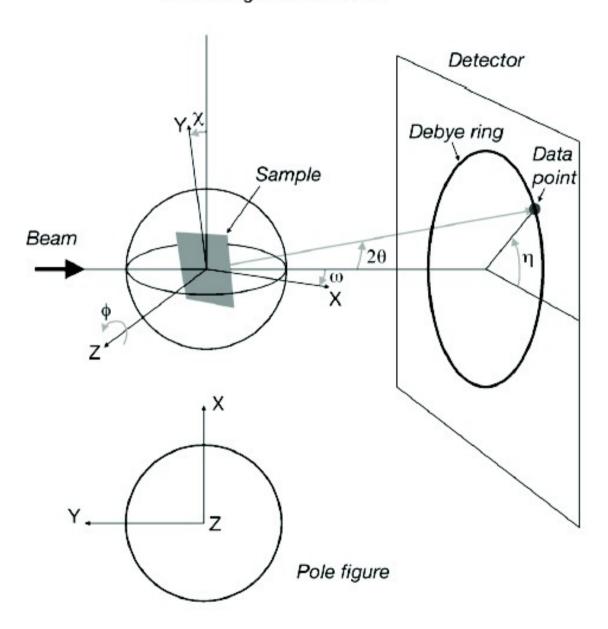
MgFeO XRD-2D combined analysis using MAUD

macro-strain texture crystallite size micro-strain

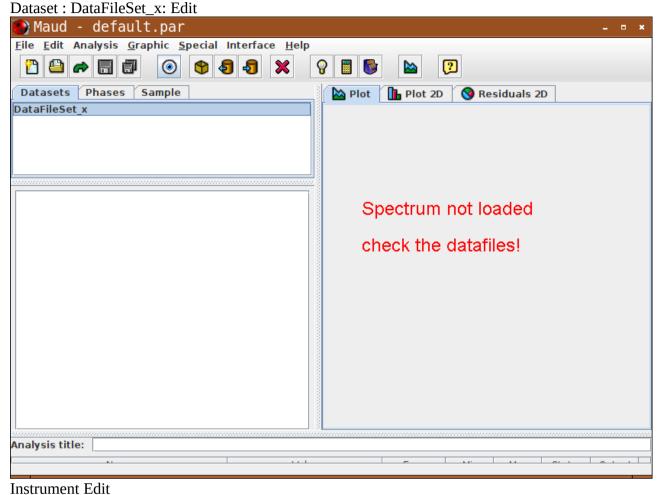
a tutorial transcription from the XRD school in Trento December 2018

# Maud angles convention



#### 1) Instrument calibration:

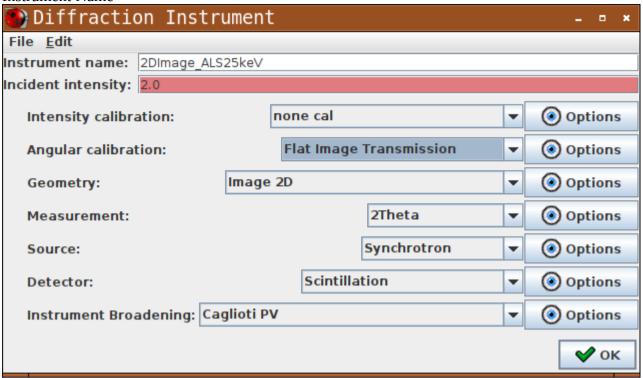
2



#### DataFileSet x File <u>E</u>dit <u>T</u>ools General Datafiles Excluded regions Background function Data block id: Date/time meas ✓ Computation enabled Force random texture Force no strain Peak cutoff: 30 Replace datafile on add Dataset weight: 1.0 Instrument -Computation range Min in data units: 0 Edit Type: Diffraction Instrument Max in data units: 0 ¶ Import... Data in groups of: 1 Name: Diffraction Instrument 🖣 Store... Intensity extractor: Le Bail ▼ Options Position extractor: none pe ▼ Options Diffraction model: Basic diffraction Reflectivity model: none reflectivity Options Options ✓ Lorentz restricted Fluorescence model: none fluorescence Options

