



Newsletter

Major Analytical Instrumentation Center

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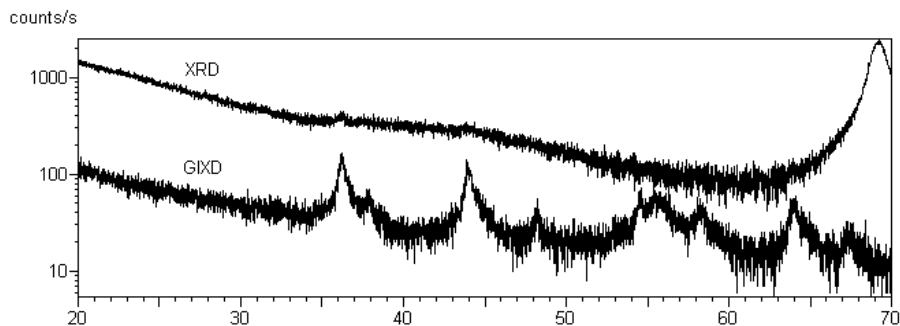
Department of Materials Science and Engineering

Grazing Incidence X-ray Diffraction (GIXD)

By V. Craciun

The X'Pert MRD system in MAIC can be used to perform grazing incidence x-ray diffraction (GIXD) studies of thin films grown on various substrates. The advantages of this technique are: (i) higher sensitivity due to a larger interaction volume (ii) absence of strong diffraction lines from the substrate material and (iii) depth profiling of various crystalline phases present. The first step in GIXD measurements is the determination of the critical angle (θ_c) of the film (see the previous MAIC newsletter for the definition and measurement of the critical angle; for typical materials θ_c is approximately from 0.1° to 1° , hence the name of the technique, grazing incidence). If the radiation incidence angle Ω is smaller than the critical angle then the x-rays penetrate into the material as an evanescent wave with a typical penetration of around 1-2 nm.

For incidence angles greater than θ_c , x-ray radiation penetrates deeper into the layer allowing for sampling at increasing depths with increasing angle. However, since the penetration depth initially increases exponentially with the increase of the incidence angle, the depth profiling is not very accurate. An example of the usefulness of the GIXD technique is presented below. Because the thickness of the deposited film was rather small, the XRD measurement in the usual Bragg-Bretano geometry (θ - 2θ) didn't reveal the crystalline structure of the film and was dominated by the substrate peak. On the other hand, the GIXD measurement performed at an incidence angle of 1° revealed much more information about the crystalline structure and lattice parameters of the deposited film by reducing the signal from the substrate.



X-ray diffraction patterns of a ~25 nm thick $\text{Li}_x\text{Mn}_{2-x}\text{O}_4$ film grown by the pulsed laser deposition technique on a (100) Si substrate; upper trace: θ - 2θ geometry; lower trace: grazing angle incidence (incidence angle $\Omega=1^\circ$). The θ - 2θ spectrum was vertically shifted for clarity.

Director's Corner by L.A. Dempere

The MAIC staff wishes you a Happy New Year 2003! We welcome the spring semester with the addition of a JEOL SEM 6400 to our facilities. This SEM with lithography capabilities will be transferred to the MAIC from the Physics Department at UF and it will be installed in MAIC by the end of January. I would like to thank Dr. David Tanner and Dr. Alan Dorsey for supporting this idea and making it possible. A new dual Focused Ion Beam (FIB) will also be added to the MAIC facilities this year through a research grant led by Dr. Kevin Jones. We expect to have the system installed and fully operational by the end of spring.

