X-RAYS

PLG 4/06/07
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In this lab, we will gain familiarity with the equipment necessary to generate and detect x-rays, and then use x-ray diffraction as a probe of crystal structure and to determine an x-ray spectrum.

X-ray generation and detection

The basic apparatus is the Tel-X-Ometer which consists of an x-ray source, a sample holder, and a rotating platform which can hold a detector and various slits, filters, etc. The entire set-up is enclosed inside a plastic radiation cover to prevent exposure to x-rays. An interlock prevents the x-ray source from being turned on unless this radiation cover is securely closed. In addition, the x-ray tube itself is enclosed in a lead-glass housing for further shielding.

The x-rays are produced by an x-ray tube in which electrons from a filament are accelerated to high energy (typically 30 keV) and slammed into a copper anode. There are two contributions to the X-ray spectrum. When the electrons are rapidly decelerated in the target, they emit "bremsstrahlung" radiation. This gives rise to a continuous spectrum with a short wavelength cut-off determined by the energy of the incident electron beam. In addition to this continuum, the x-ray spectrum contains sharp lines (i.e., well-defined wavelengths) which are characteristic of the target. These result from ionizing collisions between energetic electrons and target atoms, causing the removal of an inner core electron. As an electron from a higher energy level drops down to fill the vacancy, an x-ray photon is emitted. The energy (and therefore wavelength) of these x-rays is determined by the difference in energy levels. In this experiment, the dominant line is the K_α ($2s \rightarrow 1s$) line of copper, which has a wavelength $\lambda = 0.154$ nm.

The x-rays are detected by a Geiger-Muller tube. This device, also used for detecting radioactivity, consists of two electrodes with gas in between. An applied potential difference produces an electric field in the gas-filled region. When an x-ray photon enters the tube, it ionizes some of the gas molecules. The resulting electrons are accelerated by the electric field and collide with other gas molecules, producing more ions and electrons. This "avalanche" process continues, giving a large pulse of charge at one of the electrodes. These individual pulses, each indicating the arrival of an x-ray photon, are counted and constitute the x-ray signal. A narrow slit in front of the tube restricts the range of angles for the detected x-rays, allowing good angular resolution when measuring an x-ray diffraction pattern.

Bragg diffraction

Recall that when x-rays are incident on a crystal, the incident angles θ for constructive interference are given by Bragg's law:

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

where n is the (integer) order, λ is the x-ray wavelength, and θ is the angle between the incident x-ray beam and the diffracting crystal planes, as shown in Fig. 1. Since θ is also the angle between the outgoing (constructively interfering) x-ray beam and the crystal planes, the angle between the incident and outgoing beams is 2θ. Note that the rotary table has the angle of the detector marked as 2θ, as is the usual case in x-ray physics.

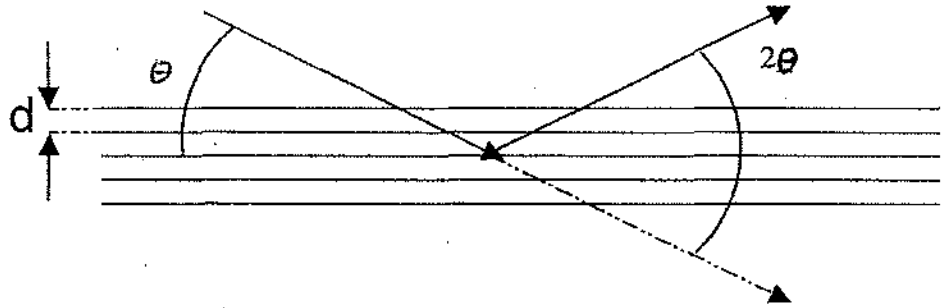

Fig. 1 Bragg scattering of x-rays.

In one experiment, we will use the Cu K_α line to locate a number of diffraction peaks from a crystal of RbCl. These measurements will then be compared with predictions based on the known crystal structure. This is an example of x-ray crystallography. In the other experiment, we will use the angular pattern of x-rays diffracted from Ge {1,1,1} to determine the x-ray continuum spectrum and the cut-off wavelength. This is an example of x-ray spectroscopy.

