TUTORIAL FOR MAUD
S. MERKEL – UNIVERSITÉ DE LILLE
2025/11 VERSION

Content

INTRODUCTION

This tutorial was originally written by Luca Lutterotti, Roman Vasin, and Hans-Rudolf Wenk and can be found at http://eps.berkeley.edu/~wenk/TexturePage/MAUD.htm along with the companion paper:

> L. Lutterotti, R. Vasin & H.-R. Wenk. Rietveld texture analysis from synchrotron diffraction images. I. Calibration and basic analysis (2014) *Powder Diffr.* **29** 76-84 doi: 10.1017/S0885715613001346

It was updated by S. Merkel at Univ. Lille France, with additional figures and up-to-date screenshots. The text was modified following student feedback and failures to follow the instructions during training labs at Univ. Lille. The original version also used a detector geometry which is considered obsolete in MAUD. This version uses the commended *Inclined Detector Geometry.*

The Lille version of the tutorial is available at http://merkel.texture.rocks/Cours/MAUD/

DOWNLOAD SOFTWARE AND DATA FILES

The latest version of MAUD can always be found at https://luttero.github.io/maud/. MAUD is written in Java and works with any operating system for which a Java implementation is available (Windows, Mac OS X, Linux, and Unix).

You will also find

- *MAUD-2025-PartI-CeO2.zip* which contains:
  - Starting files
    - Diffraction image for CeO_2 standard: CeO2-00010.tif
    - Crystallography information file (crystal structure) for CeO_2: CeO2.cif
  - Final solutions:
    - Refined instrument parameters for CeO_2: APS-BESSRC-11-ID-C-InclinedReflection.ins
    - MAUD parameter file for CeO2 with completed analysis with a 2025 MAUD version and the inclined detector geometry (for checking your attemps): CeO2_2025_calibration_final.par
- *MAUD-2025-Part-I-Coin.zip* which contains:
  - Diffraction images for Nickel coin: Nickel-00178-40.tif Nickel-00179-20.tif, Nickel-00180-0.tif, Nickel-00181+20.tif, Nickel-00182+40.tif
  - MAUD parameter file for Nickel coin with completed analysis with a 2025 MAUD version and the inclined detector geometry (for checking): NCoin_2025_06.par

A note on file extensions

MAUD analysis relies on several types of files. Please ***be rigorous and pay attention to those***. You will be lost in you data analysis if don't.

- Diffraction images, from the experiment, are often saved in "*TIF*"
- The raw data (the diffraction image), converted to MAUD format, will use "*ESG*". Those are MAUD-specific plain text files, which you can explore with a text editor.
- Crystal information are saved in "*CIF*". A plain text file defined by the International Union of Crystallography.
- MAUD analyzes are saved as "*PAR*". This is a MAUD-specific plain text format which includes all information about the analysis, the raw data, the cif information for all phases in your sample, and the physical model used to fit the data.

EXPERIMENT AND OBJECTIVES

A basic configuration for X-ray diffraction experiment, which we will use below, is shown in Figure 1.

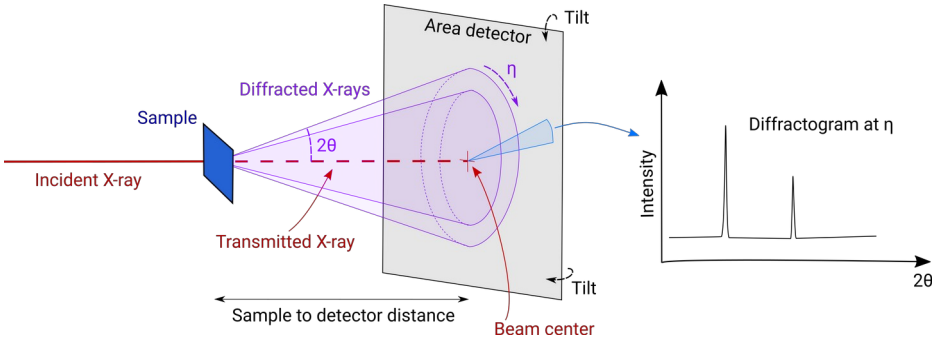

Figure 1: Experiment geometry

The sample properties are measured using X-ray diffraction, on a detector placed away from the sample. We are interested in analyzing the diffraction pattern (intensity as a function of 2θ) but how the diffraction pattern changes with the azimuth angle η. All contain information on the crystal structure (atomic positions, crystal symmetry, cell parameters, etc) but also microstructural features (grain size, strain, texture, etc).

Before you start looking at the sample properties, however, you need to know the properties of your instrument:

- the characteristics of the your X-ray source: wavelength, spatial distortion, etc,
- your detector:
  - where it is, in distance, but also whether it is tilted, etc,
  - whether it induces a specific peak shape, which is not related to sample properties.

The first section of the tutorial will guide you on how to define the properties of your instrument, using data measured on a CeO_2 standard for which you know all sample properties. The calibrated instrument can then be used to study the actual sample you are interested in.

The second section will guide you through the analysis of a real dataset. Part II is optional. You will be working on another dataset in the following lesson.

PART 1: STEP-BY-STEP PROCEDURE OF IMAGE PROCESSING AND INSTRUMENT CALIBRATION WITH CeO_2 STANDARD

When you run MAUD for the first time, a window will open with many options and displays as shown in Figure 2. It has three main tabs (left side, top): "*Datasets*", "*Phases*" and "*Sample*".

Most frequently used commands are present as icons in the toolbar, *e.g.*, "*Disk*" to save current analysis, "*Eye*" to edit a selected object, "*Calculator*" to compute model diffraction patterns, "*Hammer*" to start a refinement, etc.

Please take some time to explore the MAUD interface, the three tabs for Datasets, phases, and sample. Test the effect of the Eye, Calculation, and Hammer icons.

1. Defining the instrument

An instrument is defined for each dataset.

