University of Colorado

Experiment No. 7
Physics 2150 Laboratory

Electron Diffraction from Crystals

INTRODUCTION

In 1924 a French student, Louis de Broglie proposed in his thesis that matter, as well as light, has a dual character, behaving in some circumstances like particles and in others like waves. He suggested that a particle, such as an electron, that has a momentum p, has associated with it a wavelength, λ, where:

$$\lambda = \frac{h}{p} \tag{1}$$

and h = Planck's constant. This is the same relationship between momentum and wavelength that is appropriate for light. In 1927 C. J. Davisson and L. H. Germer directly verified de Broglie's ideas by observing interference patterns formed when a beam of 54 eV electrons was reflected from a single crystal of nickel. Figure 1 illustrates the basic ideas involved. The solid horizontal lines form one set of reflecting planes.

The Bragg condition for constructive interference is given by:

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

where θ is a grazing angle, d is the spacing between Bragg planes, and n is an integer. In the Davison and Germer experiment a maximum in the reflected electron intensity was observed at the value of θ predicted by the Bragg law when the wavelength was taken from Eq. (1) with the value of the electron's momentum, p, derived from its known energy. (See below). In discussion of phenomena involving the interaction of waves (or particles) with material objects, the words "reflection", "scattering", and "diffraction" are often used interchangeably.

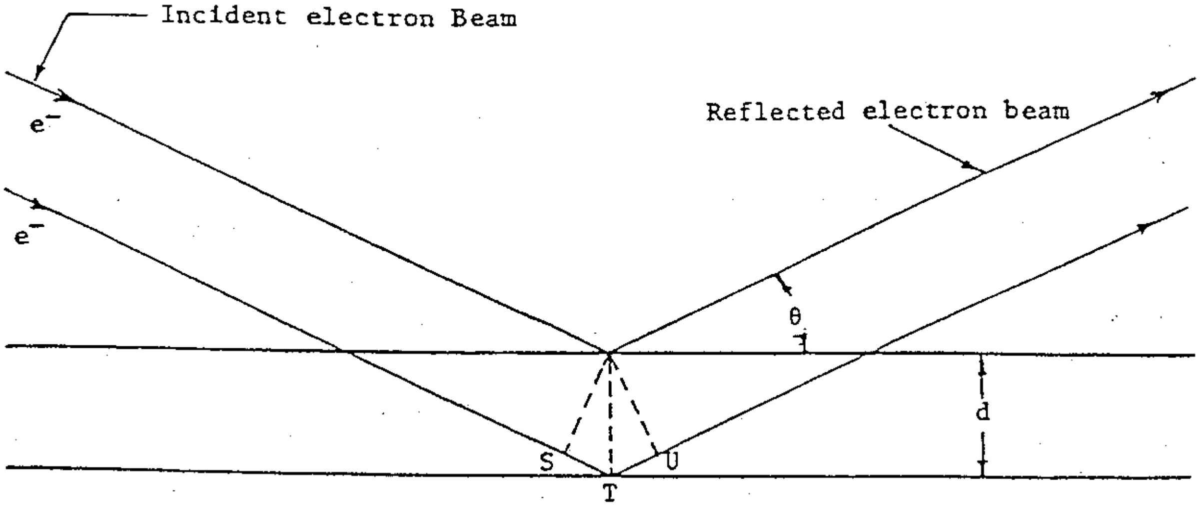

Figure 1. Reflection of a beam of electrons from planes of a crystal. The path length difference STU of reflections from two adjacent planes is 2dsinθ.

The present experiment has a somewhat different configuration to permit students to observe electron diffraction patterns more conveniently, but the concepts are the same. The object of the experiment is to verify de Broglie's basic hypothesis and to learn something about crystalline structure and diffraction patterns.

The experiment utilizes an evacuated diffraction tube that contains an electron gun which forms a small beam of electrons, an accelerating anode to provide a known energy to the electrons in the beam, crystalline targets made of either aluminum or graphite, and an observing screen for viewing the electron diffraction pattern. A schematic view of the essential features of the electron diffraction tube is shown in Figure 2.

