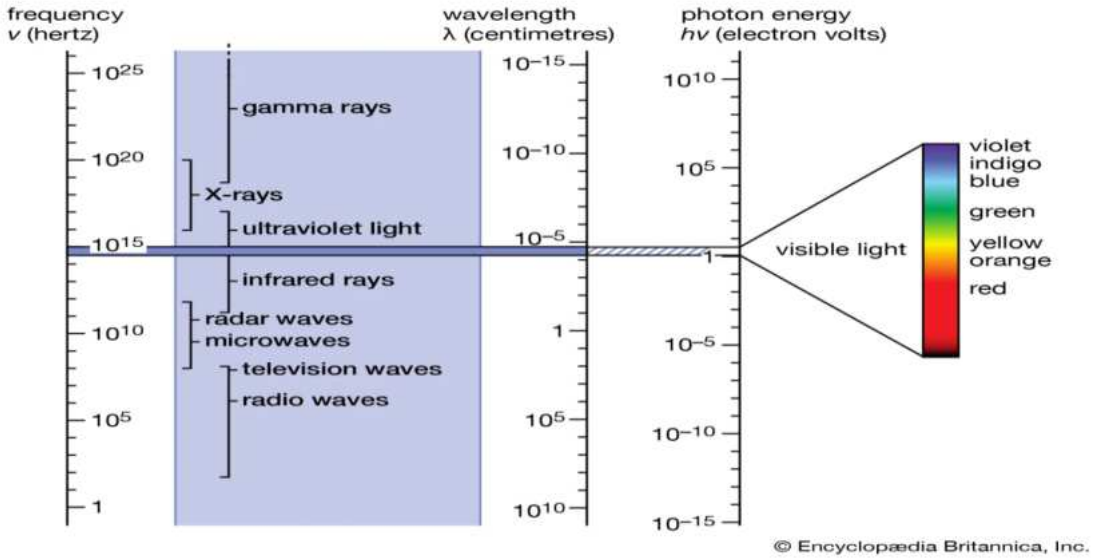
Chapter Two

Methods for Structure Investigation:

Introduction

X-rays were discovered in (1895) by the German physicist Wilhelm Röntgen and were so named because their nature was unknown at that time. X-rays are part of the electromagnetic radiation, which have high frequency ranging from (3×10^{16} Hz to 3×10^{19} Hz), short wavelength ranging from (0.1 to 10000 Å) and energies in the range (100 eV to 100 keV).

X-ray wavelengths are shorter than those of UV rays and typically longer than those of gamma rays. X-rays were invisible and they traveled in straight lines could easily pass through the human body, wood, quit thick pieces of metal and other opaque objects.

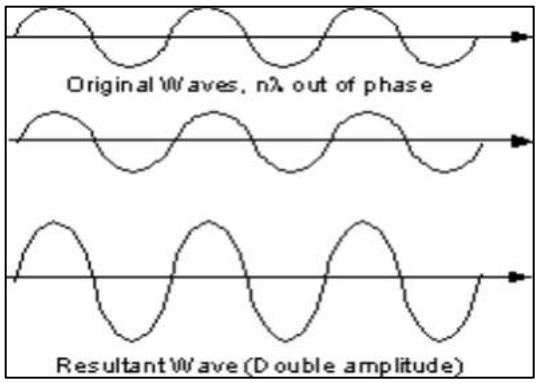


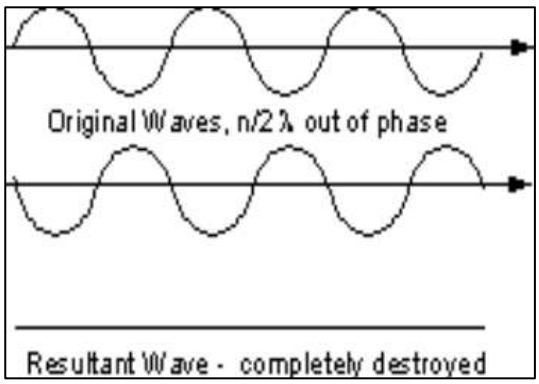
Fundamentals of Diffraction:

A beam of X-rays consists of a bundle of separate waves, the waves can interact with one another. Such interaction is termed interference.

If all the waves in the bundle are in phase, that is their crests and troughs occur at exactly the same position (the same as being an integer number of wavelengths out of phase, (nλ), n = 1, 2, 3, 4, etc.), the waves will interfere with one another and their amplitudes will add together to produce a resultant wave that is has a higher amplitude (the sum of all the waves that are in phase.

If the waves are out of phase, being off by a non-integer number of wavelengths, then destructive interference will occur and the amplitude of the waves will be reduced. In an extreme case, if the waves are out of phase by an odd multiple of (½ λ) [(2n+1)/2 λ], the resultant wave will have no amplitude and thus be completely destroyed.





Diffraction Methods:

1. **X-ray crystallography:**

Is a technique in crystallography in which the pattern produced by the diffraction of x-rays through the closely spaced lattice of atoms, which arranged in a periodic array with long-range order in a crystal. It is recorded and then analyzed to reveal the nature of that lattice.

The wave length of X-Ray can be calculated by;

$E = h\nu = \frac{hc}{\lambda}$ for X-ray

h = 6.626x10^{-34} J.s Planck's constant.

c= 3x10^{8} $m.sec$ Velocity of Light.

1 eV = 1.6 x 10^{-19} J.

$$\lambda = \frac{12.4}{E(keV)}$$

Amorphous materials like glass do not have a periodic array with long-range order, so they do not produce a diffraction pattern. Their X-ray scattering pattern features broad, poorly defined amorphous 'humps'.

2. **Electron and Neutron Diffraction:**

❖ **Wavelength for Neutron:**

Since waves are diffracted by a crystal, we can also use electrons and neutrons instead of X-rays, provided the wavelengths of matter waves associates with these particles are in the range of (1 to 10 Å). Wavelength for neutron

$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2m_n E}} = \frac{6.626\times10^{-34}}{\sqrt{2\times1.675\times10^{-27}E}} = \frac{1.145\times10^{-20}}{\sqrt{E}} = \frac{0.286}{\sqrt{E}}$$

Where (λ) is in Angstroms and (E) in eV. Thus the energy of a neutron of wavelength of (1 Å is ≈ 0.08 eV). The thermal neutrons in an atomic reactor have (the energy ≈ 0.08 eV) and hence can be used for the study of crystal diffraction.

