ENGINEERING PHYSICS

MODULE – 5

Material Characterization Techniques and Instrumentation: **8 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), X-ray Photoelectron Spectroscopy(XPS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) Numerical problems.

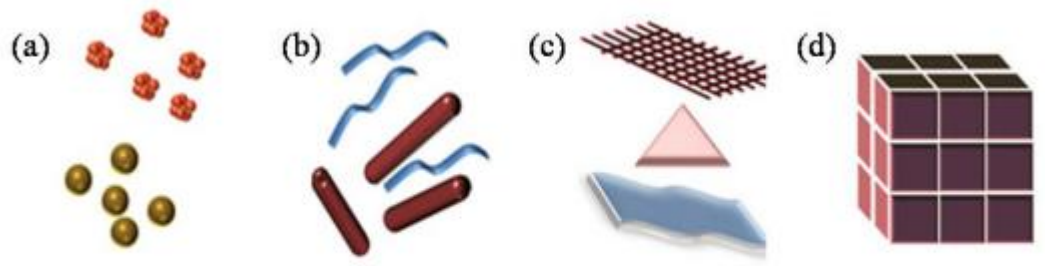
Nanomaterials

Materials having at least one dimension less than approximately 100 nanometers are classified as nanomaterials.

Nanomaterials are of increased interest because of the unique optical, magnetic, electrical, and other properties at this scale. These emergent properties have the potential for great impacts in electronics, medicine, and other fields.

Engineered nanomaterials are resources designed at the molecular (nanometre) level to take advantage of their small size and novel properties which are generally not seen in their conventional, bulk counterparts. The two main reasons why materials at the nano scale can have different properties are increased relative surface area and new quantum effects. Nanomaterials have a much greater surface area to volume ratio than their conventional forms, which can lead to greater chemical reactivity and affect their strength. Also at the nano scale, quantum effects can become much more important in determining the materials properties and characteristics, leading to novel optical, electrical and magnetic behaviours.

Nanomaterials have extremely small size which having at least one dimension 100 nm or less. Nanomaterials can be nanoscale in one dimension (eg. surface films), two dimensions (eg. strands or fibres), or three dimensions (eg. 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



Classification of Nanomaterials (a) 0D spheres and clusters, (b) 1D nanofibers, wires, and rods, (c) 2D films, plates, and networks, (d) 3D nanomaterials.

Fig. 5.1

CLASSIFICATION OF NANOMATERIALS

(i) Zero-dimensional nanomaterials: Here, all dimensions (x, y, z) are at nanoscale, i.e., no dimensions are greater than 100 nm. It includes nanospheres and nanoclusters.

(ii) One-dimensional nanomaterials: Here, two dimensions (x, y) are at nanoscale and the other is outside the nanoscale. This leads to needle shaped nanomaterials. It includes nanofibres, nanotubes, nanorods, and nanowires.

(iii) **Two-dimensional nanomaterials:** Here, one dimension (x) is at nanoscale and the other two are outside the nanoscale. The 2D nanomaterials exhibit platelike shapes. It includes nanofilms, nanolayers and nanocoatings with nanometre thickness.

(iv) **Three-dimensional nanomaterials:** These are the nanomaterials that are not confined to the nanoscale in any dimension. These materials have three arbitrary dimensions above 100 nm. The bulk (3D) nanomaterials are composed of a multiple arrangement of nanosize crystals in different orientations. It includes dispersions of nanoparticles, bundles of nanowires and nanotubes as well as multinanolayers (polycrystals) in which the 0D, 1D and 2D structural elements are in close contact with each other and form interfaces.

Nanocomposite

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 nanometre range to design and create new materials with unprecedented flexibility and improvement in their physical properties.

The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite will differ markedly from that of the component materials. Size limits for these effects have been proposed:

1. <5 nm for catalytic activity
2. <20 nm for making a hard magnetic material soft
3. <50 nm for refractive index changes
4. <100 nm for achieving super paramagnetism, mechanical strengthening or restricting matrix dislocation movement

In mechanical terms, nanocomposites differ from conventional composite materials due to the exceptionally high surface to volume ratio of the reinforcing phase or the aspect ratio (the ratio of its sizes in different dimensions). The reinforcing material can be made up of particles (e.g. minerals), sheets (e.g. exfoliated clay stacks) or fibres (e.g. carbon nanotubes or electrospun fibres). The area of the interface between the matrix and reinforcement phase(s) is typically an order of magnitude greater than for conventional composite materials. The matrix material properties are significantly affected in the vicinity of the reinforcement.

Ceramic-matrix nanocomposites

Ceramic matrix composites (CMCs) consist of ceramic fibers embedded in a ceramic matrix. The matrix and fibers can consist of any ceramic material, including carbon and carbon fibers. The ceramic occupying most of the volume is often from the group of oxides, such as nitrides, borides, silicides, whereas the second component is often a metal. Ideally both components are finely dispersed in each other in order to elicit particular optical, electrical and magnetic properties as well as tribological, corrosion-resistance and other protective properties.

Metal-matrix nanocomposites

Metal matrix nanocomposites can also be defined as reinforced metal matrix composites. This type of composites can be classified as continuous and non-continuous reinforced materials. One of the more important nanocomposites is Carbon nanotube metal matrix composites, which is an emerging new material that is being developed to take advantage of the high tensile strength and electrical conductivity of carbon nanotube materials. (eg.carbon nanotube metal

matrix composites, boron nitride reinforced metal matrix composites and carbon nitride metal matrix composites).