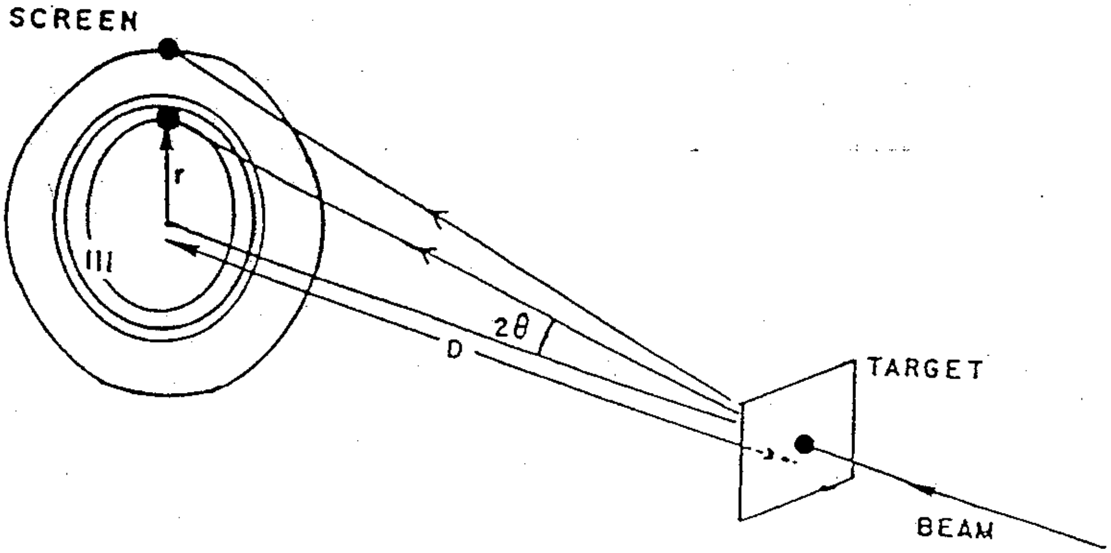

Figure 2. Spatial geometry of target and screen

The incident beam is scattered through an angle 2θ which is consistent with the angle θ defined in Figure 1. If the electrons are accelerated by a potential, V, the kinetic energy of the electrons is given by:

$$\frac{p^2}{2m} = eV \qquad (3)$$

where e is the charge on the electron.

The de Broglie wavelength is then:

$$\lambda = \frac{h}{\sqrt{2meV}} \qquad (4)$$

when the values of h, m and e are substituted into the expression, it becomes:

$$\lambda = \sqrt{\frac{150.4}{V}} \qquad (5)$$

where λ is expressed in Angstroms and V in volts. Since the electron energies used are 10 keV or less non-relativistic relationships are appropriate.

I. DIFFRACTION FROM CRYSTALS AND MILLER INDICES

When the electron beam passes through polycrystalline aluminum, ring diffraction patterns as in Figure 3 are produced by diffraction from the various families of planes within the randomly oriented crystals in the thin film target.

