

# MAUD tutorial - Instrumental Broadening Determination

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## 1 Introduction and purpose

In this tutorial we will see how to measure the instrumental broadening on a typical Bragg-Brentano instrument and store it in Maud for subsequent use. This analysis step is fundamental for a successive measurement of particle size and microstrain as line broadening analysis requires as a prerequisite to separate the broadening due to the sample (crystallite size and microstrain) from the broadening arising from the instrument. This pre-analysis must be performed each time the instrument configuration is changed or for any different instrument setup. As a different instrument configuration we consider here every change that can potentially change the instrumental broadening, in particular slits, beam size, goniometer radius, monochromator etc. The entire procedure consist of three steps. The first step is the preparation of the standard sample with the necessary characteristics and we will discuss some details in the following. The second step is the measurement of the standard using the same experimental setup as in the line broadening experiment; a good idea is to measure the large  $2\theta$  as possible in order to characterize the instrumental broadening nearly over the entire range covered by the instrument (the standard sample should be chosen to have peaks in the full range). The third step is the analysis with a fitting program (Maud in this tutorial) to determine the instrumental function to be used for every line broadening analysis subsequently. The most difficult part of the procedure is the preparation of the standard sample to be used for the instrumental broadening measurement. The idea is to prepare a sample so well crystallized that the broadening due to finite size of the crystallites and microstrain is negligible, i.e. sufficiently large crystallites and very low defects concentration leading to near zero microstrain. The goal is not very easy as increasing the crystallization of the sample may cause so called graininess problems when the crystallites becomes too large compared to the x-ray beam investigated sample volume. At that point only few grains contribute to the diffraction process and

their number is not statistically sufficient to describe the instrument peak shape. As an example of the problem you can read the reference [2] in which the procedure of a broadening standard sample preparation and characterization is explained in detail. In that paper a KCl sample was used as it can be easily crystallized free of defects. Otherwise other materials are used by different laboratories, the yttrium-oxide is one widely used, another one is the lanthanum boride; the NIST was working on the subject to prepare one standard good for the purpose, but the fact that every diffractometer class need a different one in practice was precluding the preparation of a unique standard. Different instruments may have a completely different sampled volume, from very small (few cubic microns) at synchrotron facilities to very large (cubic centimeters) in some neutron spectrometers. So the notion of a sufficiently large statistical number of crystallites determines a maximum crystallite size that it is quite different in one case respect to the other. One final important note and recommendation: the Si standard distributed by NIST or the one included with some diffractometers is normally certified or prepared for line-spacing or peak position measurements and it is not very good for instrumental broadening determination. The principle of a instrumental broadening standard sample preparation is following the idea of choosing a materials that can be crystallized very well (consider also that the defect density, point or line, must be reduced to a very low value) and has an absorption factor not too different from the sample class you measure normally. This because the absorption controls the measured volume and thus the instrumental broadening function.

## 2 Requisite

This tutorial is based on Maud version 1.9992. But any version from 1.98 can do the tutorial. In version 2.0 the interface has been changed but you can use this tutorial just keeping in mind that some of the windows may be located or appear different. As an example of standard broadening sample measurement to analyze we will use the spectrum of the yttrium-oxide measured during the size-strain round-robin that can be found at:

[http://www.du.edu/~balzar/s-s\\_rr.htm](http://www.du.edu/~balzar/s-s_rr.htm)

A copy of that file can be downloaded along with the analysis final step completed (for checking purpose) at:

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

Download the datafile containing the yttria spectrum and put it in a location on your computer were you can subsequently load it from Maud. The standard sample broadening preparation and measurement is not described here, some recommendations are reported in the previous paragraph. Refer to the bibliography for further reading.

### 3 Procedure

Maud is quite different from other Rietveld programs in how it treats the peak broadening. Instead of using a unique classical Caglioti and similar formulas to describe the broadening change with  $2\theta$  as a sum of the instrumental and sample broadening, Maud treats separately the instrumental from the sample aberrations.

The instrumental broadening is stored in the *instrument* object through a Caglioti like formula, whereas the sample broadening is computed for each phase from the crystallite size and microstrain (plus eventually other microstructure/defects parameters). These are stored in the phase objects.

In the following procedure we will determine by fitting the standard broadening sample the instrumental part supposing the sample broadening is negligible. In Maud this can be achieved by setting to *none* the size-strain model in the phase and refining the Caglioti like parameters in the instrument object. Once this is done, in the future with normal samples, the user can use this determined instrumental function as known and fit only the microstructure parameters of the phases as broadening parameters.

Now we go to the procedure. Start Maud, if not already opened. Press the "New analysis" shortcut on the toolbar or choose "New analysis" from the "File" menu. With the "New analysis" Maud will load the analysis file "default.par" present in the directory/folder where Maud has installed all the configuration files and databases (search for it on your computer if you are not sure where it is). This file contains a starting point for the analyses and can be customized by the user to reflect its own preferred instrument and configuration. We will see at the end of this tutorial how we will use the results obtained on the instrumental broadening to update our default analysis starting point to make use of the new function measured. If the user or who has installed Maud has not changed the default.par file, it should start with a Bragg-Brentano instrument that was created using as a reference the Rigaku IID max diffractometer used in [2]. First step, you need to modify your instrument to reflect your own diffractometer that it could be not a Bragg-Brentano. In the case of this tutorial the instrument we will use is a Philips Bragg-Brentano, so click on the *Instruments* tab-panel (version 2.0 or later only: the instrument is located inside the dataset, each dataset has one, so select the datasets tab-panel, click on the available dataset and press the edit button on the toolbar, the one featuring an eye, or choose from the menu "edit" → "edit object"; in the opening window, under the general tab panel press the edit button in the instrument view), select the instrument present there and press the *edit* button. In the new window appearing you can make the following changes (some can be already done)

- change the name of the instrument to Philips
- set a initial *incident intensity* to 10
- check that the calibration models (both the angular and intensity) are set to the respective *none* model
- select the Bragg-Brentano geometry
- select the Theta-2Theta type of measurement

- select X-ray as radiation (you can initially use also the *tube radiation* for the Cu  $K\alpha$ , but because this instrument shows the presence of a small  $K\beta$  component we will use the general X-ray model and we will specify the radiation wavelengths manually to be able to refine the  $K\beta$  component at end)
- select the scintillation detector (also the standard detector is ok for this case)

The final result should be like in figure 1.

