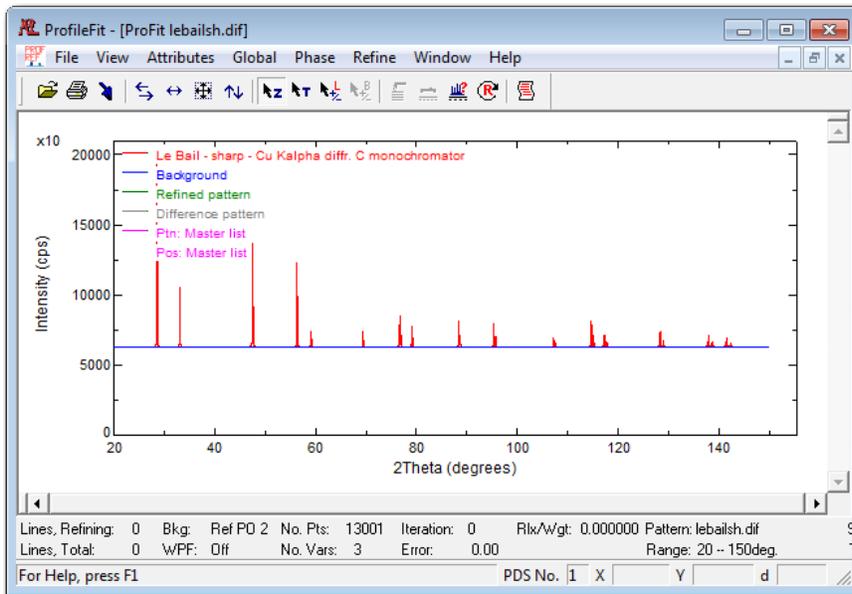
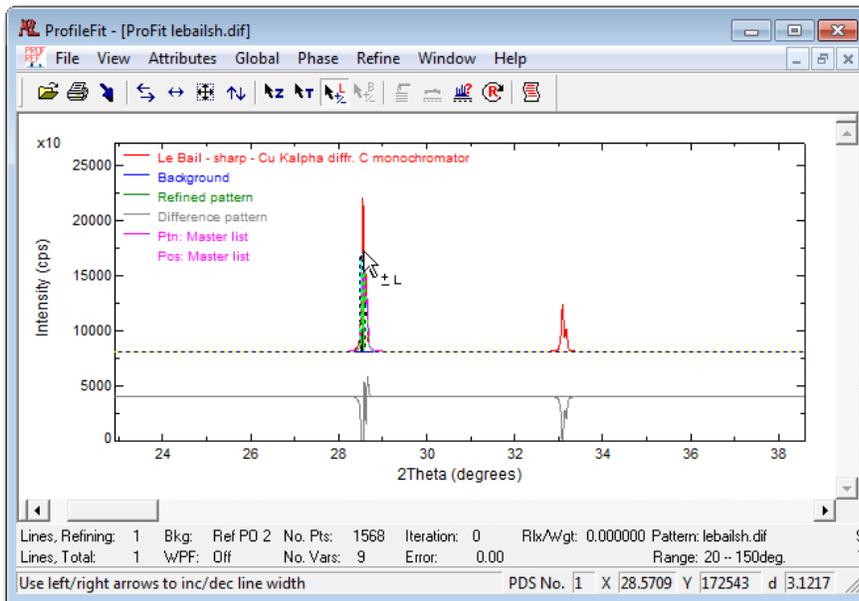


