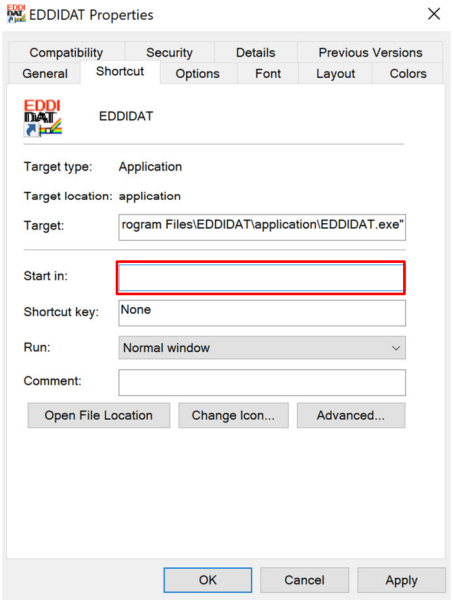
EDDIDAT User Manual V1

Contents

1. Installation instructions

The program EDDIDAT is installed by executing the EDDIDAT_Installer.exe. You will be prompted to install Matlab Runtime. This is necessary in order to be able to use the program. However, Matlab itself is not needed.

You can choose the path to install the program to but be sure you have administrator rights in this folder. It is recommended not to select the drive where Windows is installed (usually C:\). During the installation routine you are given the choice to create a desktop shortcut. Unfortunately, the desktop shortcut created does not work since it is missing the proper link to the installation folder ("Start in" is empty). You can change the entry for "Start in" manually ("YourPath\HZB\EDDIDAT\application\") or you can simply create a new desktop shortcut manually from within the installation directory.



The main folders are found in

"YourPath\EDDIDAT\application\Data".

Here, you can manage the measurements and results. Your measurements need to be copied to the folder "Measurements". The results are saved to the folder "Results". For each analysis, a separate folder is created, named after the filename of the measurement and the current date. You can save your analysis in the folder "GUIData".

2. Introduction

Before you analyze your measurements, make sure that the measurement file is free of “artefacts”, such as test scans, aborted scans, empty scans, additional commands etc. Those are the main reasons why a measurement won’t load.

In the program GUI, there are currently four tabs: “Fitting”, “Stress Analysis”, “Universal Plot” and “Plot Fit Data”.

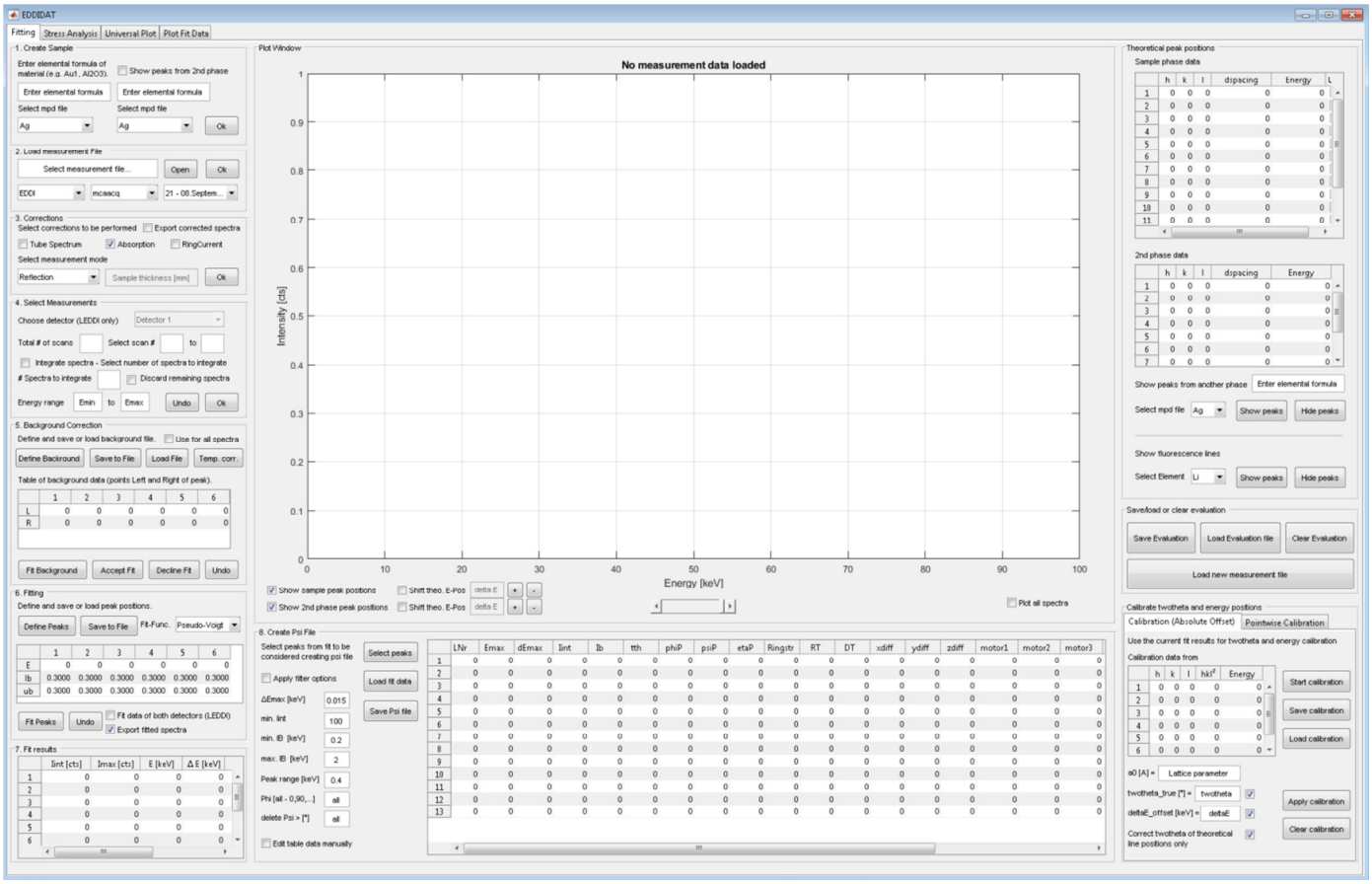


Fig. 1 The GUI of EDDIDAT.

3. Fitting of Measurement data

In the "Fitting tab", the measurement data is fitted. The user is guided through the fitting process in steps 1 to 8. The single steps are as following:

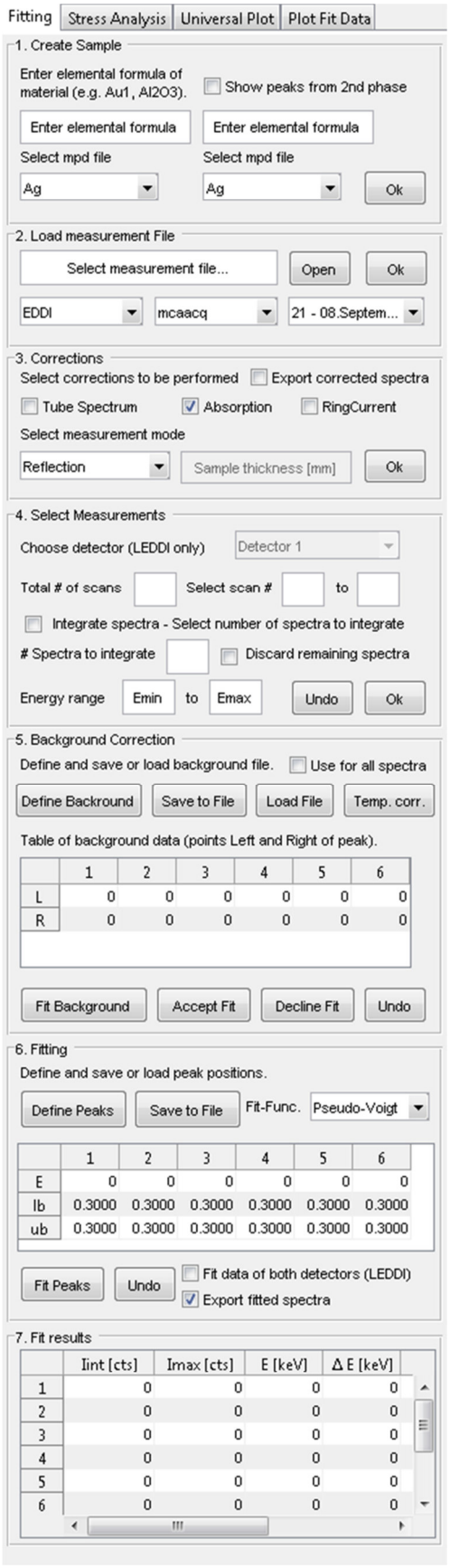


Fig. 2 The steps 1-7 of fitting a measurement.

1) Create Sample

The user has to enter the elemental formula of the material that has been measured. The corresponding mpd-file (material parameter data) has to be selected (if not available in the database, it can easily be created). This file contains information about the material, e.g. atom weight, density, crystal structure and lattice parameter.

2) Load measurement File

Select the measurement file that should be analyzed. The user has to choose the diffractometer, scan mode and dead time correction function for the measurement.

3) Corrections

Select the corrections that should be applied to the measurement data. The user can choose from different measurement modes. The data can also be fitted without corrections, just uncheck all checkboxes and press 'Ok'.

4) Select Measurement

The total number of scans is shown and the user can select the scans to be analyzed (e.g. from scan 1 to 30). It is also possible to integrate over a selected number of spectra (e.g. measurement data contains 32 Scans and 5 spectra should be used for integration, which leaves 2 scans, which the user can discard from the analysis). In order to define the energy range that should be analyzed the user can select Emin and Emax.

5) Background correction

Define points for background reduction. Mark a point left and right of each peak. Background information can be saved to a file to be reused. In case of temperature data, a temperature correction function can be created, in order to account for temperature induced peak shift. After fitting the background the user has to accept the corrected data before continuing the analysis.

