**3.014 Order-Disorder Transitions**

**Data Analysis using PANalytical X’Pert HighScore Plus v3.0a**

Before beginning work, select the menu ***View > Desktop > 3014***

**WORKING WITH MULTIPLE FILES**

Files have been created so that you can view multiple scans at the same time.

To view the in-situ measurements collected using a furnace mounted on the PANalytical X’Pert Pro diffractometer:

* 1. Select menu ***File > Open***
  2. Select the file “HTXRD CoPt.hpf” in the folder ***C:\temp\3014 CoPt F10***

To view the ex-situ measurements collected by quenching samples from different temperatures:

* 1. Select menu ***File > Open***
  2. Select the file “CoPt Quenched.hpf” in the folder ***C:\temp\3014 CoPt F10***

***!! In the Graphics Window, make sure you are in the Compare tab (at the bottom of the screen)***

* To change the color of a scan
  1. In the Lists Pane on the right-side of the screen, select the Scan List tab
  2. For any scan, double-click in the column Line Color
  3. Select a new color for the scan
* To save an image,
  1. Select menu ***File > Save As***
  2. Set the file type to *JPEG bitmap graphics (\*.jpg)*
  3. Give the file a name and save it in a folder that you will be able to find later

**WORKING WITH AN INDIVIDUAL FILE**

1. Open the experimental data
   1. To open one file at a time
      1. Select menu ***File > Open***
      2. Find the data file, with a **\*.xrdml** or **\*.raw** extension, in ***C:\temp\3014 CoPt F10***
2. Open the reference pattern
   1. Select menu ***Reference Patterns > Retrieve Pattern By > Reference Code***
   2. In the window that opens, type the reference code and then click **Load**
   3. After the reference pattern is loaded, click **Close** in the retrieval window

CoPt ordered: 99-999-0004

CoPt disordered: 99-999-0003

1. To open the reference card so that you can read information, such as the published crystal system, space group, and lattice parameter:
   1. In the Lists Pane (right side of screen) select the Pattern List tab
   2. Double-click on the entry for the reference pattern
      1. The Reference Pattern window will open
      2. When done, close the reference card by clicking on the **Close** button in the lower right corner
2. To adjust the height of the reference pattern sticks
   1. In the Lists Pane (right side of screen) select the Pattern List tab
   2. Click once on the entry for the reference pattern to highlight it
   3. In the Lists Pane (right side of screen) select the Object Inspector tab
   4. Use the **Manual Scale** slider to change the height of the reference pattern
   5. You can also change the color of the stick pattern overly in this window
3. To save the graphic image,
   1. select menu ***File > Save As …***
   2. set the file type to *JPEG bitmap graphics (\*.jpg)*
4. For the calculations in this exercise, you need to precisely determine the peak positions, intensities, and widths. This is done by profile fitting.
   1. Manually insert peaks
      1. Select menu ***Treatment > Insert Peak***
      2. Left-click in the main graphics window anywhere you would like to insert a peak.
      3. Select menu ***Treatment > Insert Peak*** to turn the cursor off
   2. Profile fit the peak
      1. Select menu ***Treatment > Profile Fit > Fit Profile > Default***
      2. You should see a calculated peak profile in blue overtop the experimental data (in red). If you don’t, select menu ***View > Display Mode > Show Calculated Profile*** to see the full profile fit peak
      3. If the calculated profile does not fit very well, refine again by selecting ***Treatment > Profile Fit > Fit Profile > Default***
   3. Read peak information
      1. In the Lists Pane (right side of screen) select the Peak List tab
      2. In the Peak List, both K-alpha1 and K-alpha2 peaks are listed. To get rid of K-alpha2 peaks from the list, right-click in the Peak List and select *Delete All K-alpha2 Peaks*
      3. For each peak, record the peak position, area, width, and (hkl).
         1. You will have to read the (hkl) from the graphics display.
         2. The peak width is labeled FHWM, “Full Width at Half Maximum”.
      4. To see additional information about any peak, double-click on that peak in the Peak List
5. To save the image,
   1. Select menu ***File > Save As***
   2. Set the file type to *JPEG bitmap graphics (\*.jpg)*
   3. Give the file a name and save it in a folder that you will be able to find later

**Optional Steps and Controls**

* Left-click and drag the mouse to zoom in; double-click to zoom out
* To read the agreement indices which tell you how good the profile fit is
  + in the Lists Pane (right side of screen) select the Refinement Control tab.
  + Double-click on “Global Variables”, which will take you to the object inspector
  + Find and expand the section **Agreement Indices**
  + **R profile** and **Weighted R profile** are the residuals that tell you how well the calculated profile fits your data (a good fit is below 10%)
* *To change the y-axis between linear and square root scale, select menu* ***View > Display Mode > Y-Axis***
* To change the widths of lines:
  + select menu *Customize > Document Settings*,
  + select the Graphics Display tab.
  + Change the widths and then click **Apply** to see the change
* To change the color of the scan:
  + in the Lists Pane (right side of screen) select the Scan List tab.
  + Double-click on the scan title, which will take you to the object inspector.
  + change the Line Color and other elements as you like

**SOME USEFUL INFORMATION THAT YOU NEED TO KNOW**

* The wavelength of radiation that we used is 1.54056 Å. This is the characteristic wavelength for Cu K-alpha1 X radiation.
* The instrument peak width is approximately B(instrument)= 0.09 degrees. To determine the broadening due to your sample:

B(sample)= B(measured) – B(instrument)

You will then use B(sample) in the Scherrer formula to calculate the crystallite size.