Crystal Structure and Electron Diffraction

References:

Kittel C.: “Introduction to Solid State Physics”, 8th ed. Wiley 2005
University of Michigan, PHY441-442 (Advanced Physics Laboratory Experiments, Electron Diffraction).
PHYWE Laboratory Experiments, Electron diffraction TEP 5.1.13
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Introduction

The Electron Diffraction (discovered by Davisson and Germer in 1927) is one of the most famous experiments in the history of Physics. It demonstrated the wave-particle duality, showing that fast electrons hitting a smooth nickel sample yielded a strong diffraction peak.
The hypothesis that particles should behave like waves was predicted by De Broglie in 1926. The De Broglie relation related momentum *p* and wavelength *λ*:

$$\lambda = \frac{h}{p} \qquad (1)$$

Where: $h = 6.626 \times 10^{-34}\ J \cdot s = 4.136 \times 10^{-15} eV$ is Planck’s constant.

In order to observe a wave-like behaviour of electrons, a “grating” was needed, with “slits” separation of the same order as the wavelength. In a typical lab setting (a couple of kV of accelerating potential) the De Broglie wavelength of an electron is about 1 Å (10^{-8} cm), corresponding to the inter-atomic spacing in common crystals. Crystals contain periodic structural elements serving as a diffraction grating that scatters the electrons in a predictable way. The diffraction pattern of an electron beam passing through a layer of a crystalline material contains information about the crystal structure.

Some theory

In a cathode ray tube, electrons are accelerated through a high voltage U_A, acquiring a kinetic energy: $\frac{1}{2}mv^2 = \frac{p^2}{2m} = e \cdot U_A$ (2)

The wavelength associated with electrons is obtained from (1) and (2):

$\lambda = \frac{h}{\sqrt{2m \cdot e \cdot U_A}}$ where $e = 1.602 \times 10^{-19} A \cdot s$ (the electron charge) and $m = 9.109 \times 10^{-31} kg$ is the rest mass of the electron.

Note: at the voltages used (less than 10kV), the relativistic mass can be substituted by the rest mass with an error of only 0.5%.

Diffraction of electrons on a crystal assumes that the regular arrays of atoms are reflective planes, acting as simple plane mirrors. Reflection occurs in accordance with the Bragg condition:

$$2d \sin\theta = n \cdot \lambda \qquad (3)$$

where d is the spacing between the planes of the crystal structure and θ is the Bragg angle (between the beam and the lattice planes). n is an integer (n = 1, 2, 3.....). Bragg formulated (3) by analyzing X-ray diffraction on crystals.

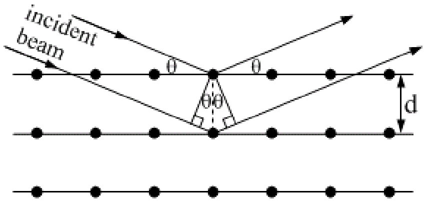

Figure 1: Bragg's model of crystal diffraction as multiple reflections on lattice planes. θ is the Bragg angle θ_{Bragg}.

Davison and Germer's electron diffraction data were completely accidental. They observed a strong peak in electron scattering from nickel. Using Bragg's condition for wavelength and the known lattice constant of nickel, they verified the De Broglie hypothesis.

The experiment will be done with a graphite (carbon) crystal that has a hexagonal structure.

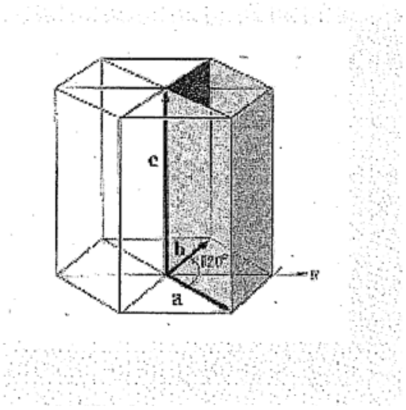

Figure 2 Graphite unit cell

In polycrystalline graphite, the bond between the individual layers is broken, so their orientation is random. A poly-crystal (powder) is a conglomerate of a large number of small crystal domains. An electron beam is spread out in form of a cone and produces interference rings on a fluorescent screen. In Figure 3, the crystalline planes (100) and (110) which give rise to the inner and outer rings in the electron diffraction tube are presented. These spacings have been defined in terms of the unit vectors a and b where a = b in the hexagonal structure. The indices (100), (110), etc. are known as Miller indices:

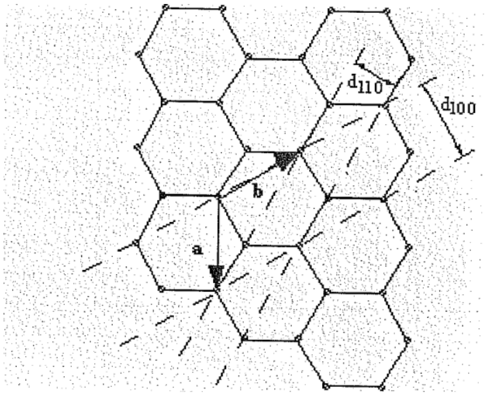

Figure 3 Two dimensional lattice spacings for graphite

The ratio of the d-spacings in a hexagonal structure is $d_{100}/d_{110} = \frac{\sqrt{3}}{1}$.

