Experiment 5

Electron Diffraction

Powder crystalline samples produce iconcentric-ring diffraction patterns that can be used to characterize the structure of the material. Electron diffraction, in particular, uses the wavelength that is well-suited to the analysis of the crystal structure of metals. In this experiment you will measure the de Broglie wavelength for fast moving electrons, directly demonstrating both the wave-particle duality and its practical use in the analysis of crystal structures of solid-state materials.

5.1 Introduction

In 1924, de Broglie proposed the hypothesis of wave-particle duality, associating with each particle a wave, the de Broglie wave, whose wavelength λ depended on the momentum p of the particle according to

$$\lambda = \frac{h}{p} \tag{5.1}$$

where h is the Planck's constant. The 1923-27 experiments of Davisson and Germer, in which electrons were scattered off a crystal of nickel and produced a diffraction pattern, were the first experimental confirmation of this hypothesis (the diffraction off a crystal came about quite accidentally, when a vacuum seal failed, and they heated their amorphous nickel target to get rid of the oxide film on its surface, which formed large nickel crystals). Independently, Thompson (1927) developed a scheme for firing the electrons through metal films, which is what is used in the apparatus for this experiment. Either method allows one to measure the de Broglie wavelength of fast moving electrons, and show that it is related to its momentum according to Eq. 5.1.

Scattering electrons through a metal film

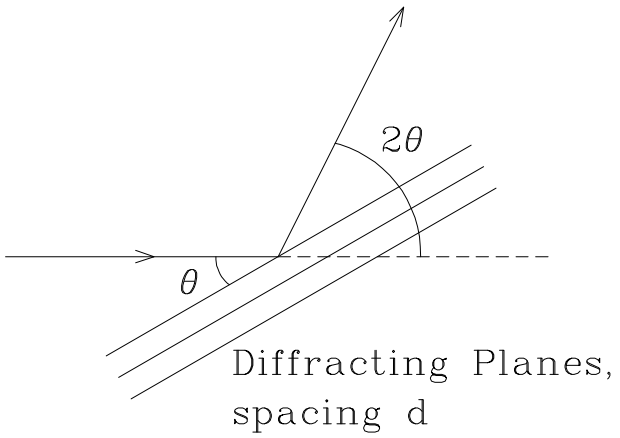

Figure 5.1: A schematic illustration of the formation of diffraction peaks in electron scattering.

If an electron leaves a cathode with zero velocity, then its kinetic energy after traversing an accelerating potential V is given by:

$$\frac{1}{2}mv^2 = eV \tag{5.2}$$

where m is the electron mass, v is it's speed after traversing the potential V, and e is it's charge. The momentum p of the electron is then given by

$$p = mv = \sqrt{2meV}$$

and its de Broglie wavelength

$$\lambda = \frac{h}{\sqrt{2meV}}. \tag{5.3}$$

If this electron strikes a crystal, its de Broglie wave will be diffracted by the 3-dimensional periodic

structure of the crystal. The electron will pass through the crystal only in those directions in which the de Broglie wave is diffracted strongly by the crystal. These directions are given by Bragg's Law:

$$2d\sin\theta = n\lambda \tag{5.4}$$

Therefore, if we know the d-values for a crystal (and we can calculate these if the crystal structure is known), then the wavelength λ can be found by measuring the directions (2θ) in which the waves are strongly diffracted, as illustrated schematically in Fig. 5.1.

Powder samples

Consider now the situation where a parallel beam of waves is incident on many crystals at once, oriented randomly with respect to direction of the beam. In such a random aggregate of crystals (*i.e.*, a powder) there are always crystals with the same set of lattice planes in an orientation satisfying Bragg's Law. All these crystals will diffract the incident beam strongly over the same angle 2θ, while other directions will result in destructive interference. The result is a diffracted beam in the shape of a cone with top angle 4θ.

