

DIFFRACTION FROM CRYSTALS: MATTER WAVES

INTRODUCTION

The experiments performed in this lab will demonstrate the dual wave/particle nature of matter. Matter is said to have a dual nature because depending on the type of measurement being made matter may seem to be composed of either particles or waves. As an example consider Young's famous experiment in which an interference pattern is produced on a photographic plate by passing a beam of light (photons) through two closely spaced slits. Interference is solely a wave phenomena inferring that the motion of the photons must be wavelike. However, upon close examination of the photographic plate it is seen that the interference pattern is composed of numerous individual spots. Spots where individual photon particles hit! In Young's experiment it is found that light propagates like a wave but is detected by the photographic film as individual particles! In this lab we will examine a similar phenomena; diffraction of an electron beam by a crystal lattice (diffraction is another wave phenomenon caused by interference.)

The discovery that matter has a dual wave/particle nature was one of the fundamental underlying developments of modern physics. It was predicted in 1924 when a French student, L. deBroglie suggested in his doctoral thesis that the wavelength of a matter wave was given by the same equation as for light.

$$\lambda = \frac{h}{p} \quad (1)$$

In this equation h is Planck's constant and p is the momentum of the particle. As support to this highly speculative proposal deBroglie pointed out that by substituting $p = h/\lambda$ into Bohr's quantum condition for the stable orbits of electrons in a hydrogen atom,

$$\text{Angular Momentum} = m v r = \frac{n h}{2 \pi} \quad (2)$$

one gets,

$$n \lambda = 2 \pi r = \text{Circumference.} \quad (3)$$

If deBroglie's relation was correct it suggested that Bohr's stability condition was equivalent to stating that an integral number of standing electron waves must fit around the circumference of the electron orbits. In 1925 Erwin Schrodinger developed a the mathematica theory of wave mechanics which he used to describe the motion of electrons in an electric field. His results provided further support for deBroglie's realition.

The first experimental evidence of the existence of the wave nature of electrons was discovered accidentally by Davison and Germer in 1927 at Bell Telephone Laboratories. When heating an aluminum electron beam target to evaporate the oxide coating that had formed on the surface they inadvertently crystallized

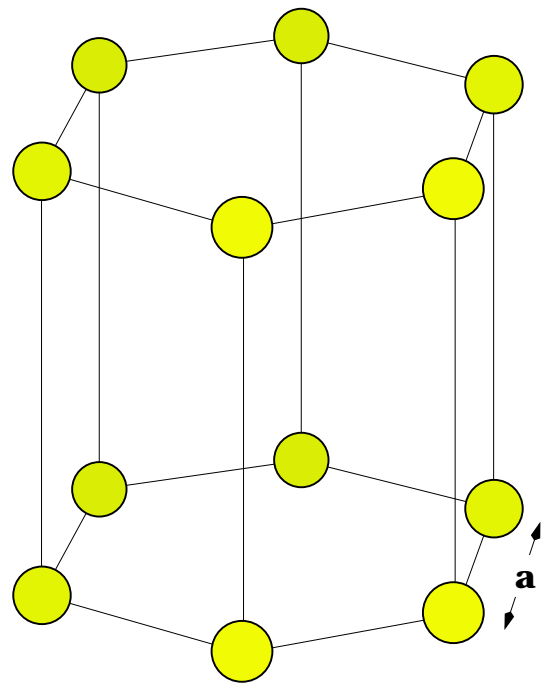


Figure 1 Hexagonal Crystal Structure

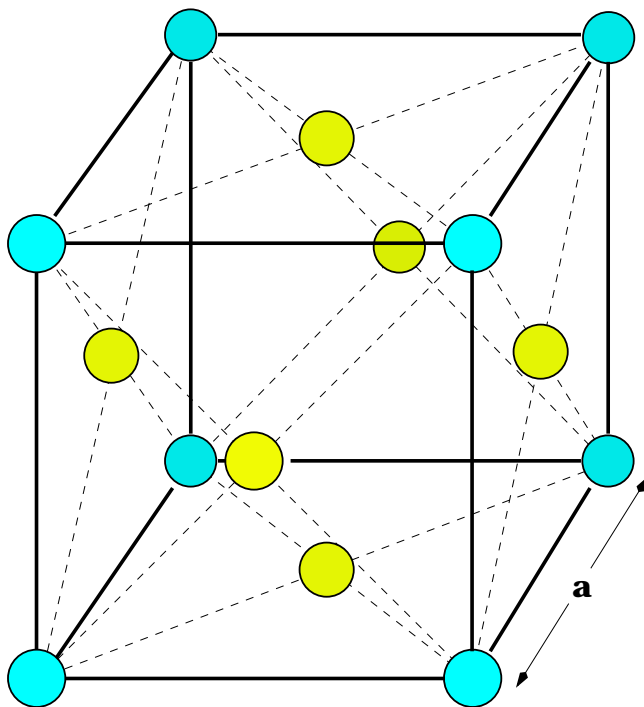


Figure 2 Face Centered Cubic Crystal Structure

it. Afterwards they discovered that the intensity pattern of 'slow' electrons scattered off the aluminum target showed minimum and maximum. Subsequently the two scientists prepared a single crystal of aluminum to use as their target. By measuring locations of the minima and maxima of the resulting diffraction pattern, the wavelength of the beam was calculated and shown to match the deBroglie relation exactly. DeBroglie's theories were further confirmed that year by similar experiments performed by G. P. Thompson using polycrystalline metallic foil targets. He found that identical patterns were produced by both electron and X-ray beams when they were of the same wavelength.

