

Materials Science 142

Applications of Diffraction Techniques in Materials Science

Laboratory Exercises 4

Assigned: 5/03/06

Due: 5/10/06, 5pm

Ab initio Unit Cell Determination

Goal: Determine, for a completely unknown compound, the lattice type and lattice parameters. Seven sets of data are to be analyzed. Of these seven, five are cubic. Of the cubic patterns, one has systematic absences (due to a glide plane or screw axis). The other two patterns may or may not have systematic absences.

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

Procedures: You will collect six out of the seven datasets as part of the lab using the Phillips diffractometer (Cu $K\alpha$ radiation, $K\alpha_1 = 1.5406 \text{ \AA}$, and $K\alpha_2 = 1.5444 \text{ \AA}$) using an internal 2θ standard. The seventh dataset has been collected on the inel diffractometer (also Cu $K\alpha$ radiation) using silicon as an internal 2θ standard.

- 1) Prepare the samples (using an internal 2θ 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). Each student will collect data for at least one sample. Name the datasets 'example1' etc. as identified and exchange data after all data collection is complete.
- 2) Perform a peak search using the X'Pert Plus software and export the peak list to format for use in a spreadsheet or other program. The dataset 'example7.mdi' can be imported directly into the software for analysis.
- 3) For at least one of the datasets, eliminate $K\alpha_2$ peaks from your data using the relationship:
$$\lambda_1 / \lambda_2 = \sin(\theta_1) / \sin(\theta_2)$$
to establish which peaks are due to $K\alpha_2$. For the rest, you may use the X'Pert Plus software to eliminate these peaks before exporting the peak list.
- 4) Prepare and apply a 2θ calibration correction. For at least on the samples, prepare the calibration curve manually. For the remainder, you may use the X'Pert Plus software to do this automatically before exporting the peak list.
- 5) Calculate $\sin^2\theta$ for each peak.
- 6) Test for cubic lattices: prepare a table listing line #, $\sin^2\theta$, s(SC), s(BCC) and s(FCC); divide the $\sin^2\theta$ column by the s columns and establish which

yields a constant value of $\sin^2\theta/s$. Keep in mind that some peaks may be missing because they are of weak or zero intensity. If the lattice does not appear cubic, go on to step 7.

- 7) Test for hexagonal lattices: divide each $\sin^2\theta$ by the $\sin^2\theta(1)$. See if any peaks can be identified as (hk0) lines. If not, repeat the procedure by dividing each $\sin^2\theta$ by $\sin^2\theta(2)$, etc.
- 8) Test for tetragonal lattices: the procedure is the same as that for tetragonal, but the expected sequence of s values are different.

There are no cells of lower symmetry in the examples. But beware of systematic absences!

The seventh dataset has been collected from the material ZnCrSb. The material has a density of 7.33 g/cm³.

Report: Prepare a table of $2\Theta_{\text{obs}}$, d_{obs} , $2\Theta_{\text{calc}}$, d_{cal} hkl for each pattern (using the calibration corrected values for $2\Theta_{\text{obs}}$) for only the $K\alpha_1$ peaks. Identify the lattice type and give the lattice constant(s). For the cubic pattern that has systematic absences, give the absence condition (e.g. h 0 0, $h = 2n + 1$) and the symmetry operation that gives rise to these absences. In an appendix provide the manually determined 2Θ calibration curve and an indication of the $K\alpha_2$ peaks for the one sample that was manually evaluated.

HW: Show, using structure factor calculations, that the following symmetry operations imply the indicated absences:

1. 2_1 along [1 0 0] $\Rightarrow h = 2n + 1$ absent for (h 0 0)
2. 4_1 along [0 0 1] $\Rightarrow l = 2n + 1$ absent for (0 0 l)
3. d in (0 0 1) plane, along [1 1 0] $\Rightarrow h + k = 2n + 1$ absent for (h k 0)
4. n in (0 0 1) plane, along [1 1 0] $\Rightarrow h + k \neq 4n$ absent for (h k 0)