Lawrence Berkeley National Laboratory

Recent Work

Title

Workshop on application of X-ray microdiffraction to materials and environmental Sciences

Permalink

<https://escholarship.org/uc/item/2ps289wn>

Journal

Synchrotron Radiation News, 16(1)

ISSN

0894-0886

Author

Tamura, N

Publication Date

2003

DOI

10.1080/08940880308602991

Peer reviewed

Workshop on Application of X-ray Microdiffraction to Materials and Environmental Sciences

Nobumichi Tamura

To cite this article: Nobumichi Tamura (2003) Workshop on Application of X-ray Microdiffraction to Materials and Environmental Sciences, , 16:1, 6-7, DOI: [10.1080/08940880308602991](https://doi.org/10.1080/08940880308602991)

To link to this article: <https://doi.org/10.1080/08940880308602991>

Published online: 23 Sep 2008.

Submit your article to this journal [↗](#)

Article views: 7

ALS Users Meeting and Workshop

Workshop on Application of X-ray Microdiffraction to Materials and Environmental Sciences

The Workshop on Application of X-ray Microdiffraction to Materials and Environmental Sciences was held on October 12, 2002. It was organized by Nobumichi Tamura (Berkeley Lab), King-Ning Tu (University of California, Los Angeles), and Jim Patel (Berkeley Lab and Stanford Synchrotron Radiation Laboratory). It is the second workshop on X-ray microdiffraction to be held during an ALS Users Meeting.

The first workshop in October 2000 was intended to demonstrate the potential of using X-ray microdiffraction as a microstructural characterization tool and to attract new users to the then-recently commissioned X-ray microdiffraction end station on Beamline 7.3.3. The station was then officially opened to the general users in December 2001. Today, 60 percent of the time on this beamline is allocated to X-ray microdiffraction, while the remaining 40 percent is taken up by high-pressure research.

The intent of this second one-day workshop was to give an overview on the diversity of the scientific projects carried out at the ALS microdiffraction facility. The layout of the station consists of a four-crystal monochromator (allowing use of either white or monochromatic radiation), Kirkpatrick-Baez focusing optics capable of placing a sub-micron X-ray beam on the sample, scanning sample stages, a large-area CCD detector to collect diffraction patterns, and a solid-state detector to collect fluorescence signals. Specific software routines have been developed to automate the data collection and analysis.

The applications range from the study of the mechanical properties of composite materials and microelectro-mechanical systems (MEMS) devices, to the characterization of biomaterials and superconducting films, to en-

vironmental and earth sciences. They can be classified into three major categories according to the scientific goals:

1. Understanding microstructure dependent material properties such as strength and fatigue resistance at granular (mesoscopic) level (composites, etc.);
2. Measuring spatially resolved texture and strain/stress in very small devices during in-situ service conditions (microelectronics, etc.); and
3. Characterizing in a non-invasive way complex heterogeneous samples (soils, etc.)

In the first talk of the morning session, Alain Manceau (Joseph-Fourier University, Grenoble, France) showed how a combined and synergistic use of four non-invasive synchrotron-based techniques (X-ray microfluorescence [μ SXRF], X-ray microdiffraction [μ XRD], Scanning X-ray microdiffraction [μ SXRD], and X-ray absorption spectroscopy [EXAFS and μ EXAFS]) can be used to understand sequestration mechanisms of trace metals in soils and sediments. This promising approach provides a solid scientific basis and educated strategies for the cleaning of contaminated soils [1].

Bryan Valek (Stanford University) introduced the technique of white-beam μ SXRD, which allows mapping grain orientation and strain/stress in materials with submicron spatial resolution. He then showed the technique's use to study the local mechanical behavior during a thermocycling experiment of aluminum thin films deposited on silicon and the effect of current flow on the microstructure of aluminum interconnects during an in-situ electro-migration experiment [2].