6) Fitting

Define peak positions by marking the peak maximum of the peak that has to be analyzed. Those values will be used as starting values for the refinement procedure. In case of overlapping peaks, the lower and upper boundary of the energy position can be adjusted. The fit function can also be chosen. The following functions are available: Pseudo-Voigt, Thompson-Cox-Hastings Pseudo-Voigt, Gauss and Lorentz.

7) Fit results

Table with some of the fitted line profile parameters for a quick check.

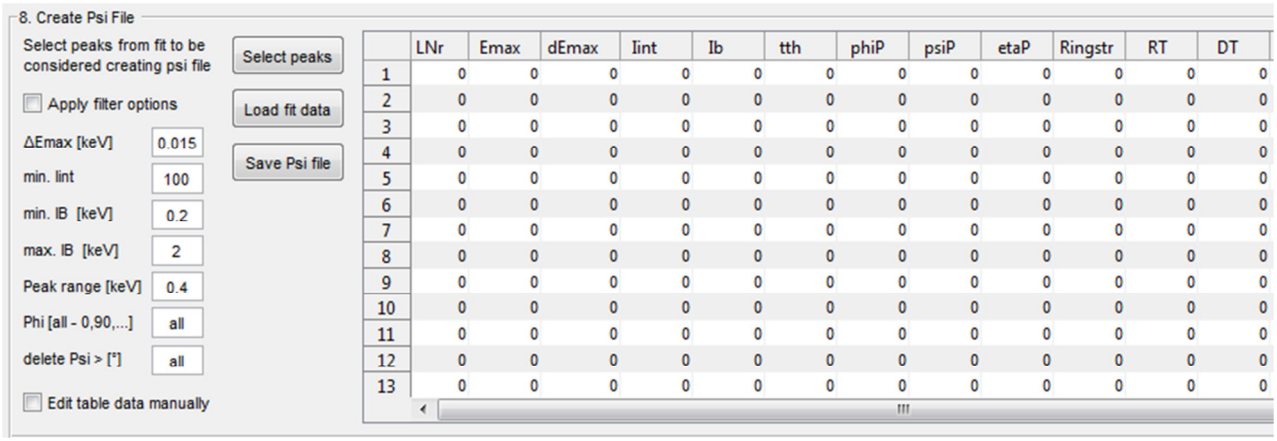


Fig. 3 The step 8 of fitting a measurement.

8) Create Psi File

The user can select the peaks ('Select peaks') he wants to add to the results file ("Psi" file). Clicking "Load fit data" adds the information of the fitted peaks to the table. In order to filter the data (e.g. zero intensity peaks, large Emax errors, very broad or narrow peaks etc.) the user can select from different filter options. Clicking on "Save Psi file" creates an ASCII file containing the data from the table.

After a finished fitting procedure the GUI looks like this.

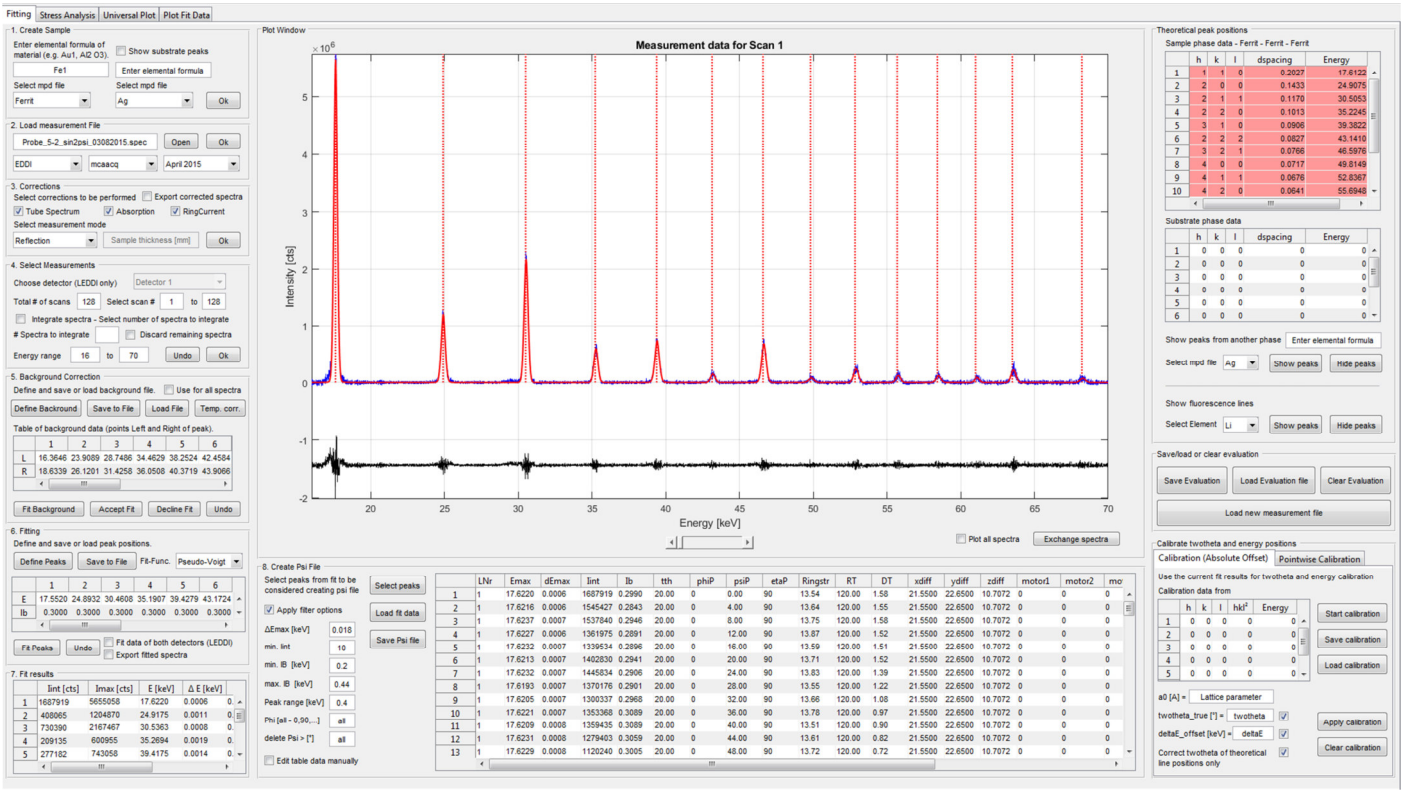


Fig. 4 GUI after a finished fitting procedure.

The finished analysis can be saved to a 'GUIData'-file. This file can be used to process the data at a later time or to share results with other users.

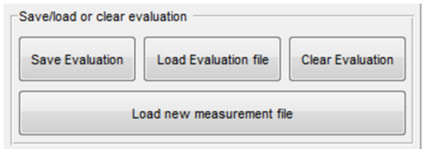


Fig. 5 Save/load evaluation file.

4. The Plot Window

The intensity of the current scan is plotted as a function of energy. The user can switch through the scans by clicking on the slider beneath the plot. To make it easier for the user to identify the peaks of the investigated material, lines describing the theoretical energy positions are displayed in the plot window. The corresponding peak information of all peaks available in the chosen energy range is summarized in a table. Additionally, the user can plot the theoretical energy positions of any element/material/fluorescence line that is saved in the data base. In case of textured samples, it might be useful for the user to plot all scans simultaneously. This can be done by clicking the checkbox "Plot all spectra".

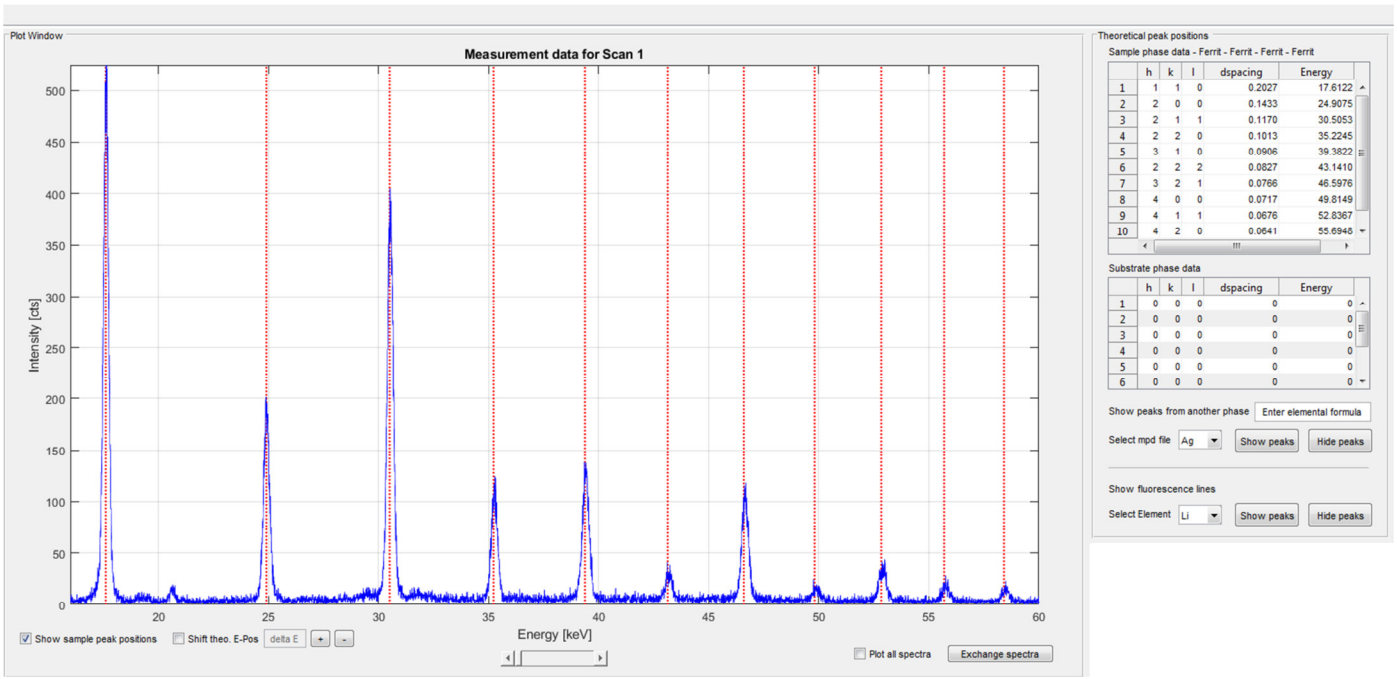


Fig. 6 Plot window of the GUI.

