PF

PeakFit – Analysis of BT8/X-ray Diffraction Data

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Introduction

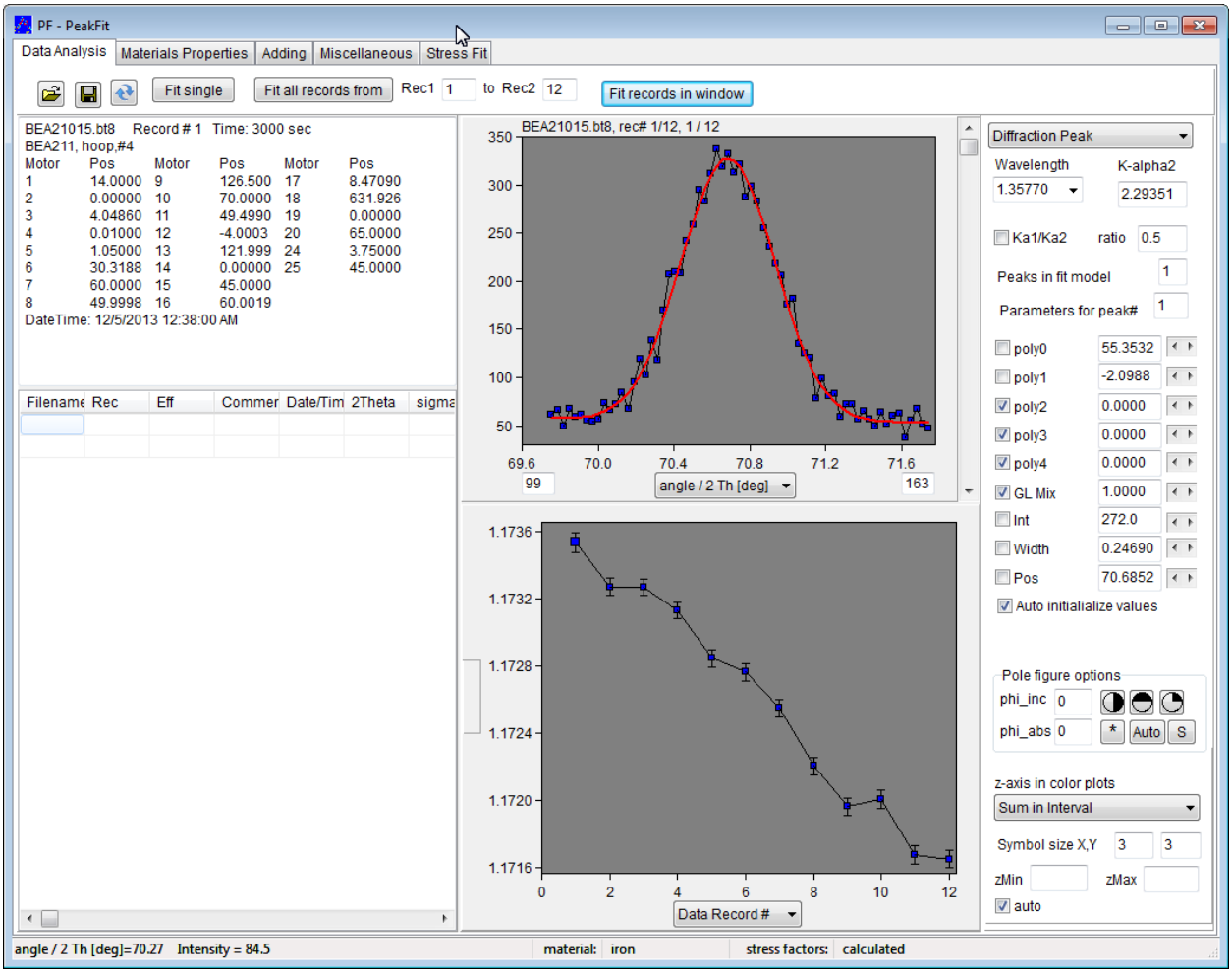
1. Purpose

The purpose of PF is the analysis (the fitting of peaks) and the calculation of stresses from the peak fit results. The stress calculation can also be done with imported values that were entered manually or copied from clipboard. PF is designed to make this as easy as possible by automating many of the time consuming sorting and bookkeeping tasks that stem from processing vast amounts of data. Other uses include pole figure analysis and wall scans (entering curves).

