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12. Characterization Techniques

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Abstract:

Characterization of materials is the basic and important aspect of nanotechnology. Various extra ordinary properties can be explored deeply by characterization techniques. The nanomaterials possess various features in terms of surface morphology, optical properties, chemical properties, mechanical properties etc.

These all properties are further studied in detail with the help of characterization techniques. Hence, this chapter gives emphasis on X- ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM). Photoluminescence (PL).

Keywords:

Nanomaterials, Characterization, Nanotechnology.

12.1 Introduction:

Nanomaterials are known to be prominent for various applications due to their extraordinary properties. Various Characterization techniques came forward for the detail investigation of these Nanomaterials, Which involves X-ray diffraction (XRD), Scanning electron microscope (SEM), Transmission Electron Microscopy (TEM), Photoluminescence (PL), etc.

These techniques are popular for identification of crystal structure, size, surface topography, purity and elemental composition etc. The Characterization techniques plays important role in the field of nanotechnology and material science.

12.2 X-Ray Diffraction (XRD):

Crystallographic structure and physical properties of materials can be reveal by XRD which is non-destructive technique which are in the form of powders and thin films. Wavelength of X-rays is of the order of an angstrom (Å = 10-10 m), which is falls in the range of inter planer distances.

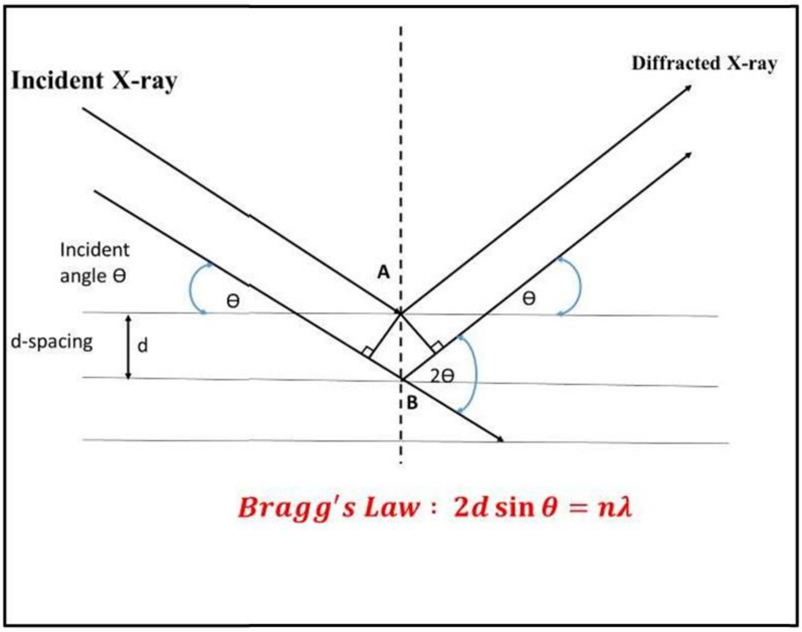

Figure 12.1: Schematic of X- Ray Diffraction Process

Hence, crystalline materials give response to X-rays and constructive diffraction pattern forms. Collective information is presented in the form of diffracted peaks of uneven intensities.