5. Calibration of measurement data

It is possible to calibrate the 2θ angle and the energy positions E^{hkl} using a calibration measurement (e.g. from a stress-free powder sample). The user has to fit the energy positions of the respective sample material and the calibration routine is started by clicking on 'Start calibration'. The program then calculates the "true" 2θ angle and the energy offset ΔE. Clicking on "Apply calibration" corrects the values in the psi file table, whereas the user can choose between correcting 2θ or the energy positions.

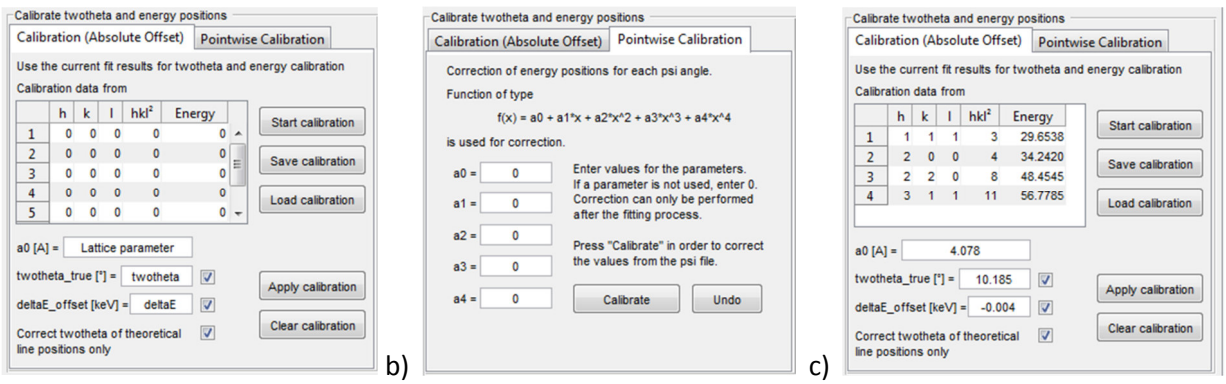


Fig. 7 Tab with calibration routine. The user can correct an (a) absolute offset or (b) correct the energy positions using a pointwise calibration. In c) shown is the result from the calibration of an Au-powder sample ($2\theta = 10°$).

It is also possible to use this tab to correct/calibrate the theoretical line positions (unmark the checkboxes twotheta_true and deltaE_offset). This could be helpful in cases where the adjusted 2θ angle is not as desired (e.g. $2\theta_{desired} = 16°$, $2\theta_{adjusted} = 16.45°$), which would lead to a significant offset of the measured and theoretical line positions. A too large offset could lead to problems during fitting.

6. The "Plot Fit Data" Tab

After the fitting procedure, it is possible to plot the fit results. The user can choose multiple parameters for the x- as well as the y-axes. The plots are created for each peak analyzed. The plots are exported as TIFF-images as well as ASCII files.

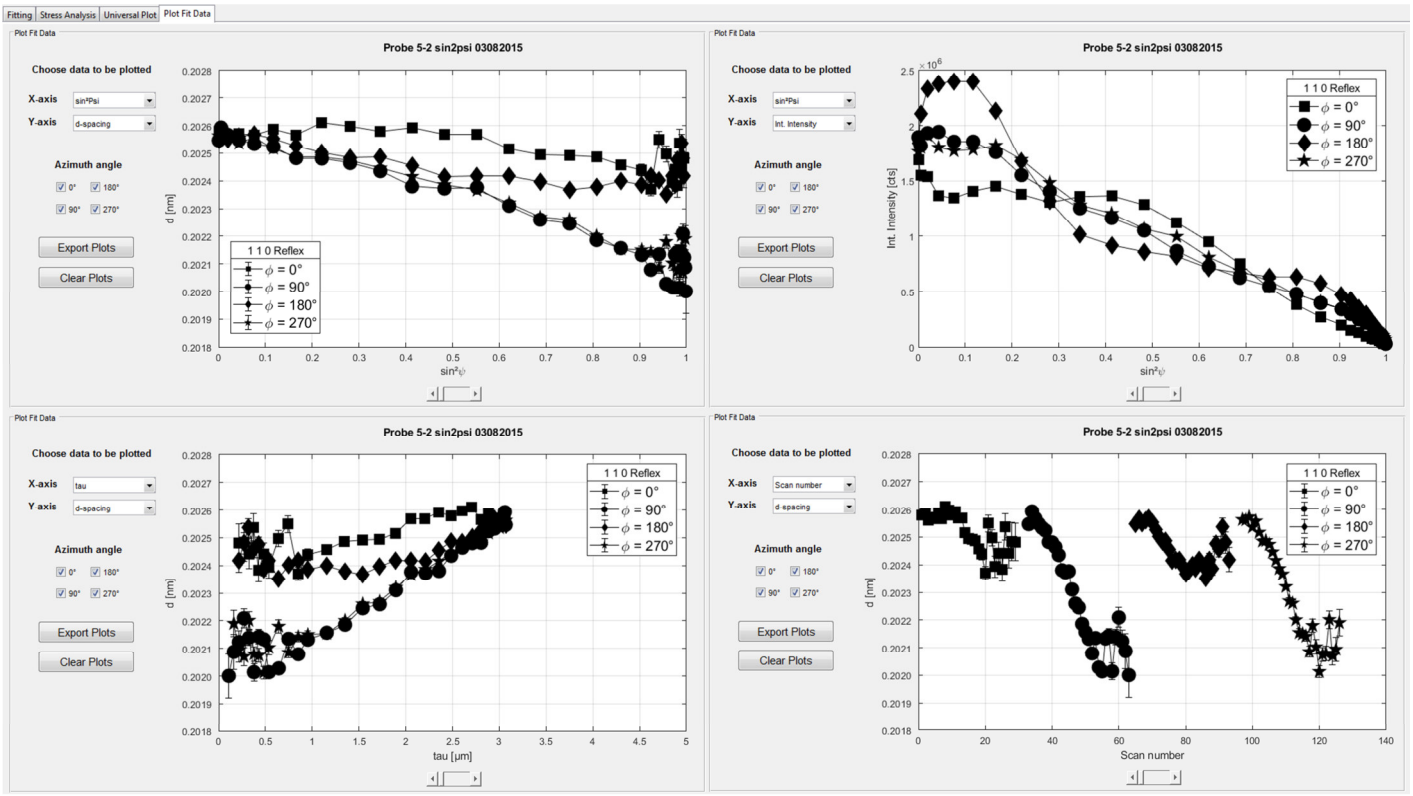


Fig. 8 The Plot Fit Data tab.

7. The "Stress Analysis" Tab

In the "Stress Analysis" tab the user can calculate stresses from the previously fitted measurement data based on the modified multi-wavelength method [1]. Before calculating stresses, the user has to enter the diffraction elastic constants (DEC) (see Fig. 9). If the DEC of the material under investigation is saved in the data base the program automatically finds the correct DEC for the fitted *hkl* peaks. Otherwise, the user has to enter the values manually. The new values can then be saved to the data base.

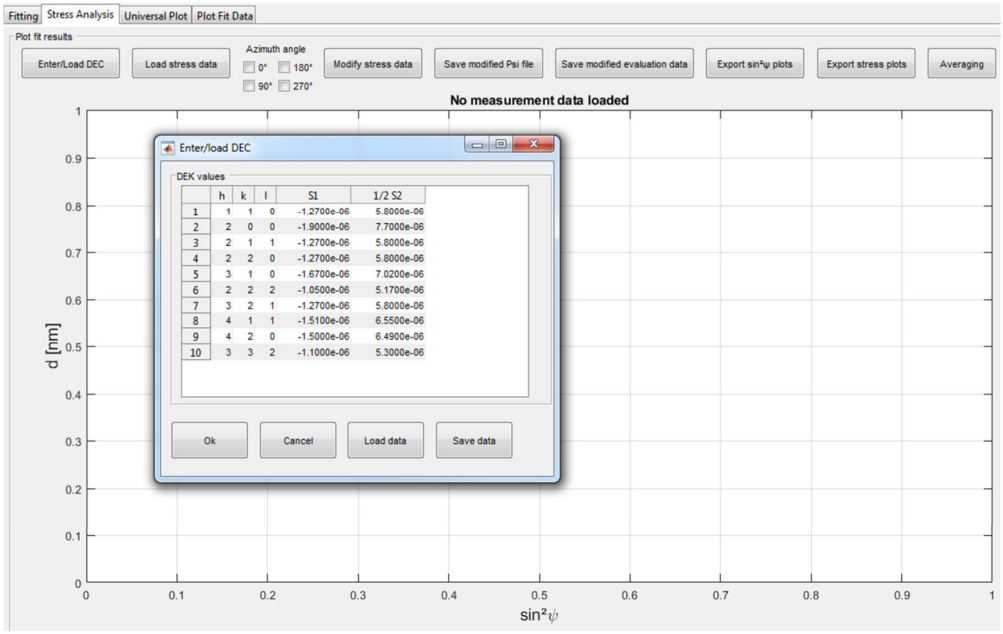


Fig. 9 Enter/load DEC values.

Clicking on "Load stress data" starts the calculation of stresses. The program automatically detects under which azimuths ($\varphi = 0°, 90°, 180°, 270°$) the measurements were conducted and calculates the values for the corresponding stress component. The $d - \sin^2\psi$ distributions and the resulting regression lines are plotted in the plot window for each *hkl* used in the analysis. On the right side of the plot the calculated residual stress distributions are shown. The individual stress components can be accessed by clicking the slider. The axes limits and thick marks can be defined by the user for each stress plot (Change X-scale/Y-scale).