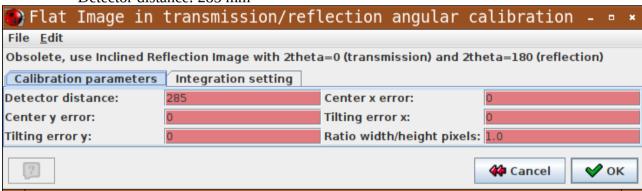
**У** ок

Instrument Name



Angular callibration: Flat Image Transmission

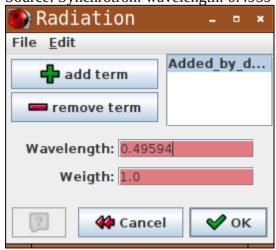
Detector distance: 285 mm



Geometry: Image 2D

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

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

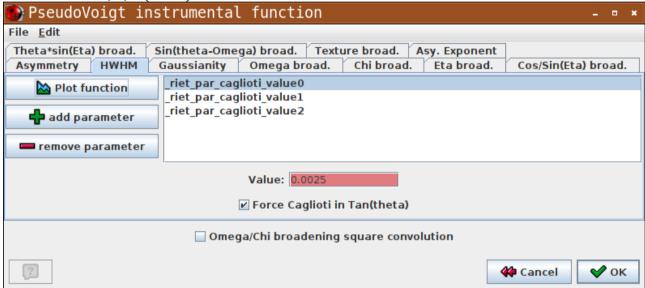


Detector: Scintillation (only change for XRF)

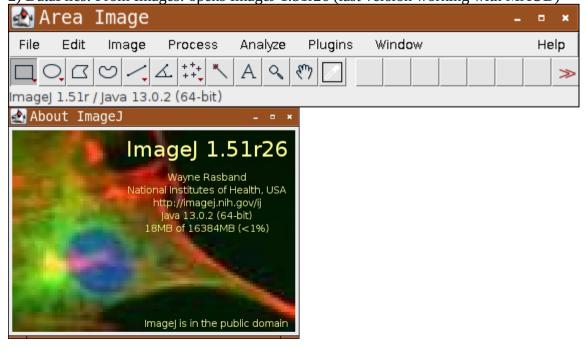
Instrument Broadening: Cagliotti PV

Asymmetry: 0, 0 (Fixed); Use reciprocal value: check PseudoVoigt instrumental function File Edit Theta\*sin(Eta) broad. Sin(theta-Omega) broad. Texture broad. Asy. Exponent Asymmetry HWHM Omega broad. Chi broad. Gaussianity Eta broad. Cos/Sin(Eta) broad. riet\_par\_asymmetry\_value0 Plot function riet par asymmetry value1 add parameter remove parameter Value: 0 ✓ Force Tan(theta) correction ✓ Use reciprocal value Truncation angle [deg or d]: 0.4 Omega/Chi broadening square convolution Cancel 🗱 💜 ок

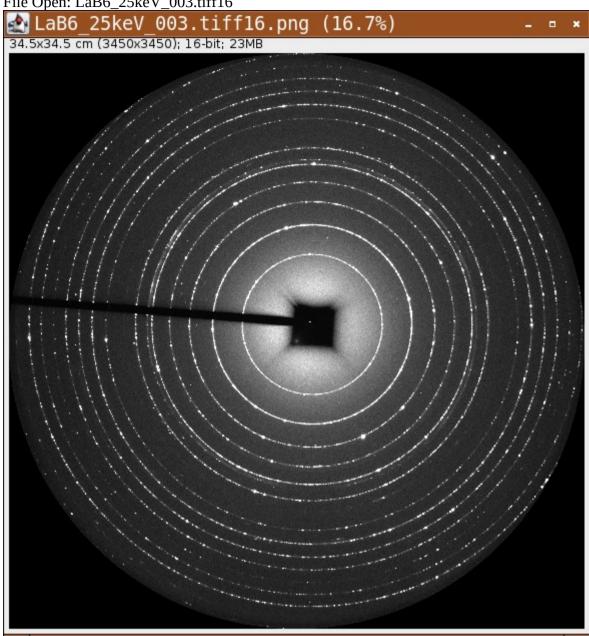
HWHM: 0.0025, 0, 0 (Fixed)



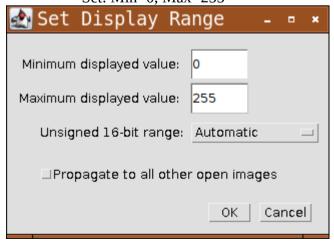
DataFiles: From Images: opens ImageJ 1.51r26 (last version working with MAUD)



