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Mesoscopic and Nanoscopic Materials Characterization

with the 3D X-ray Crystal Microscope and the Princeton Instruments PI•SCX:4300 Camera

Figure 1. The 3D X-ray Crystal Microscope.

Figure 2. The PI•SCX:4300 from Princeton Instruments.

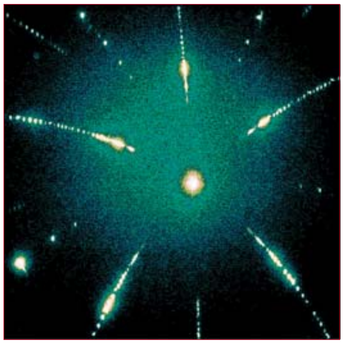

Figure 3. A microLaue pattern from a pendeoepitaxial film of GaN grown on single-crystal SiC. The SiC has had grooves machined into the surface that allow the GaN to grow with reduced strain. Laue image collected by Rosaliya Barabash (ORNL) and Wenjun Liu (ORNL) on beamline 34-ID-E at the Advanced Photon Source. The x-ray beam was approximately 0.5 x 0.5 µm squared. Sample from S. Einfeldt (a), Z. Reitmeier (b), A. M. Roskowski (b), and R. F. Davis (b).

(a) Institute of Solid State Physics, University of Bremen, 28334 Bremen, Germany

(b) Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA

The work of Dr. Gene Ice, Dr. Bennett Larson, and their collaborators could have a profound influence on future integrated circuits, superconducting films, and structural and electro-active materials. Ice, who leads the X-ray Research and Applications Group of the Metals and Ceramics Division at Oak Ridge National Laboratory (ORNL), and Larson, who leads the X-ray Diffraction Group of the Condensed Matter Science Division at ORNL, have collaborated with several other ORNL researchers, including Drs. Wenjun Liu, John Budai, and Jonathan Tischler, to create and refine a revolutionary new instrument for materials characterization.

The penetrating, nondestructive tool — known as the 3D X-ray Crystal Microscope (see **Figure 1**) — has been designed specifically for three-dimensional characterization of mesoscopic and nanoscopic materials structure. The first system is already in use on beamline 34-ID at the Advanced Photon Source. Microscope installations are now being planned for additional synchrotrons in the United States and abroad.

"The prototype microscope has a routine spatial resolution of approximately 0.5 x 0.5 x 1 µm cubed and can probe tens to hundreds of µm below a sample surface depending on the composition of the sample," the ORNL researchers[1] note. "For each volume element measured, the microscope can determine between 10 and 16 parameters… The time required to collect each volume element varies between 1 and 14 seconds depending on the precision of the parameters and the sample complexity."

"In a typical experiment," Ice and Larson[2] explain, "a polychromatic x-ray beam impinges on an achromatic total-external-reflection Kirkpatrick-Baez mirror pair. The elliptical mirrors focus the x-ray beam to a submicron spot on the sample. The sample is rastered in the x-ray beam by a precision 3-axis stage to select a line through the sample for analysis. The Laue patterns generated by each volume element (voxel) within the sample are collected by an x-ray sensitive CCD [PI•SCX:4300 camera from Princeton Instruments, see **Figure 2**]. The overlapping Laue patterns from each voxel along the x-ray beam are decoded by a differential aperture that scans across the surface of the sample. From the decoded Laue patterns, it is possible to determine the phase and orientation of local crystalline structure within each voxel. In addition, it is often possible to determine the deviatoric strain tensor and/or the deformation tensor. The full elastic strain tensor can be determined from the deviatoric strain tensor and from a single measurement of the Laue energy of one reflection."

The 3D X-ray Crystal Microscope represents an invaluable tool for learning about the evolution of local stress in materials, as well as the effect local inhomogeneities have on materials behavior. "Indeed, most materials are formed from misoriented crystalline grains with anisotropic crystalline properties and with a complex network of grain and phase boundaries," Ice and Larson[2] elaborate. "Although the volume-average behavior of polycrystalline materials can be modeled with various levels of sophistication, the overall behavior ultimately is a consequence of materials behavior at the mesoscopic and atomic scales."

The semiconductor, electronics, and auto industries could potentially realize significant benefits from the information attainable with the new instrument. According to Ice, the 3D X-ray Crystal Microscope is proving extremely effective for studying electromigration in IC interconnects — with an eye towards someday helping to improve device reliability. The microscope allows researchers to look along the interconnects and acquire quantitative data on strain-induced deformities attributable to processing and electron wind.

Ice says the tool is also being used to characterize thin, epitaxially grown films (see **Figure 3**), particularly high-temperature superconductors (see **Figure 4**). In addition, it can be used to map the tetragonal domain walls of ferroelectric and other electro-active materials. The novel microscope will have a considerable impact on the field of nanotechnology research as well.

