Operating Manual for ProcessDiffraction Version 8.7.0

J.L. Lábár

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Starting Essentials

ProcessDiffraction is a computer program, written for PC compatible machines under the *Windows* operating system to extract quantitative information from electron diffraction patterns, recorded from thin specimens in a transmission electron microscope (TEM).

Most of the features of the program deal with selected area electron diffraction (SAED) or incoherent nanobeam diffraction (NBD). Fourier transforms (FT) of lattice resolution TEM images (HRTEM) can also be processed with the program, similarly to SAED. Orientation information for a crystalline grain can also be obtained with the program from the Kikuchi-bands in convergent beam electron diffraction patterns (CBED).

This operating manual presents a few examples for frequent usage. A few sample data files (referenced in this Manual) are also included in the ***Examples*** ZIP file in the same home page from where you download the program. You can download and install them anywhere on your computer. To access it, its path must be specified to ProcessDiffraction as explained below in section Start of the program. More details are described in the published papers that you can also download separately from the same home page as a separate ZIP file named ***For_Reading_&_Referencing***.

Disclaimer

The ProcessDiffraction program is not a commercial product. It is produced out of enthusiasm of the author. Although a thorough testing is attempted, the author does not take any responsibility if any programming bug remained in it.

Operating system / computer / display

The program has been developed and most extensively tested under the Windows XP operating system. It should also run under other Windows systems, but possible differences have not been systematically tested. (The author is aware of successful users under Windows 7, too.)

Memory requirements depend on the size of image files you intend to use. An average PC with 1 Gb RAM should be adequate.

The screen appearance of the program is optimized for a 1400*1050 resolution, however it also works with other display sizes, only some scrolling operation might be needed. Occasionally, resize operation of a window by the Mouse might also be required.

Notation

Names of menu items are shown in this manual in ***blue bold italics*** to help locate them in the text. Each window within the program is identified with a name at its Title bar. That name is referred to in the text as ***red bold italics***. Hyper-references are shown with blue dashed underline. Jump to this reference is by <Ctrl>Click. File names are shown in **bold**. Names of control objects are shown in *italics*.

File formats to input your measured diffraction patterns

Four file formats are supported in the present implementation.

1. Uncompressed BMP (8 bit integer format). Less recommended.
2. Uncompressed TIF (8 bit integer format)
3. Raw format (integer numbers per pixel, with zero length header).
4. Floating point raw format (4-byte or 8-byte real format with zero header length as saved by Digital Micrograph with ".dat" extension as an example). This format is not supported by Adobe Photoshop.

5. Proprietary format used by the DITABIS imaging plates (IPs) with “*.IPL”, “*.IPH” (both 16 bit formats) and “*.IPC” extensions (24 bit format where the useful data extend to 20 bits).

Operation of Child-windows and menus

Different tasks appear in different windows. These are so-called “child-windows” of the main window, what means that they appear within the main window and when they are active the menus of the main window will change depending on which child window is active. When the main window is minimized or closed, all child windows go with it automatically.

Child windows that do not have the controls  at their top right corner are not supposed to be minimized or closed separately. Closing them would close the entire program.

Glossary

- ***pattern***: the 2D image, containing a diffraction pattern
- ***distribution***: the 1D distribution of circularly (elliptically) averaged intensity [determined from the 2D pattern] presented as a function of the length of the scattering vector (or alternatively as of the scattering angle of simply as of the non-calibrated channel number).
- ***Marker***: a set of diffraction lines layed over the measured pattern to visualize their positions and intensities (and also used in quantitative phase analysis to compare the measured intensities to).

Basic Operations

Start of the program / Specifying File locations

After clicking OK for the Start-up screen called ***About ProcessDiffraction***, you are left with an empty image window (the ***SAED*** window) with active menu items. At the very first start-up the program offers to create a default data structure for you (see below).

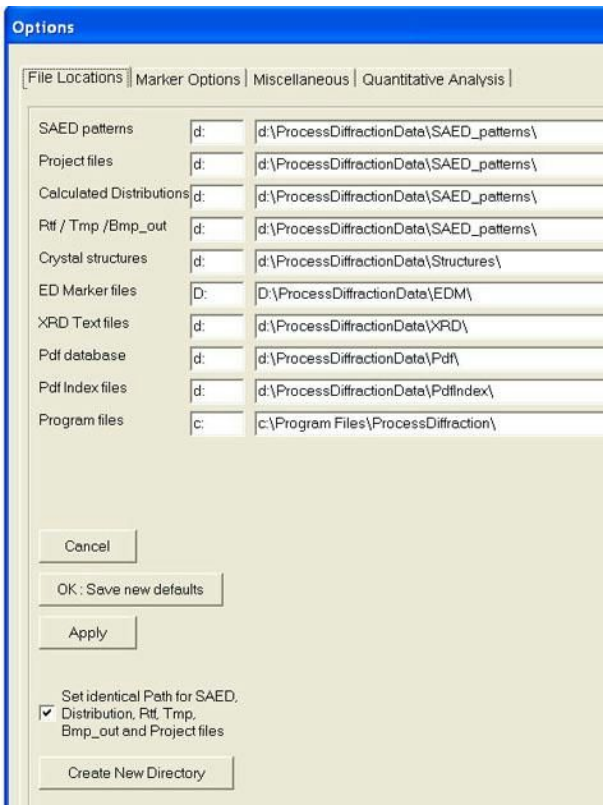
The very first operation should be that you specify to the program where your data files are located and where you want to store your results and intermediate (or temporary) files. This is done by selecting menu ***Options***. The appearing new window should look like the one below. (Make sure the "ear" *File locations* is selected.)

Any valid drive and directory can be selected for any one of the listed items. Validity of drives and directories is tested before you can leave ***Options***. It is also tested when you start up the program. The ***Options*** window pops-up if any of the (previously) specified items is invalid (it might also be the default name if the default data directory structure has not been created and no other directory name was specified by you or a directory was renamed).

A convenient (but not compulsory) way is to let the program generate a default data directory structure for you. If you did not do so during the first start (when creation of the data structure was offered) you can do so any time later by selecting ***SAED*** window, menu item ***File / Create Default Data Structure***. Remember that a newly created data directory structure is empty. To access your measured diffraction patterns you should either move them to the default directory (first item, "SAED patterns" in the list on the right) or preferably you modify that directory in "File locations" by clicking on the text-box next to the name of the item.

Observe the usage of the ***Check-box*** at the bottom of the window (many people keep the data listed in the first four lines in the same directory).

Figure Options / File Locations



The "***Project***" contains all processed data. By saving the Project, you can resume operation at any later time after interruption. Its location is in the 2nd line.

Legal users of the ***Pdf-2 XRD database*** can access data of the database directly from within ProcessDiffraction (details are explained separately). Separate locations are intentionally specified for the "Pdf database" and "Pdf Index files", because the index files are generated by ProcessDiffraction and need a writable directory, while the database itself frequently comes in a read-only CD. You may not have a copy of the Pdf-database. Even if you do not have it, valid directories should be specified both in the line "Pdf database" and "Pdf Index files".

If you do not have the Pdf-2 database, you can still supply XRD data to the program ***manually*** (from a printout, you received from someone). Example format is given in the Examples directory in file "Cu_fcc.xrd". The location of such manual files is specified in line "XRD text files".

The directory of the "***Program files***" (where you installed ProcessDiffraction) also contains other files needed for the operation of ProcessDiffraction. Do not divert it from its true value!

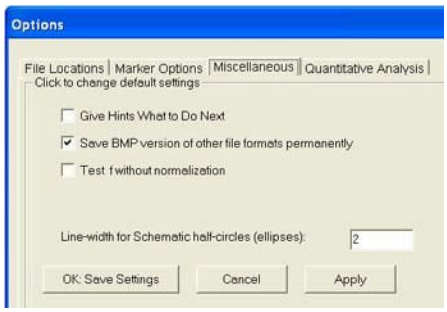
Ring pattern: qualitative phase analysis

Specifying essential information for the loaded pattern

ProcessDiffraction displays your diffraction pattern on screen and you can manipulate the appearance on screen without altering the original data that are used for data evaluation.

For that reason the image on screen is stored separately in a Bmp file (while your data are stored unaltered in the TIF or IPC,… file). This rendering file can be either a temporary file or a permanently stored file, in accordance with your choice in menu ***Options*** (selecting "ear" *Miscellaneous*"). Here you can select a Check-box *Save BMP version of other file formats permanently*. That BMP version only contains the rendering data. Section Optimized rendering on SAED screen explains how to obtain the best appearance.

Figure Options / Miscellaneous



To practice with your first pattern, set the directory for SAED patterns (in Options) to the location of the Example files! First select Save BMP permanently (to see the difference between the original and the rendering version of the same pattern)! In ***SAED*** window select menu item ***File / Reset Project and Load a New Image***! Select file **Cu-kalibr_883.tif** in the Open window! Let the program save **Cukalibr_883.bmp** as the rendering file (as it is suggested by the program)! From menu ***Window*** select the ***Info*** window!

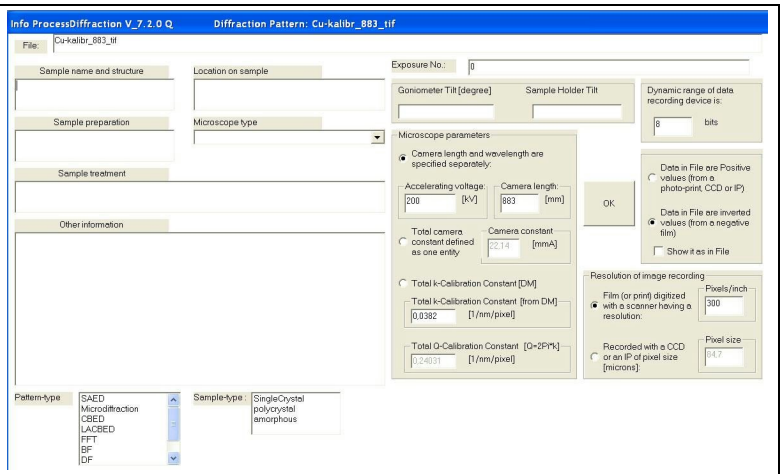
Select Radio-button *Data in file are inverted (negative)*!

Leave Check-box *Show it as in File* unchecked!

Specify 200 kV, L=883 mm (for the camera length) and 300 Pixels/inch! The other selection possibilities are unimportant for the moment.

Click Command-button *OK*! The ***SAED*** window must appear.

