

# MAUD and MTEX

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UNIVERSITY OF TRENTO - Italy

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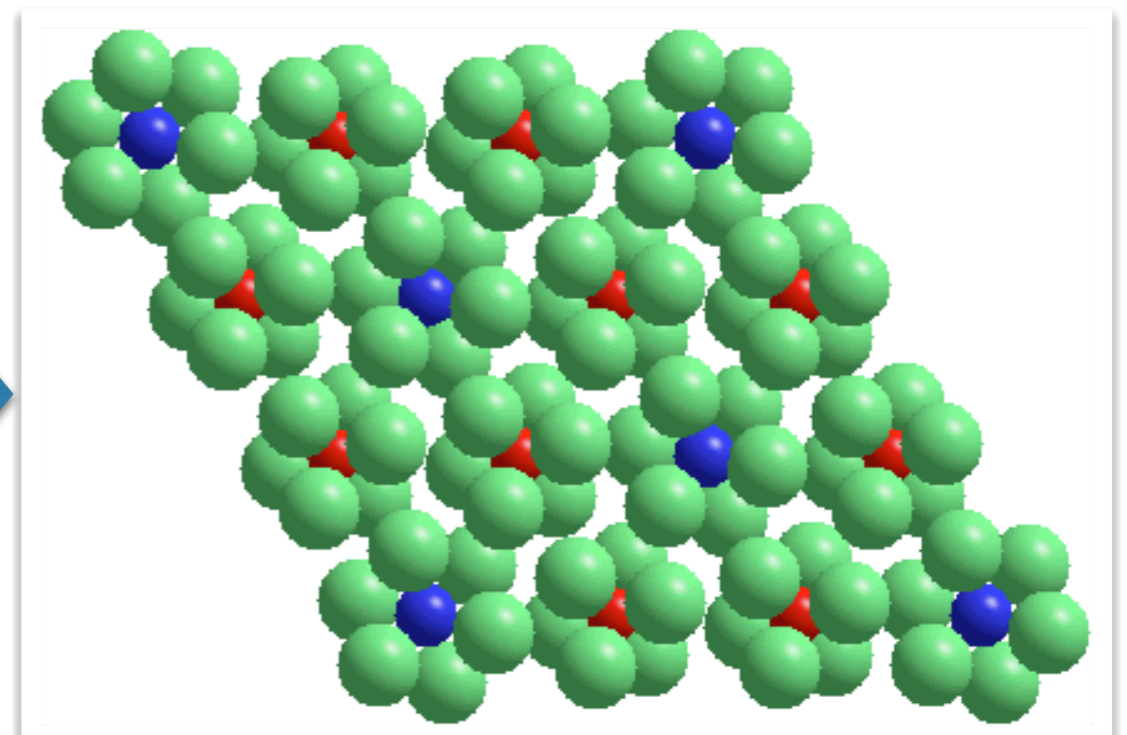
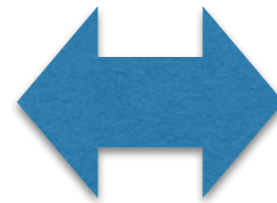
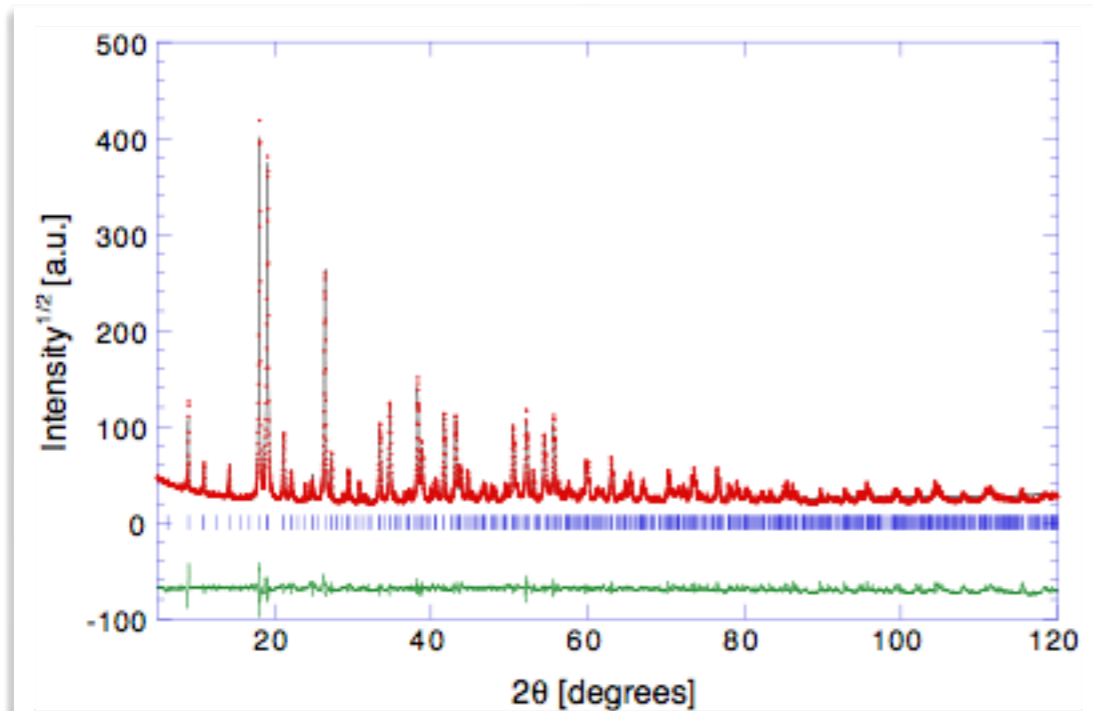
Department of Industrial Engineering

Combined analysis:  
general principles and  
theory

# Pattern fitting: the Rietveld method

- Least squares minimization of:

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



# XRD Pattern calculation: general formula

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

Crystal structure

Crystallite sizes and microstrains

Phase quantities

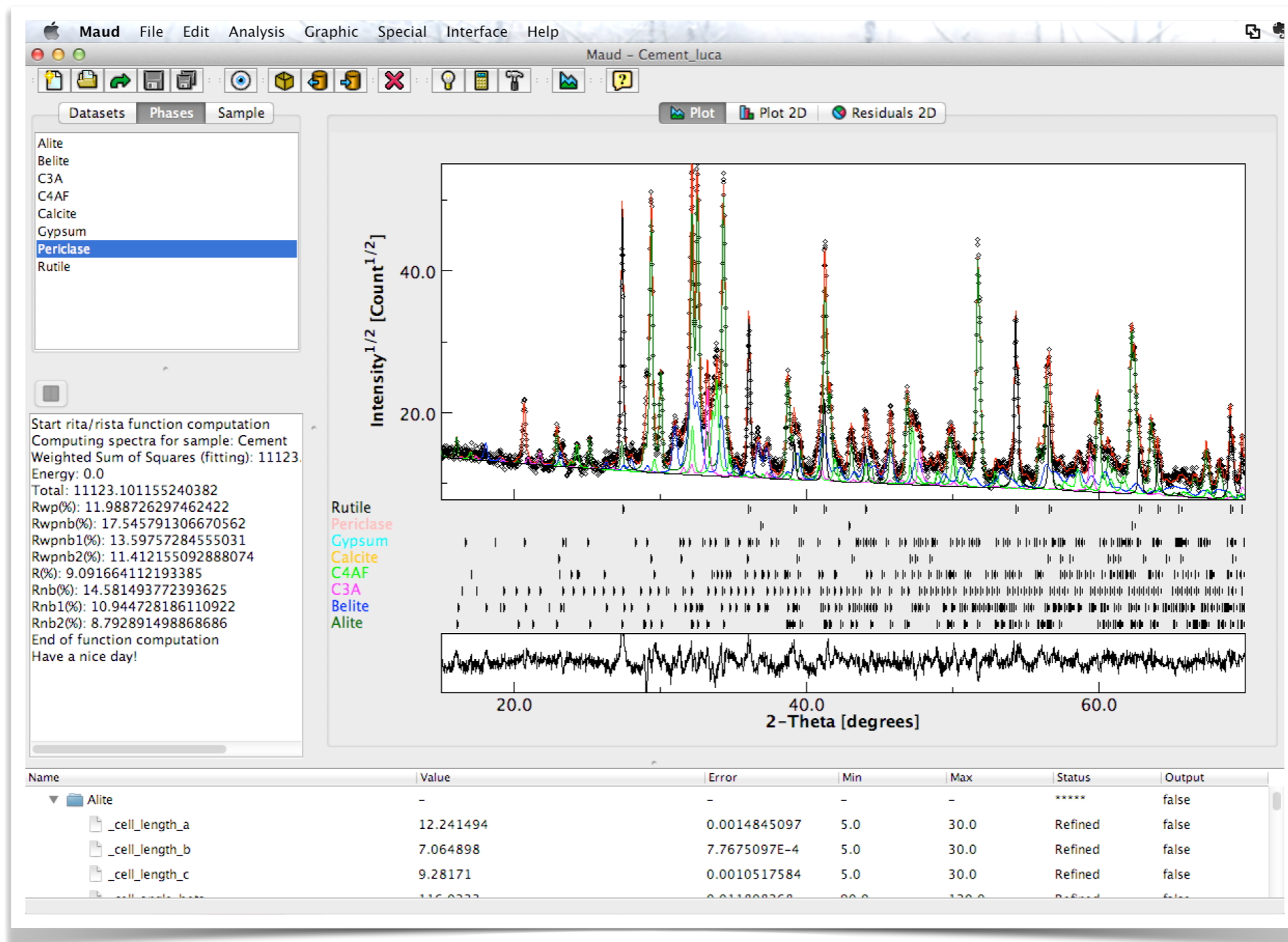
Residual stresses (and cell parameters)

Texture (ODF)