The thermal neutrons from the nuclear reactors have a large spread in energy and therefore we have to monochromatize them. These neutrons are allowed to fall on a crystal and a particular reflected beam is selected. This is allowed to fall on the test

sample under examination. The intensity of the scattered beam is measured through a counter, which is depend on the neutron scattering cross – section of nucleus.

We must note that though the neutron diffraction studies are very similar to those of X-rays, there are many differences;

1. X-rays are scattered by electrons, whereas neutrons are scattered by nuclei.
2. Many of low atomic weight elements and discrimination between neighboring elements of periodic table can be study by neutron diffraction, where X-rays are incapable of giving precise results.
3. We know that a neutron possesses a magnetic moment and hence has an additional scattering due to magnetic ordering.
4. Neutron diffraction studies have become important in magnetic structure determinations, X-ray or electron diffraction studies do not reveal this structure.
5. Lattice vibrations can also study using neutron diffraction technique.
6. X-rays are easier to produce as compared to neutrons which require a nuclear reactor.

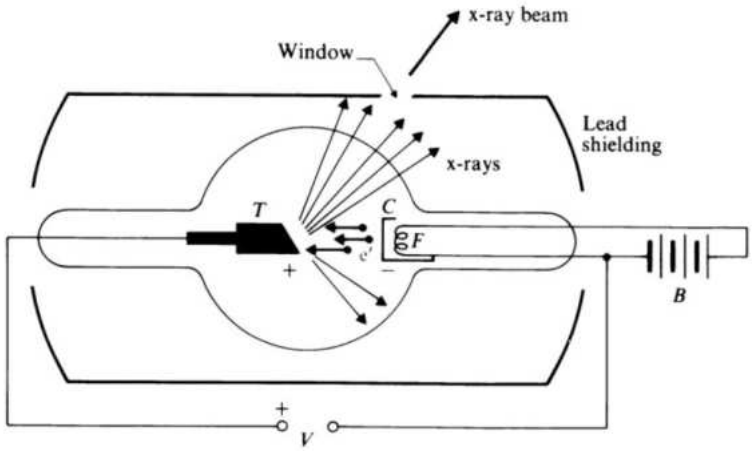
❖ **Wavelength for electron:**

$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2m_e E}} = \frac{6.626 \times 10^{-34}}{\sqrt{2 \times 9.109 \times 10^{-31} E}} = \frac{4.91 \times 10^{-19}}{\sqrt{E}} = \frac{1.227}{\sqrt{E}}$$

where (λ) is in Angstroms and (E) is in kilo eV. Like X-rays or neutrons, electrons can also be used. However, the electrons are much more easily scattered and hence the penetration depth is small. This is why electron diffraction method is used in surface studies-bonds, defects, energy states, etc.

Production of X-rays:

X-rays can be generated by an X-ray tube, a vacuum tube (low pressure 10^{-10} mm/Hg) that uses a high voltage to accelerate the electrons released by a hot cathode to a high velocity. The cathode is a filament like the lamp filament which is used as a source of electrons is heated by the electric current of about (3 Amp) and emits electrons which rapidly drawn to the target by high voltage (up to 60 kV) is across the tube. When an electron collides the metal target (the anode), part or all of its kinetic energy is converted into X-rays (0.2 %) and the power rest generates heat (99.8 %), so cooling water is circulated through the anode to keep it from melting. The kinetic energy of the generated electrons depending on the voltage difference between the filament and the target. X-rays are emitted through two or more windows in the tube. Lead shielding is necessary to contain scattered electrons and x-rays by absorbing them. As well as, filter usually made of Aluminum (Al) or Nickel (Ni) is used to absorb low energy photons. The metal target is usually tungsten, molybdenum. In crystallography, a copper target is most common.



The wavelength of the x rays is controlled by the applied voltage between the cathode and anode. For the higher potential differences (short wavelengths) the term hard x rays is used and for the lower potential differences (long wavelengths) the term soft x rays is used to describe the quality of the radiation. The type used in medical and dental diagnosis — are known as soft X rays.

The intensity of x – ray beam produced when the electron strike the anode is highly dependent on the anode material :

1. The higher the atomic number (Z) of the target, the more efficiency x-ray are produced.
2. The target material used should also have a high melting point since the heat produced when the electrons are stopped in the surface of the target is substantial .

Properties of X Rays:

1. X rays have a wide range of wavelengths.
2. X rays can penetrate some substances more easily than others. For example, they penetrate flesh more easily than bone, and bone more easily than lead. The ability of penetrate depends not only on their wavelength, but also on the density and thickness of the substance.
3. X rays affect photographic film in the same way as light rays do.
4. X rays ionize gases and cause certain substances to glow, or fluoresce.
5. They diffract in crystals.

Applications of X-rays (uses):

1. Measure the average spacing and the angles between layers or rows of atoms.
2. Determine the orientation of a single crystal or grain.
3. Find the lattice constant of lattices.
4. Studying the crystalline structure of an unknown material.
5. Measure the size, shape of atoms, the length and types of chemical bonds and internal stress of small crystalline regions.
6. They are used to detect the mean positions of the atoms and defects in the crystals.

Types of x-rays:

There are two different mechanisms by which X-rays are produce. One give rise to Bremsstrahlung (continuous) X-rays and the other is characteristic X-rays.

1. **Bremsstrahlung (continuous)(Soft) X-Ray:**

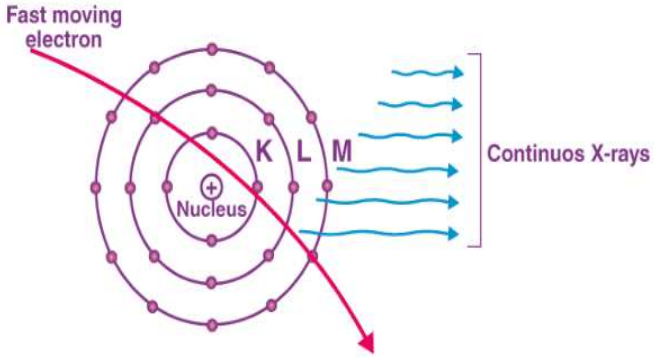
They can be generated when the velocity of the colliding electrons (coming from cathode) is reduced upon passing through a retarding electric field due to the electrons of the target material; (Many times electrons get close enough to the nucleus of a target atom). It divert from its path and emits an x-ray photon that has some of its energy. X–rays produced in this way have a fancy German name, bremsstrahlung, which means "braking radiation". Bremsstrahlung also called white radiation since it analogous to white light and has a range of wavelengths. The factors affecting the wavelength, is the potential difference between the filament and the target, ($\lambda \propto 1 / V$) and it does not change with the changing of the target material.