Polymer-matrix nanocomposites

In the simplest case, appropriately adding nanoparticulates to a polymer matrix can enhance its performance, often dramatically, by simply capitalizing on the nature and properties of the nanoscale filler (nanofilled polymer composites). This strategy is particularly effective in yielding high performance composites, when uniform dispersion of the filler is achieved and the properties of the nanoscale filler are substantially different or better than those of the matrix. Nanoparticles such as graphene, carbon nanotubes, molybdenum disulfide and tungsten disulfide are being used as reinforcing agents to fabricate mechanically strong biodegradable polymeric nanocomposites for bone tissue engineering applications.

Magnetic nanocomposites

Magnetic nanocomposites can be utilized in a vast number of applications, including catalytic, medical, and technical. For example, palladium is a common transition metal used in catalysis reactions. Magnetic nanoparticle-supported palladium complexes can be used in catalysis to increase the efficiency of the palladium in the reaction.

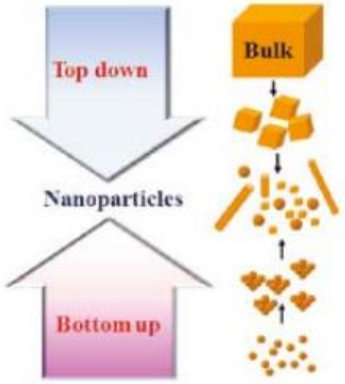
Magnetic nanocomposites can also be utilized in the medical field, with magnetic nanorods embedded in a polymer matrix can aid in more precise drug delivery and release.

Heat resistant nanocomposites

In the recent years nanocomposites have been designed to withstand high temperatures by the addition of Carbon Dots (CDs) in the polymer matrix. Such nanocomposites can be utilized in environments wherein high temperature resistance is a prime criterion

Nanomaterial - synthesis and processing

A nanometer is a billionth of a meter. Nanomaterials can be prepared by both the ‘bottom up’ or the ‘top down’ approaches. i.e. either to assemble atoms together or to dis-assemble (break, or dissociate) bulk solids into finer pieces until they are constituted of only a few atoms.



preparative methods of nanoparticles.

Fig. 5.2 Preparative methods of nanomaterials

Examples:

1. Mechanical grinding

Mechanical grinding or milling is a top down method and is typically achieved using high energy planetary ball. The energy transferred to the powder from steel balls depends on the rotational (vibrational) speed, size and number of the balls, ratio of the ball to powder mass, the time of milling and the milling atmosphere. Nanoparticles are produced by the shear action during grinding

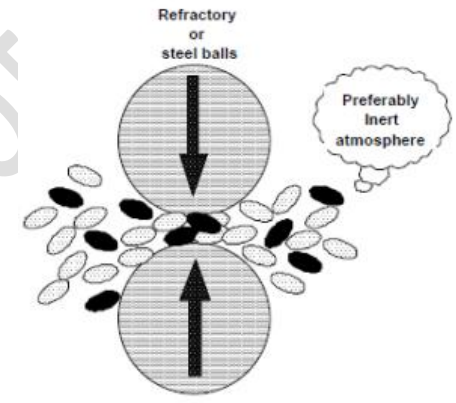


Fig. 5.3 Mechanical grinding

2. Physical Vapour Deposition

The simplest bottom up method to produce nanoparticles is by heating the desired material in a heat resistant crucible containing the desired material. This method is appropriate only for materials that have a high vapour pressure at the heated temperatures that can be as high as

2000°C. The atoms are evaporated into an atmosphere, which is either inert (e.g. He) or reactive (so as to form a compound). The hot atoms of the evaporated matter lose energy by collision with the atoms of the cold gas and undergo condensation into small clusters via homogeneous nucleation. And are collected on a cold plate.

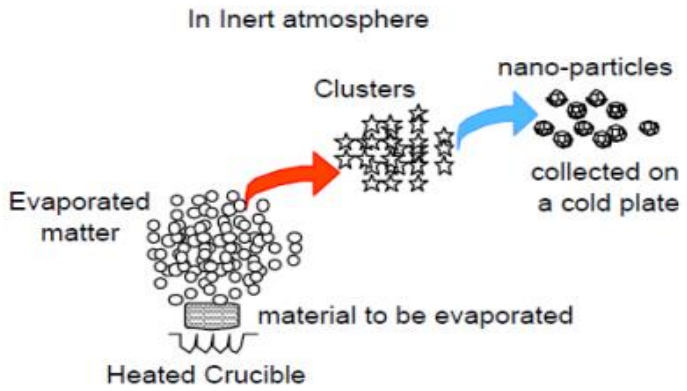


Fig. 5.4 Physical Vapour Deposition

Bragg's law

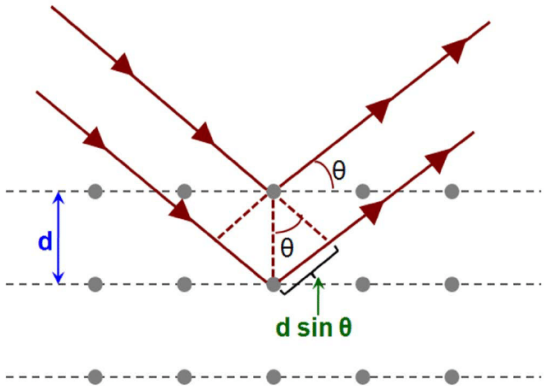


Fig. 5.5 Bragg's law of diffraction

Suppose, and X-ray bean is incident on a solid, making an angle θ with the planes of the atoms. These X-rays are diffracted by different atoms and the diffracted rays interfere. In certain directions, the interference is constructive and we obtain strong reflected X-rays. The analysis shows that there will be a strong reflected X-ray beam only if

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

where n is an integer. this equation is known as Bragg's law.