2. Quick Start

The fastest way to process the data is as follows:

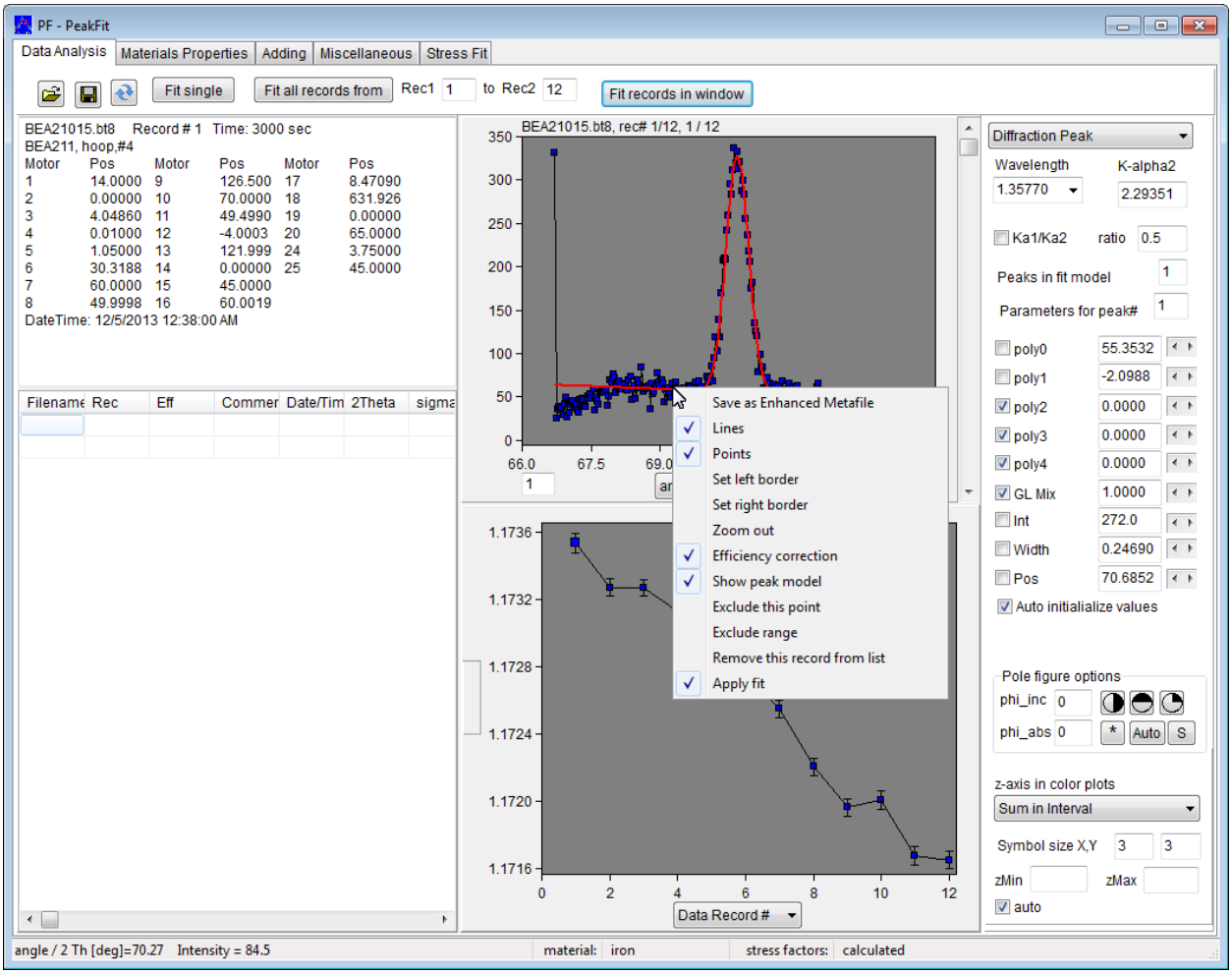
1. Load a measurement file (either BT8 or X-ray format) using the File Open button .



