^{\exp} - I_i^{calc} \right)^2 , w_i = \frac{1}{I_i^{\exp}}$$





XRD Pattern calculation: general formula

$$I_i^{calc} = S_F \sum_{j=1}^{Nphases} \frac{f_j}{V_j^2} \sum_{k=1}^{Npeaks} L_k \left|F_{k,j}\right|^2 S_j\left(2\theta_i - 2\theta_{k,j}\right) P_{k,j} A_j + bkg_i$$

Crystal structure

Phase quantities

Crystallite sizes and microstrains

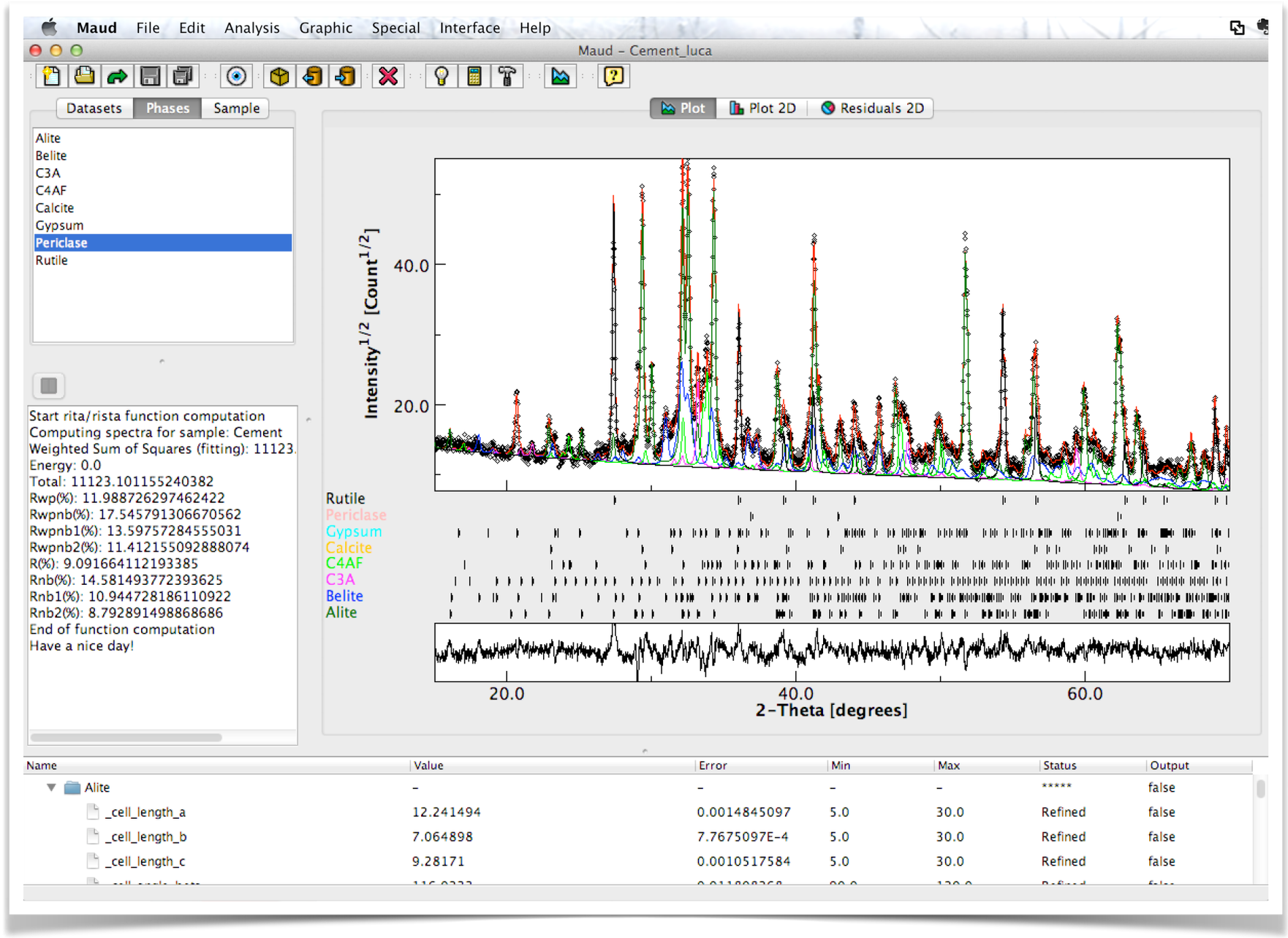
Residual stresses (and cell parameters)

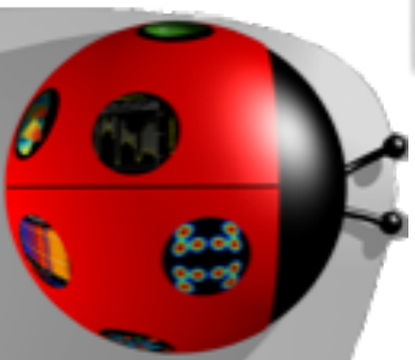
Texture (ODF)

Film thickness

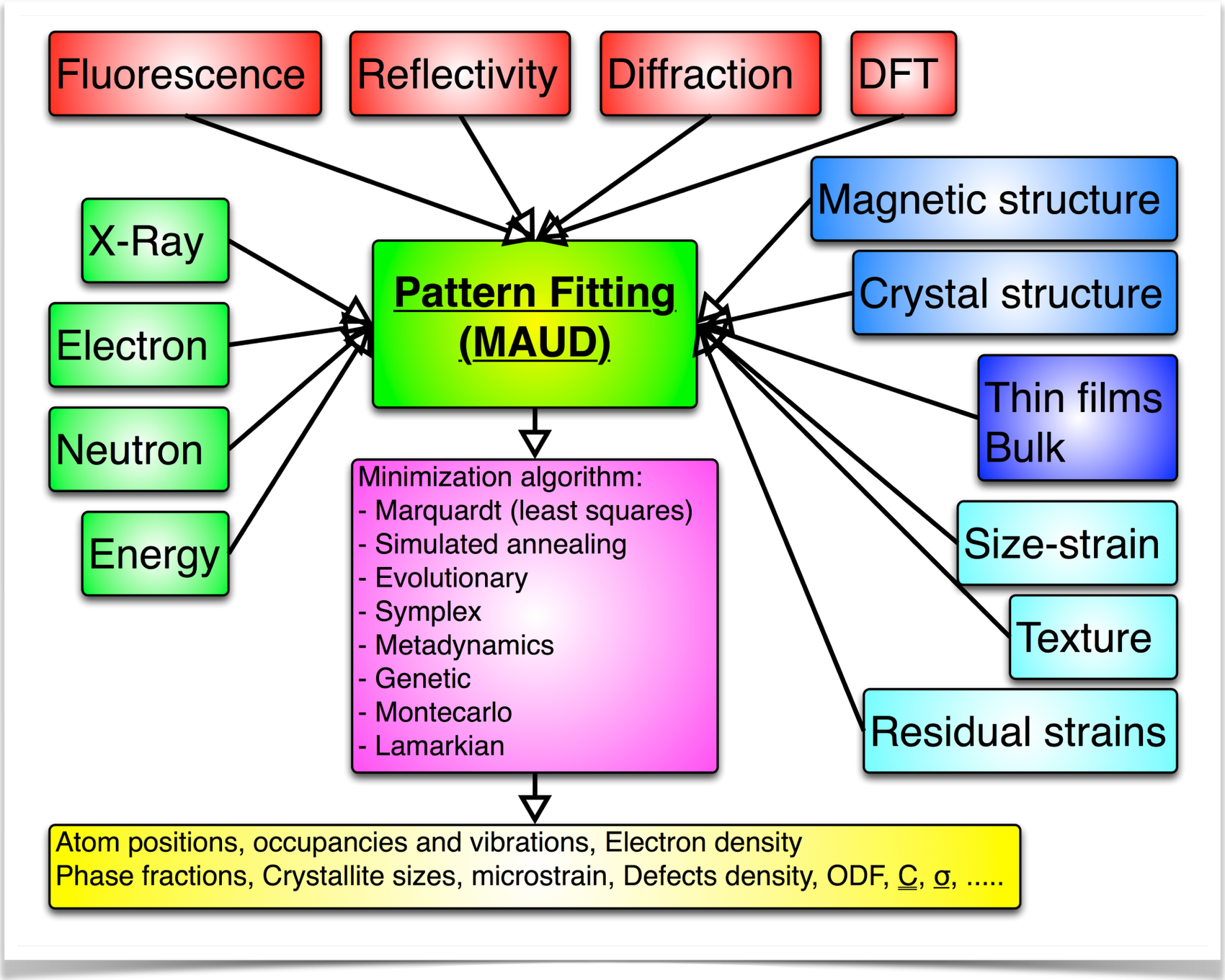
The MAUD program

Materials Analysis Using Diffraction





- http://maud.radiographema.com



MAUD structure and models

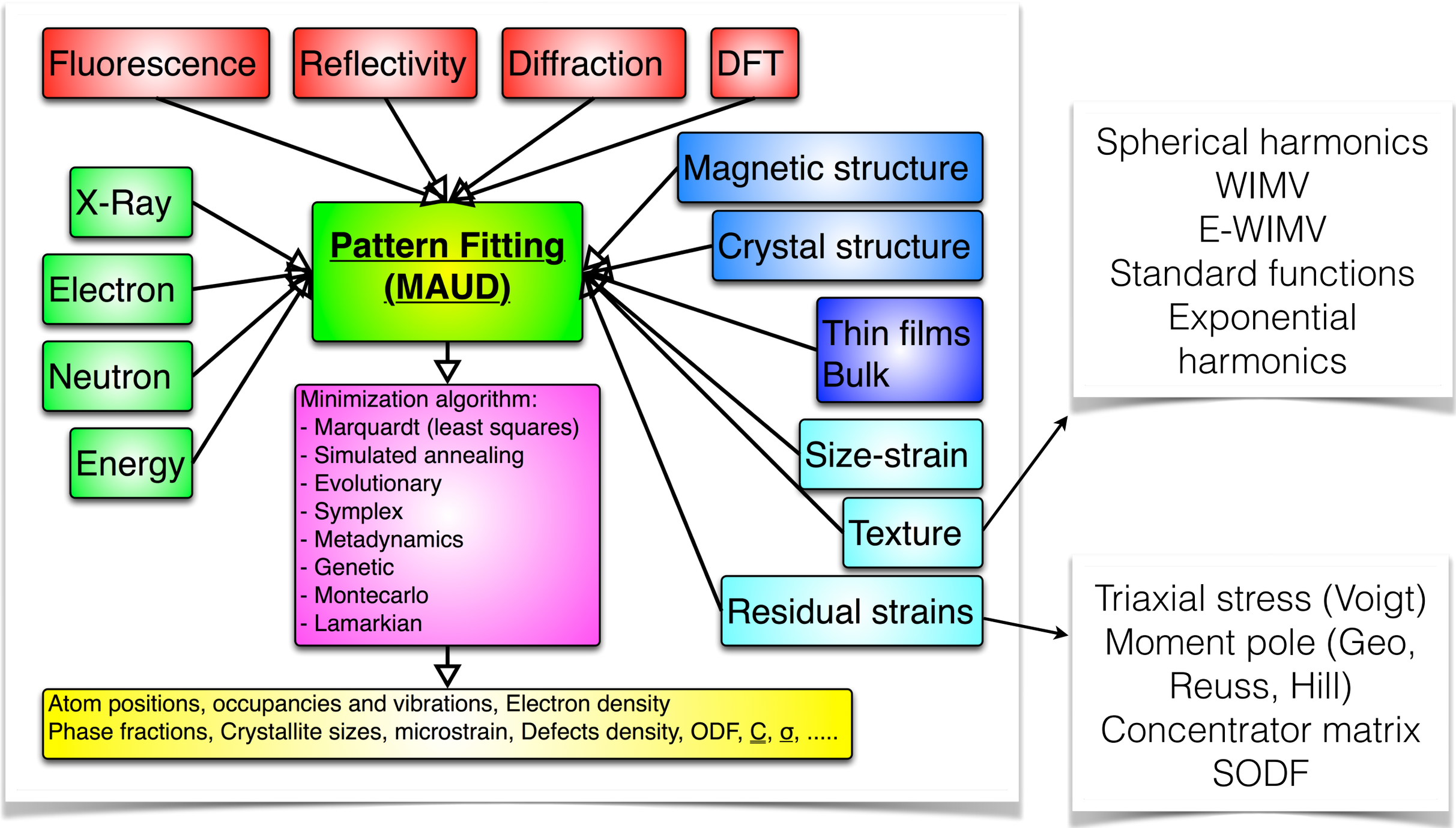
actual structure

Fluorescence
Reflectivity
Diffraction
DFT
X-Ray
Electron
Neutron
Energy
Pattern Fitting (MAUD)
Magnetic structure
Crystal structure
Thin films
Bulk
Size-strain
Texture
Residual strains
Minimization algorithm:
- Marquardt (least squares)
- Simulated annealing
- Evolutionary
- Symplex
- Metadynamics
- Genetic
- Montecarlo
- Lamarkian
Atom positions, occupancies and vibrations, Electron density
Phase fractions, Crystallite sizes, microstrain, Defects density, ODF, C, σ, ...

Spherical harmonics
WIMV
E-WIMV
Standard functions
Exponential
harmonics

MAUD structure and models

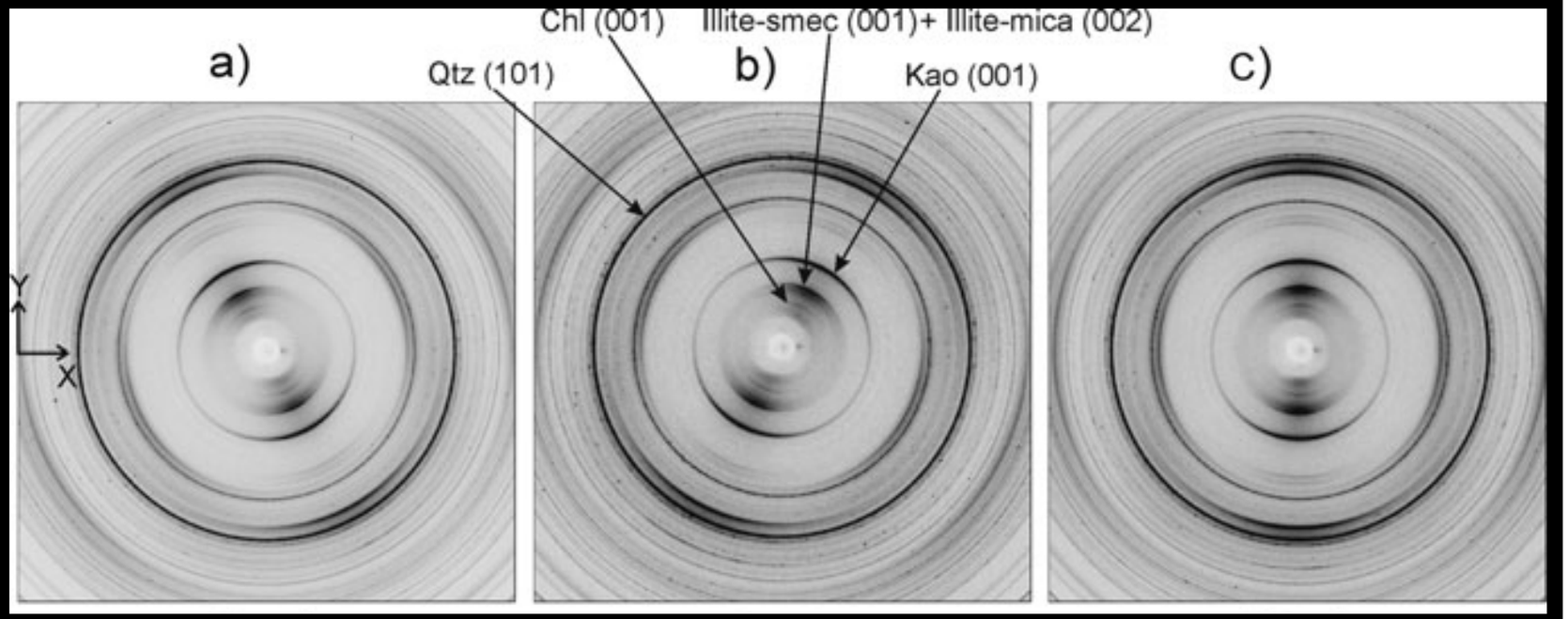
actual structure

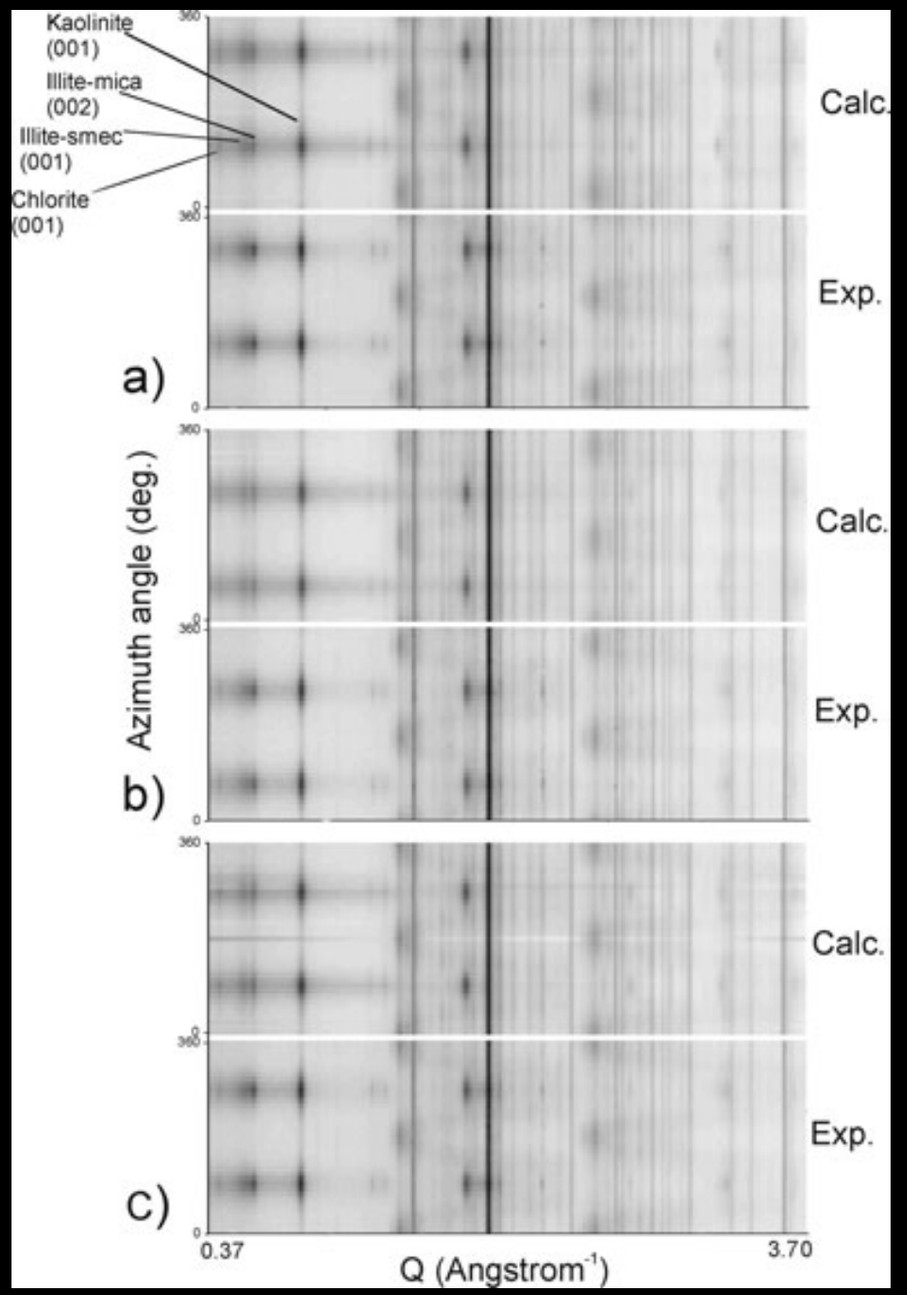


MAUD structure and models

actual structure

Texture in shales





Geophysical Prospecting

EAGE EUROPEAN ASSOCIATION OF GEOSCIENTISTS & ENGINEERS

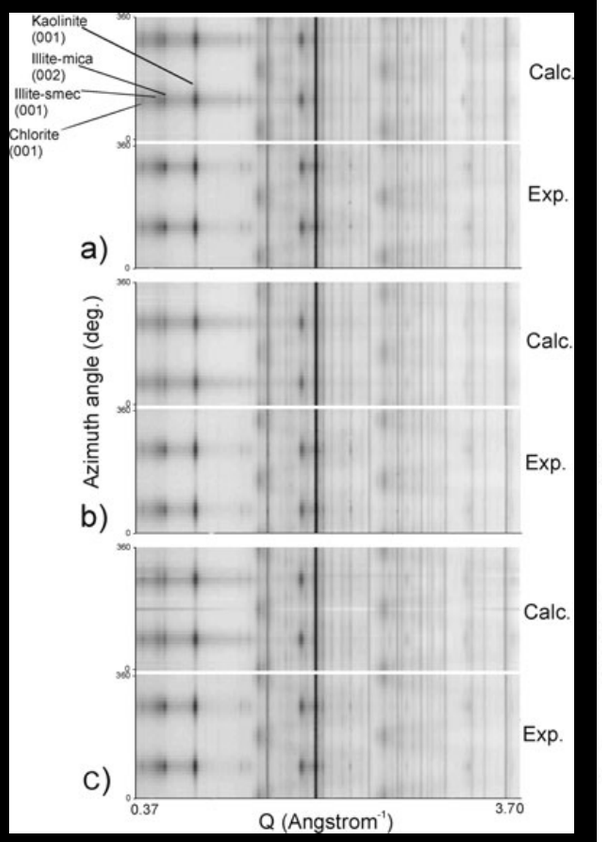
Geophysical Prospecting, 2011, **59**, 536–556

