

# Characterization and Analysis: An Innovative Technique for Nanotechnology

K.G. Joshi<sup>1</sup>, S. B. Bajaj<sup>2</sup>, S.S. Ingle<sup>3</sup>

<sup>1</sup>(Research Scholar, Research center- Dept., of Physics, JES College, Jalna, India)

<sup>2</sup>(Department of Physics, JES College, Jalna, India)

<sup>3</sup>(Department of Mechanical Engineering, SRES, College of Engineering, Kopergaon, India)

**ABSTRACT :** Nanotechnology is a multidisciplinary area of applied science and Engineering that deals with design and manufacture of extremely small components of the system. The mankind has seen several technology revolutions in the past: industrial, agricultural, medical, and InfoTech in a course of two centuries. But through each of these, we have been able to exploit

only a small fraction of the total possibilities. We have been still dealing with the matter at a bigger scale. The building blocks, our engineering skills & products were bigger than the nano size and hence had limitations in manipulation. With its capacity to manipulate the smallest possible component of the matter, the Nanotechnology has the potential to bring that cycle of technological revolution to completion. As the nanomaterials are not new as for centuries, we are preparing & using nanoparticles but during that period we were lacking of the powerful instrument which measure size of particles & correlation of properties. Recently new techniques are evolved for characterization and analysis of nanomaterials which gives a revolution to development of Nanotechnology. This paper highlights some of the techniques used for analysis & characterization of nanomaterials.

**Keywords** - Nanomaterial, nanoscale, characterization, synthesis, diffraction etc

## I. INTRODUCTION

Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale size-dependent properties are often observed. Thus, the properties of materials change as their size approaches the nanoscale and as the percentage of atoms at the surface of a material becomes significant. In order to explore novel physical properties & phenomenon, real size potential applications of nanostructure & nanomaterial's, the ability to fabricate & process nanomaterials is the first corner stone in nanotechnology. There are various ways to fabricate nanomaterial's like physical methods, chemical methods, biological methods & hybrid methods such as vapor phase growth, liquid phase growth, solid state reaction, hybrid growth, high energy ball milling, sputtering, sol-gel, chemical bath deposition, molecular beam epitaxy. Nanotechnology is new but research on nanoscale is not new at all. The study of biological systems & the engineering of many materials such as colloidal dispersion, metallic quantum dots, and catalyst have been in nanometer regime for centuries. What has changed recently is an explosion in our ability to image, engineer and manipulate system in the nanometer scale. What is new about nanotechnology is the combination of our ability to see and manipulate matter on the nano scale and our understanding of atomic scale interaction [5]. Nanomaterials are characterized by various techniques. The technique to be used, depend upon type of material & information need to know. Usually first knowing the size, crystalline type, composition & then chemical state, etc & other optical magnetic properties are important. For analysis & characterization of nanomaterial recently various methods are introduced like microscope, diffraction techniques, spectroscopies & electric and magnetic measurements. Out of which diffraction methods gives average particle size of nanomaterial. It is bulk characterization method whereas methods microscopes like Scanning Electron Microscope, Transmission Electron Microscope, Scanning Probe Microscope gives the atomic level characterization of nanomaterial. Depending upon which type of information about the material is needed, analysis technique is selected. In this paper the some of the analysis techniques are reviewed [1][2].

## II. DIFFRACTION TECHNIQUES

Diffraction techniques are often used in average particle size analysis as well as structural determination. Some of the diffraction techniques are X-ray Diffraction (XRD), electron diffraction, small angle X-ray Scattering (SAXS), small angle Neutron scattering (SANS).

