Electron Diffraction of Polycrystalline Materials

Introduction

Starting in 1897 with its “discovery” by J.J. Thomson (which was really the culmination of a long series of experiments by Thomson and his contemporaries on the mysterious “cathode rays”), the electron has occupied a central spot in our understanding of matter. Thomson found that the charge to mass ratio $e/m$ was nearly 2000 times larger than that for ionized hydrogen. This factor could have been due to a larger charge on the newly minted electron or a smaller mass or both. Appealing to physical intuition and simplicity, Thomson and others tended to believe the difference was due to a different mass. It was not until Millikan’s famous oil drop experiments in 1913 that the size of the charge on the electron was determined and more importantly that charge was quantized and came only in integer multiples of this basic unit.

Since the early part of the 20th century, the electron has been increasingly used as a probe of materials rather than an object of study itself. There are, for example, a number of techniques like electron energy loss spectroscopy and inverse photoemission spectroscopy that use electrons to measure various energy levels in matter. Another important class of techniques uses the wave nature of electrons to measure the structure of materials at their surfaces and in their bulk. This laboratory experiment is designed around an electron diffraction apparatus that uses a beam of electrons with a well defined wavelength to measure the interatomic spacing of bulk materials.

The wave nature of what we tend to think of as material particles was first postulated in 1923 by Louis de Broglie in his doctoral dissertation. One of the motivations for this work was the apparent asymmetry in the way physicists talked about light and matter: Light, which had been traditionally thought of as a wave since at least 1865 when J.C. Maxwell introduced his electromagnetic wave theory (much of which was confirmed experimentally by H. Hertz in the 1880s), had been shown to exhibit particle-like behaviors in its interactions with matter (particularly in the photoelectric effect and Compton effect). But matter was treated only as a collection of particles. In his dissertation, de Broglie developed his hypothesis by analogy with the case of light, noting that a photon of energy $E = hf$ also possesses momentum $p$ and they are related by

$$hf = cp$$

For an electromagnetic wave $c = f\lambda$ so that

$$\frac{hc}{\lambda} = cp.$$  

Solving for the wavelength yields

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

and de Broglie asserted that this wavelength should also be associated with particles of momentum $p$.

For an everyday particle moving at everyday speeds (say $m = 1$ kg and $v = 1$ m/s) the de Broglie wavelength is of order $\lambda \sim 10^{-34}$ m. To observe diffraction or interference effects, of course, the opening through which a wavefront passes or the difference in path length between
two rays needs to be of the order of the wavelength. Given the incredibly small de Broglie wavelength of our everyday particle (keep in mind that the diameter of a typical nucleus is of the order $10^{-15}$ m) it is no wonder that these effects are not observed. The inherent difficulty of verifying de Broglie’s hypothesis was one factor in the lukewarm reception his work initially received.

Verification eventually arrived, however, in 1927 in the form of the famous experiments carried out by C.J. Davisson and L.H. Germer who were measuring the intensity of a beam of electrons reflected from a piece of nickel. After a vacuum accident contaminated their sample with an oxide layer, they heated it in an attempt to clean the surface. As a result, the nickel surface crystallized, and when the electron beam was turned back on they noticed that the reflected beam intensity was localized in a particular direction in space, $50^\circ$ with respect to the incident beam. This direction was consistent with a model where electron waves reflecting from regularly spaced atoms on the surface give rise to constructive and destructive interference. The basic geometry and results are shown in Figure 1 below:

![Diagram](image)

The kinetic energy of the electrons in these experiments was 54 eV which, according to de Broglie’s hypothesis, gives them a wavelength of 1.67 Å. Using the known lattice spacing of nickel and the direction of the intensity peak, the path difference between electrons reflected from adjacent rows of atoms is easily calculated to be $2.15 \text{ Å } \sin 50^\circ = 1.65 \text{ Å}$, which agrees quite well with the hypothetical wavelength of the electrons.

Curiously enough, another article by G.P. Thomson (son of J.J.) and A. Reid that discusses the diffraction of electrons transmitted through thin films of material appeared in the same volume of *Nature* where Davisson and Germer published their results. Davisson and Thomson later shared the Nobel Prize for their discovery of electron diffraction which is the foundation for a number of experimental techniques widely used today. Also, make sure you appreciate the historical irony here: J.J. “proved” that cathode rays were particles while G.P. “proved” they were waves.
**Goals**

The primary scientific goal of this experiment is to measure the spacings between lattice planes of a material using a beam of high energy electrons to create a diffraction pattern. The plane spacings are related to the fundamental lattice constant of the solid, in this case aluminum. This can be accomplished using de Broglie’s hypothesis, a bit of geometry, and measurements from the diffraction pattern created by an electron beam passing through the sample. Along the way, you will also learn about vacuum technology and the operation of several pieces of research equipment.

A reflection high-energy electron diffraction (RHEED) apparatus has been installed in a vacuum chamber in the Surface Science Laboratory in Halsey 342. This RHEED gun is capable of producing a well collimated beam (spot size $\approx 100 \, \mu m$) of electrons with an energy in the range of 5–20 keV. In addition to selecting the energy you have control over the position and focus of the beam. The instructor will guide you in the safe and proper use of the RHEED gun as well as some basic vacuum procedures. A film of aluminum approximately 1000 Å thick supported by a 90% transmitting nickel mesh has been placed on the sample manipulator so that it lies in a plane perpendicular to the electron beam. A phosphor screen opposite the electron gun will show the diffraction pattern consisting of concentric circles created by the electrons after they have passed through the film. Patterns can be viewed directly (the viewport is made of leaded glass which blocks bremsstrahlung radiation) or by using the digital video camera to download images to the computer for analysis.