CRYSTAL LATTICE STRUCTURE

A crystal is a three dimensional array of atoms or molecules arranged in a regular pattern which consistently repeats itself at definite spatial intervals. A crystal lattice may be viewed as being composed of many atomically thin layers of atoms/molecules stacked on top of each other. A basic unit of a hexagonal lattice structure like that formed by hexagonal pyrolytic graphite is depicted in Figure 1. For a given crystal type there are many ways in which the crystal lattice may be 'sliced up' into identical repeating layers or planes. Such a plane is specified in terms of its Miller indices (H K L). Miller indices describe the angle of the direction normal to a plane with respect to fundamental crystal lattice unit. Some of the planes of a face-centered-cubic type lattice and the corresponding Miller indices is shown in Figures 2 and 3. The face-centered-cubic lattice is a crystal structure formed by many metals (aluminum, gold silver, nickel), alkali halides such as table salt, and a large number of other substances.

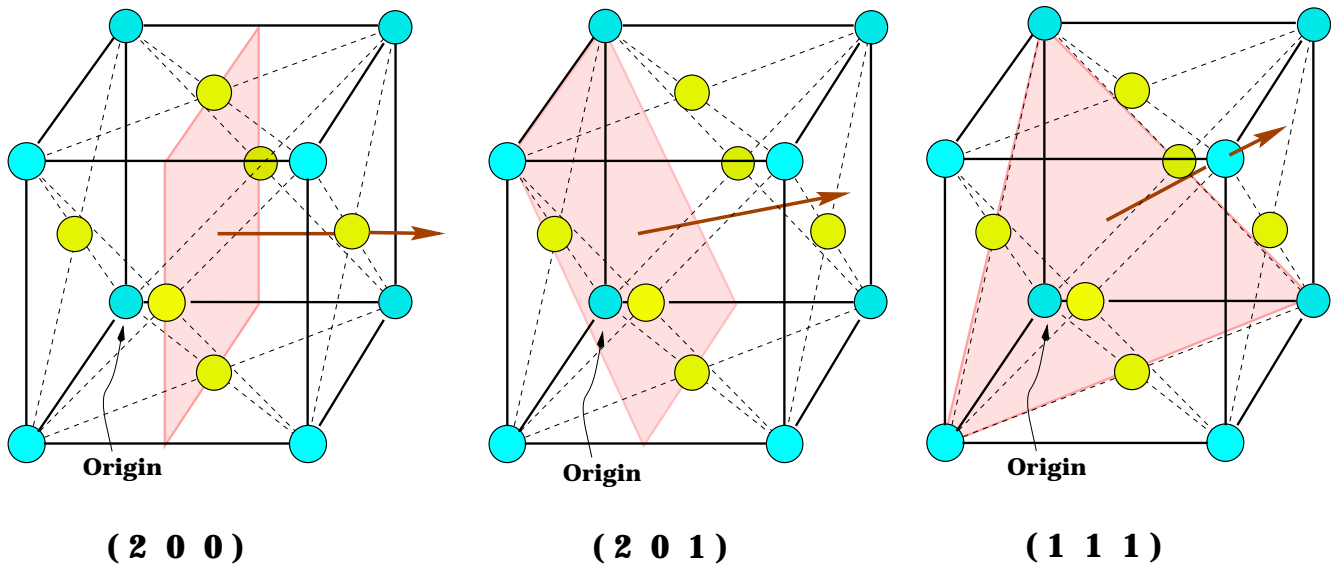


Figure 3 Miller Indices of Reflection Planes for Face Centered Cubic Lattice

BRAGG'S LAW

When a crystal lattice is exposed to a wave of photons (electromagnetic waves), electrons, protons, or other particles, each atom/molecule interacts with the wave and becomes a secondary wave source (essentially absorbing and then re-emitting a particle of the original wave.) These secondary waves may sum constructively or destructively with each other and the original wave at different points in space as the waves propagate away from the crystal. The resulting diffracted wave consisting of the original wave plus the sum of the secondary waves will exhibit a specific pattern of minima and maxima corresponding to the crystal type and the wavelength of the particle beam.

