

MAUD tutorial - Hippo Texture Analysis Wizard

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1 Introduction and purpose

This tutorial is based on the presentation made for the *Third HIPPO Workshop*, Dec. 9 2006, held at UC Berkeley CA (Sven Vogel and Rudy Wenk the organizers) that was attached to the *Neutron Scattering in Earth Science* short course of the AMS (Figure 1). The content and an extensive work from which this tutorial has been extracted is under publication in the LANSCE highlights (H.-R. Wenk, L. Lutterotti and M. Bortolotti, *Neutron Texture Analysis with HIPPO at Your Fingertips*) 2007.

It illustrates how to do a texture analysis with Hippo data. The example chosen is a Quartz textured sample from H.-R. Wenk. In principle the same procedure may apply for each neutron TOF spectrometer (and not TOF) for which you have a GSAS instrument parameter file and some GSAS datafiles. But I have not tried myself it without something different from a TOF spectrometer.

Mineralogical Society of America Short Course Neutron Scattering in Earth Sciences

Berkeley Dec. 7-8, 2006; Conveners: H.R. Wenk and N. Ross
http://www.minsocam.org/MSA/SC/Neutron_descrptn.html; support for students

Neutron properties
Diffraction
Inelastic scattering
SANS
Crystal structures
Magnetism
Hydrates
High pressure
Liquids / Glasses
Strain / Texture
Tomography
etc.

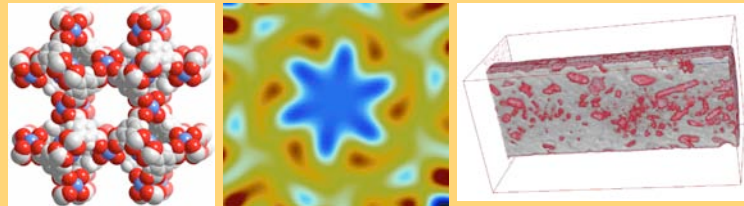
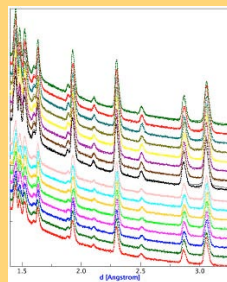
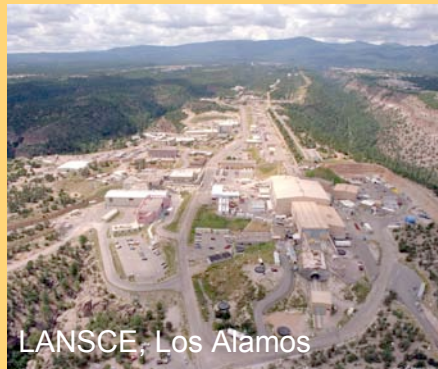


Figure 1: The AMS short course connected to the Hippo third workshop.

2 Requisite

The tutorial is based on Maud version 2.06 released in 2007.

Before to start you need to download the datafiles and instrument parameter file for this tutorial. They are downloadable from the same web page of this pdf (zip format, extract them to the folder where you want to use them for the tutorial).

<http://www.ing.unitn.it/~maudTutorial/HippoWizard/hippotexture06.zip>

Download MAUD from the web and install it on your computer (<http://www.ing.unitn.it/~maud/>).

You also need a recent version of Java (<http://java.com>, JVM 1.5 or higher preferred). Most modern

computers have Java already installed with their operating system.

3 Entering and preparing data

Start MAUD. The menu option *File*→*New*→*Hippo wizard* opens a window (Figure 2) that allows you to conveniently import the experimental data and prepare it for the automatic analysis.

