

# Electron Diffraction

## 1 Introduction

It is well established that every “particle” with a rest mass  $m$  moving with momentum  $p$  can behave like a “wave” with a wavelength  $\lambda$  given by Louis de Broglie’s relation:

$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2mE}} . \quad (1)$$

In this equation,  $h$  is Planck’s constant and  $E = p^2/(2m)$  is the kinetic energy of the particle in the non-relativistic regime. The wave aspect of particles was demonstrated first by an experiment on electron diffraction performed by C. H. Davisson and L. H. Germer. For his hypothesis on the wave nature of particles de Broglie was awarded the Nobel Prize in Physics in 1929, and Davisson and Germer received the 1937 Nobel Prize for their experiment. The Davisson and Germer experiment had a beam of electrons with a well defined wavelength shorter than the spacing between atoms in a target metal crystal *reflecting* from its surface; at particular angles of reflection they detected a large increase in the reflected intensity corresponding to diffraction peaks much like one can see nowadays on reflection from a grating using a laser. An elementary description of the de Broglie hypothesis and of the Davisson and Germer experiments can be found in any introductory physics book with some modern physics chapters [1, for example]. An excellent description of a more modern electron source and reflection diffraction from a graphite crystal and the crystal with a single layer of Krypton atoms on top can be found in the short article by M. D. Chinn and S. C. Fain, Jr. [2].

Our setup is a *transmission* electron diffraction apparatus, but the physics is similar to the one of Davisson and Germer. Our apparatus consists of a sealed unit, thus it is not possible to change the target samples or do adjustments other than the ones on the control panel of the instrument. In these notes we briefly describe the apparatus, provide notes on crystallography which will help understand what is called a “spot” pattern and a “ring” pattern. The same methods and basic diffraction physics in this experiment are used in more sophisticated diffraction experiments with protons, positrons, x-rays, light, microwaves, neutrons and atoms (everything with a wavelength shorter than the distance between the “slits” of the diffraction grating), so this relatively simple experiment is a good introduction to a wide range of experimental techniques used in current research.

## 2 Electron diffraction apparatus

Typical electron diffraction apparatus consists of an electron gun, similar to the ones used in old oscilloscopes except that the voltage ( $V$ ) accelerating the electrons (charge  $e$ ) can be varied between zero and about 10 kilovolts. The accelerating voltage controls the kinetic energy of the electrons emitted from a heated filament (essentially at rest compared to their final velocity). The final energy of the electrons (or their momentum) can be found from

$$eV = \frac{p^2}{2m} = E . \quad (2)$$

Figure 1 shows a schematic of the apparatus inside the glass tube and shows the geometry of the electron beam’s path. (See Fig. 7 below for an outside view). Electrons are accelerated by

the potential difference  $V$  between the cathode and an anode that they pass through. There are also additional electrodes (not shown) used to focus the beam. The electrons then drift between the horizontal and vertical deflection plates so that they pass through metal or graphite samples mounted on a grid. The electrons eventually strike the fluorescent screen. If the sample has an aligned single crystal usually one can obtain diffraction “spots” on the screen, while if the sample is polycrystalline one sees “rings” on the screen.

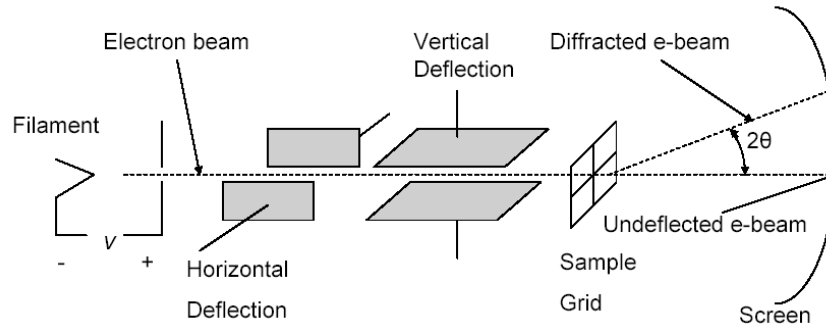


Figure 1: Schematic of electron tube. The horizontal and vertical deflection plates allow the beam to be steered so that it strikes different sample areas on the sample grid. Notice that the scattering angle is  $2\theta$ , not  $\theta$  as is used in linear diffraction gratings in optics. The sample grid is much smaller in dimension than what it appears here. See the text for explanation of Bragg diffraction.

Figure 2, found on the internet, shows a comparison between electron and x-ray diffraction from polycrystalline aluminum. Notice the similarity in the ring patterns created by either method.

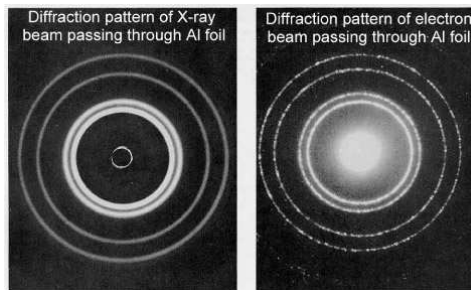


Figure 2: A similar diffraction pattern is seen from polycrystalline aluminum, regardless of whether it is made with electrons or x-rays.