Braggs spectrometer

Braggs spectrometer used to determine the wavelength of X rays is shown in Fig. Braggs spectrometer is similar in construction to an ordinary optical spectrometer.

Xrays from an X-ray tube are made to pass through two fine slits S1 and S2 which collimate it into a fine pencil. This fine X-ray beam is then made to fall upon the crystal C (usually sodium chlo- ride crystal) mounted on the spectrometer table. This table is capable of rotation about a vertical axis and its rotation can be read on a circular graduated scale S. The reflected beam after passing through the slits S3 and S4 enters the ionization chamber. The X-rays entering the ionization chamber ionize the gas which causes a current to flow between the electrodes and the current can be measured by galvanometer G. The ionization current is a measure of the intensity of X-rays reflected by the crystal.

The ionization current is measured for different values of glancing angle . A graph is drawn between the glancing angle and ionization current.

For certain values of glancing angle,the ionization current increases abruptly. The first peak corresponds to first order, the second peak to second order and so on. From the graph, the glancing angles for different orders of reflection can be measured. Knowing the angle and the spacing d for the crystal, wavelength of Xrays can be determined.

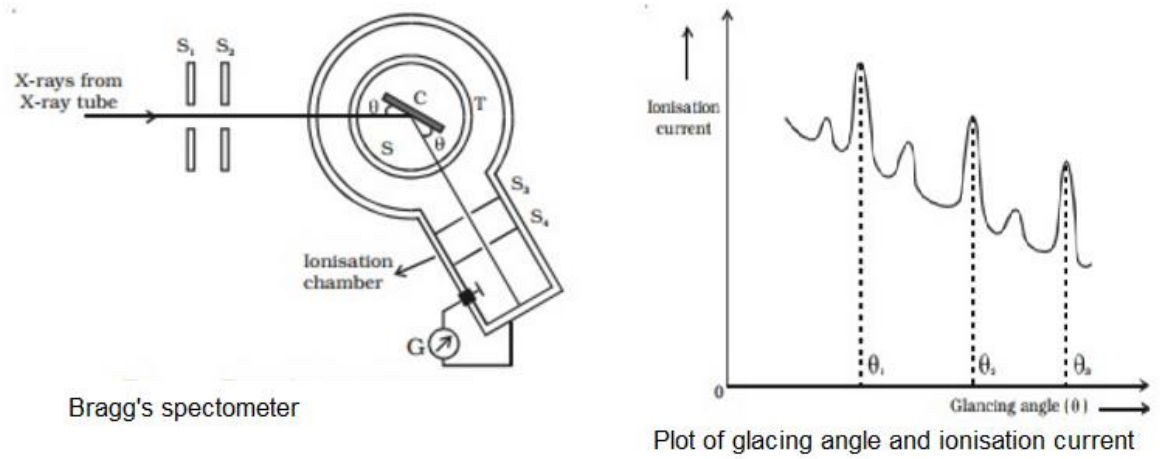


Fig. 5.7 (a) Bragg's Spectrometer (b) Plot of the glancing angle and ionization current

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. XRD works by irradiating a material with incident X-

rays and then measuring the intensities and scattering angles of the X-rays that leave the material

Scherrer formula

$t = 0.9 \lambda / B \cos \theta_B$ is known as the Scherrer formula

where: t- partical size

B- Full width Half Maxima (FWHM)

θ_B- Bragg's angle.

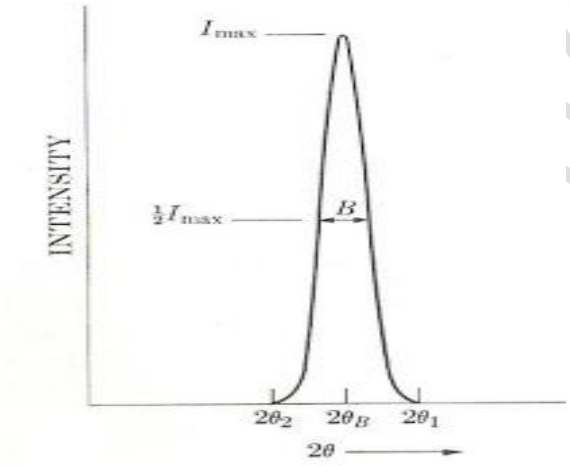


Fig. 5.8 Intensity vs. theta plot

It is used to estimate the particle size of very small crystals from the measured width of their diffraction curves

Atomic Force Microscopy (AFM)

Atomic force microscopy or scanning force microscopy (SFM) is a very-high-resolution type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction-limit. AFM consists of microscope cantilever with a sharp tip (probe) at its end used to scan the specimen surface. Instead of using an electrical signal, the AFM relies on forces between the atom on the tip and in the sample. The force present in the tip is kept constant and the scanning is done.

AFM Working Principle

The AFM principle is based on the cantilever/tip assembly that interacts with the sample; this assembly is also commonly referred to as the probe. The AFM probe interacts with the substrate

through a raster scanning motion. The up/down and side to side motion of the AFM tip as it scans along the surface is monitored through a laser beam reflected off the cantilever. This reflected laser beam is tracked by a position sensitive photo-detector (PSPD) that picks up the vertical and lateral motion of the probe. The deflection sensitivity of these detectors has to be calibrated in terms of how many nanometers of motion correspond to a unit of voltage measured on the detector.

The detector of AFM measures the deflection (displacement with respect to the equilibrium position) of the cantilever and converts it into an electrical signal. The intensity of this signal will be proportional to the displacement of the cantilever.

Applications

The AFM has been applied to problems in a wide range of disciplines of the natural sciences, including solid-state physics, semiconductor science and technology, molecular engineering, polymer chemistry and physics, surface chemistry, molecular biology, cell biology, and medicine.