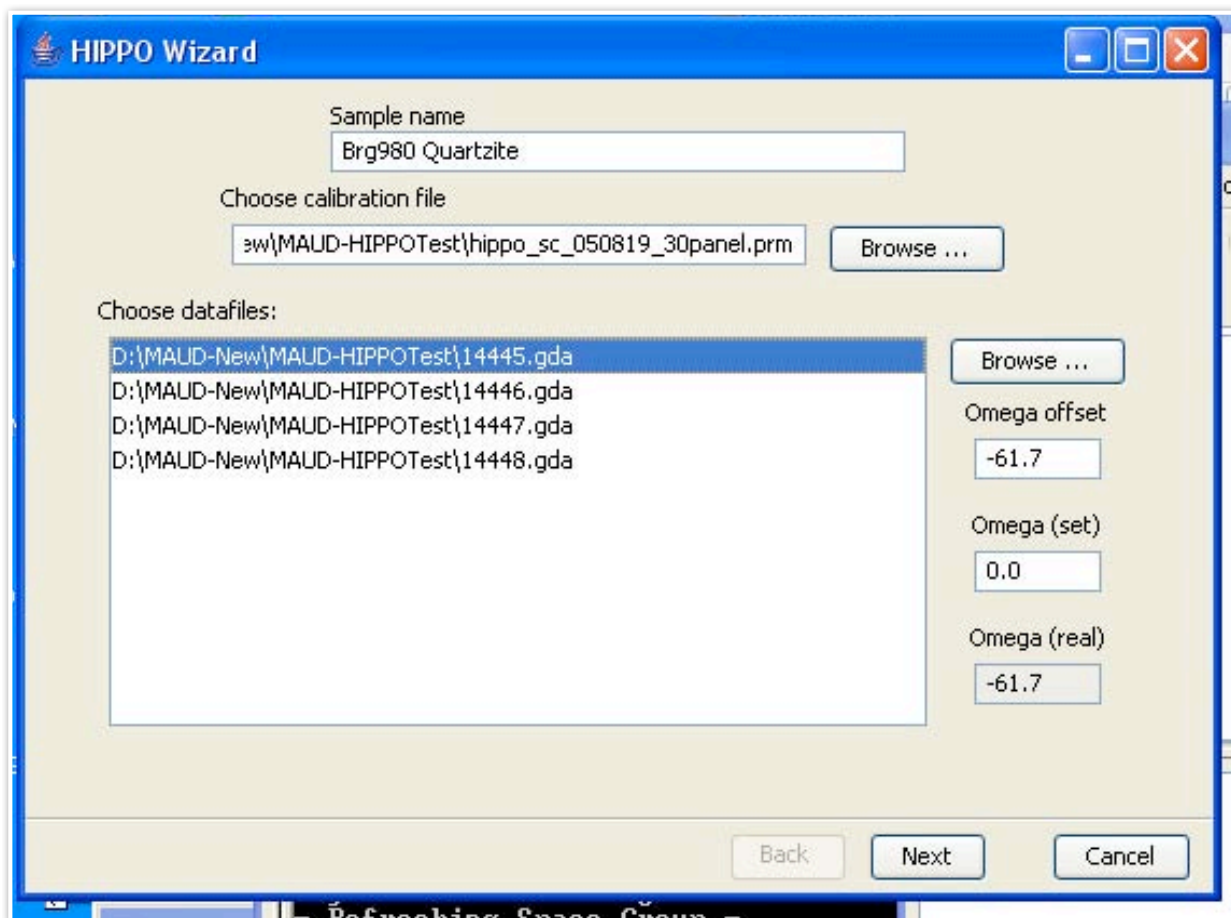


Figure 2: Entering data in the Hippo wizard.

In the Hippo wizard window:

- Input a title in the sample name.
- Select a parameter file (for this demonstration we use hippo_sc_050819_30panel.prm) by

clicking on the *Browse* button next to the *Choose calibration file* field and then select data files (browse for 14445.gda to 14448.gda).

