In situ X-ray Peak Shape Analysis of Embedded Individual Grains during Plastic Deformation of Metals

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Introduction

Three-dimensional x-ray diffraction (3DXRD) microscopy is an emerging tool for structural characterisation in materials science. Based on the use of highly penetrating hard x-rays and a "tomographic" approach to diffraction, the method enables a nondestructive 3-D description of the microstructural evolution within polycrystals. Under favourable conditions, hundreds of embedded grains can be characterized simultaneously with respect to position, shape, volume, crystallographic orientation, and average strain tensor. The 3DXRD method is extended to the generation of reciprocal space maps of a set of reflections, all of which arise from the same embedded grain [1]. Each of these 3-D reciprocal space maps has information about the intensity distribution as function of Bragg angle 2θ (i.e., the radial peak profile) as well as a complete description of the mosaic spread of the peak (i.e., the azimuthal spreading).

In comparison to conventional macroscopic peak profile investigations, the 3DXRD-based method has several advantages. First, the measurements are specific to each grain. As such, the amount of averaging over various spatial heterogeneities is substantially reduced. The importance of such variations can be judged by comparing results from different grains. Likewise, the macroscopic results are based on ensembles of grains that have parallel reflecting lattice planes but different orientations around the lattice plane normals. Second, instead of measuring a few 1-D profiles, tens to hundreds of 3-D distributions are available for each grain, one for each reflection characterized. Hence, the information content in the data is substantially enhanced. The feasibility of the 3DXRD-based method of peak shape analysis is demonstrated on a polycrystalline Al specimen subjected to tensile strain. The data presented are the first true-bulk results for the plastic response of individual grains [1].

Methods and Materials

The experiment was performed at X-ray and Operations Research beamline 1-ID at the APS. A flat Si(311) double-crystal monochromator provided a collimated beam of 30-keV x-rays with a narrow bandwidth ($\Delta E/E$ of $<0.5 \times 10^{-4}$). A 1-mm-thick tensile specimen of commercially pure aluminium (AA 1050) was mounted in a displacement-controlled stress rig, which, in turn, was installed on a three-circle goniometer.

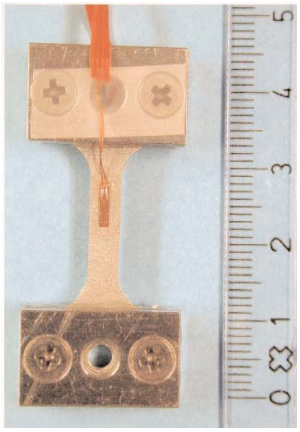

FIG. 1. Tensile specimen of commercial aluminium AA1050 after 4.5% tensile deformation with mounted strain gauge and specimen holders. A metric ruler is shown for comparison.

For characterization of individual peak shapes with respect to the reciprocal space parameters, a medium-resolution charge-coupled device (CCD) was positioned a large distance (about 3 m) from the specimen. The instrumental resolution in the radial (2θ) direction was estimated from a LaB_6 powder calibration peak to be $\Delta d/d$ of $\approx 10^{-4}$. In addition, a large-area CCD was positioned closer to the sample. This detector sampled all low-index diffraction spots arising from all illuminated grains. The multi-grain indexing program GRAINDEX [2] sorted the diffraction spots acquired during sample rotation around the tensile axis according to the grain from which they originated and subsequently determined the orientation of the grains. One grain with a size of about 100 μm, positioned near the center thickness of the specimen, was selected and centered with respect to the beam and goniometer. This grain was found initially to have its [4 11 3] direction along the tensile axis.

The selected grain was characterized *in situ* under tensile loading to strains of 0.01%, 1%, 2.5%, and