Additionally, plots of the integral breadth, integrated intensity and the fitted peak for a specific ψ angle are shown (see Fig. 10). Those plots can be used by the user to identify data points that might not fit the distribution, e.g. due to bad fitting. Those data points can be selected and deleted by the user through clicking on "Modify stress data". In case of measurements that where conducted under more than one φ angle the user has to select the $d_\varphi - \sin^2\psi$ distribution he wants to delete data points from first by clicking on the proper checkbox. The stresses are than recalculated "on the fly", so the user can directly see the effect of deleting the point. All plots can be exported as TIFF-image as well as ASCII-files. In case of the stress plots, a tau-file is created. This file contains the respective d_0 and stress values $\sigma[\tau(hkl)]$. The made changes can also be saved to a corrected psi-file and GUIData-file. Additionally, the stress plot data is saved to a file so that the user can load the stress plot (button "Load tau data") at a later time and make changes to the axis limits, axis ticks or the hkl values that should be plotted, if desired.

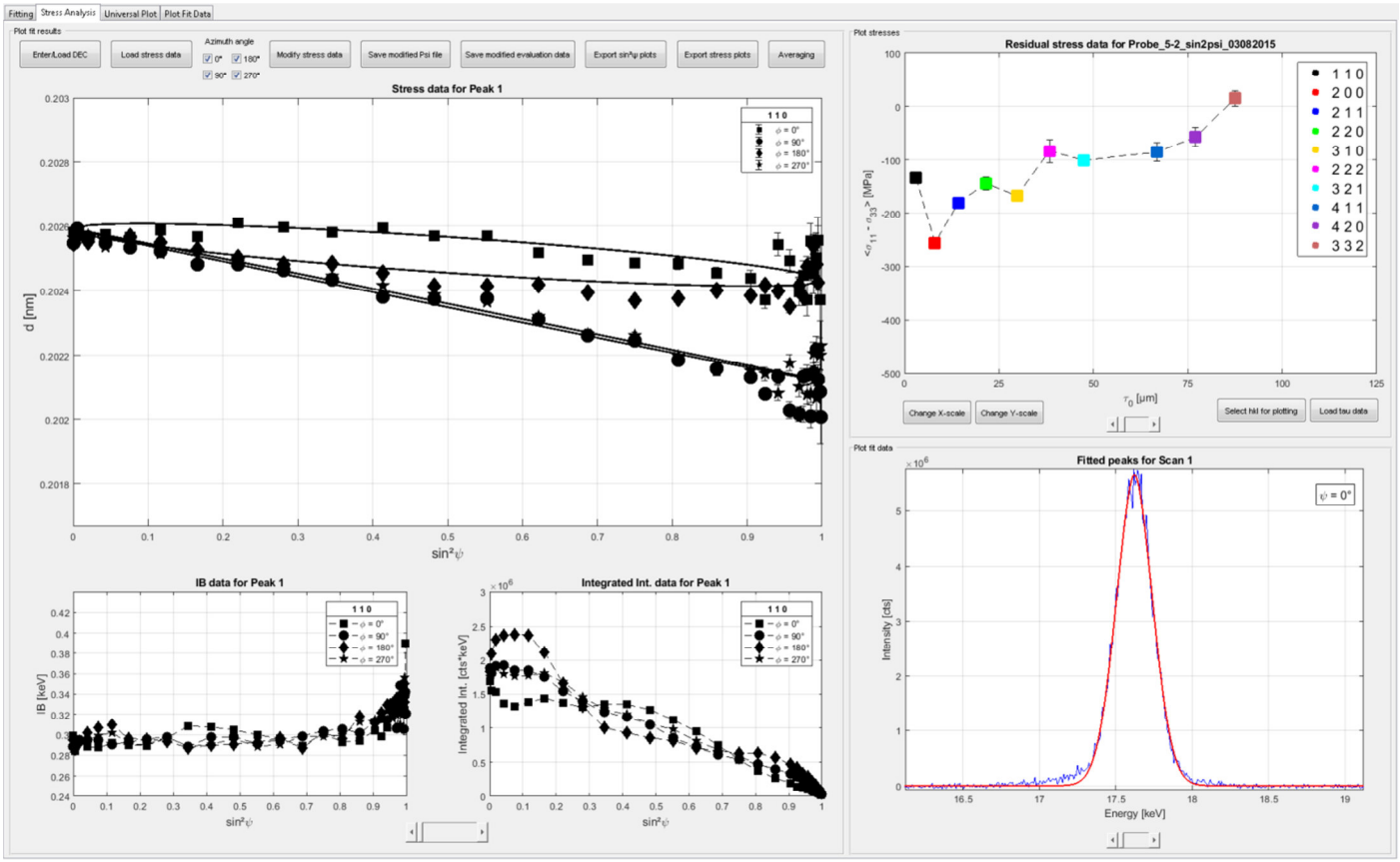


Fig. 10 The „Stress Analysis" tab.

For cubic crystal symmetry, averaging over several lattice planes *hkl* can be performed. The program weights the contributions of the individual lines according to the multiplicity H(*hkl*). The result is a $\sin^2\psi$-distribution which is normalized to a(100).

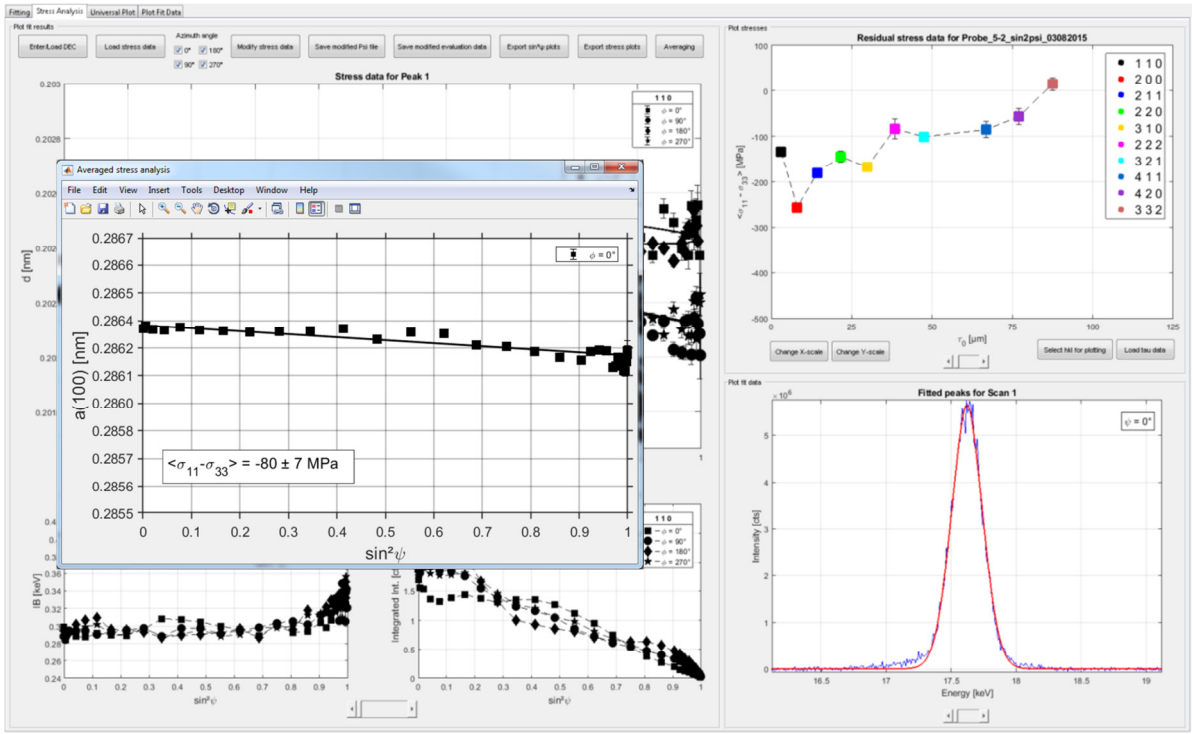


Fig. 11 Results of averaging over several lattice planes.

8. The "Universal Plot" Tab

In the "Universal Plot" tab the user can calculate stresses from the previously fitted measurement data based on the universal plot method [2] (see Fig. 12). Here, the stresses can be plotted as a function of the corresponding information depth τ into one universal plot. It is then possible to determine the Laplace space $\sigma(\tau)$ and the real space stress depth profile $\sigma(z)$, respectively, by fitting appropriate (polynomial) functions to the resulting stress depth distribution. To start the evaluation the user has to load the peak information by clicking the button "Load peak information". The *hkl* and d_0 values of all fitted peaks are loaded into a table. From the table, the user can select the peaks that should be used for the analysis. Furthermore, the user can modify the d_0 values that are used for the analysis (by changing them in the table and clicking on the corresponding checkbox). If there is a $d_0(z)$ gradient present in the sample the user can also enter a function to account for the gradient.

In order to prepare the data for fitting the user can select the $\sin^2\psi$-range, for which data should be plotted. Additionally, the user can chose different filter options to further improve the data or delete data points with large errors (Filter options). For the fitting of the $\sigma(\tau)$ data, the user can choose between undamped and damped polynomials of different degrees (Fit properties). The fitting process is started by clicking on the button "Plot data". The resulting stress depth profiles are shown in the plot windows of each stress component (see Fig. 13). The data from the plot windows can be exported by clicking on "Export data". One way to assess the quality of the fitted stress depth profiles is to recalculate the *d*-$\sin^2\psi$-distributions using the determined (fitted) depth profiles. This can be done by clicking on "Plot *d*-$\sin^2\psi$-distributions" where a new window opens showing the measured *d*-$\sin^2\psi$-distributions and the recalculated ones (see Fig. 14). The corresponding data can also be exported.