- Assign each data file a omega rotation angle (0° for 14445.gda, 45° for 14446.gda, 67.5° for 14447.gda and 90° for 14448.gda; the bulk rotation $\omega = -61.7^\circ$, *Omega offset*, accounts for the HIPPO sample changer oddity that the sample holder notch is displaced from the incident beam direction. Applying this rotation realigns the sample, with the arrow pointing at the incident beam in the reconstructed pole figures). Click on each file again to verify that the angles were assigned correctly.
- Click *Next* and choose the d-ranges to be considered for each detector bank. This range depends on the number of peaks in your patterns. For instance, if the maximum reflection d-spacing is at 2.3\AA , choose a maximum d-spacing of 2.5\AA since a lot of data represents only background. For this example, we use 1 to 2.75\AA . Data at lower d-spacings do not improve the analysis in this case because of the large number of overlaps. Restricting the range also speeds up the computation. Generally, you consider each bank and each rotation as a different instrument and refine instrumental parameters (namely the scaling factors and conversion factors for TOF to d-spacing), accordingly. Hence, leave the corresponding option in the dialog checked (*Treat rotations as separate datasets/instruments*).
- *Finish* the wizard. In MAUD you may now inspect your data using the *Plot 2D* panel and by clicking on each *Dataset*. You may want to change the plotting options if you wish to plot the intensities as calibrated or on a linear scale: choose from the menu *Graphic*→*Plot options*. Next click on the *Phases* panel and load phases from a data base (structures.mdb or CIF compliant files containing crystal structures), or entering your own structure information (space group, lattice parameters and atomic coordinates). For this example use *Quartz* from the MAUD data base, structures.mdb.

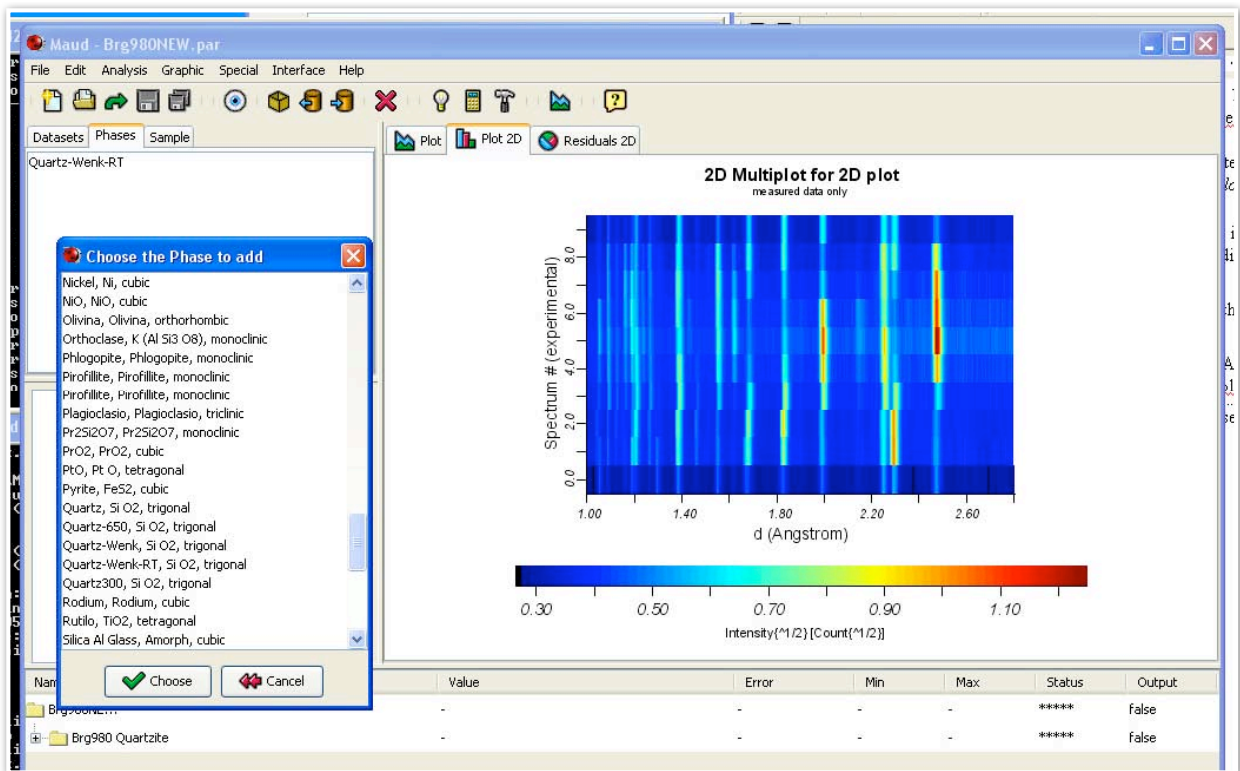


Figure 3: Entering the quartz phase from the Maud structural database.