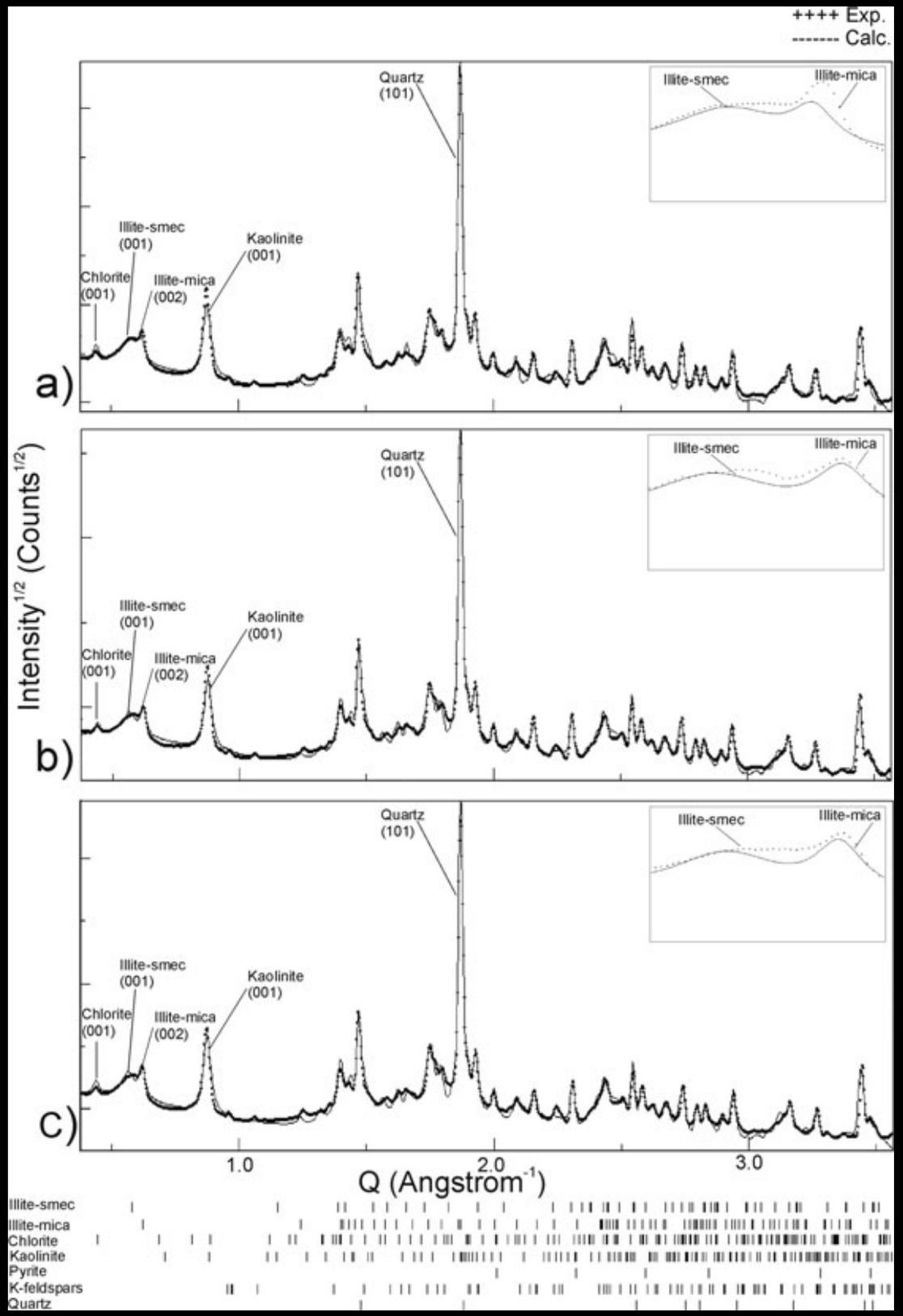
doi: 10.1111/j.1365-2478.2010.00942.x

Texture and anisotropy analysis of Qusaiba shales

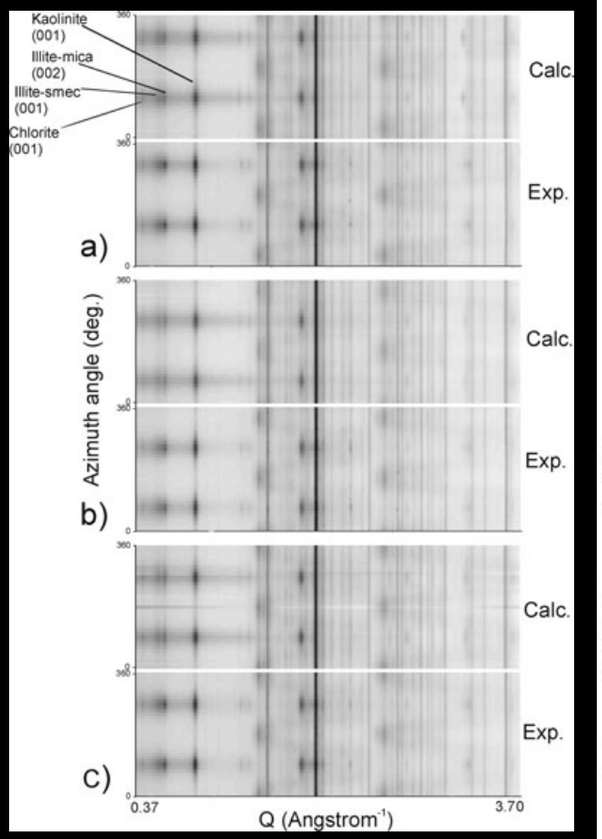
Waruntorn Kanitpanyacharoen[1], Hans-Rudolf Wenk[1]*, Frans Kets[2,3], Christian Lehr[3] and Richard Wirth[4]