Figure Info window

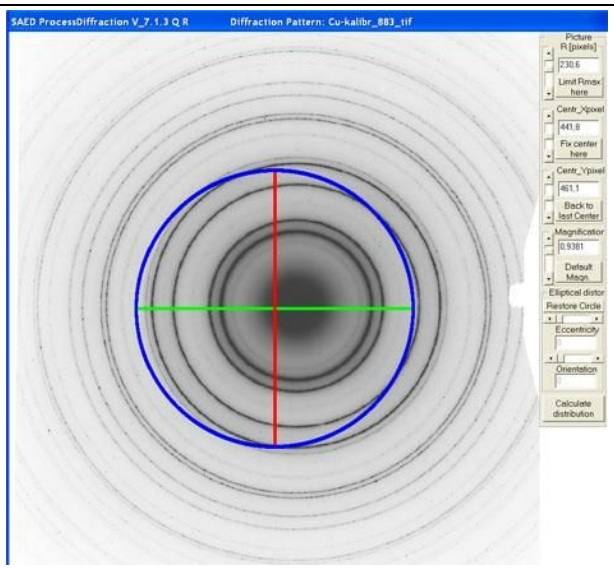


Finding the pattern center

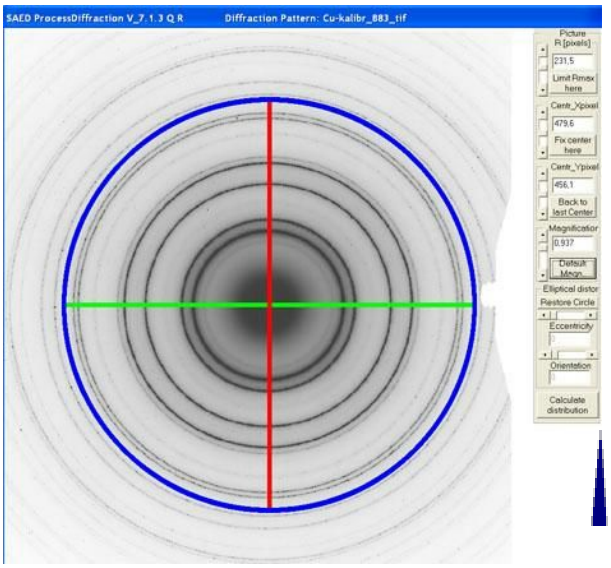
Check if menu item ***Overlay / Reference circle*** is checked! A reference circle with a cross-hair must appear overlaid on your measured pattern[1]. The center of the pattern is found manually by shifting the pattern over the reference circle using the vertical Scroll-bars *Centr_Xpixel*, *Centr_Ypixel* and *Magnification* as seen in the two images below (Figure Pattern centering).

[1] The line-width of the reference circle is set in menu ***Options*** (selecting "ear" *Miscellaneous*) by changing the number in the Text-box "Line width for the schematic half-circles". (Line width=7 is selected in the examples of this manual for better visibility in print.)

When the pattern is centered, a circularly[2] averaged intensity distribution can be calculated from it by clicking the Command-button *Calculate distribution* at the lower right corner (marked by an arrow in Figure Pattern centering). The same function can also be evoked from menu ***Process / Calculate distribution***, as an alternative. Two-dimensional (2D) rings (as in Figure Pattern centering) will transform into peaks in the one-dimensional (1D) intensity distribution (as in Figure Raw Distribution).



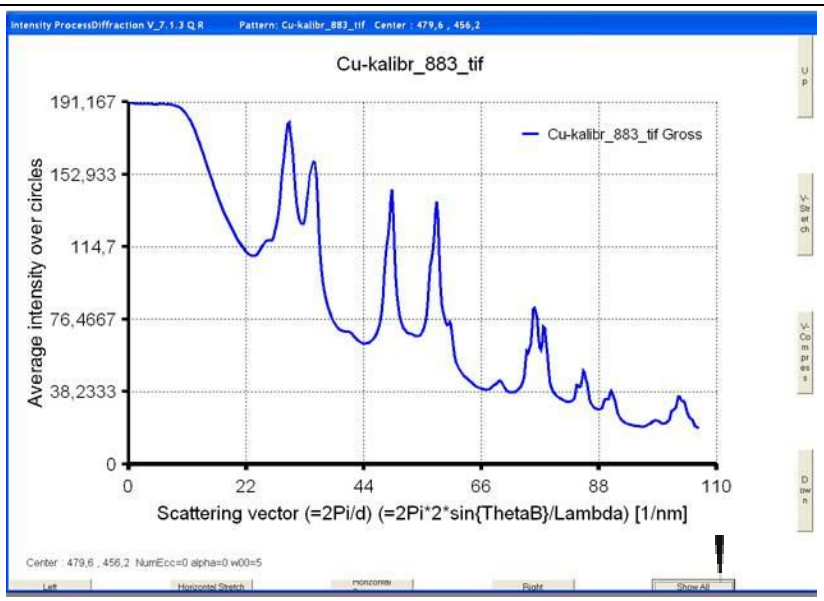
Un-centered rings



Centered rings

Figure Pattern centering

The distribution should appear in a separate window, called ***Intensity*** and look like this:



If the distribution fails to appear, click on Command-button *Show All* at the lower right corner of the ***Intensity*** window (see arrow in Figure Raw Distribution)!

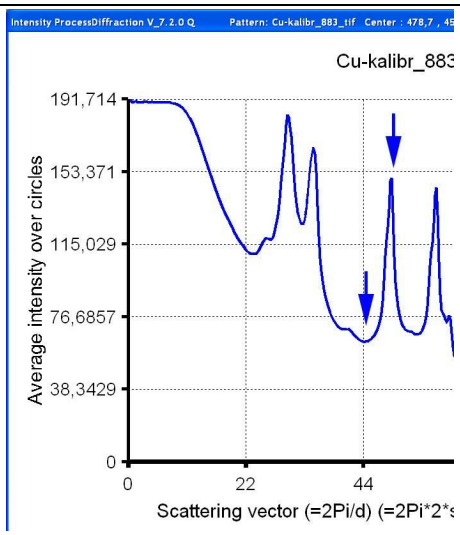
Although the center was aligned manually, there is an automatic refinement of the center routine build in.

However, the image to the left demonstrates that occasionally it can fail.

Figure Raw Distribution

Whenever the program detects that the automatic center refinement failed it asks for your manual help, asking you to show a significant peak and a nearby valley. The same refinement routine can also be activated from menu ***Process / Refine Center***.

[2] Correction for elliptical distortion is treated later.



You should use the Mouse to double-click on the best, well separated peak. Its position should appear in the Text-box *Peak_top*. Next you should double-click on a valley. Its position should appear in the Text-box *Valley_bottom*. (Possible selections are indicated in Figure P/B by arrows). When both are selected, the Command-button *Refine center* is activated and must be clicked.

The distribution is recalculated with the new center and should result narrower and higher peaks.

Figure P/B

IDENTIFYING THE PHASE(S)

The obtained intensity distribution can now be used for qualitative phase identification ("fingerprinting"). A piece of information extracted from the XRD database and pre-stored for you in the Examples is used in the first instance here. Select menu ***Marker / Show more Markers / Old Marker Sources / from Text files (*.XRD)***! If you correctly specified the location of the **Cu_fcc.xrd** file[3] in ***Options***, now its name appears in the Open window and you can select it. You can produce similar files for any XRD-card you take from the literature. (More convenient if you are a legal user of the Pdf-2 database making the manual data entering method obsolete.)

The distribution must be shown with the diffraction lines overlaid. Both positions and relative intensities of the X-ray diffraction lines are shown. Obviously the intensities are only to be treated as a rough indication which lines are strong, since electron diffraction intensities are not exactly the same as the XR ones. (Later we shall see how to work with electron diffraction intensities in by finding which Marker(s) fit your measured peaks you identified which phases are present, so you performed your first qualitative phase analysis.

Generate true electron diffraction Markers,

but this is not needed here.) Automatic labeling of rings is shown in section Automatic labeling of rings.

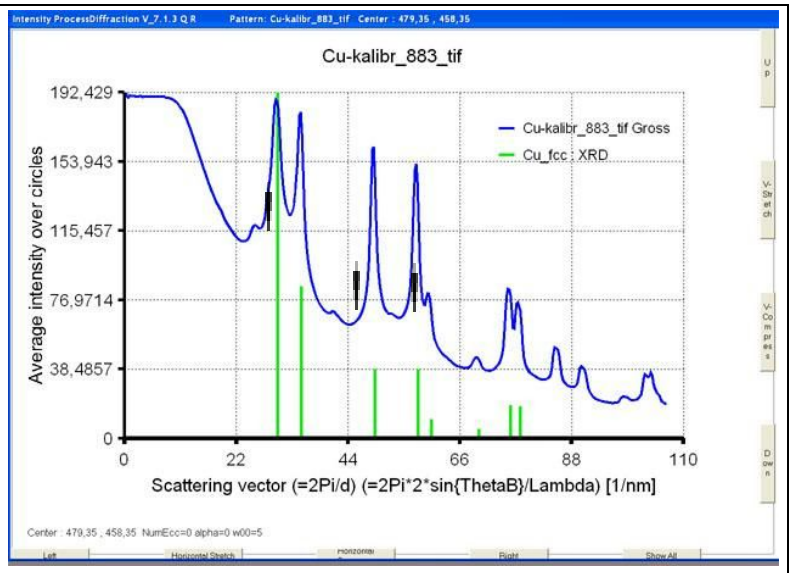


Figure Additional Peaks

You can see that

1. The major lines are from diffraction of fcc Copper.

[3] The ***structure of the file*** is a simple text file with "XRD" extension. The first line contains the name of the program with version number information followed by one diffraction line per text-line in coma-separated format: d-value, XR intensity, and label (Miller index).

2. The XRD database contained a more limited range in the scattering vector (so in scattering angle) than what we measured with electrons here (no Marker lines are above 75 [1/nm]).
3. There are also minor lines (indicated by arrows) that are not explained with Cu diffraction (we shall see later that it is oxide of Cu).
4. A better comparison to the Marker lines can be done if background-corrected net peaks are used.

REMOVAL OF BACKGROUND: NET PEAKS

Select menu ***Process / Calculate Background***! Select *log-Normal* for BKG shape and *Gross* for source! Double-click on 4 Background (BKG) points! Their position will appear in the list and will be marked in the distribution (see the left figure below!). Select menu ***Process / Calculate Net Peaks***! The results should look like the right figure below.

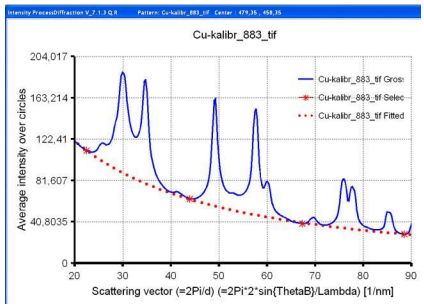


Figure BKG

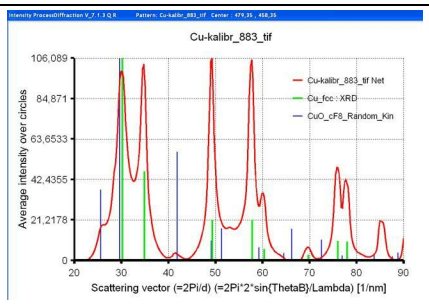


Figure Net Peaks

Deviations of the measured intensities from those indicated by the Markers are caused by both dynamic scattering and texture (and to a lesser extent by the difference between XRD and SAED).

Lines of an oxide phase are also shown (see below).

By finding which Marker(s) fit your measured peaks you identified which phases are present, so you performed your first qualitative phase analysis.

Single crystal patterns

INDEXING A SINGLE CRYSTAL PATTERN

Set the directory for SAED patterns (in ***Options***) to the location of the Example files! Select menu item ***File / Reset Project and Load a New Image***! Select file **Si-112.tif** in the Open window!

Fill in calibration data in the ***Info*** window: 300 pixel/inch, 200 kV, L=883 mm, negative data! Roughly center the pattern and click Command-button *Fix center here* in the ***SAED*** window!