Applications in the field of solid state physics include

(a) the identification of atoms at a surface,

(b) the evaluation of interactions between a specific atom and its neighboring atoms, and

(c) the study of changes in physical properties arising from changes in an atomic arrangement through atomic manipulation.

In molecular biology, AFM can be used to study the structure and mechanical properties of protein complexes and assemblies. For example, AFM has been used to image microtubules and measure their stiffness.

In cellular biology, AFM can be used to attempt to distinguish cancer cells and normal cells based on a hardness of cells, and to evaluate interactions between a specific cell and its neighboring cells in a competitive culture system. AFM can also be used to indent cells, to study how they regulate the stiffness or shape of the cell membrane or wall.

X-ray Photoelectron Spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA), is used to determine quantitative atomic composition and chemistry. A sample is irradiated with monochromatic x-rays, resulting in the emission of photoelectrons whose energies are characteristic of the elements within the sampling volume. An XPS spectra is created by plotting the number of electrons verses their binding energy.

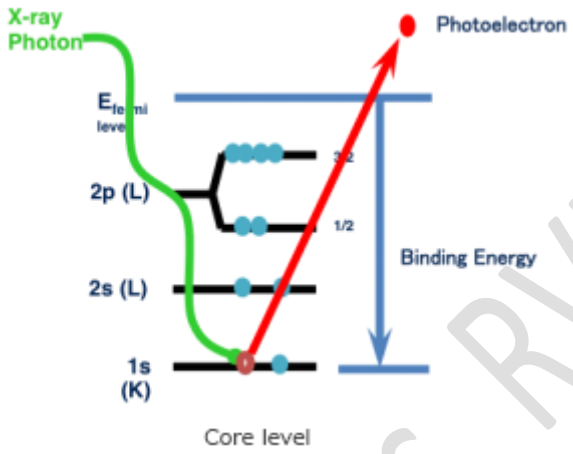


Fig. 5.9 Emission of photoelectrons

Principle: Based on Einstein's photoelectric effect. Many materials emit electrons when light shines upon them and ore electrons with kinetic energy Ek are ejected by incident X-rays.

$$\mathbf{E_K = h\nu - E_b - \varphi_{sp}}$$

E_K = kinetic energy

E_b = binding energy

h = Planck's constant

ν = frequency of X-rays

φ_{sp} = spectrometer work function

Construction: The logical components of an XPS instrument are shown below.

X-rays illuminate an area of a sample causing electrons to be ejected with a range of energies and directions. The electron optics, which may be a set of electrostatic and/or magnetic lens units, collect a proportion of these emitted electrons defined by those rays that can be transferred through the apertures and focused onto the analyzer entrance slit. Electrostatic fields within the hemispherical analyzer (HSA) are established to only allow electrons of a given energy (the so-called Pass Energy PE) to arrive at the detector slits and onto the detectors themselves.

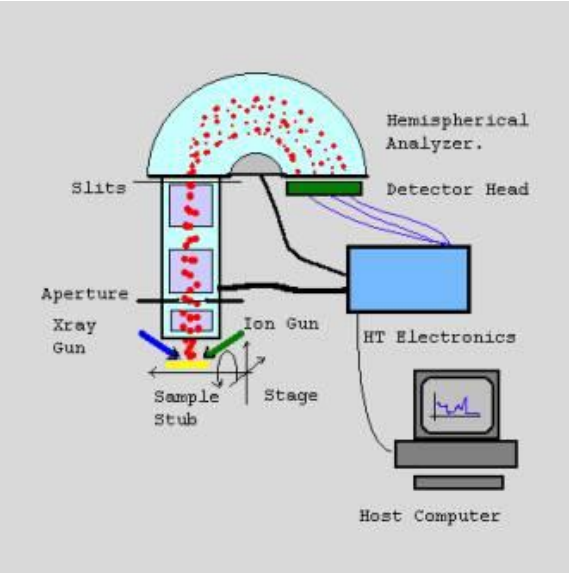


Fig. 5.10 Components of an XPS instrument

Electrons of a specific initial kinetic energy are measured by setting voltages for the lens system that both focus onto the entrance slit the electrons of the required initial energy and retards their velocity so that their kinetic energy after passing through the transfer lenses matches the pass energy of the hemispherical analyzer. To record a spectrum over a range of initial excitation energies it is necessary to scan the voltages applied to these transfer lenses and the prescription for these lens voltages is known as the set of lens functions. These lens functions are typically stored in some configuration file used by the acquisition system.

A hemispherical analyzer and transfer lenses can be operated in two modes, namely, Fixed Analyzer Transmission (FAT), also known as Constant Analyzer Energy (CAE), or Fix Retard Ratio (FRR) also known as Constant Retard Ratio (CRR). In FAT mode, the pass energy of the analyzer is held at a constant value and it is entirely the job of the transfer lens system to retard the given kinetic energy channel to the range accepted by the analyzer. Most XPS spectra are acquired using FAT.