Bragg's law is necessary condition for X-ray diffraction,

$$2d\sin\theta = n\lambda \qquad (1)$$

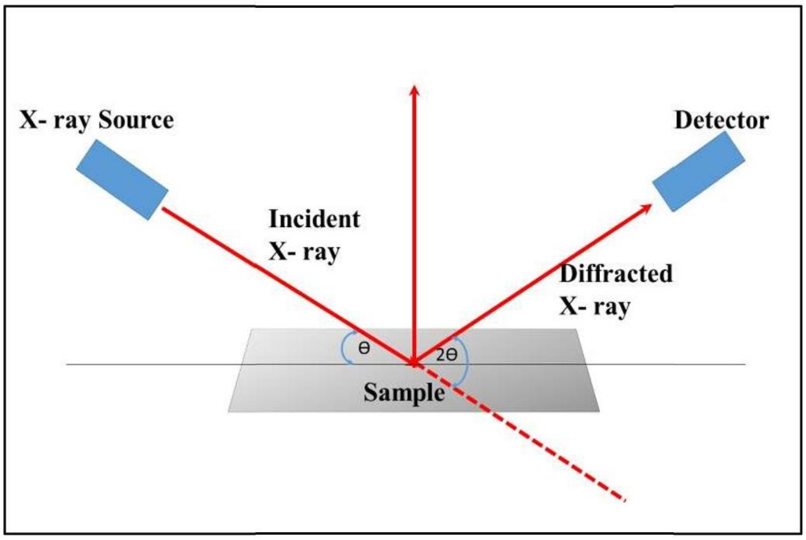

Figure 12.2: Schematic of X-Ray Diffraction Set-Up

The information such as type of crystal structure as well as inter atomic spacing associated to crystal can be withdrawn from XRD pattern. XRD is most popular for high accuracy and also it is absolute technique.

12.3 Scanning Electron Microscope (SEM):

In case of nanometric film the surface topography plays an important role. The information of surface topography can be directly achieved by SEM. Out of all possible interaction secondary electron is responsible for the image formation in SEM. The electron beam is scanned over the specimen. The SEM is useful for organic and inorganic specimens. In addition to surface topographic information SEM has additional facility of elemental mapping, which is useful for identification of various element.