In the ***SAED*** window select menu ***Process / Show Cursor and correlation Start***! You can move the new window named ***Cursor*** not to cover the interesting part of your pattern. Select Check-boxes *Jump to center of gravity of Zoomed-window* and *Refine center with mirror spots* (as in Figure Cursor_single_crystal)!

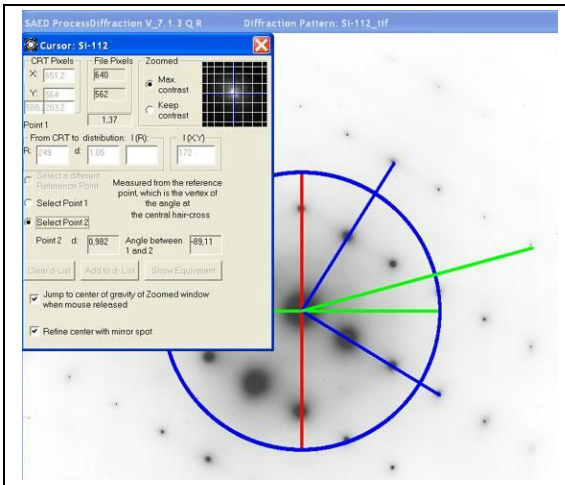


Figure Cursor_single_crystal

When Radio-button *Select Point 1* is active move the *Mouse* to one of the points with the highest d-value, or its multiples! In the figure the third point from the center is selected. A magnified image of the region around the *Mouse* is shown in the upper right corner of window ***Cursor***. Select Radio-button *Select Point 2* and Move the *Mouse* to a linearly independent high d-value point! In the figure the second point from the center is selected. Both the two d-values and the angle between the two reciprocal vectors (shown as blue lines in the figure) are listed and the vector sum is also indicated with a different color.

Select menu ***Copy to / new Crystallographic Calculator***!

In addition to the appearance of a new ***Crystallographic Calculator*** window, a pop-up message warns you not to forget to specify the orders of reflections you selected. Remember! It was 3 for the first point and 2 for the second point in the above example. Specify it in the appropriate text-boxes as seen in Figure_Crystallographic_Calculator_SC! Now your measured data are ready for evaluation.

You should tell the program which structure is expected to explain the measured pattern (i.e. select a Model structure to index the pattern). As the first alternative, you can enter the unit cell data manually. The second alternative that you have a stored structure file and you can load it (menu ***File / Load Structure*** in the ***Crystallographic Calculator*** window). The additional data on the atomic coordinates in the structure file are not needed and are disregarded here.

In the present example data for Si are loaded from the example structure file **Si_cF8.str**.

Figure_Crystallographic_Calculator_SC

Select menu ***Solve / Report results into a document***! The menu item will remain selected until you deselect it or start a new project.

Select menu ***Solve / Solve this spot pattern automatically***! The 48 symmetrically equivalent solutions are listed in the Document. You can save it separately from the Project if you wish.

If the solution is not found, try to increase tolerances *d-tolerance* and *plane angle-tolerance* and solve again! In the present example <112> zone type is the solution.

Automatic labeling of single crystal SAED patterns is shown in Figure_Labelled_sc_pattern.

Indexing a single crystal SAED pattern gives a rough orientation of the crystal. A more accurate orientation can be obtained from the Kikuchi lines in a CBED pattern as shown in an example in Crystal orientation from CBED.

Solving a set of SAED patterns (from a tilt series) together is shown in Solving a set of SAED patterns.

Dealing with FFT of HRTEM

A typical application is that you want to use the FFT of a HRTEM to identify a phase like from a SAED pattern. Many users compute the FFT in Digital Micrograph[4] (DM) where the calibration of the FFT is as good as the calibration of the image was. That calibration value can be read out and fed to ProcessDiffraction. The recommended procedure is that the calibration value is included in the file name of the FFT. The example shown here also uses an FFT saved from DM.

Start a new project! Load the pattern **FFT_HRTEM_Pt_0p094083.tif** from the examples! It is an FFT of the image of a Pt particle (shown in Figure HRTEM of a Pt particle) calculated by DM. The calibration value (0.094083)[5] is coded into the file name. A message comes up during loading: "Look-up table (LUT) was found in the file". Select: *Keep the original data and use the LUT for displaying*! The loaded pattern looks like in Figure FFT of HRTEM.

In the ***Info*** window select Radio-button *Total k-calibration constant [DM]*! Enter the calibration value of 0.094083! Select FFT in the list *Pattern type*! Accept the recommended 256 points from the drop-down list! It should look like in figure Info_FFT. Click OK to switch to the ***SAED*** window! Set the center to 128, 128 (=half the number of points), since it is always at the geometric center of the image for FFT! Select menu ***Process / Show Cursor and Correlation Start***! Select two short reciprocal vectors as in section Indexing a single crystal pattern! Should look as in Figure Vectors_FFT. Copy to a new ***Crystallographic Calculator*** window using menu! Now the first reflections were selected. Load structure

[4] Trade Mark of GATAN inc.

[5] That calibration value can be read out in DM from menu Display / Image Display and selecting Calibration.

Pt_fcc.str from the examples! Solve the pattern as in Indexing a single crystal pattern! The result should be <110> type zone axes. Label the pattern as in Automatic labeling of single crystal SAED!

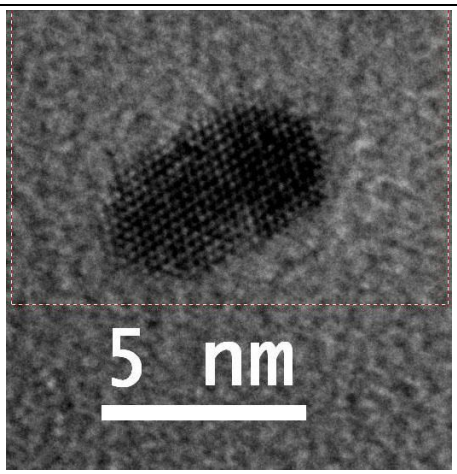


Figure HRTEM of a Pt particle

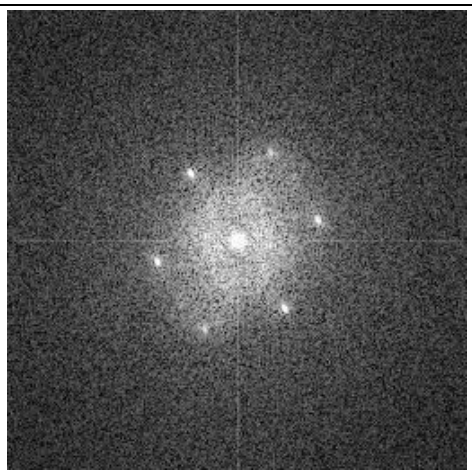


Figure FFT of HRTEM

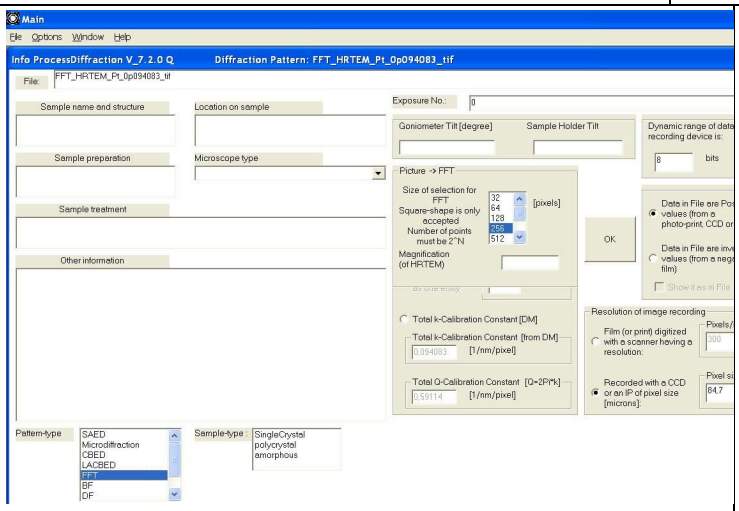


Figure Info_FFT

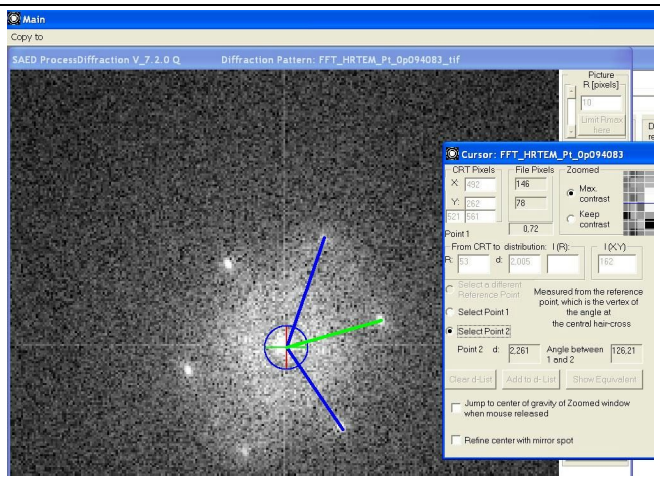


Figure Vectors_FFT

Crystal orientation from CBED

Load the CBED pattern[6] **CBED_Si_265mm_3x9001.IPC** from the Examples! The name of the phase and the camera length are coded into the file name. Fill in calibration data in the ***Info*** window! (Accelerating voltage=200 keV, Camera length=265 mm). You can see that the information about pixel size was filled in for the value pertinent to the IP. If you are not happy with the visibility of the Kikuchi-bands, you can enhance the rendering as in Optimized rendering on SAED screen. Select menu ***Structure / Determine orientation from Kikuchi lines***! A new small window named ***Mark Kikuchi line-pairs*** appears. Resize it to see all information and move it not to block the interesting part of the pattern! Center the reference circle to the center of the direct beam! Click menu ***Set Beam direction***! A new message acknowledges that the beam direction was set. Close the message window by clicking OK! Now select 3 Kikuchi bands that form a triangle as described below

1. Select one side of a narrow Kikuchi band from a triangle by the Mouse! (Click by left Mouse button, hold down while moving and release when happy!) If dark and bright lines can be distinguished, start with the dark one!
2. Click radio-button *Select Bright Kikuchi Line*!

[6] CBED here only means that the pattern was recorded with convergent beam. However, Kikuchi lines and bands are used from the pattern in this section.

3. Select the line at the other side of the Kikuchi band!
4. Click Command-button *Add Kikuchi line-pairs to the list*! The dark and bright lines of your selection disappear and a blue line will indicate the middle of the selected band as in figure First_Kikuchi.
5. Select the second and third lines similarly as in 1-4![7] The result should look as in figure All_Kikuchi. The selected bands and the zones at their intersections are labeled. The parameters of the lines are listed within window ***Mark Kikuchi line-pairs***.

Click menu item ***Solve here***! A window, named ***Select a Model structure to solve Kikuchi-triangle*** appears allowing you to select from pre-stored structure files. (For preparing Model structure files, see section Generate true electron diffraction Markers!) Select **Si_cF8.str**! A message appears, informing you how many solutions are offered for line-1, line-2 and line-3, as in Figure Kikuchi_NSol. If all of them are >0, select OK! If the photo number was not filled in the ***Info*** window at the beginning, the program asks for the photo number here. A name for the orientation matrix, to be saved is asked for. If the microscope type, sample name and location on sample fields were not filled in previously in the ***Info*** window, they are also asked here. A Document window appears with a list of the found solutions. For Si (cubic system) 24 solutions are expected[8]. Go through the content of this document to see the listed information!