### 3 Crystal structure and diffraction geometry

A three-dimensional crystalline solid is a periodic array of atoms (or molecules) in all three directions of space. For example, if we assume the usual  $x, y, z$  rectangular coordinates and there is one atom at every point of space with coordinates  $(ba, ca, da)$  with  $b, c, d$  being all possible integers and  $a$  being the shortest distance between two atoms, then the simplest array of atoms is one in which there is one atom at every corner of a cube, and the cubes stack neatly in all directions forming a three dimensional lattice, as shown in Fig. 3(a). Each cube is a representation of the *unit cell*. A unit cell is a structure that if repeated in all directions will fill space according to all possible

integers  $b, c, d$ . The smallest possible unit cell (by volume) is a *primitive cell*. The atoms in a unit cell do not have to be located at the corners of the unit cell, but if there is more than one atom in a unit cell, the coordinates of the atoms within the unit cell form what is called the *basis*.

The *simple cubic* (sc) lattice typically uses a primitive unit cell and is called a primitive (P) lattice. In total, there are 14 ways of arranging atoms in crystals, which are called the 14 Bravais lattices [3]. Then from these there is a total of 32 different crystalline structures in three dimensions, with  $a, b$ , and  $c$  equal or not equal, and with rectangular and non-rectangular coordinates.

The simple cubic lattice of atoms is not common in nature. Two common arrangements are called *face centered cubic* (fcc) and *hexagonal close packed* (hcp), which are formed when atoms pack first in planes forming a “triangular” lattice, then these triangular lattice planes stack on top of each other, with atoms in the top layer lying in the hollows formed by three touching atoms of the under layer. If we call a packed plane by A, B, and/or C, then when the planes are stacked on top of each other in the ABCABCA... sequence, the structure formed is fcc; see Fig. 3(b). In the fcc lattice the third over layer is back exactly on top of the first one (back to A). The *primitive* unit cell of the face-centered cubic (fcc) lattice is rhombohedral (not cubic!) and contains only one atom, but the larger “conventional” unit cell for fcc is indeed cubic and contains four atoms: one on a corner and one at the center of each face that is common to that corner.

If the triangular planes stack like ABABAB... , with the second over-layer stacked exactly on top of the first layer (back to A) then the structure is hcp, see Fig. 3(c). The primitive unit cell of the hcp lattice has a rhombic base and the height from A to A as shown in the figure, and it contains two atoms: one at a vertex and one in the center of the rhombohedron (labeled B in the figure).

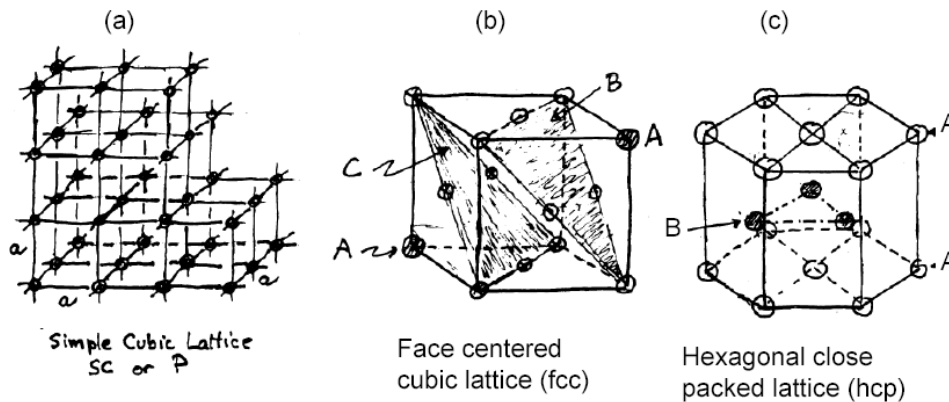


Figure 3: Simple cubic, face centered cubic and hexagonal close packed lattices. A, B, and C in (b) and (c) refer to the order of stacking planes of packed atoms.

## Bragg Diffraction

When radiation (electrons, x-rays, neutrons) strikes a substance, some goes through unchanged, some scatters into other directions and some gets absorbed. Electrons and x-rays interact with the electronic cloud of the atoms, and neutrons with the nucleus of atoms. Each type of radiation penetrates into substances in different ranges, and the probability of a unit of radiation (i.e., an electron or a photon) interacting with a material has the units of area and is called the *cross section*. Electrons in particular are very sensitive to atoms on the surface or are used to study very thin films (due to the strong Coulomb interaction). X-rays penetrate considerably more and the scattering

cross-section increases with atomic number (due to more electrons). Neutrons can go through rather thick samples since they have no charge and the nucleus of an atom is very small, but neutrons are sensitive to different isotopes and to whether the nucleus has spin or not; they are very good to study deuterium versus and hydrogen, which have different neutron scattering cross-sections but have the same atomic number. If the scattering substance is crystalline, then the ordered atoms in the crystal lattice act as a three-dimensional diffraction grating (or two-dimensional, or one-dimensional depending on how many dimensions the crystal has) provided that the wavelength of the radiation/particles is smaller than the atom-atom spacing.

