Materials Science 142

Assigned: 5/17/06

Laboratory Exercises 6 Due: 5/24/06

Instrument Peak Profile and Particle Size Analysis

Goal: Determination of the particle size and possible microstrain in a sample. Evaluate the various contributions to the instrumental peak broadening.

Part A: Instrument Calibration

You are provided with a sample of lanthanum hexaboride (LaB_6) In principle, the NIST standard reference material (SRM) 660 is the preferred peak shape standard because it is prepared so as to have no microstrain and a particle size that is large enough to prevent particle size broadening yet small enough to ensure randomization of the diffraction orientations. At the time of this writing, it was unclear whether SRM 660 would be available or conventional LaB_6.

1) Prepare the sample and collect diffraction data over the 2Θ range 15 to 150°. Record the instrument settings.
2) Using the X-Pert Highscore software, analyze the peak profiles. Strip the $K\alpha_2$ contribution from the peaks and turn on the 'shape parameter' for the profile fitting. This shape parameter corresponds to η as defined in class, implying that the pseudo-Voigt function is used.
3) Record the total peak integral breadth, the total full width half max, the area and the intensity for each peak. Record also (or compute) the Gaussian and Lorentzian peak breadths.
4) Plot β_{tot}, β_G, and β_L as functions of 2Θ. Also plot the shape parameter as a function of 2Θ. Comment on the form of these plots.
5) Fit β_{tot}, β_G, and β_L to polynomials in 2Θ to serve as calibration curves for the instrumental contribution to the broadening.

Part B: True Sample Contributions to Peak Broadening

You are provided with a sample of nickel with an unknown but small particle size. The material may also have microstrain.

1) Prepare the sample and collect diffraction data over a 2Θ range that is appropriate for nickel, and using the instrument settings under which the LaB_6 data were collected.

2) Using the X-Pert Highscore software, analyze the peak profiles. Strip the $K\alpha_2$ contribution from the peaks and turn on the 'shape parameter' for the profile fitting.

3) Record the total peak integral breadth, the total full width half max, the area and the intensity for each peak. Record also (or compute) the Gaussian and Lorentzian peak breadths. *See appendix.*

Data Analysis

4) Use the Warren method to obtain the peak broadening due to the sample, in which it is assumed that peak widths add as Gaussians:

$$(\Gamma_{sample})^2 = (\Gamma_{meas})^2 - (\Gamma_{instr})^2 \qquad \text{Warren relation}$$

Here, use the FWMH for peaks measured before deconvolution. Use the calibration curve established in part A to obtain Γ_{instr} at the same 2Θ as the sample peak positions.

5) Use the Scherer method to obtain the peak broadening due to the sample, in which it is assumed that peak widths add as Lorentzians:

$$\Gamma_{sample} = \Gamma_{meas} - \Gamma_{instr} \qquad \text{Scherrer relation}$$

Use similar procedures as in step (4).

6) Prepare Williamson-Hall plots using the Γ_{sample} values determined in steps (4) and (5). That is, prepare plots of $\Gamma_{sample}\times\cos(\theta)$ vs $\sin(\theta)$ and evaluate the intercepts and slopes to obtain the mean particle size and the mean strain. Comment on the differences in the results.

Now use the deconvoluted peak breadths to obtain the particle size and microstrain.

7) Evaluate $\Gamma_{sample,L}$ and $\Gamma_{sample,G}$ using the deconvoluted calibration curves and deconvoluted $\Gamma_{meas,L}$ and $\Gamma_{meas,G}$ values.

8) Taking $\Gamma_{sample,L}$ to be due to particle size effects and $\Gamma_{sample,G}$ to microstrain, calculate the mean particle size and the microstrain. Do $\Gamma_{sample,L}$ and $\Gamma_{sample,G}$ exhibit their expected dependences on diffraction angle?

Part C: Instrumental Effects on Peak Broadening

Using LaB_6, investigate the influence of diffractometer instrument settings, in particular, the divergence slit, the antiscatter slit and the beam mask, on peak broadening. In all cases, collect diffraction data from 15° to 150° in 2Θ.

The divergence slits control the equatorial divergence of the incident beam and thus the length of the sample that is irradiated.

The beam masks control the axial width of the incident beam and thus the width of the sample that is irradiated.

The antiscatter slits limit stray radiation from reaching the detector.

Default values are: divergence slit = ½, anti-scatter slit = 1, and beam mask = 10.

1) Keep the beam mask and anti-scatter slit at their usual values. Collect diffraction data for various divergence slits: 1/8, [½], 1, 3/2, 2.
2) Keep the divergence slit and beam mask at their usual values. Collect diffraction data for various anti-scatter slits: [1], 2.
3) Keep the anti-scatter slit and the divergence slit at their usual values. Collect diffraction data for various beam masks: 5, [10], 20.
4) Using the X-Pert Highscore software, analyze the peak profiles. Strip the $K\alpha_2$ contribution from the peaks and turn on the 'shape parameter' for the profile fitting.
5) Record the total peak integral breadth, the total full width half max, the area and the intensity for each peak. Record also (or compute) the Gaussian and Lorentzian peak breadths.
6) Prepare plots as necessary to illustrate the most important influences on peak profiles. You may wish to comment on changes in intensity as well as changes in peak breadths.

Appendix. Relationship between Lorentzian, Gaussian and Voigt peak breadths:

$$\frac{\beta_L}{\beta_V} = 2.0207 - 0.4803\phi - 1.7756\ \phi^2$$

$$\frac{\beta_G}{\beta_V} = 0.6420 + 1.4187(\phi - \frac{2}{\pi})^{1/2} - 2.2043\phi + 1.8706\ \phi^2$$

$$\phi = \frac{\Gamma_V}{\beta_V} \qquad\qquad \frac{2}{\pi} \le \frac{\Gamma_V}{\beta_V} \le \frac{2\left(\ln(2)\right)^{1/2}}{\pi^{1/2}}$$