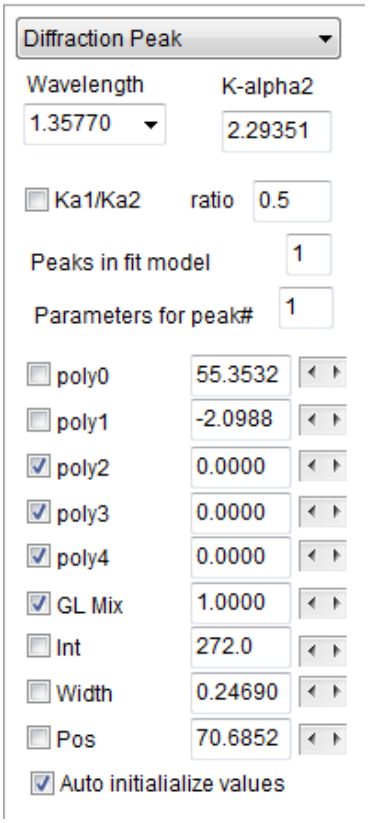
After loading one or more file(s) the measured diffraction peaks can be inspected (upper graphics window). The scrollbar on the right side of the window allows scrolling through all loaded measurements (records) for visual inspection. Blue symbols are the measured data, the red line is either the initial estimate before the peakfit or the fitted peak function after completing the peakfit. The lower graphics window allows the second stage analysis by plotting quantities such as Sum in Interval, d-spacing, FWHM (both after fitting the peaks) and so on vs. data record # (when opening the files, each measurement record is assigned a number), motor positions, $\sin^2\psi$ or other. The upper text window presents all information available from reading the file for the particular measurement on display in the upper graphics window.

Preparing for Fit

It is recommended to narrow the range for the peak fit in the upper window by **right clicking** at the x-value desired for the left (click left of peak) or right (click right side of peak) border:



Initial estimates for the fit can be adjusted manually or determined automatically:



←choose between diffraction peak fit and entering curves

←wavelength is determined automatically for neutron diffraction

←Doublet is relevant for X-rays

←for fitting multiple peaks

←peak for which parameters are shown below

←background, up to 4th order polynomial

← mix parameter using [m × Gaussian] + (1-m) × Lorentzian
← intensity
←width(sigma) as defined in Gaussian

←position
← initialize fit parameters automatically

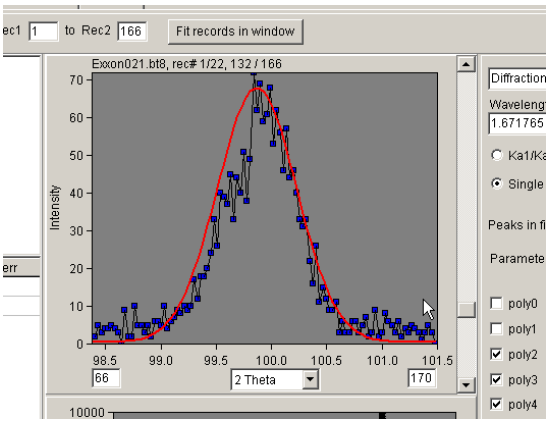
A fit can be performed either by fitting just the peak in the upper graphics window **(Fit Single**), the data sets starting from (**Fit all records from**) Rec1 to Rec2, or all records visible in the lower graphics window **(Fit records in window**). The latter is useful if the data sets shown there have been narrowed down using a **right click** in the lower window and subsequent setting **left border/right border/ upper border or lower border**.

The complete fit results are written into the lower text window. Though not needed in most cases, they can be save either by clicking the save button at the top or by manually copying (CRTL-C) them into EXCEL or some other program. For stress calculations, a condensed summary of the current fit (records fitted after the most recent click on one of the fit buttons) can be loaded and saved on the ‘Stress Fit’ tab by clicking the ‘Load’ button.

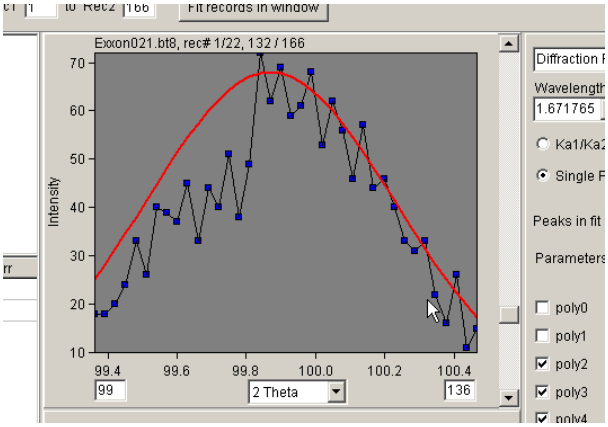
Entering Curves (EC)

Entering curves are measured by moving the specimen stepwise into the gage volume (whose position is fixed in space). By fitting an EC for a particular entering geometry (reflection geometry, transmission and so on) the position of the sample surface (in terms of motor coordinates) can be determined. The position is the motor value where half the gage volume is immersed in the sample.
Data for EC are usually intensity data. It is recommended to define the window rather narrow to reduce the background for the EC:

Good:

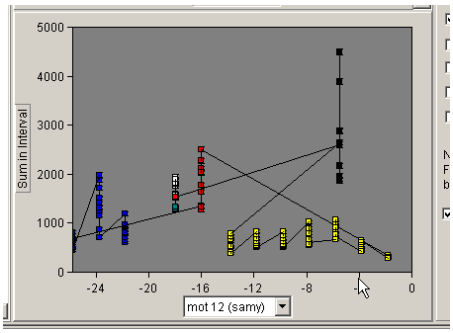


Better:



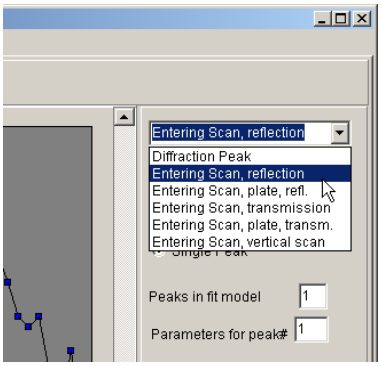
The sum of all counts in the upper window should be displayed in the lower window by clicking on the lower y-axis title bar:

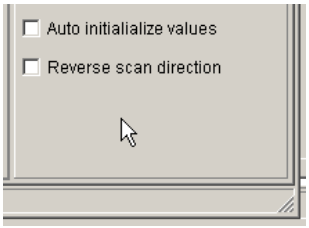
Title bar➔



The actual scan-motor should be chosen as x-axis. Next, double click the lower graphics window. This activates fitting for data displayed here.

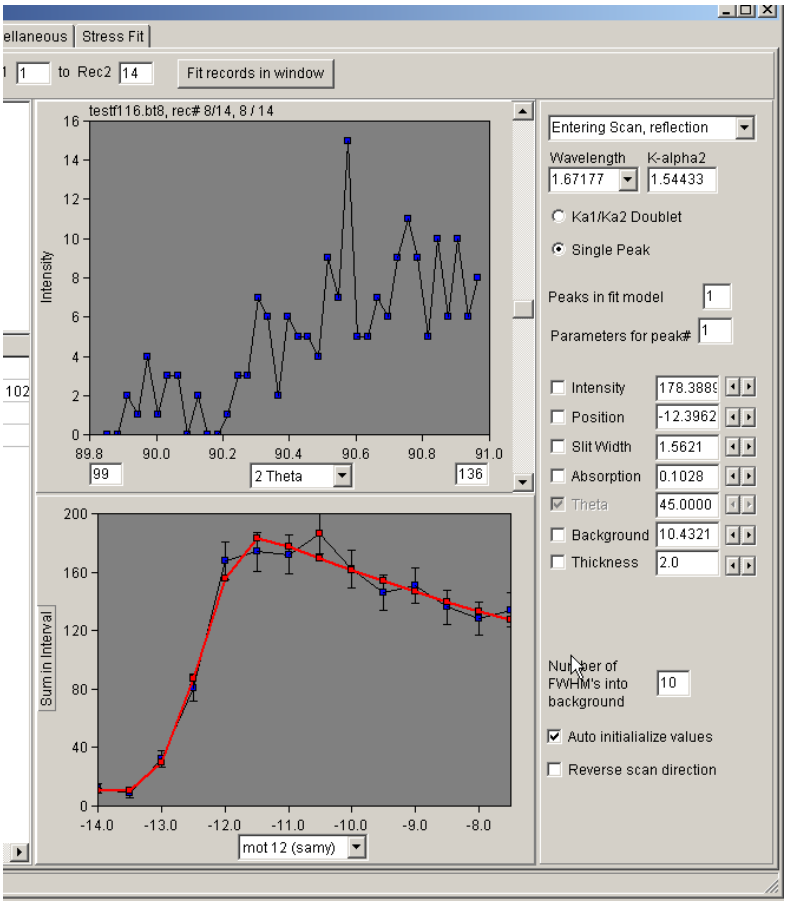
Then choose the appropriate model from the fit function list in the upper right corner.