SizeStrain – For XRD profile fitting and Warren-Averbach analysis

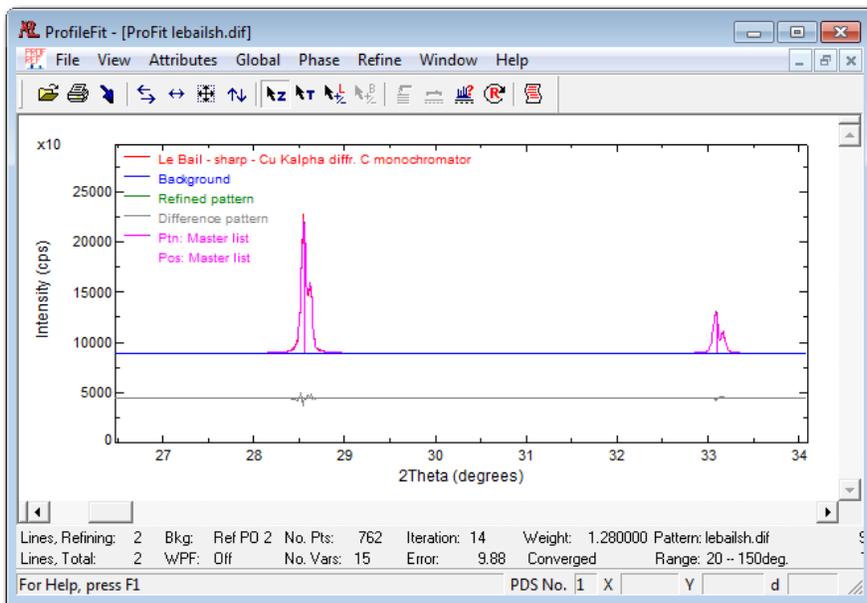
I. How to use MDI SizeStrain?



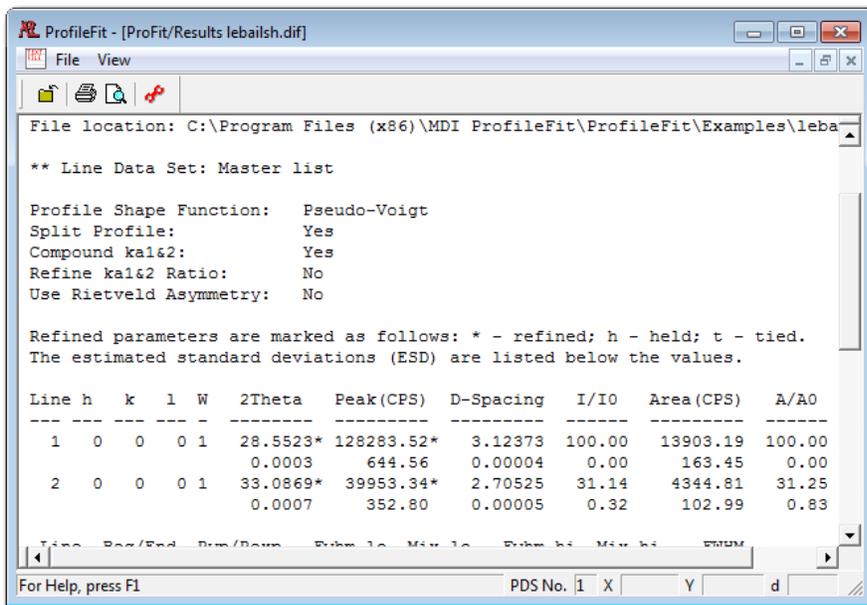
1. Start MDI SizeStrain, use menu *File -> Open Pattern for new Analysis* to open an XRD pattern file from *Program Files\MDI SizeStrain\Program\Example\lebaish.dif*.
 - a. Note that the program would open the most recently opened pattern file or work file upon starting.
 - b. To modify the program startup behavior, use menu *File->Preferences*, and select the desired behavior.
2. Zoom into a region with left-mouse drag. The zoomed area can be scaled and/or shifted by right-mouse drag.



3. Activate the line editing cursor (), then click on the peaks in the window (see above).
4. Click on the **R** button () to start refinement.
5. Press the ESC key to stop a refinement if it takes too long to finish.



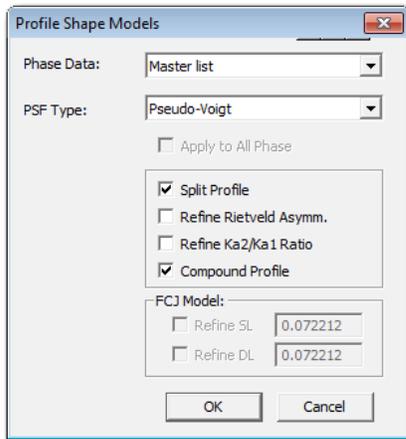
6. Save the current work using *File -> Save Work*. It can be loaded back later.
7. To view the refinement results, press the toolbar button ().



II. How to do a Warren-Averbach analysis:

A. Create instrument broadening calibration curves:

1. Open an XRD pattern from a reference sample that does not have any size broadening effect. For example, *LeBailsh.dif*, in *Program/Examples* folder.
2. Set profile shape function: Select menu *Phase -> Profile Model*, and select *Pseudo-Voigt* function. Check the box, "Split profile", to compensate the profile asymmetry.