According to Maxwell-Hertz theory, the decrease in the energy of the electrons converts into electromagnetic radiation contains all the different possible wavelengths because the electrons lose their energy gradually.

The amount of Bremsstrahlung produced for a given number of electrons striking the anode depends upon two factor:

a. The (Z) of the target, the more protons in the nucleus the greater the acceleration of electrons.

b. The kilovolt peak-the faster the electrons, the more likely they will penetrate into the region of the nucleus.



For continuous X-Ray The total intensity is given by:

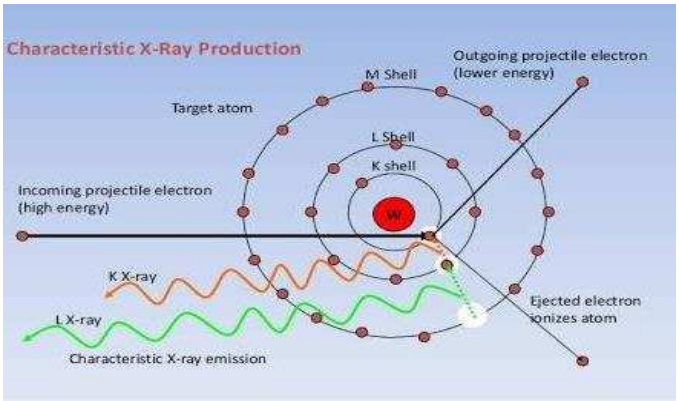
$$I_{Continues} = AIZV^m$$

where (A) is a proportionality constant, ,(I) electrical current intensity, (Z) atomic number of the target, (V) potential difference between cathode and anode and m is a constant depend on (Z), with a value of about (2).

2. Characteristic (K-Line) X-ray:

They are called Hard X-ray or trough X-ray, They can be generated when an emitted accelerated electron (coming from the cathode) collides with (K) electron close to the nucleus of the target material atom then if the later electron receives sufficient energy, it jumps to a higher level or leaves the atom altogether and is replaced by another electron from a higher level.

When an electron falls from the (L) level to the (K) level, the emitted radiation is called a (K_α) characteristic x-ray and that emitted when an electron falls from the (M) shell to the (K) shell is called a K_β characteristic x-ray.



The difference in energy appears as radiation with a definite wavelength which is determined from the relation:

$\Delta E = E_2 - E_1 = h c / \lambda$

$\lambda = h c / \Delta E$

The wavelength of the characteristic radiation does not depend on the potential difference between the cathode and the anode, although it does not appear at low potential difference. It changes by changing the target material as the wavelength decreases by increasing the atomic number of the target element.

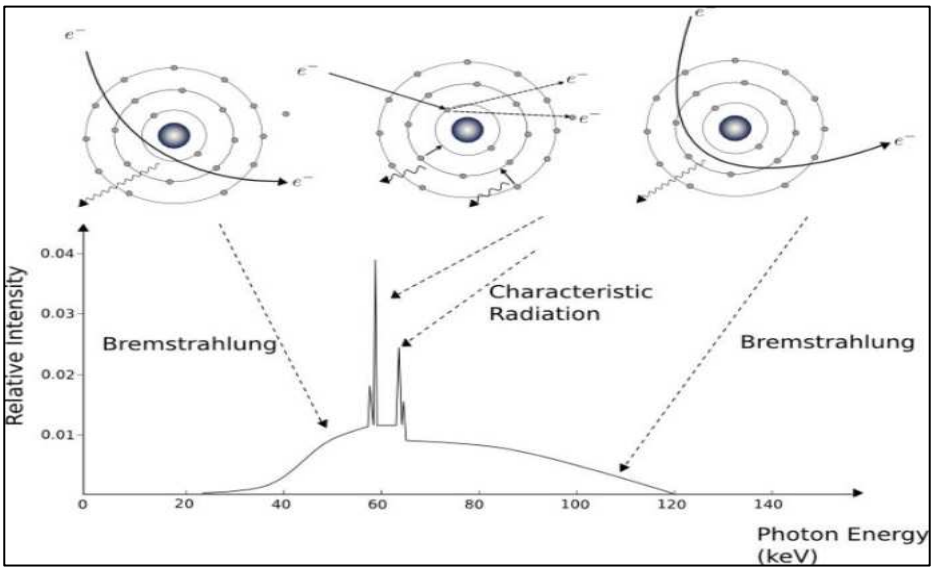
Conditions to obtain a line spectrum (characteristic) of a target material:

For a (K) line, the total X-Ray intensity is given by:

$$I_{k-line} = BI(V - V_k)^N$$

Where (B) is proportionality constant,(V) potential difference between cathode and anode,(I) electrical current intensity, (V_k) the (K-line) critical excitation voltage and (n) is constant with a value of about (1.5).

Analyzing a beam of X-rays generated from a target to compounds of different wavelengths, we find that the spectrum consists of two components, as illustrated below,



The Relation Between (λ_{SWL}) and (V_{max}):

The continuous spectrum is due to rapid deceleration of the electrons hitting the target. Not every electron is decelerated in the same way, however some are stopped in one impact and give all their energy at once, while the others are deviated this way and that by the atoms of the target losing fractions of their total kinetic energy until it is all spend. Those electrons which are stopped in one impact will give rise to photons of maximum energy i.e. x-rays of minimum wavelength, such electrons transfer all their energy (eV) into photon energy, and we may write.

$$eV = h\nu_{\max} = h\frac{c}{\lambda_{SWL}}$$

$$\lambda_{SWL} = \frac{hc}{eV} = \frac{12400}{V}$$

This equation gives the short wavelength limit in Å

Examples:

1. An x-ray operates at 30Kv and the current through it is 2.0mA.Calulate:

(i) The electrical power output

(ii) The number of electrons striking the target per second.

(iii) The speed of the electrons when they hit the target

(iv) The lower wavelength limit of the x-rays emitted.