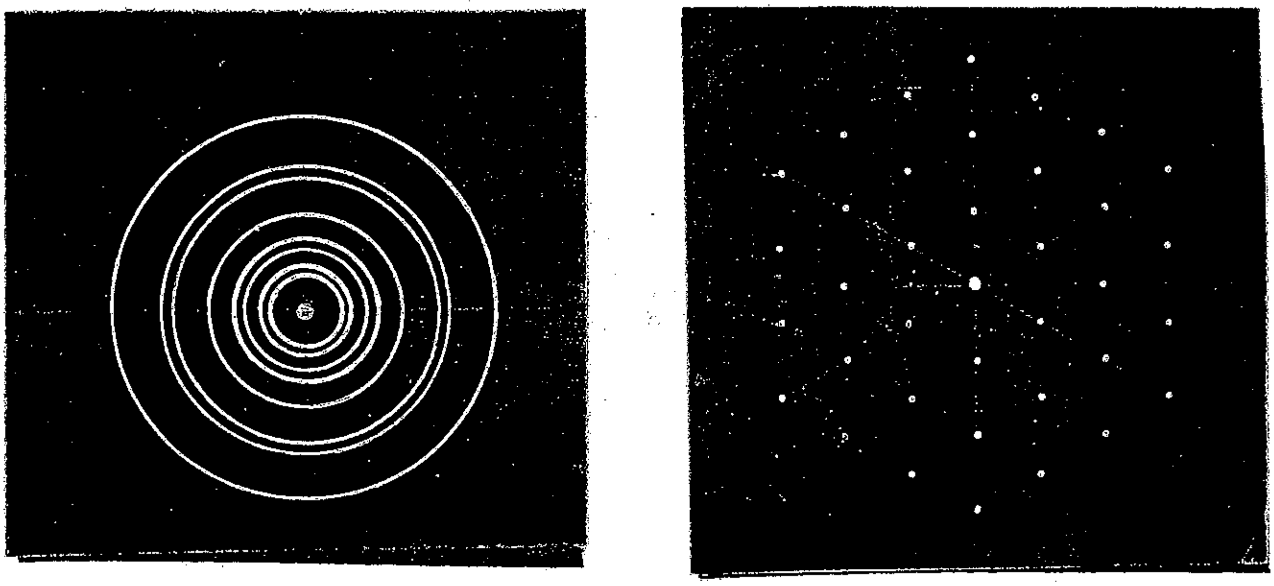
(a) (b)

Figure 3. Transmission electron diffraction patterns of (a) polycrystalline aluminum and (b) pyrolytic graphite

The separation d between parallel reflection planes for a face-centered cubic structure such as aluminum is given in terms of the integers h, k, and ℓ (Miller indices) as

$$d = \frac{na}{(h^2 + k^2 + \ell^2)^{1/2}} \tag{6}$$

where a is the length of the edge of the unit cell, diagrammed in Figure 4 and where n is the order of the reflection. The integers h, k, and ℓ can be shown to be proportional to the direction cosines of the normal to the reflecting planes.

Some understanding of the Miller indices can be obtained by observing several of the reflecting planes shown in Figure 4. A unit cell is the simplest repeated array of atoms out of which the larger crystal is made. Crystallographers designate the planes according to their intercepts on the three axes in integral numbers of the corresponding lattice constants. For example, the intercepts of the plane shown with Miller indices (111) are just 1 unit cell spacing on each of the three axes. The Miller indices are formed by taking the reciprocals of the these integers which in general will yield fractions. These fractions are then reduced to the smallest three integers having the same ratio. These three integers are the Miller indices (hkℓ). The three integers define the plane and the family of planes parallel to it and are equally spaced. For example the plane with x, y, and z intercepts (not shown) of 2, 2, and 1, respectively, and all parallel equally spaced planes are designated by the Miller indices (hkℓ) = (112).

The separation between planes with Miller indices 2, 0 and 0 shown in Figure 4 can be seen to be a/2 which is exactly what Eq. (6) would yield.

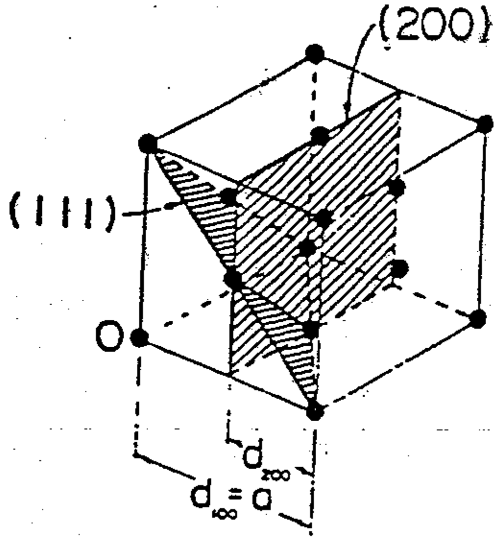

Figure 4. The face-centered cubic lattice of aluminum. Two possible reflecting planes are shown and labeled with their Miller indices ($hk\ell$). The separation between the (100) planes is called the lattice constant ($d_{100} = a$).

