

ELECTRON DIFFRACTION

REFERENCE

Instruction Manual: *Electron Diffraction Tube - Welch Scientific Co. Cat. No. 2639* - (available at the Resource Centre).

INTRODUCTION



This experiment is a demonstration of the wave nature of the electron, and provides a confirmation of the de Broglie relationship:

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

where λ = electron wavelength, h = Planck's constant, p = electron momentum. It also provides an introduction to the use of diffraction in the analysis of crystals.

This guide sheet outlines a method for the analysis of cubic crystal forms, this being useful to you for interpreting the transmission diffraction pattern produced by scattering electrons off a thin film target of polycrystalline aluminium. The apparatus also contains samples with hexagonal structures. These are pyrolytic graphite targets, and are available both as single crystals and in polycrystalline form.

For the methodology of analysis of the hexagonal crystal, and for additional material on cubic crystals, see the reference.

THE DE BROGLIE WAVELENGTH

The voltmeter measures the accelerating potential of the electrons in the tube. Thus:

$$\frac{1}{2} m v_f^2 = eV \quad \text{or} \quad p = m v_f = \sqrt{2meV} \quad (2)$$

where v_f is the final velocity of the electrons after being accelerated through a potential V . The above assumes the non-relativistic approximation. To what degree is this justified?

Substituting in the de Broglie relationship, equation(1);

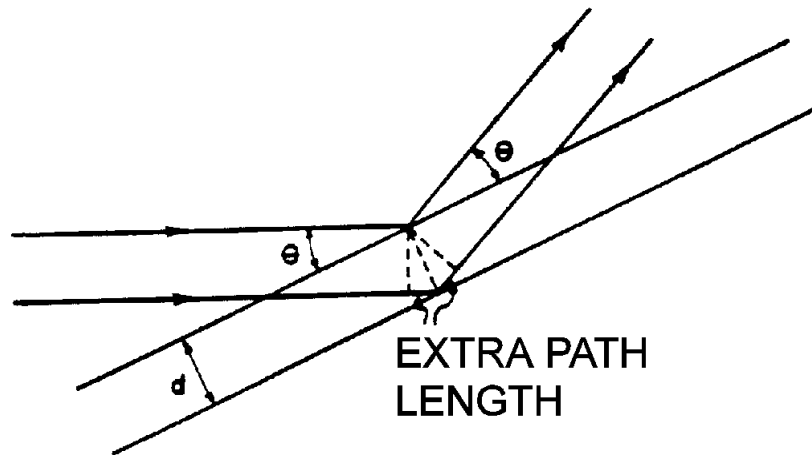
$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2meV}} = \sqrt{\frac{h^2/2me}{V}} \quad (3)$$

When the values of h , m , and e are substituted:

$$\lambda \text{ (nm)} = \sqrt{\frac{1.505}{V(\text{Volts})}} \quad (4)$$

BRAGG'S LAW

The case of waves (electromagnetic waves such as x-rays or "matter" waves such as electrons) scattering off a crystal lattice is similar to light being scattered by a diffraction grating. However, the three-dimensional case of the crystal is geometrically more complex than the two- (or one-) dimensional diffraction grating case. Bragg's Law governs the position of the diffracted maxima in the case of the crystal. A wave diffracted by a crystal behaves as if it were reflected off the planes of the crystal. Moreover there is an outgoing diffracted wave only if the path length difference between rays "reflected" off adjacent planes are an integral number of wavelengths. Thus considering a beam scattering off two parallel planes of atoms as shown in figure.



A beam incident on a pair of planes separated by a distance d . (For reinforcement of the scattering from atoms in **one** plane the usual condition for reflection applies, - angle of reflection equal angle of incidence, as indicated.)

The extra path length of the lower ray may be shown to be $2d \sin \theta$ so that maxima in the diffraction pattern will occur when:

$$2d \sin \theta = n\lambda, \quad n = 0,1,2,\dots \quad (5)$$

ELECTRON DIFFRACTION

This is Bragg's Law. Furthermore, the beam is deflected a total angle 2θ . Thus, for our electron diffraction tube, with maxima registered as spots or rings on the face of the tube, the distance of the spot from the incoming beam axis = R , so

$$R = D \tan(\text{deflection}) = D \tan 2\theta \sim D \cdot 2\theta \quad (6)$$

where D = distance from target to screen. Combining equations (5) and (6), and taking $\sin\theta \approx \theta$, then:

$$R = \frac{n\lambda D}{d} \quad (7)$$

(Note that for the polycrystalline samples mentioned below, r is the radius of the ring.)

