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A BRIEF REVIEW ON NANO-STRUCTURED THIN FILM CHARACTERIZATION TECHNIQUES

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Abstract:
In the present study, the characterization techniques, which have been employed to measure and evaluate the physical properties viz. film thickness, crystal structure, surface morphology, film composition, optical transmittance, optical band gap, electrical parameters of the deposited thin films are described.

1. Introduction:

Synthesis of TCO materials with desired structure and properties is of prime importance in condensed matter physics and material science. Since the discovery of TCOs, there has been renewed interest in developing novel methods for the preparation of TCO thin films. The field of material science and engineering community's ability to conceive the novel materials with an extraordinary combination of physical, chemical and mechanical properties has changed the modern society. There is an increasing technological progress. Modern technology requires thin films for distinct applications [1].

Thin film technology is the basic of astounding development in the solid state electronics. The usefulness of the optical properties of metal films, and scientific curiosity about the behavior of two-dimensional solids has been responsible for the immense interest in the study science and technology of the thin films. Thin film studies have directly or indirectly advanced many new areas of research in solid state physics and chemistry which are based on phenomena uniquely characteristic of the thickness, geometry, and structure of the film [2].

2.1. X-ray diffraction studies:

A non-destructive X-ray diffraction is a fundamental characterization technique for the identification of the lattice structure and phase identification [3]. This technique is applicable to solids (powders) or films on substrate. It is useful in providing wide variety of information like crystallinity of the material, lattice parameters, phase, average crystallite/grain size, dislocation density, stress etc. In the present studies, glancing angle XRD is used to analyze the synthesized CdZnO samples. The purpose of the GAXRD is to limit the penetration of X-rays to few nanometers from the surface i.e. the diffraction pattern thus obtained is only by the contribution of film and not by the substrate. The detailed technical specifications found elsewhere [4].

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The basic law involved in the diffraction method of structural analysis is the Bragg's law. When the monochromatic X-rays impinge upon the atoms in a crystal lattice, each atom acts as a source of scattering. The crystal lattice acts as series of parallel reflecting planes. The intensity of the reflected beam at certain angles becomes maximum when the path difference between two reflected rays from two different planes is an integral multiple of λ. This statement is called Bragg's law and is given by the relation[5],

$$2d\sin\theta = n\lambda \quad \text{----- (1)}$$

where n is the order of diffraction, λ is the wavelength of X-rays, d is the inter-planar spacing and θ is the glancing angle. Using d values, the set of lattice planes (hkl) can be identified and the lattice parameters are evaluated. For Hexagonal structure the lattice planes (hkl) can be identified using the relation[6]

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \quad \text{----- (2)}$$

where a and c are lattice parameters.

In the present investigation, the X-ray Diffraction (XRD) data has been collected at room temperature using the Bruker AXS D8 Advance Diffractometer (figure 1) with Cu K_α radiation (λ=0.15054 nm). The diffractometer works in θ-θ Bragg-Brentano geometry which is very useful for dealing with different types of samples.

X-ray Diffraction studies give the diffraction patterns of the samples which are compared with the standard powder diffraction films published by the international centre for diffraction data (ICDD). The average crystallite size of the films (D) is calculated using Debye-Scherrer formula[7],

$$D = \frac{0.94\lambda}{\beta\cos\theta} \quad \text{----- (3)}$$

where β is the Full width at half maximum (FWHM). The lattice period c (c-axis constant) for the hexagonal crystal can be calculated by using Bragg's law[8]:

$$c = \frac{\lambda}{\sin\theta} \quad \text{----- (4)}$$

where λ is the wavelength of X-ray radiation (0.15054 nm), θ is the Bragg's angle of the dominant peak (degrees).

Fig. 1 Bruker AXS D8 Advance Diffractometer (IUAC, New Delhi).

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The number of defects in the films can be determined by calculating dislocation density (δ) using the following relation [9]:

$$\delta = \frac{1}{D^2} \quad \text{----- (5)}$$

where D is the average crystallite/grain size (nm).