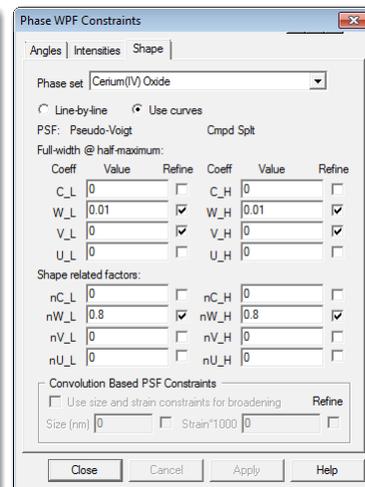
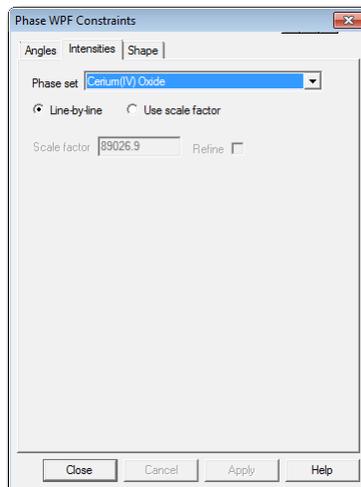
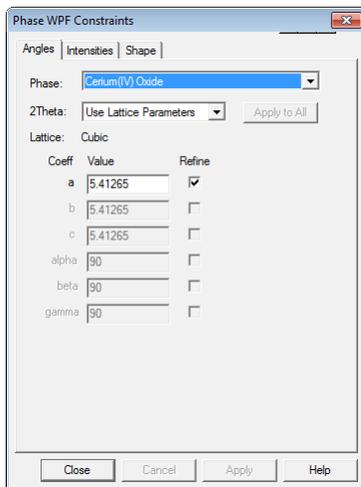


3. Select peaks to refine:

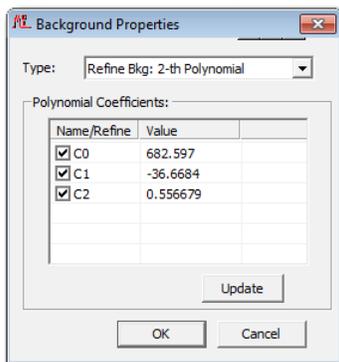
Load phase data created in Jade 9, CeO2.xml, using *Phase -> Add Phase from Jade PDF file*.

4. Set whole pattern fitting parameters: Select menu *Phase -> Whole-Pattern Fitting*:

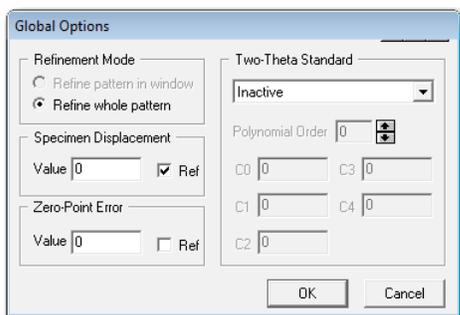
- Angles tab: select Use Lattice parameters, and check the box Refine.
- Intensity tab: select *Line-by-Line*.
- Shape tab: select Use curves and check the boxes (except C_L).



5. Set background refinement parameters: select menu *Global->Background model*. Use refined 2nd order polynomial as background model.

