Time Of Flight (TOF) Neutron Diffraction

Luca Lutterotti

UNIVERSITY OF TRENTO - Italy

Department of Industrial Engineering

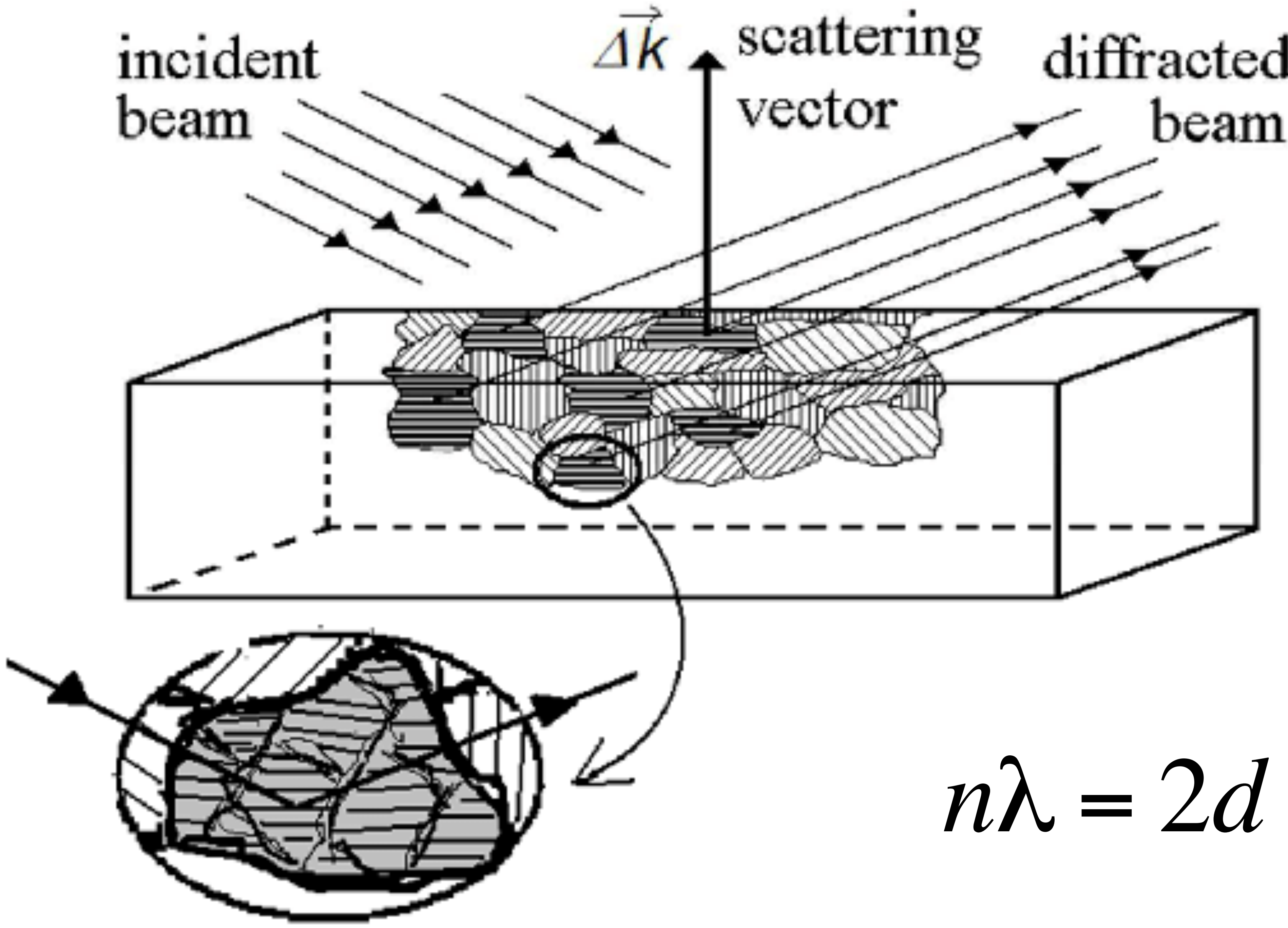
Time Of Flight (TOF) Neutron Diffraction

Luca Lutterotti

UNIVERSITY OF TRENTO - Italy

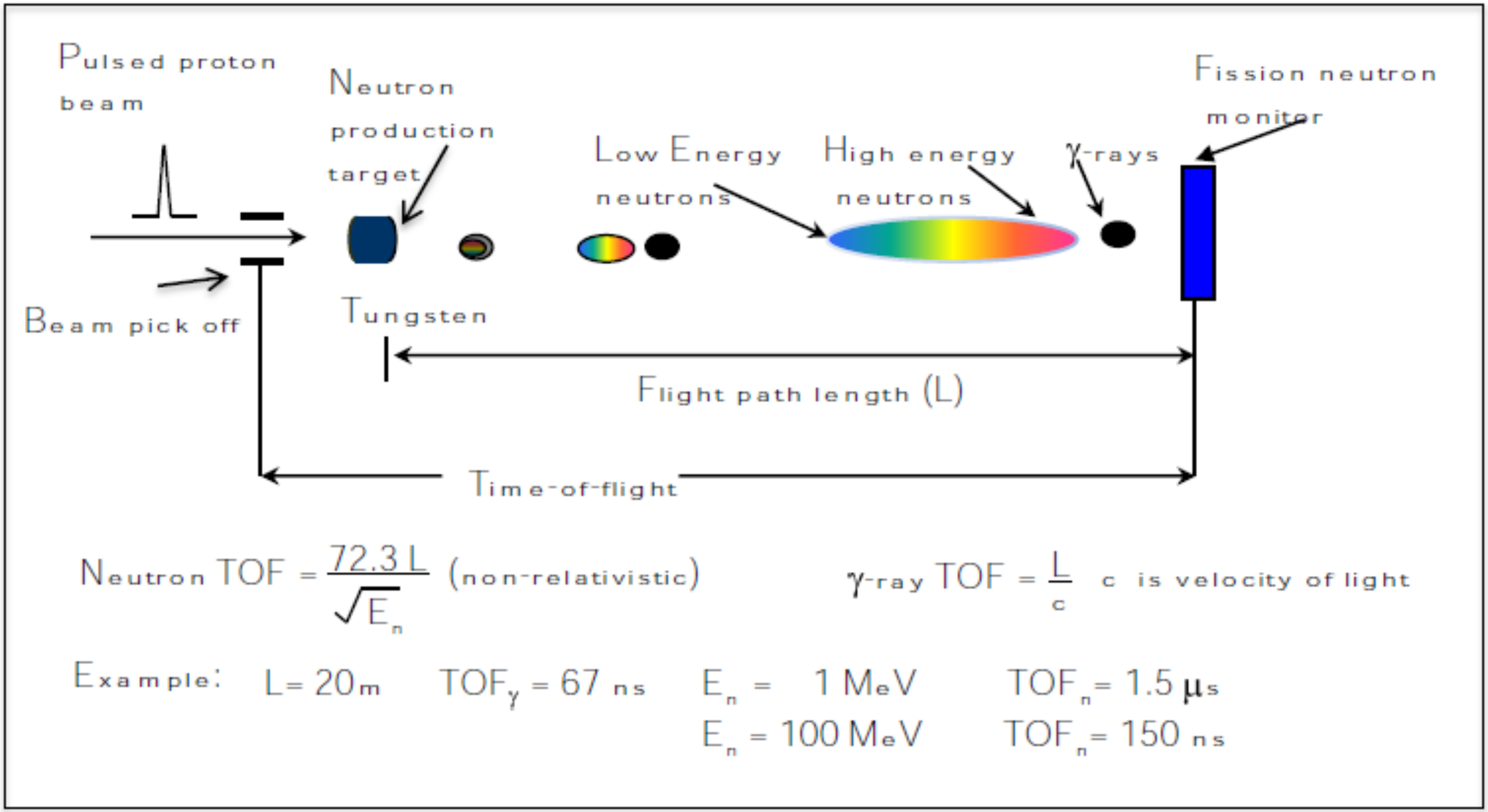
Department of Industrial Engineering

Powder diffraction



$$n\lambda = 2d\sin\theta$$

Neutron TOF: a pulsed source



TOF to d-spacing

From GSAS manual

$$T_{ph} = \mathrm{DIFC}\ d_p + \mathrm{DIFA}\ d^2{}_p + \mathrm{ZERO}$$

Time Of Flight

d-spacing

TOF to d-spacing

From GSAS manual

$$T_{ph} = DIFC\ d_p + DIFA\ d^2_p + ZERO$$

Time Of Flight

d-spacing

$$DIFC = 252.816 \cdot 2 \sin\Theta \left(L_1 + \sqrt{L_2^2 + L_3^2/16} \right)$$

TOF to d-spacing

From GSAS manual

$$T_{ph} = DIFC\ d_p + DIFA\ d^2_p + ZERO$$

Time Of Flight

d-spacing

θ angle of the bank/detector

$$DIFC = 252.816 \cdot 2 \sin\Theta \left(L_1 + \sqrt{L_2^2 + L_3^2 / 16} \right)$$

Neutron mass / Planck constant

TOF to d-spacing

From GSAS manual

$$T_{ph} = DIFC\ d_p + DIFA\ d^2{}_p + ZERO$$

Time Of Flight

d-spacing

From sample to bank

θ angle of the bank/detector

$$DIFC = 252.816 \cdot 2 \sin \Theta \left(L_1 + \sqrt{L_2^2 + L_3^2 / 16} \right)$$

Neutron mass / Planck constant

Primary path length

TOF to d-spacing

From GSAS manual

$$T_{ph} = DIFC\ d_p + DIFA\ d^2_p + ZERO$$

Time Of Flight

d-spacing

From sample to bank

θ angle of the bank/detector

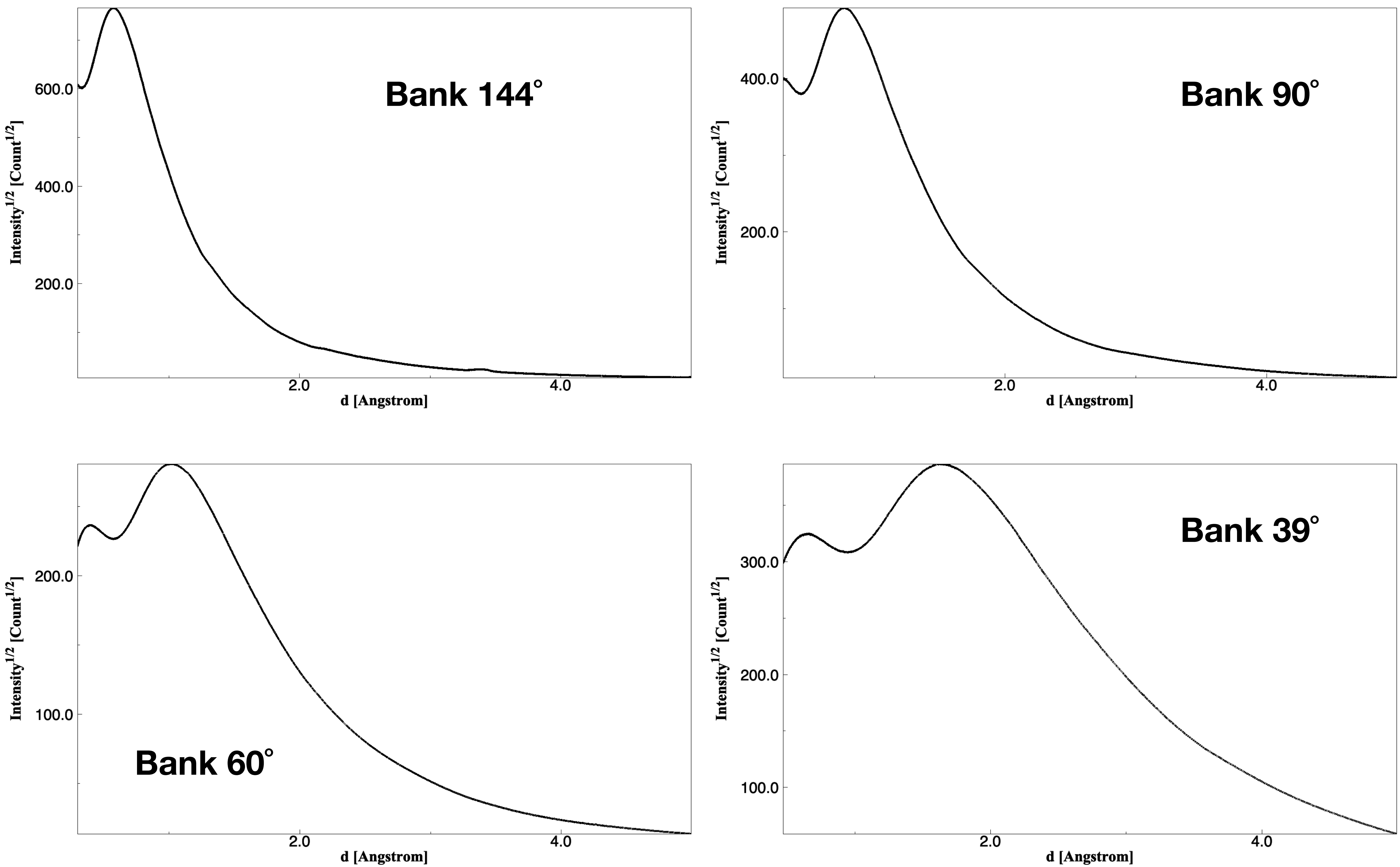
$$DIFC = 252.816 \cdot 2\sin\Theta\left(L_1 + \sqrt{L_2^2 + L_3^2/16}\right)$$