The variation of c-axis constant is inherently related to the presence of stress in the films. Including the thermal expansion coefficients of CdZnO (α_{CdZnO} = 6.1 × 10^{-6} K^{-1}) and the glass substrate (α_{glass} = 9 × 10^{-6} K^{-1}), the stress (σ) in the films [10,11] can be derived using the following relation:

$$\text{Stress } (\sigma) = \frac{2c_{13}^2 - c_{33}(c_{11}+c_{12})}{2c_{13}} \times \left[\frac{c_{film} - c_{bulk}}{c_{bulk}} + \left(\alpha_{film} - \alpha_{glass}\right) \times \Delta T \right] \quad \text{----- (6)}$$

where ΔT is difference of the substrate temperature and the room temperature, c_{film} represents the calculated c-axis length of the film, and c_{bulk} = 0. 52054 nm, the values for elastic constants used are: c_{11} = 208.8, c_{33} = 213.8, c_{12} = 119.7 and c_{13} =104.2 GPa [12]. The strain in the films in the direction of the c-axis can be determined by XRD as

$$\varepsilon = \frac{c_{film} - c_{bulk}}{C_{bulk}} \quad \text{----- (7)}$$

2.2. Atomic force microscopy:

The AFM probes the surface of the sample with a sharp tip, a couple of microns long often less than 100 Å in diameter. The tip is located at the free end of a cantilever, which is 100 to 200 μm long. The forces between the tip and sample surface cause the cantilever to bend or deflect. A detector measures the cantilever deflection as tip is scanned over the sample or the sample is scanned under the tip. The measured cantilever deflection allows the computer to generate a map or surface topography.

Several forces typically contribute to the deflection of an AFM cantilever. AFM operates by measuring the attractive or repulsive forces between a tip and the sample. The forces most commonly associated with atomic force microscopy are inter-atomic force called the Van der Waals force. The dependence of the Van der Waals force upon the distance between the tip and the sample is shown in figure 2. The two distance regimes are labeled in the figure (a) the contact regime and (b) non-contact regime.

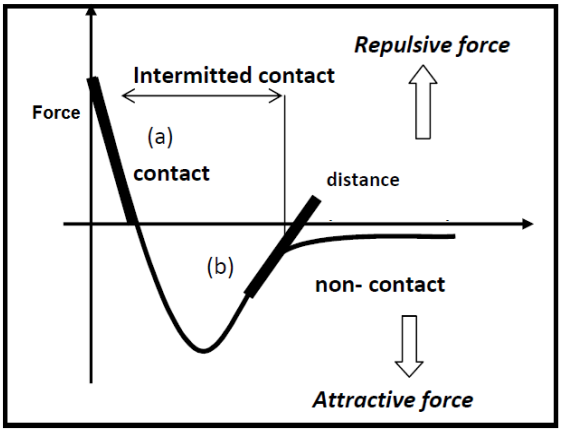

Fig. 2 Inter-atomic force versus distance curve for the operation of AFM

In the contact regime, the cantilever is held at a distance less than few angstroms from the sample surface, and the inter-atomic force between the cantilever and the

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sample is repulsive. In the non-contact regime, the cantilever is held at a distance of the order of tens to hundred of angstroms from the sample surface, and the inter-atomic force between the cantilever and sample is attractive. Figure 3 shows schematic diagram of AFM [13].

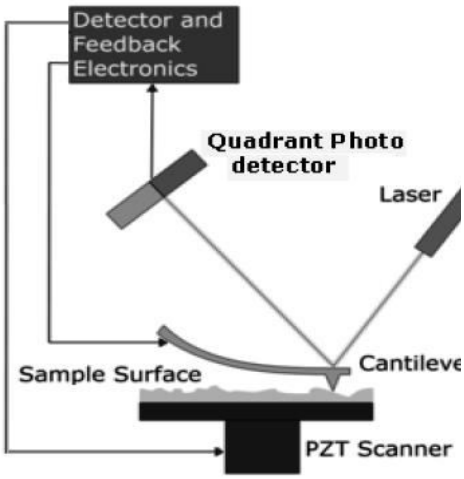

Fig. 3 Schematic diagram of AFM

In principle, AFM resembles the record player as well as the surface profilometer. However, AFM incorporates a number of refinements that enable it to achieve atomic– scale resolution: Sensitive detection, flexible cantilever, sharp tips, high-resolution tip sample positioning and Force feedback.

2.3. Field emission-scanning electron microscopy:

The scanning electron microscopy is a versatile, non-destructive technique that reveals detailed information about the morphology and the composition of natural and manufactured materials at higher magnification, higher resolution and depth of focus as compared to an optical microscope. Researchers in biology, chemistry and physics apply this technique to observe structures that may be as small as 1 nanometer [14,15](= billion of a millimeter). The FESEM may be employed for example to study organelles and DNA material in cells, synthetic polymeres, and coatings on microchips.

