Bragg Diffraction of X-rays– measuring the interplanar spacing in NaCl

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The purpose of this experiment is to measure the interplanar spacing of adjacent lattice planes of a salt crystal (NaCl). Students will use the Leybold X-ray apparatus (554 801) equipped with a molybdenum target to produce x-rays with two pronounced peaks (wavelengths). The x-rays are collimated and directed to a NaCl crystal where constructive scattering occurs at well-defined angles of incidence. Data will be collected over a range of angular steps using a Geiger-Muller tube, and analyzed off-line in order to determine the interplanar spacing of adjacent crystalline planes in NaCl.

I. BACKGROUND

Bragg diffraction is a phenomenon named after the early pioneers of x-ray diffraction, Sir William Bragg and his son Lawrence Bragg. Bragg diffraction is made up of two parts. The first part is called Bragg reflection and this is shown in Fig. 1. The second part is constructive interference and this occurs when the path length difference between the two paths is equal to an integral number of wavelengths. The equation describing this constructive interference is shown in Eq. 1

$$n\lambda = 2d\sin\theta \qquad (1)$$

where n is the order of diffraction, λ is the wavelength of the x-rays, d is the interplanar spacing between adjacent crystalline planes, and θ is the the incident angle of the x-rays measured with respect to the surface of the NaCl crystal. Take a look at Fig. 3 to better understand the angle θ.
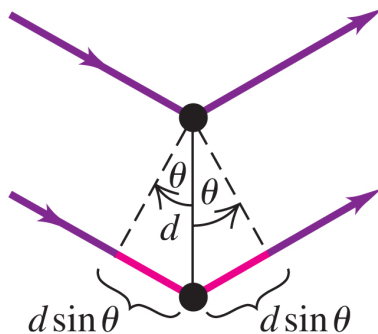

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FIG. 1. The incoming x-ray is actually a wavefront coherently scattering off multiple atoms (in this case two atoms) where the top atom is on the surface and the lower atom is located in the first layer underneath the top layer. Constructive interference occurs when the path length difference ($2d\sin\theta$) is equal to an integer number of wavelengths, $n\lambda$.

II. THE X-RAY MACHINE

The first step in producing x-rays is to accelerate electrons through a large potential difference of 25-35 kV. As the electrons impact the molybdenum target they are decelerated through a process called bremsstrahlung (breaking radiation). The x-rays are produced when a small-mass charged particle (e.g., an electron) passes near a massive charged particle (e.g., a molybdenum nucleus). The rapid deceleration of the electron by multiple scattering off nuclei leads to multiple x-rays being emitted, and on rare occasions, a single high-energy x-ray is emitted when an electron gives up all of its kinetic energy to a single nucleus. This last process corresponds to the *end-point energy* of the x-ray energy spectrum, and this can be observed by viewing the left end of the spectra shown in Fig. 2.

The molybdenum surface (anode) is angled with respect to the incoming electron beam to favor the production of x-rays in a particular direction. The spectrum of energies from the molybdenum target is shown in Fig 2. Not far from their production is a collimator tube which allows a narrow, horizontal ribbon of x-rays to pass through on their way to the crystalline NaCl target.

When the angular tilt of the NaCl target (resting on the goniometer) with respect to the incoming x-rays is just right (θ), constructive interference occurs and an increased counting rate (*counts/sec*) is observed in the Geiger-Müller tube located at an angle 2θ. This is shown in Fig 3.