Neutron mass / Planck constant

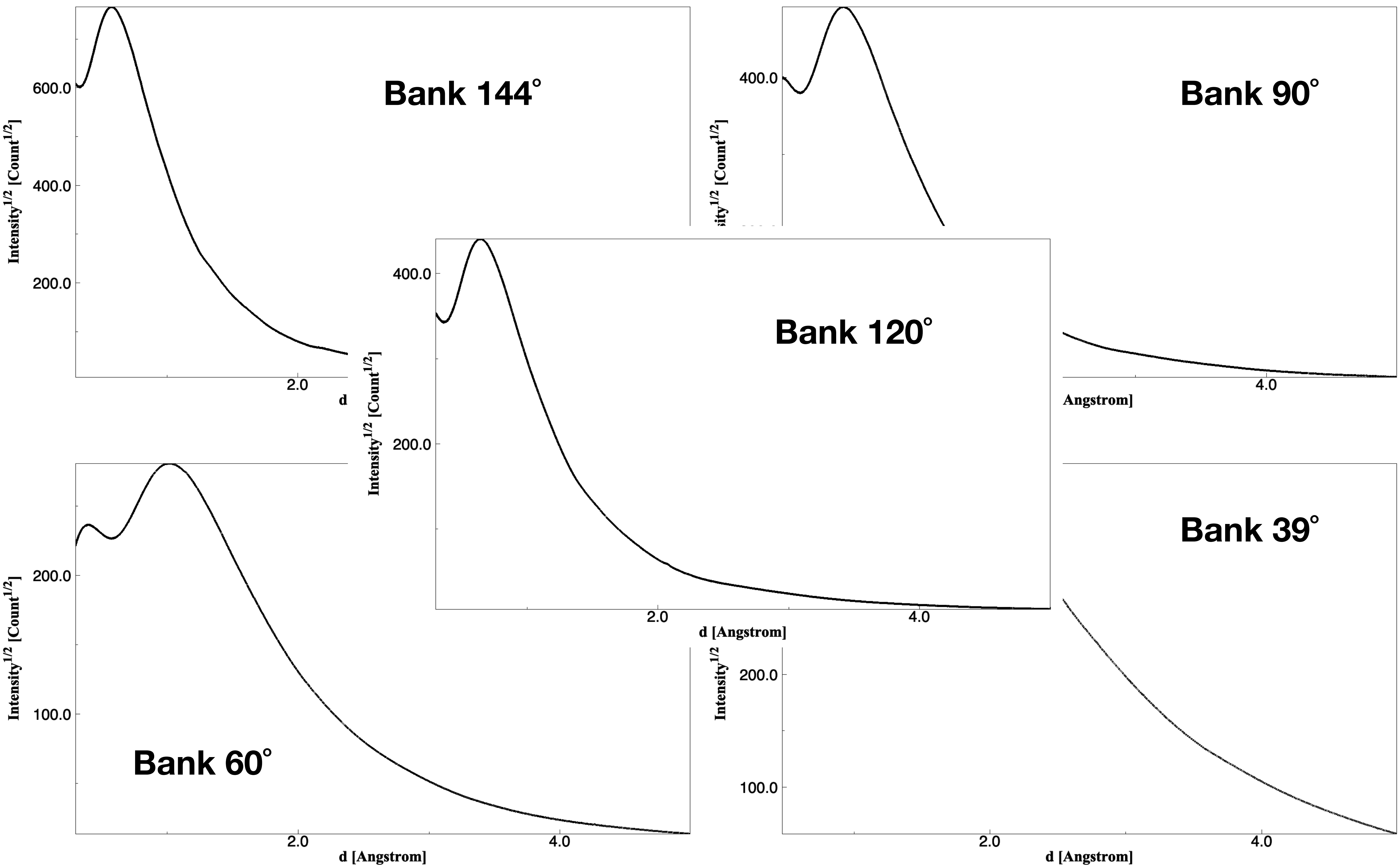
Tube length

Primary path length

Incident intensity: measurement by Nb-V



Incident intensity: measurement by Nb-V



Incident intensity functions

- Correct the intensity on your sample using the directly measured incident intensity function
- Or: fit the incident intensity function using an analytical function and use it for the normalisation

Incident intensity functions

- Correct the intensity on your sample using the directly measured incident intensity function
- Or: fit the incident intensity function using an analytical function and use it for the normalisation

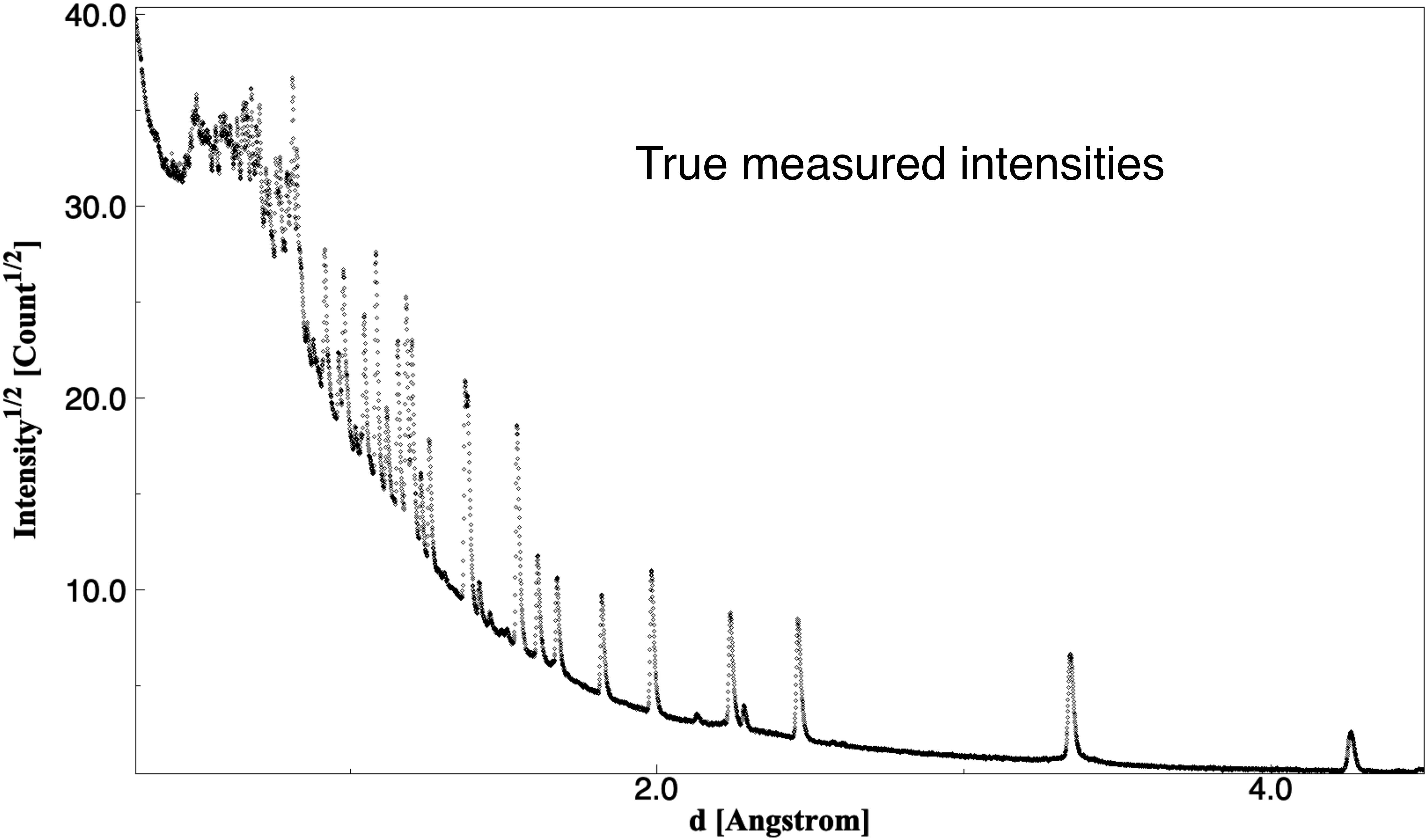
From GSAS manual

Function 1: $$I_i = P_1 + P_2\, exp\left[-P_3 T\right] + P_4\, exp\left[-P_5 T^2\right] + \cdots$$

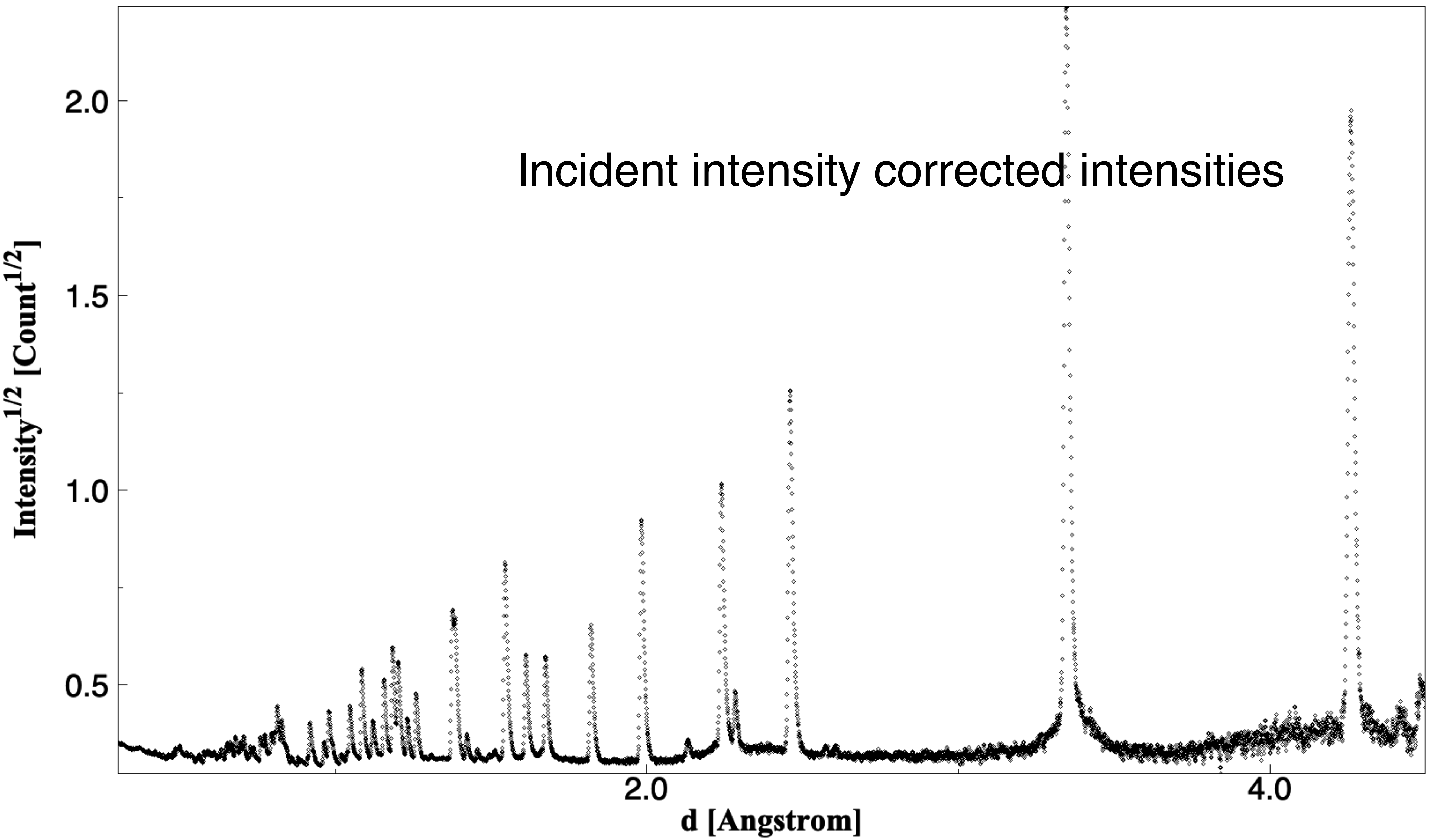
Function 2: $$I_i = P_1 + \frac{P_2}{T^5} exp\left[-P_3 / T^2\right] + P_4\, exp\left[-P_5 T^2\right] + \cdots$$

Function 3: 12 Chebyschev coefficients function

A diffraction experiment: Quartz



A diffraction experiment: Quartz



Hippo layout: more banks is good!

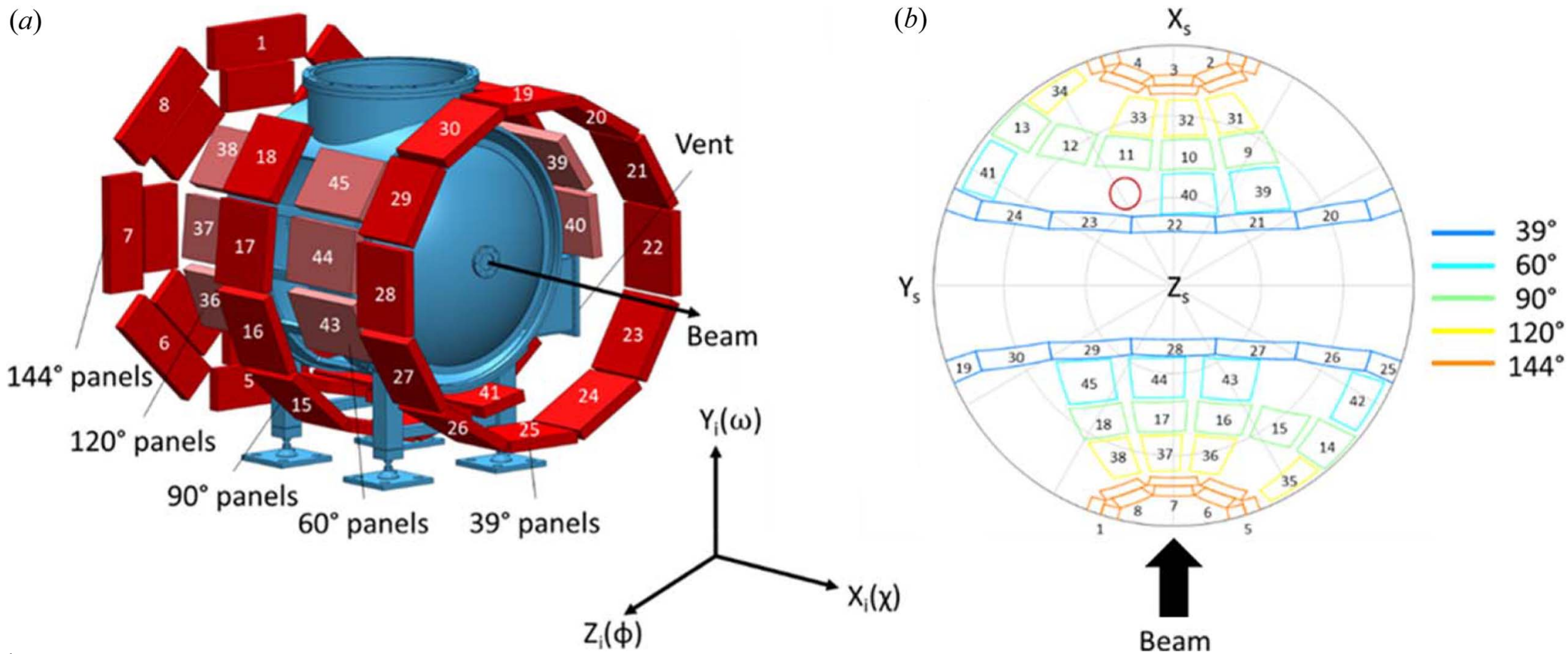
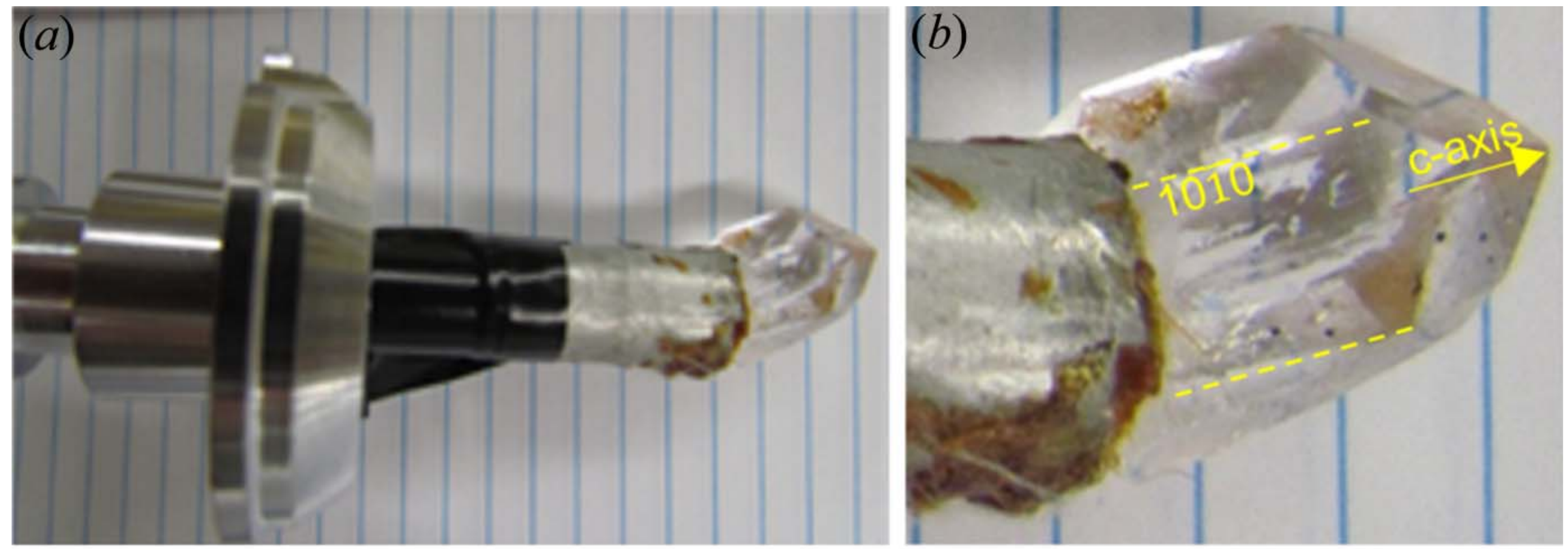
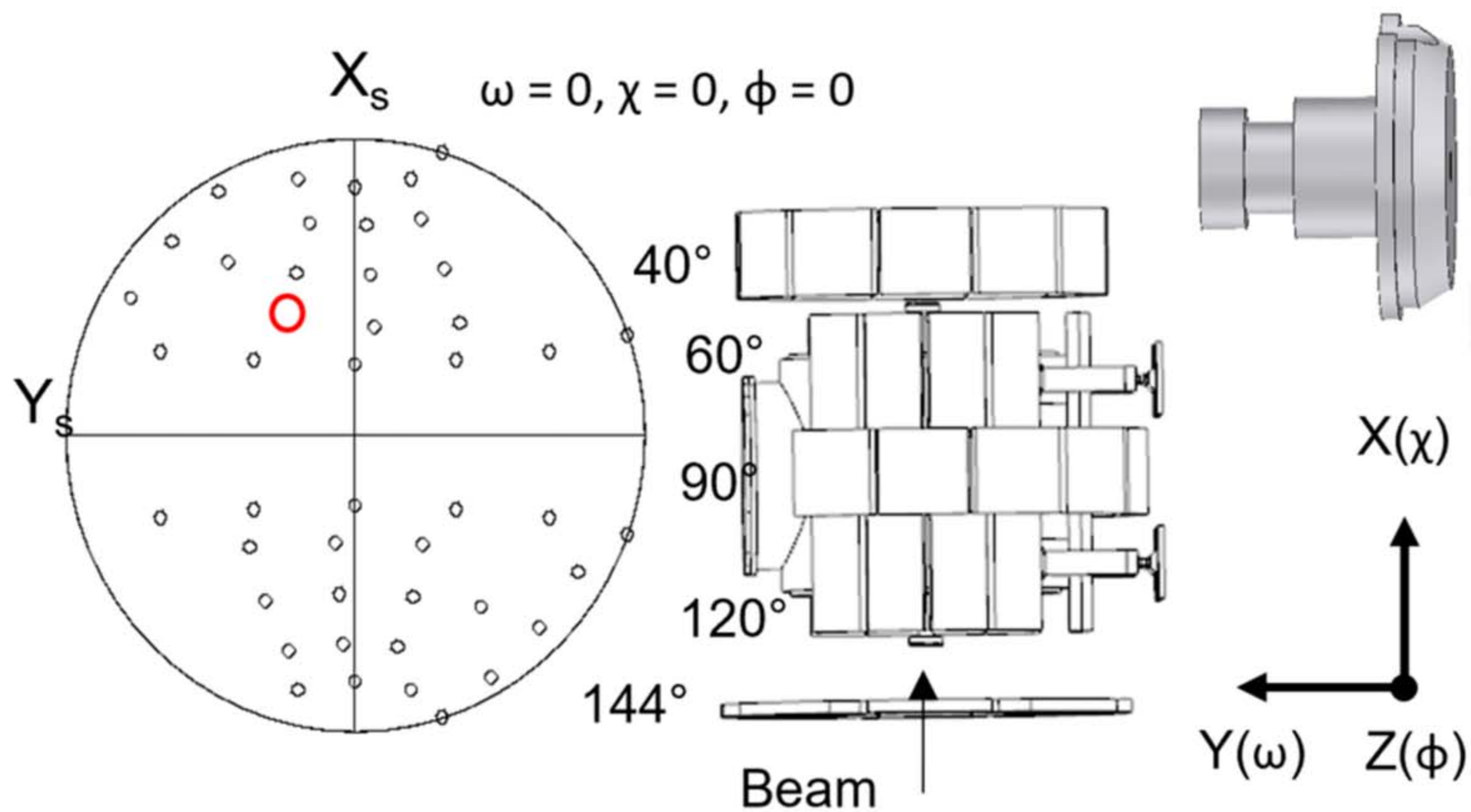