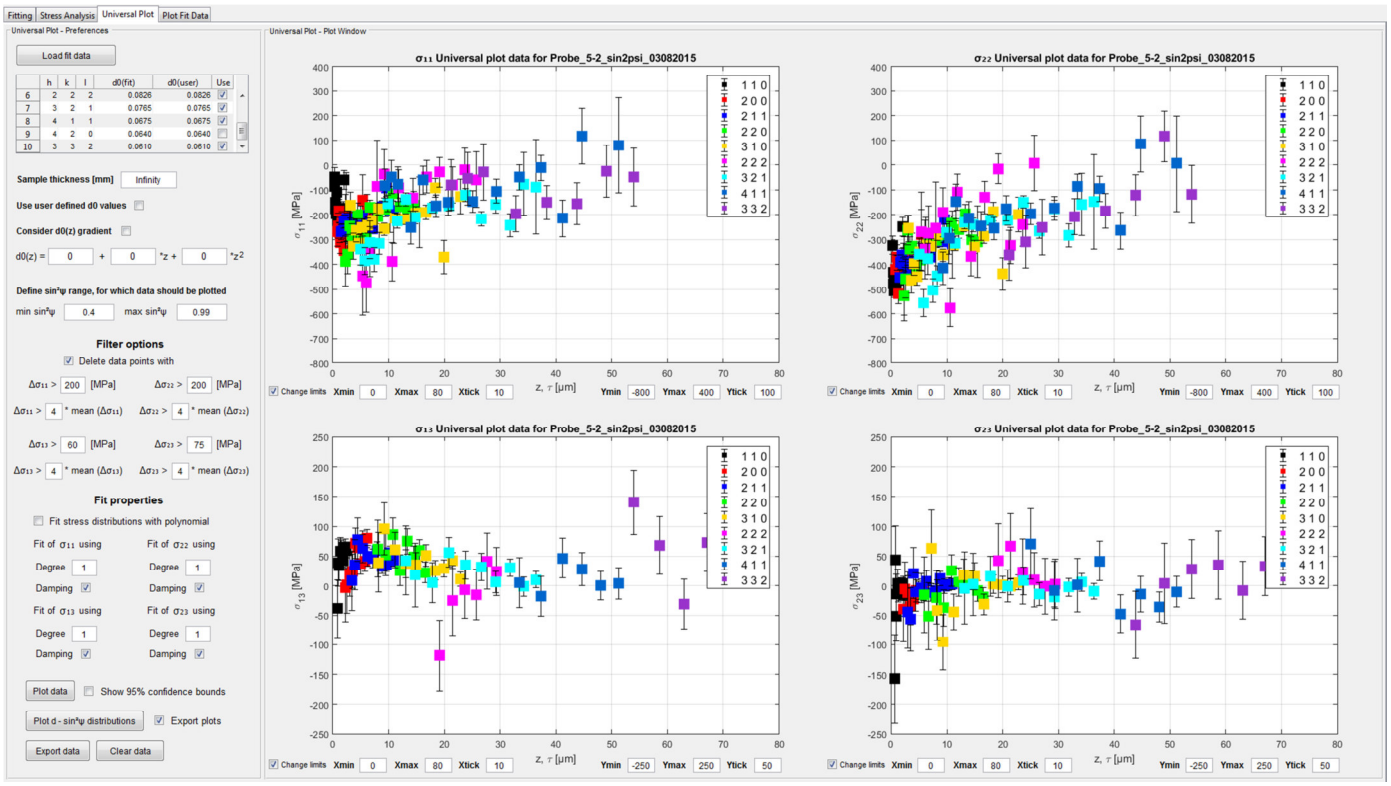


Fig. 12 The „Universal Plot" tab.

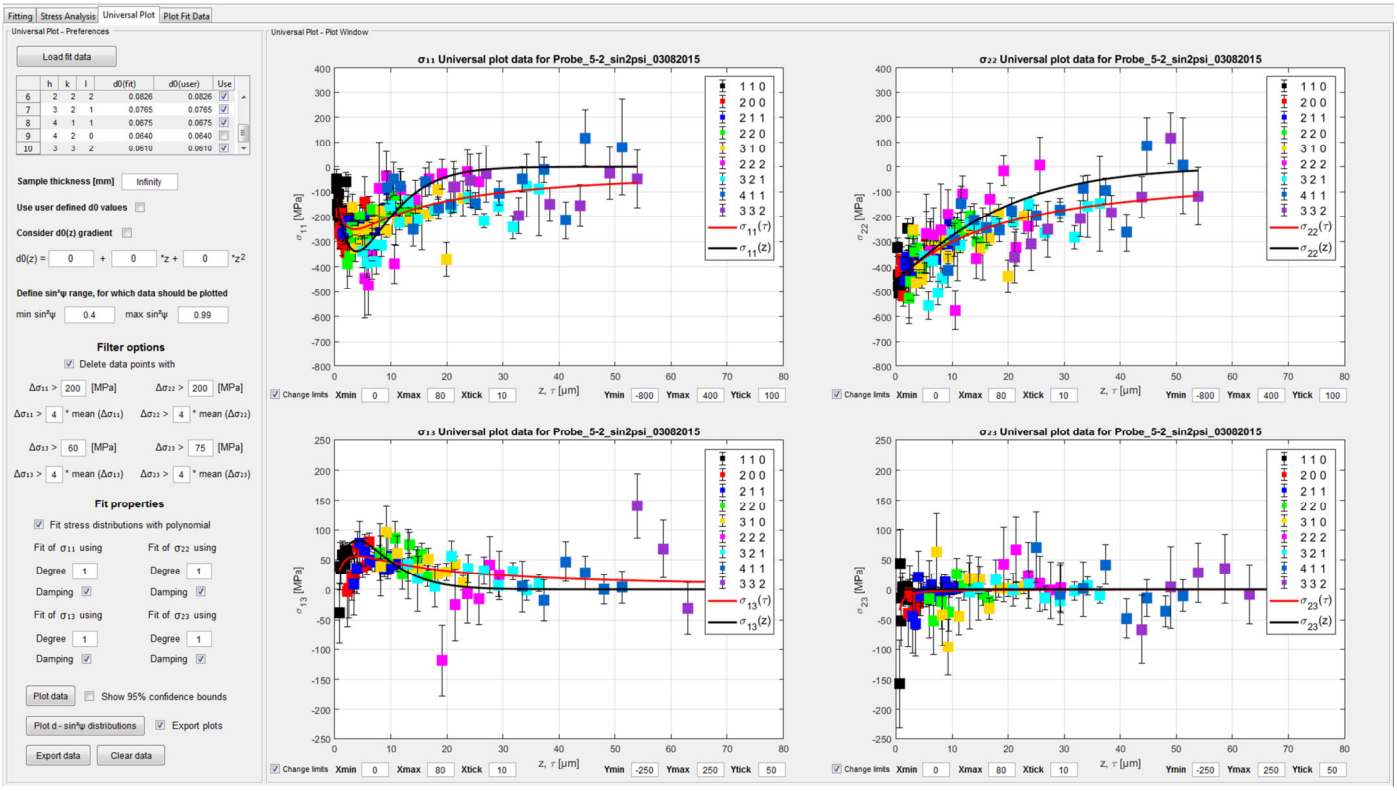


Fig. 13 Fitted Laplace space $\sigma(\tau)$ and real space $\sigma(z)$ stress depth profiles.

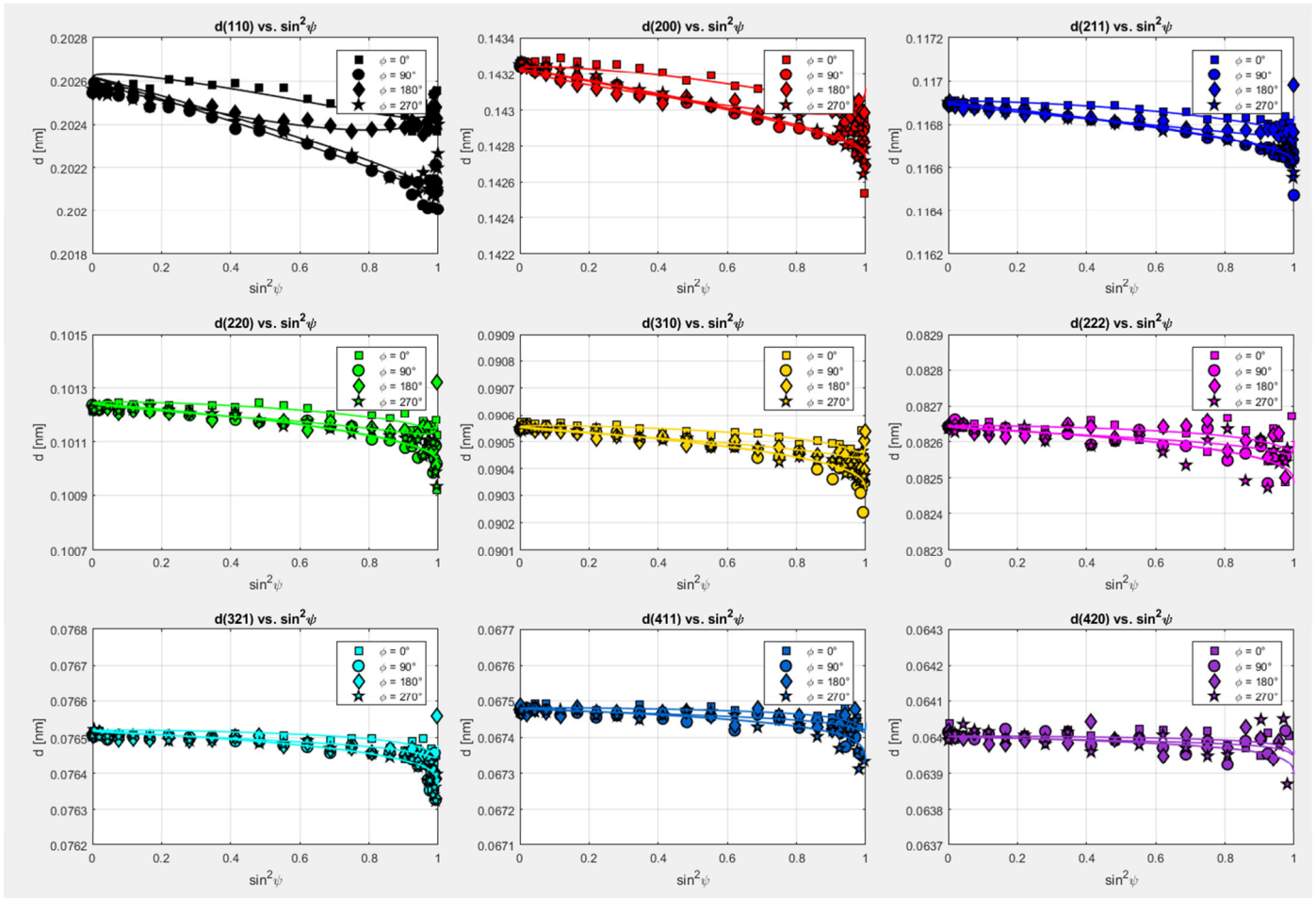


Fig. 14 Recalculated d-sin$^2\psi$-distributions using the fitted stress depth profiles.