File Open: LaB6\_25keV\_003.tiff16

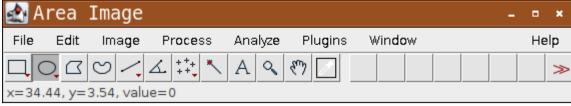


Image>Adjust>Brightness/Contrast: Set: Min=0, Max=255



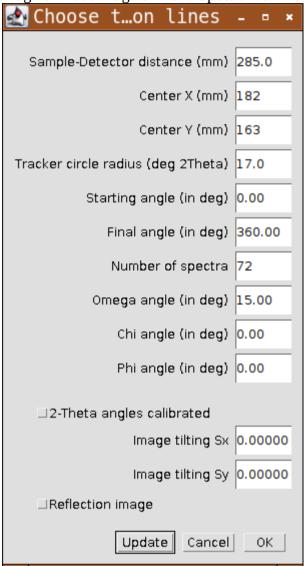
Check if the image has size in pixels and mm: we need the pixel size Image>Properties:

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]

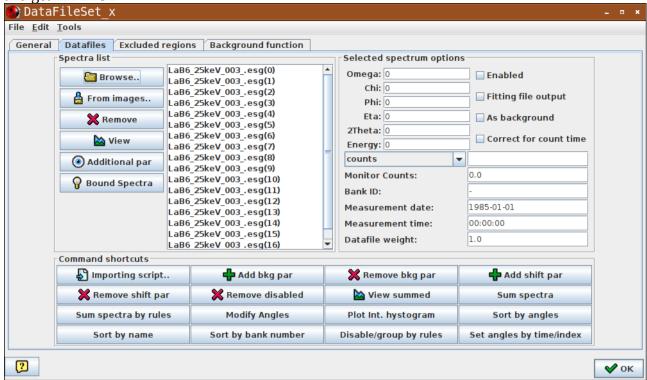
Integrate from 0 deg to 360 deg, 18 spectra For capillary: omega=0, chi=0, phi=0

(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

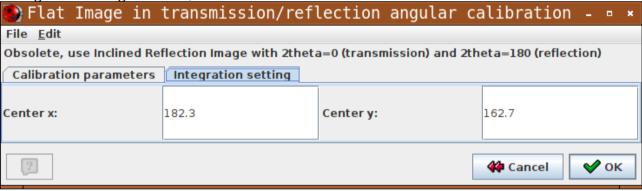
and get in MAUD

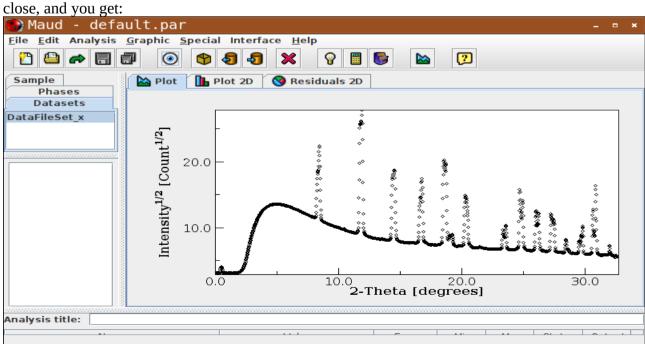


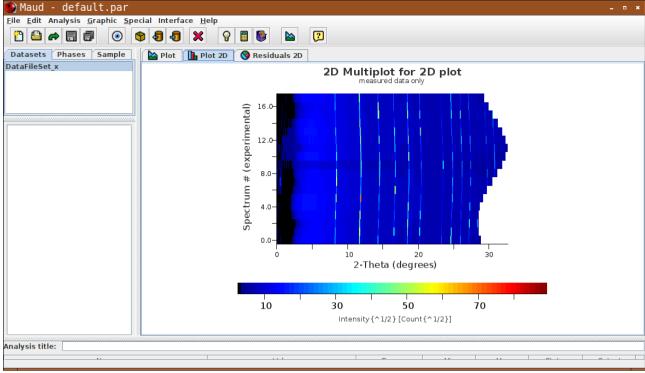
#### then check:

