ELECTRON DIFFRACTION

INTRODUCTION

The first electron diffraction experiment, performed by Davisson and Germer in 1927, provided the first direct confirmation of de Broglies wave theory of matter. Since then, electron diffraction has become an important tool for the study of both crystal structure and molecular structure, as have x-ray diffraction and neutron diffraction. There are several important differences between these techniques (see the excerpts from the book by Vainshtein), but the basic principles behind all three phenomena are the same.

Each atom in the crystal being studied scatters the incident wave in all directions, with an intensity which is determined by the structure of the individual atoms and depends on the wavelength λ of the radiation being used and the scattering angle θ. The intensity of scattered radiation is proportional to the square of a quantity known as the atomic structure factor $f(\lambda, \theta), I \sim f(\lambda, \theta)$. The scattered waves from all of the atoms in the crystal are generally out of phase, and give zero intensity when added together at an observation point outside the crystal. For certain values of the scattering angle, however, the scattered waves are in phase. The angles at which coherent scattering takes place contain information about the geometrical arrangement of the atoms in the lattice, much in the same way that the angular dependence of the intensity of scattered radiation from an atom contains information about the atomic structure. Thus it is the scattering angles of the diffracted beams which are of interest to a solid state physicist trying to determine the structure of a cresol, and the intensities of the beams which are important to a biologist trying to determine the structure of a protein molecule (it was x-ray diffraction of crystallized DNA that allowed the determination of its structure by Watson and Crick). A reference on this subject can be found at:

`http://perch.cimr.cam.ac.uk/Course/Adv_diff2`

`/Diffraction2.html#crystal_diffraction`

The simplest way to derive the condition for constructive interference from a crystal is that devised by W. Bragg. The crystal can be regarded as being composed of a set of parallel planes which contain all of the atoms in the crystal. A portion of the incident electron, x-ray, or neutron beam is specularly reflected by each of the planes. By requiring that the scattered waves from adjacent planes (and hence all planes) are in phase, i.e. that the path difference is an integral number of wavelengths, one arrives at the Bragg condition, that the total extra difference in paths between two planes must be an integer number of wavelengths.

This picture is actually an oversimplification, but it gives the correct results. There are many different sets of planes which can be drawn through the atoms of a crystal, each set with its own interplanar spacing, and each set of planes will therefore give a diffraction peak at a different scattering angle for a given electron wavelength. By determining which sets of planes are present and what their spacings are, one determines the crystal structure. For more information, see the references.

THE EXPERIMENT

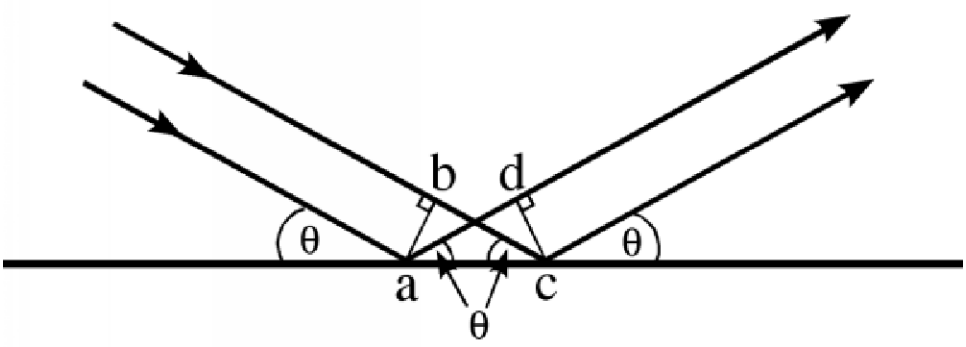

Figure 1:

In this lab, you will determine the crystal structure of gold by diffracting electrons with energies of about 30 keV. The steps involved in performing and analyzing the experiment are: (1) making the thin plastic film which serves as a substrate for the gold; (2) evaporating a thin layer of gold on top of the substrate; (3) placing the gold targets in the diffraction apparatus and observing and photographing the diffraction pattern; (4) carefully measuring the radii of the diffraction rings and estimating their relative intensities; (5) determining the crystal structure and lattice constant of gold from the radii of the rings, and comparing the measured relative intensities with calculated values; (6) cleaning up. Parts (2) and (3) involve working with various types of vacuum pumps and gauges, some of which are easily damaged by improper handling, so be sure to read the separate handout on vacuum systems.

