Materials Science 142

Applications of Diffraction Techniques in Materials Science

Assigned: 4/26/06

Laboratory Exercises 3 Due: 5/03/06, 5pm

Precision Lattice Parameter Determination

Goals: Determine the lattice constant of a cubic material to high precision using both extrapolation methods and an internal standard; determine the primary source(s) of errors in peak position measurement.

Gain experience in scientific writing – *submit as a complete lab report*.

Part A: Powder diffraction data will be collected from $Cs_6(H_2SO_4)_3(H_{1.5}PO_4)_4$ (space group: I-43d, a_o ~ 14.5 Å) using the Philips X'Pert Plus diffractometer and Cu Kα radiation: $K\alpha_1$ = 1.5406 Å, $K\alpha_2$ = 1.5444 Å.

1) Prepare the sample (without an internal standard) as instructed by the TA and collect data over the 2θ range 10 to 60°. Record the diffractometer settings used (current, voltage, step size, dwell time).

2) Peform a peak search using the X-Pert Plus software.

3) Eliminate $K\alpha_2$ peaks.

4) Save the peak list as a text file.

5) Index the peaks using the approximate lattice constant. Note that some peaks may be absent due to the relfection conditions, and others may be absent simply because the intensities are (coincidentally) low. Reflection conditions: $h+k+l = 2n$; for hhl peaks, $2h+1 = 4n$; for h00 peaks, h = 4n. Idenfity any peaks that cannot be indexed, and by definition, must be due to an impurity phase.

6) Calculate the lattice constant, a, for each peak.

7) Prepare plots of (i) a vs $\cos^2(\theta)/\sin(\theta)$and (ii) a vs $\cot^2\theta$. Establish the best value for a_o by extrapolation (estimate the error). Comparing the linearity of these plots, identify the most dominant error in the measurement.

Report: Show the raw diffraction data with labels for the peak indices. Prepare a table of $2\theta_{obs}$, intensity, peak index, and *a* for each peak. Show the plots requested in step (7) and report the results requested in this step.

Part B: Analyze the diffraction pattern of the same material making use of an internal 2θ standard.

8) Compare the measured 2θ values of the Si peaks with their expected positions. Prepare a plot of $2\theta_{obs} - 2\theta_{calc}$ vs. $2\theta_{obs}$. Fit an nth order

polynomial (select the value of n that is most appropriate) to this plot. This polynomial will serve as a $2\theta_{obs}$ correction function.

9) Apply the $2\theta_{obs}$ correction function to the measured values of 2θ for the material.

10) Using the indexing established above, calculate the lattice constant for each peak, then calculate the average value of *a* and the standard deviation.

11) Prepare plots of (i) *a* vs $\cos^2(\theta)/\sin(\theta)$and (ii) *a* vs $\cot^2\theta$. Comment on the appropriateness of using both an extrapolation and an internal standard simultaneously.

Report: Prepare a table of $2\theta_{obs}$ and $2\theta_{calc}$ for Si and show the calibration plot. Also prepare a table of $2\theta_{obs}$, $2\theta_{corr}$ and $2\theta_{calc}$ for the material, where $2\theta_{corr}$ is the $2\theta_{obs}$ with the calibration correction applied, and $2\theta_{calc}$ is the value derived using the average lattice constant. Give the indices for each peak, and indicate unobserved and unindexable peaks. Give also the values of d_{obs}, d_{corr} and d_{calc} for each peak. Report the results of steps (9) and (10).

Part C: Determine the extent of sample displacement using extrapolation methods. Data have been previously collected from sintered alumina standard, corundum phase, intentionally displaced by specified amounts. A Siemens D500 instrument with Cu $K\alpha$ radiation was used.

1) Perform a peak search; eliminate $K\alpha2$ and $K\beta$ (1.3922 Å) peaks. The Siemens D500 is not equipped with a monochromator to eliminate $K\beta$ peaks.

2) Search the ICSD database to find the expected 2θ (and hence d) positions of the diffraction peaks of corundum.

3) Using the relationship:

$\Delta d/d = -(S/R)\cos^2(\theta)/\sin(\theta)$ and R = 200.5 mm for the D500

calculate the sample displacement. Estimate the error in this value.

Report: Turn in appropriate plots, derive the slope and the intercept. Report the sample displacement for each pattern.