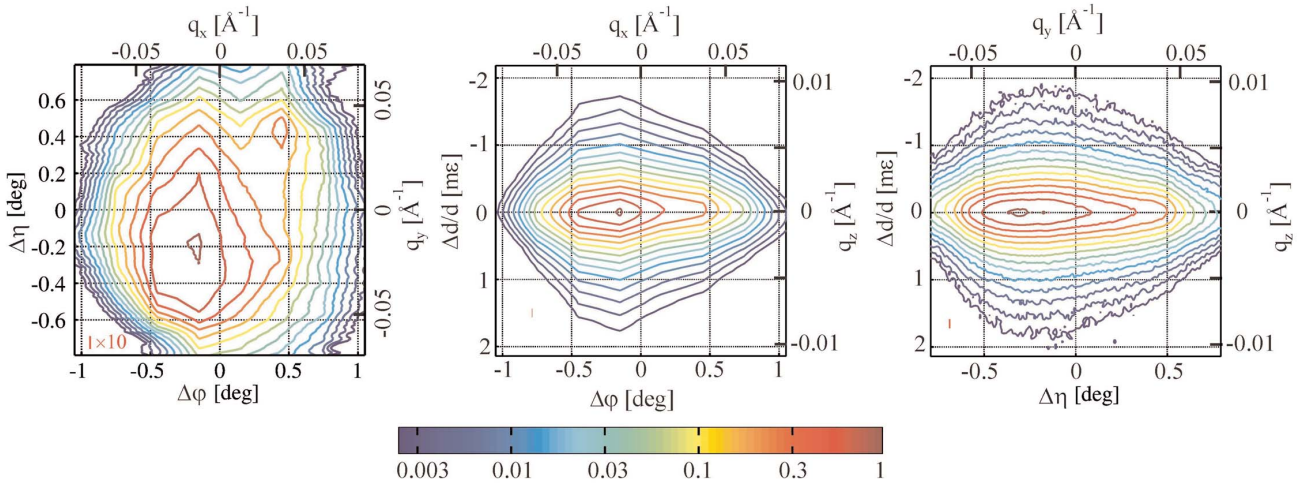

FIG. 2. Reciprocal space projections for the (-113) reflection of a single embedded Al grain at a strain of 2.5%.

4.5%. The grain was always fully illuminated by the synchrotron beam. At each load, the orientation of the grain was determined on the basis of data acquired with the closer CCD. A fixed set of 20 reflections was characterized with the more distant CCD (by choosing appropriate specimen tilts and adjusting the vertical position of the detector). Among these reflections from five different {hkl} families were six {311} reflections.

Results

Three 2-D projections of the 3-D peak shape of the (-113) reflection are reproduced in Fig. 2 for a strain of 2.5%. The azimuthal broadening in φ (rotation of the sample around tensile axis) and η (along diffraction ring) is rather smooth, with only a slight distortion due to a weak secondary maximum. The radial peak profiles generated by integrating over these angles are shown in Fig. 3 as a function of tensile strain. The increasing peak width clearly demonstrates the sensitivity of the peak profiles to structural changes upon plastic deformation. All profiles of the (-113) reflection are almost symmetric.

The radial peak profiles obtained for the other five {311} reflections were also nearly symmetric. The most pronounced example of asymmetry arose for the (1-13) peak, where the intensity decreased more slowly for smaller lattice spacings than for larger lattice spacings. In contrast, the asymmetry of the (11-3) peak [which is nearly perpendicular to the tensile axis as is the (1-13) peak] is as weak as the asymmetry of the (-113) peak.

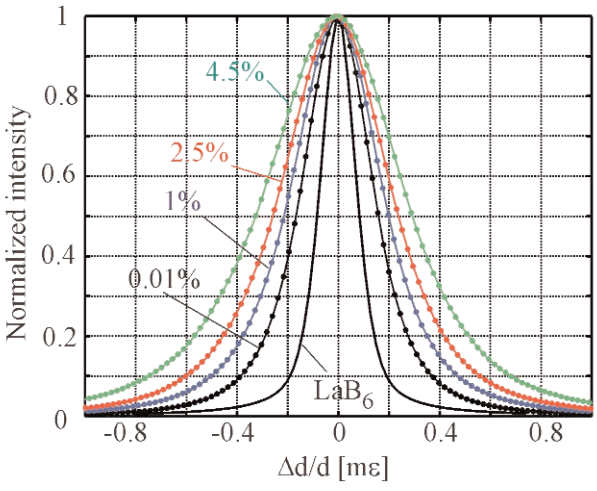

FIG. 3. Radial profile of (-113) peak from a single embedded Al grain as function of strain. Also plotted is the profile of a LaB_6 calibration powder.

Discussion

Asymmetric broadening of radial peak profiles from individual grains is understandable in terms of the internal stresses that arise in deformation structures in which dislocation boundaries act as harder obstacles and develop forward internal stresses. The largest asymmetry arises for the diffraction vector parallel or perpendicular to the tensile direction, whereas only small asymmetries are expected for intermediate angles [as for the (-113) peak]. The marked difference between the asymmetries of the (1-13) and (11-3) peaks, despite similar angles between the diffraction vector and tensile axis, is caused by an anisotropy of the dislocation structure around the tensile axis. Transmission electron microscopy (TEM) investigations of tensile-deformed polycrystalline aluminium of high purity show the existence of well-defined dislocation boundaries already at tensile strains of 5%. For grains with orientations similar to the selected grain, a single set of parallel, extended dislocation boundaries has been

observed preferentially aligned with one of the {111} planes.

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References

[1] W. Pantleon, H.F. Poulsen, J. Almer, and U. Lienert, "In-situ x-ray peak shape analysis of embedded individual grains during plastic deformation of metals," Mater. Sci. Eng., A (to be published, 2003).

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