1. Making the substrate film

The gold must be placed on a substrate which is as transparent as possible to high-energy electrons, to avoid excessive scattering. A thin film of formvar, a lightweight plastic, is what we will use. The film is made by dissolving the formvar in a solvent and then placing a drop of the solution on the surface of some distilled water. The drop spreads out and the solvent quickly evaporates, leaving a thin film of formvar on the surface of the water. This film can then be easily transferred to a target holder.

a) Weigh out 80 mg. of formvar powder, using the analytical balance. This is a delicate instrument, and your T.A. will show you how to use it properly. Place the formvar in a small glass vial. (These vials are disposablemake sure you are using a fresh one.)

b) Bring the needed chemicals and glassware to the fume hood in Room 438. You will need the bottles of acetone and ethylene dichloride, the 10 ml. graduated cylinder, the glass petri dish, and the glass vial containing the formvar. You will work in the exhaust hood to avoid inhaling the solvent vapors, especially in the case of a spill.

c) Measure out 10 ml. of ethylene dichloride in the graduated cylinder and gently pour it over the formvar. Then measure and add 1 ml. of acetone. Cover the vial and let it sit until the formvar is completely dissolved (about 45 minutes). Do not shake the vialthe softened formvar will stick to the walls and will not be dissolved. While the formvar is dissolving, rinse out the petri dish with acetone to make

sure that it is clean and free of dust, impurities, etc.

d) Bring everything back to Room 449. Get the bottle of distilled water from the refrigerator in the 4th floor Common Room. Place about a quarter of an inch of distilled water in the petri dish.

e) Take a clean target holder and place it in the petri dish. Shake loose any air bubbles that form on the surface or in the holes. Using a clean eyedropper (these are also disposable), gently put one drop of the formvar solution on the surface of the distilled water. You should be able to see it spread out over the whole surface almost instantaneously. If you drop it from too high, it may sink to the bottom instead of spreading over the surface. The solvent portion of the film evaporates in about 5 seconds. You can see the evaporation taking place by watching the colored interference fringes, which will disappear when the film is dry.

f) Lift the target holder by the handle and pull it towards the edge of the petri dish so that it breaks the surface near the edge. It will pull most of the formvar film with it. Scrape the bottom surface of the target holder against the edge of the dish to get rid of the extra film, leaving the top surface covered, and place the target holder on a paper towel to dry. Since this can take several hours, it is easiest to let the targets dry overnight.

g) Remove whatever formvar film remains on the surface of the water with the tweezers, and repeat step (f) with another target. Put a film on all of the targets available (there are about 12).

h) When you are done, you can dispose of the eyedropper. Bring the petri dish and vial to the fume hood. The vial can be left open in the fume hood to evaporate the solvent, and then thrown out. The petri dish should be cleaned with acetone and returned to Room 449.

2. Evaporating the gold film

Before doing this, read the handout on vacuum equipment and make sure that you understand the basic principles of operation of the turbo pumping station, thermocouple gauge, and ionization gauge (the latter is not used until part (3)). That handout also contains a schematic diagram of the vacuum system. NOTE THAT YOU SHOULD NOT TURN THE TURBO PUMPING SYSTEM OFF AND THEN ON WITHIN A 5 SECOND TIME FRAME. THE TURBO SYSTEM SHOULD NOT BE TURNED ON IN AN EXISTING VACUUM CONDITION, IF YOU TRY IT WILL VENT ITSELF.

a) Start by closing off the valve that connects the turbo pump system to the bell jar and then opening the vent valve. Remove the glass cover and wipe off the thin gold film on the inner surface if the previous user did not do so. Place four of your targets in the jar as shown in the photograph on the wall, with the top surface of the film facing the tungsten filament. Place the targets at varying distances from the source.

b) After checking that the O-ring on the base is free of debris, replace the glass cover on the O-ring and make sure that it is centered. Close the vent valve and open the valve which connects the turbo system to the bell jar. Turn on the turbo system, when the green light is on the turbo is up to speed (about 20,000 rpm).

c) The pressure in the bell jar should drop below 30 microns in about 5-10 minutes.

d) Take the large vacuum bottle down to the Stockroom on A-level and fill it about 2/3 full with liquid nitrogen. Ask Claudine, the Stockroom Manager, how to fill the bottle and record your withdrawal on the Physics 311/312 sign out sheet. If you ask him in French, he will be extra nice to you if your accent is Haitian.