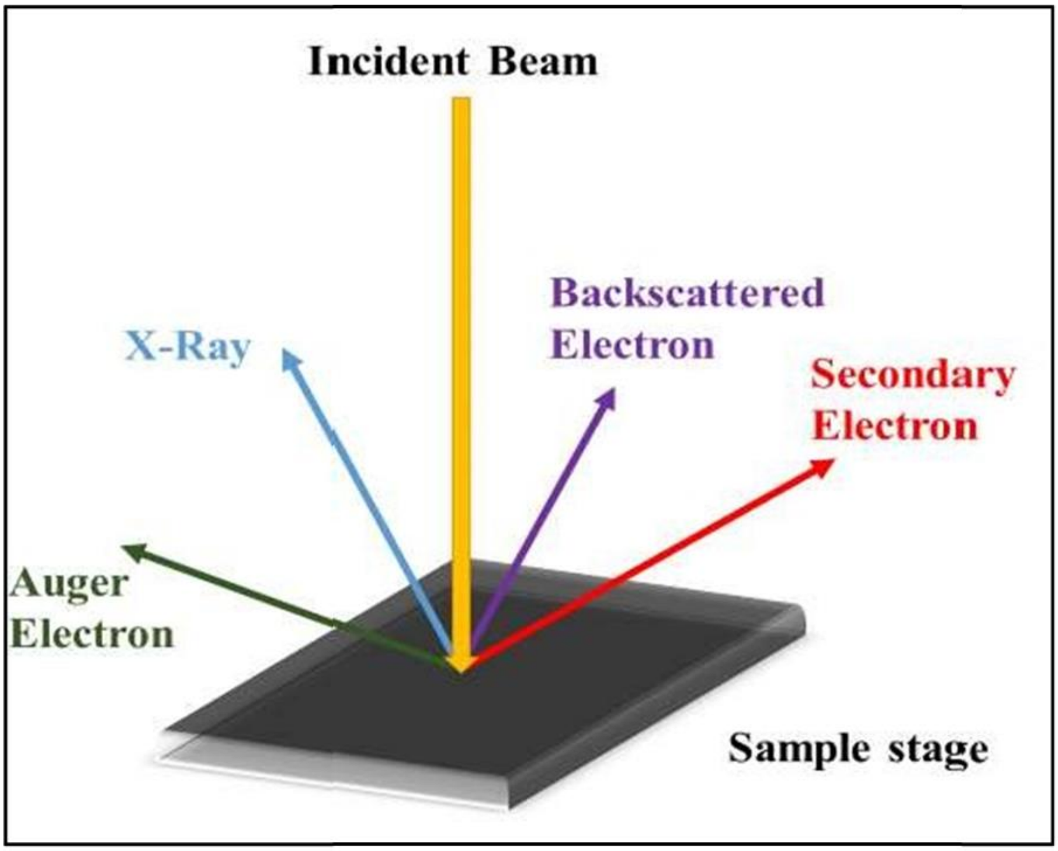

Figure 12.3: Schematic of Electron Interaction Processes

When primary electrons interact with specimen, various processes take place out of all secondary electron’s emission is quite important. The amplification of output signal is further carried out for the formation of clear and high-resolution image. Elemental mapping is quite important when we doped some element is host material. By analysing the energy associated to high energy electrons, elemental mapping is possible. All elements one can be detect in the elemental mapping except few.

