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Bragg Scattering

GOAL

- To see evidence that atoms in a crystal are arranged periodically.
- To acquire experience in counting statistics and its associated error.
- Learn the technique of background subtraction.
- To study the production and uses of X-rays.

X-ray Production

Broadly speaking, X-rays (photons with wavelengths from about 0.01 nm to about 0.1 nm) are produced when high-energy electrons collide with matter. We will be interested in the wavelength spectrum of these X-rays. A typical X-ray production device is illustrated in Fig. 2. A potential difference is maintained between the cathode (usually a hot tungsten filament) and the anode, usually a metal with a high atomic number. The anode in our X-ray tube is copper, Z=29.

The heated hot cathode emits electrons via "thermionic emission." Electrons near the surface of the metal find themselves in a potential well of some depth and can be considered free since the cathode is metal and therefore, to a good approximation, an equipotential, implying an electric field $\vec{E} = -\nabla\phi = 0$. Inside the well, these electrons occupy a stack of energy levels (think of particles in a box from your modern physics course or particles in a simple harmonic oscillator potential well), consistent with the detailed shape of the well and the Pauli exclusion principle. It is useful to note the minimum energy required to lift an electron in the top most energy level completely out of the well so that it is no longer confined in the metal. We call this particular energy the "work function" of the metal, conventionally labeled ϕ, where this ϕ is a completely different quantity from any potential we may apply to the metal, also often labeled with the very same symbol! A typical work function for a metal is a few eV. See Fig. 1.

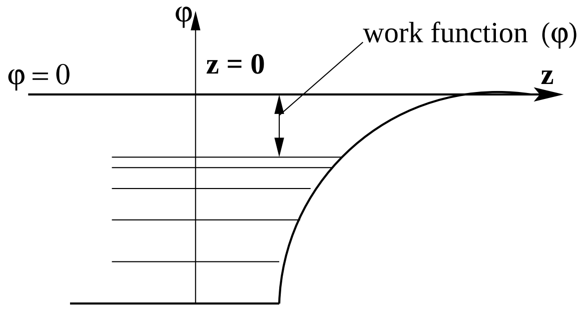

Figure 1: Energy levels of electrons inside a metal.

As the metal cathode is heated, energy is transferred to the electrons in the potential well, exciting some of them, and if an electron acquires sufficient kinetic energy, it can escape the well. Accounting for Fermi-Dirac statistics, the current density J (amps/m^2) due to electrons escaping from the metal surface at a temperature T is given by *Richardson's* equation,

$$J = A_0 T^2 \exp(-\phi/kT),$$

where A_0 is a constant, ϕ is the metal's work function and k is Boltzmann's constant. The exponential term dominates the thermal behavior of the emission.

After being accelerated toward and striking the anode, some filament electrons scatter off of anode electrons, emitting *bremsstrahlung* photons and slowing in the process. Some of the filament electrons knock out inner atomic shell electrons in the anode and the resulting vacancies in these shells are then filled by the atom's other electrons. The photons emitted when the vacancies are filled have a discrete wavelength (i.e., the *characteristic spectrum*) that depends both on the shell that has a vacancy and the shell from which the vacancy filling electrons originates. (This should be familiar to you from your knowledge of quantum mechanics.) The overall X-ray tube emission spectrum is then the superposition of a broad, relatively featureless spectrum from the bremsstrahlung emission, with a spectrum that has sharp "spikes" corresponding to vacancy filling electronic transitions.

There are 2 dominant X-ray wavelengths emitted by the Cu anode and each corresponds to electronic transitions that fill vacancies in copper's K shell (i.e., principal quantum number $n = 1$). If the vacancy filling electron originated from the L shell ($n = 2$), the emitted X-ray is called a K_α X-ray and its wavelength $\lambda = 0.154\,\text{nm}$. If the vacancy filling electron originated from the M shell ($n = 3$), the emitted X-ray is called a K_β X-ray and its wavelength $\lambda = 0.138\,\text{nm}$