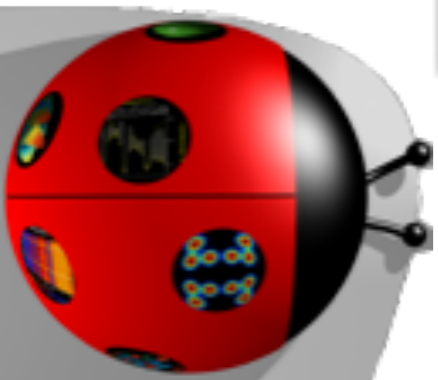
Film thickness

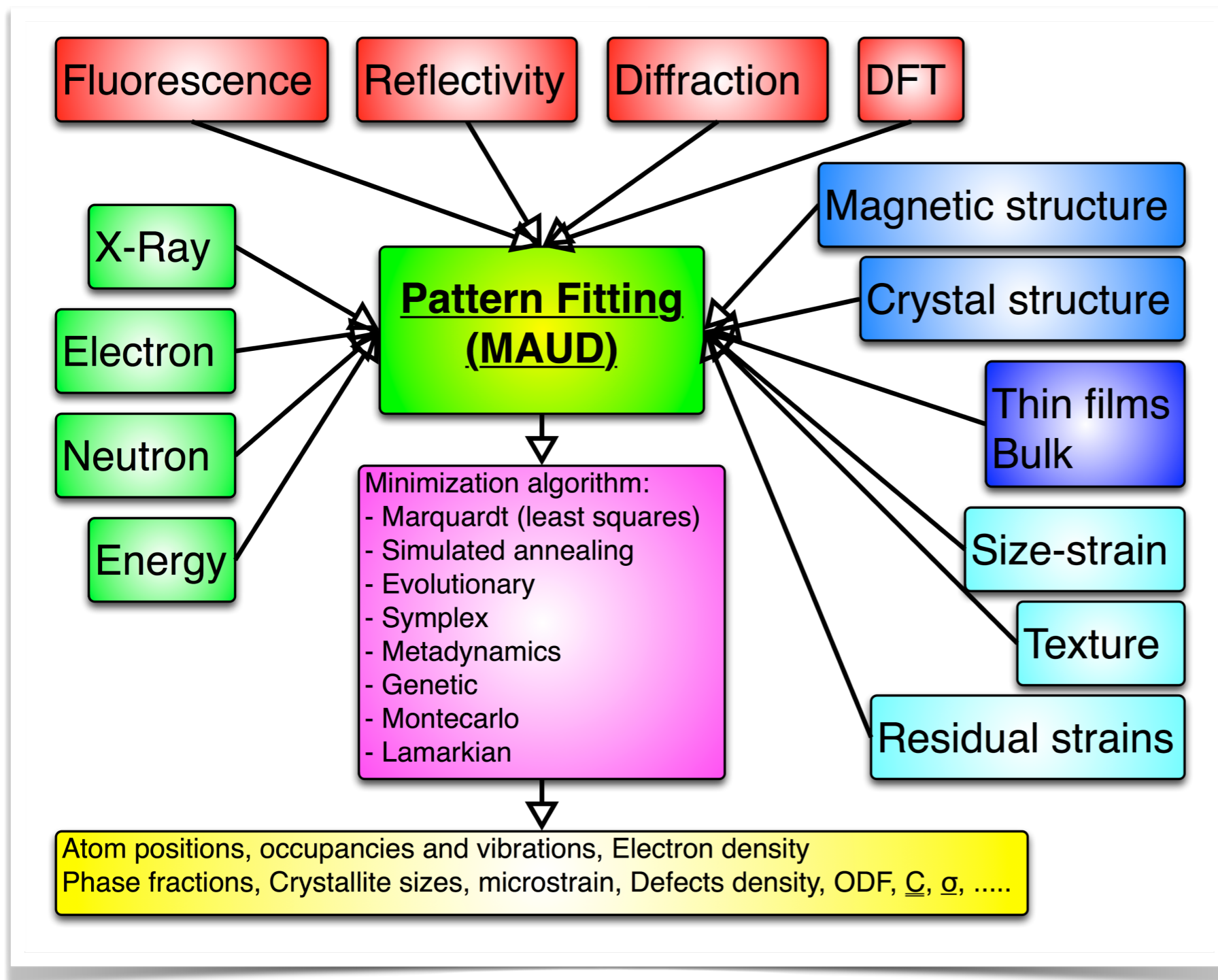
# The MAUD program

## Materials Analysis Using Diffraction



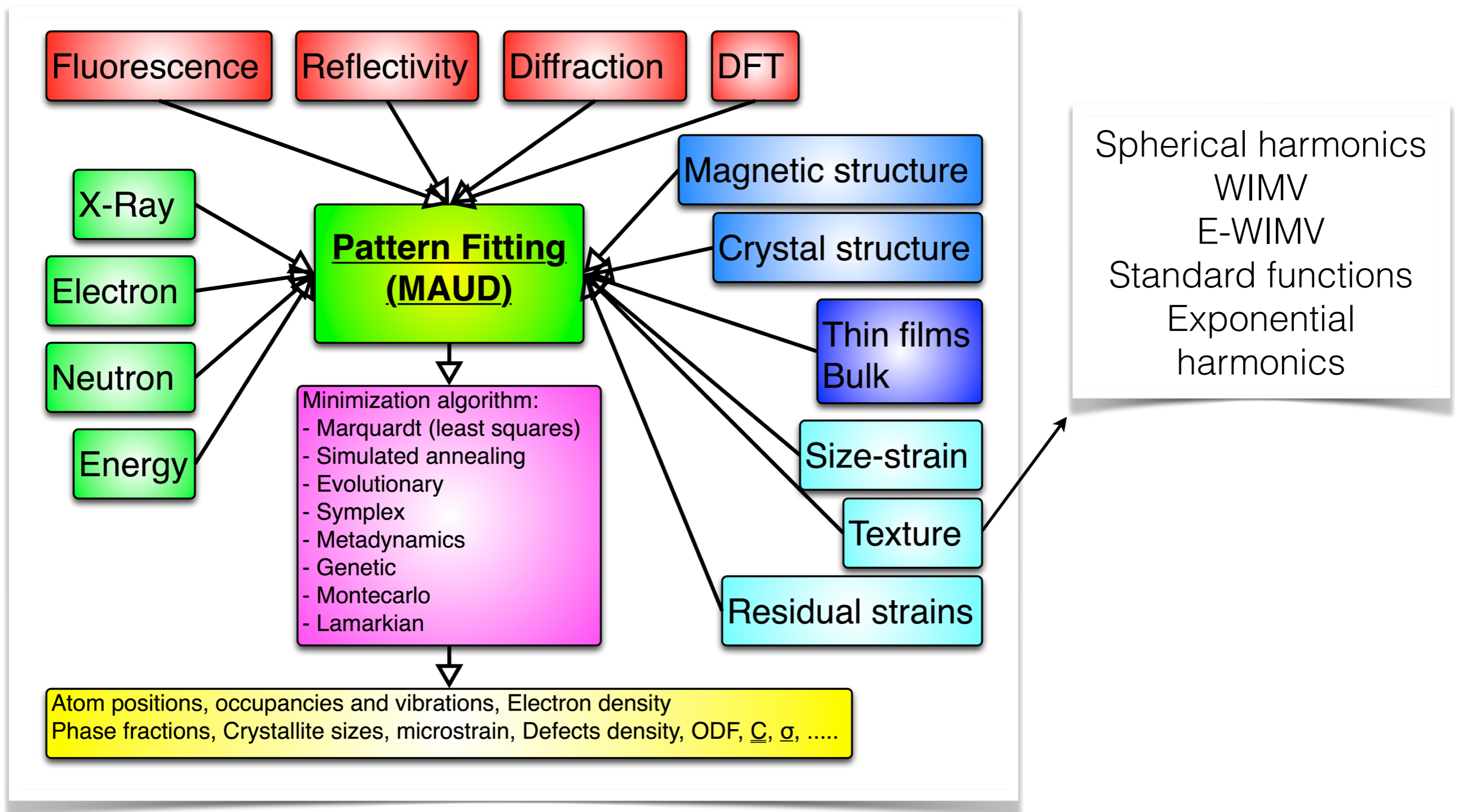
- <http://maud.radiographema.com>





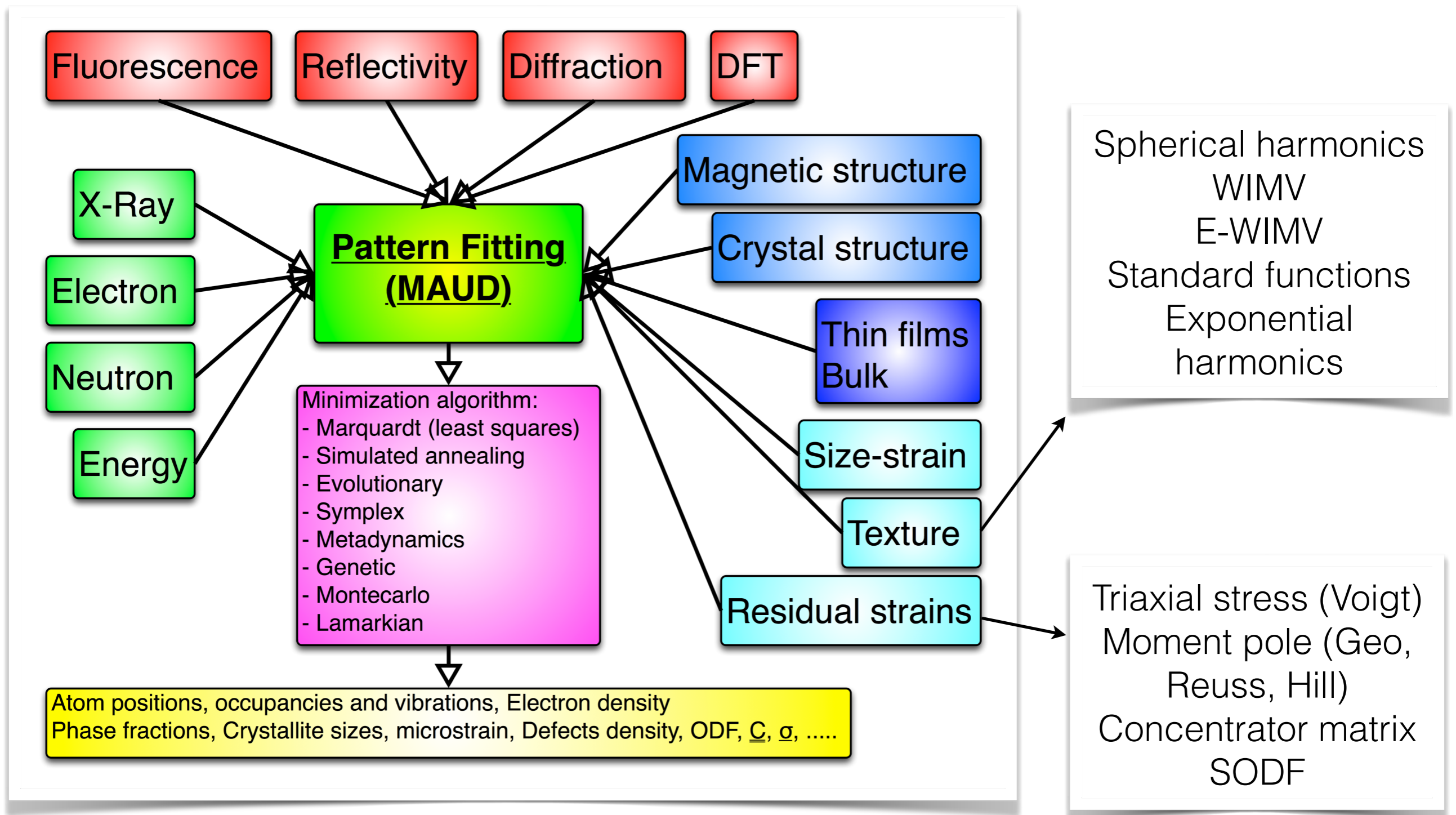
# MAUD structure and models

actual structure



# MAUD structure and models

actual structure

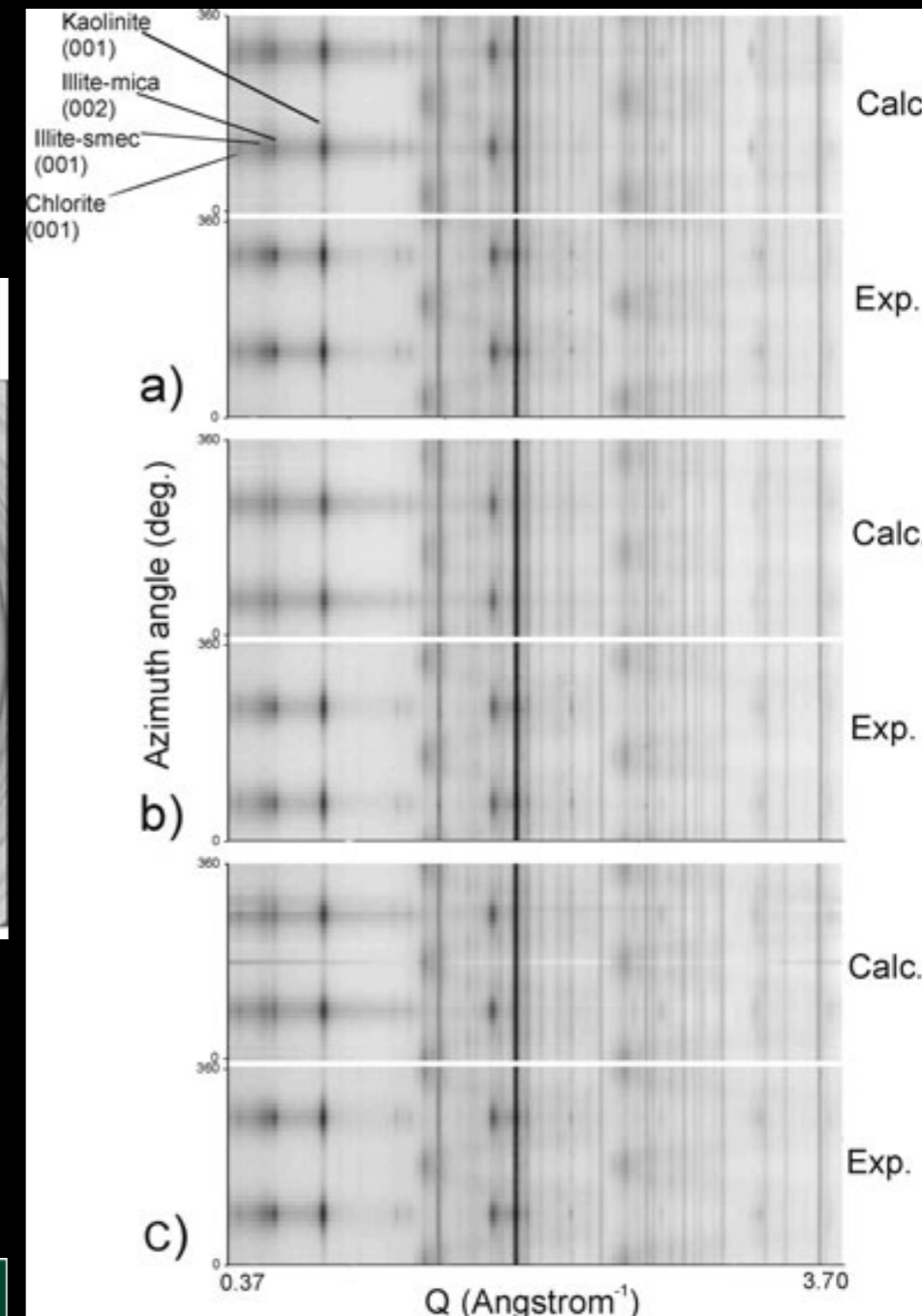
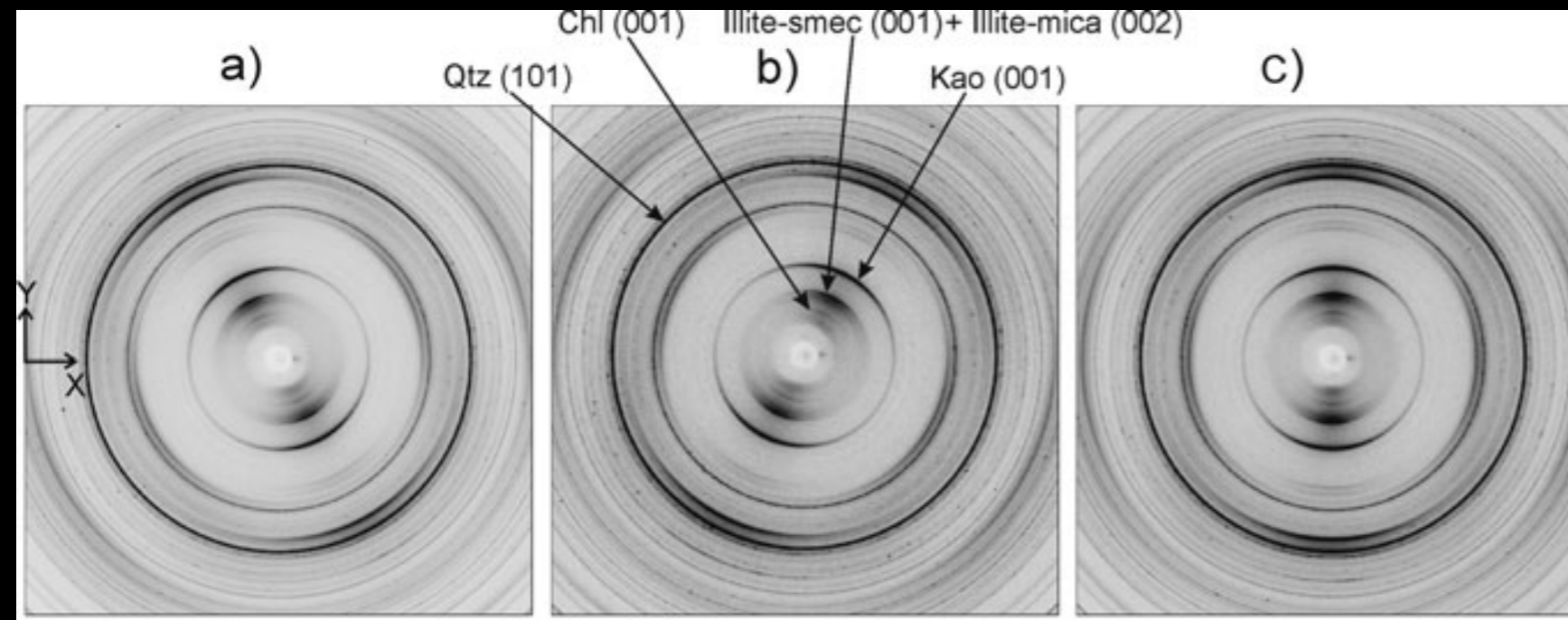


# MAUD structure and models

actual structure



# Texture in shales



Geophysical Prospecting

EAGE

EUROPEAN  
ASSOCIATION OF  
GEOLOGISTS &  
ENGINEERS

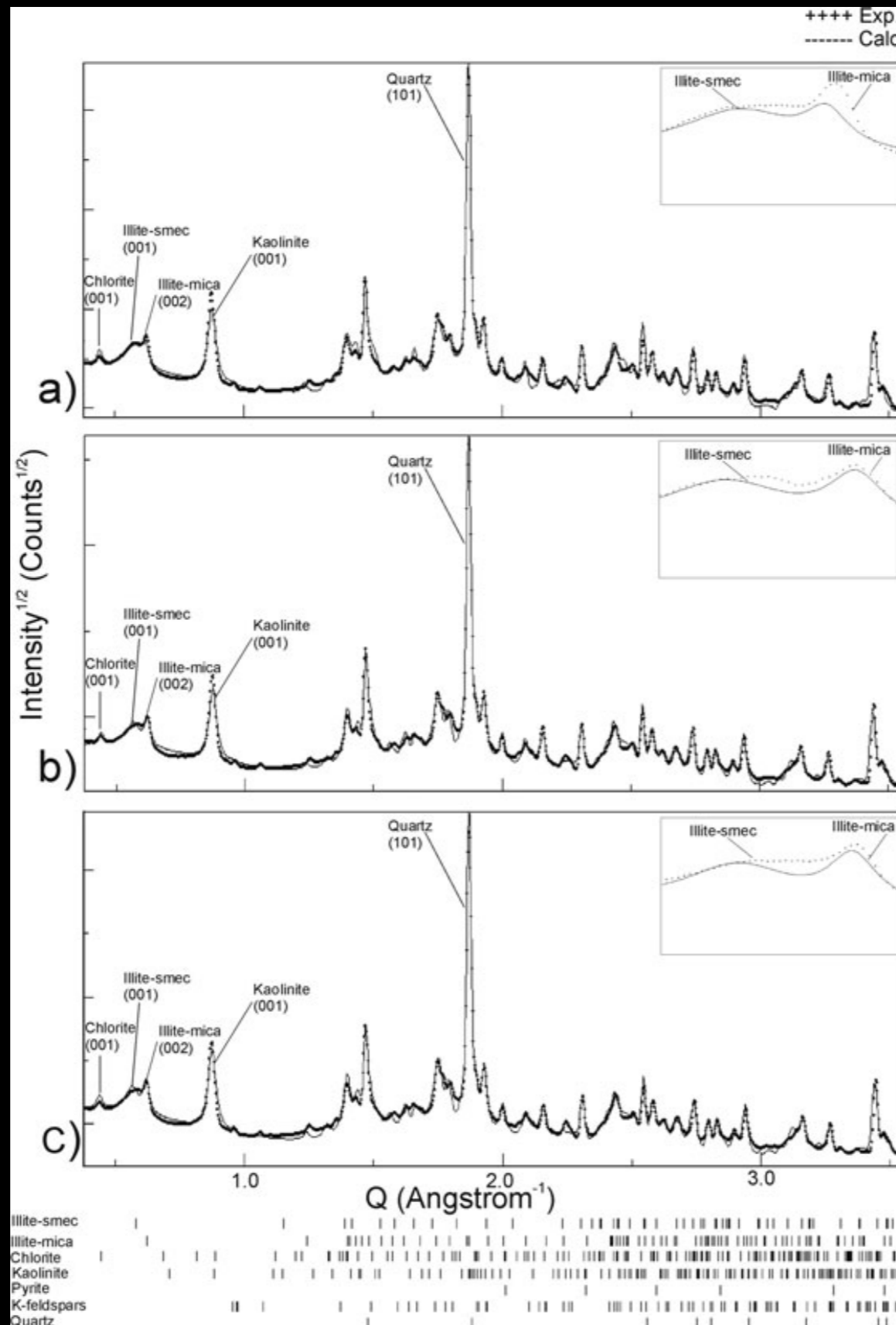
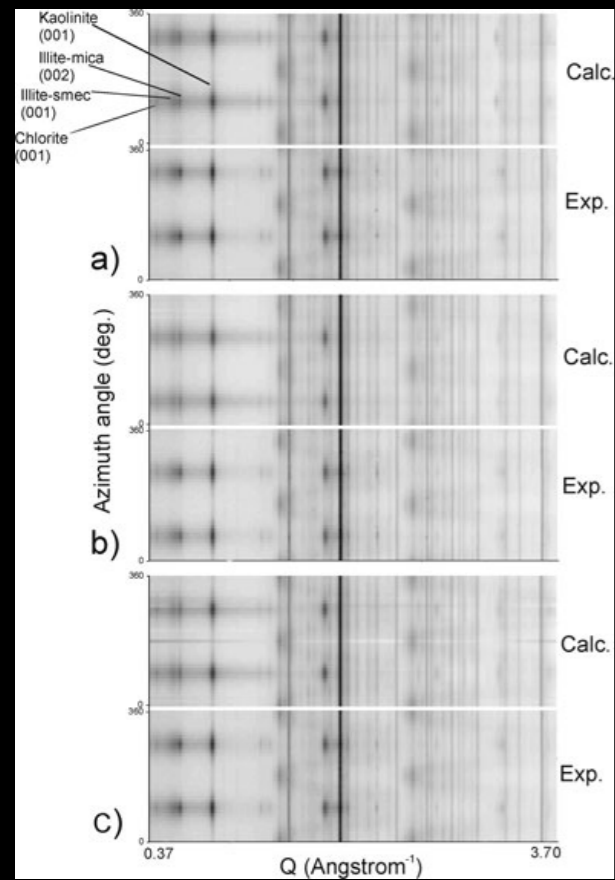
*Geophysical Prospecting*, 2011, 59, 536–556

doi: 10.1111/j.1365-2478.2010.00942.x

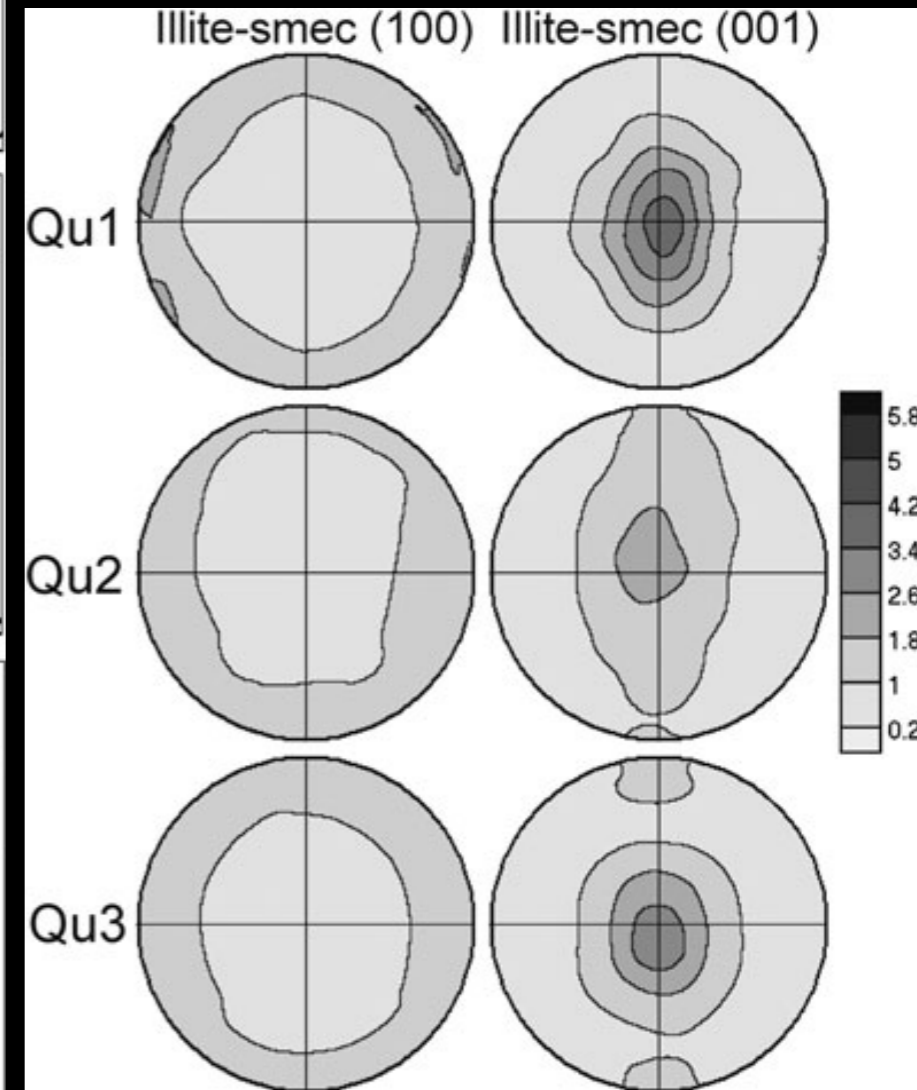
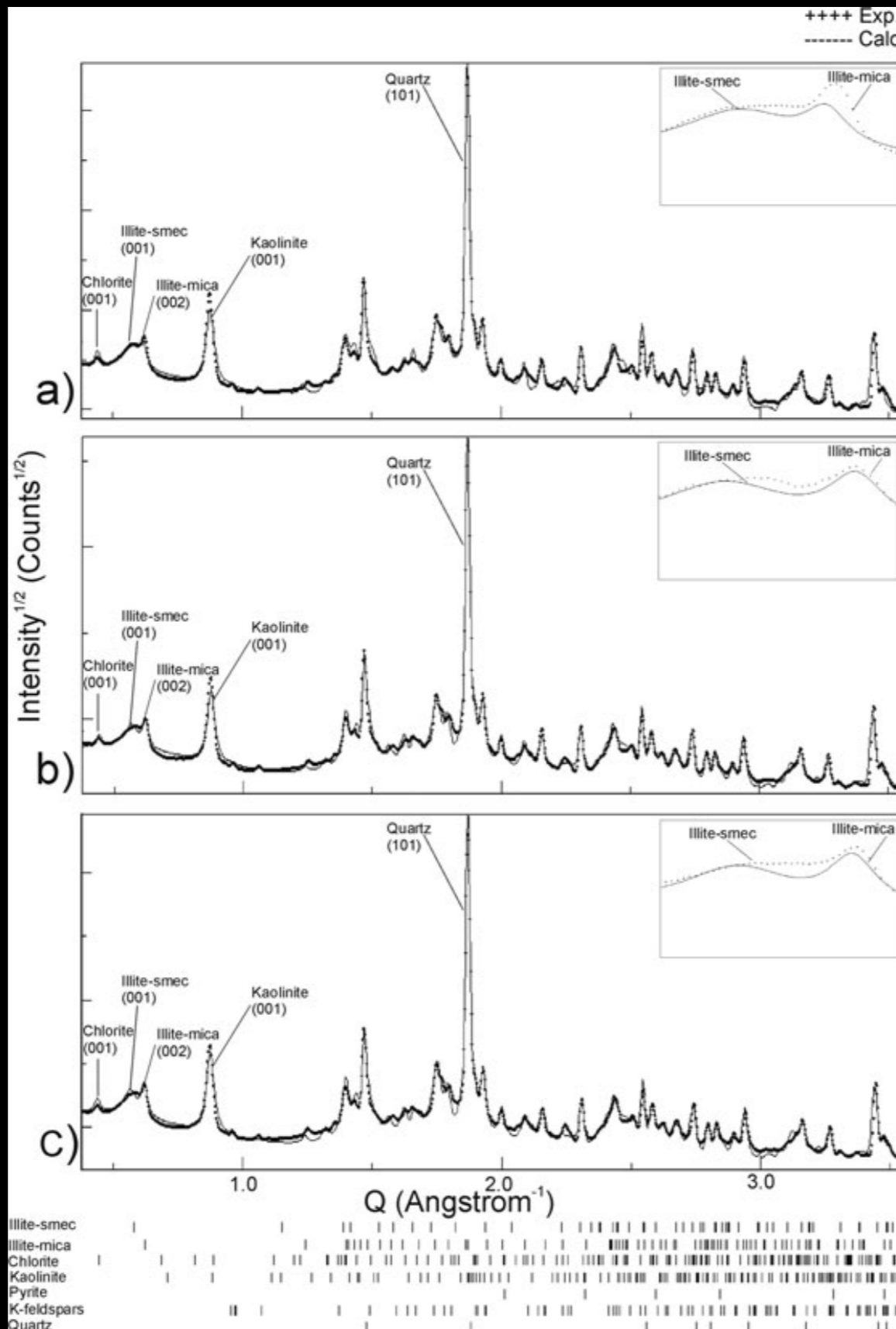
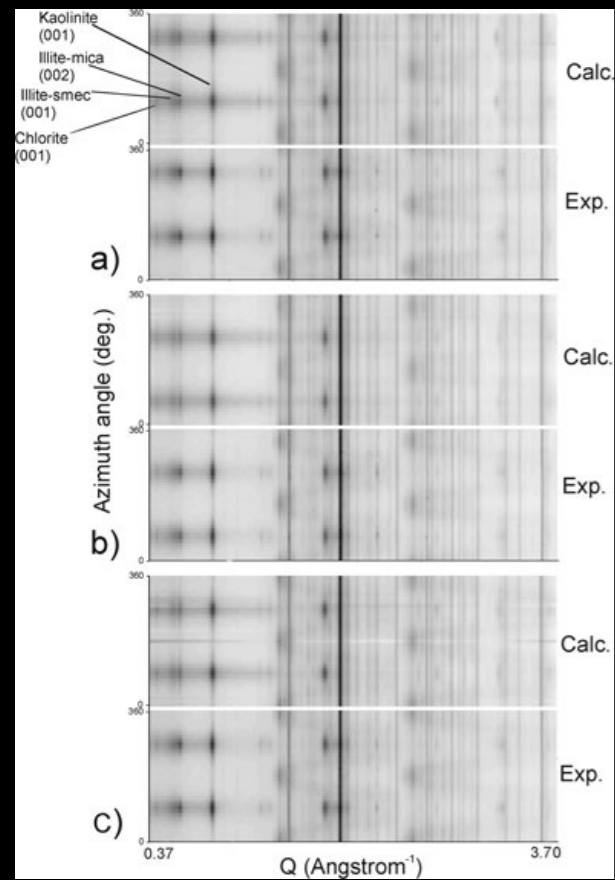
## Texture and anisotropy analysis of Qusaiba shales

Waruntorn Kanitpanyacharoen<sup>1</sup>, Hans-Rudolf Wenk<sup>1\*</sup>, Frans Kets<sup>2,3</sup>,  
Christian Lehr<sup>3</sup> and Richard Wirth<sup>4</sup>

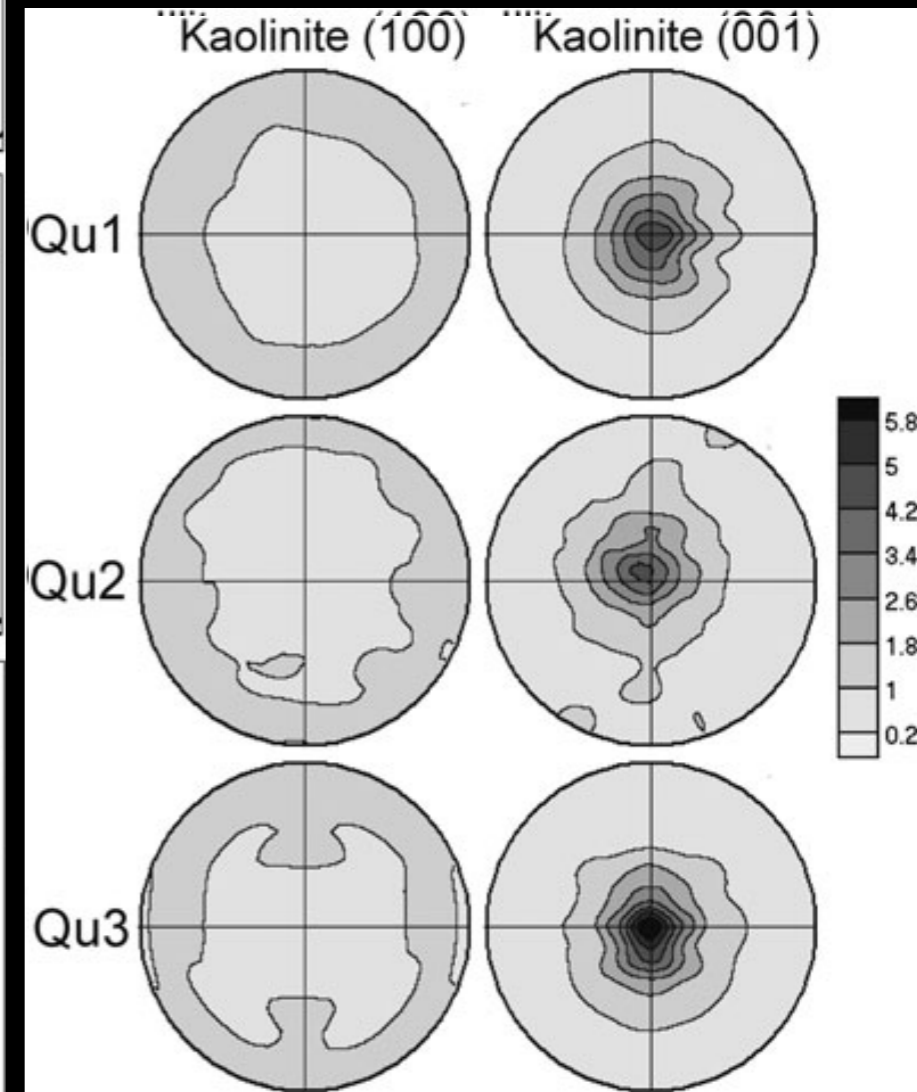
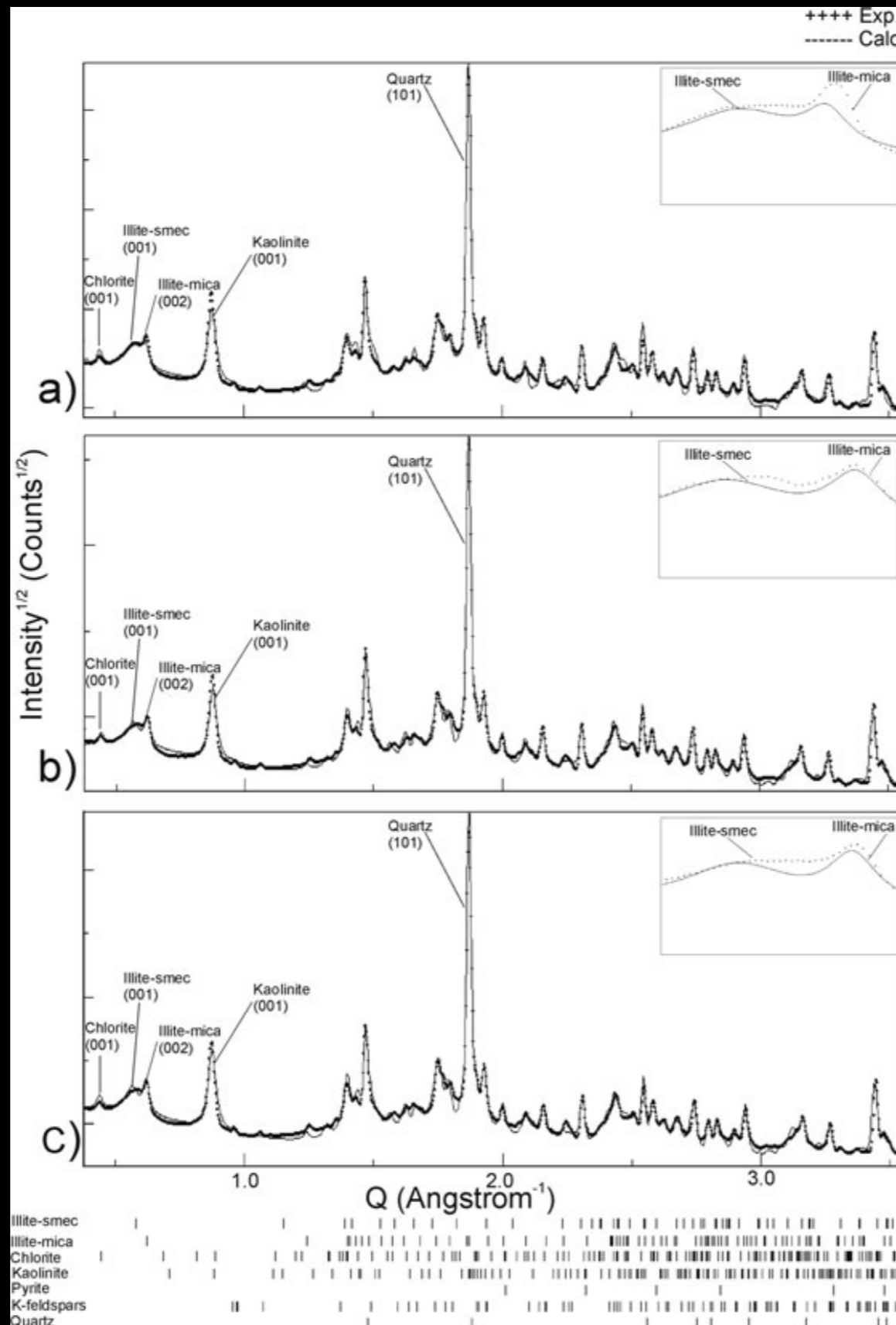
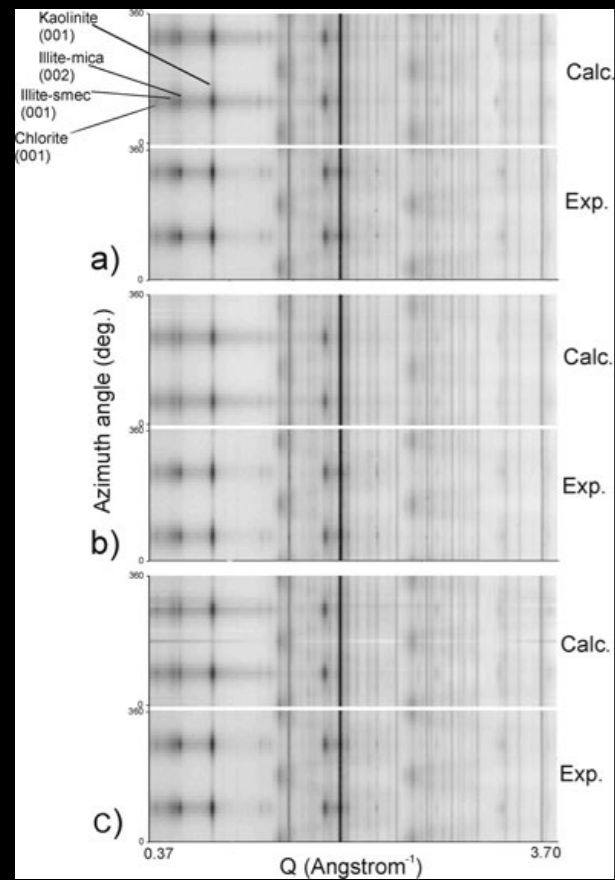
# Texture in shales



