

PREFACE



The science and technology of modern materials is undergoing revolutionary changes. Materials are now increasingly being tailored to provide a specific function as efficiently as possible. This has necessitated the use of a host of new processing techniques, many of which are classified as thin film technologies. And, thus, low dimensional micro-materials and nano-materials have become the emerging frontier. The new tailored materials are exotic, both structurally and chemically. The use of metastable, non crystalline, heterogeneous, multi-layered, graded composition, superlattice and non stoichiometric materials is now common place both for fundamental studies and for device applications.

The development of tailored materials is possible only by exploiting the synergetic effect of microscopic structure on properties. This has led to revolutionary developments of scores of micro-analytical techniques using such probes as photons, electrons, ions and neutral particles. The higher the sensitivity and/or spatial resolution, the more qualitative the techniques become. Therefore, quantitative analysis by these techniques has also emerged as a major scientific field by itself.


Of all the analytical techniques commonly used today, the ones based on X-ray probes continue to dominate the field largely because these are more reliable, simpler, and relatively more quantitative and provide a variety of information. Such techniques include X-ray Diffraction (XRD), X-ray Topography, X-ray Photoelectron Spectroscopy (XP-S), X-ray Fluorescence (XRF), Extended X-ray Absorption Edge Fine Structure (EXAFS), Small Angle Scattering (SAS). Ever since its discovery, X-ray diffraction continues to enjoy the status of a significant fundamental quantitative tool for analysis of materials. The developments in detectors, X-ray optics, sophisticated goniometers, high intensity rotating anode and synchrotron radiation sources, and on-line data acquisition and processing facilities provided by computers have changed the whole field remarkably. Besides being much simpler and faster, the analysis has become much more precise and effectively very powerful.

With the advent of equilibrium and non-equilibrium micro- and nano-materials with their spatial gradients in their physical and chemical structures, it has become necessary to develop X-ray diffraction techniques suited to such low dimensional materials. Instead of the conventional Bragg-Brentano geometry, a renewed interest has developed in utilizing the Seemann-Bohlin para-focussing geometry because of increased intensity without sacrificing resolution and also offering the feasibility of employing a multiple detector system for real time phase transformation studies. Analysis of surfaces and ultrathin films requires new XRD geometries in which the X-rays probe materials only on the surface. In one modification, the angle of incidence of the X-ray beam can be varied from a fraction to several degrees. This angle of incidence (θ) with respect to the sample surface is kept fixed while the detector on 2θ axis scans the XRD pattern. The small angle of incidence allows the beam to sample a large volume on the surface thereby increasing the diffracted intensity. The beam optics allows a very well defined parallel beam. By varying the angle of incidence, it is possible to conduct depth profile of the physical and chemical structure of the films/surface. With sensitive detectors, computer based electronics and high (4-12kW)

intensity X-ray source, about 100 Å thick films can be characterized within a short duration of a few seconds. Our Thin Film Laboratory has utilized this technique to establish some completely unexpected results such as depth variation of the composition and phases (crystallographic structures) of films of carbides, nitrides and oxides of several transition elements. These results have an important bearing on the mechanical hardness, and wear and tear properties of these coatings. In the case of multilayer films, very useful information on the metallurgy of the interfaces has been obtained. With increased X-ray intensity, it should be possible to conduct some real time in-situ experiments on the growth of multilayers.

Another grazing incidence diffraction technique having the capability of analysing very thin epitaxial layers has come to prominence recently. Diffraction is obtained from lattice planes perpendicular to the surface where a monochromatic X-ray beam irradiates the sample surface at a grazing angle which is slightly larger than the critical angle of total reflection. Conventional diffraction and radial scans yield information on the crystallite size, strain, epitaxial relation and orientational spread. A variety of thin film epitaxial systems such as Cu/Al₂O₃ (0001), W/Al₂O₃ (1 $\bar{1}$ 02), Mo and Nb on GaAs (001), GaAs/Al₂O₃ (0001) and InAs/GaAs (001) have been studied to yield very valuable information.

With impressive strides in the development of X-ray diffraction techniques for depth profiling of surface and ultrathin films with precision, speed and high spatial resolution, a new era is being ushered in. The study of real time dynamics of interactions at surfaces and interfaces is indeed feasible and is expected to become a major tool in the rapidly expanding arsenal of micro- and nano-analytical techniques.



K. L. Chopra
Director
Indian Institute of Technology
Kharagpur, India