Principle: FESEM is a microscope that works with electrons (particles with a negative charge) instead of light. These electrons are liberated by a field emission source. The object is scanned by electrons according to a zig-zag pattern. In standard electron microscopes electrons are mostly generated by heating a tungsten filament by means of a current to a temperature of about 2800°C. Sometimes electrons are produced by a crystal of lanthanum hexaboride [16-18](LaB_6) that is mounted on a tungsten filament. This modification results in a higher electron density in the beam and a better resolution than with the conventional device. In a field emission (FE) scanning electron microscope no heating but a so-called "cold" source is employed. An extremely thin and sharp tungsten needle (tip diameter 10^{-7} –10^{-8} m) acts as a cathode in front of a primary and secondary anode. The voltage between cathode and anode is in the order of magnitude of 0.5 to 30 KV. Because the electron beam produced by the FE source is about 1000 times smaller than in a standard microscope, the image quality is markedly better. As field emission necessitates an extreme vacuum (10^{-8} Torr) in the column of the microscope, a device is

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present that regularly decontaminates the electron source by a current flash. In contrast to a conventional tungsten filament, a FE tip last theoretically for a lifetime, provided the vacuum is maintained stable.

FESEM: MIRA II LMH from TESCAN, with a resolution of 1.5 nm at 30 kV [19,20] has been installed to boost research activities in nanomaterials, in a project funded under Nano Initiative program of Department of Science and Technology (figure. 4). It has a secondary electron (SE) and a backscattered electron (BSE) detector for imaging. An energy dispersive X ray detector INCA PentaFET3 with 133 keV resolution from OXFORD has also been installed in this system for elemental analysis.

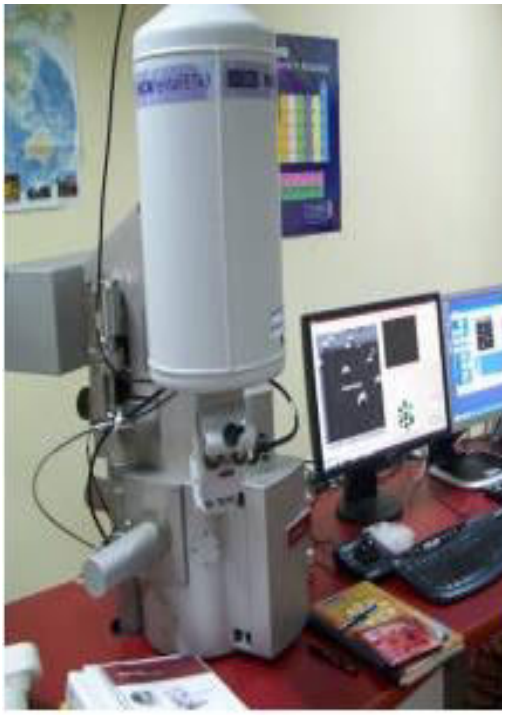

Fig. 4 FESEM with EDX attachment (IUAC)

2.3.1. Energy dispersive analysis by X-rays:

In EDX technique a sample is made the target in an X- ray tube and is bombarded with electrons of suitable energy, it emits characteristics X-rays. This is the basis of a method of chemical analysis. The emitted X-rays are analyzed in an X-ray spectrometer and the elements present in the sample are qualitatively identified by their characteristics wavelengths. For compositions greater than or about 1% and elements separated by few atomic numbers[21,22], energy dispersion analysis is very useful because the intensities are increased about 100-Fold [23-25]. The resolution however, of an energy dispersion instruments is as much as 50 times less than the wavelength dispersion spectrometer using a crystal; thus overlapping of lines from nearby elements may occur. If a sample is irradiated with X-rays of sufficiently high energy, it will emit fluorescent radiation. This radiation may be analyzed in an X-ray spectrometer and the elements present in the sample can be identified by their characteristics wavelengths. Study of thin films, ferrites, composites, biological samples and pharmaceutical samples are the common application areas.

3. Conclusions:

We have analyzed the thin film characterization techniques in detail. Mainly focuses on glancing angle x-ray diffraction to study the structure and lattice parameters

of the thin film samples. In GAXRD, we studied, how to measure the different parameters basic parameters based on the XRD data. AFM studies gives the information about the surface topography of the thin film samples and measure the surface rms roughness. FESEM with EDS give the information about the surface morphology and estimated the grain/particle size and also the elemental analysis.

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