Lab Book: X-Ray Diffraction

Elisa Jacquet, Alev Orfi, An Vuong

09/01:

- Learnt how to use the x-ray diffractometer and set up the programs
- Samples need to be analysed
    - 1 mystery sample
    - 2 Cu3Au alloys
    - CuNi and PbSn alloys

- Diffractometer parameter1 (param1 from now on) file:
    - Scantype: locked coupled
    - Scan mode : step scan
    - step size (for 2 theta): 0.05deg / 2s
    - initial 2 theta angle: 10 deg
    - final 2 theta angle: 110 deg
    - default generator: 40kV and 40mA

- Wavelength of the diffractometer that is incident upon our sample is: lambda = 0.154nm

Data Collection Step #1:

- Obtain the data for CuNi and PbSn alloys using parameter 1 set-up
    - The data we obtain from the x-ray diffractometer is: the number of x-rays incident upon the detector when the detector is at a particular angle relative to the sample.
    - We call the angle of the detector 2 theta.
    - The way in which we set up the equipment, the detector moves in increments of 0.05 degrees and counts the x-rays for 2 seconds at each position.

Data Collection Step #2:

- Collect data for the rest of the samples: 2 Cu3Au samples with parameter1 set-up

Data Collection Step #3:

- If we need to, do another run of data collection on all samples by focusing the diffractometer to collect more data points around the peaks.

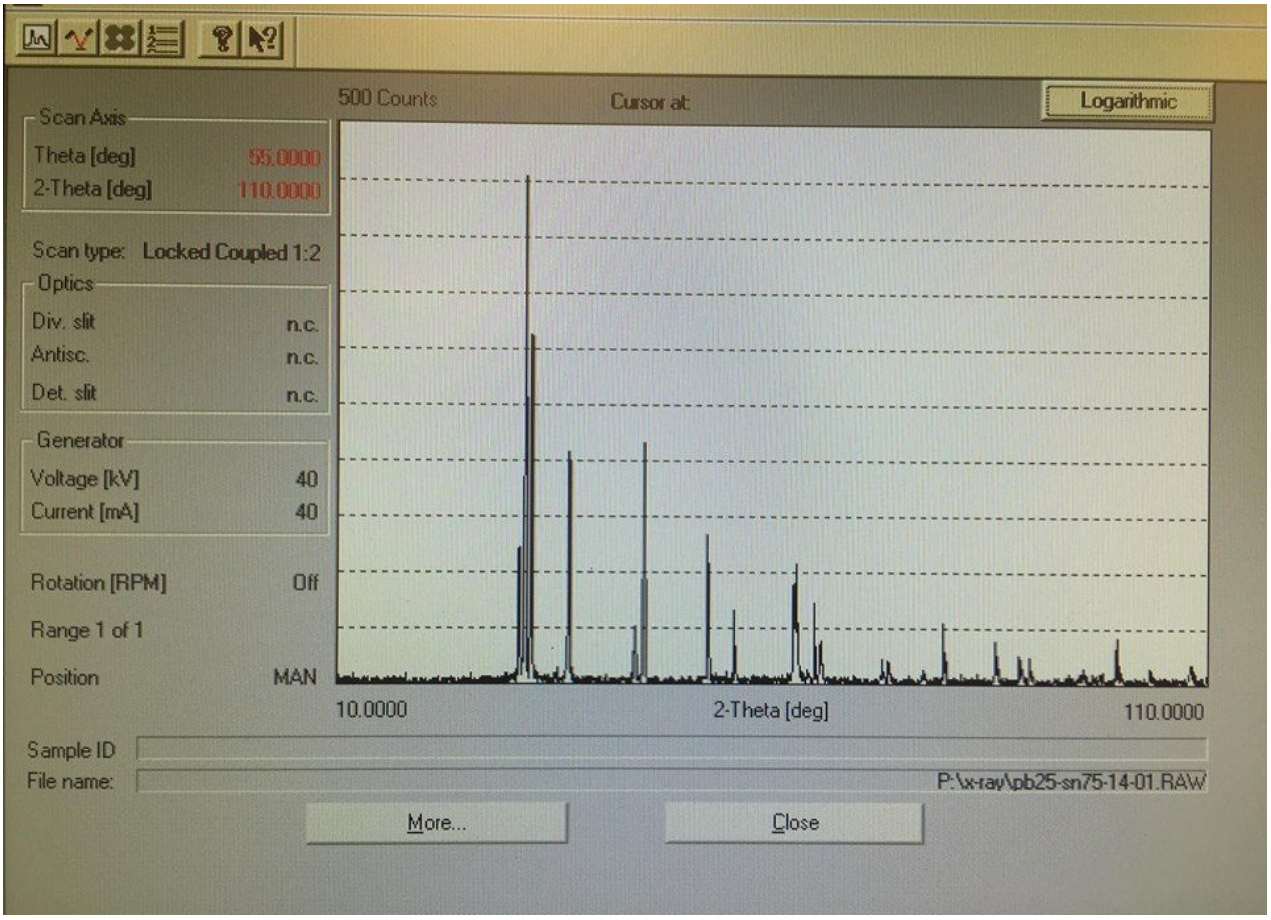
Data Analysis Step #1:

- Solve for the lattice structure of the CuNi and PbSn alloys :
    - Calculate “a”: the lattice constant
    - The data will show peaks in counts at particular values of 2*theta. From this, we can solve for “a” and for the miller indices.
- Need to get an idea of how the different concentrations of metals within the alloy reflects on the data and miller indices / lattice constants. -> place to start: plot the counts vs angle for each alloy on the same set of axes to compare.
- Starting point: conduct analysis for 100% Cu to get the code running and the fit working. (Work on automating the process for the other trials).

Samples run in the day : (using param1)

- Cu 100%
- Ni 100%
- Cu 75% , Ni 25%
- Cu 50%, Ni 50%

Example of monitoring data as diffractometer is running:



- The angle 2 theta at each peak is what we will need in order to solve for the lattice constant and miller indices.

10/01:

Samples run in the day : (using param1)

- Cu 25%, Ni 75%
- Pb 100%
- Sn 100%
- Pb 75%, Sn 25%

14/01:

Samples run in the day: (using param1)

- Pb 50%, Sn 50%
- Pb 25%, Sn 75%
- Cu3Au sample A
- Cu3Au sample B

Miller Indices and Characterisation of lattices:

Copper is FCC
Nikel is FCC
Lead is FCC
Gold is FCC
Tin is Cubic Diamond structure

For FCC, h, k and l must be either all odd or all even to have peaks
Combinations are: 111, 200, 220, 222

01/23:

Observations:

From looking at the PbSn graphs we can see that the Pb and Sn exist in different sections in the lattice. The 100% Pb showed certain peaks and the 100%Sn showed others and the alloys showed all the peaks. This is different than the CuNi alloys where the peaks only moved position, indicating a change in *a*.

Updated Goals:

1. While graphing the PbSn it's evident we need more data points to obtain good resolution of the double peaks. Goal: go back to the lab and rerun with new parameters
2. Try plotting voigt on double peak (addition of two voigt functions)
3. Get "a" values for all the different alloys - CuNi and PbSn: see how the value of "a" changes for each alloy, an explanation why, and what the structure looks like.
4. Compare our values of "a" with accepted data - especially for pure copper and pure nickel
5. Gold/Copper alloy:
    a. Each sample (A and B) was made with different methods and so they should have a different structure.
    b. Find the structure of each sample and compare them

01/24:

- New data collection for 100% Pb alloy with new parameter file (x-ray diffractometer set up)
    - NOTE: this takes 3hr to run so need to give enough time to run through all PbSn samples !
- Param2 (new set-up for experiment):
  - Scantype: locked coupled
  - Scan mode : step scan
  - step size (for 2 theta): 0.025deg / 3s
  - initial 2 theta angle: 10 deg
  - final 2 theta angle: 110 deg
  - default generator: 40kV and 40mA

- After talking to Robert Gagnon, decided it is better to both decrease the step size in the diffractometer and increase the amount of time for each step. This is the best way to minimize noise and make the peaks really stand out.

01/28:

Peak Fitting Tutorial:

- Reduced Chi^2 values should be as close to 1 as possible.
- The single - peak fits that we did for CuNi alloys are actually not as good as we thought: need to go over the code and write a new fitting function to fit for double peaks
    - This comes from the fact that the beam incident upon the sample in the x-ray diffractometer is not really monochromatic since it contains the two k(alpha) copper wavelengths.
    - We need to treat all the peaks as double peaks.