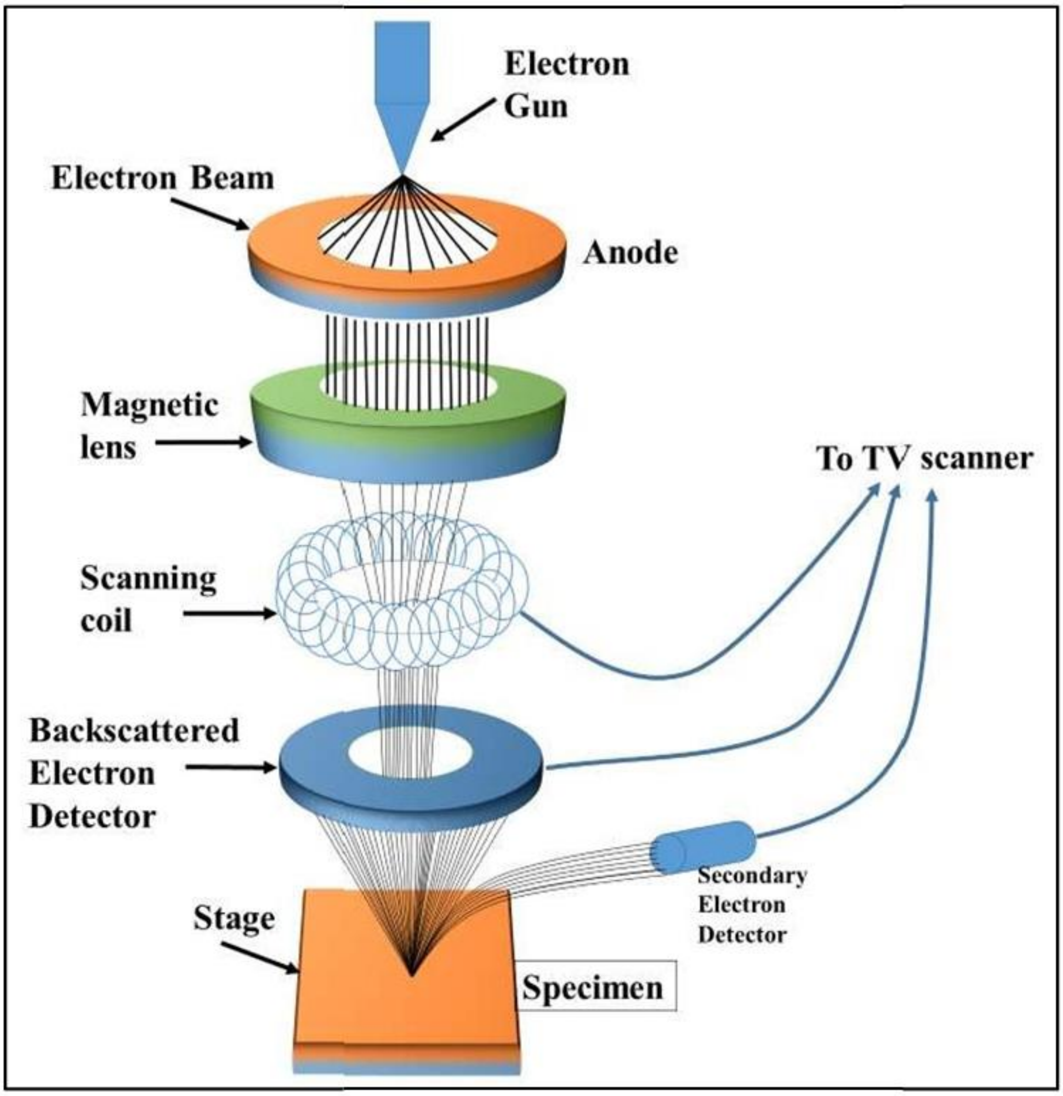

Figure 12.4: Schematic of Scanning Electron Microscopy (SEM) Technique

12.4 Transmission Electron Microscope (TEM):

The crystal structure, inter atomic spacing and defect in the crystalline materials can be obtained by one of the powerful technique such as (TEM). In addition, to physical properties of nanomaterials their crystal structures are also quite important which has most of hidden information.

Such information can be explored by TEM technique. Further, detects and crystallographic planes can be viewed by High resolution TEM (HRTEM) technique. Constructive interference pattern is the Key parameter for TEM [1].

High vacuum is minimum requirement of TEM. Voltage range of 40-200 kV is capable to accelerate the electron which are coming out from thermionic gun. The maximum thickness of the order of 0.1–0.3 µm for corresponding 100 keV is required [2]. Like optical microscope TEM has facility of various lenses. Because of these electromagnetic lenses coherent and parallel beam of electron is produced. The feature which defines angular range of electron is the aperture. The process of clear TEM image is depends combinations of lenses used.

12.4.1 Bright Field Imaging:

Elastic and inelastic scattering are responsible for change in energy spread as well as angular range. Stopping of unnecessarily scattered electron is done by aperture kept in the back focal plane (Figure. 12.5). Bright field image is captured in the absence of image if the aperture is placed at optical axis [1]. Dark portion will appear in image due to high thickness and high density of material/specimen.

