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Common Techniques Used to Characterize Nano-Crystalline Thin Films

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Abstract – This paper presents some important techniqueslike X-ray diffraction, scanning electron microscopy, energy dispersive analysis of X-ray, optical transmission measurement, which are commonly used to characterized nano-crystalline thin films. Structural parameters like crystallite size, micro-strain, grain size, stoichiometric composition of constituted elements, and optical band gap etc. which are dependent upon these techniques also highlighted briefly.

Keywords - XRD, FE-SEM, EDAX, Band Gap.

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1.1 INTRODUCTION

In the last years, the interest on the nano-crystalline thin films have been considerable increased due to their practical importance in technology of thin-film devices as high efficiency solar cells (Nataren, *et. al.,* 1982), field effect transistors (Schon, *et. al.,* 2001), detectors (Konstantatos, *et. al.,* 2006), sensors (Chopra, *et. al.,* 2004), diodes (Shirakawa, 2002), memory switching (Sreeram, *et. al.,* 1991), etc. The properties of thin films can be studied by using various characterization techniques like X-ray diffraction (Cullity, 1978), scanning electron microscopy (McMullan, 1988), transmission electron microscopy (Fultz and Howe, 2007), atomic force microscopy (Binnig, *et. al.,* 1986), energy dispersive analysis of X-ray (Goldstein 1968), optical absorption measurement (Tauc 2003), electrical conductivity, etc. In order to study transport properties of thin films, some special techniques are used like time-of-flight (Seynhaeve, *et. al.,* 1989), steady-state photo-grating (Meeder, *et. al.,* 2002), constant photocurrent measurements (Ritter, *et. al.,* 1986), etc. In the present paper, some common characterization techniques like X-ray diffraction, scanning electron microscopy, energy dispersive analysis of X-ray, optical transmission measurement are highlighted.

1.2 X-RAY DIFFRACTION

X-rays are electromagnetic radiations of wavelength about 1 Å (10^{-10} m), which is about the same size as an atom. The discovery of X-rays in 1895 by Roentgen enabled scientists to probe crystalline structure at the atomic level. X-ray diffraction (XRD) has been in use in two main areas, for the fingerprint characterization of crystalline materials and the determination of their structural properties. Each crystalline solid has its unique characteristic X-ray powder pattern which may be used as a "fingerprint" for its identification. Once the material has been identified, X-ray crystallography may be used to determine its structural properties (unit cell type, lattice parameters, grain size, strain, dislocation density, preferred orientation etc.). X-ray diffraction is one of the most important characterization tools used in solid state physics and material science.

The dominant effect that occurs when an incident beam of monochromatic X-rays interacts with target material is scattering of those X-rays from atoms within the target material (Fig. 1). In crystalline structure, the scattered X-rays undergo constructive and destructive interference. This is the process of diffraction. The diffraction of X-rays by crystals is described by Bragg's law:

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

where θ is angle of reflection, d the lattice spacing, 'n' the order of diffraction and λ the wavelength of incident X-rays.

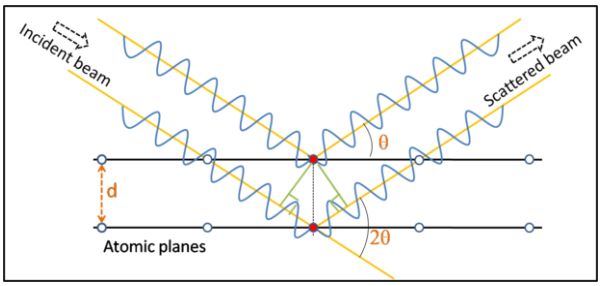

Fig. 1 Schematic diagram of reflection of X-rays at a monocrystalline lattice.

The directions of possible diffractions depend on the size and shape of the unit cell of the material. The intensities of the diffracted beam depend on the kind and arrangement of atoms in the crystal structure. However, most materials are not single crystals, but are composed of many tiny crystallites in all possible orientations called polycrystalline. If the experimental angle is systematically changed, all possible diffraction peaks from the polycrystalline material will be detected.

The structural parameters such as crystallite size, micro-strain and dislocation density can be calculated from XRD spectra in nano-crystalline thin films.

1.3 FIELD EFFECT SCANNING ELECTRON MICROSCOPY

Field Effect scanning electron microscope (FE-SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the sample surface. The electrons interact with the atoms that make up the sample producing secondary electrons, backscattered electrons, X-rays that contain information including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. 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 (i.e. for rapid phase discrimination). X-ray generation produced by inelastic collisions of the incident electrons with electrons in discrete orbitals (shells) of atoms in the sample give information about chemical composition in sample.

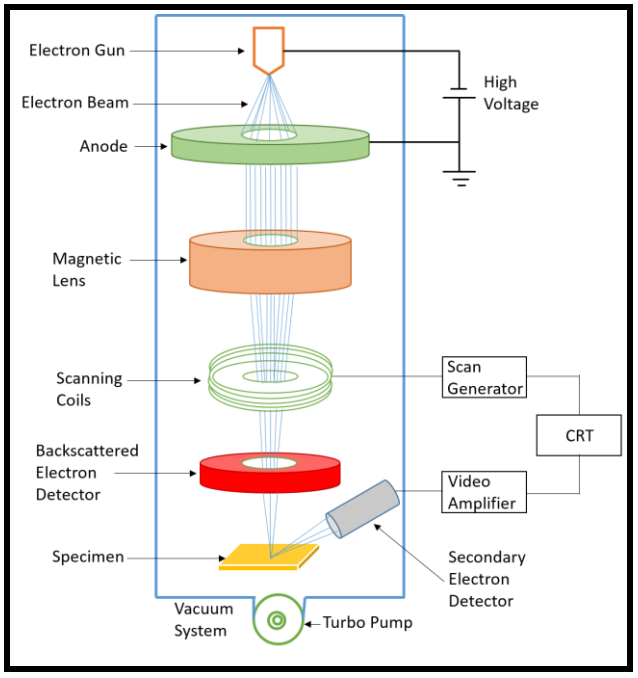

Fig. 2 Schematic diagram of FE-SEM used to take SEM images and EDAX spectra.

