Determining the Crystal Structure of Metal Alloys via X-ray Diffraction

Elisa Jacquet, An Vuong, Alev Orfi

McGill University Department of Physics

Supervisor: Prof. Brunner & Prof. Sankey

January 21, 2019

Abstract

X-ray diffraction can be use to obtain microstructural quantities of metal alloys. A Voigt function provides a good approximation for X-ray diffraction profiles allowing the determination of the mean crystallite size of several cubic crystals. We analyzed Copper and Nickel alloys, noticing a changing profile depending on the alloy ratio. An initial analysis was performed on pure copper, fitting its diffraction profile with a voigt function to obtain the peak angle. Using this value we obtained $3.61 \pm 0.03 \mathring{A}$ to be the lattice constant of pure Copper.

Experimental Set-up

This study makes use of an x-ray diffractometer to obtain information about the crystal structure of various metal alloys. Such a diffractometer produces x-rays by decelerating and exciting electrons in a copper target. Through this process, copper produces different x-ray wavelengths that are then filtered out to produce a monochromatic beam with average wavelength $k_{\alpha\ \mathrm{avg}} = 0.154\mathrm{nm}$. This monochromatic beam is incident on the sample at an angle θ. The sample - in this case an alloy of copper and nickel - diffracts the incident beam at varying angles depending on both theta and the samples' crystal structure. A detector that is locked in place with the sample counts the number of rays diffracted at an angle 2θ. The sample (and detector) are rotated so that the diffractometer can count the number of rays diffracted at angles ranging between 5°and 55°. A diagram of the basic set-up and geometry of the diffractometer is shown in 1. As θ is changed, the number of counts recorded

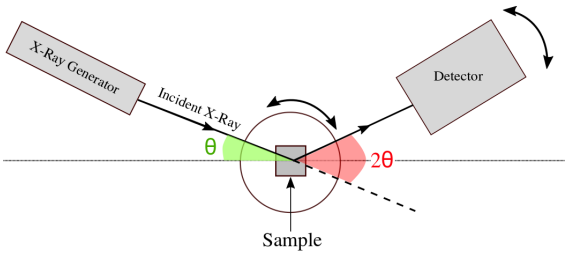


Figure 1: Diagram of the set-up of the x-ray diffractometer. The apparatus records the number of counts at each angle 2θ. Both sample and detector are free to rotate independently, though for this study, both were locked together.

by the diffractometer changes as well. The measurements show peaks in counts at various angles due to a diffraction pattern described by the Bragg Law relation: [1]

$$2dsin\theta = n\lambda \tag{1}$$

The relation between the interplanar spacing d, the Miller indices of the crystal and the lattice constants is dependent on the crystal system. The relation for the cubic system is shown below in Equation 2.

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \tag{2}$$

Combining this relation and the Bragg Law we get a general relation for cubic structures. [3]

$$sin^2(\theta) = \frac{\lambda^2}{(4a^2)}(h^2 + k^2 + l^2) \tag{3}$$

In our study, the experiment was conducted for the following alloys of copper and nickel: 100% Cu, 75% Cu and 25% Ni, 50% Cu and 50% Ni, 25% Cu and 75% Ni, and 100% Ni. For each sample, the apparatus counted the number of diffracted rays for 2 seconds, and rotated in intervals of 0.05°.