(i) $p = VI = 30x10^{-3} x2.0x10^{-3}$

$= 60w$

(ii) $I = ne$ *where* n *is the number of electrons stricking the target persecond*

$$n = \frac{I}{e} = \frac{2x10^{-3}}{1.6x10^{-19}} = 1.3x10^{16}$$

(iii) $\frac{1}{2}mv^2 = eV$

$$v = \sqrt{\frac{2eV}{m}} = \sqrt{\frac{2x1.6x10^{-16}}{9x10^{-31}}} = 1x10^8 \frac{m}{s}$$

$$\lambda_{\min} = \frac{hc}{eV} = 0.41x10^{-10} m$$

2. Electrons are accelerated from rest through a p.d of 10Kv in an x ray tube. Calculate:

(i) The resultant energy of the electrons in eV. *($10^4 ev$)*

(ii) The wavelength of the associated electron waves. *($1.23x10^{-11} m$)*

(iii) The maximum energy and the minimum wavelength of the x ray radiation generated (assume, m_e,e, h=6.62x10^{-34}Js, c=3x10^{8}m/s): *($1.6x10^{-15} J$, $1.24x10^{-10} m$)*

$$\lambda = \frac{h}{P} \quad where \quad p = \sqrt{2eVm_e}$$

Hint:use the de Broglie equation $\lambda = \frac{h}{\sqrt{2eVm}}$

and $\lambda_{\min} = \frac{hc}{eV} = 1.24x10^{-10} m$

3. What is the k.e of an electron with a de Broglie wavelength of 0.1nm.through what p.d should it be accelerated to achieve this value? Assume e, m_e, h.

$$\lambda = \frac{h}{p} = \frac{h}{m_e v}, v = \frac{h}{m_2 \lambda} \qquad p = mv \qquad but \qquad k.e = \frac{1}{2}mv^2 = \frac{1}{2}m_e \frac{h^2}{m^2{}_e \lambda^2}$$

(use since $v^2 = \frac{h^2}{m^2{}_e \lambda^2}$)

also $eV = k.e,$ *so* $V = \frac{h^2}{2em_e \lambda^2}, \; V = 0.151Kv$

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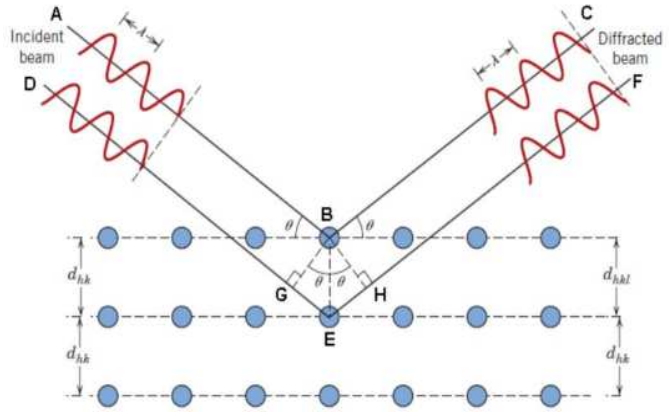
Bragg's Law:

When a beam of x-rays falls at a glancing angle (θ) on a set of parallel planes with coordinates (h k l) , separated from each other by a distance of (d_{hkl}) , it scattered in all directions inside the crystal.



It is noticeable in figure that the wave path in the direction (DEF) which is reflected in (E) is longer than the path of the wave. The wave is in the direction (ABC) that is reflected in (B), so if these two sets of waves are in the same phase (inphase), then the difference between (DEF) and (ABC) paths must be an integer number of lengths waveform (nλ), where (n) is equal to an integer (n = 1,2,3, …..)

In order to find the difference between the two paths we plot (BG) and (BH) perpendicular to (DE) and (EF), we find that:

AB = DE
BC =HF
So the difference between the two paths is equal to the sum;

$GE + EH = n\lambda$ ------ (1)
From triangle (GBE), we find that;

$BE = d_{hkl}$
$\therefore GE = d_{hkl} \sin(\Theta)$ ------ (2)
Also from triangle (HBE), we find that;

$\therefore EH = d_{hkl} \sin(\Theta)$ ------ (3)
By substituting equations (2) and (3) into equation (1), we find that;

$d_{hkl} \sin(\Theta) + d_{hkl} \sin(\Theta) = n\lambda$
$2\, d_{hkl} \sin(\Theta) = n\lambda$ ------ (4)

The Bragg reflection can only occur when the wavelength (λ) in equation (4) which is used to obtain on a reflection from a plane having Miller coordinates (hkl), **smaller than or equal to twice the interplane distance between two successive planes (d_{hkl})**.

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The Bragg condition necessary for reflection is;

$\lambda \leq 2\, d_{hkl}$

It is for this reason that visible light cannot be used to study the crystal structure. In order to obtain reflections Bragg from levels with large Miller indices, we need X-rays with short wavelengths.

Bragg's law is a logical consequence of the periodicity of the lattice and does not depend on the arrangement of the atoms accompanying its points.

Examples:

Determine the interplanar spacing when a beam of X-ray of wavelength 1.54 Å is directed towards the crystal at angle 20.3° to the atomic plane.

Solution

$2d \sin\theta = n\lambda$ | $\lambda = 1.54$ Å

$\therefore \quad 2d \sin 20.3° = 1 \times 1.54$ | $\theta = 20.3°$

$$\therefore \quad d = \frac{1.54}{2\sin 20.3°} = \frac{1.54}{2 \times 0.3469} = 2.22 \text{ Å}$$

X-rays with wavelength of 0.58 Å are used for calculating d_{200} in nickel. The reflection angle is 9.5°. What is the size of unit cell?

Solution

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad \Big| \quad \lambda = 0.58 \text{ Å}, \quad \theta = 9.5°$$

$$\therefore \quad d_{200} = \frac{a}{\sqrt{2^2 + 0^2 + 0^2}} = \frac{a}{2} = 0.5a$$

Now, from Bragg's law

$2d \sin\theta = n\lambda$, we have

$2\, d_{200} \sin 9.5° = 1 \times 0.58$

$2 \times 0.5a \times 0.165 = 0.58$

$$\therefore \quad a = \frac{0.58}{1.165} = 0.52 \text{ Å}$$

Calculate the Bragg angle if (111) planes of a cube (a = 3.57 Å) crystal are exposed to X-rays (wavelength = 1.54 Å)

Solution We have, Miller indices of the (111) planes, $h = 1$, $k = 1$ and $l = 1$; $a = 3.57°$ and $\lambda = 1.54$ Å. Let θ be the Bragg's angle for the first order reflection.