This is the powder method of diffraction (Cullity, Chapter 3, p.96) and is the technique used in this experiment to measure the wavelength λ. Our beam of electrons is incident on a polycrystalline foil of aluminum. The cones of diffracted electrons strike a fluorescent screen at a distance D, forming a set of fluorescent rings of radius r. It is evident that diffraction angle 2θ is given by

$$\tan 2\theta = \frac{r}{D}. \tag{5.5}$$

The Bragg's Law

$$2d\sin\theta = n\lambda \tag{5.6}$$

may then be rewritten as

$$2\left(\frac{d}{n}\right)\sin\theta = \lambda\,. \tag{5.7}$$

In Eq. 5.4 we refer to θ as the direction of the n-th order beam diffracted from the set of atomic planes with spacing d. In the alternative formulation (Eq. 5.6) we may refer to θ as the direction of the first-order beam diffracted from a set of planes with spacing d/n. For cubic crystals the distance d between successive lattice planes in the set specified by the Miller indices $hk\ell$ is

$$d = \frac{a}{\sqrt{h^2+k^2+\ell^2}} \tag{5.8}$$

where a is the unit cell side length. Hence we may write

$$\frac{d}{n} = \frac{a}{n\sqrt{h^2+k^2+\ell^2}} = \frac{a}{\sqrt{H^2+K^2+L^2}} \tag{5.9}$$

where $H = nh$, $K = nk$, $L = n\ell$ are the Miller indices of a set of planes with spacing d/n.

In the present experiment, the diffraction angles are sufficiently small that $\tan 2\theta \approx 2\theta$ and $\sin\theta \approx \theta$. Hence, combining Eqs. 5.6–5.7, and 5.9 gives an expression for the electron beam wavelength in terms of the radii of the diffraction rings on the fluorescent screen, namely

$$\lambda = \frac{a}{\sqrt{H^2+K^2+L^2}}\,\frac{r}{D}. \tag{5.10}$$

Finally, by equating the expression for the diffraction measurement of the electron beam wavelength (Eq. 5.10) to the de Broglie expression for the electron-beam wavelength written in terms of the electron tube accelerating voltage V (Eq. 5.3) one obtains

$$r = \sqrt{H^2+K^2+L^2}\left(\frac{D}{a}\,\frac{h}{\sqrt{2me}}\right)\frac{1}{\sqrt{V}} \tag{5.11}$$

Eq. 5.11 gives an explicit expression for the radii of a set of diffraction rings as a function of accelerating tube voltage and its validity rests on that of the de Broglie postulate.