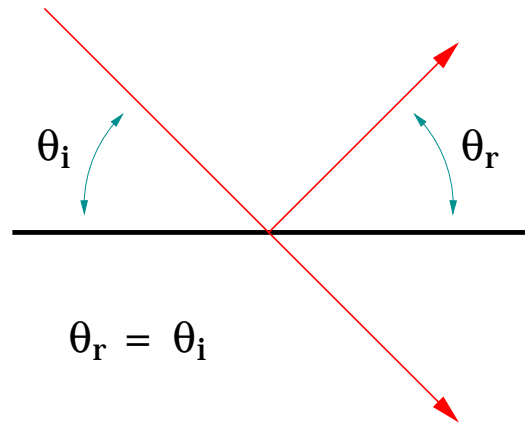


Figure 4 Snell's Law
Angle of Incidence Equals
Angle of Reflection.

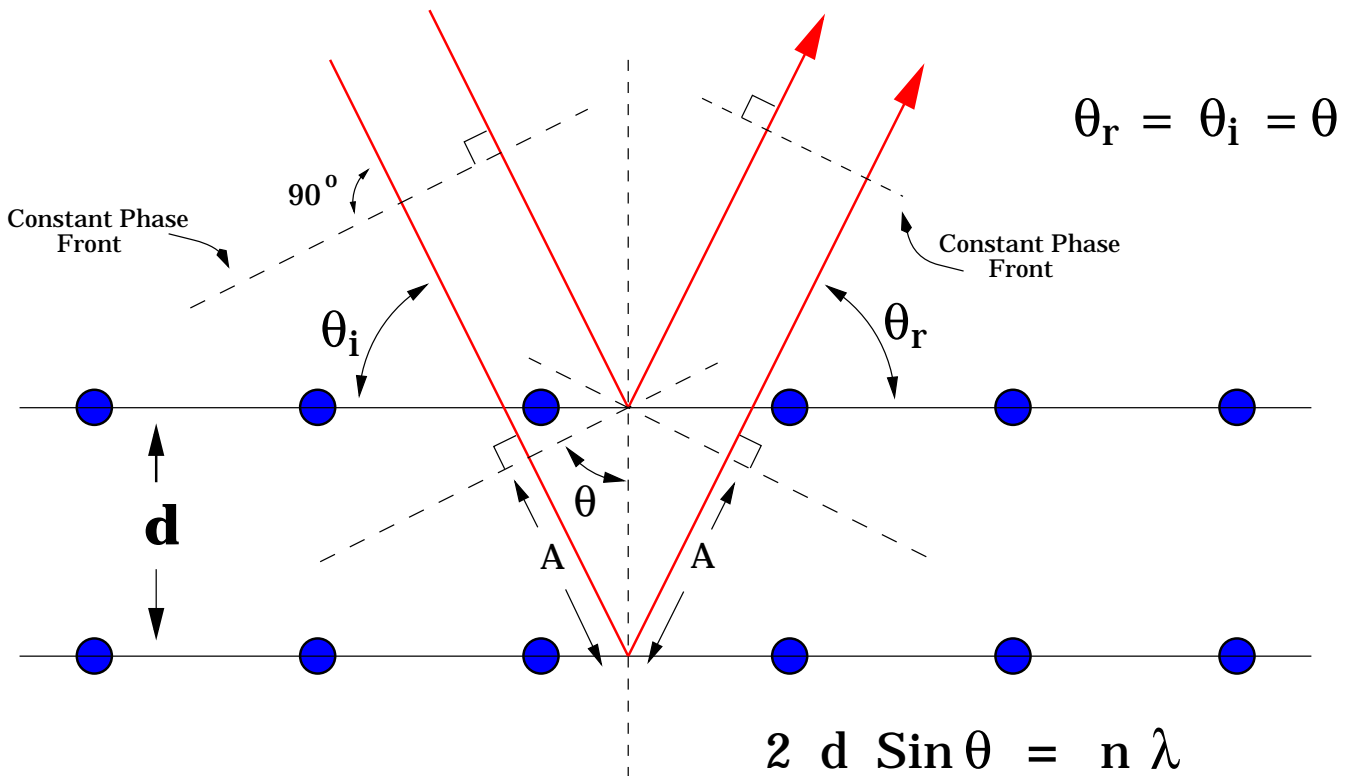


Figure 5 Derivation of Bragg's Law

In order for a plane wave to maintain a constant wave front upon reflection from a crystal lattice plane, the difference in distance traveled between the rays 1 and 2 in the figure above must be an integral number of wavelengths.

