LAB 01
X-RAY EMISSION & ABSORPTION

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ABSTRACT

The goal of this experiment was to examine the production of x-rays, the nature of electromagnetic radiation emitted from an x-ray source, and the absorption of x-rays.

In this report, we characterized the Copper-Target s-ray emission spectrum and tested eight different filters for x-ray absorption effects. We inspected the effect of filter thickness and type, and ultimately found that the mass absorption coefficient increases with filter thickness and atomic number.

INTRODUCTION

X-RAY EMISSION

In this lab we address x-ray emission by examining the spectrum of radiation emitted from an x-ray tube with a specific target. The resulting spectrum represents the energy dispersion of the electromagnetic radiation released by the target during x-ray emission, and is quantified by determining intensity of diffracted beam at particular emission wavelengths. The specific I vs λ plot that results from an unknown target can be used to identify the makeup of the target using characteristic material traits. In our case, we will be examining the specific plot of a copper target.

The x-ray diffractometer allows us to control the angle at which the x-rays strike a secondary dispersive element (in this case 111 silicon crystal) but not the wavelength of the emitted x-ray. However, the dispersive element has the ability to scatter radiation of different energies (and therefore different wavelengths) into different directions. Using the known angle of collection (representing the direction that the energy is scattered) and Bragg's Law, we can calculate the relationship between intensity measurements and the wavelength of the emitted radiation. This information can also be used to calculate the Intensity vs. Energy plot for the particular x-ray tube target.

X-RAY ABSORPTION

There are two forms of absorption that a beam can undergo, absorption due to elastic or inelastic scattering. A small part of an x-ray will be scattered elastically when hitting a target, this part will not lose energy but will scatter in a different direction. The majority of absorbed x-rays will be inelastically scattered (this is true absorption) and lose energy when striking a material.

Inelastic absorption is dependent on the energy of the x-ray striking the target. There are two cases where a beam will pass through a material, a beam with more energy than one of the absorber's electronic transitions will pass straight through the material without releasing energy into removing an electron, and beam with less energy will similarly pass through the material without the ability to remove an electron. Only an incident x-ray photon that closely matches the energy for an electronic transition will be absorbed, causing an absorption edge corresponding to the level of that electron and visible on the intensity plot at the absorption wavelength. Using the intensity plot, it is possible to determine a material's mass absorption coefficient as well.

EXPERIMENTAL PROCEDURES

For this experiment, our materials were an x-ray diffractometer, a copper target, a (111) Silicon crystal, and 8 different absorption filters (10 μm, 20 μm, 40 μm, 80 μm Ni, 10 μm Al, 12.7 μm Ti, 25 μm Cu, 25 and μm Mo).

Before beginning both sections of the lab, we initialized the Rigaku Miniflex II X-Ray Diffractometer. This included turning on the cooling water, initializing the data collection software, mounting the (111) Silicon wafer crystal (this was already done before the experiment began by the instructor) and checking for background radiation. The voltage for this diffractometer is preset at 30kV and 15 mA.

X-RAY EMISSION

The collection of pure x-ray emission data (with no filter) was straightforward. We closed the cabinet door and energized the x-ray beam after ensuring that the equipment was in the initial position. We then began recording with the software and recorded the Intensity of x-ray photons diffracted at specific diffraction angles (2θ) over the entire possible range for the diffractometer. Obviously this was limited by the x-ray emitter, so we were able to record data from 2θ = 3° to 140°. The recording resolution was set at 2.00°/min in order to get a clean spectrum.

X-RAY ABSORPTION

After determining the standard x-ray photon spectrum for a copper target, we tested the effects of different filters on the detected intensities of the x-ray beam. Filters are inserted into the machine just in front of the detector between the beam and the detector. These filters are of various thickness and materials (10 μm, 20 μm, 40 μm, 80 μm Ni, 10 μm Al, 12.7 μm Ti, 25 μm Cu, 25 and μm Mo).

Each filter was tested over a specific relevant diffraction angle range to reduce testing time. It was possible to determine the specific wavelengths over which the filter would absorb using Bragg's law around the absorption edges of the absorber. The following table indicates the calculated ranges that were used. Note that the K Absorption edges were selected for use because L and M edges required energies outside of the relevant range.