- Edit each phase and in *Advanced models* choose E-WIMV as a texture method. Under the texture options select an ODF resolution of 10° or 15° , which approximates the angular resolution of HIPPO.
- If you need the sample rotation axis in the center of the pole figure, edit the *Sample* and for *Sample position* choose $\chi = -90^\circ$. This orients the system so that the rotation axis points to the bottom and the incident neutron beam enters from the south of the pole figure. In the final pole figure you are looking down on the sample as in the figure 4. In the sample-editing window you can enter the volume fractions for polyphase materials in the *Layers* dialog.



Figure 4: The quartz textured sample mounted for the experiment.

If you close this window and go to the main menu clicking on *Graphic*→*texture plot* you may choose to plot the *Pole figure coverage* for the selected hkl peak and obtain the coverage like in figure 5(a) or 5(b) by enabling the datasets you are interested to look for (to enable or disable a dataset, you must edit the specific dataset and check/uncheck the *computation enabled* check box in the *General* tab panel).

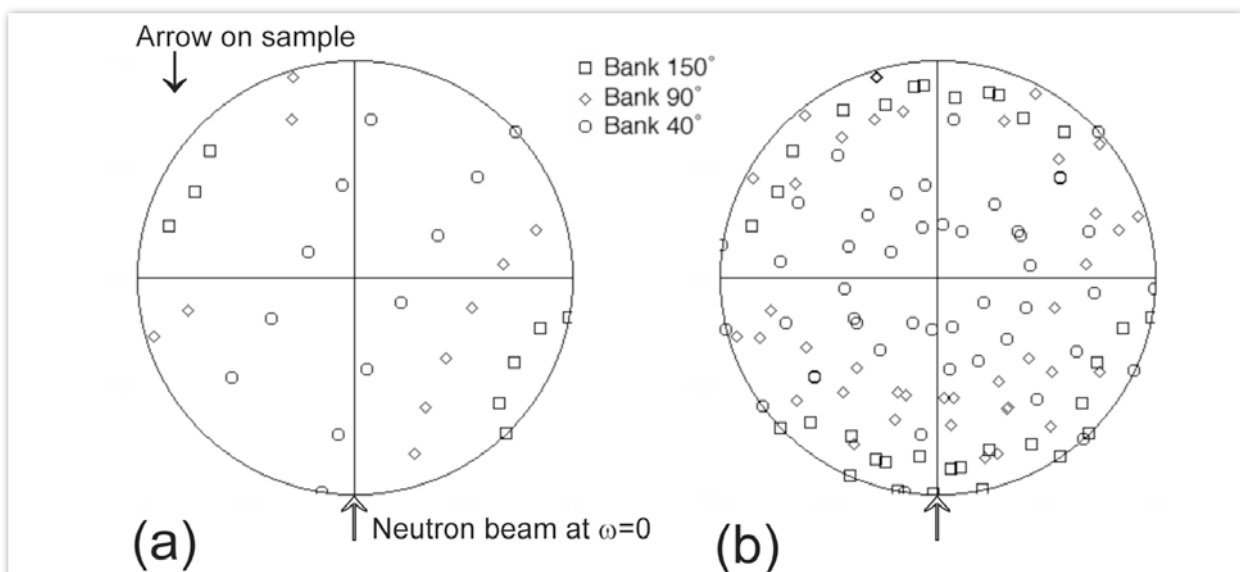


Figure 5: Pole figure coverage from the experiment considering only one sample rotation (a), or all rotations (b).

- Data are displayed on the MAUD main window either as average profile (Plot) or as a color

stack of all spectra for a given *Instrument* (Plot 2D). In the Plot 2D panel you can check all data sets by selecting them one at a time in the data list.