Note then, that the obtaining of a diffraction maximum requires that two conditions be met. Not only must the angle of deflection bear an appropriate relationship to d and λ , but also the crystal orientation must be correct to provide an apparent "reflection" off the crystal planes. The way the crystals are oriented relative to the incoming beam will thus determine the appearance of the diffraction pattern,

ELECTRON DIFFRACTION PATTERNS

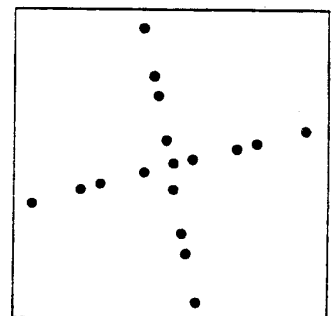
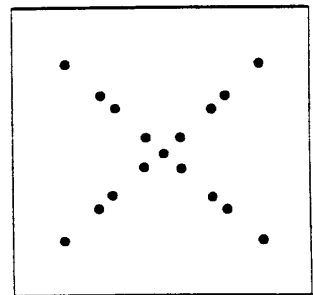
In relation to diffraction patterns it is interesting to consider three types of solid matter: single crystals, polycrystals and amorphous materials.

SINGLE CRYSTALS

Single crystals consist of atoms arranged in an orderly lattice. Some types of crystal lattices are simple cubic, face centre cubic (f.c.c.), and body centre cubic (b.c.c). In general, single crystals with different crystal structures will cleave into their own characteristic geometry. You may have seen single crystals of quartz, calcite, or carbon (diamond).

Single crystals are the most ordered of the three structures. An electron beam passing through a single crystal will produce a pattern of spots. From the diffraction spots one can determine the type of crystal structure (f.c.c., b.c.c.) and the "lattice parameter" (*i.e.*, the distance between adjacent (100) planes).

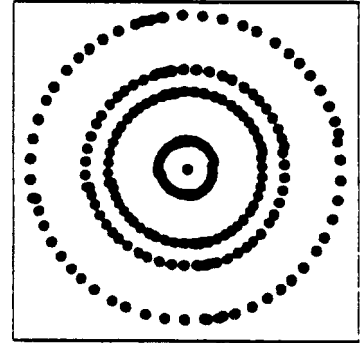
Also, the orientation of the single crystal can be determined: if the single crystal is turned or flipped, the spot diffraction pattern will rotate around the centre beam spot in a predictable way.



POLYCRYSTALLINE MATERIALS

Polycrystalline materials are made up of many tiny single crystals. Most common metal materials (copper pipes, nickel coins, stainless steel forks) are polycrystalline. Also, a ground-up powder sample appears polycrystalline. Any small single crystal "grain" will not in general have the same orientation as its neighbours. The single crystal grains in a polycrystal will have a random distribution of all the possible orientations.

A polycrystal, therefore, is not as ordered as a single crystal. An electron beam passing through a polycrystal will produce a diffraction pattern equivalent to that produced by a beam passing through series of single crystals of various orientations. The diffraction pattern will therefore look like a superposition of single crystal spot patterns: a series of concentric rings resulting from many spots very close together at various rotations around the centre beam spot. From the diffraction rings one can also determine the type of crystal structure and the "lattice parameter". One cannot determine the orientation of a polycrystal, since there is no single orientation and flipping or turning the polycrystal will yield the same ring pattern.