- Select the default dataset under "*Datasets*" and edit it (menu: "*Edit→Edit object*" or click the "*Eye*" icon in the toolbar).
- Locate "*Diffraction Instrument*" in "*Instrument*" panel under "*General*" tab.
- Click "*Edit*" button. This opens the Instrument editing window (Figure 3)

Step by step preparation of the instrument:

- Rename the instrument to "*APS-BESSRC 11-ID-C*" so you can add it later to the instrument database and recognize it when needed.
- Adjust "*Incident intensity*" value to 0.001.

- Check that the option for "*Intensity calibration*" is "*none cal*".
- Choose as "*Angular calibration*" model:
  - Choose "*Inclined Reflection Image*";
  - Click "*Options*" button next to it and
    - Change the "*Detector distance*" to 1850 mm. The distance is needed for the conversion of image coordinates to 2θ, for now it is just an estimate and this value will be refined later.
    - Set the "*Center x error*" and "*Center y error*" to 0
    - Set all detector angles to 0 ("*Detector 2theta*", "*Detector tilt*", "*Detector rotation*", "*Detector eta*")
    - All will be optimized during the tutorial by a calibration model.
- For "*Geometry*" choose "*Image 2D*".
- For "*Measurement* choose "*2Theta*".
- For "*Source*":
  - Select "*Synchrotron*",
  - Click "*Options*" and change default wavelength to 0.10798 (Å).
- In "*Instrument Broadening"* we should set the default parameters to be in line with synchrotron diffraction!
  - Click "*Options*" button next to the "*Caglioti PV*" model (Caglioti *et al.*, 1958)
  - Remove all asymmetry parameters (or set them zero)
  - Under "*HWHM*" tab set all parameters to zero except for the first (the constant value, equal to W of the Caglioti formula), set it equal to 0.00025.
- Close the Instrument editing window.

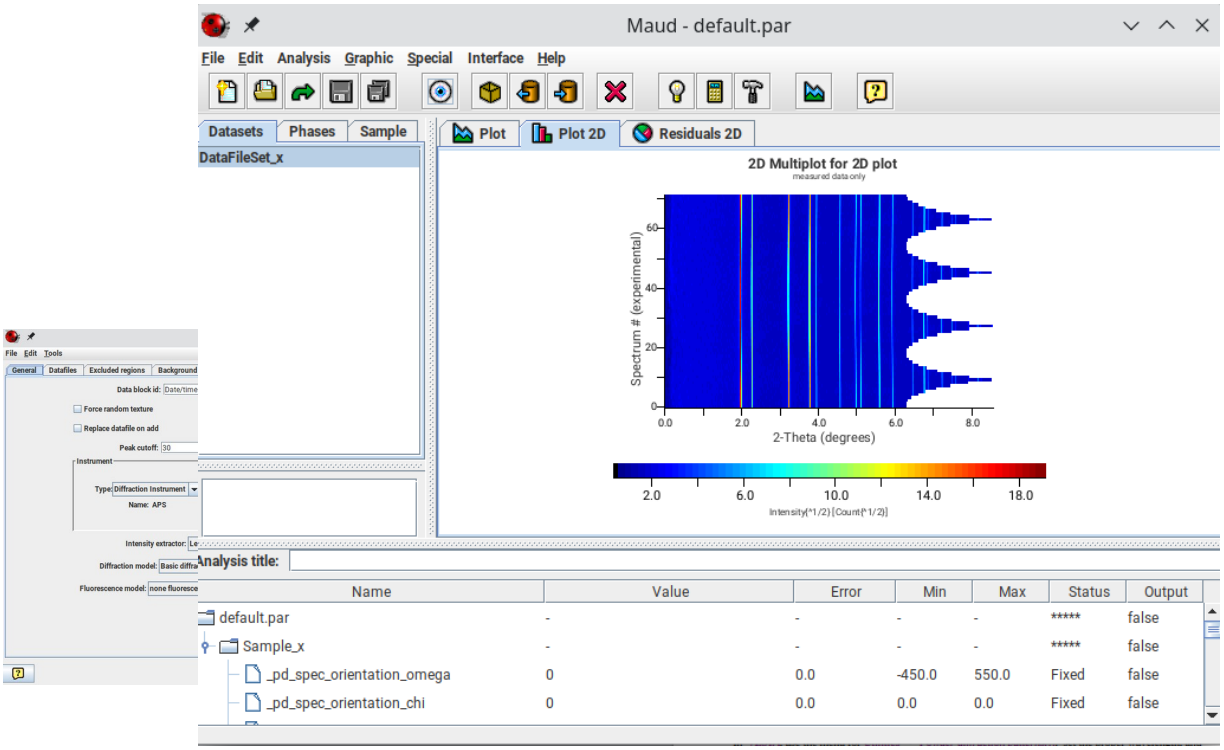

Figure 2. Main MAUD window on computer running Linux in 2024. Superposed is a window "DataFileSet" with options and settings for the selected Dataset. At the start the plot window is blank. Here data for CeO2 standard have been entered but not refined.

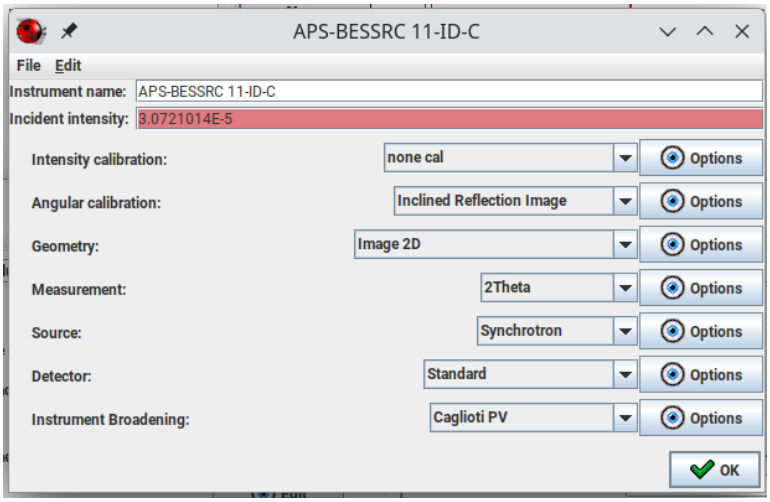

Figure 3. "Diffraction Instrument" window with general setup for synchrotron diffraction images.

