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Correlating Observations of Deformation Microstructures by TEM and Automated EBSD Techniques

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Correlating observations of deformation microstructures by TEM and automated EBSD techniques

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Abstract

The evolution of the deformed microstructure as a function of imposed plastic strain is of interest as it provides information on the material hardening characteristics and mechanism(s) by which cold work energy is stored. This has been extensively studied using transmission electron microscopy (TEM), where the high spatial and orientational resolution of the technique is used to advantage to study local phenomenon such as dislocation core structures and interactions of dislocations. With the recent emergence of scanning electron microscope (SEM) based automated electron backscatter diffraction (EBSD) techniques, it has now become possible to make mesoscale observations that are statistical in nature and complement the detailed TEM observations. Correlations of such observations will be demonstrated for the case of Ni-base alloys, which are typically non-cell forming solid solution alloys when deformed at ambient temperatures. For instance, planar slip is dominant at low strain levels but evolves into a microstructure where distinct crystallographic dislocation-rich walls form as a function of strain and grain orientation. Observations recorded using both TEM and EBSD techniques are presented and analyzed for their implication on subsequent annealing characteristics.

1. Introduction

Engineering of the grain boundary microstructure in low to medium stacking fault energy (SFE) FCC materials, as for instance Cu and Ni-base alloys is effectively achieved by thermomechanical processing [1-3]. One approach has been a multi-cycle treatment of moderate strain levels (5-30%) with annealing treatments at relatively high temperatures but for very short times, typically on the order of 5 to 30 minutes. An important aspect is that the total forming reduction is broken up into several cycles of strain and annealing. The microstructure thus obtained contains a higher fraction of special boundaries as compared with the as-received condition.

This is unlike general industrial practice of large cold reductions followed by a recrystallization anneal, or hot-working. In this case the annealed microstructure after single-step deformation on the order of the cumulative strain from the multi-step treatment does not show such dramatic differences when compared with the as-received condition [3]. It is well understood that a material after undergoing deformations on the order of 50-70% (or higher) completely recrystallizes within a short period of time on thermal treatments above $0.5T_m$. The process of nucleation sites being randomly seeded in the deformed microstructure as a consequence of the large strains is generally accepted.

Current understanding of the evolution of the microstructure and the mechanism(s) responsible for the increase in the grain boundary character distribution (GBCD) after several moderate strain and annealing cycles is rather empirical in nature. In particular, the deformation microstructure at moderate strain levels of 5-30% has not been well characterized for non-cell forming FCC materials. Therefore, the objective of this study is to investigate the deformation microstructure as a way of better understanding the annealing behavior.

2. Experimental procedure

TEM and EBSD investigations were conducted on sections perpendicular to the compression axis to enable determination of the orientation dependence of slip in the polycrystalline samples of a Ni-16Cr-9Fe alloy. These observations provided information at different length scales. The typical resolution obtained with the EBSD technique is about 1 μm, while features can be routinely resolved to about 1-2 nm in the TEM. Similarly, the orientational resolution is better in the TEM as compared to the EBSD technique, but not by orders of magnitude as in spatial resolution. Instead the comparison shows that the resolving power of EBSD is about 1°, which compares with about 0.1° in the TEM.

Individual dislocations are not sampled in EBSD (as in the TEM) but rather lattice rotations that arise as a consequence of deformation are indicated as orientations that deviate from the referential point. Thus point-to-point correlations (misorientations) are obtained. This data can then be converted into information on texture and the character of grain boundaries. As an extension, the effects of lattice rotations (due to deformation) near grain boundaries on the character of the grain boundary, as defined by the coincident site lattice (CSL) model [4], can be easily studied [5,6].

3. Observations

The dislocation substructure as a function of room temperature compressive strain is shown in the TEM micrographs of Fig. 1. After 5% strain, planar arrays of dislocations were observed (Fig. 1a). This was indicative of negligible cross-slip in the alloy, which is a concentrated Ni-base solid solution and in all likelihood has a low SFE (< 70 mJ/m^2) [7]. In addition, there was minimal evidence for interactions between neighboring slip bands. Further deformation to about 15% strain revealed a well-developed Taylor lattice type dislocation substructure (Fig. 1b). Interactions between the planar arrays of dislocations did indicate the incipient stages of deformation banding. This was observed to be more pronounced after 25% (Fig. 1c), and almost all of the grains exhibited some evidence of deformation banding at 45% strain. The widths of the bands, or domains as called by Hughes [8], typically were in the range of 200-250 nm. No evidence for a cellular sub-structure was seen within or outside these bands at the strain levels of 5-45%.

The deformation bands, as seen in Fig. 1c, did not particularly form near grain boundaries. Based on detailed observations that were made of this deformation microstructure it appeared that the dislocation rich walls formed on the primary slip plane (dotted line in Fig. 1c). This was also verified by diffraction where it was evident that the misorientations across the walls are of the order of 1°.

