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Radiation Facilities for Materials Science and Nuclear Beam Material Science

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Abstract

Synchrotron Radiation Facilities, supported by the Materials Science and Engineering Laboratory of the National Institute of Standards and Technology, include beam stations at the National Synchrotron Light Source at Brookhaven National Laboratory and at the Advanced Photon Source at Argonne National Laboratory. The emphasis is on materials characterization at the microstructural and at the atomic and molecular levels, where NIST scientists, and researchers from industry, universities and government laboratories perform state-of-the-art x-ray measurements on a broad range of materials.

Keywords: Ceramics; Materials Science; Metallurgy; Polymers; Semiconductors; Synchrotron Radiation; x-ray Instrumentation

INTRODUCTION

Synchrotron radiation sources provide intense beams of x rays for leading-edge research in a broad array of scientific disciplines. The National Institute of Standards and Technology (NIST) Materials Science and Engineering Laboratory (MSEL) program to characterize materials by means of this powerful probe began in the early 1980s, with the design, construction and commissioning of a monochromatic x-ray topography [1] station (X23A3) at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. The topography station, which was home to the only dedicated monochromatic topography instrument of its kind in the country, also included experimental facilities for hard x-ray microscopy [2] and parallel-beam x-ray microradiography. By 1989, an ultra-small-angle x-ray scattering (USAXS) instrument [3] had been added to X23A3, specifically for the purpose of enabling anomalous ultra-small-angle x-ray scattering. It was later used for a wide variety of USAXS measurements on ceramics [4], metals [5], polymers [6] and biomaterials [7].

Today, the NIST/MSEL synchrotron radiation program includes the utilization and operation of three additional beam stations at the NSLS. X23A2 serves a large x-ray absorption fine structure (XAFS), diffraction anomalous fine structure (DAFS), and x-ray spectroscopy community. It provides stable scanning of monochromatic x rays in the energy range from 4.9 keV to over 30 keV. X24A, which is a shared beam station, provides incident x rays in the 1.7 keV to 6 keV energy range, and is used by NIST researchers for standing-wave x-ray measurements. Finally, the U7A station, which is also a joint effort, utilizes x-ray photoemission spectroscopy (XPS) and near-edge x-ray absorption fine structure (NEXAFS) to study the structure and chemical nature of diverse materials from a surface and bulk perspective either in vacuum or under atmospheric reaction conditions. Taken together, these three NIST/MSEL beam lines at the NSLS provide continuously tunable x rays from 0.18 keV to over 30 keV, which covers spectroscopically nearly the entire periodic table.

Looking toward the future, NIST/MSEL became a partner in 1995 with the University of Illinois at Urbana/Champaign, Oak Ridge National Laboratory, and UOP LLC, in a collaboration designated "UNICAT," to design and construct x-ray instrumentation for sector 33 at the Advanced Photon Source (APS) at Argonne National Laboratory. The APS is present-

ly one of the premier synchrotron x-ray sources in the world. In 1998, a new NIST USAXS facility was installed and commissioned on the sector 33 undulator beam line. In 2001, the next generation monochromatic topography and XAFS began commissioning on the sector 33 bending magnet beam line. The emphasis throughout is on state-of-the-art x-ray measurements on advanced materials. Today, the combined portfolio of NIST facilities with our partners at the NSLS and with UNICAT at the APS offers measurement capabilities in ultra-small-angle x-ray scattering, high-resolution x-ray topography, hard and soft XAFS, and standing-wave x-ray diffraction. It also offers access to leading edge instruments for structural crystallography and time-resolved structural scattering, surface and interface scattering, diffuse scattering, and magnetic scattering.

This paper is a highly selective review, by members of the current Materials Microstructure Characterization Group, of the past two decades of research efforts of the scientists who built these facilities and currently are responsible for their operation. The emphasis is on the broad range of science performed by NIST researchers and others, enabled by facilities that were designed, constructed, commissioned and operated by NIST. Many additional contributions of colleagues and collaborators can be found in the references and acknowledgements at the end.