General>Instrument>Edit>Angular Calibration>Flat Image Transmission>Options>

>Integration setting: center X, center Y







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

and

Fix lattice value and refine detector distance and centering (also tilt), and instrument broadening

To fit instrument broadening:

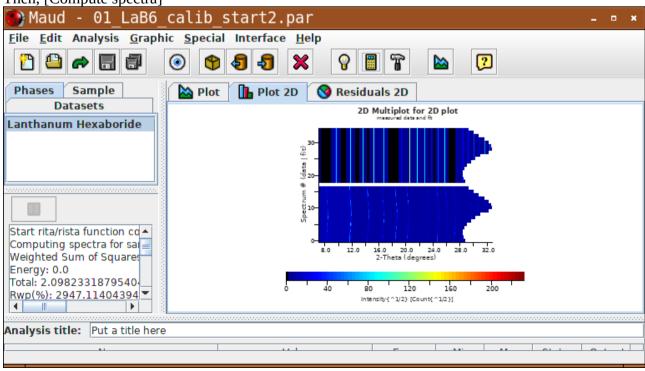
Phase>Edit>Microstructure> Line broadening model>None

or

Line broadening model>Delft

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]

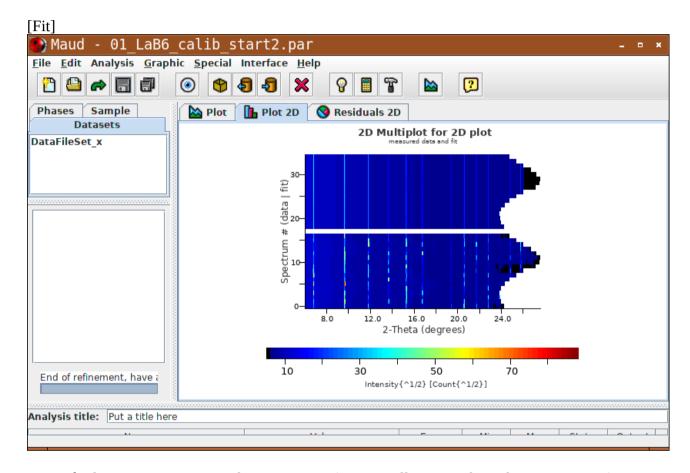
instrument> incident intensity: refine! (\_pd\_proc\_intensity\_incident)

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)



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 2<sup>nd</sup> 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

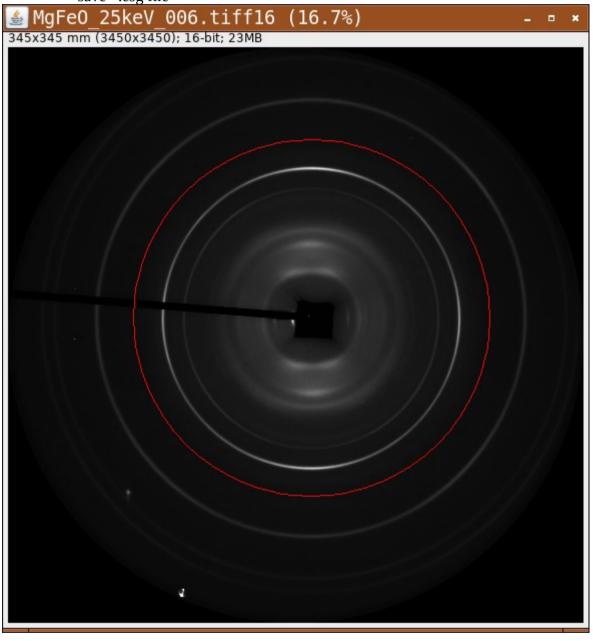
ImageJ: Toolbar: Oval: Select exposed area