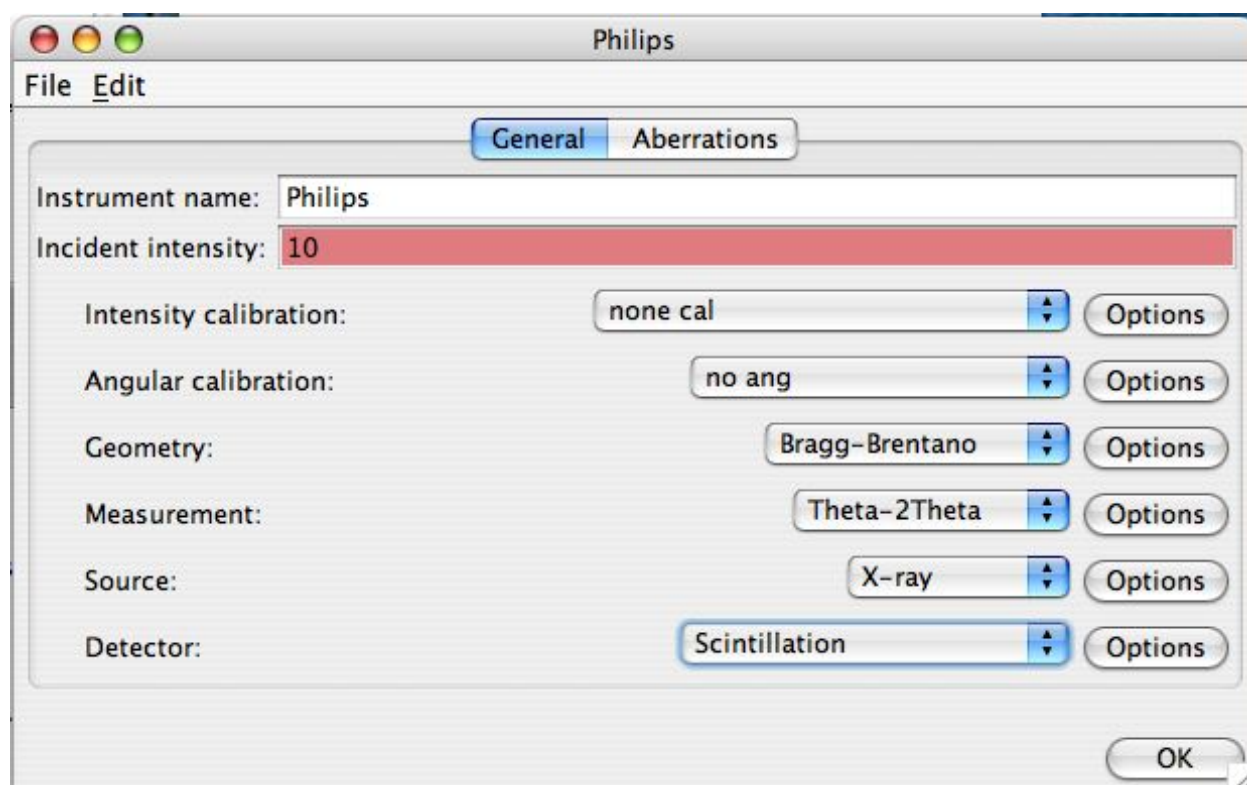


Figure 1: Starting instrument general setting.

Now for completeness we will adjust also some parameter for the Bragg-Brentano geometry to reflect more the Philips instrument, but these steps will in reality not affect the refinement or final result. For the geometry press the *Options* button near the Bragg-Brentano combo-box. A new window will appear, change the fields inside until they look like in figure 2.

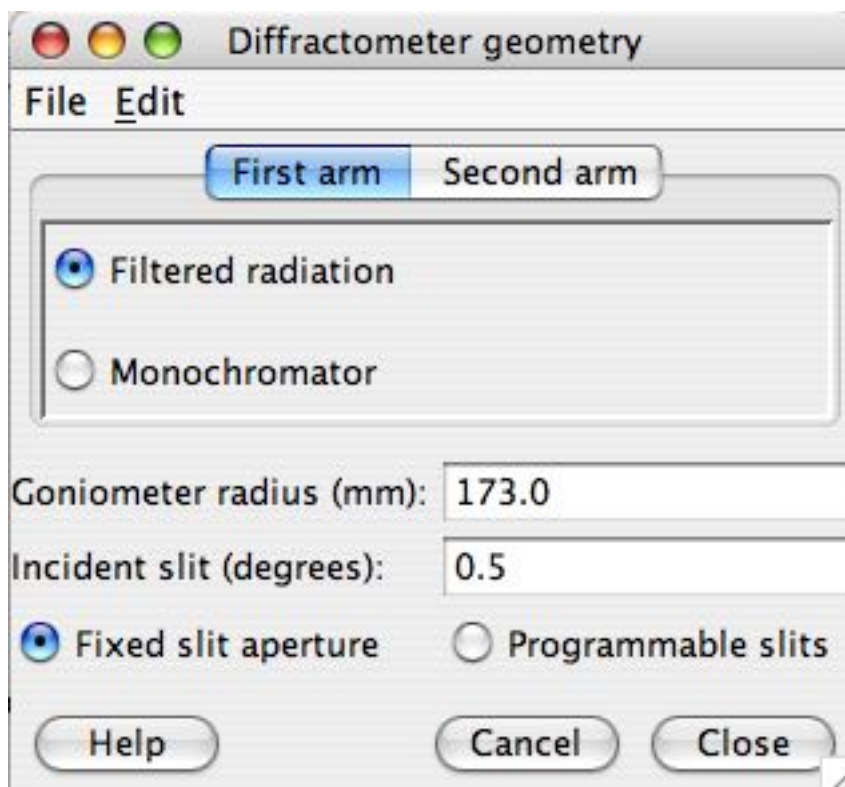


Figure 2: Bragg-Brentano setting.

For the *second arm* you should also select *filtered radiation* as the instrument was not having a monochromator (as proved by the  $K\beta$  component). Close the box using the *Close* button; closing the box with the window closing small button on the window bar does not assure that the last field changed was confirmed (this is valid in general). Now do the same for the *X-ray* radiation. Be sure to select as source X-ray instead of X-ray tube (this is not customizable as we need later for the  $K\beta$  component). After pressing the *Options* button for it, add and change the terms in there until you have the two radiations *Kalpha1* and *Kalpha2* settled up to 1.5405981 and 1.54447 for the wavelengths and 1.0 and 0.5 for the respective weights. To change the name of the radiation, double click the radiation name (only for aesthetic purposes). The final result should resemble that in figure 3.