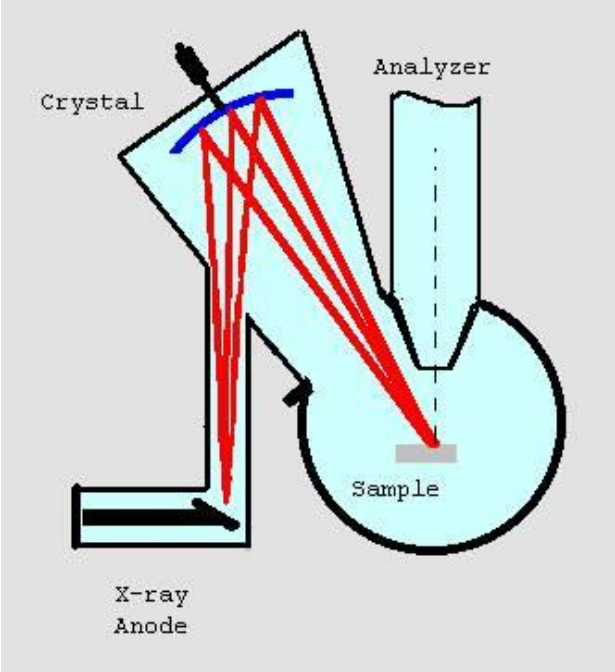


Fig. 5.11 Hemispherical Analyser of XPS

Monochromator

Energy Resolution

A number of factors influence the energy resolution achieved within a spectrum. The diameter of the analyzer, the pass energy and the spread of energies in the X-ray source play a major role in determining the full width half maximum (FWHM) for a given photoelectric line. Sample dependent considerations are also important where localized charging may broaden lines regardless of the precision built into the instrument and therefore effective charge neutralization is an important part of any system.

Small Area Analysis

Electrons are dispersed through the hemispherical analyzer so that different energy electrons arrive at different positions in the radial direction; further, they are also spatially dispersed around the circumference of the sphere. This relationship has been exploited by the Scienta ESCA300 and SPECS delay-line-detector systems, where images can be recorded that show both energy dispersion and spatial information in the form of sets of line-scans. VG ESCALABS 220i instruments use additional lenses at the entrance slit and before an imaging detector, which perform a Fourier transform and the inverse operation to allow an energy-resolved stigmatic image to be recorded through a hemispherical analyzer whilst operating in deflection mode.

Working:

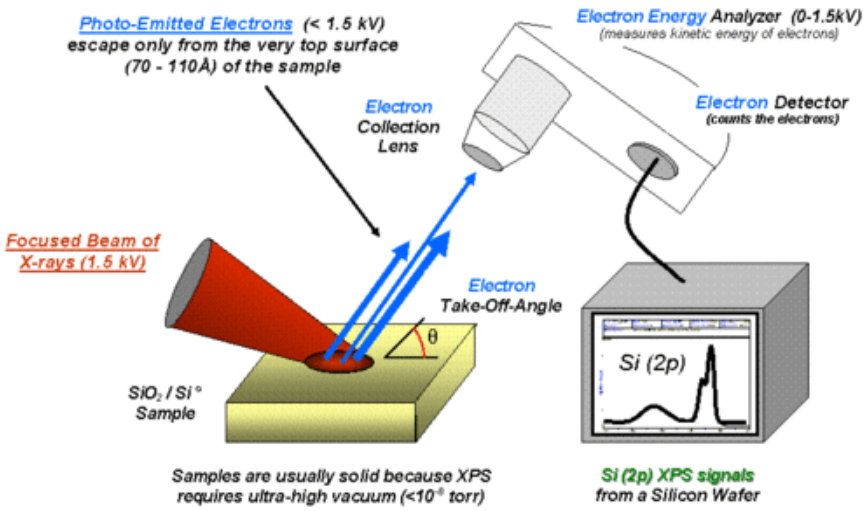


Fig. 5.12 Working of an XPS

X-rays (photons) are shot onto a sample, and when electrons in the sample absorb enough energy, they are ejected from the sample with a certain kinetic energy. The energy of those ejected electrons is analyzed by a detector and a plot of these energies and relative numbers of electrons is produced. Electrons of different energies follow different paths through the detector which allows the computer to differentiate the electrons and produce the spectra seen below.

Atoms present in compound being tested by XPS are determined according to the equation:

$$E_{\text{binding}} = E_{\text{photon}} - (E_{\text{kinetic}} + \phi)$$

Here, binding energy is the energy of an electron attracted to a nucleus; photon energy is the energy of X-ray photons being used by the spectrometer, and the kinetic energy is the energy of the ejected electrons from the sample. The work function is a correction factor for the instrument and correlates to the minimum energy required to eject an electron from an atom. The work function and photon energy are known and the kinetic energy is measured by the detector. That leaves the binding energy as the only unknown, which can then be determined. As electrons are in orbitals farther from the nucleus, less energy is required to eject them, so the binding energy is lower for higher orbitals. Electrons contained in different subshells (s,p,d, etc.) have different energies as well. By showing the energy of electrons emitted from a material, XPS allows for the composition of a material to be determined.