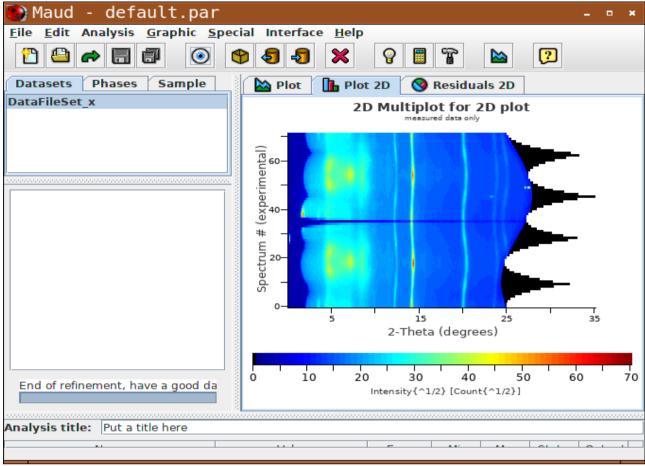
ImageJ: Plugins>Maud Plugins>Multi-Spectra from normal transmission/Reflection Image

Number of spectra: 72 (every 5 deg)

[ok]

save \*.esg file



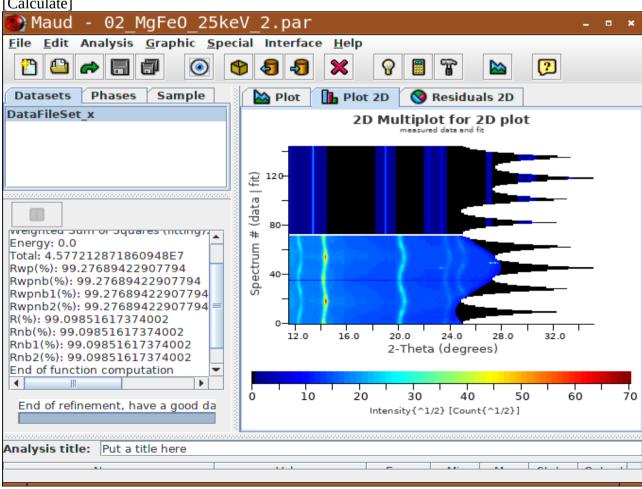


Back in MAUD

Zoom in at first line: change dataset range: min=11.5, max=50

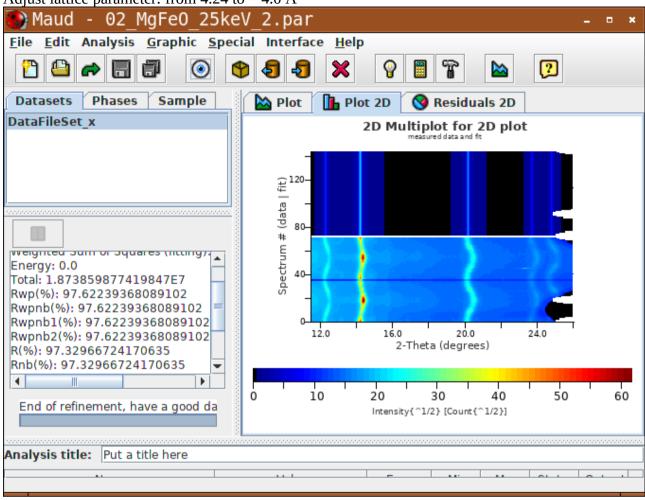
Phase>Import>MgFeO.cif

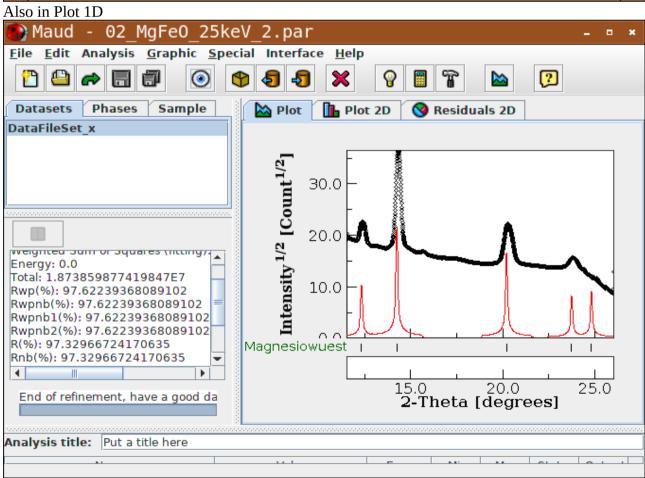
[Calculate]