Table 1: Calculated Testing Bragg Angle Ranges for Various Metal Absorbers

Metal	λ at K Absorption Edge (Å)	Angle, θ	2θ	Testing Range
Ni	1.488[2]	13.73°	27.46°	20-35°
Al	7.948[2]	Undefined	Undefined	120°-140°
Ti	2.497[2]	23.47°	46.94°	40°-55°
Cu	1.380[2]	12.72°	25.44°	17°-32°
Mo	0.619[2]	5.67°	11.34°	3°-18°

Calculations were completed using Bragg's Law:

$$n\lambda = 2d_{hkl} \sin\theta$$

With values of λ determined from known values of K absorption edges. When rearranged to solve for angle, we get

$$\theta = \sin^{-1}\left(\frac{n\lambda}{2d_h kl}\right)$$

Where n = 1, and d_{hkl} is a property of the (111) silicon determined by:

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

Which is a known equation for cubic interplanar spacing. Here, *a* is the lattice parameter of (111) Silicon – 5.4309 Å^2.

Therefore, our final θ calculation was based on the equation

$$\theta = \sin^{-1}\left(\frac{\lambda}{2\sqrt{3}(5.431)}\right)$$

The range used was then calculated by doubling this angle and creating a window of integer values 15° around 2θ.

To test each sample, we adjusted the 2θ range to what we calculated and recorded the resultant intensities in a similar manner to the "X-Ray Emission" procedure. However, this time the collection speed was 4.00°/min. Changing this collection speed was expected to reduce the quality of the signal in favor of reducing testing time.

Notice that Al produced an undefined angle range. This is because the interplanar spacing of silicon (111) is too small to reflect the wavelength needed for the Al absorption edge at a reasonable diffraction angle. To image Al's absorption edge, we would need to use a different dispersive element. Instead, we chose to image an angle range near the upper bounds of what is possible in order to confirm our hypothesis.
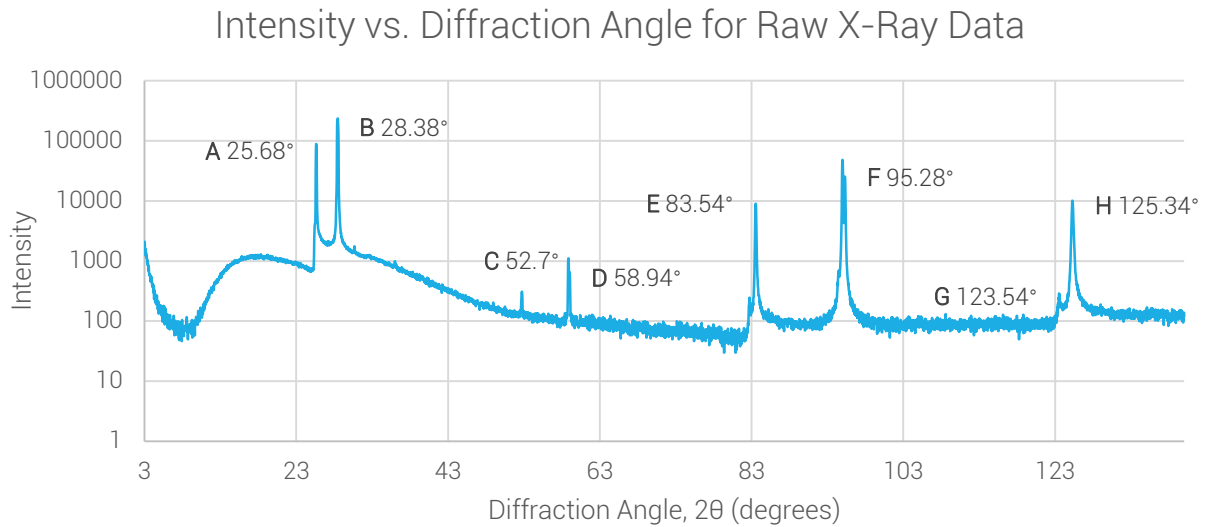
EXPERIMENTAL RESULTS

In all x-ray experiments, the background radiation was found to be 0.02 mR/hr using a Geiger counter.