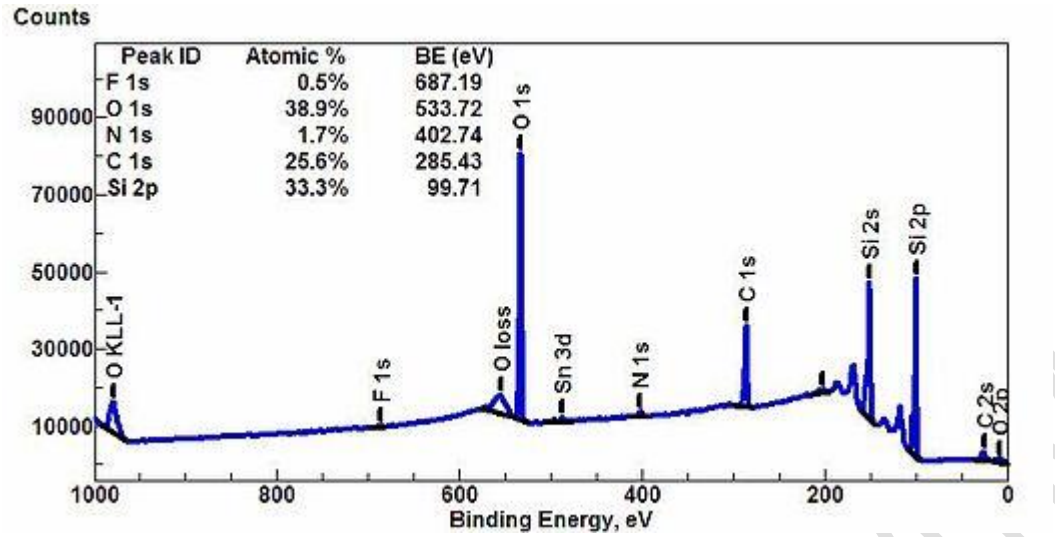


Fig. 5.13 Plot of Counts vs Binding Energy of an XPS ouput

Additionally, chemical shifts can be determined using XPS. This is owing to the fact that binding energy doesn't only depend on the shell of the electron. It also depends on the environment, that is, the bonds that the atom in question partake in. Therefore, a primary carbon would have a slightly different binding energy than a carboxyl carbon, for example.

Applications:

XPS is useful for investigating almost all surface problems. XPS data can be used to solve problems with existing surface interactions, or to investigate new materials. It is used for **biological** applications like in the analysis of a Wound Dressing and the characterization of Chemical Gradients and Antibody Immobilization. It is used in surface Chemical-State Analysis of catalyst. It is used in Defect Analysis and to analyze Contact Lens Samples. It is used in the surface analysis of oxides, Polymers ad in the field of microelectronics.

Scanning Electron Microscopy (SEM)

Principle: A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern (raster scanning, is the rectangular pattern of image capture and reconstruction in television) , and the position of the beam is combined with the intensity of

the detected signal to produce an image. In the most common SEM mode, secondary electrons emitted by atoms excited by the electron beam are detected using a secondary electron detector. The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on specimen topography. Some SEMs can achieve resolutions better than 1 nanometer. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples. Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis is considered to be "non-destructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyse the same materials repeatedly.

Construction: Essential components of all SEMs include the following:

Electron Source ("Gun"): electron microscopes employ an electron beam for imaging. In SEM, two types of electrons are primarily detected:

- backscattered electrons (BSE),
- secondary electrons (SE),

Backscattered electrons are reflected back after elastic interactions between the beam and the sample. Secondary electrons, however, originate from the atoms of the sample: they are a result of inelastic interactions between the electron beam and the sample.

The electron beam travels through the electron column, which consists of a set of lenses that focus the beam onto the sample surface. Electron microscope lenses can be electrostatic or magnetic, depending on whether they use an electrostatic field or a magnetic field to focus the electron beam.

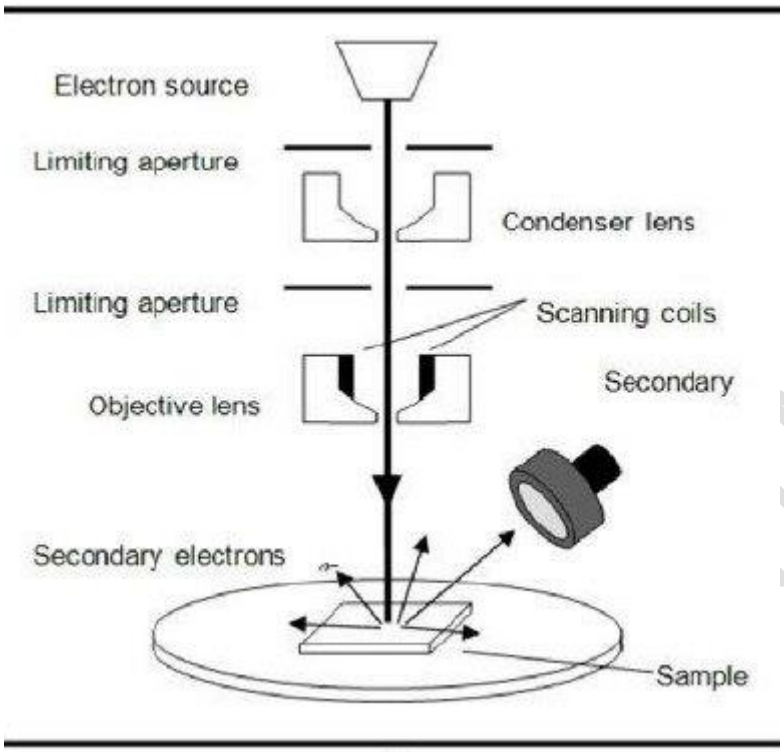


Fig. 5.14 SEM

Deflectors

Electrons are negatively charged particles and travel through the electron column at high energy and high speed. One way to deflect these particles is to let them travel through an electric field generated by two plates at potential +U and -U, as shown in Fig. 1a.

Under the influence of the electric field, the electron is deflected at an angle that depends on the electron energy, the electric field applied in between the plates, and the length of the plates. The faster, or the more energetic the electron, the smaller the deflection angle. The higher the electric field and the longer the plates, the bigger the deflection angle. A device consisting of two plates at different potential is called a deflector.

Electron Lenses:

Electrostatic lenses consist of metallic plates connected to high voltage with an aperture that the electron beam travels through. Single-aperture lenses consist of a single metallic plate at high voltage and can often be found in electron sources.

lens.

Magnetic lenses

Magnetic lenses use the Lorentz force, that is proportional to the electron charge and velocity, to deflect electrons. Magnetic lenses consist of a metallic body (called the ferromagnetic circuit) that ends with two pole pieces.