The Method

The electron beam emitted by a hot filament hits a polycrystalline graphite sample located in a vacuumed tube. The beam is spread out and forms **interference patterns**. Maxima will be arranged in the form of circles on the fluorescent screen, corresponding to cones with apex on the graphite sample (see Figures 4 and 5).

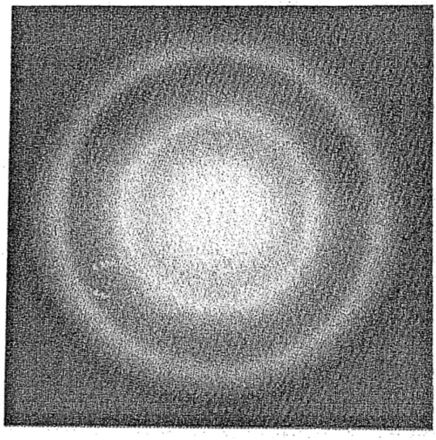

Fig. 4 Electron diffraction experimental result.

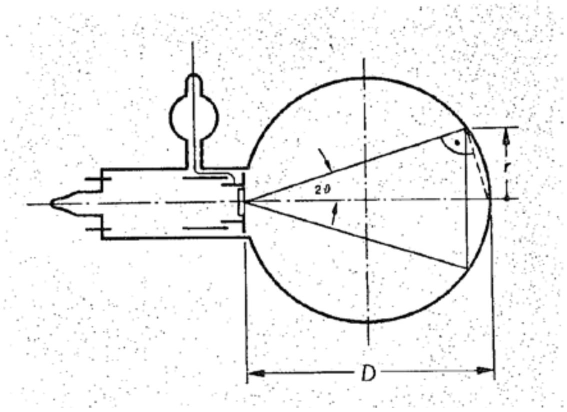

Fig. 5 The electron diffraction angle.

The diffraction cones have half-angles given by:

$$2\theta_{Bragg} \cong \tan^{-1}\left(\frac{r}{2R}\right) \qquad (4)$$

where r is radius of the 2-dimensional diffraction pattern (circle) and D = 2R is diameter of the tube.

Assuming small diffraction angles and negligible error due to the tube curvature, $\sin\theta \simeq \tan\theta$ and $\sin 2\theta \simeq 2\sin\theta$

Combining (3) and (4), we obtain:

$$r = \frac{2R}{d} n\lambda = \frac{2R}{d} n \frac{h}{\sqrt{2meU_a}} \qquad (5)$$

Equation (5) shows clearly that the observed diffraction/interference radii r depend on the anode voltage U_a.

The experiment

The equipment will be wired for you. Setup will consist of:

- Electron diffraction tube, radius R = 65 mm
- Power supply unit (6.3V AC, 0-20V DC, 0-300V DC, 0-380 V DC)
- High voltage supply unit, 0 – 10 kV
- Plastic vernier caliper.

Adjust the bias voltage G1 at ~20V. Do not change voltages at grids G4 and G3 as they are pre-set at the right values.

Turning on: turn on the voltmeter and the Leybold power supply unit. Wait for 30 seconds (this allows the electron gun to warm up), then turn on the high voltage and wait another 1 minute.

Turning off: slowly turn down the high voltage to zero and then turn off the Leybold power supply unit and the voltmeter.

Read the anode voltage at the display of the HV power supply. To determine the diameter of the diffraction rings, measure the inner and the outer edge of the rings with the plastic vernier caliper and take the average.

You may also measure any further interference rings which you can recognise. Dimming the room lights would help.

Note: the bright spot in the middle of the screen may damage the fluorescent layer of the tube. Reduce the light intensity after each reading as soon as possible.

- Measure the first two radii of the first order of diffraction (n = 1) at different anode high voltages between 2.5 kV and 6.0 kV and plot r as a function of λ (use Python to plot). Using (5), calculate the lattice constants d_{100} and d_{110} of graphite.
  Note: DO NOT exceed 6.2 kV! It may damage the tube!
- Estimate and discuss the possible errors of the measurement. Some errors are: using a non-relativistic formula for electron momentum; using approximate relations for small angles, neglecting the curvature of the tube face, etc.

Questions

- Have you observed the wave nature of the electron? Give one example of the electron showing its particle-like nature in this experiment. Discuss your results.
- Graphite consists of layers of carbon atoms with an average separation between layers of 3.4 Å. Assume that each layer has the hexagonal structure shown in Figure 6:

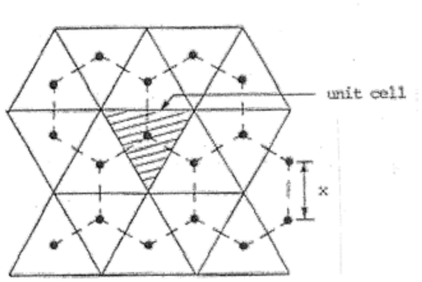

Figure 6. Hexagonal structure of graphite

Considering the unit cell being an equilateral triangle, calculate:

a) The nearest neighbor distance x between atoms within each layer.

b) The average spacing between carbon atoms. Compare the average spacing with the two measured lattice constants and also with the nearest neighbor distance.

