

# Module 5

## Material Characterization and Instrumentation Techniques



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**Subject:** Engineering Physics

**Subject Code:** 21PHY12/22



**Wilhelm Conrad Röntgen** was a German mechanical engineer and physicist, who, on 8 November 1895, produced and detected electromagnetic radiation in a wavelength range known as X-rays or Röntgen rays, an achievement that earned him the inaugural Nobel Prize in Physics in 1901.

Röntgen was awarded the first Nobel Prize in Physics. The award was officially "in recognition of the extraordinary services he has rendered by the discovery of the remarkable rays subsequently named after him" Röntgen donated the 50,000 Swedish krona reward from his Nobel Prize to research at his university, the

University of Würzburg. Like Marie and Pierre Curie, Röntgen refused to take out patents related to his discovery of X-rays, as he wanted society as a whole to benefit from practical applications of the phenomenon. Röntgen was also awarded Barnard Medal for Meritorious Service to Science in 1900.

### **Course Syllabus:**

#### **Material Characterization and Instrumentation Techniques: 08 Hours**

Introduction to materials: Nanomaterials and nanocomposites. Principle, construction and working of X-ray Diffractometer, crystal size determination by Scherrer equation, Principle, construction, working and applications of Atomic Force Microscopy (AFM), Fourier transform infrared spectroscopy(FTIR), X-ray photoelectron spectroscopy(XPS), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM).

**5.1 INTRODUCTION TO NANOMATERIALS:**

Nanomaterials are cornerstones of nanoscience and nanotechnology. Nanostructure science and technology is a broad and interdisciplinary area of research and development activity that has been growing explosively worldwide in the past few years. It has the potential for revolutionizing the ways in which materials and products are created and the range and nature of functionalities that can be accessed. It is already having a significant commercial impact, which will assuredly increase in the future.

**5.1.1. BASICS OF NANOMETRES:**

We know materials are composed of atoms with different sizes, which has movement with one another. There exists a special cause of materials in which the atoms do not move away from each other and its size will be in the order of 1 to 100 Nano meters. These new materials are called as Nano materials and the developed technology is called nanotechnology. Using this highly sophisticated latest technology, the Nano materials can be formed from metals, ceramics, Polymers and even from liquids.

**5.1.2. EXPLANATION:**

Nano material is one billionth of a metre. For comparison, single human hair is about 80000 nm or 80 micrometre, red blood cell is approximately 7000 nm or 7 micrometre wide and water molecule is almost point 0.3nm across. Scientist and engineers on our days interested in nanoscale materials, which is ranging from 1 to 100nm. At nanoscale, the properties of materials are very different from those at larger scale. Therefore, the Nano world is in between Quantum world and macro world. Nanoscience is concerned with the study of phenomena and manipulation of materials at nm scales. Nanotechnology is the design, production, characterization and application of structures, devices and Systems by controlling shape and size at the nanometer Scale.

**5.1.3 SPECIAL FEATURES OF NANO PHASE MATERIALS:**

In addition to smaller grain size, nanomaterials contain less number (a few ten thousand) of atoms. In contrast, the grain size of conventional materials varies from a few microns to few millimetres and contains several billions of atoms. These basic differences make nanomaterials unique and special. Nano materials contain a very large number of grain boundaries, which may be 50% or more of the total volume of the material. This unique micro structure makes this material to exhibit different properties such as tremendous strength, form ability toughness.

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For example, crystalline copper is five times stronger than ordinary copper. Further, the increased grain boundary increases magnetic and chemical properties.

## 5.2. INTRODUCTION TO NANOCOMPOSITES

Nanocomposites are materials that incorporate Nano-sized particles into a matrix of standard material. The result of the addition of nanoparticles is a drastic improvement in properties that can include mechanical strength, toughness and electrical or thermal conductivity.

Composite structures whose characteristics dimensions are found at nanoscale. Nanocomposite is a multiphase solid material where one of the phases has one, two or three dimensions of less than 100 nanometers (nm) or structures having Nano-scale repeat distances between the different phases that make up the material. The idea behind Nanocomposite is to use building blocks with dimensions in nanometer range to design and create new materials with unprecedented flexibility and improvement in their physical properties.

### 5.2.1 DIFFERENT FORMS OF NANOCOMPOSITES

#### 1. Nanostructured material

The structures whose characteristics variations in design length is at the nanoscale.

#### 2. Nano tubes or Carbon nanotubes

Carbon nanotube is a hollow tube made up of carbon of nanoscale diameter. In short, it is represented as CNTS. Carbon nanotubes are also called Bucky tubes. Nanotubes are formed by folding or rolling two-dimensional graphite into a cylindrical shape structure. The particle size is in the order of  $10^9$  m.

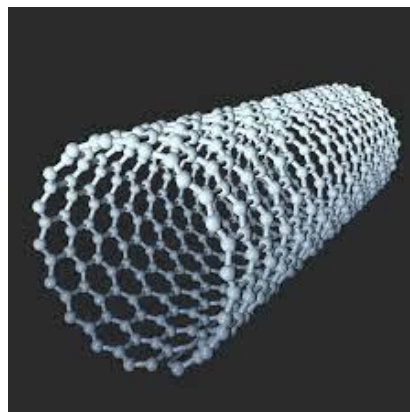


Fig: Carbon Nanotubes

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### 3. Nano Dots

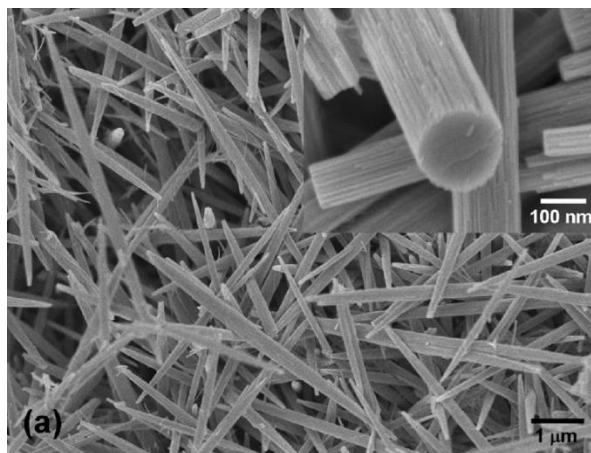
Nanoparticles, which consists of homogeneous material, especially they are almost spherical or cubical in shape.



