Introduction

In this lab you will explore the X-ray spectrum produced by copper in an X-ray tube and investigate how X-ray diffraction can be used to study the crystallographic structures of materials. X-ray physics has played a major role in at least three Nobel prizes in Physics. The 1914 Nobel prize in Physics was awarded to Max Von Laue for the discovery of the diffraction of X-rays by crystals. The 1915 Nobel prize in Physics was awarded to Sir William Henry Bragg and his son Sir William Lawrence Bragg for their analysis of crystal structures by means of X-rays. The 1962 Nobel prize in Physiology or Medicine was awarded to James Watson, Francis Crick and Maurice Wilkins, for their determination of the molecular structure of deoxyribonucleic acid (DNA) using X-ray diffraction.

In a typical x-ray machine like the one you will be using, thermal electrons emitted from a heated filament in a vacuum are accelerated by a voltage \( V \) to impinge on a copper target. The resulting radiation (X-rays) emitted from the copper has a broad spectrum with sharp peaks at certain wavelengths. The broad spectrum is called Bremsstrahlung (or braking radiation) which is due to the radiation emitted by the deceleration of the electrons in the copper. The minimum wavelength of this radiation corresponds to the maximum energy of the electrons and Planck’s constant, \( h \), and can be determined from \( E = \frac{hc}{\lambda_{\text{min}}} \) where \( c \) is the speed of light and \( E = eV \) is the energy of the electrons. The sharp peaks in the spectrum are due to the excitation of atomic (core) electrons in the copper by the incoming electron beam, which then emit X-rays of specific wavelengths when they de-excite. The peak at shorter wavelength is called the K\( \beta \) line and corresponds to transitions from the atomic M (n=3) to K (n=1) state. The peak at longer wavelength is the K\( \alpha \) line due to transitions from the L (n=2) to the K (n=1) state.

Since the wavelengths of X-rays are so small (\( 10^3 \) to \( 10^4 \) shorter than the wavelengths of visible light), optical gratings are difficult to fabricate for study of X-ray spectra. As suggested by von Laue in 1912, one can use instead crystal lattices to reflect or diffract the X-rays and thereby determine the X-ray wavelengths. In 1913 Bragg pointed out that constructive interference would occur for reflection of radiation of
wavelength $\lambda$, from layers of atoms separated by a distance $d$. For an angle of incidence $\theta$, the condition for Bragg reflection is

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

where $n$ is an integer indicating the order of diffraction and $n\lambda$ is the optical path difference traveled for beams reflected from adjacent layers. We will use this relationship to examine the spectrum emitted from the copper anode of the X-ray tube.

We will also use the spectrum to study the properties of crystals. One generally uses the K\(_{\alpha}\) part of the copper spectrum to approximate a mono-energetic beam. The X-ray beam focused onto a crystal will reflect or diffract from the planes of atoms in the crystal into specific directions.

In a crystal lattice the $x,y,z$ directions are denoted [100], [010] and [001] respectively. For example, the [210] direction corresponds to the direction that has vector components of 2 lattice spacings in the $x$ direction and 1 lattice spacing in $y$ direction. For X-ray work one also defines the orientation of sets of crystal planes. The common notation used is Miller indices denoted (note the round brackets) as (hkl) where h,k and l are integers. For cubic lattices these are easy to visualize and determine. For a given plane the Miller indices are determined as follows: 1) find the intercepts of the plane along each axis, expressed in units of the lattice constant; 2) take the reciprocal of each of these three numbers; 3) scale the three resulting numbers to eliminate fractions (See Preston and Dietz, Ch. 10).

**Apparatus**

1. Tel-X-Ometer 580 X-ray diffractometer system including a Geiger-Mueller tube, copper anode X-ray tube, crystal mount, rotatable slide and detector mount, and scatter shield. This machine, specifically designed for student use, operates at standard X-ray energies but at somewhat lower currents than a standard machine. The machine has a dome plastic cover, which shields the X-rays. The X-rays will shut off whenever the cover is opened.

2. ratemeter
3. digital multimeter (DMM)
4. NaCl crystal
5. Ni filter and various collimators
Procedure

1) Exploring the X-ray emission spectrum of Copper

(a) Mount the NaCl crystal (TEL 582-004) in the crystal post. Place one of the short edges onto the step in the crystal post; ensure that the long broad face, with a "flat matte" appearance, a (100) cleavage plane, is butting against the chamfered protrusion of the post. Screw in the clamp until the crystal is held securely by the rubber jaw.