We have

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

$$d_{111} = \frac{3.57}{\sqrt{(1)^2 + (1)^2 + (1)^2}} = 2.06 \text{ Å}$$

Now, from Bragg's law, we have

$2d_{111} \sin\theta = n\lambda$

$\therefore \quad 2 \times 2.06 \times \sin\theta = 1 \times 0.54$

$$\sin\theta = \frac{1 \times 0.54}{2 \times 2.06} = 0.131$$

$$\theta = 7° \; 32'$$

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For a certain BCC crystal, the (110) plane has a separation of 1.181 Å. These planes are indicated with X-ray of wavelength 1.540 Å. Show that the maximum order of the Bragg's reflection that can be observed is n = 1.

Solution

$2d \sin\theta = n\lambda$

$d = 1.181$ Å
$\lambda = 1.540$ Å

$\therefore \quad n = \dfrac{2d\sin\theta}{\lambda}$

$= \dfrac{2 \times 1.181 \sin 90°}{1.540} = 1.53$

Determine interatomic spacing when glancing angle of 30° is observed during first order reflection in a crystal having Miller indices as (111). The wavelength of X-rays is 2 Å.

Solution

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

$\lambda = 2$ Å
$\theta = 30°$
$n = 1$
$h = k = l = 1$

$\therefore \quad d = \dfrac{a}{\sqrt{(1)^2 + (1)^2 + (1)^2}} = \dfrac{a}{\sqrt{3}}$

Now, $2d \sin\theta = n\lambda$

$$2\frac{a}{\sqrt{3}} \sin 30° = 1 \times 2 \text{ Å}$$

$$a = 2\sqrt{3} \text{ Å}$$

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Experimental Diffraction Methods:

There are several methods of recording the shape of the X-ray diffraction depending on:

1. The shape of the sample is in it was a single crystal or a material in the form of a powder.
2. The type of rays used if they were beams; Continuous spectrum or single wavelength spectrum.

❖ X-ray diffraction occurs only if the values of the θ and λ are coincided as well as fulfilling the Bragg condition.

❖ Solid state X-ray diffraction can be achieved in the laboratory by two principles;

1. The first principle is the constancy of the angle of incidence of rays (θ) and the wavelength variation of the rays (λ).

2. The second principle is fix the wavelength of the rays (λ) and the change of the angle of incidence of rays (θ).

❖ One of the essential properties of x-rays used in many diffraction experiments is that they should be **monochromatic.**

❖ There are two basic ways to obtain monochromatic beam of x-ray of the white beam (connected):

1. The absorption method. (H.W.)
2. The reflection method. (H.W.)

❖ The following methods are widely used for determining crystal structure;

1. Laue's method.

2. Rotating Crystal Method

3. Powder method

We have already discussed Bragg's method. Now, we will discuss other methods.

1. Laue's method:

This is one of the principal method to study;

a. Study of crystal symmetry and crystal directions.

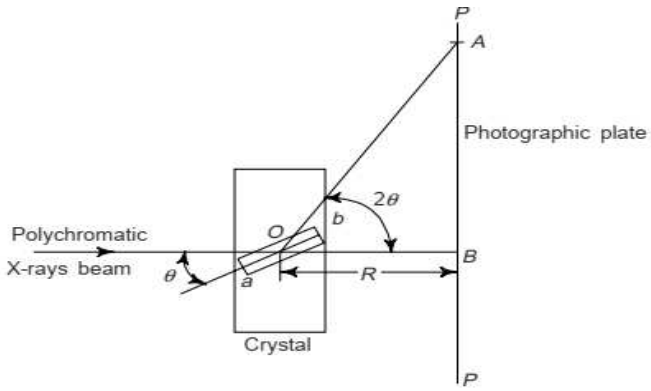
b. Information about Crystal defect which due from mechanical thermal effect.

c. Determine the unit cell shape of a solid.

❖ This method cannot be used to study the crystal structure of solids due to the wide range of wavelength of the X-rays (polychromatic X-rays) used, and it is also not possible to know the type of foundation associated with the solid material lattice under study.

In this method, a single crystal is placed in the path of X-rays beam and the glancing angle (θ) is kept constant. A white radiation having wide range of wavelengths is allowed to fall at (90°) on the crystal which having different interplanar spacings (d). Each set of crystal plane selects appropriate value of wavelength, satisfying Bragg's law and produce a black spot at (B) on photographic plate (PP), as

in figure below.



Different wavelengths are included in the primary X-rays and hence they will produce spots around this central spot, which are less pronounced.

Let (ab) represent the position of one of the possible Bragg's plane, (θ) the corresponding angle and (A) any spot (other than central spot) on (PP). From (ΔAOB), one finds;

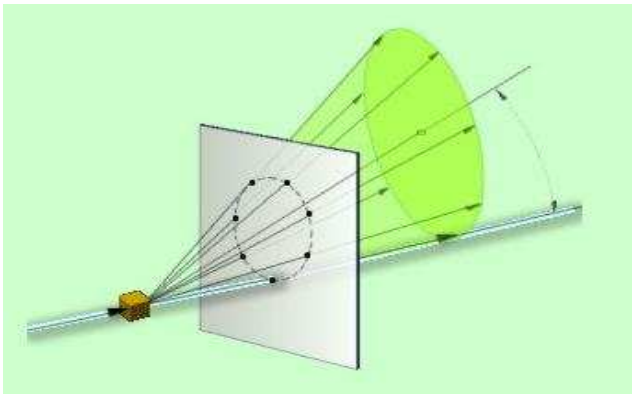
AB = R tan 2θ

Knowing (AB) and (R), one can find (θ) for the corresponding plane.

❖ There are two practical variants of the Laue method:

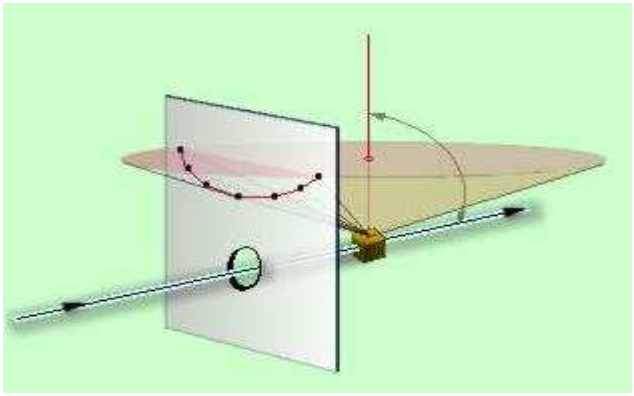
a. In the transmission Laue method, photographic film is placed behind the crystal to record beams which are transmitted through the crystal.