[1] Klaus, M. & Genzel, C. (2019) J. Appl. Cryst. 52, 94-105
[2] Ruppersberg, H., Detemple, I., Krier, J. (1989) Phys. Status Solidi (a) 116, 681

If the user experiences any problems or has suggestions for improvements, please feel free to contact me.

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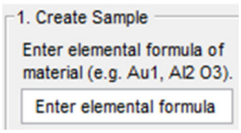
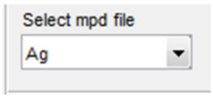
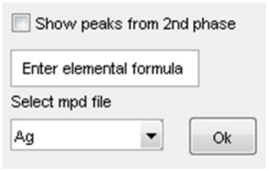
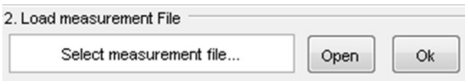
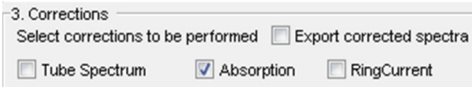
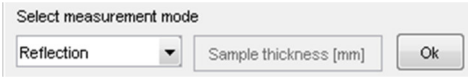
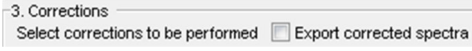
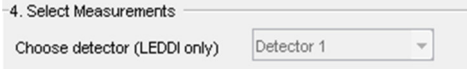
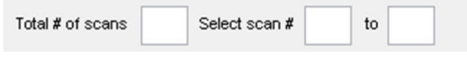
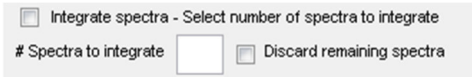
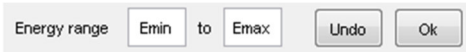
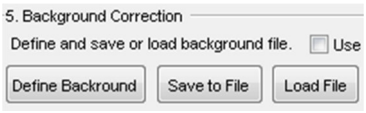
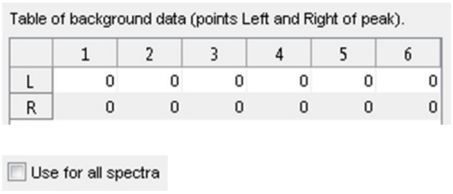
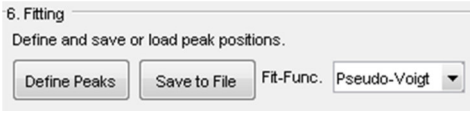
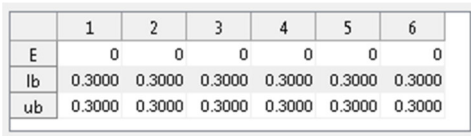
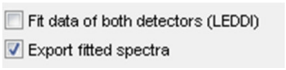
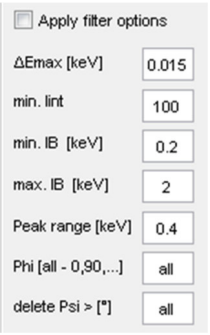
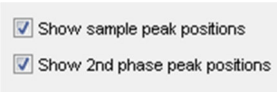
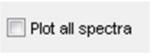
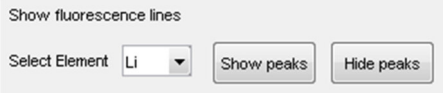
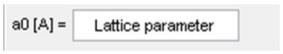
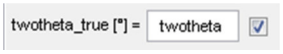
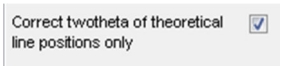
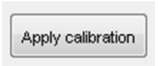
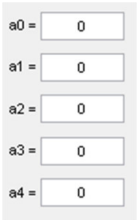
9. Appendix

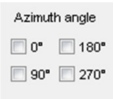
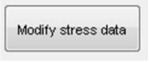
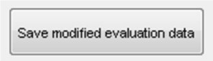
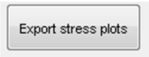
In the following, each button/edit field/checkbox and its purpose is described.

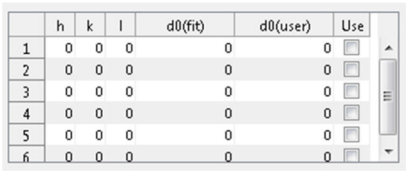
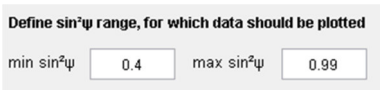
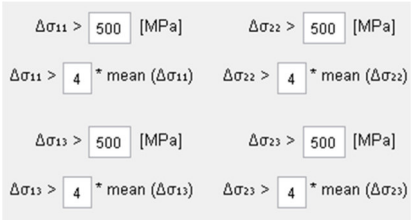
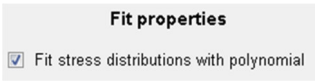
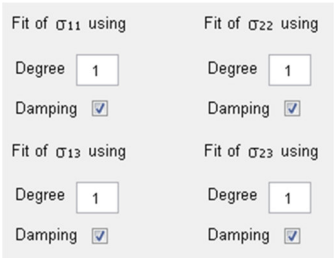
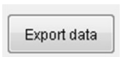
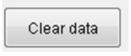
Fitting Tab	
Create Sample field	
	**Enter elemental formula** (empirical formula): enter each element followed by its relative number
	**Select material parameter file:** if mpd is missing, the user can create a new one (based on the format given in the mpd file) and simply add it to the folder \Data\Materials
	**Show peaks from 2nd phase:** the peaks from a 2nd phase can be plotted by clicking the checkbox and entering the elemental formula and selecting the mpd file (only for visual guidance and not for fitting: each phase has to be fitted separately)
Load Measurement field	
	**Select the measurement file** to be analyzed
	**1st drop down menu:** select instrument that was used for the measurement **2nd drop down menu:** select measurement mode (e.g. continuous measurement or a-scan/d-scan) **3rd drop down menu:** select dead time correction function to convert from channel to energy scale
Corrections field	
	Select corrections that should be performed to the measurement data: - **Tube spectrum** (e.g. Wiggler spectrum, W-tube spectrum, MetalJet spectrum) - **Absorption** - **RingCurrent** (only for synchrotron measurements)
	**Select measurement mode:** e.g. reflection, transmission (if chosen, "Sample thickness" edit field becomes active)
	**Export corrected spectra:** corrected spectra can be exported by checking the checkbox
Select measurement field	
	**Choose detector:** if LEDDI diffractometer has been used, choose detector data that should be plotted/analyzed
	Shown are the total number of spectra/scans, the user can select/change the first and last spectra (e.g. the total number of scans) that should be used for the analysis

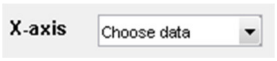
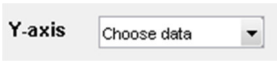
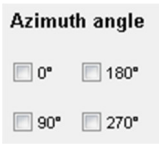
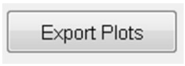
	**Integrate Spectra:** by checking the checkbox the user can select the number of spectra that should be integrated (e.g. 42 spectra were measured, the user selects that 5 spectra should be used for integration, i.e. that after the integration there are 8 "new" spectra (obtained from integrating over 5 consecutive spectra) and 2 spectra remain, which can be discarded if selected
	Select the energy range that should be plotted
Background correction field	
	**Define Background:** user can select points left and right of each peak to define background **Save to File:** background points can be saved to a file **Load File:** load previously defined background points
	**Temp. corr.:** Button to start temperature correction of background points, e.g. in case of temperature measurements
	Table showing the defined background points (for the selected spectra), which can be changed manually by clicking on the desired entry **Use for all spectra:** check checkbox in order to make changes to a background point that should be applied to all spectra rather than only the current one
	**Fit Background:** fit background based on defined background points **Accept Fit:** the fitted background is previewed, if ok, accept fit **Decline Fit:** proposed background correction is not ok, decline fit and manually change background points **Undo:** delete current background points and start over
Fitting field	
	**Define Peaks:** select peaks by marking the peak maximum **Save to File:** save peak max information to background file **Fit-Function:** select peak fit function
	Table showing the defined peak maximum positions (for the selected spectra), which can be changed manually by clicking on the desired entry **lb:** lower boundary of peak max. position for fitting routine, can be changed manually **ub:** upper boundary of peak max. position for fitting routine, can be changed manually
	**Fit Peaks:** start fitting of peaks **Undo:** delete defined peak max. positions and start over
	**Fit data of both detectors (LEDDI):** check checkbox in order to fit data from both detectors simultaneously (only if 2θ of both detectors is the same) **Export fitted spectra:** export fitted spectra