Initial Analysis

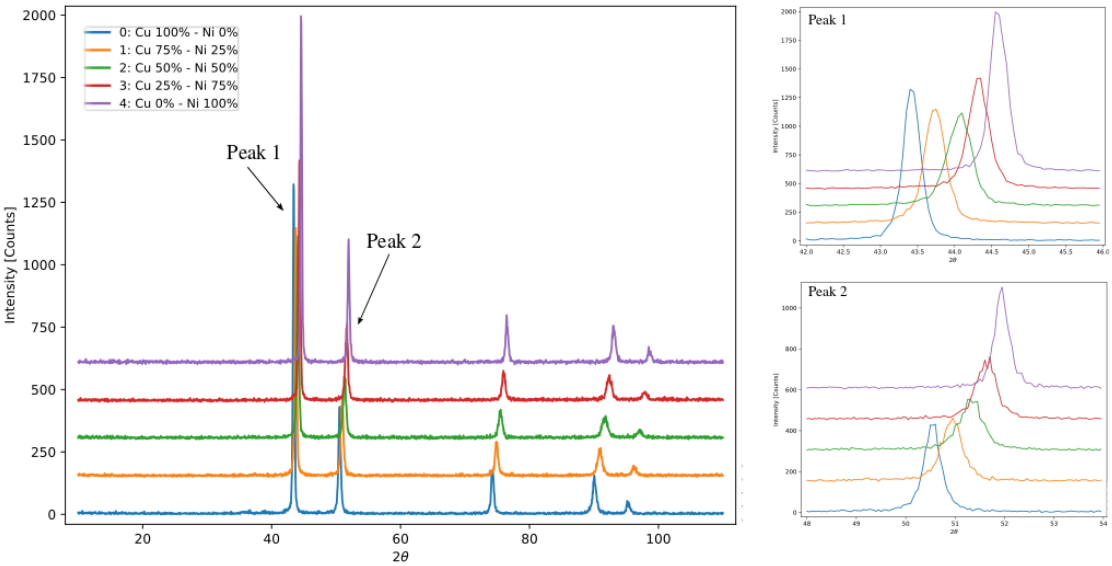


Figure 2: Plot of signal intensity vs angle 2θ of all five Cu/Ni alloy samples with different concentration. LEFT: All data plotted on top of each other with a shift of 150 counts between 2 consecutive alloys. TOP RIGHT: Zoomed in image of peak 1. BOTTOM RIGHT: Zoomed in image of peak 2.

Analysis of crystal Structure of Copper Nickel alloys

The diffraction pattern of 5 samples of Copper Nickel alloy with different concentration are plotted with respect (2θ). As seen in figure 2, the data is superimposed allowing for easy comparison. The signal intensity tends to increase as the concentration of Nickel in the studied alloy increases. Additionally, a slight right shift was also detected from all peaks as

Nickel's concentration dominates the alloy. This preliminary analysis is in agreement with the known resemblance of Cu and Nickel lattice constant.

Determining the lattice constant for 100% Cu alloy

Peak fitting analysis, using a Voigt function, was performed to determine the exact angular position of the peaks. Among 5 peaks observed, the fit was only performed for the first 4 as the signal from the last peak resembles that of background noise. Figure 3 shows a sample fit for the first peak in the diffraction pattern of the 100% Cu alloy. The residual plot on that same figure shows no systematic behaviour. The success of the fit was confirmed with a chi-squared value of 37.5 ± 0.1, indicating a probability of more than 90%.

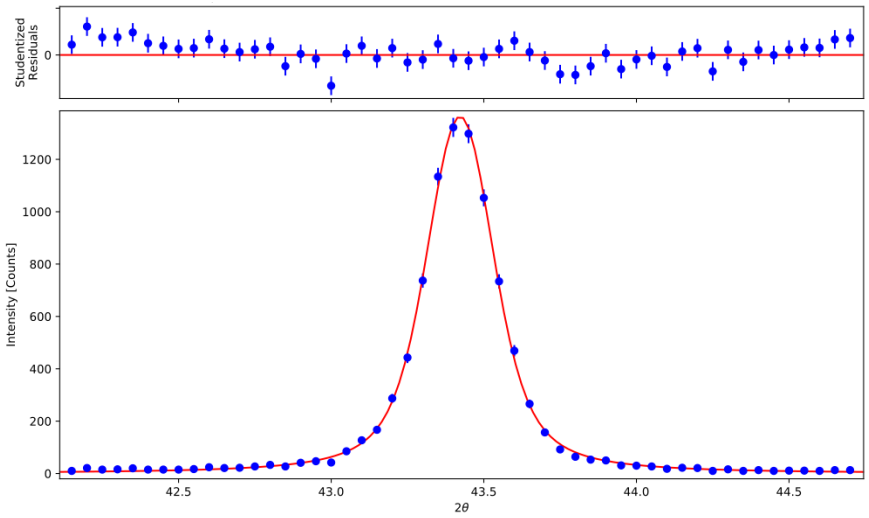


Figure 3: Fitting on the first peak of Cu 100% alloy and its standardized residuals. Errors on the intensity were calculated by taking the square-root of the number of counts. A Voigt function with Gaussian standard deviation $\sigma = 0.077 \pm 0.003$ and Lorentzian half-width $\Gamma = 0.069 \pm 0.002$ was used to fit the first peak. Chi-square value was calculated to be 37.5 ± 0.1 with 130 DOF. The peak was identified to be at angle $2\theta = 43.422 \pm 0.002°$

Once the positions of 4 peaks of Cu 100% alloy were identified, a linear fit was performed to correlate the angle with the square-root of the sum of the 3 Miller indices (h, k, and l). The slope of this fit was determined to be 0.0213 ± 0.002. From the relation in equation 3 the lattice constant of Cu 100% was calculated to be $3.61 \pm 0.03 \mathring{A}$. A special note should be made that this result assumes no error on the value of the wavelength of the incident ray, taken to be 0.154 nm. [2]

Bibliography

[1] William Bragg, *The significance of crystal structure*, Journal of the Society of Chemical Industry.

