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Gradient matrix method to image crystal curvature by processing of EBSD data and trial recognition of low-angle boundaries in IF steel

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Abstract

A gradient matrix has been introduced to derive the lattice orientation gradient, indicative of crystal substructures, from EBSD orientation data. A trial mapping of this term around a grain junction in a low deformed IF steel proves to reveal a distinct low-angle boundary extending from the junction line. The background dislocation substructure, however, is imaged rather poorly because of the mapping noise.

Keywords: IF steel; Triple grain junction; Low-angle boundary; Lattice curvature; EBSD

1. Introduction

The crystal fragmentation during large plastic strains [1], which eventually forms high-angle subgrain (fragment) boundaries in grains of deformed polycrystals, attracts a great interest from both the scientific and practical viewpoints. In particular, it gains in significance because of extraordinary mechanical properties of metals with high-angle microcrystalline structures, which form after several hundreds per cent of the true strain achievable by the SPD methods [2–4]. Furthermore, a proper treatment of the crystal orientation splitting inside constitutive grains becomes obviously important in simulating the evolution of polycrystal texture and anisotropy during plastic deformation [5–7]. Experimental investigations in the considered field are very complicated, however, since the underlying processes have an essentially multi-stage and multi-scale character [1,8,9]. Thus, the low-angle substructure evolves first, made of cells of sub-micron to micron dimensions separated by fuzzy dislocation boundaries. Next, crystalline fragments, each consisting of a number of cells, begin to disorient. Their disorientation continually increases during plastic deformation, whereas the boundaries between them become more and more distinct, approaching the regular appearance of grain boundaries. In addition, according to TEM data [1,9], the interaction among whole grains on a scale of tens microns is directly involved in the fragmentation. Indeed, these data often show fragment boundaries to originate at the grain junctions or at morphological features (kinks, ledges, etc.) of the grain boundaries.

Under the above circumstances, relevant methods of crystallographic analysis should conform to rather contradictory requirements. On the one hand, in order to image the dislocation cells and to quantify the low-angle boundaries, sufficiently high resolution and angular accuracy are necessary. On the other hand, these resolution and accuracy should be combined with a panoramic view of the considered microstructure. In particular, it is desirable to map clusters of whole fragments, containing cells, as well as clusters of grains, containing fragments or/and cells. From this viewpoint, the rapidly progressing technique of the orientation mapping, based on the electron backscatter diffraction (EBSD), seems to be very promising [10–12]. At present, as applied to plastically deformed metals, this technique provides an angular accuracy of about 0.5°, still tending to improve, and a practicable spatial resolution of the order of 0.1 μm. This is quite enough to image boundaries between fragments disoriented by several degrees or, all the more, high-angle grain boundaries. At the same time, both these accuracy and resolution are very poor to discern low-angle dislocation boundaries between the cells or between the freshly nascent fragments. This drawback of EBSD technique, particularly as compared to TEM, notably limits its application to deformation microstructures. Nevertheless, while waiting for more efficient equipment, untapped opportunities should be sought among methods to process

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the raw EBSD data, and the present paper is an effort in this direction.

A rational way to better distinguish low-angle dislocation substructures, as proposed after the invention of EBSD orientation mapping [10,11], is to directly map the corresponding gradient of crystal orientation (lattice curvature) rather than the orientation as such. A closely related treatment of the orientation gradient has been used in texture simulations as well [13]. Certain attempts to implement this idea may be perceived in recording the disorientation between neighboring EBSD data points [14] and in factoring out the average orientation of the crystal containing a substructure of interest [15,16]. However, a satisfactorily rigorous and flexible approach to the spatial differentiation of discrete orientation data (recorded over some grid) has not been formulated as yet. The flexibility of such an approach, in particular, should facilitate a fitting of both the shape and dimensions of the differentiation domain in order to find the best compromise between the resolution and noise reduction.