Procedure

You should first verify that the x-ray source is working. Place the phosphor screen in the filter holder and align it directly across from the x-ray source, i.e., at 0° on the rotary table. Close the plastic cover and shift it until it "clicks". This is the interlock which allows the x-ray source to be turned on. Note that the "power on" key must also be engaged and the timer must be activated in order for the "x-rays on" switch to work. Since x-rays are not visible to our eye, we use a phosphor screen which gives off visible light when illuminated with x-rays. With the x-rays on, you should see a faint glow on the phosphor screen. It may be necessary to turn off the room lights and/or cover the x-ray tube with aluminum foil (which blocks light from the glowing filament, but transmits the x-rays) in order to see the glow.

In the first experiment, you will look at the diffraction pattern from a crystal of RbCl. Carefully place the crystal in the sample holder, making sure that a flat surface of the crystal is flush with the vertical surface of the holder. In order to have a collimated x-ray beam incident on the crystal, place the 1 mm slit (oriented vertically) in the opening in front of the x-ray tube. The Geiger-Muller detector should be placed in the radial spectrometer arm. For good angular resolution of the diffracted x-rays, place a 1 mm slit (oriented vertically) in front of the detector. The signal from the detector should be connected to the counter (TEL-Atomic Digicounter). Starting at the minimum possible angle, measure the number of counts at 1° intervals, up to a maximum angle of $2\theta = 60°$.

You should see several peaks within this range. In the vicinity of each peak, you may want to take smaller angular steps. The counting rates may not be very high, so to reduce your statistical uncertainties, you should take three measurements at each angle, counting for 10 seconds each time. Make a plot of x-ray counts vs. 2θ. Identify the observed peaks and match them with predictions based on the data in Table 1. Note that for each set of planes (labeled by the Miller indices {h,k,l}), you need to apply Bragg's law to determine 2θ. Use n=1 and assume that the x-rays are from the Cu K_α line.

h	k	l	d (nm)	Intensity
1	1	1	0.371	60
2	0	0	0.321	80
2	2	0	0.227	80
2	2	2	0.185	100
4	0	0	0.160	60

Table 1 Data for RbCl. {h,k,l} are the Miller indices for the planes responsible for the reflection, d is the spacing between planes, and I is the relative intensity in the powder pattern (an x-ray pattern from a large array of powdered particles).

In the second experiment, you will use a Ge {1,1,1} crystal to measure the x-ray continuum spectrum. Carefully place the thin section of germanium wafer in the sample holder, again making sure that one of its flat surfaces is flush with the vertical surface of the holder. As in the first experiment, record x-ray counts as a function of 2θ. You should see a small signal at all angles, with one strong peak superimposed. Here, we are primarily interested in the small signals. The large peak is the Cu K_α Bragg peak (n=1) from the {1,1,1} planes, which have a spacing d = 0.325 nm. Calculate where this peak should be and compare to your measurement.

Now make a plot of x-ray signal vs. θ. The continuous (small) signal is due to the presence of various wavelengths in the x-ray spectrum. We would like to determine this spectrum. Assuming that Bragg diffraction is only possible from the {1,1,1} planes, each wavelength will give a signal at a unique angle, as given by Eq. (1). The data gives I(θ), the intensity (per unit angle) as a function of θ. We would like the actual x-ray spectrum I(λ), the intensity (per unit wavelength) as a function of λ. This transformation must be done carefully – it is not simply a matter of transforming the axes from θ to λ. Spectra are measured at constant angular aperture dθ. At each angle θ, the total x-ray intensity in a small range dθ (determined by the slit width) is detected: I(θ)dθ. However, in the intensity vs. wavelength spectrum, it is the wavelength aperture dλ which is constant. For each value of λ, the total intensity of x-rays within a range dλ, centered at λ, is given by I(λ)dλ. The functions I(θ) and I(λ) are related by: $I(\theta)d\theta = I(\lambda)d\lambda$. Using Eq. 1 to transform a range of angles dθ into a range of wavelengths dλ, show how to transform from I(θ) to I(λ). Check your argument with the TA. Using this conversion, make a plot of the x-ray spectrum I(λ). From this plot, extrapolate to find where the intensity goes to zero at short wavelengths. This is the cut-off wavelength. Compare this with the predicted cut-off wavelength based on the 30 kV accelerating voltage.

Finally, use a similar procedure to convert the wavelength spectrum $I(\lambda)$ to the energy spectrum I(E). Recall that the photon energy (in eV) is related to the wavelength (in nm) by: $E = hc / \lambda = 1240 \text{ eV nm} / \lambda$. Again, check your argument with the TA. Make a plot of I(E). From this plot, extrapolate to find where the intensity goes to zero at high energies. Is this cut-off energy consistent with the cut-off wavelength obtained above?