[2] B D Cullity, *Elements of x-ray diffraction*, (1978).

[3] Charles Kittel, *Introduction to solid state physics*, **5** (1976).

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 and 1 mystery sample 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.

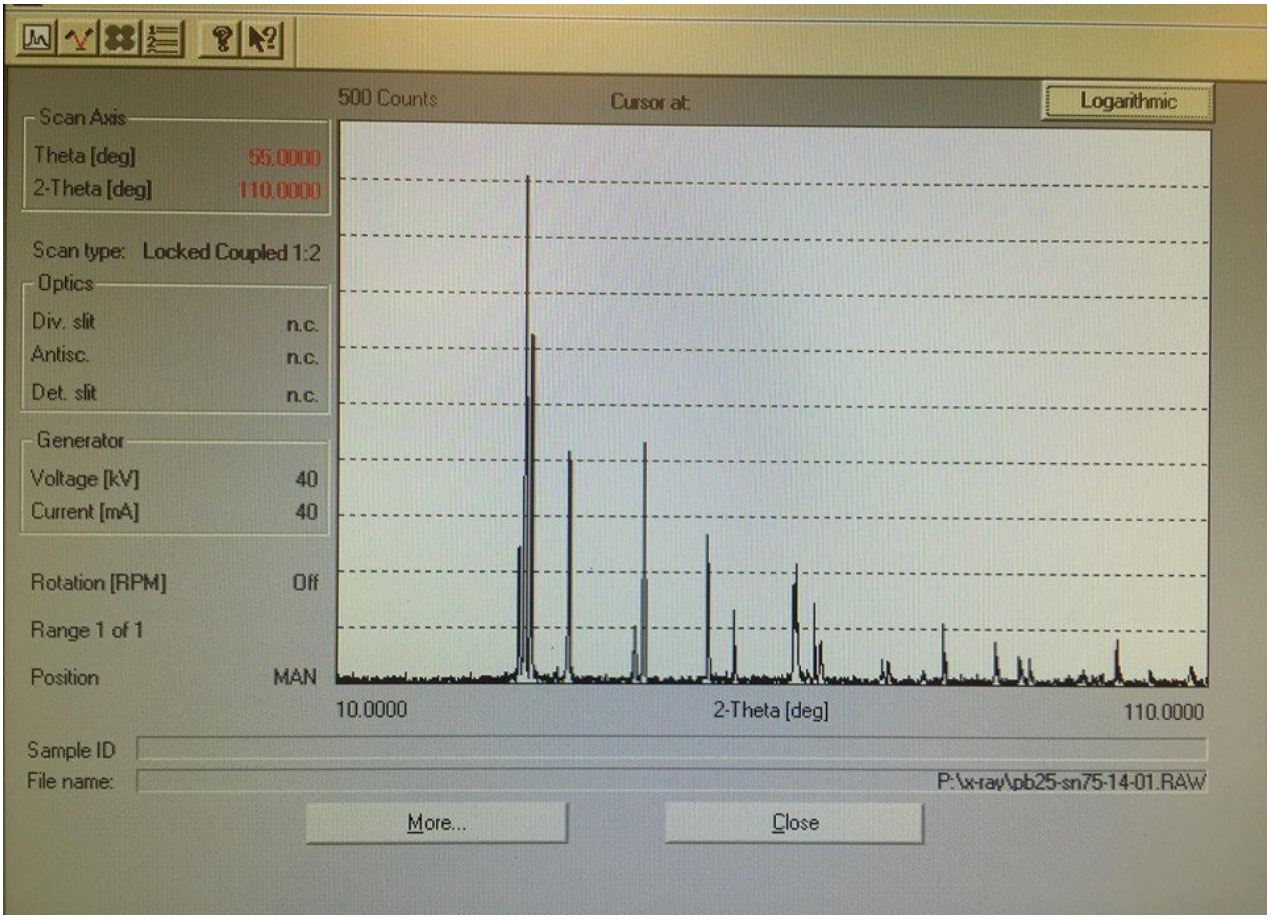
Data Analysis Step #1:

- 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

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