Ge 116

Lab project Week 9: Preparation for X-Ray Powder Diffraction

Report due March 18, 2013

Part 1

A. Radiation Safety Training

Obtain Radiation Safety training from the Safety Office (Haick Issaian, Radiation Safety Officer). *Completed on Monday 3-4pm.*

**Complete the take-home quiz and bring it to class or lab by the time we meet for XRD measurements.**

B. Sample Preparation

Meet in 352A Arms. Powder X-ray Diffraction relies, in part, on two assumptions: that the individual grains are randomly oriented, and that there are enough randomly oriented grains that we sample all lattice reflections. To achieve these conditions, we want to prepare powders roughly the consistency of flour (grain size of ~100 m down to ~2 m).

Crush and weigh out “Unknown 0” and “Unknown 6” provided by the GPS working collection. We’ll aim for about 30 mg of sample, though powder spectra can be obtained on samples as small as a few mg. First, measure out a portion of ~30 mg of uncrushed sample, then crush it into a powder under acetone, then grind under acetone in an agate mortar to the correct consistency (flour). For ultra-purity, grind under ethanol.

Measure the average grain size of your samples on the microscope in the adjacent room. Also, take a small portion of the ground bulk sample and unground NaCl and measure the average grain size for each. Note them in the table below:

|  |  |  |
| --- | --- | --- |
| **Sample** | **Average grain size (m)** | **Range of grain sizes/Notes** |
| Unknown 0 |  |  |
| Unknown 6 |  |  |
| Ground bulk-rock |  |  |
| Unground NaCl |  |  |

We will use the already prepared (ground) bulk sample and NaCl. When finished, clean mortar and pestle by grinding in amorphous silica at least three times.

**Store your sample in a labeled vial.**

**Part 2.**

C. X-Ray Powder Diffraction Experiments

339 Steele. *We will be in a lab facility, closed-toed shoes are required.* We will be using the Haile Group Inel CPS-120 powder diffractometer system equipped with a conventional X-ray source (Cu Kα). We will be guests in Prof. Sossina Haile’s lab and want to be as thorough and clean as possible.

The x-ray source is usually left in standby mode—40 kV, 10 mA, with a water cooling system left on.

Familiarize yourself with how the machine works, from sample mounting to data collection. There is a step-by-step procedure written out that one can look through. Mount each sample on the provided holder, packing your sample in the entire volume, and place it on the sample stage. Connect to the XRD using the *X-pert Data Collector*, and use it to ramp up the voltage/current to 45 kV/40mA.

Login/password: geology/geology

The most recently created collection program for geologists (geo4) is fine for for NaCl (2θ range: 5° to 75°, 0.01 step size, 10 second dwell time). Using this configuration, measure an x-ray diffraction spectrum for each of your samples. We may use a longer dwell time for the unknowns and bulk rock sample.

Run: NaCl unground, NaCl ground (ground 02/08/2012 at ~11am CST), Unknown 0, Unknown 6, and bulk sample. Note the figure and table on page 3.

When finished, return the machine to standby and sign the logbook. Make sure your sample area is clean before you leave. Email the data files to yourself.

D. Data Analysis

**Now, either in this lab or on the offline computer in the microprobe lab, identify your unknowns.** The *X-pert high score* software is equipped with a searchable powder diffraction database. Use the software to subtract the background, eliminate Kα2 peaks, match up your unknowns, save the peak lists as a text file, and export the raw NaCl data (choose “\*.rd” or “\*.di” formats).

1. *NaCl—salt*.NaCl forms crystals that are face-centered-cubic (“*fcc*”, space group Fm3m). In a cubic system, lattice constants for each face-edge are equivalent. (that is, a=b=c, implying (002) =(200) = (020) ).
	1. **Index your NaCl spectra (ground & unground) with the help of Table 1: match the (*hkl*) reflections with your peaks. Fill in Table 1. Calculate the lattice parameter, “a” (in units of Å), from each reflection.**
	2. **Determine the volume of NaCl (ground & unground) and describe your procedure.**
	3. **Determine the density of NaCl (ground & unground) in units of g/cm3.**
	4. **Comment on the differences in the XRD spectra of the unground NaCl sample and the ground NaCl sample: peak intensities, relative peak intensities, peak widths, peak positions, and other features you may notice.**
2. *Unknown O.* I**dentify this mineral using the peak matching analysis software. Note the lines of different phases that the program suggests and the peaks of your data.** This mineral is a phosphate that you analyzed using EMPA and SEM; it forms crystals with hexagonal symmetry (space group P63/m).
3. *Unknown 6.* **Identify this mineral using the peak matching software** Note the lines of different phases that the program suggests and the peaks of your data. Recall the color of the unground sample. It is a magnesium silicate containing some iron.
4. *Bulk Rock.* **Using the mineral list from your EMPA analyses as an aid, identify the peaks associated with each mineral.** **Are there peaks you have not identified? If so, make an attempt to explain them.**
5. **In your report, include all the XRD spectra you collected, including your indexed NaCl spectra (ground and unground) from question 1a.**
6. **List the strengths (at least 3) and weaknesses (at least 2) of X-ray diffraction.**

Please contact either Ted Present (tpresent@caltech.edu) or Jennifer Jackson (jackson@gps.caltech.edu) if you have any questions.



**Figure 1.** Recall that X-rays interact with electric field surrounding the atoms. The movement of the electron cloud re-radiates waves with the same frequency as the X-rays (elastic scattering). Those waves interfere with one another creating constructive and destructive interferences which is the very phenomenon we are going to measure in the x-ray diffraction lab. The nature of this interference can be described by Bragg’s law, which is a kinematic description that states that the interference is constructive when the phase shift is proportional to 2π. This gives the relation n = 2dsin

**Table 1.** The first six allowed reflections for NaCl, an *fcc* lattice, listed with increasing 2

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **(hkl)** | **Reflection** | **2**ground, unground | **d-spacing (Å)**ground, unground | **a (Å)**ground, unground |
| (111) | Very Weak |  |  |  |
| (200) | Strong |  |  |  |
| (220) | Strong |  |  |  |
| (311) | Weak |  |  |  |
| (222) | Strong |  |  |  |
| (400) | Strong |  |  |  |