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Preface

Probing strains and dislocation gradients with diffraction

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This collection of special topics papers Materials Science and Engineering A highlights novel methods for the analysis of strain and dislocation gradients using X-ray, neutron and electron diffraction. The development of ultra-brilliant synchrotron X-ray sources and recent developments in neutron diffraction, provide new opportunities for the analysis of inhomogeneous strain and dislocation gradients on length scales from nanometers to millimeters. These new developments compliment powerful Electron Back Scattering techniques that are themselves rapidly developing.

The special topic starts with a paper by Ice et al. that describes recent progress in polychromatic X-ray microdiffraction (PXM). The paper has an emphasis on opportunities to further improve spatial, temporal and momentum transfer resolution. As described in the paper, the smaller the focused beam size, the smaller the volume probed and the higher the spatial resolution in all three dimensions. The current performance and possible upgrades to station 34-ID-E at the Advanced Photon Source (APS) were described since this station is the only microdiffraction station currently able to resolve crystal structures with submicron resolution in all three dimensions. The authors further discussed ultimate limits set by sample damage, and the possibility of merging polychromatic microdiffraction and coherent imaging techniques.

Example applications of polychromatic X-ray microdiffraction (PXM) are presented for several technologically important problems in the following five papers of Barabash et al., Jing Chao et al., Tamura et al., Magdid et al., and Swygenhoven et al.

The paper of Barabash et al. describes a combined PXM and Electron Back Scattering Diffraction (EBSD) study of structure and phase transitions induced in a Ni-based superalloy during friction stir processing. The combination of PXM and EBSD methods allowed the authors to confirm the existence of vortex deformation modes in the stir zone, and determine lattice rotations, the formation of geometrically necessary dislocations and strain gradients in the narrow boundary region between the thermal–mechanical and stir zones.

Comparison between the results of PXM and neutron residual elastic- and plastic-strain measurements in uniaxial tensile strained nickel-based Alloy 600 samples is given in the paper Jing Chao et al. The paper is focused on local strain effects in polycrystalline Alloy 600 introduced by tensile extension or by placing calibrated scratches in the surface. The authors conclude that PXM and neutron diffraction measure different strain and strain gradient tensor components.

A superbend X-ray microdiffraction beamline at the Advanced Light Source (Berkeley) is described in the manuscript of Tamura et al. Several example measurements of strain/stress in engineered materials demonstrate the current status of the beamline hardware and software.

Polychromatic microdiffraction studies at the Swiss Light Source are described in the papers of Magdid et al. and Swygenhoven et al. In the paper of Magdid et al. the low-temperature grain rotations in polycrystalline thin films are understood as disclination–dislocation interactions. High temperatures – electromigration induced grain rotation and hillock growth are considered as the materials reaction to stress gradients involving diffusive flux and dislocation climb.

Latest developments in the in situ Laue diffraction of metallic micropillars during micro-compression tests are described by Swygenhoven et al. using a transmission geometry with a polychromatic beam. The “smaller is stronger” problem is discussed in connection with in situ pillar tests under different boundary conditions.

Complementary methods using monochromatic radiation has been an ongoing research in several scientific groups. The current work performed at APS I-ID beamline is presented by the manuscripts of Lienert et al., and by the paper of Wejdemann et al., Lienert et al. describe in situ single grain peak profile measurements on Ti–7Al during tensile deformation. The method allows determination of the average strain tensors in individual grains as well as intra-granular strains and misorientations.

The paper of Wejdemann et al. describes the analysis of deformation structures under varying loading conditions followed by in situ high angular resolution Three-Dimensional X-ray Diffraction (3DXRD). The dislocation cell and wall structure is probed by monochromatic beam while rocking the sample in a relatively narrow angle range. The intensity maxima in split rocking curves are assumed to correspond to different cell interior. Peak positions fluctuate from cell to cell due to formation of inhomogeneous internal strains.

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Moscicki et al. exploited Friedel-pair based indexing method of individual grains in polycrystals investigated with hard X-rays. This method is complementary to existing indexing methods used for 3DXRD. The authors use the symmetry properties of the Friedel pairs enabling a clear separation of the contributions from grain orientation and position as well as average strain.

The further development of the above method performed at European Synchrotron Radiation Facility (ESRF) opened new opportunities for 3D materials science of polycrystalline materials at the micrometer length scale by combined use of X-ray diffraction and X-ray imaging (paper Ludwig et al.). Using a monochromatic X-ray beam, both the direct and diffracted beams are registered on a single detector located behind the sample. Further reconstruction allows imaging the shape of individual grains.

Zolotoyabko et al. used high-resolution neutron powder diffraction in order to accurately measure bond-length difference between the mollusk-made and geological calcium carbonate. This approach allowed the authors to identify specific bonds which are significantly affected by organic–inorganic interactions. The maximal bond change reached about 1% for both mollusk-made and geological calcium carbonate samples. The above change of the bond length can modify frequencies of normal vibrations measured by Raman or FTIR techniques.

The possibilities of depth-resolved measurements using laboratory diffractometry is presented in the manuscript of Krost et al. The group of Prof. Krost designed a convergent beam, concurrent detection X-ray diffractometer, as well as the grazing incidence diffractometer. Adjusting the line focus of the conventional sealed X-ray source in the plane of the wafer surface allows depth-dependent measurements at high intensity. These diffractometers were used for micrometer scale characterization of group-III nitrides.

The collection of papers is a sampling of the ongoing research at scientific laboratories around the world which probes strains and dislocation gradients in novel materials. The combination of small beams, high flux synchrotron radiation, new neutron techniques and advanced computer reconstruction, allowed researches to obtain unique information about deformation mechanisms at different length scales.

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