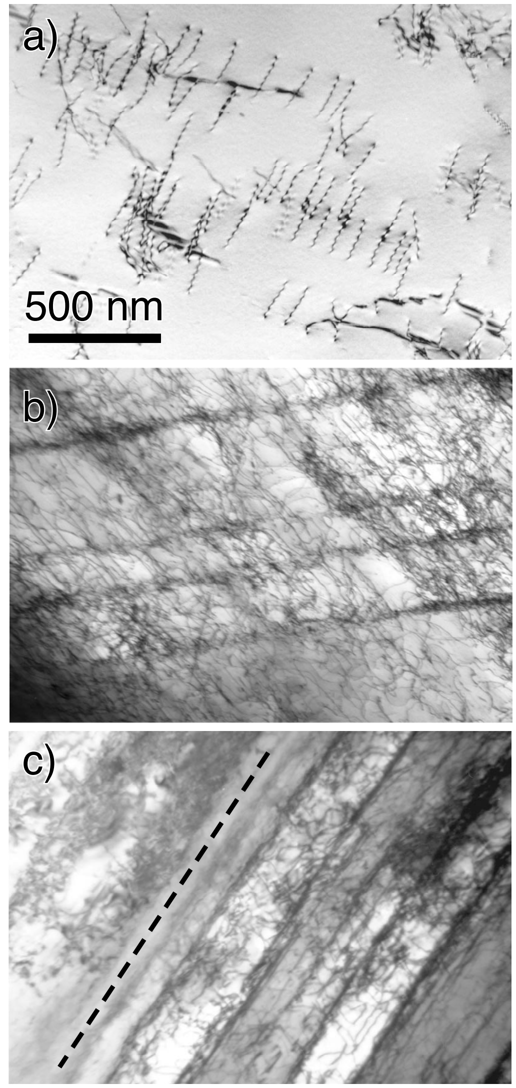

Figure 1. TEM micrographs of dislocation structure after a) 5%, b) 15%, and c) 25% strain.

A statistical sampling of the deformed microstructure revealed that deformation (after 5% strain) within the grains was pronounced in regions near the grain boundaries, as evidenced by the rotations that are mapped by EBSD in Fig. 2b. The rotations (Fig. 2b) within the grains could be due to deformation incompatibility or formation of dislocation walls that manifest as misorientations. These are indicated by the fine grayscale segments (1-15°) observed near the high angle grain boundaries that are shown in bold. In comparison, the EBSD map from the undeformed, well-annealed sample is

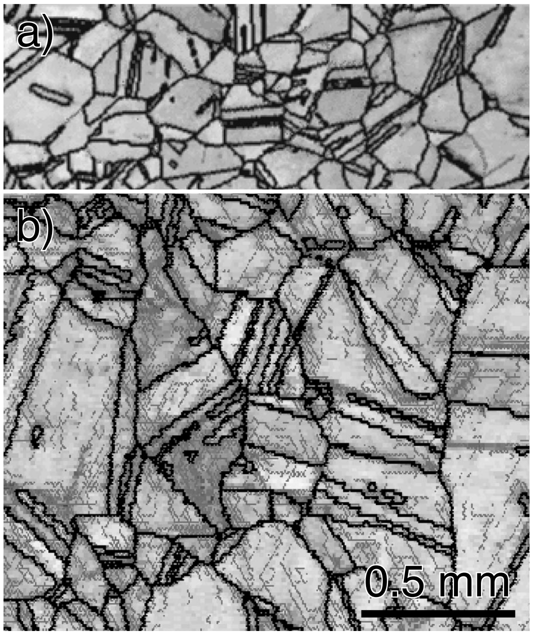

Figure 2. EBSD maps showing lattice rotations within grains in a) solutionized sample, and b) after 5% strain.

shown in Fig. 2a.

Significantly more rotation was observed by EBSD mapping after 15% strain. The EBSD maps in Fig. 3 illustrate substantial slip activity in all grains and well within the grain interiors. The maps show special and random high angle grain boundaries mapped in black and light grayscale color, respectively. An interesting observation was of high angle boundaries changing character as a consequence of lattice rotations in their vicinity. Examples of have been marked as ‘A’ in Fig. 3a, where it is observed that the random boundary becomes special in character as its deviation from Brandon criterion [9] changes locally. Special boundaries locally converting to random character were also observed, and one such example is marked as B in Fig. 3b.

Low-angle boundaries between 1 and 2° mapped as fine grayscale segments (in Fig. 3a) are observed at a higher frequency when compared with Fig. 2b and are uniformly distributed. The interesting point is that at this strain level the frequency of low-angle boundaries between 2-5° (mapped in Fig. 3b) increases dramatically over that of the 5% sample, where practically none were observed. These observations are in agreement with the TEM images of Fig. 1, which suggest that regions of local rotation appear within the grains with increasing strain. The length scale of EBSD mapping would suggest that several of the domains (typically 250 nm in width) separated by dislocation rich walls (or domain boundary [6]) could be sampled as one singular region of high misorientation.

Formation of low-angle boundaries of angular rotation greater than 2° was correlated with an orientation dependence. It was generally observed that such rotations were observed in grains where the stress axis was closer to the <110> or <111> directions. Grains with an axis near <100> typically had higher densities of misorientations close to 1°.