One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an ellipse.



b. In the back-reflection Laue method, the beams that are back diffracted are recorded on a film located between the source and crystal. The beams which are diffracted in a backward direction are recorded.

One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an hyperbola.



2. **Rotating Crystal Method:**

This method differs from Laue method by;

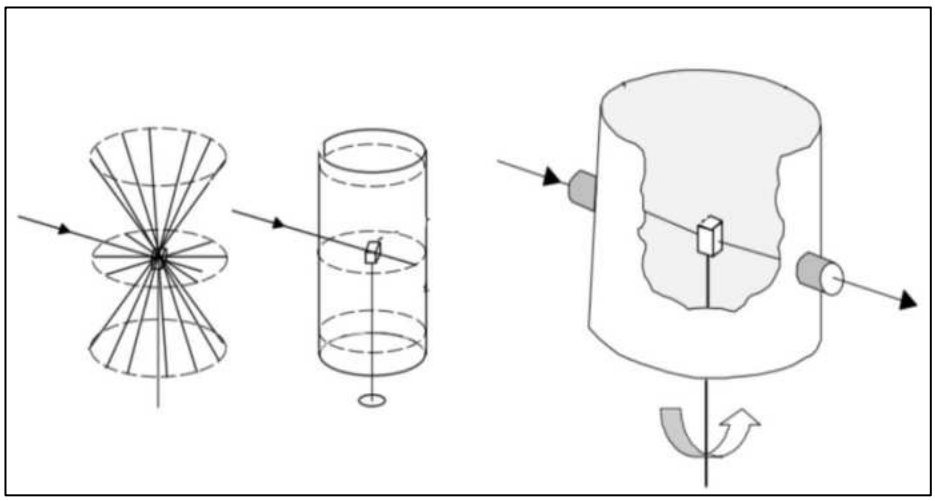
a. The crystal is allowed to rotate continuously around a fixed axis, perpendicular to the direction of the incident X-ray, meaning that this method depends on the change of Bragg's angle.

b. This method uses single-wavelength (monochromatic) x-rays.

This method is used to ;

a. Reveal the crystal structure, as the interplanar distance between the horizontal lines of the black spots on the photographic film which is perpendicular to the axis of rotation of the crystal is directly proportional to the intermediate distance in the reciprocal lattice.

b. You can also find the coordinates of the unit cell.



3. **Powder Method:**

This method is quite useful when single crystals of large size are not available. Method is used to;

a. Study the crystal structure of solids that cannot be obtained in the form of single crystals.

b. Size of crystal grains.

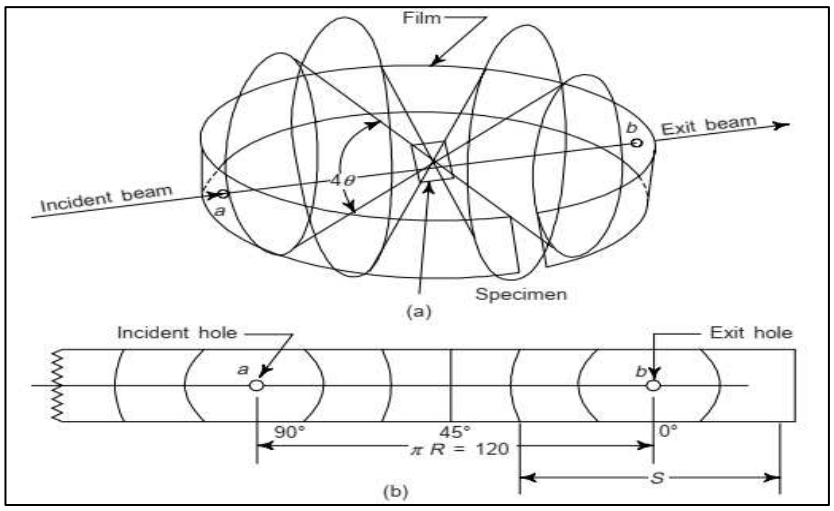
c. Orientation of crystals in crushed solid samples.

d. Identifying stresses in crystalline materials.
e. determination of unit cell dimensions.
f. measurement of sample purity.

The sample used is in the form of a fine powder containing a large number of tiny crystallites with random orientations, So the parallel atomic planes that are diverging a certain distance reflect the rays incident on them in the direction in which constructive interference occurs and fulfills Bragg's law.

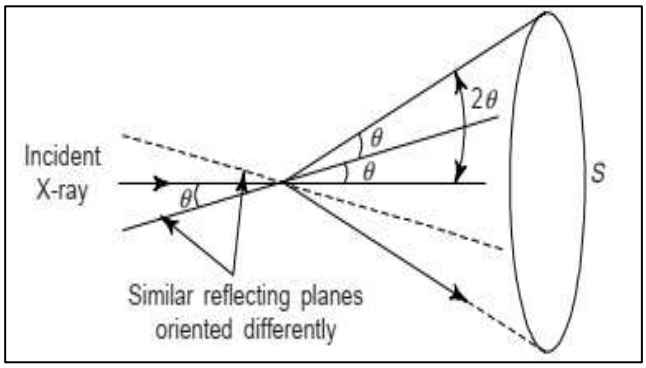
It is prepared by crushing the commonly available polycrystalline material, thus eliminating the tedious process of growing the single crystals. The powder is placed in a capillary tube or pasted on a thin wire or pressed and cemented into a thin spindle.

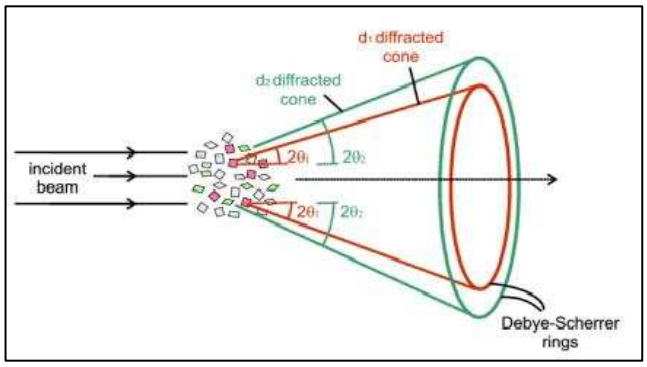
This powder specimen is mounted at the centre around which a strip of circular photographic film is positioned, as in figure below.