X-RAY EMISSION

X-ray emission results were intensity values for the entire usable diffraction angle range possible using our equipment.
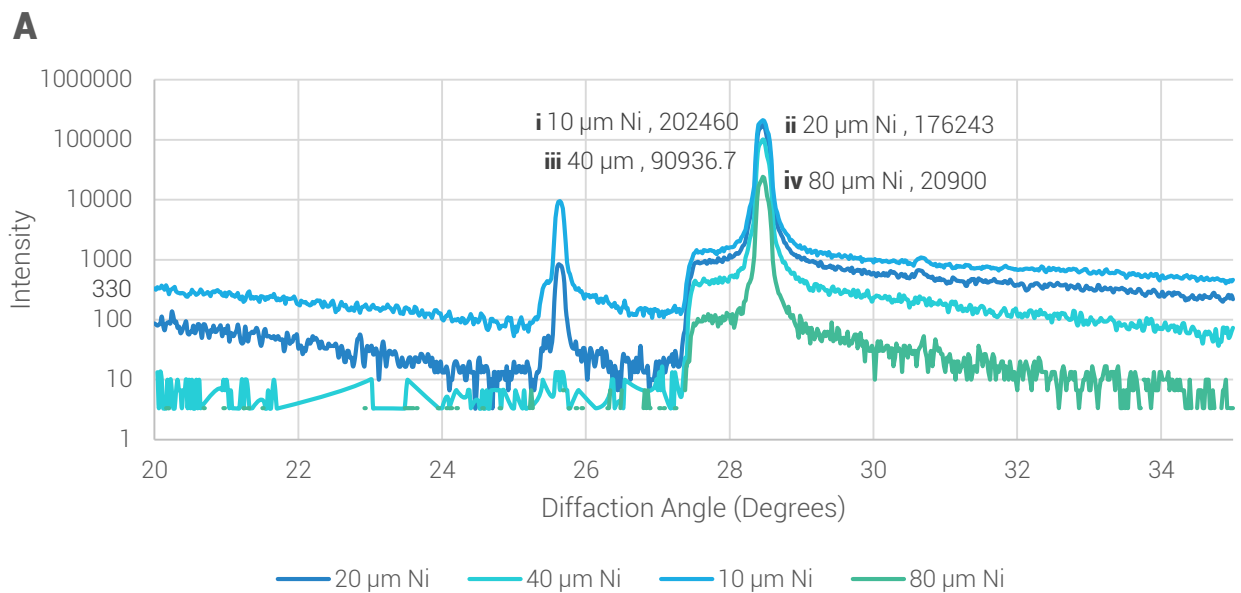
Figure 1: Intensity vs. Diffraction Angle for Raw X-Ray Emission from a Copper Target. Bold letters correspond to various characteristic lines that will be further explored in the "Discussion" Section of the Report.