To calculate properly the intensity of a refracted beam one should use each atom or molecule in the crystal as a scattering point or cloud for the particular radiation involved, and sum the scattered wave in all directions of space to find out where the diffracted intensity is strongest. In 1912, W. L. Bragg proposed that the elastic scattering of x-rays from a crystal could be easily analyzed if we consider that through any perfect crystalline structure we can always draw families of parallel planes equidistant from each other, each family containing all the atoms of the crystal. For example, if one looks at Fig. 3(b) and imagine a crystal with many units like the one pictured, the A, B, and C planes are parallel to each other, each containing the same number of atoms, separated by a distance  $d$  between adjacent planes. For every crystalline structure there is an infinite number of different families of planes with different  $d$  spacing between them. If every plane acts like a mirror, Bragg's equation for finding the angle of strongest scattering (or perfect diffraction) is

$$2d \sin \theta = n\lambda; . \tag{3}$$

The index  $n$ , an integer from 1 up to some maximum, is called the “order” of the diffraction peak. We usually analyze only the first order peaks. Figure 4 shows the angles and distances in this equation. Each family of planes has a specific set of indices, called Miller indices. The planes shown in Fig. 3(b) have indices  $h = 1, k = 1, l = 1$ , usually indicated by the notation “(111).” Miller indices are integers specifying the length of vectors in what is called *reciprocal space*, or *wavevector space*, which has dimensions of inverse distance.

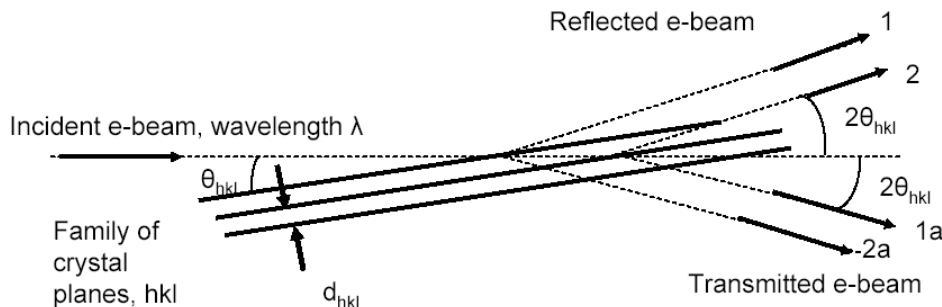


Figure 4: Bragg diffraction. When the path difference between rays 1 and 2 (or 1a and 2a) is  $\lambda$  or a multiple of  $\lambda$  there is constructive interference at that specific angle.

A full explanation of Miller indices and reciprocal space is in too long to include here; we refer the reader to the texts by Kittel [3, Chapters 1,2] and Ashcroft and Mermin [4, Chapter 5]. The principal points may be summarized:

- If the unit cell in a crystal is defined by three vectors  $\mathbf{a}_1$ ,  $\mathbf{a}_2$  and  $\mathbf{a}_3$ , then any unit cell in a

crystal can be specified by a vector  $\mathbf{T} = n_1\mathbf{a}_1 + n_2\mathbf{a}_2 + n_3\mathbf{a}_3$ , where  $n_1, n_2, n_3$  are integers. The set of vectors  $\{\mathbf{T}\}$  is the “real space lattice.”

- For the set of vectors  $\{\mathbf{T}\}$  there is another set of vectors  $\{\mathbf{G}\}$  such that  $e^{i\mathbf{G}\cdot\mathbf{T}} = 1$ . The set of vectors  $\{\mathbf{G}\}$  is called the “reciprocal space lattice.” Reciprocal space is also called “k-space,” “momentum space,” or “wavevector space.” It has the units of inverse length.
- Any  $\mathbf{G}$  that satisfies the above conditions may be written as  $\mathbf{G} = k_1\mathbf{b}_1 + k_2\mathbf{b}_2 + k_3\mathbf{b}_3$  where  $k_1, k_2, k_3$  are integers and  $\mathbf{b}_1, \mathbf{b}_2, \mathbf{b}_3$  are vectors that define the unit cell in reciprocal space.
- For any family of planes in the real space lattice separated by a distance  $d$ , the shortest reciprocal space vector has a direction that is perpendicular to these planes with a magnitude of  $2\pi/d$ . This vector may be written  $\mathbf{G} = h\mathbf{b}_1 + k\mathbf{b}_2 + l\mathbf{b}_3$ . The integers  $h, k, l$  are the Miller indices which specify this particular reciprocal space vector.