III. PROCEDURE

This equipment is expensive and potentially hazardous; however, safeguards are built into the apparatus to prevent health hazards from occurring. For example, all the glass you see in the experiment is lead glass, and this reduces the x-ray level to the background x-ray exposures you normally receive ($\sim 1\mu S/hr$). Likewise, if any of the glass doors are ajar or open, the interlock will immediately halt the electron beam on the Molybdenum target that produces the x-rays.
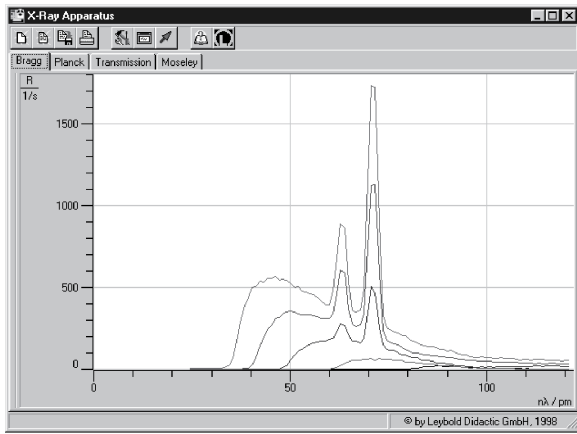
FIG. 2. The x-ray spectrum of the K_α and K_β peaks (right and left respectively) are shown as a function of wavelength. The longer wavelength ($\sim$71.04 pm) corresponds to the K_α line while the shorter wavelength ($\sim$63.14 pm) corresponds to the K_β line. The different curves correspond to different accelerating voltages–15, 25, 30, and 35 kV.
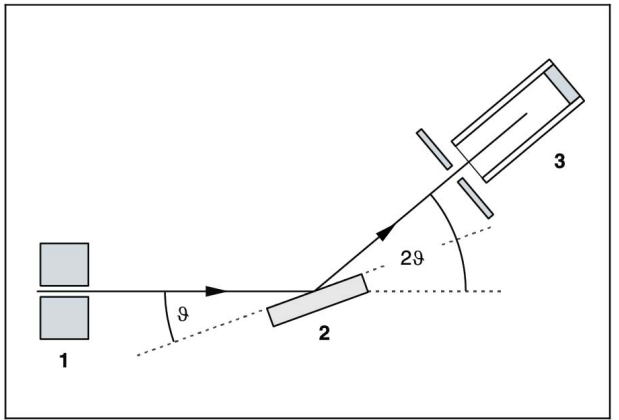

FIG. 3. The goniometer apparatus is shown in the figure above. The x-rays exit the collimator (1) and reflect off the crystal (2) and enter the Geiger-Müller tube where they are recorded as a rate (s^{-1}). Note that the angle of incidence (θ) is with respect to the surface of the crystal and not the normal.

You should not have to open the glass doors while the experiment or calibrations are underway. Because of the interlocks, it is not possible to produce x-rays while the glass door is open and you are accessing the NaCl crystal, or the Geiger-Müller tube. The lab assistant or instructor will connect the Geiger-Müller tube to goniometer before you operate the machine.

A. Setting up the experiment

1. Make sure the glass doors in front of the experiment are closed. If they are open or ajar, the experiment will not operate.

2. Turn the power on the X-ray machine. The switch is on the lower left-hand side.

3. Turn on the computer and start the `X-Ray Apparatus` program. The `X-Ray Apparatus` program should be located on the laboratory desktop computers. Starting in 2024, the laboratory computers in STEM-221 and STEM-222 have the Cassy software used to operate the x-ray machines. Make sure that the USB cable between the computer and the x-ray machine are connected **before running the Cassy program**.

4. Once the `X-Ray Apparatus` program is running, you must perform a calibration run. Push the **Zero** button on the front panel of the machine. This will zero both the crystal angle and Geiger-Muller tube angle. Go to the settings in the `X-Ray` program and click on the calibration tab. This procedure will take 1-2 minutes. You will see the x-ray tube glowing while the x-rays are generated for the calibration run. When the calibration is complete, click on the "adopt" tab to accept the calibration. You only have to do the calibration once and you can continue to collect all the measurements you need. If either the machine or the computer are disconnected, or turned off and then turned on, you must perform another calibration run.

5. Now you are ready to proceed with taking data.

6. I would suggest taking a first run with the following parameters to get the "lay of the land." Set U = 30kV. Set I=0.8mA. Set the lower and upper range of the angles (β) to be 3.0º to 30º. Set the rotational step size $\Delta\beta$=0.2º. Start taking data by clicking on the Take Measurement button in the upper-left tabs in the software program. Sit back and watch the x-ray machine take your data.

When you collect data in the range 3.0º $< \beta <$ 30º, you should see something similar to what is shown in Fig 4.