Studies conducted with the aim of completely eliminating lead from the electronic packaging industry have showed that tin and tin compounds offer a cost-effective alternative for solder joints and surface finish. Tin is, however, known to spontaneously grow whiskers, thereby greatly affecting the reliability of lead-free circuits. Woojin Choi (University of California, Los Angeles) demonstrated by a combined use of FIB, transmission electron microscopy, and white-beam μ SXRD the microstructural mechanisms underlying the spontaneous growth of tin whiskers on a tin-finished copper-lead frame [3].

Ferroelectric ceramics are widely used in devices such as actuators, MEMS, and sensors. In an effort to understand and accurately model the electromechanical behavior of these materials, Ersan Üstündag (California Institute of Technology) is conducting an active research program to study them with μ SXRD. In particular, he was able to measure for the first time the elastic strain field around individual domains in BaTiO_3 , thereby providing the experimental data for the refinement of micro-mechanical models [4].

PVD deposited metallic thin films often exhibit high compressive stresses, resulting in the spontaneous formation of bucklings and wrinkles. Philippe Goudeau (University of Poitiers, France) applied monochromatic μ SXRD to measure the stress field on and around individual bucklings of nanocrystalline metallic films of gold, molybdenum, or tungsten on a silicon substrate. Such measurements yield experimental assessments to finite element calculations [5].

In the first talk of the afternoon session, Tamura showed how μ SXRD could give new insights into understanding the mechanical properties of polycrystalline thin films. Plas-

**G-Chamber of SGM for U-10 Beamline
at Pohang Light Source in Korea
(Energy Range : 10~1000eV using 6 gratings)**

Supporting cutting-edge science with the best in ultra-high vacuum & precision machining technology.

If you have a good concept that needs to be developed, TOYAMA is the place to come!

We have the technology to turn your ideas into reality.

Main Products:

- Beamline Components
- Monochromators
- Mirror Positioners
- Mirror Bending Mechanisms
- UHV Systems
- Standard Feedthroughs

Pioneering New Horizons in Science

TOYAMA Co., Ltd.

13-16, Hibarigaoka 4-chome, Zama-shi,
Kanagawa Prefecture, 228-0003, JAPAN
Tel: +81(46)253-1411 Fax: +81(46)253-1412

E-Mail: salesdept @toyama-jp.com

Internet Home Page: <http://www.toyama-jp.com>

ticity at the level of individual grains appeared to be highly complex, and the final local stress state of the film was shown to strongly depend on its local environment (first-neighbor grains). Grains deformed in a non-homogeneous way lead to the concept of an "effective" grain size that is smaller than the actual grain size of the material [6].

Sander Caldwell (ALS) showed how X-ray microdiffraction could be a quick and effective alternative to reciprocal-space mapping for the characterization of thin-film microstructure. From a single Laue pattern of a $\text{YBa}_2\text{Cu}_3\text{O}_x$ superconducting film deposited on SrTiO_3 , he derived its twinning properties. White beam μSXR D was then used to determine the distribution of twins inside the film.

In an effort to assess existing strain gradient theories, Monica Barney (Lawrence Liver-

more National Laboratory) conducted μSXR D measurements on single-crystal aluminum while it was being subjected to pure shear. She presented preliminary results, showing evidence of subgrain formation and rotation.

The workshop ended with a round-table discussion to assess the needs of the X-ray microdiffraction user community for a fully dedicated microdiffraction beamline, to review funding issues, and to discuss future upgrades for faster data collection and analysis, as well as better spatial resolution and strain sensitivity. ■

NOBUMICHI TAMURA

Advanced Light Source, Berkeley Lab

References

1. A. Manceau et al., *American Mineralogist* **87** (2002) 1494.

2. B. C. Valek et al., *Appl. Phys. Lett.* **81** (2002) 4168.
3. W. J. Choi et al., Proceedings of the 52nd IEEE Electronic Component & Technology Conference, 28-31 May 2002, San Diego, CA, IEEE Catalog number 02CH3734-5 (2002) 628.
4. R. C. Rogan, et al., *Appl. Phys. Lett.* (2002) submitted.
5. P. Goudeau, P. Villain, N. Tamura, R. S. Celestre, and H. A. Padmore, *J. Phys IV France* **12** (2002) 409.
6. R. Spolenak et al., *Phys. Rev. Lett.* (2003) in press.