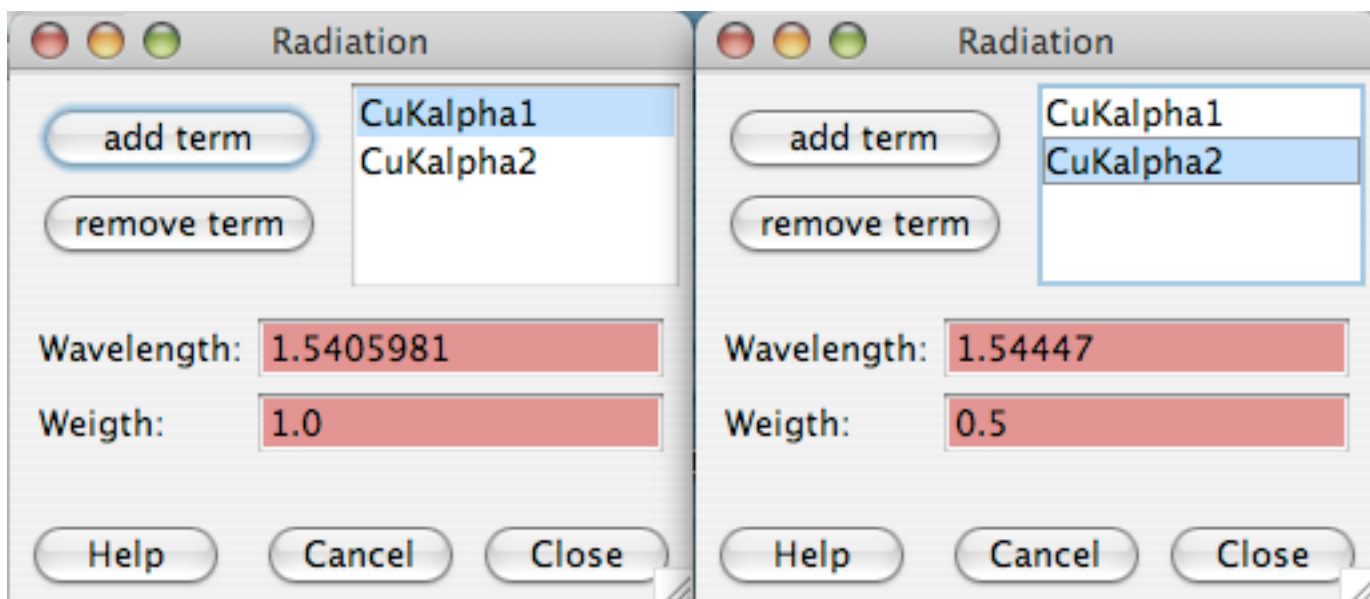


Figure 3: Setting for the  $K\alpha_1$ ,  $K\alpha_2$  radiation.

Now we need to check and setup the starting aberration parameters (instrumental broadening function and  $2\theta$  or d-spacing instrument shifts). Still in the instrument edit window click on the *Aberrations* tab-panel. Set the starting parameters by removing/adding, if necessary, and changing the values of them as in the following:

- Asymmetry: only 2 parameters, both 0.0; *Truncation angle* : 0.4; *force Tan(theta) correction* : on
- HWHM: 3 parameters, all 0.0 except the first equals to 0.005; *Force Caglioti in Tan(theta)* : on (in version 2 you don't need to turn it on for  $2\theta$  like measurements)
- Gaussianity: 2 parameters, the first equals to 0.5, the second 0.0 (the first is the intercept, the second the slope; the Gaussianity goes from 0.0 to 1.0)
- $2\theta$  displacement: one parameter equal to 0.0
- no Omega, Chi or Eta broadening (these are to be used only if you are determining the instrumental function for several spectra collected also changing Chi, Omega or Eta angles; typical of texture-stress instruments).

Figure 4 reports how it looks for the Caglioti (HWHM) tab-panel selected.

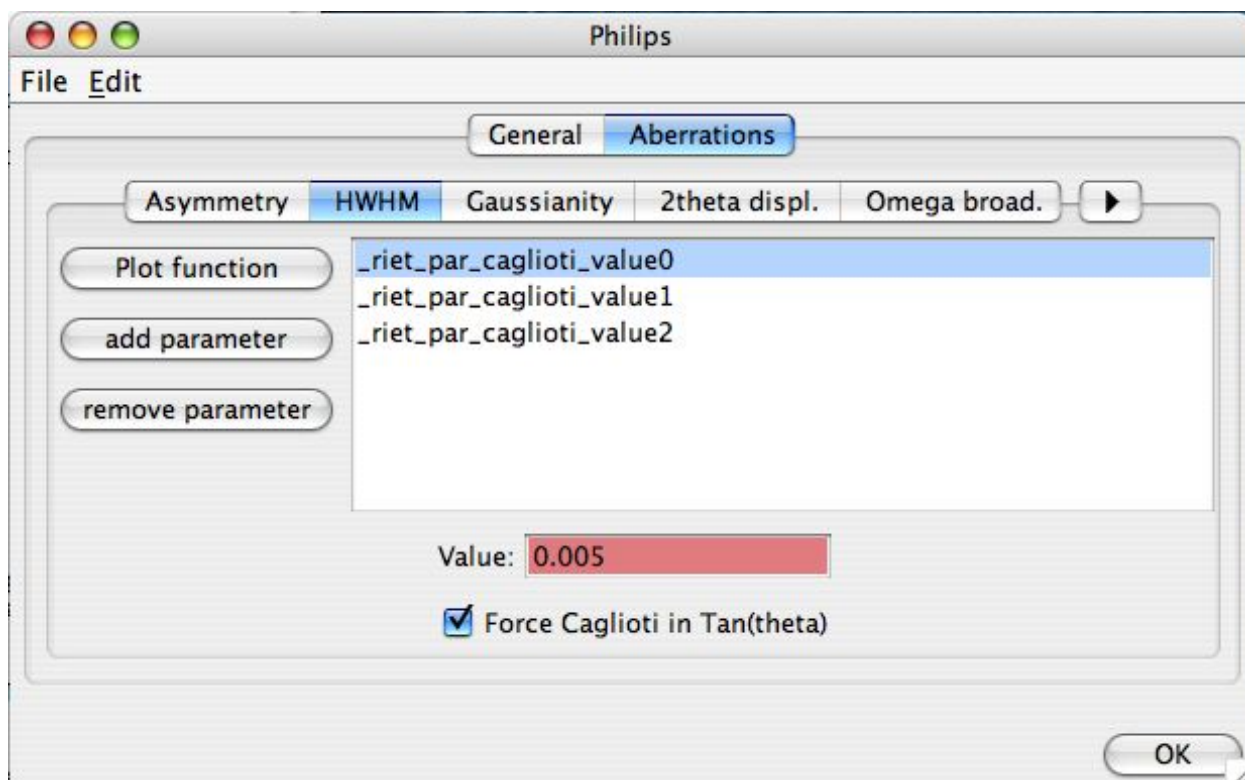


Figure 4: Starting setting for the HWHM or Caglioti part of the instrumental aberrations.

Now we can close the instrument edit panel pressing the *OK* button in the lower-right corner of the window.

In the main window of Maud select the *Data sets* tab-panel (Version 2.0 or later only: closing the instrument window you are already in the dataset edit window so you don't need to open it). Select the available dataset and press the edit button to show the Data sets edit window. First we load the spectrum from the datafile. Go to the *Datafiles* tab-panel and press the *Browse* button, in the file dialog search, select and load the CPD-Y2O3.PRN datafile previously downloaded with this tutorial. The spectrum was collected for the size-strain round-robin [1] and the original can be downloaded at [http://www.du.edu/~balzar/s-s\\_rr.htm](http://www.du.edu/~balzar/s-s_rr.htm). Note: the help button in the lower-left corner will present a window containing the datafile formats supported by Maud.

Select the datafile you have loaded in the datafiles tab-panel and press the *View* button to see a plot of the spectrum. It should look like the one in figure 5. The spectrum has an increasing background at low angle without peaks. To save time we can avoid that part by selecting a narrower range. Press and hold the right mouse button (ctrl+click for Mac OS X if your mouse has only one button) inside the plot window to show a small menu and select *Show coordinates* from it. A small window will appear in the upper-left corner of your screen and moving the mouse on the plot you can follow the actual coordinates. As you can check we can safely start our spectrum from 15 degrees up to 150 and avoid the no peaks region.