- Note the bottom (first) spectrum of each 90° bank has a very low intensity and you need to remove it from the analysis. Edit *Datasets*, go to *Datafiles*, and *disable* the first spectrum on the list (number 9) for each omega rotation (after selecting the spectrum, uncheck the *Enabled* check box in the selected spectrum options).
- At this point you should save your analysis in your data directory (*File*→*Save analysis as*) as a xxxx.par file (better to save it near the datafiles). Now you are ready to start the refinement.

4 Rietveld refinement

Under *Analysis* select *Wizard*. In the appearing window choose *Texture analysis* in the right panel and press the button *Go!* You may watch the progress of the refinement in the graphics window. This will refine the most important parameters in four cycles. Select no more than five iterations. To guarantee convergence you may, at the end, perform 35 additional iterations (selecting from the menu *Analysis*→*Refinement*). What the program does during the wizard refinement is briefly described in the following:

- Cycle 1: Refining the scale, background parameters and phase fractions for multiphase systems. The intensity calibration (12 parameters) are directly loaded from the HIPPO calibration file and then kept constant. A single scale factor is refined for each detector. Backgrounds are expressed with a polynomial. Three coefficients are refined for each bank and in addition two for each panel. Backgrounds start to look reasonable and so does the overall intensity (Figure 6). Note that the high-angle 150° bank detectors have a much higher resolution than the 40° bank, but lower counting statistics. Texture is not reflected in the recalculated spectra. Also, the wavy line is not matched due to mispositioning of the sample, which is particularly serious for the 40° bank.

