Materials Science X-Ray Analysis Laboratory

The Department of Mechanical Engineering supports and houses the X-Ray Analysis Laboratory with two Philips x-ray diffractometers, a Materials Research Diffractometer and a Multi-Purpose Diffractometer. The instrumentation was funded from National Science Foundation, Division of Materials Research and the U of R with equipment help from Bausch & Lomb. The facility is about 10 years old. The Materials Research Diffractometer, MRD, is well suited for very high-resolution work especially for thin films and single crystal multilayer diffraction analysis; the Multi-Purpose Diffractometer, MPD, is for powder or polycrystalline diffraction analysis at room or non-ambient temperatures with controlled atmospheres. These instruments have a wide range of applications: they perform qualitative and quantitative analysis of powder diffraction data, high resolution rocking curves for electronic epitaxial wafers, quantifying defects and perfection in high quality crystals, measuring thin film thickness, layer densities and interface quality. In bulk and thin film materials, one can determine stresses, textures and grain sizes. This is made possible by pre-aligned, interchangeable x-ray optical modules for pre and post processing of the x-ray beam. The user can select from a variety of Prefix optics, depending on requirements for intensity, focusing and tolerance for beam divergence. These modules can be exchanged in a matter of minutes, without the need for complicated dismantling, reassembly and realignment. Generally, higher intensity is accompanied with increased beam divergence. For example, an asymmetric crystal module, using a Ge (220) 4 crystal monochromator selects Cu Kα1 radiation at a beam divergence as low as 18 arc-seconds and an intensity of 5 million counts per second. The line and spot foci have intensity 100 times higher, but at the expense beam divergence and dispersion. The new goniometer has a resolution on θ and 2θ of 0.0001°. The Eulerian cradle has 5 degrees of freedom for sample orientation and position. The x-ray software is extensive with
programs for performing comparison of the diffraction pattern with known patterns, Rietveld phase analysis, crystal orientations, textures, residual stresses, particle size analysis, film thickness, epitaxial layers, etc. The results obtained on complex structures are precise yet very fast. The data can be exported to spread sheets and other graphical programs. The instruments are housed in the refurbished x-ray room in Hopeman 111 on River Campus. Both diffractometers are using the same software so they are operationally transparent. The MPD and MRD units including the very high resolution modules and goniometer are used in selected Mechanical Engineering and Materials Science courses. For further information about the instruments and their capabilities, contact Professor Stephen Burns or Chris Pratt. See above for the color pictures of the X-Ray Optics for Selected Applications in Materials.

Contacts: Professor Stephen J Burns, at (585) 275-4082, he can be reached in Hopeman 219 or by e-mail at burns@me.rochester.edu; Chris Pratt, schedules operation and use, she can be contacted at (585) 275-7807, she is in Hopeman 115 and her e-mail is Pratt@me.rochester.edu.

Below are descriptions of the two x-ray diffraction systems we use for analysis of crystalline materials.

1. General Purpose X-ray Diffraction System for phase analysis of polycrystalline micro or bulk samples with a high temperature, controlled atmosphere sample stage, plus focusing and parallel beam optics.

2. Eulerian Cradle, High Resolution, Horizontal X-ray Diffraction System with these capabilities:
   o High-Resolution X-Ray Diffraction, HRXRD, double-crystal or 4-bounce x-ray geometry for studying large-face single crystals and epitaxial thin films, rocking curve analysis.
   o Bragg geometry for studying stress and texture in polycrystalline solids; crystallographic alignment of single crystals.
   o Grazing-Incidence geometry for phase analysis of polycrystalline thin films and x-ray reflection for film thickness.
   o High resolution powder diffraction.

The following information briefly describes these x-ray diffraction systems and provides an operational overview to facilitate user planning. Additional information is available via the links shown at the end of this page.

System Configurations and Operational Overview

All work with the following systems and in the X-Ray Analysis Laboratory must be performed in compliance with established practices and procedures. See x-ray safety procedures below.
1. **General Purpose X-ray Diffraction System: Philips MPD**

The general purpose X-ray diffractometer is a Philips X'Pert MPD system with a vertical $\Theta$-2$\Theta$ goniometer (160 mm radius). The x-ray source is a long-fine-focus, ceramic x-ray tube with Cu anode. Normal operating power is 40 kV, 30 mA (or less than 1.8 kW). The system optics consist of fixed divergence, anti-scatter, and receiving slits, incident and diffracted beam Soller slits, a curved graphite diffracted beam monochromator, and a proportional counter detector in Bragg-Brentano, parafocusing geometry. The principal application of this system is for phase analysis of polycrystalline samples. The Bulk Multi-Purpose Sample Stage supports typical powder mounts but primarily accommodates large monolithic specimens of a wide variety of sizes and shapes. In addition, specialized micro-capillary sample tubes mounted on Supper goniometers with a rotatable sample stage has been constructed. An environmental chamber is available for use with non-ambient conditions. See details on the Anton-Paar TTK 450 from -193°C to +400°C; the non-ambient sample stage can accommodate powder specimens and small, flat monolithic solids e.g., thin film specimens in controlled atmosphere.

