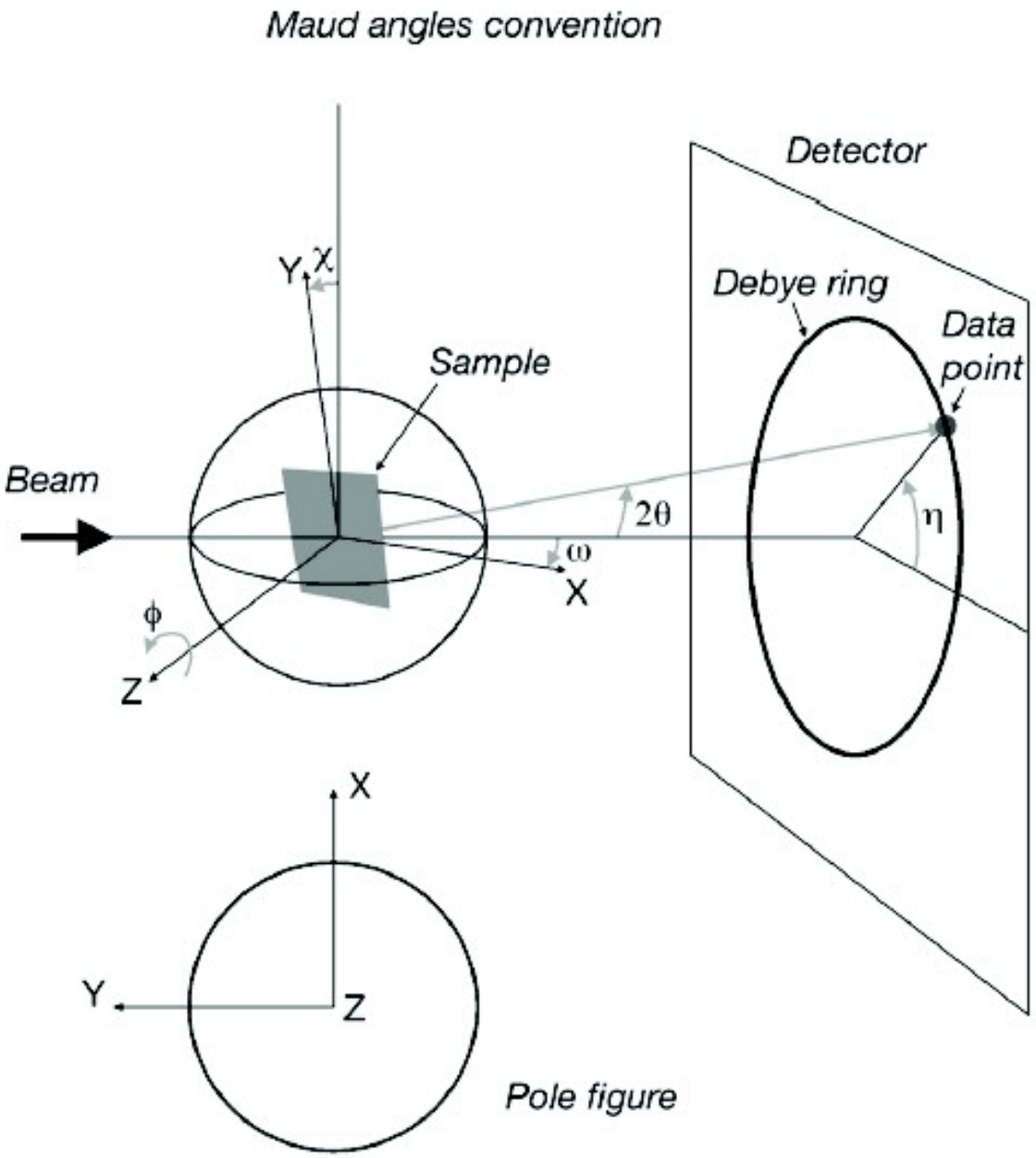
MgFeO XRD-2D combined analysis using MAUD

- macro-strain
- texture
- crystallite size
- micro-strain

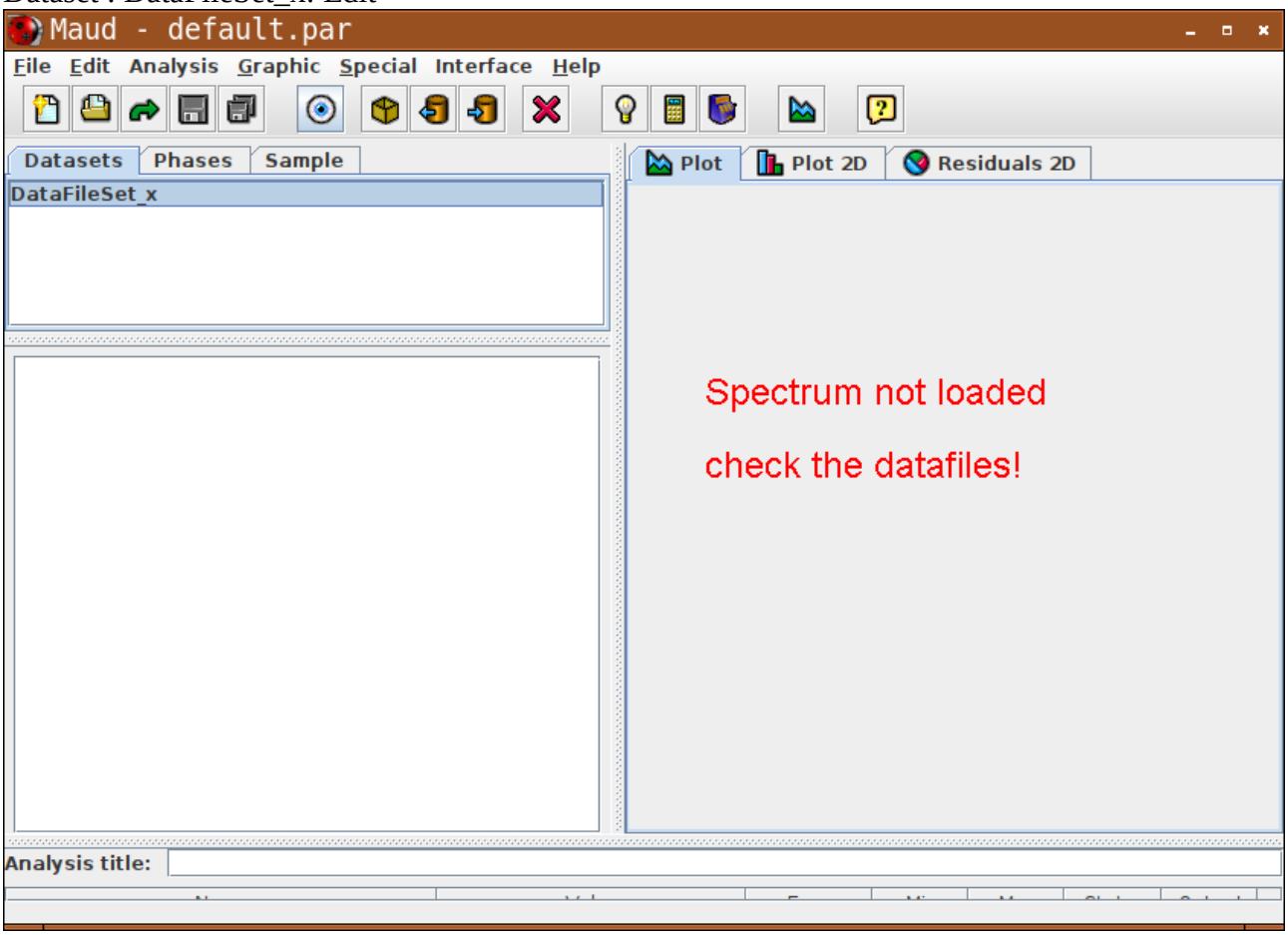
a tutorial transcription from the XRD school in Trento December 2018



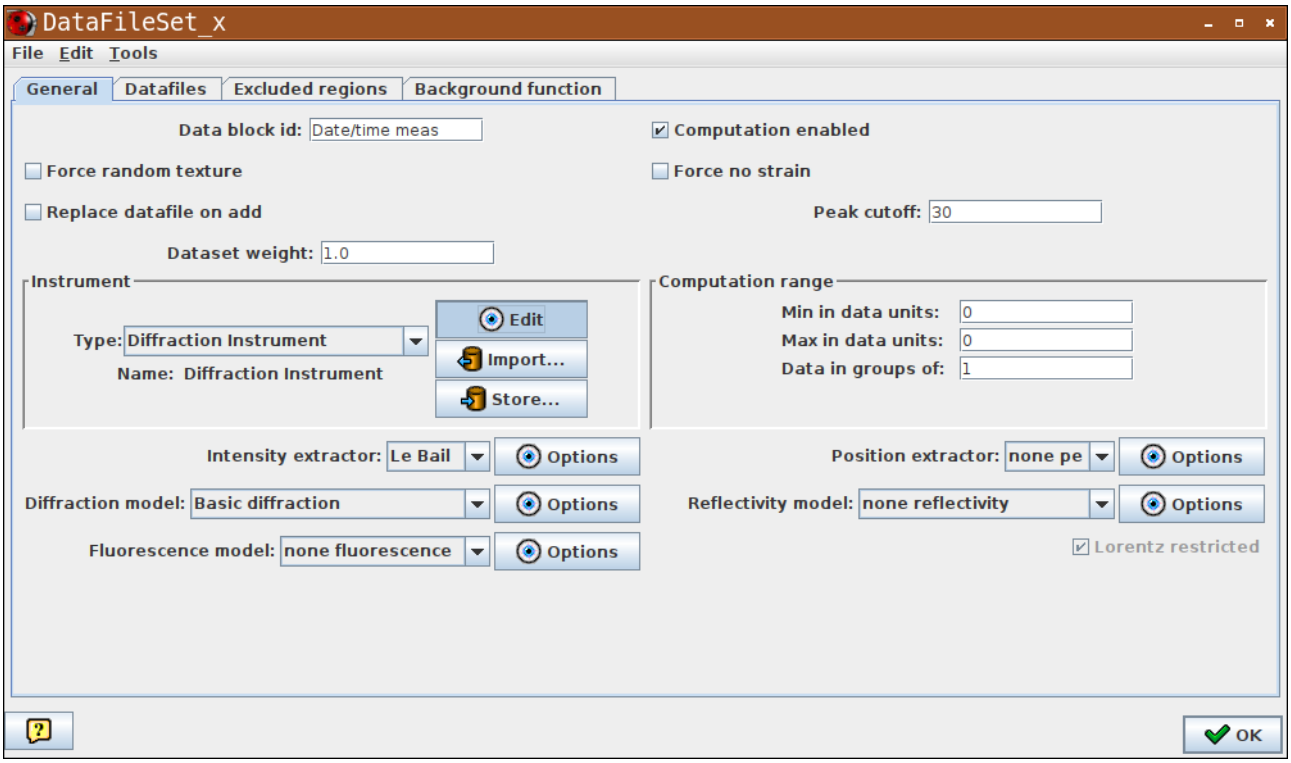
MAUD image 2D calibration

1) Instrument calibration:

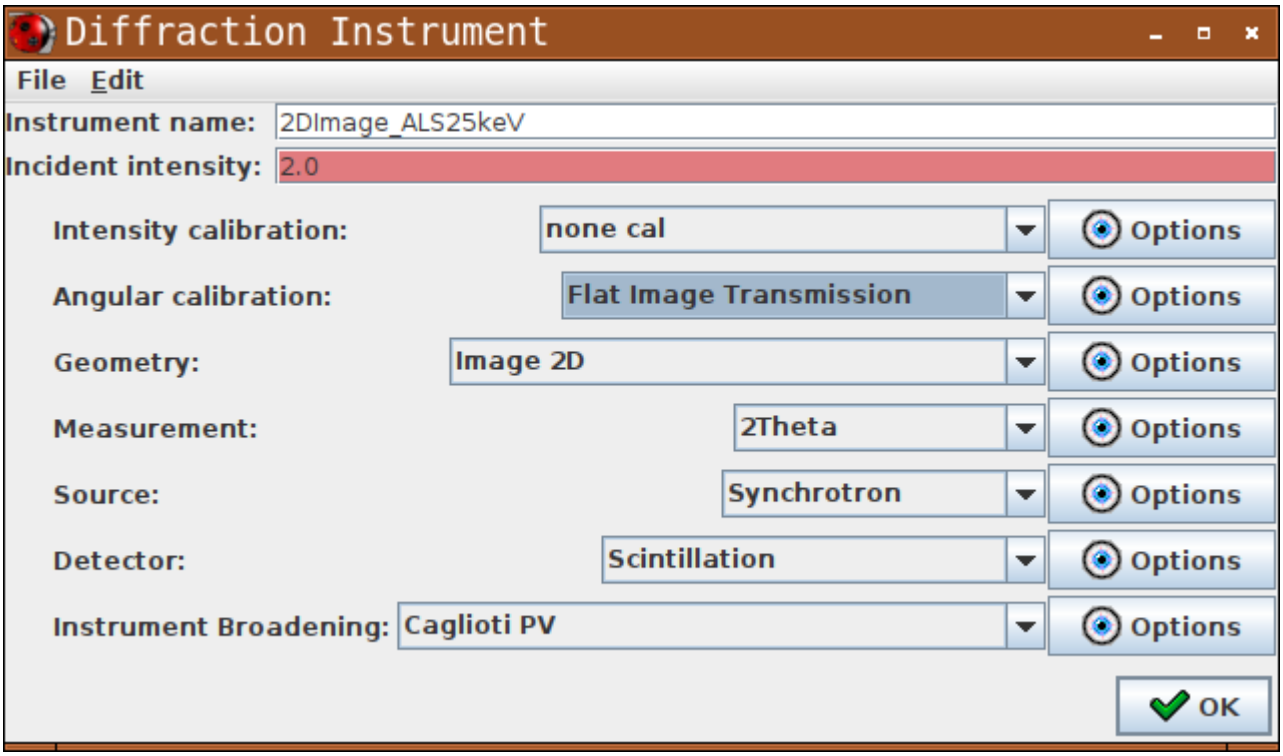
Dataset : DataFileSet_x: Edit



Instrument Edit

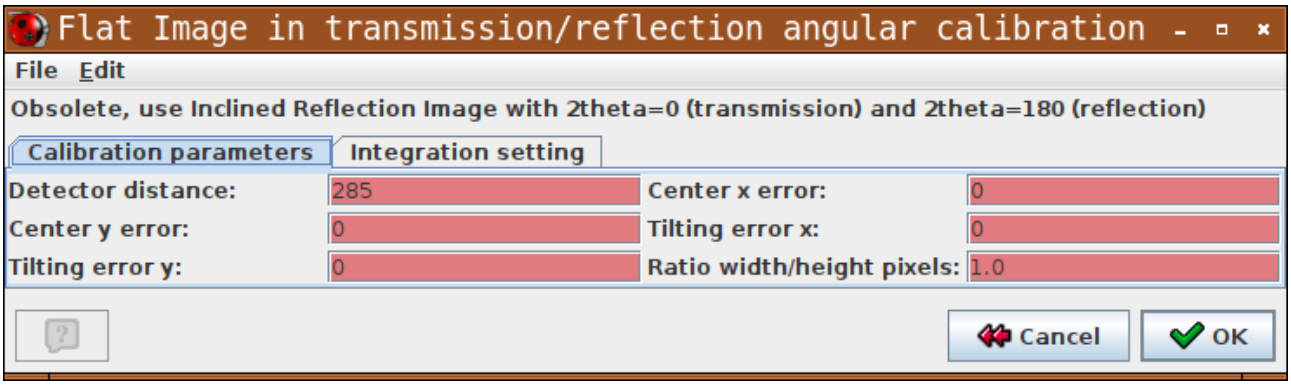


Instrument Name



Angular callibration: Flat Image Transmission

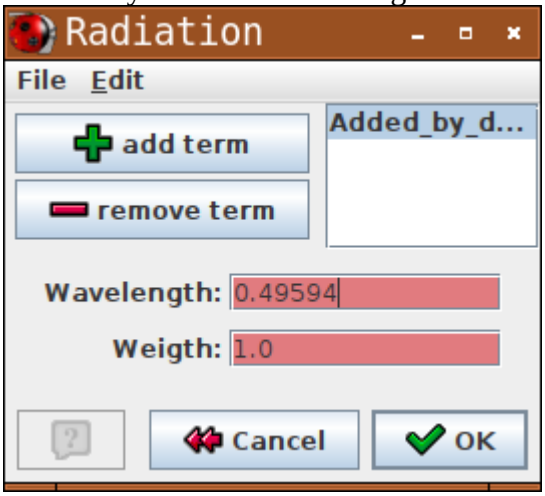
Detector distance: 285 mm



Geometry: Image 2D

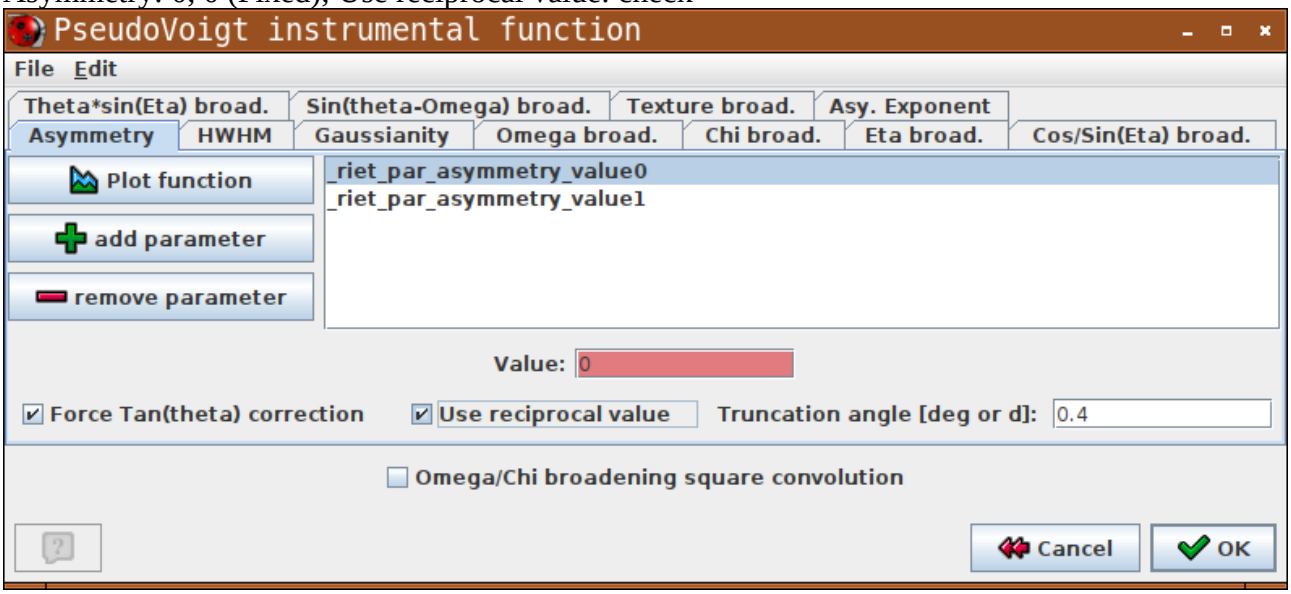
Measurement: 2Theta (means no scan, as opposed to Theta-2Theta)

Source: Synchrotron: wavelength: 0.49594 A (25 keV)

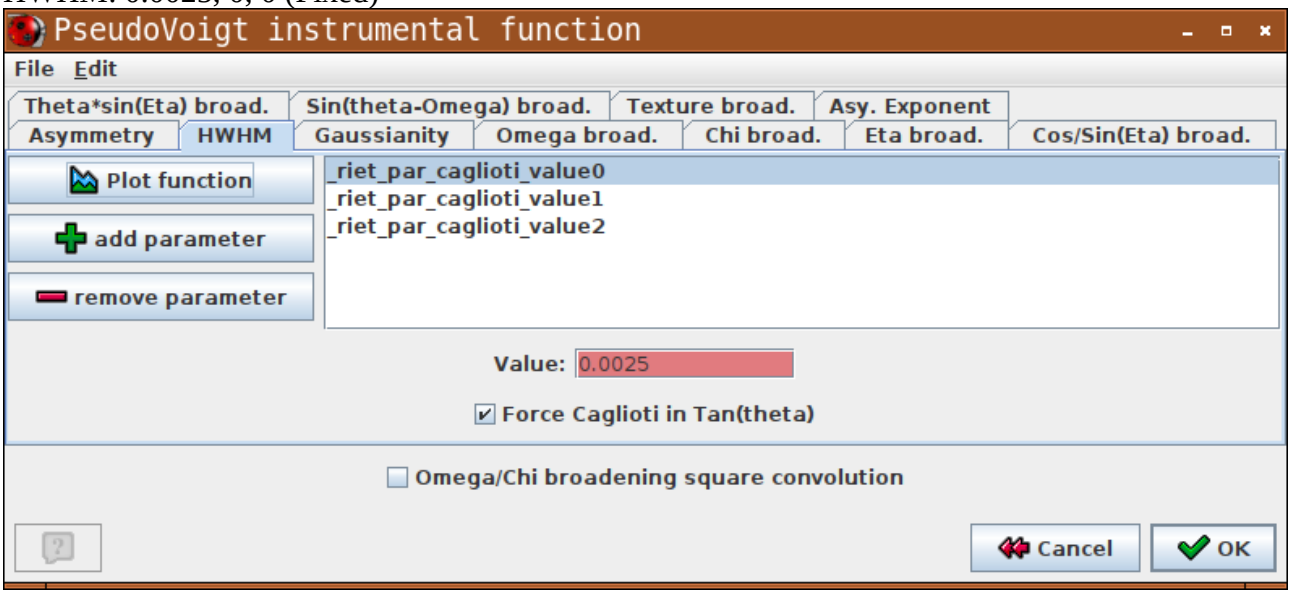


Detector: Scintillation (only change for XRF)

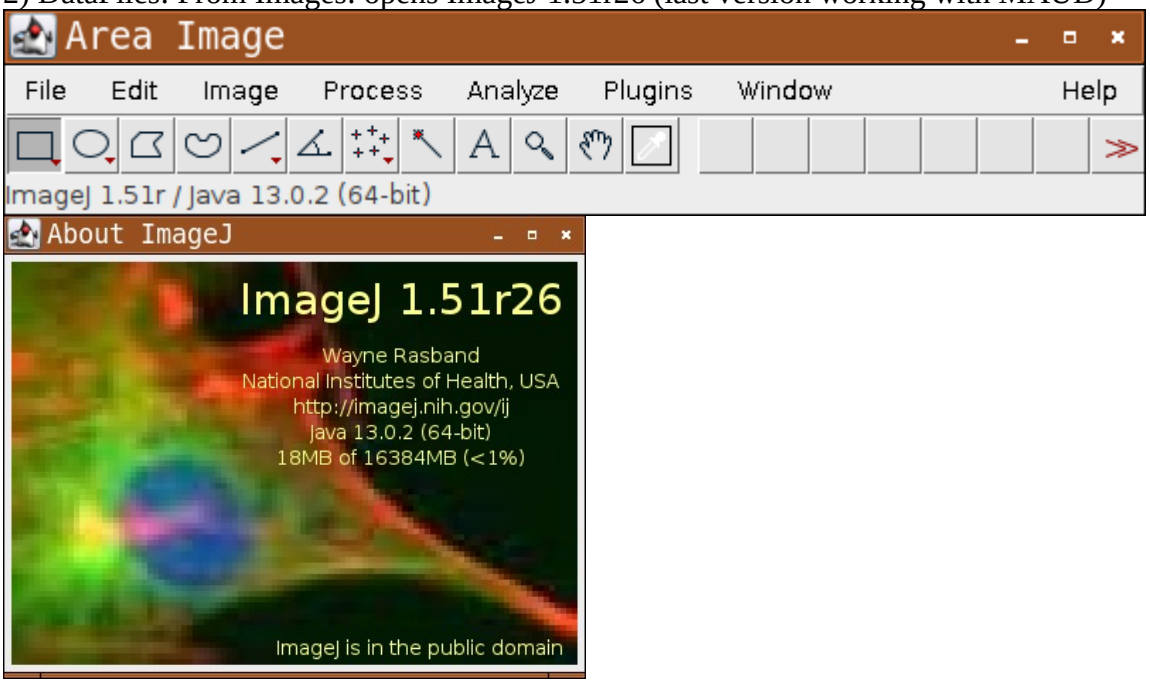
Instrument Broadening: Cagliotti PV
Asymmetry: 0, 0 (Fixed); Use reciprocal value: check



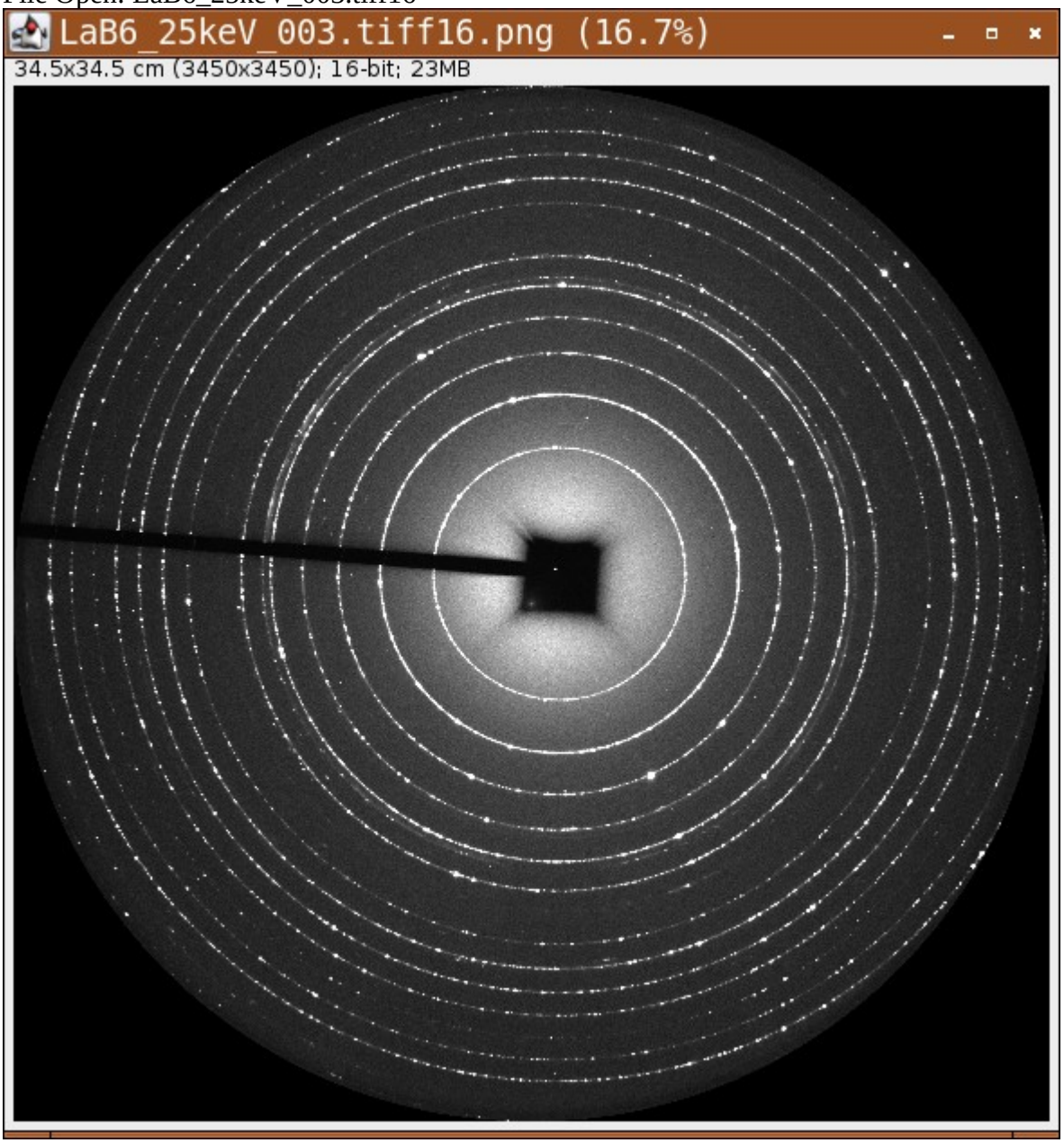
HWHM: 0.0025, 0, 0 (Fixed)



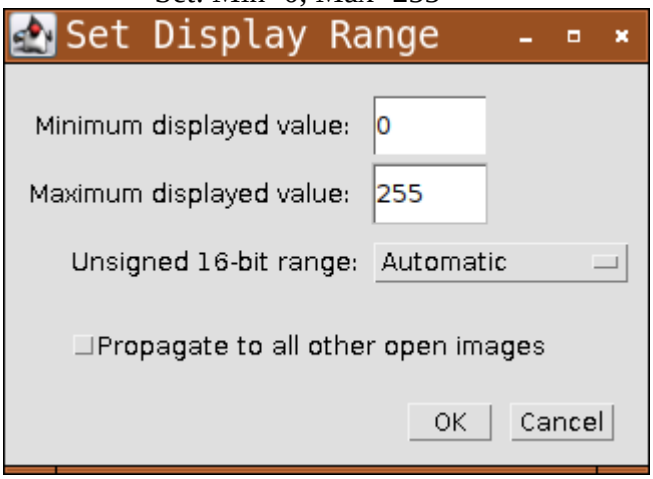
2) DataFiles: From Images: opens ImageJ 1.51r26 (last version working with MAUD)



