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ScienceDirect

Acta Materialia 85 (2015) 301–313

Acta MATERIALIA

www.elsevier.com/locate/actamat

Deformation-induced orientation spread in individual bulk grains of an interstitial-free steel

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Received 18 July 2014; revised 10 November 2014; accepted 21 November 2014

Abstract—Three-dimensional X-ray diffraction was employed to characterize the lattice rotations of individual bulk grains in a 9% tensile deformed sample of interstitial-free steel. Three grains of initially close orientation that are representative of the scatter of all investigated grains with tensile axes near <522> were identified. Their rotation paths and intragranular orientation spread were analysed in detail, using crystal plasticity modelling to evaluate the nature of the orientation spread. It was found that the same set of most stressed slip systems are active in the three grains and that variations in the relative activities of the two most stressed systems account for the dominant orientation spread in the grains. The distribution of slip on these systems varies from grain to grain and also within each grain. While the grain orientation controls the identity of these slip systems, the variations are attributed to grain boundary and grain interaction effects.

Keywords: Ferritic steels; Tensile behaviour; High-energy X-ray diffraction; Individual bulk grains; Crystal plasticity

1. Introduction

Thermo-mechanical processing of polycrystalline metals has been a subject of research for almost a century. The field has been significantly advanced through the development of novel experimental techniques giving crystallographic information at the level of individual grains, such as electron microscopy in two-dimensions [1,2] and, recently, high-energy X-ray diffraction at synchrotrons in three-dimensions [3,4]. Three-dimensional X-ray diffraction (3DXRD) using synchrotron radiation is capable of characterizing individual grains embedded in the bulk of a polycrystalline metal. As the technique is non-destructive, it enables study of the same set of bulk grains before and after some external stimuli, e.g. elastic [5–7] and plastic deformation [8–11] or thermal annealing [12–14]. Several experimental setups are possible, each with specific advantages, e.g. high temporal resolution [12,15], high spatial resolution of grain maps [11,16] or high resolution of crystallographic orientations [17,18].

In parallel, polycrystal plasticity models have evolved from the classical Sachs [19] and Taylor/Bishop–Hill models [20,21] based primarily on the crystallographic orientation of the grains to increasingly complex models, accounting for interactions between clusters of grains [22,23], interactions between a grain and a matrix, representing the average of the other grains [24], and finite-element-based models, accounting for the detailed interaction between neighbouring grains [25–27] as well fast-Fourier-transform-based methods [28]. The different behaviour of grains with initially similar orientations as well as the evolution of intragranular orientation differences during plastic deformation are currently key topics being studied. The critical scientific questions are to what extent the behaviour of the grain is controlled by its crystallographic orientation, and what role the interaction with the neighbouring grains plays. Assessment of the former is important for advancing constitutive relations for slip.

The present paper addresses this subject by investigating the intergranular variations in lattice rotations and the active slip systems for selected grains of a specific initial orientation, as well as the intragranular slip system differences leading to orientation gradients across these grains. The grains selected for such studies should be representative of the ensemble of grains of that particular orientation, and the experimental basis is therefore a 3DXRD setup optimized for characterization of a large number of grains in a tensile-deformed interstitial-free (IF) steel with a fairly strong rolling texture. From this data set, representative grains are then identified for detailed studies.

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http://dx.doi.org/10.1016/j.actamat.2014.11.038
1359-6462/

In the 3DXRD experiment each grain gives rise to a number of diffraction spots, and the evolution of these is tracked as the sample is deformed. As the grain rotates, the diffraction spots move and, at the same time, they broaden because of the evolving intragranular orientation spread. The method employed here is to index the individual reflections to determine an average (centre-of-mass (CMS)) orientation of each individual grain before deformation and after 9% elongation. The rotation paths of ~100 grains are analysed, and three representative grains with initial tensile axes near <522> are selected for detailed analysis. The active slip systems of these grains are identified by crystal plasticity analysis, and the variations in intragranular slip system causing the orientation spread within each of the three grains are also investigated by crystal plasticity simulations. The simulated orientation spread is converted to simulated diffraction spots, the shape of which is then compared with the experimental reflections.