2. Entering 2D image data

With the ImageJ plugin embedded in MAUD a *.tif* image file can be directly entered into MAUD and saved as *.esg* files, which are ASCII files containing a list of radial positions or diffraction angles and corresponding experimental intensities. If the diffraction data is in another detector format not recognized by ImageJ, it has to be converted into a regular *.tif* file, preserving the original intensities.

To perform the integration in MAUD using the ImageJ library:

- Remain in the "*Datasets*" window, select "*Datafiles*" tab (Figure 4)
- Press the button "*From images..*" to start the ImageJ plugin.
- A small window with the typical ImageJ toolbar will appear (Figure 5, top right).

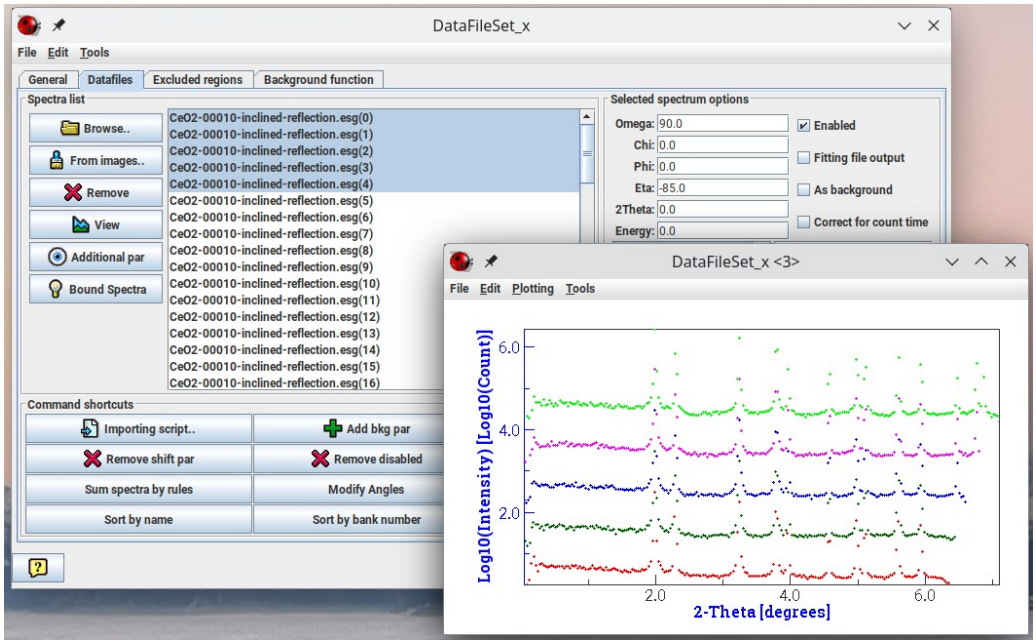

Figure 4. "Datafiles" in Dataset options window for CeO2 standard, viewing five of the diffraction patterns (.esg file) in the insert. Initially there are no data in the window. The screenshot has been taken after they have been loaded "From images".

Step by step conversion of a Tif image to MAUD own ESG format:

- From the ImageJ menu "*File→Open…*" load the image *CeO2-00010.tif*.
- From menu "*Image→Adjust→Brightness/Contrast*" use the "*Auto*" button as many times as you need, until you can see clearly also weaker diffraction circles or spots. Close this window.
- Select "*Image→Properties*", change "*Unit of length*" to mm and "*Pixel width*" and "*Pixel height*" to 0.2 for the Perkin-Elmer detector (200 µm/pixel) and press "*Ok*". Now on the top of the image window you should read "409.60x409.60 mm (2048x2048); 32-bit; 16MB".
- Next we have to rotate the image 90° to bring the rotation axis for sample tilt into the origin to conform to the angle convention in MAUD (Figure 5). Select "*Image→Transform→Rotate 90 Degrees Anticlockwise*". When using later the calibration done with this standard we have to apply the same rotation to all sample images.
- Now proceed to the "*Plugins→Maud plugins→Multi spectra from normal flat reflection image*". The diffraction image appears as illustrated in Figure 5 (left) and next to it a "*Choose the integration lines*" window shows a list of parameters (Figure 5, right). For this sample,
  - Sample-Detector distance is 1850 mm.
  - Adjust the tracker radius to 2θ = 5 degrees and update the plot (click "*Apply*" not "*OK*" to view changes). Update it to 7 degrees and click again. Update it to 10 degrees and click again. The tracker is indicating where MAUD thinks where a Debye-Scherrer cone with the selected 2θ should be on the detector. It is just a visual indication.
  - If necessary adjust "*Center X (mm)*" and "*Center Y (mm)*" to bring the tracker circle (red) into the center (the solution is X = 204.85 and Y = 204.79).
  - Set the "*Eta step*" value to to 5°. The image will then be integrated in 72 sectors of 5° each, with the azimuth η from 0 to 360° (or -90° to +270° in some cases). The start of the integration is