e) Pour some liquid nitrogen into the small insulated container. BE SURE TO WEAR PROTECTIVE EYEWEAR WHEN POURING THE LIQUID NITROGEN! Place this on the platform under the bell jar, raise the platform until the cryopump tube is immersed in nitrogen, and clamp it in place. Be careful not to break the bottom of the container by pushing it up too hard. After a few minutes of cryopumping, the vacuum is good enough for evaporation to proceed.

f) Turn on the Variac starting at 0 volts and begin to smoothly increase the voltage. Look through the glass jar at the white wall behind as you do this, so that you will be able to detect the color changes caused by evaporation as soon as they occur. When the Variac is at about 40 volts, the filament will start to glow. At a slightly higher voltage than this, gold will begin to evaporate from the filament and the walls of the bell jar will appear to turn light blue-green. As soon as this color change has become clearly visible, turn the Variac quickly to zero and turn it off. Lower the platform holding the liquid nitrogen and wait for the cryopump tube to warm up (about 15 minutes). If you do not let it warm up, then a lot of water will condense on the inside when you open the bell jar, and this takes a long time to pump out.

g) Close the turbo pump off from the bell jar. Turn the turbo pump system off. Open the vent valve only a crack and let the jar vent slowlya sudden inrush of air can damage the fragile target films. Lift the cover off and remove your targets.

h) Hold your targets up to the light or a white wall and look through the center hole. The film should be fairly transparent and a light blue-green color. When light is reflected off the top surface, it should appear gold and shiny. If one of your targets appears to be of high quality, shut down the diffusion pump. Otherwise, try again with a new set of four targets.

i) Clean off all gold from the inside of the jar.

3. Making the diffraction pattern

Definition of valves used:

A: VALVE FROM DIFFUSION PUMP TO CHAMBER. This lets the diffusion pump evacuate the chamber to a high vacuum.

B: VALVE FROM BACK OF DIFFUSION PUMP TO MECHANICAL ROUGHING PUMP. This removes the gas that the diffusion pump has removed from the chamber. It can only be closed for a short period of time when the diffusion pump is hot, or the oil will spread throughout the system making a mess.

C: VALVE FROM CHAMBER TO ROUGH PUMP. This is used to bring the chamber down to a low enough pressure so that the diffusion pump can work.

The diffusion pump on the diffraction pump on the diffraction apparatus is air-cooled and is left running (hot!) all the time. The “standby” condition is: C:closed. B: open. A: open. Close off the diffusion pump to chamber valve (A) and vent the chamber. Be sure valve A is closed or you will burn up the pump

oil! Unscrew and remove the cover plate from the top-center of the apparatus and mount your target on the mounting plate, using the two pins and two outer holes to align the target and the top center hole for screwing it on. Rotate the flap so that the target is covered and replace the cover. Pump out the chamber first with the rough pump by closing valve B temporarily and opening valve C. After the pump stops gurgling and the thermocouple gauge reads less than 50 microns (this should take less than 5 minutes) close valve C, open valve B then valve A. When A is opened the thermocouple gauge reading for the chamber should quickly hit the bottom of the scale (high vacuum condition).

At this point, turn the scale multiplier of the ion gauge control and readout box to ”log” and turn on the filament. When it settles down after the initial outgassing (up to 5 minutes), set the selector knob to ”log calibrate” and adjust the calibration. Switch the selector back to the ”pressure” setting and switch the multiplier knob to successively higher multipliers until the needle is somewhere in the middle of the scale. You will need a pressure of about 2-3 X 10^{-5} Torr to get good diffraction patterns (you should know how to roughly calculate the mean free path for an electron at a given pressure), and for this you must let the diffusion pump run for awhile, depending on how clean and dry the chamber is. When the pressure reaches 4-5 X 10^{-5} Torr, fill the cold trap with liquid nitrogen. This should bring the pressure down to the desired range.

When the pressure is low enough, you are ready to expose the target to the electron beam. First, swing the Polaroid camera out of the way so that you can see the screen. It is coated with a phosphor which glows when struck by electrons, much like a TV screen. Turn the coarse and fine adjust of the high voltage power supply (on the table behind the diffraction apparatus) all the way down and switch it on. Bring the voltage up one step at a time, first with the coarse adjust and then with the fine, until it reaches 30-35 kV. Turn on the filament, and increase the current until the diffraction rings are clearly visible, but do not turn it up all the wayit can damage the screen. The rings are easier to see in the dark. Adjust the magnetic focus coil until the rings are as sharp as you can make them. Trying increasing and decreasing the voltage and see what happens to the size of the rings. Does it make sense?