Shift is caused by pressure: 40 Gpa

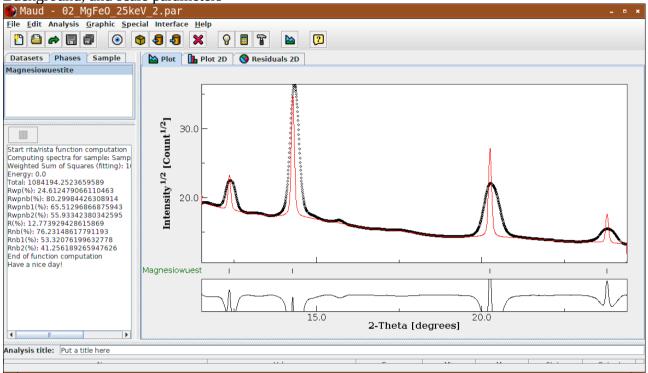
Adjust lattice parameter: from 4.24 to ~ 4.0 A





### 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

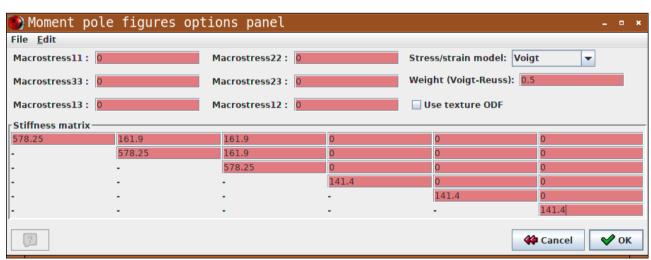
C44: 141.4

> hydrostatic stress (Macrostress)

S11=S22

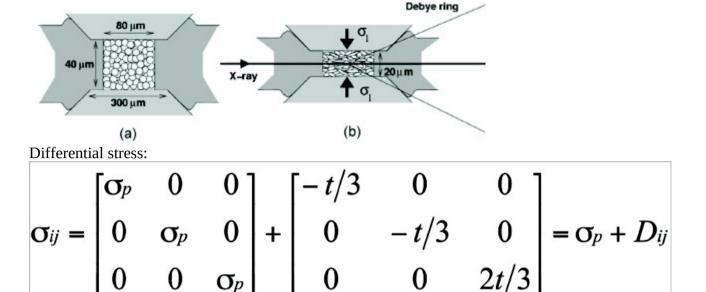
S33= -2\*S11

neglect shear: S12, S13, S23

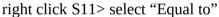


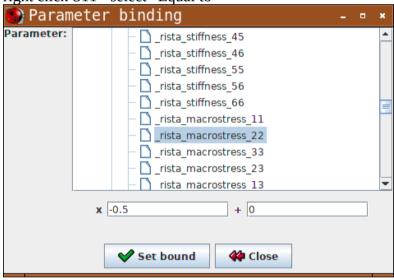
"One question ... From where do we get these value?"

"From literature ..."



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





[Set bound]

Do same for S33

[OK] and open from Options again, and check: Moment pole figures options panel File <u>E</u>dit Stress/strain model: BulkPathGEO -Macrostress11: 1.5 Macrostress22: -3 Weight (Voigt-Reuss): 0.5 Macrostress33: 1.5 Macrostress23: 0 Macrostress13: 0 Macrostress12: 0 Use texture ODF Stiffness matrix-578.25 578.25 161.9 578.25 141.4 0 141.4 0 141.4 **Cancel У** ок

Stress/strain model: BulkPathGEO (geometrical mean path)

#### Refine also:

- > lattice parameters
- > crystallite size and micro-strain

Before, just check: >Compute >Nothing!?

