High-Resolution Three-Dimensional X-Ray Microscopy

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Guest Editors

Abstract

This issue of *MRS Bulletin* focuses on the rapid progress that is ongoing in the development of hard x-ray microscopies with three-dimensional spatial resolutions ranging from micrometers to nanometers. The individual articles provide a crosscut of developments in hard x-ray projection tomography microscopy for imaging density and chemical fluctuations in crystalline and noncrystalline materials; large-angle diffraction-based, spatially resolved imaging of local structure, orientation, and strain distributions in crystalline materials; and emerging coherent diffraction imaging for nanometer-range Fourier transform imaging of crystalline and noncrystalline materials.

Keywords: *microbeams, microstructure, three-dimensional x-ray microscopy, tomography, x-ray optics.*

The availability of high-brilliance synchrotron x-ray sources, recent developments in high-precision x-ray focusing optics, and the development of new x-ray diffraction and contrast imaging techniques have stimulated revolutionary advances in three-dimensional x-ray microscopy using hard (e.g., >5–6 keV) x-rays. Electron microscopes have long provided high-resolution structure and spectroscopy tools for the investigation of thin-section samples, and electron backscattering diffraction (EBSD) microscopy routinely provides surface or near-surface microstructural information.[1] Similarly, soft x-rays (e.g., <3–5 keV) enable a rich variety of two-dimensional structure and spectroscopic microscopy tools.[2] However, hard x-ray microscopy tools to probe the interior of bulk materials with three-dimensional spatial resolution in the micrometer or submicrometer range have, until recently, been missing from the scientific toolbox for structure and spectroscopy investigations.

Considering that almost all technological and biological materials are inhomogeneous on length scales ranging from nanometers to millimeters, nondestructive probes with a range of penetration power and resolutions are needed for the investigation of the structure and evolution of materials. With single-crystal diamond and silicon as notable exceptions, the important technological properties of materials are often linked directly to inhomogeneous density and chemical distributions or to crystal grain-size and grain-orientation distributions; grain-boundary configurations and crystalline or noncrystalline second phases can be important as well. The generation and control of the evolution of such microstructural features are of central importance to the structural metals and ceramics industries, and they play critical roles in determining the properties of materials such as composites (hard/soft), functionally graded materials, and layered materials.

The articles in this issue of *MRS Bulletin* describe hard x-ray microscopy techniques that provide 3D spatial resolution ranging from a few micrometers to nanometers. The individual articles include (1) x-ray absorption and phase contrast imaging of density fluctuations and chemical structures in both crystalline and nanopatterned materials with micrometer and submicrometer resolution; (2) x-ray diffraction imaging of the crystal structure, grain orientation, and elastic and plastic strain distributions with resolution from a few micrometers to the submicrometer range; and (3) coherent diffraction imaging of both crystalline and noncrystalline materials with resolution capabilities below 10 nm. Each of these techniques is experiencing rapid progress. The nondestructive nature of these x-ray techniques makes them complementary to electron microscopy techniques. Electron microscopy provides atomic resolution for structural features that are not lost by the destructive technique of slicing samples into thin sections. However, there are many cases in which nondestructive measurements are needed. Spatially resolved measurements of elastic strain in materials or investigations of microstructural evolution such as grain growth and plastic deformation under bulk conditions require nondestructive techniques over sample sizes of up to millimeters in some cases. Moreover, irreplaceable samples or samples in which there is a potential for contamination or artifact introduction during thinning fall into the category requiring nondestructive measurements as well.

As indicated here, the high-intensity and highly collimated x-ray beams from third-generation* (i.e., high-brilliance) synchrotron sources—such as the European Synchrotron Radiation Facility (ESRF), the Advanced Photon Source (APS), the Japanese SPring-8 synchrotron source and the Advanced Light Source (ALS)—have played central roles in driving the development of these x-ray microscopies. The development of high-precision hard x-ray Fresnel zone plates, multiple refractive x-ray lenses, and total reflection x-ray mirrors has been critical as well. Hard x-ray Fresnel zone plates and total reflection mirrors now provide high-intensity x-ray beams with diameters of <100 nm. Some scientists predict that beam sizes of a few tens of nanometers will be possible in the next few years.