Figure 1

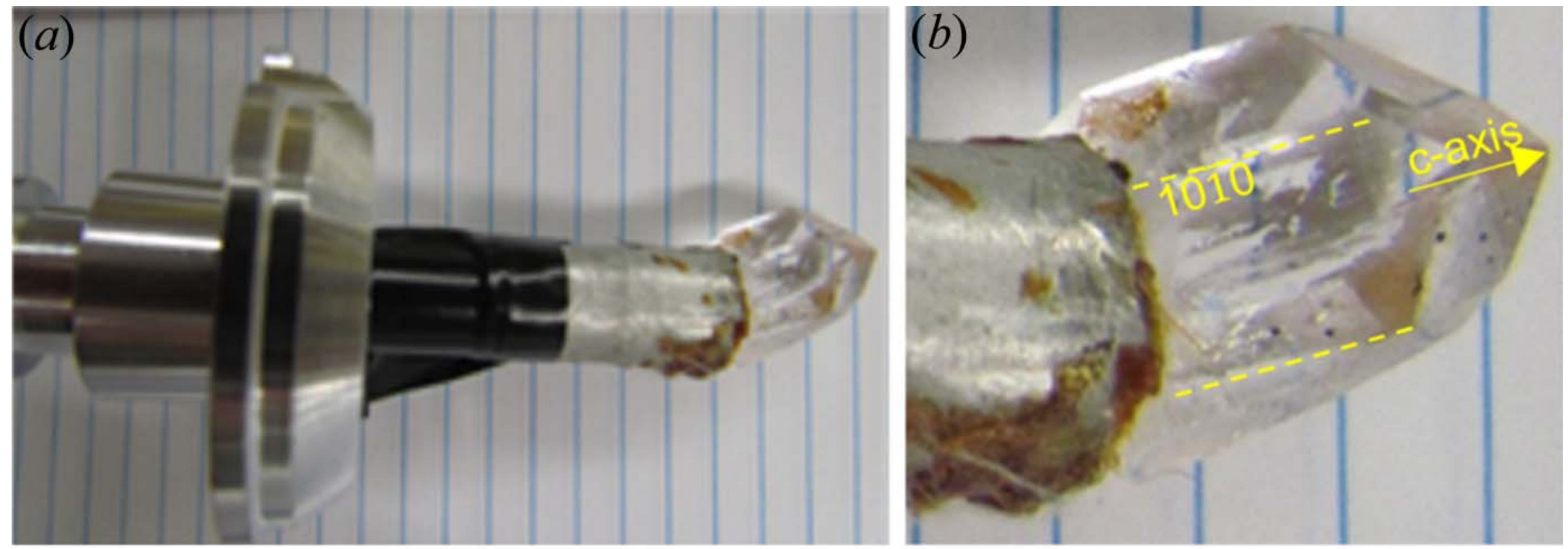
(*a*) Schematic of the HIPPO instrument at LANSCE. Note the location of the flange of the sample chamber (top) and the vent (right side in the 60° detector ring) where the neutron detectors are missing. (*b*) Equal area projection of HIPPO detectors onto a generic (*hkl* independent) pole figure. Differently from constant-wavelength texture measurements, the pole figure coverage in a neutron TOF experiment is the same for all (*hkl*). Note the resulting large gap in the detector coverage from the HIPPO sample chamber flange and the missing detector panel (red circle) due to the vacuum vent which would mirror panel No. 43 with respect to a vertical mirror plane. The correlation of the pole figure coverage plot at $\omega_s = 0°$, $\chi_s = 0°$, $\varphi_s = 0$ with the instrument detector layout is shown in more detail in Fig. 4.

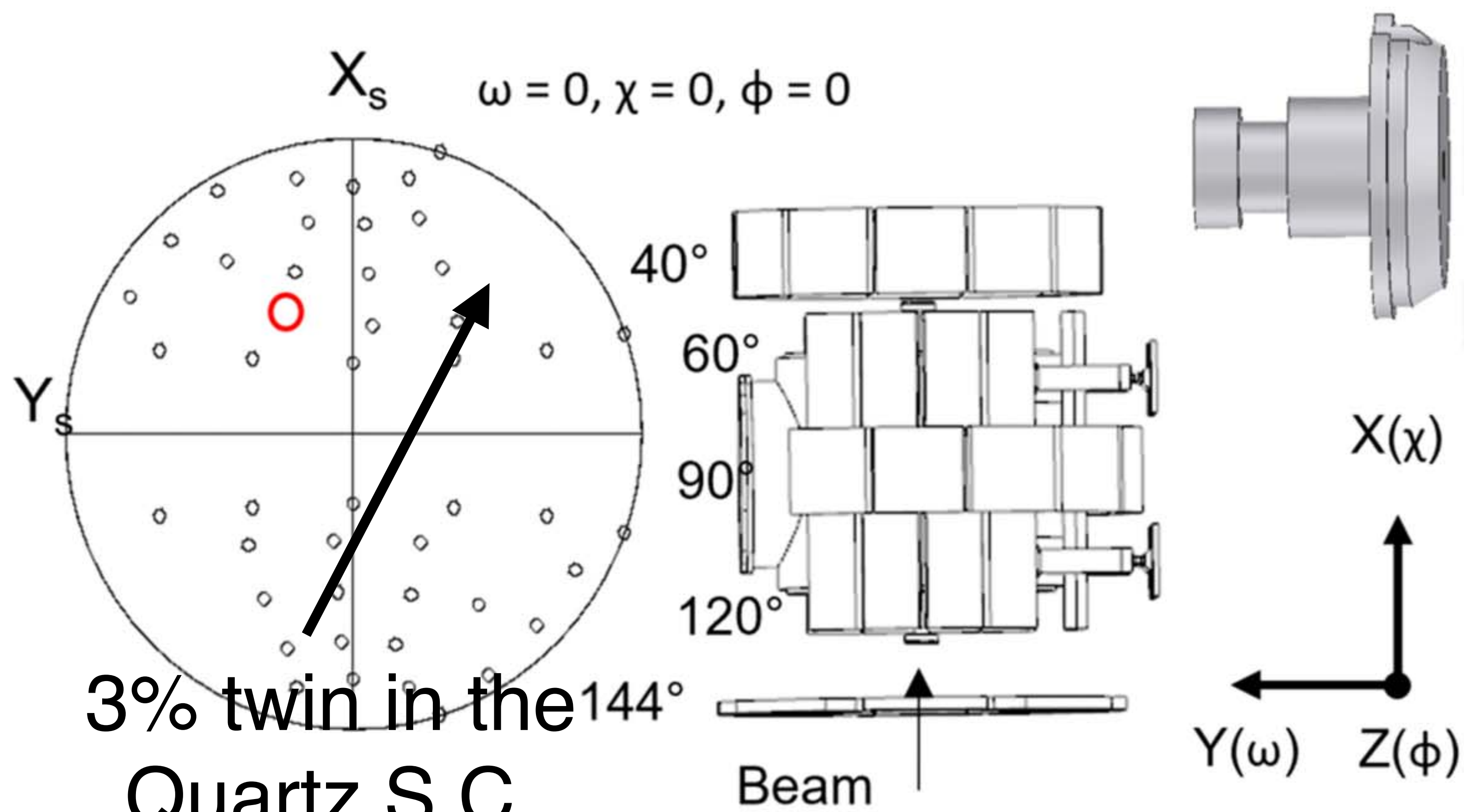
You can measure a single crystal!





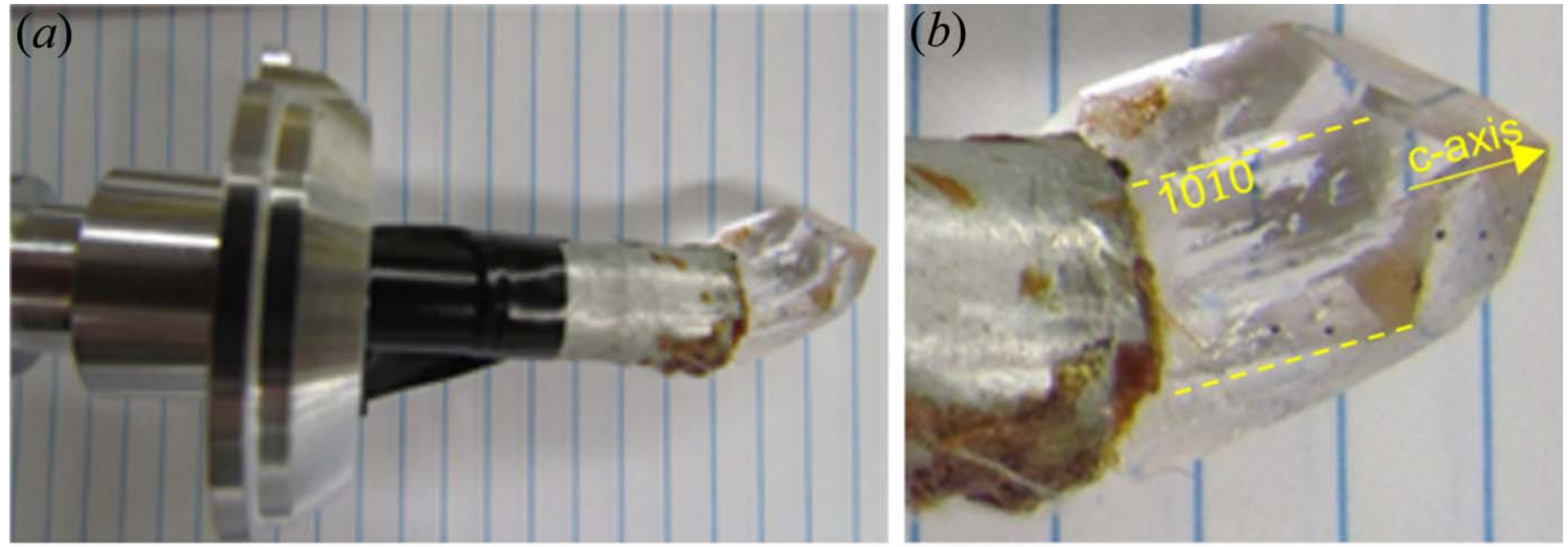
You can measure a single crystal!





3% twin in the Quartz S.C.

You can measure a single crystal!



3% twin in the
Quartz S.C.