In Fig. 4 we show an incident beam of radiation of wavelength  $\lambda$ , scattered or transmitted from a family of parallel planes separated by  $d_{hkl}$ . The reflected radiation direction is shown by rays 1 and 2, and the transmitted radiation direction by rays 1a and 2a. The angle of incidence on a particular family of planes is  $\theta_{hkl}$ , measured with respect to the *lattice planes* (not the incident beam direction as is done in 1-dimensional diffraction in optics). There is a reflected beam at angle  $2\theta_{hkl}$  with respect to the incident beam direction, and also a transmitted beam at angle  $-2\theta_{hkl}$  if there is not strong attenuation through the crystal (or index of refraction change that would deviate the diffracted beam). With the construction shown in Fig. 4 it is easy to derive Bragg’s law.

**Exercise 1** *The kind of scattering assumed by Bragg’s law is elastic scattering, wherein the energy of the scattered radiation is the same as the energy of the incident radiation. This implies that the wavelength of the radiation does not change after it is scattered by the crystal. We can define a wavevector  $\mathbf{k}$  associated with a radiation beam as having a magnitude  $k = 2\pi/\lambda$  and a direction equal to the direction of the radiation. With this in mind,*

1. *Derive Bragg’s law from the geometry shown in Fig. 4. Remember that the condition for constructive interference is that the path lengths followed by the interfering waves must differ by integer multiples of the wavelength.*
2. *Show that de Broglie’s relation can be written in vector form as  $\mathbf{p} = \hbar\mathbf{k}$ .*
3. *Let the wavevector of the incident e-beam in Fig. 4 be  $\mathbf{k}_i$  and the wavevector of the reflected e-beam be  $\mathbf{k}_r$ . These wavevectors have the same magnitude. Show by geometric construction that Bragg’s law is satisfied when  $\mathbf{k}_r - \mathbf{k}_i = \mathbf{G}$  where  $\mathbf{G}$  is a vector whose magnitude is  $2\pi/d_{hkl}$  and whose direction is perpendicular to the family of planes specified by the Miller indices  $h, k, l$ . This shows that the reciprocal lattice  $\{\mathbf{G}\}$  is the set of points that satisfy Bragg’s law.*

**Exercise 2** *The diffraction condition in a lattice may be expressed as  $\mathbf{H} \cdot \mathbf{T} = \text{integer}$ , where the vector  $\mathbf{H} = \hat{\mathbf{n}}/d$ , with  $\hat{\mathbf{n}}$  as the unit normal to the set of Bragg planes and  $d$  the spacing between them. The vector  $\mathbf{T}$  is a translation vector in the lattice. For a simple cubic lattice, the translation vector is a linear combination of  $\mathbf{a}_1 = [a, 0, 0]$ ,  $\mathbf{a}_2 = [0, a, 0]$ , and  $\mathbf{a}_3 = [0, 0, a]$ , so that  $\mathbf{T} = a[n_1, n_2, n_3]$ , where  $n_i$  is an integer. Show that the vector  $\mathbf{H} = [h, k, l]/a$  with  $d = a/\sqrt{h^2 + k^2 + l^2}$  satisfies  $\mathbf{H} \cdot \mathbf{T} = \text{integer}$  in a simple cubic lattice for all integers  $h, k, l$ , with the vector  $[h, k, l]$  perpendicular to the Bragg plane. Note:  $\mathbf{G} = 2\pi\mathbf{H}$ .*

There is a very large number of parallel equidistant planes in a macroscopic crystal (thousands), thus at the angle of constructive interference there is in-phase addition of wave amplitudes and the intensity is proportional to the square of the amplitude. If the angle is not “perfect” then there is almost complete cancellation of the amplitude due to phase differences between rays and the intensity is practically zero. There is another reason why the intensity may be zero in a particular direction, even if Bragg’s equation says that the interference should be constructive: the placement of atoms in the crystal plane is important, thus there may be crystal structures in which the reflected intensity from atoms in one plane may be 180° out of phase with the one of the next parallel plane. If this occurs, then intensity in that specific direction is zero. This leads to atomic *form factors* and lattice *structure factors* and allowed and disallowed Miller indices when identifying a diffraction pattern.

### The Bragg equation using Miller indices and our electron diffraction apparatus

The samples in the four quadrants of the sample holder shown in Fig. 1 are either graphite or aluminum. Graphite crystallizes in a structure that resembles Fig. 3(c) with important differences, as noted in Fig. 5: it does not have all of the close packed atoms in each crystal plane A (or B). Instead, the carbon atoms are tied together with covalent bonds forming hexagons, so graphite is “missing” the center atom of Fig. 3(c). Also the c-axis (vertical, from one plane to the next) is much longer in graphite than for a close packed structure since the bonding between the A and B layers is due to much weaker “van der Waals” forces (fluctuating electric dipoles), rather than the strong covalent bonds that tie the atoms into hexagonal nets. The strong cohesion in the A (or B) planes and the very weak cohesion between planes A and B is what makes graphite so good as a lubricant and the reason why one can fabricate nowadays one-atomic-layer carbon films (called graphene).

