X-RAY DIFFRACTION

${\it CASPAR\ LANT}$

Intermediate Experimental Physics II

Section: 002

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The Objective of this week's experiment was glean a glimpse into the atomic and structural composition of a couple elements, primarily with the aid of x-ray diffraction.

1. Theoretical Background/ Abstract

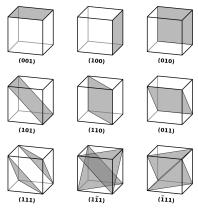
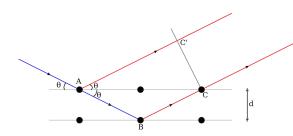


Figure 1

The structures of crystal latticies can be examined by using the techniques of x-ray crystallography, which extend from basic principles of light diffraction. Cubic latticies present in materials like sodium chloride (table salt) and aluminum form parallel planes in regions of high atom-density (see figure 1). The most apparent planes (corresponding to regions of largest atom-density) are orthogonal to the lattice structure, and can be seen as "extensions" of each cubic face. These orthogonal planes are seperated by what's referred to as the "latice spacing" of the material. Bragg's law will help us calculate the lattice spacing of our samples:

Bragg's Law:
$$m\lambda = 2d\sin(\beta)$$

Where β is the angle of reflection, λ is the wavelength of incident light, and d is the lattice constant; the spacing between "planes" in the material. $m=0,\pm 1,\pm 2,$ and so on. We may remember from our studies in optics that when light is incident on a material boundary between two media, some of it will reflect off the boundary, and the rest will pass through into the next layer, in a ratio that corresponds to the relative indeces of refraction of the two media. This effect is illustrated in the following diagram:



In this diagram, dots represent atoms, and the thin lines depict the planes that they form. Again, d is the lattice constant. It's important to note that usually the lattice "constant" seperated into distinct orthogonal components to account for non-cubic latticies, but in the case of Al and NaCl, d is the same in all directions.

The energy of the electrons is given by the electron charge, e^- , time the voltage in the tube, which tends to be very high. This metric is also the energy of the emitted x-ray. This can be seen by extending the Planck-energy formula $E = h\nu$, where h is the Planck constant, 6.62×10^{-34} m²kg/s.

$$E = e^{-}U = \frac{hc}{\lambda}$$

Mosley's Law, as shown on the right, gives us the wavelength and frequencies of the x-rays emmitted from our tube. It just so happens that n = 2 gives us K_{α} , our lower-bound for frequency, and n = 3 yeilds K_{β} , our upper bound. R is the Rydberg constant, equal to $1.097 \times 10^7 \,\mathrm{m}^{-1}$. Z is the atomic number of the x-ray-emitting material, which in this case is 42 (for Molybdenum).

$$\frac{1}{\lambda} = R (Z - 1)^2 \left(1 - \frac{1}{n^2} \right)$$

$$\frac{R (m^{-1})}{1.10 \times 10^7} \begin{vmatrix} Z & n & \nu \text{ (Hz)} & \lambda \text{ (m)} \\ 42 & 2 & 1.38 \times 10^{10} & 7.23 \times 10^{-11} \\ 1.10 \times 10^7 & 42 & 3 & 1.64 \times 10^{10} & 6.10 \times 10^{-11} \end{vmatrix}$$

2. Experimental Procedure

- (1) Plug the X-Ray machine into the power supply and turn it on.
- (2) Start the "X-Ray Apparatus" software on the nearby compupeter.
- (3) Select USB Connection in the settings menu in the recently opened software
- (4) Open the righthand sliding door of the X-ray apparatus and place the collimator in the designated slot, located between the x-ray tube and the larger compartment. The collimator should click into place with satisfying precision.
- (5) Mount the geiger counter into the rotating arm about 10cm away from the sample podium. Tighten the thumpscrew and plug the bayonet connector into the signal jack.
- (6) Place the NaCl sample into the sample holder, and tighten another thumbscrew to hold it in place.
- (7) Close the righthand sliding door, and ensure that the lefthand door is fully closed. Don't worry, the machine will not start if either door is open.
- (8) Make sure that the step angle, step time, current, voltage, and minimim and maximum angles are set correctly.
- (9) Begin a run by pressing the 'start' icon in the software interface.
- (10) Repeat the experiment with different values for voltage and current, and observe the effect.
- (11) Shield the measurement from certain frequencies with any of the supplied metallic filters.
- (12) When finished, repeat steps 6 though 11 for the aluminum sample.
- (13) If you still have the time/patience, remove the geiger counter, collimator, sample holder, and filter from the x-ray enclosure, and replace them an object of personal interest that you would like to have x-ray'd.
- (14) Remove the round panel from the right side of the machine, close the door, and perform a scan. It's possible-but highly unlikely- that you'll see an image on the exposed phosphor sheet. This, ideally, would function in a manner similar to an oscilloscope.