**Fig:** Nanodots

### 4. Nano Rods

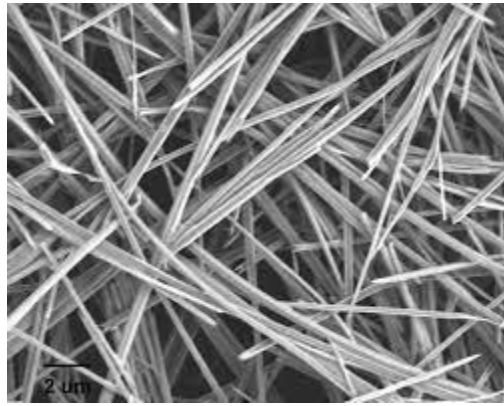
Nano structures which are shaped like sticks or rods with diameter in nanoscale and their length very much longer.



**Fig:** Nano Rods

### 5. Nanowires

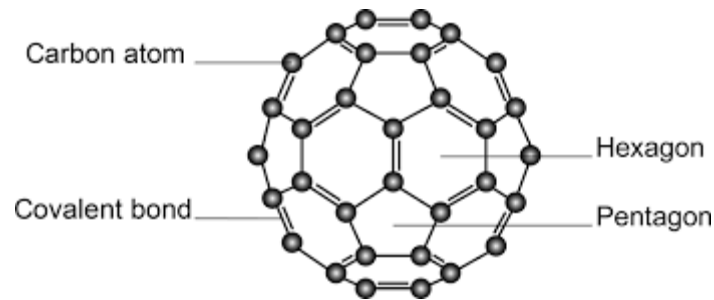
Nanowires are Nano rods, which especially conduct electricity.



**Fig:** Magnesium oxide Nanowire

## 6. Fullerenes

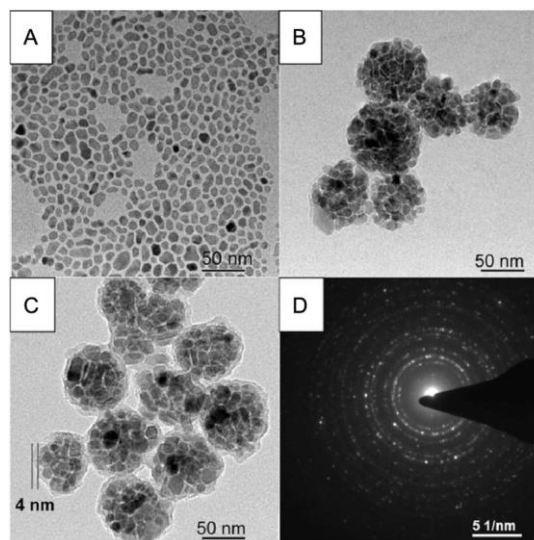
A form of carbon having a large molecule sting of an Empty cage of 60 or more carbon atoms.



**Fig:** Fullerene

## 7. Cluster

A collection of units atoms are reactive molecules up to about 50 units.

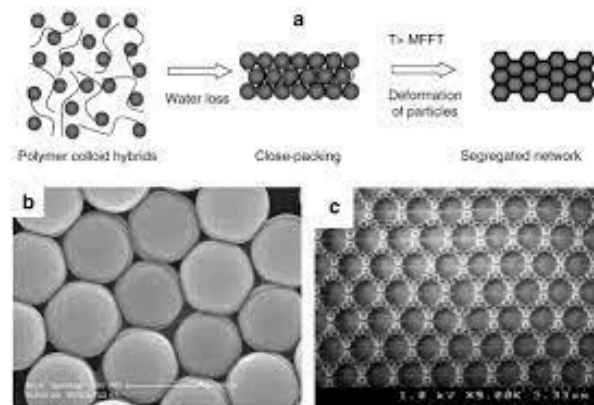


**Fig:** Cluster of SPIONs.



## 8. Colloids

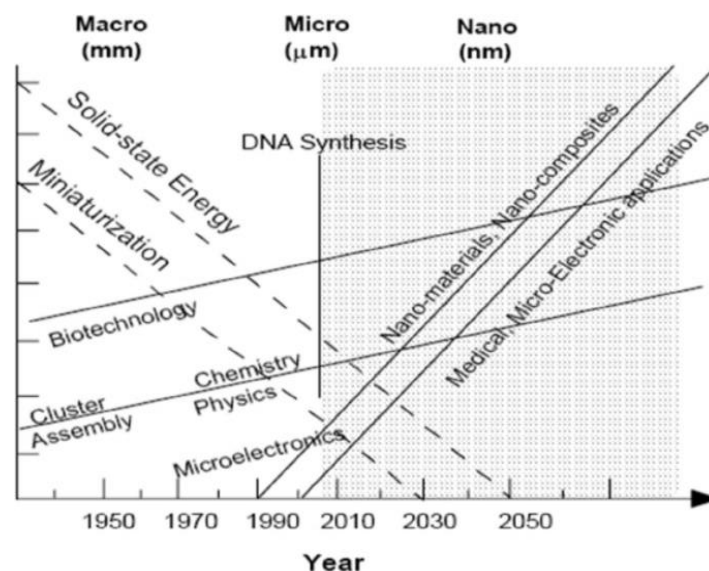
Stable liquid phase containing particles of size 1 1000nm range. A number of metals, alloys intermetallic, ceramics, polymers as well as composites are synthesized in Nano form.



**Fig:** Colloid-Polymer Nanocomposite

### 5.3. WHAT ARE NANOMATERIALS?

Nanoscale materials are defined as a set of substances where at least one dimension is less than approximately 100 nanometres. A nanometre is one millionth Of a millimetre approximately 100,00o times smaller than the diameter of a human hair. Nanomaterials are of interest because at this scale unique optical, magnetic, electrical, and other properties emerge. These emergent properties have the potential for great impacts in electronics, medicine, and other fields.

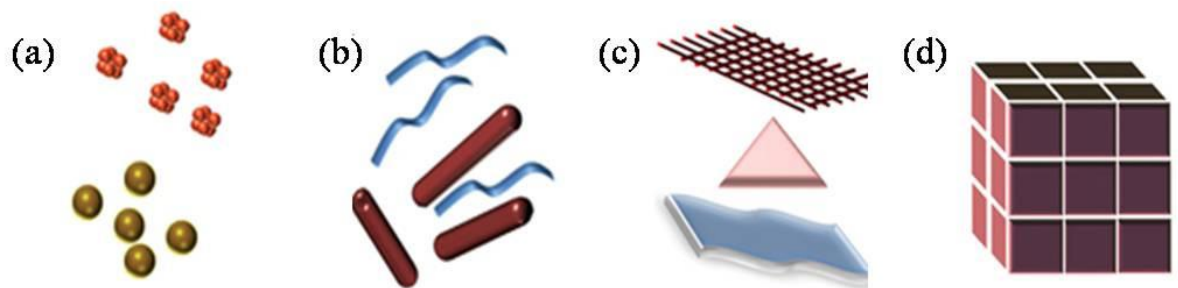


**Fig 1.** Evolution of science and technology and the future

### 5.3.1. CLASSIFICATION OF NANOMATERIALS

Nanomaterials have extremely small size which having at least one dimension 100 nm or less. Nanomaterials can be nanoscale in one dimension (e.g. surface films), two dimensions (e.g. strands or fibers), or three dimensions (e.g. particles). They can exist in single, fused, aggregated or agglomerated forms with spherical, tubular, and irregular shapes.