If the window ***Mark Kikuchi line-pairs*** is active, you can select menu ***Show selected Kikuchi solution***. You can select one of the 24 symmetry equivalent solutions to be used for labeling the solved pattern. Is should look as in figure Solved_Kikuchi.

The appearance of the figure was enhanced by increased letter size, shift and magnification to better show the labeled part of the pattern. See section Automatic labeling of single crystal SAED for a description how add, size and move labels on the screen! It is important that when you want to shift and magnify a part of the labeled pattern, you shift both the center and the labels together with the pattern. To do so, select menu ***Overlay / Controls for shift of Reference circle***! In the appearing control select check-box *Fix reference circle to picture*! Now each shit and magnification change will affect both the pattern and the center simultaneously. The action is reserved while the check-box is selected even if the controls themselves are hidden again.

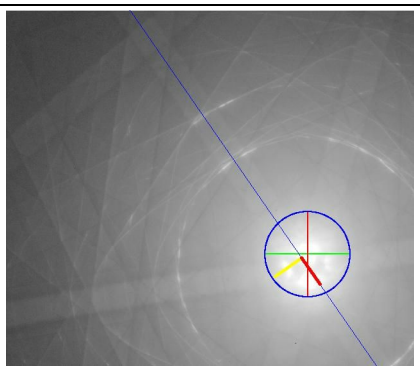


Figure First_Kikuchi

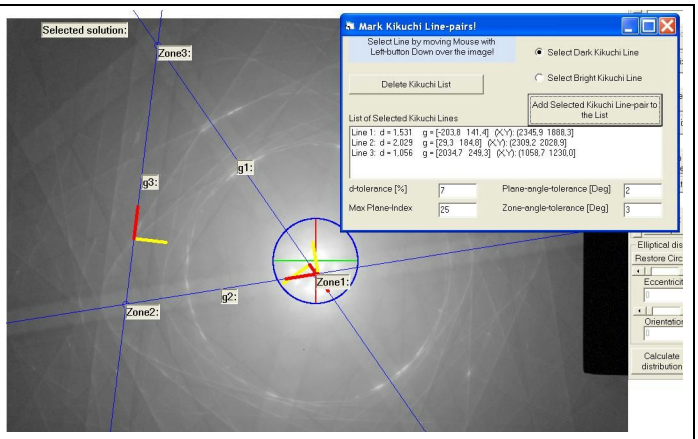


Figure All_Kikuchi

[7] Generally sequence of lines is suggested clockwise. However, if selection was made anticlockwise, the program automatically renumbers the lines the 1-2-3 follow each other clockwise.

[8] Since right-handed solutions are only searched for, only the proper rotations of the point group transform possible solutions into each other.

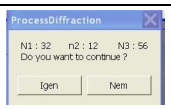


Figure Kikuchi_NSol

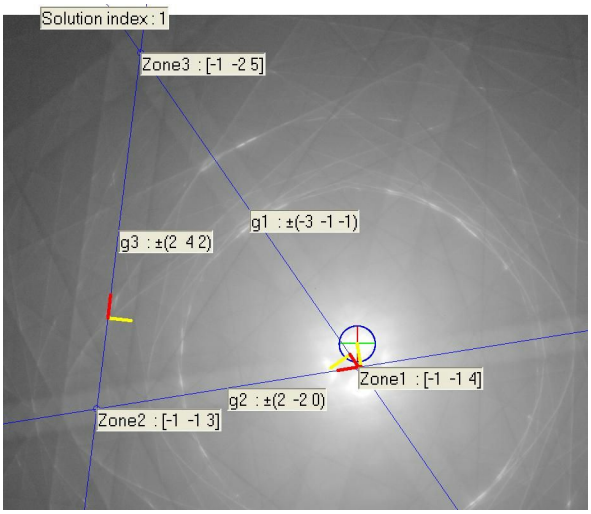


Figure Solved_Kikuchi

Using a priori information

A priori structural information comes from the literature. You store that information in separate structure files. ProcessDiffraction renders the diffraction lines from these structure files. Qualitative analysis only relies on the position of the lines, so the unit cell parameters are only needed. That is why XRD files can also be used for qualitative analysis as an alternative to our structure files. Quantitative analysis uses intensities of the diffracted lines, too. Calculation of the intensities needs the atomic positions within the unit cell, which are also stored in these structure files. Since XRD intensities are different from those of ED, XRD data cannot be used for quantitative analysis.

Generate true electron diffraction Markers from structure data

As we saw above, a "Marker" is a collection of diffraction lines that belong to a given phase[9] as also shown in Figure Net Peaks. Positions of the diffraction lines can be calculated from the parameters of the unit cell. However, the intensities of these diffraction lines are determined by the content of the unit cell. Consequently, we can only calculate the intensities if we know the positions of all atoms within the unit cell. Such detailed information is not available for all the phases that are listed in the diffraction data bases. Structure information with atomic coordinates is listed e.g. in Pearson's Handbook of Crystallographic Data for Intermetallic Phases, P. Villars and L.D. Calvert eds., American Society for Metals, 1985. The new Pdf4+ database from International Centre for Diffraction Data also contains that information for many phases and that number is increasing year by year. A search on the internet for a given phase may also show up original publications about the structure with atomic coordinates. Structures of many phases are also available in the free database at http://crystdb.nims.go.jp/index_en.html. Usage of that information is shown below.

Select menu ***Structure / Define new structure***! The ***Crystal structure definition*** window should appear, as in Figure Structure Definition.

[9] "Phase" is used in a thermodynamic sense. It means a homogeneous piece of material with a well defined crystal structure (and composition).

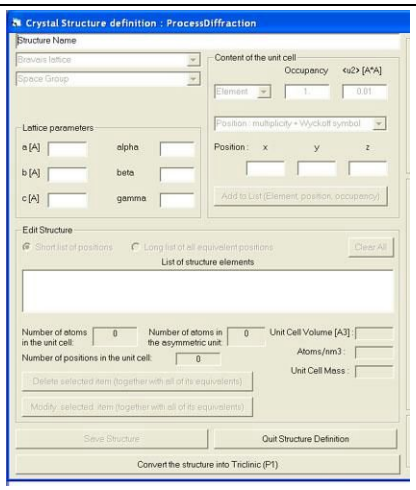


Figure Structure Definition

As you enter information, new and new controls will be activated facilitating further steps.

Enter *Structure Name*! Select *Bravais lattice* and *Space group*! Enter *Lattice parameters*! You see that only the independent parameters are asked, the rest is filled in automatically. Select an *Element* from the list then select *Position: multiplicity and Wickoff symbol* from the list! You can see that positions relevant to the selected Space group (SG) are only listed. Enter atomic coordinates into *Position x, y, z* as fractions (0≤value<1)! Click Command-button *Add to list*! Check the difference between *Short list* and *Long list* for your education only (by clicking Radio-buttons)! You need to enter the content of the asymmetric unit only. The rest is generated for you by the symmetry operations of the SG.

When all atomic coordinates are entered click *Save Structure*! The directory, specified in ***Options*** is used as a default location to store your new structure file. It is recommended that you give "talking names". It is recommended that you include the name of the phase and Pearson's symbol for the structure, like "**Al3Pt2_hP5.str**". The extension of the file name is always "str". You can see calculated data in the window (number of atoms, volume, density and mass).

You can edit an existing structure by loading it: menu ***Structure / Load crystal structure***! To edit, click on an item in *List of structure elements*! Two Command-boxes should be activated: *Delete selected item* and *Modify selected item*. Modify the values in *Element*, *Wickoff* and *Position* boxes and click *Modify selected item*! The original values for the selected item is deleted from the list and the new, modified values are added to the end of the list. You can save the modified structure when you finished all modifications. The structure file is a simple text file, so you can read its values. Modification is only recommended by using the Crystal structure definition module (as described above).

When you finished, either close the window (upper right corner) or click Command-button *Quit Structure Definition*!

Your new structure is now ready to be used either for generating Markers for a measured distribution or for indexing a single crystal pattern or for calculations in the *Crystallographic Calculator*.

Generate (or load an existing) distribution of measured diffraction intensities into the ***Intensity*** window! Select ***Marker / Show more Markers / Calculate >>On the Fly<< (from Structure file)***![10] The list of your existing structure files (stored in the directory specified in ***Options***) appears. Select one from the list! A small window pops-up to inquire if you want to generate the Marker for a random orientation distribution of the grains of that phase. Click Command-button *Yes*! (We shall discuss preferred orientation distributions later.) The new Marker should appear overlaid your measured distribution as in Figure Net Peaks. That Marker is calculated for kinematic conditions. (Dynamic effects will be discussed later.)

The name of a Marker in Legends always shows the type of the data source. XRD information entered manually end at "XRD". Names for data from the Pdf2 database always end at "Pdf" and the number of the Pdf-card. Diffraction Markers calculated for electron diffraction show if the calculation was done for "random" distribution (or the axis of the texture is shown) and if it is "kinematic" or "dynamic".

Using the Pdf-2 database

This XRD database can only be used for phase identification, but cannot be used for quantitative phase analysis. It is NOT part of the ProcessDiffraction program, however, legal users of it may be able to use it

[10] Markers can only be generated if calibration data are completely specified in the ***Info*** window.

from within ProcessDiffracttion. The last database version is from 2003 that was tested. Newer possible modification to the database structure by ICDD might create an issue for the operation here. This is now a dis-preferred way of using a priori knowledge. It is rather recommended that information, collected from a free online database (as e.g. http://crystdb.nims.go.jp/index_en.html) be supplied to ProcessDiffraction to create a structure file and use that structure file for both qualitative and quantitative phase analysis.

Pdf-2 can be used either manually to examine XRD information from a phase or alternatively, it is used to generate a Marker for qualitative analysis.

For manual examination select menu ***XRD Database*** in the ***SAED*** window! A new ***XRD*** window appears. Select (multiple) element(s) from the element list and click Command Button *Search in Pdf database*! Alternatively, specify the JCPDS Card Number and Search! When the list of possible phases appears, select on by clicking with the Mouse and click Command Button *Show details of the selected phase*!

For generating a Marker, load a pattern, process it to obtain a distribution (as e.g. Gross intensity) [or alternatively, re-load a previously processed project with a distribution]! Select menu item ***Marker / Show more Markers / from powder diffraction database*** in the ***Intensity*** window! The Marker lines will appear overlayed your measured distribution in the ***Intensity*** window, provided you previously specified (at least rough) calibration data in the ***Info*** window.

Calibration

Calibration of scattering angle: 1D

A simple rough calibration is described here. More accurate calibration is obtained as described in Fine tuning calibration.

Load the ring pattern from file **Cu-kalibr_883.tif**, center and correct for distortions as in section Pattern distortion!

Calculate a Marker for random orientation distribution grains from structure file **Cu_fcc.str** in the Examples as shown in section by finding which Marker(s) fit your measured peaks you identified which phases are present, so you performed your first qualitative phase analysis.

Change the axes of the Graph in ***Intensity*** window to [40-70 1/nm] and vertical full scale 200 as shown in section Optimized information in the Intensity window!