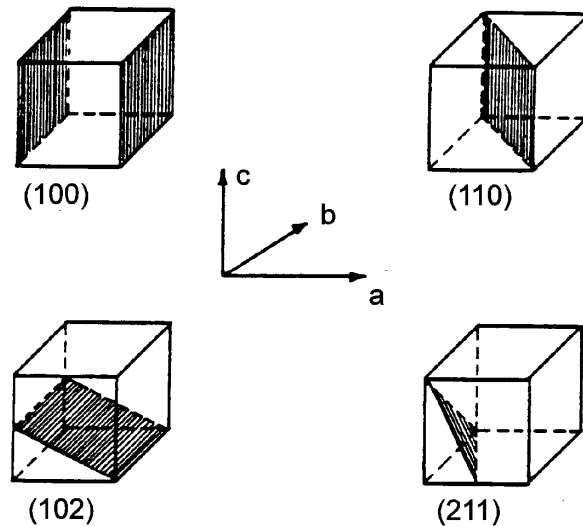


AMORPHOUS MATERIALS

Amorphous materials do not consist of atoms arranged in ordered lattices, but in hodgepodge random sites. Amorphous materials are completely disordered. The electron diffraction pattern will consist of fuzzy rings of light on the fluorescent screen. The diameters of these rings of light are related to average nearest neighbour distances in the material.

THE MILLER INDICES FOR CUBIC CRYSTALS

The Miller indices characterize various planes through a crystal lattice. First choose crystallographic axes, \vec{a} , \vec{b} and \vec{c} with the origin at one atom. The Miller indices are defined to be the reciprocals of the fractional intercept of the plane with the three axes, as shown in the figure. If the plane is parallel to a given axis, the index is = 0, corresponding to an intercept of infinity.



Miller indices of some lattice planes

For a cubic crystal, the distance of the specified plane from the parallel plane which passes through the origin is:

$$d = \frac{a}{\sqrt{H^2 + K^2 + L^2}} \quad (8)$$

where H, K, L are the Miller indices and $a =$ length of a side of the cube. The proof of this is a simple matter of solid geometry.

The lower the Miller indices, the greater the separation between adjacent parallel planes. In addition, the lower indexed planes have a greater density of lattice points.

The aluminium target is a powder of small crystal flakes, *i.e.*, it is polycrystalline. Thus the diffraction pattern appears as rings. There are also many possible planes that may be drawn through the crystal lattice, and in a powder sample in general all of these will be present. A simple example of this is shown in Figure 1.

A more useful form (as shown below) may be found by multiplying numerator and denominator by the order of interference, n :

$$d = \frac{na}{\sqrt{h^2 + k^2 + \ell^2}} \quad (9)$$

where $h = nH, k = nK$ and $\ell = nL$; $n, k,$ and ℓ are also integers.

METHOD OF RING RATIOS

A convenient way of obtaining information from a polycrystalline material's ring pattern is through the "method of ring ratios".

We will first consider a simple cubic crystal structure. Each ring is a reflection off of planes with different interplanar spacings. We will show later that the larger the interplanar spacing the smaller the ring. Thus the smallest ring is a result of reflections off a family of planes which all have the same interplanar spacing, and being the smallest ring, these planes must be the ones with the biggest interplanar spacing. These are called the (100) family of planes.

Figure 1 shows that the (100) family of planes has the largest interplanar spacings. We call this spacing the lattice parameter of the material (" a ") and it is one of the values listed amongst the constants of any material.

Lattice Parameter "a"

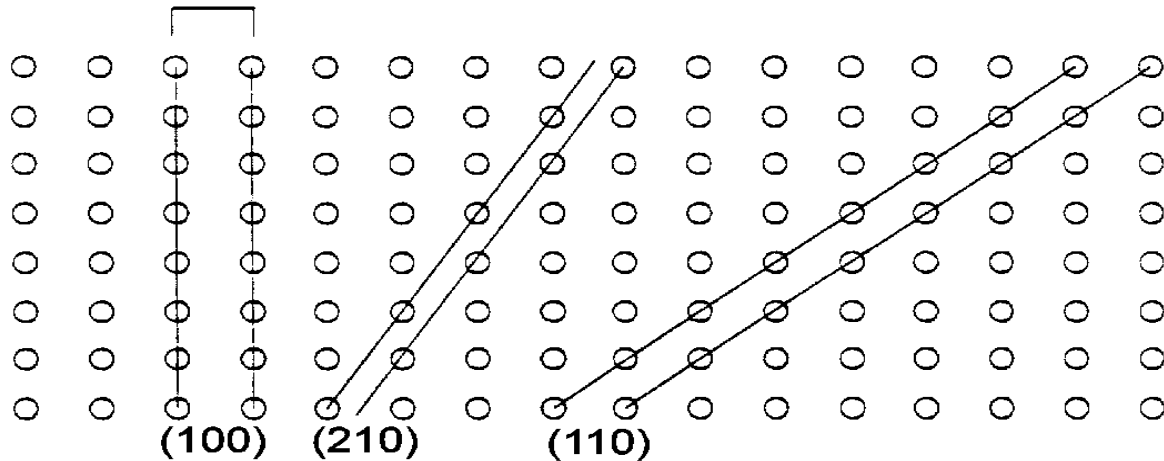


Figure 1.