To get a feeling for the relationship between a photon's energy and its wavelength, you may find the following formula useful for converting between photon energy, when measured in electron-Volts, and photon wavelength, when measured in nanometers:

$$E_\gamma\,(\text{eV}) = \frac{1240\,\text{eV} - \text{nm}}{\lambda_\gamma\,(\text{nm})}$$

X-ray Absorption

The intensity of a photon beam decreases exponentially with distance through an absorbing material. This process is described by the relation

$$I = I_0 e^{-\sigma_a n t},$$

where t is the thickness of the material, σ_a is the cross-section for absorption, and n is the number of atoms per cubic centimeter of the material. There are three important processes for photon absorption: the photoelectric effect at low energies, Compton scattering at intermediate energies and pair production at high energies. The total absorption cross section is then given by

$$\sigma_a = \sigma_{pe} + \sigma_{cs} + \sigma_{pp}$$

It is convenient to combine the absorption cross-section σ_a with the atomic number density n and rewrite the above equation governing the intensity of the transmitted photon beam as

$$I = I_0 e^{-\mu t},$$

where $\mu = \sigma_a n$ is the total absorption coefficient. Given a collimated, monochromatic X-ray beam, it is possible to measure μ by measuring the intensity of the transmitted beam for various thicknesses of the material. We will do this using aluminum.

Bragg Scattering

It is possible to measure the wavelength of X-rays by scattering them from a crystal with several known properties. This is known as the Bragg diffraction method. An implicit assumption accompanying this technique is that atoms of such a crystal are arranged in a regular cubic three-dimensional pattern as shown in Fig. 3. The inter-atomic spacing d may be calculated using known properties of the material such as the molecular weight M, the density ρ, and the Avogadro number N. For example, NaCl has the following properties:

- $M = 5.846 \times 10^{-2}\ kg/mole$
- $N = 6.02 \times 10^{23}\ molecules/mole$
- $\rho = 2.16 \times 10^{3}\ kg/m^3$

Since NaCl is diatomic, the number of atoms per unit volume is $2\rho\frac{N}{M}$. The distance between the atoms in the lattice, therefore, is obtained from the equation

$$d^3 = \frac{1}{2\rho N/M}; \quad d = (M/2\rho N)^{1/3}$$

The first condition for Bragg reflection is that the angle of incidence θ equals the angle of reflection. This is identical to optical reflection and implies that any detector of the reflected rays must move through an angle 2θ as the crystal is rotated through the angle θ from the position that gives direct back-reflection. The second condition is that reflections from several lattice planes must add constructively (see Fig. 4):

$$n\lambda = \overline{AB} + \overline{BC} = 2d\sin\theta$$

With these two conditions in hand, the wavelength of X-rays may be determined by measuring the intensity of the scattered light as a function of the scattering angle.

Peak hunting and errors

When you hunt for peaks in your Bragg diffraction spectra, you will need to make a judgement about how large a count rate needs to be at a given value of 2θ to actually call it a peak. How do you estimate the expected variation in the count rate at an arbitrary value of 2θ? For example, if you measure 1025 counts in a particular 30-second time interval at $2\theta = 30$ degrees, what is a reasonable estimate for the *uncertainty* in the count rate per second? If you repeat the measurement a second time, you do not expect necessarily to collect the *exact* same number of counts. When you move to a new value