Select menu ***Calibrate / Camera constant by Marker***! A small control appears at the top right corner with 2 empty Text-boxes. Double-click on a peak! The channel number of the marked peak position appears in the Text-box *SAED*. Double-click on the corresponding Marker line! The channel number of the marked line position appears in the Text-box *Marker*. Click on Command-button *Start* and the selected line will exactly match the position of the selected peak. The value of the *Camera length* is also updated in the corresponding Text-box in window ***Info***. If your original calibration was too good to see any change, first modify the *Camera length* in window ***Info*** to a slightly different value (make it wrong) and repeat the procedure!

Calibration of pattern distortion: 2D

Load a ring pattern and center it as in section The simplest operations with your first ring!

Make sure Check-box *Fix Reference Circle to Picture* is Un-checked and its parent controls are hidden as in section Indexing a single crystal pattern!

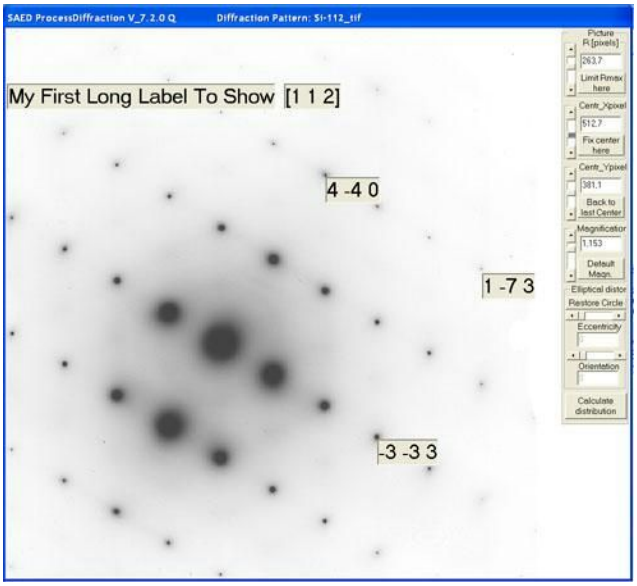
Adjust magnification of the pattern and radius of the reference circle that a separate measured ring with good contrast almost overlaps the reference circle! Adjust Scroll-bars *Eccentricity* and *Orientation* at the right side of window SAED until the best match is achieved between the reference “circle” (which is an ellipse now) and the measured ring! Generate elliptically averaged intensity distribution (and compare it to the circularly averaged one as in Comparing and saving distributions)!

The same values of *Eccentricity* and *Orientation* can be used for other patterns (either rings or single crystal spots) recorded under identical conditions at the same TEM to correct for their distortions. Values read out with the Cursor are also automatically corrected for those distortions.

Rendering

AUTOMATIC LABELING OF 2D SINGLE CRYSTAL SAED PATTERNS

After you have solved the single crystal pattern, select menu ***Solve / Label Spot Pattern with Solution*** in the relevant ***Crystallographic Calculator*** window! A pop-up window appears with a list of solutions (48 equivalent solutions in the example above). Click on your selection in the list then click on Command-button *OK: Show Selected*! From menu ***Window*** in the ***Crystallographic Calculator*** window select ***SAED***! You can see that controls for the reference circle also appeared (usually they are hidden) and the Check-box *Fix Reference Circle to Picture* is checked. Its effect is that when you move the image with the controls *Centr_X-pixel*, *Centr_Y-pixel* and *Magnification* (at the right side of window ***SAED***) both the reference circle and the labels (of the reflections) move together with the diffraction pattern. The Label with the zone axis indices [in squared bracket] does not move, but stays at the middle of the upper part of the screen. In the ***SAED*** window select menu ***Edit / Size Labels***! Enter the value 20! Move your pattern and magnify to get the best appearance. You can add an additional label manually by selecting menu ***Edit / Add Manual Label***. Enter the text "My First Long Label To Show"! You can see that major part of the Label is covered by the controls of the reference circle. Select menu ***Overlay / Controls for shift of the Reference Circle***! The control disappears and you can see your manually entered label. Select menu ***Edit / Move Manual Label***! Click on the label and hold down the left Mouse button and drag the label. Your Manual Label moves to the position where you release the Mouse button.

The result should look like this. You can copy that image to the ClipBoard by <Alt> <PrtScr> and insert into any document by <Ctrl>V. If the contrast of the spots in your measured pattern does not satisfy you, see section Optimized rendering on SAED screen! Figure_Labelled_sc_pattern:	

OPTIMIZED RENDERING ON SAED SCREEN

1. Displaying or not of automatically generated labels, the Reference circle and controls for moving the Reference circle in window ***SAED*** is controlled from submenus of menu ***Overlay***. They are simple Toggle-switches. Status is indicated by checking of the submenu item. Examples of usage are given in section Automatic labeling of single crystal SAED and Crystal orientation from CBED.
2. The appearance of the pattern can be manipulated in the ***SAED*** window in several ways. First, a scanned in negative can be shown either as a negative or as a positive print (controlled by the *Show as in the File* Check-box in the ***Info*** window). Second, linear or logarithmic rendering with variable lower and upper intensity values can be selected from menu ***Edit / Edit Gray level Histogram of Viewed Image***. When this menu is selected, the ***Gray Level Histogram*** window

appears, with gray levels on its X-axis. By double-clicking on the axis, the ***Gray Level Histogram Axis*** window appears where you can select lower and upper gray level values to be displayed (as shown in Figure Gray_Level_Axis). Selection of the values for the X-axis is the important. Do not forget the select Radio-button *Manual* for the X-axis! You may select if different renderings of the same pattern are saved under different names (if permanent storage was selected in ***Options***) or the default bmp-file is overwritten each time.

3. Visual appearance of a pattern can be further enhanced by removing the non-linear background below the spots and/or rings. This is to be applied if faint features are only seen over a large background. First, load and image, correct for elliptical distortion, center and calculated distribution from it! Calibrate if necessary! Then in the ***Intensity*** window select menu ***Process / Find Minimal Values along each Ring***! Next, select only these minimum values to be shown! Then select menu ***Process / Calculate Background***! Select **Minimal Values** as source and **Spline** (or **log-Normal** if you wish) as shape then select the background points at the upper envelope of the visualized **Minimal Values** curve along the entire interval! Check if the fitted curve follows the trend as you want it! As a last step, select menu point ***Process / Correct Pattern for BKG***! Give a name for the BKG corrected image! You can see it in the ***SAED*** window or use the saved version in any other programs!

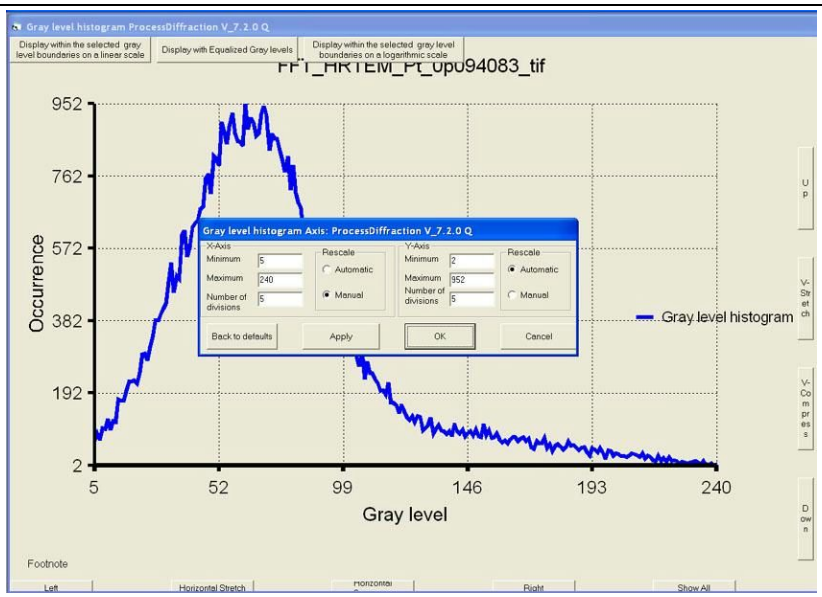


Figure Gray_Level_Axis

If you click on Command-button *Display within the selected gray level boundaries on a linear scale*, the display file (bmp) is re-generated and the pattern shows the new appearance.

Logarithmic display is obtained if the Command-button *Display within the selected gray level boundaries on a logarithmic scale* is clicked on.

Usage of logarithmic intensity scale is recommended for files with large dynamic range (like IP files). The default value is logarithmic for IPs and linear for the rest.

Automatic labeling of rings

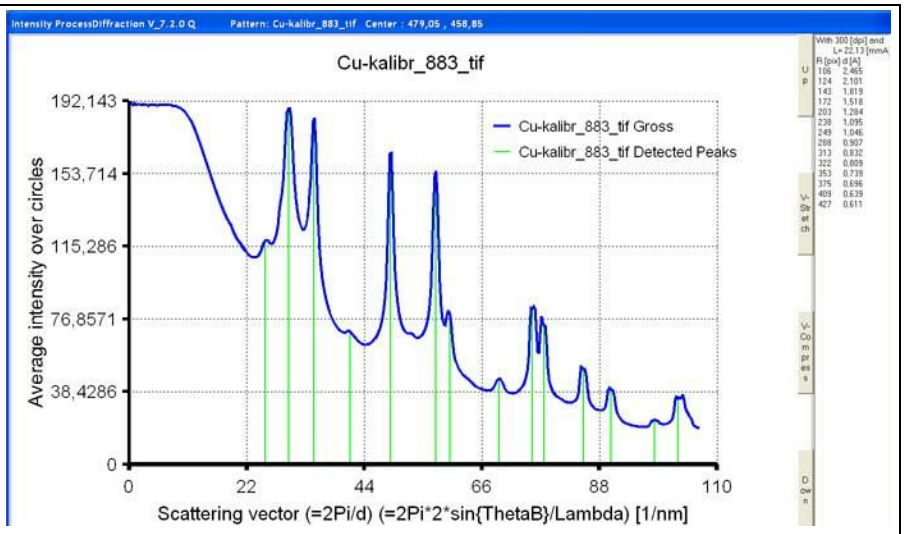
Load and center a ring patterns and calculate the Gross intensity distribution averaged over circles (or ellipses) as in section The simplest operations with your first ring! Correct for elliptical distortion as in Pattern distortion! Calibrate Camera length if necessary as in Calibration of Patterns! Select menu ***Process / Find Peaks***!

A small window ***Filter*** pops-up with filter parameters. If you can not see all parameters, resize the window! Keep the default parameters and click Command-button *Start Peak-search*!

The result should like this.

Vertical lines mark the detected peaks. Positions (and corresponding d-values) of peaks are listed at the right of the figure.

Figure Found Peaks



In case of false identification the peak-list can be corrected manually. Click on the undesired item in the Peak-list (at the right) and hit <BackSpace>. The item disappears from the list and the corresponding vertical line also disappears from the Graph.

Overly Marker lines of the given phase as in The simplest operations with your first ring!

Select menu ***Process / Schematic Half Circles***! Half of your measured ring pattern will be replaced with schematic half-circles at the positions where peaks were found. The calculated composite image is saved in a different Bmp file (used for display only). Whenever a peak is close to a Marker line, the corresponding circle is labeled with the name of the diffraction line. The top corner of the label (closest to the central vertical) identifies which ring the label belongs to.

Fix the reference circle to the pattern as in section Indexing a single crystal pattern!

