Three-Dimensional High-Energy Diffraction Microscopy of Polycrystalline Bulk Materials

U. Lienert[1], J. Almer[1], B. Jakobsen[2], W. Pantleon[2], H.F. Poulsen[2], D. Hennessy[3], C. Xiao[3], R.M. Suter[3]
[1]Argonne National Laboratory, Argonne, IL U.S.A.;[2]Risø National Laboratory, Roskilde, Denmark.;
[3]Carnegie Mellon University, Pittsburgh, PA, USA

Introduction

The microstructure of polycrystalline materials is characterized by a hierarchical arrangement of crystalline elements (grains, microbands, and subgrains). The arrangement is often highly heterogeneous, especially with respect to dynamics during processing. Conventional experimental methods either lack sufficient spatial resolution or are surface or thin foil probes. To obtain three dimensional spatially resolved results with these latter techniques requires that samples be sectioned. This destructive procedure prohibits studies of the dynamics of the individual crystalline elements. Thus, there is a need for a nondestructive method that provides comprehensive structural information for each of the crystalline elements within macroscopic volumes of the material. Furthermore, the method should be sufficiently fast to record the dynamics of 10–1000 elements simultaneously during processing.

Three-dimensional x-ray diffraction (3DXRD) microscopy is an emerging method that aims to fulfill these requirements [1]. The method is distinguished by two principles. The first is the use of a beam of high-energy x-rays generated by a synchrotron source for transmission studies. Hard x-rays (in the range 50–100 keV) can penetrate 4 cm of aluminum or 5 mm of steel. The second principle is a "tomographic" approach to diffraction. The conventional approach for providing spatially resolved information with diffraction is to scan the sample with respect to the beam. However, probing the sample point-by-point is generally too slow for dynamic studies. Hence, it has been replaced by an approach that provides information on many parts of the material simultaneously.

Here we report on activities establishing 3DXRD capabilities at the Advanced Photon Source (Argonne National Laboratory) within the high-energy program at beamline 1-ID.

High energy x-ray diffraction microscopy [2]

High energy x-ray diffraction microscopy non-destructively maps the internal microstructure of bulk polycrystalline materials. It offers the opportunity to study the response of ensembles of grains to external stimuli such as heat or stress.

Data are collected using 50keV x-rays focused in the vertical direction to a line 1.5 – 2.5 μm high. A bent Si Laue crystal or refractive optics are available for focusing. A high resolution CCD detector images diffracted beams at three distances as the sample is rotated. The shapes of diffraction spots are, ideally, projections along the diffracted beam of the shapes of diffracting grains.

A forward modeling approach has been developed to reconstructing microstructure. Raw CCD images are treated for noise reduction and diffraction spot identification. Each spot is thresholded at a fraction of its peak height to generate a binary data set. A simulation program then attempts to define the microstructure. The sample plane is covered by a triangular grid. The crystallographic orientation of each area element is adjusted and optimized by a Monte Carlo process in an attempt to maximize overlap of its Bragg diffraction with experimentally observed intensity. An iterative procedure refines the area elements and focuses on boundary regions where the orientation changes rapidly.

In a verification exercise, it has been shown that a well-defined subarea of a silicon single crystal can be reconstructed to about 10-μm resolution. Furthermore, six successive layers within an Al-1050 alloy have been reconstructed.

High resolution reciprocal space mapping [3]

When metals deform plastically, dislocations are introduced. Within existing grains, these self-organize into dislocation structures (subgrains) with a size of about 1 μm. With increasing external load the subgrains become progressively smaller – a phenomenon known as scale refinement. We have recently developed a novel ultra high angular resolution (0.004°) 3DXRD technique which allows investigation of bulk subgrains, and hence monitoring the dynamics on this length scale, something which previously has been impossible at least on bulk samples. The technique is based on the observation, that high resolution reciprocal space maps of broadened diffraction spots from a deformed grain show a remarkable structure, consisting of bright sharp peaks on top of a cloud of enhanced intensity. The peaks are interpreted as diffraction arising from individual subgrains, whereas the cloud tentatively is interpreted as coming from the dislocation filled areas (walls) between the subgrains.

Remarkably, no formation of new boundaries within subgrains have been observed with increasing strain. The subgrains rather materialize temporarily as dislocation-free islands in a sea of dislocations. From observed peak spliting it is concluded that the technique might give information about single dislocation events.

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