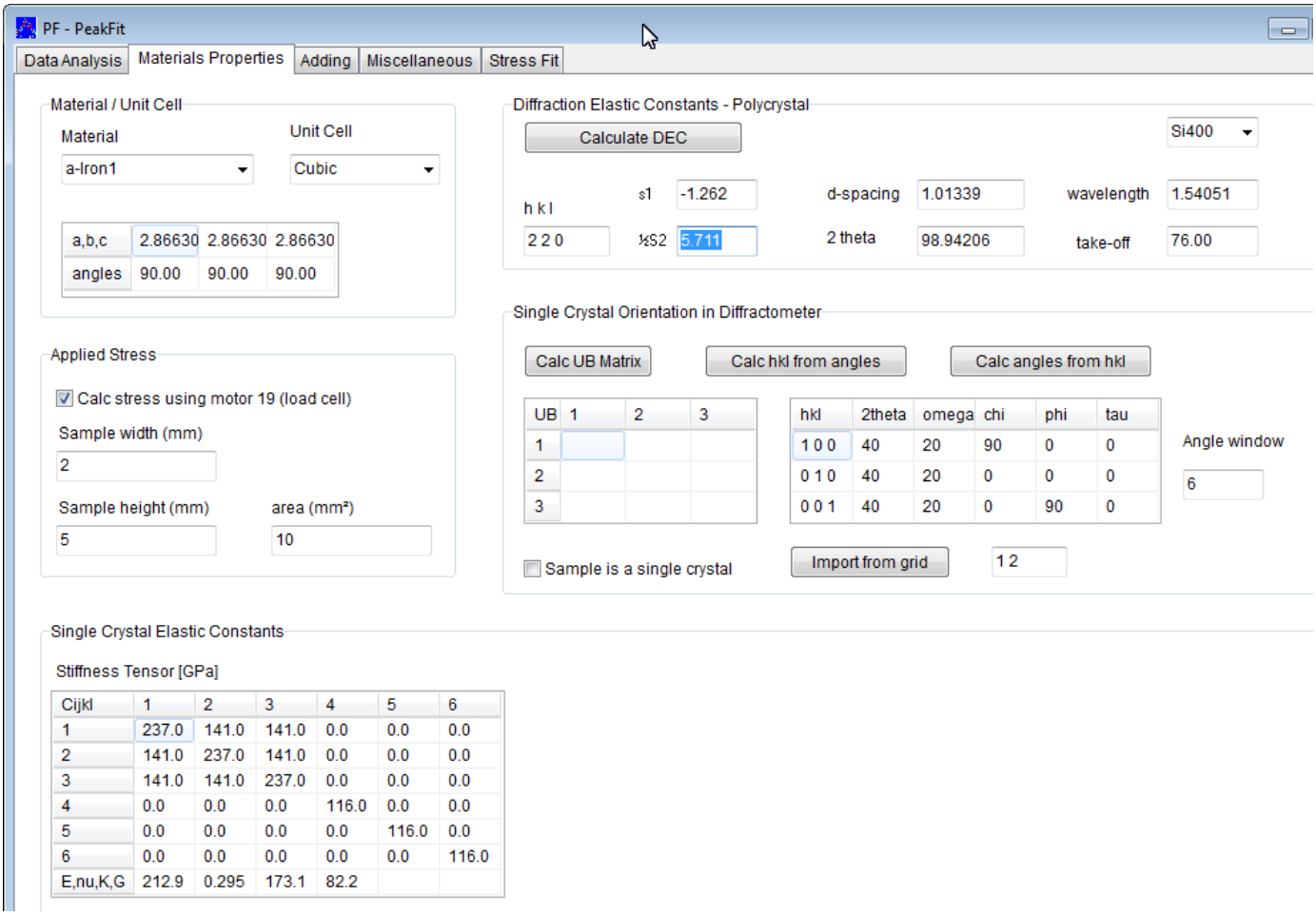
In most cases, '**Auto initialize values**' will give good results. If this is not the case then uncheck this box and adjust values manually before the fit. Also, set the correct scan direction through '**Reverse scan direction**'.

An example for an EC in reflection (psi=0) is shown here:



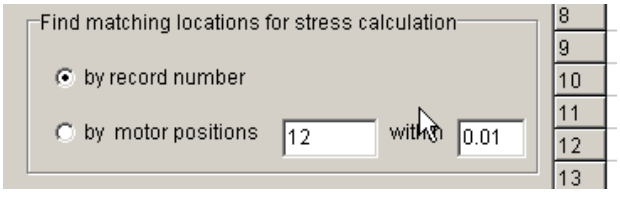
Stress Fit

The first step should be selecting the elastic constants in the ‘Materials Properties’ tab. For common engineering materials (isotropic polycrystals), only the selection of the material (top left) and entering the {hkl} are necessary to calculate the DEC. Further options are available for working on single crystals. Note that the isotropic DEC s_1 and $½s_2$ do not appear in the stress-worksheet (see next paragraph). Instead they are used to calculate the stress factors F_{11}, F_{22}, …