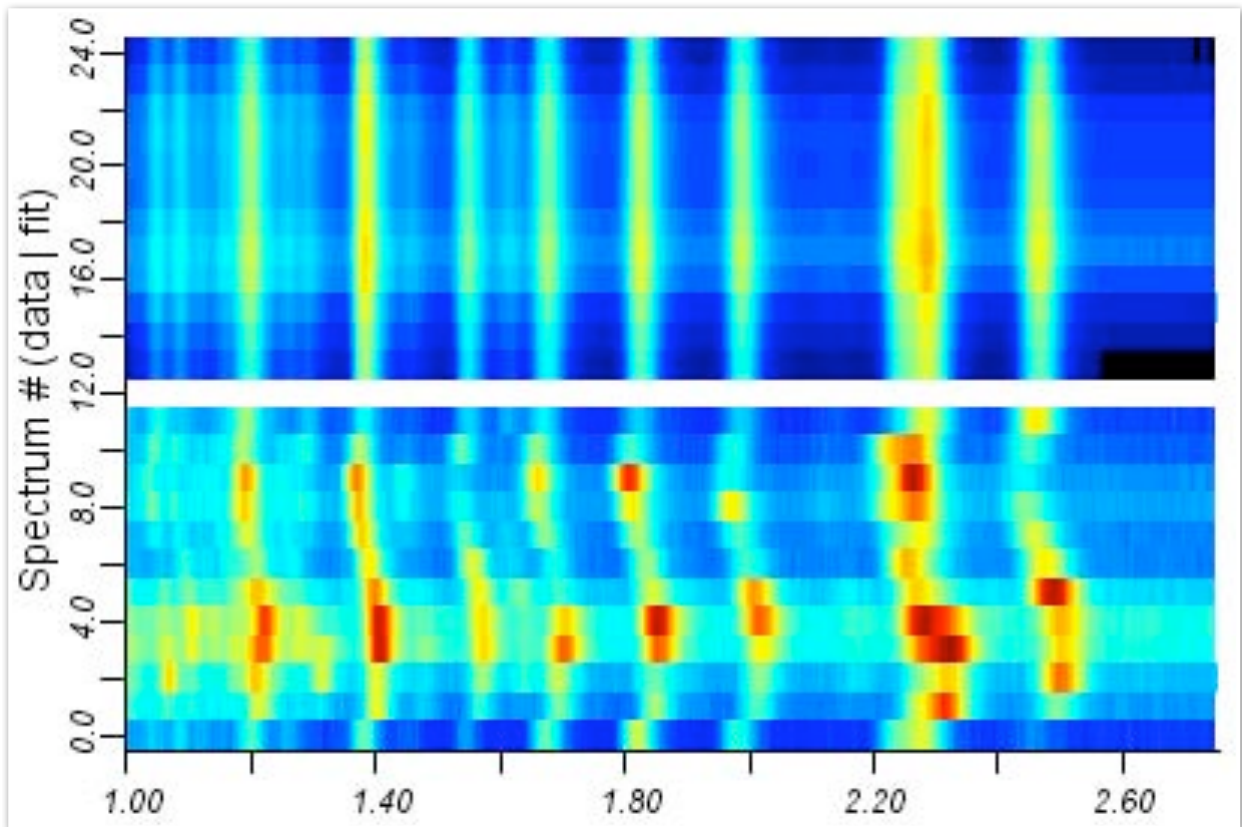


Figure 6: Starting data and fit of the refinement as seen in the 2D plot panel.

- Cycle 2: In addition to the previous parameters peak positions are adjusted in during this cycle by refining cell parameters and instrument errors based on observed d-spacings. The waviness is corrected.

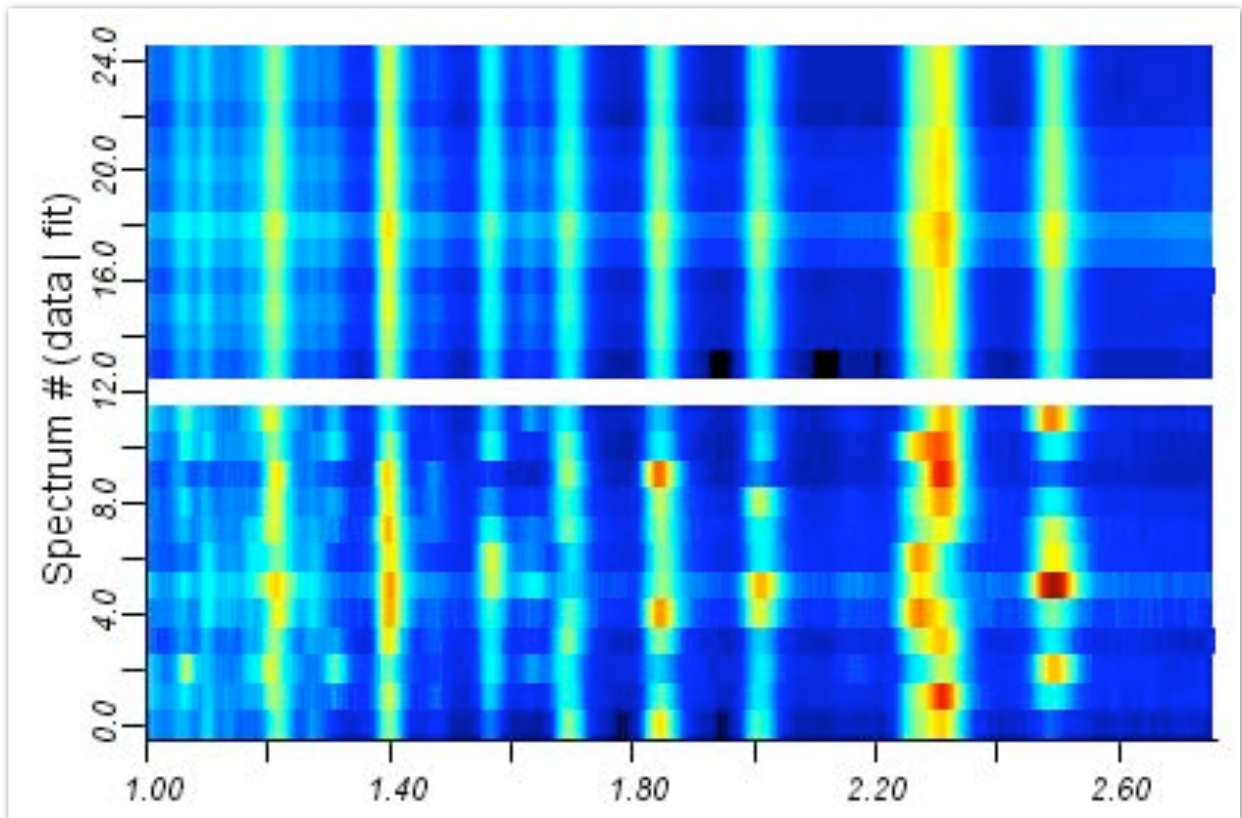


Figure 7: Progress of the fitting after the second cycle.

