XRDUG Seminar IV

Mathematical Profile Fitting of XRD Data - DMSNT Operation

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XRDUG SESSION IV
Profile Fitting Using DMSNT:

The following discussion and screen shots of the software will all focus on the manual operation of the profile fitting program. Recall, in the Session III we discussed the whole pattern profile fitting and how to use this option. However, I recommend a manual profile fitting (or after the whole pattern fitting) to best fit all peaks especially those with low intensity or those near large peaks.

General Comments:

- Make sure the background is fit properly. Try to pick background points (using Spline fit) that are at true background and do not pick points that are in between peak tails but do not touch true background.

- Pearson VII is typically is the most appropriate function to use.

- Realize that the accuracy of the position of the peak inserted is dependent on the resolution of the cursor so it is important to zoom on small regions of the spectrum at a time to insert peaks.

- Selecting the curve fitting (icon with the curve function in blue) will fit all data visible in the graph. Subsequently, make sure that the data on the graph for each side of the peak or peaks is at true background. Failure to ensure true background on the extreme left and right sides of the graph area could result in serious errors in the reported peak information in the peak file.

- The closer to the data the initial peak insertions are, the quicker it will converge. More importantly, these choices can have great influence on the final fit particularly with peak overlaps that do not have nice distinct features.

- Error values reported for each convergence are dependent on many factors (e.g. step size, counting error, etc.) and should not be used to compare fits between different data or different regions. This only serves to help define the best fit (e.g. profile function, assist in

defining existence of peaks), a minimum error can indicate the best fit (be careful here), within the same region of a sample.

> Low intensity peaks will have a larger error than high intensity peaks.

> Scans with a lower count time will have a larger error compared to the same scan with a higher count time.

> Ranges on the graph with more background can increase the error value; more importantly, the range in general will affect the error value.

Basic Profile Fitting Procedure:

This procedure assumes the file is open, and a proper background fit has been accomplished; subsequently, the net intensity file is displayed.

1. Select the range using the cursor based zoom (press and hold the right mouse button and select a box of the area to zoom). Select the smallest region that includes a peak(s) with background on the extreme left and right. You may also use the **zoom window** to numerically pick the areas of interest.

2. Insert peaks using the **Insert Peak** icon (6th one from the left on the toolbar menu); you must select this icon for each peak inserted. Peaks are inserted by selecting the left FWHM, right FWHM, and center/top (figure 1). If you insert a peak on the shoulder of another in which features are a minimum, estimate the shape of the peak visually and use this visual guess to insert the points (figure 5). Figure 2 is an example of a manually inserted peak profile.

3. Select the **Curve Fitting** icon (5th from the left on the toolbar menu), and select the proper parameters; typically, **Fit KAlpha2** is selected (figure 2).

4. Select **Calculate** to begin the convergence (figure 2).

5. After convergence (figure 3) select **Save**, this only saves the data in temporary memory. In order to preserve the data on file after each

range select **File, Save** on the program menus. If this is not done and the program crashes, which it does frequently, all data will be lost and all peaks will have to be refit. Please note that the profile fitting window is the only place where the profile function parameters are available. Select **Print** if the profile functions for the peak(s) are going to be recreated.

6. Select another region and repeat the process.

7. If a peak has to be deleted, put the cursor on the green stick for the peak in question and double click the right mouse button. Select **Delete** to remove the peak (figure 4). You can also double click on a peak in the peak fitting window.

<u>Examples of Various Profile Fitting Situations:</u>

- Figure 6 shows an example of manually fitting $K_{\alpha 2}$. When the $K_{\alpha 2}$ is automatically fit (figure 3) a similar (although it is no longer independent) fit is accomplished in the background. Note the peak parameters when Fit $K_{\alpha 2}$ is chosen are based on the $K_{\alpha 1}$ peak profile for position and FWHM, however, the area is the sum of both curves.

- L/R given in various peak parameters are for the Split Pearson peak which is asymmetric and subsequently, the FWHM and shape or exponent can change independently (remember this is essentially two Pearson VII functions).

- Figure 7 shows an example of the most important utility of profile fitting, determining quantitative peak parameters from diffraction peaks that overlap. This is the famous "5 Finger of Quartz" used as XRD standard to test resolution.

- Figures 8-11 demonstrate a use of the Sigma = 1 (when selected, weighting of intensity) parameter in profile fitting. Note figure 8 shows the diffraction pattern of the 2θ region of interest with full scale intensity; **always** zoom in on the background to look for low intensity peaks as in figure 9.

  - Figure 9 shows the initial peak insertions.

o Figure 10 shows the convergence when Sigma = 1 is selected, essentially weighting higher intensities more which affects the low intensity peak fits.
o Figure 11 shows the convergence when Sigma = 1 is deselected, with a much better fit to the low intensity peaks.

- Figure 12 and 13 demonstrate the affect that step size has on the profile functions. These affect the step used in the profile function creation and subsequently, affect the area value since it appears the numeric integration may be based on this step size.