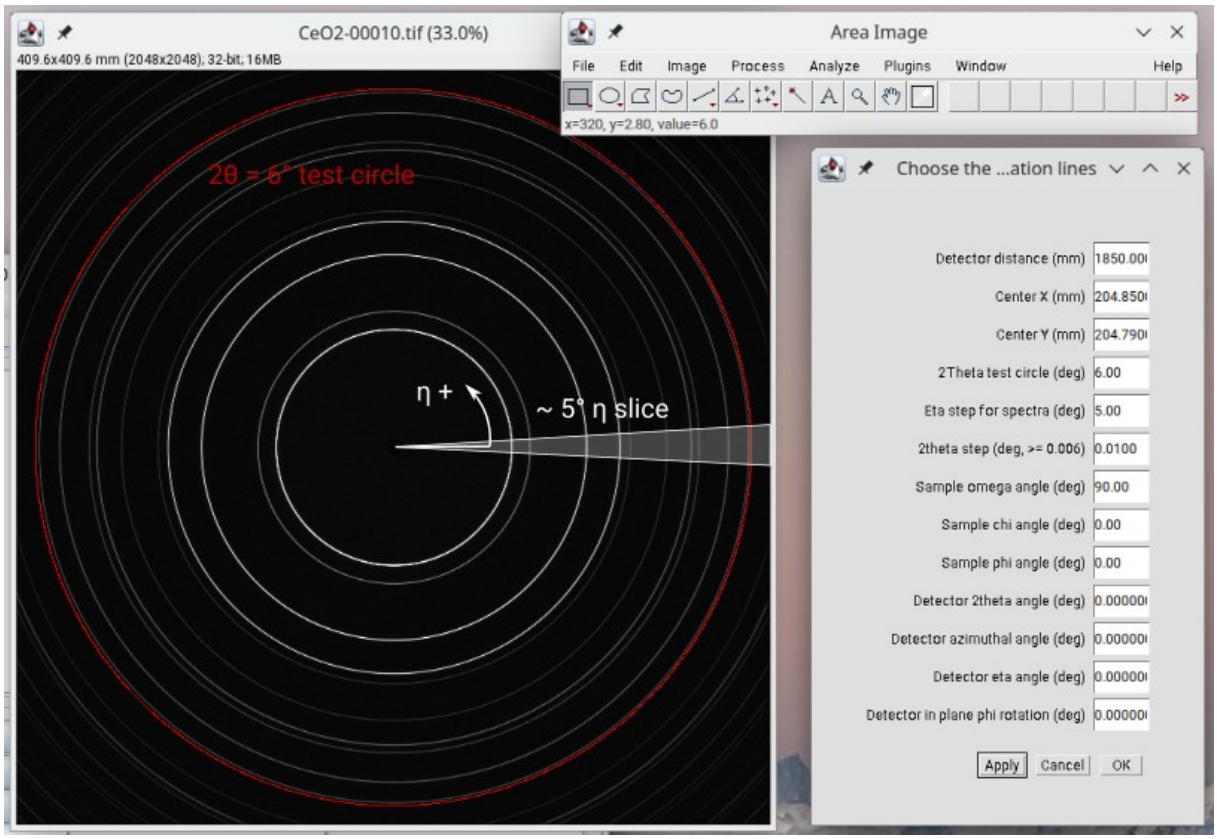

Figure 5. ImageJ setup for image processing to generate diffraction patterns by azimuthal integration of the 2D image. The start of the azimuth η (diffraction pattern #0) is indicated. The red circle is used for aligning the center of the image. The image will be integrated into azimuthal slides. Here, 72 slices of 5°, from 0 to 360° (or -90° to +270° in some cases).

indicated on Figure 5 (left) and the sense is counterclockwise.
    - Set the "2theta step" to 0.01° (otherwise, you will not have enough resolution in 2θ)
    - Set *Sample omega* to 90° to bring the normal to the sample into the center of the pole figure (see later and Figure 5).
    - Have the other parameters set to 0.
- Hit "*OK*" and the integration should start right away. After the integration is done, the program will ask to save the files as ASCII data. Choose the directory in which you have your data and want to perform the analysis (better not the MAUD directory). Here we give it a name *CeO2-00010* (it is good practice to use the same name as the image) and the program will automatically assign "*.esg*" as file extension.
- Close the diffraction image and ImageJ windows and return to the MAUD dataset editing window, where the *.esg* files are now listed in the "*Spectra list*" panel. If you close the dataset window, a summation of all patterns should be visible in the plot panel of the main MAUD window (Figure 2).

Now is a good time to save. Use "*File → Save analysis as*" and save it to "*CeO2_calibration_01.par*". Do not just "*Save analysis*" because this would overwrite the default MAUD parameter file in a random location of your computer. Your parameter file contains all information about instrument, phase and sample parameters and can be used as a starting analysis file for other images.

3. Refinement range and background

There are no diffraction peaks at $2\theta < 1.6°$, so we restrict the refinement range in the "*General*" tab of the dataset editing window (Fig. 2, insert) by changing "*Min in data units*" and "*Max in data units*" values. Units are those of the actual diffraction data (in this case 2θ in degrees). Enter 1.6 as Min and 8 as Max.

Be sure to enter both the Min and the Max values. Otherwise, you might get a black screen.

Choose the background model (switch to "*Background function*" tab). By default a polynomial background is used in MAUD, and we use 5 parameters to approximate background by a 4th order polynomial (click "*Add parameter*" or "*Remove parameter*" buttons to change the number of parameters to 5).

With this the initial setup of the diffraction instrument and the input of diffraction data is completed

Save your analysis to "*CeO2_calibration_02.par*".

4. Phase and initial parameters adjustment

Crystal structure

The "*Phases*" tab in main MAUD window contains crystallographic and microstructural information on all the phases in the sample such as lattice parameters, space group symmetry, atomic positions, grain size, texture, etc. By default it is empty. The basic parameters can be entered as "crystallographic information files" (*.cif*) which can be downloaded, e.g., from databases like the ICSD (ICDD) or the COD (http://www.crystallography.net, Gražulis *et al*., 2009) or AMCSD (http://rruff.geo.arizona.edu/AMS/amcsd.php, Downs and Hall-Wallace, 2003).

To import a structure from a .cif file click the "*Cylinder with arrow out*" icon on the toolbar or select "*Edit→Load object from CIF…*" menu.

A structure model can also be built manually. Then, navigate through the tabs to define your crystal structure parameters (space group, cell parameters, atomic positions, etc). The procedure is described in the box but not required here.