The magnetic field is given by a coil positioned at the top of the ferromagnetic circuit, as shown in Fig. 5.15. The strength of the lens can be altered by varying the magnetic field B. This is done by modifying the geometry of the pole piece, namely the distance between the pole pieces, and the current flowing into the coils (excitation).

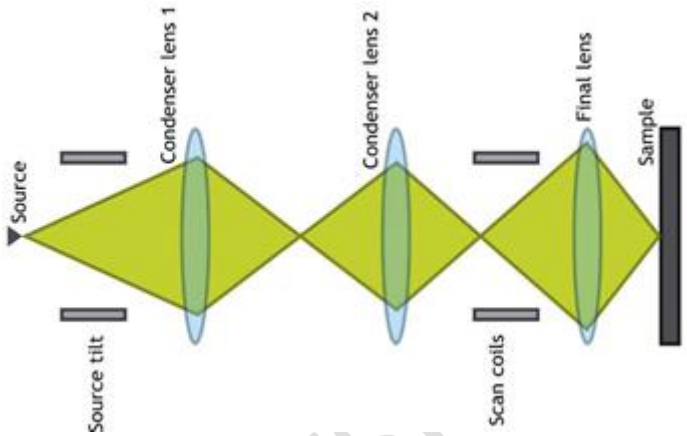


Fig. 5.15 Magnetic lens schematic.

SEMs always have at least one detector (usually a secondary electron detector), and most have additional detectors. The specific capabilities of a particular instrument are critically dependent on which detectors it accommodates.

Infrastructure Requirements:

Power Supply, Vacuum System, Cooling system, Vibration-free floor

Room free of ambient magnetic and electric fields

Working: At the heart of a scanning electron microscope is a high-energy electron source positioned above a series of condenser lenses and apertures which focus these electrons into a beam. The position of this beam is altered by sets of deflection or scanning coils before the final lens aperture. A sample is placed in the path of the electron beam which is continuously deflected into a raster scanning pattern by the deflection coils.

When electrons impact a surface, they generate secondary and backscattered electrons (BSE), as well as x-rays. BSE and x-ray detectors in the sample chamber acquire these signals, which are characteristic of the sample's elemental composition, morphology, and crystalline

structure. Scanning electron microscopy can subsequently be used for imaging the elemental composition of a sample surface and determining topographical sample features with a significantly increased resolving power.

Scanning electron microscopy can generate 3D chemical surface maps of a sample with a magnifying capacity of up to 50,000x. This offers high lateral resolution ranging from millimeters to nanometres (>10nm), while energy dispersive x-ray (EDX) analysis provides chemical detection limits of 1000 – 3000 parts per million (ppm).

Application: The SEM is routinely used to generate high-resolution images of shapes of objects (SEI) and to show spatial variations in chemical compositions: 1) acquiring elemental maps or spot chemical analyses using EDS, 2)discrimination of phases based on mean atomic number (commonly related to relative density) using BSE, and 3) compositional maps based on differences in trace element "activitors" (typically transition metal and Rare Earth elements) using CL. The SEM is also widely used to identify phases based on qualitative chemical analysis and/or crystalline structure. Precise measurement of very small features and objects down to 50 nm in size is also accomplished using the SEM. Backscattered electron images (BSE) can be used for rapid discrimination of phases in multiphase samples. SEMs equipped with diffracted backscattered electron detectors (EBSD) can be used to examine microfabric and crystallographic orientation in many materials

Scanning electron microscopy is a robust analytical tool with a broad range of practical applications in the commercial, analytical, and industrial spaces. It is broadly used for quality control (QC) and good-bad testing of pharmaceutical products and has proven useful for detecting and identifying unknown contaminants in manufactured goods.

Transmission Electron Microscopy (TEM)

Transmission electron microscopes (TEM) are microscopes that use a particle beam of electrons to visualize specimens and generate a highly-magnified image. TEMs can magnify objects up to 2 million times. In order to get a better idea of just how small that is, think of how small a cell is. It is no wonder TEMs have become so valuable within the biological and medical fields.

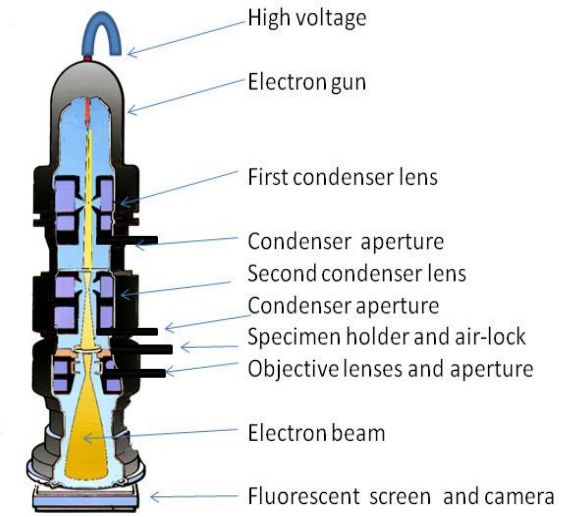
The transmission electron microscope is a very powerful tool for material science. A high energy beam of electrons is shone through a very thin sample, and the interactions between the

electrons and the atoms can be used to observe features such as the crystal structure and features in the structure like dislocations and grain boundaries. Chemical analysis can also be performed. TEM can be used to study the growth of layers, their composition and defects in semiconductors. High resolution can be used to analyze the quality, shape, size and density of quantum wells, wires and dots.

The TEM operates on the same basic principles as the light microscope but uses electrons instead of light. Because the wavelength of electrons is much smaller than that of light, the optimal resolution attainable for TEM images is many orders of magnitude better than that from a light microscope. Thus, TEMs can reveal the finest details of internal structure - in some cases as small as individual atoms.