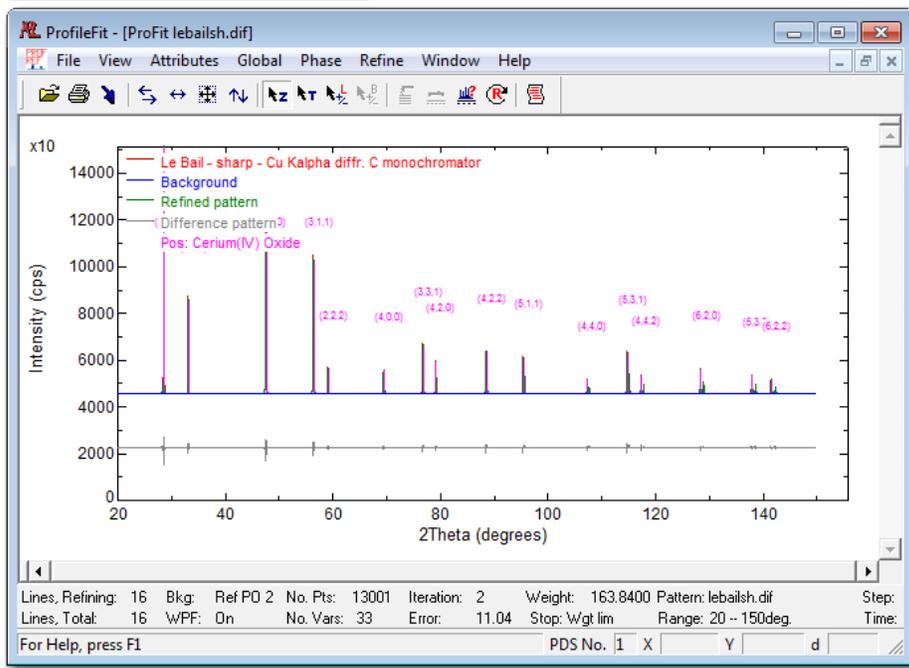
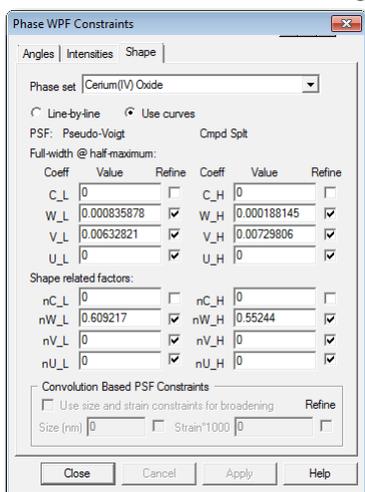


6. Set specimen displacement refinement: Select menu *Global -> Option*. Check the Ref. box for Specimen Displacement.

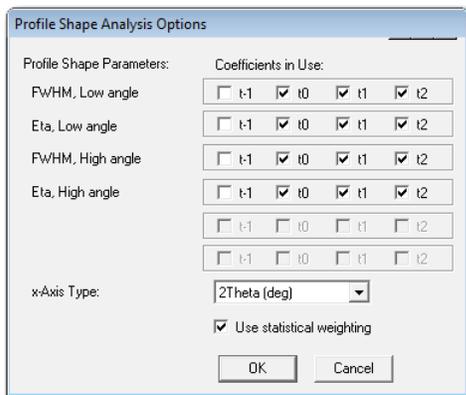


This step is not needed if individual line profile fitting is performed rather than the Whole-Pattern fitting.

7. Start the refinement by clicking on button . The error should be ~14% when finishes.
8. Select menu *Phase* -> *Whole-Pattern Fitting* (as in Step 4). On the Shape tab, check the Refine boxes as below. Start the refinement again, and it should be satisfactory with *Error*=~11%.



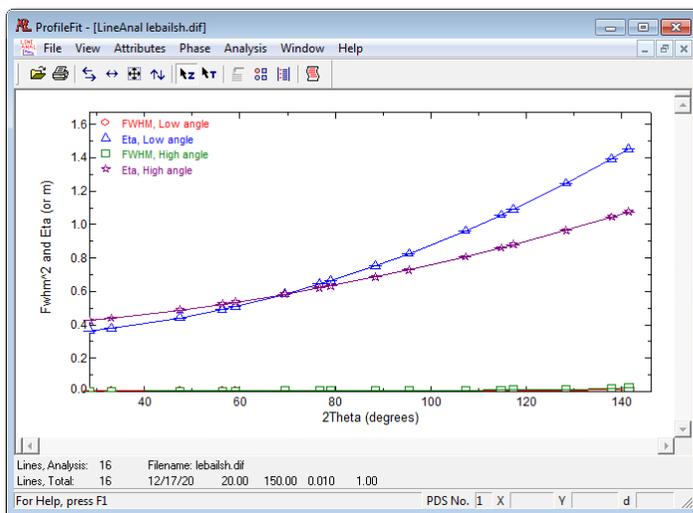
9. Select *View* -> *Line Shape Analysis*.
10. Select menu *Analysis* -> *Options*. Check all the boxes ($t_0 - t_2$) that correspond to the Shape curves.



a.
$$Fwhm^2 = c-2/\tan(\theta)^2 + c-1/\tan(\theta) + c0 + c1*\tan(\theta) + c2*\tan(\theta)^2 + c3*\tan(\theta)^3.$$

b.
$$Eta = s-2/(2\theta)^2 + s-1/(2\theta) + s0 + s1*(2\theta) + s2*(2\theta)^2 + s3*(2\theta)^3.$$

You may remove data points that have large errors, by using menu *Analysis-> Select Peaks*, and de-select those lines.