2. 3DXRD experiment and grain reconstruction

The material selected for the study was a fully recrystallized IF steel (99.6 wt.% Fe) with a well-developed body-centred cubic (bcc) rolling texture and grains with average dimensions $50 \times 50 \times 70\ \mu m^3$. A tensile sample with square cross section 0.7×0.7 mm and length 30 mm was machined by spark cutting, i.e. with ~200 grains in the cross section. Consecutive layers perpendicular to the tensile axis of the sample were mapped by 3DXRD in the undeformed state with a beam height of 10 μm. Subsequently, the sample was tensile deformed ex situ first to 3%, then 6% and finally 9% elongation. When positioning the sample in the stress rig as well as in the 3DXRD setup for mapping between tensile steps, extreme care was taken to align the tensile axis in the same way, using the entire sample length of 30 mm as a guideline. Owing to the combination of the strong texture of the sample, the number of grains in the cross section and the deformation-induced intragranular orientation spread, peak overlap in the 3DXRD data became severe after 6% elongation. To reduce the number of grains in the cross section, and thereby the peak overlap after 9% deformation, the sample was mechanically polished to about half the original dimensions of the cross section. For the polished sample, 60 layers with an interlayer spacing of 20 μm were mapped with a beam height of 10 μm. Note that, as the polishing was done after the final deformation step, all investigated grains were bulk grains far from the free sample surface as far as the tensile deformation is concerned.

2.1. 3DXRD set-up and analysis

The 3DXRD experiment was performed at beamline ID11 at the European Synchrotron Radiation Facility. Two detectors were used simultaneously: (1) a Quantix near-field detector with 1536×1024 pixels of dimensions $4.3 \times 4.3\ \mu m^2$ placed at a sample-to-detector distance of 9.3 mm, and (2) a Frelon4M far-field detector [29] with 2048×2048 pixels of $50 \times 50\ \mu m^2$ placed at a distance of 195 mm; see Fig. 1 for a schematic of the setup. The X-ray energy was 69.5085 keV (W K-edge); the beam was monochromated using a bent Laue monochromator, which also provided vertical focusing to 10 μm, while a slit was used to confine the horizontal beam dimension to 1.5 mm. The planar beam was used to map the sample layer-by-layer. For each layer, the sample was rotated around the tensile axis, acquiring diffraction images for ω-angles in the range of $[-22.5°;22.5°] \cup [67.5°;112.5°]$ in steps of 0.5°.

The FABLE software [30] was used to identify the individual grains within the sample from the diffraction reflections. The philosophy was to use the far-field data to index the reflections coming from individual grains (i.e. determine their crystallographic orientations), and get a rough estimate of their positions within the sample, while the near-field data were used to improve the spatial resolution. The indexing was performed using GrainSpotter [31]. For the subsequent refinement against both near- and far-field data, the FitAllB module [32] was applied. Owing to extensive reflection overlap in the 3DXRD data for the 6% elongated sample, only the data after strains of 0, 3 and 9% were reconstructed. In the present context, the focus is on analysing the structure after 9% deformation.

2.2. Reconstruction of grains

Because the beam height (10 μm) was substantially less than the average grain size along the tensile direction (70 μm), the grains could be observed in several subsequent

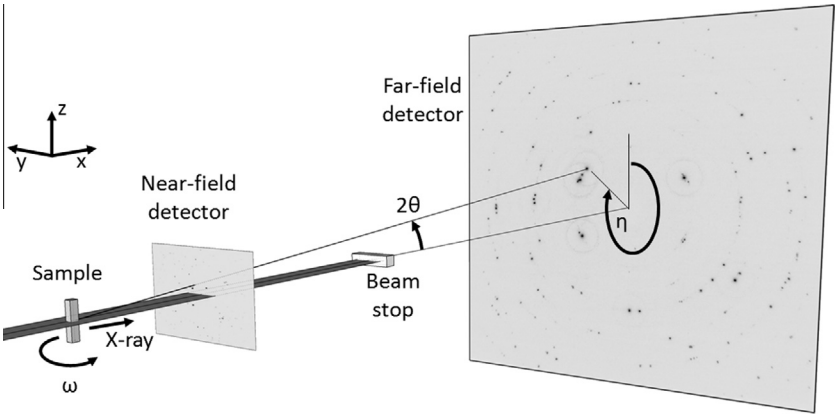

Fig. 1. The 3DXRD setup with a far-field and a near-field detector (distances are not to scale). Angles 2θ, η and ω and directions *x*, *y* and *z* are defined. The detectors show spots from the undeformed sample.