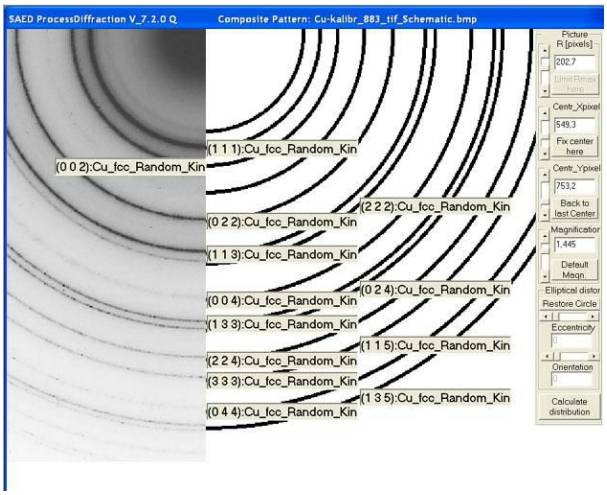
Magnify and move your pattern and see how the labels stick to the circles! When you are happy, you can copy to the ClipBoard and save the publication-ready image into your document or into any image processing software.

The result should look like this.

Remember, you can also change label size and add a Manual Label to it as in section Indexing a single crystal pattern!

You can also save the Project.

Figure Indexed_Rings



Optimized information in the Intensity window

The 1D intensity distributions can be displayed with either linear or logarithmic intensity scale. The Xaxis units can be selected as either [pixels] or [Q=2π/d] in [1/nm] units or [mrad] for the scattering angle of electrons. These parameters, together with lower and upper limits for either axis can be selected from the ***Axis*** window that is evoked by double-clicking on either of the axes. Example is shown in Figure Graph Axis.

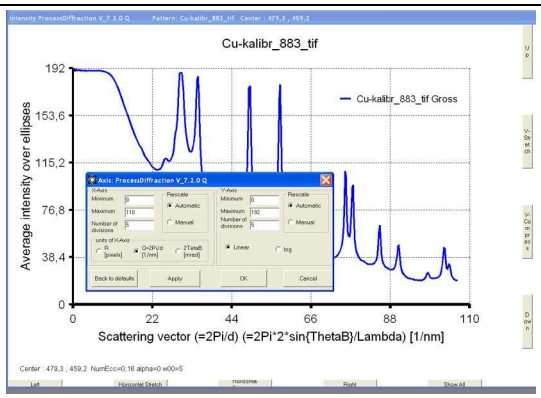


Figure Graph Axis

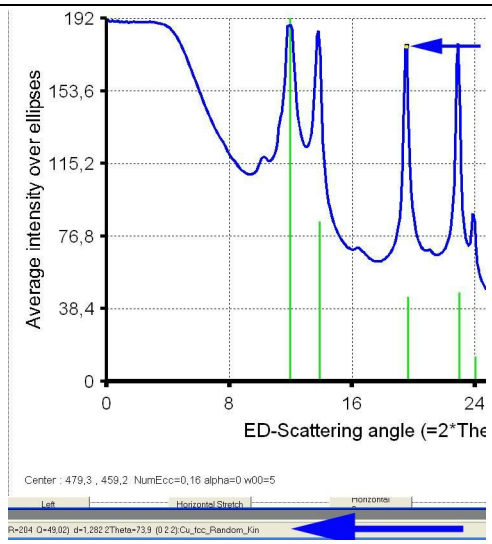


Figure X readout

When a point on the graph is clicked on, information appears at the bottom line of the window as in Figure X readout. Both the selected (clicked) point and the information line are marked with arrows in the figure. The position of the selected point is listed as pixels and Q[1/nm] with the corresponding d-value [1/Å]. It is shown how large 2θ would correspond to this peak at XRD with a Cu source. Also listed which line of the generated Markers is within experimental error.

Alternatively the range of display can also be manipulated with the Command-buttons at the bottom and at the right side of the window.

Comparing and saving distributions

The basic idea in the program is that only calibrated patterns are to be compared. Four dedicated memories are reserved for comparing different distributions. Saving is done in tow steps. First the distribution is copied into a Compare Memory and next it is saved into a file from there. Saved distributions later can be loaded into one of the Compare Memories of another project.

After proper calibration of your distribution (as in The simplest operations with your first ring pattern: qualitative phase analysis and Calibration of Patterns) select menu ***Compare / Copy distribution to next compare memory /*** and select the distribution you want to copy! Now you can save it by selecting menu ***File / Save distribution data / form Compare memory x***!

In a new project you can reload it by selecting menu ***File / Load distribution data / to Compare memory x***! Rendering can be controlled by using menu ***Show*** and checking the wanted distributions to show.

Reading out data points

Individual values can be read out from the 2D pattern using the ***Cursor*** window (the ***Cursor*** window can be activated from either the ***SAED*** window or the ***Intensity*** window using menu ***Process / Cursor and Correlation Start***). There are two ways of reading out individual data values from a 1D distribution. First, if you click on a point in your distribution in the ***Intensity*** window, the values at that point are shown at the bottom line of the window. Second, the ***Cursor*** window also gives you the value and can show the ring on the 2D pattern that corresponds to the point in the 1D distribution.

Separate and disregard image components: Masking

The aim of Masking is to separate certain parts of the recorded pattern (shadow of beam stop, shadow of numbering system, single crystal spots unintentionally collected from traces of incompletely removed single crystal substrate below the interesting polycrystalline layer, etc.) and exclude them from processing.

Load, center, calibrate and correct a pattern! Select menu ***Edit / Select a rectangular part of pattern*** in the ***SAED*** window! Select the region to be excluded from processing with the Mouse! Select menu ***Edit / Masking / Add the last selected rectangle to the composite Mask*** in the ***SAED*** window! Be sure that rendering of Mask is checked in menu ***Overlay / Mask***! Continue with selecting and adding new and new rectangular parts (and circular parts similarly)! Advice is also given to how to generate a set of circular disks to block out single crystal spots.

When ready, you can save the Mask by using menu ***File / Save Mask***. To apply the Mask in the afterwards operations, select menu ***Process / Apply composite Mask*** in the ***SAED*** window! The masked rendering file is sawed as a Bmp file separately and you can swap between the original and the masked rendering file from menu Show in the SAED window. These rendering files do not affect the measured and processed intensities. The usage of the masked intensities (in 1D distribution) is from the ***Intensity*** window by selecting ***Process / Calculate Gross Distribution from Corrected Pattern***. After that you probably will continue by evaluating that GrossFromCorrected distribution (e.g. in quantitative phase analysis).

For a new pattern with the same geometry of unwanted components (e.g. the next item in an exposure series) you can load a previously saved Mask and apply it in the same way.

Detecting faint features

Presence of a minor phase can be inferred from the appearance of a set of a few spots that do not form complete rings. The contribution of the few points to the intensity averaged over the entire circle may be too low to be seen in the 1D Distribution. The procedure that we discuss here makes these faint features appear distinct in the 1D Distribution facilitating their identification by comparing them to Markers.

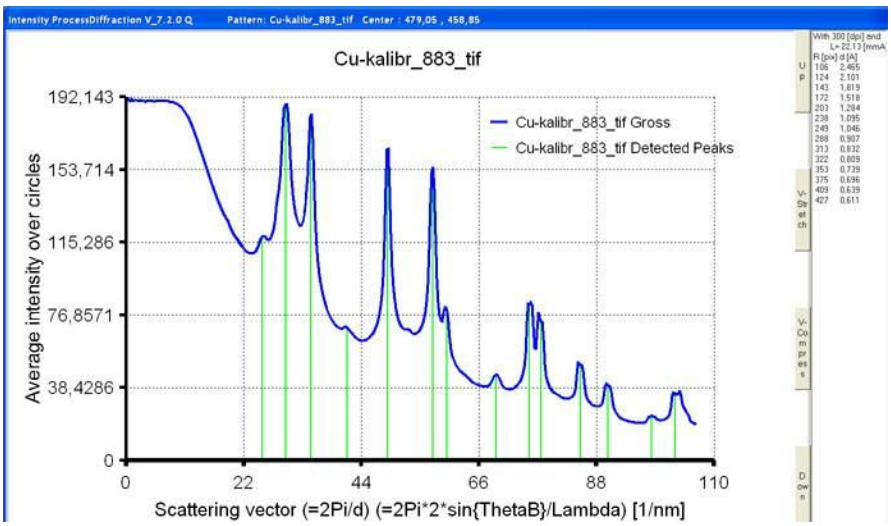
Load a pattern that contains scattered points in addition to the rings (as in Ring patterns)! Center the pattern and correct for elliptical distortion (as in Pattern_distortion)! Calculate the 1D distribution (as in Ring patterns)!

Select menu item ***Process /Find Maximal Values Along Each Ring*** in the ***Intensity*** window!

Estimating unknown cubic cell parameters from the distribution

This is a simple calibration procedure to obtain a rough estimate of a cubic cell parameter, using the position of the first strong peak in the distribution.

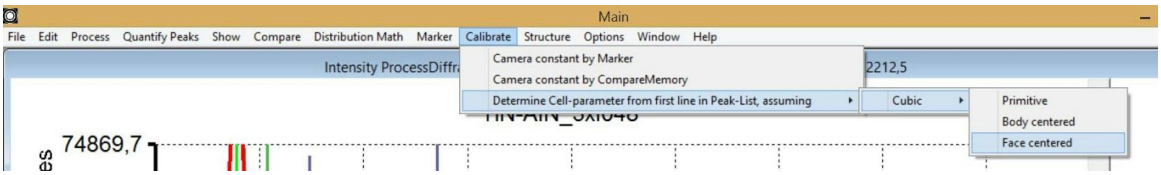
Load a distribution from a cubic phase! Specify its calibration parameters in the ***Info*** window! Select menu ***Process / Find Peaks*** and click the *Start Search* button in the new small window. A list of the detected peaks appears at the right side of the ***Intensity*** window. The detected peaks are also shown graphically.



Peak List

This list can be cleared or additional peaks can be added from the ***Cursor*** window (the ***Cursor*** window can be activated from either the ***SAED*** window or the ***Intensity*** window using menu ***Process / Cursor and Correlation Start***). To delete an individual item, click on it (at the right side) and press "Backspace" in your keyboard!

When the first strong peak is left as the first item in the list, select menu ***Calibrate / Determine cell-parameter from the first line in Peak-list, assuming / Cubic / Face-centered*** (if your structure is fcc)!



Now you see a new Marker, called "Cubic_F_a=4.788_LastCalculated". The determined cell parameter is in the name.

Advanced topics

Unified information from several diffraction patterns

Extending dynamic range

Dynamic range (difference between minimal and maximal data values) can be limited in any single patterns. It can be extended by merging several patterns, recorded with different exposure times. Merging is performed with the 1D distributions that were calculated from the 2D patterns. The individual distributions are normalized together using a manually selected interval (where both distributions have significant, but not saturated values). Calibrated patterns can only be merged!!!

Load the first pattern from the exposure series (as in Ring patterns)! Center the pattern and correct for elliptical distortion (as in Pattern_distortion)! Calculate the 1D distribution (as in Ring patterns)!