From the Bragg relation, Eq. (2), we obtain with use of Eq. (6)

$$\lambda = \frac{2a \sin \theta}{(h^2 + k^2 + \ell^2)^{1/2}} \tag{7}$$

When the angle θ in Eq. (7) is small, referring to Figure 2, the factor $\sin \theta$ can be replaced with

$$\theta = r/2D$$

and Eq. (7) becomes

$$\lambda = \frac{ar}{D\ (h^2 + k^2 + \ell^2)^{1/2}} \tag{8}$$

where D is the distance from target to screen and r is the radius of the rings. The lattice constant a is known from x-ray measurements. D and r are obtained by direct measurement.

The observed diffraction pattern, consisting of rings of various radii is produced by the constructive interference of the electron waves diffracted from the various families of planes within the randomly oriented crystals in the thin film target. The intensity at some position in the diffraction pattern is proportional to the square of a factor known as the structure amplitude $F(hk\ell)$. See any crystallogrophy text for a derivation of this factor.

$$\text{the Intensity } I \propto \left|F(hk\ell)\right|^2 \tag{9}$$

Eqn (9) thus gives the effect of interference of diffracted waves from atoms within the unit cell. For the face-centered cubic structure of aluminum, F(hkl) is given by:

$$F(hkl) = 1 + e^{i\pi(h+k)} + e^{i\pi(k+l)} + e^{i\pi(l+h)} \qquad (10)$$

When squared, the structure factor vanishes unless h, k, and l are all odd or all even, in which case F = 4. See Table 1 for the allowed FCC reflections for aluminum. Thus, the only rings that occur are those for which h, k, and l are all odd or all even.

Table 1. Allowed FCC reflections for aluminum

(hkl)	$(h^2+k^2+l^2)^{1/2}$
111	1.732
200	2.000
220	2.828
311	3.316
222	3.464
400	4.000
331	4.358
420	4.472
422	4.898
511, 333	5.196
440	5.656
531	5.916
442, 600	6.000
620	6.325
533	6.557
444	6.928
551	7.141

III. PROCEDURE

1. Precautions In order to observe electron diffraction patterns, accelerating voltages for the elections in the range of 6000 to 10,000 volts are used. The electron gun, crystalline targets and observing screen are enclosed in an evacuated glass tube in the arrangement shown in Figure 2. Care should be exercised to avoid contact with any of the high voltage connections in the rear of the electron diffraction tube.
2. Obtaining the Diffraction Patterns
   Before turning on the AC power, rotate the intensity and high voltage controls to their counter-clockwise (lowest) positions. After the AC power

switch is turned on, it is well to wait a few minutes for the diffraction tube filament to warm up before turning up the high voltage control. The experiment is to be carried out at voltages of about 6000, 8000 and 10,000 volts. In order to make the adjustments easier in obtaining an optimum diffraction pattern one should start with an accelerating voltage of 10,000. This corresponds to a scale reading of 100 on the voltmeter. A chart attached to the equipment provides a more accurate voltage determination for the various scale readings. With the accelerating voltage at about 10 kV turn the intensity control up until a spot or pattern is seen on the screen. As this is done the tube current should be carefully monitored by observing the microammeter. It is very important for the life of the tube and the targets not to exceed a current of 10 microamperes. The microammeter should be frequently checked during the course of the experiment to see that this upper limit is not being exceeded. Suggested current settings are 5-10 μa for 6,000 and 8,000 volts and 5 μa for 10,000 volts. Always work with the minimum current needed to give good patterns for the several voltages used.