2. **Eulerian Cradle, High Resolution, Horizontal X-ray Diffraction System: Philips MRD**

The x-ray diffractometer is a Philips X'Pert MRD PRO system with a horizontal, high-resolution $\Omega$-2$\Theta$ goniometer (320 mm radius). An open Eulerian cradle and sample holder provides two additional axes of rotation (-90° < $\Psi$ < 90°, and -360° < $\Phi$ < +360°). The X-ray source is a long-fine-focus, ceramic x-ray tube with Cu anode. Normal operating power is 45 kV, 40 mA (again less than 1.8 kW). The sample stage supports monolithic and powder specimens of a wide variety of shapes and sizes.

In the high resolution configuration, the x-ray source is used in spot-focus and the incident-beam optic is a Bartells 4-bounce Ge (220) monochromator which provides an intense, highly collimated beam with a divergence of $\Delta\Theta \sim 18$ arc-seconds. The typical receiving optic is a sealed Xe proportional counter detector. This configuration is for high-resolution, double-crystal diffraction, or HRXRD. Alternatively, the detector can be placed behind thin film attachments using the Ge (220) prefix optics or more divergent spot or line focus x-ray sources. The principal application of this setup is structure analysis of epitaxial thin films (rocking curve measurements, etc.).

In Bragg geometry, the x-ray source is used in line-focus. The incident beam optic is a crossed-slit collimator or a programmable slit. Receiving optics consist of a programmable receiving slit, soller slit, curved graphite monochromator, and a Xe proportional counter detector. Typical applications include powder diffraction, stress and texture analysis of polycrystalline solids.
In Grazing-Incidence geometry, the x-ray source is used in line or spot focus. The incident beam optic is typically a fixed divergence slit assembly. Receiving optics consist of a 0.027 radian parallel plate collimator, flat graphite monochromator, and proportional counter detector. Applications include phase analysis of polycrystalline thin film samples and x-ray reflectivity (XRR).

Analysis Software

The principal x-ray diffraction data analysis program is X’Pert High Score and X’Pert Plus. There are extensive software packages available on line but they are not supported by the X-Ray Analysis Laboratory. It is suggested that any interested researcher consider using Jade (Materials Data Inc., Livermore, CA.). Jade supports comprehensive analysis of x-ray diffraction patterns, including Search/Match (phase identification), peak profile fitting, indexing, unit cell refinement, etc. Other programs include Riqas (MDI) and X’Pert Plus (Philips) for Rietveld analysis. In addition, the Philips programs X’Pert Texture, X’Pert Epitaxy, and WINGIXA, and the Bede Scientific programs RADS and REFS, facilitate analysis of the various data obtainable from the Special Applications and Four-Circle X-ray Diffraction Systems.

Databases

The principal reference database is the current issue of the Powder Diffraction File (International Centre for Diffraction Data, Newtown Square, PA.). The lab also can obtain access to the Inorganic Crystal Structure Database (NIST, Gaithersburg, MD and FIZ-Karlsruhe, Germany). These data are now incorporated into ICDD Powder Diffraction Data Base V.

Additional Information

PANalytical (formerly Philips Analytical)
http://www.panalytical.com/

Anton-Paar
http://www.anton-paar.com/

International Centre for Diffraction Data: PDF Database
http://www.icdd.com/

FIZ/NIST: ICSD Database
http://www.nist.gov/srd/nist84.htm

International Union of Crystallography
http://www.iucr.org/iucr-top/welcome.html

Materials Data Inc.
http://www.materialsdata.com/
Introduction to X-ray Diffraction

This is intended as a (very) brief introduction to some of the common x-ray diffraction techniques used in materials characterization. It is designed for people who are novices in this field but are interested in using the techniques in their research. Extensive and authoritative discussions can be found in the numerous books and journal articles on this subject. Some references are listed below.

3. *Elements of X-ray Diffraction, 2nd Ed.*, by B.D. Cullity, Addison-Wesley, 1978 (Covers most techniques used in traditional material characterization)