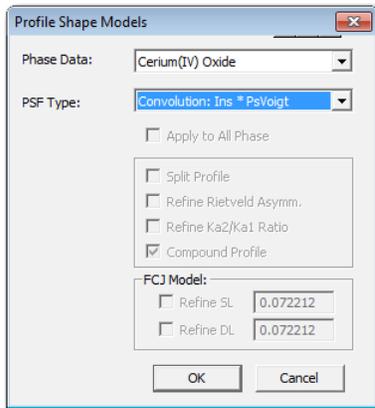


- If the plot does not look like the plot above, select menu *Attributes -> Hide Instrument Curves* to hide invalid instrument curves from showing, which may overwhelm the fitted curve.
- If the plot looks different, make sure the enabled polynomial coefficients are shown as above.
- If modification of the pattern refinement is needed, close the *Line Analysis view*, and refine the pattern. Then switch to the *Line Analysis view* again.

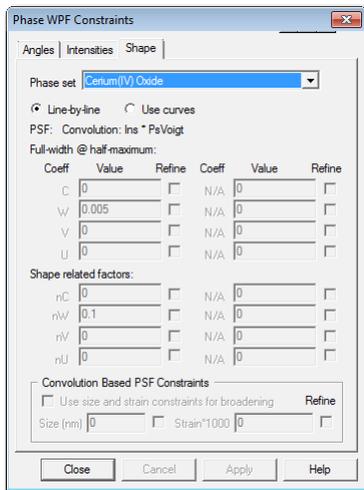
11. Save the instrument broadening curve (select menu *Analysis -> Save as new instrument broadening curves*). Note: Once the instrument curves are updated here, it remains the same for all analysis from this point on, until it is changed using the above approach.
12. Save work by selecting *File -> Save Work*. This process has been saved in *lebailsh_WPF_FitShapeCurve.pfxml* file in the example folder. It can be loaded back at a later time to generate the calibration curves again (menu *File->Load work*).

B. Warren-Averbach analysis:

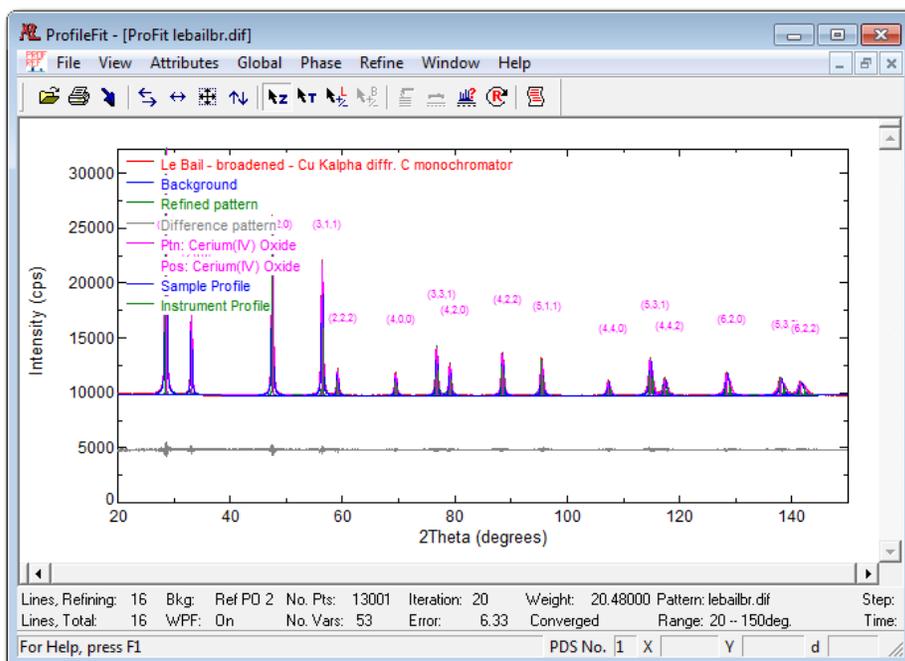
- Open a pattern for analysis. For example, *LeBailbr.dif*, found the in the same example folder as above.
- Select menu *Phase -> Add Phase from Jade PDF file*, and select *CeO2.xml*, as in the last section above.
- Change the profile model to *Convolution: Ins * Pseudo-Voigt*: Select menu *Phase -> Profile Model*.