File Open: LaB6_25keV_003.tiff16

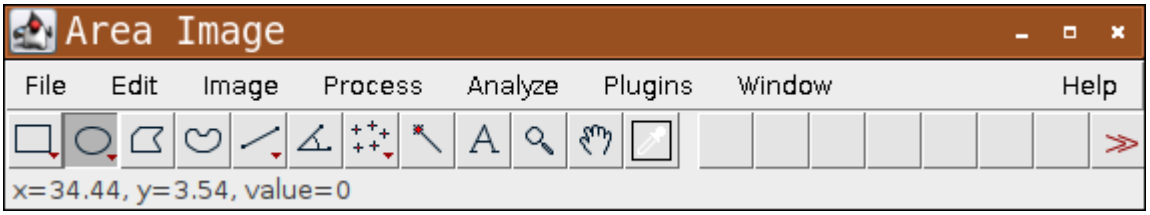


Image>Adjust>Brightness/Contrast:
Set: Min=0, Max=255

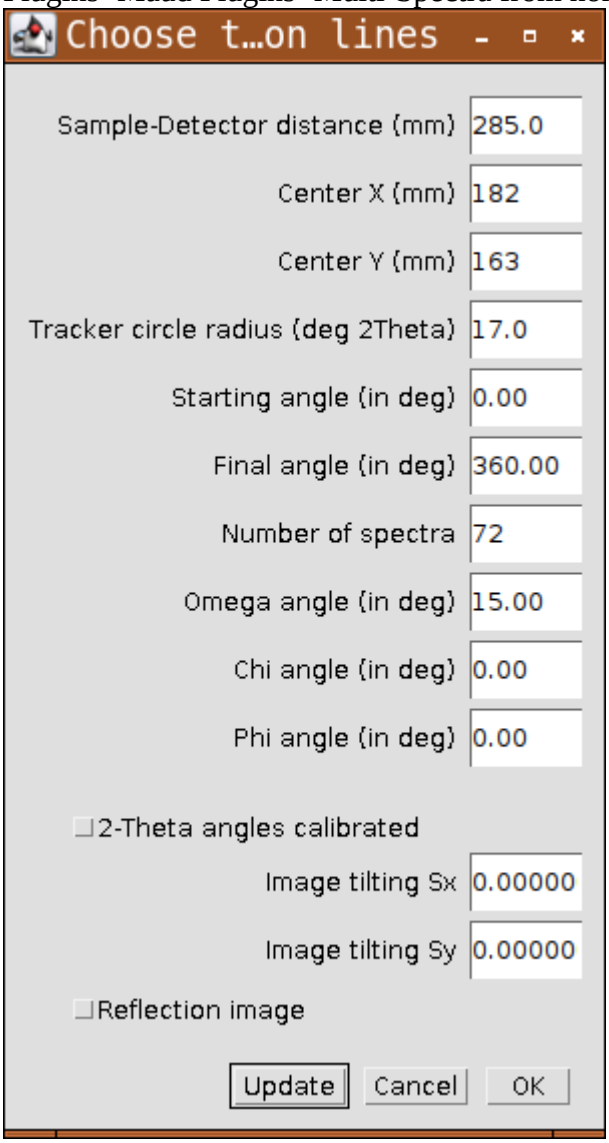


Check if the image has size in pixels and mm: we need the pixel size
Image>Properties:

Create a mask to avoid all pixel with value = 0 or value < 0
Use “Oval” selection tool

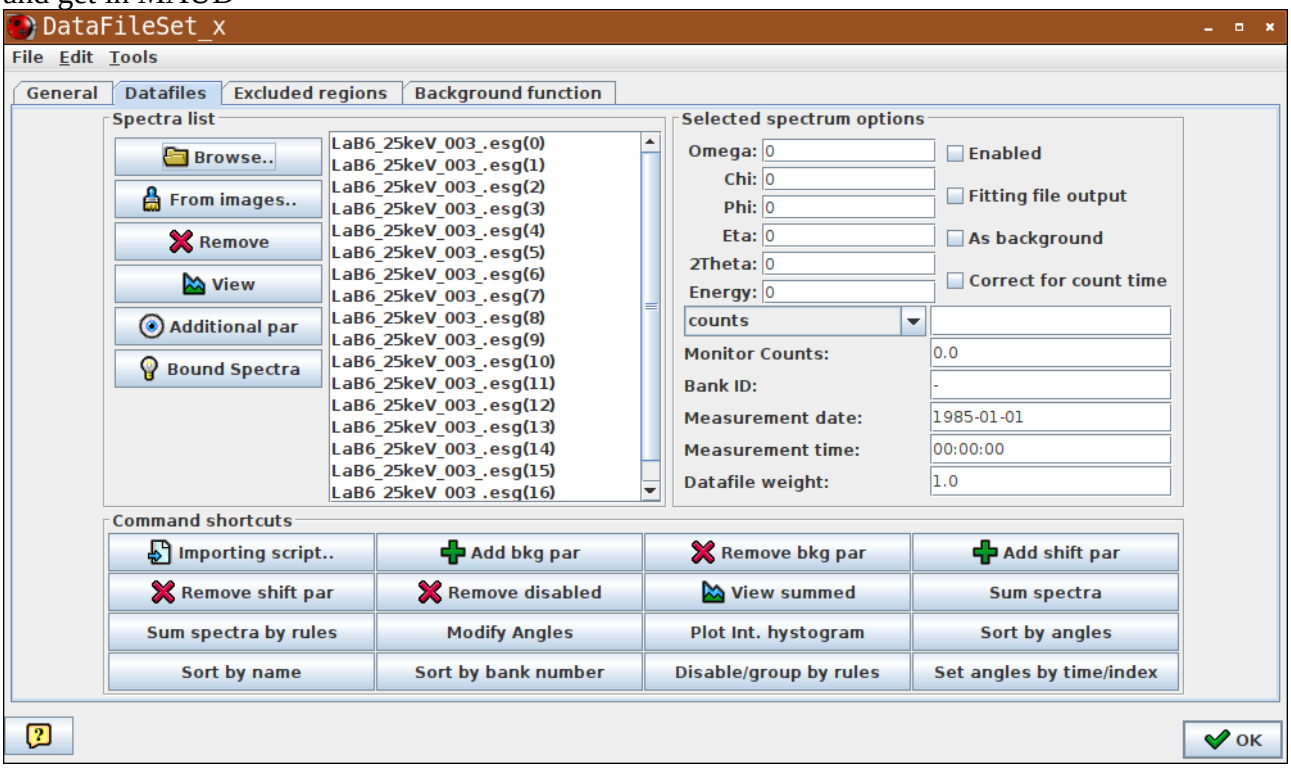


and adjust a circle a little bit inside the exposed area:
then,
Plugins>Maud Plugins>Multi-Spectra from normal transmission/Reflection Image

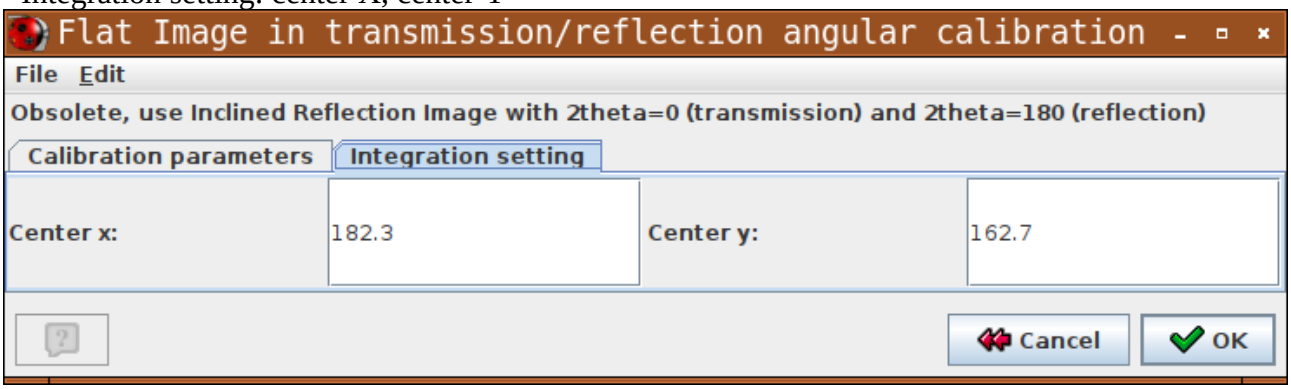


Distance: 285 mm
center X: 182.3 mm
center Y: 162.7 mm
Tracker circle radius: 17.0 deg (2tht for LaB6)
[Update]
Integrate from 0 deg to 360 deg, 18 spectra
For capillary: omega=0, chi=0, phi=0
(for slabs: explanation in 20181129_143839-46-cont.mp4 at t=00:28:30)
[OK] , wait for integration and save with “.esg” extension
Close IamegeJ, Don’t save image changes

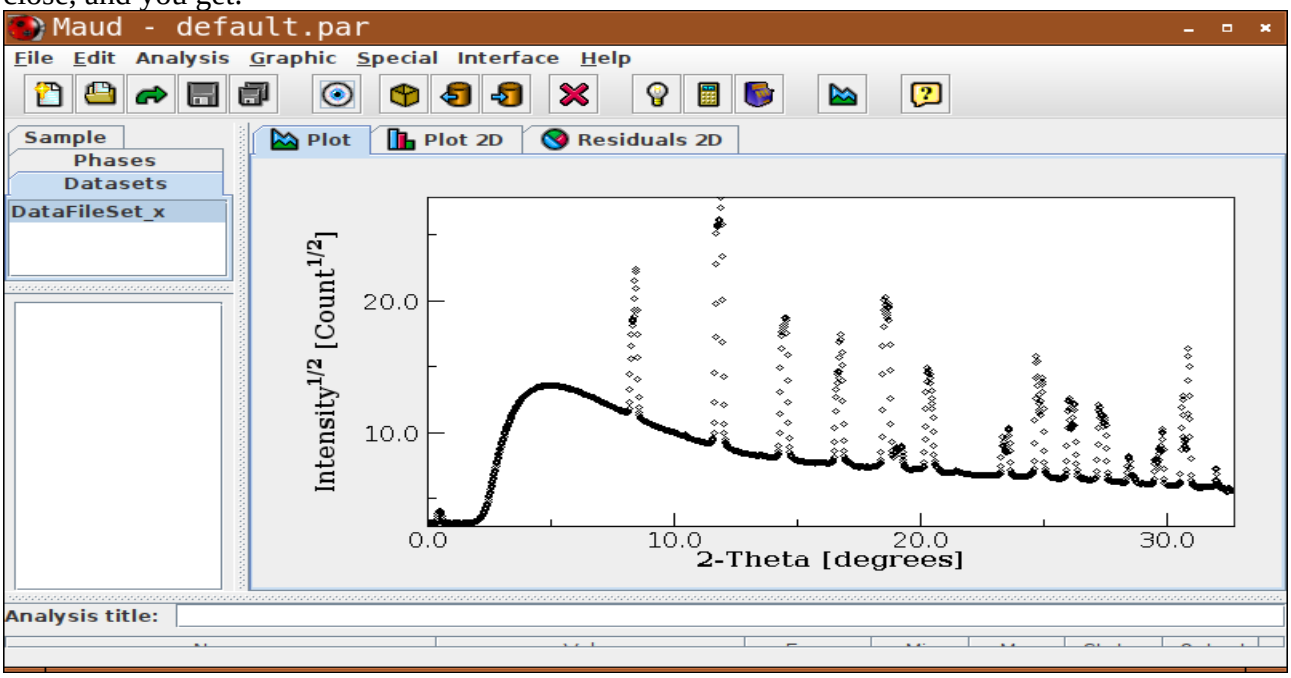
and get in MAUD

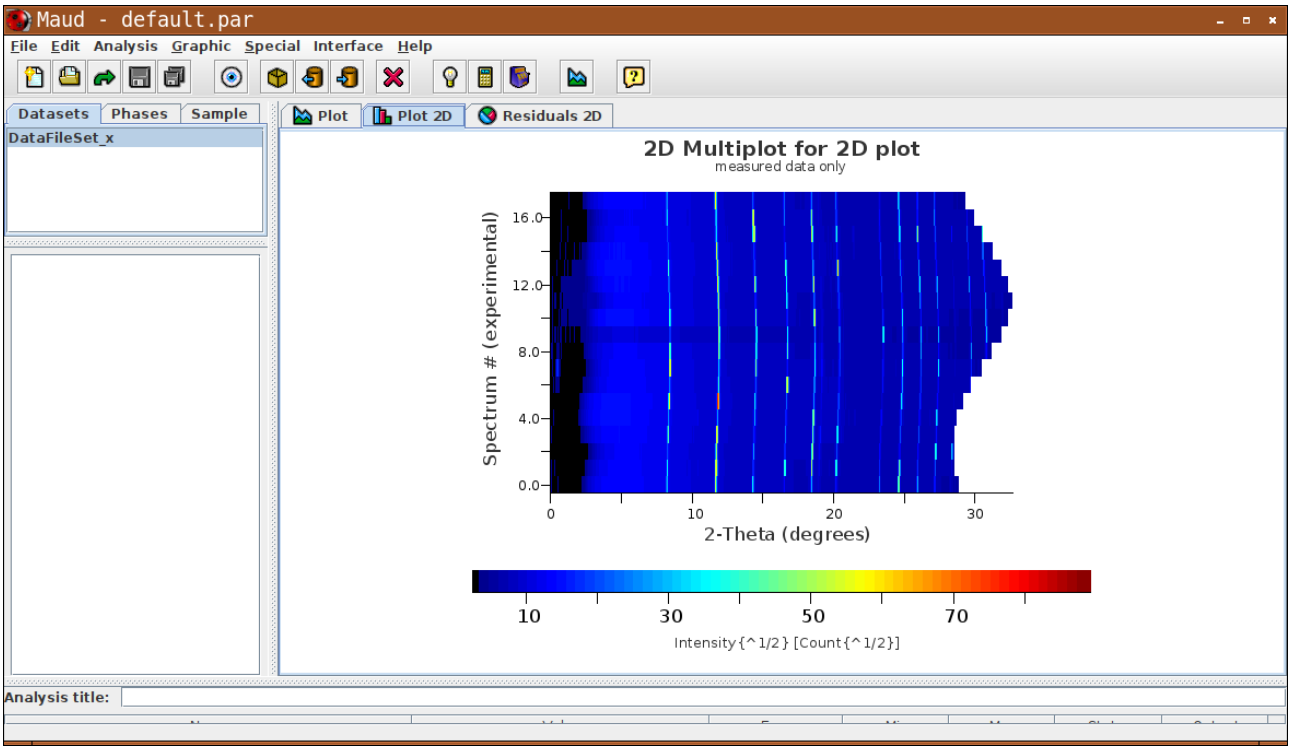


then check:
General>Instrument>Edit>Angular Calibration>Flat Image Transmission>Options>
>Integration setting: center X, center Y



close, and you get:

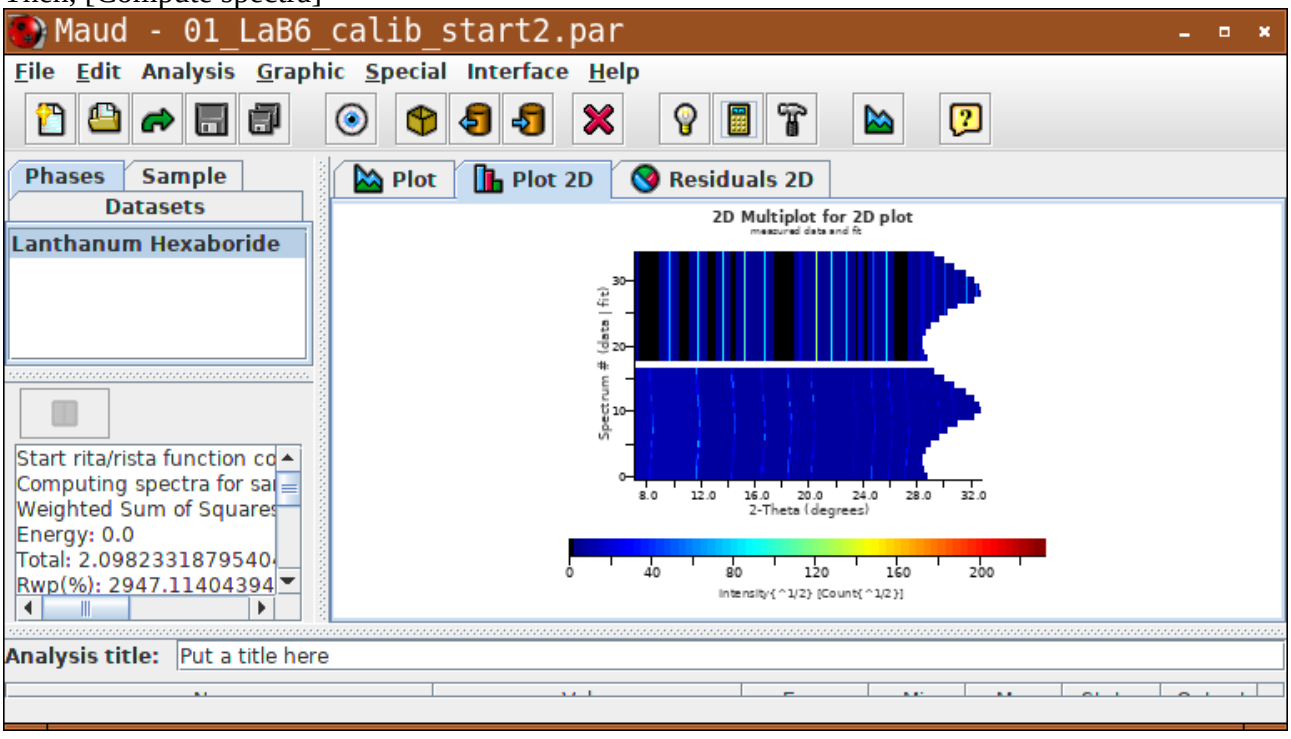




and in [Plot 2D] one can see the beam-stopper in spectrum #9
DataFiles> file .esg(9) > Enable : check disable
Also observe small deviation due to centering

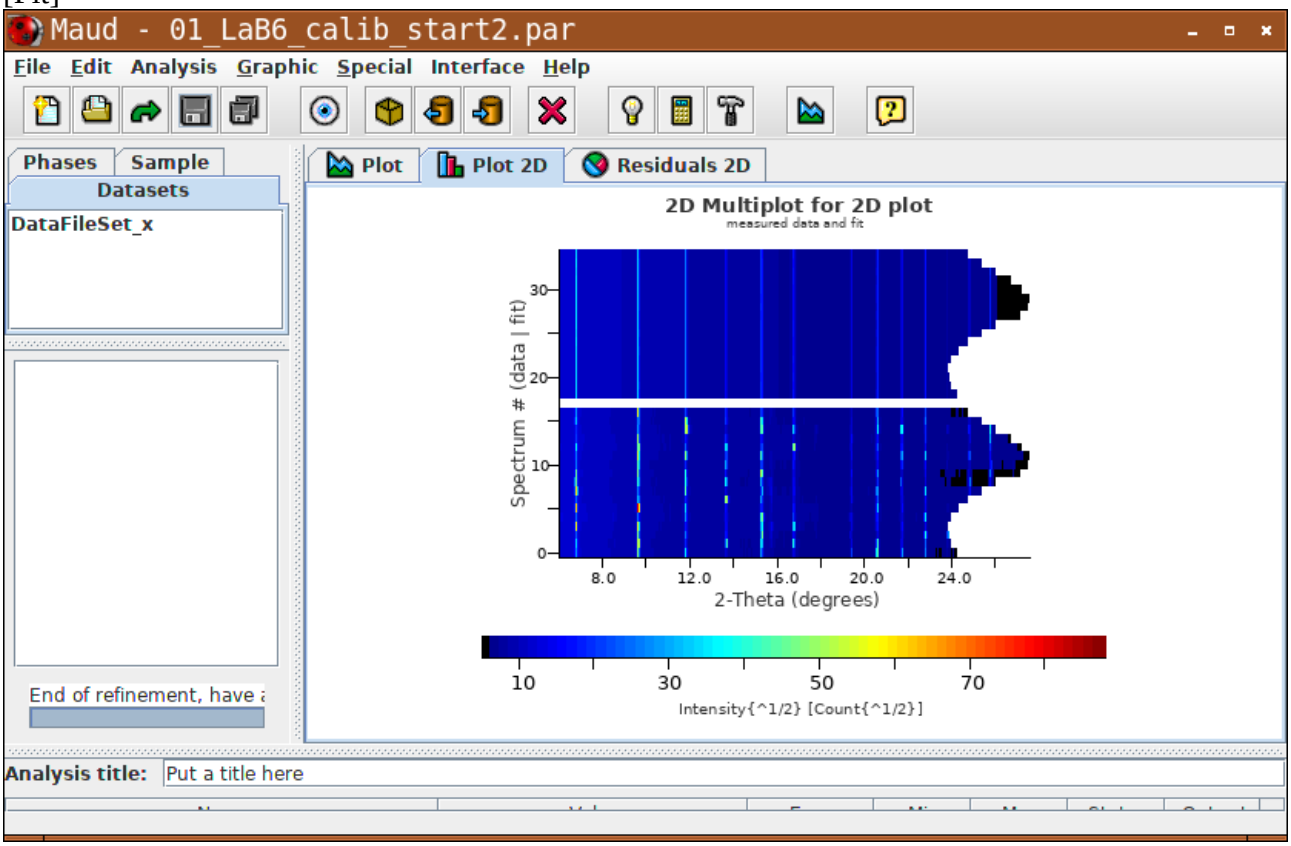
Datasets> General > Range of Computation> Min in data units: 6 and !!! Max : 40
(if you leave Max: 0, the program will stuck !!!)

Phases>Load from CIF: LaB6.cif Lattice value: 4.15689 A
Fix lattice value and refine detector distance and centering (also tilt), and instrument broadening
To fit instrument broadening:
Phase>Edit>Microstructure> Line broadening model>None
or
Line broadening model>Delft
and Size-strain model>Isotropic>Options> 0, 0
Then, [Compute spectra]



We observe that the distance is not good: change to 350 mm and calculate again:
and start to refine:
Background: normal one ~ 100
Intensity: (instrument edit) : decrease 20-40 times
Analysis> Parameter list> Expand All:
and [Free Backgrounds]
instrument> incident intensity: refine! (_pd_proc_intensity_incident)
Flat Image Transmission: refine!:
distance, center x,y, tilt x,y
Image 2D: polarisation effects for highly polarized beam
(in the vertical direction, or horizontal at high angles; otherwise: 0,0)

[Fit]



Try to fit the grain-iness using arbitrary texture (not actually texture but arbitrary intensity)
Phase> Edit>Advanced model>Texture>Arbitrary tex
before Fit, fix incident intensity !!!!
[Fit] again
Next, zoom a peak and observe the asymmetry:
Instrument>Cagliotti>Asymmetry 1st parameter refine
and Truncation angle: set negative! -0.4
means, asymmetry increase toward higher angles (for 2D detectors)
[Fit]
Refine 2nd asymmetry parameter
Refine HWHM Cagliotti parameters
[Fit] :
Refine Gaussian Cagliotti parameters
[Fit] :
Dataset>Edit>Exclude region>Add> from 15.5 to 16.0

Analysis>Parameter List>Fix All Parameters
Dataset>Instrument>Save>LaB6_ALS_image2D.ins

2) MgFeO Magnesiumwuestite high pressure

New Analysis

Dataset>Instrument>Import>LaB6_ALS_image2D.ins

Dataset>DataFiles>From Image>ImageJ

ImageJ: File>Open>MgFeO_25keV_006.tiff16

ImageJ: Image>Adjust>Brightness/Contrast: [Set] min=0, max=255

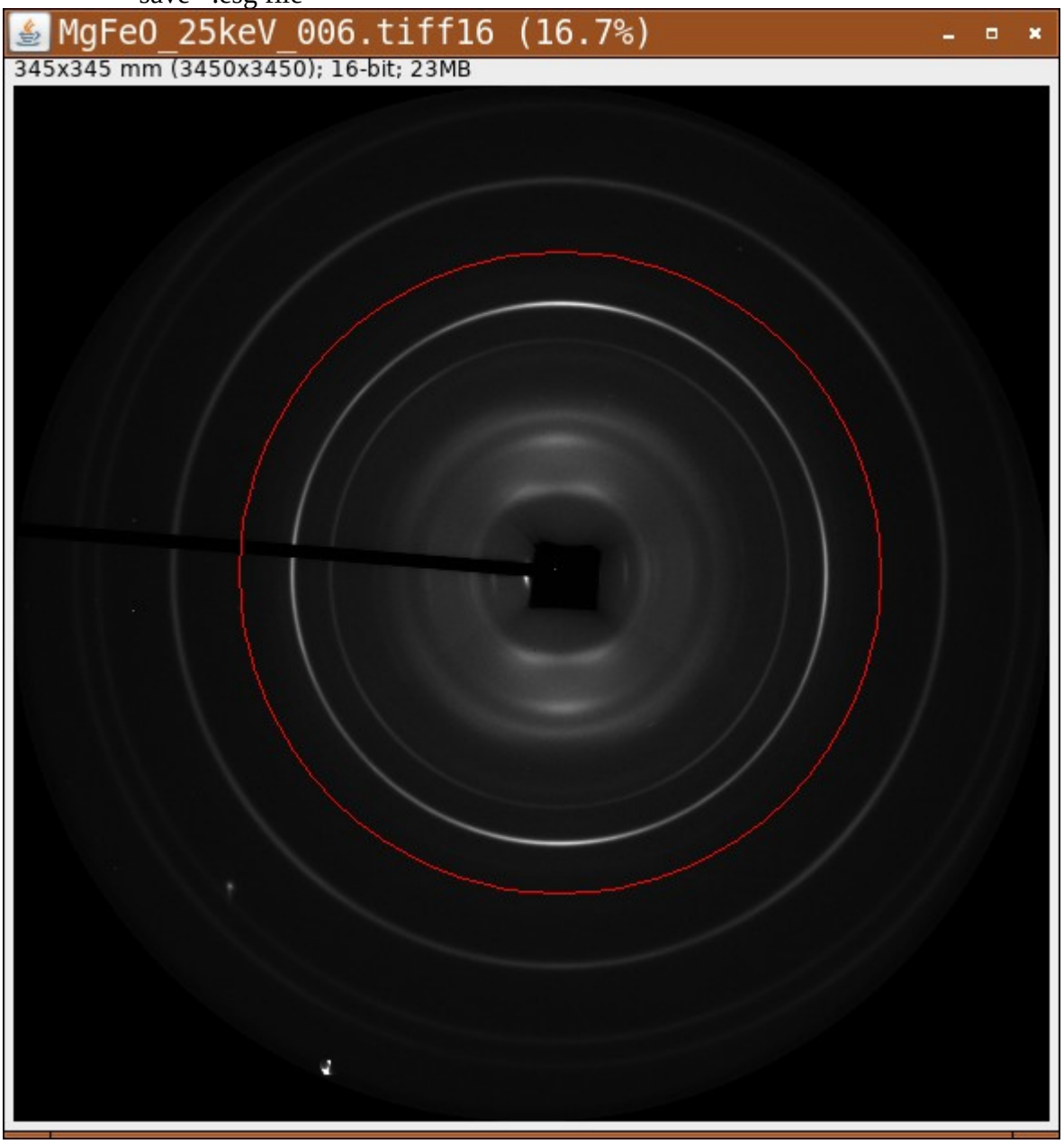
ImageJ: Toolbar: Oval: Select exposed area

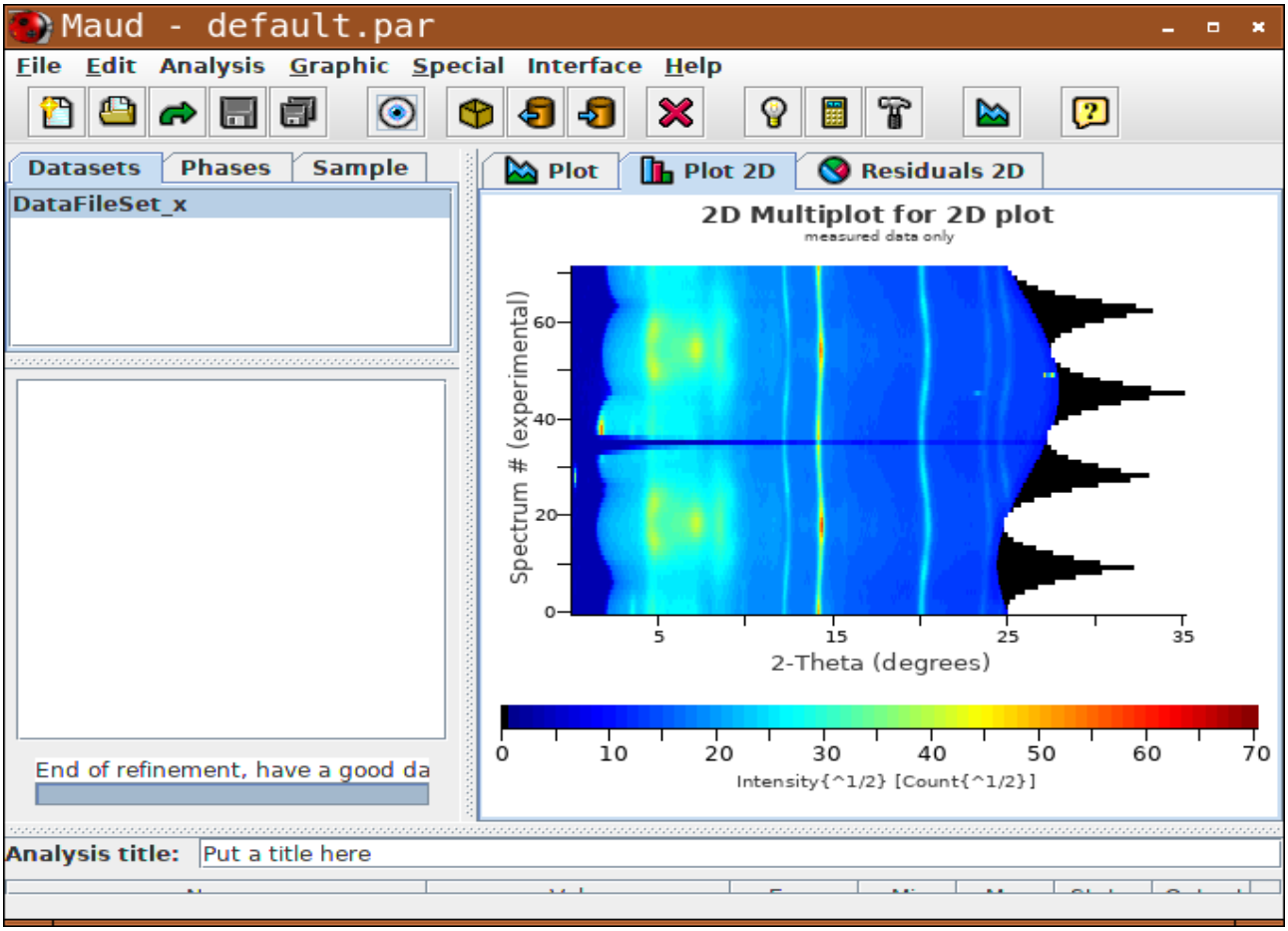
ImageJ: Plugins>Maud Plugins>Multi-Spectra from normal transmission/Reflection Image