1. Select menu item ***Process / Merge Present Distribution with / Another unprocessed SAED in file*** in the ***Intensity*** window! Select menu item ***Process / Reset Merging*** in the ***Intensity*** window! Select the source (e.g. "Gross") in the pop-up window! A message will appear explaining that you should process the next pattern.
2. Load the next pattern from the exposure series! Center the pattern and correct for elliptical distortion! Calculate the 1D distribution! Switch the x-axis to "R [pixels]", since the interval will be asked in pixel units! Read out and remember which interval is good for normalization (intensity values must be significantly above statistical noise and below saturation values)! Select again menu item ***Process / Reset Merging*** in the ***Intensity*** window! Specify lower limit and upper limit for the normalization interval in the Input-box! The merged distribution is calculated for you.
3. Repeat step 2 if more patterns are to be merged!

Do not forget to save the project with the merged data to avoid losing the results of your merging work!

Cumulating several patterns into a single pattern

Occasionally you need to unite several patterns in order to

- Improve statistics
- Average orientations in a nanocrystalline sample
- Protect beam sensitive samples
- Measure predefined objects that can not be selected with a single selection of the selected area aperture (SAA).

Typically you record a series o diffraction patterns by either scanning the electron beam over an area or by using a small SAA in a series of locations (e.g. thin layer in cross section, of and irregularly shaped inclusion, etc.).

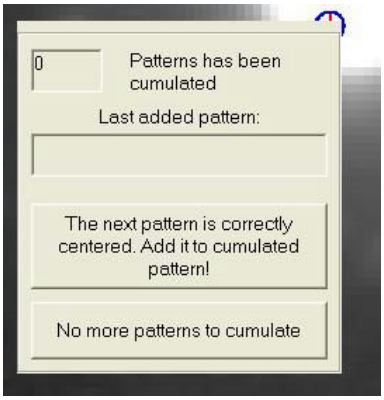
Uniting those patterns is done with the "Cumulate" function of ProcessDiffraction.

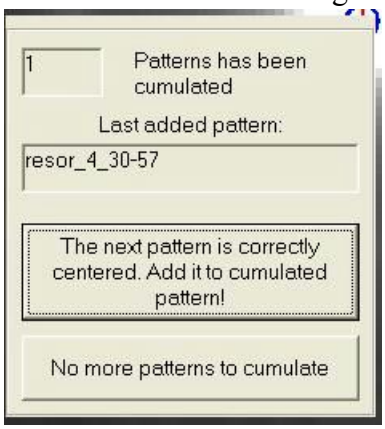
Load the first pattern! Fill-in the calibration data!

Click menu item ***Process / Start to sum-up (cumulate) common part of individually centered SAED patterns***!

A message appears: 

Click OK!

A Frame appears with command buttons: 

After centering the first pattern, click command button "*The next pattern is correctly centered. Add it to the cumulated pattern*"! The above frame changes showing the number of cumulated patterns and the name of the last one: 

.

Select menu item ***File / Keep present Project and load a new pattern into it***! You can see that the calibration data are preserved. The program also checks if a pattern with the same size was loaded.

Center the pattern and click command button "*The next pattern is correctly centered. Add it to the cumulated pattern*" again!

Repeat until the last pattern was also cumulated. Then click command button "*No more patterns to cumulate*"!

Now the common part of all cumulated patterns is kept (crop operation) and saved in tow formats: First the rendering file is saved in Bmp format. Next the real data are saved in 4-byte floating point format with ".dat" extension. This format saves the data pixel-by-pixel with no header added. The format information is coded into the filename. "B4" means 4-byte pixels, "H0" means header of zero length, "Wabc" means with of "abc" pixels and "Hefg" means height of "efg" pixels. You can modify the file name at that point however modification of the format data is not advised. This cumulated image can be reloaded later again for any other processing without the need to redo the cumulate process.

The cropped cumulated pattern is now ready for any processing in the same way as if a single pattern had been loaded.

Solving a set of single crystal SAED patterns

Intended use: record a set of single crystal patterns and record the goniometer settings for each. Solve them one-by-one. For some of them more than one group of symmetrically equivalent solutions might result. This ambiguity can be resolved by evaluating the entire set together, using the angular relations between the directions of their observations.

Load and solve a single crystal pattern. Specify the goniometer settings and sample name in the ***Info*** window! When the solution is ready, select menu ***Solve / Manage Set*** in the ***Crystallographic Calculator*** window! The list of available patterns will show your solved patterns. Click the solved pattern(s) to select it/them and click the Command Button *Add selected items to the SET* in the ***Manage SET*** window! Now

they will appear in the List *Members of the SET*. If either the name or the goniometer setting were not specified, they will be asked here. When the set is specified, you can try to solve it by clicking menu item ***Show SET Results*** in the ***Manage SET*** window. Only a single group of symmetrically equivalent solutions should remain.

Misorientation at Grain Boundaries

Misorientation is deduced from previously calculated and saved orientation matrices (see section Crystal orientation from CBED!) Determine the orientations from two CBED patterns (**CBED_Si_265mm_3x9001.IPC** and **CBED_Si_265mm_3x6312.IPC** from the Examples) and save the orientation matrices!

Select menu ***Structure / Grain Boundary / Grain Boundary Solve*** in the ***SAED*** window! The result will appear in a new ***Document*** window.

Quantitative analysis of nanocrystalline ring patterns

The 1D distribution obtained from the ring patterns is processed by fitting both a background-shape and peak-shapes and adjust them to mimic the measured pattern as closely as possible. The number and kind of parameters varies during the procedure, as described below.

The two main types of fitting operation are *Pattern decomposition* on the one hand and (Quantitative) *Phase analysis*[11] on the other hand.

All fitting procedures that aim at minimizing the deviation between measured and modelled quantities are prone to fall into local minima and the closer we can start the procedure to the true values in parameter space, the higher the chance of finding the global minimum.

Fitting a single peak: fine tuning calibration

In order to fine-tune the calibration of the camera length and to determine the peak shape you can perform the simplest case of Peak deconvolution: fitting a peak shape to a single, separated peak. This can be useful to generate the best starting estimate for these parameters for the later, more complex fitting procedure, when more parameters are to be varied. You can use "inheritance" to copy that peak shape to the starting value of all diffraction peaks by using menu ***Markers/Let next Marker inherit the peak shape of Marker(1)***. (It is effective until you deselect that menu item again.)

First, produce a measured 1D distribution from the measured 2D pattern (e.g. "**TiN_3xQ842.IPC**"[12] from the examples)! Calculate Net intensities! Generate the Marker of the phase to be analyzed that contains the peak in question[13] (use the structure file **TiN_cF8.str**)! Calibrate it roughly as in Calibration of Patterns[14]. Specify fitting limits by selecting menu ***Quantify peaks/Set fitting interval*** and then by double-clicking on the start channel and the stop channel. The program acknowledges your selection by changing the text in the Status bar at the bottom. Select the strongest peak (between Q≈27.5 and Q≈32) Next, specify the mode and parameters of fitting. Select menu ***Quantify peaks/Setup fit parameters***. In the "General" ear, select "Net" for "Type of experimental data to fit to" and "R-weighted pattern" for the "Criteria to fit"! Specify 10^{-4} for tolerance! Click to the "Analysis type" ear! Select "Pattern decomposition"! Ensure that parameters for both Debye-Waller factor and grain size and cell parameters are NOT selected for optimization (NOT varied)! Go to "Peaks in the fitted profile"! Select "Adjust peak positions together (with camera length)", "Vary peak shapes" and select Pseudo-Voigt shape, with identical width for both the Gaussian and the Lorentzian component and "Proportion of the Gaussian fraction varied"! Select "Different constant peak width per structure" as a "constraint"! When click on the

[11] that results in numerical values of phase fractions

[12] It was recorded at 200 kV with nominal camera length of 1 m.

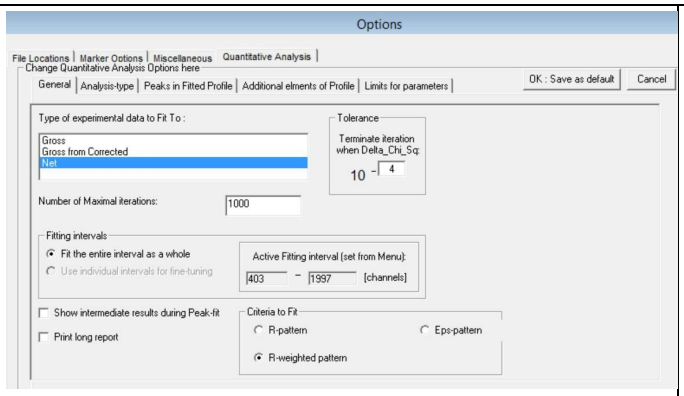
[13] That should be the only Marker. No other Markers must be present at that stage.

[14] The resulting camera length should be around 970 mm.

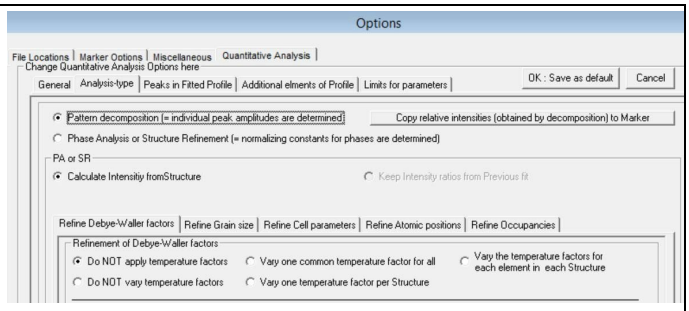
"OK" command button, the Options panel disappears. Select menu ***Quantify peaks/Fit***. When ready (fast) continue fitting (by selecting menu ***Quantify peaks/Continue Fit starting from the previous fit results***)! Continue fitting again and observe in ***Results*** window, how the final result is reached! (The camera length in the ***Info*** window should now be 969 mm.) At that point save[15] your project for later reuse!

Pattern decomposition: measuring intensities of a set of individually fitted peaks

First produce a measured 1D distribution from the measured 2D pattern (e.g. "**TiN_3xQ842.IPC**"[16] from the examples)! Calculate Net intensities! Generate the Marker of the phase to be analyzed that contains the peak in question[17] (use the structure file **TiN_cF8.str**)![18] Calibrate it roughly as in Calibration of Patterns[19]. (The above steps in this section can be replaced by loading the previously saved project file: **TiN_3xQ842_Net_Shape.pde**.) Specify fitting limits by selecting menu ***Quantify peaks/Set fitting interval*** and then by double-clicking on the start channel and the stop channel. The program acknowledges your selection by changing the text in the Status bar at the bottom. Select all the large peaks (between Q≈23 and Q≈96) Next, specify the mode and parameters of fitting. Select menu ***Quantify peaks/Setup fit parameters***. In the "General" ear, select "Net" for "Type of experimental data to fit to" and "R-weighted pattern" for the "Criteria to fit"! Specify 10^{-4} for tolerance! (At that point you can save your project as **TiN_3xQ842_Fit-start.pde** for later re-use.)



Fit Options General



Fit Options Analysis type / Debye-Waller

Check the check-box "Print long report"! Click to the "Analysis type" ear! Select "Pattern decomposition"! Ensure that parameters for both Debye-Waller factor and grain size and cell parameters are NOT selected! Go to "Peaks in the fitted profile"! Select "Adjust peak positions together (with camera length)", "Vary peak shapes" and select Pseudo-Voigt shape, with identical width for both the Gaussian and the Lorentzian component and "Proportion of the Gaussian fraction varied"! Select "Different constant peak width per structure" as a "constraint"! When click on the "OK" command button, the Options panel disappears.