For graphite, the scattering we observe is from the basal plane atoms and Bragg’s equation takes the form

$$\sin \theta_{hkl} = \sqrt{h^2 + hk + k^2} \left( \frac{\lambda}{\sqrt{3}a} \right), \quad (4)$$

where  $a$  is the magnitude of the unit cell base,  $2.46 \text{ \AA} = 0.246 \text{ nm}$ , formed by the equal length “real space” vectors  $\mathbf{a}$  and  $\mathbf{b}$ . One can complete the three-dimensional unit cell by drawing a vector  $\mathbf{c}$  perpendicular the plane of  $\mathbf{a}$  and  $\mathbf{b}$ , such that its length  $c$  is the repeat distance of the A planes. This cell is orthorhombic, with four carbon atoms per unit cell. (Can you draw the rest of the unit cell and check that there are four atoms in the cell?). All integer  $h$  and  $k$  are allowed values, with the index  $l = 0$ .

For cubic structures, Bragg’s equation can be written

$$\sin \theta_{hkl} = \sqrt{h^2 + k^2 + l^2} \left( \frac{\lambda}{2a} \right). \quad (5)$$

Here  $a$  is the side of the cube. There are specific rules that govern the allowed values of  $h, k, l$  due to the fact that the common cubic structures have a *basis* (not just an atom at a corner) and thus a *crystal structure factor* which creates phase cancellation for scattering from some planes: for sc lattices,  $h, k$ , and  $l$  can be any integer from 1 up until  $\sin \theta_{hkl}$  exceeds one (since there is no basis). For fcc lattices the  $(hkl)$  integers must be either all odd or all even. Another cubic lattice is *body centered cubic* or “bcc,” which is like simple cubic but with an additional atom located at the center of the cubic unit cell. In this lattice, all integers are possible but their sum must be even. The

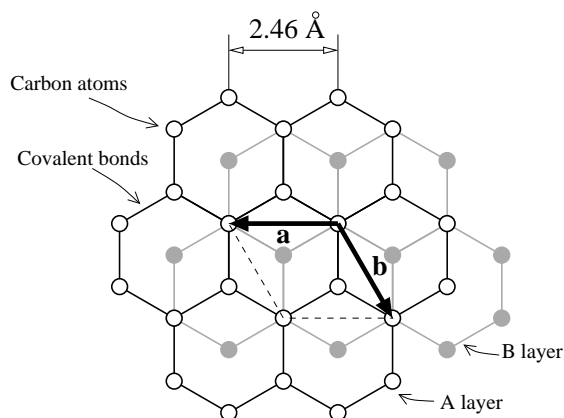


Figure 5: Top view of a graphite crystal. The crystal is formed by stacked layers of carbon. The carbon atoms are attached to other carbon atoms in the layer by covalent bonds in a hexagonal pattern. In the picture above the top (A) layer is the one with the darker lines. Notice how tight the covalent packing is, and that the atom at the center of the hexagons is missing. In the vertical direction, perpendicular to the basal plane, another layer (B) of hexagons is formed, shifted in such a way that there is a carbon atom at the center of each hexagon of the top layer. The next layer down is at the same position as the top A layer. The arrows and dotted line indicate a possible crystal “unit cell,” with  $\mathbf{a}$  and  $\mathbf{b}$  axes in the basal plane, and a  $\mathbf{c}$  axis perpendicular to the basal plane through the origin which goes from the top layer to two layers above (since the planes are stacked ABABAB...).

number zero is considered an even integer. Thus, for example, if one finds a particular scattering corresponding to indices (110) the structure is not fcc because there are two odd integers and one even integer, but such a scattering peak could come from a sc structure (all integers allowed) or a bcc structure (the sum of the integers is even). When analyzing a diffraction spectrum on the screen of the oscilloscope (or a more sophisticated recording scheme) one must use several spots or rings at different distances to the center spot to determine the crystal structure.

**Exercise 3** Equations (4) and (5) above can be derived from Bragg’s equation with a little bit of trigonometry. Try it for the simple cubic lattice: draw a cube, put  $x, y, z$  axes on it. Then, draw two parallel planes—one intersecting the three axes at distances  $a$  from the origin and the other one parallel to it either through the origin or through the cube corner opposite to the origin. Figure 3(b) shows the same planes for an fcc structure. Calculate the distance between the two planes as a function of  $a$  using trigonometry. Then compare your result to Bragg’s law in the form of Eq. (5). What Miller indices do your planes have?

**Exercise 4** For the face-centered cubic (fcc) lattice, the translation vectors  $\mathbf{T}$  are linear combinations of  $\mathbf{a}_1 = (a/2)[0, 1, 1]$ ,  $\mathbf{a}_2 = (a/2)[1, 0, 1]$ , and  $\mathbf{a}_3 = (a/2)[1, 1, 0]$ . Show that the condition  $\mathbf{H} \cdot \mathbf{T} = \text{integer}$  for all possible values of  $\mathbf{T}$  leads to the requirement that  $h, k, l$  be either all even or all odd.

## 4 *The Experiment*

In our apparatus the graphite sample is composed of a collection of mostly-oriented crystalline material with its  $c$  axis parallel to the electron beam. Since many regions are mostly oriented, with effort one can obtain superimposed sets of diffraction “spots” which clearly show the hexagonal symmetry of the carbon-net planes.