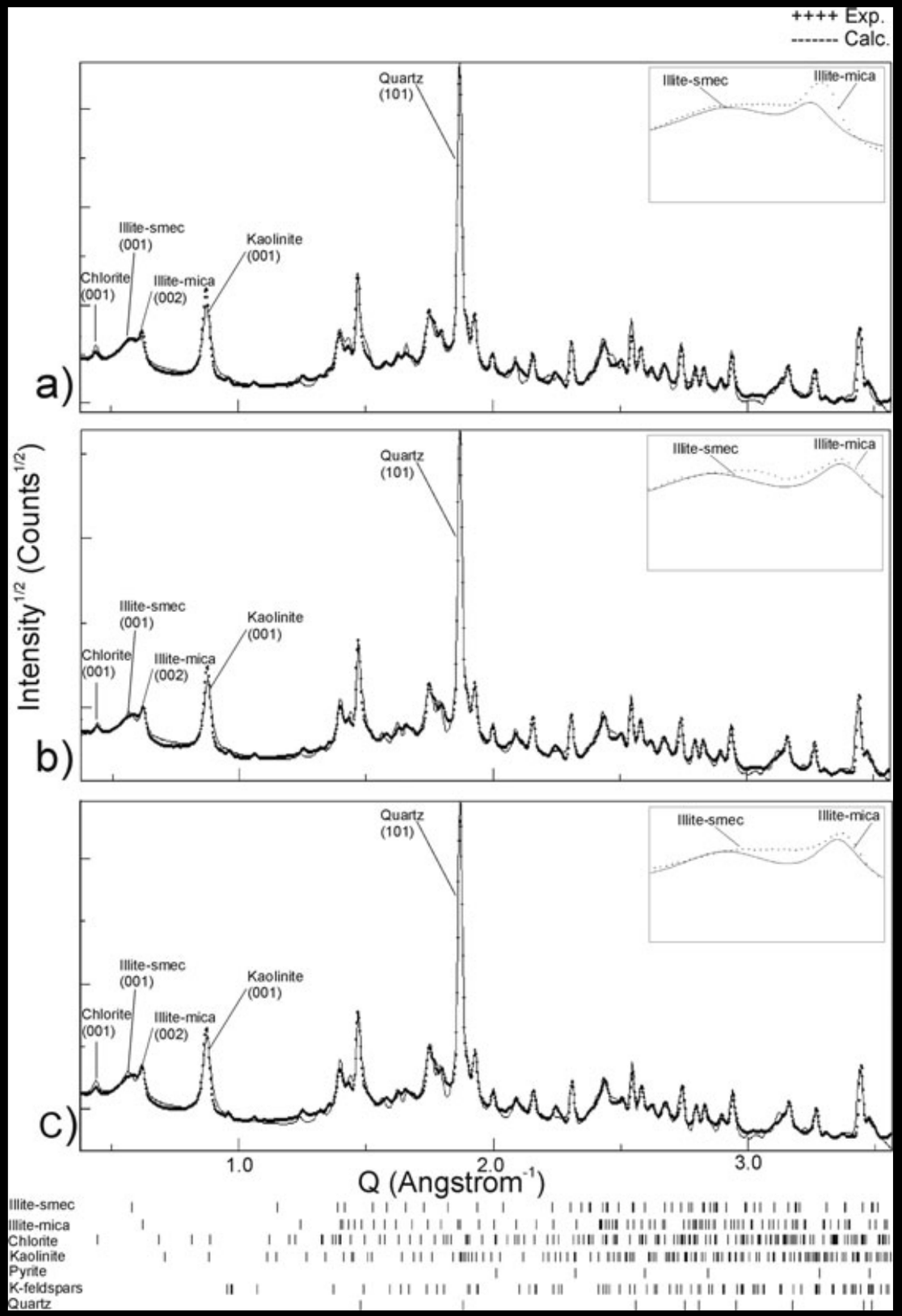
Texture in shales

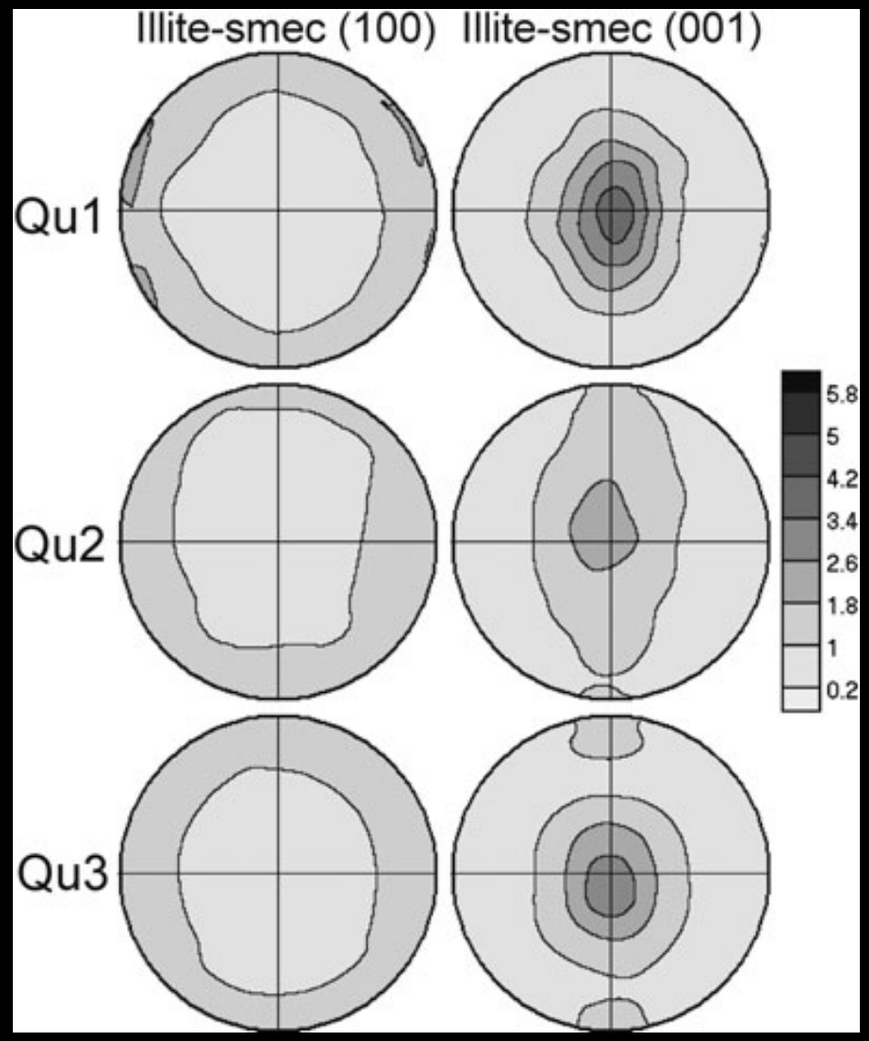




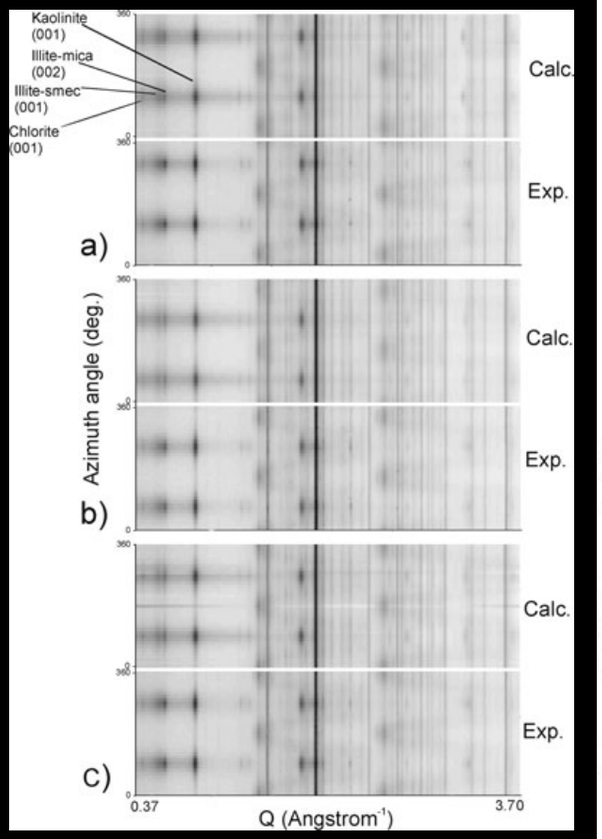
Texture in shales

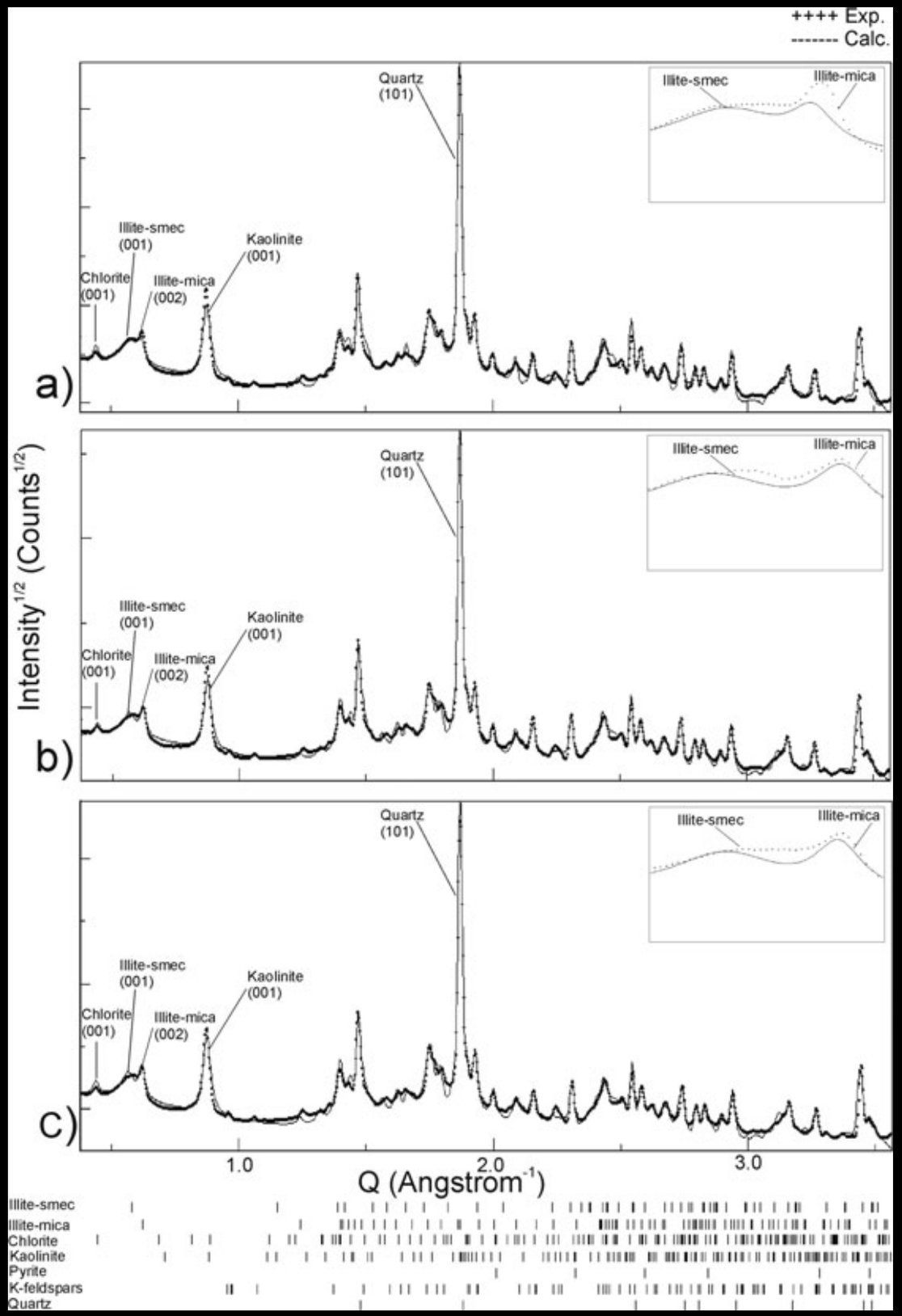


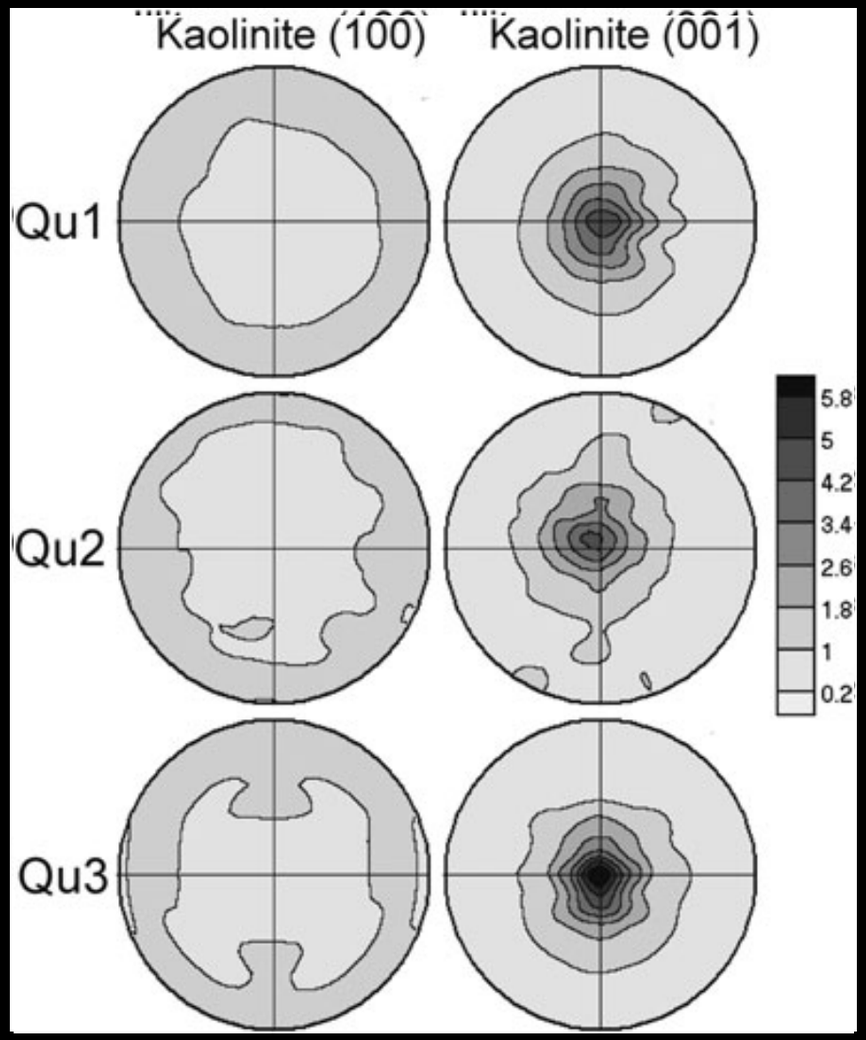




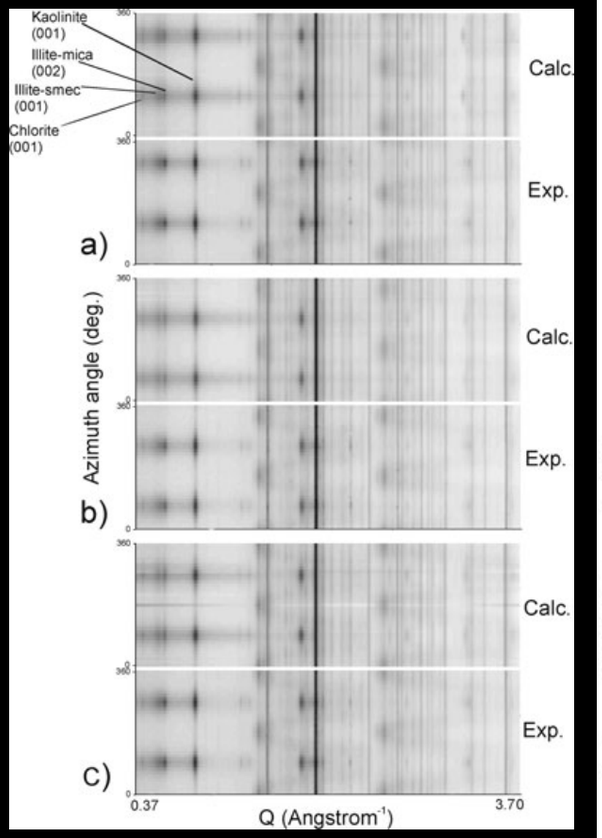
Texture in shales

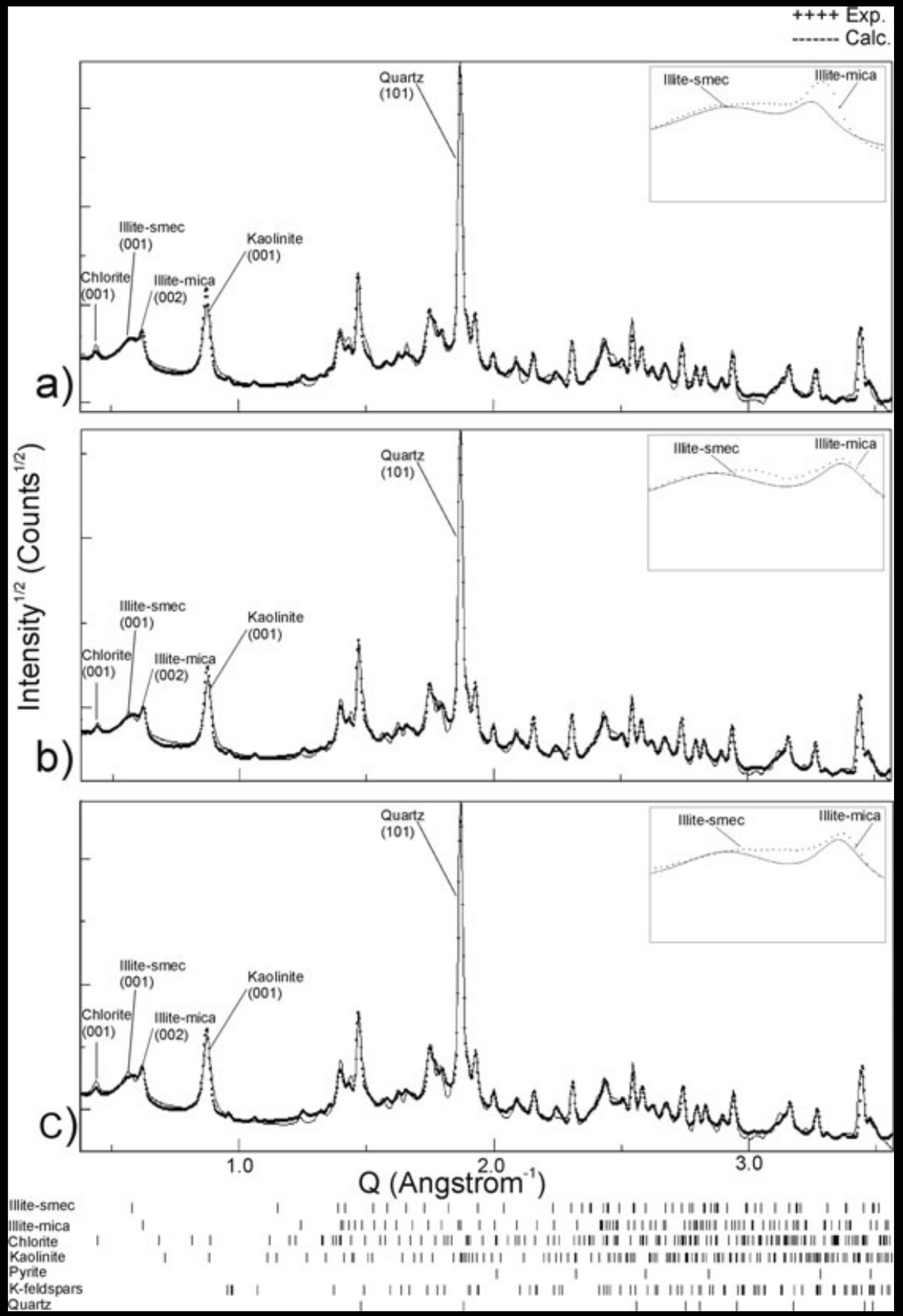


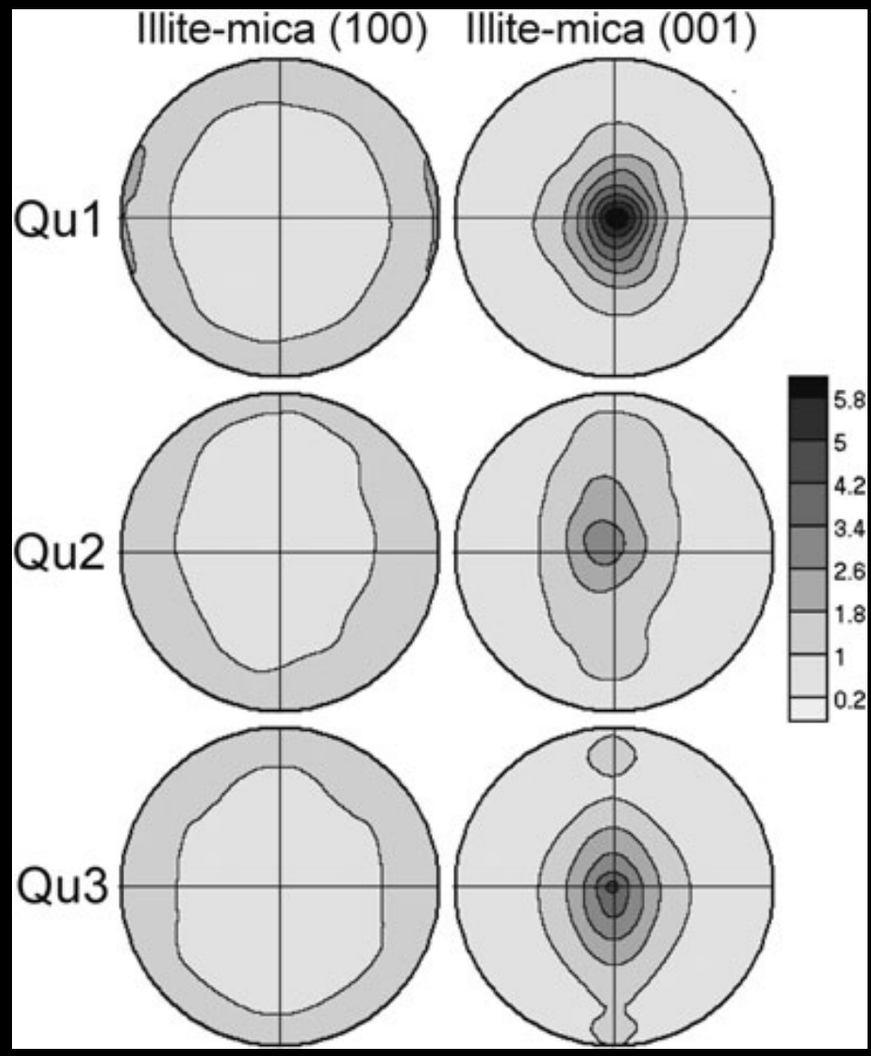




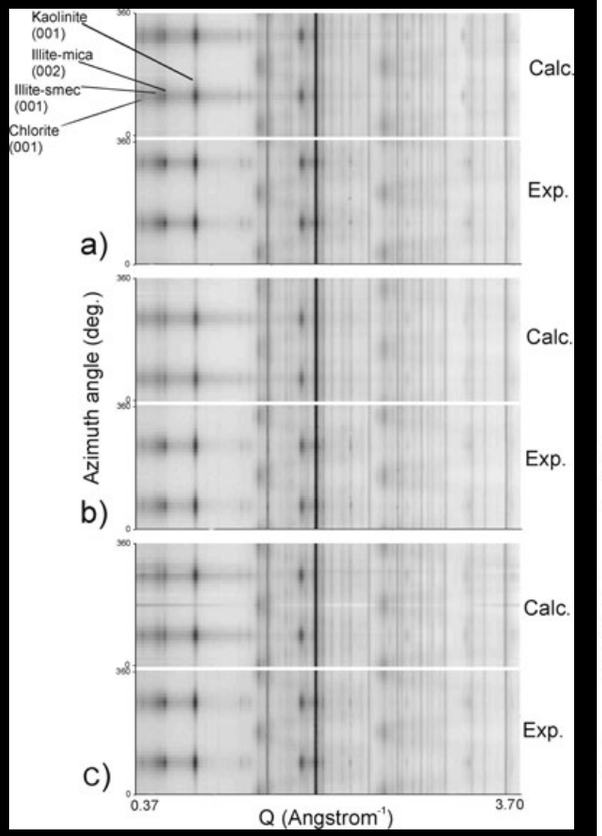
Texture in shales

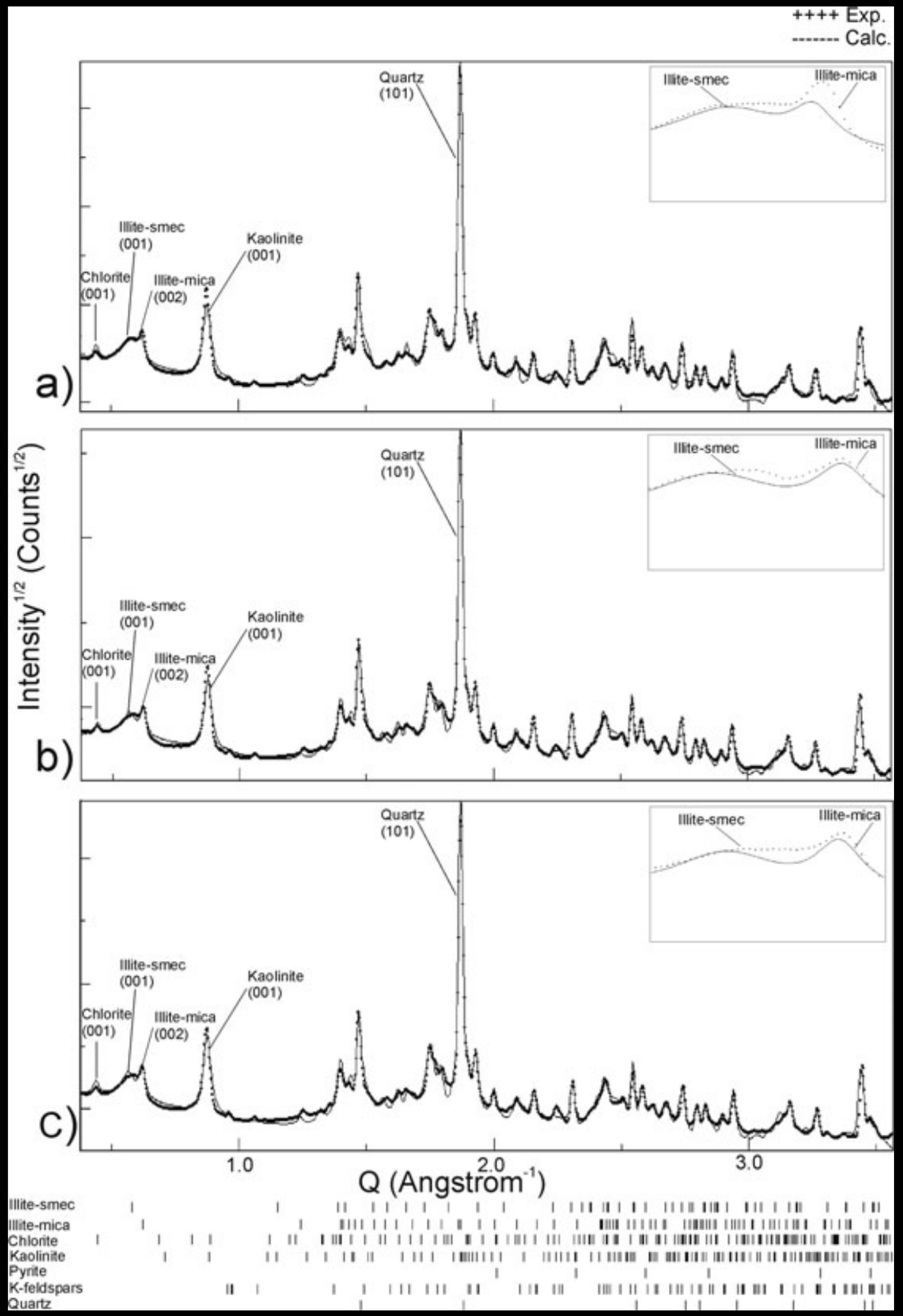


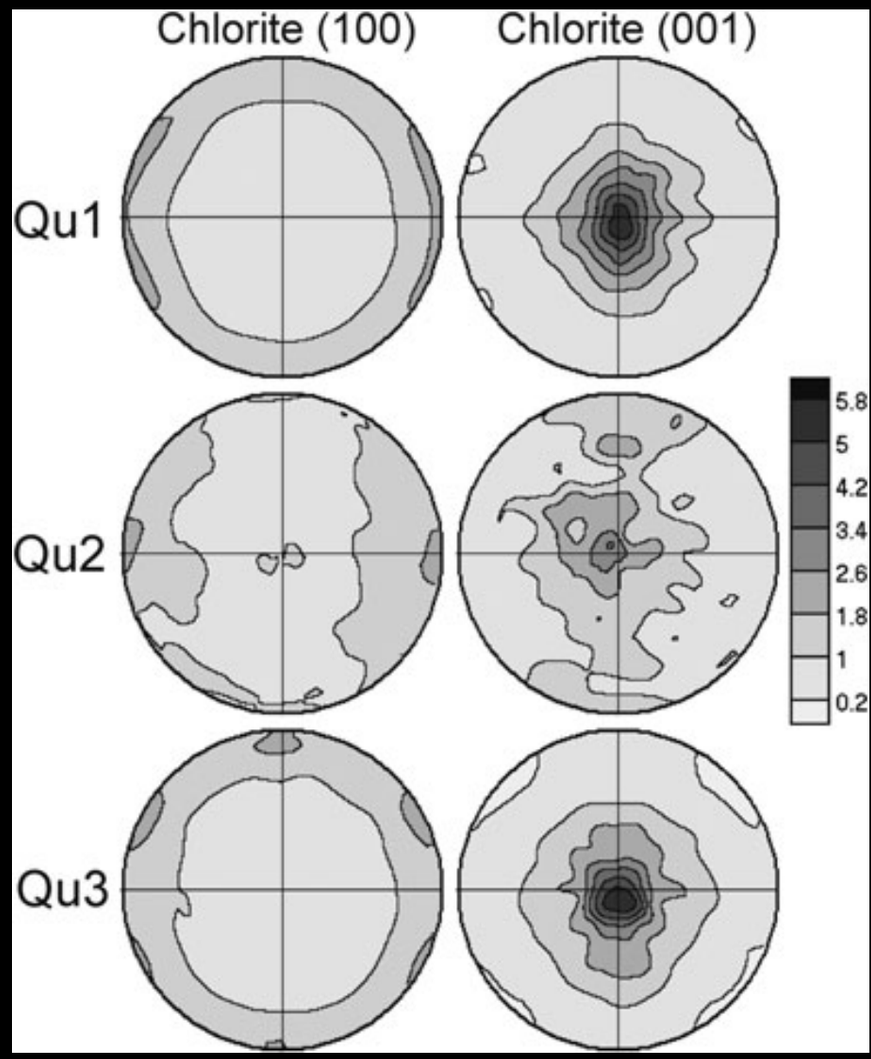




Texture in shales

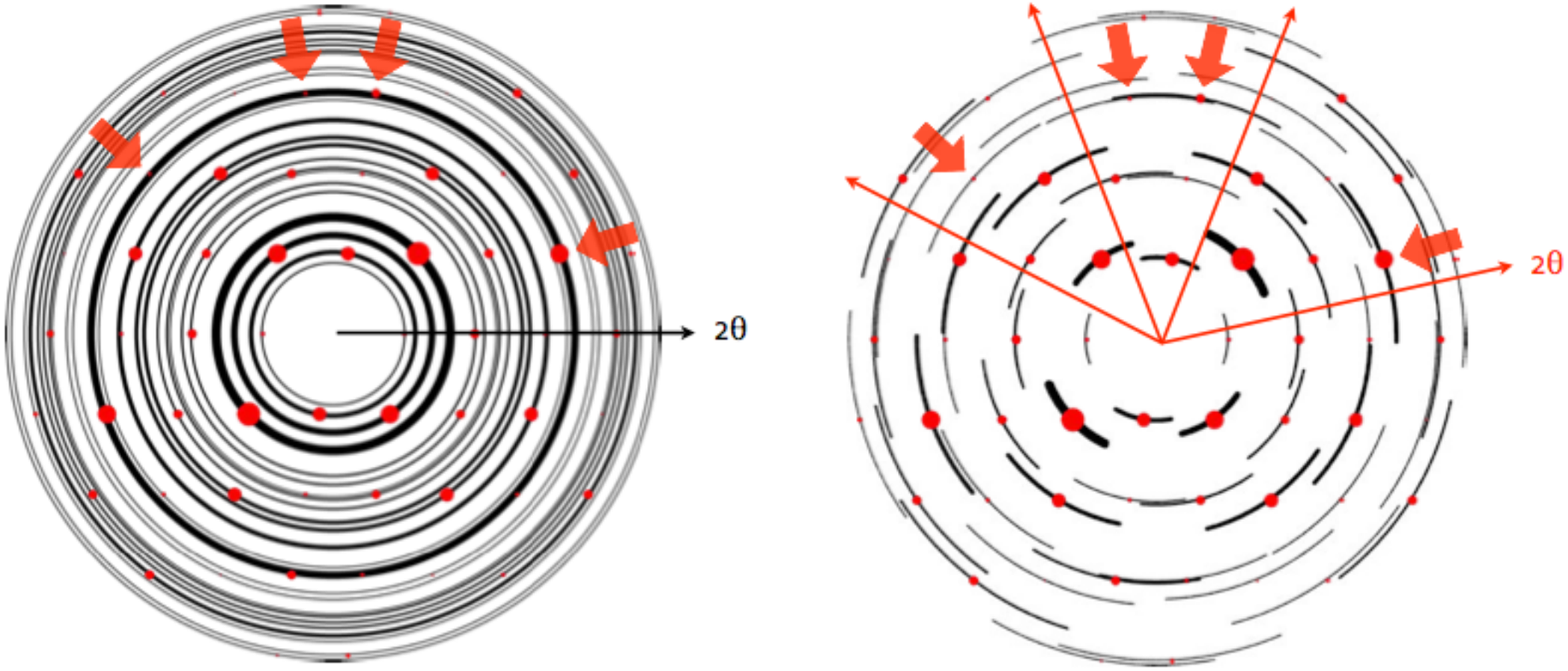






Crystal structure solution by texture

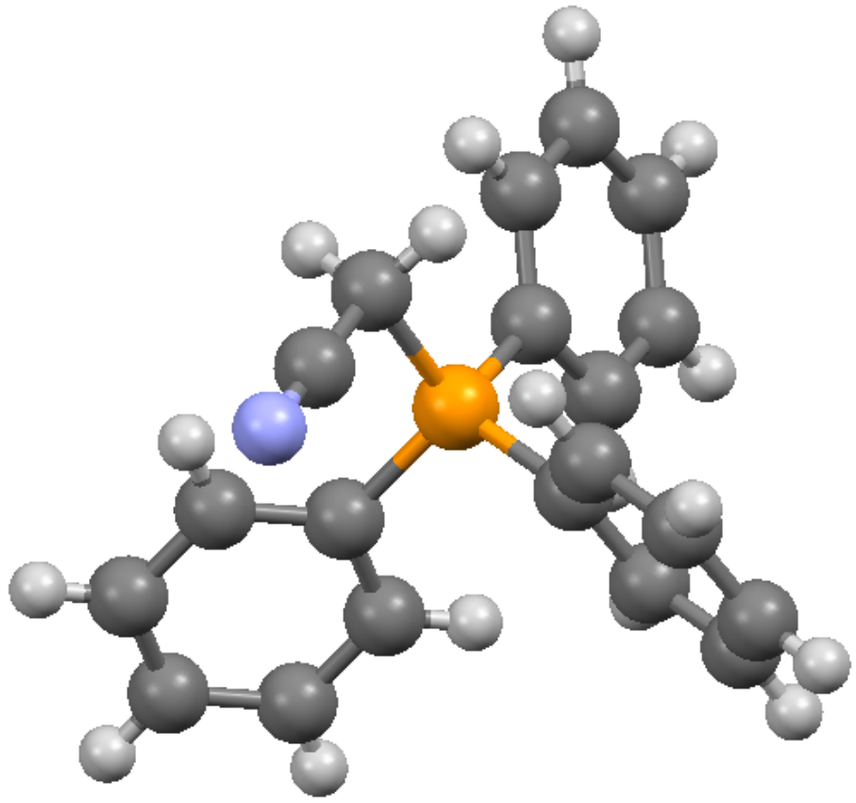
Wessels T., Baerlocher Ch. and McCusker L.B., Science, 284, 477-479, 1999