The development of innovative x-ray imaging and diffraction techniques exploiting high-resolution CCD area detectors and the development of advanced computational and analysis techniques in combination with high-brilliance beams and high-precision focusing optics have been the driving force behind the current revolution in 3D x-ray microscopy. That is, harnessing all of these aspects *simultaneously* has been the key to the powerful x-ray microscopies that can now be ap-

*First-generation x-ray sources used x-rays that were the by-products of high-energy storage rings, while second-generation sources were dedicated to x-ray production, but did not have the high intensity and collimation now available.

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plied routinely in materials investigations. This has not been possible in the hard x-ray regime previously.

The availability of these microscopies is creating new opportunities in materials research and in the broader (biological, geological, physical, environmental, etc.) areas of materials science as well. The dynamics and evolution of the nonuniform structure of materials on mesoscopic length scales of tenths of micrometers up to hundreds of micrometers and down to subnanoscale sizes are in general not predictable in detail with our present theoretical understanding and computational capabilities.[3] Accordingly, quantitative 3D x-ray microscopy measurements are critically needed over these size ranges. Such measurements will provide the (currently missing) link with increasingly powerful computer simulation and multiscale modeling required for continued progress toward a fundamental understanding of materials properties and advanced materials processing on all length scales.

The articles that follow provide a crosscut of developments and activities in their respective microscopies. As has been the case with electron and soft x-ray microscopy, the hard x-ray microscopies discussed in these articles are vibrant and progressing at an ever-quickening pace as instrument technologies advance and new techniques develop and mature.

The first article, by Schroer et al., discusses absorption and phase contrast imaging microscopy and fluorescence microscopy. These techniques are sensitive to electron density and chemical-specie distributions and are independent of the presence or absence of crystallinity in the sample. Combining tomography with imaging makes it possible to determine the three-dimensional structure of opaque samples nondestructively. Moreover, when tomographic imaging is combined with absorption or fluorescence spectroscopy, 3D imaging with chemical specificity is possible, and in some cases, the valences of atoms can be determined in addition to their spatial distribution. This is a completely novel approach that will be of great interest for many areas such as chemistry, environmental sciences, materials science, and physiology. Examples chosen for illustration include the structure and composition of micrometeorites, radioactive uranium particles released during the Chernobyl accident, eutectic binary alloys, and the internal structure of interconnect circuits with multiple planes of integration, the last of which was performed using a laboratory-scale x-ray generator.

The imaging process used for determining 3D structure is described by two characteristic parameters: contrast and lateral resolution. Absorption contrast is determined by the absorptive term in the refractive index. The dispersive term in the refractive index is much larger for hard x-rays, and as a result, phase contrast is determined by the dispersive term in the refractive index. This provides much higher sensitivity, particularly for low-atomic-number (low-Z) materials. However, phase contrast requires coherent illumination of the sample, which in turn requires a distant x-ray source of small dimensions. The second characteristic of an image is the lateral resolution. From optics, spatial resolution is given by $\sim 0.61\,\lambda/\mathrm{NA}$, where λ is the x-ray wavelength and NA is the numerical aperture. Since the NA is $<10^{-3}$ for hard x-rays, structural features comparable in size to the wavelength cannot be resolved, and in particular, hard x-rays with wavelengths of ~ 1 Å cannot provide atomic-resolution imaging using forward scattering. Electron microscopes also have rather small NAs, but atomic resolution is achieved by using electrons with energy of a few hundred kiloelectronvolts, so that $\lambda \ll 1$ Å. The limiting "image resolution" that can be reached with x-rays is a point of discussion at present, but it seems to be somewhere between 10 nm and 20 nm.