The aluminum on the other hand, which are foils thin enough for the beam of electrons to go through, shows what is called a “ring pattern.” The ring pattern is due to micrometer size crystals next to each other that are randomly aligned to form the foil. Because of the complete randomness of orientation of each tiny crystal making up the foil, there will be a few crystals aligned “right” to satisfy Bragg’s condition. The superposition of diffraction spot patterns produces the rings we observe. Figure 6 shows how “spots” from a hexagonal (graphite) pattern single crystal, would, when added to other spot patterns that are randomly oriented, generate a “ring” pattern. You can check in the figure that the “rings” you see correspond to the  $(hk)$  numbers allowed for these structure.

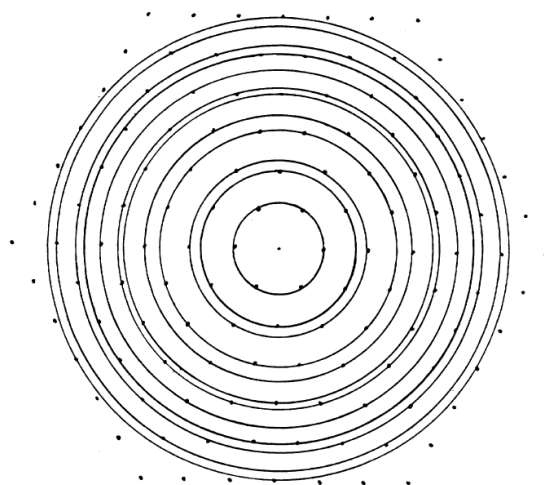


Figure 6: Spot pattern becoming a ring pattern for a two-dimensional hexagonal (e.g., graphite) crystal. Notice that if the target crystal is composed of many randomly oriented crystals, then the different spot patterns superimpose on each other and one gets a ring pattern. In this diagram, the random orientations all lie in the plane with the “ $c$ ” axis staying oriented along the beam direction.

### Getting started

It takes a little practice to find a good region of the sample screen and make a pattern that is clear enough to measure. You should explore how the apparatus works and get familiar with the controls before diving into data collection.

The first task is to “find the beam.” Here’s how:

- Make sure the power switch is off. Turn the ANODE VOLTAGE (the big knob) all the way down.



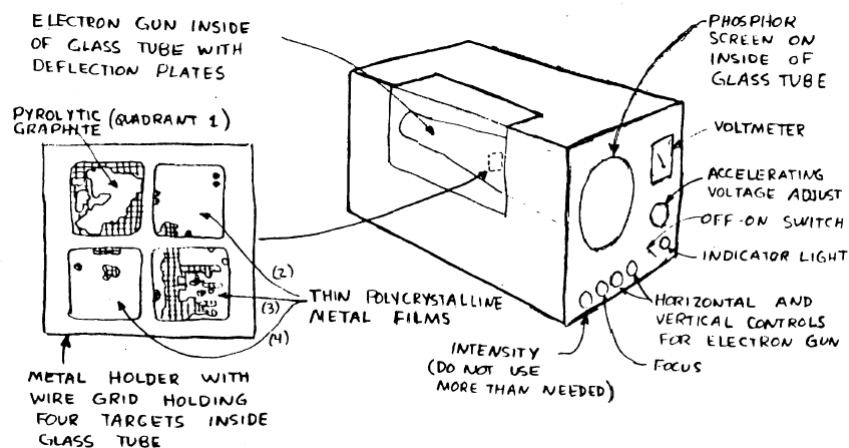


Figure 7: Drawing of electron diffraction apparatus box and the layout of the sample holder inside the tube. The drawing of the four quadrants where samples are mounted. The apparatus has been modified to allow a digital voltmeter to monitor the anode voltage to increase the precision of the voltage measurements, but use the analog meter on the front panel while setting up.

- First set all four control panel knobs, INTENSITY, FOCUS, VERT., and HORIZ. (see Fig. 7) so that the white lines on the knobs are straight up. Then set the INTENSITY knob so that it points directly to the right (the “3 O’clock” position).
- Turn the power switch on and wait about 30 seconds for it to warm up. Turn on the digital voltmeter.
- Bring up the ANODE VOLTAGE relatively slowly to between 8 and 10 kV. You should see a white spot appear and move in the upper right of the screen. If you do not see it, leave the ANODE VOLTAGE at about 8 kV and play with the VERT. and HORIZ. knobs.
- When you see the spot, use the VERT. and HORIZ. knobs to bring the spot to a visible point near the center of the screen.
- Finally, look at two things: (1) notice how the spot moves when you change the ANODE VOLTAGE. Why does this happen? (2) Notice the relationship between the voltage measured on the panel meter and the voltage measured on the digital voltmeter. What is the conversion factor between these two readings, assuming that the panel meter is only approximate?