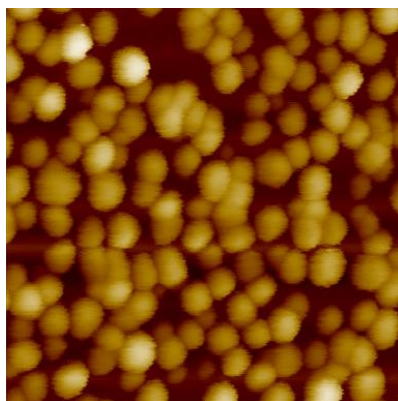
Common types of nanomaterials include nanotubes, dendrimers, quantum dots and fullerenes. Nanomaterials have applications in the field of nano technology, and displays different physical chemical characteristics from normal chemicals (i.e., silver nano, carbon nanotube, fullerene, photocatalyst, carbon nano, silica). According to Siegel, Nanostructured materials are classified as Zero dimensional, one dimensional, two-dimensional, three-dimensional nanostructures.



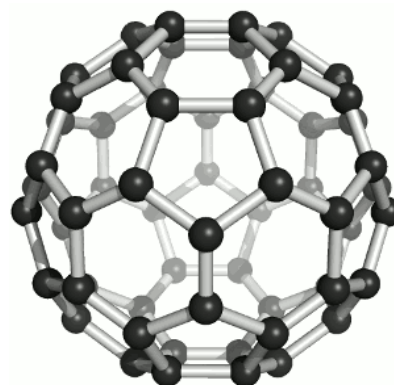
**Fig 2:** Classification of Nanomaterials (a) 0D spheres and clusters; (b) 1D nanofibers, nanowires, and nanorods; (c) 2D nanofilms, nanoplates, and networks; (d) 3D nanomaterials.

Nanomaterials are materials which are characterized by an ultra-fine grain size ( $< 50$  nm) or by a dimensionality limited to 50 nm. Nanomaterials can be created with various modulation dimensionalities as defined by Richard W. Siegel: zero (atomic clusters, filaments and cluster assemblies), one (multilayers), two (ultrafine-grained overlayers or buried layers), and three (nanophase materials consisting of equiaxed nanometer sized grains) as shown in the above figure 2.