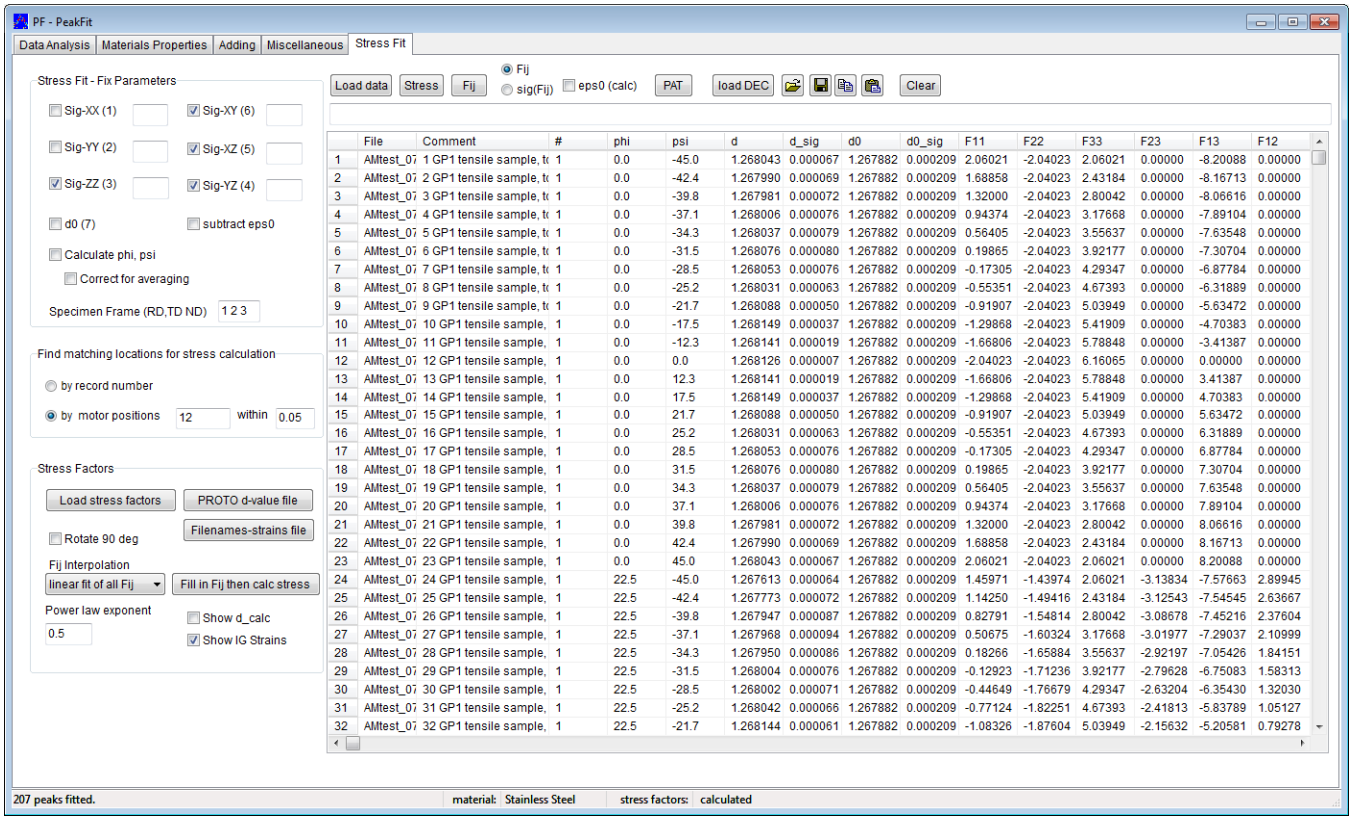


Next, go to the ‘**Stress Fit**’ tab and click ‘**Load**’. The latter loads the latest fit results in the stress calc worksheet. Before actually calculating stresses several conditions need to be set:

1. PF needs to be told which data belong together, i.e. which of the measurements (usually in different sample directions) were done at the same sample coordinates to be used for stress tensor calculation at this particular coordinate. This is done either by record number or by matching motor positions:



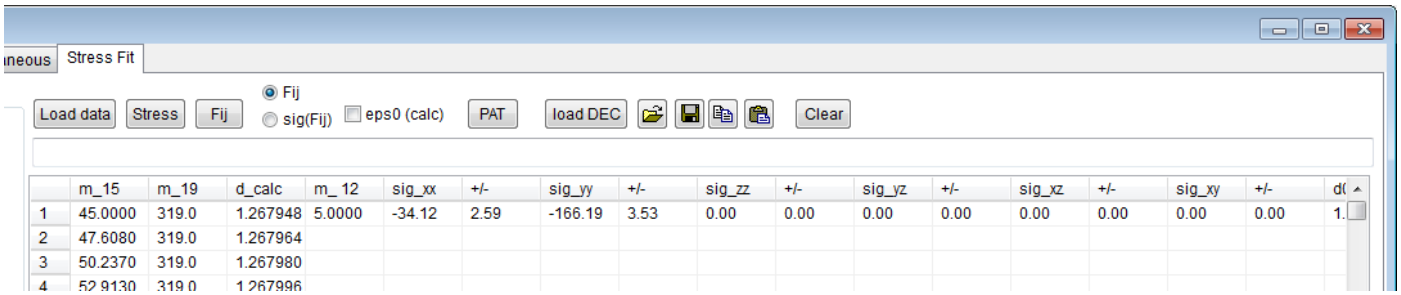
The latter is typically only useful for X-rays where no sample re-mounting is required.
Matching record numbers has to be done manually as shown in this example:



In this example, three series of measurements were done, each series has seven measurements at different coordinates as expressed in values for motor 11, 12, 13 (columns m_11, m_12, m_13). Matching the records is done by manually editing the 3rd column '#' through giving matching measurements the same number.

2. Each of the records needs to be given its correct values for **phi** and **psi** according to the convention X➔(phi=0, psi=90), Y➔(phi=90, psi=90) and Z➔(phi=0, psi=0). Usually, this has to be done manually as well. Intermediate directions will turn on shear components.
3. Enter the correct value for **d0** and its uncertainty **d_sig** (cannot be zero!!). This is required only if d0 is a fixed parameter in the stress calculation (d0 box checked).
4. Fix/Free individual stress components. For example, for thin specimen it can be reasonably assumed that sig-33=0 and this component can be fixed to zero which then allows to have d0 as a fit parameter if sufficiently many measurements are available (equal or more than the number of unknowns).

After clicking '**Stress'** the results are listed in the worksheet (scroll to the right).



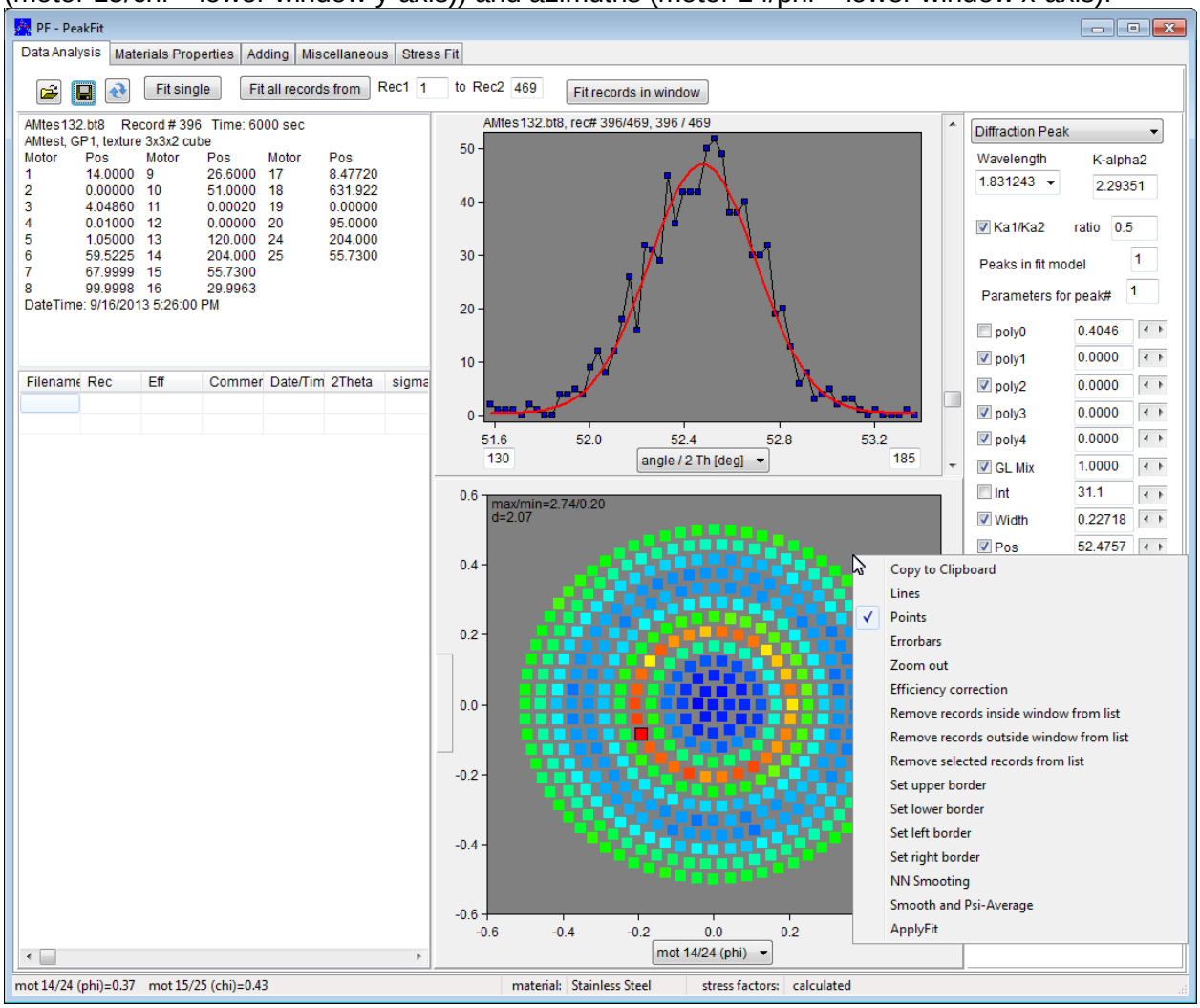
They can be saved through the save button or manually copied into EXCEL.

Recommendations

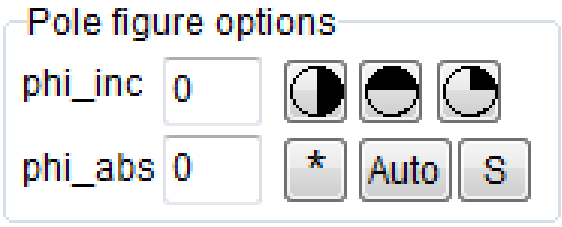
The subject of using d0 values is a tricky one because, strictly speaking, for the d0 values to be truly correct, one needs to dice up each specimen after the actual measurement and extract little coupons from exactly those locations where the neutron measurements were done. As long-range stresses are now released, a ‘stress-free’ d0 can now be measured. As this is often not possible, reference samples are used which may not be stress free. Also, the actual specimens usually have a different thermo-mechanical history which may well have changed the true d0 compared to the d0-ref from the reference sample. Also, each d0 measurement comes with its own uncertainty, thus increasing the uncertainty on the stresses.
An alternative is the use of fixed stresses, for X-rays usually the sig-33 normal stresses which for a single phase material are zero at the surface. With a fixed sig_33=0, d0 can actually be calculated! Using this condition for the bulk of the material may not be strictly valid but it should be a good assumption for thin samples, thus eliminating the need for d0 coupon extraction and d0 measurements.

Analysis of Pole Figure Data

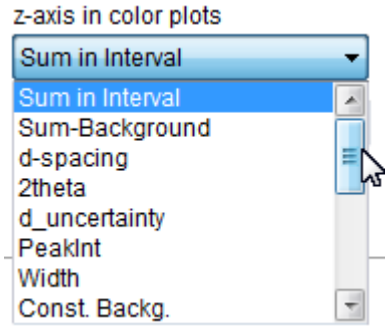
Pole figure files are measurements of intensities collected while the sample was oriented at different tilts (motor 15/chi – lower window y-axis)) and azimuths (motor 14/phi – lower window x-axis).



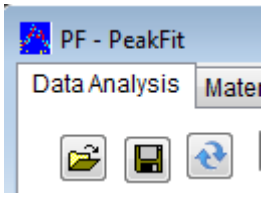
The graphical representation can be copied to clipboard by right-clicking on it, and selecting “copy to clipboard”.
If there is only one peak captured in the detector then clicking the “Auto” button will do the background correction. Otherwise, the region for the peak of interest must be selected in the upper window and all records must be fitted with constant background, preferrably with a fixed peak position and width. Pole figures can be rotated in phi and various symmetries (RD-mirror, TD-mirror, orthorhombic, phi-rotational symmetry) can be enforced through the pole figure menu operations. Clicking the button ‘S’ will perform a smoothing.



The pole figure display mode can be changed to display various other peak fit-derived quantities:



After finishing background correction, rotation or similar, pole figure data can be saved by clicking the disk button:



The “save” menu offers three file formats for saving:
1. SUM-file: four columns of data (phi, psi, total counts, background)
2. Popla-format EPF-file containing normalized intensities.
3. Popla format uncertainty pole figure containing the standard deviations of the intensities.