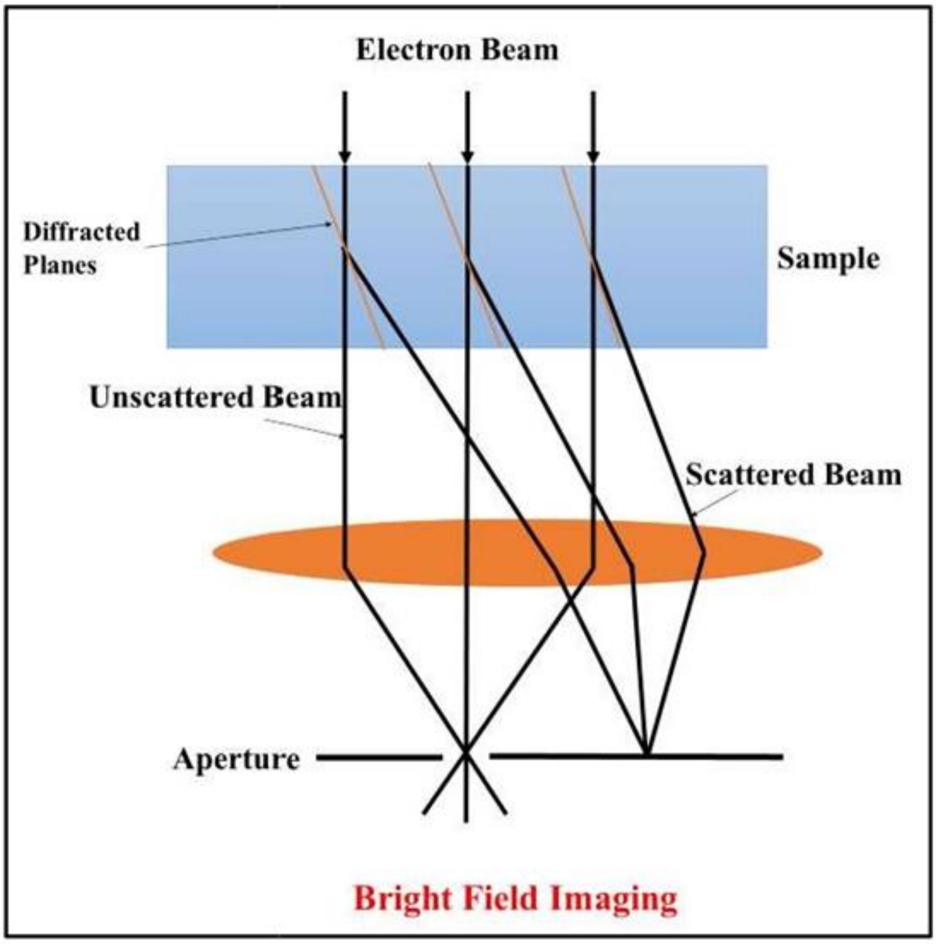

Figure 12.5: Bright Field Imaging Schematic View

12.4.2 Dark Field Imaging:

In case of dark field imaging aperture can be used for particular selection of diffracted beam. The TEM is known for generation of information in reciprocal space as well as in real space [1].