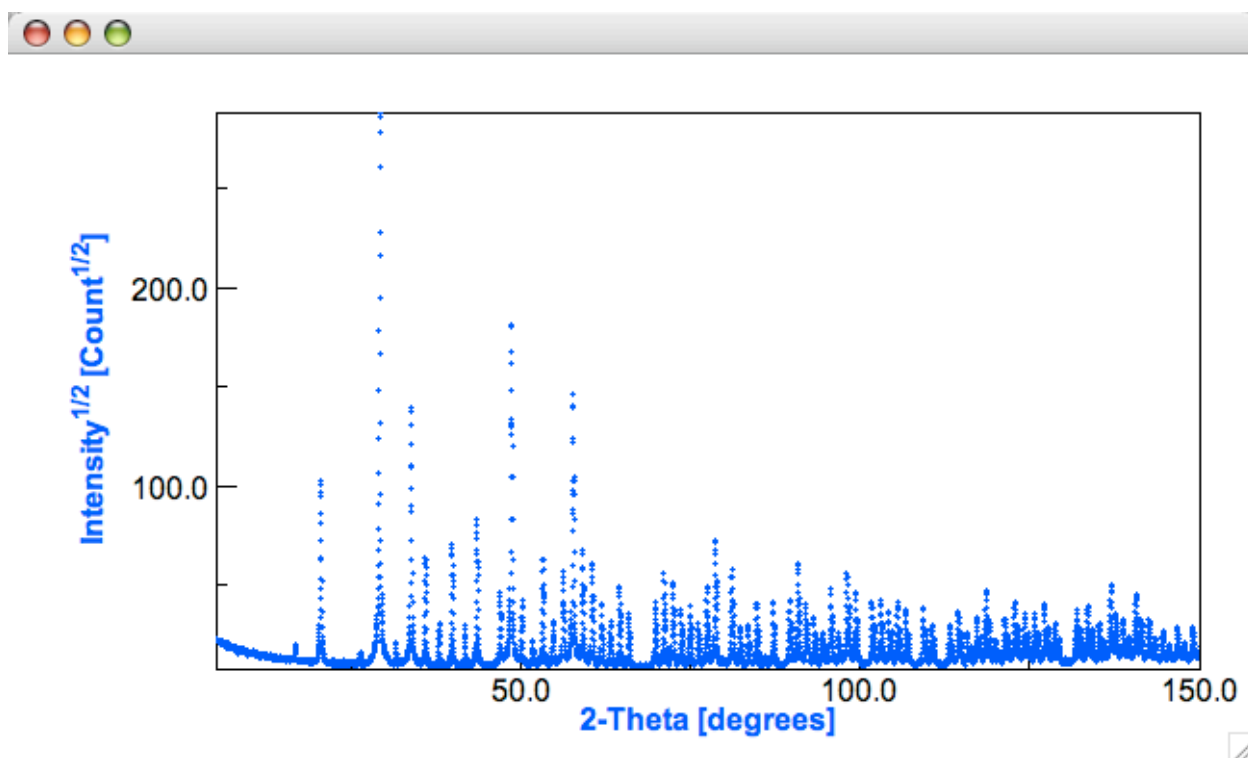


Figure 5: The CPD-Y<sub>2</sub>O<sub>3</sub> spectrum collected for the size-strain round-robin [1].

Close the plot window and select the *General* tab-panel in the dataset edit window. In the *Computation range* set the 2Theta minimum to 15 and the maximum to 150 (or >150). If you want to see now the result of this setting, press the *Apply now* button and go back to plot again (with the *View* button) the spectrum. Check the *Background function* under *Polynomial* if it is composed by 5 parameters. As the range is quite large we need a 4th degree polynomial function, i.e. 5 parameters. Adjust the number of parameters to the desired value by adding or removing parameters (to remove a parameter you need to select one). Close the dataset edit window when you have finished to come back to the main Maud window. The spectrum we just loaded was collected for a perfectly crystallized Y<sub>2</sub>O<sub>3</sub> sample that virtually should give no sample broadening. We need to add the corresponding Yttrium-Oxide phase by manually specifying its crystallographic setting or by loading it from a CIF file or database if we already have its structure somewhere. In the example files of Maud (extracted the first time you have run Maud) there is a Maud analysis file called *y2o3.par* that contain a Yttrium-Oxide phase in CIF format. We can use it as database from where to load our phase. So, in the Maud main window select the *Phases* tab-panel. There should be no phases there. In case not, remove all phases present there. Press the *Add from database* button (Version 2.0 or later only: this is located in the toolbar, let the mouse to stay over a toolbar button to get a tooltip about its function; or use the menu edit→load object from CIF) and navigate to the *y2o3.par* file (it should be in the *maud* directory containing databases, examples, the *default.par*



mentioned before and preferences). Load it, a window will appear showing a list of phases with only one name, Yttrium-Oxide (if nothing appears it means that in the file you loaded there was no CIF object corresponding to the tab-panel you are currently in; check if you are in the *Phases* tab-panel). Select the only phase and press the *Choose* button. *Note: there should be one Yttrium-oxide structure also in the structures.mdb file in the same directory. This file is a simple database containing some structures in CIF format made by adding to it phases created under Maud and saved to the database with the corresponding Maud button.*

