UC DAVIS Chemical Engineering & Materials Science

EMS-162L: Structure and Characterization of Materials Laboratory

Winter Quarter, 2004

Introduction to the X-Ray Powder Diffractometer

1. Calculate and plot the following peaks in the diffraction pattern of quartz.

Peak	d-spacing (nm)	Relative Intensity (%)
(101)	0.33420	100
Five-fingers of quartz		
(212)	0.13821	6
(203)	0.13749	7
(301)	0.13718	5

Create two plots, one for the (101) peak and spanning the 22 range from 23 to 27 degrees and the other for the 5-fingers peaks scaled from 67 to 69 degrees 22. Use a step size of 0.02 degrees 22 for both plots. For each "reflection" use the radiation listed in the table below:

Characteristic Radiation	Relative Intensity (%)
Cu- $k_{\alpha 1}$	100
Cu-$k_{\alpha 2}$	45
Cu-k_{β}	30
W-L_{α}	10

Use the Cauchy profile for each peak. The Cauchy profile is given by

$$I = \frac{1}{1 + \sigma^2 (2\theta - 2\theta_B)^2}$$

where F is the width of the peak and 2_B is the Bragg angle. Peak width is a function of 22 given by the expression F=50-0.37@22_B. (Note that this equation if valid only for this exercise.)

To obtain the correct relative intensity in your plots multiply each Cauchy profile by the relative intensity of the characteristic radiation times 100. Then, sum each profile for a given reflection and multiply this by the relative intensity of the reflection. Finally, sum each profile to obtain the total intensity for the diffraction pattern.

2. When the x-ray source is operated at potentials higher than that required to knock out an inner shell electron it will be possible to generate characteristic x-rays. As this voltage is increased the probability that this will happen increases and therefore the efficiency of the source increases. But how will adjusting the tube current effect the amount of x-rays produced and how will increasing it effect the intensity of diffracted x-rays reaching at the detector?

3. Using wide slits on the detector will increase the measured intensity of the diffraction peaks by allowing more x-rays to enter the detector to be counted. For instance, the intensity of the (101) peak for quartz will increase from 7500 to over 20,000 cps when replacing a 0.1 mm receiving slit with a 0.3 mm receiving slit. But what effect will this have on the 22-resolution? Make a sketch that illustrates your answer.

4. Another way to increase the number of x-ray photons that strike the active element in the detector is to scan the 22 range of interest more slowly. While this will not increase the measured intensity, whose units are counts per second, it will increase the total number of counts at each 22 position. How do you think this will affect the quality of your results?

5. Make a good, full-page sketch of the diffractometer you are using. Show the following:
   a. x-ray source
   b. specimen
   c. detector
   d. scatter and divergence slits
   e. scatter and receiving slits
   f. $k_{\$}$ filter
   g. the angles theta, omega and phi (2, T and N)

   Label each part and each angle and note the radius of the goniometer, the width of the slits the type of filter used, type of detector and type of x-ray source.