### 5.3.2. Examples of Nanomaterials:

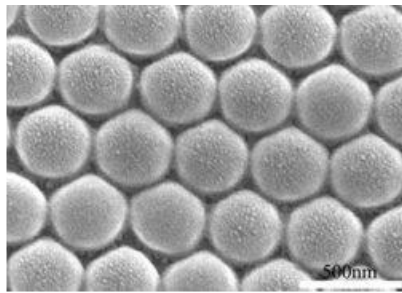


Au nanoparticle

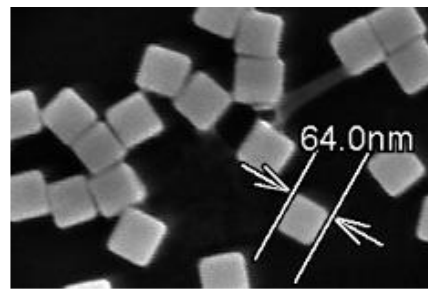


Buckminsterfullerene

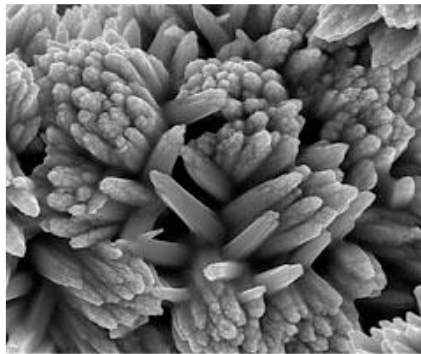




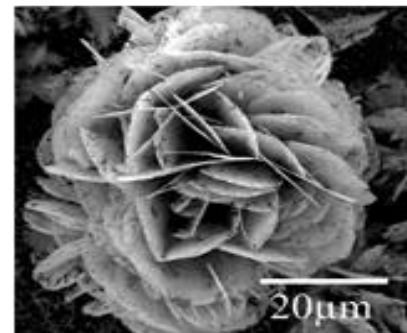
FePt nanosphere



Silver Nanocubes



Titanium nanoflower

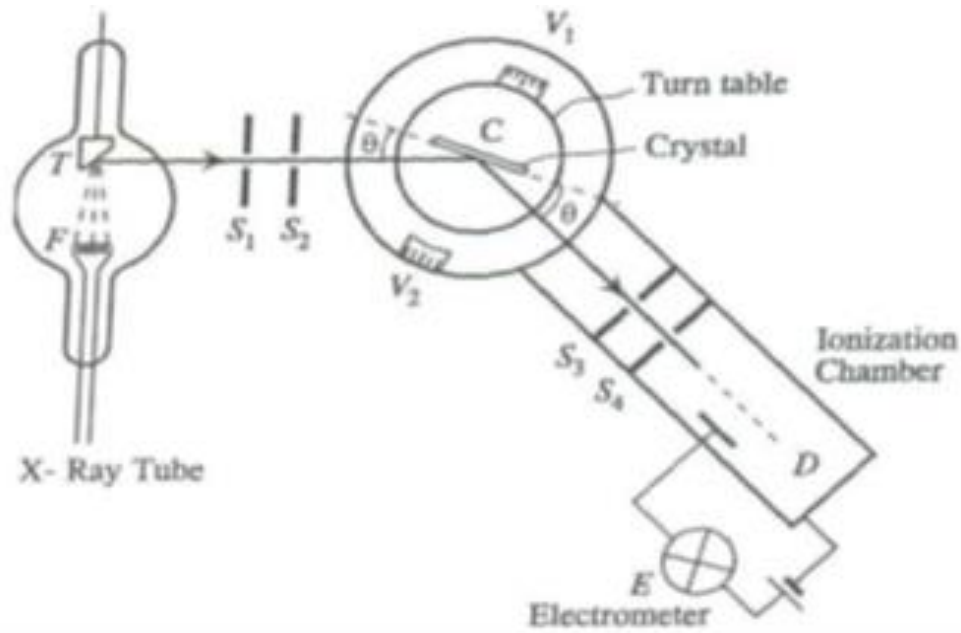
SnO<sub>2</sub> nanoflower

#### 5.4. X-RAY POWDER DIFFRACTION (XRD):

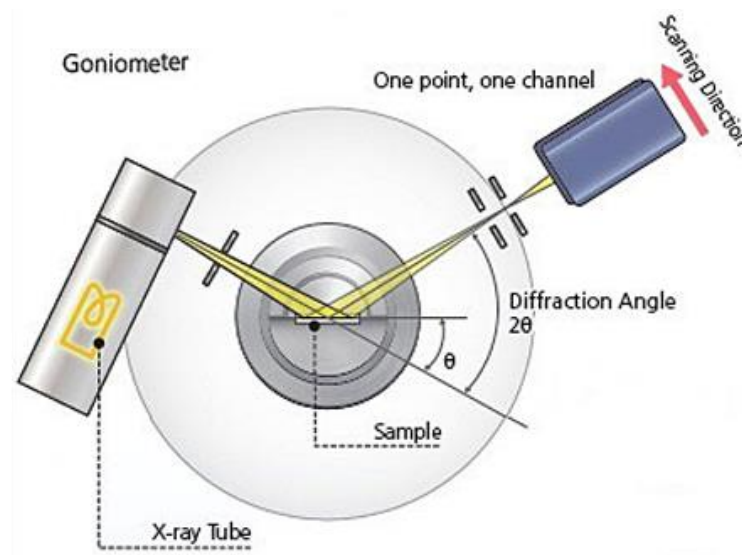
X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analysed material is finely ground, homogenized, and average bulk composition is determined.

X-Rays produced from an X-ray tube for passed through two lead slits  $S_1$  and  $S_2$  as shown in figure. This Beam is made to fall on the Crystal C on the top of the thin wire mounted exactly at the center of a circular turntable. The position of the table can be read by means of Vernier scale  $V_1$ . The X-ray beam after reflection enters an ionization chamber D filled with a gas. The ionization chamber is mounted on a mechanical arm issue, which can turn coaxially with the turntable. However, the mechanical arm is so coupled to turntable that, for every rotation by an angle  $\theta$  of the turntable, the mechanical arms turned through  $2\theta$ . This is required because, as the law of reflection, whenever a reflecting surface turns through an angle  $\theta$ , the reflected ray turns through an angle  $2\theta$ .

Hence, the X rays after reflection from the Crystal are always made to enter the ionization chamber D, and they are again made into a narrow beam by means of slits  $S_3$  and  $S_4$ . The position of the arm carrying the chamber read by the Vernier  $V_2$ . The X-rays produce ionization of the gas in the chamber D. The ionization current is measured by the electrometer E. If more X - rays enters the chamber, there will be an increase in the ionization current.



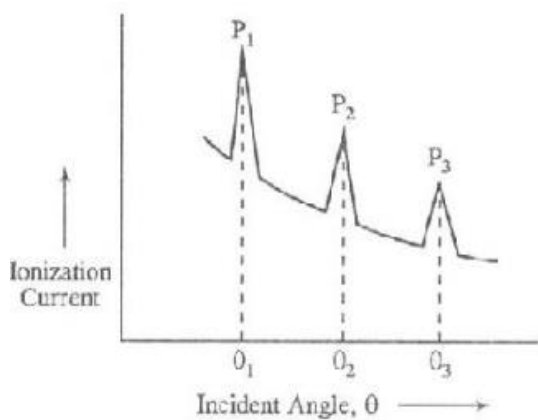
**Fig:** Schematic diagram of construction and working of XRD



When the reflected rays from the Crystal reinforce, the intensity of X rays entering the ionization chamber increases causing a rise in the ionization current. Reinforcement occurs when Bragg's law is obeyed. While, the experiment is carried out, the turntable is rotated until the Crystal receives the X-ray beam at an angle of incidence satisfying the Bragg's law. This is indicated by a sudden rise in ionization Current. The rise in current occurs more than once as  $\theta$  is varied, because the law is satisfied for various values of  $n$ .

$$2d\sin\theta = \lambda: 2\lambda : 3\lambda \text{ and likewise.}$$

If the ionization current is plotted against the incident (glancing) angle  $\theta$ , then a graph as shown in the below figure is obtained. The peaks  $P_1, P_2, P_3$  etc. are observed at  $\theta_1, \theta_2, \theta_3$  etc.

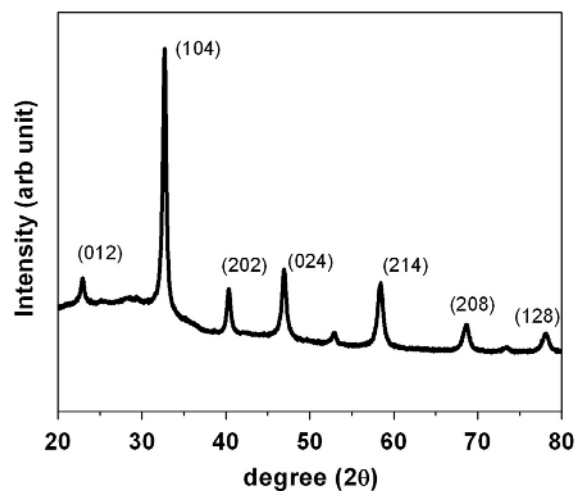


Using Bragg's law, we can write

$$2d\sin\theta_1: 2d\sin\theta_2: 2d\sin\theta_3 = \lambda: 2\lambda: 3\lambda$$

$$\sin\theta_1: \sin\theta_2: \sin\theta_3 = 1:2:3$$

This helps in assessing the accuracy of measurement of  $\theta_1, \theta_2, \theta_3$ . One can measure the important crystal parameter, the inter-planar spacing  $d$ , through the relation  $2d\sin\theta = n\lambda$ , by using the measured value of  $\theta$ , provided,  $n$  the order and  $\lambda$  wavelength of incident x-ray is known.



**Fig:** XRD Pattern of LSMO Manganite

#### 5.4.1. DETERMINATION OF CRYSTAL STRUCTURE:

One gets different values for  $d$  depending upon the particular set of parallel planes that satisfy Bragg's reflection as  $\theta$  is changed. By taking the ratios of the different values of  $d$  obtained, it is possible to decide the particular crystal system to which the experimental crystal would belong. For example, let us consider a case of cubic crystal. Let, inter-planar spacing for (100) planes be  $d_1$ , and that for (110) and (111) planes be  $d_2$  and  $d_3$ , respectively. By the geometry of the Planes in a cubic crystal, it can be shown that,

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$$d_1 : d_2 : d_3 = 1 : \frac{1}{\sqrt{2}} : \frac{1}{\sqrt{3}}$$

Thus, among the many experimentally obtained values for different  $d$  for the Crystal. If 3 of them are found to bear the same ratio above, it indicates that the crystal structure of the Crystal under study is Cubic.