Vergard's Law:

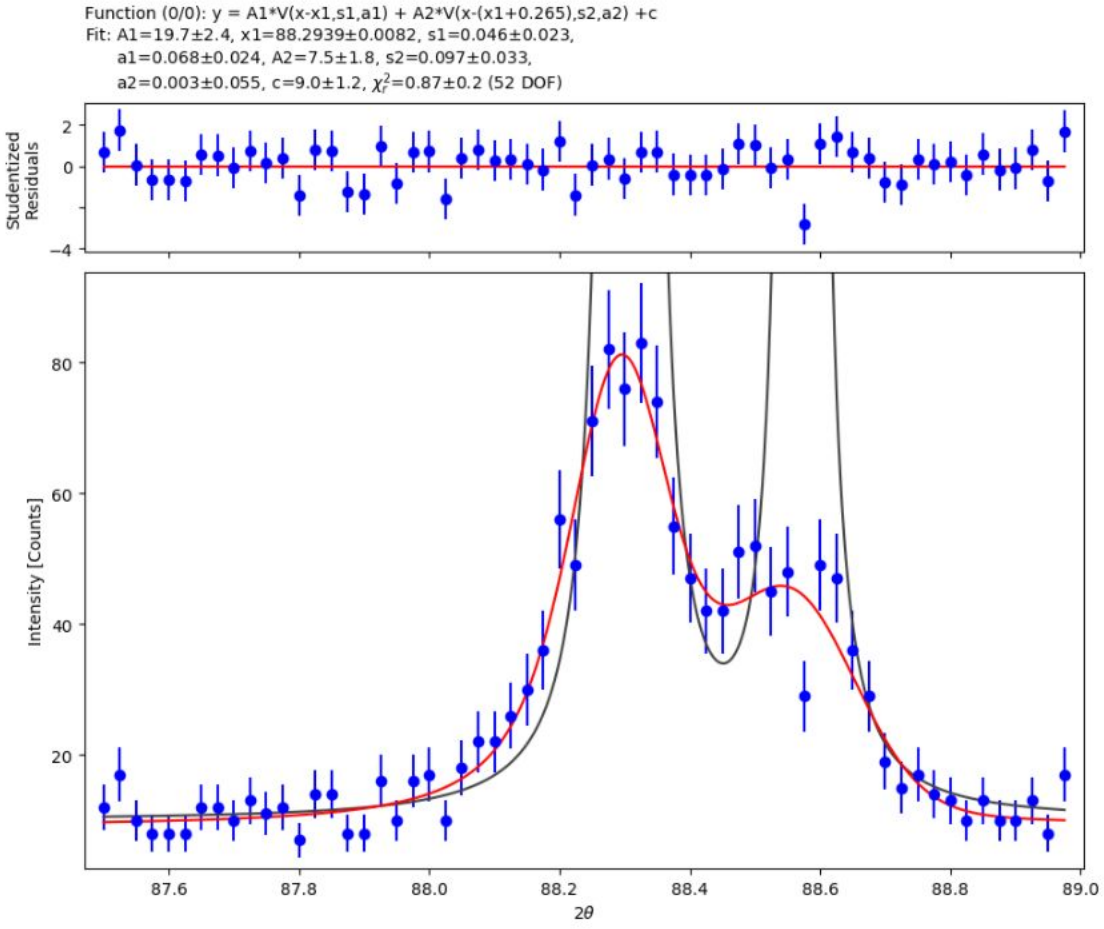
- Is based on experimental observation that some binary alloy's lattice parameters will change linearly with change in concentration.
- According to these two papers, Vergard's law does not always hold. It only holds if the two metals making up the alloy share certain property (mainly have atomic radii of similar size):
    - http://newmaeweb.ucsd.edu/~vlubarda/research/pdfpapers/mm-03.pdf?fbclid=IwAR1kvXv3dDt8MGNie6gs_OtETsXvh3l87nhg5oGd5fEDK6DTBaNrem4-EqY
    - https://www.researchgate.net/publication/235550709_Vegard's_law
- Verify this with our data, since we already found in interim report that the relationship between concentration and lattice parameter of CuNi alloys changes linearly with change in concentration .

Additional Crystal Structure Research on PbSn:

PbSn:
https://link.springer.com/chapter/10.1007%2F10542753_2419

01/30:

- Got the double peak fitting to work with much better reduced chi-squared. See below sample graph:



- **Ran sample**: 75%Pb 25%Sn with param2 file
- Performed the double fit for all the peaks in the CuNi series of data (i.e for each alloy, all the peaks were fitted and tabulated)

01/31:

- **Ran samples**: 50%Pb 50%Sn and 25%Pb 75%Sn with param2 file
- Work on analysis and report writing
- Perform double peak fit for Cu3Au data samples

02/01:

- **Ran sample**: 100%Sn with param2 file (completes data collection)
- Perform double peak fit for all PbSn data
- Keep working on writing report