Swing the camera back in front of the screen and lock it in place. If you need more film, you can get it from Andrew Dupko. Make several exposures of the diffraction pattern, increasing the exposure time by a fixed ratio for each photograph $(\theta)^2)$ is a good choice). This procedure allows a reasonably accurate determination of the relative intensities of the rings, as described by Vainshtein (200-201). In your shortest exposure, only the smallest tow or four rings should be visible, with moderate brightness and good resolution. In the longer exposures these rings will saturate the film in the center, and the fainter outer rights will be clearly visible. The filament should be turned low enough so that the shortest exposure needed lasts few seconds, making exposure times easier to control. Do not change the filament brightness once you have begun this series of exposures.

In addition to making several exposures of different lengths at one accelerating voltage, make one exposure at each of the two other electron energies, in the middle and the bottom of the useful range of the high voltage power supply. This will allow you to check the relationship between the electrons energy and wavelength. You will need to readjust the focus coil at each new energy.

When you have finished, turn off the filament, focus coil, and high voltage power supply. Isolate the chamber from the pumps, open the cover, and remove your target. Replace the cover and pump down the chamber again. You should leave the diffusion pump running to keep the chamber clean.

4. Measuring the ring diameters and intensities

With the transparent ruler provided, carefully measure the diameters of all of the visible rings, using the calibrated cross marks to establish a scale of distance. If a particular ring is too bright or dim to see well in one photo, use one of the other exposures. Estimate the relative intensities of the rings using either the exposure time method or the nine-digit visual scale, both discussed by Vainshtein, 200-201.

5. Data analysis

Using only the ratios of the measured radii of the rings, you can determine the crystal structure of gold (it is either sc, fcc, or bcc). Once you have established the crystal structure, you can make a fairly accurate determination of the lattice constant by fitting a straight line to a plot of $\sin(\theta)$ versus $(\theta)(h^2 + k^2 + l^2)$ for each ring, where (θ) is the measured Bragg angle of a ring and h, k, l are the corresponding Miller indices. This type of plot also allows you to see at a glance how well the sizes of the rings compare with the expected values. Compare this value of the lattice constant with the one which you can calculate using the density, atomic weight and crystal structure of gold, and Avogadro'ss number. Also compare it with a "book" value obtained from any solid state physics text. Calculate the lattice constant using the diffraction patterns made at other energies, to confirm that the wavelength of the electron varies with energy in the expected way. Do you need to use special relativity to calculate the wavelength?

Plot the measured values of the intensity on logarithmic graph paper (why not linear?). Calculate the expected values of the relative intensities using the scheme described by Cullity in Chapter 4. You should leave out the polarization factor, $(1+\cos^2(\theta))$ (why?), and you can use values of the atomic structure factor for electron scattering, $(\sin(\theta)/(\theta))$, interpolated from the table in Vainshtein, 404-405. Make sure that you understand the origin of the various geometrical factors in the intensity formula. Plot the calculated intensities on log paper too, and compare with the measured values.

6. Cleaning up

Take the petri dish, the solvents, and all of the targets back to the fume hood. Put some ethylene dichloride in the dish. Soak each of the targets until the formvar can be easily removed by wiping with a paper towel. When you have cleaned all of the targets, rinse out the petri dish with acetone, making sure that there are no pieces of formvar stuck to it. Return everything to Room 449.

READING AND REFERENCES

Although there are many steps to this lab, none of them is especially difficult, and all of the lab work and data analysis can be done in two or three afternoons. However, it is very important to have a solid understanding of the physical principles and the functioning of the equipment used.

1. Elements of X-Ray Diffraction, by B. D. Cullity, Chapters 2, 3, and 4. This is the principal reference and contains just about all of the basic theoretical background that you will need to understand the experiment and analyze the data. It is also a very well written book, and makes good reading.

2. Solid State Physics, by Ashcroft and Mermin, Chapters 5 and 6. This reading introduces the very important concepts of reciprocal space and the reciprocal lattice, and shows how they relate to diffraction and crystal planes.

3. Structure Analysis by Electron Diffraction, by B. K. Vainshtein, excerpts from Chapters 1 and 3. This points out the important differences between x-ray, electron, and neutron diffraction, and introduces the concept of diffraction and scattering as measuring the fourier transform of the potential or electron density of the target. It also has useful information on measuring the ring intensities.

You may also want to look up the original paper of Davisson and Germer for its historical interest, to get a feeling for how the original discovery was made. It is in Physical Review, Vol. 30, (1927) 705.

Physics 312 February 2001 Robert H. Austin