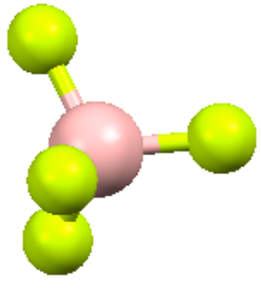


- Problems:
  - two experiments and two analyses (first texture measurement, than high resolution collection for few sample orientations)
  - time consuming experiment
  - as texture becomes sharper (better for resolving), errors in sample positioning/ orientation compromise the solution
- With sharp texture, Rietveld Texture Analysis with EWIMV is needed

Crystal structure solution

- Standard function for texture
- 4 fragments + 2 subfragments
- Lamarkian optimization





Crystal structure solution

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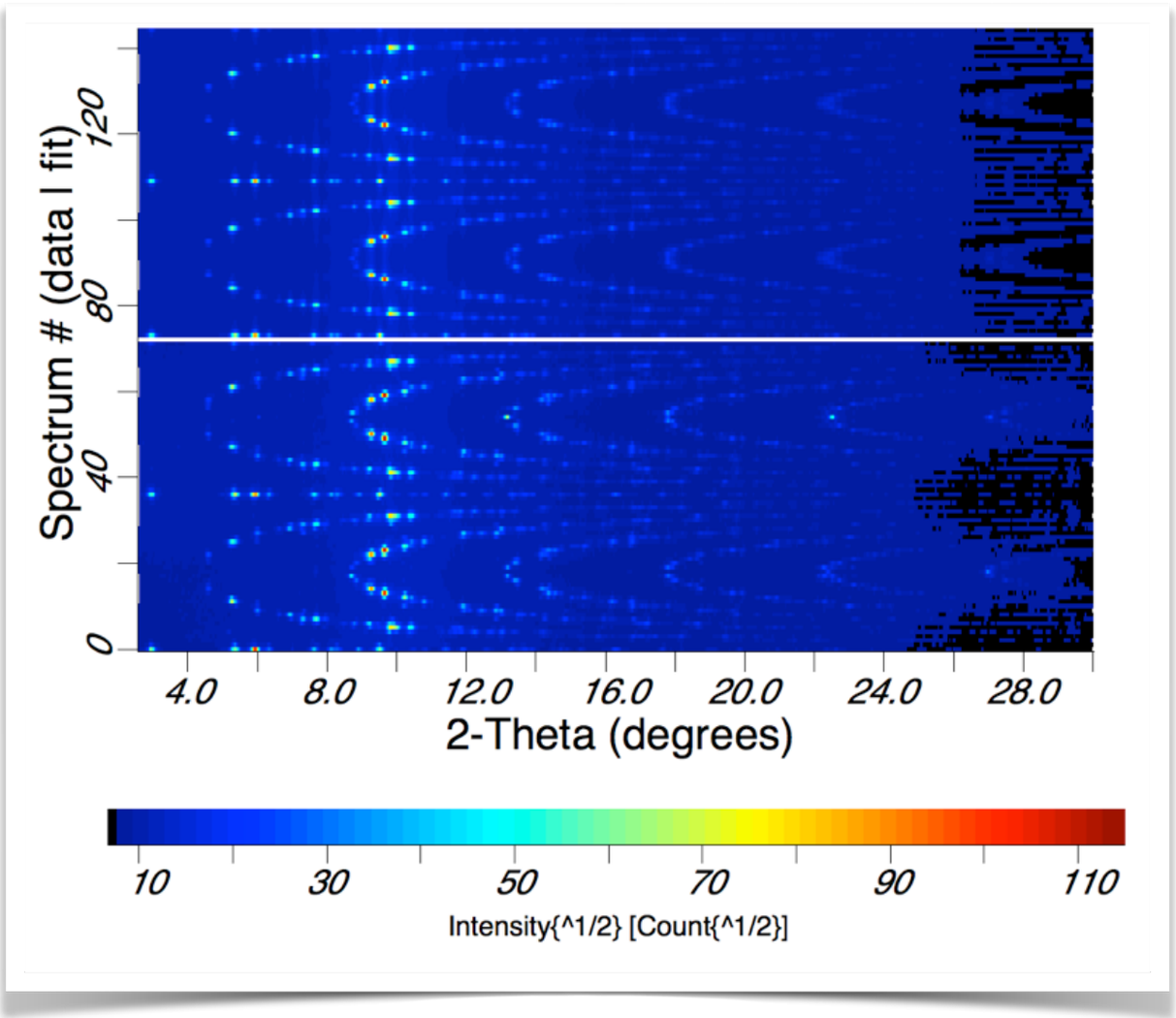
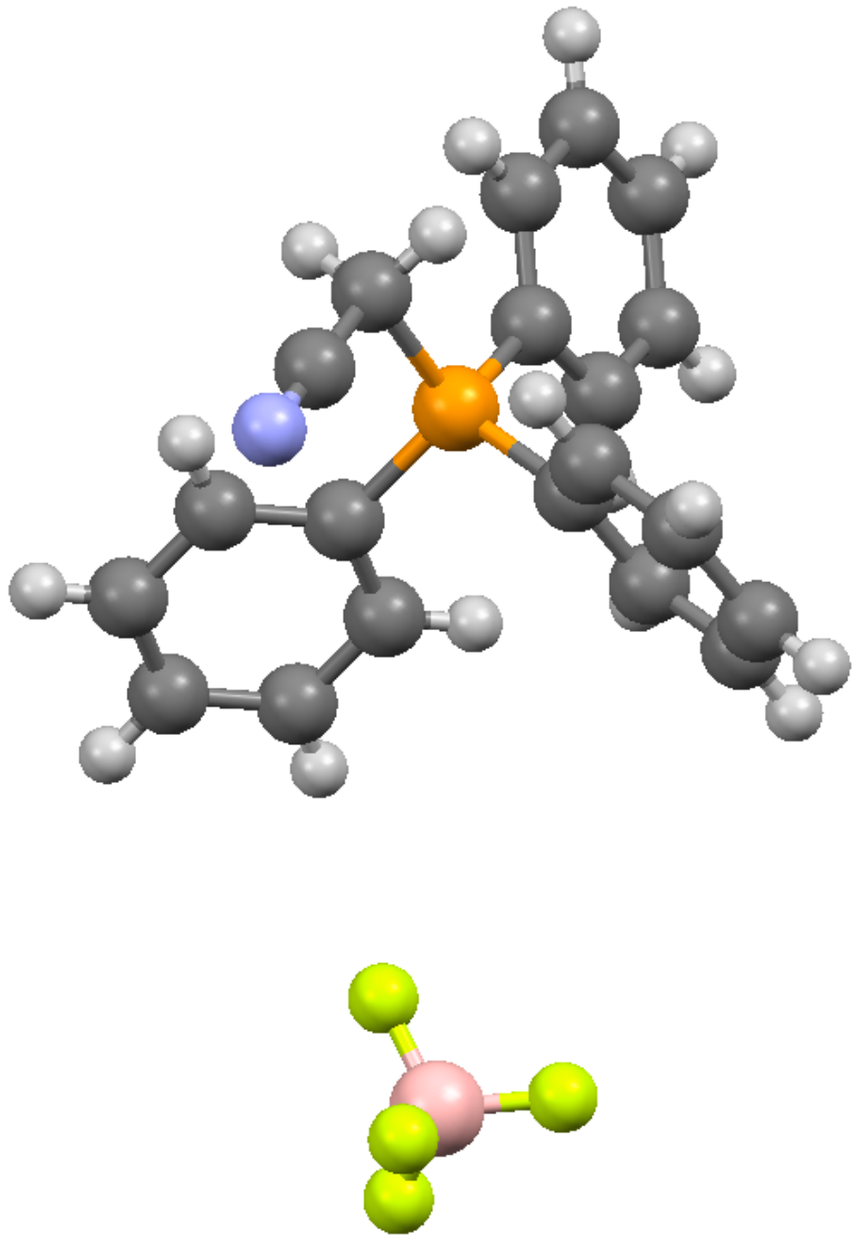
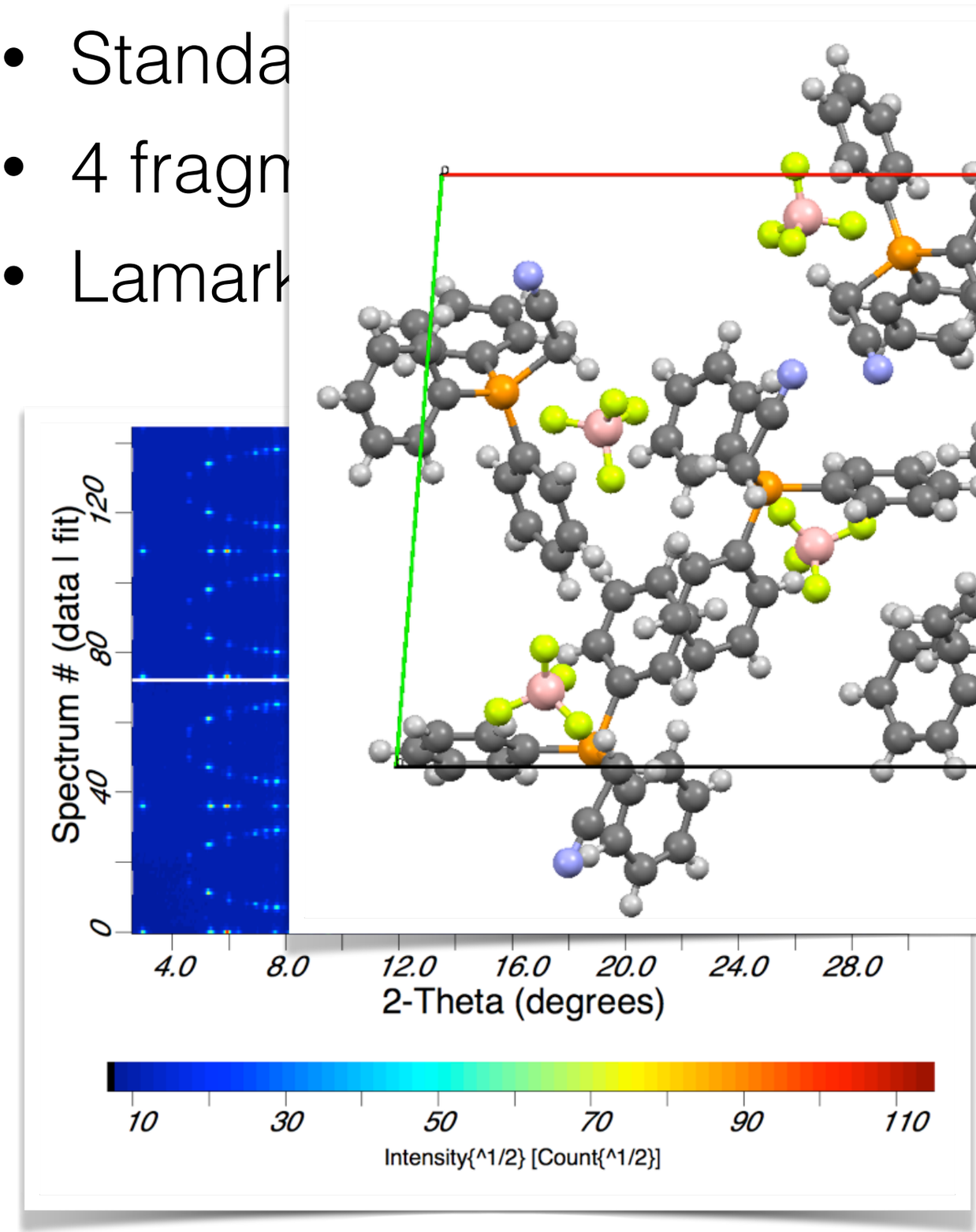