Further, for FCC  $d_1 : d_2 : d_3 = 1 : \frac{1}{\sqrt{2}} : \frac{2}{\sqrt{3}}$

And for BCC,  $d_1 : d_2 : d_3 = 1 : \frac{2}{\sqrt{2}} : \frac{1}{\sqrt{3}}$

#### 5.4.3. DETERMINATION OF WAVELENGTH:

In practice,  $\lambda$  is calculated as follows. In case of sodium chloride crystal,  $d$  has been calculated by molecular data with assistance of X-Ray data. Using sodium chloride crystal as the specimen, the experiment is performed. Following the same procedure, the various values of  $\theta$  satisfying Bragg's law condition is obtained. Since  $d$  is already known, the wavelength  $\lambda$  of X-rays is evaluated using Bragg's law and hence  $\lambda$  is determined. The Bragg's Equation is written as,

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

#### 5.4.4. SCHERRER EQUATION

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The **Scherrer equation**, in X-ray diffraction and crystallography, is a formula that relates the size of sub-micrometer crystallites in a solid to the broadening of a peak in a diffraction pattern. It is often referred to, incorrectly, as a formula for particle size measurement or analysis. It is named after Paul Scherrer. It is used in the determination of size of crystals in the form of powder.

The Scherrer equation can be written as:

$$D = \frac{K\lambda}{\beta \cos \theta}$$

where:

- $D$  is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size, which may be smaller or equal to the particle size;
- $K$  is a dimensionless **shape factor**, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite;
- $\lambda$  is the X-ray wavelength;

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- $\beta$  is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. This quantity is also sometimes denoted as  $\Delta(2\theta)$ ;
  - $\theta$  is the Bragg angle.

The Scherrer equation is limited to Nano-scale crystallites, or more-strictly, the coherently scattering domain size, which can be smaller than the crystallite size (due to factors mentioned below). It is not applicable to grains larger than about 0.1 to 0.2  $\mu\text{m}$ , which precludes those observed in most metallographic and ceramographic microstructures.

It is important to realize that the Scherrer equation provides a lower bound on the coherently scattering domain size, referred to here as the crystallite size for readability. The reason for this is that a variety of factors can contribute to the width of a diffraction peak besides instrumental effects and crystallite size; the most important of these are usually inhomogeneous strain and crystal lattice imperfection.

The Scherrer equation is a widely used tool to determine the crystallite size of polycrystalline samples. However, it is not clear if one can apply it to large crystallite sizes because its derivation is based on the kinematical theory of X-ray diffraction.

For large and perfect crystals, it is more appropriate to use the dynamical theory of X-ray diffraction. Because of the appearance of polycrystalline materials with a high degree of crystalline perfection and large sizes, the full width at half-maximum is then extracted and the crystallite size is computed using the Scherrer equation. It is shown that for crystals with linear absorption coefficients below 2117.3  $\text{cm}^{-1}$  the Scherrer equation is valid for crystallites with sizes up to 600 nm.

It is also shown that as the size increases only the peaks at higher  $2\theta$  angles give good results, and if one uses peaks with  $2\theta > 60^\circ$  the limit for use of the Scherrer equation would go up to 1  $\mu\text{m}$ .

## 5.5. ATOMIC FORCE MICROSCOPY (AFM)

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### 5.5.1. INTRODUCTION:

The atomic force microscope (AFM) is a type of scanning probe microscope whose primary roles include measuring properties such as magnetism, height, friction. The resolution is measured in a nanometer, which is much more accurate and effective than the optical diffraction limit. It uses a probe for measuring and collection of data involves touching the surface that has the probe.

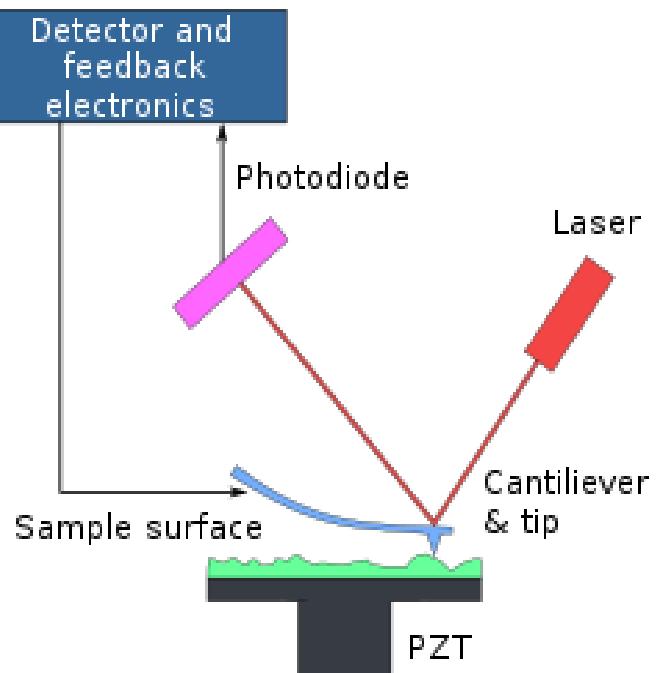
### 5.5.2. PRINCIPLE:

The Atomic Force Microscope works on the principle measuring intermolecular forces and sees atoms by using probed surfaces of the specimen in nanoscale.

### 5.5.3. CONSTRUCTION:

There are three major working principle that includes Surface sensing, Detection, and Imaging.

- An AFM uses a cantilever with a very sharp tip to scan over a sample surface. As the tip approaches the surface, the close-range, attractive force between the surface and the tip cause the cantilever to deflect towards the surface. However, as the cantilever is brought even closer to the surface, such that the tip makes contact with it, increasingly repulsive force takes over and causes the cantilever to deflect away from the surface.
- A laser beam is used to detect cantilever deflections towards or away from the surface. By reflecting an incident beam off the flat top of the cantilever, any cantilever deflection will cause slight changes in the direction of the reflected beam. A position-sensitive photo diode (PSPD) can be used to track these changes. Thus, if an AFM tip passes over a raised surface feature, the resulting cantilever deflection (and the subsequent change in direction of reflected beam) is recorded by the PSPD.
- An AFM images the topography of a sample surface by scanning the cantilever over a region of interest. The raised and lowered features on the sample surface influence the deflection of the cantilever, which is monitored by the PSPD. By using a feedback loop to control the height of the tip above the surface—thus maintaining constant laser position—the AFM can generate an accurate topographic map of the surface features.



### 5.5.4. WORKING:

Atomic Force Microscopes have several techniques for measuring force interactions such as van der Waals, thermal, electrical and magnetic force interactions for these interactions done by the AFM, it has the following parts that assist in controlling its functions.