The second article exploits Bragg diffraction from the periodic lattice structure of grains in polycrystalline materials to perform 3D x-ray structural microscopy. Poulsen et al. describe the development of a high-energy (>50 keV) 3D x-ray diffraction (3DXRD) microscope that is capable of imaging crystal structures in millimeter- to centimeter-thick samples with 3D spatial resolution of ~ 5 μm, with the sensitivity to detect the presence of grains as small as 150 nm. High-resolution CCD detectors play a critical role, as both the spatial projection and the angular orientation of individual grains in polycrystalline materials are collected on CCDs using Bragg reflections excited as the sample is rotated in the x-ray beam. This information is analyzed by computer and collated such that the 3D position, orientation, and elastic and plastic strains can be obtained for individual grains and grain boundaries. The isolation of size and orientation information for individual grains in polycrystalline materials is extremely powerful, as it makes it possible to perform *in situ* measurements of plastic deformation, grain nucleation, and grain growth in bulk materials. Quantitative measurements of this nature are providing new tests of theoretical models of processes ranging from deformation to nucleation and growth in polycrystalline materials. Three-dimensional x-ray microscopy instruments similar to the 3DXRD microscope developed at the ESRF have been built and are now operating at the HASYLAB (Hamburger Synchrotronstrahlungslabor) synchrotron facility in Hamburg, Germany, and at the APS.

The third article, by Ice and Larson, presents an overview of a polychromatic (i.e., white) x-ray microbeam technique that provides submicrometer 3D spatial-resolution measurements of the structure, orientation, grain size, morphology, and both elastic and plastic strain tensors in single crystals, polycrystals, composites, and deformed materials. Through the use of a platinum wire as a knife-edge profiler of white microbeam Laue diffraction patterns, this differential-aperture x-ray microscopy (DAXM) achieves submicrometer point-to-point intragrain and intergrain 3D spatial resolution. Examples are shown of micrometer-resolved measurements of local grain and subgrain orientations and morphologies in polycrystalline aluminum, micrometer-resolution spatially resolved strain tensor measurements in cylindrically bent silicon, and measurements of nanoindentation-induced deformation in copper. These capabilities provide a direct link to computer simulations and multiscale modeling investigations of polycrystal grain growth, plastic deformation, and microstructural evolution on mesoscopic length scales. This technique is currently optimized for energies of 8–25 keV, which yields depth ranges of tens of micrometers in high-Z materials to several hundreds of micrometers in lower-Z materials such as aluminum.

In the final article, Robinson and Miao address what might be referred to as the holy grail of 3D x-ray imaging microscopy, in the sense that it makes use of direct Fourier transformation of x-ray diffraction patterns to extract both the phase and amplitude of the illuminated sample. Although both practical and technological considerations limit this approach to sample sizes in the micrometer range, coherent diffraction imaging opens the possibility for structure determinations beyond the normal imaging and diffraction capabilities discussed in the other articles in this issue. Robinson and Miao show that when sample volumes smaller than the coherence volume of the beam are illuminated, the diffraction pattern contains detailed, full-field information on the structure of the sample, limited by the extent to which the full angular range of the diffraction patterns can be collected. While this technique is in an early stage of development, the fundamental importance of the method has attracted intense interest. This article demonstrates 3D coherent diffraction imaging first through an example

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of non-crystallographic (small-angle scattering) imaging of nanopatterned Ni structures with ~8 nm resolution and then through an example of (large-angle) coherent Bragg diffraction imaging of a micrometer-sized gold particle. Work using softer x-rays at the ALS has demonstrated 2D coherent diffraction imaging of artificially arranged 50 nm gold spheres, with 3D image reconstructions in progress.[4] These examples are just the beginning of what is certain to become an enormously rich field of microstructural research, as numerical analysis techniques, x-ray sources, and CCD detector technologies develop. Moreover, the effective NA associated with large-angle x-ray diffraction imaging as discussed in this article is, in principle, capable eventually of achieving atomic resolution.

While single-atom imaging has been reported for the case of double-walled carbon nanotubes using electron diffraction,[5] atomic-resolution imaging using x-rays has significant hurdles to overcome, including the requirement of x-ray beams with even higher brilliance than exist today, such as the so-called fourth-generation free-electron laser x-ray sources.[4] Although significant radiation damage issues are anticipated for biological structures, less problematic applications to inorganic micro- and nanostructured materials can be envisioned in ultrahigh-resolution, *in situ* investigations of processing-induced structural evolution.

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