Principle: TEMs employ a high voltage electron beam in order to create an image. An electron gun at the top of a TEM emits electrons that travel through the microscope's vacuum tube. Rather than having a glass lens focusing the light (as in the case of light microscopes), the TEM employs an electromagnetic lens which focuses the electrons into a very fine beam. This beam then passes through the specimen, which is very thin, and the electrons either scatter or hit a fluorescent screen at the bottom of the microscope. An image of the specimen with its assorted parts shown in different shades according to its density appears on the screen. This image can be then studied directly within the TEM or photographed. Below is shown a diagram of a TEM and its basic parts.

Construction:



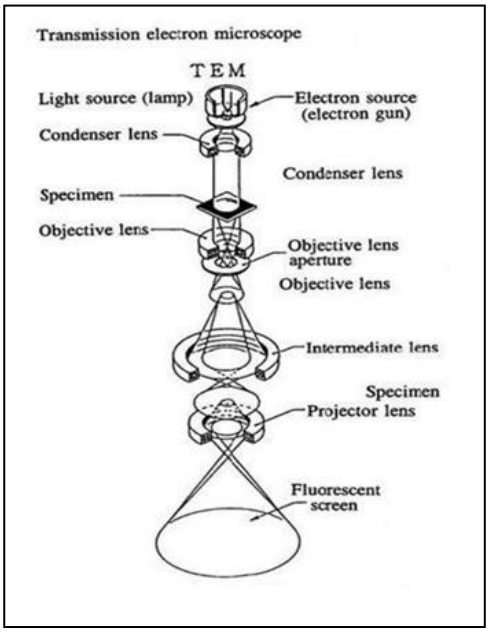


Fig. 5.16 Construction of TEM

Working:

In a transmission electron microscope, an electron gun, fires a beam of electrons. The gun accelerates the electrons to extremely high speeds using electromagnetic coils and voltages of up to several million volts.

The electron beam is focused into a thin, small beam by a condenser lens, which has a high aperture that eliminates high angle electrons. Having reached their highest speed, the electrons zoom through the ultra-thin specimen and parts of the beam are transmitted depending on how transparent the sample is to electrons.

The objective lens focuses the portion of the beam that is emitted from the sample into an image. Another component of the TEM is the vacuum system, which is essential to ensure electrons do not collide with gas atoms.

A low vacuum is first achieved using a either a rotary pump or diaphragm pumps which enable a low enough pressure for the operation of a diffusion pump, which then achieves vacuum level that is high enough for operations. High voltage TEMS require particularly high vacuum levels and a third vacuum system may be used.

The image produced by the TEM, called a micrograph, is seen through projection onto a screen that is phosphorescent. When irradiated by the electron beam, this screen emits photons. A film camera positioned underneath the screen can be used to capture the image or digital capture may be achieved with a charge-coupled device (CCD) camera.

Imaging

The beam of electrons from the electron gun is focused into a small, thin, coherent beam by the use of the condenser lens. This beam is restricted by the condenser aperture, which excludes high angle electrons. The beam then strikes the specimen and parts of it are transmitted depending upon the thickness and electron transparency of the specimen. This transmitted portion is focused by the objective lens into an image on phosphor screen or charge coupled device (CCD) camera. Optional objective apertures can be used to enhance the contrast by

blocking out high-angle diffracted electrons. The image then passed down the column through the intermediate and projector lenses, is enlarged all the way.

The image strikes the phosphor screen and light is generated, allowing the user to see the image. The darker areas of the image represent those areas of the sample that fewer electrons are transmitted through while the lighter areas of the image represent those areas of the sample that more electrons were transmitted through.

Typical accelerating voltages for a biological TEM range up to 125,000 Volts.

Abbe's equation: $d = 0.753/aV^{1/2}$
d = resolution in nm
a = half aperture angle
V = accelerating velocity
Resolution is defined as the distance at which two points or objects can be distinguished. Therefore as r approaches zero we say that the resolution is increased.

DeBroglie's formula:
$\lambda = h/mv$
h = Plank's constant
(6.626 x 10 −23 ergs/ sec)
m = mass of the electron
v = electron velocity
DeBroglie's formula states that if the accelerating voltage is increased, electron velocity will increase as will resolution.

Application: This technology can be used in various industries from medical research where it is employed to investigate viruses and bacteria, for example, to forensic science, gemology and materials science.

What Are the Differences Between a TEM and a Light Microscope?

Although TEMs and light microscopes operate on the same basic principles, there are several differences between the two. The main difference is that TEMs use electrons rather than light in order to magnify images. The power of the light microscope is limited by the wavelength of light and can magnify something up to 2,000 times. Electron microscopes, on the other hand, can produce much more highly magnified images because the beam of electrons has a smaller wavelength which creates images of higher resolution. (Resolution is the degree of sharpness of an image.) Figure 2 compares the magnification of a light microscope to that of a TEM.

QUESTION BANK:

1. Differentiate between nanomaterials and nanocomposites.
2. Explain any three types of nanocomposites.
3. Explain top down and bottom up processes of nanomaterial synthesis with examples.
4. What is Bragg's law?
5. Explain the construction and working of Bragg's spectrometer.
6. Write down the Scherrer formula and explain it.
7. What is the principle of Atomic Force Microscopy?
8. Explain the construction and working of AFM.
9. Explain the principle, construction and working of a FTIR spectroscope.
10. Explain the principle, construction and working of an X-ray Photoelectron Spectroscope.
11. Explain the principle, construction and working of a scanning electron microscope.
12. Explain the working principles of electron lens and magnetic lens.
13. Explain the principle, construction and working of a transmission electron microscope.
14. Explain the principle, construction and working of a Scanning tunnelling microscope.