A collimated beam of monochromatic X-rays is produced by passing the X-ray through a filter and a collimator. This is allowed to strike the specimen (in capillary tube or wire or spindle) through a small hole. The specimen (spindle etc.) is slowly revolved inside the specially constructed powder camera. The X-ray beam enters through hole ‘a’, passes through the specimen and the unused part of beam exit through hole ‘b’.

Let us consider that the incident beam make an angle (θ) with a set of parallel crystal plane. If Bragg condition is satisfied, then there will be reflection. Since there are a large number of randomly oriented crystals in the powder sample and hence there are several possible orientations of this set of planes of same angle (θ). Consequently, the reflected rays will not be in the form of parallel beam but they will lie on the surface of a cone with its apex at the sample and the semivertical angle (2θ), as in figure below.





The angle between the surface of cone, i.e. reflected ray and exit of beam is (2θ), i.e., the apex angle is (4θ). The angle (θ) corresponding to a particular pair of arcs is related to the distance (S) between the arcs is;

$$4\theta(radiantions) = \frac{S}{R}$$

where (R) is the radius of the camera

$2\pi R \propto 360^\circ$ (circumference)

$$\frac{S}{2\pi R} = \frac{4\theta}{360^o} = \frac{\theta}{90^o}$$

$$S = \frac{2\pi R\theta}{90^o}$$

If (θ) is measured in degrees, the above equation is modified as;

$$4\theta(degrees) = \frac{360S}{2\pi R} = \frac{360S}{2(3.14158)R} = \frac{57.296\,S}{R}$$

The calculations can be made simpler by taking the radius of the camera in multiples of (57.296). For example, taking (R = 57.296 mm), we get;

$$\theta(degrees) = \frac{S(mm)}{4}$$

Obviously, one fourth of the distance between the corresponding arcs of a particular pair in (mm) is a measure of the angle (θ) in degrees. Knowing all the possible (θ's) and considering only the first order reflections from all the possible planes, relation ($2d \sin\theta = n\lambda$) is used to calculate the interplanar spacings for various sets of parallel planes which contribute to these reflections. Thus, we have;

$$d = \frac{\lambda}{2\sin(\theta)}$$

These (d) values are used to determine the space lattice of the crystal structure.

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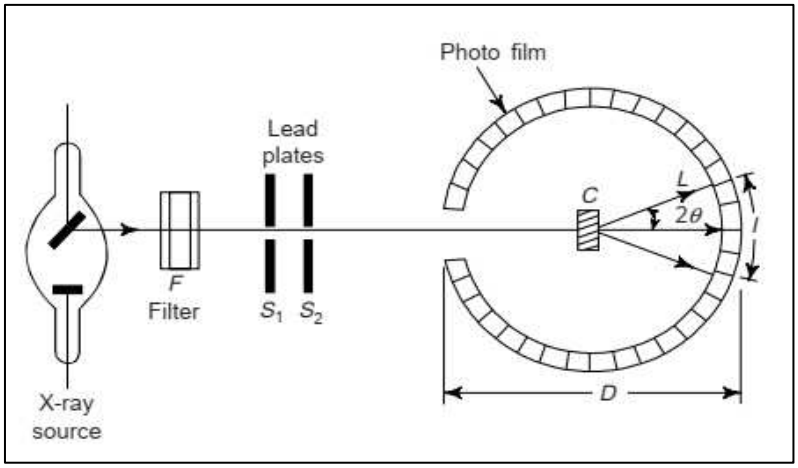
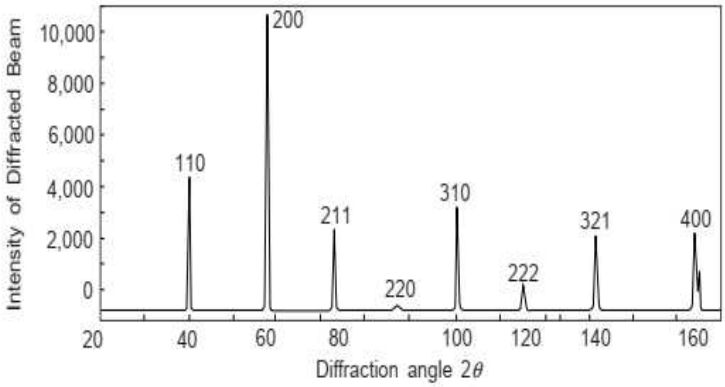
The experimental arrangement of powder method (Debye-Scherrer Camera) is shown in figure below.

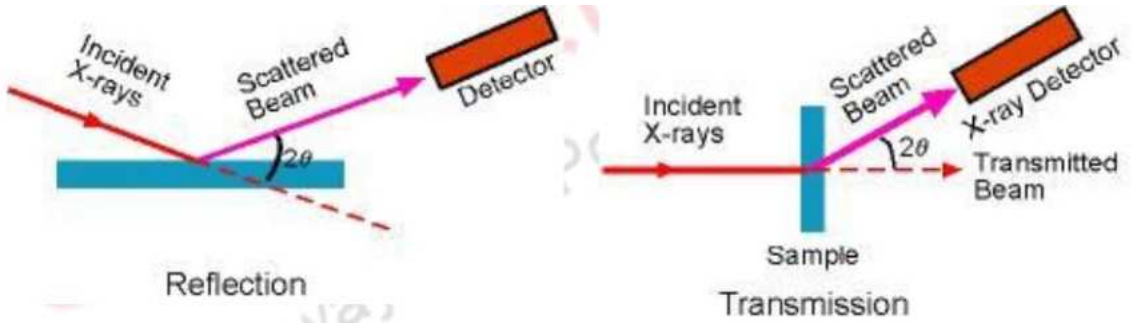


It consists of a cylindrical camera, called the (Debye-Scherrer) Camera , whose length is small as compared to the diameter.

Figure below shows the diffraction pattern of tungsten metal. The numbers on the peak are the indices of the planes which produce that peak. One can compute the interplanar spacings by measuring the value of (θ) and using Bragg's law.



Note : Powder diffraction data can be collected using either reflection or transmission geometry, as shown below.



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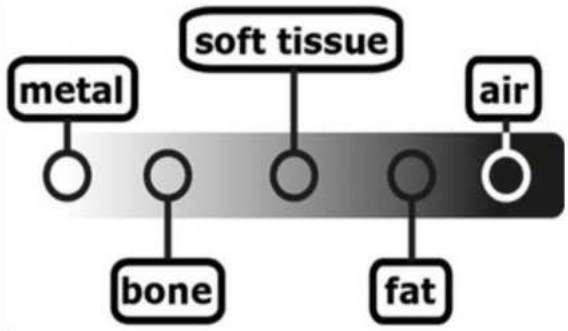
Because the particles in the powder sample are randomly oriented, these two methods will yield the same data.

