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Design in Light Metals

GENERATING 3-D MICROSTRUCTURES USING 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 50-500 nm with the entire dataset reconstructed to generate detailed 3D crystallographic maps of the microstructure [1-4].
- 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 a particle-containing nickel alloy that contains various interesting microstructural features such as particle deformation zones, and faceted recrystallizing grains containing twins [4].

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 partly recrystallized Ni-0.3%Si alloy containing spherical silica particles of size 1-5 μm (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.

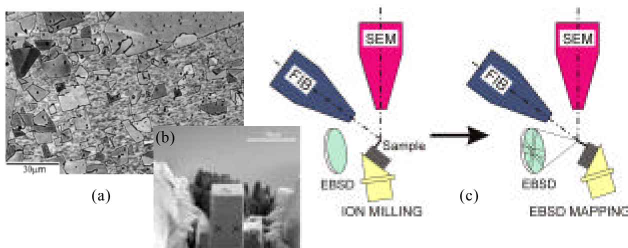

Figure 1. (a) Electron channeling contrast micrograph of partly recrystallized nickel. (b) SEM micrograph of a typical ion milled protrusion showing the starting face for serial sectioning. (c) Relationship between the sample and the FIB/SEM beam directions and EBSD detector.

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 fresh surface.
- Each milling stage removed a 0.2 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 0.2 μm which generated ~ 20,000 data points per slice.
- Figure 2 gives a series of EBSD micrographs of the Ni sample showing the distribution of silica particles, the orientation spread of the deformation microstructure and recrystallizing grains containing twins.

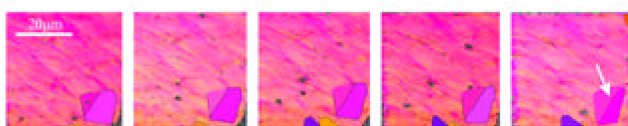

Figure 2. EBSD micrographs of a series of Ga^{+} ion milled sections of partly recrystallized nickel. The distribution of silica particles, orientation changes in the deformation microstructure and recrystallizing grains containing twins can be seen (particles and twin boundary are arrowed).

3-D Reconstruction of Recrystallizing Grains

- Figure 3 shows a reconstructed 3D-EBSD micrograph highlighting the features shown in Fig. 2. The deformed regions have been stripped away to highlight the relatively uniform distribution of silica particles and two groups of twinned grains. The external boundaries of the recrystallizing grains shown below are clearly faceted, as shown by TEM [4], with crystallographic analysis by EBSD showing that they are parallel to low index planes.

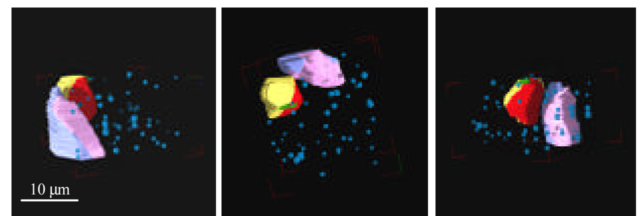

Figure 3. Reconstructed 3D-EBSD micrographs showing detail not clear in the 2D-EBSD micrographs such as the spatial distribution of silica particles and two groups of twinned grains containing faceted interfaces (deformation substructure stripped away for clarity).

Crystallographic Analysis of Twinned Grains

- A common feature of the recrystallizing microstructure is the presence of groups of recrystallizing grains containing twin boundaries (Figs 2 and 3). Figure 4a shows three twinned grains with the {111} pole figure in Fig. 4b showing that grains 1-2 and 2-3 are twin related (i.e. rotated 60° about <111> axis) and all grains are misoriented by 5-15° from the deformation substructure (matrix) An interesting feature of Fig. 4d is the large silica particle at the core of the grains with a further particle situated ~2 μm below the twin boundary in (a) (not shown).
- The recrystallizing grains originating at the silica particle in Fig. 4 are twin-related with the boundary having both planar (coherent) and ledge (incoherent) morphology. Two of the grains are seen to grow away from the particle in a directional manner with the {111} (twin) boundary parallel to the principal growth direction.

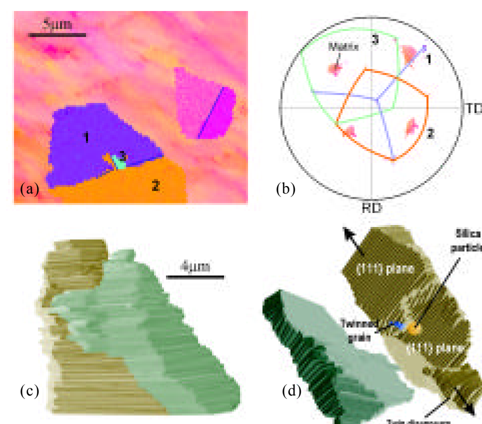

Figure 4. (a) 2D-EBSD section showing recrystallizing grains (1-3). (b) {111} pole figure showing that grains 1-2 and 2-3 contain 60°<111> coherent (Σ3) twin boundaries. (c) Reconstructed grains in (a) separated in (d) to reveal a silica particle at the core of the recrystallizing grains, planar twin boundaries parallel to {111} plane and incoherent ledges, and the eventual disappearance of the twin boundary during growth (principal growth directions are arrowed).

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