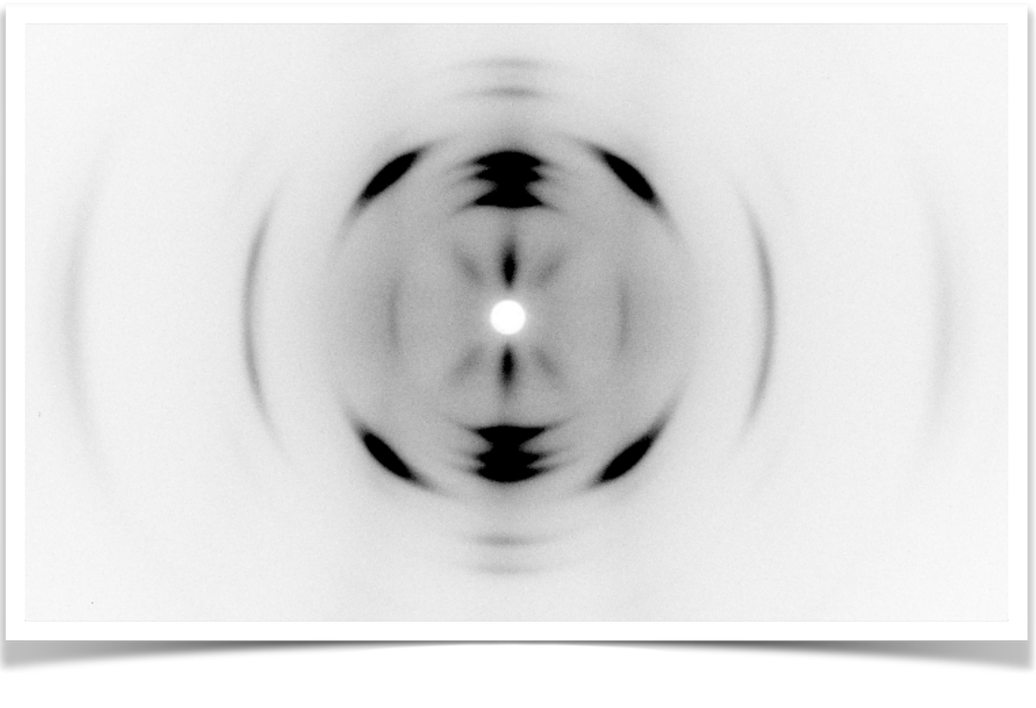


Crystal structure solution

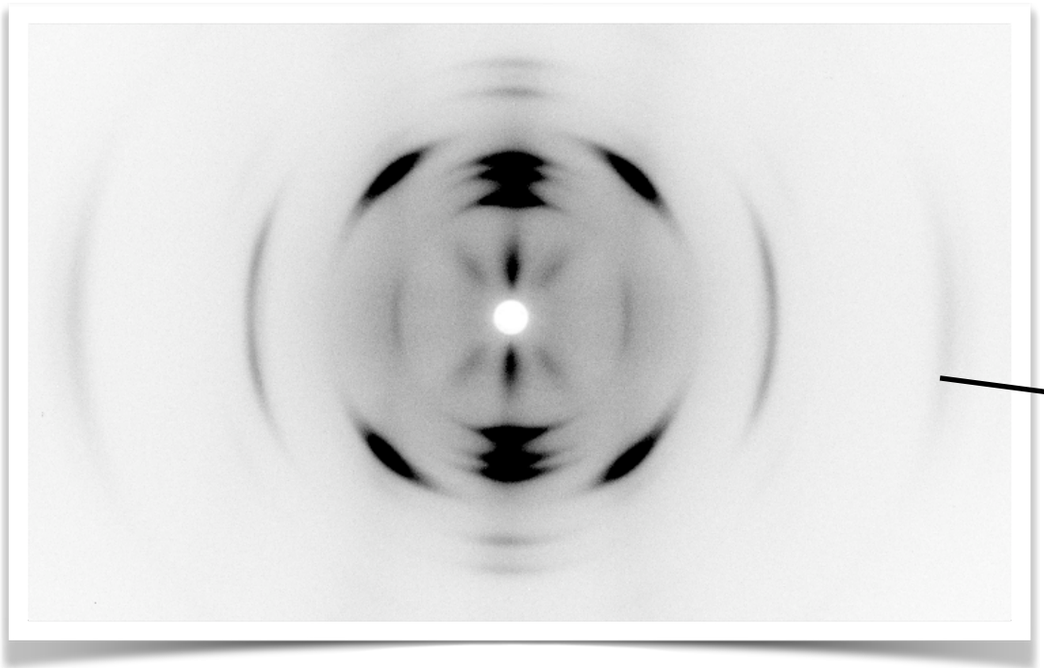
- Standa
- 4 fragn
- Lamark

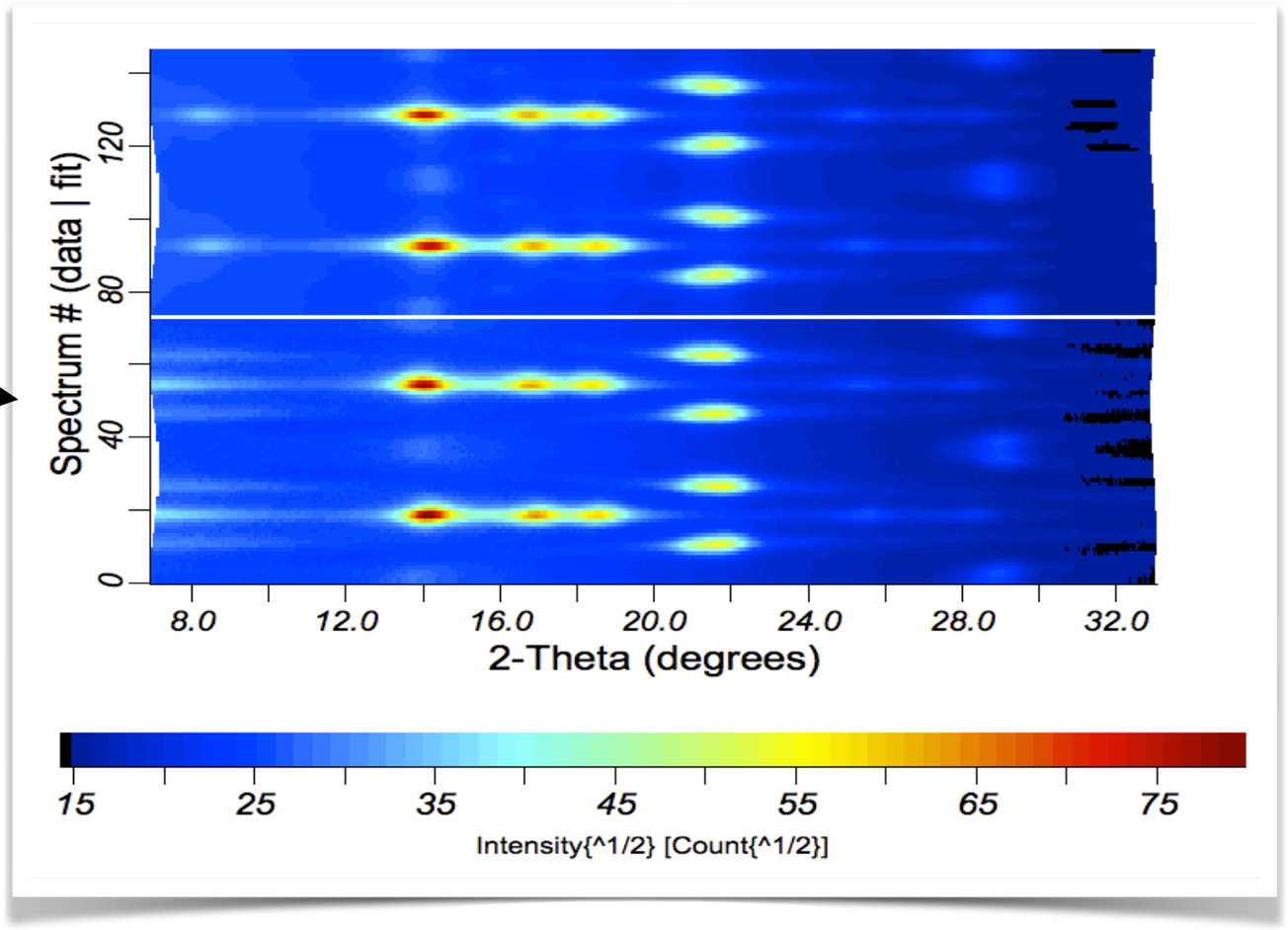


Isotactic polypropylene:RTA

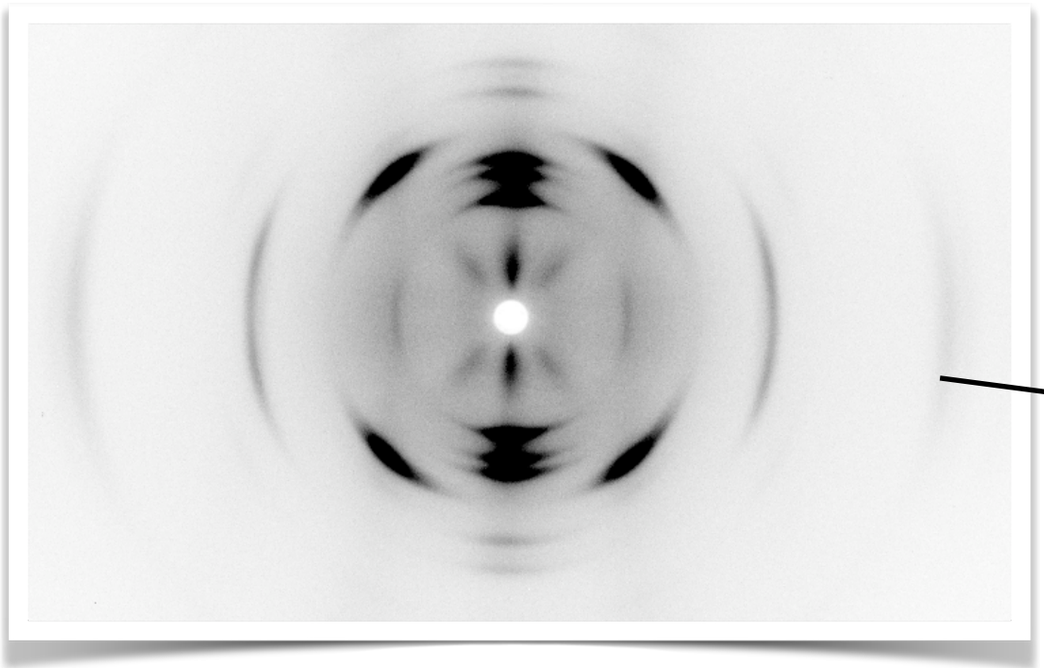


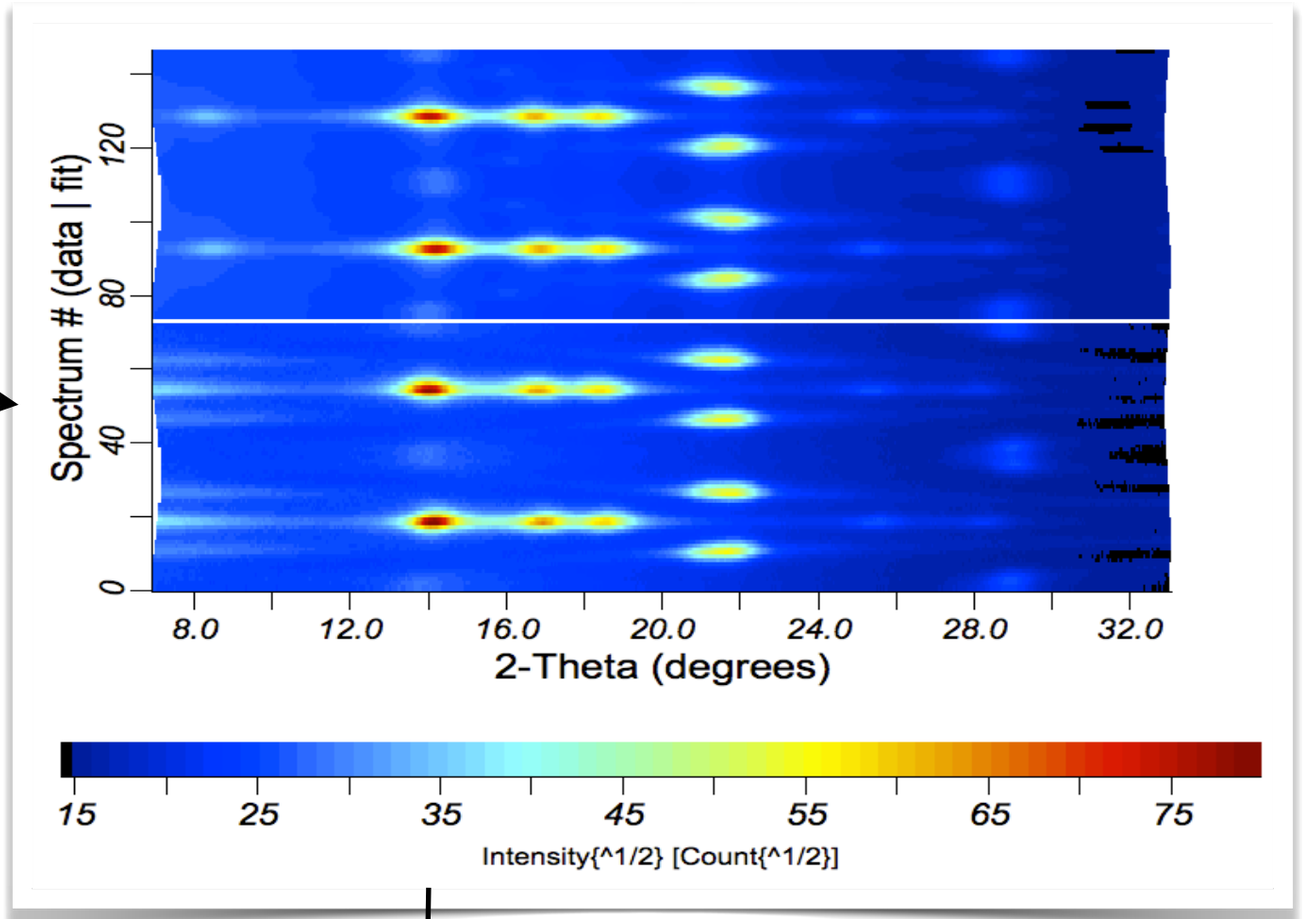
Isotactic polypropylene:RTA



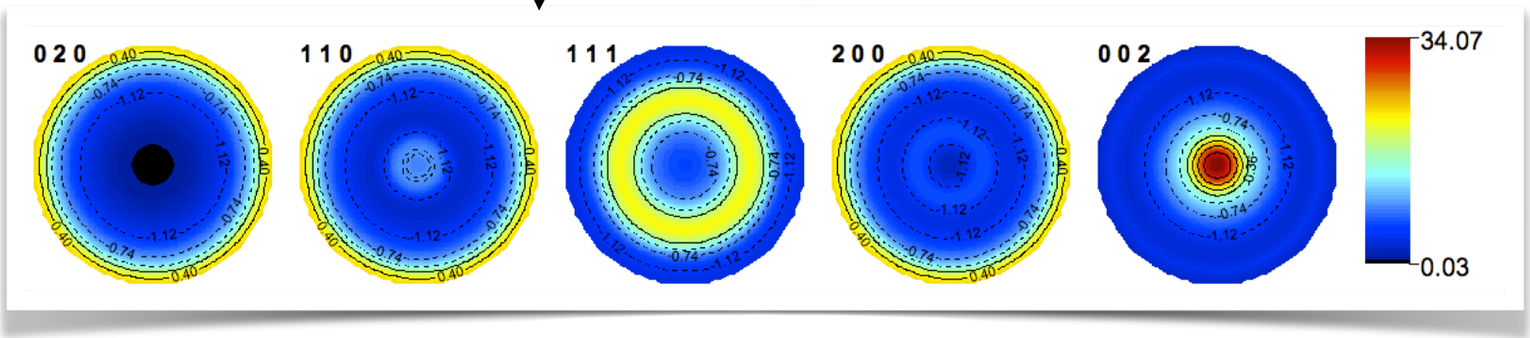


Isotactic polypropylene:RTA

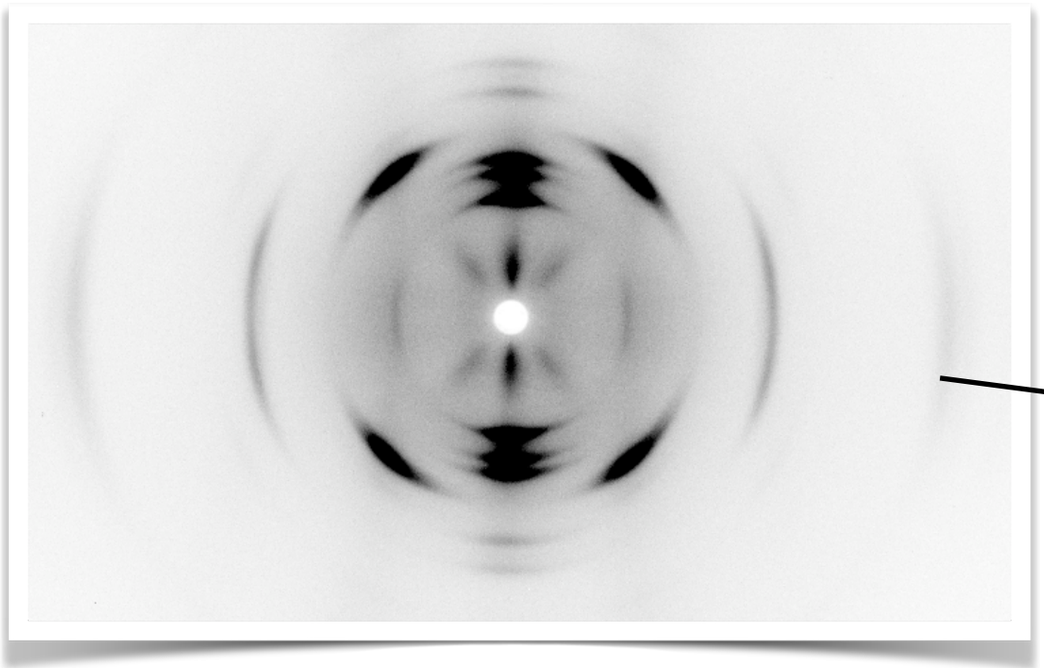


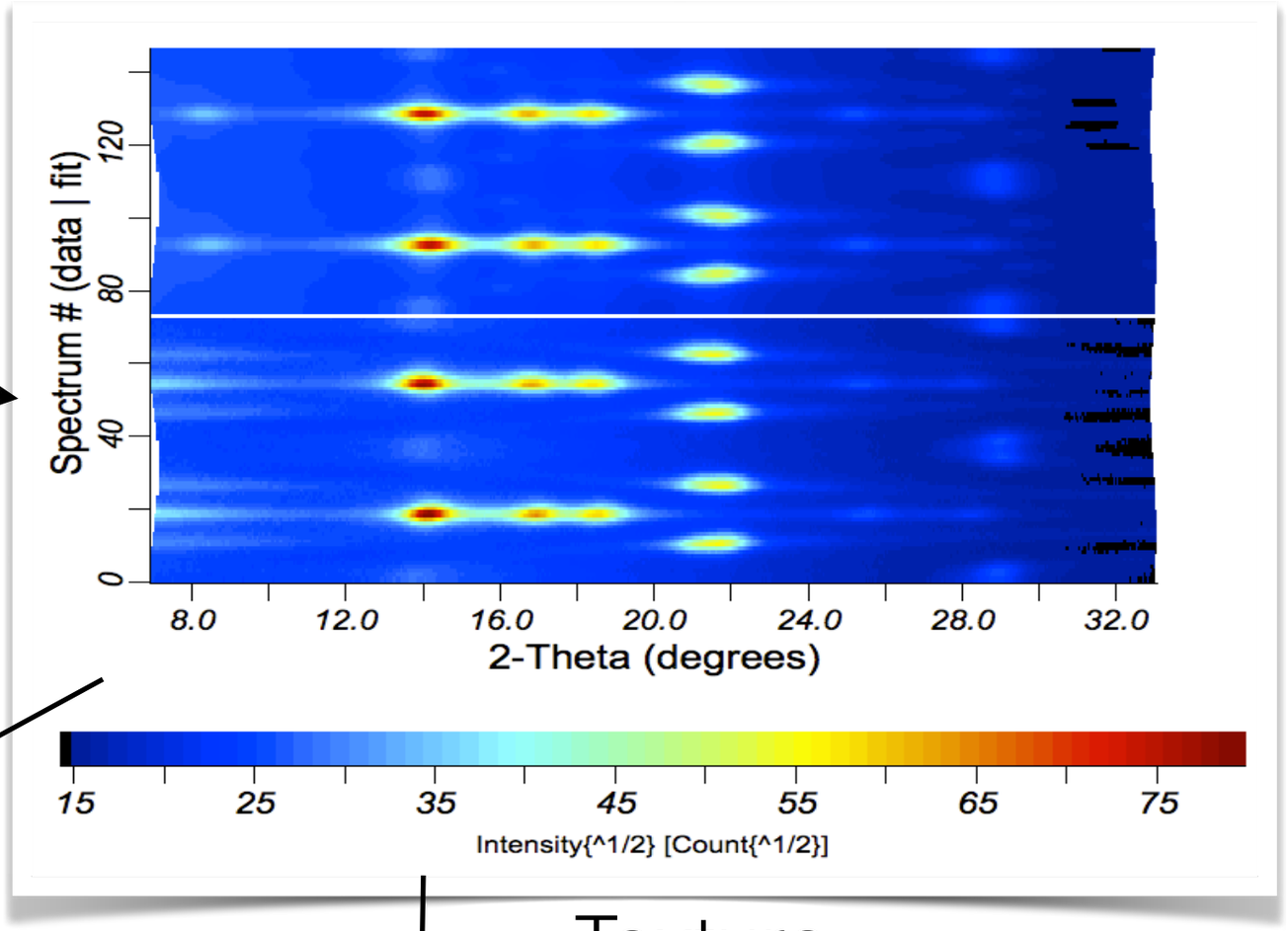


Texture

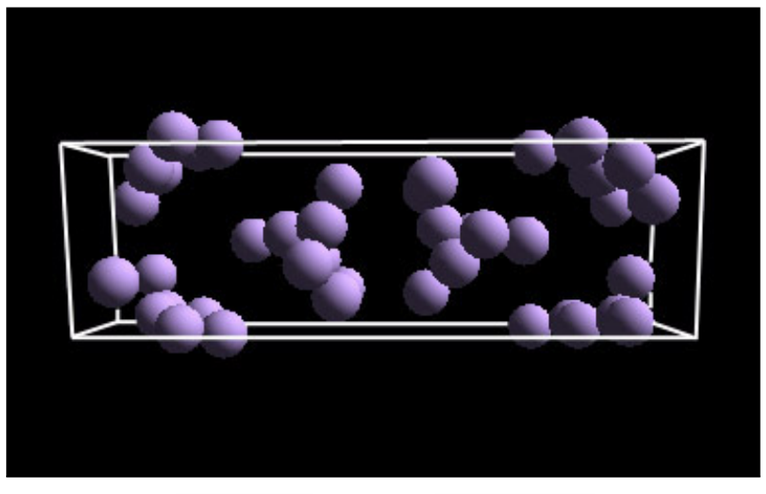


Isotactic polypropylene:RTA

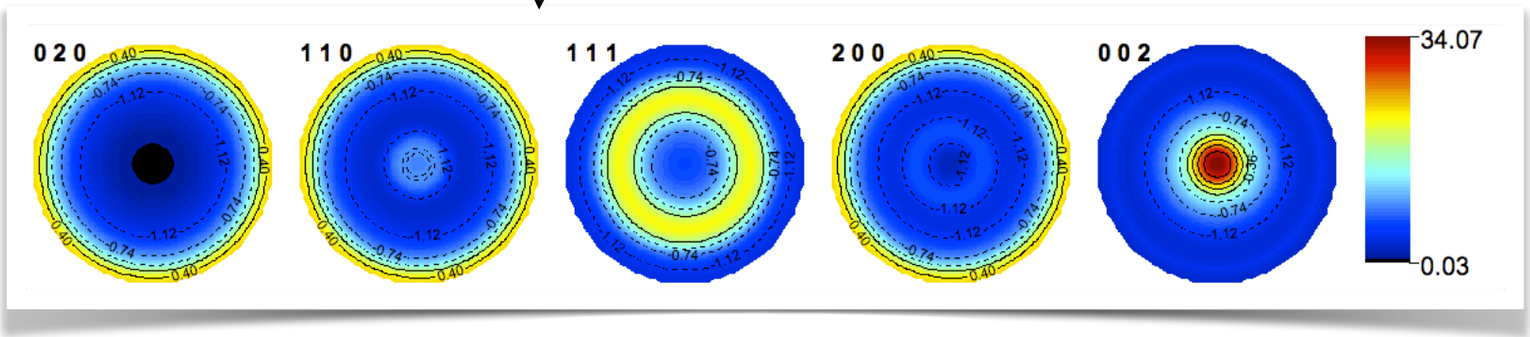




Structure

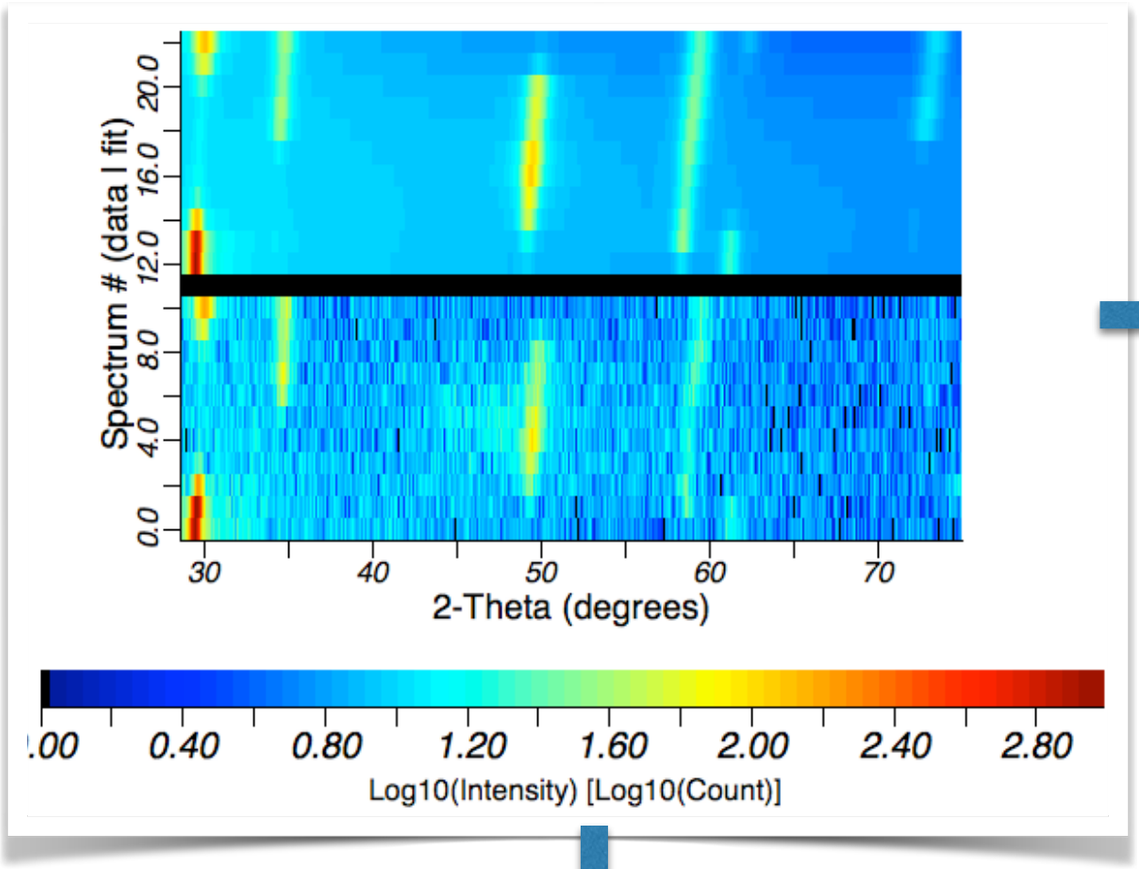


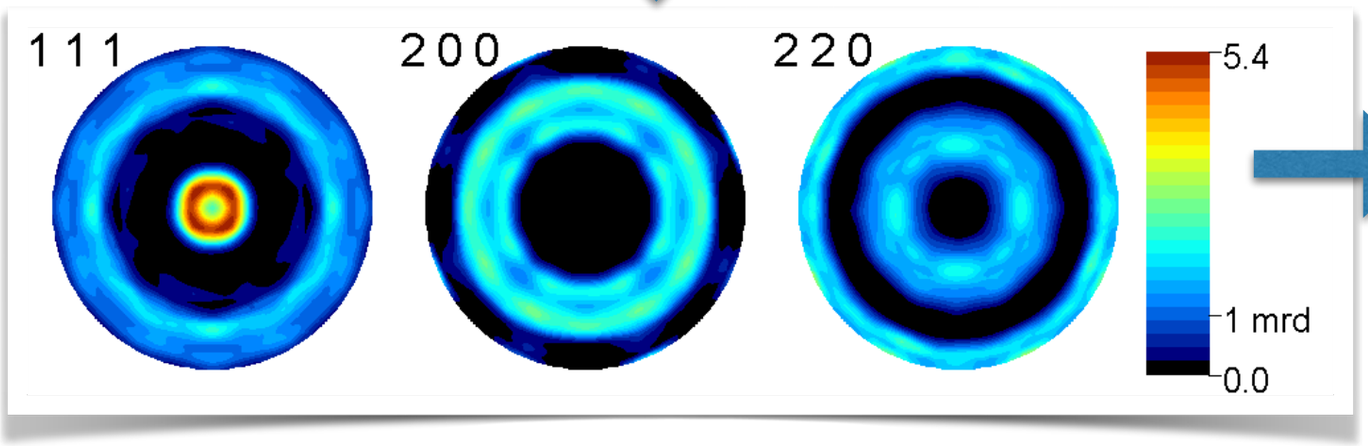
Texture

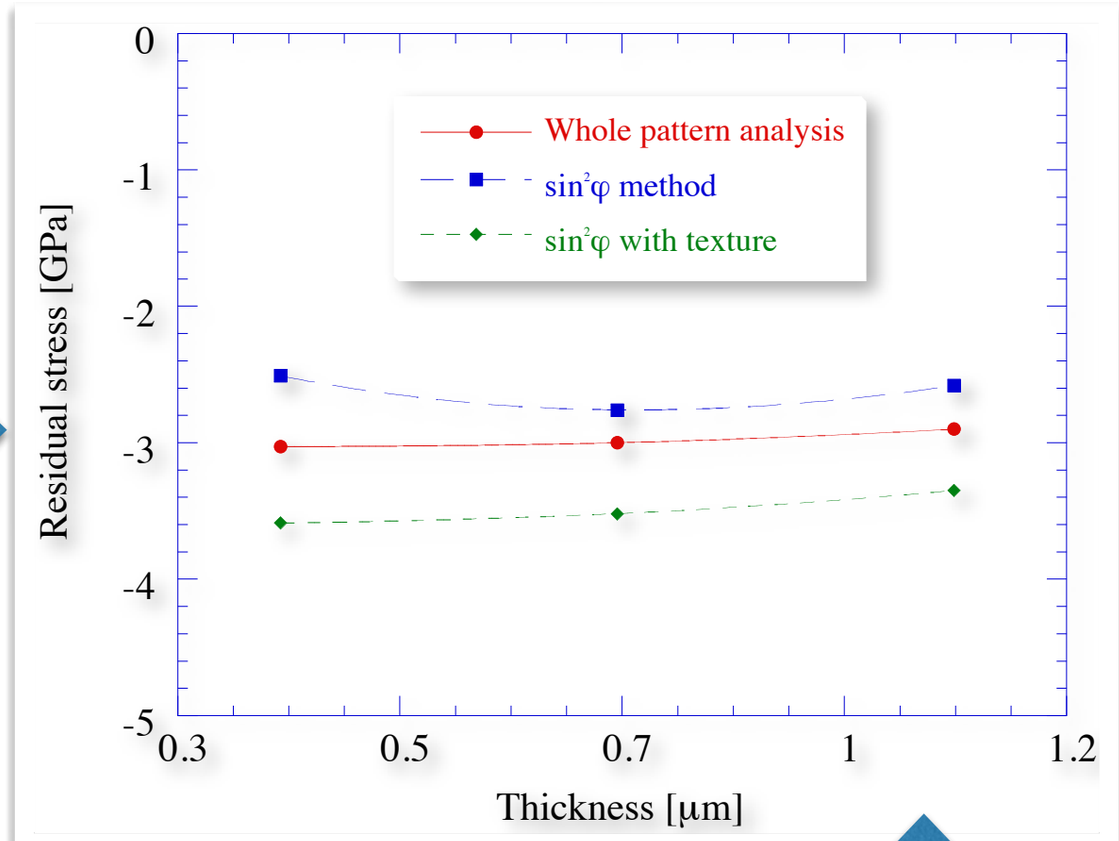


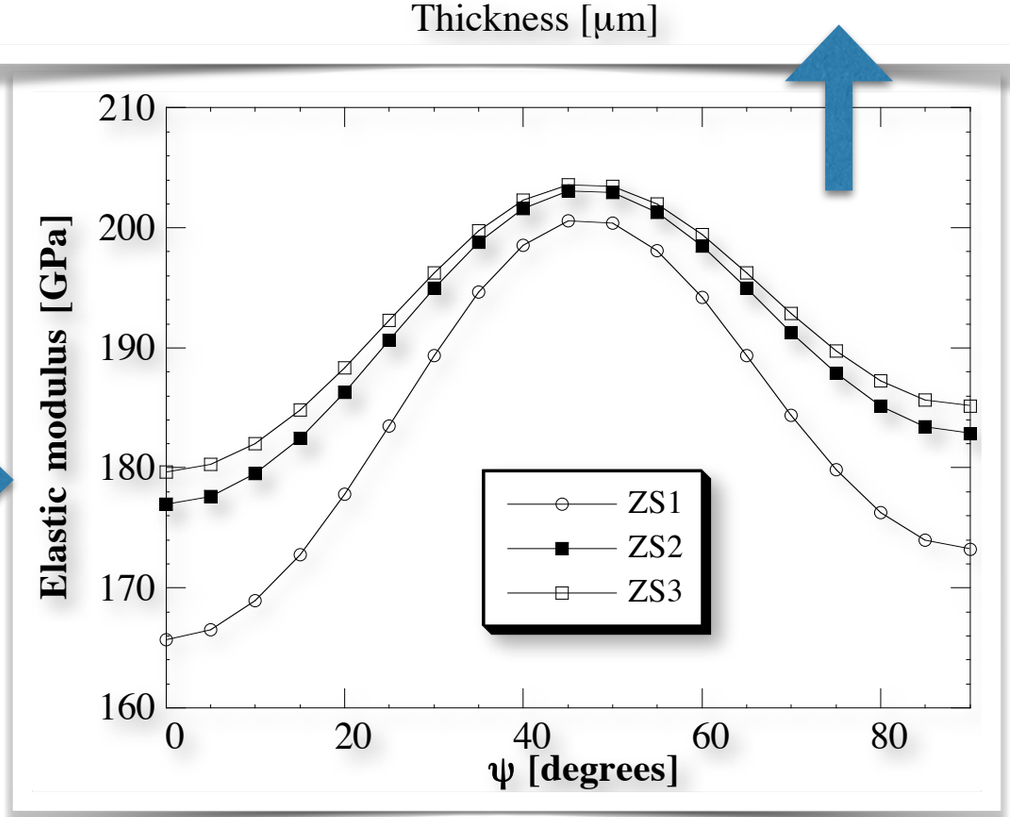
First experiment: residual stress and texture (1992)

c-ZrO_2 by magnetron sputtering

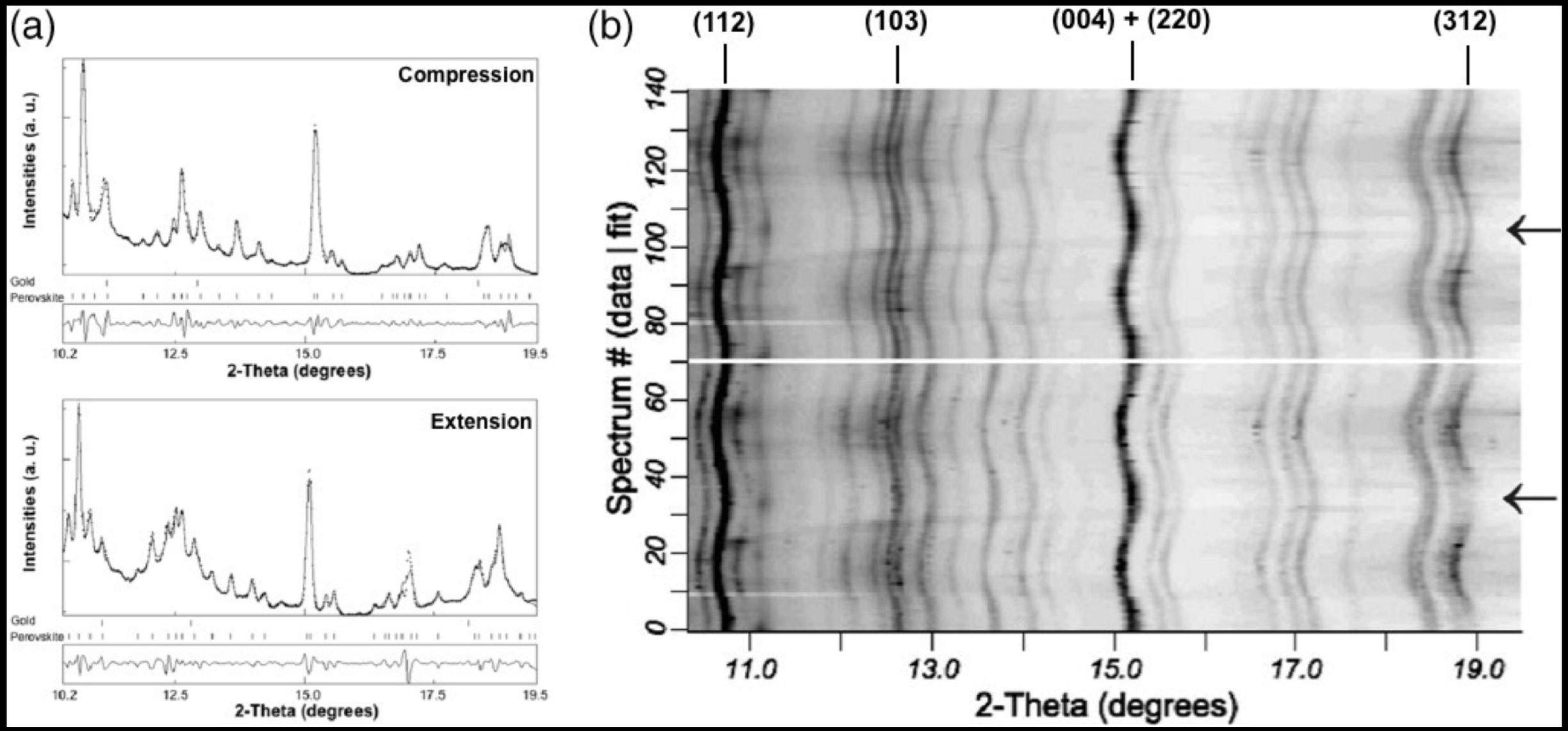








$MgSiO_3$ perovskite at 43 GPa



H-R Wenk, I Lonardelli, S Merkel, L Miyagi, J Pehl, S Speziale and C E Tommaseo

Deformation textures produced in diamond anvil experiments, analysed in radial diffraction geometry

J. Phys.: Condens. Matter 18 (2006) S933–S947

Rietveld texture analysis (RTA)

The powder pattern intensity:

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

Rietveld texture analysis (RTA)

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

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

Rietveld texture analysis (RTA)

The powder pattern intensity:

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

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

Harmonic method:

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

Harmonic coefficients

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

WIMV

The ODF space is discretized in cells:

$$f_{n+1}(g) = f_n(g)\left(\frac{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{\exp}(y)}{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{cal,n}(y)}\right)^{\frac{c}{NM_h}}$$

WIMV

The ODF space is discretized in cells:

$$f_{n+1}(g) = f_n(g)\left(\frac{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{\exp}(y)}{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{cal,n}(y)}\right)^{\frac{c}{NM_h}}$$

- Always positive
- Ghost conditional corrections
- Complex and sharp textures
- Works for lower symmetries

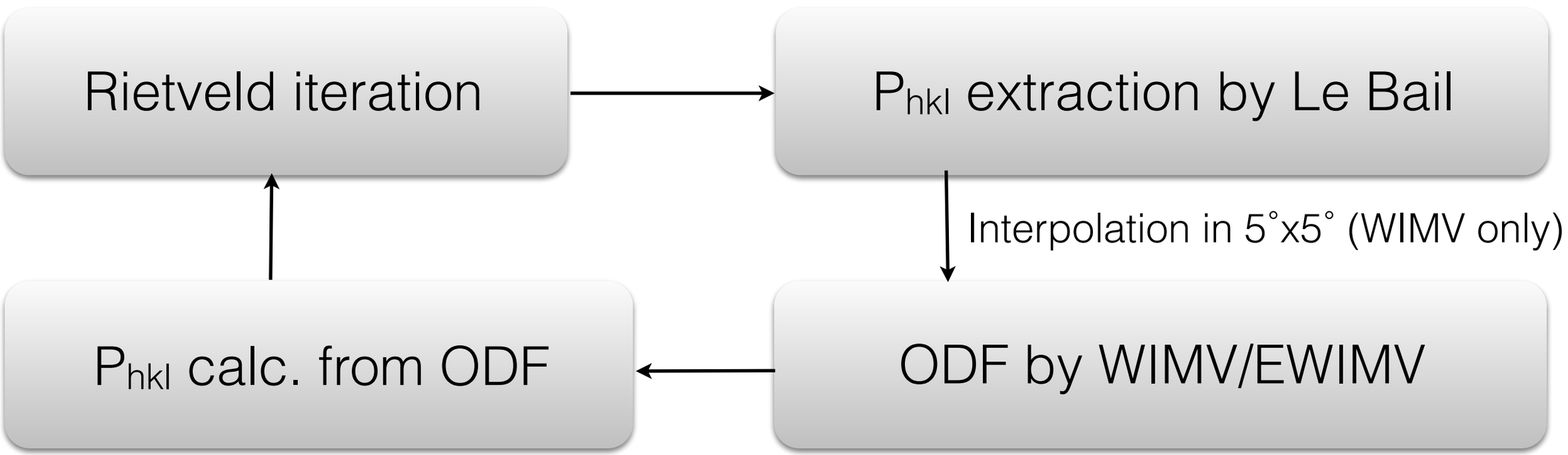
WIMV

The ODF space is discretized in cells:

$$f_{n+1}(g) = f_n(g)\left(\frac{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{\exp}(y)}{\prod_{h=1}^{N}\prod_{m=1}^{M_h} P_h^{cal,n}(y)}\right)^{\frac{c}{NM_h}}$$

- Always positive
- Ghost conditional corrections
- Complex and sharp textures
- Works for lower symmetries

The Rietveld integration:

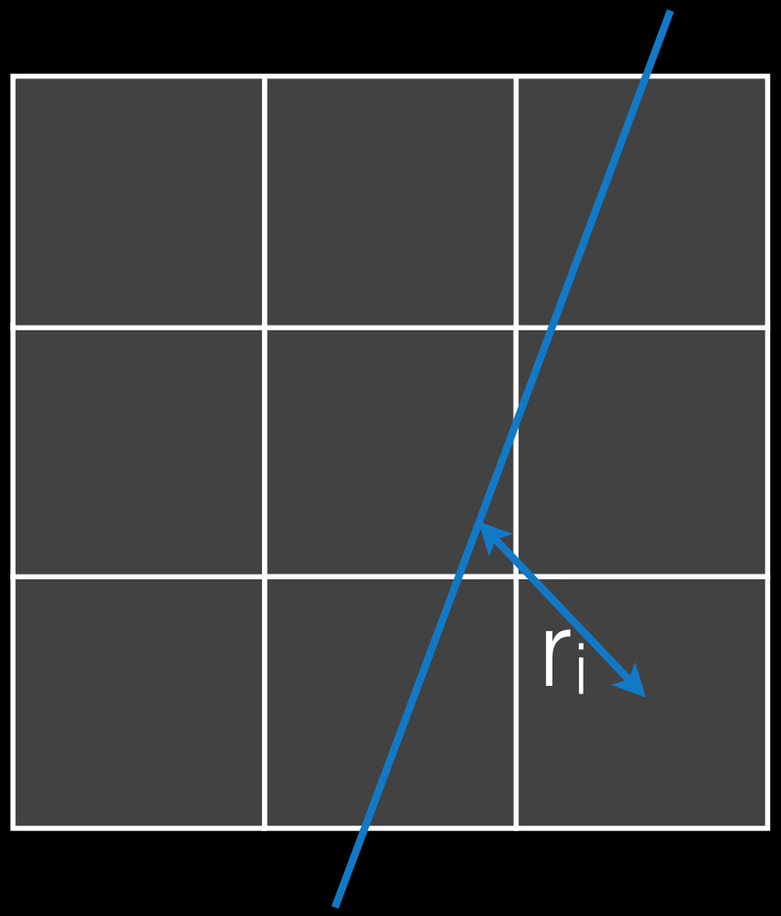


EWIMV

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

w_h = intensity of reflection h

Weighting of neighboring cells



WIMV: uses value of containing cell

EWIMV:

$$P_h = \int \frac{1}{K} \sum_{i=1}^{N} \frac{f_i}{r_i}, \quad K = \sum_{i=1}^{N} \frac{1}{r_i}$$

MTex in MAUD

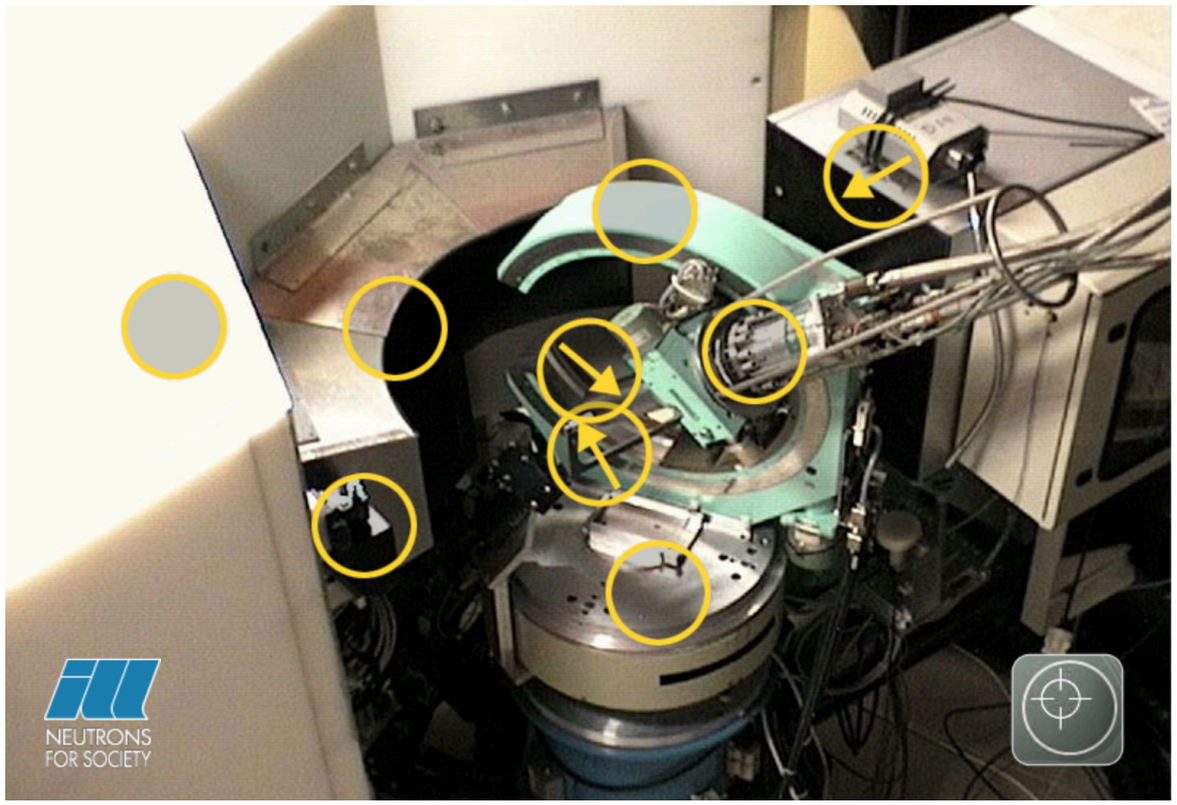
- This was possible because of Florian

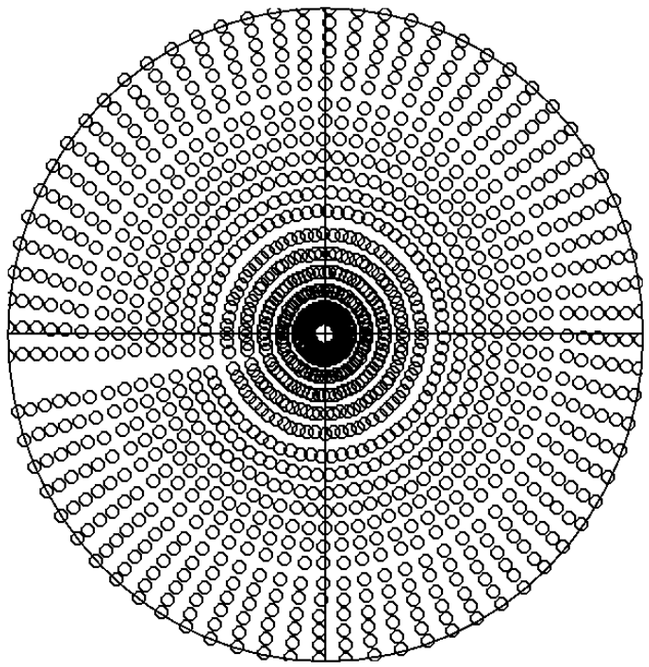
- Florian developed a Java interface for MTex (the binary executables, version 4.5.1)

- The Florian interface is used inside for a so-called MTex model along with the others texture model

- Each Rietveld iteration cycle, MAUD extracts pole figures, selects a subset and from them MTex calculates and ODF. From the ODF MTex calculates the value of the pole figure for each reflection of each pattern on request.

Calcite @ D19

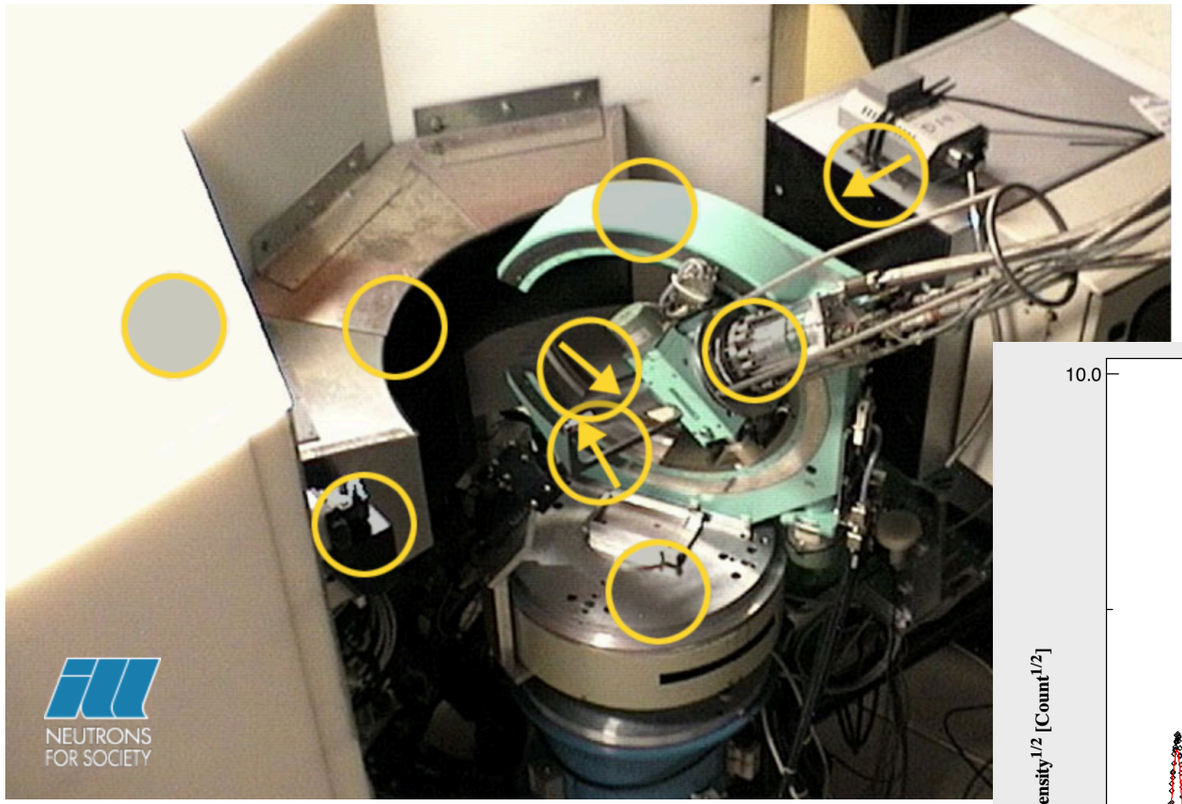


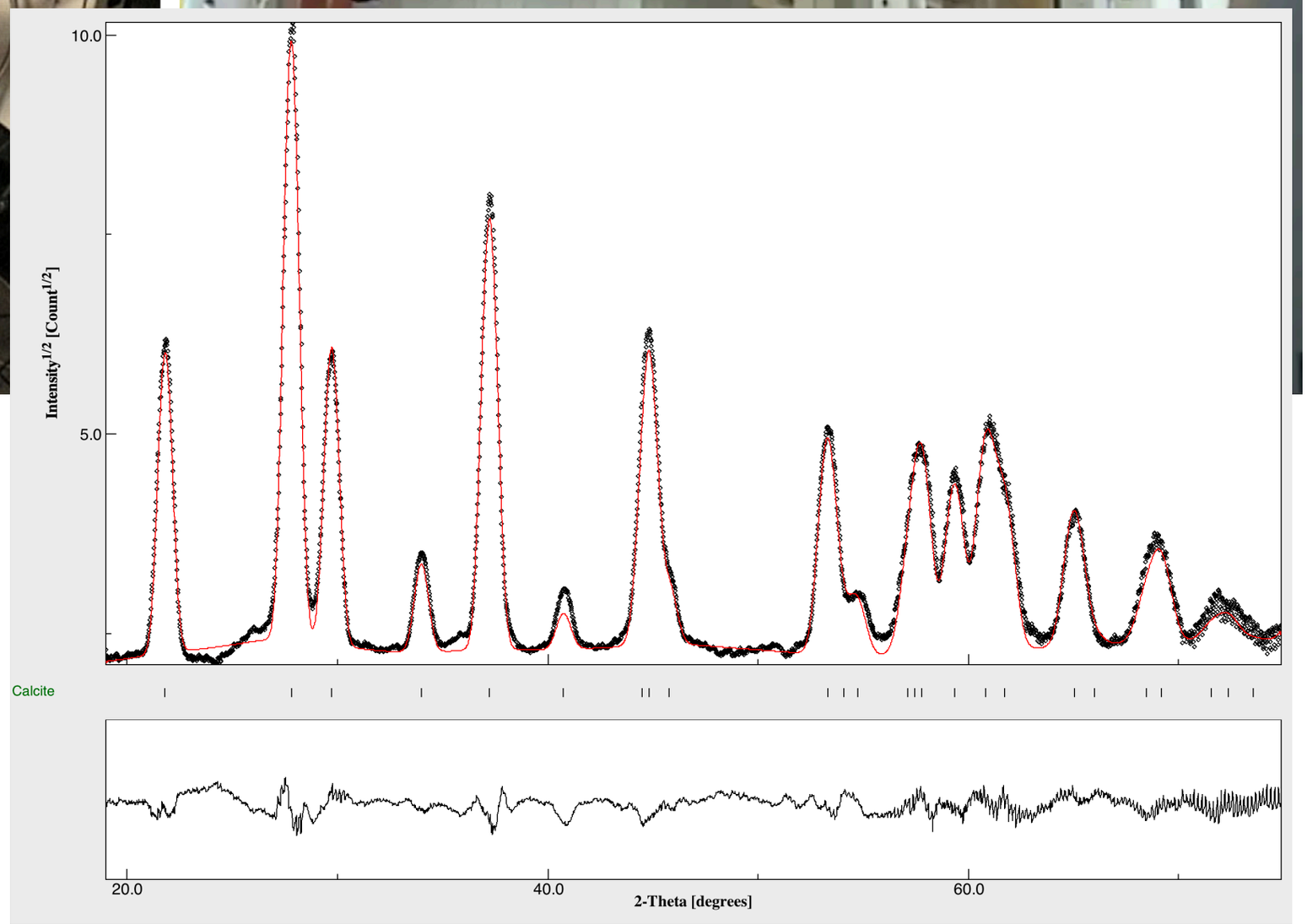


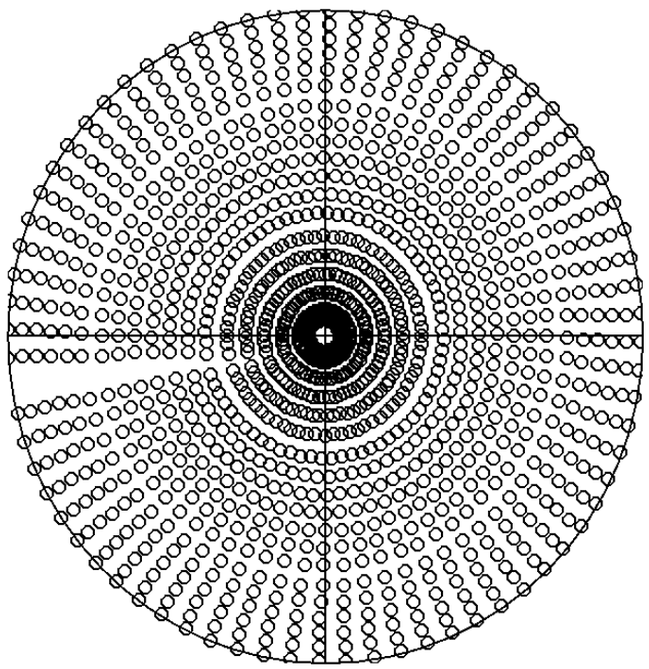
Experimental coverage for 006

Experiment conducted by Michele Zucali

Calcite @ D19





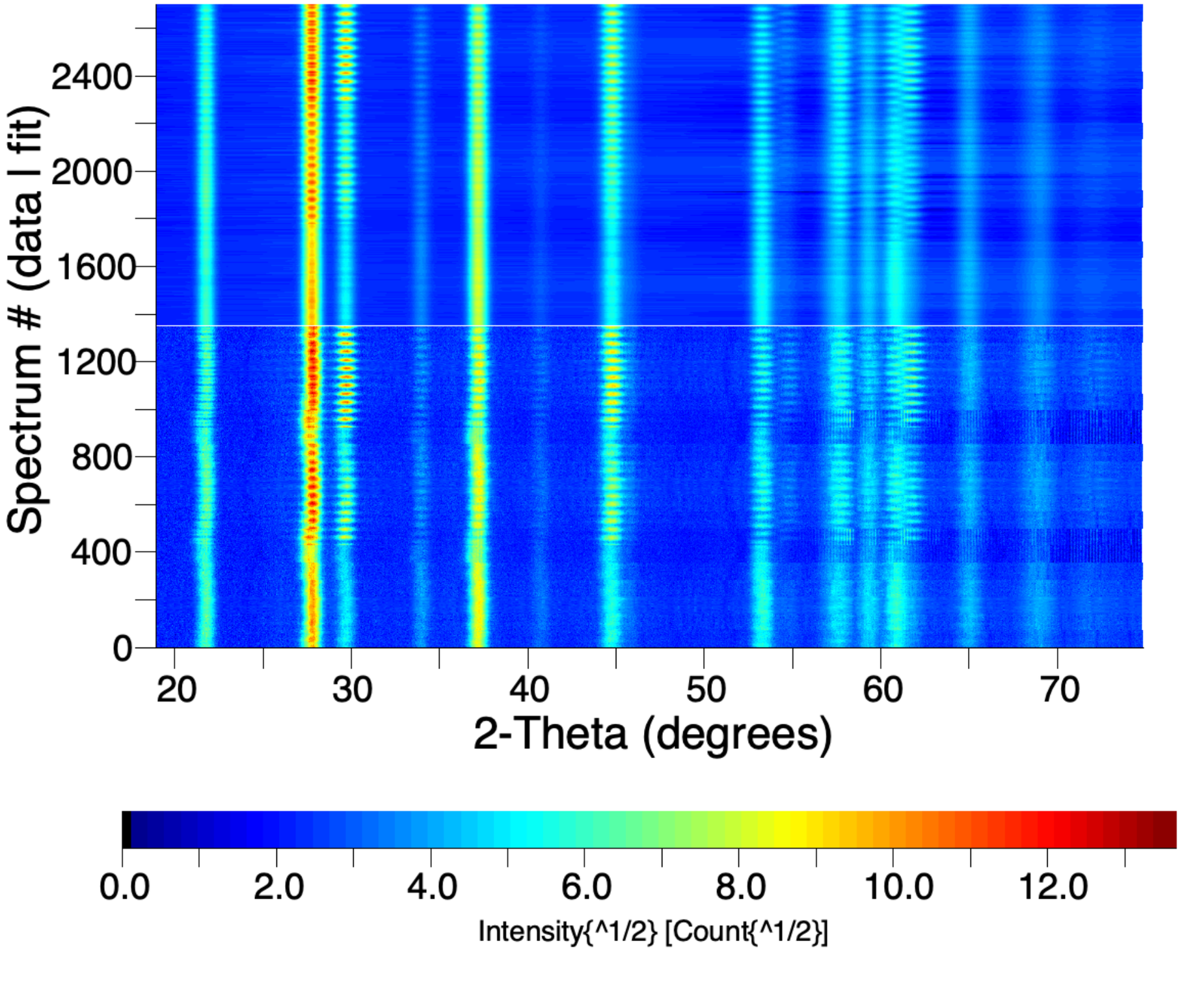


Experimental coverage for 006

Experiment conducted by Michele Zucali

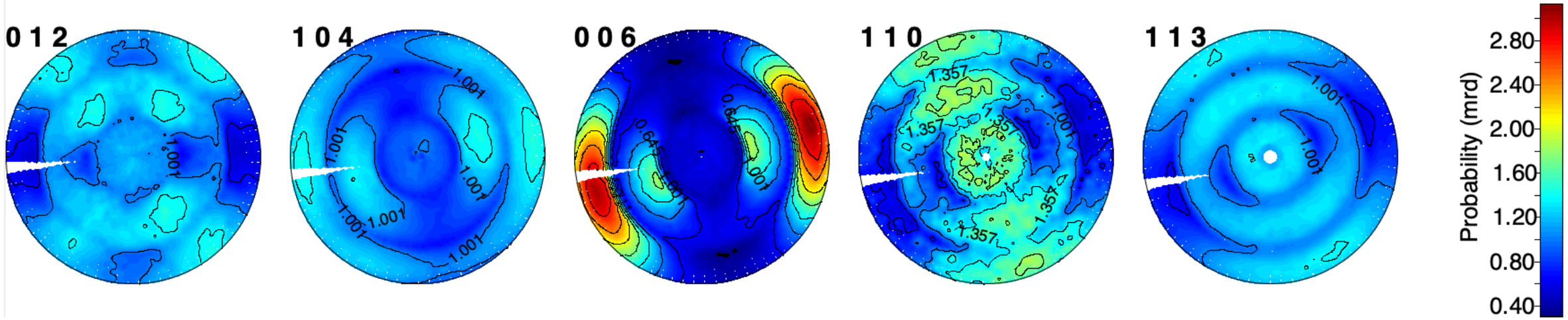
2D fitting of calcite patterns

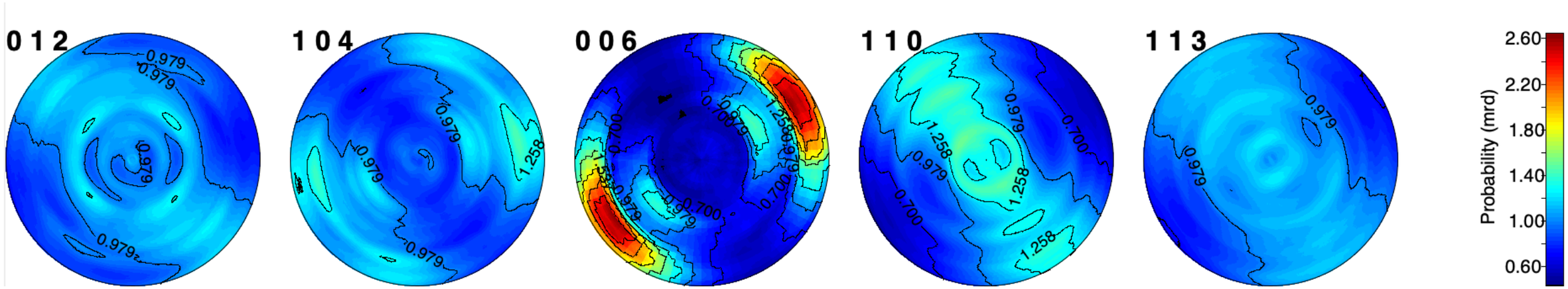
Using EWIMV texture model



Texture analysis of calcite

Extracted (experimental) pole figures (some)

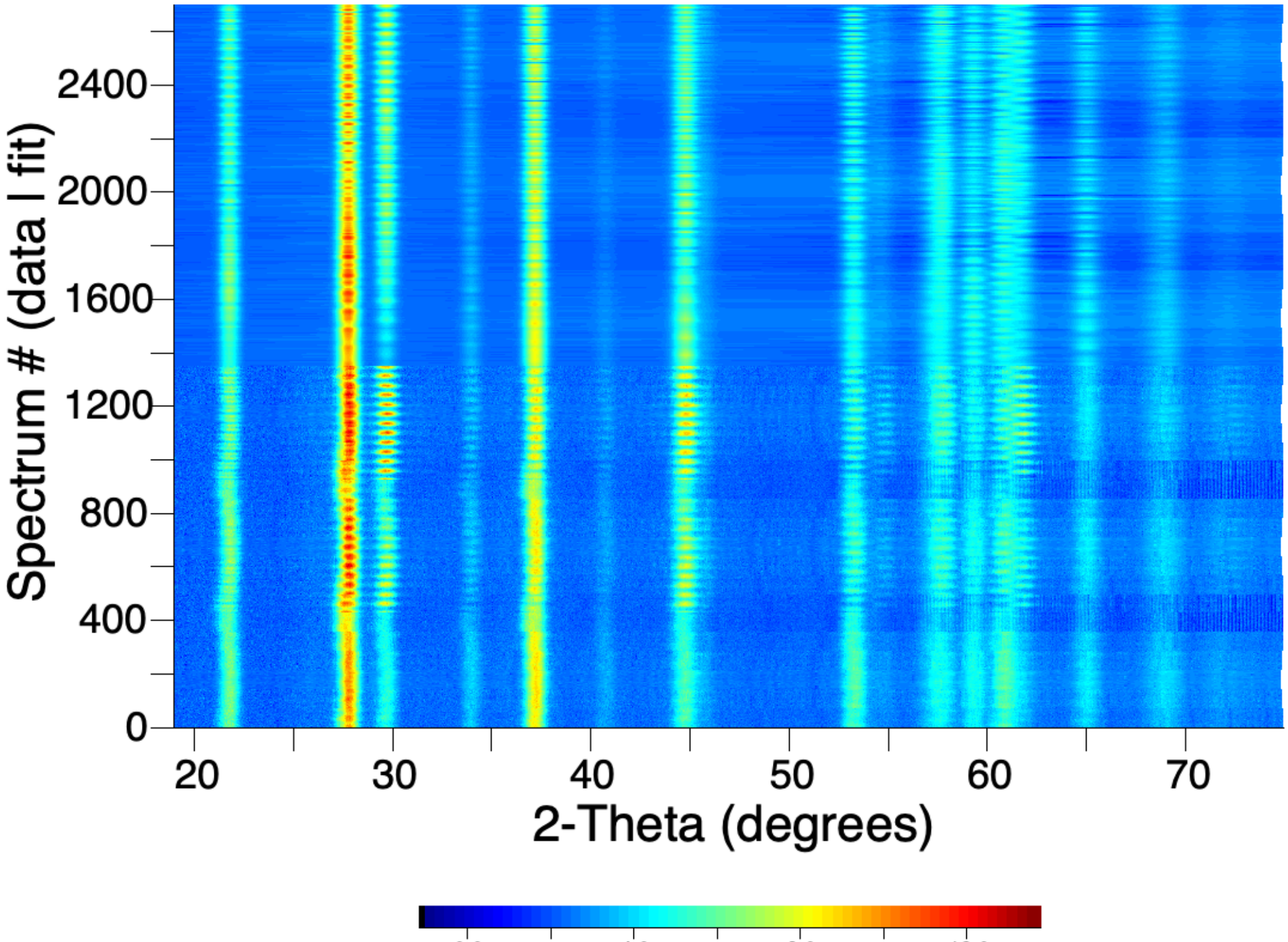




Recalculated pole figures (using EWIMV, 10° as texture model)