- Cycle 3: The microstructural parameters (crystallite size and microstrains) are added to the refinement to adjust peak shapes. Only one overall temperature factor, B, is used for the entire analysis to avoid correlations and minimize possible errors. Other structural parameters such as atomic coordinates are not refined with the Texture Wizard. You may refine them separately at the end.
- Cycle 4: The orientation distribution is refined. Intensities of individual peaks start to match. End of the analysis

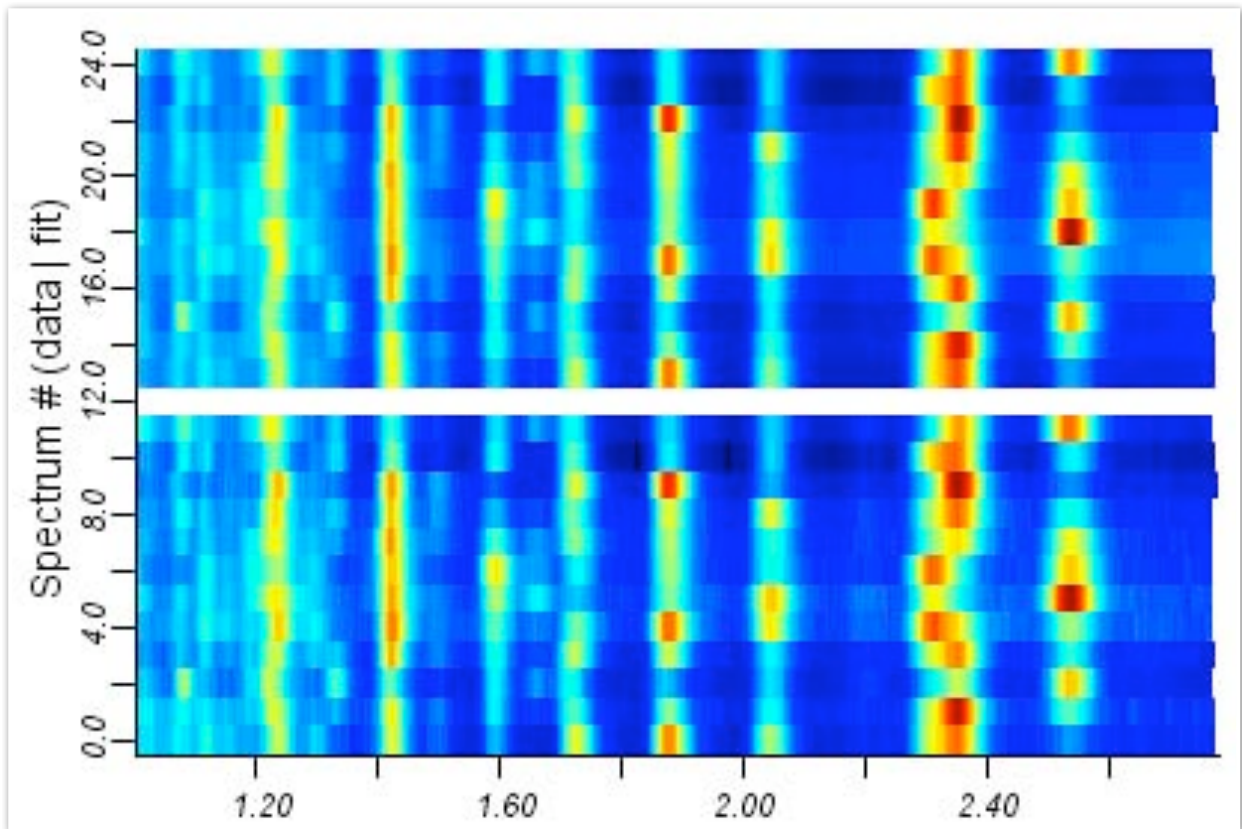


Figure 8: End of the refinement: this is the fitting result shown in the 2D plot panel.