These observations were further investigated by TEM. An example has been taken from a sample deformed 15%, where the stress axis is perpendicular to the plane of view. It is quite apparent that the deformation microstructure, as seen in Fig. 4, on either side of the grain boundary is vastly different. In the case of the top grain with only two active slip systems (hard deformation direction, ~<111>) numerous dislocation rich walls (as in Fig. 1) along the primary slip planes could be observed. Presence

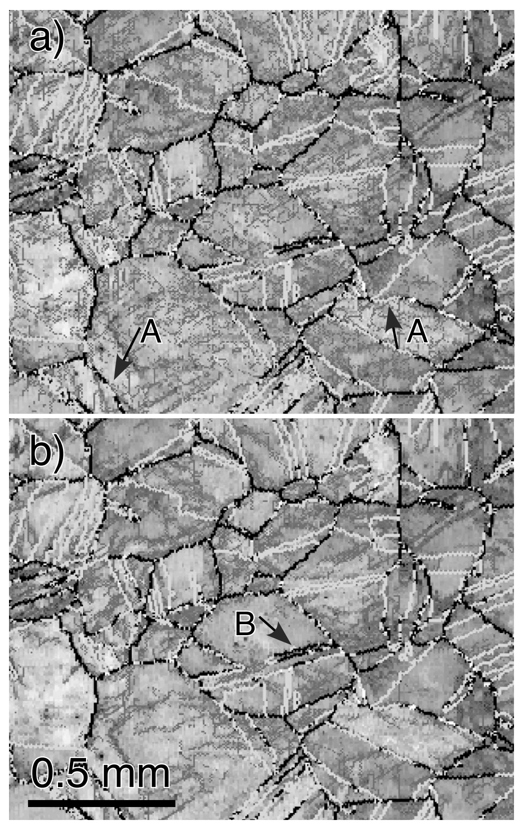

Figure 3. EBSD maps showing rotations within grains of a) 1-2°, and b) 2-15° after 15% strain.

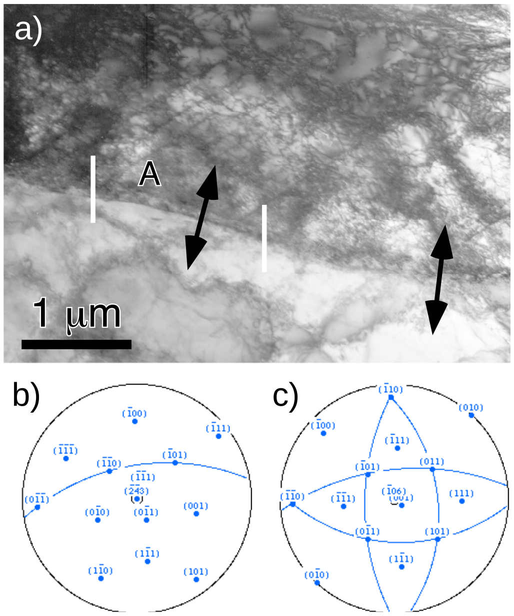

Figure 4. a) TEM micrograph of a grain boundary region showing the formation of dislocation walls in a grain with hard orientation, b). Stereographic projection of c) shows the bottom grain in a soft orientation.

of these walls lead to spot splitting in the diffraction pattern tilted almost perpendicular to the grain axis thus indicating substantial local rotations. The deformation microstructure appeared to be more uniform in the bottom grain with multiple possible slip systems (soft deformation direction, ~<001>) and no discernable rotations within the grain. In addition, deformation bands, as at position 'A', could also be observed in the top grain in the vicinity of the grain boundary. From diffraction evidence taken across several points along the boundary, it was possible to ascertain that the locally the segment of the boundary had changed character from a $\Sigma 91_d$ (<111>/54°) to $\Sigma 37_c$(<111>/50.5°). This is agreement with the EBSD observations of Fig. 3 and points to the local fluctuations in the deformed microstructure [5,6] that need to be investigated in order to predict the annealing behavior.

Grain sub-division into regions of misorientations over a wide range of angles was postulated even for non-cell forming materials [10] and experimentally verified in an Al-5.5at%Mg alloy [8]. It has been shown in this study that a combination of TEM and EBSD is an effective way of extracting information on the evolutionary process of such deformation microstructures. TEM investigations enabled identification of the character of individual dislocations and their participation in planar slip processes that evolve into dislocation tangles or organize themselves into the so-called Taylor lattices [10]. This process was also captured by EBSD observations, albeit at a length scale close to the resolution limit of the technique. This enabled a statistical sampling of the collective behavior of an ensemble of dislocations that manifests itself as lattice rotations due to the presence of geometrically necessary dislocations.

The arrangement of dislocations informs us about the storage of cold work. For instance, in the present study it is clear that new high angle boundaries that could serve as recrystallization nuclei are not introduced. This has indicated that in the process of grain boundary engineering, where the material is only deformed 25% strain in each cycle [1-3], annealing in all likelihood proceeds by boundary migration mechanisms that rely on local variations in the stored energy of cold work [11].

Acknowledgments

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