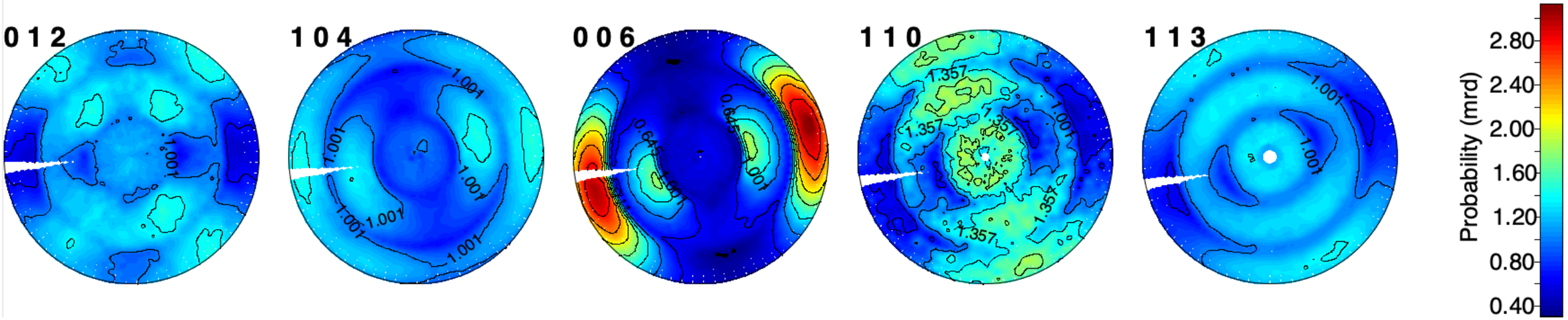
2D fitting of calcite patterns

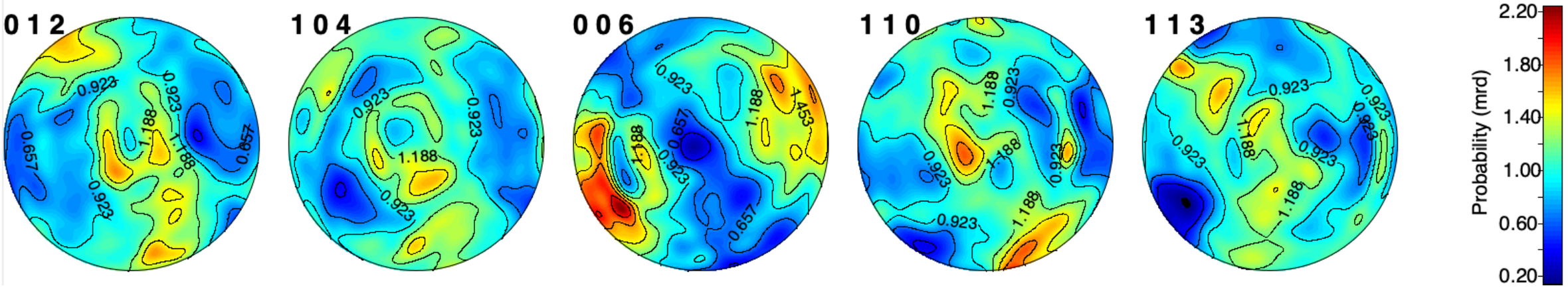
Using MTEX texture model



Texture analysis of calcite

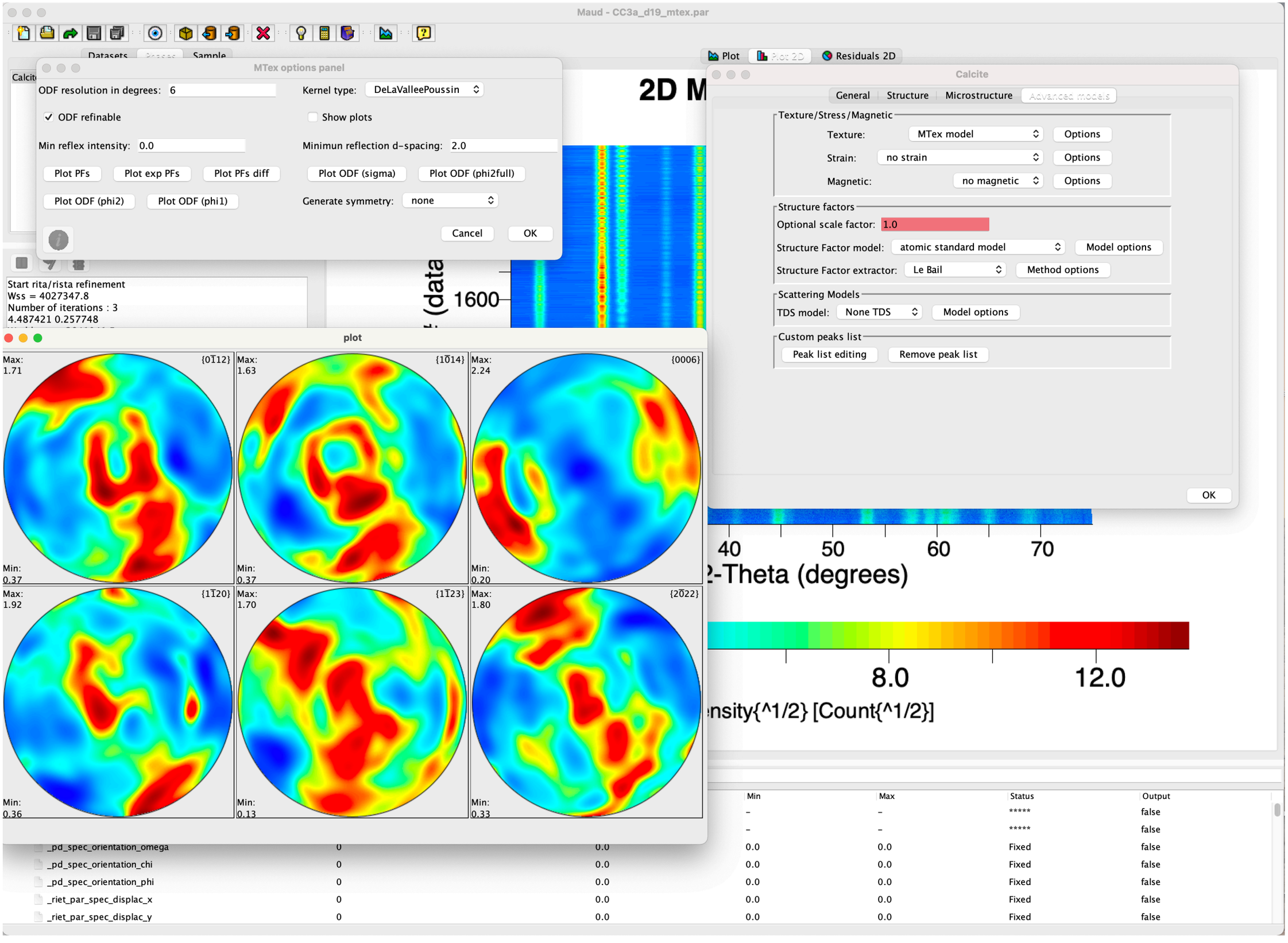
Extracted (experimental) pole figures (some)



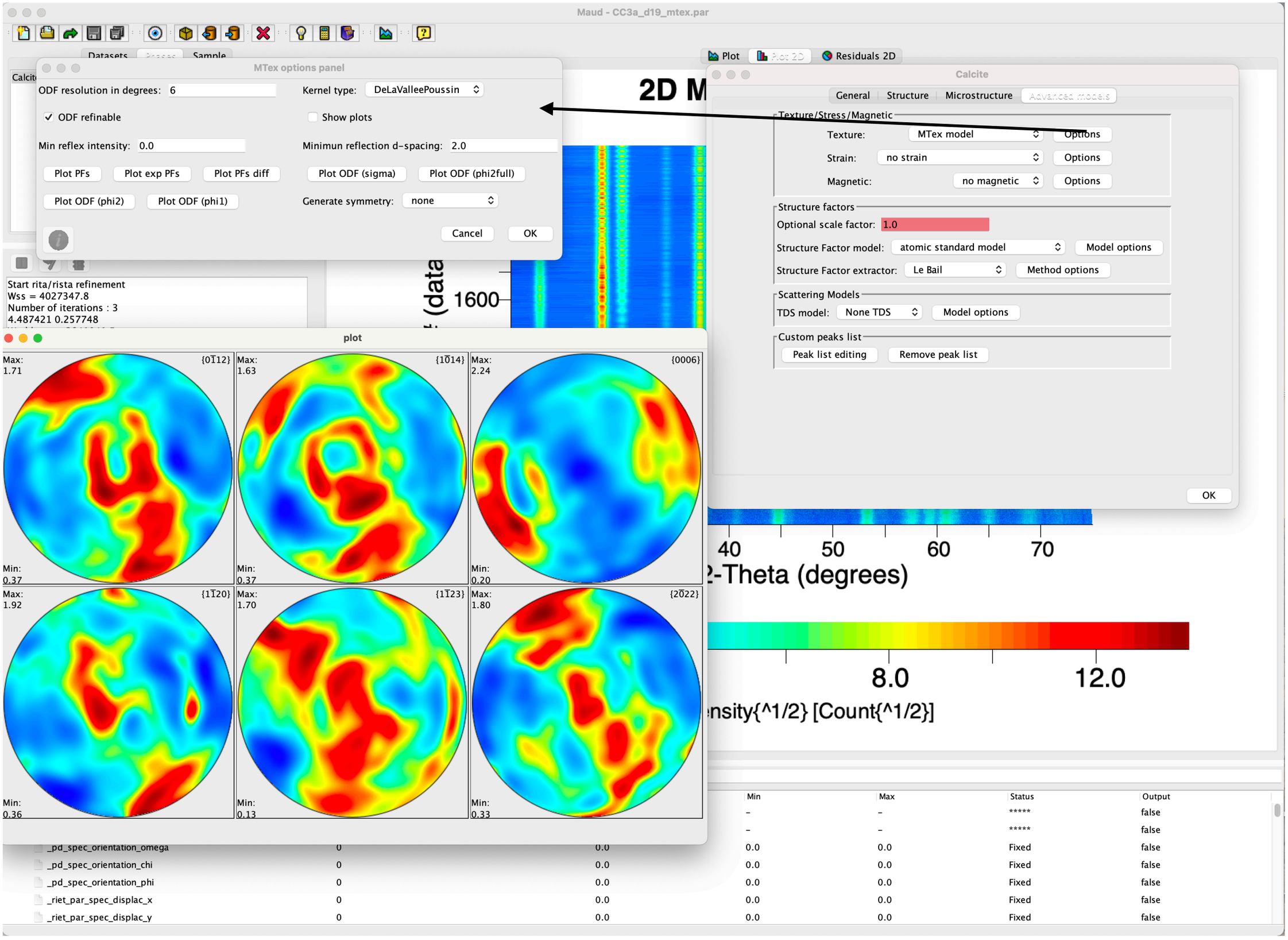


Recalculated pole figures, using MTEX (6˚, De La Vallee Poussin) as texture model)

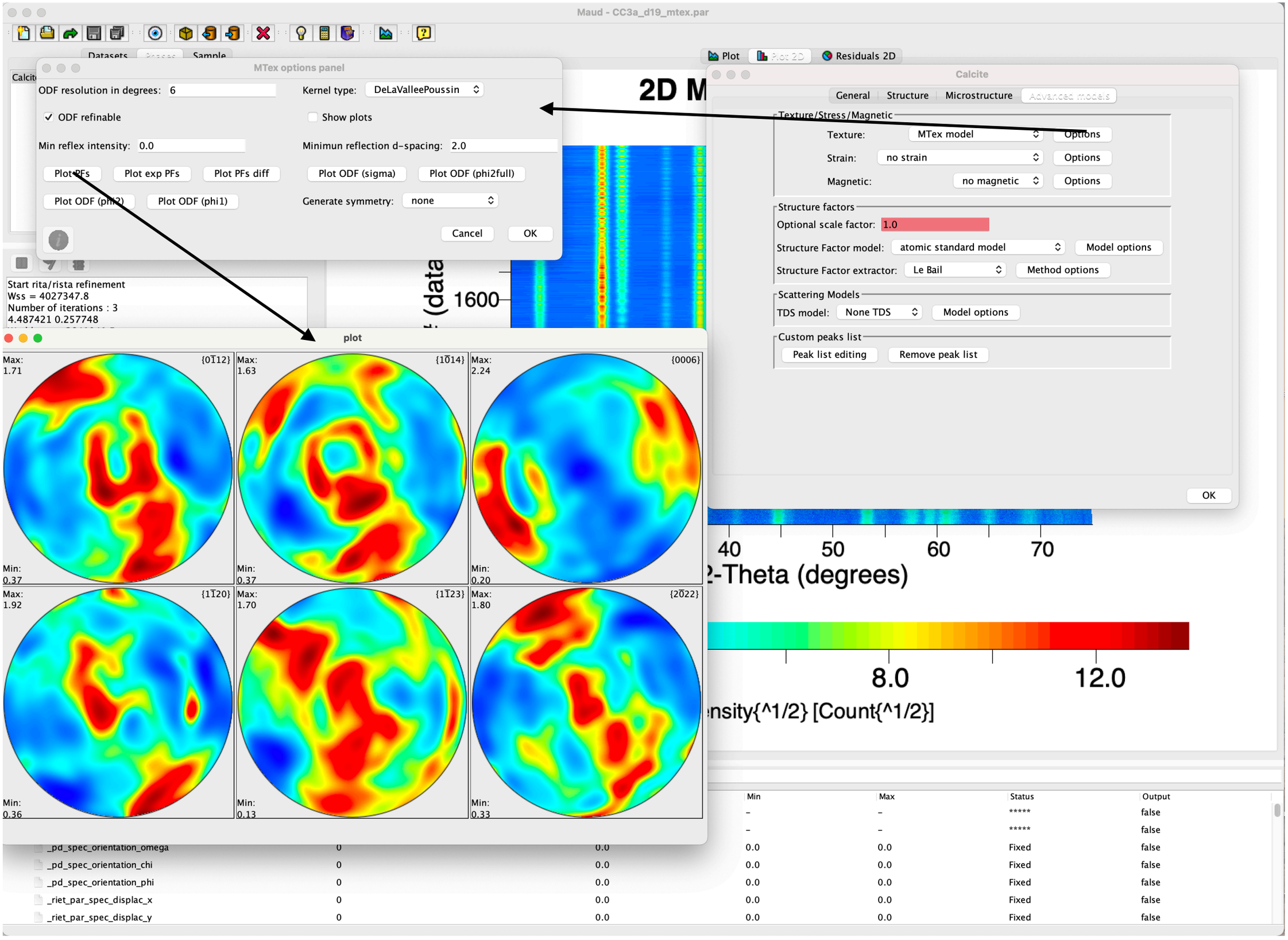
The model in MAUD



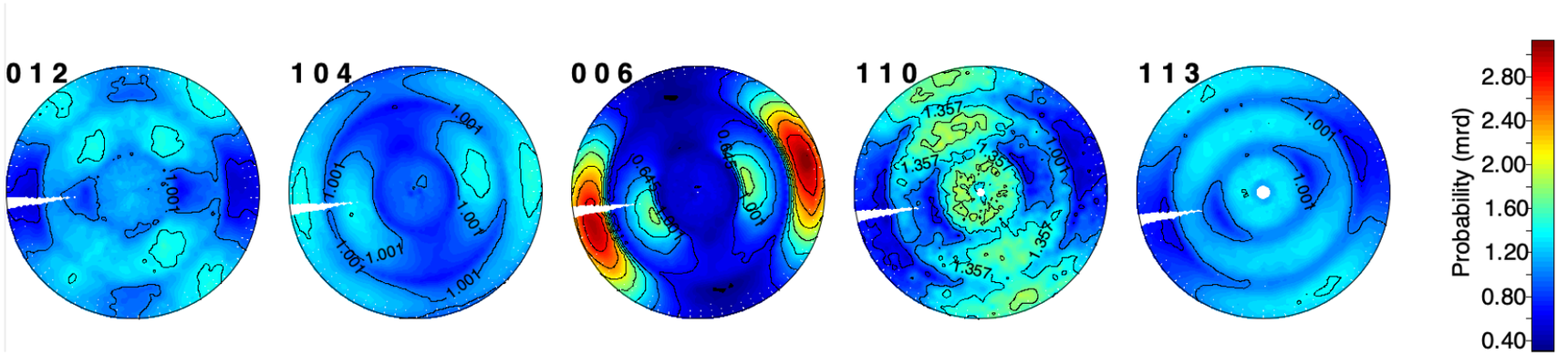
The model in MAUD



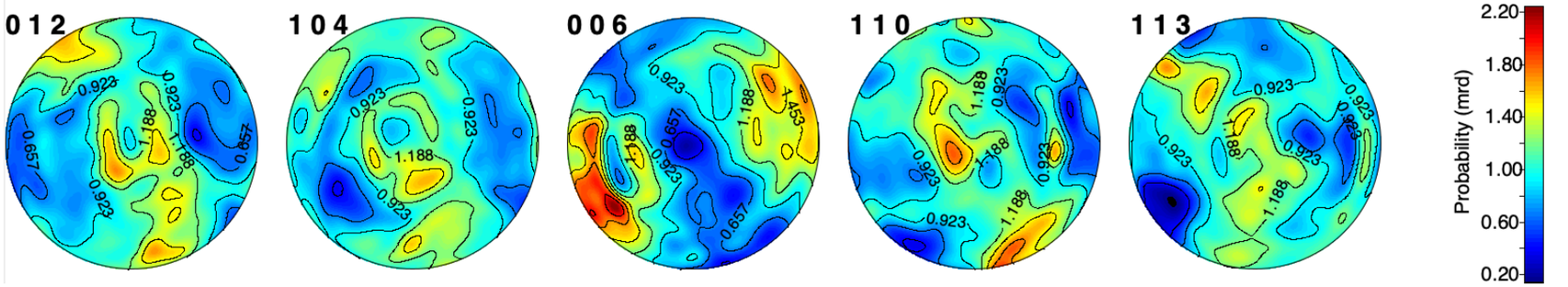
The model in MAUD



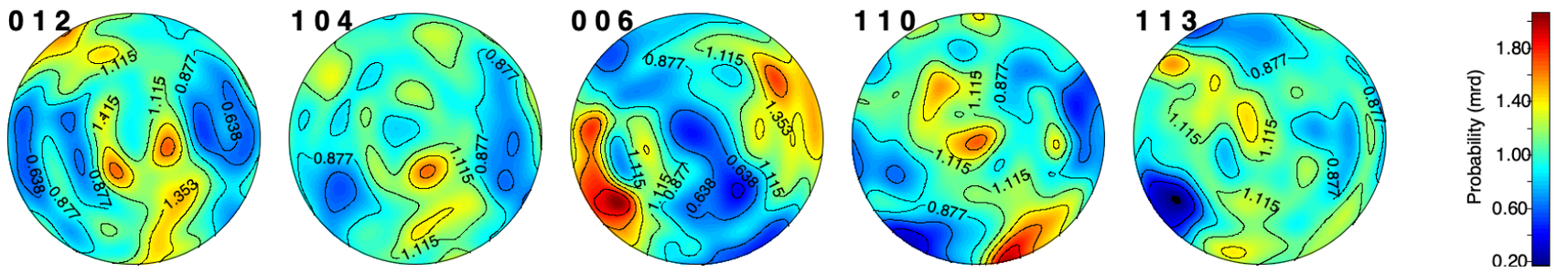
Testing different options in MTex



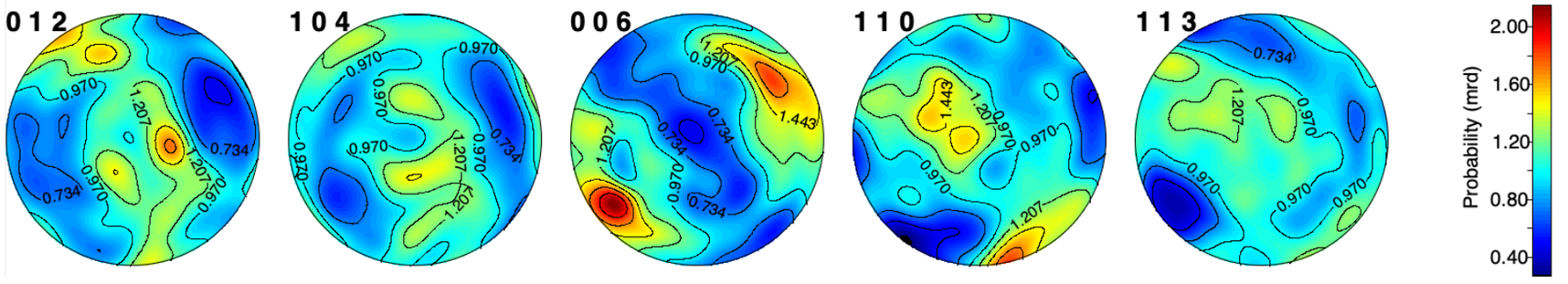
Extracted (experimental) pole figures (some)