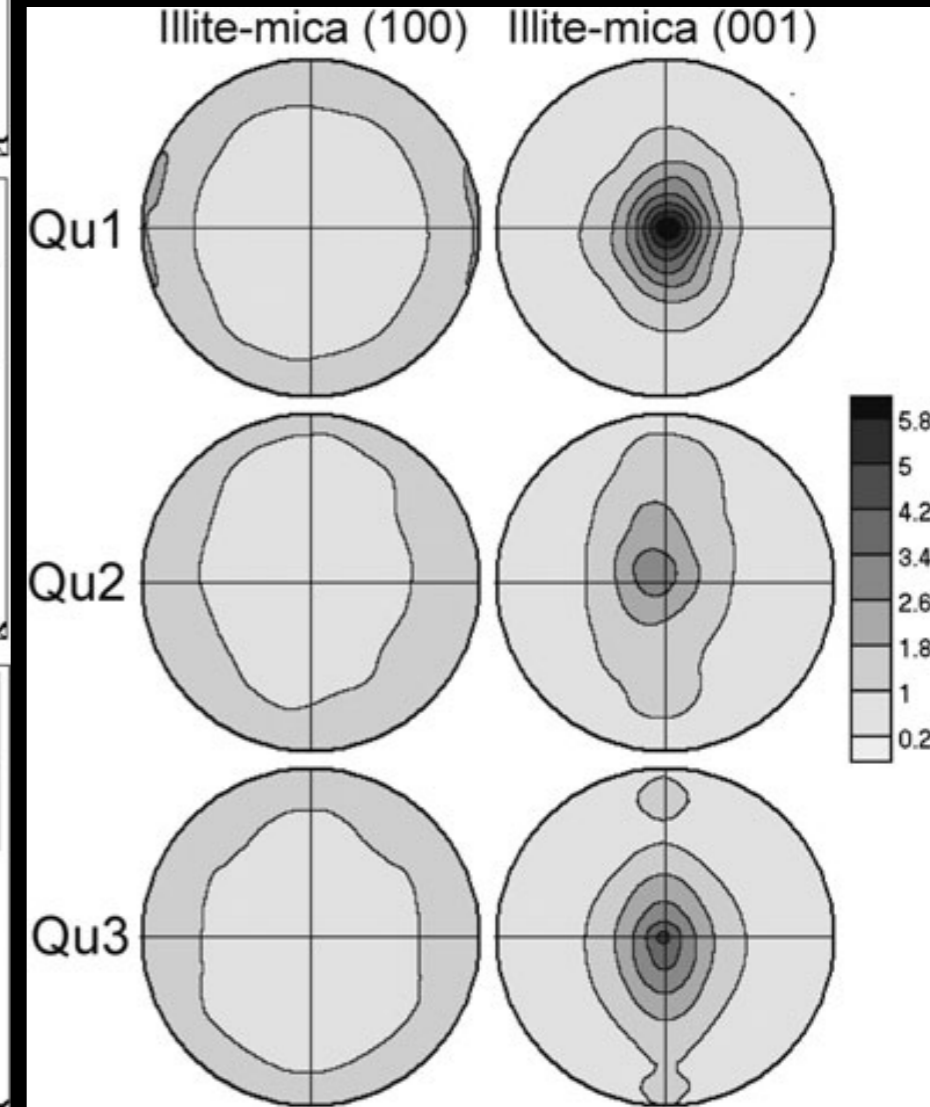
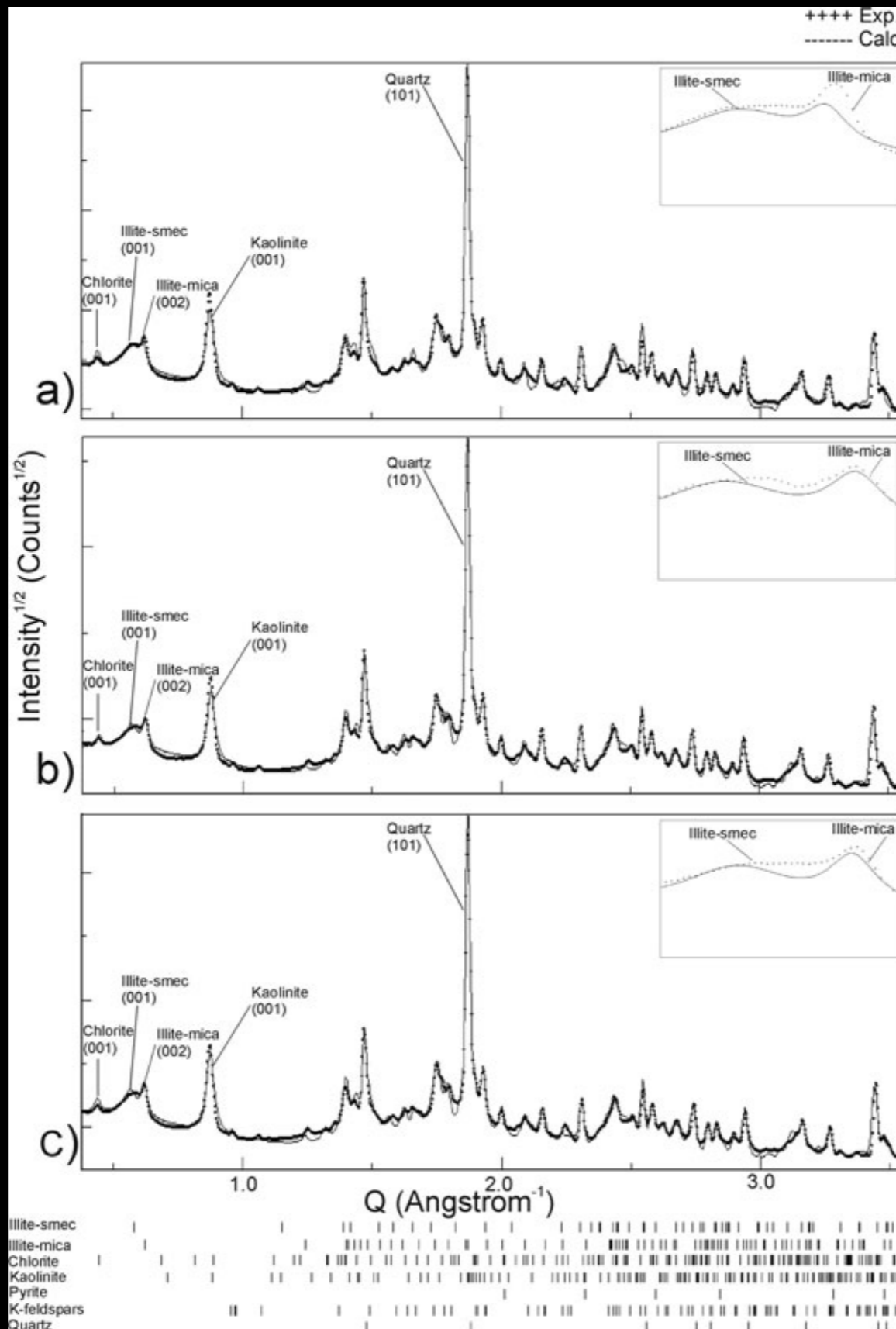
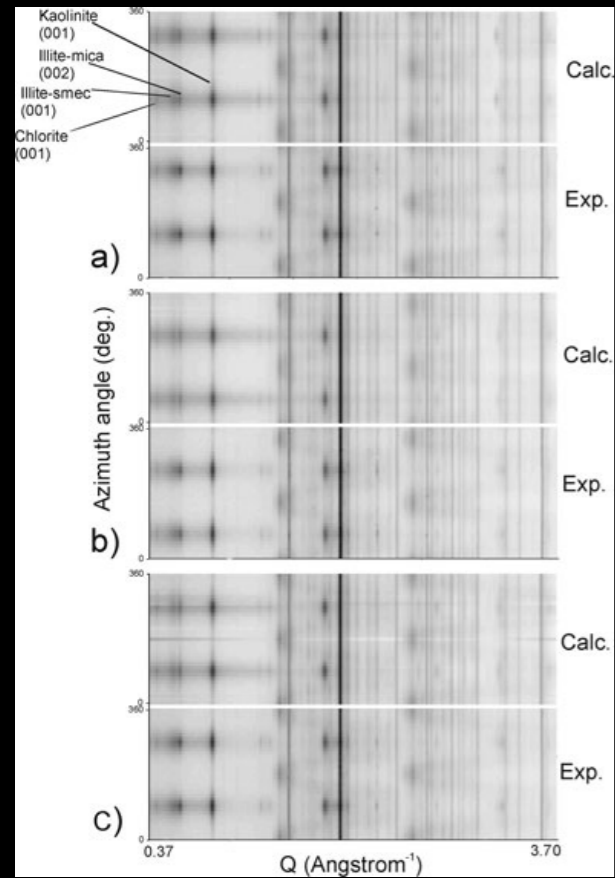
# Texture in shales



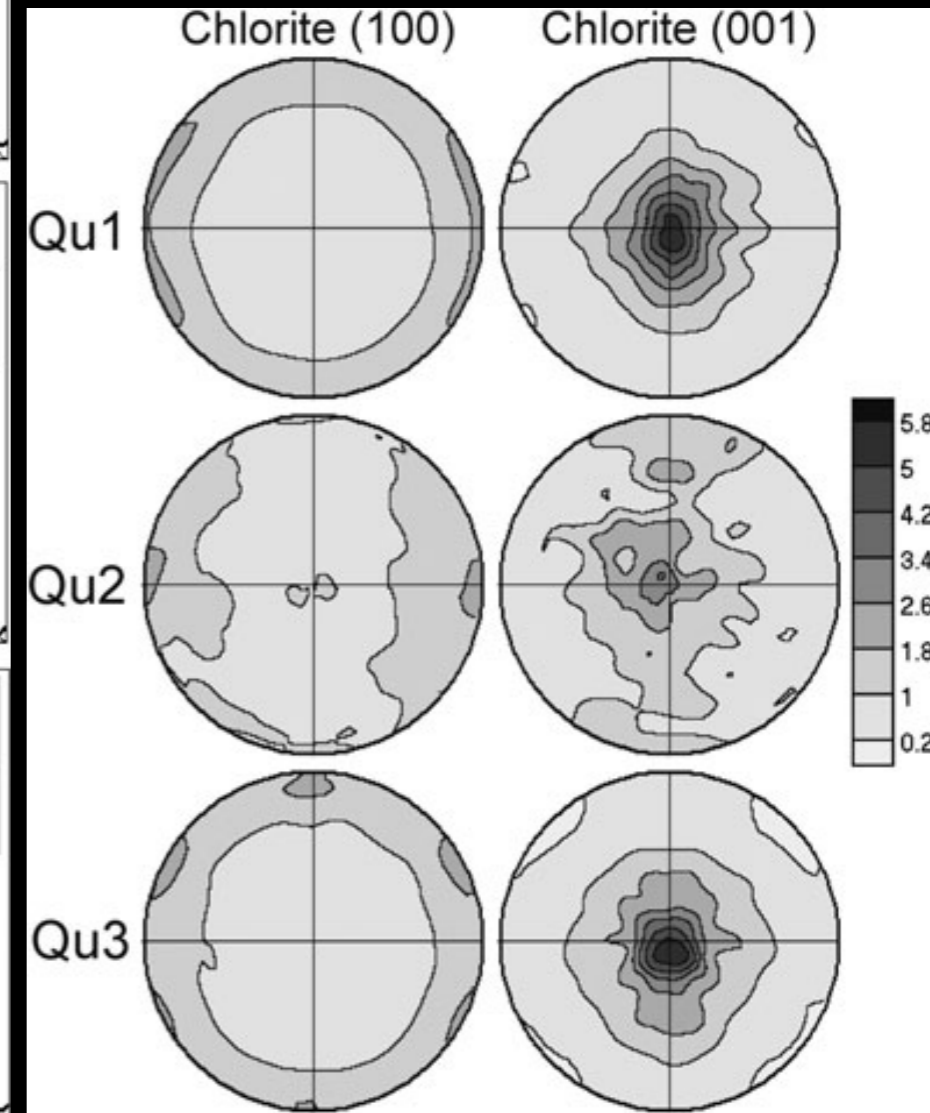
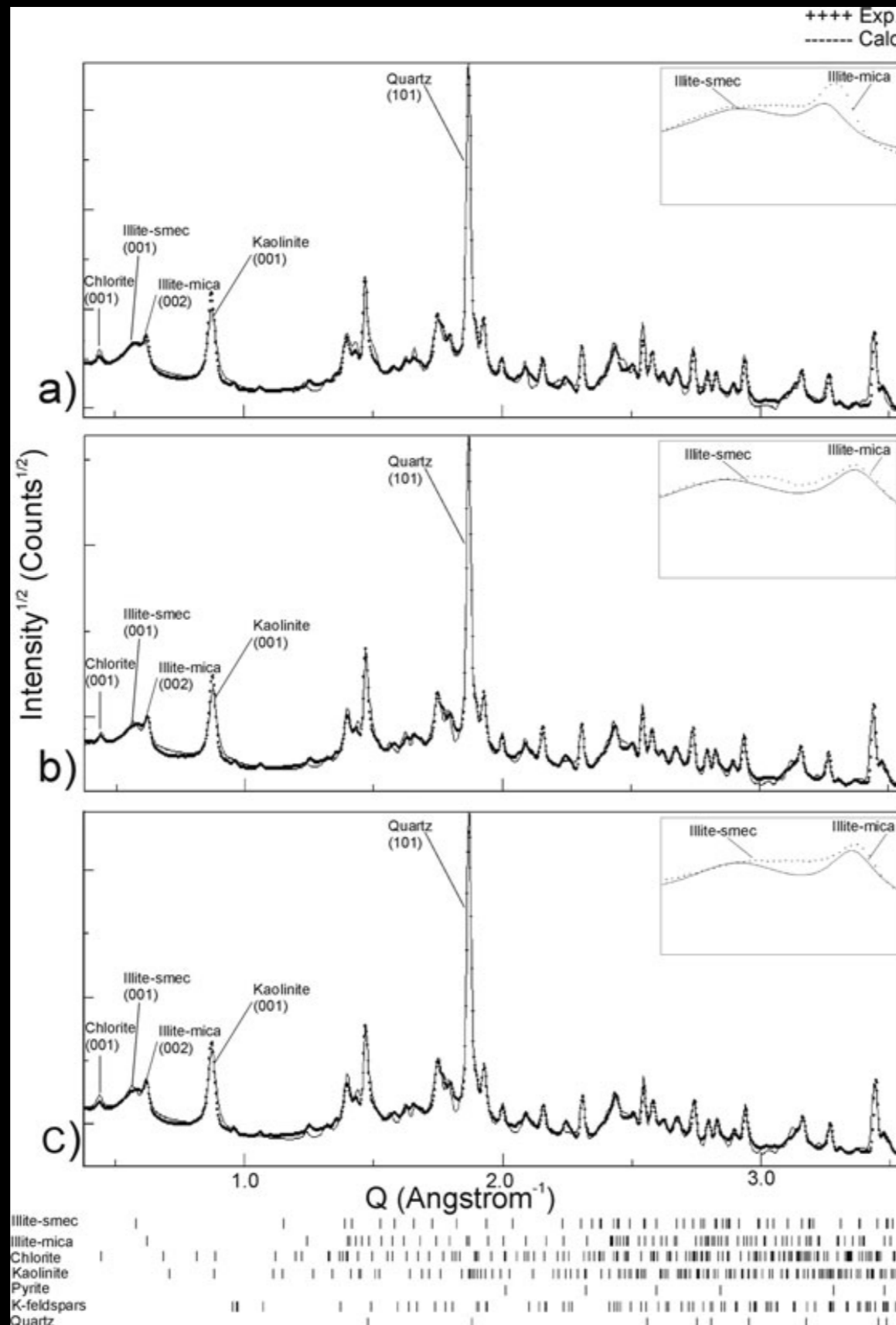
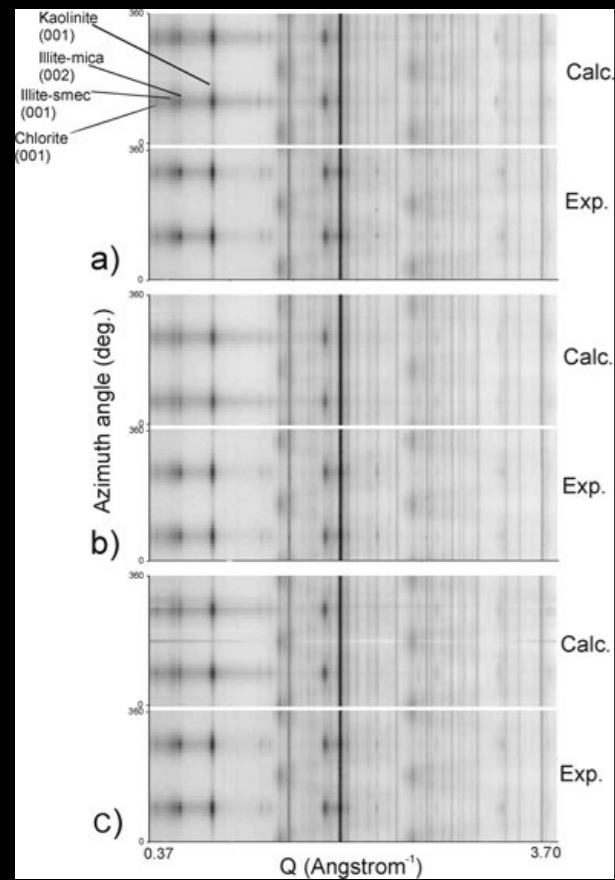
# Texture in shales



# Texture in shales

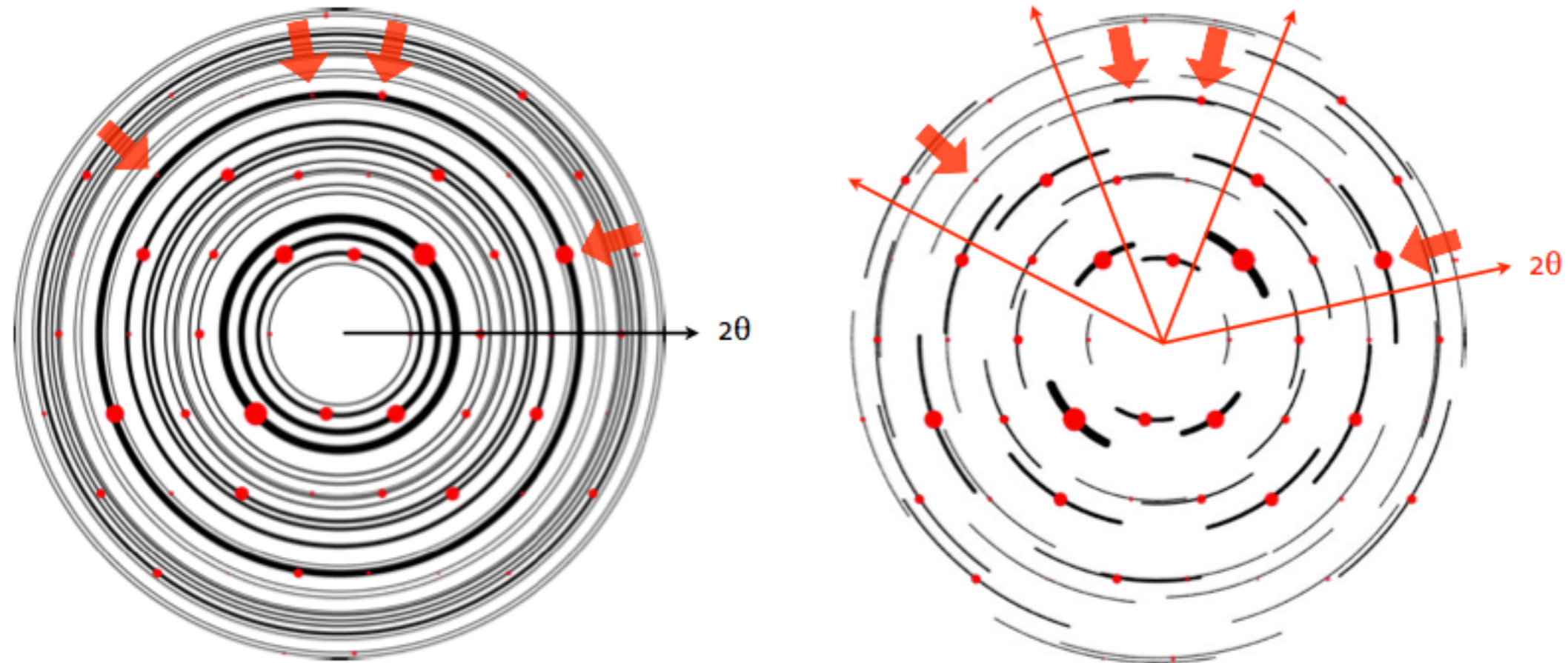


# Texture in shales



# Crystal structure solution by texture

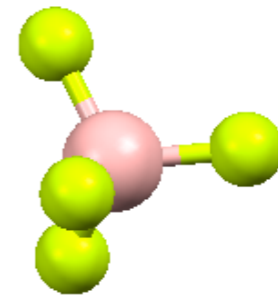
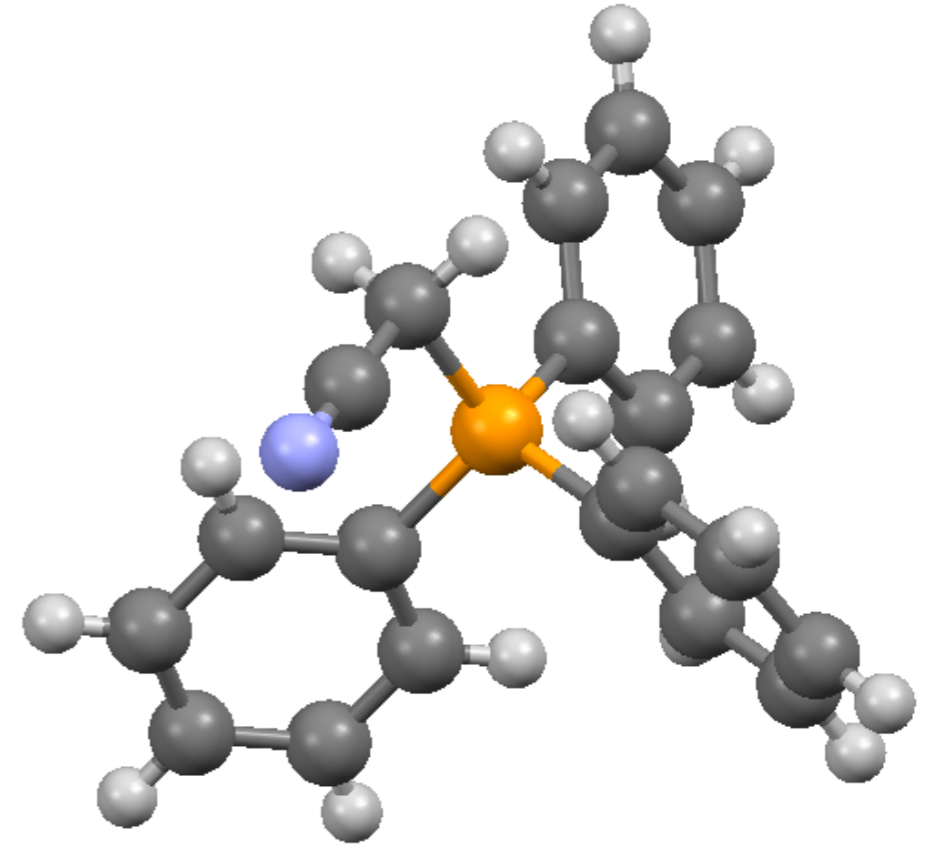
Wessels T., Baerlocher Ch. and McCusker L.B., Science, 284, 477-479, 1999