Families of planes with identical spacings are known by their "Miller index" name (h,k,l) . The distance between successive planes in any material can be calculated from the lattice parameter:

$$d_{(hkl)} = \frac{na}{\sqrt{h^2 + k^2 + l^2}} = \frac{na}{\sqrt{N}} \quad (9)$$

where, for convenience we have defined $N = h^2 + k^2 + l^2$.

Thus N gives a measure of the distance between the planes: the larger the N the smaller the interplanar spacing.

Substituting equation (9) in equation (7) gives:

$$R^2 = \frac{N\lambda^2 D^2}{a^2} \quad (10)$$

Thus R^2 is proportional to N .

Here is a list of all the possible non-redundant combinations of h,k,l and their associated N values:

(h,k,l)	N
(1,0,0)	1
(1,1,0)	2
(1,1,1)	3
(2,0,0)	4
.....	..
.....	..
<i>etc.</i>	<i>etc.</i>

When looking at a simple cubic polycrystal diffraction pattern, you can measure the ring radii and the smallest will be (100), next (110), *etc.* this is called "indexing" the pattern.

To obtain the lattice parameter you simply use equation (9). Each indexed ring will yield a value for the lattice parameter. How do they compare? Should you average them? If the values are not close, maybe the specimen has an f.c.c. or b.c.c. crystal structure.

F.c.c. and b.c.c. crystal lattices have extra planes compared to the simple cubic lattices. these extra planes will result in absent diffraction rings. F.c.c. crystals will only have diffraction rings for which h, k, l are all even or all odd (zero is considered even). B.c.c. crystals on the other hand, will only have rings associated with planes for which $(h + k + l)$ is even. Thus the simple cubic, f.c.c and b.c.c. crystals will have a different series of N values.

Write down the first ten possible miller indices for f.c.c. and b.c.c. crystals and their associated N values as in the table above.

We use the notation that R_i is the i^{th} ring. The method of ring ratios makes use of the fact that

the series $\frac{R_i^2}{R_1^2}$ will be different for each of the three crystal structures and match the series $\frac{N_i}{N_1}$

for the appropriate crystal structure.

Does aluminum have a simple cubic, f.c.c. or b.c.c. crystal structure? Determine the lattice parameter of the aluminum polycrystal by plotting R^2 versus N .

OPERATING NOTES

- Before turning the apparatus "on", turn the intensity and voltage controls "off".
- Turn the A.C. Line switch on; allow a few minutes for the power supply and the cathode ray tube filament to warm up.
- Turn up the Anode Voltage to a value between 6 and 10 kV. (The meter is accurate to about $\pm 3\%$, and cannot be easily calibrated.)
- Slowly turn up the intensity control to the minimum current that allows good visibility, being careful to **never exceed a current of 10 μA .**
- Use the vertical and horizontal position controls to get the electron beam scattering off the aluminium target, which is characterized by a diffraction pattern of concentric circles. (The spot diffraction patterns are due to the graphite target.)
- Adjust the focus control until the central spot size is as small as possible for each selected voltage.
- Check the voltage after using the position and focus adjustments, since there is some interaction between the controls.
- The parameters of the experiment are the radii of the circles of the diffraction pattern, and the accelerating voltage.
- Turn the intensity and voltage controls off before switching off the AC line voltage.

THE EXPERIMENT

For one diffraction ring, measure R versus V (the accelerating voltage). Does your data agree with the V dependence predicted by equations (4) and (7)?

We suggest you do a full analysis of the polycrystalline aluminum. Do you find aluminum to be FCC or BCC? Compare the lattice constant you observe with the value of $0.4049 \pm .0001$ nm obtained from x-ray scattering data.*

You should also look at a single crystal of pyrolytic graphite, identify the spot pattern and obtain the lattice constant. (See page 4 of the instruction manual for details.)

(dh - 76, rw & jbv - 89, mf-95)

* B.D. Cullity, Elements of x-ray Diffraction, Addison Wesley (1956) p482