

# STUDY OF RECRYSTALLIZATION IN COLD ROLLED IF STEEL and NICKEL USING 3-D FIB-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 **FIB-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 nucleation behavior of an 85% cold rolled interstitial free steel, and a 60% cold rolled Nickel single crystal containing a SiO<sub>2</sub> particle, including the nucleation sites, growth behavior of nuclei, and orientation relationships between recrystallizing grains and the surrounding substructure, and the effect of particle and voids formed around particle on the nucleation behavior.

## 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) and 30% recrystallized Ni (Fig. 1b).
- Figure 1c shows the initial sample preparation using FIB to generate a protrusion suitable for serial sectioning and concurrent EBSD mapping. Figure 1d illustrates slice milling and the following EBSD positions of the sample stage.

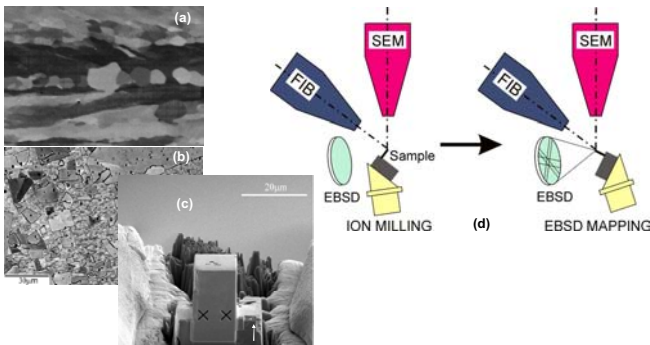


Figure 1. Electron channeling contrast micrograph of partly recrystallized (a) IF steel and (b) Ni containing SiO<sub>2</sub> particle. (c) EBSD micrograph of a typical ion milled protrusion showing the starting face for serial sectioning. (d) Relationship between the sample and the FIB/SEM beam directions 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 SEM imaging and EBSD mapping of each RD-TD fresh surface.
- Each milling stage removed a 0.15 and 0.2 μm thick slice of steel and Ni, and eighty slices were analyzed by SEM and EBSD. The electron beam step size was 0.1 μm which generated ~ 22,500 data points per slice.

Figure 2a gives a series of EBSD micrographs of the partly recrystallized steel showing a nucleus (dislocation free as shown in slices 1-4) is formed from an <111> deformation subgrain (containing dislocation in slices 5-7). Figures 2b shows a growing nucleus formed in the vicinity of a SiO<sub>2</sub> particle and the voids around the particle. The deformation structure where the nucleus originated can be seen from the yellow part around the particle in slice 1.

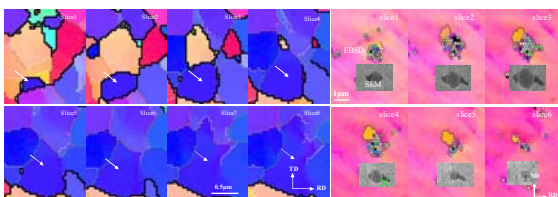


Figure 2. EBSD micrographs of a series of Ga<sup>+</sup> ion milled sections: (a), a nucleus in partly recrystallized IF steel, having an {111} orientation, originated from a deformation subgrain which contains many dislocations, (b) a nucleus formed in Ni sample in the vicinity of a SiO<sub>2</sub> particle and voids, with SEM images showing the size and shape of the SiO<sub>2</sub> particle and the voids formed around it.

## 3-D Microstructure and Crystallography in Nickel

- Figure 3 shows two groups of reconstructed 3D FIB-SEM-EBSD micrographs of SiO<sub>2</sub> particles, 2 voids formed in each side of each particle during cold rolling, and the nuclei and nucleation eye formed in the vicinity of particle and voids in the Ni sample during annealing, with Fig. 2b showing a selected slice view in Fig. 3a.
- The orientations of nucleus 1, 2 and the nucleation eye in Fig. 3 have their origins in the deformation structures in the green low angle grain boundary (LAGB) regions, with Fig. 2b showing a selected slice view of nucleus 1. The LAGB fraction tends to decrease with increasing nucleus size.
- The particles have a spherical shape, and are rotated during the cold rolling process according to the 3-D void shape. Two voids formed in each side of each particle have a non-symmetrical shape and different size.

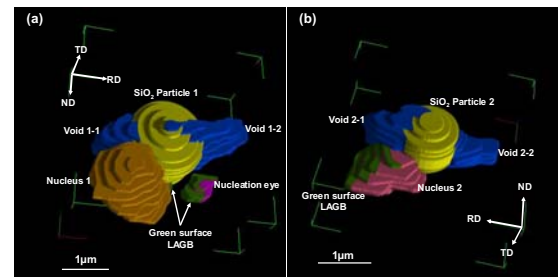


Figure 3. Reconstructed 3D FIB-SEM-EBSD micrograph showing detail not clear in the 2D micrographs such as the 3-D shape of amorphous SiO<sub>2</sub> particles, voids formed around these particles, and the nuclei and nucleation eye nucleated in the vicinity of particle and voids. The green surface of nuclei and nucleation eye represents LAGB, where the nuclei and nucleation eye originated.

## Misorientation Relationships between Recrystallizing Grain Segments and the Deformation Substructure

- Figure 4 shows two groups of recrystallizing grains growing into the deformation substructure (stripped for clarity) whereby a given color on each grain surface denotes a misorientation range between the growing grain and the surrounding substructure, i.e.:

$\theta_m < 5^\circ$	–	white surface
$5^\circ < \theta_m < 10^\circ$	–	yellow surface
$10^\circ < \theta_m < 15^\circ$	–	green surface
$\theta_m > 15^\circ$	–	predominant surface colours (blue, pink etc.)

- It can be seen that both the nucleation (Fig. 4a) and growth stages (Fig. 4b) are anisotropic. The selected slices for nucleus 1 is shown in Fig. 2a illustrating the LAGB regions (i.e. white, yellow and green grain surfaces) grow at a slower rate compared to the HAGB segments (arrows). Such growth behavior is a clear indication of **orientation pinning**. The figures also highlight the complex nature of boundary migration during recrystallization.
- The LAGB regions in nucleus 1, 2 and 3 are deformation subgrains which contain dislocations while their HAGB region are almost dislocation free.

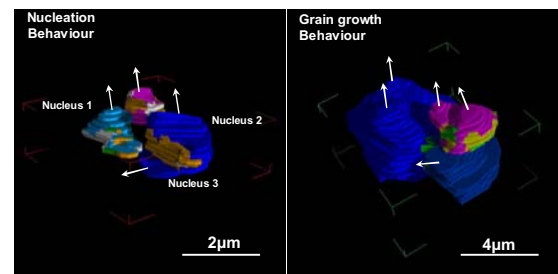


Figure 4. Reconstructed 3-D EBSD micrographs showing two groups of grains at various stages of recrystallization: (a) nucleation stage and (b) further growth. The given colour denotes a certain boundary misorientation between a growing grain and the deformation substructure (described above).

## References

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