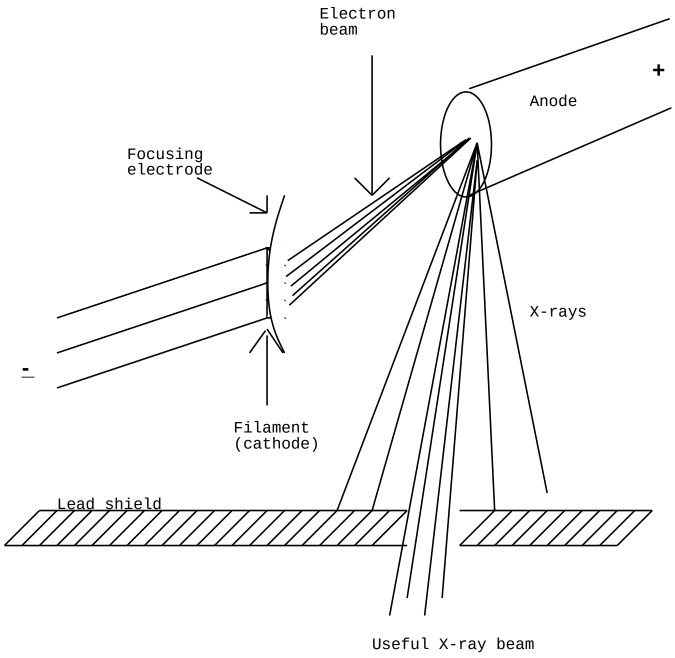

Figure 2: Schematic of a typical X-ray production device. Normally, the entire setup is contained inside a vacuum tube.

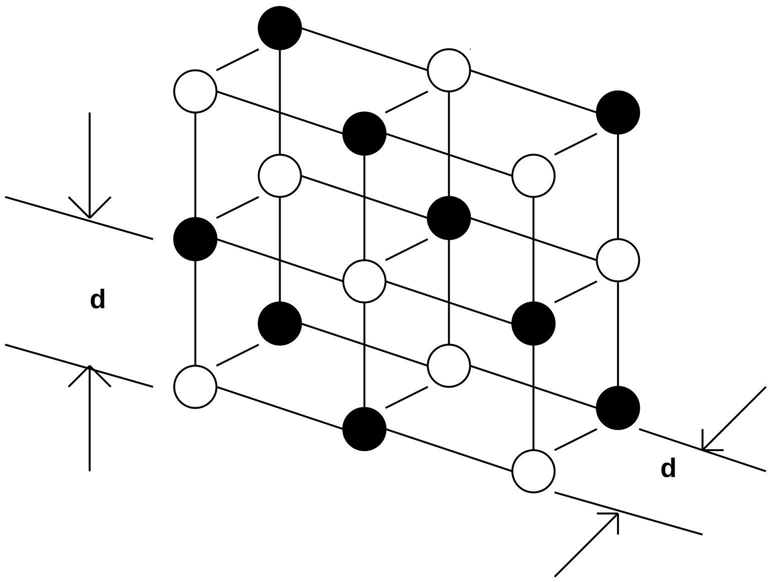

Figure 3: Representation of NaCl crystal.
Regular cubical representation of a NaCl crystal. The distance between atomic centers is d.

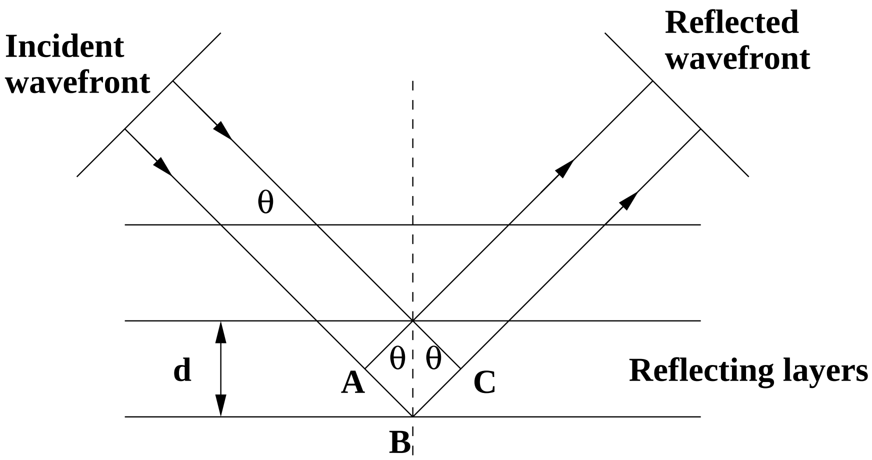

Figure 4: Schematic of Bragg reflection.
Schematic representation of Bragg reflection from crystal planes.

of 2θ and perform a new measurement, you would like to know whether an increased count rate is a genuine real peak due to Bragg diffraction or just a statistical fluctuation.