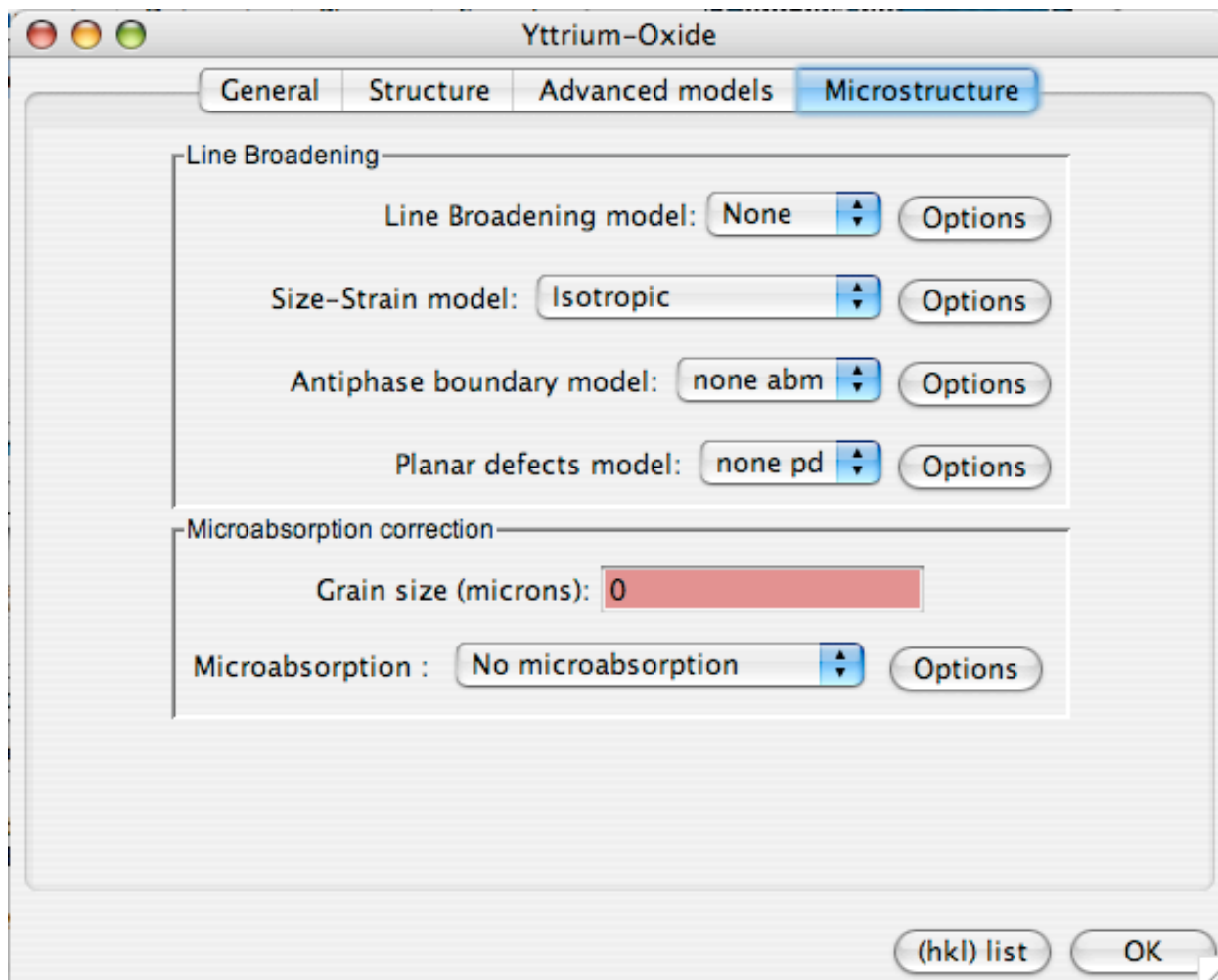
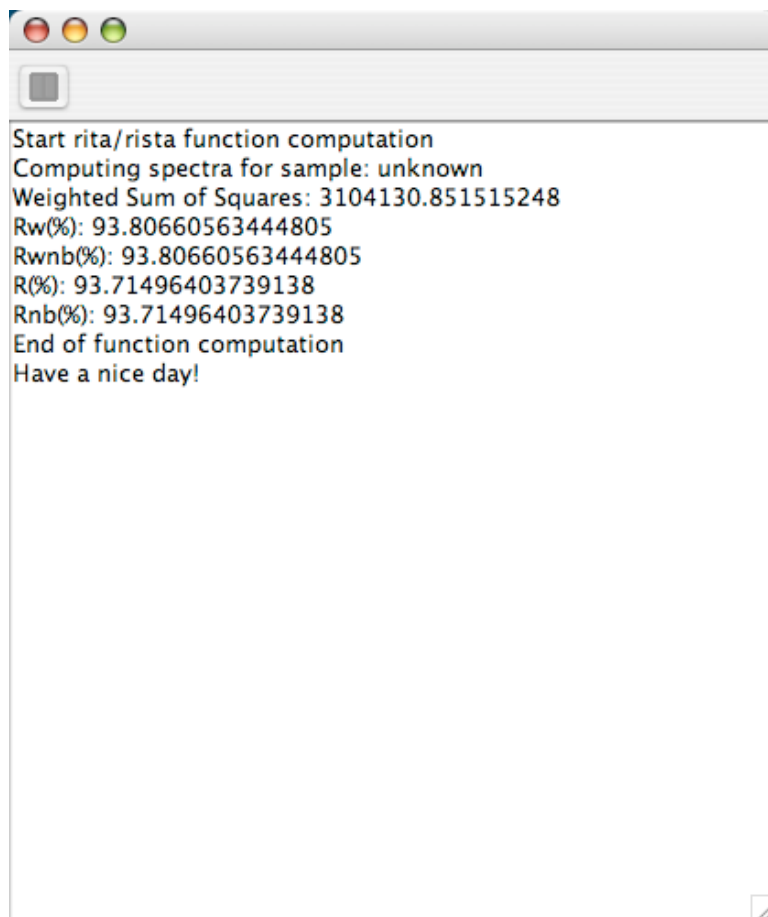


Figure 6: Setting for the microstructure in order to avoid sample broadening effects.

Now, select the Yttrium-Oxide phase in the tab-panel and press the edit button (Version 2.0 or later only: refer always to the toolbar edit button or under the edit menu). You can check the space-group setting and atomic positions. Now we need to specify that our sample has virtually no broadening due to crystallite sizes and microstrains. This can be done in two ways, both equally valid. Go to the *Microstructure* tab-panel and check the *Line Broadening model* . If you have loaded the

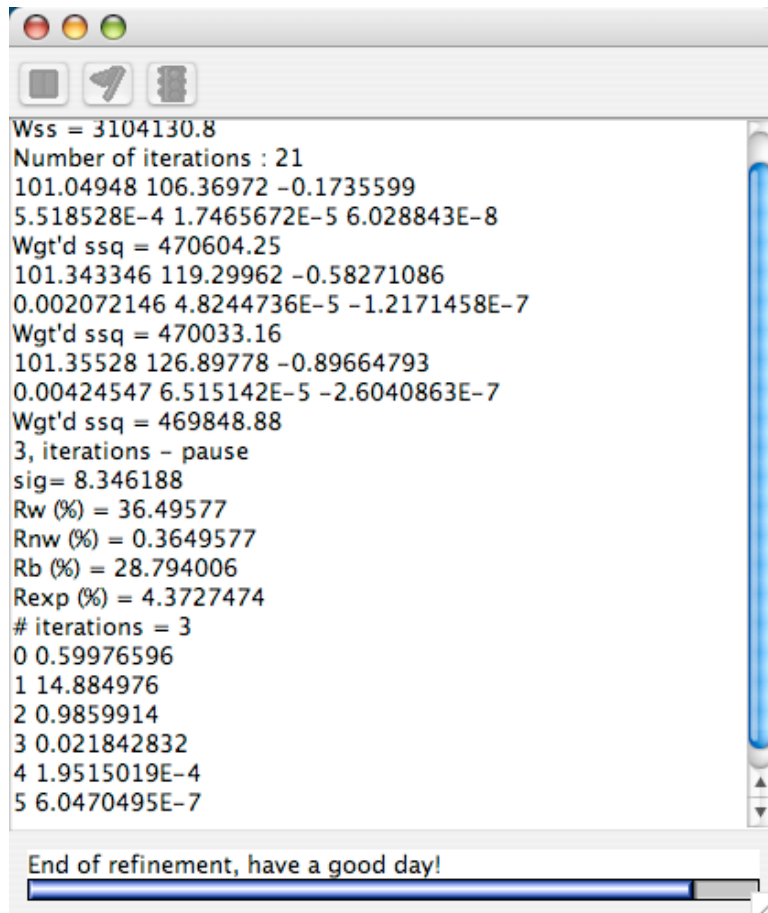
phase from the y2o3.par file it is already set to *None* (the Maud default value is *Delf*, the model described in the Rietveld book [3] by De Keijser et al.; in this case the y2o3.par file correspond to the analysis of this CPD-Y2O3.PRN file, so the model was already set to *None*). Otherwise set the model to *None*. This is the first way to avoid sample broadening. The second way of doing it, is to leave or set the line broadening model to *Delf* or whatever and in the *Size-Strain model* (the default is *Isotropic*) under Options set all crystallite size and microstrains parameters to zero. With the sample broadening set to zero, the entire broadening of the spectrum is controlled by the instrumental broadening in the aberrations tab-panel of the instrument edit window and mainly by the Caglioti (HWHM) formula and asymmetry. The final result should look like that in figure 6. Close the phase edit window and come back to the main window. Now we are ready to start with the analysis. First we check that we have the correct phase and everything is ok. Go to the main menu, under *Refinement* select *Compute spectra*. Or, to do the same, press the calculator button in the toolbar. The program will compute the spectrum based on the actual setting of the parameters (without any refinement) and will show the relative fitting indexes in a new window. The result is shown in figure 7.



```
Start rita/rista function computation
Computing spectra for sample: unknown
Weighted Sum of Squares: 3104130.851515248
Rw(%): 93.80660563444805
Rwnb(%): 93.80660563444805
R(%): 93.71496403739138
Rnb(%): 93.71496403739138
End of function computation
Have a nice day!
```