(b) Place primary beam collimator 582.001 in the X-ray tube port with the 1 mm slot vertical.

(c) Mount the slide collimator (3 mm) 562.016 at slide position #13 and the 1 mm collimator 562.015 at position #18.

(d) Check that the zero of the mounting plate has been set accurately and locked. (See section 10.6 page 4 of the Teltron Manual). Ensure that the correct face of the crystal is facing the X-ray source: this should be the face of the crystal that is in contact with the chamfered crystal post. Look through the collimating slits and verify that the primary beam direction lies in the surface of the crystal.

(e) Mount the Geiger-Mueller (GM) detector tube in the carriage arm behind the collimating slits in position #26. Check that its entrance slits are oriented correctly. Move the carriage arm to the side and close the dome with the spectrometer set to 30 kV. Turn on the power with the key and then rotate the timer knob to the right. Press the red button switch to initiate X-ray production. The red indicator light inside the dome should illuminate. If it doesn’t make sure the timer switch is rotated on and that the dome is closed and properly in its centered (interlocked) position.

(f) Connect the coax cable from the GM tube to the high voltage input of the ratemeter. Turn the ratemeter on using the electrical outlet strip switch. Select channel 3 to display on the ratemeter and turn knob 3 to set the GM voltage to 450 V. Turn the sound knob midway between its limits. You should hear an occasional beep indicating the detection of an x-ray. Move the carriage arm to $2\theta \sim 32^\circ$. Around this angle you
should hear an increase in counts indicating a large number of x-rays being diffracted into this angle. Measure the count rate accessed through port 1 of the ratemeter by using the DMM. The signal will be in the mV range.

(g) Record the spectrum starting at $2\theta = 20^\circ$ up to $2\theta = 120^\circ$. You should find several (up to six) strong and sharp peaks on top of a weak, slowly varying background. Try $2^\circ$ intervals except near peaks where $0.2^\circ$ steps may be more appropriate. Note that the carriage arm moves twice as fast in angle as the crystal does, in order to ensure that the reflection always occurs from the same set of atomic planes as $\theta$ is varied. Try to get a dozen data points on each significant spectral peak. Estimate your uncertainty in both the angle and in the count rate. In your notebook make a plot of Count Rate (mV) versus $2\theta$.

(h) From this tabulated data, find the characteristic wavelengths of x-ray emission from copper using Bragg’s law. Compare these wavelengths to the accepted values: $\lambda = 0.154178$ nm for $K\alpha$ and $\lambda = 0.139217$ nm for $K\beta$. What orders of diffraction did you observe? Do your measured values for $\lambda$ agree with the accepted values taking into account your uncertainties?

(i) After completing your analysis compare your results with the spectrum displayed on page 17 of the Teltron manual.

2) Observing the Absorption Edge of Nickel

(a) Insert the Ni filter 564.004 at position #17 in front of the GM detector tube. Repeat the measurement from part 1 and make a new plot of Count Rate (mV) versus $2\theta$. By comparing this plot to the previous one (with no Ni filter) you should be able to determine where the Ni edge lies. Use Bragg’s law to express the location of the Ni edge in units of nm. Include an uncertainty analysis and compare your results to the spectrum shown on page 20 of the Teltron manual.
3) Using Miller indices to predict Bragg Scattering

(a) Build a unit cell of NaCl. See the ICE Solid State State Modeling Kit pages 1-8 and 33 (face-centered cubic). Identify the (100) and the (110) planes of the crystal structure. Make a sketch that shows these two planes in the context of the NaCl unit cell. See Preston and Dietz, Chapter 10.

(b) Using $n\lambda = 2d \sin \theta$, calculate the possible first, second and third order angles $\theta$ for reflection from the (110) plane of NaCl. Use $\lambda = 0.154178$ nm for the K\text{\textalpha} line. Show your work in your notebook.

(c) Scan the carriage arm to search for the peaks predicted by your calculations in part (a). Use the same set-up as in part 2) except you’ll need to adjust the alignment of the crystal. To detect reflections from the (110) plane, the crystal must be rotated 45° so that the normal to the (110) plane is perpendicular to the X-ray beam when $2\theta = 0°$. Get help to make this adjustment to the apparatus. It will require loosening the 3 flat head screws securing the mounting plate underneath the crystal post.

(d) How many peaks did you find? Do the angular positions of the observed peaks agree with your predicted values to within your uncertainties?

References
3) Blakemore, Solid State Physics.
5) Wyckoff, Crystal Structures.

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