



THE 3-D STRUCTURES OF NUCLEUS AND ITS FORMATION IN COLD ROLLED STEEL USING 3D-EBSD TOMOGRAPHY

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Overview

- The emerging technique of focused ion beam (FIB) tomography has been combined with electron backscatter diffraction (EBSD), whereby consecutive sections of a sample are ion milled by FIB and each fresh surface characterized by EBSD. This advanced serial sectioning technique, termed **3-D EBSD tomography**, generates up to several hundred slices of thickness 30-500 nm with the entire dataset reconstructed to generate detailed 3D crystallographic maps of the microstructure [1-3].
- This newly-developed technique allows the quantification of the spatial distribution of various features such as the orientation relationships between phases, and grain boundary characteristics of partly and fully recrystallized microstructures etc. [1-3].
- In the present work, FIB-EBSD tomography was used to investigate the recrystallization behaviour of an 85% cold rolled interstitial free steel, including the nucleation sites, growth behaviour of nuclei and orientation relationships between recrystallizing grains and the surrounding substructure.

Sample Preparation, FIB Milling & EBSD

- An FEI Novolab 200 DualBeam™ platform interfaced with a TSL™ EBSD facility was used to develop a reliable method for concurrent serial sectioning and EBSD mapping of a cold rolled and ~2% recrystallized IF steel (Fig. 1a).
- Figure 1b shows the initial sample preparation using FIB to generate a protrusion suitable for serial sectioning and concurrent EBSD mapping. Ion milling was carried out using an accelerating voltage and milling current of 30 kV and 3 nA, respectively.

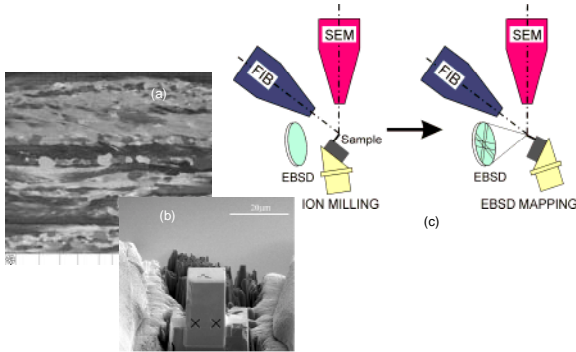


Figure 1. (a) Electron channeling contrast micrograph of partly recrystallized IF steel. (b) SEM micrograph of a typical ion milled protrusion showing the starting face for serial sectioning. (c) Relationship between the sample and the FIB/SEM beam directions and EBSD detector.

Serial Sectioning and Data Analysis

- Following the initial ion milling procedure (Fig. 1b), the sample is ready for ion beam milling and EBSD mapping. The method of serial sectioning and EBSD imaging is given in Fig. 1c which involves consecutive steps of sectioning by FIB and automated stage movement for EBSD mapping of each RD-TD fresh surface.
- Each milling stage removed a 0.15 μm thick slice of material and eighty slices were analysed by EBSD using an SEM accelerating voltage, probe current and working distance of 10 kV, 2.1 nA and 10 mm, respectively. The electron beam step size was 0.1 μm which generated ~ 22,500 data points per slice.
- Figure 2 gives a series of EBSD micrographs of the partly recrystallized steel showing a recrystallizing grain within the deformation substructure. The micrograph on the far left shows a nucleus at a grain boundary but further sectioning reveals the true complexity of this newly developed grain.

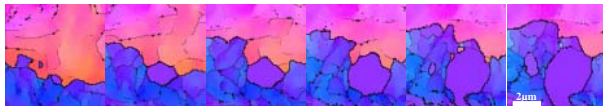


Figure 2. EBSD micrographs of a series of Ga^+ ion milled sections of partly recrystallized IF steel. The new grain has the $\{111\}\langle uvw \rangle$ orientation (γ -fibre) and is bounded by α -fibre (upper) and γ -fibre (lower) regions of the deformation substructure.

Nucleus' Internal Structure and Its Misorientation with Surrounding Deformation Structures

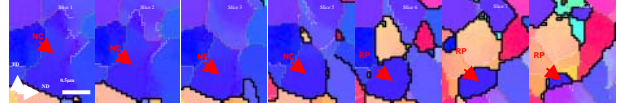


Figure 3. A series of EBSD micrographs reveal the internal orientation spread and misorientation with its surrounding deformation structures in different part of a nucleus N1. The coarse black, white, pink and green line denotes a misorientation range $>15^\circ$, $10-15^\circ$, $5-10^\circ$ and $1-5^\circ$.

- Figure 3 shows nucleus N1 can be divided into two parts according to its misorientation with surrounding deformation structures. Part one, from slice 1-5, is mainly bounded by LAGBs but with a segment of HAGBs. Part 2, from slice 6 to 8, is mainly bordered by HAGBs. Figures 4a & 4b illustrate part one and two are dislocation-containing and dislocation-free segments respectively. So part one has the features of deformation subgrain and can be termed nucleation core (NC) formed from deformation subgrain. Part two has the features of recrystallized grains, and can be termed recrystallized part (RP) formed by NC consuming its adjacent high store energy HAGBs deformation regions.

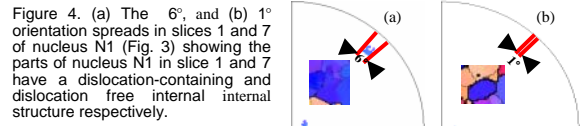


Figure 4. (a) The 6° , and (b) 1° orientation spreads in slices 1 and 7 of nucleus N1 (Fig. 3) showing the parts of nucleus N1 in slice 1 and 7 have a dislocation-containing and dislocation free internal structure respectively.

The 3-D Structure of Nucleus and Its Formation

- Figures 5a and 5b shows the 3-D structures of three nuclei and their formations in deformation structure (stripped for clarity). The different surface colours of these 3-D nuclei in Fig. 5a indicate their misorientation range with surrounding deformation structures, while in Fig. 5b the different orientations of these deformation structures surrounding the nuclei.
- It can be seen in Fig. 5a that the nucleation is anisotropic. The nucleation cores, grows at a slower rate compared with recrystallized parts. The preferred growing directions of recrystallized parts in the nuclei as arrowed in Fig. 5a, are clearly shown in Fig. 5b, to be the HAGBs regions between differently oriented deformation structures.

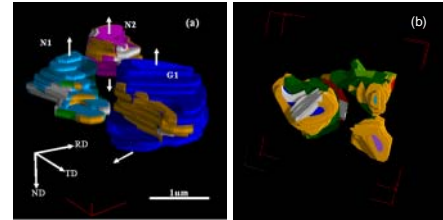


Figure 4. Reconstructed 3-D EBSD micrographs showing the 3-D structures of three nuclei. (a) The surface colour showing the nucleus misorientation range with its surrounding deformation structure: white $<5^\circ$, orange $5-10^\circ$, green $10-15^\circ$ and rest $>15^\circ$, with nucleation core mainly covered by LAGBs ($<15^\circ$), recrystallized parts by HAGBs. (b) The surface colours representing the orientations of deformation structures surrounding the nuclei, showing recrystallized parts mainly grow along the grain boundary of deformation structures.

Conclusion

- It can be concluded that the nucleation starts by nucleation core formation from a deformation subgrain with one segment of HAGB adjacent to high stored energy regions, the nucleation core starts to grow by anisotropically consume its adjacent high store energy HAGBs deformation regions, to form the recrystallized part. Such growth behaviour is clear indication of **orientation pinning**. The figures also highlight the complex nature of boundary migration during recrystallization.

References

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