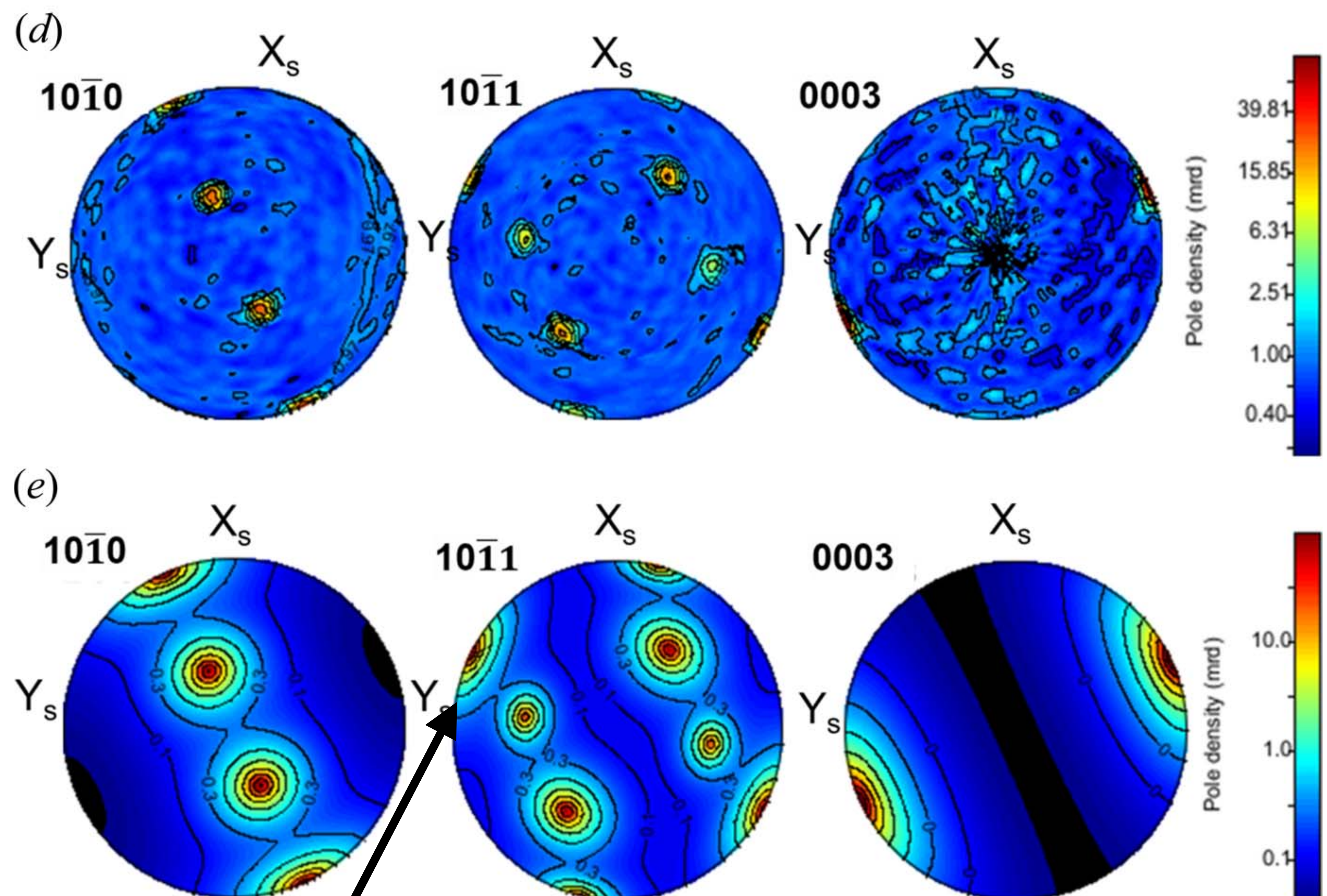
You can measure a single crystal!

3% twin in the
Quartz S.C.

You can measure a single crystal!

Measured/Analysed

Modelled (Standard Functions)



3% twin in the Quartz S.C.

The Ötzi/Icemen's axe

Its copper axe, the oldest copper manufact from the the alpine region (4000 b.C.)

The Ötzi/Icemen's axe

Its copper axe, the oldest copper manufact from the the alpine region (4000 b.C.)

The Ötzi/Icemen's axe

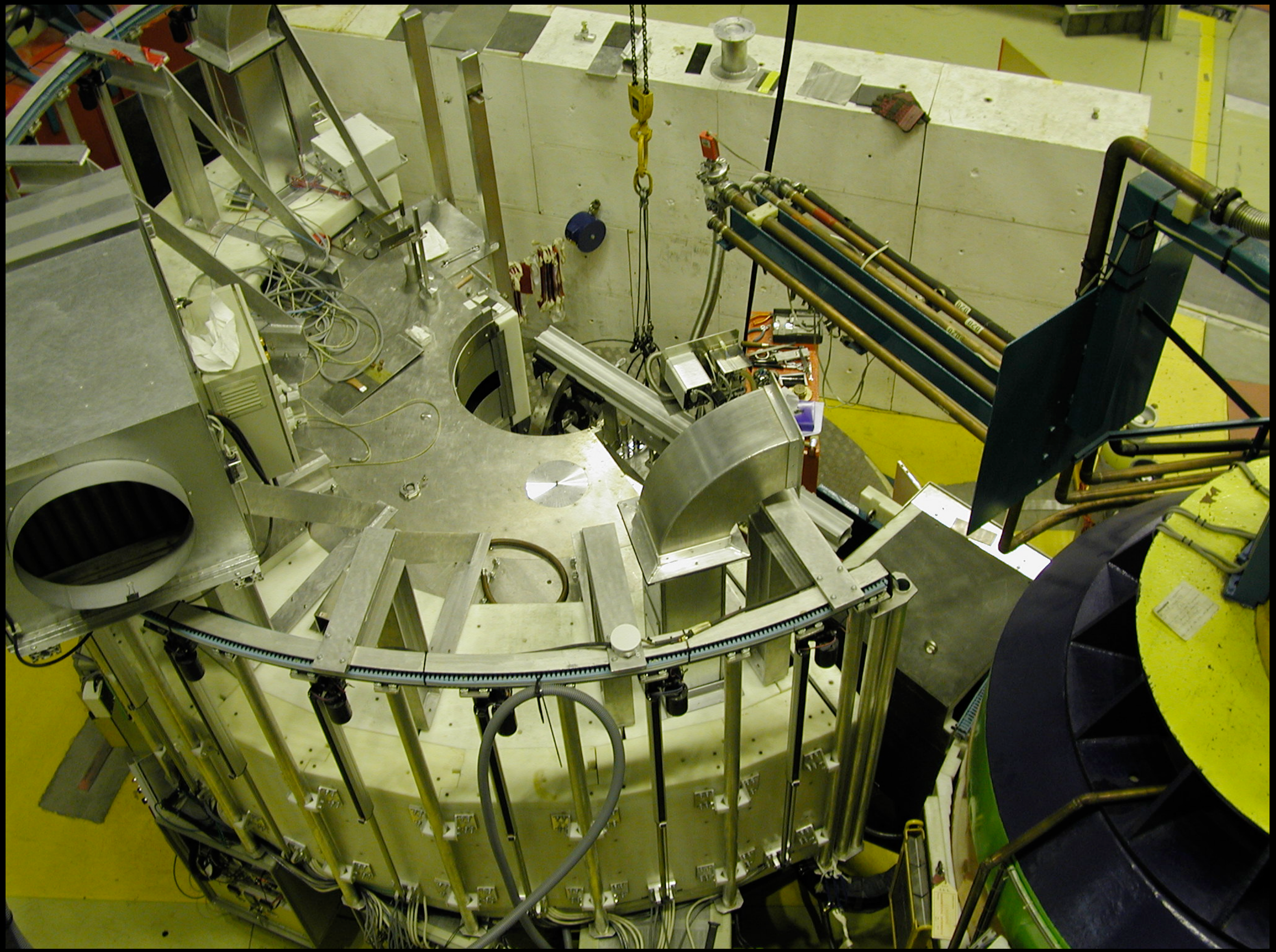
Its copper axe, the oldest copper manufact from the the alpine region (4000 b.C.)

Texture analysis (neutron diffraction)

648 different orientations in χ, φ (~1 day)

Texture analysis (neutron diffraction)

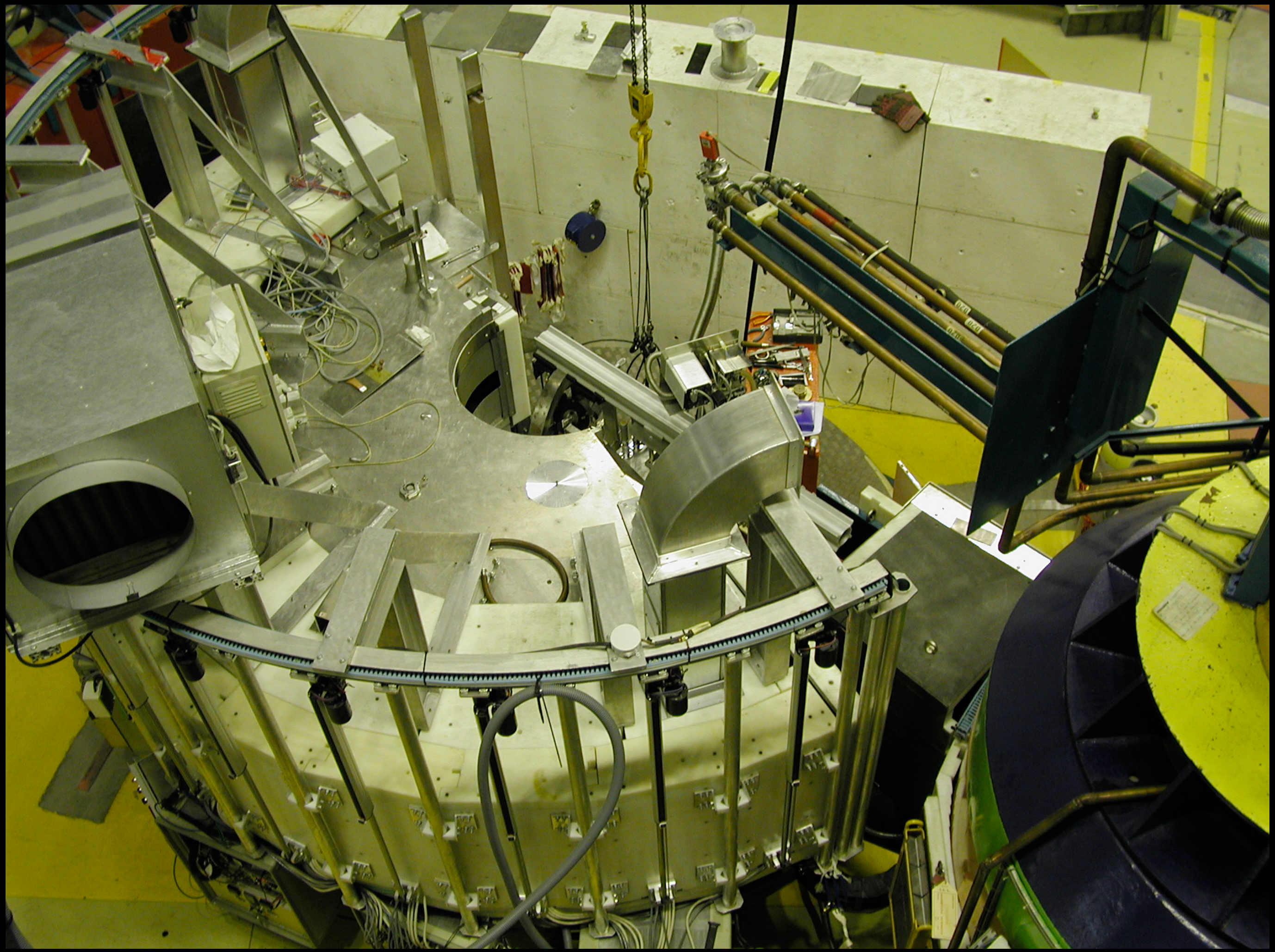
648 different orientations in χ, φ (~1 day)



ILL – D20
Grenoble, F

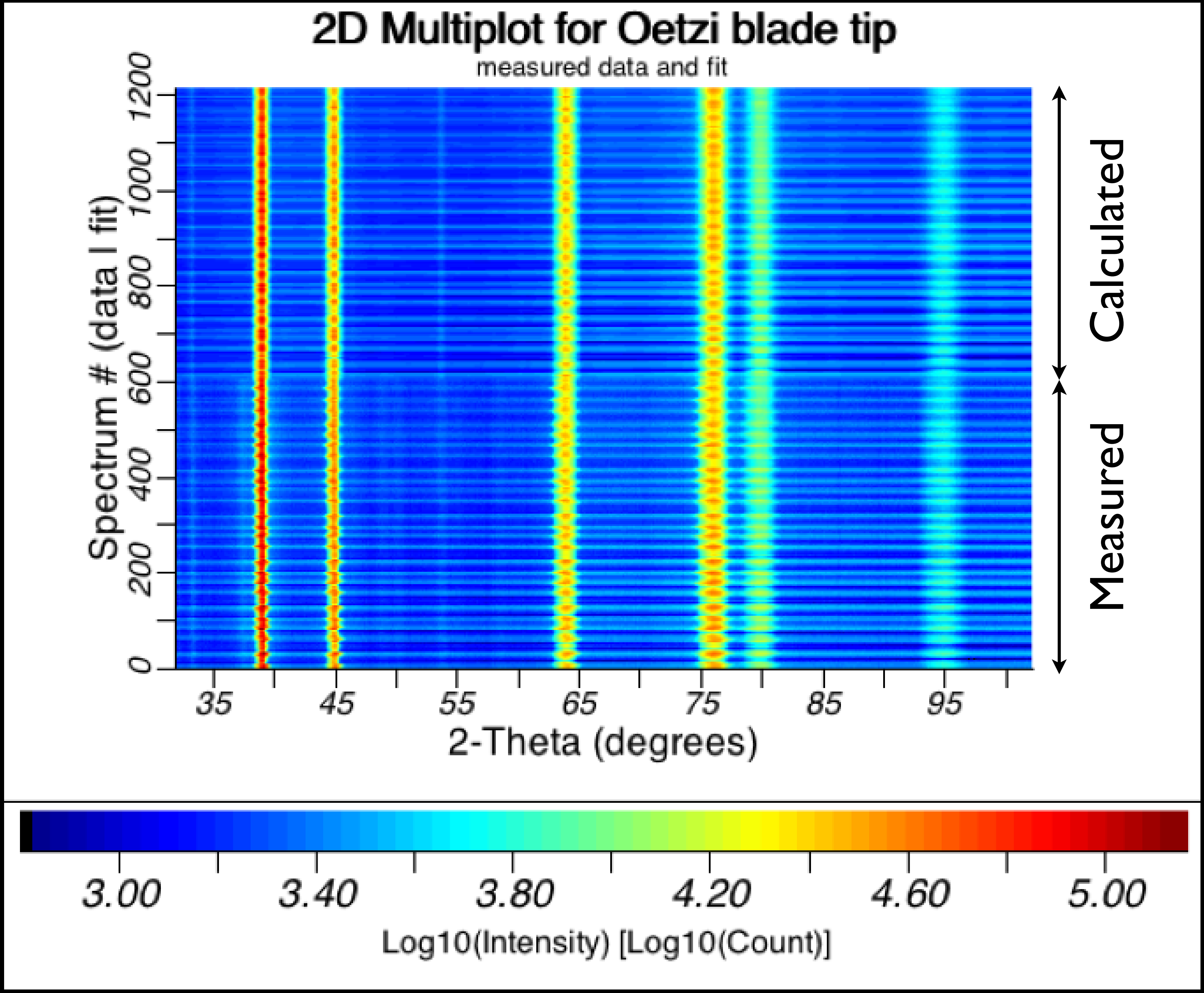
Texture analysis (neutron diffraction)