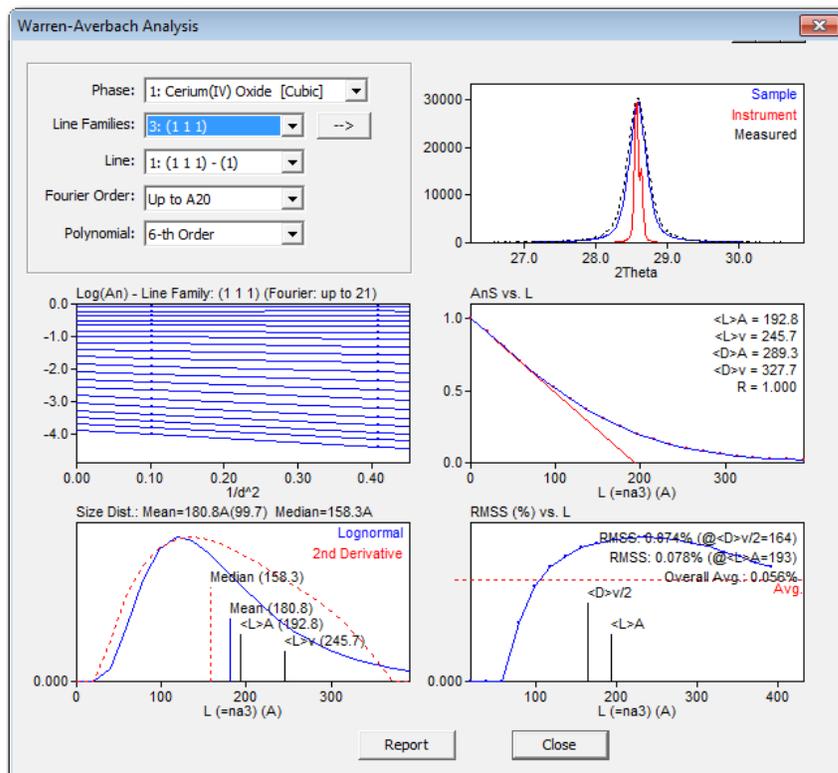
- Set to who pattern fitting: select menu *Phase -> Whole-Pattern Fitting*. The first two tabs are the same as in Step A-4, but the Shape tab is set to use *Line-by-Line*.



- Also set to refine the Specimen Displacement as in Step A-6. The background mode is the same as was used before.
- Start the refinement.