Here we import the provided cerium oxide structure file *CeO2.cif*. After it is loaded, edit the CeO_2 phase. In its properties window switch to "*Microstructure*" tab, and click "*Options*" button next to "*Size-Strain model: isotropic*". There set the crystallite size and microstrain values to 5000 (Å) and 1.0E-5 (in means very little peak broadening from the phase, which has been fabricated to do so).

Calculation and visualization

After having entered all this information, pressing the "*Calculator*" button (or from menu

"*Analysis→Compute spectra*") in the main page (Figure 2) computes diffraction patterns based on user-provided instrumental and phase parameters and compares them with experimental data.

The results of the calculations can be viewed

- With the "*Plot*" tab above the pattern in MAUD main window which is an average over all patterns from a dataset, or,
- With the "*Plot 2D*" tab in which a stack of all individual 72 patterns is displayed (experimental patterns are at bottom and model patterns are on top).

For diffraction data a square root scale for intensity is quite useful, as smaller peaks are more visible. This can be set or changed in "*Graphic→Plot options*" menu.

Save your analysis to "*CeO2_calibration_03.par*".

Manual parameters adjustment

Next we want to adjust the scale factor (beam intensity in MAUD) as the calculated intensity data are different from experimental values. This can be done "live" by using the parameter list at the bottom of the main MAUD window (Figure 2). Enlarge the tree-table and scroll until you see the parameter "*_pd_proc_intensity_incident*" under instrument in the dataset. Make sure the column "*Value*" of the table is sufficiently large by dragging its border if necessary. Click on the value of the parameter you want to change (one click). Two arrows and a field to the right of the parameter appear. The text field contains the increment by which you can change the parameter by clicking on corresponding arrows. The updated plot is displayed right away. After finishing adjustments click the mouse anywhere in the tree-table (except a value) to exit from the "*editing*" mode.

Build a structural model by hand (not required here)

- While on "*Phases*" tab click the icon with the picture of a box in the toolbar (or select "*Edit→Add new object*" menu).
- In the "*Phases*" list "*Phase_x*" appears. It can be renamed by double clicking on it.
- Select the desired phase and "*Edit*" it.
- In "*General*" tab enter space group, and lattice parameters (for monoclinic structures first setting has to be used and for rhombohedral lattices, hexagonal setting needs to be used to comply with texture analysis conventions).
- In "*Structure*" tab add atomic sites to create the structure. Once a site is added, the name can be changed by double clicking the entry in the list.
- For each site, the atom type can be changed by clicking "*Atom type*" and selecting the appropriate atom from the Periodic table that will appear. It is possible to select the oxidation state (meaningful for x-ray scattering only) or isotope (for neutron scattering only).
- Adjust atom coordinates, site occupancies, ionization state and thermal factors if needed. Sometimes thermal factors (especially anisotropic) are loaded incorrectly from the .cif file. Check them after you import a structure.
- The created structure can be viewed in 3D by clicking the "Pointer finger" icon in the upper right corner of the structure tab window.

Then adjust the first background parameter "*_riet_par_background_pol0*" in the same way.

Save your analysis to "*CeO2_calibration_04.par*".

5. Numerical optimization: 'refinement' procedure

There are various ways to refine the parameters:

- For a manual refinement the user goes through all ***relevant*** parameters listed in the tree-table in the bottom part of the MAUD main window and changes their status from "*Fixed*" to "*Refined*" (or "*Equal to…*" to set a linear dependence between different parameters). This is quite cumbersome and only applicable for experienced users. Some parameters are correlated, and some have very low impact on the calculated diffraction pattern unless other parameters values are correct, and the refinement has to proceed in a systematic order.
- The parameters can also be viewed by editing objects under "*Datasets*", "*Phases*" and "*Sample*" tabs. This is more intuitive because there the parameters are illustrated within their context, and with

a right-click on the parameter you can also set it to "*Refined*" or "*Fixed*". Once you have set all the parameters you want to refine, click the "*Hammer*" (sometimes transforms to "*Cash register*") icon in the toolbar of the main page (or select "*Analysis→Refine*" menu).

- Semi-automatic refinement is possible if the user selects the "*Analysis→Parameters list*" menu, which again displays the parameters list (click "*Expand all*" button below to see the entire tree-table of parameters expanded) but at the bottom there are "*Commands*" that allows user to change certain groups of parameters. A good start is to first "*Fix all parameters*" and then proceed with, *e.g.*, "*Free scale pars*" (general intensities and phase volume fractions are set to "*Refined*"), "*Free backgrounds*" (all parameters for backgrounds are set to "*Refined*"), etc. in cycles.
- Automatic refinement is yet another way. For this option click the "*Light bulb*" icon in the toolbar (or go to "*Analysis→Wizard*" menu) to open "*Refinement wizard*" and proceed with refinement from top to bottom of the list on the left side, or a complete analysis on the right side. ***But there is no wizard option for instrument calibration as it can not be standardized. Calibration should never be done with the Wizard (you are guaranteed to be wrong). Even later, you should know what you do and exercise caution with the Wizard options.***

The refinement can be lengthy, depending on the complexities (number of patterns, number of diffraction peaks per pattern, utilization of complex texture/microstructure models, etc.). Do not change anything during the optimization of a refinement! If you are reviewing some parameters in the course of the refinement, it is important to close all frames using the window close button (on all systems it is the button on the window title bar) and not with the "OK" button. The latter changes or refreshes the parameters in midst of the refinement and may cause the optimization to behave erratically.

6. Refine scale factors and background

We proceed with the mixture of semi-automatic and manual refinements:

- Start with "*Analysis→Parameters list*" and first click "*Fix all parameters*", then "*Free Backgrounds*" and "*Free scale pars*". You may leave parameters list window open to return to it later.
- In the MAUD main window click the "*Hammer*" button in the toolbar and the least squares refinement will start. Choose the number of cycles with the slider appearing at the left side of the window (usually 3-5 is sufficient).
- If the refinement goes well, the calculated and experimental patterns on the display should become more similar (dots are experiment, line is model).

Save your analysis to "*CeO2_calibration_05.par*".

7. Refine instrument parameters

Detector location

Next we refine instrument parameters.