- 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

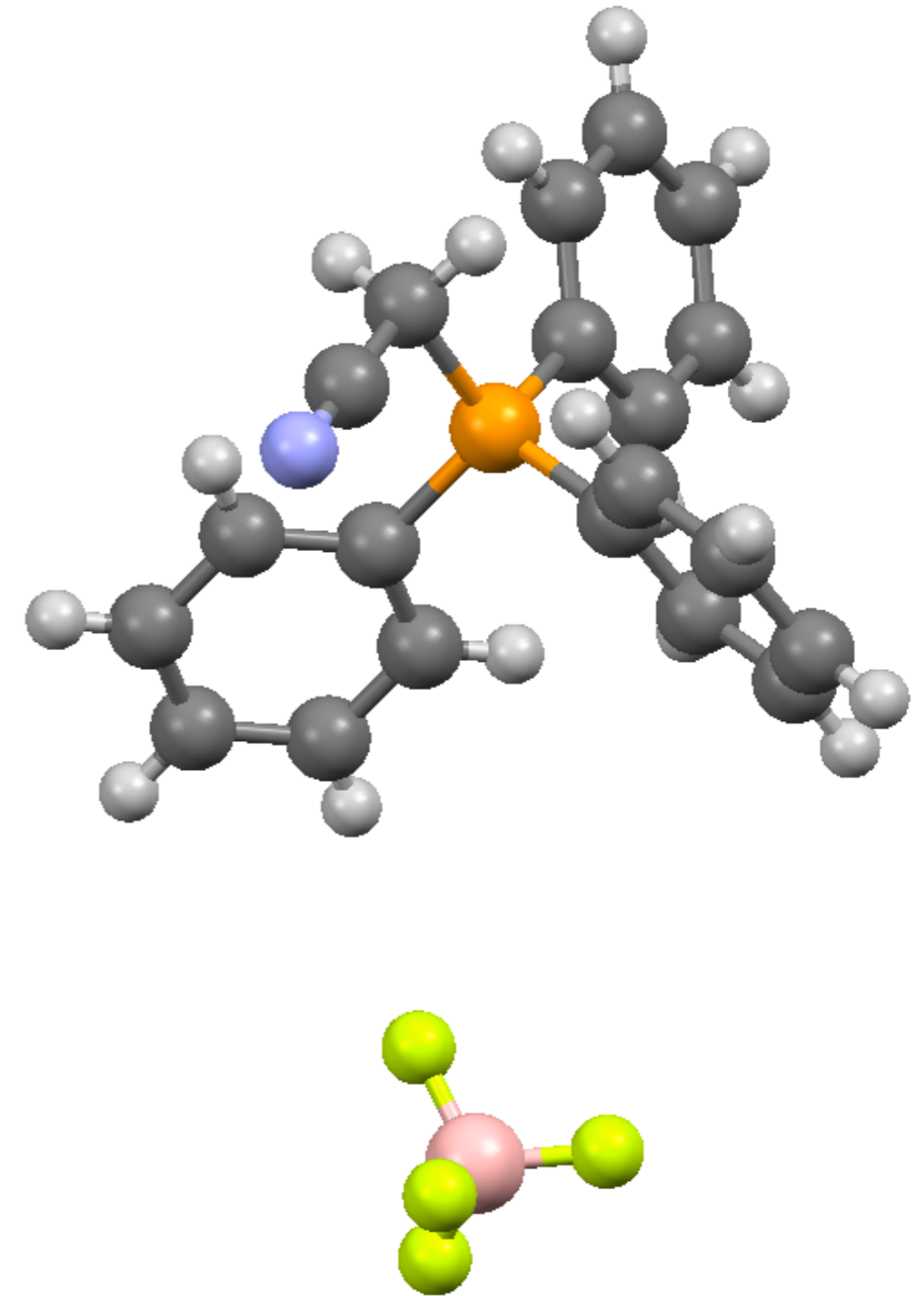
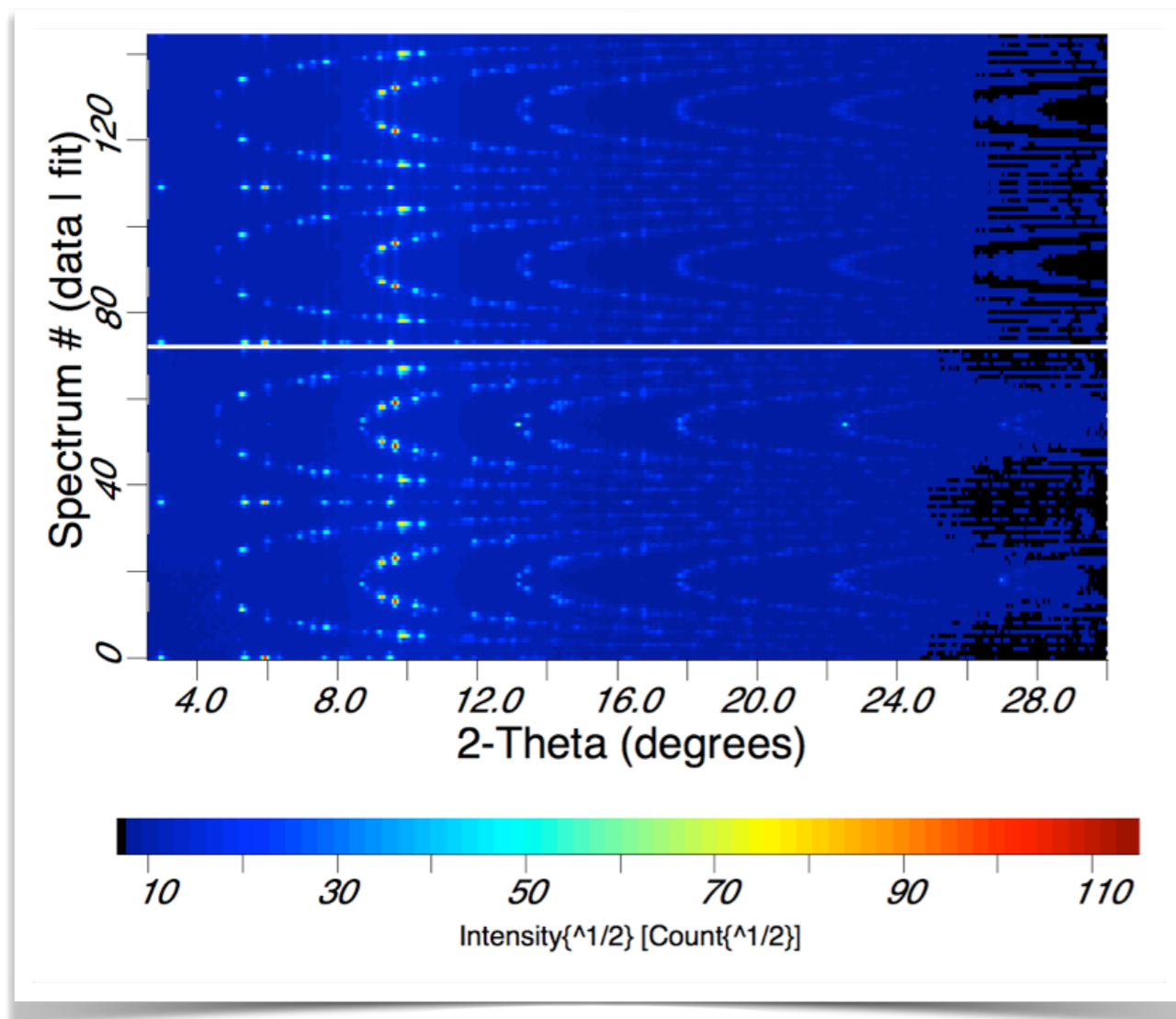
- Standard function for texture
- 4 fragments + 2 subfragments
- Lamarkian optimization





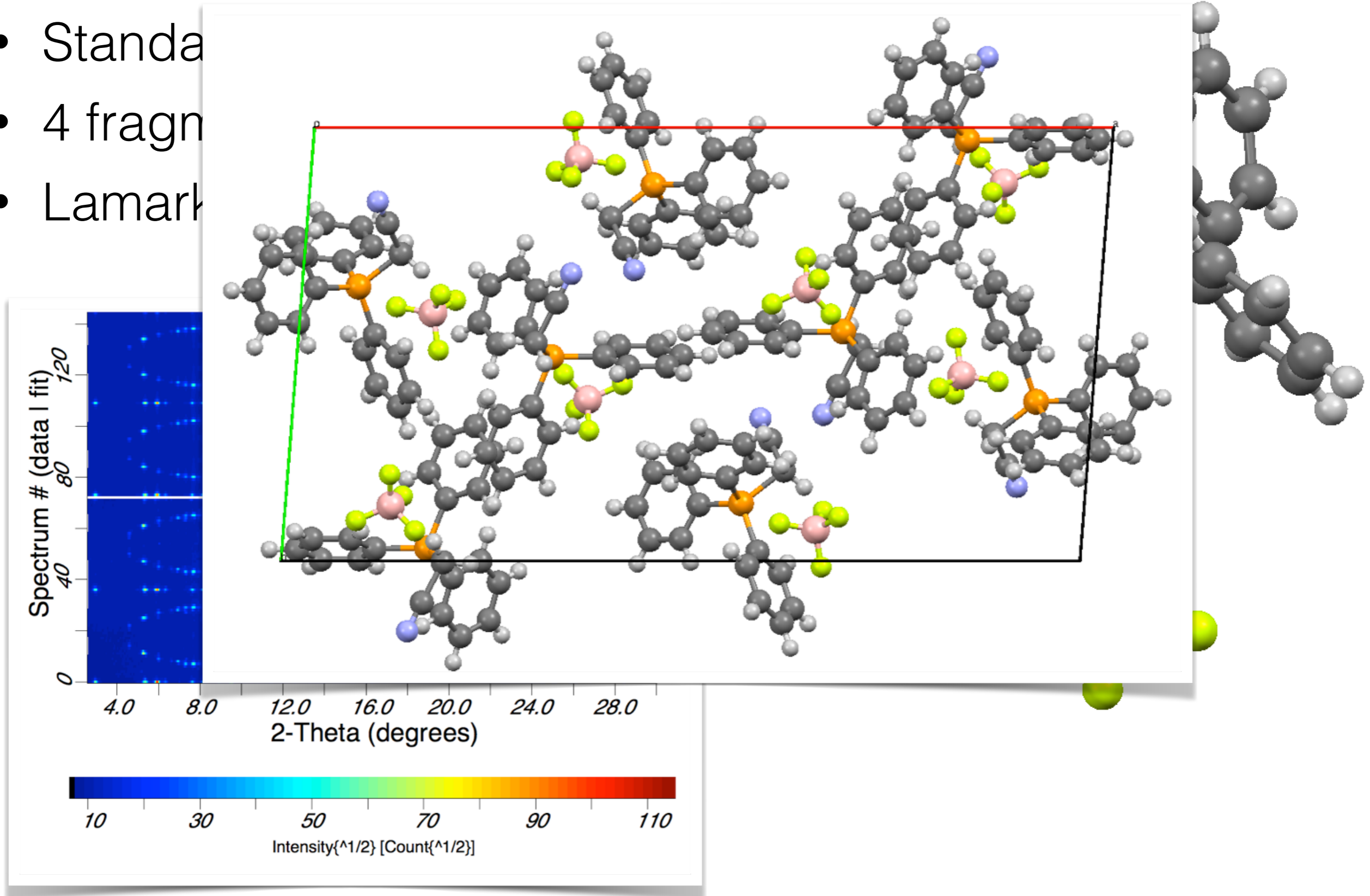
# Crystal structure solution

- Standard function for texture
- 4 fragments + 2 subfragments
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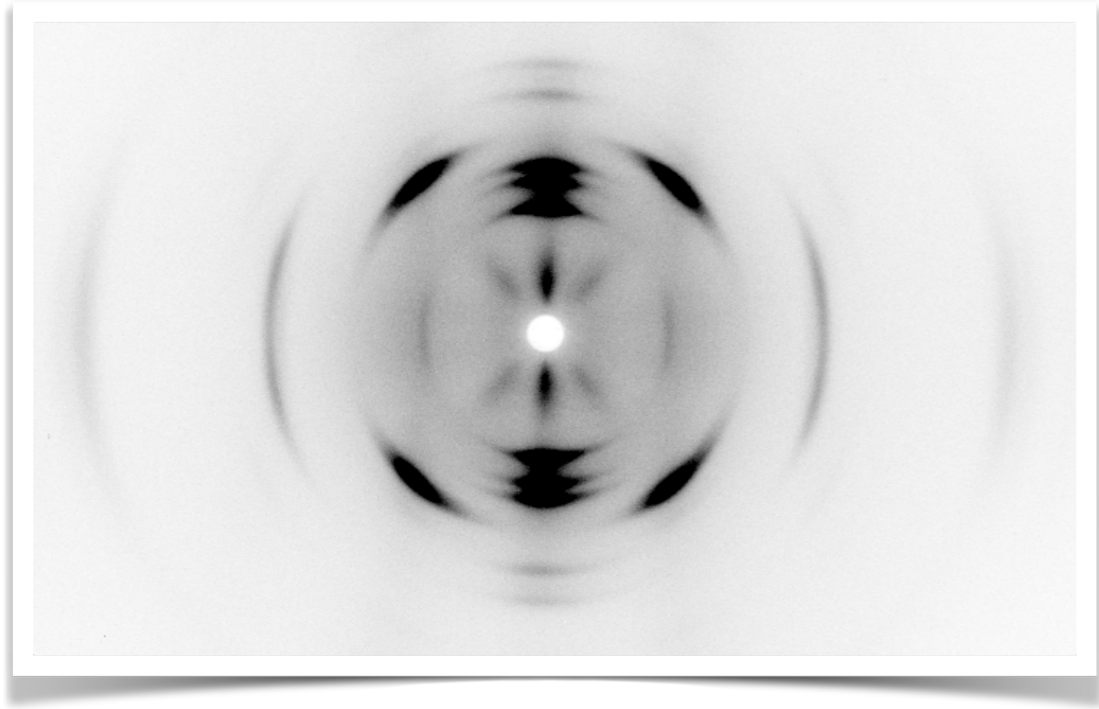


# Crystal structure solution

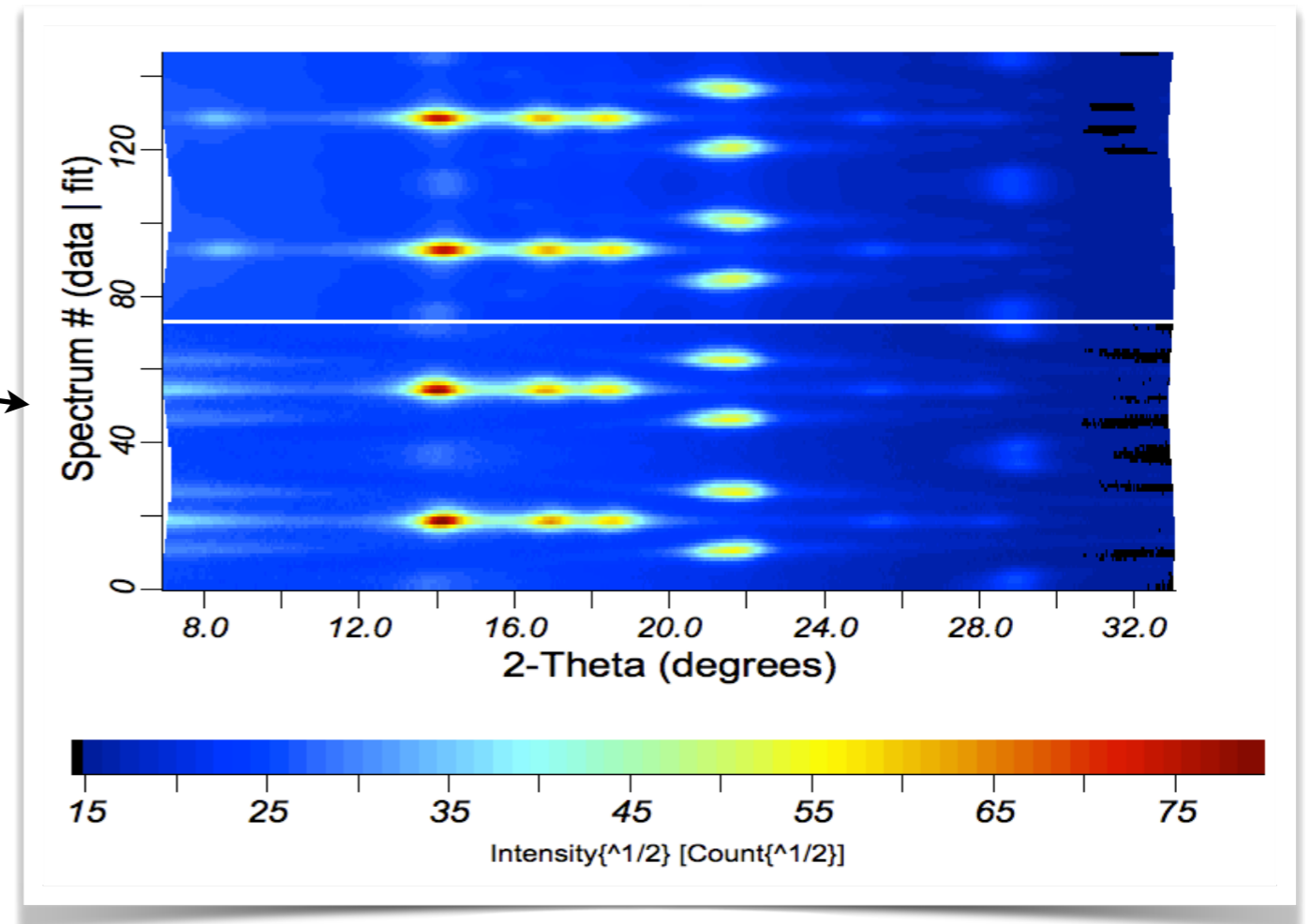
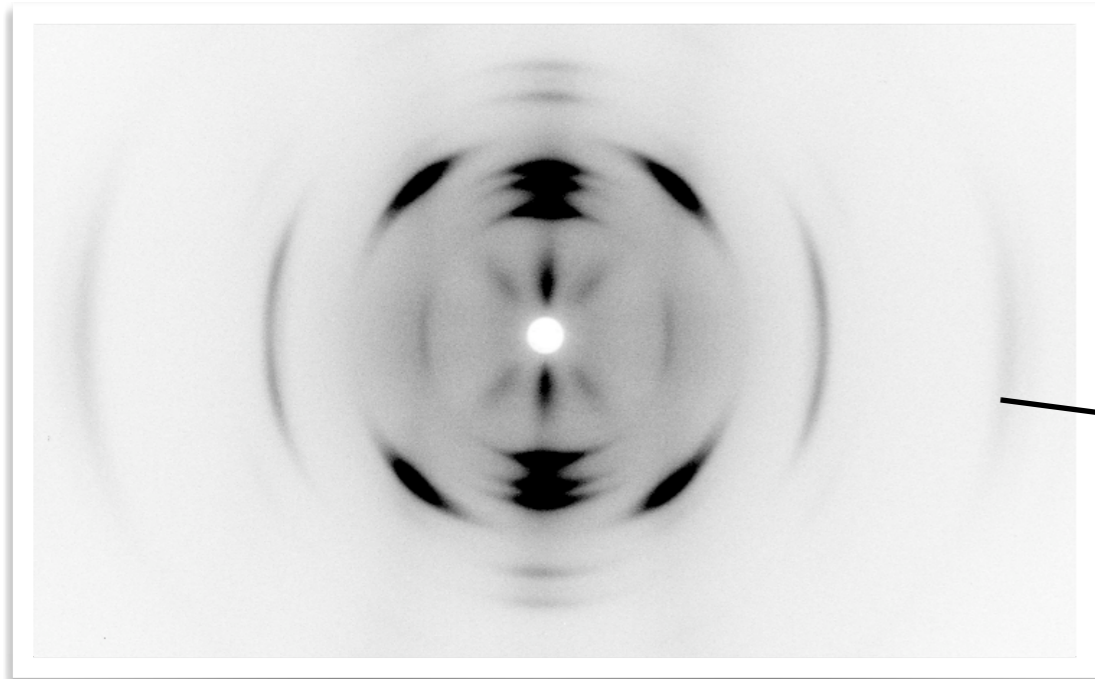
- Standard
- 4 fragments
- Lamark