Figure 7: Fitting indexes from the starting model, no refinement done.

Select the dataset in use in the Data sets tab-panel and press the toolbar button with a plot icon (or select from the *Graphic* menu *Plot selected dataset* ). A window will appear showing the spectrum with the computed function, the peak positions and a difference plot panel (Version 2.0 or later only: you don't need to open a plot window, the plot is already in the main window). As you can notice background and intensity of the phase will appear lower than the experimental spectrum, but nearly all peaks seems to be corrects. It is a sufficiently good starting point. Close the plot window and we proceed. Before to actually do the refinement it is better to save the starting analysis. So from the menu select *File* and *Save analysis as* and specify a location and name for your analysis. It is advised to save the analysis file using the *.par* extension to recognize a Maud analysis file and in the same directory or near by the datafile containing the spectrum, as this will be not saved entirely in the analysis file, but only a relative reference to its name and location. So when you move the analysis you will move both the datafile and analysis file to maintain the validity (and always leave them in the same relative position, otherwise you will have to reload again the datafile as the analysis file will loose its location; just remember the datafile location is saved as a relative path to the analysis file and not as an absolute path)(Version 2.043 or later only: in the menu *Analysis-ζoptions* a window will appear, checking the option button *store spectra in the analysis file* you don't need to care any more to the datafile location). Select from the menu *Refinement* (version 2.0 or later only: the menu has changed name to *analysis* ) and *Wizard* . In the window appearing we will proceed by refining first the background and scale parameters. So in the left panel select *Background and scale parameters* and press the *Go!* button. The program will refine these parameters and the progress will be shown in the usual window as in figure 8.



```
Wss = 3104130.8
Number of iterations : 21
101.04948 106.36972 -0.1735599
5.518528E-4 1.7465672E-5 6.028843E-8
Wgt'd ssq = 470604.25
101.343346 119.29962 -0.58271086
0.002072146 4.8244736E-5 -1.2171458E-7
Wgt'd ssq = 470033.16
101.35528 126.89778 -0.89664793
0.00424547 6.515142E-5 -2.6040863E-7
Wgt'd ssq = 469848.88
3, iterations - pause
sig= 8.346188
Rw (%) = 36.49577
Rnw (%) = 0.3649577
Rb (%) = 28.794006
Rexp (%) = 4.3727474
# iterations = 3
0 0.59976596
1 14.884976
2 0.9859914
3 0.021842832
4 1.9515019E-4
5 6.0470495E-7

End of refinement, have a good day!
```

Figure 8: Refinements indexes after the scale and background parameters refinement.

You can plot the spectrum as before to check visually the refinement progress after this first cycle. Now select again the wizard panel from the refinement menu and proceed with the second cycle: *Previous + basic phase parameters* . Press the *Go!* button again.

Name	Value	Min	Max	Error	Status	Output
y2o3.par	-	-	-	-	*****	false
Philips	-	-	-	-	*****	false
_pd_proc_intensity_incident	130.50658	0.0	10000.0	0.9001812	Refined	false
_riet_par_asymmetry_truncation	0.4	-0.44	0.36	0.0	Fixed	false
_riet_par_asymmetry_value0	200	-271.641...	332.00662	0.0	Refined	false
_riet_par_asymmetry_value1	0	-2.522707	2.064033	0.0	Refined	false
_riet_par_caglioti_value0	0.005	-0.00124...	0.001520...	0.0	Refined	false
_riet_par_caglioti_value1	0.0	-0.02445...	0.029883...	0.0	Refined	false
_riet_par_caglioti_value2	0.0	-0.02445...	0.029883...	0.0	Refined	false
_riet_par_gaussian_value0	0.5	0.0	1.0	0.0	Fixed	false
_riet_par_gaussian_value1	0.0	-0.108	0.132	0.0	Fixed	false
_riet_par_2-theta_offset0	0.009974...	0.0	0.0	9.917948...	Refined	false
none cal	-	-	-	-	*****	false
none	-	-	-	-	*****	false
Bragg-Brentano	-	-	-	-	*****	false
_diffn_radiation_polarisn_norm	0.0	0.0	360.0	0.0	Fixed	false
_diffn_radiation_polarisn_ratio	0.0	0.0	1.0	0.0	Fixed	false
Theta-2Theta	-	-	-	-	*****	false
X-ray	-	-	-	-	*****	false
CuKalpha1	-	-	-	-	*****	false
_diffn_radiation_wavelength	1.5405981	0.1	10.0	0.0	Fixed	false

Commands			
Fix all parameters	Free all parameters	Free backgrounds	Free scale pars
Free basic pars	Bound B factors	Free microstructure	Free crystal struct
Free texture	Free strain	Fix backgrounds	Fix texture
Fix strain	Bound datafiles by bank	Bound datafiles by angles	Free all spectra monitors
Fix all spectra monitors	Bound all banks coeffs	Uniform all banks	Shrink min max values
Enlarge min max values	Center min max range		

Parameter preferences	Expand all	Close
-----------------------	------------	-------

Figure 9: Status of the aberration parameters in the *Parameter List* window after changing it.