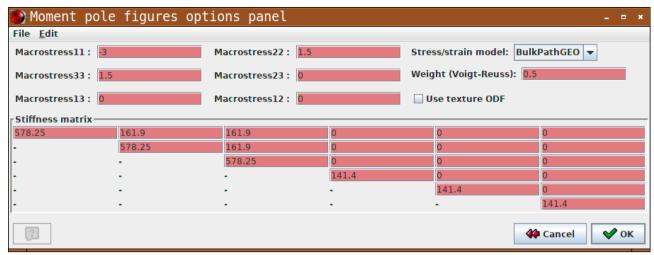
>Graphic>Texture plot>Pole Figure Coverage> (2,0,0) and (2,2,2) > Plot Texture plotting File Edit Options Pole figure Sample and Phase Active h Sample: Sample\_x ▼ Phase: Magnesiowuestite -Pole figure options Pole figure coverage Reconstructed intensity 2D map Experimental intensity 0 3D surface Inverse pole figures 0 Log scale Sample shape absorption Gray shaded Reconstructed strain Experimental strain Max azimuthal angle: 90 Number grid points: 101 ODF map plot Zoom factor: 1.0 plot limits ODF angles Start (deg) End (deg) Step (deg) Gauss smooth width: 0.0 Alpha/Gamma 0.0 360.0 5.0 Number of colors: 180.0 5.0 32 64 96 128160192224256 Plot ODF 2D Ŧ Optimize coverage **Close** 陷 Plot 🎒 Pole figure c…rage: 2 0 0 🖎 Pole figure c…rage: 1 1 1 File <u>E</u>dit File <u>E</u>dit o #1 0 #1 ∞∞∞ooooooooooooooooo 

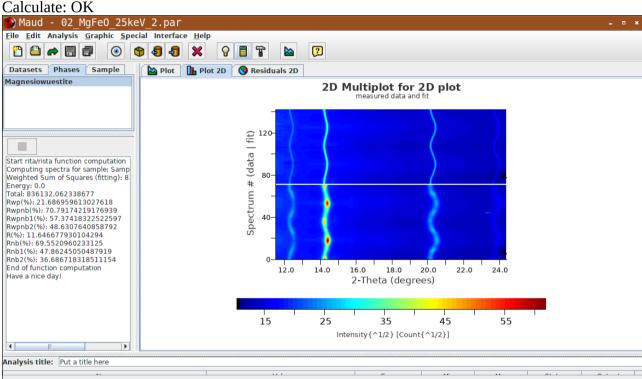
It's OK: the reason is the stress is along S11 and not S33. Let's change:

S11 = -3 Gpa (Refine)

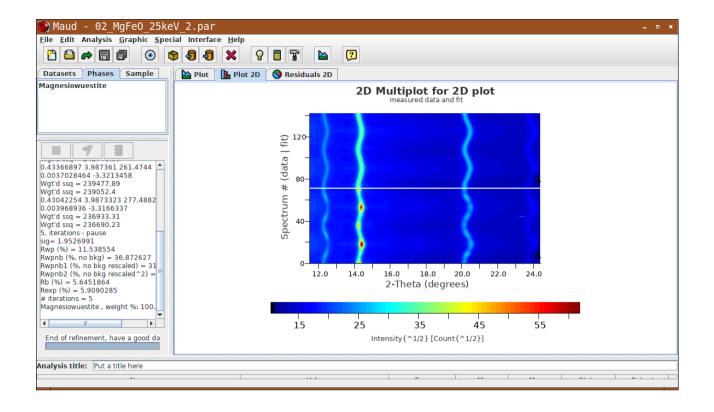
S22 (set equal to) S11: \*(-0.5) + (0)

S33 (set equal to) S11: \*(-0.5) + (0)









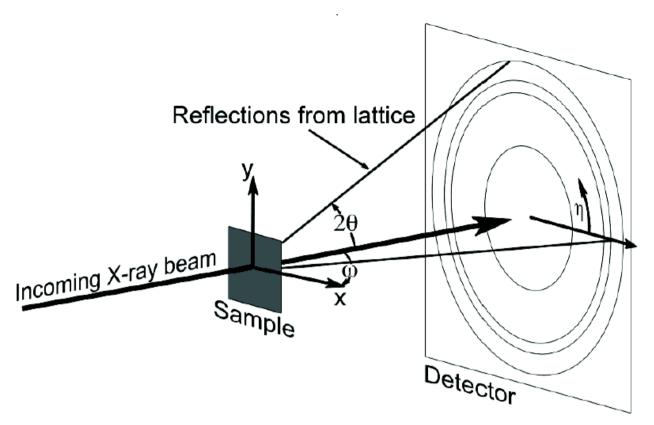
Now we need Texture
Phase>Edit>Advanced Models>**Texture**><u>Standard Functions</u>>Options
Fiber or Spherical

Here: Fiber along S11
add component
ThetaY = 90
PhiY = 0
PhiH = c axis
ThetaH=0
C
ThetaH=90
PhiY