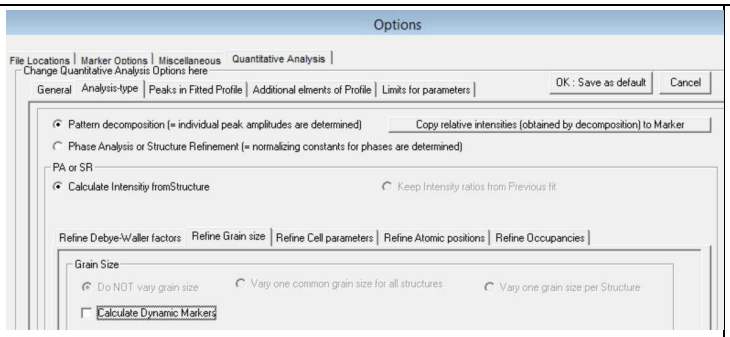
[15] e.g. specify file name **TiN_3xQ842_Net_Shape.pde**

[16] It was recorded at 200 kV with nominal camera length of 1 m.

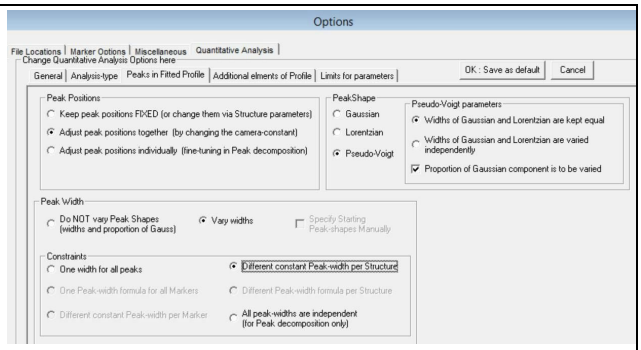
[17] That should be the only Marker. No other Markers must be present at that stage.

[18] As an (less preferred) alternative, manually marked peaks can also be used.

[19] The resulting camera length should be around 970 mm.



Fit Options Analysis type / Grain Size



Fit Options Peak shapes

Select menu ***Quantify peaks/Fit***. When ready (fast) continue fitting (by selecting menu ***Quantify peaks/Continue Fit starting from the previous fit results***)! Then continue fitting again! Leave most of the conditions as they were, only select "All peaks are independent" for constraint! Re-fit again and again and observe in ***Results*** window, how the final result is reached! You can see that the intensity ratios deviate significantly from the values predicted by kinematic scattering. Such deviation can mainly be caused either by the presence of preferential orientation distribution of the nano-grains (textured component) or/and by dynamic effects. (Temperature factors and changes in occupancy factors or in atomic positions also affect intensities.) Pattern deconvolution in itself cannot reveal the origin of the observed changes in the intensities. (Tilting of the sample proved that preferential orientation distribution (texture) is present in the present layer. The next two sections show how to quantify the components in the measured distribution.)

Parameter selection in pattern decomposition

In order to help the program find the global minimum, parameters must be selected as close to the true value as possible. This selection is approached step-by-step. The following sequence generally helps:

1. Calibrate camera length!
2. Calibrate peak shape if a separate good peak is available. Anyhow, even if you fit all peaks in a single step, start with fitting a common peaks shape to all peaks first. Do NOT use either D-W factors or dynamic markers.
3. In the last step you can release individual peak shapes (if it makes sense) and camera length.

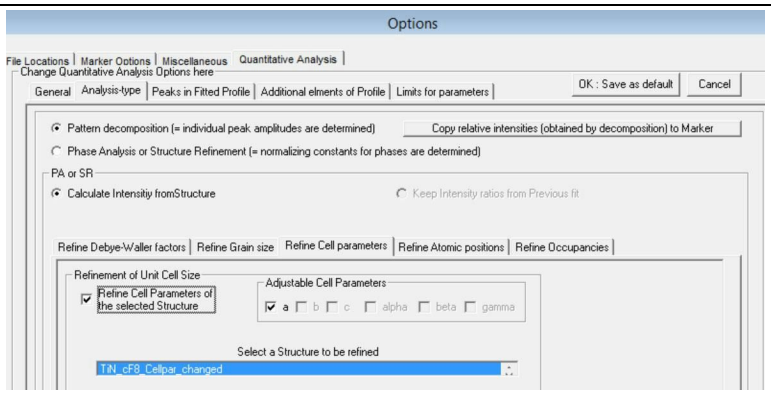
Quantitative phase analysis: random and textured components, dynamic effects and temperature factors

Load the project file **TiN_3xQ842_Net_Shape.pde**! You have one Marker in the project (TiN_cF8_random). Select menu ***Marker/Show more Marker/Calculate >>On-the-fly<<***! Select the same structure (TiN_cF8) again, but now answer "NO" to the question if random orientation distribution is to be calculated. Specify "100" texture! Now the random component and the 100-textured component of the same structure act as the two phases in quantitative phase analysis. Select menu ***Quantify peaks/Setup fit parameters***. Uncheck the check-box "Print long report"! Click to the "Analysis type" ear! Select "Phase analysis"! Ensure that parameters for both Debye-Waller factor and grain size and cell parameters are NOT selected! Go to "Peaks in the fitted profile"! In the "Peaks in the fitted profile" ear select "Keep the positions fixed" and "Do not vary peak shapes"! When click on the "OK" command button, the Options panel disappears. Select menu ***Quantify peaks/Fit***. When ready continue fitting (by selecting menu ***Quantify peaks/Continue Fit starting from the previous fit results***)! Then continue fitting again! Leave most of the conditions as they were, only select "Refine grain size" in the "Analysis type"ear of the Setup panel and check the checkbox "Calculate dynamic Markers" and select "Vary one common grain size for all structures"! Then continue fitting again! Then select "Refine Debye-Waller factors" in the "Analysis type" ear and select "Vary on common temperature factor for all"! Then

continue fitting again! Then leave most of the conditions as they were, only select “Vary peak shapes” and select Pseudo-Voigt shape, with identical width for both the Gaussian and the Lorentzian component and “Proportion of the Gaussian fraction varied”! Select “Different constant peak width per structure” as a “constraint”! Continue fitting! In the setup keep most of the parameters, only select “Adjust peak positions together (with camera length)”. Continue fitting again and again! Test the effect of increasing tolerance and continue fitting! The final results should be close to 70Vol% random and 30Vol% [100]textured components!

Measuring Cell distortions

In case of observed cell distortion (peaks of one phase are shifted) first the rough shift must be taken into account by creating a virtual phase (manual selection of cell parameter that produces Marker lines roughly coinciding with the measured peaks). Use this phase (and others) to roughly fit the measured distribution (see Quantitative phase analysis: random and textured components, dynamic effects and temperature factors)! As a last step of fitting, release the cell parameter of the selected phase for fine tuning!



Fit Options Analysis type / Cell parameters

Parameter selection in phase analysis

In order to help the program find the global minimum, parameters must be selected as close to the true value as possible. This selection is approached step-by-step. The following sequence generally helps:

4. Calibrate camera length!
5. Calibrate peak shape if a separate good peak is available.
6. Check if significant cell parameter change is present for one or more phases! If so, it shifts the corresponding peaks from the calibrated value. If so, generate a new structure with the roughly estimated cell parameters and use them when starting the fit!
7. Determine (during the experiment) if texture is present. If so, try to roughly fit textured components!
8. Refine longitudinal grain size (=dimension along beam direction) by activating dynamic Markers in Fit Options.
9. Refine temperature factors (Fit Options).
10. Release parameters of the peak shape(s) for fine tuning!
11. If no cell parameter change is present, release camera length for fine tuning. If the camera length was calibrated, but the cell parameter was changed, release the value of the cell parameter for fine tuning in this step. These are alternatives. Camera length and cell parameters are NOT to be refined simultaneously!

Polar coordinates and sectors

The aim of this function is to evaluate different sectors of the 2D pattern separately to see if there is any deviation from perfect rotational symmetry of the rings. You should start with converting the measured pattern into polar coordinates (only in the computer memory; it is not saved, since 2D patterns can be huge with no benefit in duplicating huge storage space). Select menu ***Process / Re-Map to Polar coordinates*** in the ***Intensity*** window! This gives the basis for the next two optional actions:

Select menu ***Process / Show sector as distribution*** in the ***Intensity*** window! The distribution is only calculated from the selected angular sector of the pattern and the legend will include the angular range (e.g. [0-30]) in degree units.

To check visually which part of the pattern was included select menu ***Process / Color the sector on the pattern*** in the ***Intensity*** window! The appropriate part of the pattern will be colored in the ***SAED*** window.

In future versions more functions will be based on polar coordinate representation.

Short range order: ePDF analysis

At the moment this function is only available to close cooperating partners. It may change in future.

Additional tools

A suit of crystallographic calculations

For a give structure useful data are calculated. Use the menu ***Structure / Use the Crystallographic Calculator***! The new ***Crystallographic Calculator*** window appears. Load a structure from menu ***File / Load Structure***! Specify the indices of planes or direction you want to involve in a calculation and observe the effect!

Angle between planes

Angle between directions

Conversion between 3-index and 4-index description

Direction and plane, normal to each other

List Zones at an angular interval from a direction

List Zones that lay in a plane

Solve a single crystal pattern manually or automatically

Also need the d-values of two measured spots and the angle between them. Can be specified either manually or taken from the ***Cursor*** window (see Indexing single crystal patterns).

Calculate angle between two goniometer settings

Distribution Math

Select the operation first. That will update the list of available input items (one or two lists, depending on the operation) and also the list of possible targets for the results.

Scale a distribution

Reduce a distribution by a constant

Add two distributions

Subtract two distributions

Multiply two distributions

Divide two distributions

Fit a polynomial to a distribution

Fit a spline to manually selected points in a distribution

Convolve two distributions

Calculate the Fast Fourier Transform (FFT) of a distribution

Integrate a distribution

What is new in Version 8

- Parts of the measured pattern can be “Masked”, i.e. excluded from the evaluation. It that way pixels can be excluded that show photo numbering, covered by the Beam-stop or by single-crystal spots from the substrate, while you only want to process the intensities of the rings from the layer. The masked image is saved separately and rendering can be alternated between original and masked pattern. The Mask itself is stored in the Project-file and can be saved in an individual Mask file from which it can be reloaded for another pattern with almost identical geometry (e.g. exposure series).
- Standard deviation is calculated for the ring-averaged measured quantities (and those that are deduced from them, like Net counts, etc.). Those standard deviations are used as weights during fitting models to the measured distribution in both “Decomposition” and “Phase analysis”.
- The program helps locating files if you happened to move the project and its component to a new drive/directory, or simply renamed the directory. Since the locations and file names are stored in the project it caused a problem in previous versions of ProcessDiffraction.
- Selection between "R-patterns" and "R-weighted pattern" is provided in the new version as minimization criteria. (See “The Rietveld method”, Young R.A. ed., IUCr Monographs on Crystallography 5.) A bug is removed from examination of convergence criteria, which reduced the probability of finding the global minimum (instead of local minima) in quantitative decomposition and phase analysis.
- Merging (used to extend dynamic range by merging an exposure series) is restructured for simpler unambiguous operation.
- New possibility is to convert the measured pattern into Polar coordinates and evaluate angular sectors separately.
- For the request of a user, a new format (*.chi) is introduced for saving distribution data.

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