The program will refine also the cell parameters and a 2Theta shift parameter. To check at end which parameters were refined you can use the *Parameter List* window (from the refinement menu), expanding all objects and checking which have the status to *refined*. By looking at the spectrum fitting (try to zoom some parts by dragging a rectangle with the mouse around the region you want to enlarge), you can see that the main error is now due to the peak broadening. We need to refine the instrumental function by optimizing the aberrations parameters (HWM, Gaussian content, Asymmetry). So, open again the *Parameter List* window. Expand the instrument object

(if you changed the name it should be Philips or whatever name you specified) and set the status to *refined* for the asymmetry and Caglioti parameters. Also we cannot start from an asymmetry function equal to 0.0 because apart this particular value, in general small values means high asymmetry. So change manually the asymmetry value of the first parameter to 200. We will not refine the Gaussian parameters at the moment as they are less sensible until we start to have a satisfactory good fit on the peak broadening. The status of the instrument parameters should look like in Figure 9. We set refinable also the crystal structure to provide extra degree of freedom for the fitting of peak intensities. You can easily do that by pressing the *Free Crystal struct* button in the bottom part of the window. If you don't see buttons try to enlarge down the window by dragging the corner. By expanding all objects you can check which parameters of the Yttrium-Oxide phase were set *refined*. Check in particular the B factor for the Oxygen. If the status is *Equal to*, set it to *refined*.

```

Wgt'd ssq = 24967.73
131.6745 128.63358 1.3899084
0.010573943 -0.0033049823 0.006399411
0.40144056 0.0029162238 0.01821225
192.56476 -6.2486815 0.11173496
-8.2631665E-4 2.2145691E-6 10.602886
0.27189 0.4675444 0.10872592
0.34777004 0.119037695 0.4971475
Wgt'd ssq = 24861.082
15, iterations - pause
sig= 1.921996
Rw (%) = 8.395053
Rnw (%) = 0.08395053
Rb (%) = 6.106691
Rexp (%) = 4.3678823
# iterations = 15
0 0.24676965
1 4.958355
2 0.17398432
3 7.169882E-5
4 1.3281315E-4
5 2.760222E-5
6 0.0050180024
7 8.818533E-5
End of refinement, have a good day!

```

Figure 10: End of refinement and fitting indices for this example.

Now close the window and start the refinement by pressing the hammer icon in the toolbar or choosing the corresponding *refine* menu item in the *refinement* menu. After the iterations you can

check again the plot, the  $R_w$  should arrive near 10 %. Now we add to the refinement also the Gaussian content (two parameters) using again the *Parameter List* window inside the Philips instrument object, set both parameters to *refined* . Now perform one, two or more refinement cycles until you see the Weighted Sum of Squares (Wgt'd ssq) converging to a unique value. In case adjust the number of iterations to let the program converging more. The final result should resemble that of Figure 10. We can further improve the refinement by optimizing some few addition parameters and in particular the incident radiation the  $K\alpha_1$ ,  $K\alpha_2$  separation and intensity ratio. And additionally we can notice in the plotting window by zooming near the 26.3 degrees that there is a small  $K\beta$  component (see Figure 11).

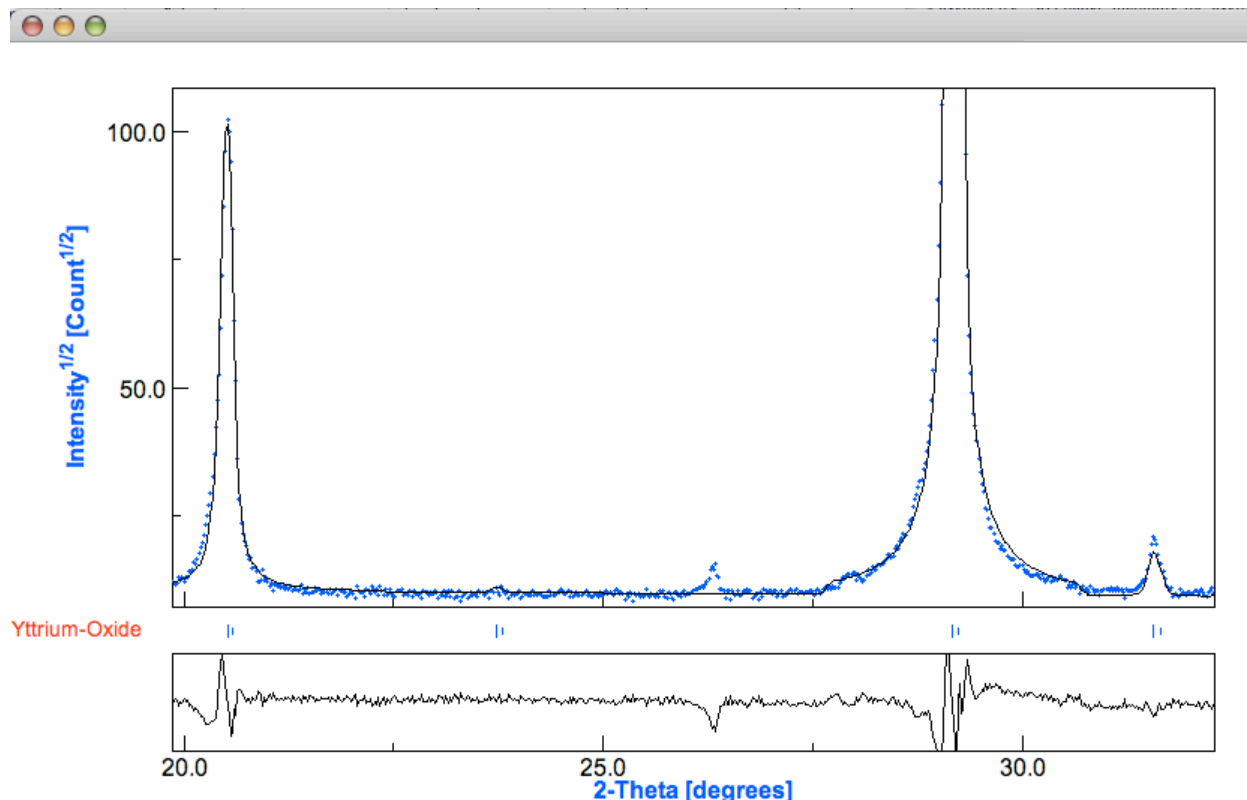


Figure 11: Zoom in the plot fitting window to see the  $K\beta$  component around 26.3 degrees in  $2\theta$ .

Go to the instrument edit window and open the radiation frame using the *Options* button for the radiation model. Add a third wavelength and call it  $K\beta$ . Set as initial values for the parameters a wavelength of 1.39 and an intensity (weight) of 0.005 (see Figure 12). Click the right mouse button over the wavelength and weight fields to change the status of the parameters to *refined*. Do it for both the  $K\alpha_2$  and  $K\beta$  radiations.