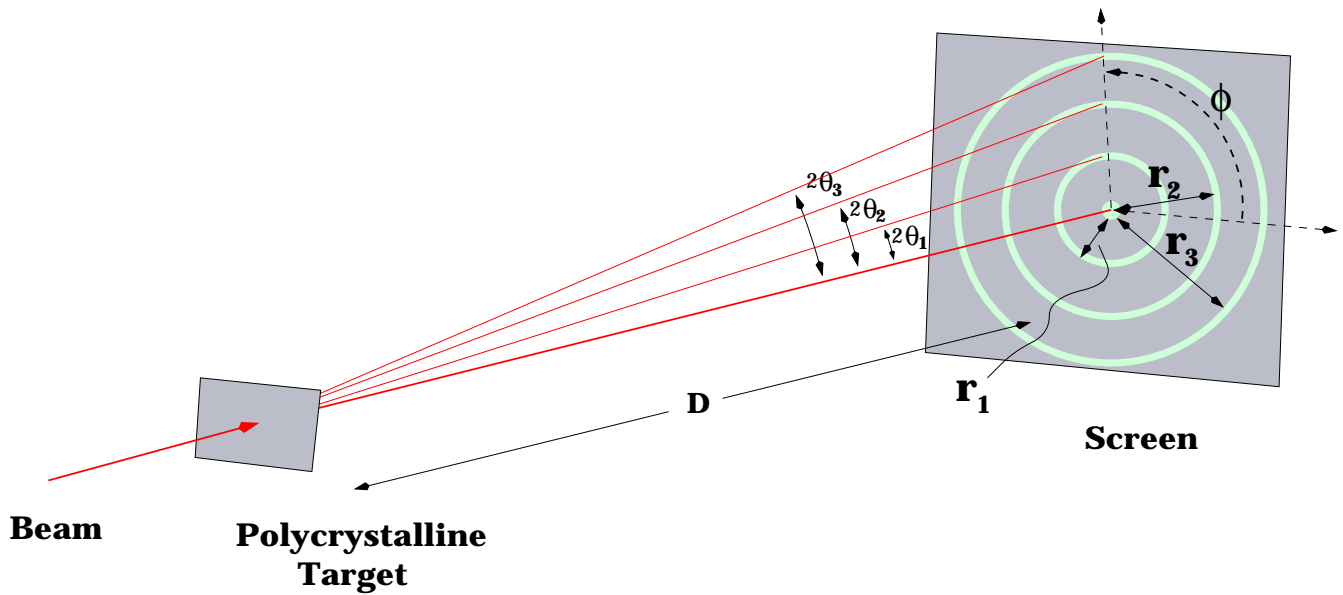


Figure 6 Rings Formed by Diffraction from a Polycrystalline Material

A simple condition describing the directions in which the secondary waves will add constructively is given by Bragg's equation,

$$2 d \sin \theta = n \lambda \tag{4}$$

where d is the separation between a particular set of planes of the lattice structure, θ is the angle between the beam and the surface of these planes, and λ is the wavelength of the beam. This relation is the primary basis of the phenomena to be measured in this lab.

In order to derive Bragg's equation it is first assumed that any plane of a crystal lattice can act as a partially reflecting mirror obeying Snell's law. This law states that a wave incident on a plane of the crystal lattice will reflect at an angle equal to the angle of incidence (Figure 4.) Secondly, Bragg's equation states that a true reflection will only be seen when the sum of small individual reflections formed by a stack of crystal planes adds constructively or in phase. Examining Figure 5 it is seen that for a plane wave to retain a constant flat wavefront upon reflection, the distance $2 A$ must be an integral number of wave lengths.

$$2 A = n \lambda \tag{5}$$

Substituting,

$$A = d \sin \theta \tag{6}$$

into Eq.5 gives Bragg's Law,

Bragg's Law	$2 d \sin \theta = n \lambda$
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(7)

WAVE DIFFRACTION FROM POLYCRYSTALLINE ALUMINUM

Upon examination under a microscope many metals such as aluminum foil are found to be composed of millions of tiny randomly oriented crystalline domains. Such a state is often called polycrystalline. When a beam of electrons is transmitted through a thin film of polycrystalline material a bullseye pattern consisting of a bright center spot (corresponding to direct transmission of the incident beam) along with many surrounding rings is seen (see Figure 6.) Each ring corresponds to one of the material's lattice planes for which there is an angle of reflection satisfying Bragg's law.