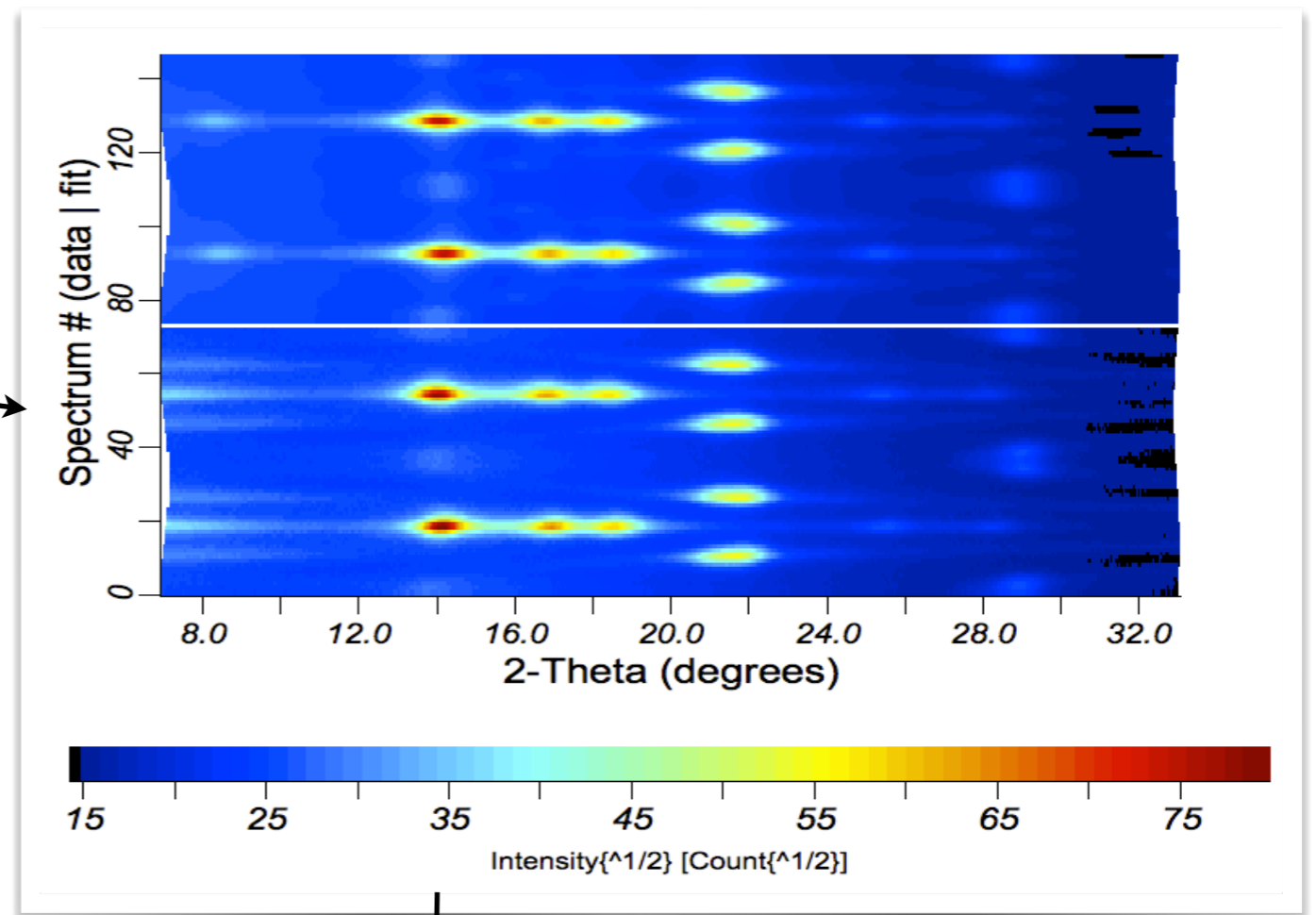
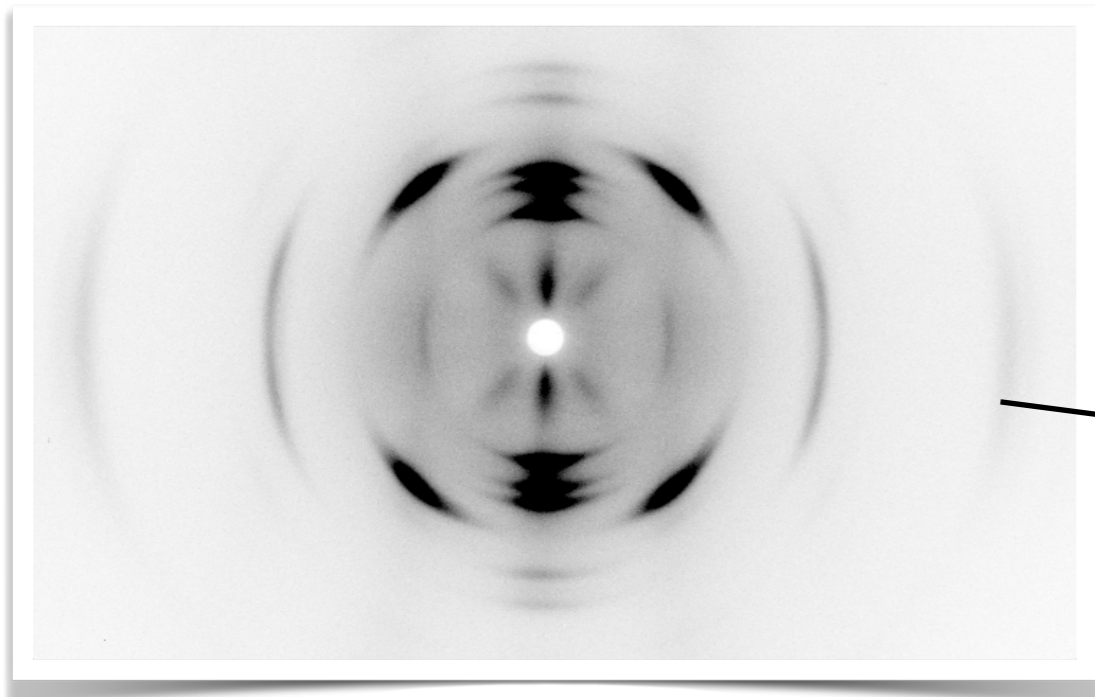
# Isotactic polypropylene:RTA



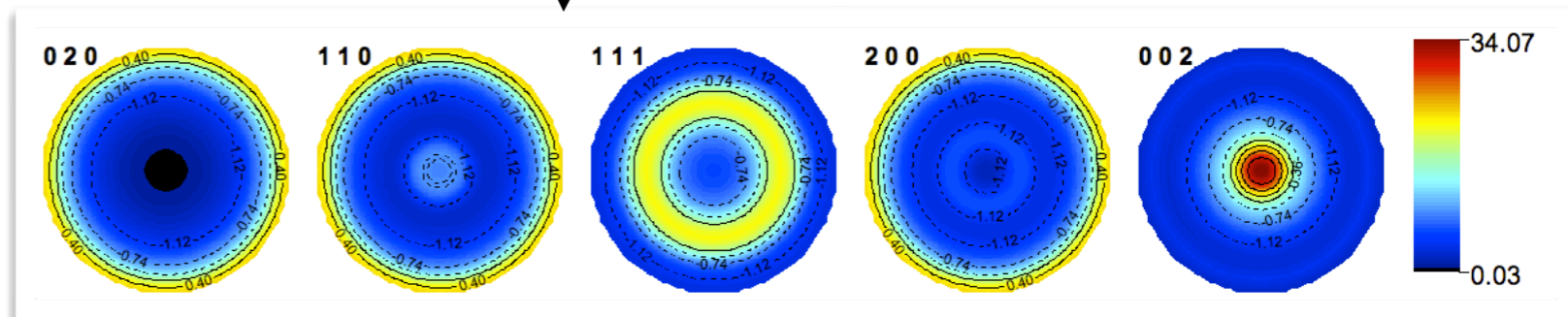
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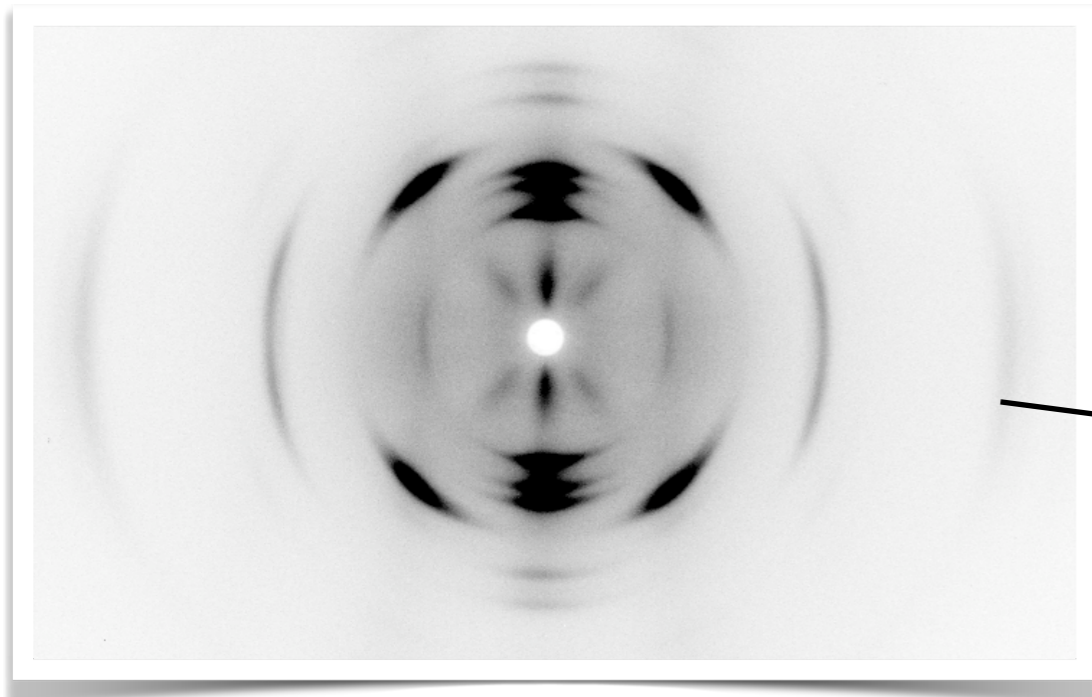
# Isotactic polypropylene:RTA



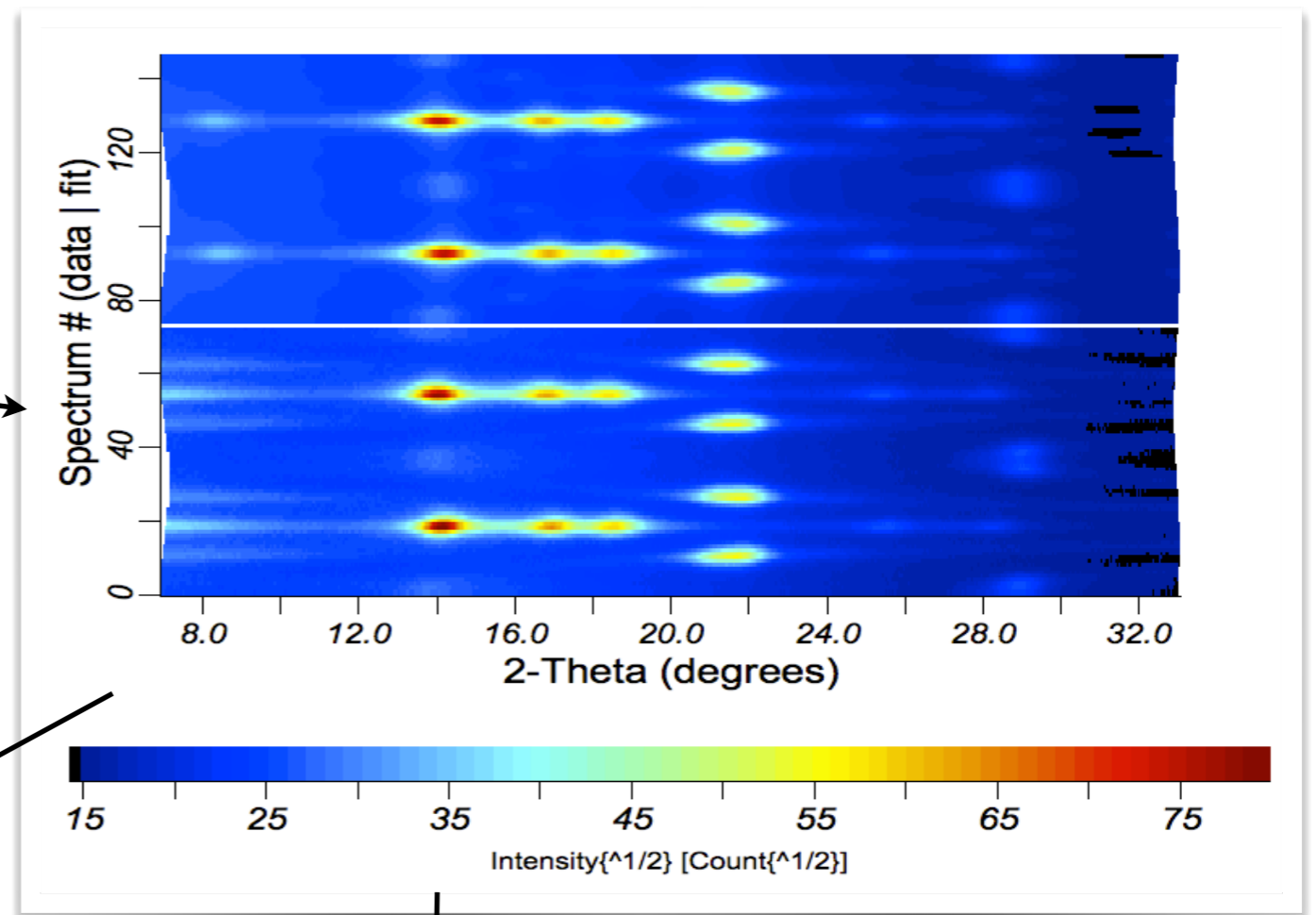
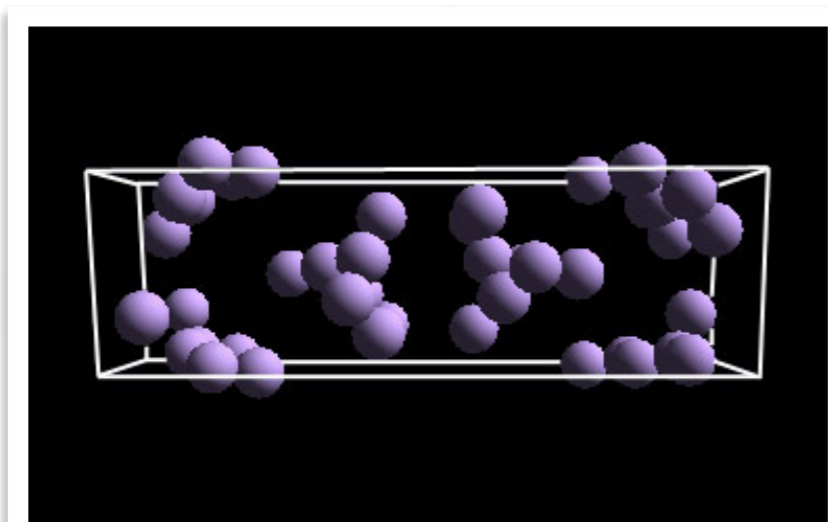
Texture



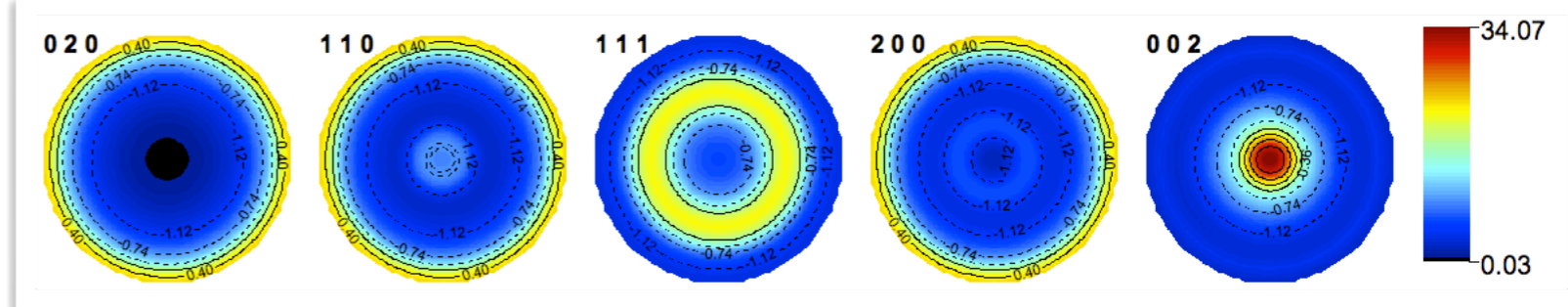
# Isotactic polypropylene:RTA



Structure

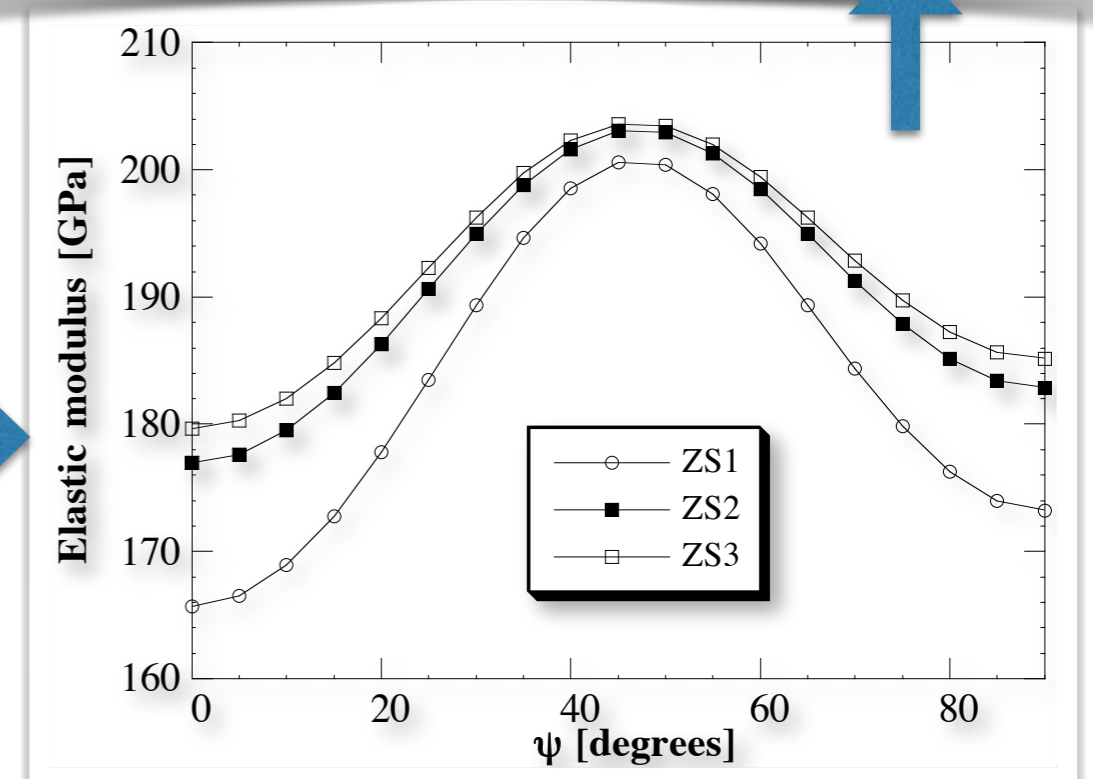
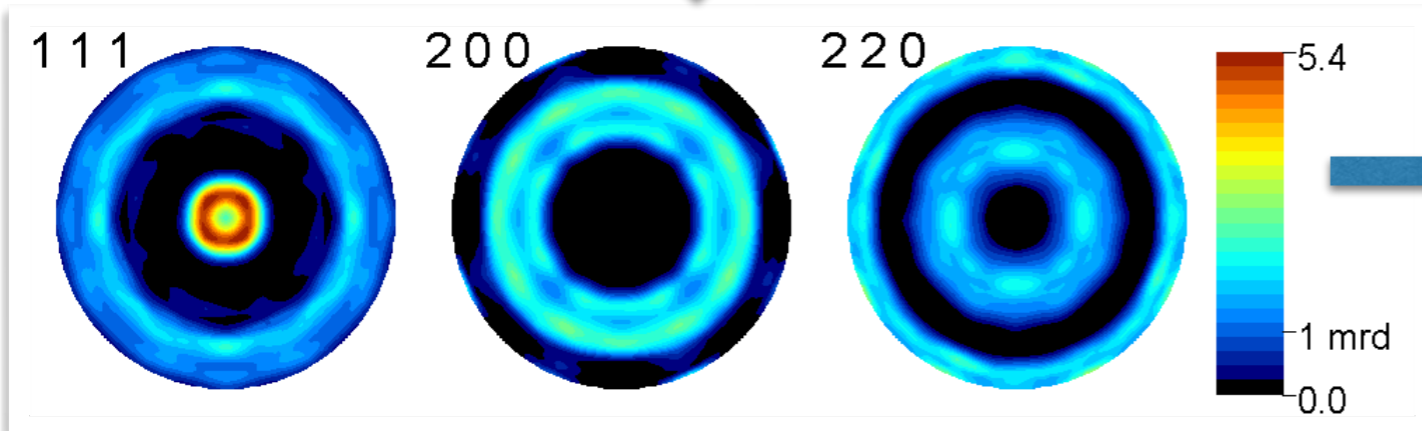
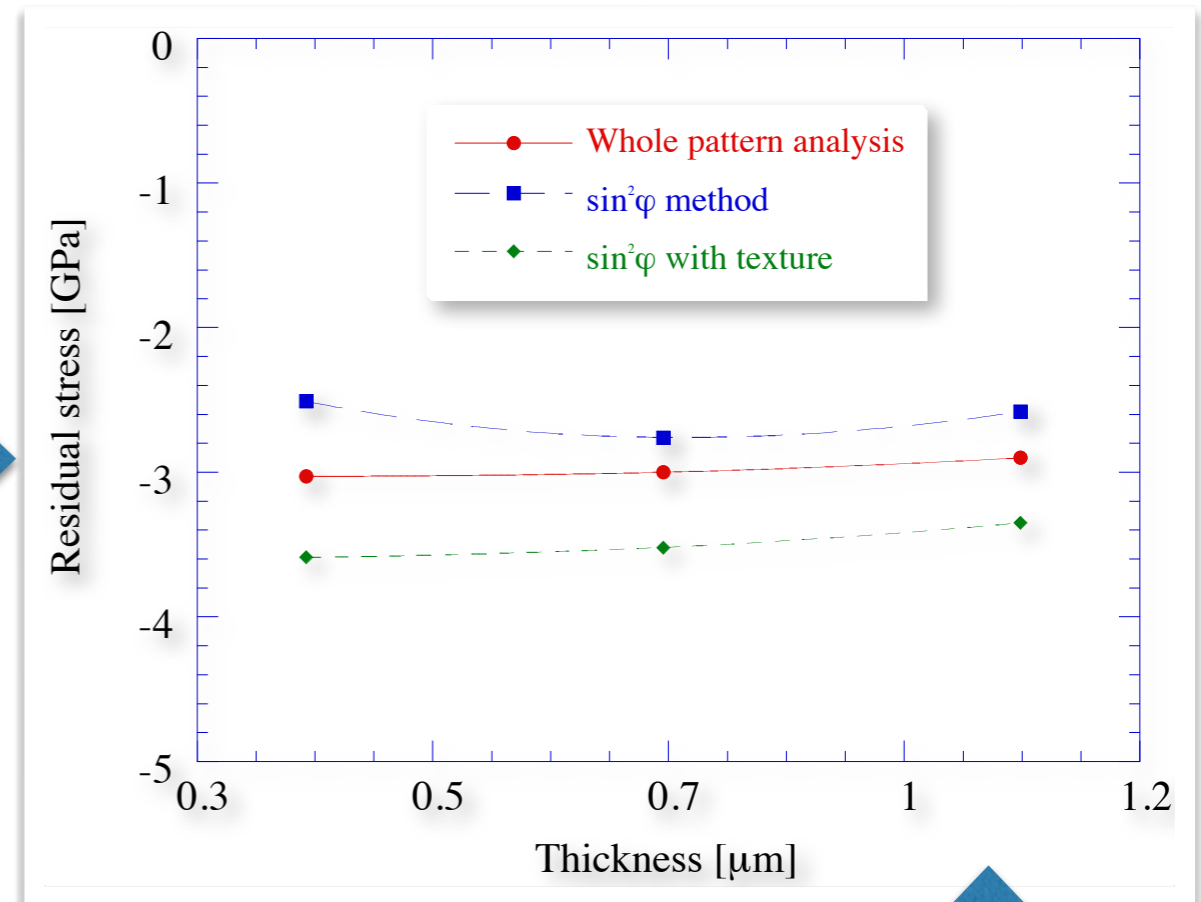
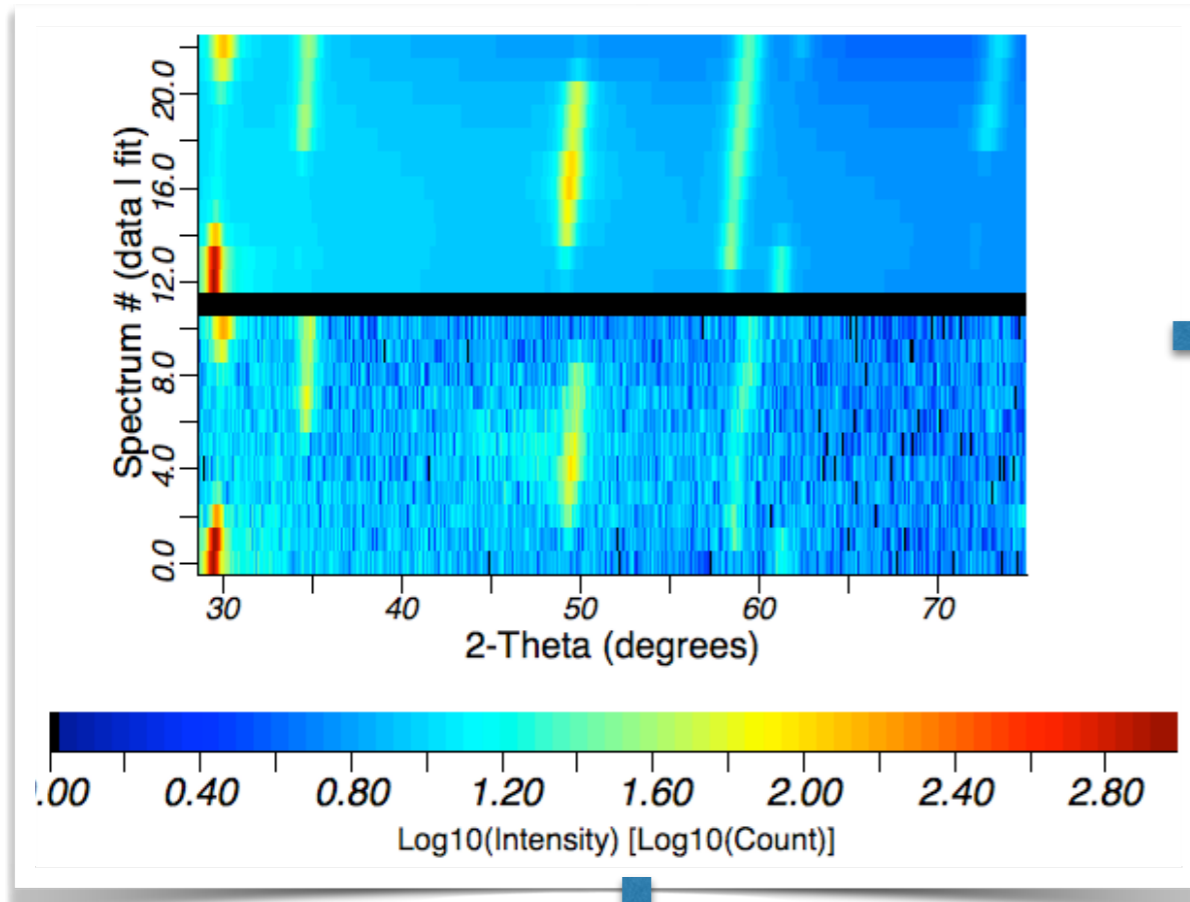