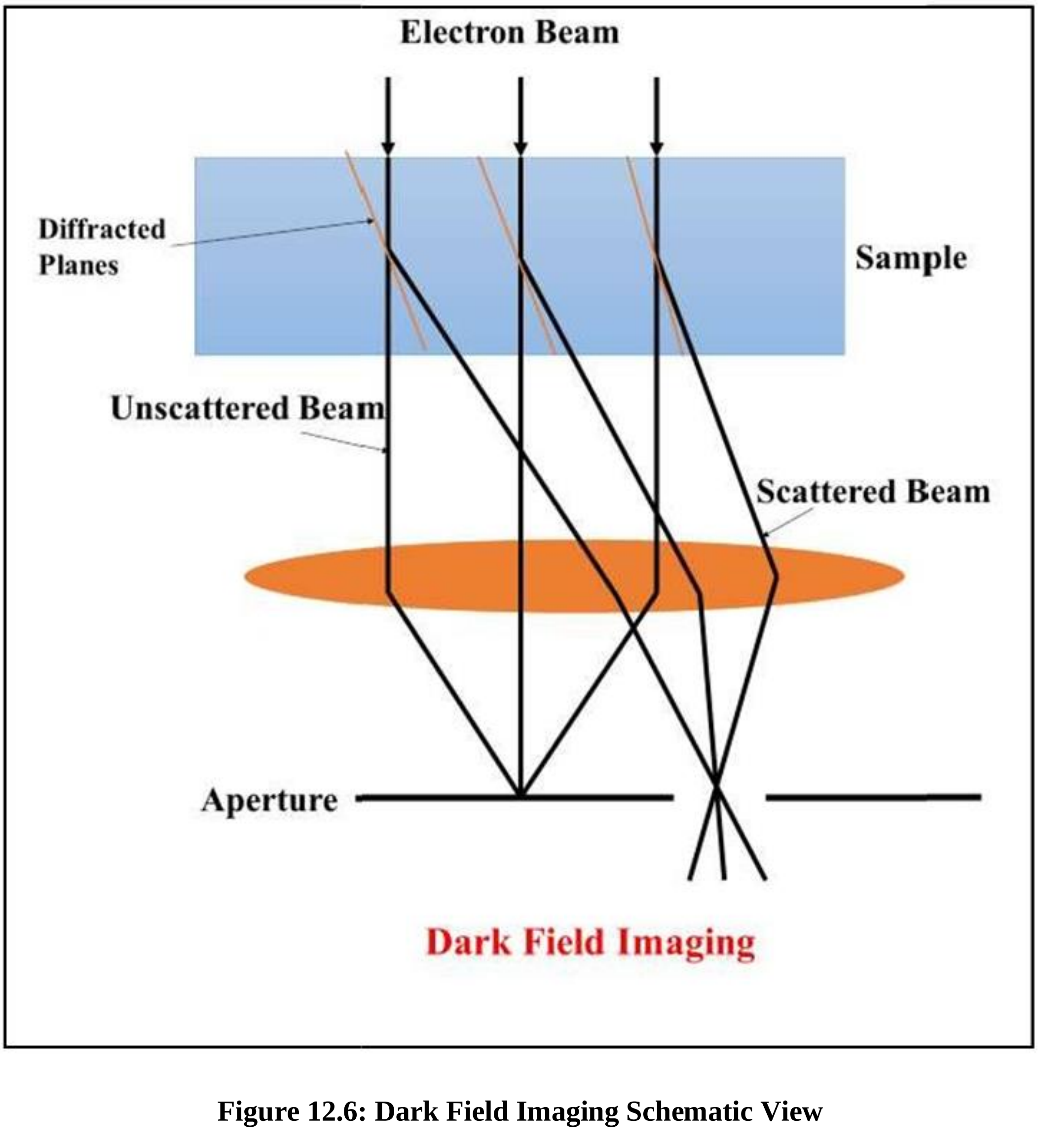

Figure 12.6: Dark Field Imaging Schematic View

12.5 Photoluminescence (PL):

As the various defects and impurity occurred in the nanometric film, the detection of such defects is quite important. PL spectroscopy has capability to withdraw above said information. The PL is known for non contact and non-destructive technique.

In typical process light beam interact with specimen and various processes take place (Figure 12.7). Upon the absorption of light electron excitation and recombination is takes place through various processes, as given below and also in Figure 12.7

a. Electron hole pair generation,
b. Recombination through defects states,
c. Recombination through band to band,
d. Emission of hole

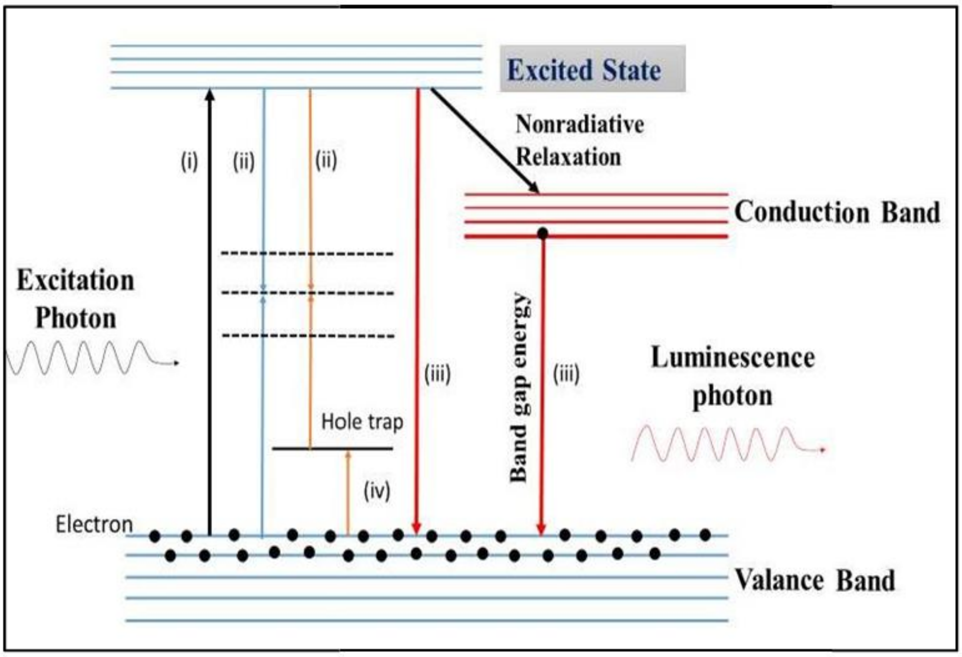

Figure 12.7: Schematic of Various Interactions in Photoluminescence Spectroscopy [3]

If the recombination follows laws of radiatively then one can identify defect or impurity in state. In this process energy levels lies near to conduction or valence band are dominantly take participation. Core levels are known for non-radiative recombination.

In detailed experimentation, Xenon (Xe) arc lamp is usually used. The wavelength range of Xe lamp is 300 to 1100 nm. A filter is usually used for the wavelength selection which further relates with the analysis of specimen.

12.6 References:

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