2.1 X-ray Diffraction (XRD)

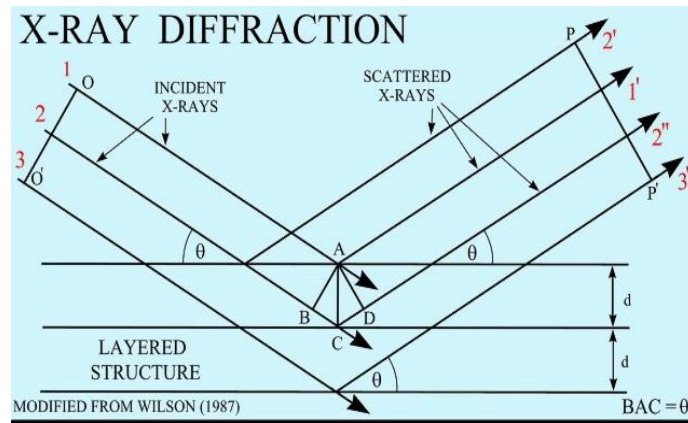


Fig.1 Bragg's Law

Crystalline materials give rise to the most obvious applications, but there is also important information to be obtained from semi crystalline and even amorphous materials. Crystallite size is a fundamental characteristic of nano-materials and microstructure is often a key to understanding and controlling bulk properties. From XRD we get the information like Phase identification and quantification, Crystal structure variations (e.g. by lattice parameters), Crystallite size and shape, and lattice distortion, Crystallinity, Non-crystalline periodicity and size, Orientation (crystalline and amorphous) etc.

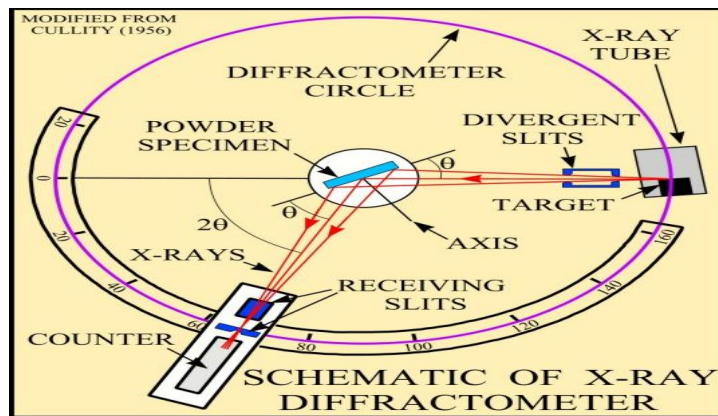


Fig.2 . Schematic of X-Ray Diffractometer

In an X-ray diffraction measurement, a crystal is mounted on a goniometer and gradually rotated while being bombarded with X-rays, producing a diffraction pattern of regularly spaced spots known as reflections. The two-dimensional images taken at different rotations are converted into a three-dimensional model of the density of electrons within the crystal using the mathematical method of Fourier transforms, combined with chemical data known for the sample. Poor resolution (fuzziness) or even errors may result if the crystals are too small, or not uniform enough in their internal makeup as shown in Fig. 2. Crystals are regular arrays of atoms, and X-rays can be considered waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms electrons as shown in fig1. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as elastic scattering, and the electron (or lighthouse) is known as the scatterer. A regular array of scatterers produces a regular array of spherical waves as shown in Fig. 1. Although these waves cancel one another out in most directions through destructive interference, they add constructively in a few specific directions, determined by Bragg's law:

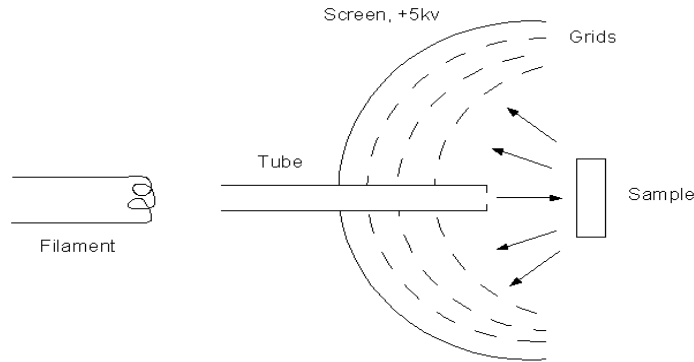
$$2d \sin \theta = n\lambda$$

Here  $d$  is the spacing between diffracting planes,  $\theta$  is the incident angle,  $n$  is any integer, and  $\lambda$  is the wavelength

of the beam. These specific directions appear as spots on the diffraction pattern called reflections. X-rays are used to produce the diffraction pattern because their wavelength  $\lambda$  is typically the same order of magnitude (1–100 angstroms) as the spacing  $d$  between planes in the crystal. [7, 6]

## 2.2 Electron diffraction

**Electron diffraction** refers to the wave nature of electrons as shown in Fig. 3. It is a technique used to study matter by firing electrons at a sample and observing the resulting interference pattern. Electron diffraction is most frequently used in solid state physics and chemistry to study the crystal structure of solids. Experiments are usually performed in a transmission electron microscope (TEM), or a scanning electron microscope (SEM) as electron backscatter diffraction. In these instruments, electrons are accelerated by an electrostatic potential in order to gain the desired energy and determine their wavelength before they interact with the sample to be studied.