Texture

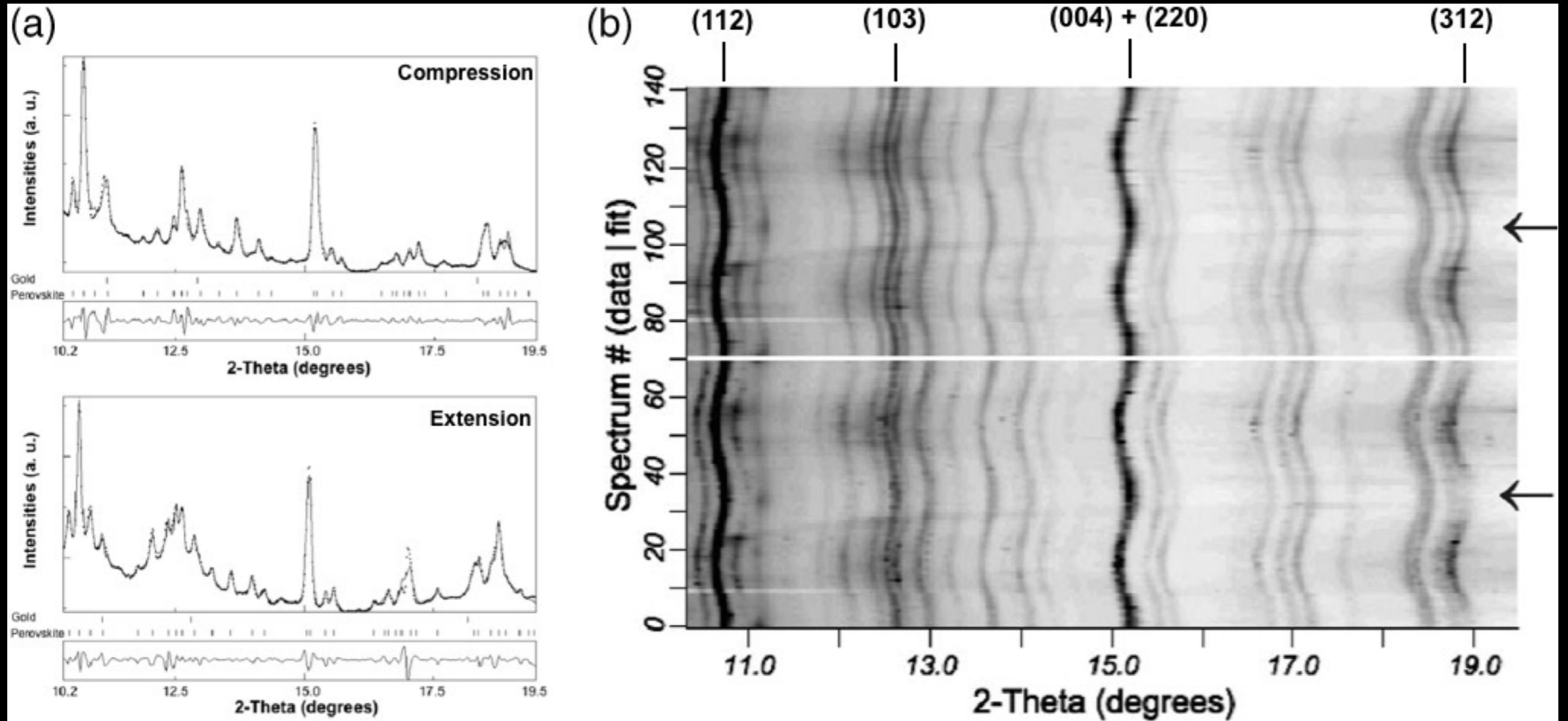


# First experiment: residual stress and texture (1992)

c-ZrO<sub>2</sub> by magnetron sputtering



# MgSiO<sub>3</sub> 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)

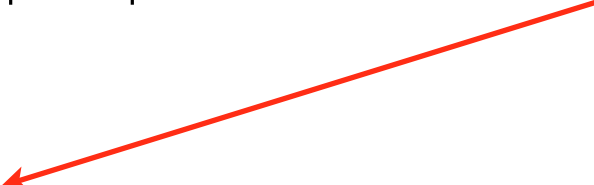
The powder pattern intensity:

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

# Rietveld texture analysis (RTA)

The powder pattern intensity:

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


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

# Rietveld texture analysis (RTA)

The powder pattern intensity:

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

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

Harmonic method:

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

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:

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

# WIMV

The ODF space is discretized in cells:

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

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

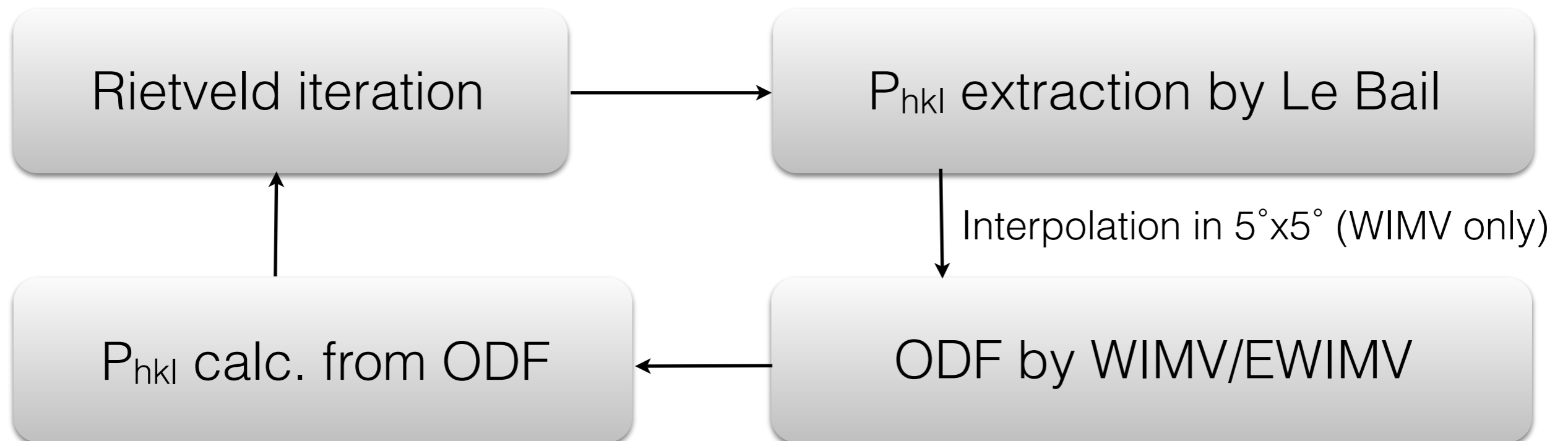
# WIMV

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

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

The Rietveld integration:

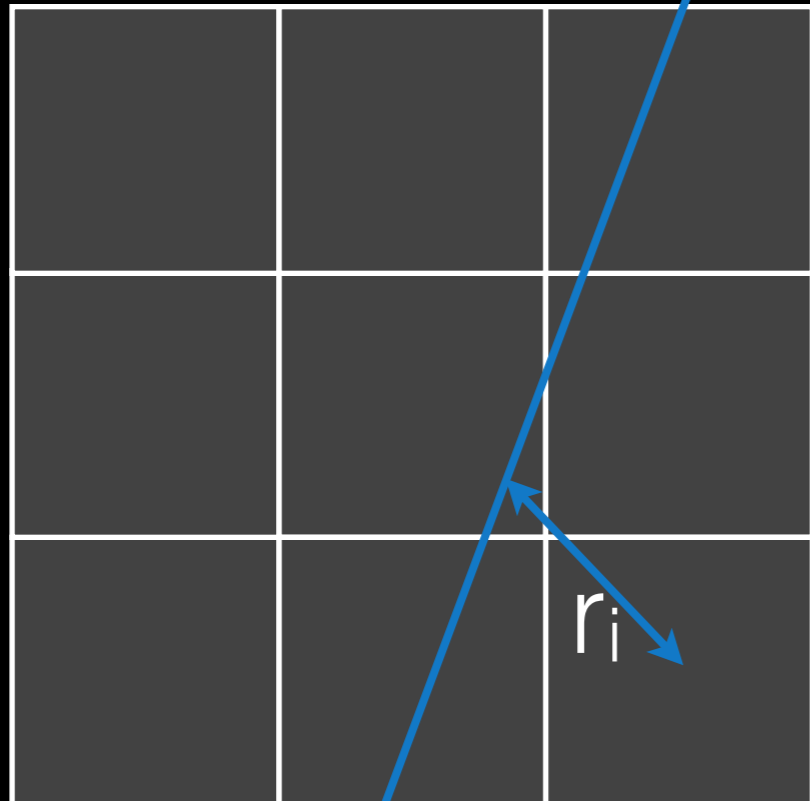


# EWIMV

$$f^{n+1}(g) = f^n(g) \prod_{h=1}^I \prod_{m=1}^{M_h} \left( \frac{P_h^{\text{exp}}(\mathbf{y})}{P_h^{\text{calc},n}(\mathbf{y})} \right)^{r_n \frac{w_h}{IM_h}}$$

$w_h$  = intensity of reflection  $h$

Weighting of neighboring cells



WIMV: uses value of containing cell

EWIMV:

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

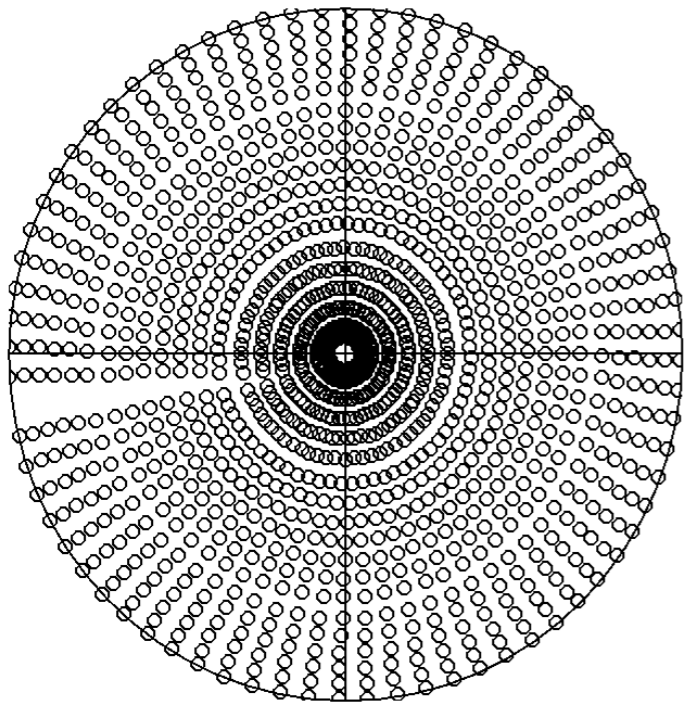
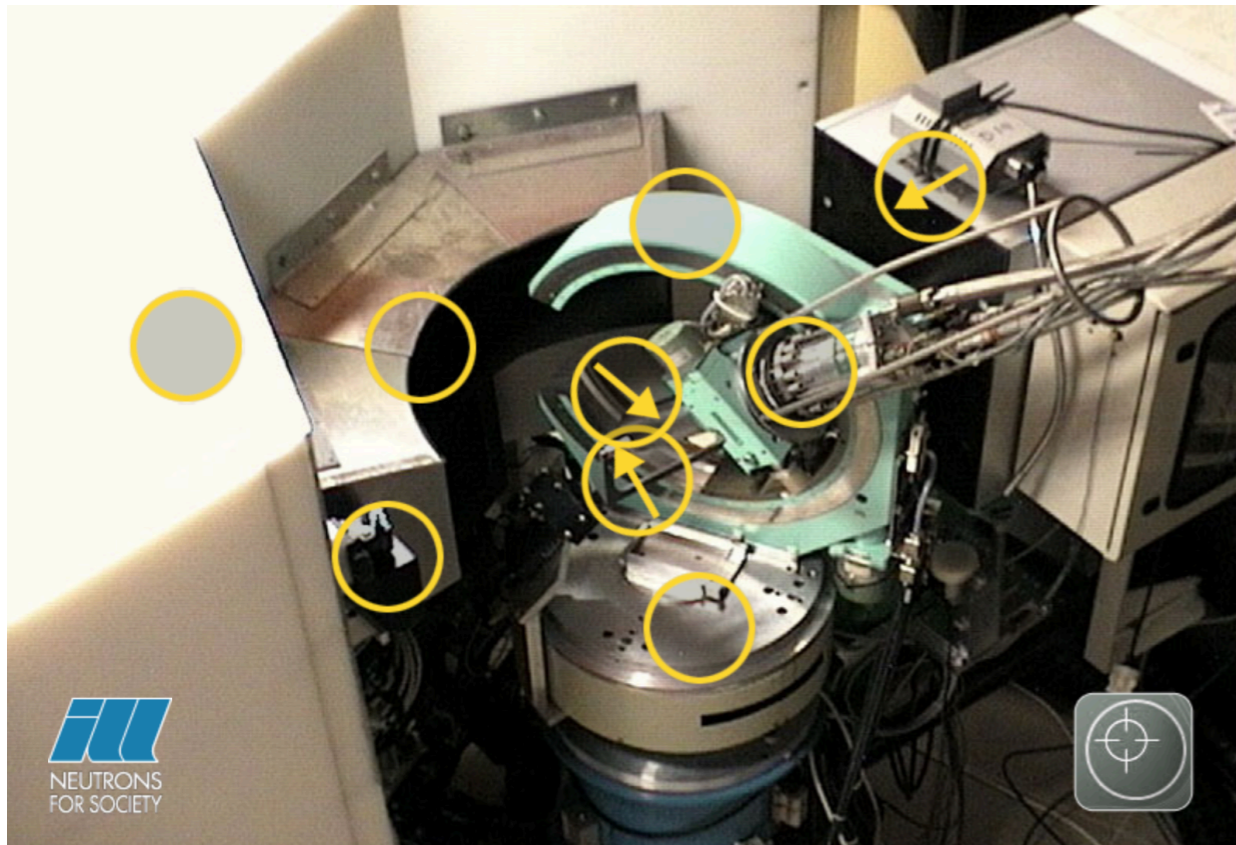
# MTeX in MAUD



- 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.



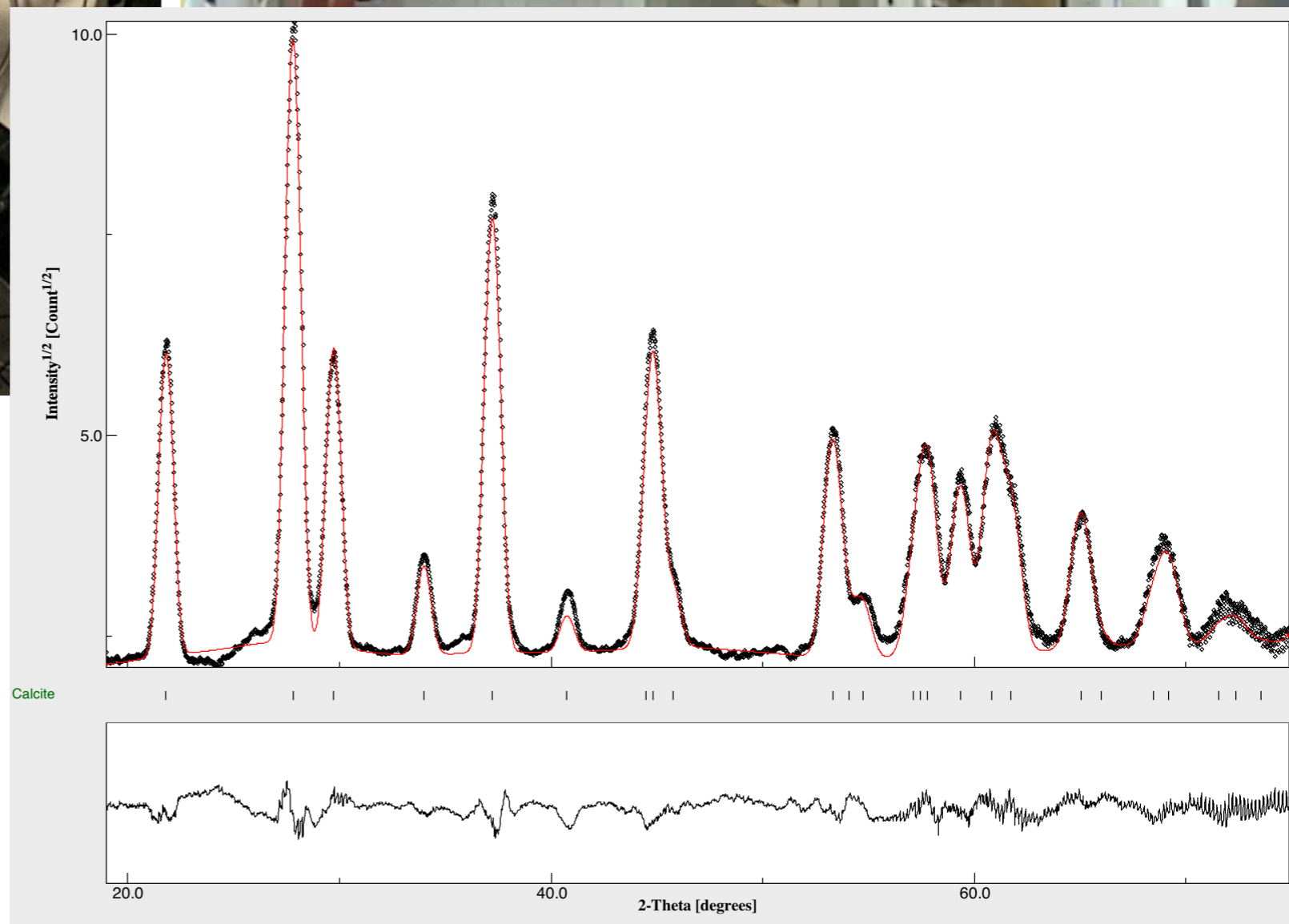
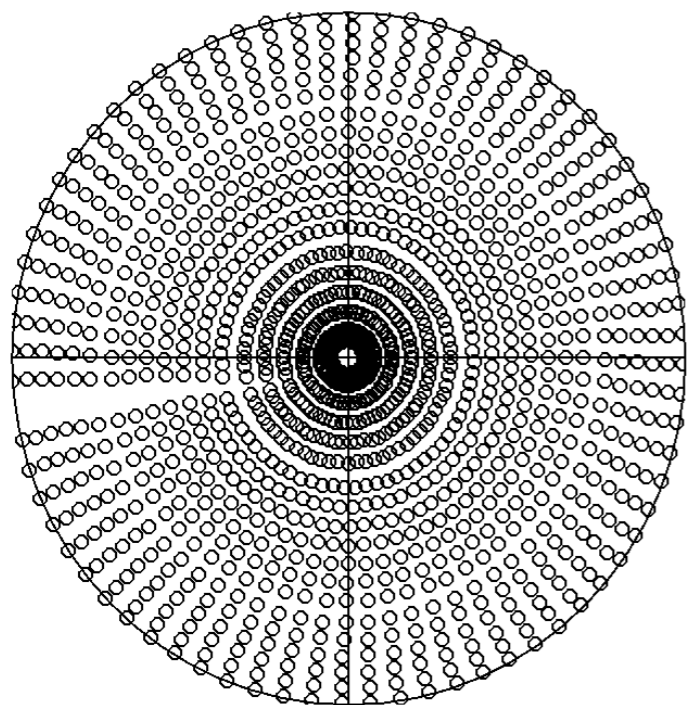
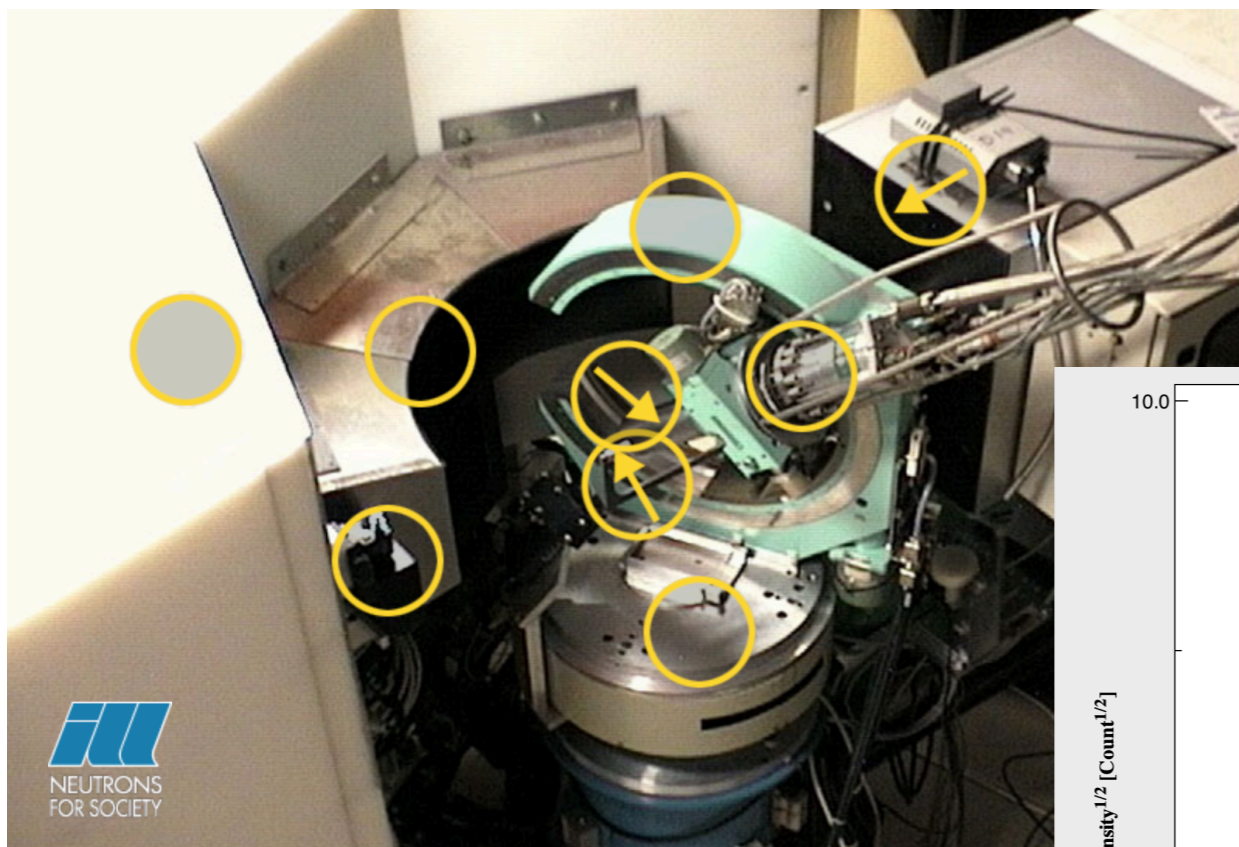
# Calcite @ D19