Create Psi file field	
	**Select peaks**: select peaks that should be considered in the psi file from a table that opens after clicking this button – each fitted peak and its *hkl* values and energy position is shown in this table, if a peak does not match the theoretical peak positions, e.g. fitted peak from 2nd phase or dummy peak, NaN's are shown instead of the *hkl* values
	**Load fit data**: load fitted peak data to psi file table
	**Save psi file**: save psi file table to file
	**Apply filter options**: by clicking the checkbox the user can define filter options for creating the psi file via the "Load fit data" button - $\Delta Emax$: maximum fit error of the fitted energy position of each peak - min. Iint: minimum integrated intensity of each peak - min. IB [keV]: minimum integral breadth of each peak - max. IB [keV]: maximum integral breadth of each peak - Peak range [keV]: max range of peak maximum defined by the user - Phi: select which phi angles should be considered - delete Psi > [°]: delete peak data for psi angles larger than …
	**Edit table data manually**: delete table data manually by checking the checkbox and clicking on the desired line in the psi file table
Plot window	
	**Show sample peak positions**: show or hide theoretical energy positions of sample **Show 2nd phase peak positions**: show or hide theoretical energy positions of 2nd phase
	**Shift theo. E-Pos**: shift theoretical energy positions of sample peaks/2nd phase peaks by the amount of delta E in + or - direction
	**Plot all spectra**: click to plot all spectra simultaneously, e.g. to check for texture effects or temperature induced peak shift
Theoretical peak positions field	
	Similar to the "Create Sample" field the user can enter the elemental formula and select the mpd file from a different phase and add the theoretical energy positions from this phase to the current plot by clicking on **Show peaks** – clicking on **Hide peaks** deletes the energy positions from the plot
	The user can select an element from the drop down menu and can add the fluorescence energy positions from the chosen element to the current plot by clicking on **Show peaks** – clicking on **Hide peaks** deletes the fluorescence energy positions from the plot
Save/load or clear evaluation field	
	**Save Evaluation**: save current evaluation to file, usually applied after creating the psi file so that the user can continue the e.g. stress analysis at a later time or share the results with other user

	**Load Evaluation file:** load evaluation file
	**Clear Evaluation:** clear all entered data and start a new evaluation (in case there are some errors after pressing Clear Evaluation, restart the program instead)
	**Load new measurement file:** load another measurement file from the same sample, the sample information and other previously entered information, e.g. the selected energy range, will be saved
Calibrate twotheta and energy positions field	
Calibration (Absolute Offset)	
	**Start calibration:** if the user analyzed the measurement of standard powder sample, e.g. gold powder, clicking on "Start calibration" will use the fitted energy positions to calculate the true 2θ value and the energy offset ΔE The results are shown in the Calibration data table and the respective edit fields
	**Save calibration:** save calibration data to a file
	**Load calibration:** load calibration data from a file
	a_0 lattice parameter from mpd file
	Calculated true 2θ value
	Calculated energy offset ΔE
	If the user checks this checkbox and unchecks the two checkboxes shown above and manually enters a value for 2θ into the field "twotheta_true" it is possible to shift the theoretical energy positions shown in the plot window
	**Apply calibration:** apply calibration to the data shown in the psi file table If a calibration file is loaded the calibration can be applied to any fitted data
	**Clear calibration:** clear current calibration data
Pointwise Calibration	
	The user can enter the parameters of a polynomial up to 4th degree in order to conduct a pointwise calibration of the fitted data The polynomial function must be determined prior to the evaluation, e.g. using a calibration measurement on a standard powder

	**Calibrate:** apply calibration function to the energy values in the psi file table
	**Undo:** undo calibration of the energy values in the psi file table
Stress Analysis Tab	
	**Enter/Load DEC:** enter or load the DEC needed for calculation of residual stresses Opens a window with a table for the DEC – if the program recognizes the phase/material it automatically loads the corresponding DEC (if available in the database), otherwise the user can enter the DEC manually (and safe them to a file for re-use)
	**Load stress data:** starts the calculation process of the residual stresses
	**Azimuth angle:** choose azimuth angle(s) for which the measurement data should be plotted
	**Modify stress data:** here the user can manually select data points that should be deleted from the d-sin²ψ plot Before selecting data select the desired azimuth angle
	**Save modified Psi file:** save psi file with modified data points
	**Save modified evaluation data:** save evaluation file with modified data points
	**Export sin² ψ plots:** export sin² ψ plots to data and graphic file
	**Export stress plots:** export stress plots and create tau file with data points shown in the stress plot Additionally, the stress plot data is saved to a file that can be loaded in order to e.g. change axes limits
	**Averaging:** start averaging procedure that allows to average over all diffraction lines (only for cubic materials)
	**Change X-scale:** change scale and tick marks of X-axis
	**Change Y-scale:** change scale and tick marks of Y-axis
	**Select hkl for plotting:** select hkl values that should be used for the stress plot
	**Load tau data:** load tau data from previous evaluation
Universal Plot tab	
	**Load fit data:** load previously fitted data from “Stress Analysis Tab”

	Table showing hkl and d0 values from fit The user can enter d0 values manually and can choose which peak should be used for the universal plot
	Enter sample thickness in case of thin film material
	Check in order to use user defined d0 values
	Consider d0(z) gradient for the analysis
	Define $\sin^2\psi$ range, for which data should be plotted
	Select filter options in order to delete data points with large error values If not selected the raw data will be plotted
	The user can select a maximum error for each stress value or a multiple of the mean error of each stress value to filter the data
	Select fit properties If not selected the stress distributions are not fitted
	Here the user can choose the polynomial degree that is sued to fit the stress distributions and whether damping should be applied or not
	**Plot data:** plot universal plot data The user can choose to show the 95% confidence bounds for the fitted curves
	**Plot d-sin²ψ distributions:** plot the recalculated d-$\sin^2\psi$ distributions and choose to export the plots
	**Export data:** export data from universal plots shown in this tab
	**Clear data:** clear plots
	**Change limits:** when clicking this checkbox edit fields to change the axis limits and tick marks appear

Plot Fit Data tab	
	**X-axis:** chose data to be plotted on the X-axis
	**Y-axis:** chose data to be plotted on the Y-axis
	**Azimuth angle:** select from which azimuth angle data should be plotted
	**Export plots:** export plot data
	**Clear plots:** clear plots

10. Temperature correction of user defined background points and peak positions

In case of a temperature induced peak shift it is possible to correct the defined background points and peak positions accordingly. After the user created the background points it is possible to create correction functions that account for the observed peak shift by pushing the button “Temp. corr.”. A new window opens as shown below.

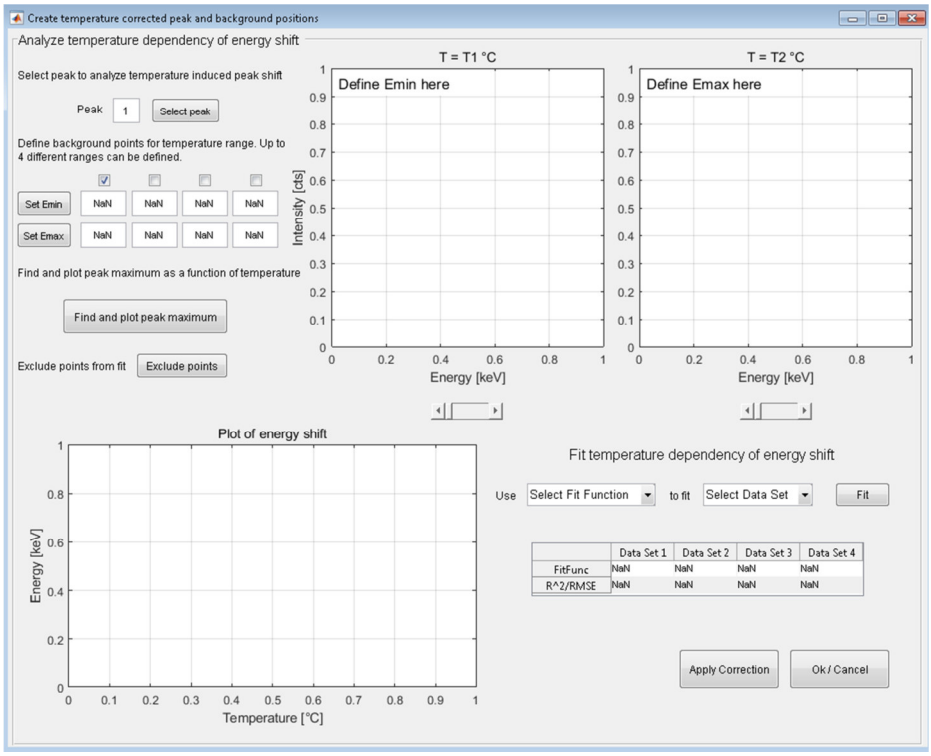


Fig. 15 Program window for the temperature correction of peak and background positions.

Here, the user can select a peak that should be used to determine the temperature induced energy shift (if more than one peak was defined). By clicking on “Select peak” the corresponding part of the spectrum is shown in the two plot windows, whereas the one on the left side is showing the spectrum for the first temperature step and the one on the right the one of the last temperature step. The temperatures of each spectrum shown are shown on top of the plot windows.

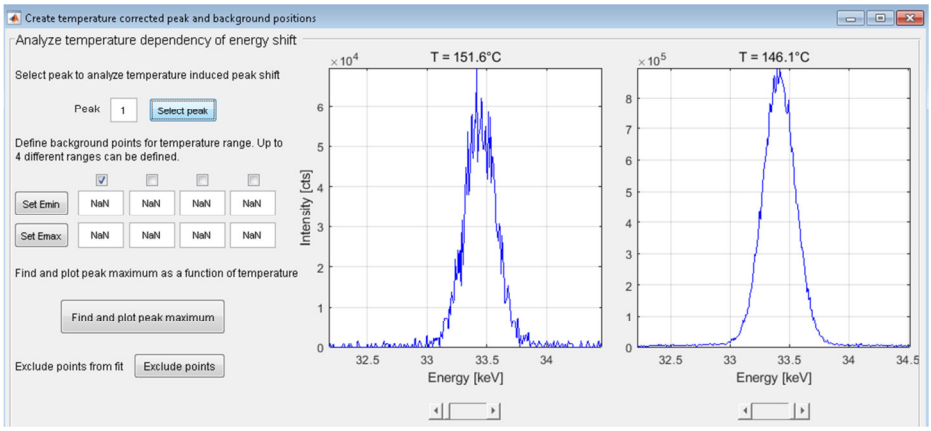


Fig. 16 Section of the diffraction spectrum of the peak chosen for correction.