MTEX (6˚, De La Vallee Poussin), reflection cut at 2.0 Angstrom

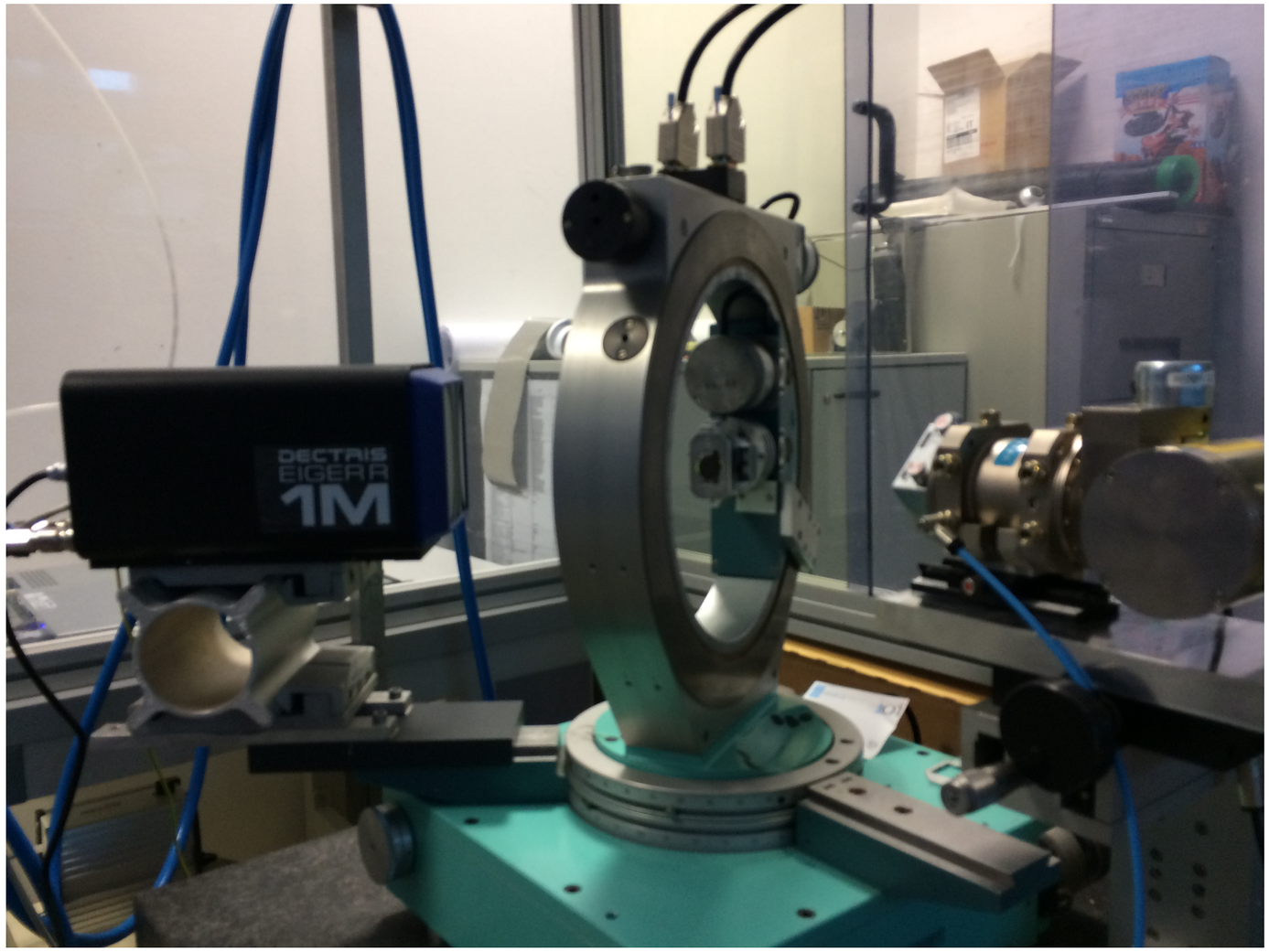


MTEX (8˚, Von Mises), reflection cut at 1.7 Angstrom



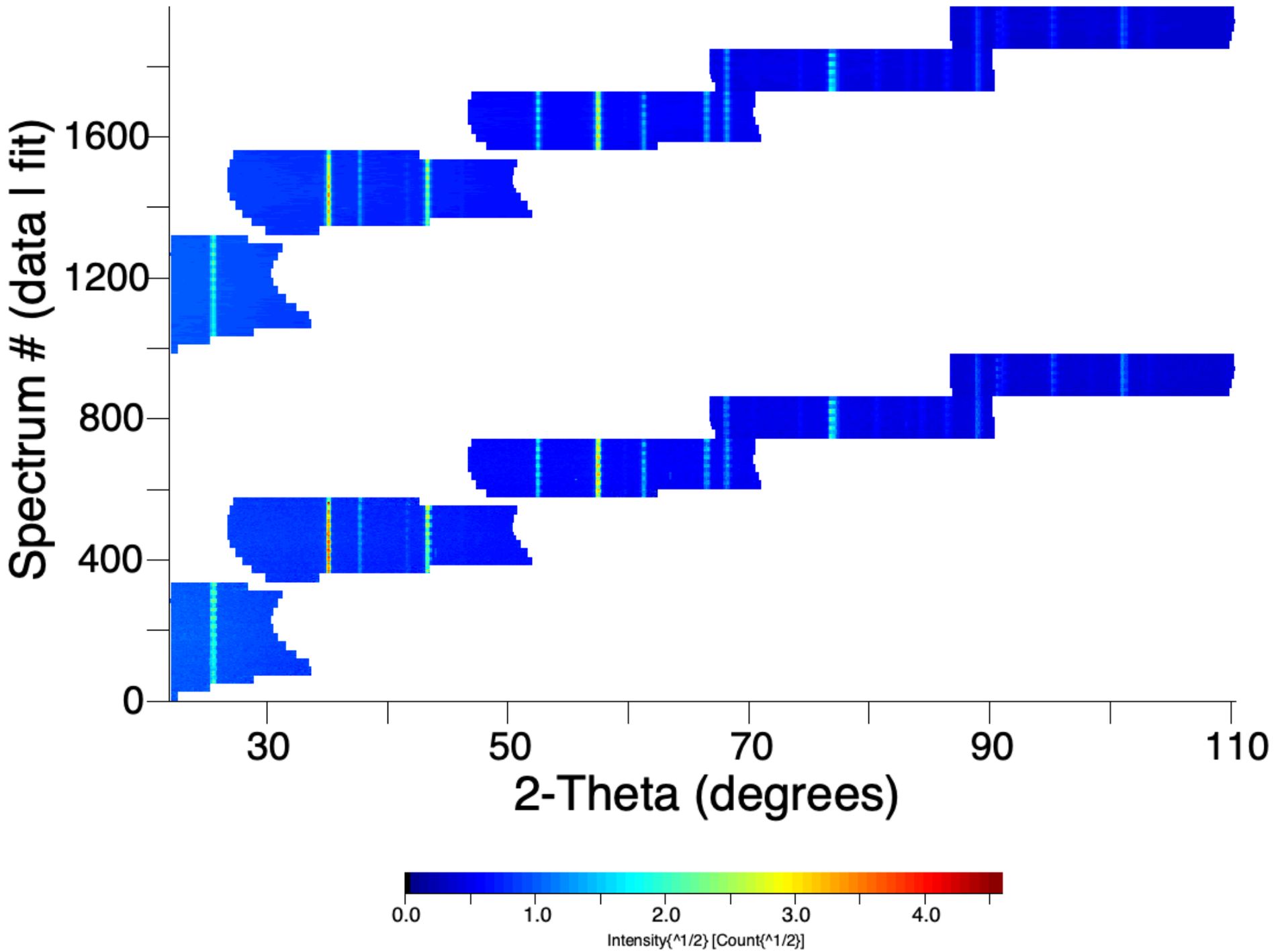
MTEX (10˚, Von Mises), reflection cut at 1.5 Angstrom

NIST 1976a standard (Corundum plate, fiber texture)

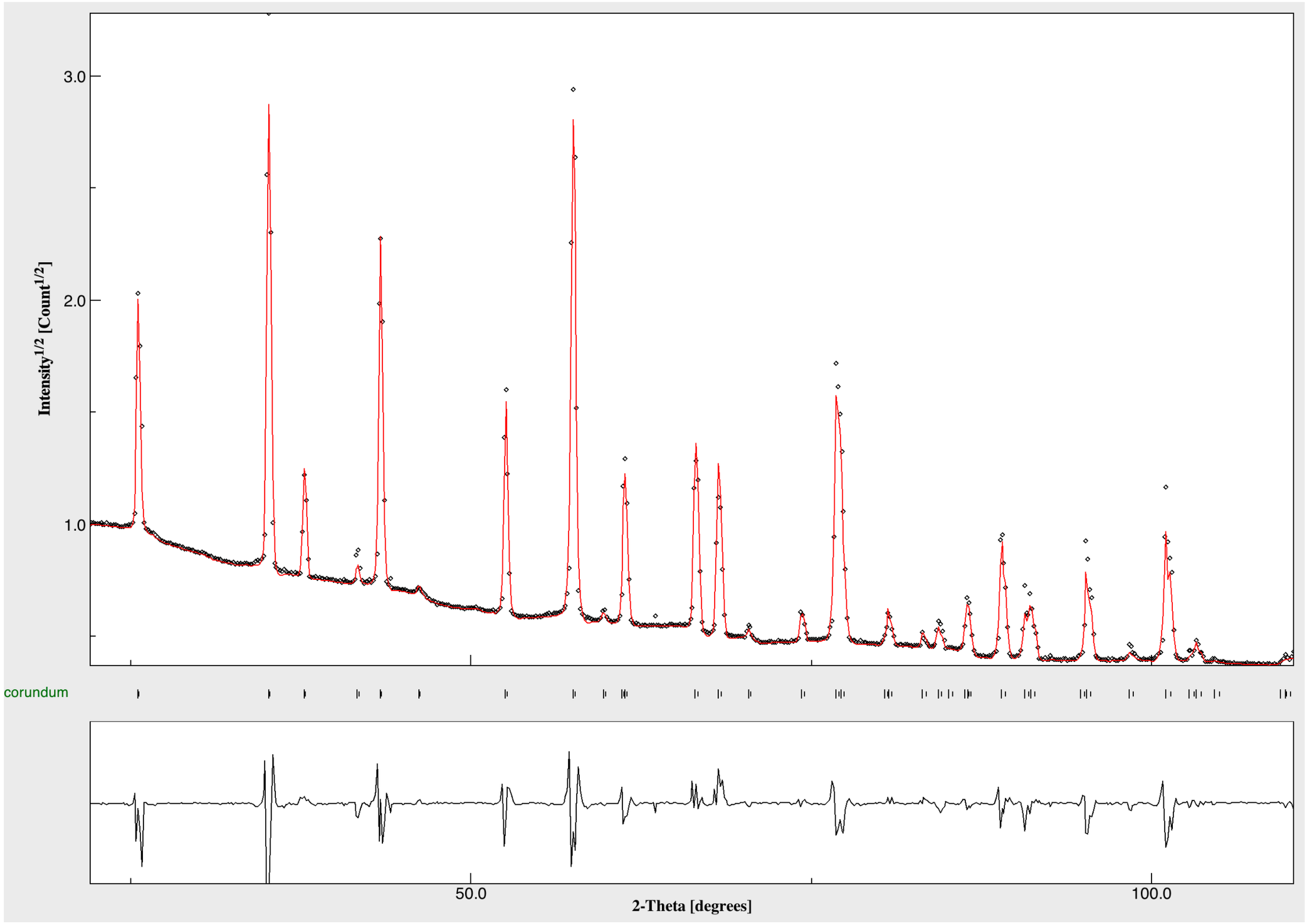


- Data measured in Lab (Trento)
- Eulerian goniometer, high brilliance micro source and Dectris 1M 2D detector
- 2D images permit faster collection of texture data

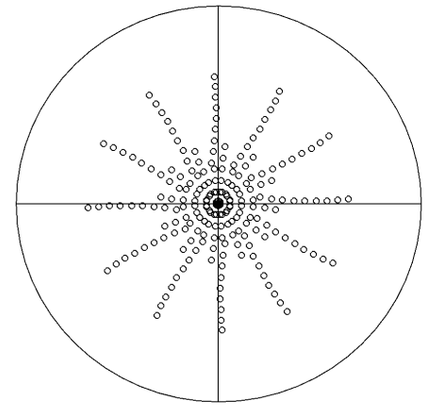
2D fitting (EWIMV)



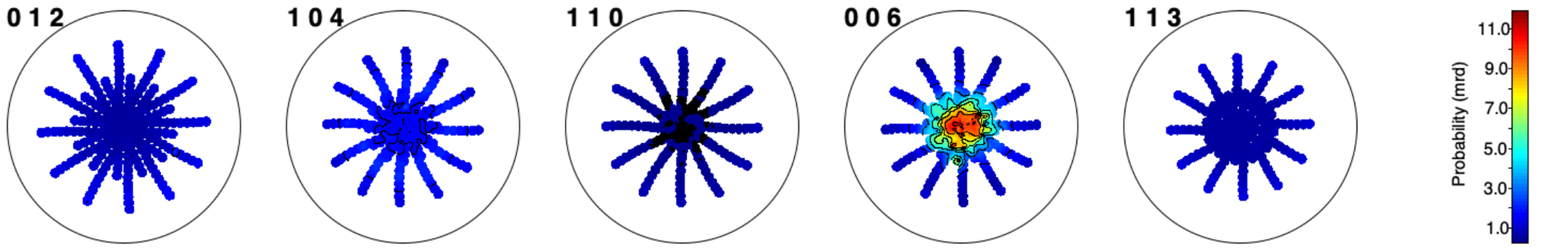
2D fitting (EWIMV)



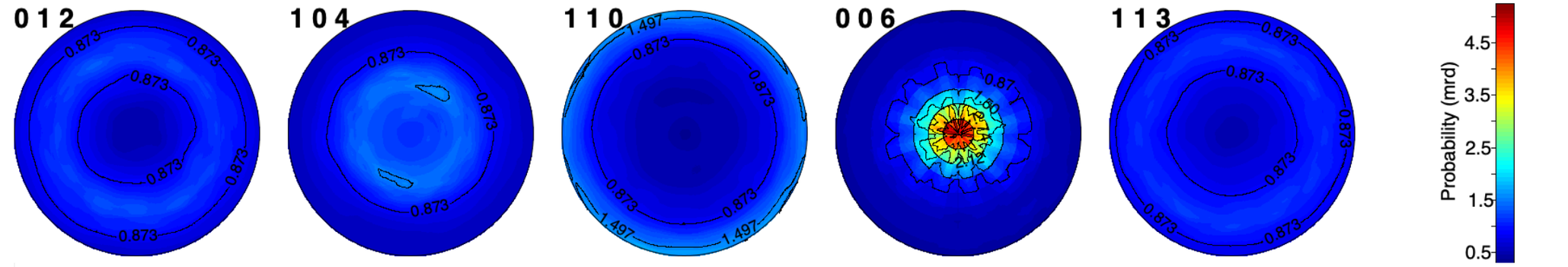
Texture analysis and comparison



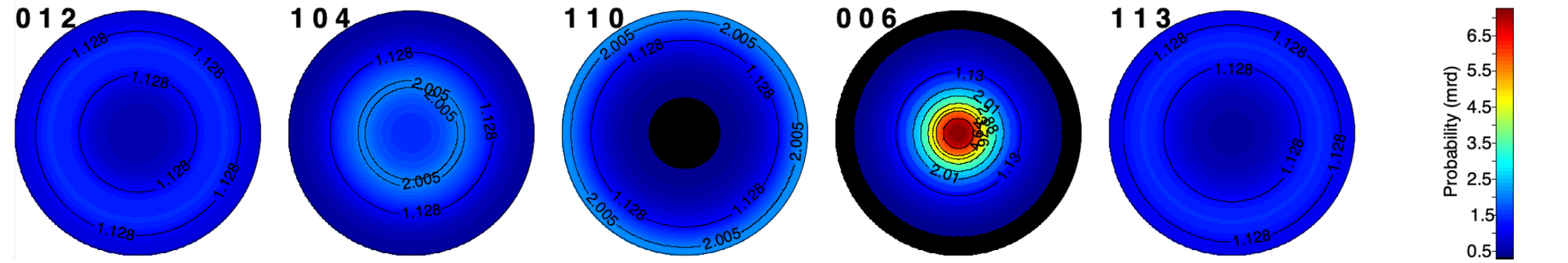
Extracted



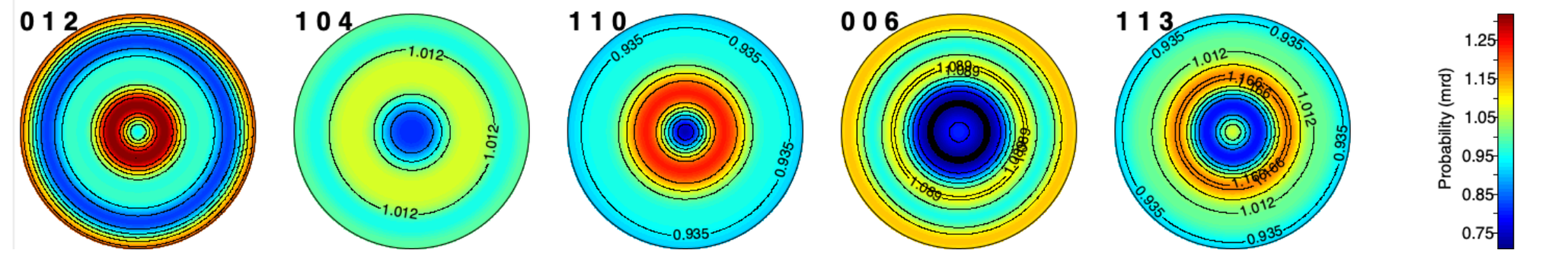
EWIMV



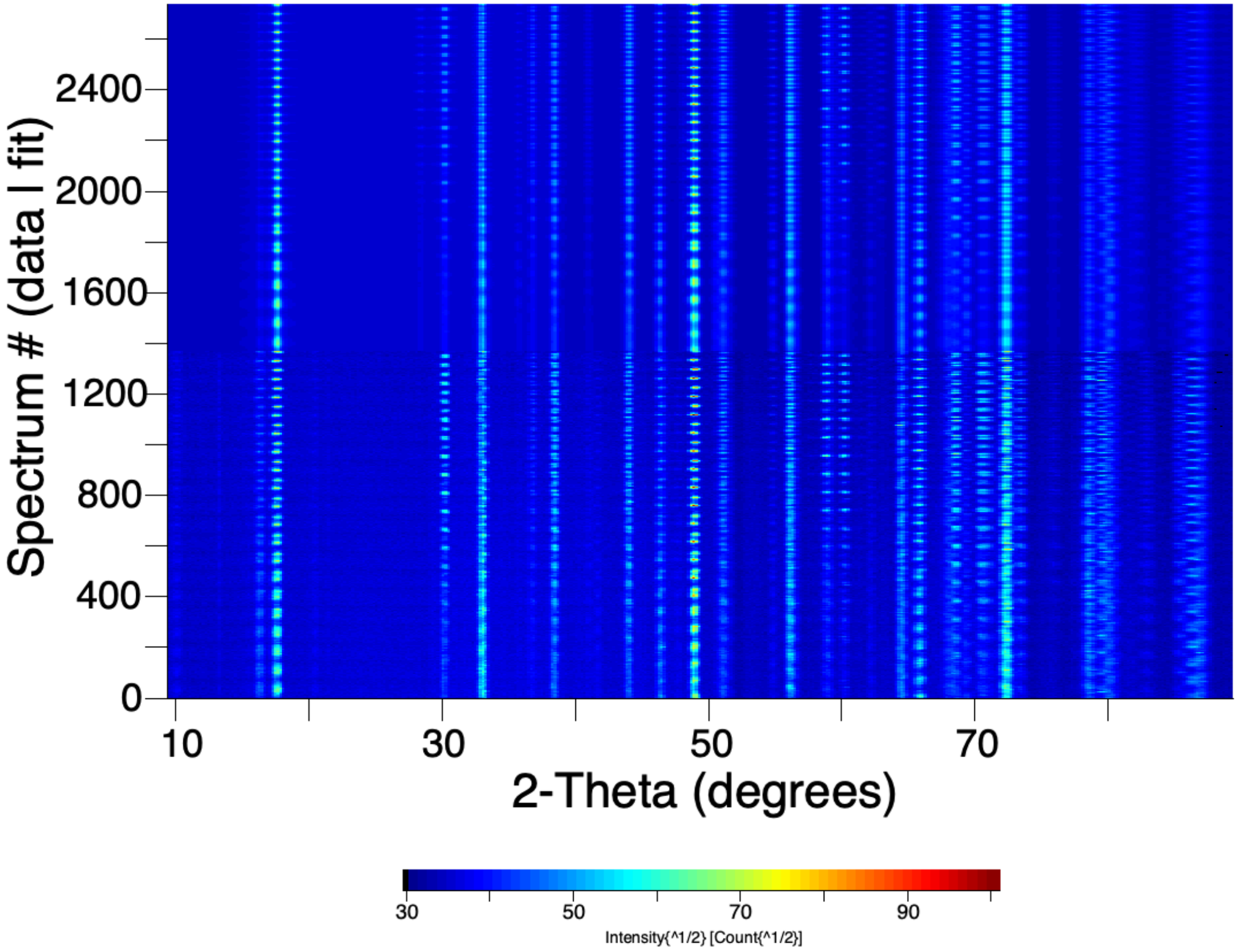
Exponential harmonics



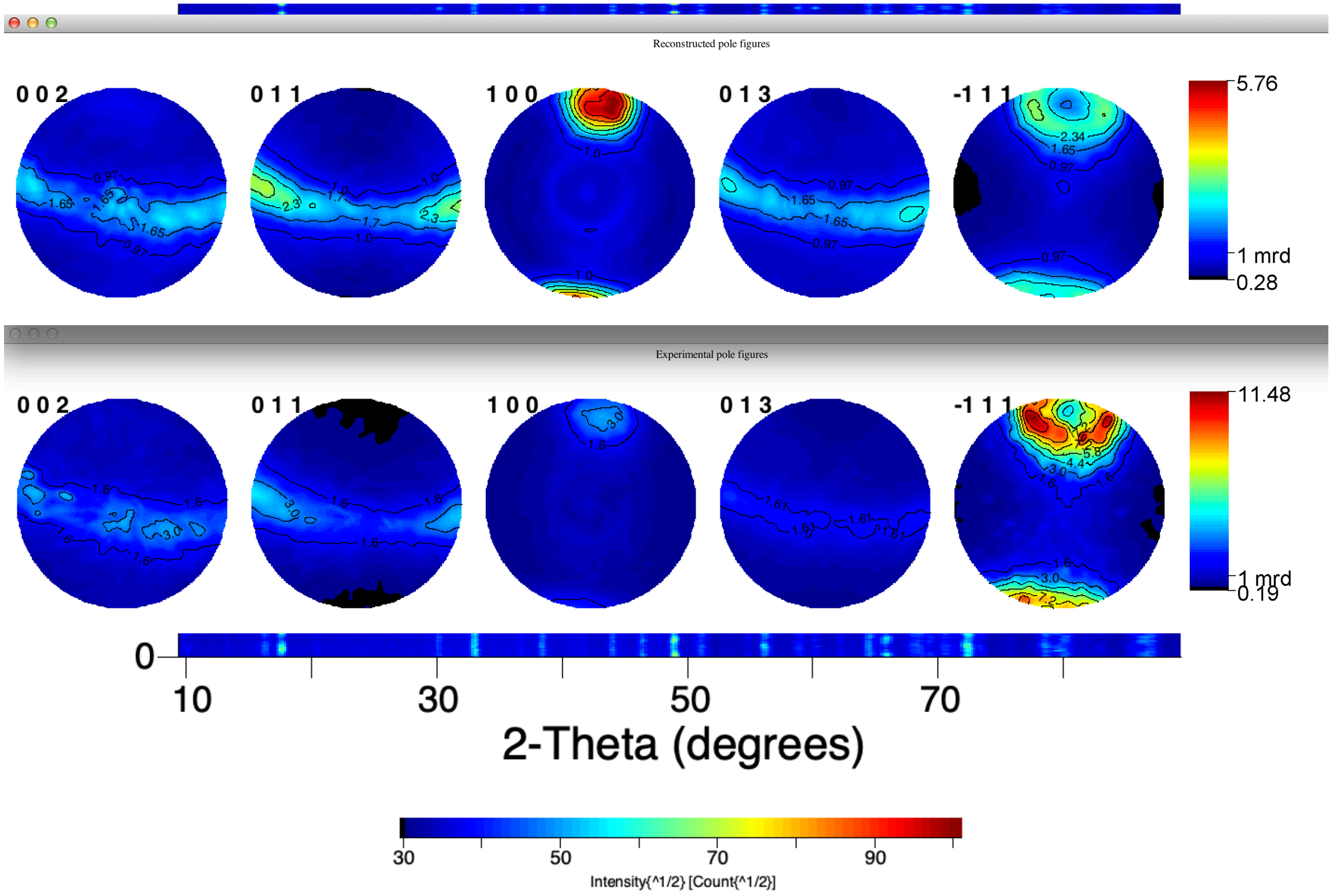
MTEX



Monoclinic glauconite (C2/m)



Monoclinic glauconite (C2/m)



MAUD and MTEX

- The basic procedure is working, there are plenty of improvements possible
- MTEX is not optimise for MAUD (more like inverse pole figures: several hkl but fewer points in the pole figure space)
- There are some problems to obtain the correct ODF (not correct options? Old version of MTEX?)
- The monoclinic does not work at all (may be on the new MTEX)