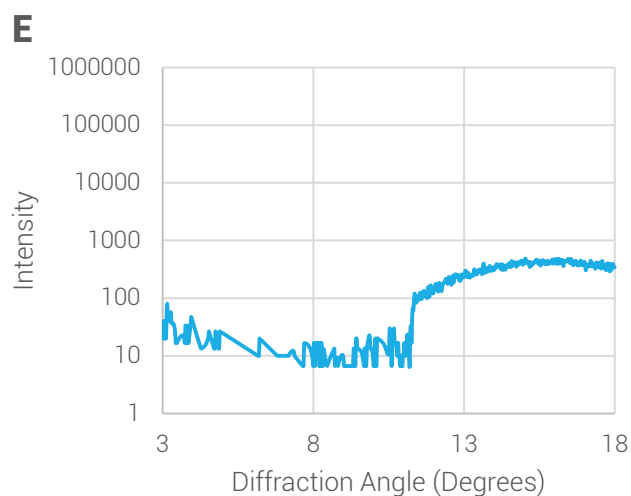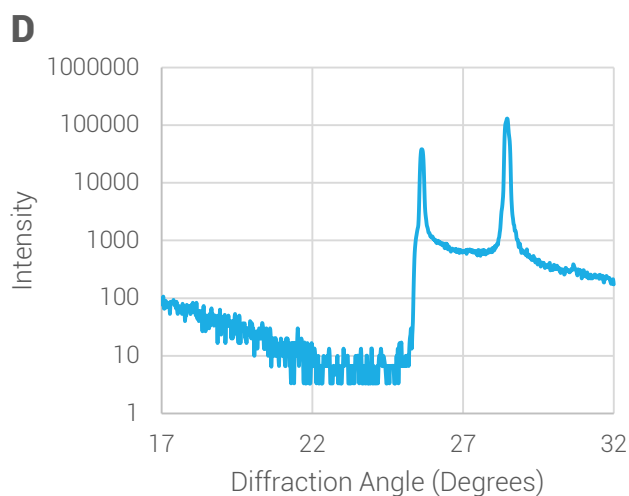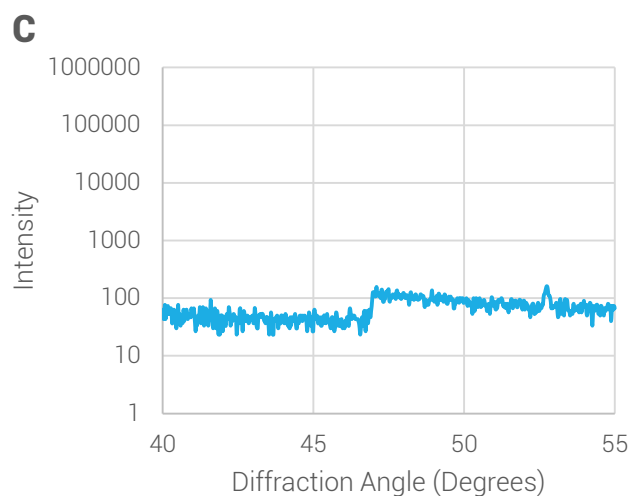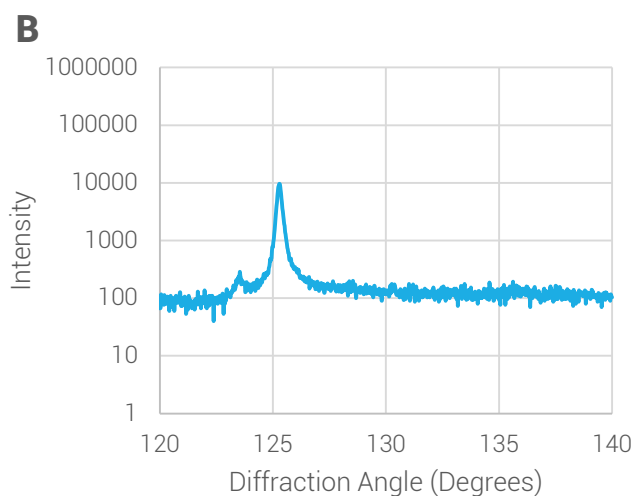Intensity vs. Diffraction Angle for Raw X-Ray Data

X-RAY ABSORPTION

These experiments focus on a specific range of the raw x-ray data for each filter. Only the experimental range of data is shown.

Figure 2: X-Ray Absorption Data for Various Filter Types. **A i** 10 μm Ni, **ii** 20 μm Ni, **iii** 40 μm Ni, **iv** 80 μm Ni **B** 10 μm Al **C** 12.7 μm Ti **D** 25 μm Cu **E** 25 μm Mo.

A

B

C

D

E

DISCUSSION

In this section we will elaborate on the significance of your results and answer the questions posed in the lab manual.

DATA REDUCTION

WAVELENGTH CONVERSION

As shown in the procedure section, Bragg's Law can be used to calculate the angular range for testing. Bragg's Law is:

$$n\lambda = 2d_{hkl}\sin\theta$$

In the form that will give us the wavelength, given that n =1. The value of d_{hkl} is a property of the (111) silicon determined by:

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

Where k, h, and l are each 1 and *a* is the lattice parameter of (111) Silicon (5.4309 Å) [2]. We know this is the correct d-spacing because we can see that peaks appear at the predicted wavelength ranges using this calculation.

CONTINUOUS SPECTRUM

The short wavelength limit is the minimum possible wavelength beam that could possibly result from voltage used to accelerate the striking electrons. This limit is theoretical, and is calculated by converting all the potential energy caused by the electric field into kinetic energy which displaces an electron in the target material.