- Edit "*Dataset→Diffraction Instrument*".
- Under "*Options*" for "*Inclined reflection image*" right click consecutively on values in red fields for the "*Detector Distance*", "*Center x error*", "*Center y error*", "*Detector 2theta*", "*Detector tilt*" and select "*Refined*" in each appearing popup menu. Close the windows and run the refinement again. This will adjust the reflection positions.
- Do not adjust "*Detector rotation*" or "*Detector eta*". They are fixed to zero here.

Save your analysis to "*CeO2_calibration_06.par*".

Peak broadening due to the instrument

Then it is necessary to refine the instrument broadening parameters in the "*Caglioti PV*" model. Caglioti parameters describe instrument broadening, especially peak asymmetry, peak width and Gaussian plus Lorentzian mixing parameter (Caglioti *et al*., 1958). These instrument peak shape parameters are essential if

you want to assess peak shape effects of real samples, such as crystallite size and microstrain, or estimate dislocation density and stacking fault probability.

Caglioti parameters are very sensitive and a strict order needs to be followed. We describe the procedure for synchrotron image data where the 2θ range is limited. With the initial parameters set as suggested in step **1,** there should be no need for manual adjustments.

If you feel (or discover during the refinement) that it is still necessary, then start adjusting the first HWHM parameter – "*_riet_par_caglioti_value0*" – manually by using the parameter tree list in the bottom of the main window with the "live" procedure (step 5**)**. After that you may adjust the second and if necessary (for large 2θ range) the third HWHM to get an approximate fit. You can view an enlarged peak by selecting an area in the plot by mouse click and dragging. Afterwards you may return to full pattern view by double clicking in the plot or right-clicking and choosing "*Reset scale*" from popup menu. If you need to adjust the asymmetry, then for synchrotron diffraction data we advise to start with only one parameter ("*_riet_par_asymmetry_value0*") and enter a large value (> 5000) for it if the asymmetry is small (which is usually the case for synchrotron data).

After this manual adjustment, you can proceed in the following order (3 iterations are sufficient in each of these steps; do not fix the parameters already set refined in previous steps or from beginning and be sure not to refine things like wavelength or parameters of the CeO_2 phase):

- "*_riet_par_caglioti_value0*", set refined and refine.
- "*_riet_par_caglioti_value1*", "*_riet_par_gaussian_value0*", "*_riet_par_gaussian_value1*" set refined and refine.
- You may add "*_riet_par_caglioti_value2*" and "*_riet_par_gaussian_value2*" for a final improvement.

Save your analysis to "*CeO2_calibration_07.par*".

8. Final step: B-factors

If you look at the plot window you can see that peak intensities sometimes remain poorly fit. You can refine an overall thermal factor

- "*Edit*" the phase,
- Switch to "*Structure*" tab,
- Right-click on the value of "*B-factor*" for each atom position.

At this point, if you used the dataset of the tutorial, you should be ready with a decent calibration (Fig. 6).

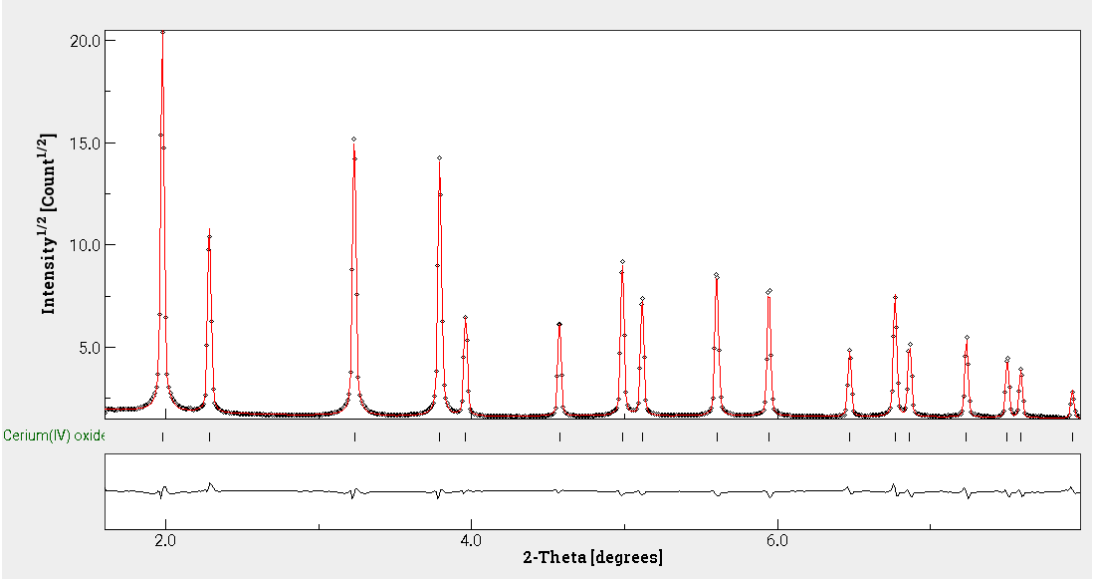

Figure 6. Plot view of the CeO_2 pattern after the end of the calibration procedure. Data (in black) and model (in red) should look identical. Similar match must be obtained in the Plot2D view.

9. Saving the calibration results

In the end, the calculated peaks should fit the experimental peaks very well, both in the "*Plot*" and "*Plot2D*" displays (Figure 2). Be sure to save the parameter file at end:

- Open with "*Analysis→Parameters list*" and select "*Fix all parameters*". This will avoid messing up the calibration if you re-open the file.
- Save your analysis as "*CeO2_calibration_final.par*".

You can also decide to export the instrument for use in the refinement of other samples, "*Edit*" the current datasets and under "*General*" tab click "*Store…*" button in the "*Instrument*" panel to save the instrument settings as a separate file in your directory (e.g. giving it a name such as *APS-BESSRC-11-ID-C-InclinedReflection.ins*).

We have now refined the relevant instrument parameters with the CeO_2 standard and can apply them to other samples measured under identical conditions.