Experimental coverage for 006

Experiment conducted by Michele Zucali

# Calcite @ D19

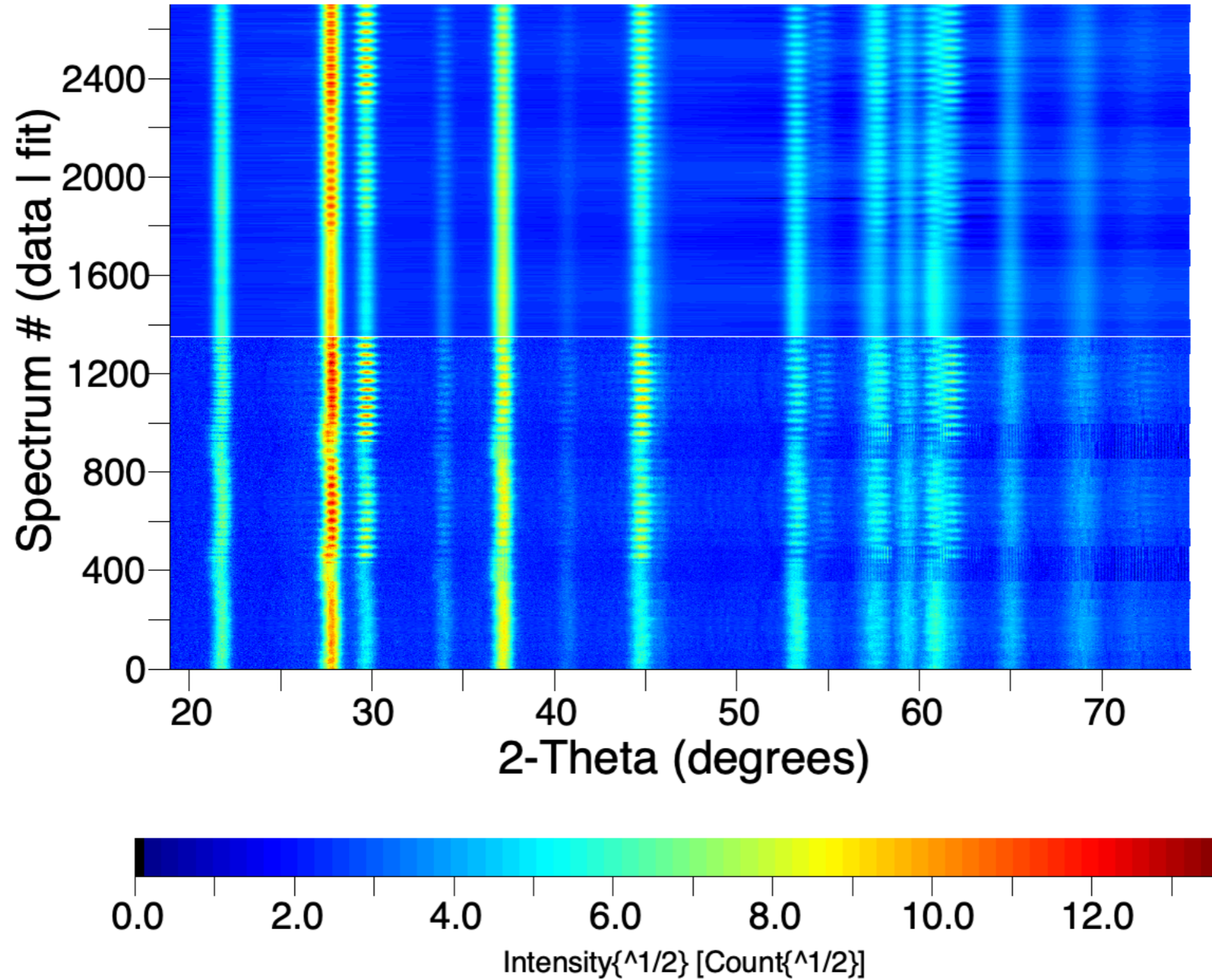


Experimental coverage for 006

Experiment conducted by Michele Zucali

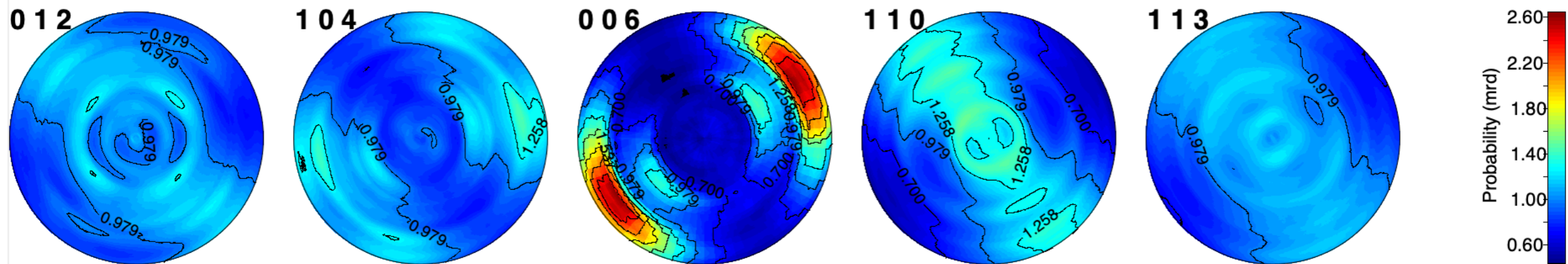
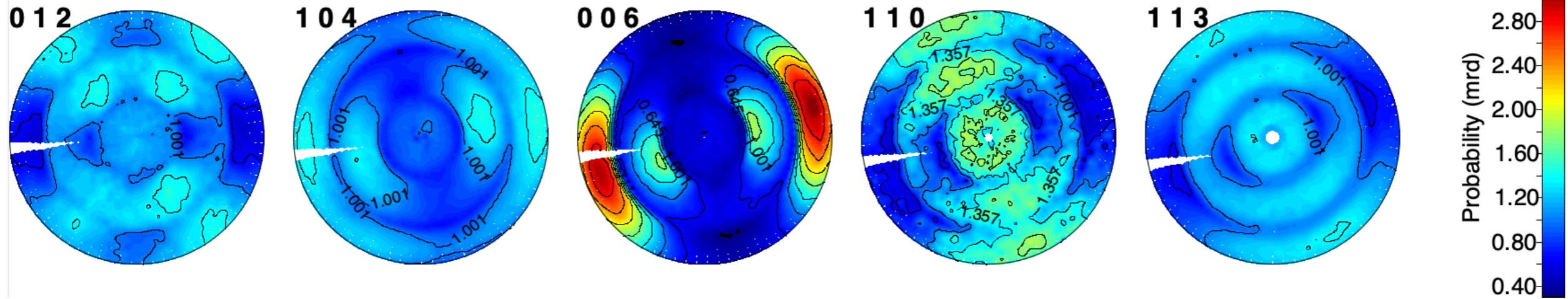
# 2D fitting of calcite patterns

Using EWIMV texture model



# Texture analysis of calcite

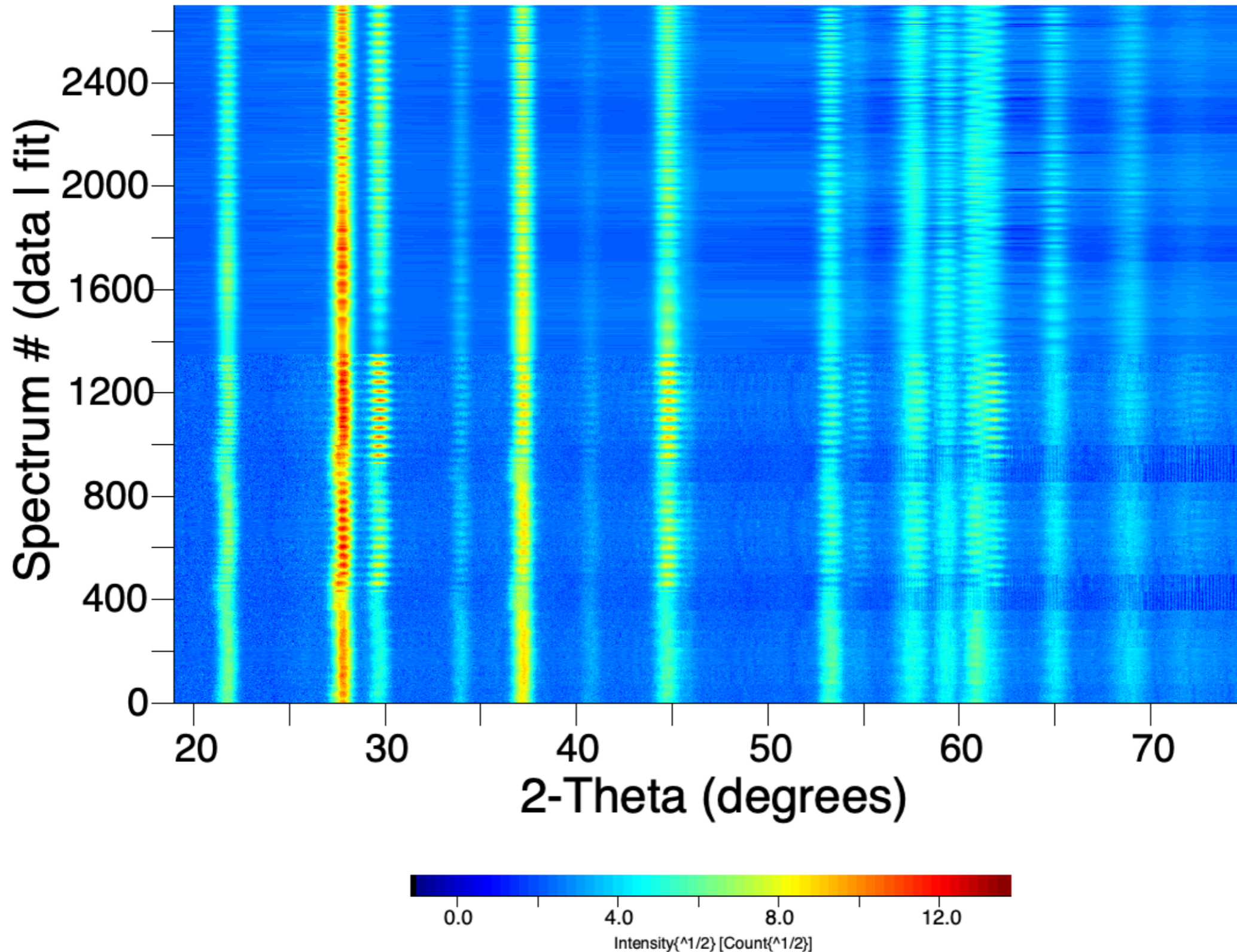
Extracted (experimental) pole figures (some)



Recalculated pole figures (using EWIMV,  $10^\circ$  as texture model)

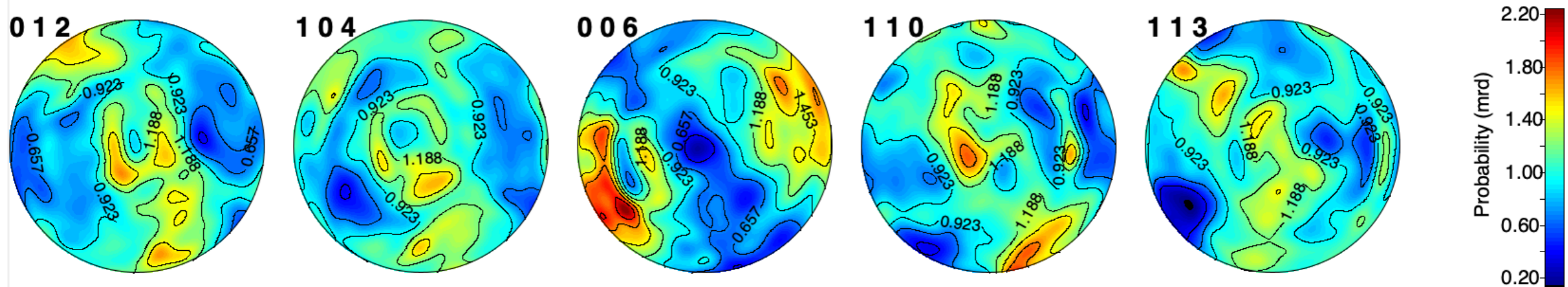
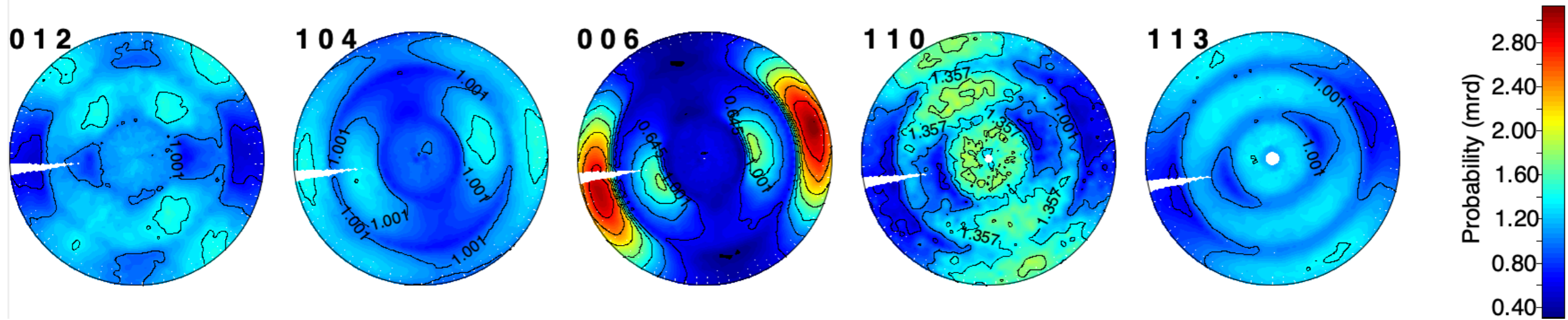
# 2D fitting of calcite patterns

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 screenshot displays the MAUD software interface for a texture analysis. The main window is titled "Maud - CC3a\_d19\_mt看.par".

**MTeX options panel:** This panel is open, showing settings for the MTeX model. Key options include:
 

- ODF resolution in degrees: 6
- Kernel type: DeLaValleePoussin
- ODF refinable
- Min reflex intensity: 0.0
- Minimum reflection d-spacing: 2.0
- Generate symmetry: none

**2D M Texture Plot:** A vertical plot showing the texture data. The y-axis is labeled "(data) 1600".

**Texture Plots:** A grid of six circular texture plots for different Miller indices:
 

- {0112}: Max: 1.71, Min: 0.37
- {1014}: Max: 1.63, Min: 0.37
- {0006}: Max: 2.24, Min: 0.20
- {1120}: Max: 1.92, Min: 1.92
- {1123}: Max: 1.70, Min: 0.13
- {2022}: Max: 1.80, Min: 0.33

**Advanced models panel:** This panel is also open, showing settings for the texture/stress/magnetic model:
 

- Texture: MTeX model
- Strain: no strain
- Magnetic: no magnetic
- Structure factors: Optional scale factor: 1.0
- Structure Factor model: atomic standard model
- Structure Factor extractor: Le Bail
- Scattering Models: TDS model: None TDS

**Intensity Scale:** A color scale for intensity, ranging from 40 to 70 degrees 2-Theta (degrees) and 8.0 to 12.0 intensity{^1/2} [Count{^1/2}].

**Parameter List:** A table at the bottom shows the status of various parameters:

Parameter	Min	Max	Status	Output
_pd_spec_orientation_omega	0	0.0	****	false
_pd_spec_orientation_chi	0	0.0	****	false
_pd_spec_orientation_phi	0	0.0	Fixed	false
_riet_par_spec_displac_x	0	0.0	Fixed	false
_riet_par_spec_displac_y	0	0.0	Fixed	false





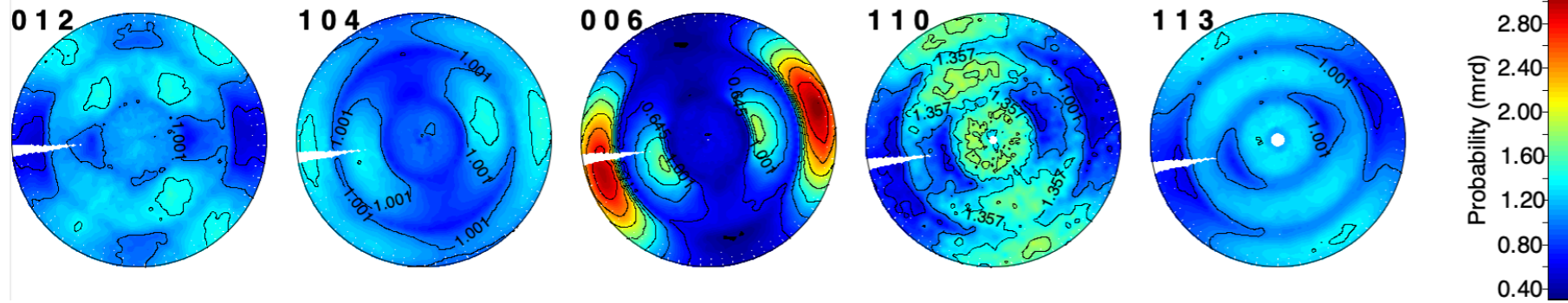
# The model in MAUD

The screenshot displays the MAUD software interface with the following components:

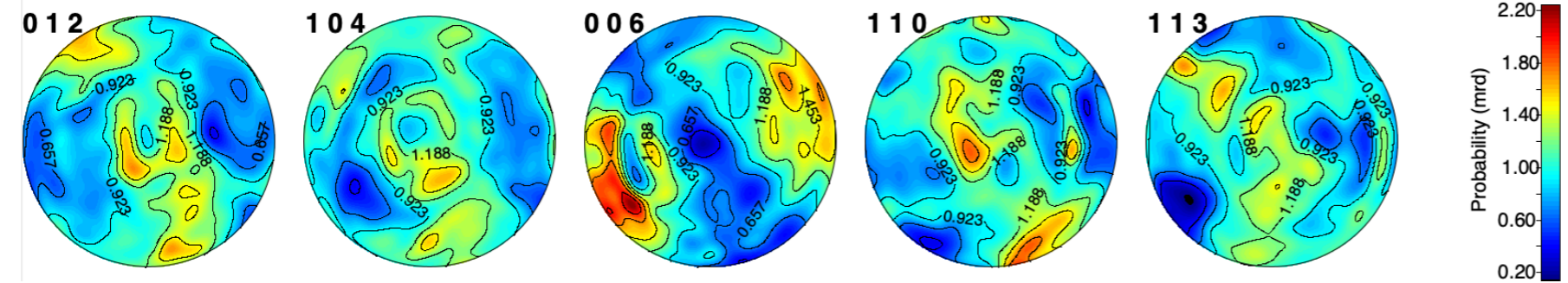
- MTex options panel:** A dialog box for configuring texture analysis. It includes:
  - ODF resolution in degrees: 6
  - Kernel type: DeLaValleePoussin
  - ODF refinable
  - Min reflex intensity: 0.0
  - Minimum reflection d-spacing: 2.0
  - Buttons: Plot PFs, Plot exp PFs, Plot PFs diff, Plot ODF (sigma), Plot ODF (phi2full), Plot ODF (phi2), Plot ODF (phi1)
  - Generate symmetry: none
  - Buttons: Cancel, OK
- 2D M texture plot:** A vertical plot showing intensity as a function of 2-Theta (degrees) and intensity<sup>1/2</sup> [Count<sup>1/2</sup>]. The x-axis ranges from 40 to 70 degrees, and the y-axis ranges from 8.0 to 12.0. A color scale at the bottom indicates intensity values from blue (low) to red (high).
- Pole Figures (PFs):** A grid of six circular plots for different hkl planes:
  - {0112}: Max: 1.71, Min: 0.37
  - {1014}: Max: 1.63, Min: 0.37
  - {0006}: Max: 2.24, Min: 0.20
  - {1120}: Max: 1.92, Min: 1.92
  - {1123}: Max: 1.70, Min: 0.13
  - {2022}: Max: 1.80, Min: 0.33
- Advanced models panel:** A sub-panel on the right with tabs for General, Structure, Microstructure, and Advanced models. It includes:
  - Texture: MTex model
  - Strain: no strain
  - Magnetic: no magnetic
  - Structure factors: Optional scale factor: 1.0
  - Structure Factor model: atomic standard model
  - Structure Factor extractor: Le Bail
  - Scattering Models: TDS model: None TDS
  - Custom peaks list: Peak list editing, Remove peak list
- Refinement Summary:** Located in the bottom left, it shows:
  - Start rita/rista refinement
  - Wss = 4027347.8
  - Number of iterations : 3
  - 4.487421 0.257748
- Parameter List:** A table at the bottom showing the status of various parameters:
 

Parameter	Min	Max	Status	Output
_pd_spec_orientation_omega	0	0.0	****	false
_pd_spec_orientation_chi	0	0.0	****	false
_pd_spec_orientation_phi	0	0.0	Fixed	false
_riet_par_spec_displac_x	0	0.0	Fixed	false
_riet_par_spec_displac_y	0	0.0	Fixed	false

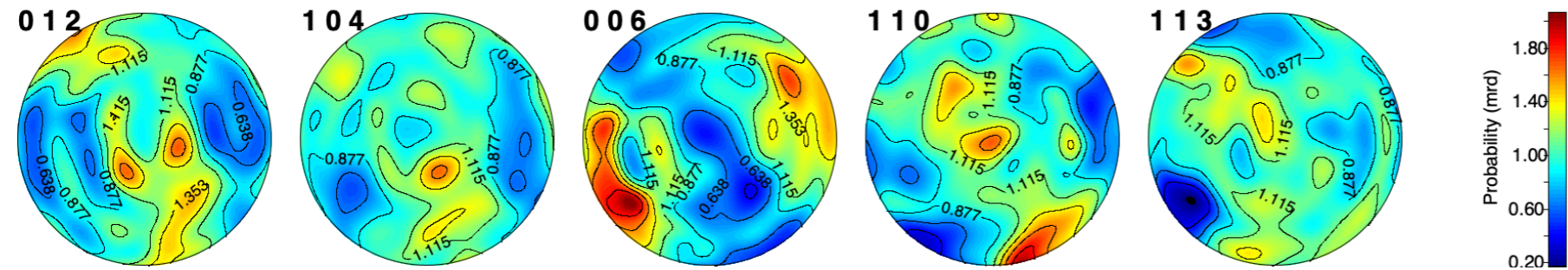
# Testing different options in MTEX



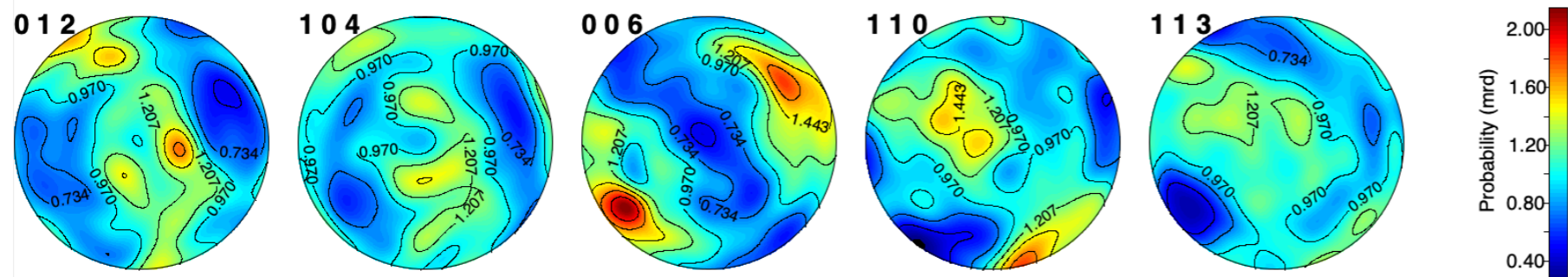
Extracted (experimental)  
pole figures (some)