**Fig.3 Electron Diffraction**

The periodic structure of a crystalline solid acts as a diffraction grating, scattering the electrons in a predictable manner. Working back from the observed diffraction pattern, it may be possible to deduce the structure of the crystal producing the diffraction pattern. Electron diffraction of solids is usually performed in a Transmission Electron Microscope (TEM) where the electrons pass through a thin film of the material to be studied. The resulting diffraction pattern is then observed on a fluorescent screen, recorded on photographic film, on imaging plates or using a CCD [4].

## 2.3 Neutron diffraction

**Neutron diffraction** or **elastic neutron scattering** as shown in Fig. 4 is the application of neutron scattering to the determination of the atomic or magnetic structure of a material. A sample to be examined is placed in a beam of thermal or cold neutrons to obtain a diffraction pattern that provides information of the structure of the material. The technique is similar to X-ray diffraction but due to their different scattering properties, neutrons and X-rays provide complementary information.

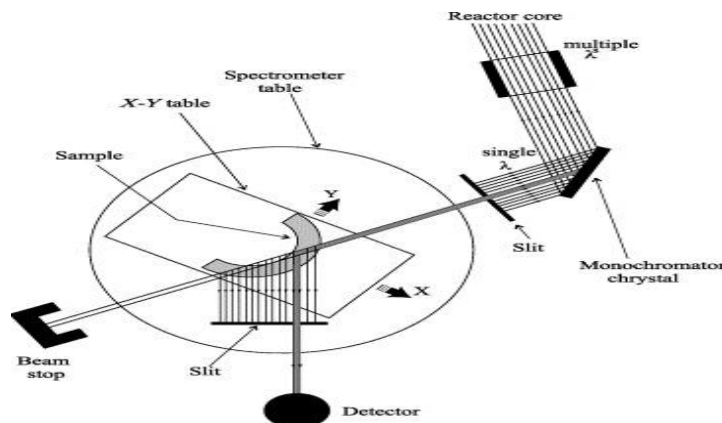


Fig. 4 Neutron Diffraction

The technique requires a source of neutrons. Neutrons are usually produced in a nuclear reactor or spallation source. At a research reactor, other components are needed, including a crystal monochromators as well as filters to select the desired neutron wavelength. Some parts of the setup may also be movable. The technique is most commonly performed as powder diffraction, which only requires a polycrystalline powder. For single crystal work, the crystals must be much larger than those used in X-ray crystallography. It is common to use crystals that are about  $1 \text{ mm}^3$ . When a beam of neutrons emanating from a reactor is slowed down and selected properly by their speed, their wavelength lies near one angstrom (0.1 nanometer), the typical separation between atoms in a solid material. Such a beam can then be used to perform a diffraction experiment. Impinging on a crystalline sample it will scatter under a limited number of well-defined angles according to the same Bragg's law that describes X-ray diffraction [8]. Neutron diffraction is an excellent method for structure determination, although it has been difficult to obtain intense, monochromatic beams of neutrons in sufficient quantities. Being uncharged, neutrons scatter much more readily from the atomic nuclei rather than from the electrons. Therefore, neutron scattering is very useful for observing the positions of light atoms with few electrons, especially hydrogen, which is essentially invisible in the X-ray diffraction.

## 2.4 Single-crystal X-ray diffraction

The oldest and most precise method of X-ray crystallography is single-crystal X-ray diffraction, in which a beam of X-rays strikes a single crystal, producing scattered beams. When they land on a piece of film or other detector, these beams make a *diffraction pattern* of spots; the strengths and angles of these beams are recorded as the crystal is gradually rotated. Each spot is called a *reflection*, since it corresponds to the reflection of the X-rays from one set of evenly spaced planes within the crystal. For single crystals of sufficient purity and regularity, X-ray diffraction data can determine the mean chemical bond lengths and angles to within a few thousandths of an angstrom and to within a few tenths of a degree respectively.