If each of the counts we record in our temporal counting interval is independent of every other count, then Poisson statistics applies and the total number of counts in our interval will be distributed according to a Poisson distribution. This means that variance in the number of recorded counts will be related to the mean number of counts. Recall that for a Poisson distribution with mean μ, the variance $\sigma^2 = \mu$. Now, if we take a *single* run at some fixed value of 2θ and record N events, then our estimate for the mean number $\bar{m}$ of counts in each counting interval is $\bar{m} = N \pm \sqrt{N}$.

Scattered photon detector

The device used to detect the scattered photons is relatively simple. It is a gas-filled (typically a noble gas and an organic vapor) tube with electrically conducting walls that has a thin metallic wire strung along its symmetry axis. The wire is biased at a positive voltage (say, 800 volts or so) with respect to the cylinder walls to produce an electric field inside the tube (provision is made to electrically isolate the wire from the tube walls). See Fig. 5.

An x-ray photon strikes the cylinder walls or the front face of the "Geiger-Müller" (GM) tube and an electron is ejected into the gaseous part of the GM tube through the compton effect.(The gas mixture itself has a density too low to be an efficient target for x-ray photons.) The liberated electron is then accelerated by the tube's ambient electric field toward the central wire.

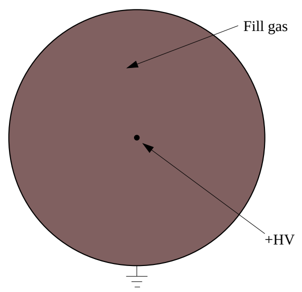

Figure 5: Cross-sectional view of a GM tube.
The central wire is biased at a positive high voltage while the outer wall is grounded. The region between is typically filled with a mixture of argon and an organic gas.

Eventually, the electron is sufficiently energetic to ionize additional gas atoms whose liberated electrons are subsequently accelerated, repeating the collision and ionization process. The result is a large "splash" of charge arriving at the central wire. This pulse of electric current ("count") is detected by electronic circuitry inside the readout box attached to the central wire by a coaxial cable. After the splash, the detector is typically insensitive to any further flux of x-rays for something like 100 μsec due to the positive ions drifting to the outer wall. The typical detection efficiency (the ratio of the number of detected photons to the total number of incident photons) is typically 1-2%. An appropriate working voltage for the GM tube can be found by adjusting the voltage of the central wire so that for a steady incident photon flux, the counting rate is constant with respect to small changes in the central wire voltage.

Literature

1. "Modern Physics, 3rd ed." P.A. Tipler and R.A. Llewellyn, Freeman, New York, 1999, sec. 3-4.

2. "Modern Physics," R.L. Sproull, John Wiley & Sons, New York, 1964, pp. 91-99.

3. Stephen T. Thornton and Andrew Rex, "Modern Physics for Scientists and Engineers," Saunders College Pub., 1993.

4. Adrian Melissinos, "Experiments in Modern Physics,"Academic Press, 1966.

5. E. Segrè, "Nuclei and Particles" 2ed., Benjamin/Cummings, 1977.

6. R. Evans, "The Atomic Nucleus," McGraw-Hill, New York, 1955.

INSTRUCTIONS

I. Equipment List

- Tel-X-Ometer, TEL 580M
- Geiger-Muller tube, TEL 547
- ST360 Counter
- Collimators
- NaCl crystal
- Al foil slides
- Micrometer

II. Setting the GM tube high voltage

You need to set the high voltage on the GM's central wire so that the tube efficiently counts photons. This is done by measuring the "plateau" curve of the GM tube. See the Melissinos or Segrè reference for more details. This activity will also verify that the x-ray tube and the GM tube still work. (You never know...)

- Place the tubular collimator (TEL 562.002) into the x-ray tube exit flange.
- Place the GM tube into slot 26 of the carriage arm.
- Place the slit collimator (TEL 662-016) into slot 13 of the carriage arm. The slit should be vertical.
- Place the narrow slit collimator (TEL 662-015) into slot 18 of the carriage arm. The slit should be vertical.
- Connect the GM tube's coaxial cable to the BNC connector on the back of the ST360 counter labeled "GM".
- Set the ST360 to count for 30 sec.
- Set the high voltage on the ST360 to 700 volts.