NIST programs at the National Synchrotron Light Source

Monochromatic Synchrotron X-Ray Topography

From 1984, until it was closed in 2000, X23A3 was a premier monochromatic x-ray topography facility. X-ray topography is a nondestructive characterization technique for imaging, by means of x-ray diffraction, the micrometer-sized to centimeter-sized defect microstructure of crystals. The NIST Group's early research concentrated largely on crystal growth and crystal perfection as a function of processing. The power of the monochromatic topography instrument was the result of the early realization [8] that it is possible to design and build topographic optics to record images of the entire sample in a single exposure. While the first topography camera was built in the laboratory, similar principles were used in the design of the X23A3 instrument. Early studies included the observation of magnetic domains within single-crystal grains of pure nickel. The formation of domain walls near the crystal surface was also observed (Fig. 1). Later on, the excellent resolution of the instrument—of the order of 1.7 μm—enabled detailed studies of bismuth-silicon-oxide (BSO). Three crystal slices from single boule of BSO were examined, and the progression of microscopic strain as a function of growth was documented [9]. Remarkably, all of the features that were recorded (Fig. 2) were growth striations that could be directly related to the growth habit of the crystal boule. Recent work has included topographic examination of biological crystals grown in space [10]. One of the outstanding questions in protein crystal growth is the effect of gravity on the growth process. The crystallization of proteins can be strongly affected by gravity through sedimentation and convection. With the minimization of these processes, through growth in microgravity, will it become possible to produce better quality crystals? To answer this question, a comparison was made between ribonuclease crystals grown on the space shuttle and control crystals grown at the same time on Earth. A comparison of x-ray topographs recorded from Earth-grown and space-grown crystals is shown in Fig. 3. The space crystal has symmetry about the vertical and horizontal axes consistent with growth sectors. As the Bragg angle is changed, the four dark regions indicated migrate together toward or away from the center of the crystal, consistent with a central nucleation point followed by homogeneous growth. The microstructure of the Earth-grown crystal is less well defined, consistent with a higher defect density. The topography results were able to delineate some of the ways in which the minimization of gravity improves the quality of space-grown crystals.

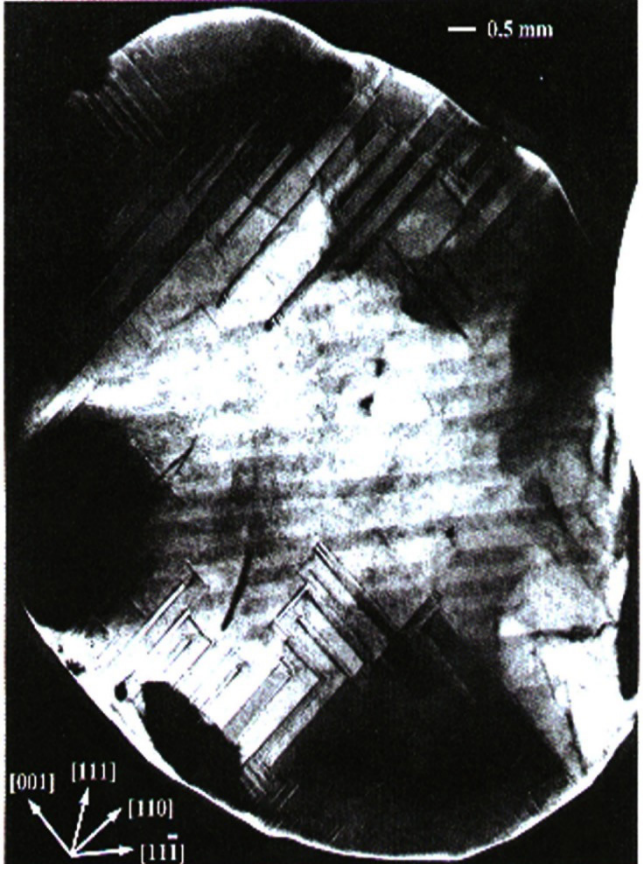

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Fig. 1: A 111^{-} transmission topograph of a Ni crystal showing 71°, 109°, and 180° magnetic domains, which intersect the surface in straight, lines along [001], [110], and $[111^{-}]$ respectively.