648 different orientations in χ, φ (~1 day)



ILL – D20
Grenoble, F

Spectra fitting by Maud



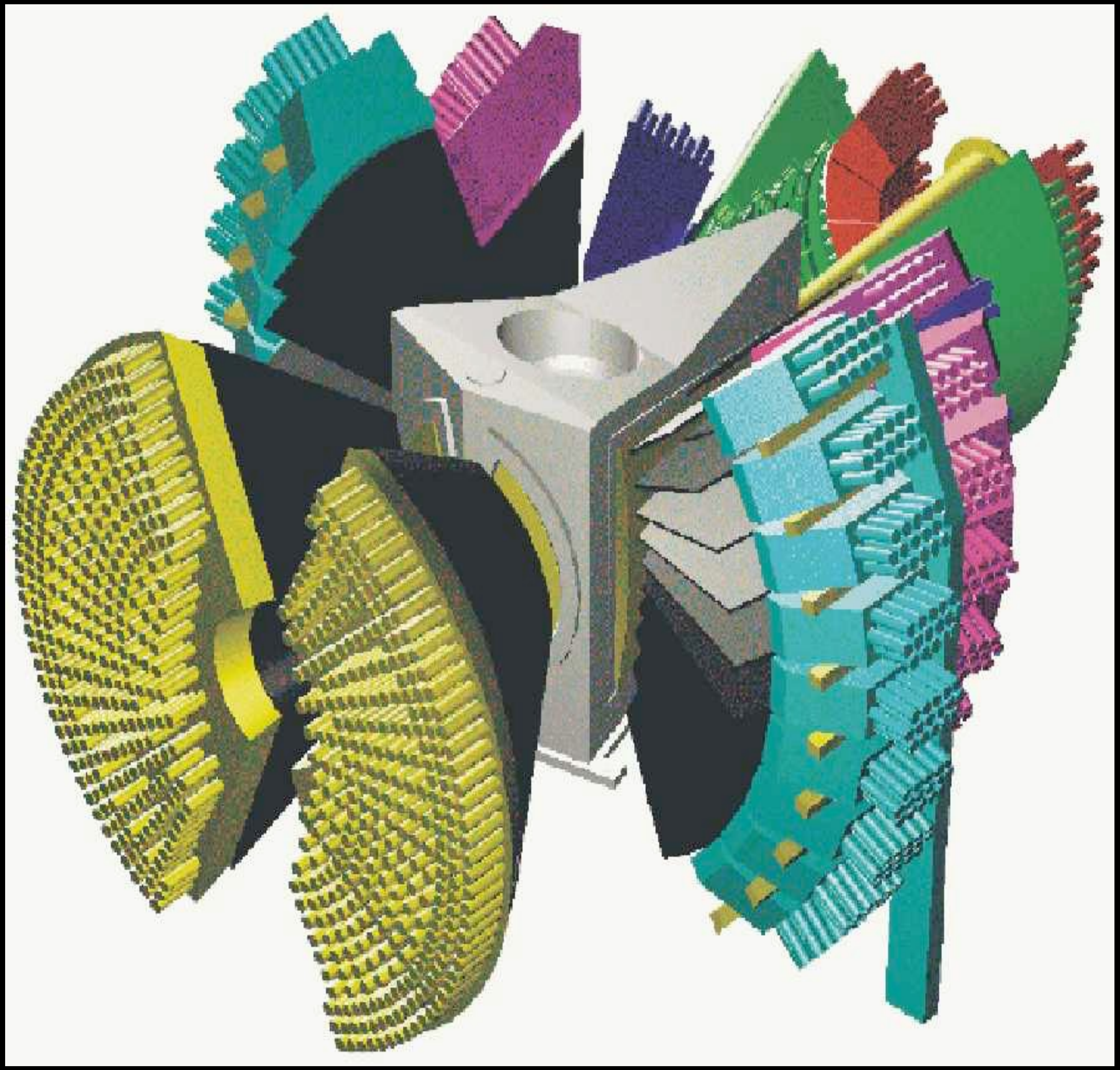
Others alpine copper axes (43)

Others alpine copper axes (43)

Texture analysis (neutron diffraction, TOF)

3 rotations
(~20 minutes)

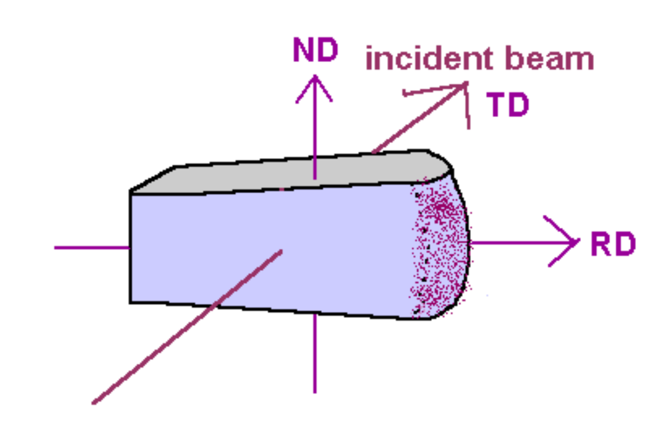
Texture analysis (neutron diffraction, TOF)

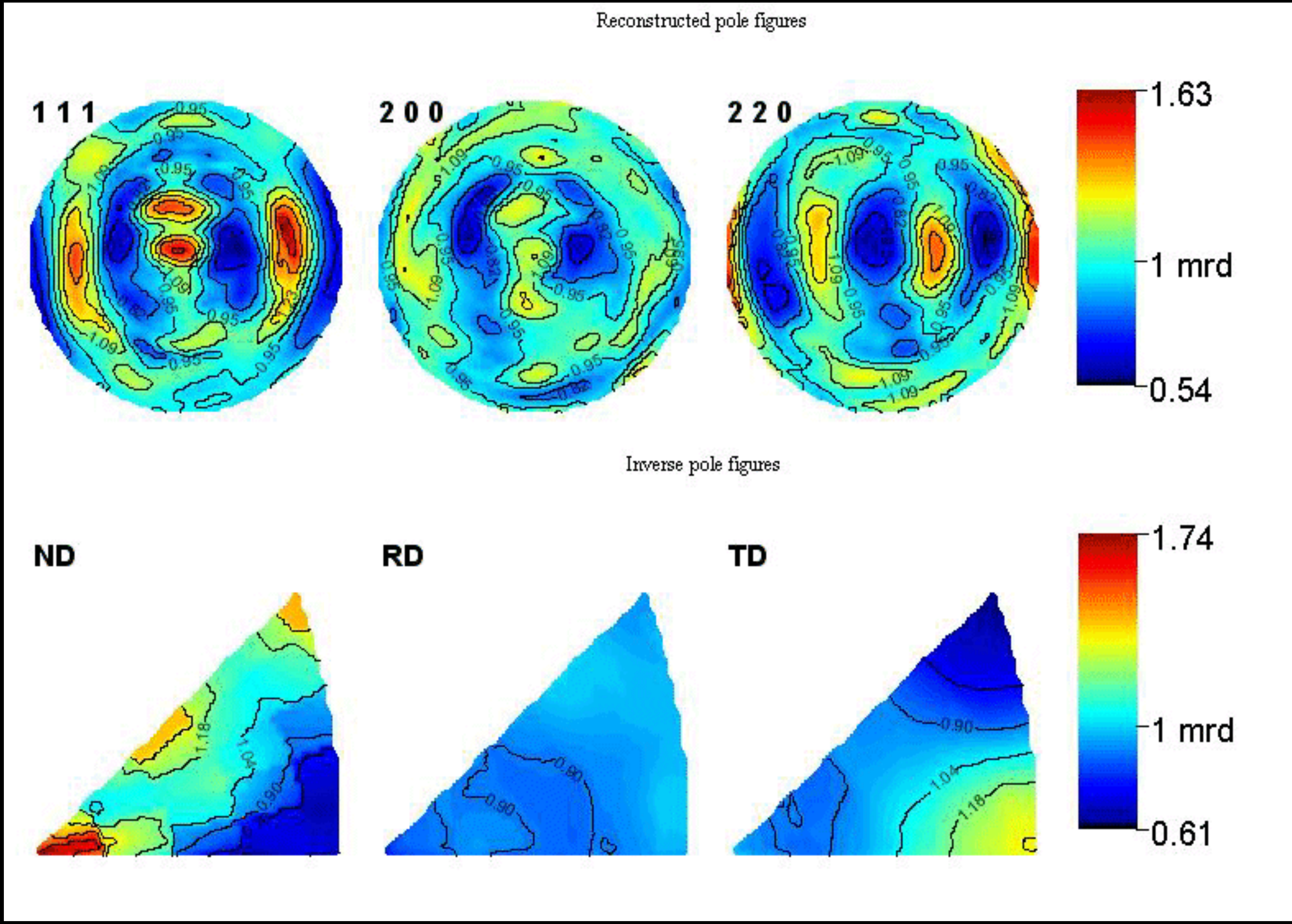


3 rotations
(~20 minutes)

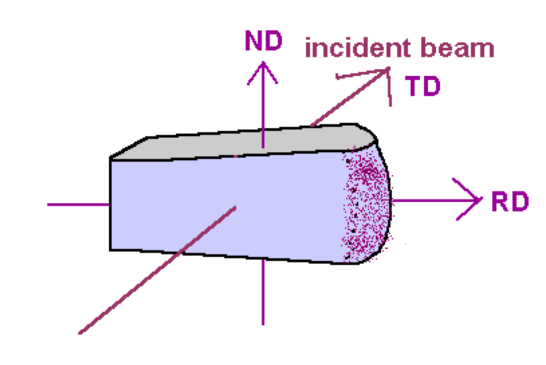
ISIS - GEM
Oxford, UK

Lovere LOV-330 (rolling texture)

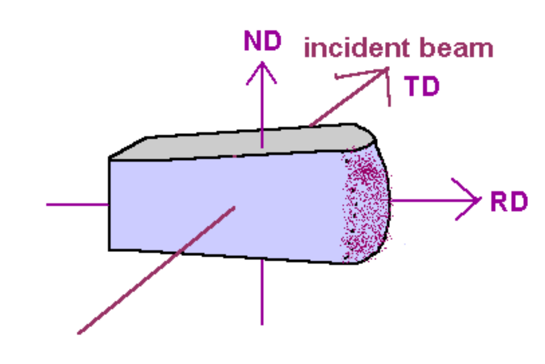


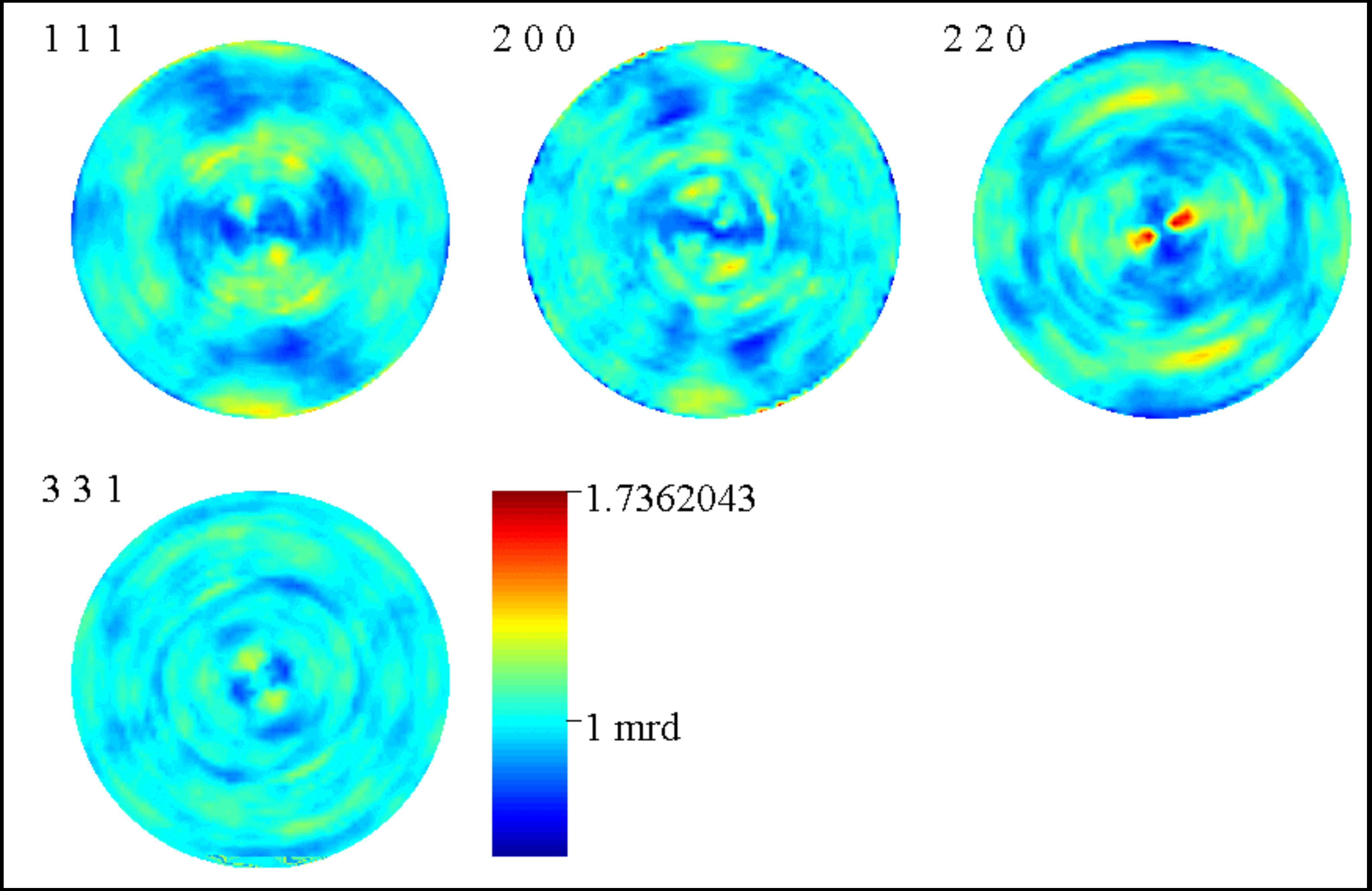


Icemen/Ötzi (no texture)