Kerry Siebein, M.S. *Electron Microscopy Analysis*

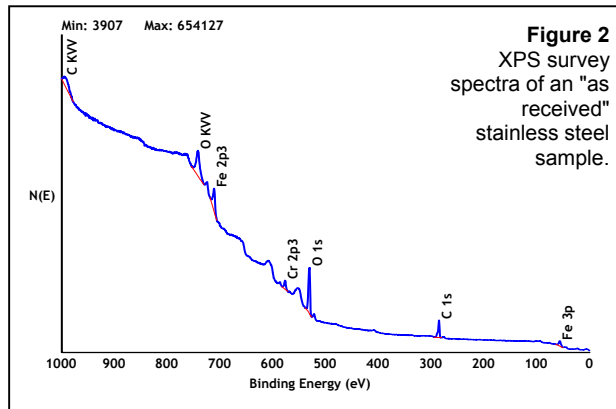
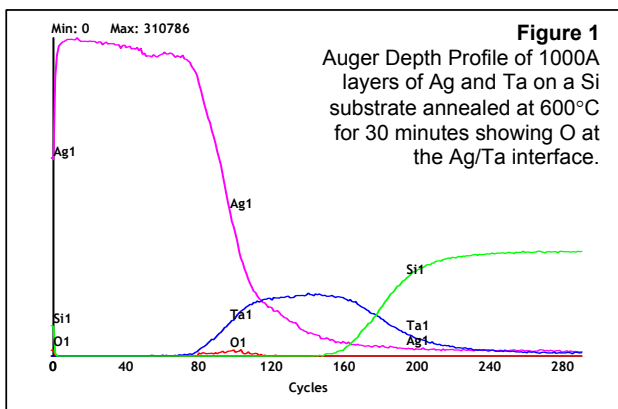
Kerry Siebein has joined the staff of the MAIC to work in the areas of Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM). She earned her Master of Science in Materials Engineering at the Worcester Polytechnic Institute in Worcester, MA. She has held several positions as a Materials Research Engineer before joining the Particle Engineering Research Center (PERC) at UF. Thus she has significant experience working with steels and experimental armor materials, characterization of ceramic materials for wear applications, coatings, thin films and polymer blends using TEM and SEM. Kerry is currently dividing her hours between the PERC and the MAIC performing materials characterization using SEM and TEM. At the MAIC, Kerry is in charge of training students in the operation of the FEG-SEM and services provided using this instrument. She is also in charge of the new Ultracryo-microtome and is coordinating training and services using this instrument.

Surface Analysis in the 21st Century

By Eric Lambers

Surface analysis at MAIC has entered a new era in user friendliness with the addition of RBD Enterprises PC Upgrades for the PHI 660 Auger Electron Spectrometer (AES) and the PHI 5100 X-ray Photoelectron Spectrometer (XPS/ESCA). While preserving the functionality of the previous software the biggest advantage of the new implementation is the ease at which the data can now be incorporated into other Windows® based programs and documents. Using the "copy" and "paste" commands the graphical data can be added to manuscripts written with Windows® based word processing programs (Figures 1&2) or presentations put together for the classroom or conferences using programs such as Power Point®. The data can also be easily exported in an ASCII format and then entered in a spreadsheet program for further analysis in any manner the user wishes. A free copy of the RBD software can be downloaded from their website at: www.rbdenter.com and with this demo

version one can also change the appearance of the graphed data. Characteristics such as line types or widths as well type faces on axis labels can be adjusted to your personal preferences. The PC 147 control unit is also part of the upgrade and enables the new software to control the instrument with state of the art electronics which in the case of the AES system replaced five printed circuit boards with a single board thereby discarding electronic control architecture which was now more than 15 years out of date. Originally developed for older generations of PHI equipment the software is constantly being updated as other equipment and new techniques are being added to the programs capabilities. We are currently involved in "beta" testing of the software for our instruments as well as working with RBD and Microfabritech® on the addition of PHI 6600 SIMS capabilities to the program. An additional feature of this software is the ability of the engineers at the company offices in Bend, Oregon to monitor and control our instruments over the Internet using a program similar to Net Meeting. This means maintenance and trouble shooting is much faster and cheaper saving both time and money.



Seminars: *Using Size Exclusion/Gel Permeation Chromatography and Tetra Detection for the Characterization of Proteins and Natural and Synthetic Polymers*

Please join Dr. Max Haney of Viscotek for a discussion of the use of SEC/GPC and tetra detection for the characterization of proteins and polymers. The seminar will cover the characterization of macromolecules using Low Angle Light Scattering, GPC-Viscometry, Refractive Index, and UV-absorbance. This multi-detector approach provides comprehensive information on absolute molecular weight distributions, molecular size, aggregation, branching, and copolymer composition.

The seminar will be held at 11 AM, February 19, 2003, in the J. Wayne Reitz Union Room 282 at the University of Florida.

Refreshments will be provided. Please call 352-392-6985 to register.

MAIC POLICIES CORNER: Documentation and Records: It is mandatory for all users of the MAIC facilities to comply with MAIC's documentation and logging protocol. An accurate/updated account number is necessary to document your work in the corresponding log books located next to each instrument.