In this case, out acceleration voltage was 30 kV. The class textbook, <u>Elements of X-Ray Diffraction</u>[3], lists a simple calculation that can be used to determine the short wavelength limit using our acceleration voltage. This is equation **1-4** from the book.

$$\lambda_{SWL} = \frac{12.40 \times 10^3}{V}$$

Here the units of V are volts. We plug in 30 kV to the formula and compute λ_{SWL} to be 0.4133 Å, or 0.41 Å with significant figures.

K ABSORPTION EDGE

The Cu K absorption edge will correspond to the energy of accelerated electrons needed to displace an electron in the K-shell of the copper target. We can look up this energy from Appendix 7 in <u>Elements of X-Ray Diffraction</u>, and compare that value to the one we calculated using our equation and the apparent absorption edge at $2\theta = 25.36°$. This edge is at the base the first peak found on the Raw X-Ray intensity plot in the "Data" section of the report, and is labeled with the angle corresponding to that peak. The known value is 1.38059 Å, and the wavelength using my data and Bragg's Law is 1.376 Å. These values are very similar, indicating that my edge is likely correct.

CHARACTERISTIC LINES AND CONTAMINANTS

The relevant characteristic lines for Copper are Kα, Kβ, and the K edge. The L edges for Copper are outside of our detector angle range and would require a different dispersive element.

Table 2: Experimental versus Known Characteristic Line Wavelengths for Copper

Characteristic Line	Known Value for Cu Source (Å)	Experimental Value of 2θ	Experimental Tabulated Value
Kα	1.543[3]	28.49°	1.543
Kβ	1.392[3]	25.65°	1.392
K Edge	1.381[3]	25.44°	1.381

Characteristic Line	Known Value for Contaminant Source (Å)	Experimental Value of 2θ	Experimental Tabulated Value
Sc Kβ	2.779[3]	52.70°	2.783
Sc Kα	3.034[3]	58.94°	3.084
Rh L III	4.129[3]	83.54°	4.176
Rh L Edge	4.597[3]	95.28°	4.633
P K Edge	5.784[3]	123.5°	5.524
P Kβ	5.796[3]	125.3°	5.569

FILTERS

Table 3: Experimental versus Known Characteristic Line Wavelengths for Various Absorbers
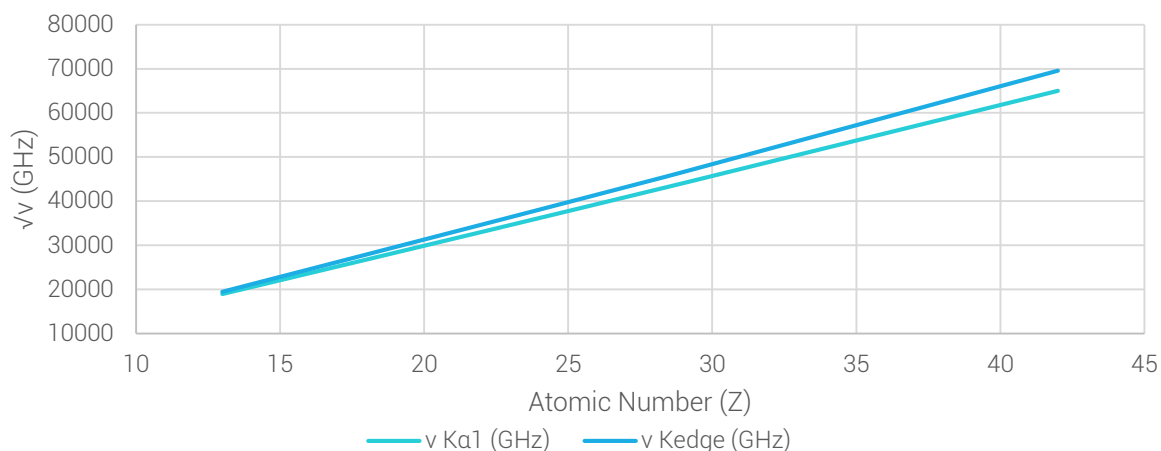
Metal Absorber	λ K_{edge} (Å)	ν K_{edge} (GHz)	Atomic # (Z)	$K\alpha_1$ (Å)	ν $K\alpha_1$ (GHz)
Ni	1.488[3]	2.014×10^9	28	1.658[3]	1.808×10^9
Ti	2.497[3]	1.201×10^9	22	2.749[3]	1.091×10^9
Al	7.948[3]	3.772×10^8	13	8.338[3]	3.595×10^8
Cu	1.380[3]	2.172×10^9	29	1.541[3]	1.945×10^9
Mo	0.619[3]	4.843×10^9	42	0.709[3]	4.228×10^9