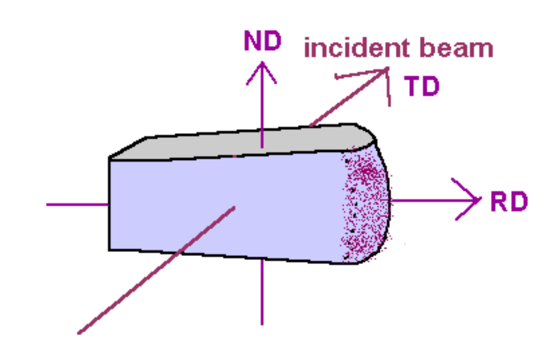


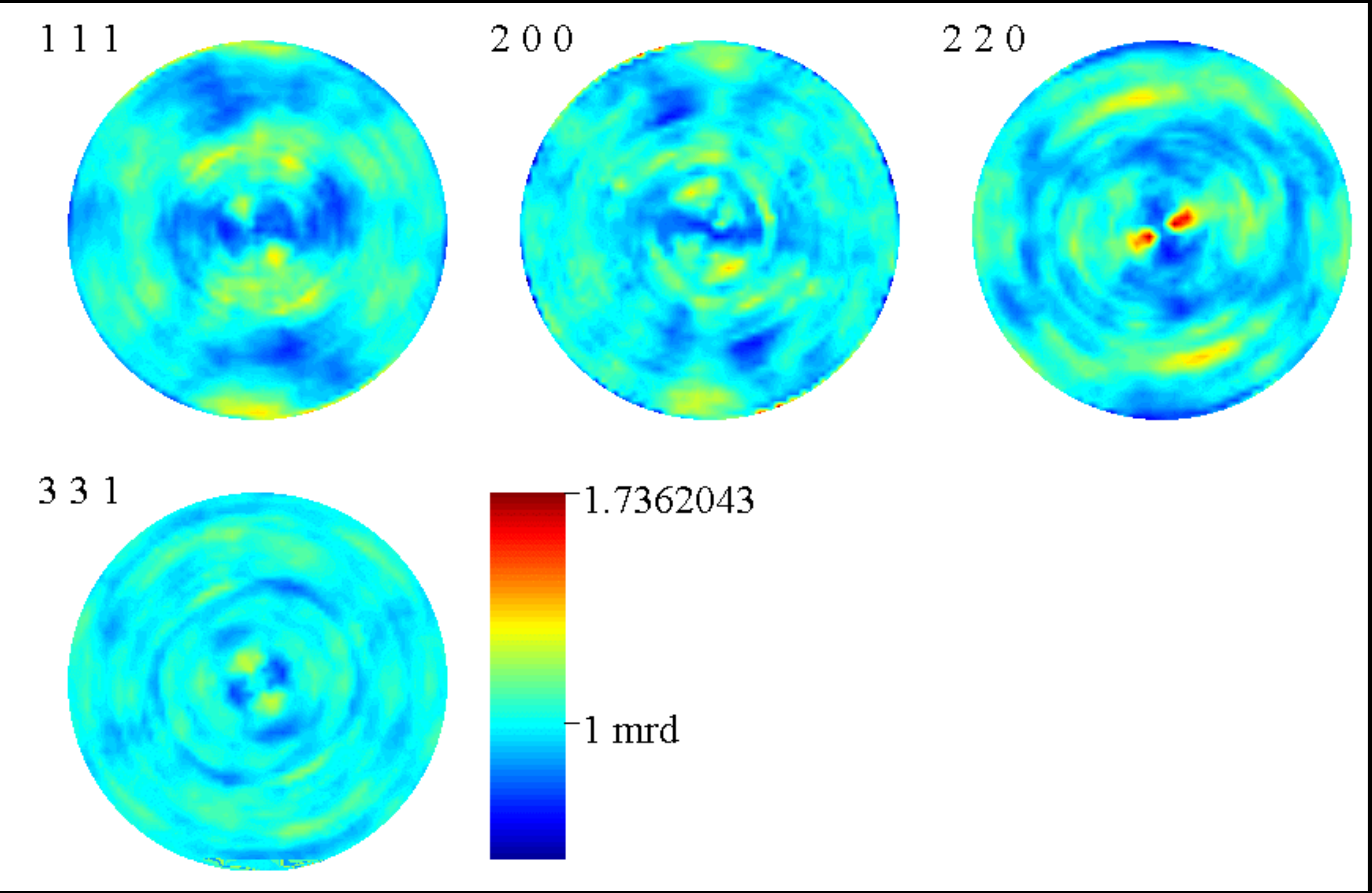
Icemen/Ötzi (no texture)



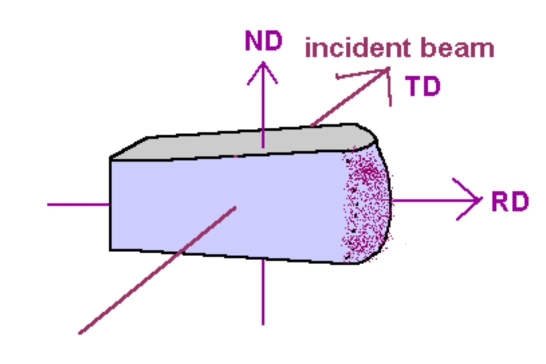


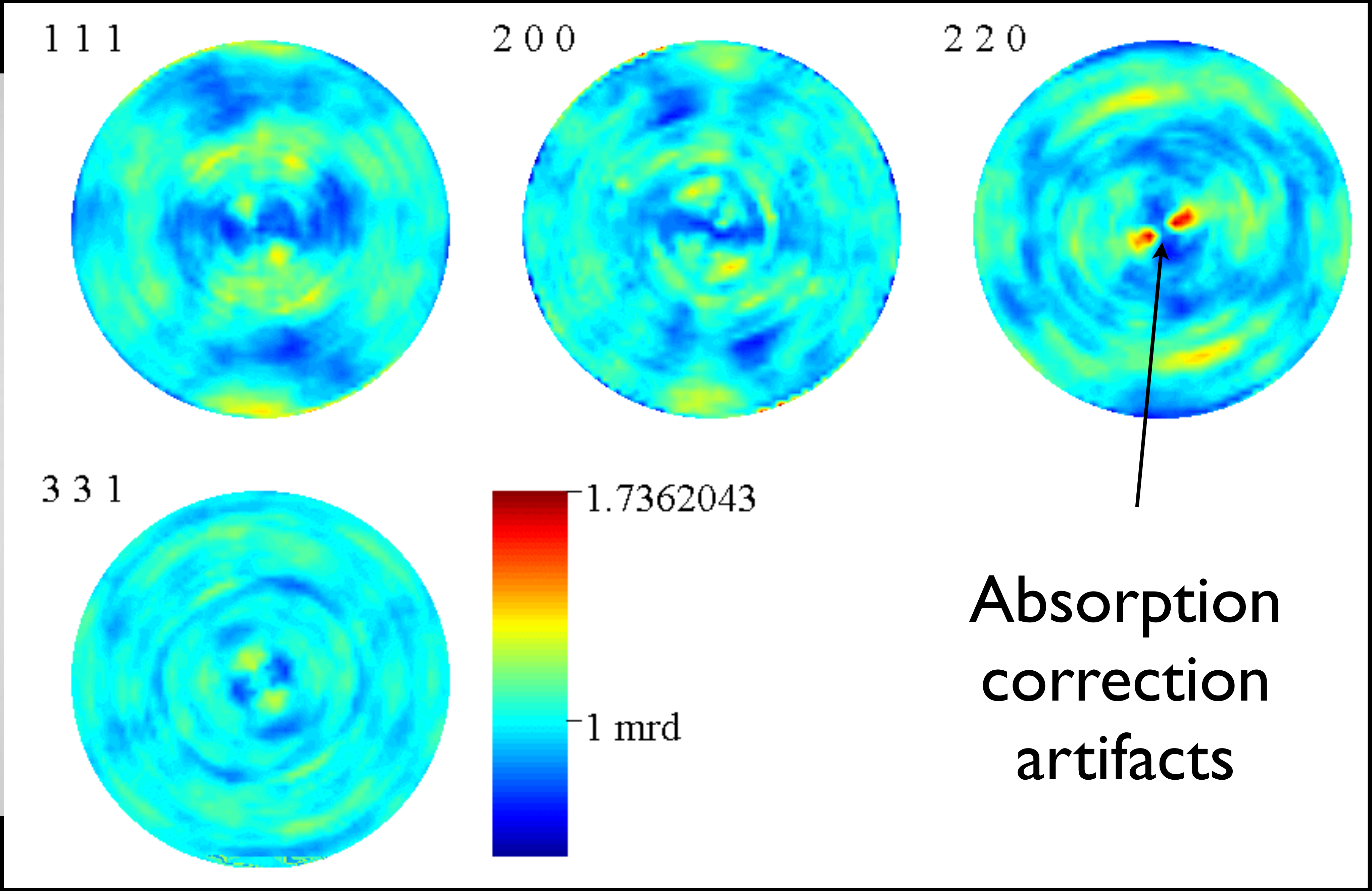
Icemen/Ötzi (no texture)



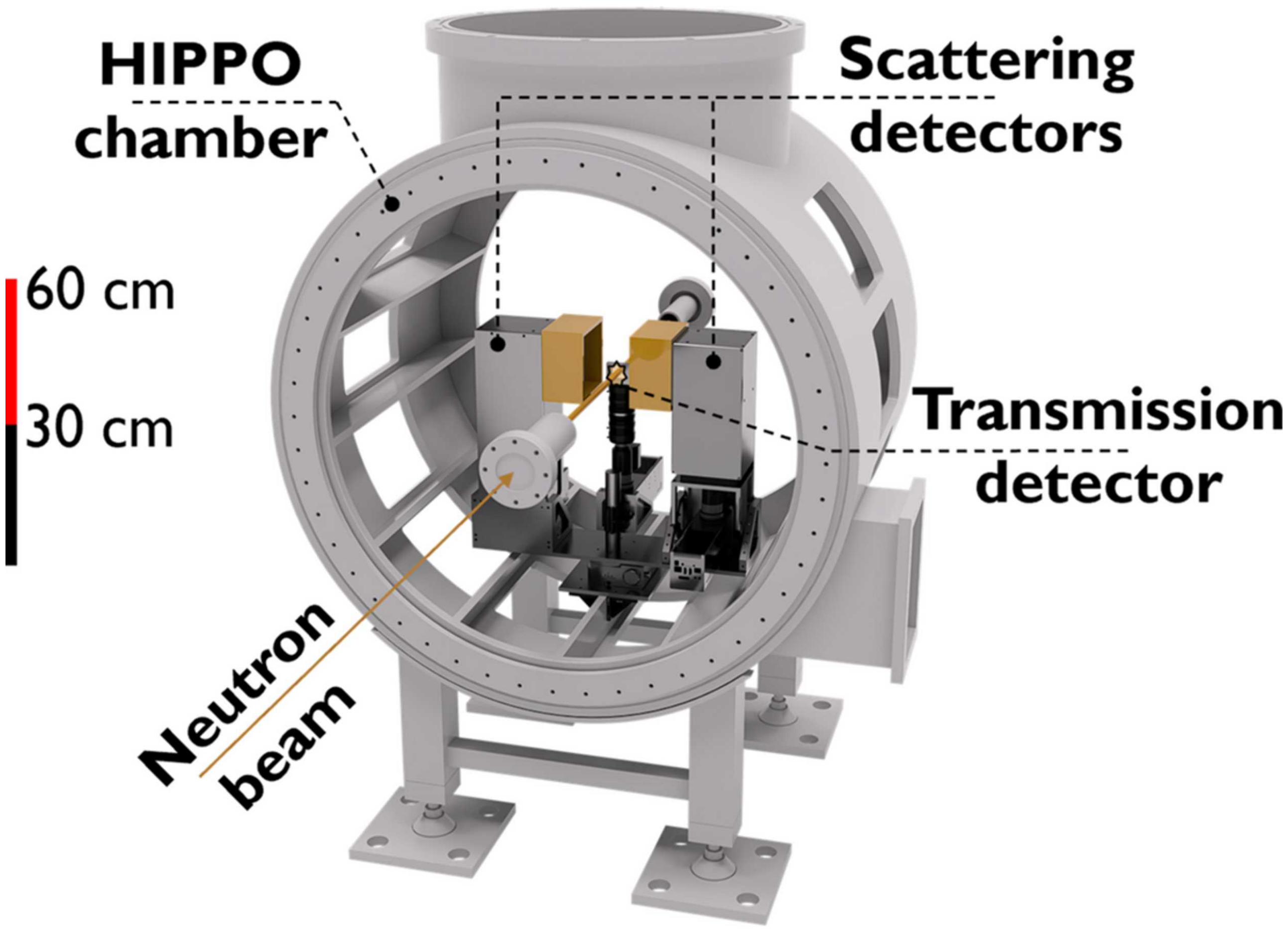


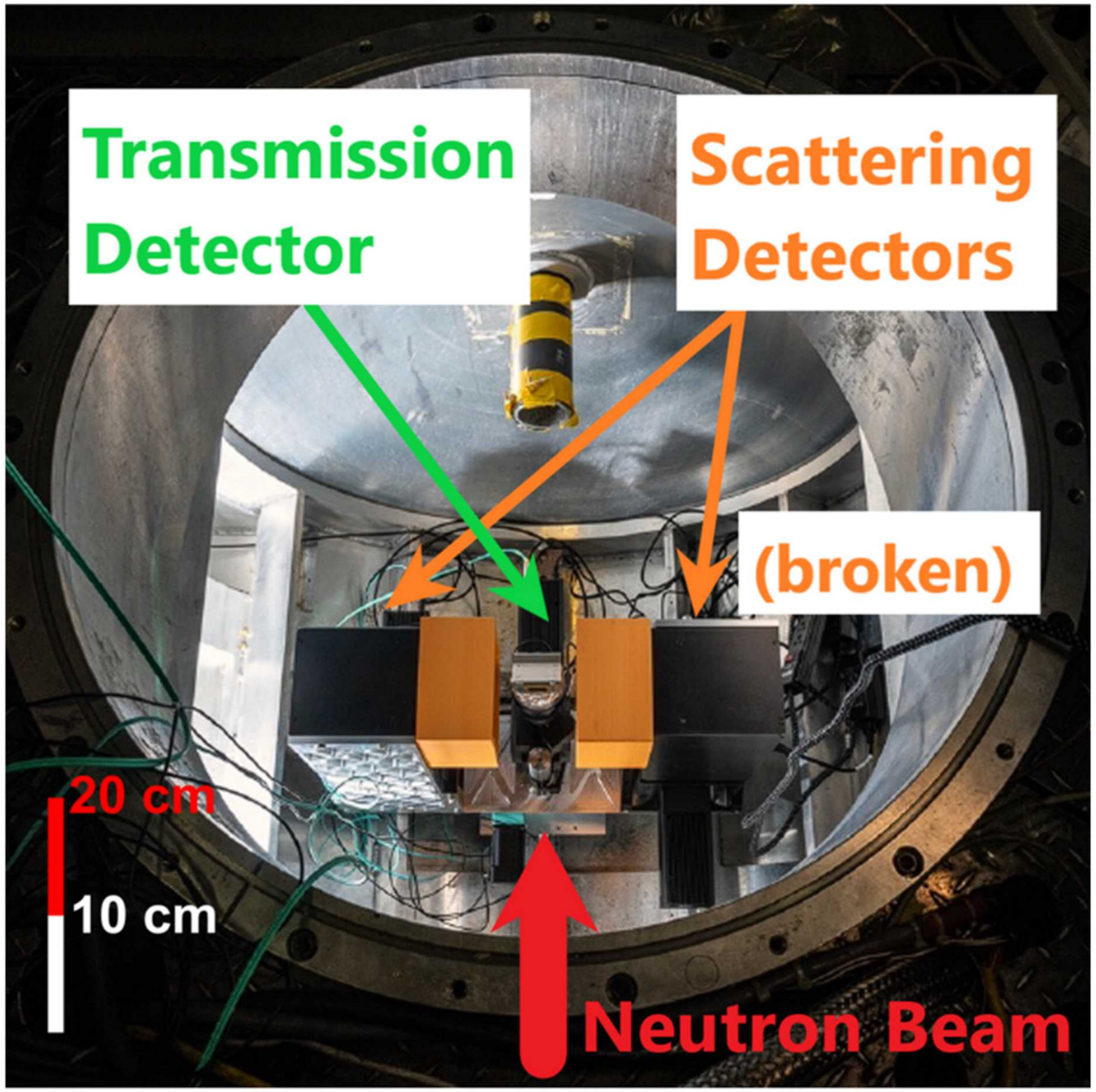
Icemen/Ötzi (no texture)