10. Exporting the figure

As for the 2D plot (with colors) the best option is to select "*Graphics → Mapplot of the selected dataset*". Adjust the color range, X-axis scale (2θ, d-spacing, or Q), Y-axis scale (linear, sqrt, log, etc) to your liking. Take a screenshot. Load the screenshot in a smart software such *Inkscape* to fix axes labels, legends, etc.

If you want to switch to grayscale in 2Dplot from color, this is done by selection "*Analysis → Preferences*", search for "*multiplot2D.grayscale*" and set it to "*true*".

As for the standard plot (i.e. Fig. 6), you can either

- Take a screenshot of the MAUD plot (which is what most people do).
- Export the data and fit for replotting in your favorite software.

To export the data and fit

- Open the *Datafiles* tab of your dataset
- Select "*View summed*" among the buttons at the bottom of the window
- Select "*Tools → Export from plot*" for a simple export of the data and fit in the plot
- Select "*Tools → Export experimental / computed data*" for a more thorough (and useful) text export with the experimental data, the computed data, as well as values of background and diffraction intensities for each phase.

ADDITIONAL NOTE: TEXTURE AND LE BAIL ANALYSIS

Sometimes, the diffraction of your calibration standard will show texture or grains. In such case, an additional step is necessary, either using a true texture model, or a Le Bail fit.

1- Fit of a true texture model

Texture is fit the "*Advanced models*" tab for the phase of interest (here CeO_2). We will not discuss the full tricks of a texture analysis in MAUD, which is beyond the scope of this document, but here is a quick tutorial on how to proceed:

- Open the "*Advanced models*" tab for the phase of interest (here CeO_2).
- Select "*E-WIMV*" from the drop-down list and press the "*Options*" button next to it.
- In "*E-WIMV options panel*" select 10° for "*ODF resolution in degrees*" (which would require a whole discussion).
- Open the " *EWIMV advanced options*" and make sure "*Normalize pole figures*" is unchecked.
- Click "*OK*" and close the all windows.
- Run a refinement. It should fit the texture and improve the correspondance between the simulated and experimental data, particularly in the Plot2D view.

2- Le Bail analysis

If your sample has many grains, you may decide to go for a Le Bail fit, for which peak intensities are matched to fit the data, whatever it means. This will lead to nice looking results but pay attention that when Le Bail is activated

- peak intensities have no meaning.
- Beam intensity should be fixed.
- B-factors will be off.
- Phase proportions can not be studied, since peak intensities have no meaning.

To do so, "*Edit*" CeO_2, switch to "*Advanced models*" tab and select "*Arbitrary tex*" as texture model. Next fix all the scale factors or intensity-related parameters as they are not needed with the Le Bail fitting (set fixed the "*_pd_processing_intensity_incident*" and the "*_atom_site_B_iso_or_equiv*" for the Ce1 atom site). Perform another refinement cycle.

This should drastically improve the visual match between model and data.

Sometimes after the refinement a message "*Cholesky negative diagonal*" appears in the MAUD output panel. This means the problem that MAUD was trying to solve was ill-conditioned, and some of the parameters are not refinable at the given conditions. The first of the three displayed numbers refers to the "wrong" parameter. You can view it on the MAUD output file (with .lst extension) or you may review the parameters list window and find a refined parameter with value "-1" in the "*Error*" column. After determining the parameter you should either adjust it manually or refrain from refining it.

PART 2. STEP-BY-STEP PROCEDURE FOR TEXTURE ANALYSIS OF NICKEL COIN

1. Introduction

This is a more advanced tutorial, for those who wish to learn how to deal with data involving multiple detectors, or data collected on the same detector, with multiple sample orientations.

This tutorial will fit texture of a Nickel coin, based on 5 diffraction images collected with 5 different sample orientations. Please see discussion in section IV of the corresponding paper for details:

> L. Lutterotti, R. Vasin & H.-R. Wenk. Rietveld texture analysis from synchrotron diffraction images. I. Calibration and basic analysis (2014) *Powder Diffr.* **29** 76-84 doi: 10.1017/S0885715613001346

The dataset consists of 5 tiff images, collected while rotation the sample ω at -40°, -20°, 0°, 20°, 40°. We will create 5 datasets, one for each image, and refine the sample texture using the 5 images simulatenously.

2. Datasets

Run MAUD, open your calibration file and save it under a new name, such as "*NCoin_01.par*".

Double-click on the dataset and rename it to "*Coin-40*".

In the main MAUD window, duplicate the dataset using "*Edit→Duplicate object*" menu item. This will create a copy of the selected dataset including instrument parameters. Repeat this until you have 5 datasets (one for each diffraction image). Then rename the datasets (by double-clicking on its name) to, *e.g.*, "*Coin-40*", "*Coin-20*", "*Coin-0*", "*Coin+20*", and "*Coin+40*".

Select the first dataset named "*Coin-40*". Remove any data that may be left in the "*Datafiles*" tab, and repeat the step to integrate and load the corresponding diffraction images of the coin sample: "*Nickel-00178-40.tif*". Use the same values for the image size, image rotation (90 degrees counterclockwise,), center X, center Y etc. that you have used for CeO_2. Do not change angles such as omega at this point.

After the diffraction image integration is completed and patterns are loaded into MAUD, under "*Datafiles*" tab select all patterns, click "*Modify Angles*" button and change the ω angle (*"New omega =" -40 + omega*) to account for the sample tilt. For the coin it is appropriate to set the refinement range to 2.5-6.2°, as there are no diffraction peaks at lower 2θ values. In "*Background function*" use 5 polynomial parameters.

MAUD tends to loose instrument parameters in recent version (it's a bug). Unless your instrument is ok, re-import your instrument you saved at the end of part 1 (*APS-BESSRC-11-ID-C-InclinedReflection.ins* in the tutorial above). This is done in the "*General*" tab of the "*Dataset*", in the "*Instrument*" subwindow.