5.2 The apparatus

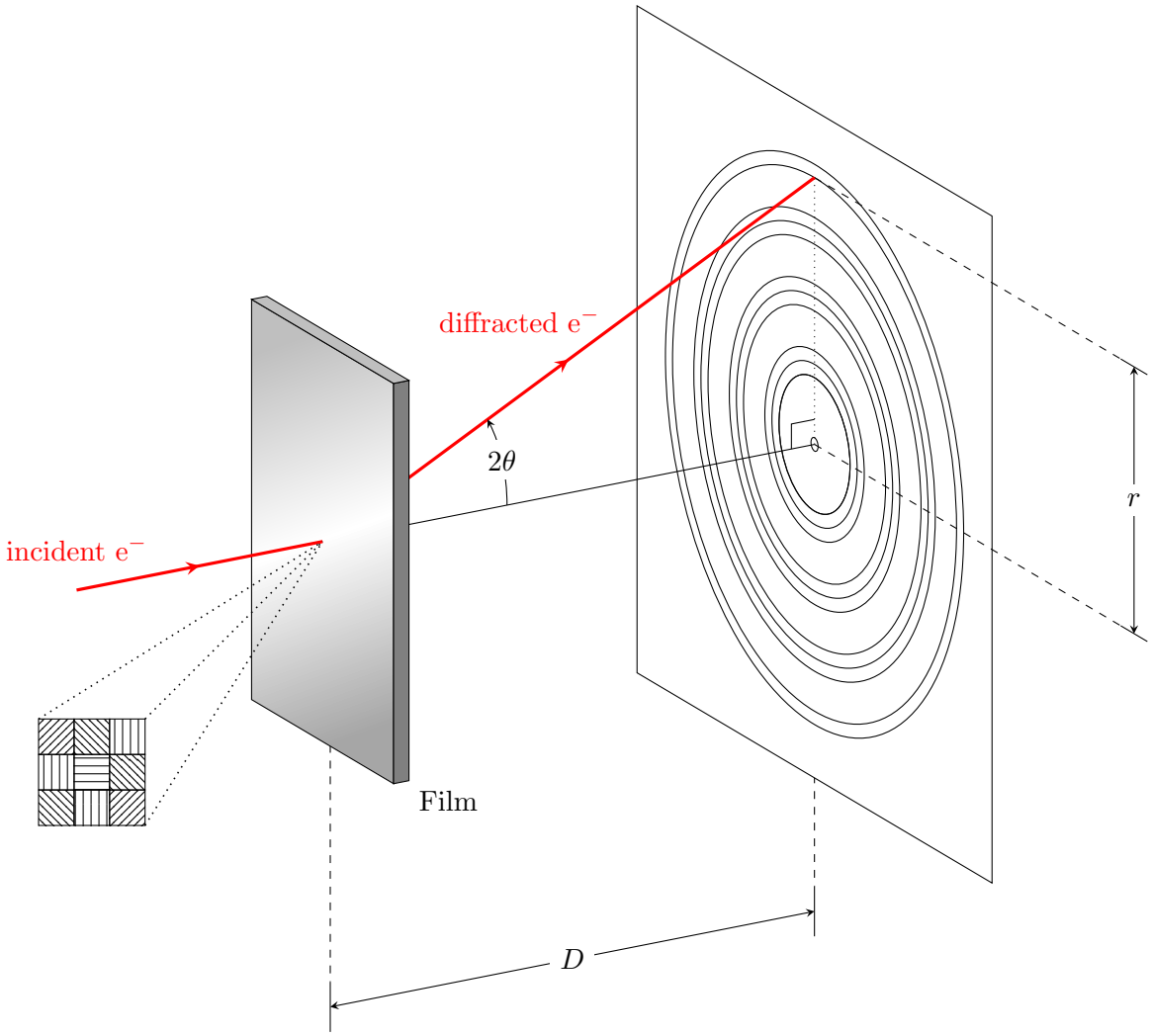

Figure 5.2: The experimental setup. Electrons are emitted from a hot cathode and accelerated through a strong electric field before striking a thin film of polycrystalline aluminium or gold. The film effectively presents itself as a collection of small crystals with their reflecting planes oriented in random directions (*i.e.* a 'powder'). Many electrons pass through the target unaffected, forming a bright, central dot on the viewing screen a distance D away. Some electrons, however, will be incident at an angle meeting the Bragg condition and diffract through the film in a cone of angle 4θ, resulting in concentric rings of radius r on the viewing screen. This drawing in tikz is by C.Wilson.

The instrument is a modified cathode-ray oscilloscope with a variable accelerating potential V. The electron beam can be deflected with horizontal and vertical deflection plates till it strikes an Al foil in the tube. The diffraction pattern of the metal foil can be measured, using of of the commonly-used techniques:

- with a ruler directly on-screen;
- taping a transparent film over the screen and using a fine marker to record several points on each visible circle for late analysis; or
- using a DSLR camera to capture the images for later analysis.

The idea of analysing the digitized data is to overlay a series of concentric circles on the image and measure their diameters as precisely as possible. In practice, at least 4–8 points per circle need to be marked and digitized by hand, or a suitable fit algorithm to the spatial distribution of the image intensity needs to be employed.

There are two instruments available in the lab, the 2639 Electron Diffraction Tube by the Welch Scientific Company (Al foil as the target), and the LEAI-62 electron diffraction apparatus by Lambda Scientific (Au foil as the target). Both share the same basic design principles, with the Welch instrument having a unique feature of having a small piece of crystalline graphite embedded in the Al foil, in a slightly off-center location. It is there to demonstrate the differences in the diffraction patterns produced by powders (circles) and crystals (discrete Laue spots, of high symmetry determined by the crystalline structure). Feel free to use one or both of the instruments, and to compare, time permitting, the results. The distances between the metal foil and the screen inside the vacuum tube are fixed, and are indicated on the outside of each apparatus. For the Welch instrument it is $D = 18.2 \pm 0.2$ cm, and for the Lambda apparatus, $D = 25.3 \pm 0.3$ cm.

