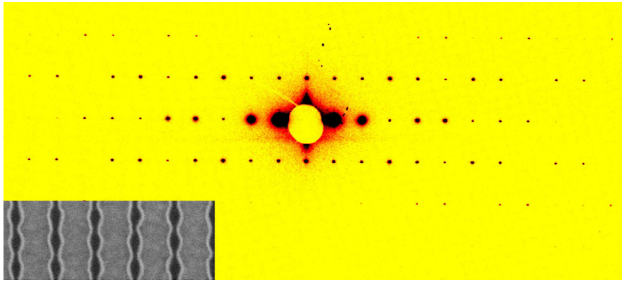
Measurement Facilities: *X-Ray Methods*



The central part of the figure shows the 2-D CD-SAXS dif-fraction pattern corresponding to the pattern in the inset.

from the Mo x-ray tube. These x-rays can penetrate a semi-standard Si wafer allowing on wafer characterization, without sacrificial cross-sectioning of the pattern. The diffraction patterns can be modeled with basic diffraction theory to quantify the pattern periodicity or pitch, the line and space widths, the pattern height, the side wall angles or taper in the pattern, and even the line edge and line width roughness.

We have also developed a complementary Critical Dimension Specular X-ray Reflectivity (CD-SXR) measurement that quantifies several of the same physical dimensions with similar precision and without sacri-ficing the sample. The advantage of CD-SXR is that it also quantifies the material density in the pattern. CD-SXR can also quantify the thickness, density, and roughness of planar films or layers that would not be amenable to transmission scattering.

Organic Semiconductor Performance

The mission of the Organic Electronics Project in the Division is to develop structural measurements that correlate the electrical performance of organic semi-conductor materials with the way they structure and morphology. For these measurements, Grazing Incidence X-ray Diffraction (GIXD) on our in house in-strument is used to determine the lattice parameters of the crystal structure in these organic molecules. These data are combined with measurements of the orientations, relative to the substrate normal, of the different chemical constituents to fully determine 3-D packing of the organic semiconductor. The details of this 3-D packing are important to describe variations in the charge mobility between different materials.

Nanoparticle Characterization

International concerns regarding the environmental health and safety of nanoparticles have spurred calls for NIST to develop reliable measurement systems and robust reference nanoparticles. Small Angle X-ray Scattering provides (SAXS) provides critical measurements of size and shape in a statistically relevant fashion. These measurements are cross-correlated with other size measurements methods.

X-Ray Porosimetry

Characterizing the porosity characteristics in thin films and nanostructures, such as those used in semi-conductor interconnects, is a significant measurement challenge. Traditional porosimetry techniques lack the sensitivity in such cases were the volumes of the porous samples are exceedingly small. To this end we have coupled our sensitive specular x-ray reflectivity technique with the capillary condensation of an organic probe vapor such as toluene. By systematically increasing the partial pressure of toluene over the porous thin film or nanostructured sample, we condense the vapor in progressively larger pores. By measuring the toluene uptake (from the change in density of the film) as a function of the partial pressure, we can determine the total porosity, the porosity as a function of vertical height, the wall density of the material between the pores, and even information about the interconnectivity of the pores.

Christopher Soles
(301) 975-8087
csoles@nist.gov

R. Joseph Kline
(301) 975-4356
joe.kline@nist.gov

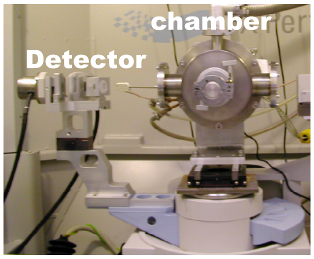
Wen-li Wu
(301) 975- 6389
wenli@nist.gov

Measurement Facilities:
X-Ray Methods

One of the core strengths of the Polymers Division continues to be developing world class x-ray scattering and reflectivity facilities. We use and continue to modify these high power measurements to address high impact measurement challenges facing our nation. At the center of our x-ray facility are two key instruments, a high resolution x-ray reflectometer and a one-of-a-kind small angle scattering instrument. In 2008, we will be augmenting these instruments with a state of the art x-ray diffractometer for solving crys-tal structures of polymers in extremely thin films. With these tools we will have one of the premier x-ray facilities for polymer research in the United Sates. While x-ray scattering and reflectivity are widely recognized as basic materials characterization tools, at NIST we use these techniques to address larger scale materials metrology needs. These are detailed below.

X-Ray Reflectivity

Our x-ray reflectometer features a 4-bounce channel cut Ge[220] cystal monochromator in the incident beam, a 3-bounce Ge[220] crystal monochromator on the reflected beam, and goiniometers with an angular accuracy of ±0.0001° . These high precision elements are coupled with x-ray focusing optics to provide a brilliant, high resolution instrument. With this configuration we can quantify the thickness, density, and roughness of films and samples from a few nm's in height up to almost 3 μm. There is also a range of environmental chambers available including ambient, vacuum, and temperature control. We have constructed a flow control unit to systematically vary the partial pressure of different vapors in the sample chamber for in-situ adsorption or swelling or thermal expansion measurements.



NIST high resolution reflectometer configured with the environmentally controlled sample chamber

X-Ray Scattering

The second critical component of our x-ray methods facilities is a custom-built small angle scattering x-ray scattering instrument. This highly versatile and configurable tool boasts a high power rotating anode generator, interchangeable Cu and Mo x-ray sources, focusing mirrors for a brilliant x-ray spot from both radiation sources, a choice of pin-hole or Bonse-Hart beam collimation, and a selection of detectors that includes a 2-D wire array, a 2-D CCD image plate, and a 1-D wire detector. Different con-figurations of these elements allow us to interrogate length scales ranging from inter-atomic distances (WAXS) up to nearly 20 μm (USAXS). Samples can be measured in either the transmission or the grazing incidence configurations.

NIST designed x-ray scattering tool configured for small angle scattering with the Cu x-ray tube

Pattern Shape Metrology

We use our expertise to develop high resolutions methods to quantify the shape of nanoscale patterns, primarily for critical dimension control in semiconductor and other nanofabrication processes. Critical Dimension Small Angle X-ray Scattering (CD-SAXS) is a transmission technique that treats the nanoscale pattern as a diffraction grating. The transmission geometry is a key aspect of the measurement made possible through the use of the high energy x-rays

FACILITIES