**Theory**

Consider the experimental geometry shown below in Figure 2. An electron beam is incident at angle $\theta$ on a material with atomic layer spacing $d$ and is partially transmitted and partially reflected. The transmitted beam is in turn partially transmitted and reflected at the next layer, and so on. According to the Bragg condition, reflected rays 1 and 2 will interfere constructively at a screen (assumed an infinite distance away) if their path difference is an

![Figure 2](image-url)
integral number of wavelengths. Note that segment $b_1b_2$ is perpendicular to both ray 1 and ray 2 and that the segments $ab_1$ and $ac_2$ have equal lengths. Therefore, the path difference between rays 1 and 2 is the sum of segments $c_2d_2$ and $d_2b_2$ which must satisfy $c_2d_2 + d_2b_2 = 2d \sin \theta = n\lambda$. Also note that the angle between the incident and diffracted rays is $2\theta$.

Now if a beam of electrons passes through a thin polycrystalline film (one where small crystalline grains are randomly oriented relative to each other) the diffracted rays will form a cone of semi-angle $2\theta$, as shown in Figure 3. If a flat phosphor screen located a distance $L$ away from the sample is used as a detector, the rays will create a circle of radius $R$ so that $R/L = \tan 2\theta$.

Using the small angle approximations $\sin \theta \approx \theta$ and $\tan 2\theta \approx 2\theta$ we see that $2d\theta = n\lambda$ and $R/L = 2\theta$. Combining these last two equations yields $Rd = n\lambda L$. Now make use of the de Broglie relation to rewrite the wavelength of the electrons in terms of the accelerating voltage $V$ of the RHEED gun:

$$\lambda = \frac{h}{p} = \frac{h}{mv} = \frac{h}{\sqrt{2mE}} = \frac{h}{\sqrt{2meV}}$$

where $E$ is the kinetic energy and $e$ is the electron charge. A final substitution for the wavelength allows us to relate the radius $R$ of the diffraction ring to the voltage $V$

$$R = \frac{nhL}{d\sqrt{2meV}}.$$ 

As you can see, this relationship can be used to find the unknown plane spacing $d$ of a material.

**Activities**

A. Align the sample in the center of the vacuum chamber and turn on the RHEED gun following the procedures outlined in the manual. You should see a diffraction pattern consisting of concentric rings around a central bright spot. Part of the challenge in analyzing these patterns is distinguishing rings that originate with planes of different spacing from higher order ($n>1$) diffraction maxima. For example, a set of planes with relatively large spacing will have a first order $n=1$ diffraction ring with a small radius $R$. Another ring of larger radius in the same pattern may be due to second order $n=2$ diffraction from those same planes or it could be the first order ring from planes with a smaller spacing.

Collect data consisting of ring radius $R$ and accelerating voltage $V$ for as many diffraction rings as possible using accelerating voltages between 15 - 20 kV. (Although the samples are only 1000 Å thick the beam transmission at lower voltages is negligible.) Note that some rings will may disappear as they move off the screen at lower voltages. Analyze the data in the context of the theory outlined above and report the value(s) of plane separation $d$ that you
obtain. The distance from the sample to the phosphor screen is \( L = 22.5 \text{ cm} \). If possible, use a fitting program that can take advantage of the uncertainties you record for ring radius. (What is the radius of a circle with a finite line width? Using the digital video equipment to record images and analyze the diffraction ring intensities as a function of position might prove beneficial for this experiment. Just remember that at some point you need to convert pixels into an actual distance. How do you do that?)

Answer the following questions:

1. Aluminum in its crystalline form exists in the face-centered cubic lattice shown in Figure 4. The lattice parameter \( a \) that defines the size of the cube is 4.05 Å. Note that you can create a number of different plane spacings depending on how you “slice” the basic cube. A theoretical x-ray diffraction intensity plot is on the following page. The Miller indices for the planes responsible for each peak are given. Note that the physical spacing for the planes can be found from

\[
\hat{d} = \frac{a}{\sqrt{k^2 + l^2 + m^2}}
\]

where \( a \) is the lattice parameter and \( k, l, \) and \( m \) are the Miller indices \((k,l,m)\) for the planes. Can you identify different lattice spacings or are the values you get consistent with one set of planes? Calculate several of these Bragg plane separations and compare them to the results of your analysis.

2. If you plot \( R \) versus \( V^{-1/2} \) in your analysis (the suggested method) do you get the expected linear relationship? If you do not, can you think of a reason why that might be?

B. The angles for the larger rings may not reasonably be called “small” for the purposes of the theory presented above. Redo the theory under the assumption that the angles are not “small”. This is not so hard to do, but you do end up with a messy expression involving an inverse tangent as the argument of a sine function. Write the final form of the equation as a direct proportion as in the original analysis shown above. In what ways, if any, does this improve the results of your analysis? What is the angle for the largest ring? Can you use the simple theory or must you resort to the more complicated analysis? Support your assertion.

References
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