5.3 Measurement checklist

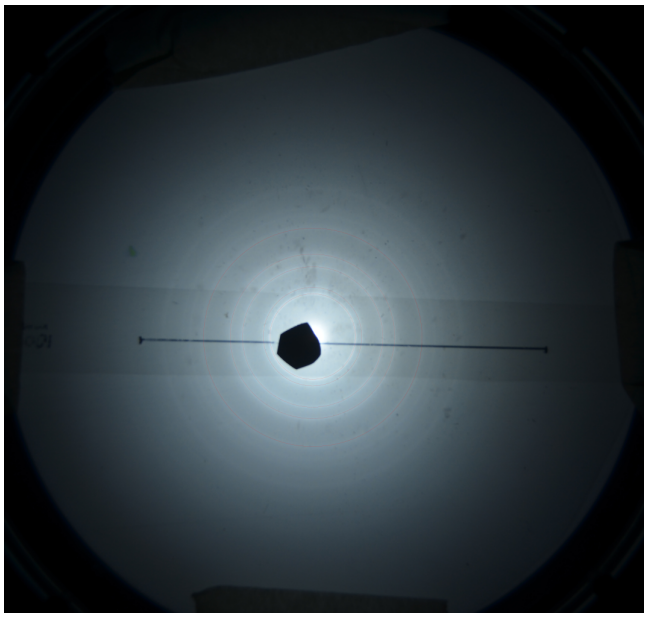

Figure 5.3: A typical diffraction pattern observed with the Welch apparatus. The black line is a scale used to convert the pixel measurements into centimeters.

To perform the experiment:

1. Review the detailed description in the manuals (see the References section below).

2. Turn the device on and slowly increase the voltage up to 9,000V. Note: turn the voltage knob and the intensity knob down to zero before powering off the apparatus; make sure they are at zero before powering it on.

3. Find a clean powder diffraction pattern (rings) by adjusting the vertical and horizontal deflection knobs. A "good" spot corresponds to a polycrystalline region of the foil; over the years, some spots were heated by overly intense electron beams and recrystallized, so they no longer act as "powder" regions.

   CAUTION: Do not exceed 5 μA beam current.

4. Set the desired intensity (4μA or so works well) and focus the beam so that the center point is as symmetric (round) as possible.

5. Place a transparent film (cut it roughly to the shape of the screen) over the diffraction pattern and fix it there using masking tape. Before taping the transparency write the voltage, intensity and your name on the transparency. A small piece of electrician's black tape placed over the bright center spot will help to see the faint outer rings. A typical pattern as observed in the Welch apparatus is shown in Fig. 5.3.

6. Make five or more diameter measurements of each ring starting at the 1st order. For each measurement draw two dots on the ring located opposite each other. The shape of the rings may be slightly distorted, and this strategy helps to account for that. You may skip over some orders if the rings

are too close together, be sure to make note of the order of the rings you do record (e.g. 1,2,4,6...) directly on the transparency.

7. Remove the transparency and measure the diameter of the rings using the calipers.

8. Optionally you may scan your transparency and use some computer-based method of drawing a circle that passes through each set of 10 (or more) dots and recording its diameter. If you are planning to use this method, drawing a precise perpendicular pair of xy-axes on the transparency before scanning it might prove useful.

9. Repeat for other values of the accelerating voltage. It is best to start from the highest end where the diffraction spots are the brightest, then lower the voltage systematically, until the diffraction spots are no longer visible. Use 5–9 kV range of accelearting voltages for the Welch apparatus, and 6–14 kV for the Lambda appratus.

10. *Optional:* On the Welch apparatus, locate the small (poly-)crystal of pyrolytic graphite (a bit above and to the left of the center of the screen) and record its diffraction pattern for at least two different voltage values. Use the data to characterize the lattice constant of pyrolytic graphite. Since the this is a poly-crystal (a cluster of a few small crystals), small adjustments to the beam position might make a big difference to the sharpness of the Laue diffraction spots.

Alternative methods of data acquisition could be considered. A DSLR (Nikon 5100) is available in the lab, and so the patterns for each chosen voltage could be recorded instantly, and the measurements analyzed using a ruler on a printout, or a computer program that would fit the observed intensity patterns to a set of concentric rings. Care should be taken to align the cameras optical axis perpendicular to the viewing screen. Set the camera to a manual exposure mode and try a variety of exposure times. Sometimes overexposing (and washing out) the brightest ring may reveal additional details on the faint ones. Automatic exposure will offer a compromise that may not reveal either very well. A typical photograph obtained using the Lambda apparatus is shown in Fig. 5.4.