The goal for this lab is to determine the interplanar spacing for adjacent layers in the NaCl crystal. When you collect your data, you will see 3 pairs of peaks as shown in Fig. 4. The first pair of peaks represent the first order diffraction (n=1) of the K_β and K_α lines. The second pair of peaks represent the second order diffraction (n=2), and so on ....

IV. ANALYZING THE PEAK VALUES

As you can see from the data you collected, there are three pairs of peaks. The first peak in the pair is the K_β peak and the second one is the K_α peak, and these pairs of peaks recur for n=2 and n=3. Using the Bragg
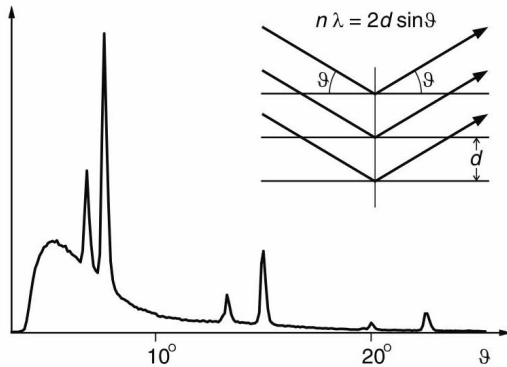
FIG. 4. This figure show three pairs of peaks when making a data run with angles $3.0° < \theta < 30°$. The position of the peaks (i.e, their angles, θ) should be recorded for further analysis.

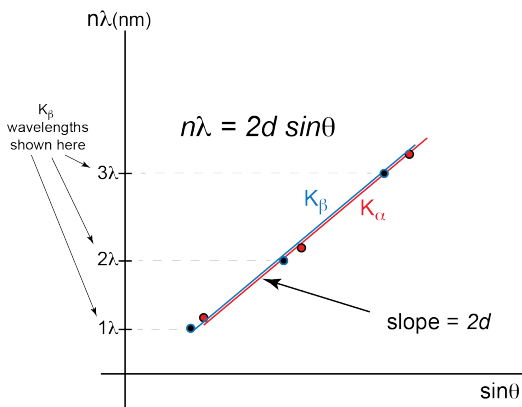diffraction equation (Eq. 1) you can plot the peak values for $n\lambda$ versus $\sin\theta$ as shown in Fig. 5.

FIG. 5. This figure shows the data corresponding to the 6 peaks collected from the Bragg Diffraction run shown in Fig. 4. The K_α peaks form one line of data, while the K_β peaks form a second line of data. The slopes of these two lines correspond to the interplanar spacing of the salt crystal (NaCl).

V. IMPORTANT CONSIDERATIONS

• Consider fitting a pair of x-ray peaks in Fig. 4 to a double gaussian function with a linear background

$$f(\theta_i) = a + b\,\theta_i + \frac{c}{\sqrt{2\pi\sigma_\beta^2}}e^{-\frac{(\theta_i - \bar\theta_\beta)^2}{2\sigma_\beta^2}} + \frac{d}{\sqrt{2\pi\sigma_\alpha^2}}e^{-\frac{(\theta_i - \bar\theta_\alpha)^2}{2\sigma_\alpha^2}}$$

(2)

• You should be able to determine the angle at the peak values to within 0.1° or better. You might want to take runs where the $\Delta\beta$=0.1, and only run with lower- and upper-values of β that encompass two adjacent peaks.

• Use the best fit parameters σ_α and σ_β for your uncertainties when plotting your error bars $\delta\theta_\alpha$ and $\delta\theta_\beta$ respectively.

• Convert your error bars in the horizontal direction into error bars in the vertical direction using Eq. 1, and then add them in quadrature with the existing error bars in the y-direction. If you have not done this before you can refer to the leaflet for the *charge-to-mass* experiment.

• **When it comes to χ^2's**, don't worry about the χ^2's for Fig. 4. However, do focus your attention on Fig. 5 when it comes to calculating the χ^2's for your straight-line fits. Your straight-line fits must make use of the vertical error bars, so make sure you calculate them correctly.
Don't forget to calculate your uncertainties in the straight-line fit so you can quote your measurement of the interplanar spacing and its uncertainty.

• The accepted value for the interplanar spacing in NaCl is 0.282 nm.

• If you are unsure about something, or if you have questions, please ask me or the Lab Assistant for assistance.