Number of spectra: 72 (every 5 deg)

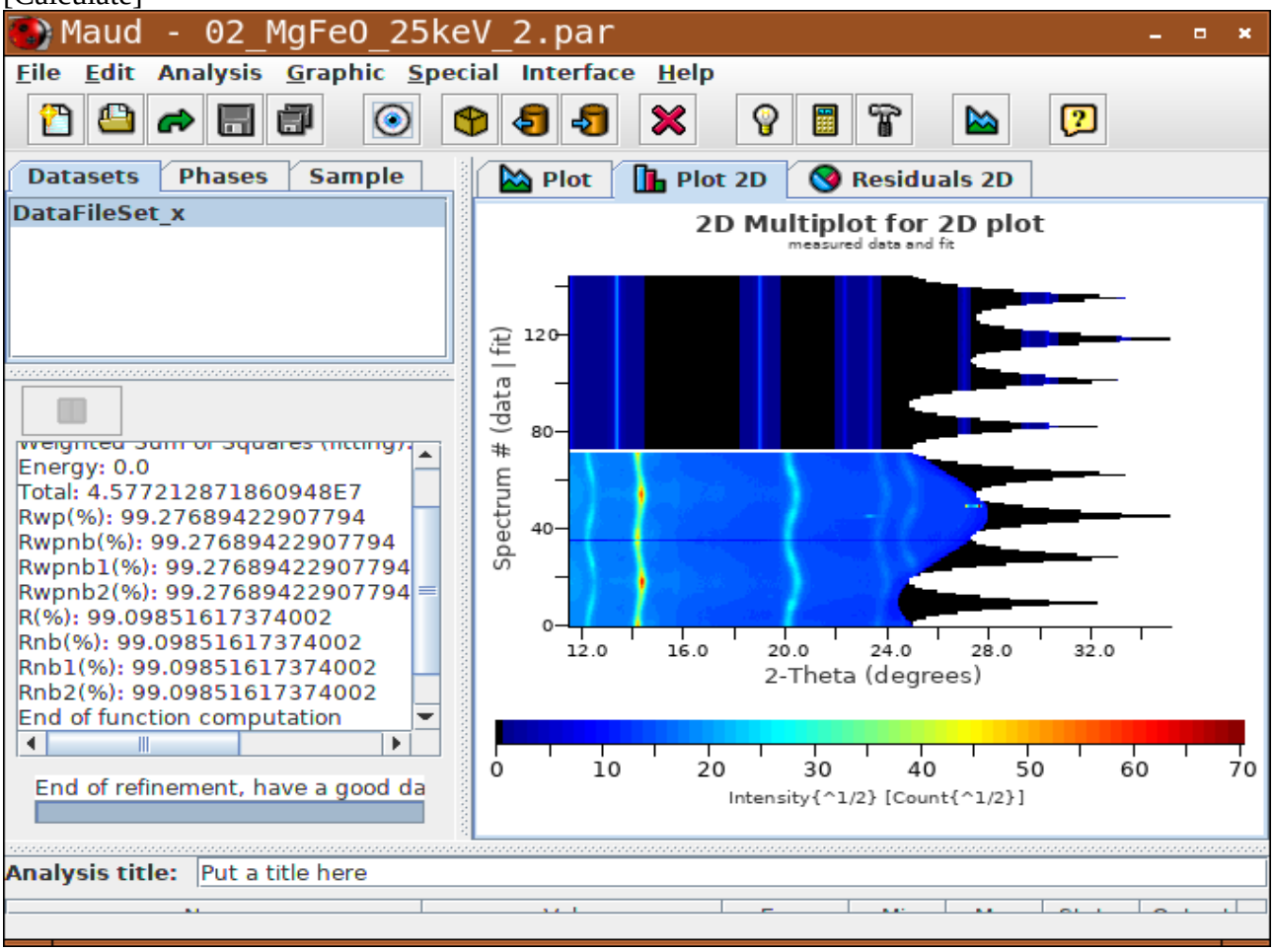
[ok]

save *.esg file

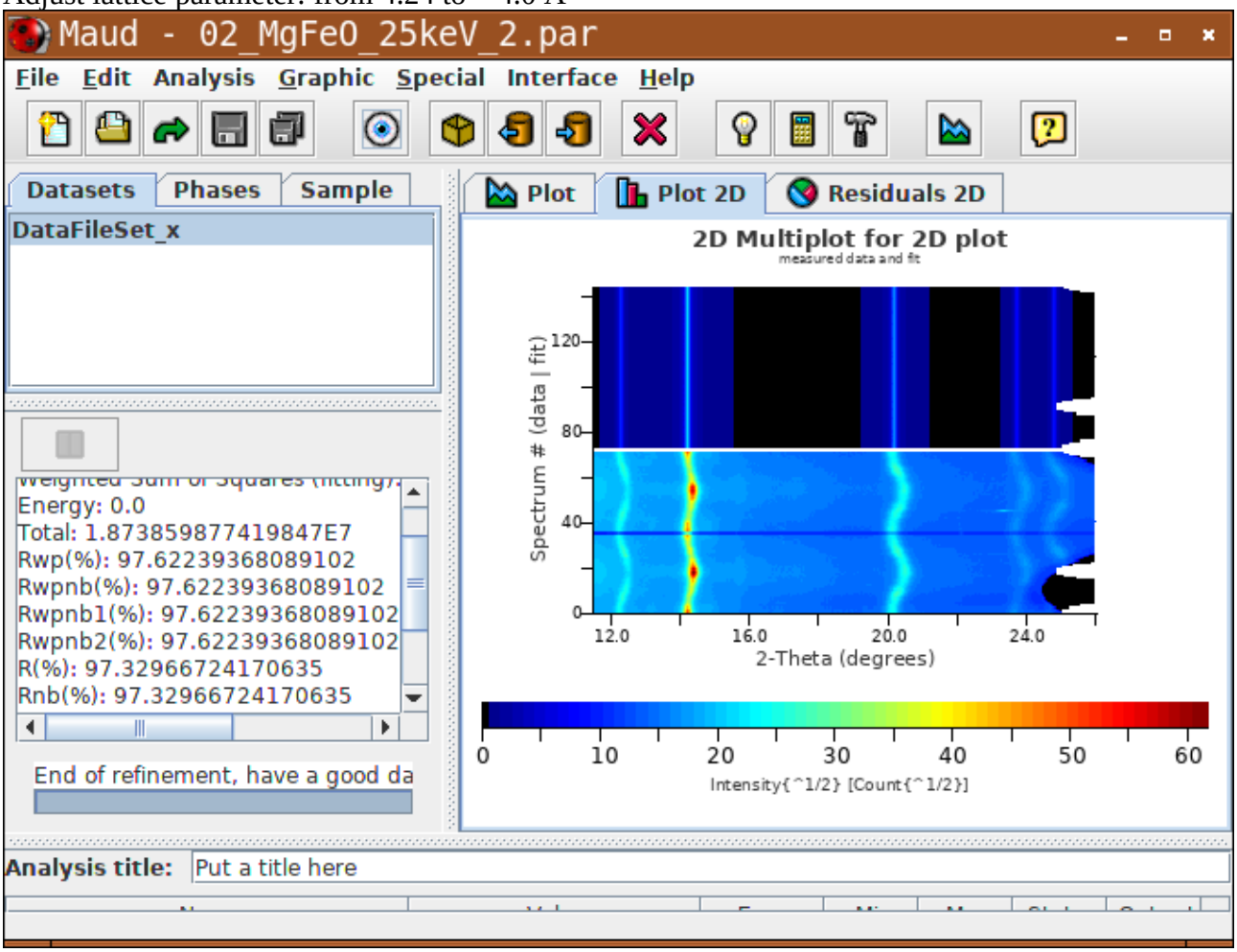




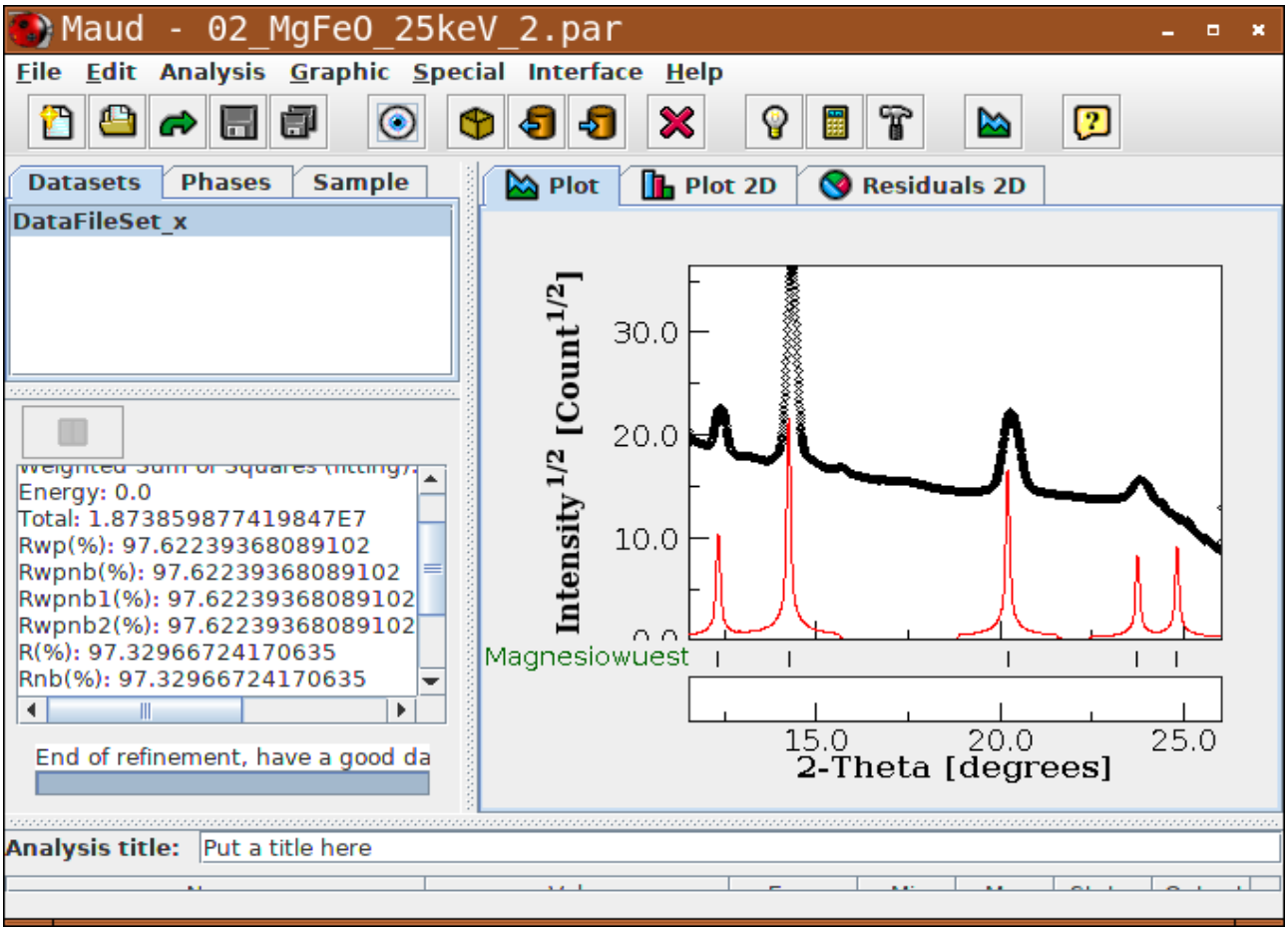
Back in MAUD
Zoom in at first line: change dataset range: min=11.5, max=50
Phase>Import>MgFeO.cif
[Calculate]



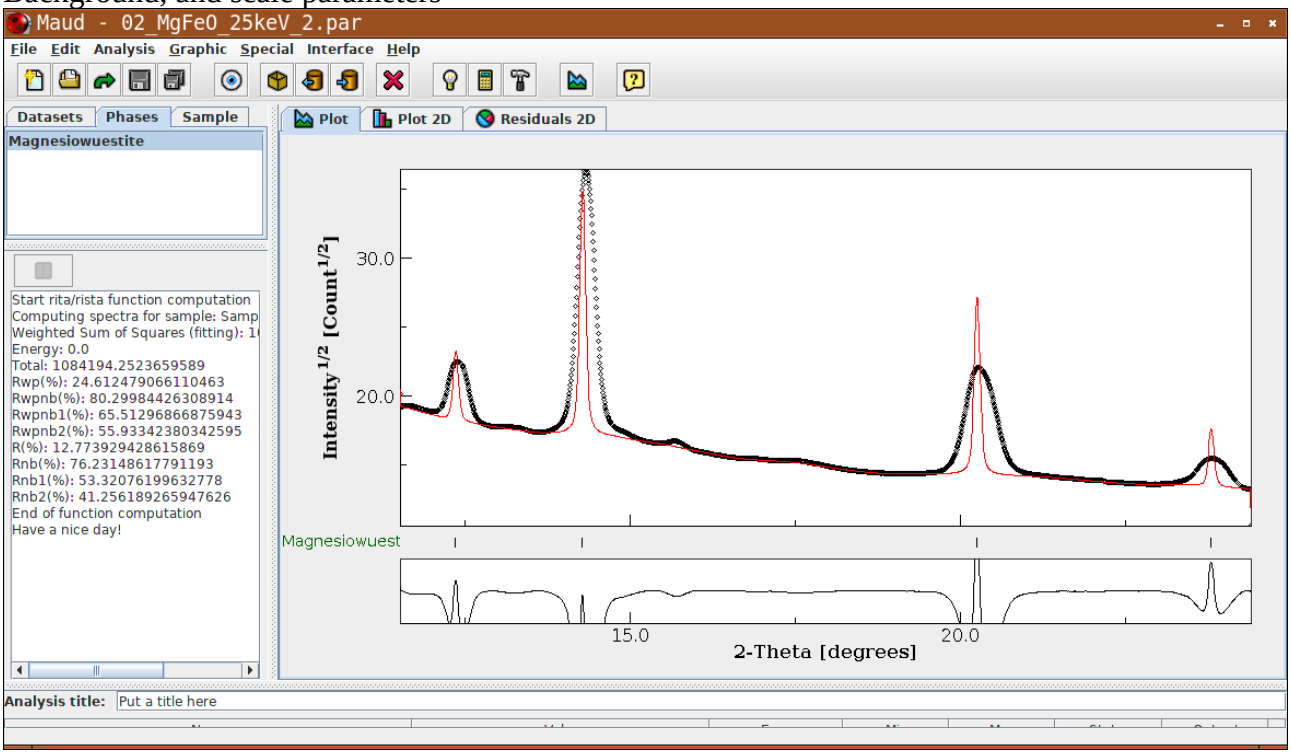
Shift is caused by pressure: 40 Gpa
Adjust lattice parameter: from 4.24 to ~ 4.0 A