LumaCam on Hippo





Diffraction data processing

```
X and Y in pixels with sub pixel resolution thanks to event centroiding
ToF in mus
Other parameters like scintillator size but feel free to ask Sven
Tiiiiim was here

X Y #Photons  ToF PSD
157.875 109.3508064516129 1.0 7.812501756632173e-10 0.0
173.0841121495327 68.3644859813084  1.0 1.0437012365471787e-09  0.0
142.0 230.1666666666666  1.0 1.3061522974311401e-09  0.0
77.14139344262296 158.89344262295083  1.0 1.8249513011880936e-09  0.0
175.546875  45.84375  1.0 2.0874024175832062e-09  0.0
```

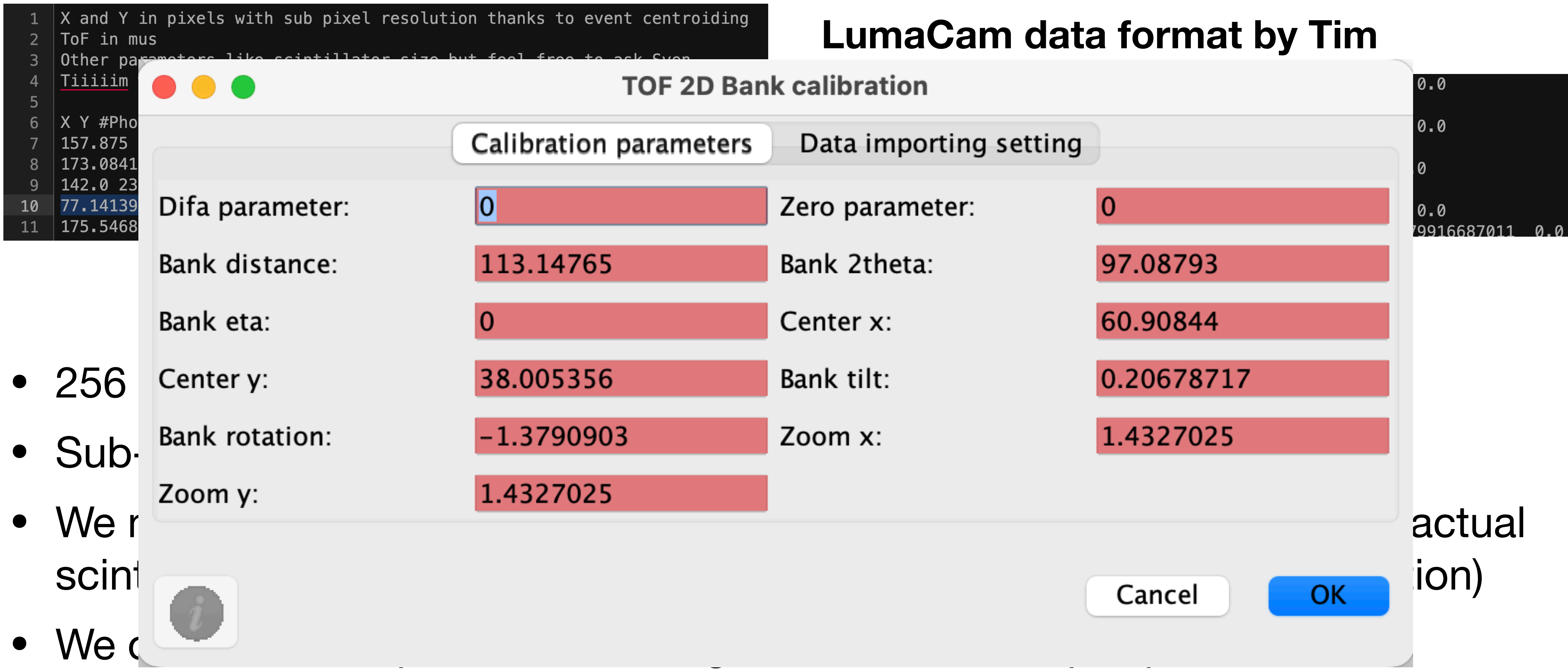
LumaCam data format by Tim

```
210.65384615384616  168.0 1.0 0.23951131198120112 0.0
237.25  39.625  1.0 0.2407752445312501  0.0
188.0 187.45238095238096  1.0 0.24083704323120125 0.0
157.625 131.325 1.0 0.24283251354370128 0.0
99.6086956521739  42.0  1.0 0.24807777448120127 0.0
197.53125 105.0 1.0 0.2483252526062012  0.0
136.0 167.63829787234042  1.0 0.24993972916870122 0.0
139.32692307692307  46.31730769230769 1.0 0.2506979916687011  0.0
```

- 256 x 256 pixels (camera, effective pixels depend on field of view)
- Sub-pixel resolution (that we can use)
- We need to exclude pixels on the border, camera uses larger FoV than actual scintillator area, and super-pixel size for binning (larger in vertical direction)
- We obtain N TOF patterns, N being the number of super-pixels
- Each super-pixel is at a different distance from sample, 2Θ and η (angular position along the diffraction cone)
- Using a standard sample (well known cell parameter) we fit, with the Rietveld method, all patterns to calibrate the detector parameters (distance, position in 2Θ, center, tilting and FoV) + TOF→d-spacing function

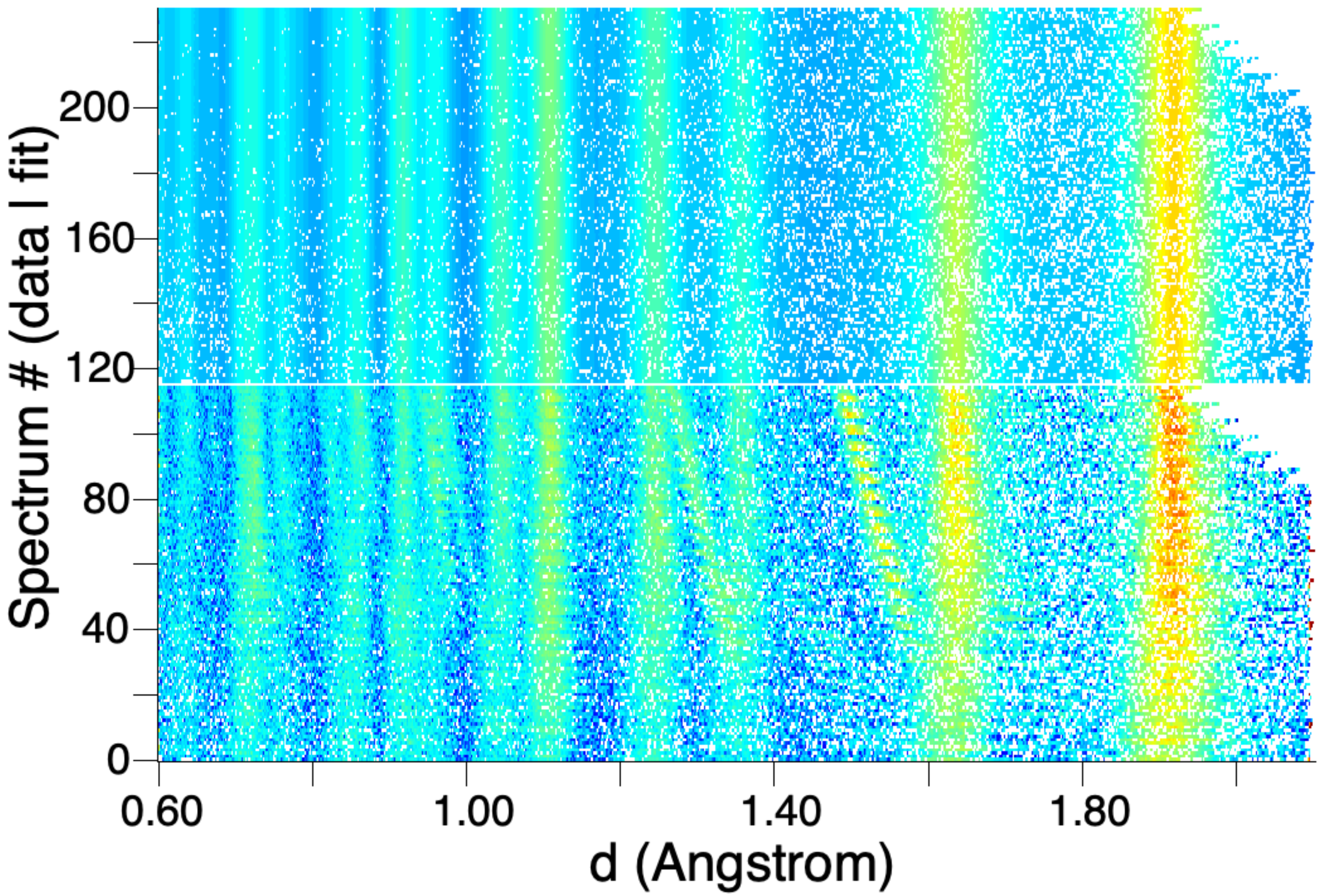
Diffraction data processing

LumaCam data format by Tim

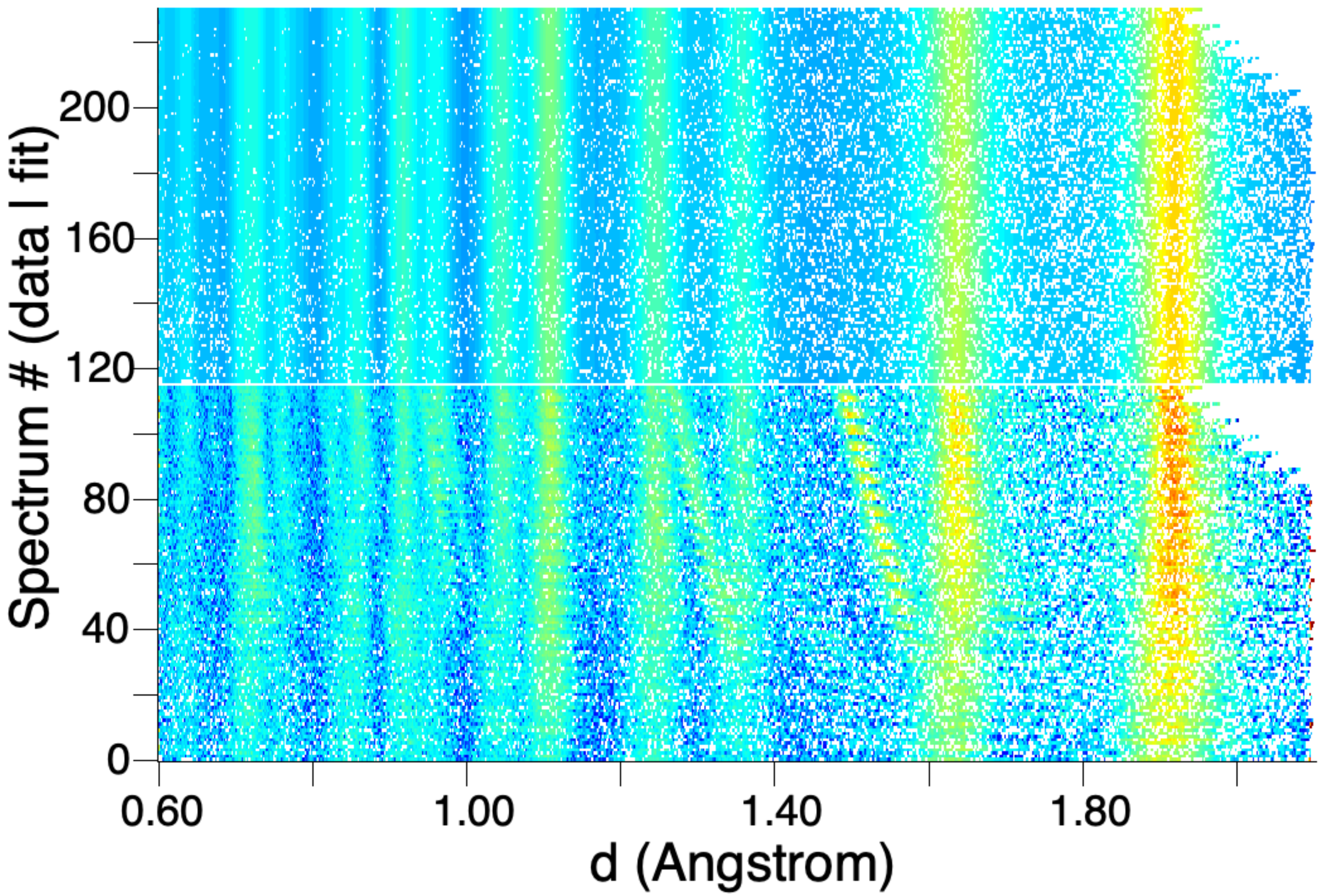


- 256
- Sub-
- We r… actual scint… ion)
- We c…
- Each super-pixel is at a different distance from sample, 2Θ and η (angular position along the diffraction cone)
- Using a standard sample (well known cell parameter) we fit, with the Rietveld method, all patterns to calibrate the detector parameters (distance, position in 2Θ, center, tilting and FoV) + TOF→d-spacing function