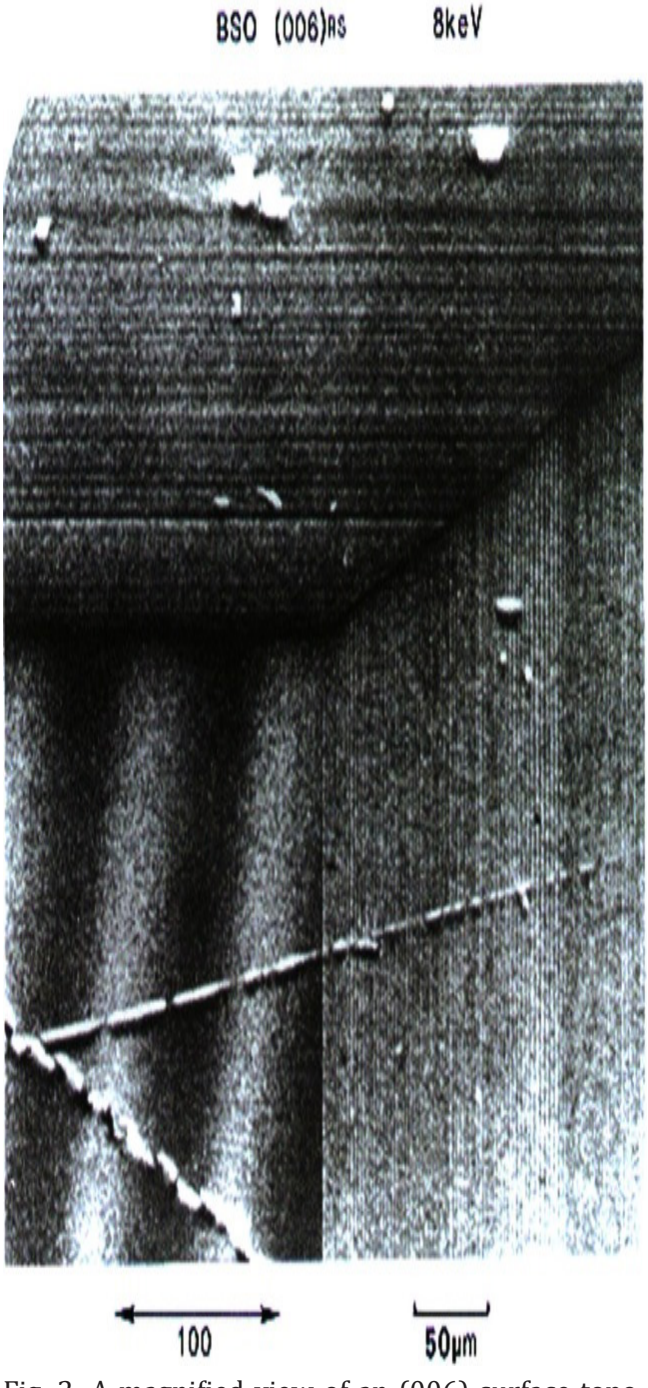

Fig. 2: A magnified view of an (006) surface topograph from a BSO crystal showing the central (001) growth sector and the peripheral (101) and (010) growth sectors. (Also visible are scratches attributable to handling damage.)

Ultra-small-Angle X-Ray Scattering

The X23A3 beam line was also host to an ultra-small-angle x-ray scattering facility [3,11]. This instrument offered high angular and wavelength resolution, large sample cross sectional area, accurate energy tuning, excellent signal-to-noise ratio at ultra-small scattering vectors, and primary calibra-