Also in Plot 1D



Start analysing:
Background, and scale parameters



Remove beam-stopper: esg(35) disable

NEXT: Stress model

Phases>Edit>Advanced Models:

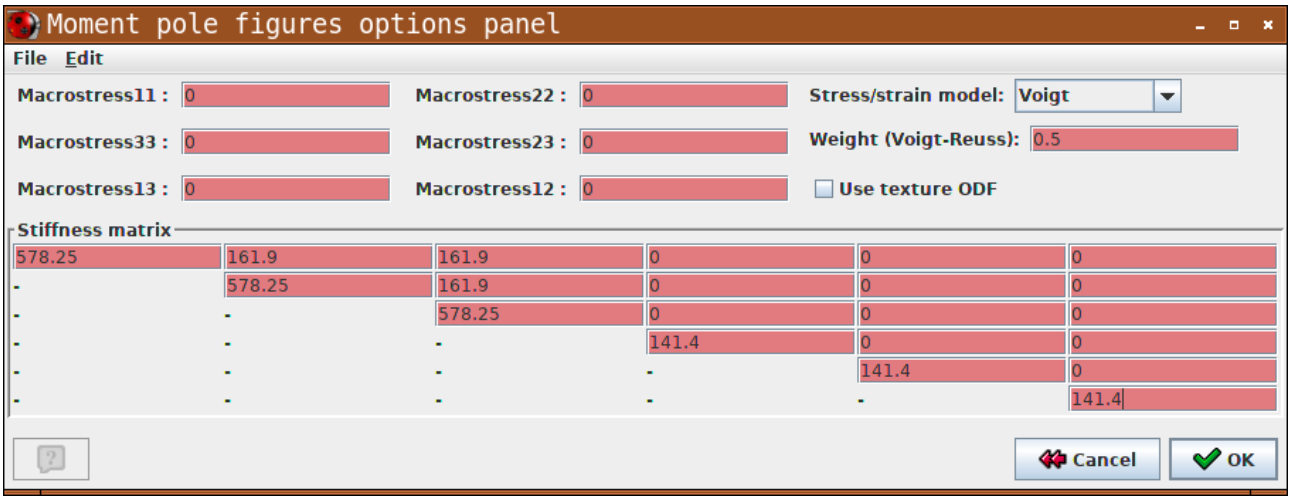
Strain: Moment Pole Stress > Options

> stiffness matrix at 40 gPa for MgFeO

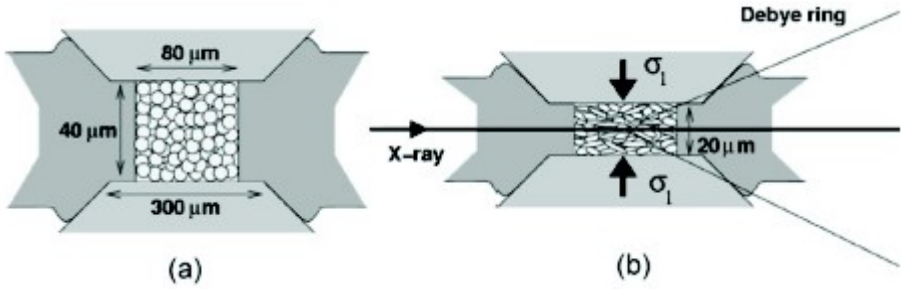
C11: 578.25
C12: 161.9
C44: 141.4

> hydrostatic stress (Macrostress)

S11=S22
S33= -2*S11
neglect shear: S12, S13, S23



"One question … From where do we get these value?"
"From literature ..."

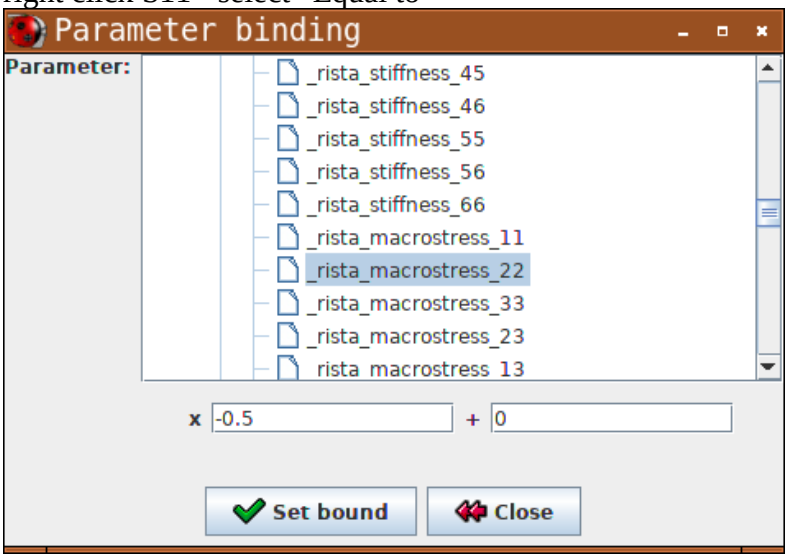


Differential stress:

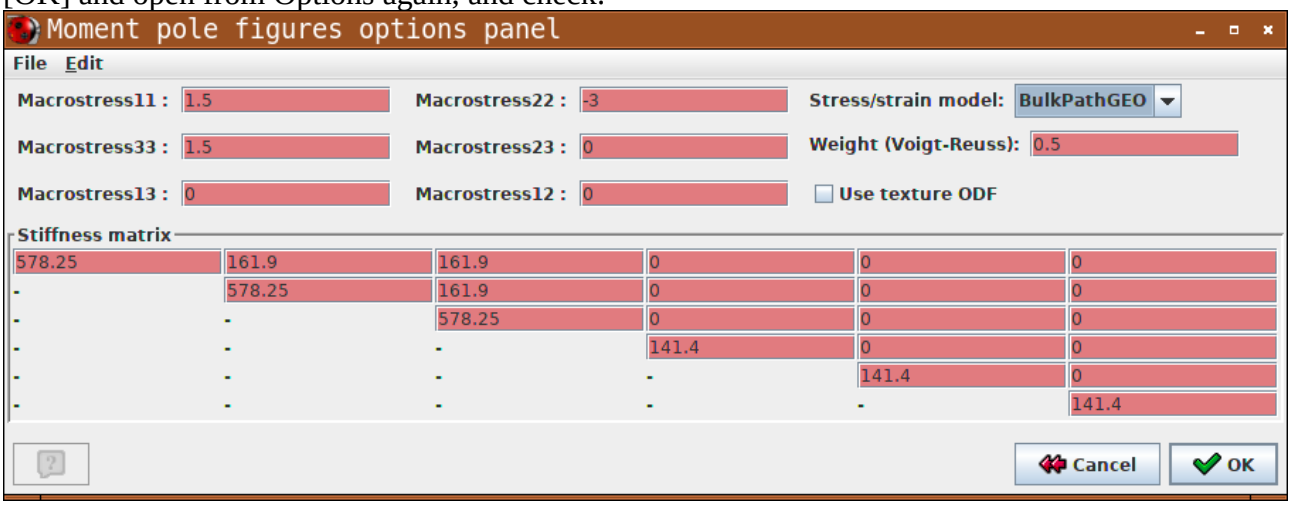
$$\sigma_{ij} = \begin{bmatrix} \sigma_p & 0 & 0 \\ 0 & \sigma_p & 0 \\ 0 & 0 & \sigma_p \end{bmatrix} + \begin{bmatrix} -t/3 & 0 & 0 \\ 0 & -t/3 & 0 \\ 0 & 0 & 2t/3 \end{bmatrix} = \sigma_p + D_{ij}$$

Let's say pressure along S22 = 3Gpa (same units as stiffness matrix) and Right-click: "refine"
Then S11 is minus half of S22

right click S11> select "Equal to"



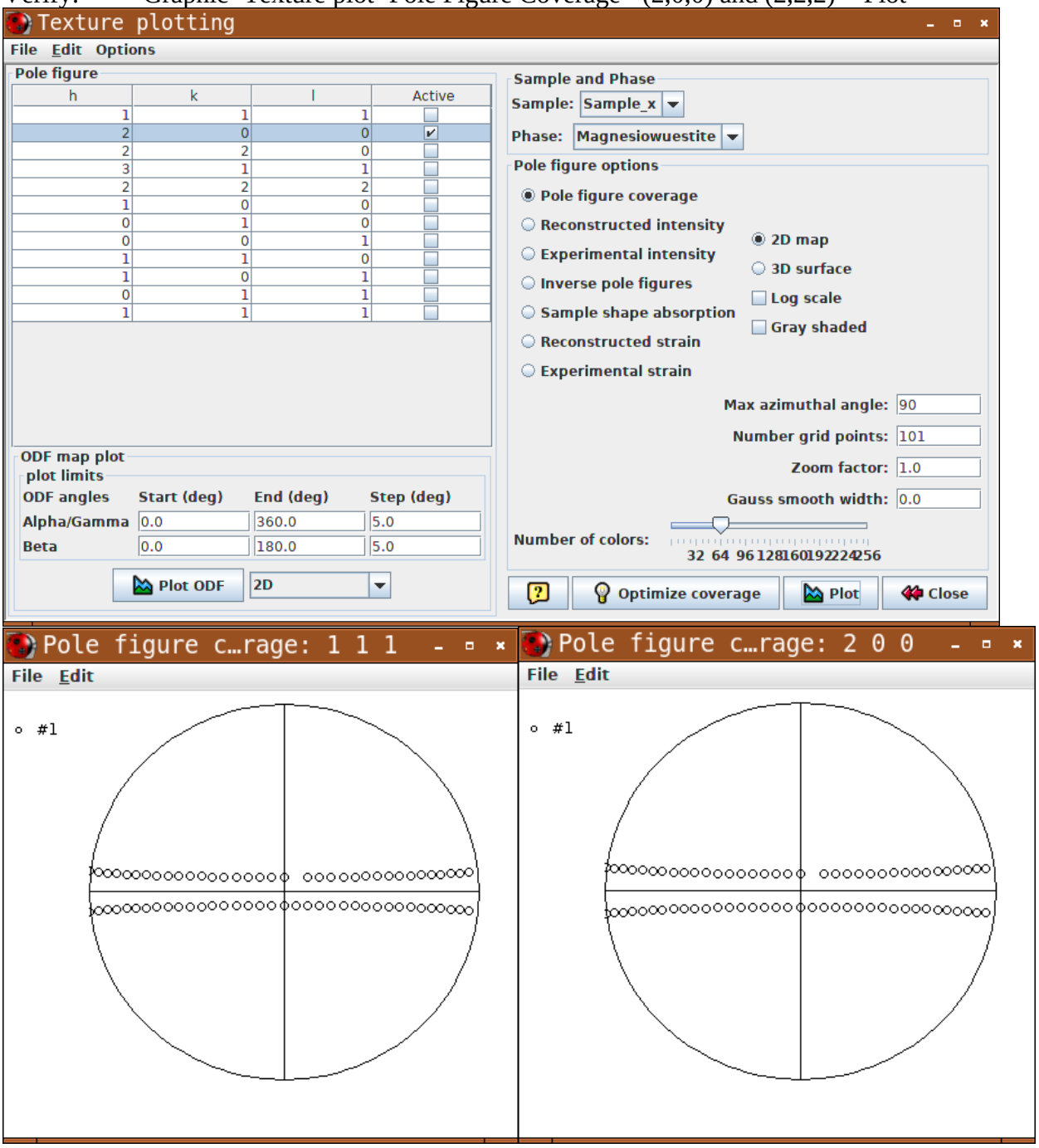
[Set bound]
Do same for S33
[OK] and open from Options again, and check:



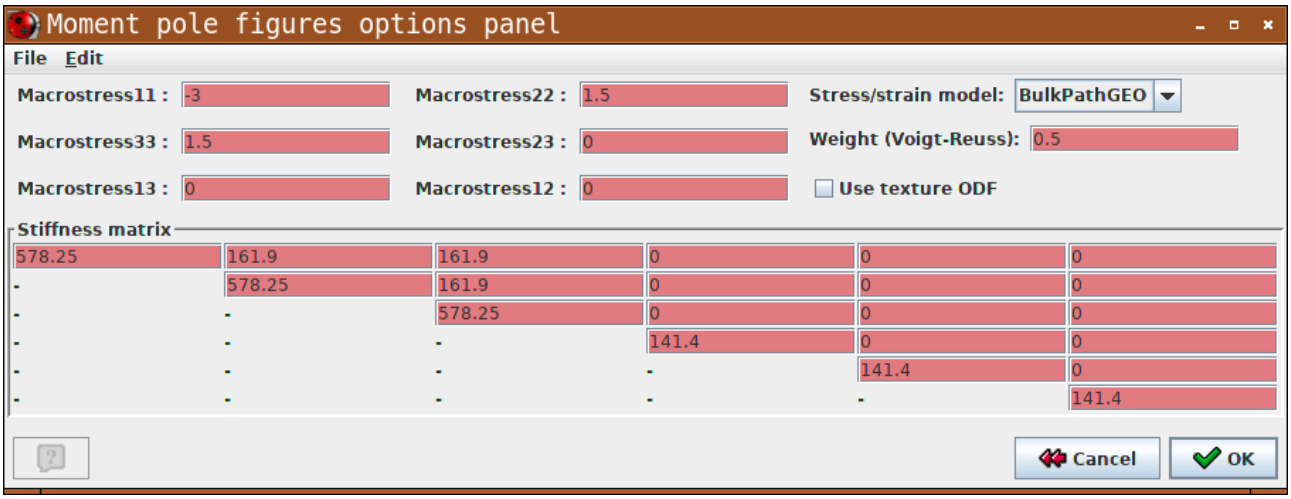
Stress/strain model: BulkPathGEO (geometrical mean path)