Rietveld calibration with LumaCam data

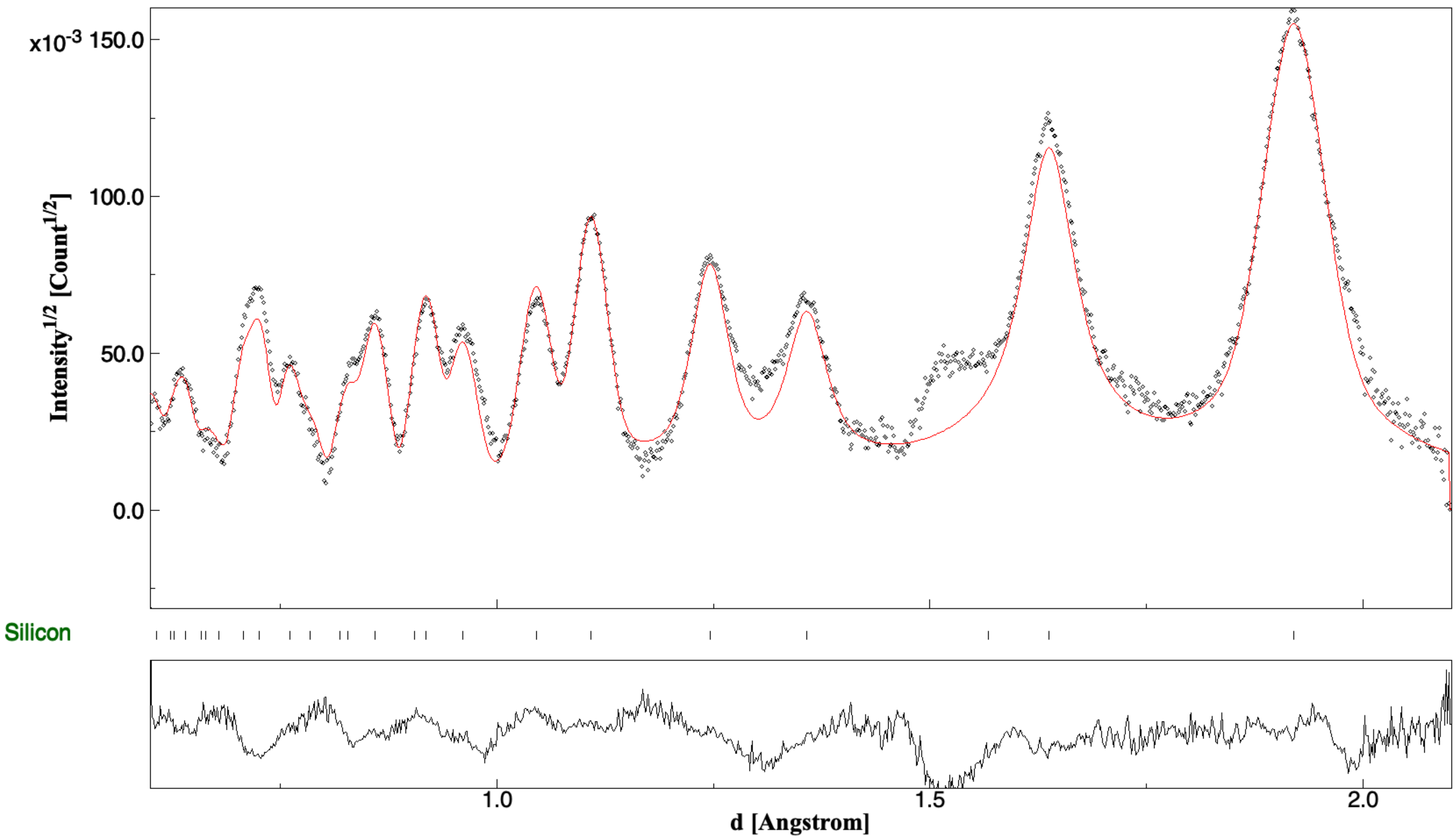


Rietveld calibration with LumaCam data



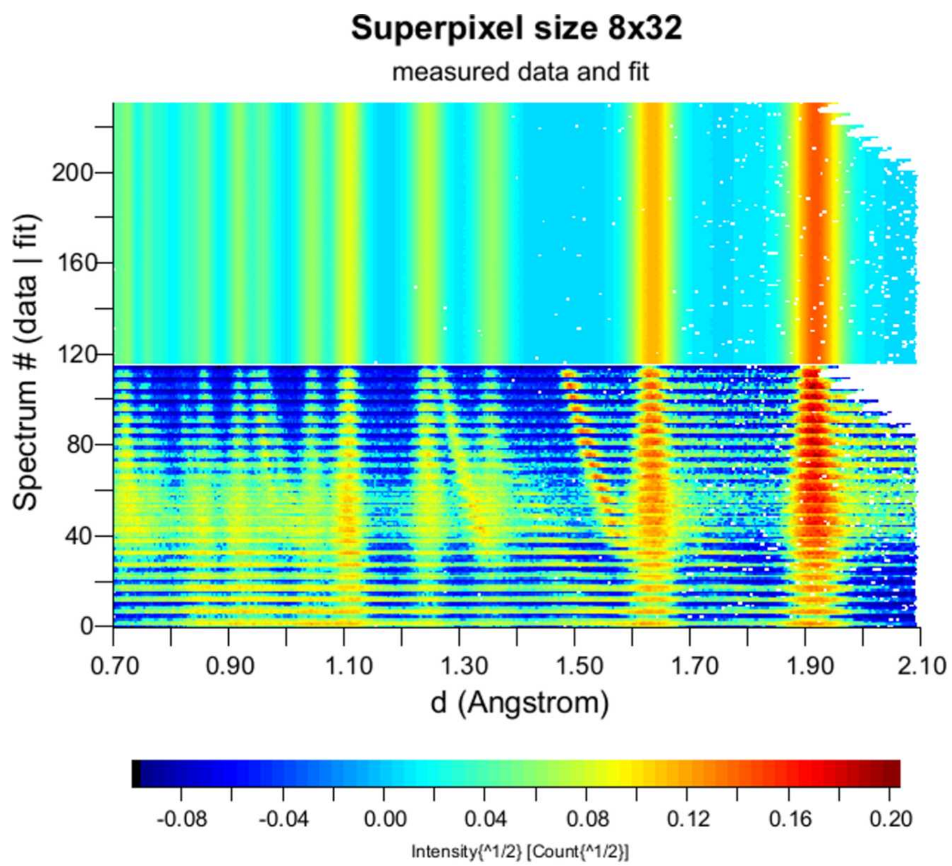
- 2θ min: 65.6° (pixel 1), 2θ max: 119.96° (pixel 111)
- η min: 49.7° (115), η max: 96.76° (111)
- Distance from sample, min: 408.22 mm (57), max: 540.29 (5)

Rietveld calibration with LumaCam data

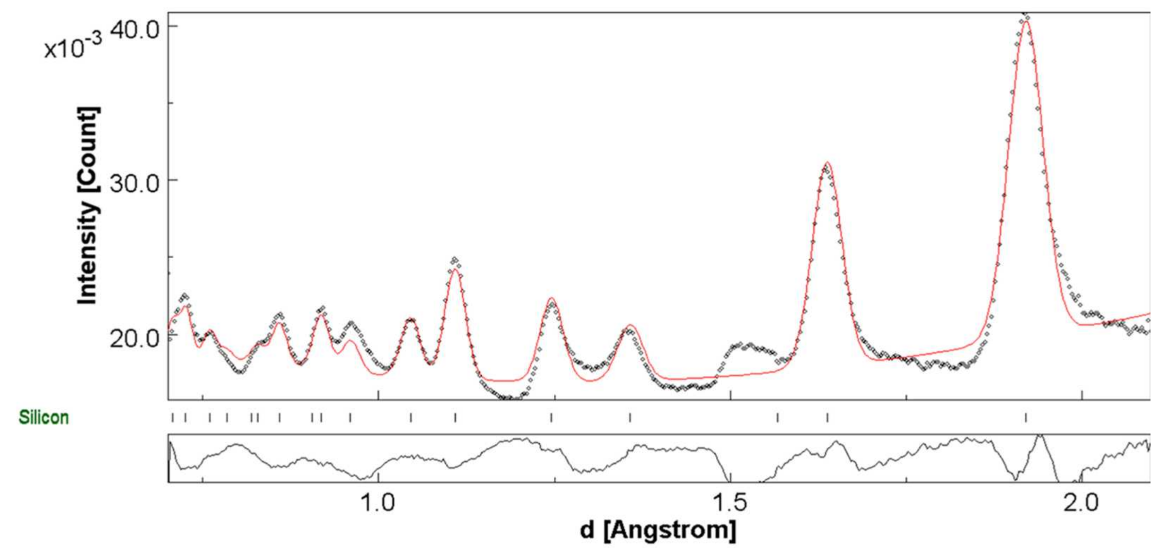


- 2θ min: 65.6° (pixel 1), 2θ max: 119.96° (pixel 111)
- η min: 49.7° (115), η max: 96.76° (111)
- Distance from sample, min: 408.22 mm (57), max: 540.29 (5)

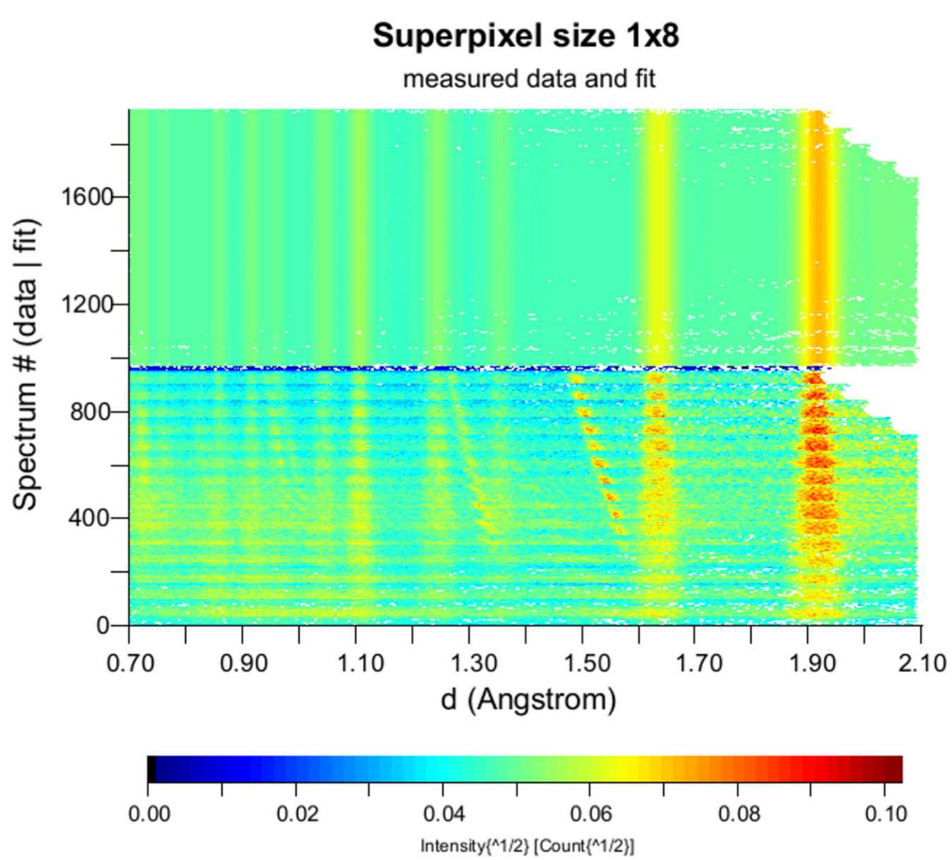
Effect of binning



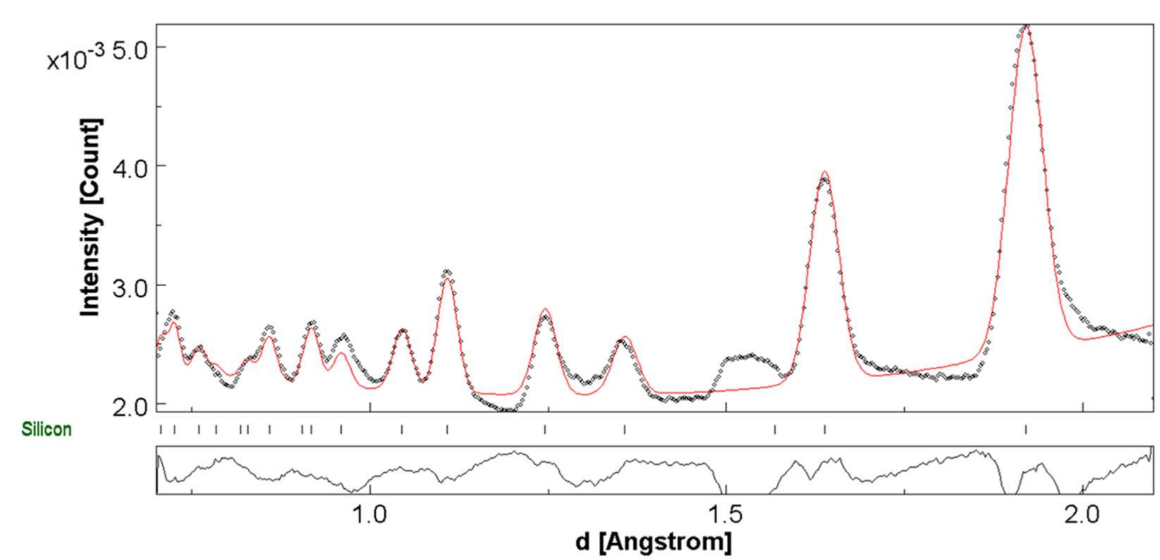
(*a*)



(*b*)

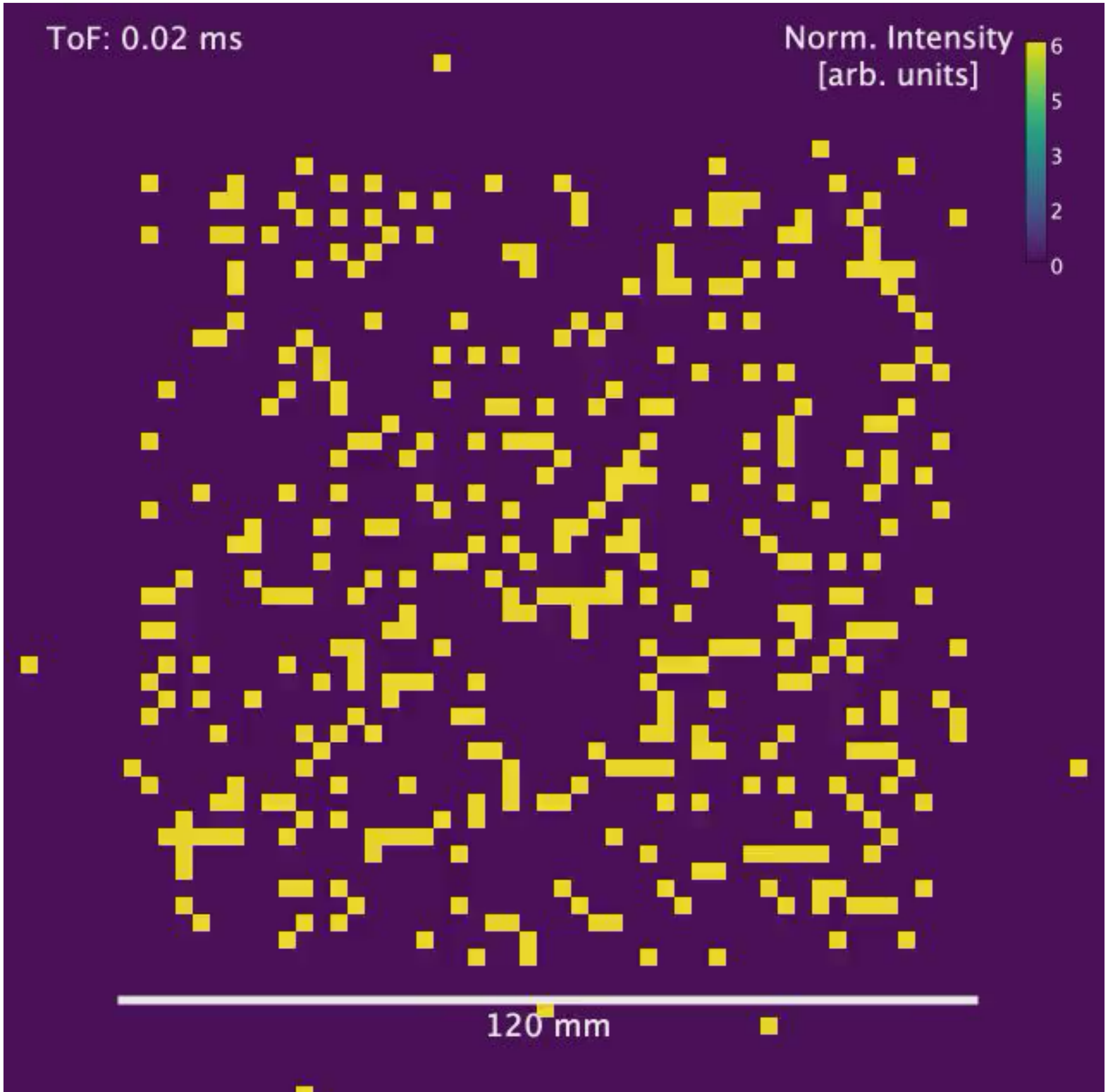


(*c*)



(*d*)

Sven: “A movie of uranium like no other, nobody has seen uranium before this way...



Sven: “A movie of uranium like no other, nobody has seen uranium before this way...