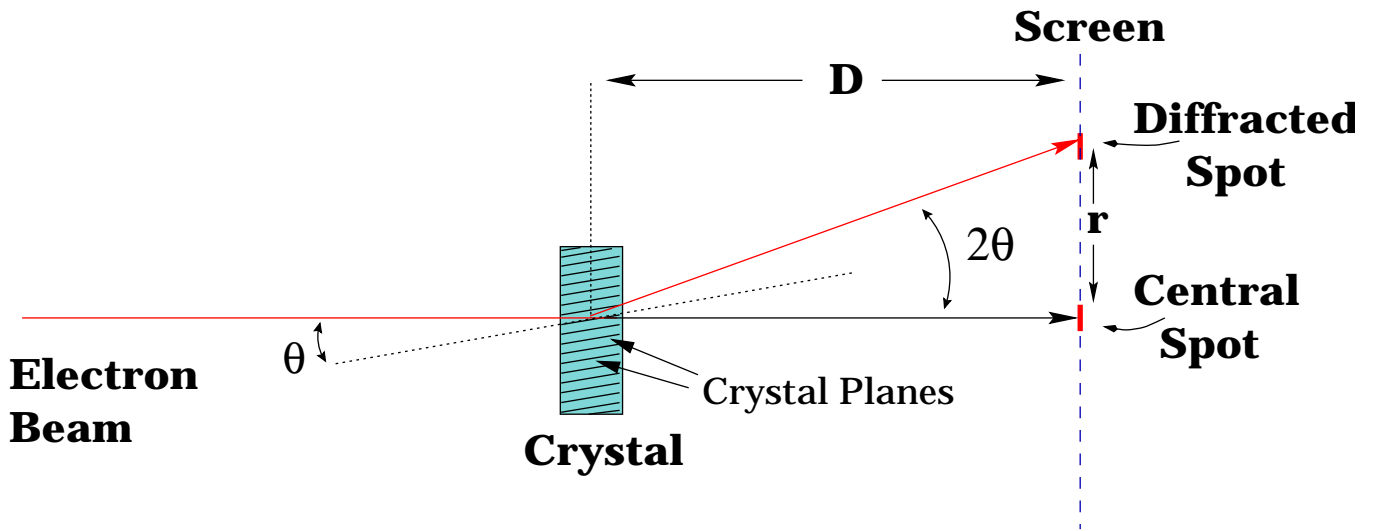


Figure 7 Crystal Diffraction Geometry

The total angle of a diffraction maxima due to reflection off of a particular crystal lattice plane is twice the angle of incidence on that plane. This angle may be approximated as the distance of the diffraction spot from the undiffracted beam over the distance between the imaging screen and the crystal, i.e. $q \approx 1/2 r/D$.

For example suppose that the separation between a particular set of the family of lattice planes of face-centered cubic aluminum is 2.02 Angstroms and the electron beam has a wavelength of 1.0 Angstrom. Bragg's equation is satisfied for first order reflection ($n=1$) at the angle

$$\theta = \sin^{-1}\left(\frac{n \lambda}{2 d}\right) = \sin^{-1}\left(\frac{1 * 1 \text{ \AA}}{2 * 2.02 \text{ \AA}}\right) = 14.33^\circ \quad (8)$$

When a crystal is oriented such that the electron beam is incident on these lattice planes with the calculated angle it will reflect a portion of the beam away with a total deviation of 2θ (see Figure 7). Due to the millions of randomly oriented crystals in a polycrystalline material there will be many crystal domains that satisfy these conditions. Although, Bragg's condition is satisfied for only particular values of the azimuthal angle θ , it may be satisfied for any polar angle ϕ . As a result rings are formed instead instead of spots (see Figure 6). An analogy is the ring pattern one sees if a board with many dots on it is spun quickly. Our eyes see the spots at all angles of rotations and the spot pattern blurs into a set of rings.

The distance between the reflection planes of face-centered cubic lattices corresponding to the Miller indices $[H K L]$ is given by,

$$d = \frac{a}{\sqrt{H^2 + K^2 + L^2}} \quad (9)$$

where a is the length of the edge of the unit cell (see Figure 2). The accepted value of a for polycrystalline aluminum is $a = 4.0496 \pm 0.0001 \text{ \AA}$.

Substituting Equation 9 into Bragg's equation and solving for λ gives,

$$\lambda = \frac{2 d \sin \theta}{n} = \frac{2 a \sin \theta}{\sqrt{(n H)^2 + (n K)^2 + (n L)^2}}. \quad (10)$$

Higher order reflections from planes $[H K L]$ are considered to be equivalent to first order reflection from the planes $[h k l] = [nH nK nL]$.

When the angle of diffraction is small, $\sin \theta$ may be approximated as,

$$\sin \theta \approx \theta \approx \frac{r}{2D} \quad (11)$$

where D is the distance between the crystal and detecting screen and r is the radius of the ring (see Figure 6.) This produces,

$$\lambda = \frac{2 a \sin \theta}{\sqrt{h^2 + k^2 + l^2}} = \frac{a r}{D \sqrt{h^2 + k^2 + l^2}}. \quad (12)$$

The intensity of reflection is proportional to,

$$I(h k l) = 1 + e^{i \pi (h+k)} e^{i \pi (k+l)} e^{i \pi (l+h)} = 1 + (-1)^{(h+k)} (-1)^{(k+l)} (-1)^{(l+h)} \quad (13)$$

which equals 4 when h , k and l are all even or odd and zero otherwise. The allowed Miller indices of face-centered cubic structures for which rings will occur are given in Table 1.

WAVELENGTH OF AN ELECTRON BEAM

The wavelength of an electron beam may be calculated from the voltage difference between the cathode (emmitter) and anode. Assuming the velocity of the electrons are significantly less than the speed of light then the nonrelativistic equation relating energy to momentum and velocity can be used. If the potential difference is V volts then the electrons will reach a kinetic energy of,

$$\text{K.E.} = \frac{1}{2} m v^2 = \frac{p^2}{2m} = q V \quad (14)$$

when they reach the anode (where m and q are the mass and charge of an electron.)