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

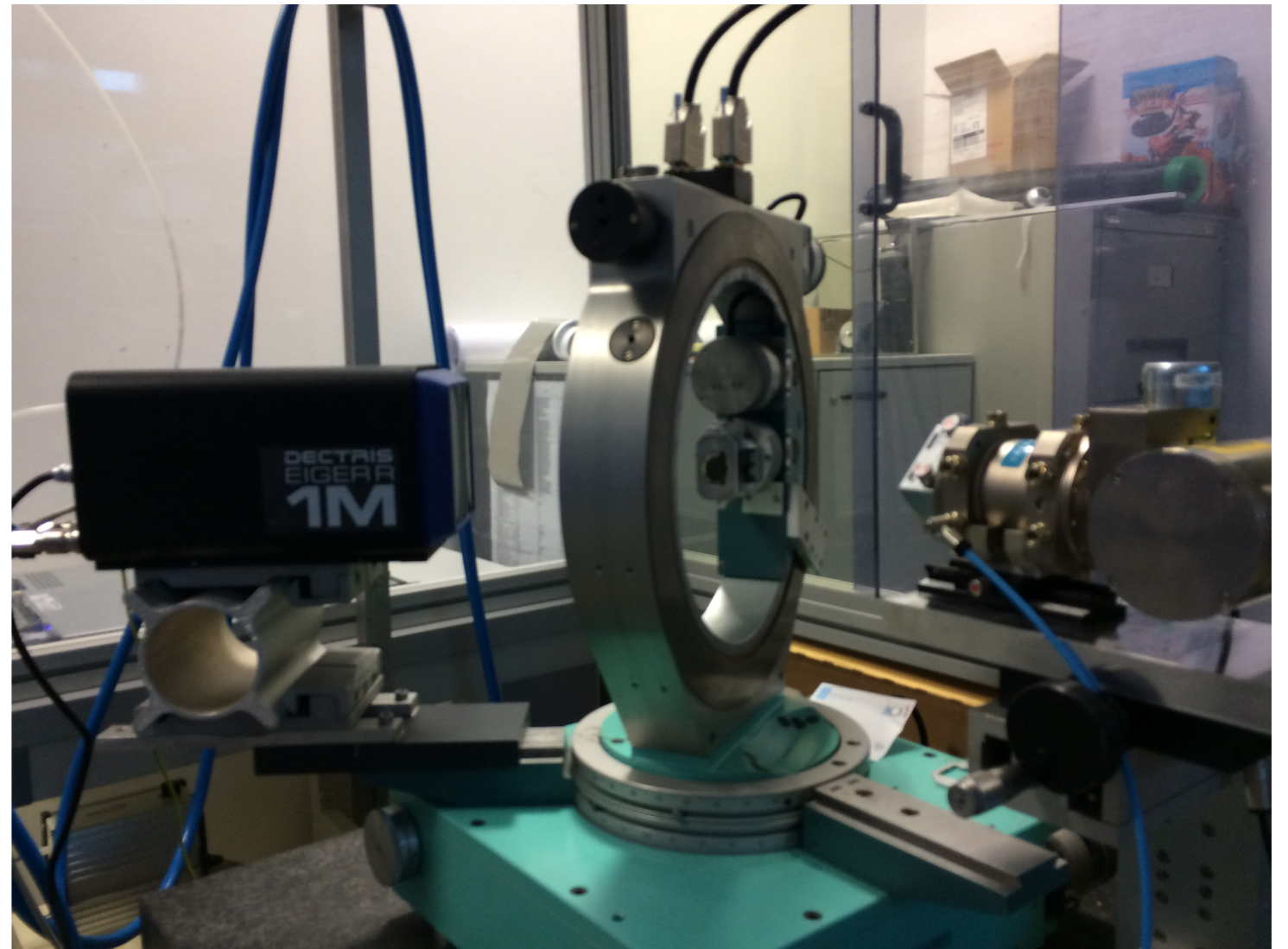


MTEX (8°, Von Mises),  
reflection cut at 1.7  
Angstrom



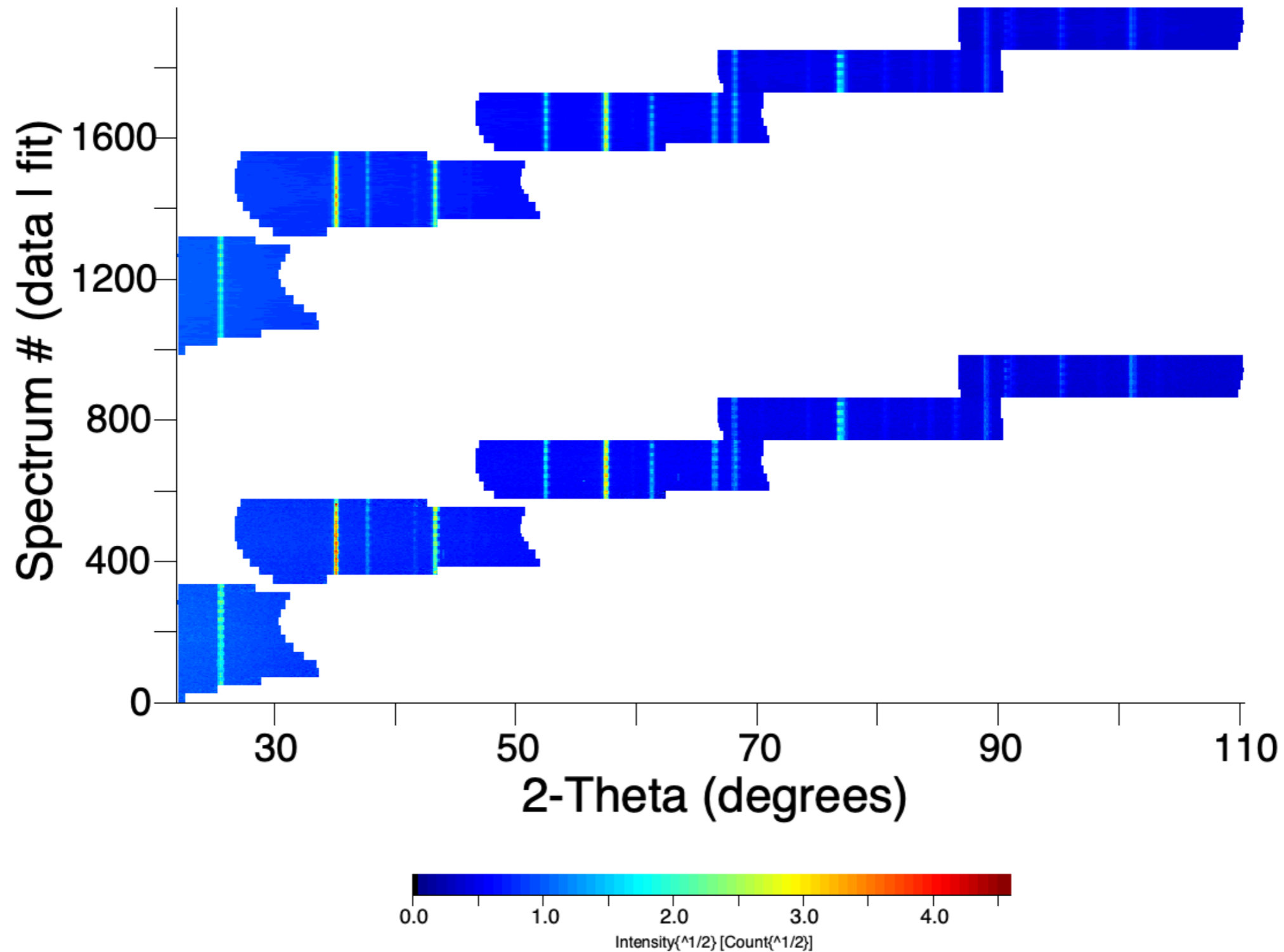
MTEX (10°, Von Mises),  
reflection cut at 1.5  
Angstrom

NIST 1976a  
standard  
(Corundum plate,  
fiber texture)

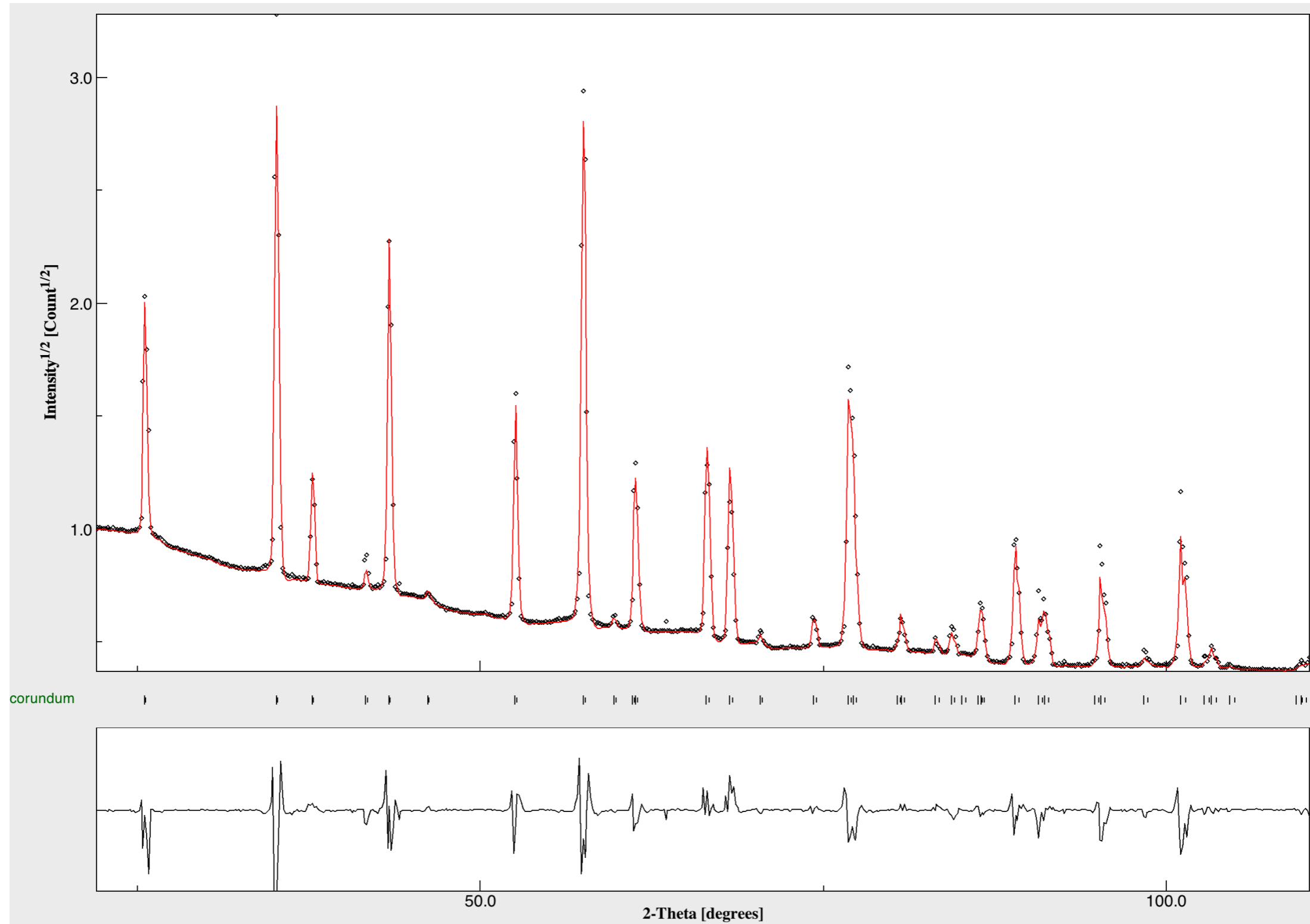


- Data measured in Lab (Trento)
- Eulerian goniometer, high brilliance micro source and Dectris 1M 2D detector
- 2D images permit faster collection of texture data

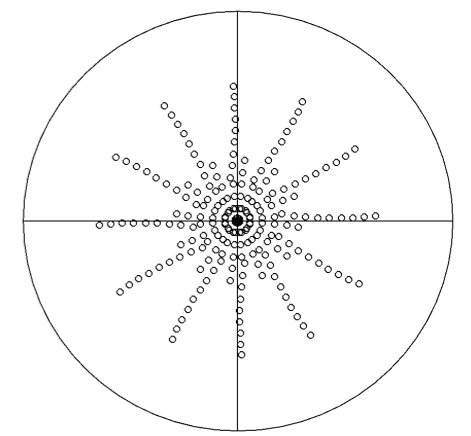
# 2D fitting (EWIMV)



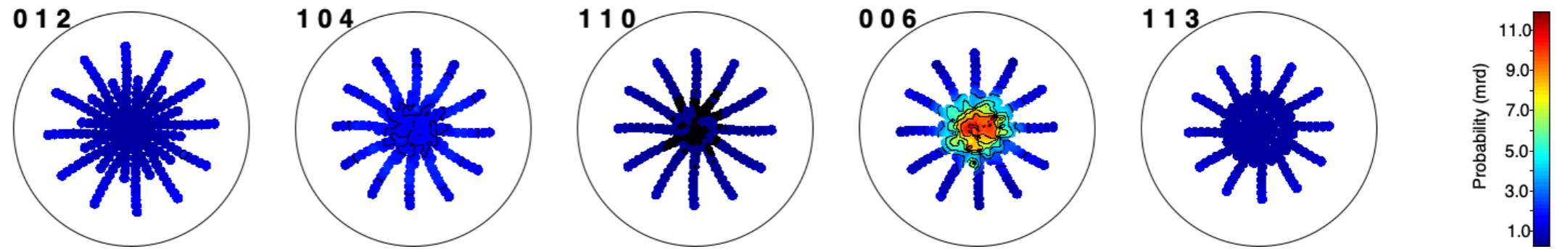
# 2D fitting (EWIMV)



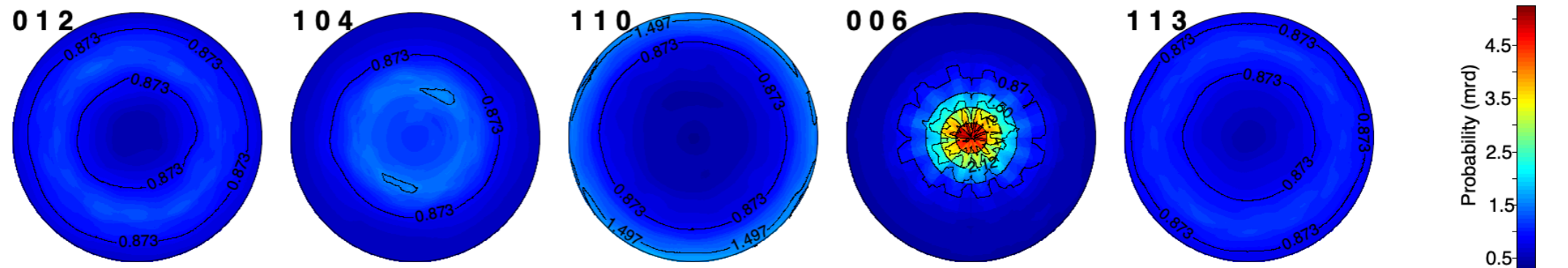
# Texture analysis and comparison



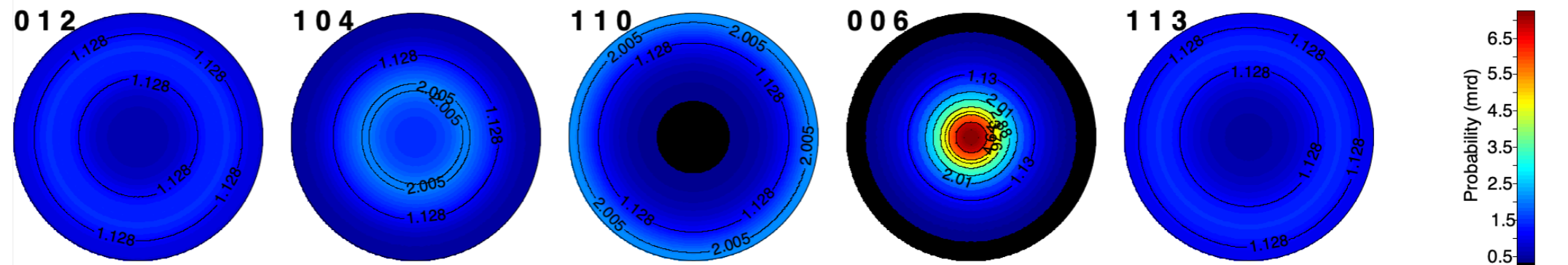
Extracted



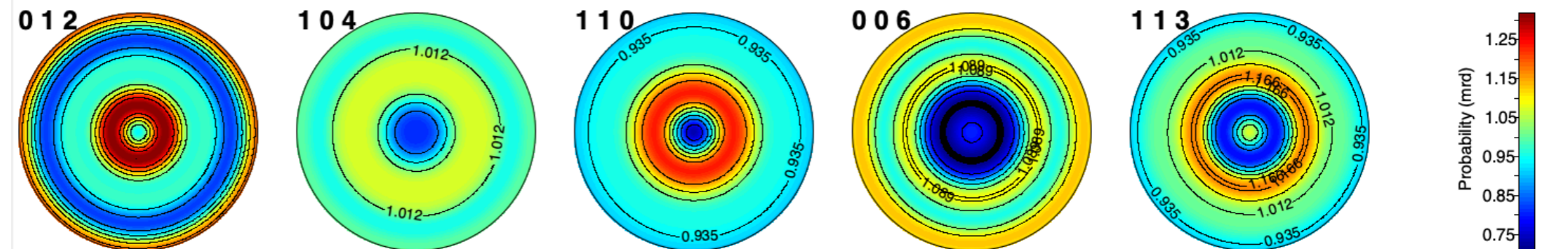
EWIMV



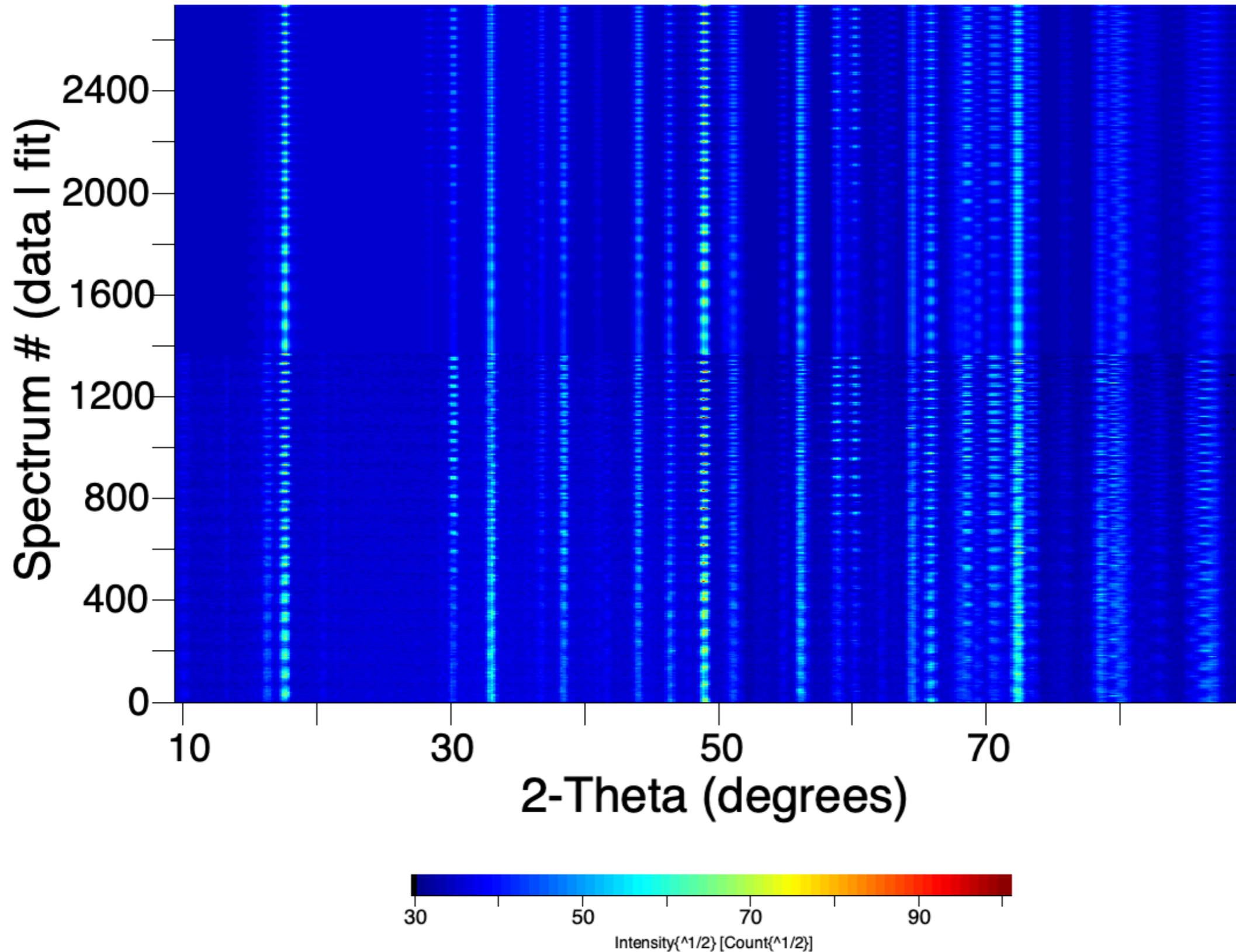
Exponential harmonics



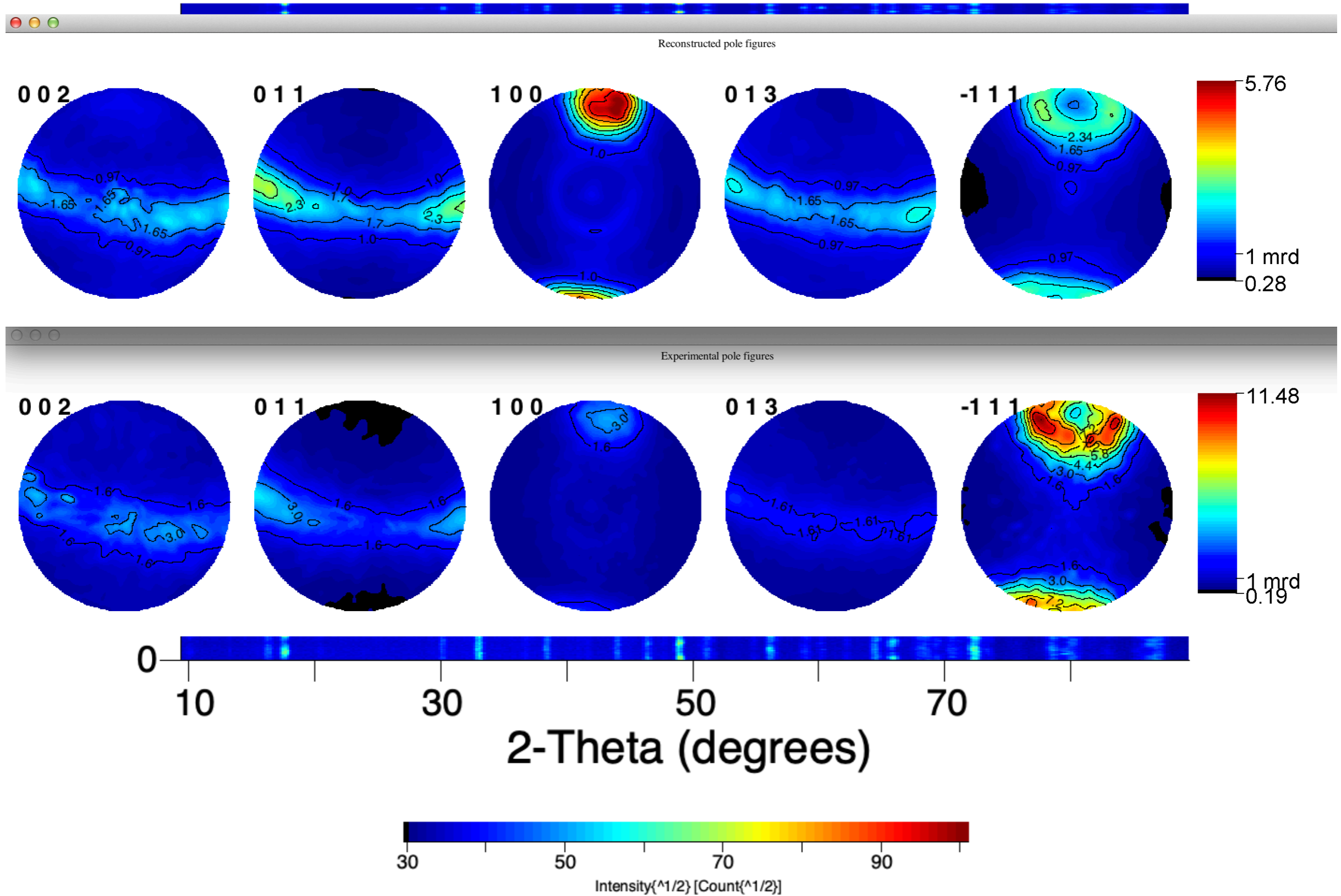
MTEX



# 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)