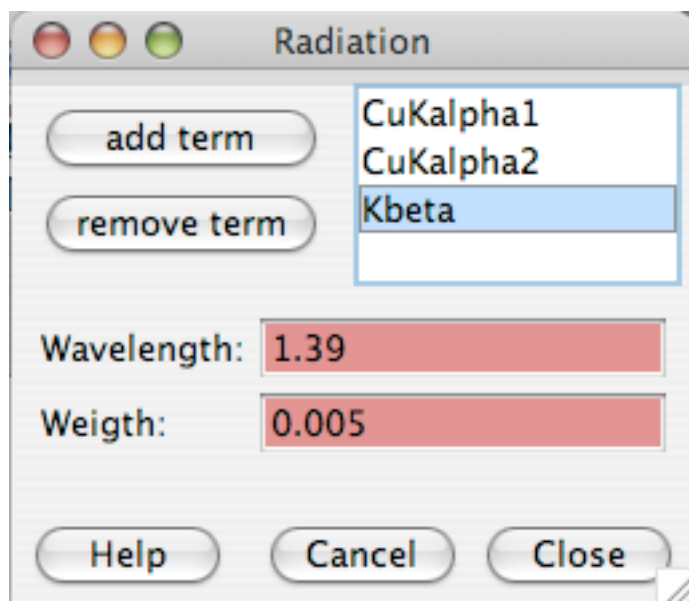


Figure 12: Adding the  $K\beta$  component in the radiation panel.

Now close the windows to come back to the main frame. Hit the compute button and plot the fitting. You will see that the peak at 26.3 is really a  $K\beta$  component; actually the weight we start with is a little bit too much, but the program will refine it. Close the plot window and start again the refinement. Do a sufficient number of iterations to reach convergence. You should arrive to an Rwp value lower than 8 %. In the case of this tutorial writing the final result is reported in Figure 13. If you want to save the analysis done up to the present choose *Save* item from the *File* menu and save the analysis. It is possible to check the broadening function by using the *Plot function* button for the HWHM, Asymmetry and other function in their respective tab-panel under the instrument edit window.

We have refined the instrumental function for this instrument using the Yttrium-Oxide well crystallized sample. Now we can store the instrument so refined for future use in all subsequent analyses, in particular when crystallite size and microstrain as to be analyzed accurately.



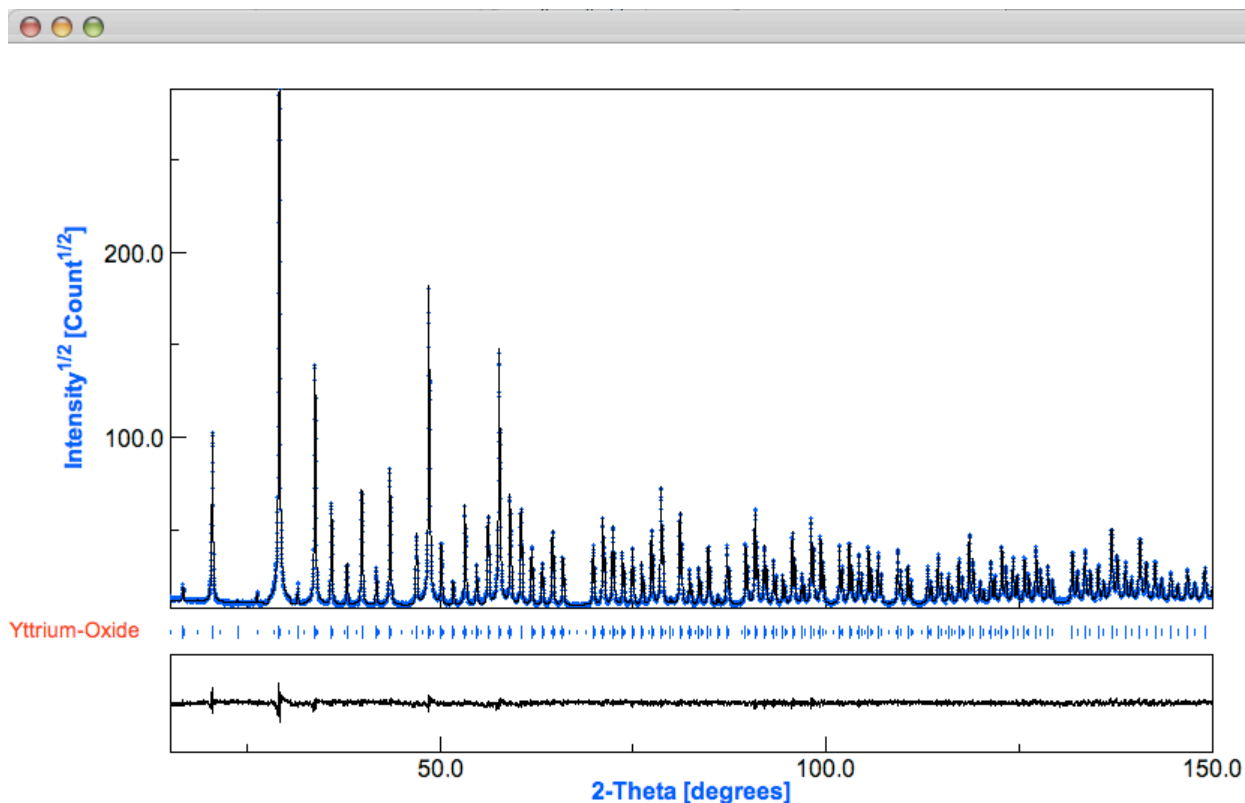


Figure 13: The final fitting result.

Now to use the aberration parameters you have refined (if you do the same as this example with your laboratory or elsewhere instrument) for future analyses, open the *Parameter List* window and press the *Fix all parameters* button to set the status of all parameters to *Fixed*. Close the window and select the instrument tab-panel. Select the instrument and press the *Save on database* button to store the instrument in the database for future easy retrieval. There is already a Instrument database in the Maud preferences and examples directory, called *instrument.mdb*. Otherwise you can start a new one by specifying a new name. Also we can set this instrument as the default one to start with when we press the *New analysis* toolbar button or the corresponding menu item. To do it, you should have already added this instrument to your instrument database. Now press the *New analysis* toolbar button and the *default.par* file from the Maud preferences and examples directory will be loaded as a starting point. Remove the instrument, by selecting it and using the *Remove* button. Now, still remaining under the instrument tab-panel, press the *Add from database* button and load the instrument you have previously saved from the database. Now save this analysis without changing the name. It will overwrite the *default.par* and this one with the instrument refined by you will become the default one.

## 4 Final notes

The accuracy of any line broadening analyses depend on how accurate is the knowledge of the instrumental broadening and so rely on the analysis of the very well crystallized reference sample, on how good is this standard in reproducing the instrumental broadening. These are not the only factor influencing the analysis, but in any case are some fundamental ones.

Any time you change the configuration of your instrument in a way that the instrument broadening may change (kind of tube/radiation, slits or monochromator etc.) you need to do again the instrument broadening determination. If you have an instrument that may have different setups you can do the analysis for each setup and store all of them.

If your instrument uses different angular tilting like a  $\chi$  tilting or fixed but different  $\omega$ , you should collect more spectra at different tilting covering your typical ranges (for example if you normally use  $\chi$  from 0 to 60 degrees, collect a spectrum for each  $\chi$  from 0 to 60 in steps of 5 degrees) and then add and refine in the instrument aberration panel omega, chi or eta parameters (at least two for each and only angle you are using).

In the next tutorial (Microstructural Analysis), we will explain how to analyze crystallite size and microstrain. It requires the knowledge of the instrumental broadening function as explained in this tutorial.

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