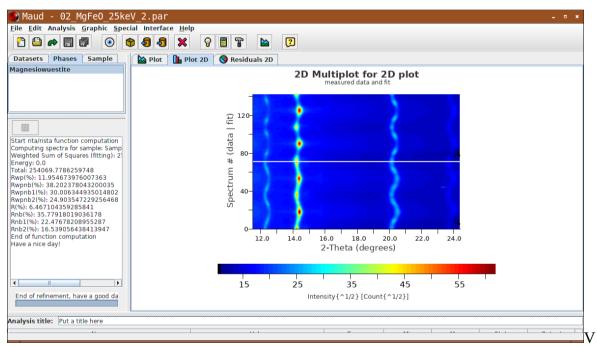
ThetaH: the crystallographic plane along the fiber component: 100 = 001

For 001: ThetaH=0, PhiH=0

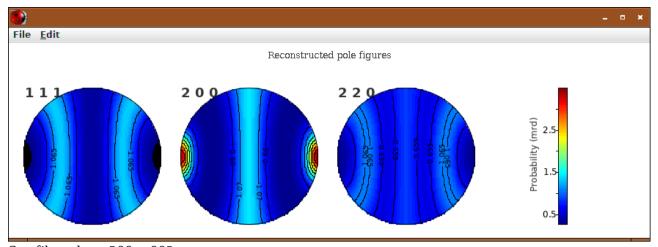
FWHM: angular spread of the fiber: 30 deg quite sharp, but we will refine



# Check if direction is OK: Calculate

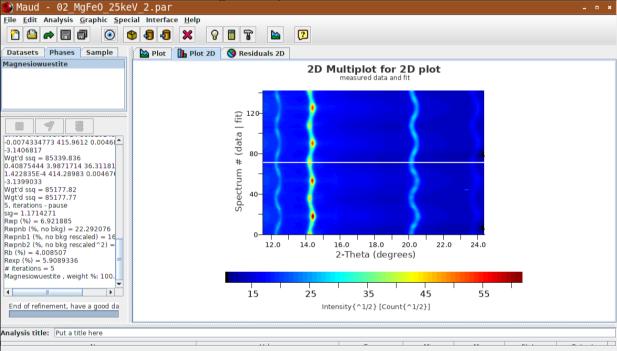


Graphic>Texture Plot>Select: 111, 200, 220 Reconstructed intensity: check > Plot!



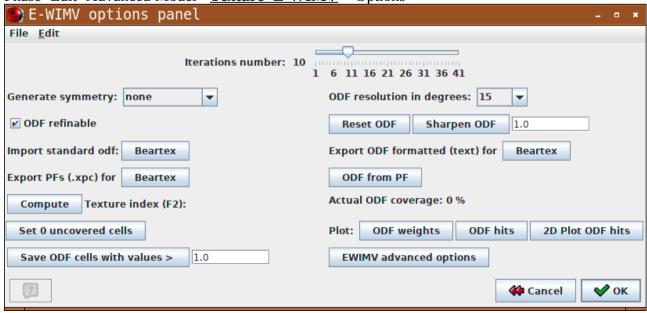
See fiber along 200 = 002

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



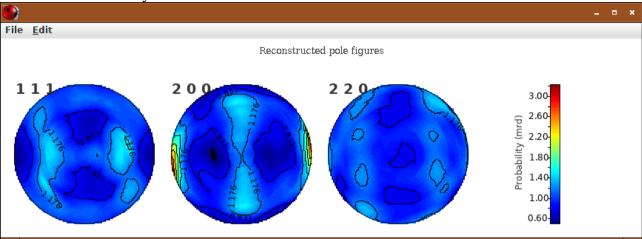
symmetry: none

ODF resolution: 15 deg ( start rough and later refine mor fine  $\sim 7.5\ deg)$ 

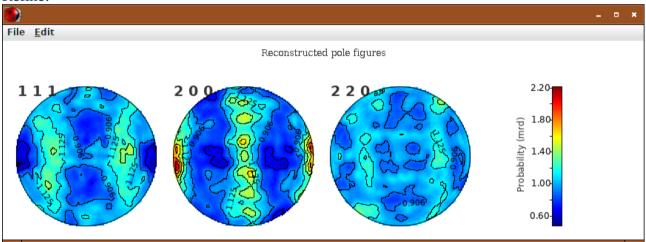
OK and refine!

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:

