

PF

PeakFit – Analysis of BT8/X-ray Diffraction Data

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Introduction

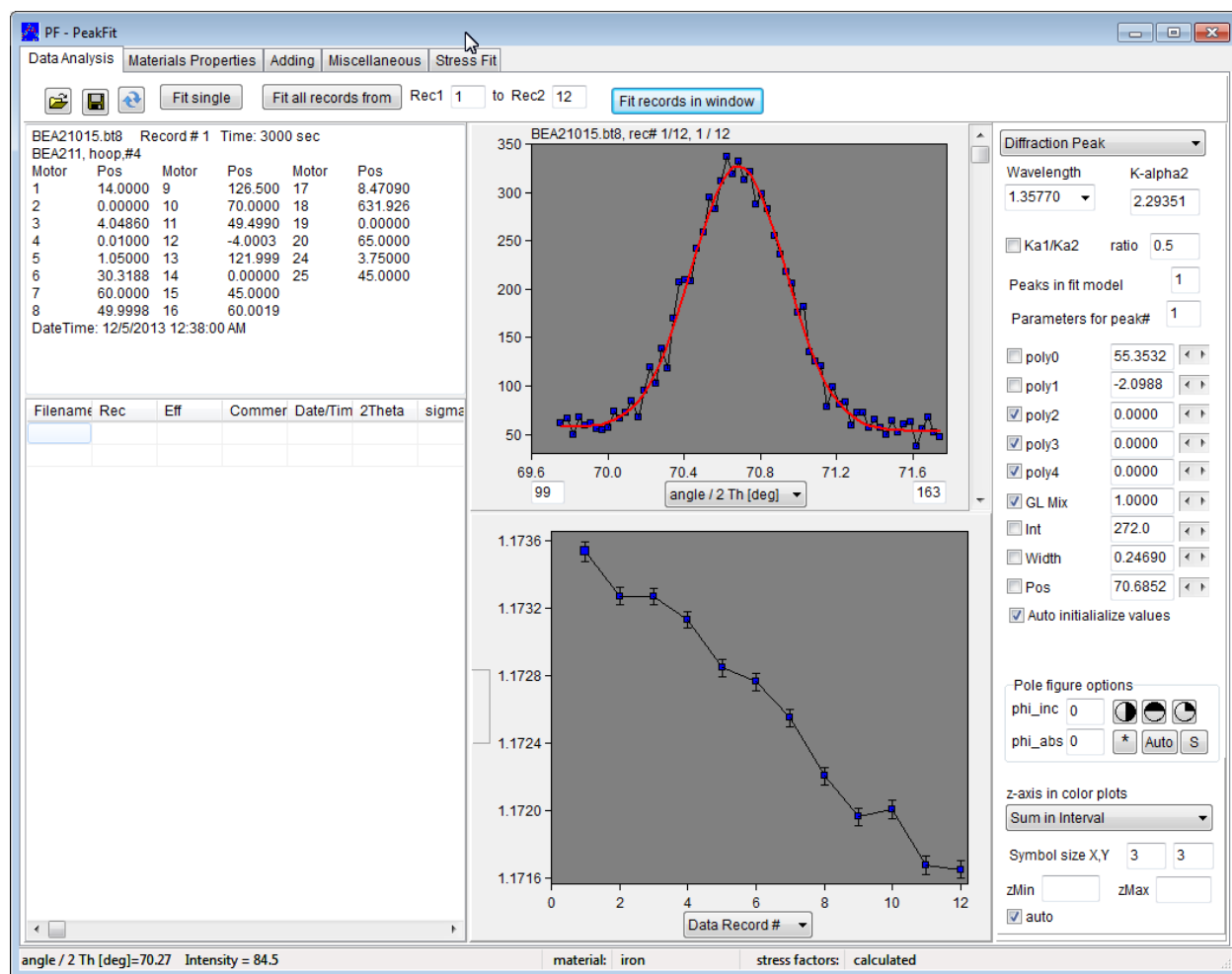
1. Purpose

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:

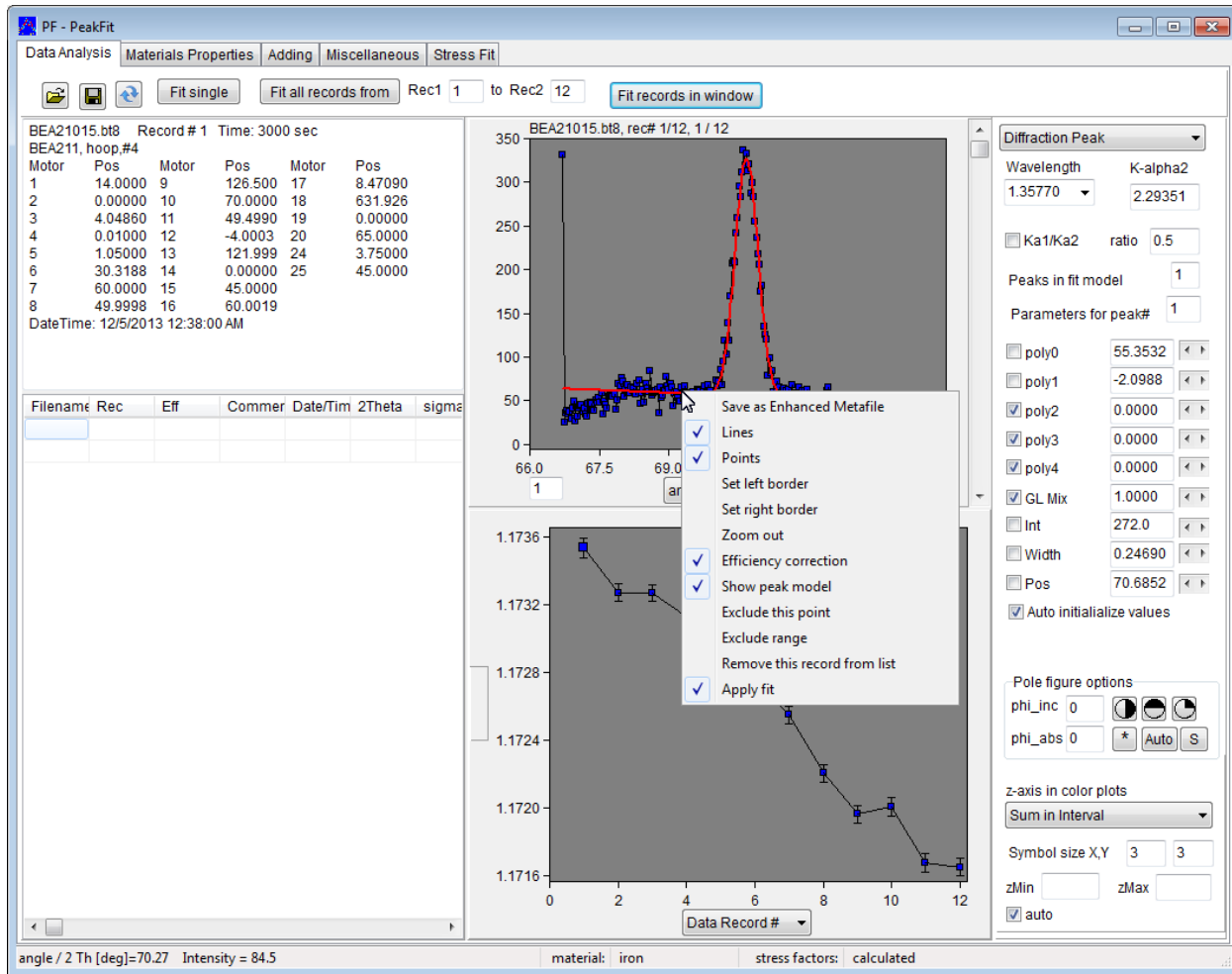
1. Load a measurement file (either BT8 or X-ray format) using the [File Open button](#) .



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:

<input type="button" value="Diffraction Peak"/>		← choose between diffraction peak fit and entering curves
Wavelength	K-alpha2	
<input type="button" value="1.35770"/>	<input type="button" value="2.29351"/>	← wavelength is determined automatically for neutron diffraction
<input type="checkbox"/> Ka1/Ka2	ratio <input type="button" value="0.5"/>	← Doublet is relevant for X-rays
Peaks in fit model	<input type="button" value="1"/>	← for fitting multiple peaks
Parameters for peak#	<input type="button" value="1"/>	← peak for which parameters are shown below
<input type="checkbox"/> poly0	<input type="button" value="55.3532"/>	← background, up to 4 th order polynomial
<input type="checkbox"/> poly1	<input type="button" value="-2.0988"/>	
<input checked="" type="checkbox"/> poly2	<input type="button" value="0.0000"/>	
<input checked="" type="checkbox"/> poly3	<input type="button" value="0.0000"/>	
<input checked="" type="checkbox"/> poly4	<input type="button" value="0.0000"/>	
<input checked="" type="checkbox"/> GL Mix	<input type="button" value="1.0000"/>	← mix parameter using $[m \times \text{Gaussian}] + (1-m) \times \text{Lorentzian}$
<input type="checkbox"/> Int	<input type="button" value="272.0"/>	← intensity
<input type="checkbox"/> Width	<input type="button" value="0.24690"/>	← width(sigma) as defined in Gaussian
<input type="checkbox"/> Pos	<input type="button" value="70.6852"/>	← position
<input checked="" type="checkbox"/> Auto initialize values		← 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**.

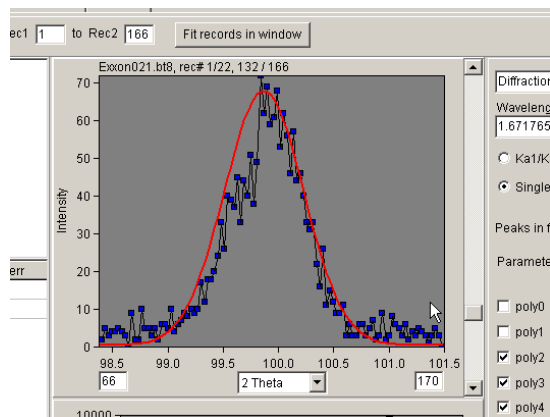
The complete fit results are written into the lower text window. Though not needed in most cases, they can be save either by clicking the save button at the top or by manually copying (CTRL-C) them into EXCEL or some other program. For stress calculations, a condensed summary of the current fit (records 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)

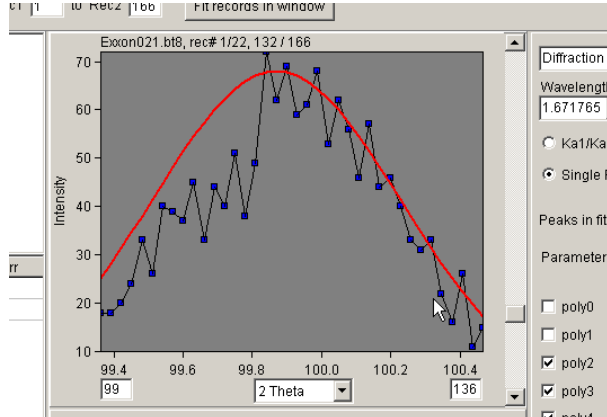
Entering curves are measured by moving the specimen stepwise into the gage volume (whose position is fixed in space). By fitting an EC for a particular entering geometry (reflection geometry, transmission and so on) the position of the sample surface (in terms of motor coordinates) can be determined. 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:

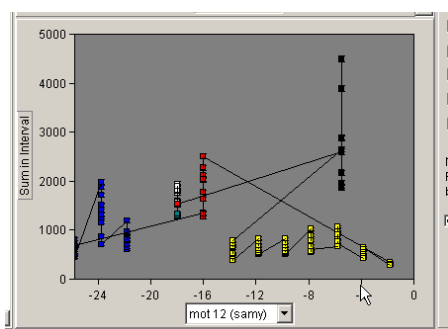


Better:



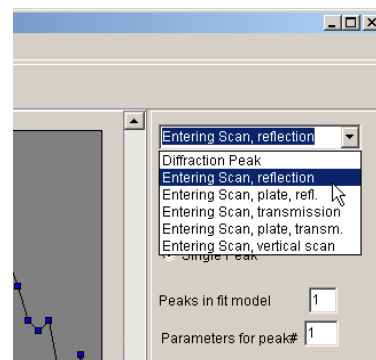
The sum of all counts in the upper window should be displayed in the lower window by clicking on the lower y-axis title bar:

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.

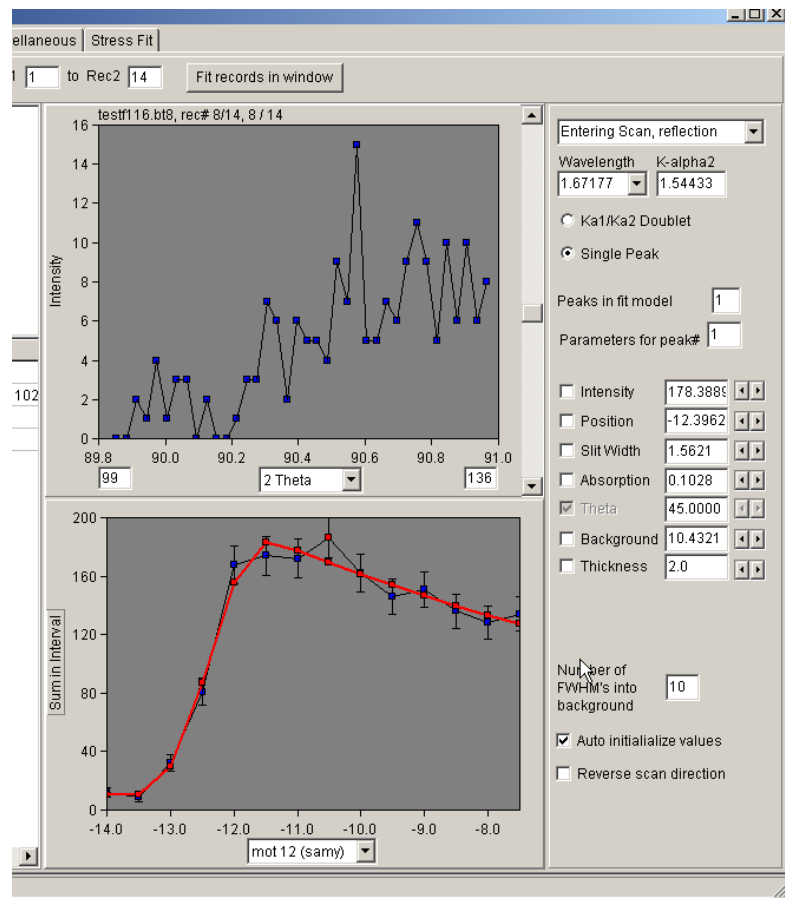


☐ Auto initialize values

☐ Reverse scan direction

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 $\frac{1}{2}s_2$ do not appear in the stress-worksheet (see next paragraph). Instead they are used to calculate the stress factors F_{11} , F_{22} , ...

Material / Unit Cell

Material: a-Iron1 Unit Cell: Cubic

a,b,c	2.86630	2.86630	2.86630
angles	90.00	90.00	90.00

Diffraction Elastic Constants - Polycrystal

Calculate DEC

h k l: 2 2 0 s_1 : -1.262 d-spacing: 1.01339 wavelength: 1.54051 $\frac{1}{2}s_2$: 5.711 2 theta: 98.94206 take-off: 76.00

Applied Stress

☒ Calc stress using motor 19 (load cell)

Sample width (mm): 2

Sample height (mm): 5 area (mm²): 10

Single Crystal Orientation in Diffractometer

Calc UB Matrix Calc hkl from angles Calc angles from hkl

UB	1	2	3
1			
2			
3			

hkl	2theta	omega	chi	phi	tau
1 0 0	40	20	90	0	0
0 1 0	40	20	0	0	0
0 0 1	40	20	0	90	0

☐ Sample is a single crystal Import from grid 1 2

Single Crystal Elastic Constants

Stiffness Tensor [GPa]

Cijkl	1	2	3	4	5	6
1	237.0	141.0	141.0	0.0	0.0	0.0
2	141.0	237.0	141.0	0.0	0.0	0.0
3	141.0	141.0	237.0	0.0	0.0	0.0
4	0.0	0.0	0.0	116.0	0.0	0.0
5	0.0	0.0	0.0	0.0	116.0	0.0
6	0.0	0.0	0.0	0.0	0.0	116.0
E,nu,K,G	212.9	0.295	173.1	82.2		

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:

Find matching locations for stress calculation

☒ by record number

☐ by motor positions 12 within 0.01

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:

PF - PeakFit

Data Analysis | Materials Properties | Adding | Miscellaneous | Stress Fit

Stress Fit - Fix Parameters

☐ Sig-XX (1) ☒ Sig-XY (6) ☐ Sig-YY (2) ☒ Sig-XZ (5) ☒ Sig-ZZ (3) ☒ Sig-YZ (4)

☐ d0 (7) ☐ subtract eps0

☐ Calculate phi, psi

☐ Correct for averaging

Specimen Frame (RD,TD,ND) 1 2 3

Find matching locations for stress calculation

☐ by record number

☒ by motor positions 12 within 0.05

Stress Factors

Load stress factors PROTO d-value file

Rotate 90 deg

Fij Interpolation

linear fit of all Fij Fill in Fij then calc stress

Power law exponent 0.5

Show d_calc

Show IG Strains

File	Comment	#	phi	psi	d	d_sig	d0	d0_sig	F11	F22	F33	F23	F13	F12
1	AMTest_07 1 GP1 tensile sample, tr	1	0.0	-45.0	1.268043	0.000067	1.267882	0.000209	2.06021	-2.04023	2.06021	0.00000	-8.20088	0.00000
2	AMTest_07 2 GP1 tensile sample, tr	1	0.0	-42.4	1.267990	0.000069	1.267882	0.000209	1.68858	-2.04023	2.43184	0.00000	-8.16713	0.00000
3	AMTest_07 3 GP1 tensile sample, tr	1	0.0	-39.8	1.267981	0.000072	1.267882	0.000209	1.32000	-2.04023	2.80042	0.00000	-8.06616	0.00000
4	AMTest_07 4 GP1 tensile sample, tr	1	0.0	-37.1	1.268006	0.000076	1.267882	0.000209	0.94374	-2.04023	3.17668	0.00000	-7.89104	0.00000
5	AMTest_07 5 GP1 tensile sample, tr	1	0.0	-34.3	1.268037	0.000079	1.267882	0.000209	0.56405	-2.04023	3.55637	0.00000	-7.63548	0.00000
6	AMTest_07 6 GP1 tensile sample, tr	1	0.0	-31.5	1.268076	0.000080	1.267882	0.000209	0.19865	-2.04023	3.92177	0.00000	-7.30704	0.00000
7	AMTest_07 7 GP1 tensile sample, tr	1	0.0	-28.5	1.268053	0.000076	1.267882	0.000209	-0.17305	-2.04023	4.29347	0.00000	-6.87784	0.00000
8	AMTest_07 8 GP1 tensile sample, tr	1	0.0	-25.2	1.268031	0.000063	1.267882	0.000209	-0.55351	-2.04023	4.67393	0.00000	-6.31889	0.00000
9	AMTest_07 9 GP1 tensile sample, tr	1	0.0	-21.7	1.268088	0.000050	1.267882	0.000209	-0.91907	-2.04023	5.03949	0.00000	-5.63472	0.00000
10	AMTest_07 10 GP1 tensile sample, tr	1	0.0	-17.5	1.268149	0.000037	1.267882	0.000209	-1.29868	-2.04023	5.41909	0.00000	-4.70383	0.00000
11	AMTest_07 11 GP1 tensile sample, tr	1	0.0	-12.3	1.268141	0.000019	1.267882	0.000209	-1.66806	-2.04023	5.78848	0.00000	-3.41387	0.00000
12	AMTest_07 12 GP1 tensile sample, tr	1	0.0	0.0	1.268126	0.000007	1.267882	0.000209	-2.04023	-2.04023	6.16065	0.00000	0.00000	0.00000
13	AMTest_07 13 GP1 tensile sample, tr	1	0.0	12.3	1.268141	0.000019	1.267882	0.000209	-1.66806	-2.04023	5.78848	0.00000	3.41387	0.00000
14	AMTest_07 14 GP1 tensile sample, tr	1	0.0	17.5	1.268149	0.000037	1.267882	0.000209	-1.29868	-2.04023	5.41909	0.00000	4.70383	0.00000
15	AMTest_07 15 GP1 tensile sample, tr	1	0.0	21.7	1.268088	0.000050	1.267882	0.000209	-0.91907	-2.04023	5.03949	0.00000	5.63472	0.00000
16	AMTest_07 16 GP1 tensile sample, tr	1	0.0	25.2	1.268031	0.000063	1.267882	0.000209	-0.55351	-2.04023	4.67393	0.00000	6.31889	0.00000
17	AMTest_07 17 GP1 tensile sample, tr	1	0.0	28.5	1.268053	0.000076	1.267882	0.000209	-0.17305	-2.04023	4.29347	0.00000	6.87784	0.00000
18	AMTest_07 18 GP1 tensile sample, tr	1	0.0	31.5	1.268076	0.000080	1.267882	0.000209	0.19865	-2.04023	3.92177	0.00000	7.30704	0.00000
19	AMTest_07 19 GP1 tensile sample, tr	1	0.0	34.3	1.268037	0.000079	1.267882	0.000209	0.56405	-2.04023	3.55637	0.00000	7.63548	0.00000
20	AMTest_07 20 GP1 tensile sample, tr	1	0.0	37.1	1.268006	0.000076	1.267882	0.000209	0.94374	-2.04023	3.17668	0.00000	7.89104	0.00000
21	AMTest_07 21 GP1 tensile sample, tr	1	0.0	39.8	1.267981	0.000072	1.267882	0.000209	1.32000	-2.04023	2.80042	0.00000	8.06616	0.00000
22	AMTest_07 22 GP1 tensile sample, tr	1	0.0	42.4	1.267990	0.000069	1.267882	0.000209	1.68858	-2.04023	2.43184	0.00000	8.16713	0.00000
23	AMTest_07 23 GP1 tensile sample, tr	1	0.0	45.0	1.268043	0.000067	1.267882	0.000209	2.06021	-2.04023	2.06021	0.00000	8.20088	0.00000
24	AMTest_07 24 GP1 tensile sample, tr	1	22.5	-45.0	1.267613	0.000064	1.267882	0.000209	1.45971	-1.43974	2.06021	-3.13834	-7.57663	2.89945
25	AMTest_07 25 GP1 tensile sample, tr	1	22.5	-42.4	1.267773	0.000072	1.267882	0.000209	1.14250	-1.49416	2.43184	-3.12543	-7.54545	2.63667
26	AMTest_07 26 GP1 tensile sample, tr	1	22.5	-39.8	1.267947	0.000087	1.267882	0.000209	0.82791	-1.54814	2.80042	-3.08678	-7.45216	2.37604
27	AMTest_07 27 GP1 tensile sample, tr	1	22.5	-37.1	1.267968	0.000094	1.267882	0.000209	0.50675	-1.60324	3.17668	-3.01977	-7.29037	2.10999
28	AMTest_07 28 GP1 tensile sample, tr	1	22.5	-34.3	1.267950	0.000086	1.267882	0.000209	0.18266	-1.65884	3.55637	-2.92197	-7.05426	1.84151
29	AMTest_07 29 GP1 tensile sample, tr	1	22.5	-31.5	1.268004	0.000076	1.267882	0.000209	-0.12923	-1.71236	3.92177	-2.79628	-6.75083	1.58313
30	AMTest_07 30 GP1 tensile sample, tr	1	22.5	-28.5	1.268002	0.000071	1.267882	0.000209	-0.44649	-1.76679	4.29347	-2.63204	-6.35430	1.32030
31	AMTest_07 31 GP1 tensile sample, tr	1	22.5	-25.2	1.268042	0.000066	1.267882	0.000209	-0.77124	-1.82251	4.67393	-2.41813	-5.83789	1.05127
32	AMTest_07 32 GP1 tensile sample, tr	1	22.5	-21.7	1.268144	0.000061	1.267882	0.000209	-1.08326	-1.87604	5.03949	-2.15632	-5.20581	0.79278

207 peaks fitted. material: Stainless Steel stress factors: calculated

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.

- Each of the records needs to be given its correct values for **phi** and **psi** according to the convention $X \rightarrow (\phi=0, \psi=90)$, $Y \rightarrow (\phi=90, \psi=90)$ and $Z \rightarrow (\phi=0, \psi=0)$. Usually, this has to be done manually as well. Intermediate directions will turn on shear components.
- 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).
- Fix/Free individual stress components. For example, for thin specimen it can be reasonably assumed that $\text{sig-33}=0$ 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).

Stress Fit

Load data Stress Fij sig(Fij) eps0 (calc) PAT load DEC Clear

	m_15	m_19	d_calc	m_12	sig_xx	+/-	sig_yy	+/-	sig_zz	+/-	sig_yz	+/-	sig_xz	+/-	sig_xy	+/-	d
1	45.0000	319.0	1.267948	5.0000	-34.12	2.59	-166.19	3.53	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.
2	47.6080	319.0	1.267964														
3	50.2370	319.0	1.267980														
4	52.9130	319.0	1.267996														

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

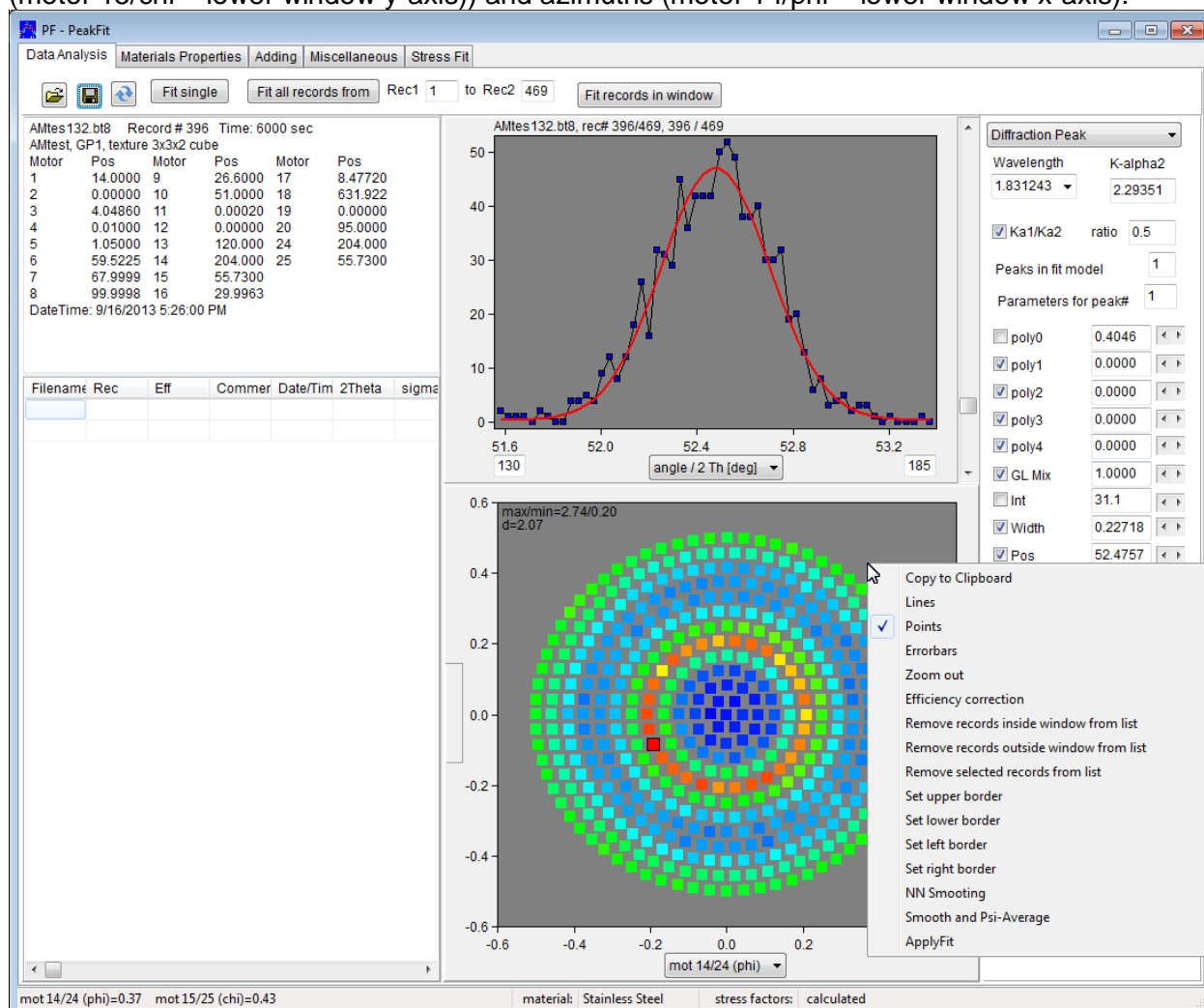
Recommendations

The subject of using d_0 values is a tricky one because, strictly speaking, for the d_0 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' d_0 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 d_0 compared to the d_0 -ref from the reference sample. Also, each d_0 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 sig₃₃=0, d_0 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 d_0 coupon extraction and d_0 measurements.

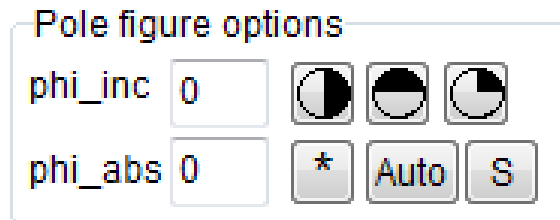
Analysis of Pole Figure Data

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).

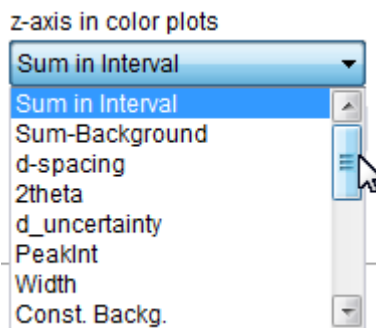


The graphical representation can be copied to clipboard by right-clicking on it, and selecting “copy to clipboard”.

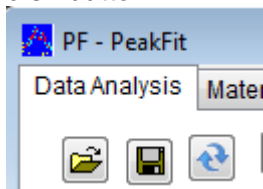
If there is only one peak captured in the detector then clicking the “Auto” button will do the background correction. Otherwise, the region for the peak of interest must be selected in the upper window and all records must be fitted with constant background, preferably with a fixed peak position and 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.