$h k l$	$(h^2 + k^2 + l^2)$	$\sqrt{h^2 + k^2 + l^2}$
1 1 1	3	1.732
2 0 0	4	2.000
2 2 0	8	2.828
3 1 1	11	3.316
4 0 0	16	4.000
3 3 1	19	4.358
4 2 0	20	4.472
4 2 2	24	4.898
5 1 1, 3 3 3	27	5.198
4 4 0	32	5.656

Substituting Equation 14 into deBroglie's relationship ($\lambda = \frac{h}{p} = \frac{h}{m v}$) gives,

$$\lambda = \frac{h}{\sqrt{2 m q V}} \quad (15)$$

If λ is expressed in Angstroms and V in volts then substituting values for the charge and mass of an electron the handy relation,

$$\lambda (\text{\AA}) = \sqrt{\frac{150}{V (\text{Volts})}} \quad (16)$$

is obtained.

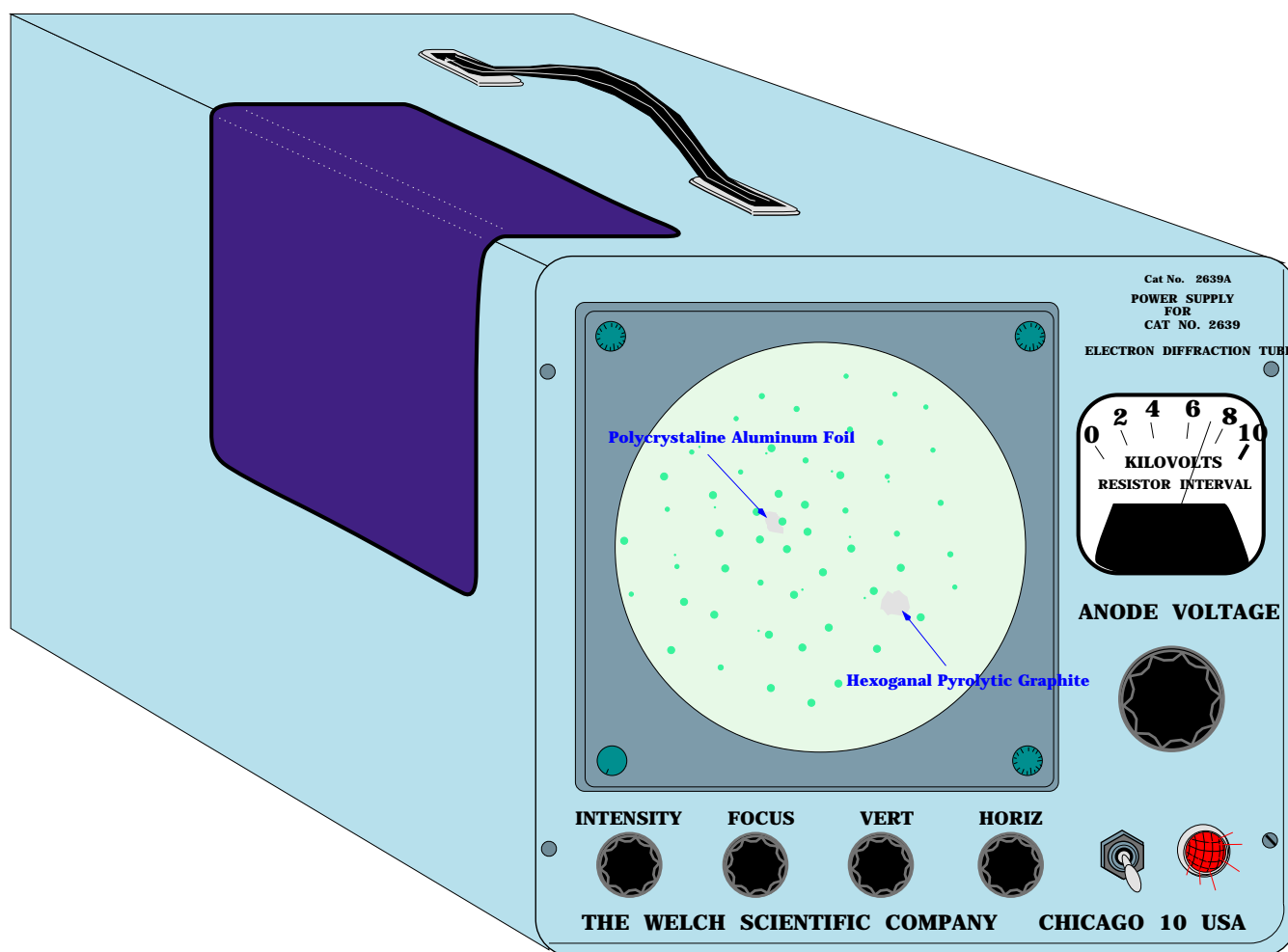


Figure 8 Electron Diffraction Tube

OPERATION OF THE ELECTRON DIFFRACTION TUBE

<<<< WARNING - TO AVOID BURNING THE PHOSPHOROUS SCREEN >>>>
<<<< TURN THE INTENSITY CONTROL UP SLOWLY >>>>
<<<< AND ALWAYS USE LOW LIGHT LEVELS EXCEPT >>>>
<<<< WHEN MOVING BEAM TO SEARCH FOR CRYSTALS >>>>