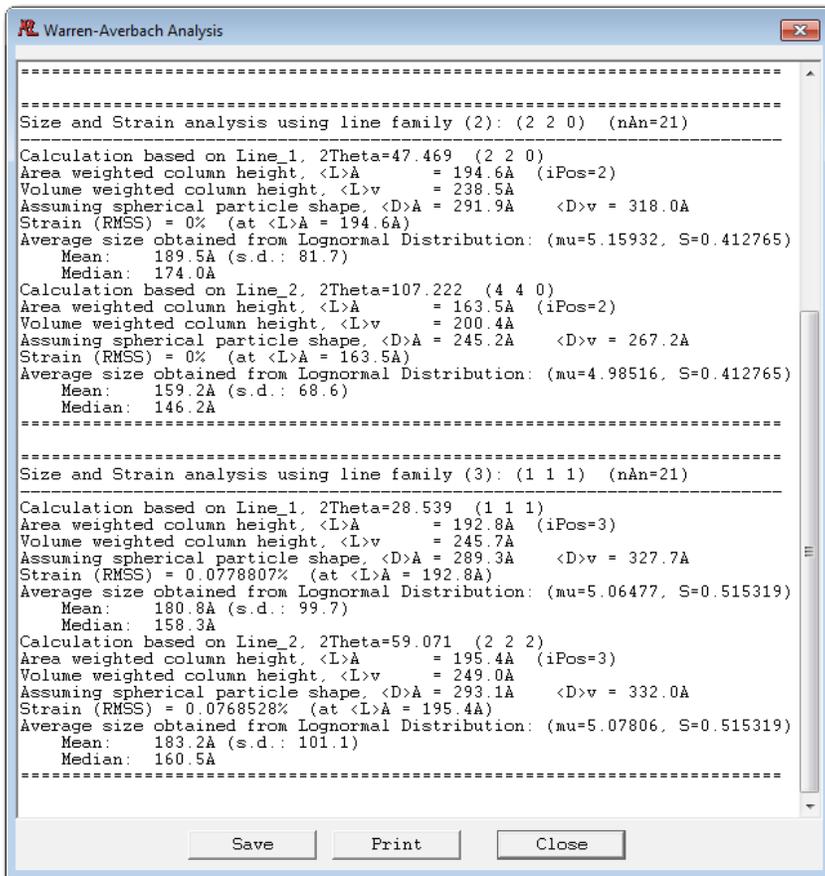


- When refinement finishes, select menu *Phase* -> *Warren-Averbach Analysis*. Note that the plot below shows the line family (111). The plots for the other line families are somewhat differently.



- The Warren-Averbach plot (logarithm of Fourier coefficients calculated from the sample profile) for the two low order reflections from the same reflection family is shown. The (111) family is shown above, that include the (111) and (222) reflections.
- The peak profile used in the calculation of size and strain is shown on upper right, which shows the peak profile for instrument, sample, and as measured.
- The AnS plot (for separation of size and strain effects):
 - Area weighted column height ($\langle L \rangle A$), calculated from the intercept of the initial slope and horizontal axis (L).
 - Volume weighted column height ($\langle L \rangle v$), calculated as twice the area under the curve.
 - Area weighted particle diameter ($\langle D \rangle A$), calculated as 1.5 times the $\langle L \rangle A$ value.
 - Volume weighted diameter ($\langle D \rangle v$), calculated as $4/3$ times of the $\langle L \rangle v$ value.
- Size distribution plot: The size distribution curve calculated as 2^{nd} order derivative of the AnS curve (red), and fitted lognormal curve (blue). The median, mean and sigma of the lognormal distribution is provided.
- RMSS (%) plot: It is the plots of the root-mean-square-strain (micro-strain) obtained from the slope of Warren-Averbach plot. This value cannot be negative. Thus, it is set to zero if it becomes less than zero.
- Functions on the dialog:
 - Line:** Select different line family for the calculation would result in different size/strain values, especially for the size distribution curve.
 - Fourier Order:** Select high enough order so that AnS vs. L plot would approach zero (or very low value). Default value is 20.

- iii. **Polynomial:** The order of polynomial used to fit AnS vs. L curve. Default: 6. Update 9th order may be selected.
- iv. **Popup menu (→):** If reflection line index (hkl) is not specified, then use this menu to specify the line families.
- v. **Report:** Show the calculation report for the Warren-Averbach analysis.

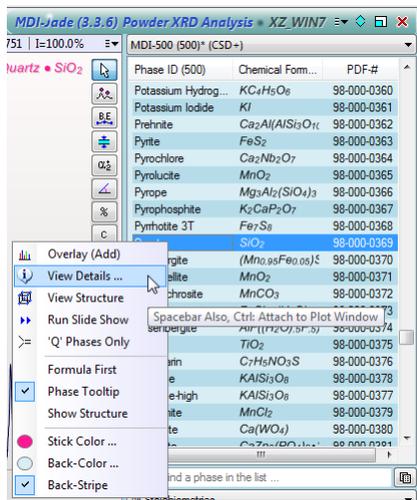


This process was also saved as a work file (lbailbr_WAanalysis.pfxml).