In the standard detection mode, secondary electron imaging or SEI, the FE-SEM can produce high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. In Fig. 2, schematic diagram of FE-SEM can be seen. In a typical FE-SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapor pressure of all metals. The electron beam, which typically has an energy ranging from a few hundred eV to 40 keV, is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam passes through electromagnetic lens and scanning coils in electron column, which deflect the beam in the x and y axes so that it scans in a raster fashion over a rectangular area of the sample surface. Once the beam hits the sample, electrons and X-rays are ejected from the sample. Detectors collect these x-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen. This produces the final image.For conventional imaging in the FE-SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for FE-SEM except for cleaning and mounting on a specimen stub. Specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultra-thin coating of electrically conducting material, commonly gold, deposited on the sample either by low vacuum sputter coating or by high vacuum evaporation. Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, iridium, tungsten, chromium and graphite. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation.

FE-SEM give visual images of grain size and shape in nano-crystalline thin films.

1.4 ENERGY DISPERSIVE ANALYSIS OF X-RAY SPECTROSCOPY

One of the most powerful attachments of SEM is Energy Dispersive Analysis of X-ray Spectroscopy (EDAX), which allows elemental analysis without destroying the sample. When a high-energy electron beam (10-20 kV) hits the atom at the point of contact, secondary and backscattered electrons are emitted from the surface. Secondary electrons leave holes in the electron shell. When electrons from the outer shell drop into these holes in the inner shell, X-rays are generated. The energy of the X-ray beam depends on the material. The energy, wavelength and the shell are unique for each atom. Since the X-rays are formed by interaction of high-energy electron beams

with sample surface, elemental analysis is possible for very small areas of the sample. By calculating the area under the peaks in the EDAX spectrum of each identified element and considering accelerating voltage of the beam, quantitative analysis can be performed. The intensity of the peaks in EDAX spectra represents the concentration of the related element in the testing area. Quantitative analysis can be performed both by a standard or standard less method.

1.5 OPTICAL CHARACTERIZATION

Optical properties of a nano-material describe the phenomena of interaction of light with the material. The optical properties of nano-materials can be tailored by controlling their structure, size, shape, and composition. Generally, spectroscopic techniques used to study the optical properties of semiconducting nano-materials are based on optical transitions. An optical transition occurs when a photon of suitable amount of energy is absorbed or emitted. The absorption of photons is established using UV-visible spectroscopy. The optical band gap of nano-materials has been obtained from an UV-visible spectroscopy. Absorption spectrum is another important tool to study the optical behavior of thin films. Optical properties of thin films are studied by recording the normal incidence absorption spectra of the films. Absorption spectra refer to the absorption of photons by the electrons of the material as a result electrons are promoted from ground state to excited state. Optical layout of monochromator-spectrograph is shown in Fig. 3. The light after passing through entrance slit 1 is routed by collimator spherical mirror onto diffraction grating. Diffraction grating is installed on a quadruple turret and change in diffraction grating is done by turning the turret at some angle around a particular axis. Grating converts the parallel beam from each point of entrance slit 1 into a fan of monochromatic parallel beam. The monochromatic beam in turn is split into two equal intensity beams by a beam-splitter or half-mirrored device. One half of the monochromatic beam passes through the reference material which is the bare glass substrate on which film will be deposited and second half of the beam is passed through the glass substrate on which film has been deposited. The intensity of lights coming out from substrate and thin film containing substrate are focused on the detector by passing the light from the lens. The monochromatic beams are converged and converted into an electric signal by the detector.The intensities of these light beams measured by electronic detectors are compared.

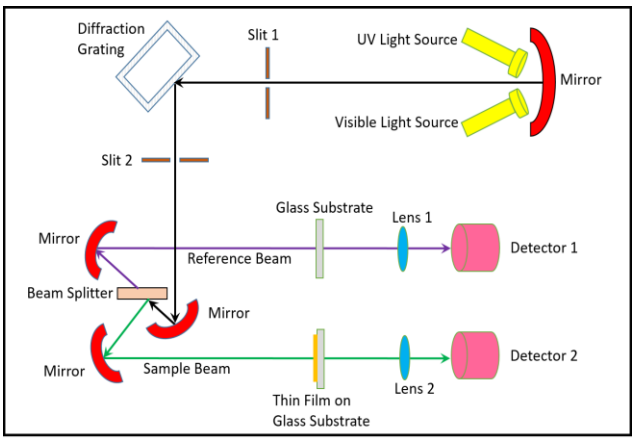

Fig. 3Optical layout of monochromator-spectrograph used to measure the absorption spectra.

After recording the absorbance of the glass substrate with and without film the required absorption spectra of the thin films are obtained after subtracting the absorbance of substrate as reference. The electric signal provided from the detector is processed by a CPU and the computational result is displayed directly on the CPU or output to record.

The optical parameters such as optical band gap, dielectric constants, film thickness and optical conductivity can be determined from transmission measurements of nano-crystalline thin films.

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