1. Before turning on the electron diffraction tube be sure that both the intensity and the anode voltage are turned to zero.
2. Turn on the power switch and allow a few minutes for the power supply and the tube filament to warm up. The anode voltage control may then be turned up and the intensity increased until the beam is seen. (Between 7 and 9 kilovolts is a good value for searching for one of the crystals.)
3. The focus control should always be adjusted to minimize the spot size on the screen whenever the voltage control is changed.
4. When taking data frequently monitor the anode voltage meter as it may drift.
5. Turn the intensity and voltage controls to zero before switching off the power to the electron diffraction tube.

EXPERIMENT I. THE ELECTRON DIFFRACTION PATTERN OF POLYCRYSTALLINE ALUMINUM

PROCEDURE

1. Once the electron diffraction tube has warmed up focus the beam and aim it at the piece of the polycrystalline aluminum foil which is slightly down and right of the center of the screen (see Figure 8.) When the beam is pointed on the foil a bright set of rings should appear.

2. For three different anode voltage levels (suggested values are 7, 8, and 9 Kilovolts) find the radii of the rings produced. A transparency with a grid of lines spaced at millimeter increments has been provided for this measurement. To obtain good data, take two or three measurements of the diameter of each ring using different orientations and average them. This will help compensate for distortions of the screen, variations in intensities, and limited accuracy of our eyes.
3. When you vary the anode voltage it will help to change it in small increments while readjusting the beam position and focus to maintain the ring pattern until the desired voltage is reached. Don't forget to monitor and adjust the anode voltage while making your measurements since it may float a bit with time.
4. The distance D between the crystal and the screen is 18.29 cm.
5. Using this data you will perform two sets of analysis:

CALCULATIONS

A. CALCULATION OF THE BEAM WAVELENGTH

For each accelerating (anode) voltage perform the following:

- i) Calculate the wavelength of the electron beam from the accelerating voltage using Equation 16.
- ii) Using the known value of the lattice constant and the measured radii of the rings, match each ring to the correct reflection plane and its corresponding Miller indices using the lattice constant of aluminum ($a = 4.0496 \pm 0.0001 \text{ \AA}$) and Equation 12. The allowed lattice spacings given in Table 1 will be necessary to identify each ring. It may be a little tricky. Keep in mind that it is possible a ring may be missing or very dim. Tabulate all values.
- iii) Once each ring has been identified, using the accepted value for the lattice constant, recalculate the wavelength of the electron beam based on the measured ring radii. Calculate and tabulate the percent error and discuss.

B. CALCULATION OF THE LATTICE CONSTANT OF ALUMINUM

- i) Determine the number of atoms associated with a unit cell of a face-centered cubic lattice. Note that most atoms are only partially within the cell. For example only one eighth of one of the corner atoms is in the cell. The other seven eighths are in the seven adjacent cells. Now using the molecular weight of aluminum, its density and Avogadro's number calculate the lattice constant a . How does this compare with the accepted value?
- ii) Rearranging Equation 13 gives the ring radius as a function of the Miller indices.

$$r = \frac{\lambda D}{a} \sqrt{h^2 + k^2 + l^2} \quad (17)$$

We can calculate the lattice constant by plotting the ring radius as a function of $\sqrt{h^2 + k^2 + l^2}$ and finding the slope ($\frac{\lambda D}{a}$). Use this method to estimate a for each set of data taken at the different accelerating voltages. To be more accurate the lattice constant may be calculated using a least-square error method.

<h2>EXPERIMENT II. THE ELECTRON DIFFRACTION PATTERN OF HEXAGONAL PYROLYTIC GRAPHITE</h2>
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PROCEDURE

1. Aim the electron beam onto the piece of graphite found slightly up and left of center to produce a hexagonal pattern of spots. You may need to play the beam around the edge and while adjusting the focus to find a thin area of the crystal which produces a sharp pattern.
2. For three different anode voltage levels (suggested values are 7, 8, and 9 Kilovolts) record the distance from the inner spot to nearest six spots. If it is easier, measure the distance between opposing spots of the pattern. For each anode voltage average these values and use the relation $\sin \theta \approx \theta \approx \frac{r}{2D}$ to obtain the first order diffraction angle at the electron beam wavelength corresponding

to that particular voltage. This gives the diffraction angle corresponding to the largest fundamental lattice plane spacing of the crystal.

3. When you vary the anode voltage it will help to change it in small increments while readjusting the beam position and focus to maintain the spot pattern until the desired voltage is reached. Don't forget to monitor and adjust the anode voltage while making your measurements since it may float a bit with time.