Refine also:
> lattice parameters
> crystallite size and micro-strain

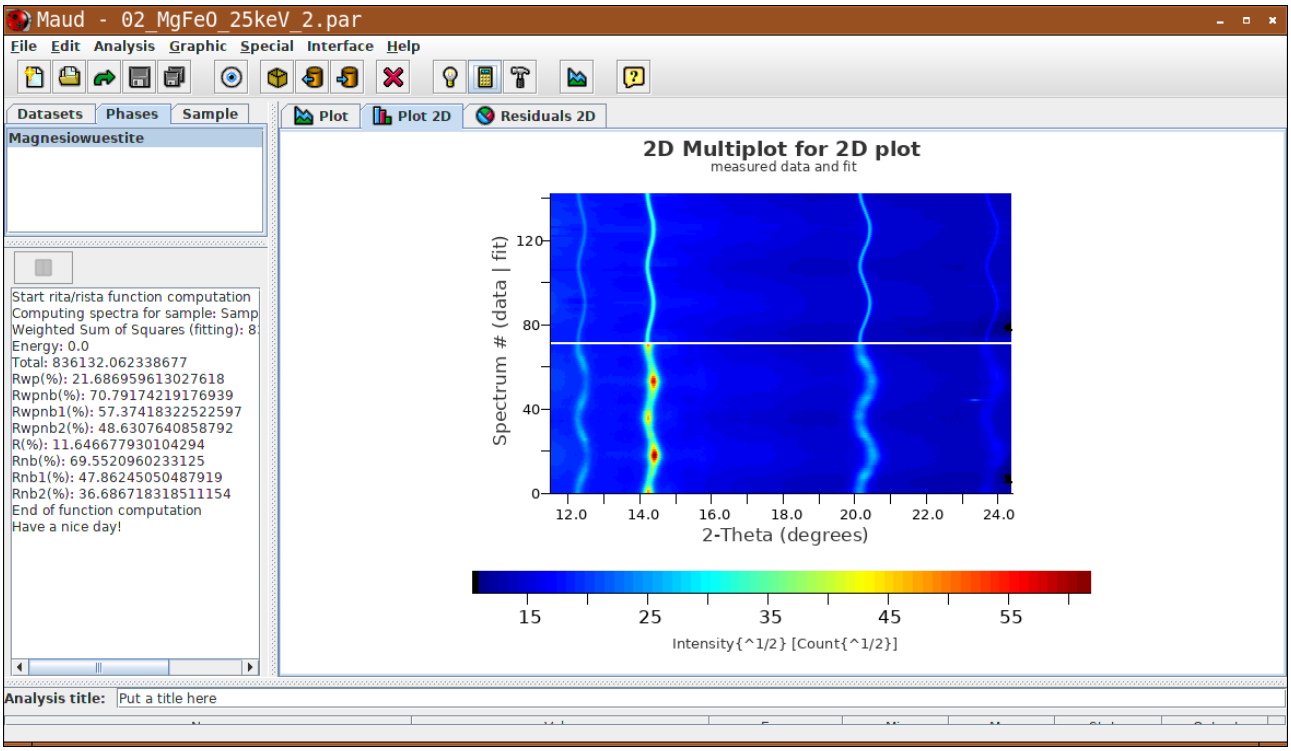
Before, just check: >Compute >Nothing !?
Verify: >Graphic>Texture plot>Pole Figure Coverage> (2,0,0) and (2,2,2) > Plot



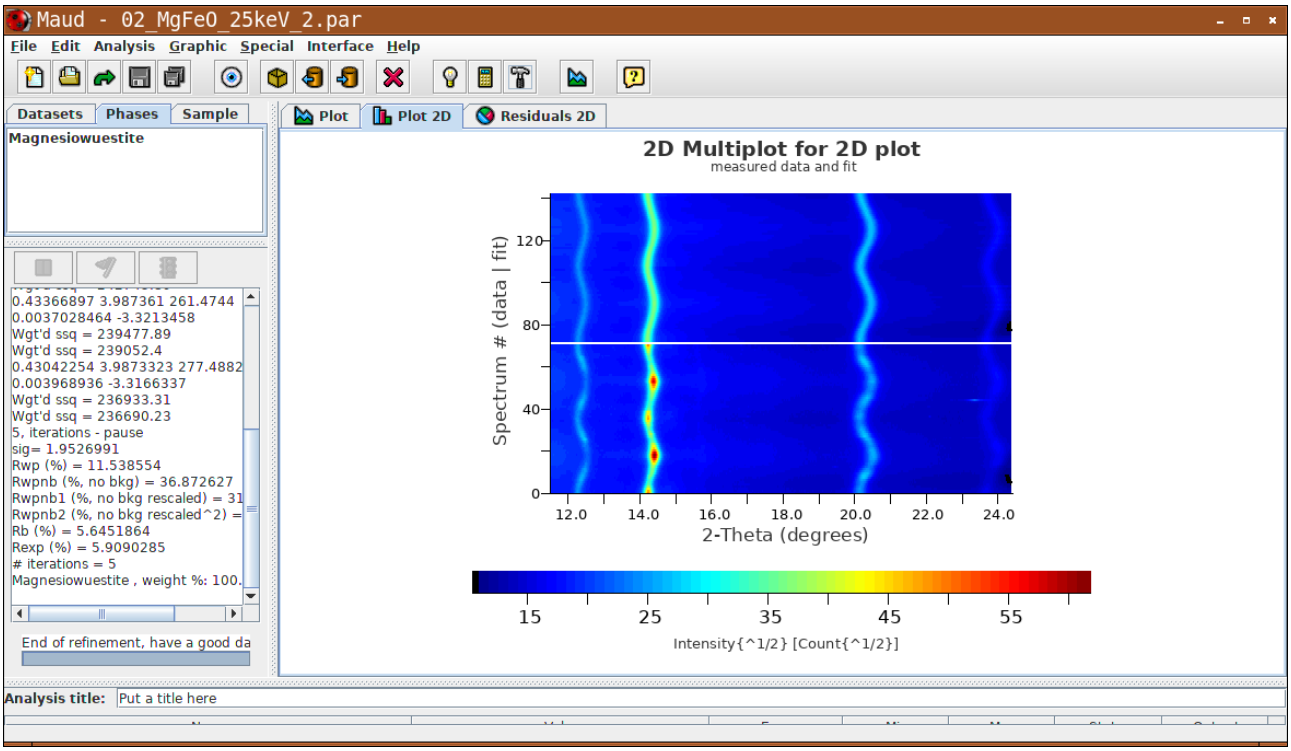
It’s OK: the reason is the stress is along S11 and not S33 . Let’s change:
S11 = -3 Gpa (Refine)
S22 (set equal to) S11 : * (-0.5) + (0)
S33 (set equal to) S11 : * (-0.5) + (0)



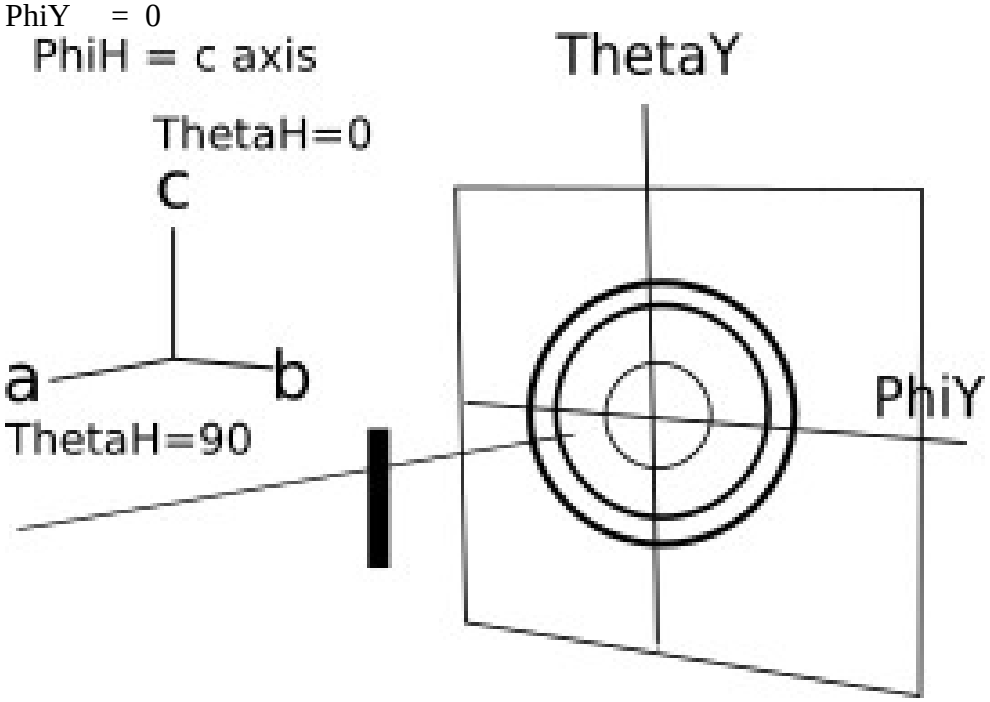
Calculate: OK



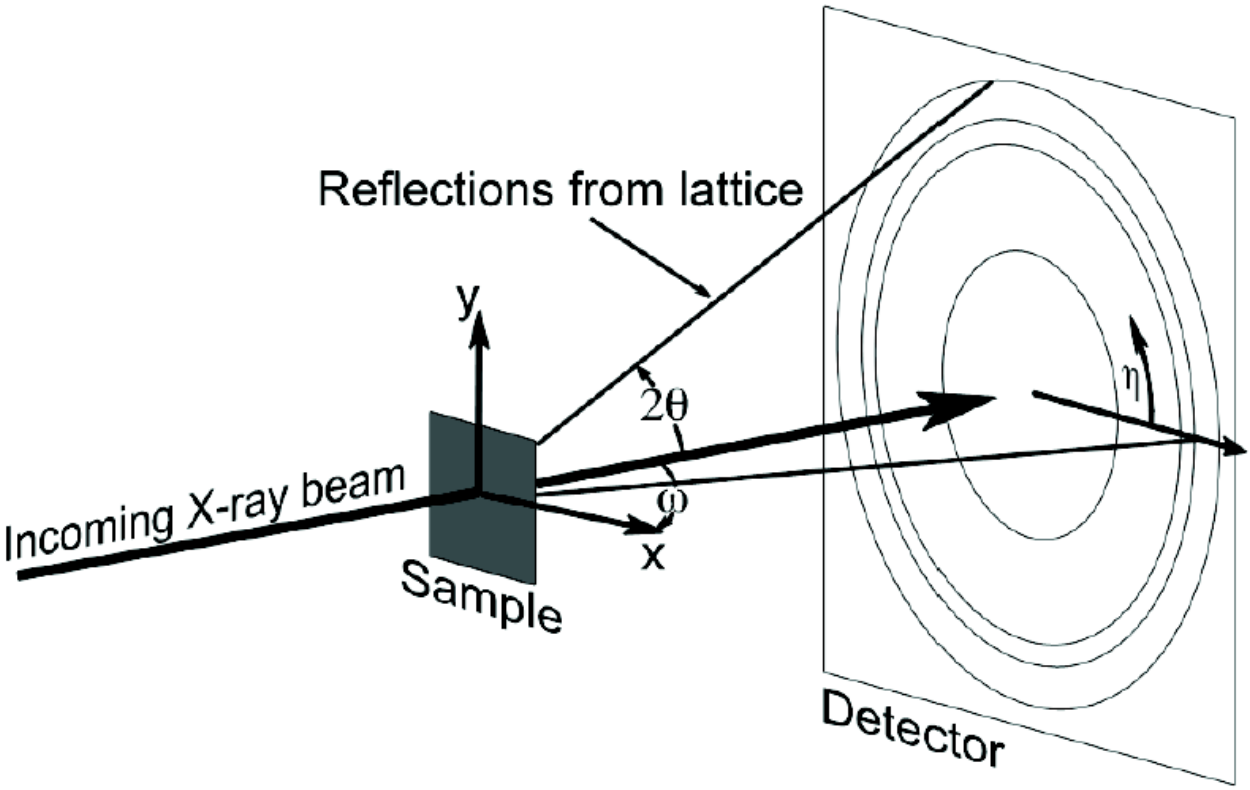
OK, let's refine: > Background > Incident intensity ! Do NOT refine detector tilt ! 1st, keep detector centering fixed > crystallite size and microstrain [Fit]	| | vS11 / ----------> S33 / |/ S22



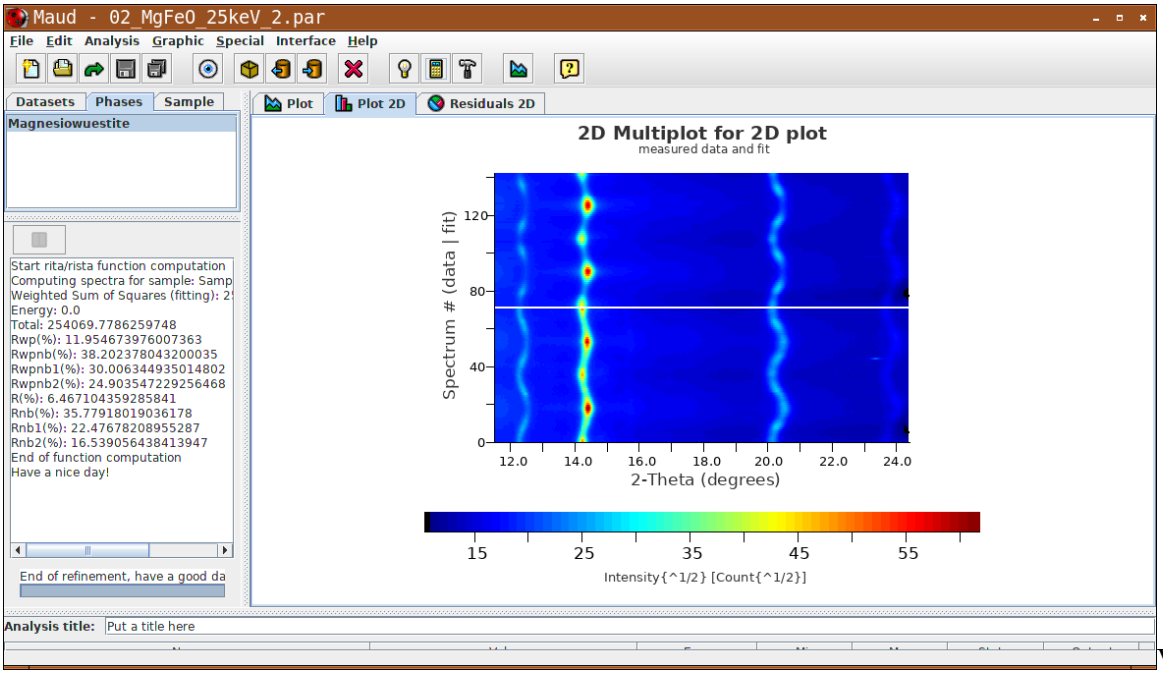
Now we need Texture
Phase>Edit>Advanced Models>**Texture>**<u>**Standard Functions**</u>>Options
Fiber or Spherical
Here: **Fiber along S11**
add component
ThetaY = 90
PhiY = 0