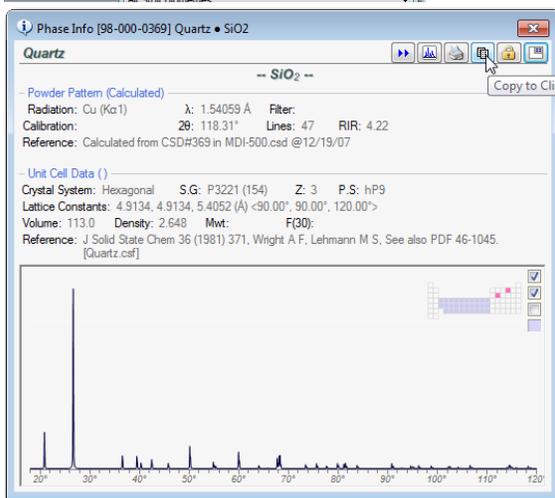
III. Importing phase data to SizeStrain for Whole-Pattern-Fitting:

SizeStrain can import line-based phase data generated from Jade 9 (classic) and JadeX. To import a phase, export the phase data (PDF) from Jade in xml format. Then select menu *Phase -> Add Phase from Jade PDF*. A phase will be appended to the phase data list if the operation is successful.

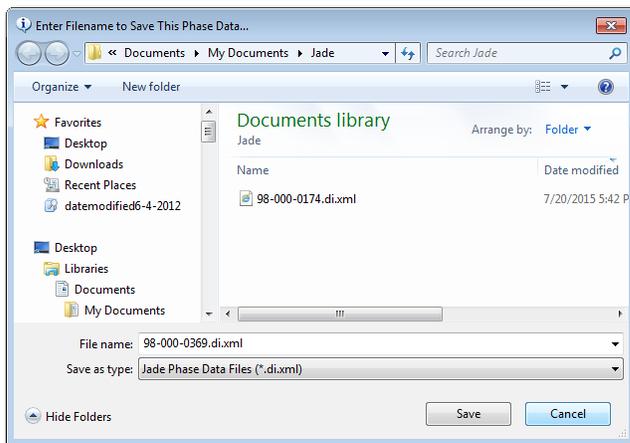
- **To export a phase from JadeX:** click a phase in the phase list (say Quartz), select *View Details* in the right-click popup menu. Then right-click on the Copy button, and select a file from the File Save dialog. A file phasename.di.xml will be saved.



a)

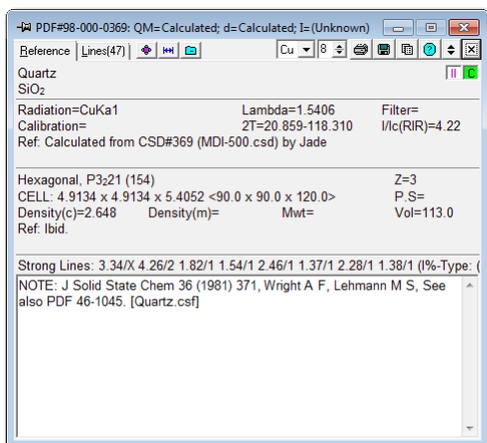


b)

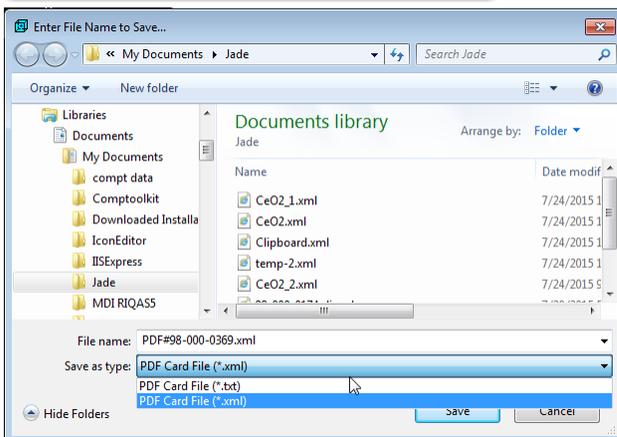


c)

- **To export a phase from Jade9:** Select a phase, and select View Details from the right-click popup menu. In the phase information dialog (a), click on the Save button, , make sure the Save as type is xml (b), then Save.



a)



b)

Known Problems:

1. Print the W-A analysis report, but not the plots.
2. The refinement may fail if the peak shape was initialized too narrow compared to those found in the pattern (manual broadening may be required – select peak editing cursor, move mouse to the peak, press the up/down key).
3. The FCJ model as implemented is very slow, so do not use it for now.

More Work:

1. Print function for W-A report with plots.
2. (done) Importing the structure-less phases from other sources.
3. (done) Implement command line switches to load pattern and procedure files for calculations.
4. Redesign the whole pattern fitting dialog. It needs to be cleaner.
5. Modify the display attributes editing dialog to use the enhanced CListCtrl object.