MOSELEY'S RELATION

$$Z \ vs. \ \sqrt{\nu K_{edge}}$$

$$Z \ vs. \ \sqrt{\nu K\alpha_1}$$

**Figure 3:** Atomic Weight vs. Frequencies at K Edges and Kα Characteristic Lines of filters.

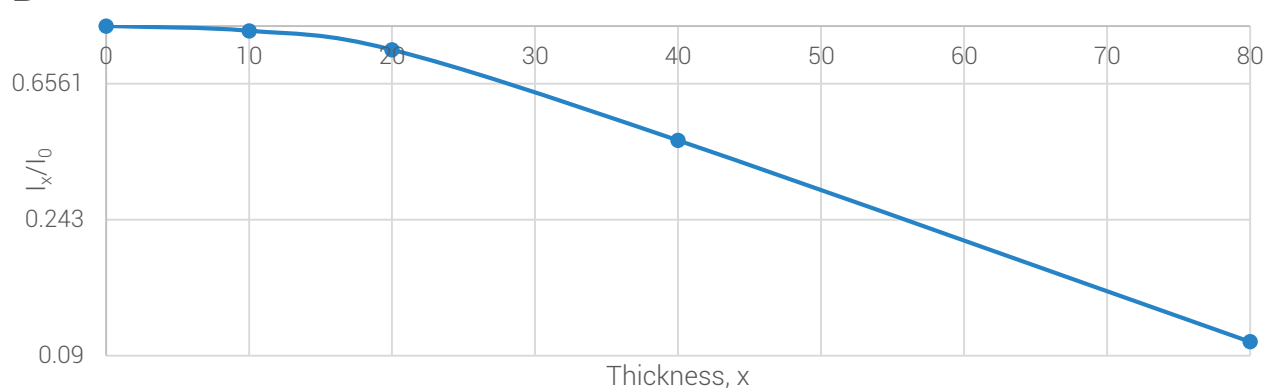## ABSORPTION COEFFICIENT

Table 4: Mass Absorption Intensity Data

Filter Sample	Thickness, x	I_x	I_x/I_0
0 Ni	00.0 μm	209735	1
1 Ni	10.0 μm	202460	0.965313372
2 Ni	20.0 μm	176243	0.840312776
4 Ni	40.0 μm	90936	0.433575703
8 Ni	80.0 μm	20900	0.099649558

Figure 4: Relative Absorption for Nickel of Various Thickness. **A** Normal relative Intensity vs. Thickness plot **B** Log base e scale Relative Intensity vs. Thickness plot.