The user can define up to four energy ranges in which a peak shift occurs (e.g. in case of heating – cooling – heating – cooling). The sample used here for explanation of the temperature correction was heated to 937°C and then cooled down to 146°C. Therefore, two different energy ranges have to be defined, one covering the heating from 151°C to 937°C and the other covering the cooling down to 146°C.

At first, the energy range for the heating section is defined. This is done as follows: with increasing temperature the diffraction peak is shifted to lower energies. The “Emin” value for the heating section has to be set in the spectrum with the highest temperature, here 937°C. The “Emax” value then has to be set in the spectrum with the starting

temperature, here the first spectrum recorded at 151°C (see red points in Fig). The "Emin" value is set in the left plot window whereas the "Emax" value is set in the right plot window.

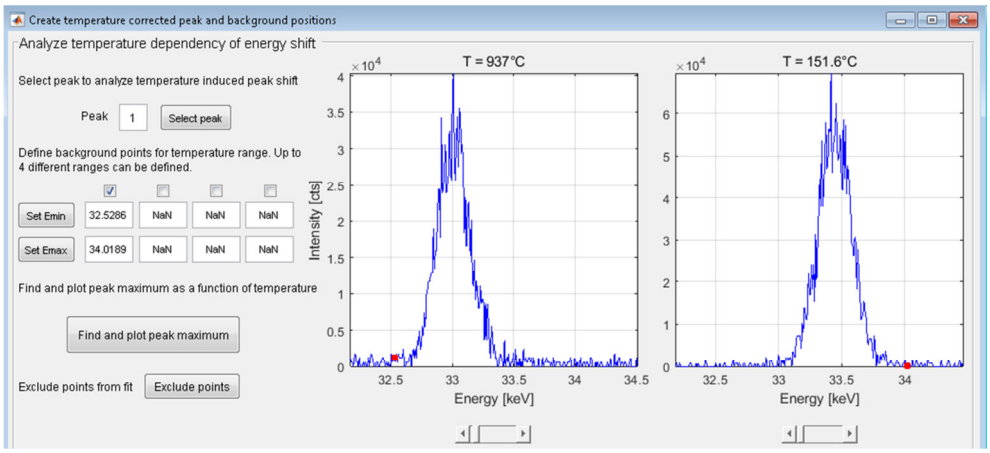


Fig. 17 Definition of Emin and Emax for the first energy range.

The second heating section is defined accordingly, here "Emin" has to be set in the spectrum at 825°C and "Emax" in the last spectrum at 146°C (see Fig. below).

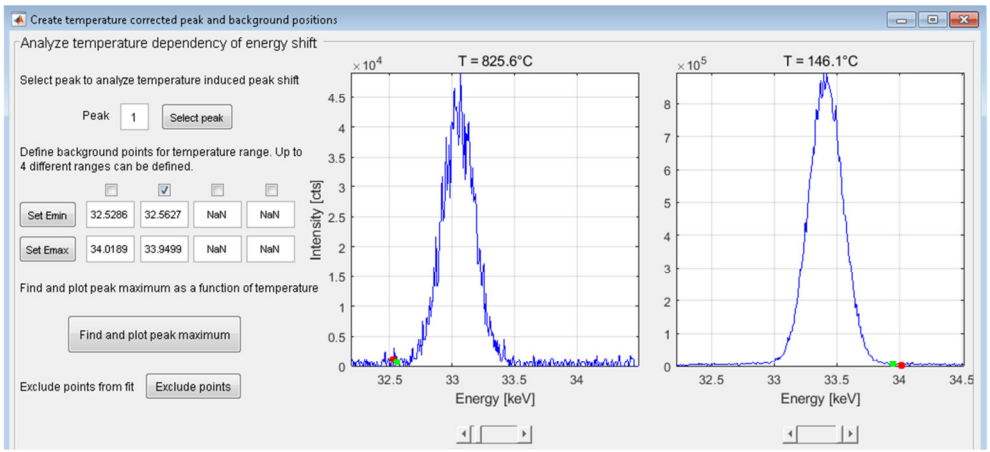


Fig. 18 Definition of Emin and Emax for the second energy range.

Clicking on "Find and plot peak maximum" starts a routine that searches for the peak maximum positions in the previously defined energy ranges and plots them as a function of temperature. Since two energy ranges were defined, two data sets created.

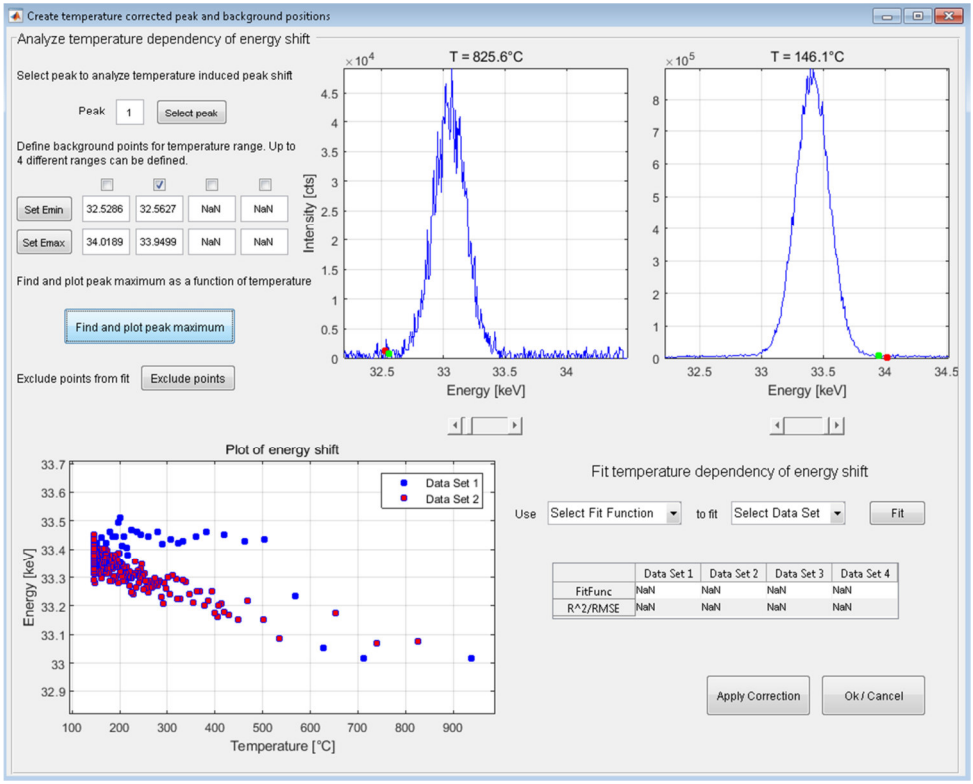


Fig. 19 Plots showing the determined temperature dependency of the peak energy position.

Now, the user can choose a fit function for each data set and fit the respective Energy-Temperature plot. Clicking on "Apply Correction" applies the respective correction function to the background points previously defined by the user (considering the temperature ranges defined here). Once defined, the correction will also be applied to the peak positions still to be defined.

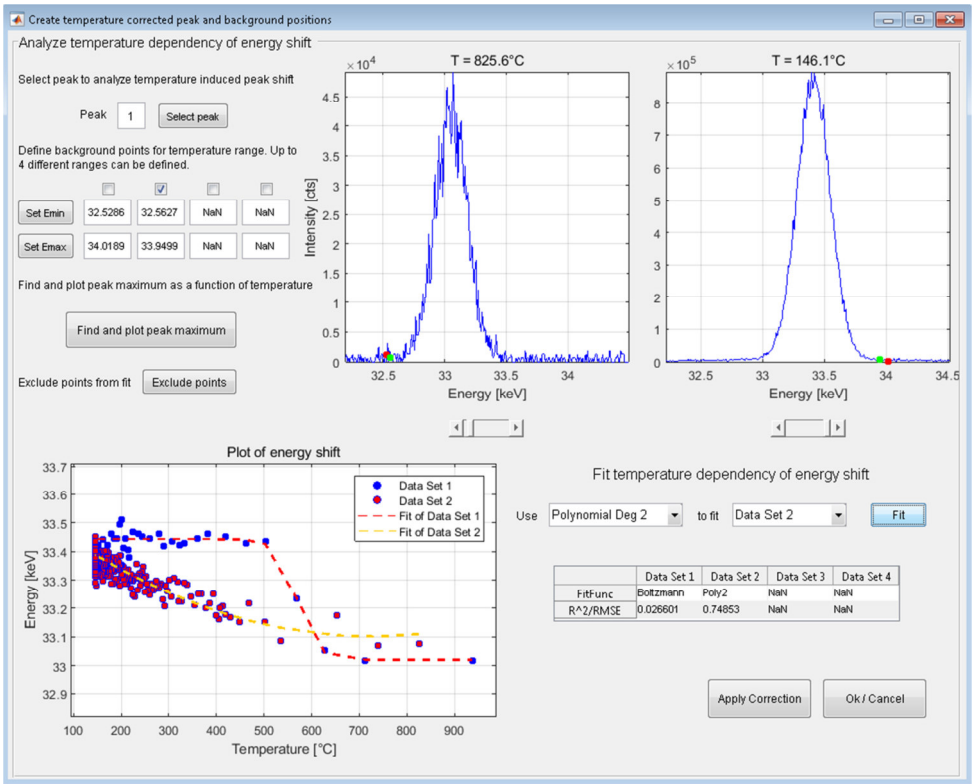


Fig. 20 Fit of the energy – temperature distributions.