- Modified tips which are used to detect the sample surface and undergo deflections.
- Software adjustments used to image the samples.
- **Feedback loop control** - they control the force interactions and the tip positions using a laser deflector. The laser reflects from the back of the cantilever and the tip and while the tip interacts with the surface of the sample,



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the laser's position on the photodetector is used in the feedback loop for tracking the surface of the sample and measurement.

- **Deflection** - The Atomic Force Microscope is constructed with a laser beam deflection system. The laser is reflected from the back of the AFM lever to the sensitive detector. They are made from silicon compounds with a tip radius of about 10nm.
- **Force measurement**- the AFM works and depends highly on the force interactions; they contribute to the image produced. The forces are measured by calculation of the deflection lever when the stiffness of the cantilever is known. This calculation is defined by Hooke's law, defined as follows:  $F = -kz$ , where F is the force, k is the stiffness of the lever, and z is the distance the lever is bent.

## 5.6. X-RAY PHOTOELECTRON SPECTROSCOPY (XPS):

### 5.6.1. INTRODUCTION:

The technique of X-Ray photoelectron Spectroscopy is also known as Electron Spectroscopy for chemical analysis. X-ray photoelectron Spectroscopy is a type of electron Spectroscopy it is an analytical technique to study the electronic structure and it is dynamic in atoms and molecules.

XPS was invented by Kai SIEGBAHN 1954 and received Nobel Prize in 1981. In x-ray photoelectron spectroscopy, primary beam x-ray Photon, which are irradiated on sample surface, the secondary beam (electron) obtained is then analyzed. The Secondary beam is made up of electrons. The spectrum of X-Ray photoelectron spectroscopy consists plot of number of electrons or power of electron as a function of energy i.e., kinetic energy or binding energy.

### 5.6.2. PRINCIPLE:

Due to the bombardment of X-Ray Photon on the sample surface K and L electron are ejected which are further analysed by the analyser. Let us consider  $E_b, E_b'$  and  $E_b''$  are binding energy of lower energy levels inner core orbitals. Where  $E_v, E_v'$  and  $E_v''$  are the energies of the valence shell electron.

The monochromatic X-ray Photon incident on the sample surface cell electron abstract the energy from this x-ray Photon and get ejected in terms of electron. Kinetic energy of the ejected electron is recorded by spectrometer and is given by

$$E_K = h\nu - E_b - \phi$$

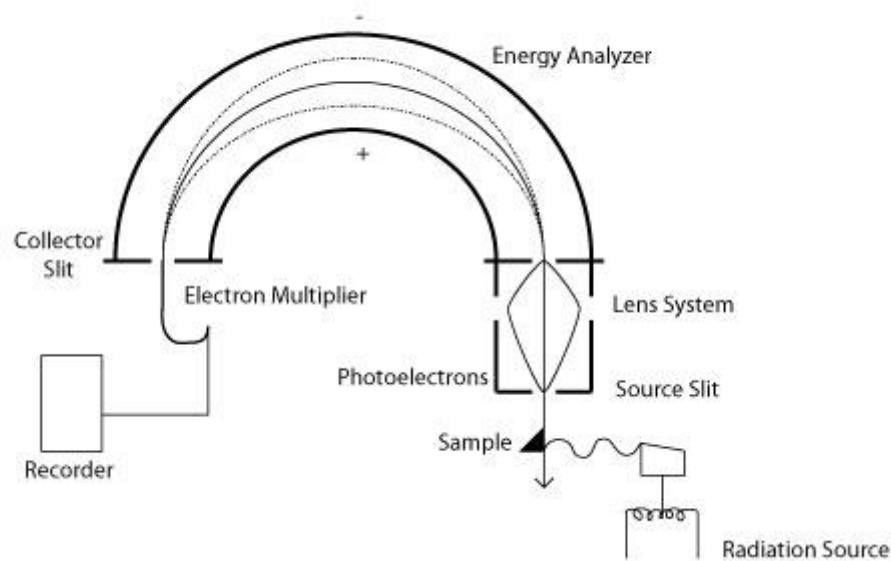
Where,  $E_K$  is kinetic energy of the ejected electron

$h\nu$ - energy associated with incident Photon

$E_b$  binding energy ejected electron

$\phi$ -Work function of the instrument

### 5.6.3. CONSTRUCTION:



The electron spectrometer made up of following components.

- Source
- Sample Holder
- Analyser
- Detector
- Processor And The Read-Out

#### SOURCE:

The simple x-ray Photon source for x-ray photoelectron spectra is an x-ray tube equipped with magnesium or aluminium metal target. Monochromator crystal can also provide having bandwidth of 0.3 electron volt. Much smaller spots on a surface to be examined.

#### SAMPLE HOLDER:

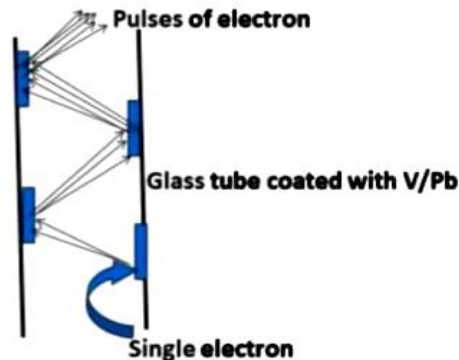
Sample holder is located in between the source and the entrance slit of spectrometer. Crystal disperser selects the photon of known energy from the source and incident on the sample. The area inside the sample holder should be evacuated within  $10^{-5}$  Torr. Pressure to avoid contamination of the surface sample. The gaseous sample is introduced into a sample compartment through a slit, to provide a pressure of  $10^{-1}$  torr. If the pressure is higher than attenuation of electron beam may take place, weaker signal may be obtained.

#### ANALYSER:

It is hemispherical in shape with very high electrostatic field is applied on analyser. Pressure maintained inside the analyser is  $10^{-5}$  torr. When the electron enters, into the hemispherical analyser, it travels in curved path and radius of curvature depends upon magnitude of field and kinetic energy of the electron.

**DETECTOR:** The electron channel multiplier tube or transducer are required of X-Ray photoelectron Spectroscopy. When single electron pass through Glass tube

coated with V/Pb the electron multiplier tube it is converted into number of electrons are pulses of electrons.



### **SIGNAL PROCESSOR AND READ OUT:**

The function of signal processor is to amplify the signal and read out device converts signal into spectrum.

### **APPLICATION OF XPS.**

- ✓ Identification of active sites
- ✓ .Determination of surface contamination on semiconductors
- ✓ .Study of oxide layers on metals
- ✓ Analysis of dust on the sample
- ✓ Determination of Oxidation State all the elements of periodic table can be determined or identified except hydrogen and Helium, as they don't emit inner core electron.