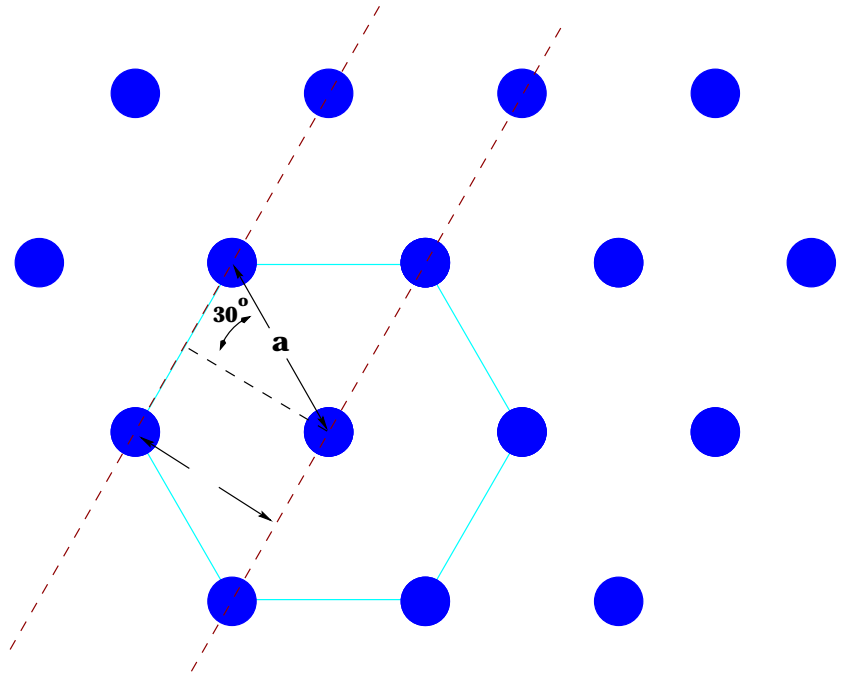


Figure 9 Lattice Plane Spacing of the Fundamental Lattice Planes with the Widest Spacing for Hexagonal Crystal Structures

4. Using the data gathered perform the following calculations.

CALCULATIONS

A. CALCULATION OF THE LATTICE CONSTANT

For each accelerating (anode) voltage perform the following,

i) The set of fundamental lattice planes with the widest separations is shown in Figure 9. For these lattice planes the spacing d is related to the lattice constant a by,

$$d = a \cos(30^\circ) \tag{18}$$

For each accelerating voltage calculate the lattice spacing using the calculated values of the diffraction angles ($\theta \approx \frac{r}{2D}$), Bragg's equation

(with $n = 1$), and the beam wavelength calculated from the accelerating potential $\left(\lambda \text{ (Å)} = \sqrt{\frac{150}{\text{Volts}}} \right)$.

- ii) Compare the calculated values for the lattice spacing with the accepted value $a = 2.4612 \pm 0.0001 \text{ Å}$.
- iii) By substituting deBroglie's relation into the Bragg Equation we can use the data from this experiment to estimate Planck's constant. For first order diffraction ($n=1$) and small θ ,

$$\lambda = \frac{h}{p} = 2 d \sin \theta \approx 2 d \frac{r}{2 D} = \frac{d r}{D}. \quad (19)$$

Substituting Equation 14 and solving for r produces,

$$r = h \frac{D}{d \sqrt{2 q m V}} \quad (20)$$

Using Equation 20, Planck's constant may be found from the slope of a line fit to a plot of r as a function of $\frac{D}{d \sqrt{2 q m V}}$. Use the linear-least-squares fit method to find the slope of line that best fits the data to find r . This may be easily performed using MatLab or with some calculators. You can check out Dr Erlenmeyer's Least-Squares Fit web page (<http://www.astro.nwu.edu/c35/PAGES/erlenmeyer.html>) and the corresponding Theory page for more details (<http://www.astro.nwu.edu/c35/PAGES/lsgerr.html>).

QUESTIONS

1. Modify Equation 15 to apply to relativistic velocities. At what voltage levels does Equation 15 become innaccurate?
2. How would the experiment change if positrons were used instead of electrons? Why would this experiment no longer work using protons?
3. What is the wavelength of a beam of alpha particle (Helium nuclei) after acceleration by a potential of 10 Kev?

4. What is an easy method of showing that the diffraction pattern is being formed by charged particles and not by light? (Hint: Remember the Planck's constant experiment.)
5. Equation 13 seems to indicate that all planes of the face-centered cubic lattice should reflect the beam with the same strength. However, it is seen that ring intensity seems to decrease with radius. Why? (Hint: A wheel with a line of lights going from the center to the outer edge is spun on its axis. Why would the outer ring seem dimmer?)
6. The wall of the electron diffraction tube, the target, and the screen are all connected to electrical ground. Can you explain why?
7. Why would the wall of the electron tube need to be covered with a thin layer of graphite?

Sections of this lab were based on:

Instructions for Catalog No. 2639 Electron Diffraction Tube, Harry F. Meiners, The Welch Scientific Company, 7300 North Under Ave. Skokie, Illinois