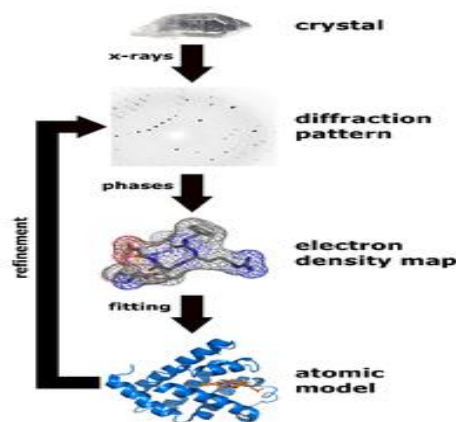


Fig.5 Schematic of Single Crystal X-Ray Diffraction

The technique of single-crystal X-ray crystallography as shown in Fig. 5 has three basic steps. The first—and often most difficult—step is to obtain an adequate crystal of the material under study. The crystal should be sufficiently large (about larger than 0.1 mm in all dimensions), pure in composition and regular in structure, with no significant internal imperfections such as cracks or twinning. In the second step, the crystal is placed in an intense beam of X-rays, usually of a single wavelength, producing the regular pattern of reflections. As the crystal is gradually rotated, previous reflections disappear and new ones appear; the intensity of every spot is recorded at every orientation of the crystal. Multiple data sets may have to be collected, with each set covering slightly more than half a full rotation of the crystal and typically containing tens of thousands of reflections. In the third step, these data are combined computationally with complementary chemical information to produce

and refine a model of the arrangement of atoms within the crystal. The final, refined model of the atomic arrangement called a *crystal structure* is usually stored in a public database [9].

## 2.5 Powder diffraction

**Powder diffraction** as shown in Fig. 6 is a scientific technique using X-ray, neutron, or electron diffraction on powder or microcrystalline samples for structural characterization of materials. Ideally, every possible crystalline orientation is represented very equally in a powdered sample. The resulting orientation averaging causes the three-dimensional reciprocal space that is studied in single crystal diffraction to be projected onto a single dimension. In practice, it is sometimes necessary to rotate the sample orientation to eliminate the effects of texturing and achieve true randomness.

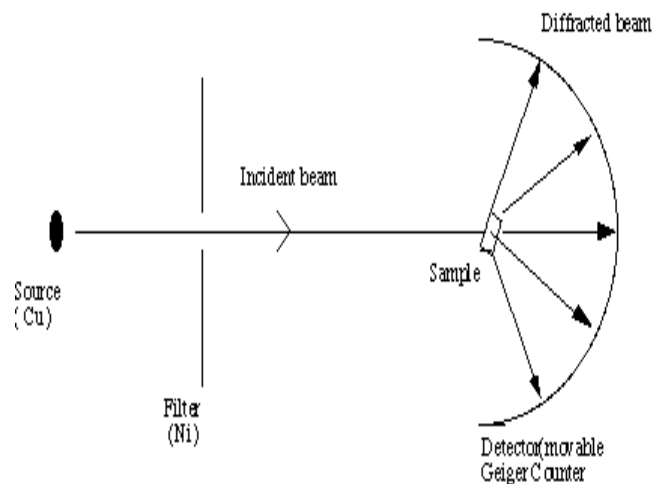


Fig. 6. Powder diffraction

When the scattered radiation is collected on a flat plate detector, the rotational averaging leads to smooth diffraction rings around the beam axis, rather than the discrete Laue spots observed in single crystal diffraction. The angle between the beam axis and the ring is called the scattering angle and in X-ray crystallography always denoted as  $2\theta$  (in scattering of *visible* light the convention is usually to call it  $\theta$ ). In accordance with Bragg's law, each ring corresponds to a particular reciprocal lattice vector  $G$  in the sample crystal. This leads to the definition of the scattering vector as:

$$G = q = 2k \sin(\theta) = \frac{4\pi}{\lambda} \sin(\theta).$$

Powder diffraction data are usually presented as a *diffraction pattern* in which the diffracted intensity  $I$  is shown as function either of the scattering angle  $2\theta$  or as a function of the scattering vector  $q$ . The latter variable has the advantage that the diffraction pattern no longer depends on the value of the wavelength  $\lambda$ . An instrument dedicated to performing powder measurements is called a **powder diffractometer** [10].

## III. MICROSCOPES

Microscopes are useful to investigate morphology, size structure & composition of solids depending upon the type of microscope. Combined with some other technique microscopes can give information about optical magnetic & other properties of nanomaterials some of the microscopes are scanning electron microscope (SEM), transmission electron microscope (TEM), Scanning probe microscopy (SPM) [3].