- Collect data for 30 seconds and record the total number of counts on the ST360.
- Make multiple runs, each time increasing the high voltage by 40 volts until you have scanned the high voltage range 700-1000 volts. Record the total number of counts for each run.
- Plot total count per 30 second run versus high voltage. Include error bars. This plot belongs in your log book and in your final lab report.

III. X-Ray Absorption

- Select a high voltage for the GM tube based on the previous exercise.
- Insert collimators as in section II.
- Insert the GM tube into slot 26 or so of the carriage arm.
- **Verify** you can close the lid and that the X-ray beam would strikce the GM tube at normal incidence.
- Insert a slot collimator into the holder, just in from of the GM tube.
- Connect the GM tube output to the ST360.
- Turn on the X-ray beam and take a 30 sec reading.
- Insert one of the aluminum targets just before the GM tube. the X-ray beam. Take another 30 second reading.
- Repeat the last two steps using a variety of Al foil target thicknesses. The table below is a template. Actual target thicknesses may vary. the following table.

No. Slides	Thickness (mm)	I cps	$\log I$	$\log I_0 - \log I$
1	0.0		–	
2	0.06			
3	0.12			
4	0.24			
5	0.36			
6	0.48			
7	0.60			
8	0.72			
9	0.84			
10	0.96			
11	1.08			
12	1.20			
13	1.32			
14	1.44			
15	1.56			
16	1.68			
17	1.80			
18	1.94			
19	2.06			
20	2.18			

- Plot a graph of I *vs. Thickness.* Theoretically, this is exponential curve where $I = I_0 e^{-\mu t}$ and $\mu = \frac{log(I_0/I)}{t}$ is the total absorption coefficient.
- Now plot $Log(I_0/I)$ vs. *Thickness.* From the equation above, this curve should be a straight line with slope μ. Note that it deviates from our simple theory.

IV. Bragg Scattering

- Mount a NaCl crystal in the crystal post. The crystal must be vertical. Is it?
- Insert the 1mm collimator into the basic port such that the slot is vertical.
- Mount the 3mm slot collimator slide in position 13 and the 1mm slot collimator slide in position 18.
- Zero-set and lock the carriage arm cursor as precisely as possible.

- Sight through the collimating slits and observe that the primary beam direction lies in the surface of the crystal. Is it?
- Mount the GM tube and it's holder in position 26.
- Connect the elapsed time module and digital scalar as before.
- Set the carriage arm at $2\theta = 11^o$, turn the X-rays on and take a reading for 30 seconds.
- Repeat the measurement in one-half degree increments, plotting $I(cps)$ vs. 2θ for all angles up to $2\theta = 124^o$ as you go. When you notice the interference peaks, take measurements at every $10'$ of arc, using the thumb wheel on the carriage arm.

You have now measured the diffraction angle spectrum for NaCl, from which you can calculate the "effective" wavelength of the X-radiation. You will notice, however, that a sizeable background appears under the spectrum. One technique that experimenters frequently employ is that of subtracting a known background. To employ this technique, simply remove the crystal from the crystal post, and repeat the above steps. What you are now measuring is the background level for each angle, in absence of the diffraction. Subtract the background ($I_{background}$) from the signal determined above and use the results to complete the following table.

Feature	2θ	θ	$\sin\theta$	$2d$ (nm)	$n\lambda$	λ
1				0.564		
2				0.564		
3				0.564		
4				0.564		

The Mystery Crystal

Now insert the "mystery" crystal into the crystal post. Repeat the above experiment using the unknown crystal. Using the determined wavelength of the X-rays, determine if this crystal is NaCl.

V. Questions

- Explain qualitatively why the "Total counts versus high voltage" curve has the shape it does.

- You might expect that the graph of $\log(I/I_0)$ *vs. Thickness* should be a straight line with slope μ. Propose an explanation as to why the curve deviates from linearity at larger thicknesses. (Hint: what is meant by the wavelength of the X-rays which we are measuring?)

- From the results of the Bragg refraction experiment, calculate the effective wavelength of the X-rays in the experiment. Don't forget to include and propagate all sources of error.

- Measure Avogadro's number using your data. (If you make assumptions, fine. Just list them.)