USA 609.587.9797 | **Benelux** +31.347.324989 | **France** +33.160.86.03.65
Germany +49.89.660.779.3 | **Japan** +81.43.274.8022 | **www.princetoninstruments.com**

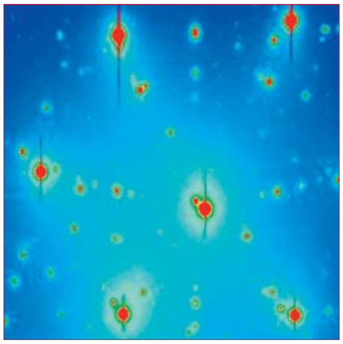

Figure 4. A microbeam Laue image from a Rolling-Assisted Biaxial Textured Substrate (RABiTS) high-temperature superconductor film. The Laue spots arise from the underlying highly textured Ni grains, from the buffer layers, and from the thin superconducting film. Samples obtained from Amit Goyal (ORNL). Image collected by Wenjun Liu (ORNL) on beamline 34-ID-E.

Figure 5. A microLaue image from a strain-free, thin-Si sample. The CCD-to-sample distance is 45 mm. The point-spread function is approximately 1.5 pixels FWHM. Image collected by Wenjun Liu (ORNL) on beamline 34-ID-E.

While the applied theory behind the 3D X-ray Crystal Microscope is actually a creative implementation of the oldest x-ray diffraction technique (i.e., Laue diffraction), the instrument's design takes full advantage of the latest technologies available. One of the new tool's key components, of course, is a high-performance CCD camera.

To collect useful data, the 3D X-ray Crystal Microscope requires a detector that delivers good sensitivity in the 8- to 20-keV energy range, a large field of view, high spatial resolution, wide dynamic range, excellent linearity, low noise, and fast frame rates. The 16-bit PI•SCX® from Princeton Instruments addresses this demanding set of criteria, Ice reports.

The camera model utilized, a PI•SCX:4300, features a 2084 x 2084-pixel CCD optically coupled to a customized CsI:Tl x-ray scintillator via a 1:1 fiber stub. (For ease of use, the front of the fiberoptic is extended outside the vacuum.) With 24 x 24-μm pixel pitch, the 2k x 2k CCD array provides a 50 x 50-mm imaging area.

Dr. Wenjun Liu states that the point-spread function is typically about 1.5 pixels FWHM (see **Figure 5**). The x-ray source size as the beam hits the sample is less than a micron, but since it's divergent (approximately 0.5 mrad) when the beam emerges from the sample, the real beam size will be around 20 μm as the x-ray hits the CCD (45 mm from the sample), which is close to the size of one pixel. In terms of camera resolution, Liu adds, the researchers fit peaks (via 2D Gaussian) and can observe peak shifts less than 0.1 to 0.2 pixels.

The camera's CsI:Tl crystalline scintillator, meanwhile, has been optimized to provide superior resolution in the energy range of interest; its special needle structure serves as a light guide, transmitting the visible light generated by the absorbed x-rays to the high-QE CCD, while also preserving spatial resolution and uniformity of light output.

The camera's signal-to-noise ratio is exceptional, due in large part to state-of-the-art Princeton Instruments electronics. To further reduce unwanted background, the scientific-grade CCD can be thermoelectrically cooled as low as -50°C, thus minimizing the noise that arises from thermally generated dark current. A 1-MHz digitizer and shutterless operation enable unprecedented frame rates for a fiberoptically bonded, large-format, 16-bit CCD camera.

The 3D X-ray Crystal Microscope is certain to become an even more powerful tool for materials characterization as the acquisition speed and spatial resolution afforded by high-performance CCD cameras continue to improve.

For more information about current x-ray research and applications at Oak Ridge National Laboratory, please visit **https://www.ms.ornl.gov/researchgroups/xray/default.htm**

To learn more about Princeton Instruments' latest x-ray CCD cameras, please visit **http://www.princetoninstruments.com/xray.html**

Figures 1, 3, 4, and **5** courtesy of Gene E. Ice, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA. Funding for the 3D X-ray Crystal Microscope provided by the Department of Energy's Basic Energy Sciences.

References

[1] Wenjun Liu, Gene E. Ice, Bennett Larson, Wenge Yang, and Jonathan Tischler, "The 3D X-ray Crystal Microscope: A New Tool for Materials Characterization", paper submitted to *Metallurgical and Materials Transactions* (2004).

[2] G. E. Ice and B. C. Larson, "The Polychromatic 3D X-ray Microscope: A New Tool for Materials Characterization", abstract for Microscopy & Microanalysis Meeting in Savannah, GA (2004).