A
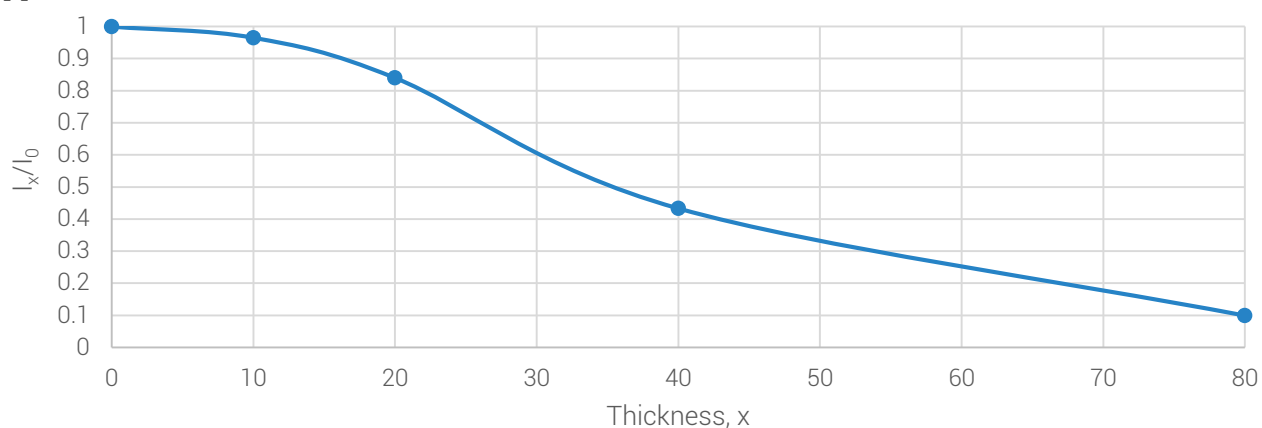
B

From the slope of the log-log plot we can calculate the mass absorption coefficient (because this plot is in base e). The following equation is **1-10** from Elements of X-Ray Diffraction.

$$\frac{I_x}{I_0} = e^{-\left(\frac{\mu}{\rho}\right)\rho x}$$

Our log plot represents:

$$\ln\left(\frac{I_x}{I_0}\right) = -\left(\frac{\mu}{\rho}\right)\rho x$$

So the slope of the plot is equal to mass absorption times the material density. In this case, the density of Nickel at room temperature is 8.91 g/cm³ from Appendix 8 of <u>Elements of X-Ray Diffraction</u>. The slope of the linear part of the plot is

$$\frac{-2.3061 - (-0.836)}{2 \times 10^{-3}\ cm} = -735.2 = -\left(\frac{\mu}{\rho}\right)\rho x = -\left(\frac{\mu}{\rho}\right)(8.91)x$$

$$\frac{\mu}{\rho}_{known} = 51.92\ \frac{cm^2}{gm}; \qquad \frac{\mu}{\rho}_{calculated} = 82.51\ cm^2/gm$$

DATA ANALYSIS

EMISSION

Our data reflected the K peaks and Edge that we expected to see in an experiment such as this, however there were definite peaks in unexpected places on the Intensity – Bragg Angle plot. From our calculation under the "Experimental Procedure" section, we expected to only see copper K shell peaks below a Bragg angle of 30˚, but many more peaks appeared further down the spectrum. These peaks can be explained by contamination by Rhodium or Phosphorus judging by the peak wavelengths (or flaws in the silicon crystal, but this explanation is unlikely).

The dispersive element (111) Silicon was useful for all filters except Aluminum. This is an efficient way to achieve dispersion, however another way to separate a spectrum according to energy is to transmit x-rays through a material rather than diffract the x-rays off the surface. My data was consistent with Moseley's law, which states that the wavelength of any particular line decreases as the atomic number of the emitter is increased. This is what was seen on my plots (which are listed under **Figure 4** above.

ABSORPTION

My measured absorption edges were very close to the tabulated values, however my mass absorption coefficient does not match well. This deviation is likely due to a difference in density of the filters used or contamination of the filter.

From tabulated data, to reduce the intensity by half we can use a formula I have mentioned before,

$$\frac{I_x}{I_0} = e^{-\left(\frac{\mu}{\rho}\right)\rho x}$$

$$0.5 = e^{-51.92 \cdot 8.91 x}$$

$$\ln 0.5 = -51.92 \cdot 8.91 x$$

Solving for x gives us a thickness of 0.001498 cm or 14.98 microns.

CONCLUSIONS

From this lab we can conclude that increased filter thickness has an effect on x-ray absorption and that filter material absorbs specific wavelengths.

Further, we have shown that Copper-target radiation has characteristic peaks correlated with K-shell absorption.

REFERENCES

[1] R. Gronsky. Lab 01 Manual: X-Ray Absorption and Emission. *MSE 104.* University of California Berkeley, Berkeley, CA.

[2] Lattice Parameter for Silicon, X-ray Powder Diffraction File No. 27-1402 (JCPDS-ICDD, 1987)

[3] B.D. Cullity and S.R. Stock, *Elements of X-Ray Diffraction*, 3rd Edition, Prentice-Hall, New York, (2001), p. 6, 11, 641*Appendix 7* and *Appendix 8*

APPENDIX

Overflow materials should appear here. Examples include extra data runs, problematic data (such as interrupted data runs), copies of pertinent literature or other documentation, computer source code listings, or derivations of equations.

No additional Information was Required.