After an electron spot has been observed on the screen, the focus control can be adjusted to minimize the spot size. There are four general positions for targets in the electron diffraction tubes being used. There will be at least one good polycrystalline aluminum target and one good hexagonal pyrolytic graphite target. The good aluminum targets are in the lower left quadrant as you face the screen. The electron beam can be moved vertically and horizontally to scan for the targets. At various positions in the lower left area a diffraction pattern similar to the one shown for aluminum in Figure 3 should be observed and at another position in the upper left part of the screen a graphite pattern should be observed. For this experiment only the aluminum diffraction pattern will be analyzed. It is well to spend some time searching and adjusting the focus control to be sure that the clearest and best defined aluminum pattern rings at each voltage are being utilized.

3. Making the Measurements

Once a good diffraction pattern has been obtained the radii of all of the observed rings should be determined. A plastic rule is available at each experiment location for these measurements. Do not use metal rules or dividers as the glass tube face can be scratched. To minimize the effects of ring distortion, make measurements across four different diameters for each ring and use the average radius in your calculations. Be sure to include the faintest rings in your measurements. As these measurements are being made the voltage should be checked periodically to see that it has not varied from its original value.

After recording all of the ring measurements at about 10 kV, repeat the procedure at 8 and 6 kV for the accelerating voltage. The position, focus and intensity controls will probably have to be changed some at these new voltages. After the measurements are completed return the voltage and intensity controls to their lowest positions.

4. Calculation of the de Broglie Wavelengths

From your knowledge of the three accelerating voltages calculate the wavelength for the electrons using equation (5) that is derived from the de Broglie equation. The actual voltages to be used in the calculations are posted on the apparatus in the laboratory.

5. Calculation of the Wavelength from the Bragg Relationship

From information presented in the introduction it is also possible to calculate the wavelength of the electrons from the observed diffraction pattern if the lattice constant of aluminum is known. The value of a for aluminum is 4.04964 A. To calculate the wavelength of the electrons, you will need to determine the average radii of diffraction rings and record the value of D, the distance from the target to the screen. Then use equation (8) to determine the Bragg wavelength, λ. The distance D is given on a label attached to the top, front surface of the plastic box containing the diffraction tube. In order to complete the calculation for λ, information on the Miller indices for the particular diffraction ring being considered is needed and this is not immediately known. It is known, in general, that the brighter inner rings are associated with the smaller values of Miller indices which are listed in Table 1. One way to determine the correct values of the Miller indices is by trial and error. Assume some sensible value of $(h^2 + k^2 + l^2)^{1/2}$ from Table I and compute a trial wavelength. If the computed wavelength does not agree reasonably well with the calculated de Broglie wavelength, the wrong Miller indices have been selected. A few trial calculations should uniquely fix the Miller indices for each of the rings. Not all of the allowed crystalline planes will produce observable rings. Sufficient information is now available to calculate the wavelength from the Bragg relationship for each of the rings at a particular voltage.

The major experimental uncertainty in the wavelengths is in the determination of the radii of the rings. Thus, for each data set (there are three, one for each voltage) determine an average wavelength and then estimate the total uncertainty for each different value of λ. Compare these values of λ to their corresponding de Broglie wavelengths to see if they agree within assigned uncertainties. Discuss briefly any possible reasons for lack of agreement.

6. Calculation of the Lattice Constant for Aluminum.

The lattice constant of aluminum should be determined in two separate ways. It can be calculated directly from the density and the molecular weight of aluminum, Avogadro's number and the number of atoms in the unit cell. The density and molecular weight of aluminum are given on page (i) of this manual. The number of atoms per unit cell ran be determined from the unit cell for a face-centered cubic lattice as shown in Figure 4. (The correct number is not 14.) Calculate the value of a for aluminum and compare its value to the standard value previously given.

The data from parts 4) and 5) can lead to a second way of determining the lattice constant of aluminum. Since the de Broglie wavelength has been determined, it can be used in the Bragg relationship, equation (8), with the measured values of r to determine a. Select two of the better determined rings at each of the three voltages to calculate six values of the lattice constant, a. Compare the average value of a and its uncertainty with the standard value and discuss any discrepancy.