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3. Graphs and Calculations

Table 1. NaCl

Table 2. Lattice Length

		m = 1		m = 2	
#	β°	K_{α} · meters	K_{β} meters	K_{α} meters	K_{β} ' meters
1	6.3	2.15×10^{-09}	1.81×10^{-09}	4.30×10^{-09}	3.63×10^{-09}
2	7.2	4.55×10^{-11}	3.84×10^{-11}	9.11×10^{-11}	7.68×10^{-11}
3	14.8	4.58×10^{-11}	3.87×10^{-11}	9.17×10^{-11}	7.74×10^{-11}
4	22.1	-3.33×10^{-10}	-2.81×10^{-10}	-6.65×10^{-10}	-5.61×10^{-10}
5	26	4.74×10^{-11}	4.00×10^{-11}	9.48×10^{-11}	8.00×10^{-11}

Bragg diffraction off of NaCl sample with X-Ray light within the wavelength of 61 to 72 pico meters

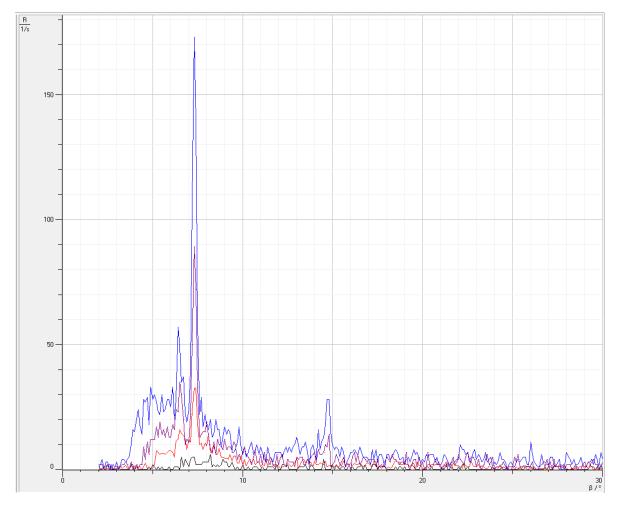


FIGURE 2. Bragg Diffraction off NaCl at 20V, 25V, 30V, 35V

Table 3. Al

Table 4. Lattice Length

			m = 1		m=2	
#	β°	R (1/s)	K_{α} · meters	K_{β} · meters	K_{α} meters	K_{β} ' meters
1	7.3	180	4.25×10^{-11}	3.59×10^{-11}	8.50×10^{-11}	7.17×10^{-11}
2	8.4	390	4.23×10^{-11}	3.57×10^{-11}	8.46×10^{-11}	7.14×10^{-11}
3	9.7	75	-1.33×10^{-10}	-1.12×10^{-10}	-2.66×10^{-10}	-2.24×10^{-10}
4	15.2	41	7.43×10^{-11}	6.27×10^{-11}	1.49×10^{-10}	1.25×10^{-10}
5	17.4	104	-3.64×10^{-11}	-3.07×10^{-11}	-7.28×10^{-11}	-6.14×10^{-11}
6	26.8	29	3.63×10^{-11}	3.06×10^{-11}	7.26×10^{-11}	6.13×10^{-11}

Bragg diffraction off of Al sample with X-Ray light within the wavelength of 61 to 72 pico meters

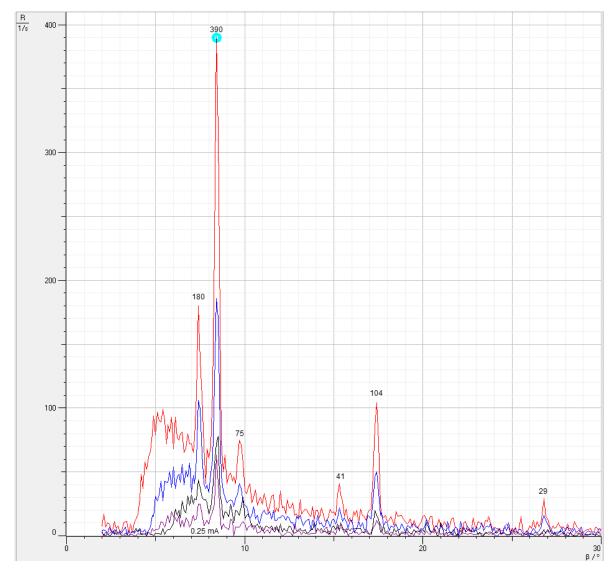


FIGURE 3. Bragg Diffraction off Aluminum Sample at 20V, 25V, 30V, 35V, and 0.25mA

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Table 5. X-Ray Wavelength

$R\left(\mathrm{m}^{-1}\right)$	Z	n	$\nu \left(\mathrm{Hz} \right)$	λ (m)
1.10×10^7	42	2	1.38×10^{10}	7.23×10^{-11}
1.10×10^{7}	42	3	1.64×10^{10}	6.10×10^{-11}

4. Questions

(1) The maximum energy xray depends on U (see above), in fact $\lambda_{min} = hc/U_{e^-}$. Find the minimum beta (maximum xray energy) for each run and plot $\sin(\beta)$ vs. $hc/2U_{e^-}$ to find the value for d for NaCl.

Answer

(2) Use this calibration to determine the wavelength of the K_{α} and K_{β} peaks seen in the plots. Compare the measured values to the prediction from Moseley's formula. Do the peak positions change with U?

Answer

(3) Calculate also the positions of the m=2,3,(4?) locations of the K peaks in the plots. Do they follow the Bragg diffraction formula?

Answer

(4) Take similar runs with the Al crystal sample and perform the same analysis steps. How does the value for d compare? Do the wavelengths of the K peaks change?

Answer

(5) Change the current used to 0.75, or 0.5, or 0.25 mA and repeat the Al run with U=35kV. What's the effect and what stays the same? Explain.

Location of peaks say the same, height of them decreases with decreasing current (see figure 4).

(6) What happens at even smaller angles than 2 degrees?

Full-frontal beam.

(7) What happens when adding xray filters such as Zr, Cu, Mo, Ag?

Cu blocked all diffracted x-rays.

(8) What if you remove the collimator?

See attached.

5. Error Analysis