Useful constants: Avogadro number: $N_A = 6.02 \times 10^{23} atoms/mol$, Atomic weight of carbon: $W = 12 g/mol$; Density of graphite: $\rho_C = 2.25 g/cm^3$

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Appendix: Miller indices

Understanding the physical correspondence between an electron diffraction pattern and the crystal structure requires a formal knowledge of the crystal lattice.

The orientation of a surface or a crystal plane may be defined by considering how the plane intersects the main crystallographic axes of the solid.

Miller indices (*hkl*) are a set of numbers which quantify the intercepts of the crystal planes with the axes, and thus may be used to uniquely identify the plane or surface. They express lattice planes and directions.

The following procedure to assign the Miller indices is simplified (it is a "recipe") and only applies to a **cubic crystal system** (one having a cubic unit cell with dimensions ***a*** x ***a*** x ***a***).

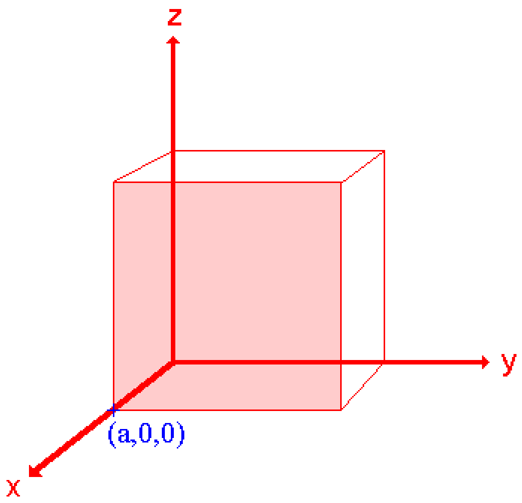
The rules are:

- Identify the intercepts of the plane with the x, y and z axes
- Specify the intercepts in fractional coordinates, dividing by the cell dimension in that direction
- Take the reciprocal of the fractional intercepts.

Consider the following plane surface in a cubic crystal of side **a**:

The intercepts are: a, ∞, ∞.

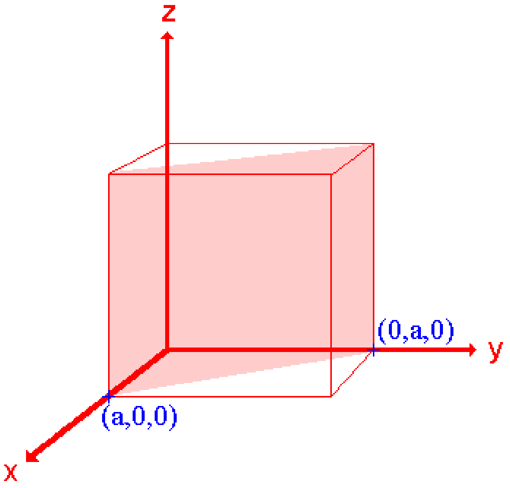
Fractional coordinate intercepts are: a/a, ∞/a, ∞/a or: 1, ∞, ∞.



Reciprocal of fractional coordinates: 1, 0, 0

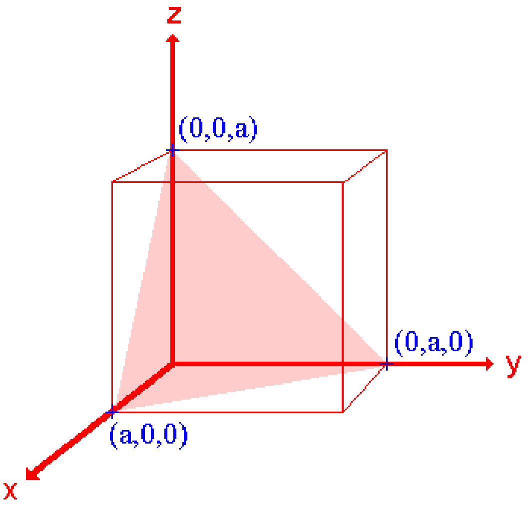
Miller indices for the surface: (100).

The (110) surface:



Intercepts: a, a, ∞
Fractional intercepts: 1, 1, ∞
Miller indices: (110).

The (111) surface:



Intercepts: a, a, a.
Fractional intercepts: 1, 1, 1
Miller indices: (111).

For a cubic crystal of side a, the distance between a generic plane (khl) and a parallel plane that passes through the origin is given by:

$$d = \frac{a}{\sqrt{k^2 + h^2 + l^2}}$$

(hkl) denotes a crystallographic plane. {khl} denotes a family of crystallographic planes ((khl), (lkh), (hlk), etc.). In the cubic system, planes having the same indices regardless of order or sign are equivalent. Negative values are expressed with a bar over the number (Example: -2 is expressed as $\bar{2}$).

In the cubic system, a plane and a direction with the same indices are orthogonal.