Materials-microstructure Measurements with Submicrometer Resolution in 3-D

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Introduction

Almost all technological materials and advanced processing techniques are based on the generation and control of nonhomogeneous microstructural features, such as precipitates and grain boundaries. Until now, however, nondestructive, *point-to-point,* 3-D structural resolution in the critical micrometer- and smaller-resolution range has not been available to investigate structure and evolution in bulk materials. As a result, fundamental investigations of 3-D mesoscale phenomena, such as intergranular grain-growth, intra- and intergranular deformation, and strain-gradient effects, have had to rely on extrapolations from 2-D measurements and computer simulation and modeling. Although the capabilities for grain-average or grain-centroid measurements approaching micrometer resolution [1, 2] and 3-D structural measurements with >10-µm resolution [3] have been available for some time, nondestructive, 3-D experimental observations with point-to-point micrometer resolution required to spatially resolve the details of microstructural evolution processes have been missing.

Methods and Materials

The generation of submicrometer-diameter polychromatic microbeams by using elliptical Kirkpatrick-Baez x-ray mirrors in connection with third-generation synchrotrons and the development of automated Laue diffraction analysis software have made submicrometer-resolution, 2-D, structural microscopy possible in thin films where depth resolution is defined by the film or buried layer thickness [4-7]. Through the development of submicrometer-resolution diffracted-beam profiling capabilities on the Michigan-Howard-Lucent Technologies-Bell Labs Collaborative Access Team (MHATT-CAT) and the University-National Laboratory-Industry (UNI)-CAT beamlines at the APS, we have developed the differential-aperture x-ray microscopy (DAXM) technique for performing structural microscopy measurements with submicrometer spatial resolution in 3-D [8]. The DAXM technique achieves micrometer depth resolution by exploiting knife-edge step-profiling and charge-coupled device (CCD) x-ray area detection of Laue diffraction patterns in a process analogous to that of a translating pin-hole camera. Computer collation and reconstruction of the depth-resolved Bragg diffraction data then make it possible to extract *complete Laue diffraction patterns* as a function of depth along the microbeam. With the depth dimension confined to a micrometer or less by the profiling analysis, the diffraction pattern analyses can then be performed by using previously developed (2-D) computer indexing and crystallographic analyses software [5, 6]. Therefore, through the use of ~0.5-µm-diameter microbeams and submicrometer depth profiling along the beam, *full diffraction information* can be obtained from submicrometer voxels in single-crystal and polycrystalline materials, deformed materials, composites, and functionally graded materials.

Results

Figure 1 shows 3-D measurements of grain structure in hot-rolled polycrystalline aluminum made by using the DAXM technique on the UNI-CAT beamline. Changes in color denote changes in orientation, defining the dimensions of grains and the positions of grain boundaries and triple junctions with micrometer resolution in 3-D. The angular resolution of the local orientation measurements is a few hundredths of a degree. Since typical intragranular orientation fluctuations in this sample [Al 1% (Si,Fe) hot-rolled at 200°C] were found to be substantially less than one-tenth of a degree, many regions appear to be fully recrystallized. However, the data analysis process is still in progress. Although these data show only a portion of the 3-D data collected, they demonstrate the capability of point-to-point micrometer-resolution structure and orientation measurements that are needed to provide a direct link between the *actual* microstructure and evolution in materials and the results of numerical simulations and modeling over the range of mesoscopic length scales.

Discussion

The DAXM method is applicable to a wide range of inter- and intragranular materials microstructures and local stress/strain investigations, including fundamental studies of grain growth and evolution in bulk materials, fracture, and plastic deformation. Micrometer-resolution inter- and intragranular grain-growth investigations are presently in progress that are complementary to grain-average, real-time nucleation and growth investigations reported by Lauridsen et al. [9].

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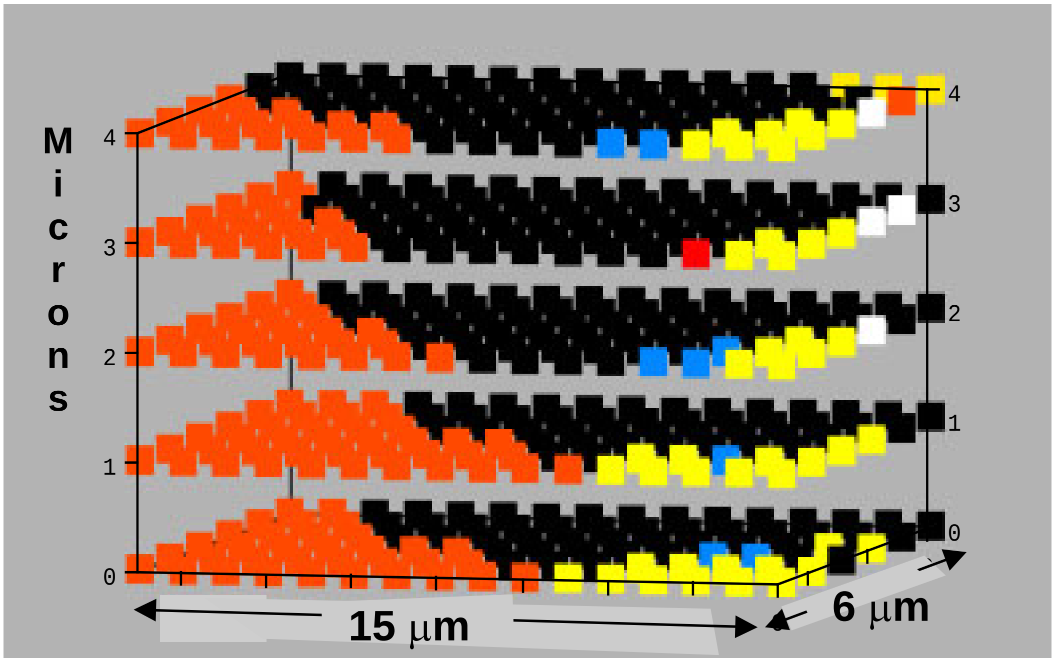

FIG. 1. Color-coded mapping of the orientation of crystal grains in polycrystalline aluminum with micrometer resolution in 3-D.