Now you need to save the analysis (use the save button in the toolbar or from the File menu *Save analysis*) and review the results. To check the texture you can go to the *Graphic* menu and click on *Texture plot* and select to plot the peak *003* as reconstructed pole figure. You should obtain a similar pole figure to the one in figure 9 (the arrow as been added subsequently).

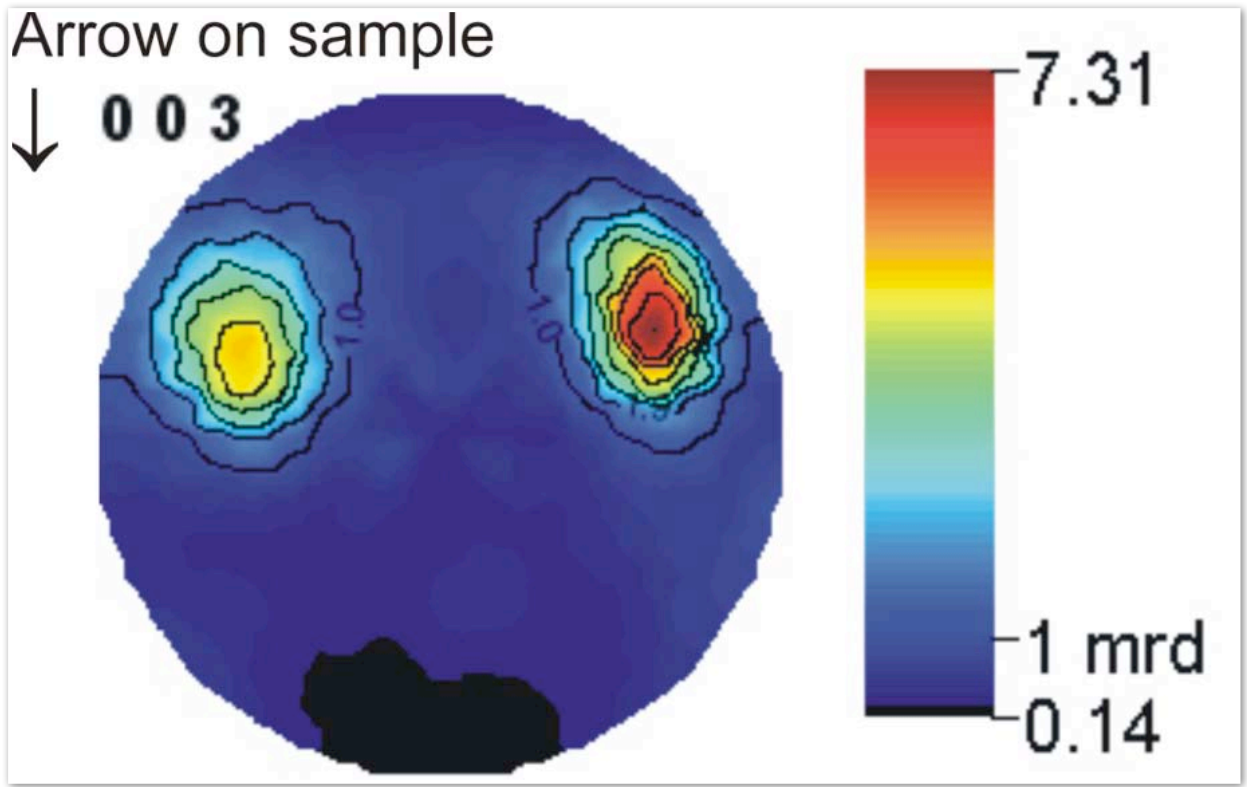


Figure 9: Reconstructed pole figure 003 for quartz from the refined ODF.