Similar problems have recently been addressed in consideration of the finite element kinematics [17] and in the strain mapping of low deformed composite materials [18]. Discrete vectorial data were successfully differentiated there with gradient matrices determined over respective grids. In order to apply this method to crystal orientations, it remains only to re-express the latter in a vector form. In the present paper this approach is realized on a low deformed interstitial free (IF) steel, where the dislocation substructure could not be imaged in grains with the conventional methods of EBSD data processing. The paper is organized as follows. First, the material and experimental procedures are briefly described. Then the raw EBSD data are reported, where the corresponding orientation maps display some evidence for a strain induced junction disclination. Next, the gradient matrix method is formulated and adapted to the orientation mapping, and the EBSD data are re-processed in order to image the lattice curvature and to recognize the low-angle dislocation boundaries. Finally, the resulting maps and the underlying substructure are discussed.

2. Material and experimental procedures

Standard flat specimens were cut from a cold rolled and then recrystallized (1300 °C, 0.5 h) IF steel and subjected to a tensile deformation of 5% in the former rolling direction. The deformation was applied at room temperature with a strain rate of $0.001\ s^{-1}$ on an Instron 4505 (100 kN) testing machine. Before further investigations, cross sections of the specimen working parts were prepared by standard metallographic procedures, using electrochemical polishing in 15% perchloric acid-ethanol solution at −5 °C as the final step. Among a number of planar sections examined by optical microscopy using a Leitz Metalloplan, one area, containing an almost perfect 120° junction of grain boundaries, has been selected and then subjected to EBSD analysis. Orientation mapping by EBSD was carried out on a hexagonal grid, following [10,12], on a Philips XL30 SEM with a tungsten filament at an accelerating voltage of 20 kV. EDAX-TSL software was used for pattern indexing. The step size and angular accuracy were 0.1 μm and 0.5°, respectively.

3. EBSD orientation mapping around triple grain junction

The present chapter considers an effort to image the low-angle dislocation boundaries, while using the conventional (EBSD) orientation imaging technique with a limited angular accuracy. Although the achievement of desired results was in no way ensured, this effort has been undertaken in order to demonstrate a reference EBSD application to a small orientation range peculiar to the low-angle substructure. Furthermore, apart from the latter, the same orientation span may be indicative of the long-range elastic rotations, which appear due to the grain interaction [19,20], and the effect of this elastic field on the substructure formation also deserves consideration.

Keeping in mind that grain boundaries in recrystallized structures tend to form equilibrium 120° junctions, we have found an appropriate planar area, where the three boundary traces make almost perfect 120° angles at their intersection. This arrangement, which was only slightly affected by the applied low deformation, will allow one to treat the grain interaction with a simple 2D model, assuming that the involved boundaries and, accordingly, the junction (triple) line are perpendicular to the investigated planar section. It should be mentioned that the above reasoning is valid only if involved grain boundaries are not special and, consequently, have equal specific energies. The accepted assumption may be motivated by rather limiting conditions for the existence of low-energy special boundaries [21] and by low probability of the situation where the perfect arrangement of the three boundary traces, found by chance on the section plane, would be made by an imperfect triple junction. In addition, the assumed orientation of the junction line will be a posteriori verified in analyzing obtained EBSD data.

3.1. Inhomogeneous lattice rotation field inside grains

The grain boundaries, forming the triple junction shown in Fig. 1a, have been conventionally drawn between EBSD data points disoriented by more than 10°. Then the average crystal orientation has been determined within each of the three grains with respect to the macroscopic Cartesian system *XYZ* associated with the imposed deformation. Axes *X* and *Y*, respectively, indicate the transversal and normal directions of the flat specimen, whereas axis *Z* is parallel to the tension direction and normal to the investigated cross section. The orthogonal matrices $\mathbf{R}_1$, $\mathbf{R}_2$ and $\mathbf{R}_3$ expressing the averaged orientations are listed in Table 1; their columns contain the components of the respective basis vectors of the *XYZ* system relative to the crystal basis. Note that the reference grain areas of 50–70 μm^2, Fig. 1a, do not cover the whole respective grains, but only their fractions forming an almost perfect (120°) triple junction. That is why, neglecting deviations from this arrangement outside the area of Fig. 1a, we will consider the boundary planes parallel to axis *Z*.

In order to confine the analyzed orientation range to that of the sought low-angle boundaries, a small vector **w** of the local