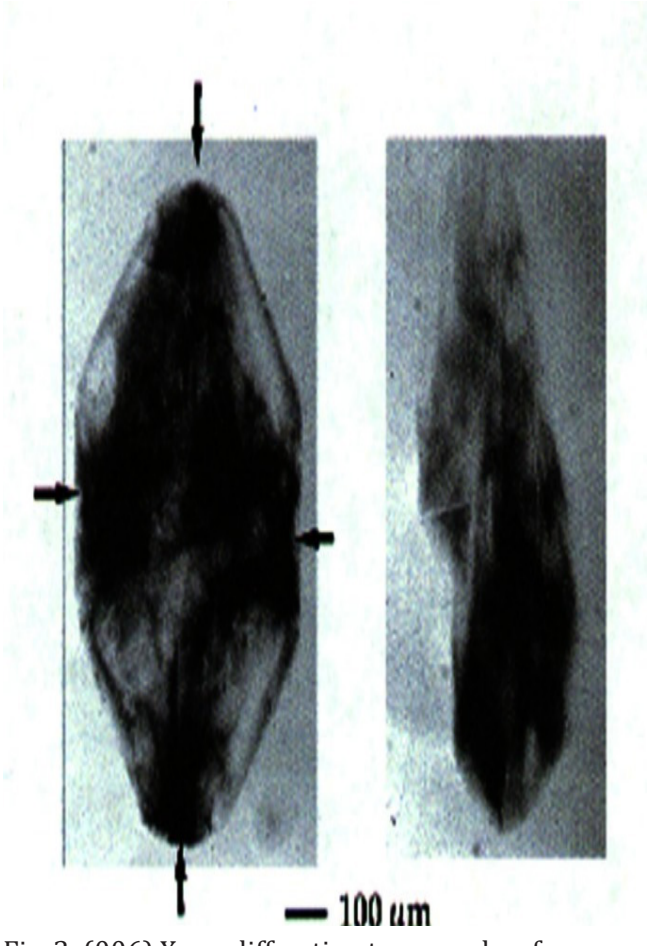

Fig. 3: (006) X-ray diffraction topographs of a space-grown crystal on the left, and an earth-grown crystal on the right. The space crystal has symmetry about the vertical and horizontal axes consistent with growth sectors. As the Bragg angle is changed, the four dark regions indicated together migrate toward or away from the center of the crystal, consistent with a central nucleation point followed by homogeneous growth. The microstructure of the earth crystal is less well defined, consistent with a higher defect density.

The absolute intensity. The scattering vector range available with this instrument served to bridge the gap between visible light scattering and standard x-ray scattering instruments. Early experiments included the quantitative determination of the microstructure of porous silica precursor bodies [12], which later led to a characterization of porosity over nearly three decades in length scale [13]. Porous materials are used in a wide variety of engineering applications, including super insulators, membranes, catalyst supports, and porous hosts for second-phase filtration. Because microstructure and performance are strongly related, the realization of different applications depends on the ability to produce porous media with specific pore sizes and pore-size distributions. The sol-gel fabrication route has been used for many polymer-based materials schemes because of the flexibility it offers to

create different microstructural products. In the study of sol-gels described here, USAXS measurements on X23A3 were complemented by electron microscopy and small-angle neutron scattering measurements to quantify the microstructural features within porous silica gels. The size distributions were derived from a maximum entropy analysis of the scattering data (Fig. 4). The materials had been prepared from 20 % mass fraction colloidal silica and 80 % mass fraction potassium silicate. These precursor bodies are typically only 15 % to 20 % of theoretical density, and before densification, they are leached to remove the alkali content. Fig. 4 compares the microstructure of leached and unleached samples. The most striking feature in this derived size distribution is a narrow peak between 2 nm and 3 nm. This feature is evident in the results from both the unleached and the leached samples. It is attributed to nanometer-scale porosity in the leached samples and is attributed to similarly sized alkali-silicate-rich regions in the unleached samples. The increased scattering contrast of silica-to-void compared to silica-to-silicate accounts for the increased intensity in the scattering of the leached sample. At large diameters, there is a peak at nearly 55 nm, which represents the silica clusters. The unusual observation of skeletal voids within the solid phase, and solid clusters within the void phase in the same material is a consequence of the extraordinary variety of microstructures in these gels.