ThetaH: the crystallographic plane along the fiber component: 100 = 001
For 001: ThetaH=0, PhiH=0
FWHM: angular spread of the fiber: 30 deg quite sharp, but we will refine

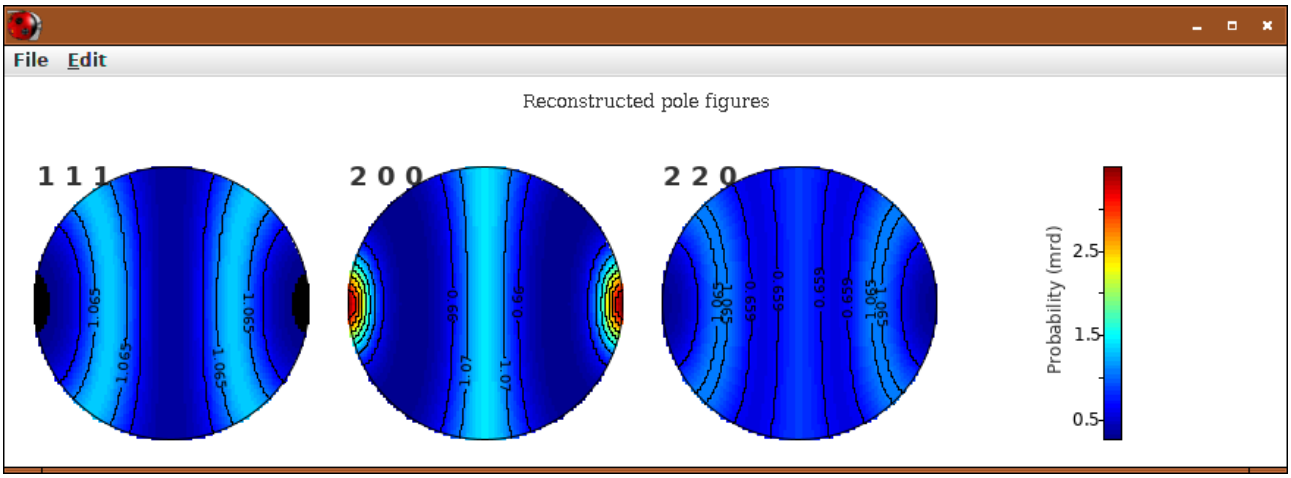


Check if direction is OK:
Calculate



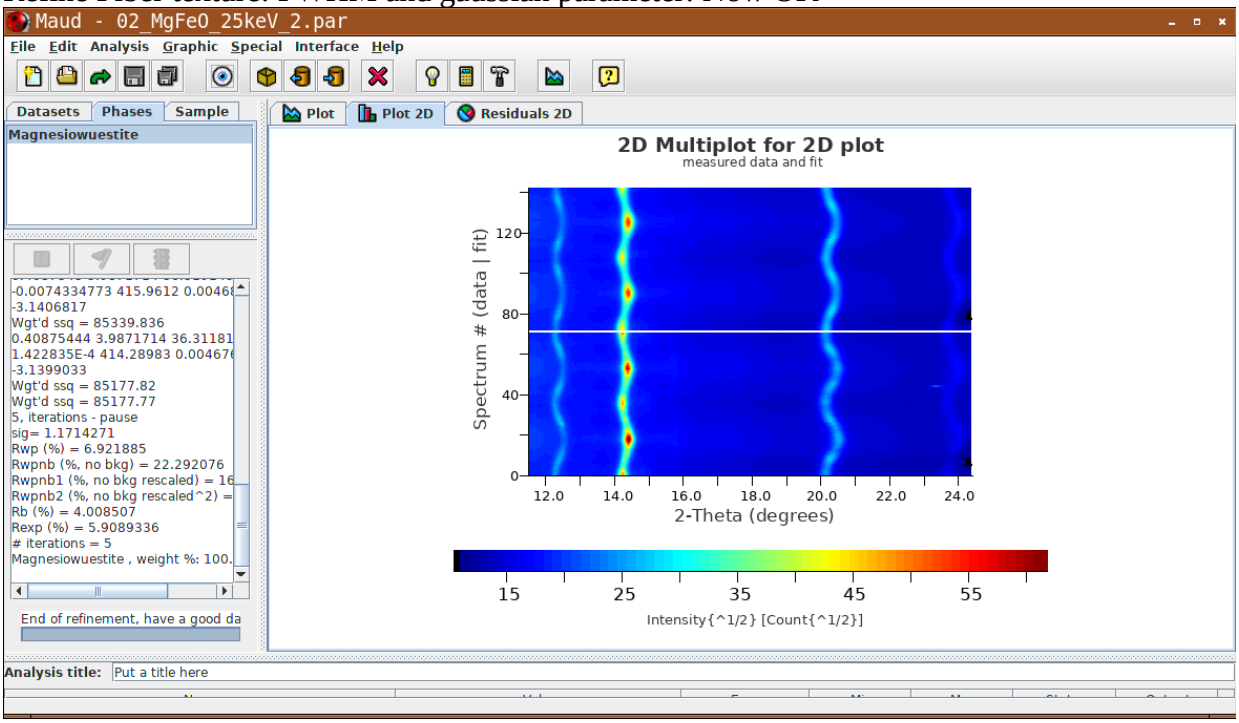
V

Graphic>Texture Plot>Select : 111, 200, 220
Reconstructed intensity: check > Plot!



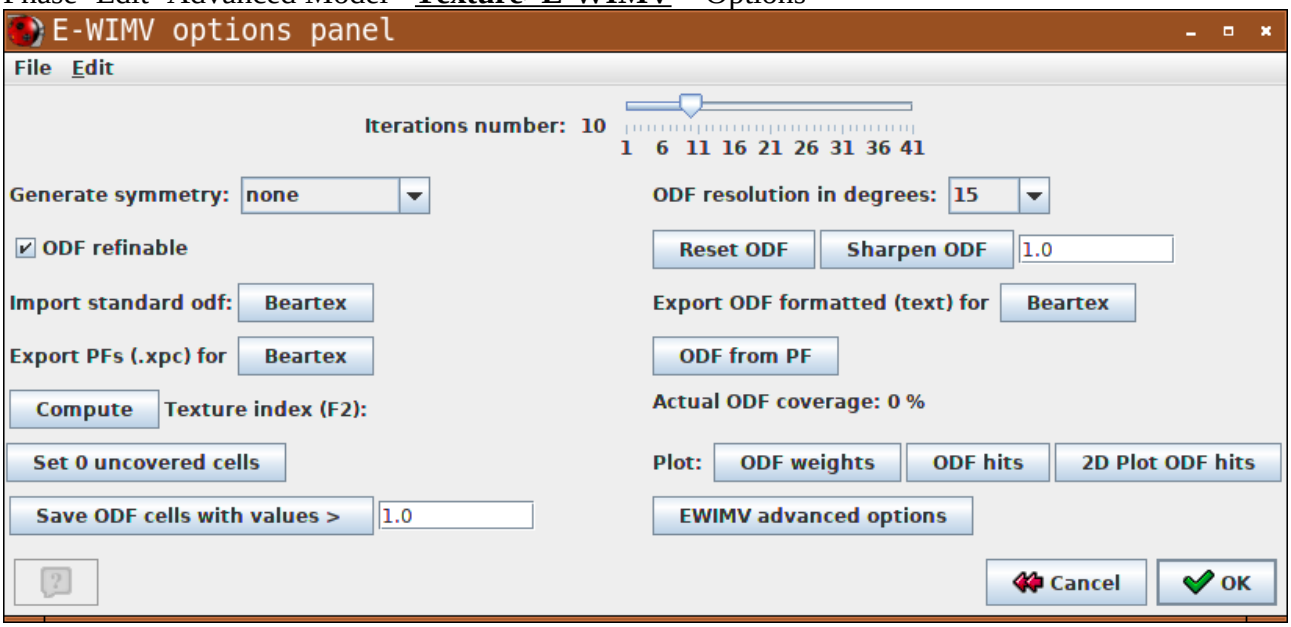
See fiber along 200 = 002

Refine Fiber texture: FWHM and gaussian parameter: Now OK



If we do not know about the texture fiber, and we start with with a model without imposing any symmetry.

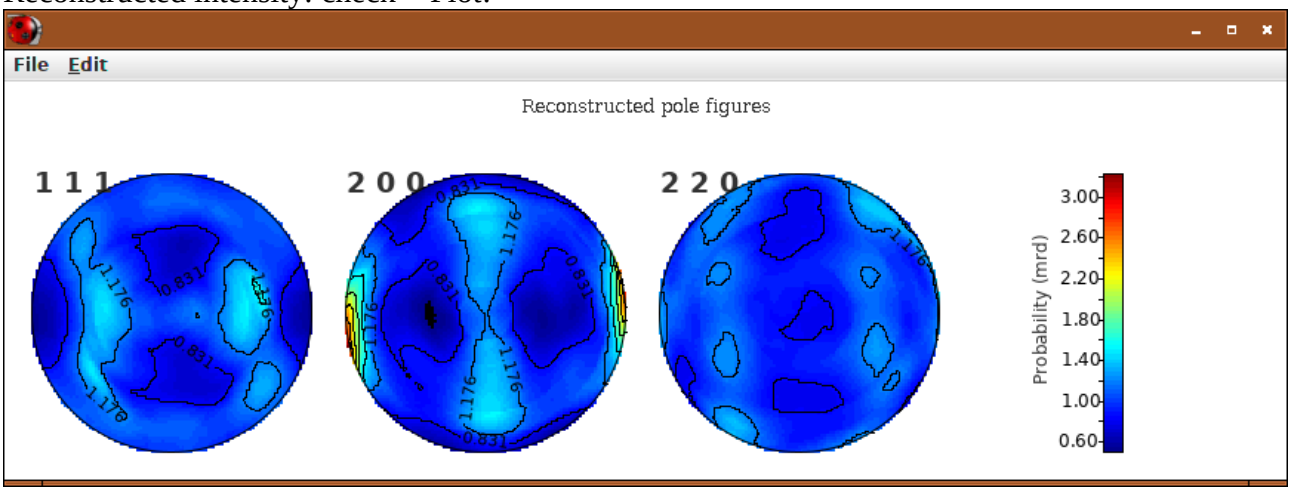
Phase>Edit>Advanced Model> **<u>Texture>E-WIMV</u>** > Options



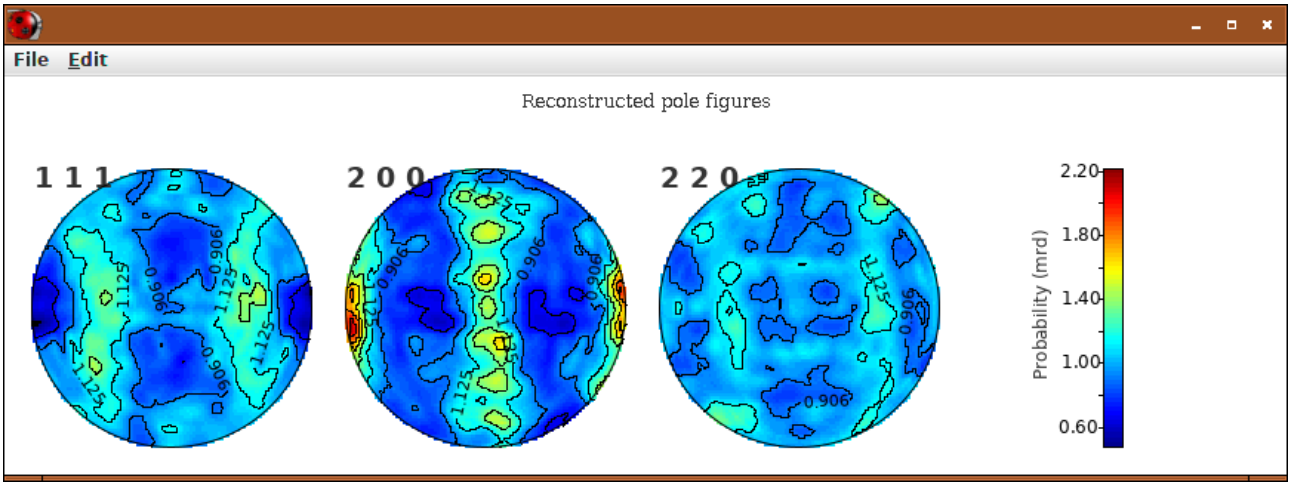
symmetry: none

ODF resolution: 15 deg (start rough and later refine mor fine ~ 7.5 deg)
OK and refine!

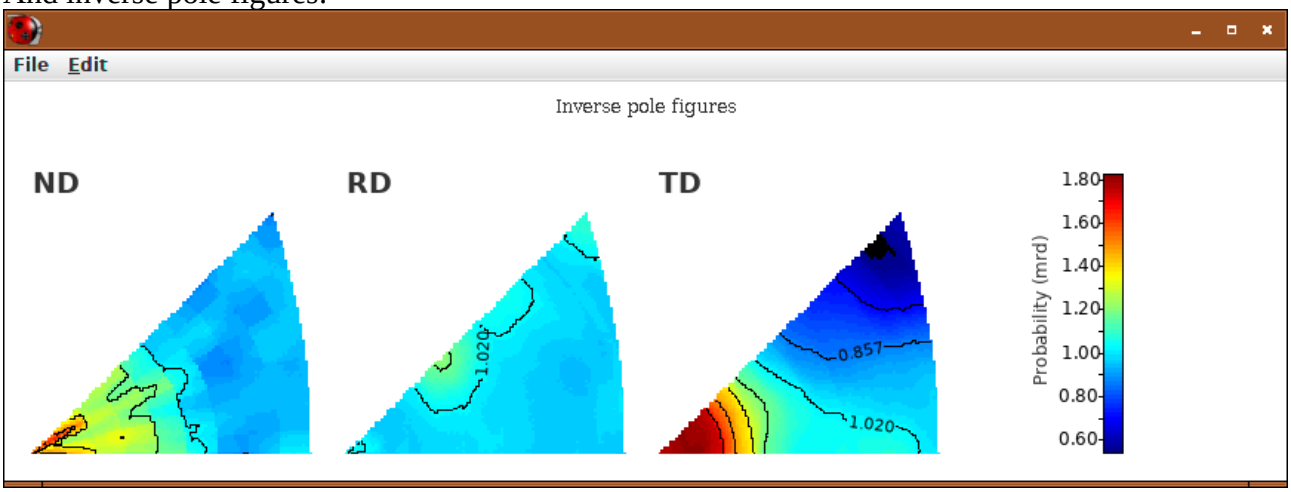
Graphic>Texture Plot>Select : 111, 200, 220
Reconstructed intensity: check > Plot!



The fiber can be recognized; let's use higher resolution: 5 deg
Refine!



And inverse pole figures:



111

100 110