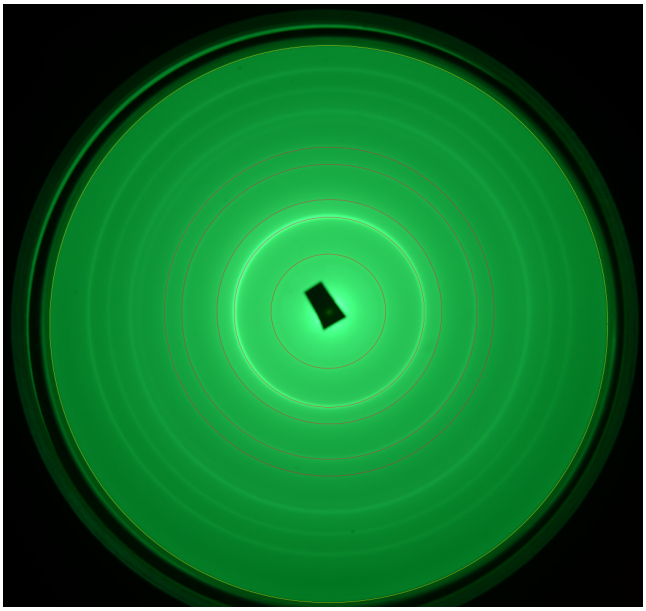

Figure 5.4: A typical diffraction pattern observed with the Lambda apparatus. The red circles have been superimposed using `inkscape` in order to measure the sizes of the diffraction rings.

To place a convenient reference scale on the image, you can pre-draw a line segment of known length (say, 10.0 cm) on the screen or measure precisely (using calipers) the size of the decorative ring on the front of the apparatus, visible as the outermost ring in Fig. 5.4.

A note of caution: avoid the use of wide-angle lenses, typical of smartphones and other hand-held devices as they may provide a significant radial non-linearity. To minimize distortions, set the zoom on the DSLR lens to 50mm equivalent (roughly the optical power of the human eye; photography lenses are often optimized for this focal distance). For DSLR this typically means around 50mm/1.5 $\approx$ 35mm.

The method of spiral powder overlays (see the References section) may prove particularly helpful, or a custom digitization and fitting program or script could be employed.

From the data calculate average values of r for each ring, at each value of V. The value of a for aluminum is $a = 4.0495$ Å, for gold $a = 4.0782$ Å (data from the Online Periodic Table). Verify Eq. 5.11.

References

Note that some of the links below require the class password for access.

- Davisson, C.; Germer, L. H. (1927). The Scattering of Electrons by a Single Crystal of Nickel. *Nature*, **119** (2998):558. `https://doi.org/10.1038/119558a0`.
- Thomson, G. P. (1927). Diffraction of Cathode Rays by a Thin Film. *Nature*, **119** (3007):890. `https://doi.org/10.1038/119890a0`.
- B.D.Cullity. Elements of X-ray Diffraction. Addison Wesley Mass. 1978 ISBN 0-201-01174-3. Available online at `https://pubs.acs.org/doi/abs/10.1021/ed034pA178`.
- N. Ashcroft and D. Mermin, Solid State Physics. Chapters 4 through 7 lay out the issues of crystallography and diffraction. See relevant excerpts:
  - From Ch.4: Crystal Lattices
  - From Ch.5: Lattice Planes and Miller indices
  - From Ch.6: Bragg and Laue Diffraction
- C. Kittel, Introduction to Solid State Physics. Chapter 1 is an introduction to crystal structure. Chapter 2 starts from diffraction and builds from there.
- Electron Diffraction Tube, instructions for catalog No.2639. The Welch Scientific Company.
- Cat.No.2639 Electron Diffraction Tube and Cat.No.2639A Power Supply, instructions for assembly. The Welch Scientific Company.
- LEAI-62 electron diffraction apparatus by Lambda Scientific (product brochure; a local copy).
- Spiral powder overlays are a novel way of analysing powder diffraction patterns, especially well-suited to electron diffraction.
- Electron Diffraction, an online book chapter.
- "Lattice Constants of the Elements." Online Periodic Table. Accessed February 22, 2021. `https://periodictable.com/Properties/A/LatticeConstants.html`.
- WebPlotDigitizer tool may prove helpful. Our Linux workstations also have quite a number of powerful graphics tools installed (`xsane` scanning tool, `engauge` image digitizer, `inkscape` drawing program, *etc.*).