The second task is to make a diffraction pattern. Once you have centered the spot with the ANODE VOLTAGE set to between 7 and 8 kV, turn the FOCUS knob completely counterclockwise. This will make the beam spot *very large*. Notice how you can see the shadow of a metal screen (a tiny grid pattern) that is decorated with dark blobs of stuff. Notice also that you can see the edges of each quadrant, and indeed, steer the beam to miss the sample area completely! (Of, course, you do not want to do this.) Use this unfocused spot to find regions where there is good material: places where the grid is covered by a dark shadow. The graphite is in the upper left. The other three regions have aluminum.

Now turn out the lights. You will need to work in the dark for most of your measurements.

Start with the graphite area. Position the spot so that it hits a piece of the graphite. Then slowly rotate the FOCUS knob so that the spot becomes smaller. When it gets small enough, if you are

lucky, you will see speckled rings appear, and you may be able to see spot patterns within the rings that show six-fold symmetry. If you do not see such a pattern right away, play with the beam position controls HORIZ. and VERT. and the FOCUS. Small adjustments can have a big effect!

## Taking measurements

Before taking measurements please follow these precautions:

- **Keep the INTENSITY as low as possible.** If the beam intensity is high, the electron beam may damage both the sample itself and the phosphor screen. You will notice that there are small dark patches on the screen: this is where the beam has burned the phosphor coating.
- **Turn the INTENSITY and ANODE VOLTAGE all the way down when you are not actively taking data.** As in the above, you want to avoid heating the phosphor screen and sample as much as possible.

## Graphite

In this first part, you will study the relationship between the voltage measured by the voltmeter (digital and/or analog) and the diameter of two sets of “spots” or “rings” of the graphite pattern. These sets are the  $\{10\}$  and  $\{11\}$  diffractions. (A note on notation conventions: when the Miller indices are enclosed in simple parens, like “(10),” this denotes a *specific set* of Miller indices, that is a specific set of crystal planes; when the indices are enclosed in square brackets, like “[10],” this denotes a *direction* that is perpendicular to those planes, that is, the direction of the reciprocal lattice vector associated with the Miller indices; when the indices are enclosed in curly brackets, like “ $\{10\}$ ,” this denotes a *family of planes that all have the same spacing*, that is, all of the diffraction spots that have the same distance from the origin.)

First, locate a reasonably bright pattern at about 8 kV anode voltage. Then defocus the beam so you can see what the sample looks like and where it is in the graphite quadrant. This will help you find your sample as you change the anode voltage.

Before you start to collect data, see how low you can set the anode voltage and still find a pattern. Notice how sensitive the beam position is to the accelerating voltage. You will also notice that the location of the sample as seen on the screen will vary with voltage. Typically the minimum useful voltage is around 4 kV. (Higher voltages are easier.)

Then, starting from your minimum, use a cell phone, tablet computer or web-cam to take pictures from the screen that show (as clearly as possible) the diffraction pattern along with the clear plastic ruler taped to the screen. You will analyze these photos to extract the ring diameters, i.e., spot-to-spot distances that pass through the center of the pattern. (You could also use plastic calipers to measure these distances, but image analysis is both easier and more precise.)

Record the voltage from the digital voltmeter before and after taking the picture, and be careful to record which voltage is associated with each image (it is easy to mess this up!), and note how much variation there is in the reading.

You should get images for diffraction patterns spanning the range of voltages between 4.5 kV and 8.5 kV, with at least 8 evenly spaced values.

## *Aluminum*

The object of the second set of measurements is to determine the structure of the polycrystalline aluminum foil (sc, fcc or bcc). In this case, however, we do not *a priori* know the Miller indices that correspond to each ring, but we will determine them from the characteristic spacing of the rings as compared to what theory allows.

Use the same procedure as above to locate a good sample area: Defocus the beam, locate a region where the grid is covered with the foil sample, then refocus the beam to see the diffraction pattern. Here you will see a set of unequally-spaced rings, similar to those shown in Fig. 2 (although the contrast will not be as sharp).

Find a good “ring” pattern for a given ANODE VOLTAGE, FOCUS and INTENSITY. Explore the range of accelerating voltage that allows you to see a clear pattern, but keep it below 9 kV.

As before, capture images of the ring patterns for different voltages that span the range and include the plastic ruler. You do not need as many pictures for this set, 5 is sufficient. Don’t forget to record the voltage for each image *carefully!*

## **Analysis of the images**

A good image analysis program to carry out the data collection from the images is called **ImageJ** [5]. It is freely available and runs on all typical computer operating systems (Microsoft Windows, Apple iOS and Linux) with the Java runtime libraries. Copies of ImageJ may be downloaded from the NIH website and the program is installed on the computers in the lab. Ask for assistance on how to use the program.