## IV. SPECTROSCOPE



Spectroscopes are useful for chemical state analysis, electronic structure & other properties of material. UV VIS IR absorption, Fourier transform Infra-Red (FTIR), Atomic Absorption spectroscopy (AAS), Electron Spin Resonance (ESR) Nuclear Magnetic Resonance (NMR) Raman Spectroscopy etc [3].

## V. ELECTRIC & MAGNETIC MEASUREMENTS

Measurement of resistivity is necessary for many applications, to understand energy states of electron in materials. Two or four probe measurements, vibrating sample measurements, superconducting quantum interference device, magneto-optical measurements etc.

## VI. CONCLUSION

These characterization & analysis techniques are complementary in the study of nanomaterials. Bulk methods are used to characterize the collective information of nanomaterial such as XRD, while surface characterization methods like SPM, SEM, TEM, etc provide possibilities at individual nanomaterial. Powder diffraction allows for rapid, non-destructive analysis of multi-component mixtures without the need for extensive sample preparation as compared to other analysis techniques. This gives the ability to quickly analyze unknown materials and perform materials characterization in such fields as metallurgy, mineralogy, forensic science. In contrast, the neutron scattering lengths of most atoms are approximately equal in magnitude. Neutron diffraction techniques may therefore be used to detect light elements such as oxygen or hydrogen in combination with heavy atoms.

## REFERENCES

- [1] Zai-Xing Yang, Wei Zhong, Peng Zhang, Mei-Hua Xu, Yu Deng, Chak-Tong Au, You-Wei Du, Controllable synthesis, Characterization and photoluminescence properties of morphology-tunable CdS nanomaterials generated in thermal evaporation processes *Applied Surface Science*, 258(19), 2012, 7343-7347.
- [2] Vicki Stone, Bernd Nowack, Anders Baun, Nico van den Brink, Frank von der Kammer, Maria Dusinska, Richard Handy, Steven Hankin, Martin Hassellöv, Erik Joner, Teresa F. Fernandes, Nanomaterials for environmental studies: Classification, reference material issues, and strategies for physico-chemical characterization", *Nanomaterials Science of The Total Environment*, 408(7), 2010, 1745-1754.
- [3] G Binnig, C F Quate, C Gerber, Atomic force microscope, *Phys. Rev. Lett.*, 56(9), 1986., 930-933.
- [4] Takaomi Matsutani, Tsuchika Yasumoto, Takeo Tanaka, Tadahiro Kawasaki, Mikiyo Ichihashi, Takashi Ikuta, Development of electron optical system using annular pupils for scanning transmission electron microscope by focused ion beam *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, 272, 2012, 145-148.
- [5] Iliana Medina-Ramirez, Sajid Bashir, Zhiping Luo, Jingbo Louise Liu, Green synthesis and characterization of polymer-stabilized silver nanoparticles, *Colloids and Surfaces B: Biointerfaces*, 73(2), 2009, 185-191
- [6] Martin Hassellöv, James W. Readman, James F. Ranville, Karen Tiede, Nanoparticle analysis and characterization methodologies in environmental risk assessment of engineered nanoparticles, *Ecotoxicology*, 17(5), 2008, 344-361.
- [7] Yongjun Chen, Suzanne K. Lunsford, Yongcheng Song, Huangxian Ju, Polycarpos Falaras, Vlassis Iliodimos, Athanassios G. Kontos, Dionysios D. Dionysiou, Synthesis, characterization and electrochemical properties of mesoporous zirconia Nanomaterials prepared by self-assembling sol-gel method with Tween 20 as a template, *Chemical Engineering Journal*, 170(2-3), 2011, 518-524.
- [8] Agnieszka Dudkiewicz, Karen Tiede, Katrin Loeschner, Louise Helene Soegaard Jensen, Eric Jensen, Rafal Wierzbicki, Alistair B.A. Boxall, Kristian Molhave, characterization of Nanomaterials in food by electron microscopy, *TrAC, Trends in Analytical Chemistry*, 30(1), 2011, 28-43
- [9] Gwénaél Goudec, Philippe Colombari, Raman, Spectroscopy of Nanomaterials: How spectra relate to disorder, particle size and mechanical properties. *Progress in Crystal Growth and Characterization of Materials*, 53(1), 2007, 1-56
- [10] B.N. Akhgar, M. Pazouki, M. Ranjbar, A. Hosseinnia, R. Salarian, Application of Taguchi method for optimization of synthetic rutile nano powder preparation from ilmenite concentrate, *Chemical Engineering Research and Design*, 90(2), 2012, 220-228.