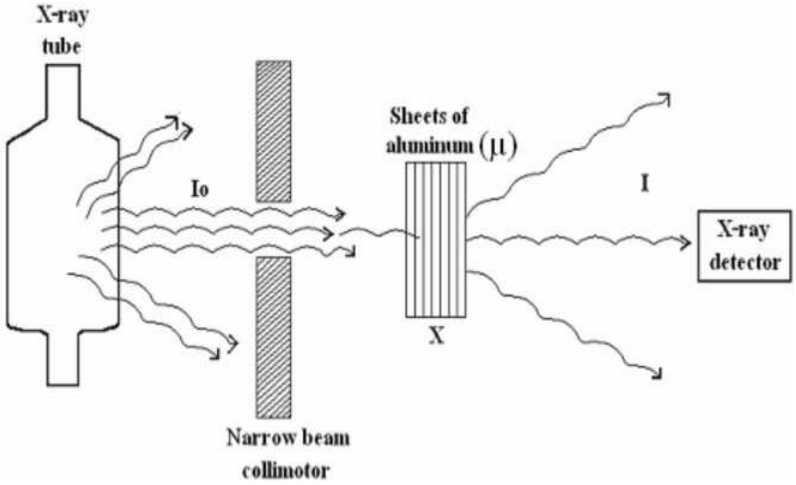
Attenuation of X-rays:

X-rays are not absorbed equally well by all materials, Heavy elements like calcium are much better absorbers of x-rays than light elements like carbon, oxygen and hydrogen, and as a result, structures containing heavy elements, like bones, stand out clearly. There are five basic radiographic densities, air, fat, soft tissue/ fluids, bone and metal. The whiteness (density) depends on the amount of x-ray radiation passing through the tissue as figure below.



The attenuation of an x-ray beam is its reduction due to the absorption & scattering of some of photons of the beam. It is an exponential process and, therefore, the beam intensity never reaches zero, figure below shows a simple method of measuring the attenuation of an x-ray beam. Attenuation of the beam can be represented numerically by:

1. Linear attenuation coefficient.
2. Half value layer.
3. Mass attenuation coefficient.



To measure the un attenuated (transmitted) beam intensity (I), we use,

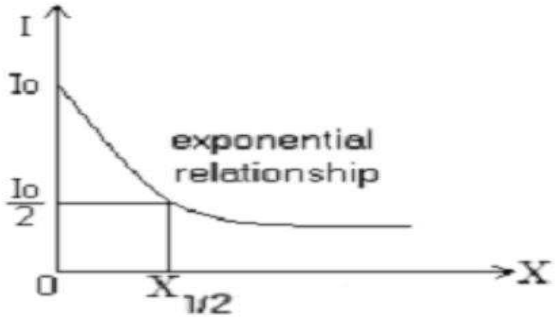
$I = I_o e^{-\mu x}$ ------ (*)

Where, I_o: un attenuated (transmitted) beam intensity, I: initial beam intensity, μ: linear attenuation coefficient, e: 2.718, x: thickness of the attenuator such as (brain tumor, bone, aluminum).

Linear attenuation Coefficient (μ) measure the probability that photon interact (absorbed or scattered) per unit length it travel in specified material, It depends on:

1. Energy of x-rays.
2. Atomic number (Z).
3. Density (ρ) of material.

Half value thickness (HVT)($X_{1/2}$) is the thickness of material which reduces the intensity of the beam of radiation one half of its value (50%), as in figure below.



At thickness $x = x_{1/2}$ then $I = 1/2\ I_o$

Substitute this condition in the equation (*):

$I = I_o e^{-\mu x}$

$1/2\ I_o = I_o e^{-\mu X 1/2}$

$1/2 = e^{-\mu X 1/2}$

$2^{-1} = e^{-\mu X ½}$

By taking Ln of both sides we get:

$-\ln 2 = -\mu X_{1/2} \times \ln e$

$0.693 = \mu X_{1/2} \times 1$

$X_{1/2} = 0.693 / \mu$ ------ (**)

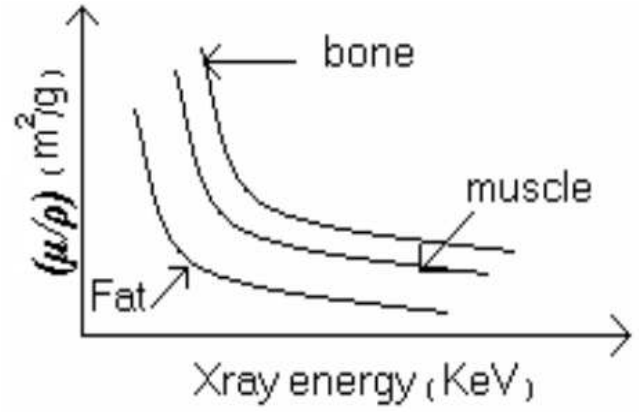
Mass attenuation coefficient (μ_m):

Which is used to remove the effect of density when comparing attenuation in several materials. The mass attenuation coefficient of a material is equal to the linear attenuation coefficient (μ) divided by the density ρ of the material. So equation (*) can be rewritten as:

$$I = I_o\, e^{(-\mu/\rho)(\rho x)} = I_o\, e^{-\mu m (\rho x)}$$

The quantity (ρx) is called the area density and is in (gm/cm^2), while ($\mu_m = \mu / \rho$) is in (cm^2/gm).

The mass attenuation coefficient emphasizes that the mass is primarily responsible for attenuating the X-rays. So if (1 gm) of lead covering an area of (1 cm^2) will absorb the same amount of x-rays whether its density is (11 gm/cm^3) or whether it is mixed with plastic to reduce its density to (2 g/cm^3).



The above quantities only really apply to a monoenergetic (one energy value) beam of X-rays from a point source (infinitely small area) travelling in a vacuum. In reality, the X-ray beam focus is not a fine point and contains photons of different energies i.e. hetrogenous beam that means the beams produced by X-ray tubes are photons of a wide range of energies, The lower-energy photons are attenuated proportionally more than the higher energy photons and are removed, leaving behind higher energy photons the resulting beam is of a higher average energy, therefore, penetrate tissue easier and the (HVL) is increased.

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