## **5.7. SCANNING ELECTRON MICROSCOPE (SEM)**

Scanning electron microscope is an improved model of an electron microscope. SEM is used to study the three dimensional image of the specimen.

### **5.7.1 PRINCIPLE:**

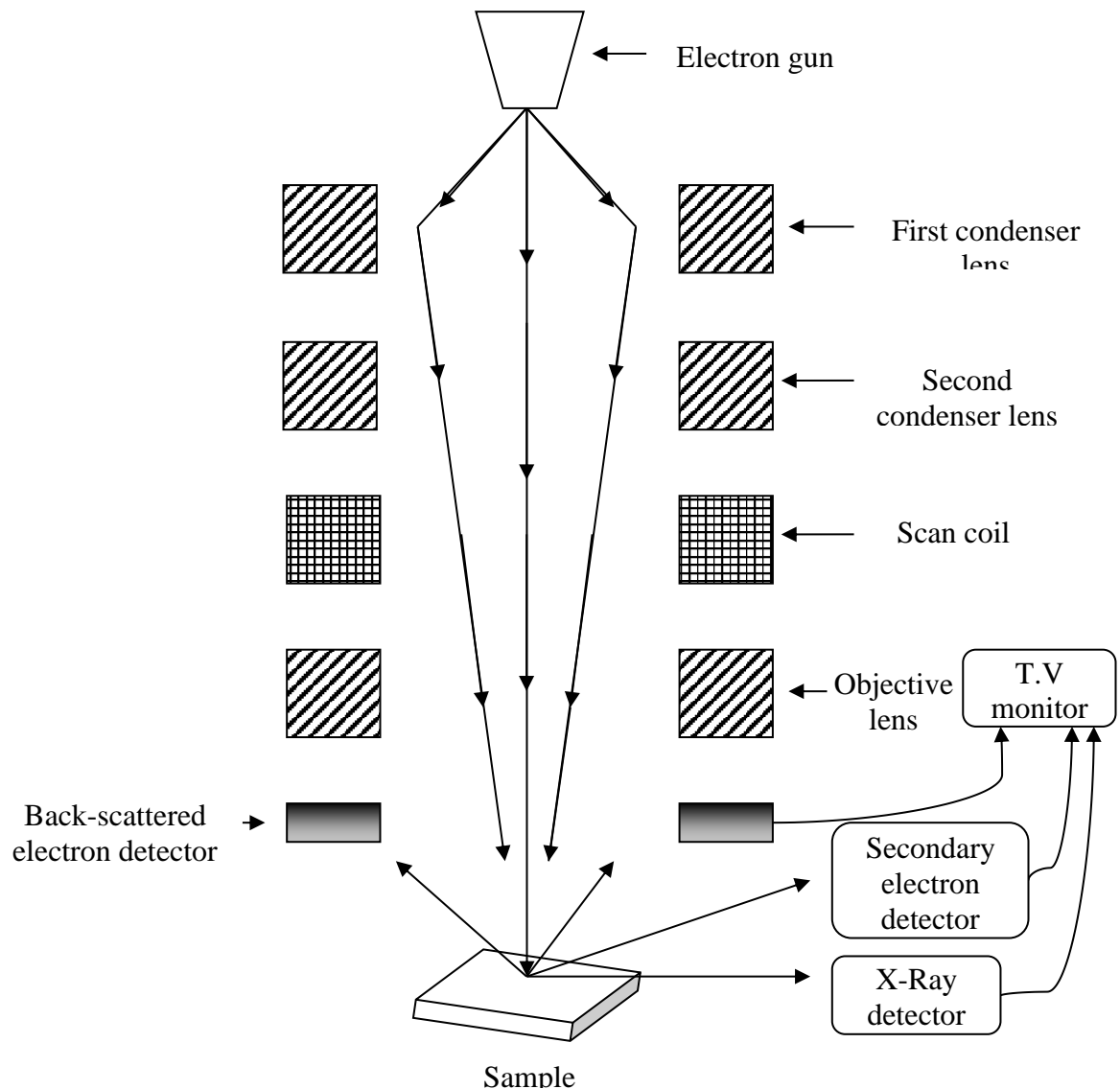
When the accelerated primary electrons strikes the sample, it produces secondary electron. These secondary electrons are collected by A positively charged electron detector which in turn gives a three dimensional image of the sample.

### **5.7.2. CONSTRUCTION:**

An electron gun is used to produce high energy electrons. Two magnetic lenses are used as condenser lenses to convert the diverging electron beam into a fine beam of spot diameter of the order of a few nanometers. A scanning coil is used to detect the electron beam to scan the sample. The objective lens is used to focus the scanning beam on a desired spot on the sample. The intensities of secondary electron, back-scattered electrons and the X-rays are recorded using detectors. The images are then displayed on TV monitors.

**5.7.3. WORKING:** when the high energy electron beam strikes the sample, some electrons are scattered due to elastic scattering (the back-scattered electrons),

some electrons are knocked off from the surface (the secondary electrons) and some electrons penetrate deep into the inner shells of the sample atoms to knock off inner shell electrons due to which characteristic X-rays are produced. These are detected using detectors and the signals are amplified and displayed on a TV monitor. Samples are required to be conducting. Non-conducting samples are coated with a thin conducting material.



#### 5.7.4. APPLICATIONS:

- ◆ SEM gives information about the surface features of the sample with resolution of the order of a few nanometers. This information can be used to study properties like reflectivity and roughness.
- ◆ SEM images give information about the elements and compounds in the sample and their relative abundance. This is used to study properties like hardness and melting point.
- ◆ SEM is used to study biological specimens like pollen grains.
- ◆ SEM is used to study the corroded layers on metal surfaces.