There are two parts you must carry out to extract the physical distances corresponding to the ring/spot diameters from each image:

1. **Set the scale.** The ruler provides the standard used to determine the diameter  $D$  of a ring or pair of spots. The natural unit of an image is the pixel separation. To “set the scale” means to determine the number of pixels per millimeter. To do this on ImageJ, first load an image (Under File>Open...). Then select the \*Straight\* tool (a line segment). Place the cursor on one marked point on the ruler, and hold the button down as you drag out a line segment to end at another marked point. You get better precision if you choose two points far apart, say 5.00 cm. With the line segment set, go to Analyze>Set Scale... and in the dialog box, enter the known distance, e.g., 5.0 cm or 50 mm and the unit of length you choose.
2. **Measure the lengths.** Under Analyze choose Measure. A “Results” box will pop up. Use the same \*Straight\* segment tool to mark out a diameter. (Hint: pay attention to the marked point at the center of the line segment: it should land on the center of the pattern for a good diameter). Take about 6 different lines across the ring or between pairs of like spots. Each measured segment will add a line to the results table by clicking Measure again, or by using Ctrl+M on the keyboard.

After you have a collection of separate measurements, you can use the Summarize tool under Results to calculate the mean and standard deviation of the columns. Only one column matters here: the Length. Record this average value and its uncertainty.

For each image you must carry out step 1, because it is typical to shift the position of the camera

as you collect the pictures, thus each image has to have its own scale set. But within each image you will have either 2 (graphite) or 5 (aluminum) diameters to collect measurements from.

### ***Analysis part 1: Graphite and de Broglie's relationship***

Make spreadsheet a table of  $D$  vs.  $V$  for each set of reflections. You should be able to convert  $D$  into  $\sin\theta_{hkl}$  given that the distance  $L$  between the graphite sample and the screen is 18.06 cm (as specified by the manufacturer). Ask yourself: am I measuring  $\theta_{hkl}$  or  $2\theta_{hkl}$ ? Am I measuring the sine or the tangent of the angle? What is the relationship between  $D$  and the angle  $2\theta_{hkl}$ ?

From Eqs. (1), (2) and (4) derive a relationship between  $V$  and  $\sin\theta$  that should give a straight line. The slope of this graph should give a relationship among fundamental constants and the graphite lattice constant  $a$  for particular values of  $h, k$ . Plot the data according to the expected relationship. Does it make a straight line? How close is the derived lattice constant to the one given in Fig. 5? Calculate possible sources of error and make a reasonable estimate of them. (Note: if you are "orders of magnitude" off, check your units! This experiment works well, with some care, to within 5% of the expected result.)

Finally, from your measurements, calculate a conversion constant between the accelerating voltage  $V$  and the wavelength of the electrons  $\lambda$ , assuming that the lattice constant is indeed 2.46 Å. You will use this constant for aluminum.

### ***Part 2: Aluminum structure determination***

In this case you want to make a spreadsheet table that gives the ring diameters  $D$  (with uncertainty) versus *ring number*. The spreadsheet should also contain the accelerating voltage. (One possibility: a table with the voltage as the column label and the ring number as the row label.)

To analyze these results, there are two things you want to find: the *type* of structure of the aluminum, which depends on the *crystal structure factor* and the *length*  $a$  of the lattice vector.

**Exercise 5** *Generate a table of the values of  $\sqrt{h^2 + k^2 + l^2}$  for cubic crystals. Your table for each type of structure should go high enough to correspond to 6 diffraction rings. For the cubic set, carry this out for simple cubic (all  $h, k, l$ ), fcc ( $h, k, l$  all even or all odd) and bcc (sum  $h + k + l$  is even). Use this table for your analysis in Part 2. (Hint: use a spreadsheet or Python program.)*

To determine the structure, first convert your  $D$  measurements into  $\sin\theta$  values, as you did earlier. Then choose one set corresponding to a particular anode voltage and plot these values versus  $\sqrt{h^2 + k^2 + l^2}$  for the allowed Miller indices for the three different types of cubic structures. You should have three data sets on your plot, one for simple cubic (which allows all combinations of integers), one for face-centered cubic (in which only indices that are all odd or all even are allowed) and one for body-centered cubic (in which the sum of the indices must be an even number). Only one of these data sets will lie on a straight line: the one corresponding to the correct structure.

For example, to test simple cubic, if you labeled your rings 1, 2, 3, ... starting from the innermost ring, and then you found  $\sin\theta_1, \sin\theta_2, \sin\theta_3, \dots$ , you would then plot these values versus  $1 = \sqrt{1^2 + 0^2 + 0^2}, \sqrt{2} = \sqrt{1^2 + 1^2 + 0^2}, \sqrt{3} = \sqrt{1^2 + 1^2 + 1^2}, \dots$ . But for the other structures, some combinations are not allowed, and the associations in your plot between your measurements and  $\sqrt{h^2 + k^2 + l^2}$  would be different.

Caution: The assignment of indices to a particular ring is not obvious. Generally they are matched in simple increasing order, but the rings vary in intensity or may be out of focus and not visible so one might be skipped. If you find that one point seems “off” the line, test whether a different choice of indices for that ring works better.

Once you have found the correct structure, fit that data to a line and extract the slope, which will be a function of  $\lambda$  and  $a$ . Repeat the analysis using the same structure for your other anode voltages. Finally, plot these slopes as a function of  $\lambda$  (use your conversion constant derived above), and extract  $a$  from a line fit, along with its uncertainty.

Compare your result with a handbook value for aluminum. (You will need to look this up.)

## ***References***

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