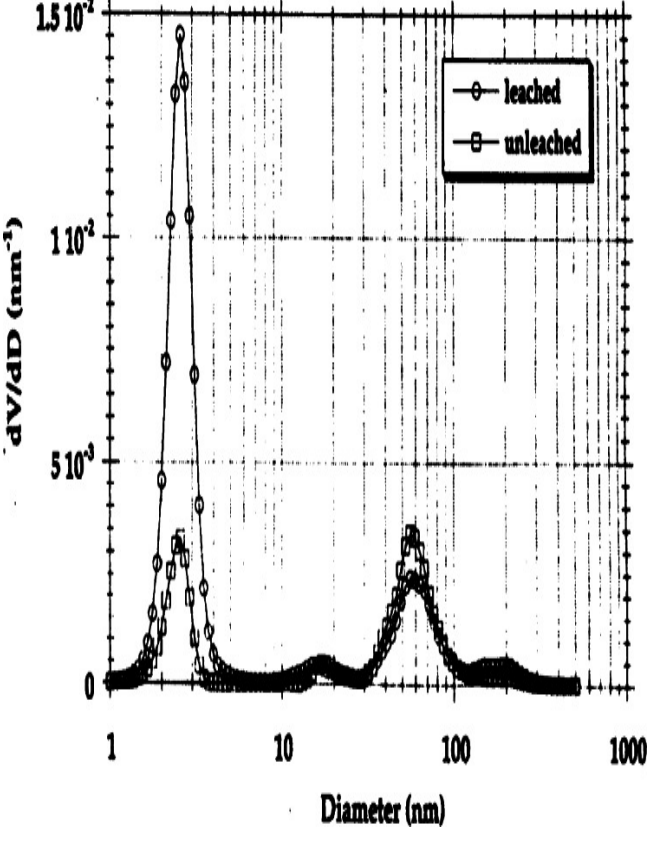

Fig. 4: Volume distributions as a function of diameter of microporous silica, derived from USAXS and SANS data from leached and unleached material. The peak at 2.5 nm represents the population of skeletal voids in the leached material, or the alkali-silicate-rich regions in the unleached material. The peaks at ≈55 nm represent the populations of silica clusters.

In another USAXS study, the extraordinary sensitivity of the X23A3 USAXS instrument enabled the in situ observation of x-ray scattering from dislocation structures during the deformation of single crystal aluminum [14]. Attempts over the years to measure small-angle scattering from dislocation structures have mostly met with limited success for various reasons. One of the earliest recognized reasons was the intrusion into the data of double-Bragg diffraction. Another problem was surface scattering from improperly prepared surfaces. Finally, unless the scattering vector is nearly perpendicular to a dislocation line, no appreciable scattering is seen. By avoiding double-Bragg diffraction conditions, meticulous sample surface preparation, and by orienting the samples for dislocation visibility, it was possible to observe: 1. correlations in the ordered fraction of dislocations, 2. the presence of dislocation dipoles, 3. the increasing dislocation content with increasing strain, and 4. the decreasing width of the interface between the dislocation walls and the surrounding, nearly-dislocation-free material. In Fig. 5, the increasing dislocation content with increasing strain can be seen directly from the slit-smeared USAXS from single-crystal aluminum at strains of 0.4 %, 0.9 %, 2.2 %, 3.6 %, 4.4 %, 5.3 %, and 6.7 %.

Conclusions

In 1984, NIST's synchrotron facilities were inaugurated with the commissioning of a single beam station at the NSLS for x-ray topography. Today, the combined portfolio of NIST/MSEL facilities with our partners at the NSLS and with UNICAT at the APS offers measurement capabilities in ultra-small-angle x-ray scattering, high-resolution x-ray topography, hard and soft XAFS, and standing-wave x-ray diffraction. It also offers access to leading edge instruments for structural crystallography and time-resolved structural scattering, surface and interface scattering, diffuse scattering and magnetic scattering. The NIST synchrotron facilities enable researchers to use these intense x-ray sources for the advancement of science and technology in materials, physics, chemistry, and, most recently, biology. The primary thrust is the application of leading-edge x-ray scattering and imaging to a broad range of scientific problems and the continued development of novel techniques for innovative research.

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