## 5.8. TRANSMISSION ELECTRON MICROSCOPE (TEM):

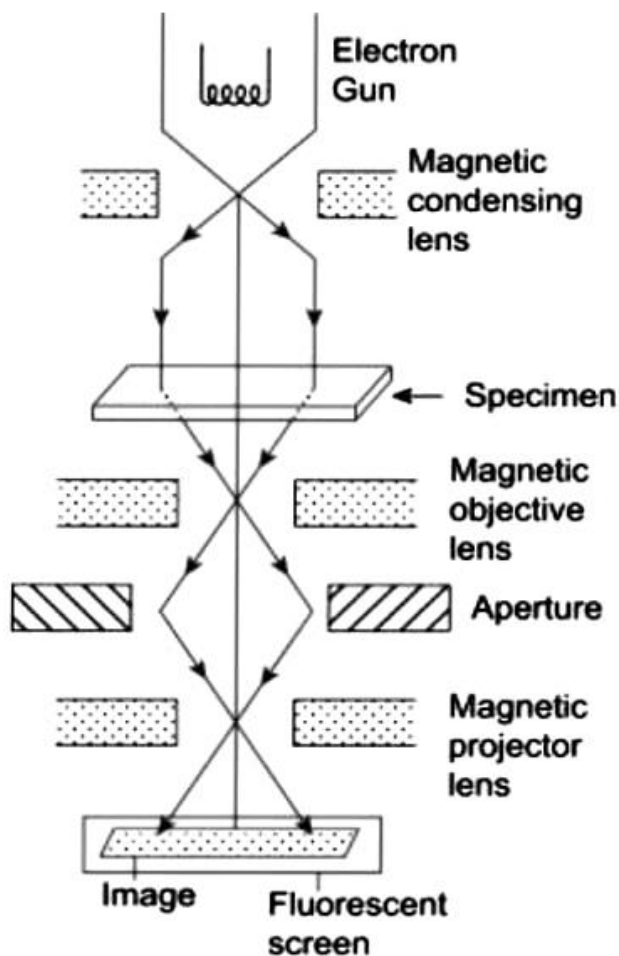
### 5.8.1. INTRODUCTION:

We know in scanning electron microscope the resolution of the image is limited only upto 10 to 20 nm. This will not be useful to view the internal features of an atom or the morphology of a sample of size  $0.2\text{nm}$ . To examine the sample of size of 2 to NM the transmission electron microscope can be used. In this microscope, the image is obtained by transmitting the electron through the specimen.

### 5.8.1. PRINCIPLE:

Electrons are made to pass through the specimen and the image is formed in the fluorescent screen there by using transmitted Beam (bright field image) or by using the diffracted beam (dark field image).

### 5.8.2. CONSTRUCTION:



It consists of an electron gun to produce electron. Magnetic condensing lens is used to condense the electron and is used to adjust the size of the electron that fall onto the specimen. The specimen is placed in between the condensing lens and objective lens as shown in figure. The magnetic objective lens is used to block the high angle diffracted beam the aperture is used to eliminate the diffracted beam and in turn, it increases the contrast of the image.

The magnetic projector lens is placed above the fluorescent screen in order to achieve high magnification. Can be recorded by using a fluorescent screen CCD charge coupled devices also.

**5.8.3. WORKING:**

Stream of electrons is produced by the electron and is made to fall over the specimen using magnetic condensing lens. Based on the angle of incidence the Beam is partly transmitted and partly diffracted as shown in figure. Both the Magnetic ERL Condensing lens transmitted Beam and the diffracted beams are recombined at the E-WALLED SPHERE of reflection, which encloses all possible reflections from the Crystal are specimen satisfying the Bragg's law image as shown in figure. The combined image is called the phase contrast image.

In order to increase the intensity and the contrast of the image and amplitude contrast image has to be obtained for stop this can be achieved only by using the transmitting beam and does the diffracted beam has to be eliminated. Now in order to eliminate the diffracted beam that beam is passed through the magnetic objective lens and the aperture is shown in figure adjusted in such a way that the diffracted image is illuminated. Thus, the final image being alone is passed through the projector lens for further magnification. Final image is recorded in the fluorescent screen or CCD this high contrast image is called Bright Field image. In addition, it has to be noted that the bright field image obtained is purely due to the elastic scattering non no energy change that is due to the transmitted beam alone

**5.8.4. APPLICATION OF TEM:**

- ◆ Transmission Electron Microscope is ideal for a number of different fields such as life sciences, nanotechnology, medical, biological and material research, forensic analysis, gemology and metallurgy as well as industry and education.
- ◆ TEMs provide topographical, morphological, compositional and crystalline information.
- ◆ The images allow researchers to view samples on a molecular level, making it possible to analyze structure and texture.
- ◆ This information is useful in the study of crystals and metals, but also has industrial applications.
- ◆ TEMs can be used in semiconductor analysis and production and the manufacturing of computer and silicon chips.

**PROBLEMS**

1. Determine the wave length of X-rays for crystal size of  $1.188 \times 10^{-6}$  m, peak width is  $0.5^\circ$  and peak position  $30^\circ$ , for a cubic crystal. Given Scherrer's constant  $k=0.92$ .

$$D = 1.188 \times 10^{-6}$$

$$\beta = 0.5$$

$$K=0.92$$

$$\theta = 30$$

Scherrer's equation is

$$D = \frac{K\lambda}{\beta \sin \theta}$$



$$\lambda = \frac{\beta \sin \theta D}{K} = \frac{0.5 \times \sin 30^\circ \times 1.188 \times 10^{-6}}{0.92} = 1.0288 \times 10^{-6} \text{ m}$$

2. Determine the crystallite size given the Wavelength of X-Rays 10 nm, the Peak Width 0.5 ° and peak position 25 ° for a cubic crystal given K = 0.94.

$$\lambda = 10 \text{ nm}$$

$$\beta = 0.5$$

$$K = 0.94$$

$$\theta = 25^\circ$$

Scherrer's equation is

$$D = \frac{K\lambda}{\beta \sin \theta}$$

$$D = \frac{0.94 \times 10 \times 10^{-9}}{0.5 \times \sin 25^\circ} = 207 \times 10^{-6} \text{ m}$$

3. An X-ray beam of wavelength 0.7 Å undergoes minimum order Bragg reflection from the plane (3 0 2) of cubic crystal at glancing angle 35°.

Calculate the lattice constant.

By Bragg's law,

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

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

$$(h \ k \ l) = (3 \ 0 \ 2)$$

$$\therefore d = \frac{a}{\sqrt{3^2 + 0^2 + 2^2}}$$

$$\theta = 35^\circ$$

$$n = 1$$

$$\lambda = 0.7 \text{ Å}$$

$$\therefore 2 \times \frac{a}{\sqrt{3^2 + 0^2 + 2^2}} \sin 35^\circ$$

$$= 1 \times 0.7$$

$$\therefore a = 2.2 \text{ Å}$$

4. Calculate the glancing angle of the (110) plane of a simple cubic crystal ( $a = 2.814 \text{ Å}$ ) corresponding to second order diffraction maximum for the x-ray of wavelength 0.710 Å.

By Bragg's law,

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

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

$$a = 2.814 \text{ \AA}$$

$$(h \ k \ l) = (1 \ 1 \ 0)$$

$$\therefore d = \frac{2.814}{\sqrt{1^2 + 1^2 + 0^2}} = 1.99 \text{ \AA}$$

$$n = 2; \lambda = 0.710 \text{ \AA}$$

$$\therefore 2 \times 1.99 \sin \theta = 2 \times 0.710$$

$$\therefore \theta = 20.9^\circ$$