Materials Science 142

Assigned: 5/10/06

Laboratory Exercises 5 Due: 5/17/06

Phase Analysis

Goal: Determination of the relative amounts of two substances in a mixture.

Part A: Effect of Absorption on Peak Intensities & Semi-Quantitative Analysis

The absolute intensity of any peak in a diffraction pattern depends on the absorption coefficient of the mixture. Collect diffraction data from 2-component mixtures of the following four compounds: quartz (SiO_2), magnesium oxide (MgO), barium carbonate ($BaCO_3$) and boron carbide (B_4C).

1) Calculate the mass absorption coefficients, μ_m, and the linear absorption coefficients, μ, of the compounds from:

$$\mu_m = \sum_n w_n \mu_m^n \qquad \mu = \mu_m \rho$$

where w_n is the weight fraction of the element n in the compound,
μ_m^n is the mass absorption coefficient of the element n, and
ρ is the density of the compound.

ρ(quartz) = 2.648 ρ(MgO) = 3.610 $\rho(BaCO_3)$ = 4.287 $\rho(B_4C)$ = 2.50 g/cc

Use samples with the following compositions (wt%)

SiO_2 + MgO	25:75	50:50	75:25	unknown
SiO_2 + $BaCO_3$	25:75	50:50	75:25	unknown
SiO_2 + B_4C	25:75	50:50	75:25	unknown
pure SiO_2				

2) Using the software of your choice, measure the integrated intensity of the quartz peak, I_Q, at ~ 3.315Å.

3) Prepare a plot of $I_Q/I_{Q,pure}$ vs composition for the three sets of mixtures, where $I_{Q,pure}$ is the intensity of the 3.315Å peak in the pure SiO_2 sample.

3) The curve $I_Q/I_{Q,\,pure}$ should follow the equation:

$$\frac{I_Q}{I_{Q,pure}} = \frac{w_Q(\mu_Q^m)}{w_Q(\mu_Q^m - \mu_Z^m) + \mu_Z^m}$$

where the subscript Z refers to the second compound in the mixture.

Plot this calculated curve on your figure of $I_Q/I_{Q,\,pure}$ vs composition. How good is the agreement? What might cause errors?

4) Use these curves to determine the compositions of the three unknowns. Alternatively, calculate the weight fraction of SiO_2 in the unknowns directly.

Part B: Quantitative Analysis

A more precise method of determining the composition of a mixture is to use an internal standard. Data have been collected from mixtures of Cu and Ta, using Mo as an internal standard. You will be comparing the ratio of intensities ($I/I_{standard}$) of peaks diffracted from these materials in order to determine the weight fractions.

The data have been collected from samples with the following compositions (wt%)

Cu:Ta:Mo 20:50:30 35:35:30 50:20:30

(unknown Cu:Ta):Mo = 70:30

1) Using the software program of your choice, perform a peak search, eliminating $K\alpha_2$ peaks.
2) Identify the peaks due to each of the materials:

   Ta: BCC, a = 3.306Å Mo: BCC, a = 3.147Å Cu: FCC, a = 3.615Å
3) For the 20:50:30 sample, generate the RIR for Cu vs Mo using 4 combinations of Cu and Mo peaks (2 from each material). The calculated RIR for each combination will be numerically identical, but use the error bars on the I^{rel} measurements to estimate an error bar for the RIR.
4) Using the software of your choice, measure the intensities of the highest intensity peaks of Ta, Cu and Mo in each sample.
5) In order to determine the composition of the unknown, generate a calibration curve by plotting $I_{Cu}/I_{standard}$ vs X_{Cu} and $I_{Ta}/I_{standard}$ vs X_{Ta} (using the mixtures with known composition). This should be a linear relationship (passing through the origin), from which you can determine the average RIR relative to Mo and the composition of the unknown.
6) Knowing that the total weight per cent must sum to 100, estimate the error in your calculation. Also estimate the error from the variability in your measured RIR (step 5). Comment on possible sources of errors.