Repeat these operations for each of the 5 datasets, importing corresponding diffraction images and adjusting ω angles accordingly. Note that if you have several datasets then the diffraction patterns shown in all displays in the main MAUD window correspond to the currently selected dataset. To see the patterns from any other dataset you must select it in "*Datasets*".

If you did well, you should have a dataset called "*Coin-40*" for which all data files are with ω = 50°, "*Coin-20*" with ω = 70°, "*Coin-0*" with ω = 0°, "*Coin+20*" with ω = 110°, and "*Coin+40*" with ω = 130°.

Save your analysis "*NCoin_02.par*".

3. Phase and first refinement

Go to "*Phases*" tab of the MAUD main window. Remove any phase that is there and create a new object. Double-click on it and rename it to "*Copper*".

Open the eye icon, and update the crystal parameter:

- Copper is cubic, Fm-3m space group, with a unit cell parameter of 3.6149 Å.
- In the "*Structure*" tab, add one atom position at (0,0,0) and set the atom type to Cu. Be careful, MAUD will set the atom type to Ca by default.

You can now compute the spectrum. Copper peaks should fall at the right 2θ. If not, you did something

wrong.

If fact, the alloy in the Nickel coin is not pure nickel, nor copper, but an alloy. Edit the copper phase, go to "*Structure*" tab, select the atom site at (0,0,0). In the "*Atom type in this site*" sub-panel, add a second atom of Ni. Adjust "Partial occupancy" of Cu to 0.75 and of Ni to 0.25.

You can now compute the spectrum again.

You are ready to start with magic. Open the "*Analysis → Wizard*"

- Run "*Background and scale parameters*", which will fit backgrounds and intensities for each dataset.
- The peaks should be slightly off. Let's play with crystal parameters. Run the wizard with "*Previous plus basic phase parameters*", which will optimize the unit cell parameters and B-factors.
- Let's improve peak shapes, by playing of grain sizes and microstrains. Run the wizard with "*Previous plus microstructure parameters*", which will optimize the unit cell parameters and B-factors.

You should now be getting closer to a solution. Save your analysis "*NCoin_03.par*".

4. Sample orientation and texture

In "*Advanced models*" tab select the texture model. Select "E-WIMV" from the drop-down list and press the "Options" button next to it. In "*E-WIMV options panel*" select 10° for "*ODF resolution in degrees*" (see the discussion in section IV of the paper). Click "*OK*" in the "*E-WIMV options panel*" to close the window.

To check the texture coverage, first compute the model diffraction patterns (use "A*nalysis→Compute spectra*", or "*Calculator*" icon), then go to the menu "*Graphic→Texture plot*"; select the hkl reflection for which you want to plot the texture coverage, in the "*Pole figure options*" panel select "*Pole figure coverage*" and press the "*Plot*" button. We set the pole figure projection plane parallel to the Nickel coin's face (as in Fig. 2 of the paper) and the corresponding coverage should be similar to the one displayed in Figure 4c in the paper.

If not, you may have messed up somewhere, which can be fixed in the MAUD main window. Go to "*Sample*" tab and "*edit*" current sample. Under "*Sample position*" change the sample orientation angle ω to 90° degrees (for instance). This will change the orientation of the sample with respect to conventional MAUD reference coordinate system and may help. Try a few values. Try to understand figure 4 in the main paper.

With the current version of the tutorial, ω values should be already set in the dataset. ω in the sample settings should be fine at 0°.

Save your analysis "*NCoin_04.par*".

5. Refine coin data, with texture

Go back to the wizard and run "*All parameters for texture*" and then "*Crystal + texture parameters*". The correspondence between data and model should improve dramatically. You may run those a few times to see if the fit improves.

Save your analysis "*NCoin_05.par*".

6. Checking the results

After completion of the refinement, a first step is to check all datasets with the "*Plot 2D*" display to see if the intensity fit looks reasonable (Figure 6 in the paper). You can also do this in a separate window by selecting "*Graphic→MapPlot of selected dataset*" menu.

It may be also important to see how individual patterns are fitted. This is done by "*Editing*" a dataset of interest. Under "*Datafiles*" tab select one or several diffraction patterns and press "*View*" button. Sometimes just one or two patterns are fitted poorly, with obviously wrong peak positions, widths or intensities. You may remove such "wrong" patterns by unchecking their "*Enabled*" property in the same window. Repeat the refinement without them.

Check the texture pattern by selecting "*Graphic→Texture plot*" menu. A new window will appear where it is possible to plot selected pole figures *hkl*, inverse pole figures, strain distributions, etc. on equal area projection.

Select the pole figures to plot (check "*Active*" next to a set of Miller indices), ensure that "*Reconstructed intensity*" (i.e., pole figures calculated from ODF) and "*2D map*" options are selected. Then press "*Plot*" button. In a sub-window you have the option to change maximum and minimum values for the pole figure plot. You can plot pole figures in color or "Gray shaded". You may adjust the number of contours to draw with a slider in a bottom right part of the window, or smooth with a Gaussian filter (change "Gauss smooth width").

Explore the texture features of MAUD. If you were good a processing, you may compare your results with Figure 7 in the companion paper.

You are now a MAUD expert. Improving the fit will involve fine-tweaking, testing new options, and understanding the physics behind them. Good luck! With the settings of this version of the tutorial, I find that one gets best texture results, in line with the supporting paper, with a better-resolved ODF, with an ODF resolution of 6°.

7. Exporting texture for external programs

Even though MAUD offers several options for plotting ODF and pole figures, it is advised to use specialized software for further texture processing. In the phase editing window under "*Advanced models→E-WIMV→Options*" you can export the ODF ("*Export ODF formatted for Beartex*"), or pole figures ("*Export PFs*") as ASCII files for the BEARTEX texture package.

The exported ODF is always interpolated into 5°x5°x5° cells and can be converted by BEARTEX into a binary format for further processing, including smoothing, rotations, representation and physical properties calculations.

You may also want to play with MTEX, which will know how to read BEARTEX files.