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Boundary migration during recrystallization of heavily deformed pure nickel

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Abstract. The detailed microstructure in front of recrystallization boundaries and their migration during annealing were traced using ex-situ electron backscatter pattern maps of one and the same surface area taken after annealing. It is observed that many protrusions/detrusions form on the recrystallizing boundaries. During annealing, the recrystallization boundary segments migrate in a stop-go type of fashion, while protrusions and detrusions alternately form and disappear. The correlation between the protrusions/detrusions and the stop-go type of migration are briefly discussed.

1. Introduction

Grain boundary (GB) migration plays a significant role in many processes in materials. For example, during recrystallization new essentially perfect nuclei develop and grow at the expense of a deformed matrix via the migration of the recrystallizing grain boundaries. The classical GB migration model suggests that the GB migrates by atoms in front of the boundary jumping from the deformed microstructure to sites belonging to the lattice of the growing grain [1]. The migration rate, v, can generally be described by the mobility of the boundary, M, and the driving force, F, on the boundary, i.e.

v = MF,

(1)

For recrystallization, the driving force is the stored energy of the deformed structure. Eq. 1 implies that the boundary will migrate continuously unless it is impinging on another recrystallized grain or a deformed grain with similar orientation (orientation pinning) [2]. However, in reality boundaries do not migrate all the time during the recrystallization process, even when there are no pinning effects. In-situ 3 dimensional x-ray diffraction (3DXRD) characterization of recrystallization has shown, for example, that the recrystallization boundaries migrate in a very complex way [3]. The migration of individual boundary segments occurs in a jerky move-stop-move type fashion, and locally fairly large protrusions (and detrusions) form on many boundaries. Subsequent ex-situ electron backscattering pattern (EBSP) investigations have indicated that on a local scale Eq. 1 is not suitable for the description of such a stop-go type of movement [4].

Early observations of protrusions can be found in the classic work by Beck et al. [5] on the recrystallization of high-purity aluminium. However, a theoretic model has only very recently been discussed by Martorano et al. [6]. In their model a driving force taken to simply vary sinusoidally was applied to a flat and circular boundary leading to the evolution of protrusions on the migrating boundary. In reality the dislocation boundaries are arranged in a much more complex fashion and so the driving force will also vary in a complex manner. The mechanism of the formation of protrusions and how they affect the GB migration are still not clear.

The aim of the present paper is therefore to investigate the detailed microstructure in front of recrystallization boundaries and to further the understanding of boundary migration during recrystallization.

2. Experimental

The sample materials chosen for the investigation were 99.996% pure nickel cold rolled to 96% reduction (ε_{vm} =3.7) in thickness (referred to as the CR sample) and 99.96% pure nickel deformed by accumulative rolling bounding (ARB) to strain of ε_{vm} =4.8 (referred to as the ARB sample) [7]. For recrystallization, the CR sample was annealed at 320°C for 5min while the ARB sample was annealed at 220°C for 10min. The microstructure was examined using a fully-automated EBSP analysis system attached to a Zeiss Supra 35 thermal field emission gun scanning electron microscope. Scanning step sizes of 75nm and 50nm were used for the CR and ARB samples, respectively. In all the EBSP orientation maps presented in this paper, black pixels are non-indexed points. Gray and thick black lines represent boundary misorientations of >1.5° and >15°, respectively.

3. Results and discussion

After annealing the samples were partially recrystallized. Typical microstructures containing recrystallized grains and their surrounding deformed matrix for both materials are shown in Fig.1. It can be clearly seen that quite similar protrusions and detrusions are seen on the recrystallizing GBs in both samples as marked by black and white arrows although the deformed structures are different: the ARB sample contains a lamellar deformation microstructure (dislocation boundaries almost parallel to the rolling plane). In contrast, the CR sample contains a mixture of lamellar boundaries as well as regions more typical of a medium strain deformation microstructure containing extended planar boundaries inclined at an angle to the rolling direction. Furthermore, the ARB sample contains much more high-angle boundaries in the deformed microstructure then the CR sample.



Fig. 1 Examples of EBSP maps showing partially recrystallized microstructures: a) CR sample annealed at 320°C for 5min; b) ARB sample annealed at 220°C for 10min. Examples of protrusions and detrusions are marked by black and white arrows, respectively. Black pixels are non-indexed points. Gray and thick black lines represent boundary misorientations of >1.5° and >15°, respectively.

Previous research on the recrystallization of the CR sample has shown that during annealing the misorientations of boundary segments that migrate can be similar to those of segments that do not migrate, as shown in Fig. 2 [4]. This implies that for boundary segments with similar misorientation some of them may move whereas others do not, or, for the same boundary segment it sometimes may move, and sometimes may not. In that experiment [4] the EBSP maps were obtained using a step size of $2\mu m$ so that the detailed microstructure of recrystallization boundaries could not be seen clearly. In the present work a much finer step size allows a more detailed investigation to be carried out. Results are shown in Fig. 3, which presents the same surface area examined after annealing at $320^{\circ}C$ for 5min (Fig. 3a) and for another 10min (Fig. 3b). Comparison of Fig. 3a with 3b shows that after

annealing most of segments of the recrystallizing boundaries have migrated, for example protrusion 1 has further developed and detrusion 3 has disappeared, instead a new protrusion 5 and a detrusion 4 has formed. However, there are still some parts of the boundary segments which have not migrated, for example a small part of detrusion 2.



Fig. 2 Distributions of misorientations for the boundaries which moved (a) and do not move (b) during recrystallization in the CR sample. (Adapt from [4])

It is interesting to note that a protrusion can evolve into a detrusion during annealing, as seen in the region shown by the white squares in Figs. 3a and 3b. Although both detrusion 2 and the boundary segment corresponding to the new detrusion did not migrate during this annealing step, it is expected that they will migrate further at some point during continued annealing.



Fig. 3 EBSP maps at the same surface area of the partially recrystallized microstructure of the CR sample annealed at 320°C for: a) 5min; b) 5+10min.

Very similar results were observed when the ARB sample was annealed, as shown in Fig. 4 which presents the same surface area after annealing at 220°C for 10min (Fig. 4a) and for another 10min (Fig. 4b). After annealing, protrusion 1 and detrusion 2 have developed further; protrusion 3 has become a detrusion; and new protrusion 4 and detrusion 5 have formed. Besides these, it can also be seen that the recrystallizing boundaries have been pinned by a similarly oriented deformed band, i.e. there are low angle misorientations across the recrystallization boundary (i.e. an orientation pinning effect [2]). The triple junctions, as marked by arrows, also act to pin the neighboring high-angle recrystallization boundaries.

Previous research shows when a protrusion/detrusion is formed, it will provide a driving force due to boundary curvature [8]. Especially for extreme protrusions/detrusions, the driving force is in the range of 10-100MJ/m³, which is comparable with the stored energy within the deformed structure [8]. Eq. 1 then has to be written as:

$$v = M \cdot (F_D - F_\sigma), \tag{2}$$

where F_D and F_σ are the driving force contributions from the stored energy of the deformed structure and boundary curvature, respectively.

It is believed that the curvature-based driving force might be important for the local boundary migration and can be correlated with the observed stop-go type of movement. For example, a detrusion might stop for a while then start to move when the neighboring protrusions are very well

developed so that the curvature driving force can drag the detrusion forward. Similarly, a protrusion might stop while the stored energy is insufficient to act against the curvature-based driving force (a drag force) and only start to move again when the neighboring detrusions have vanished so that the curvature driving force has decreased. Therefore, protrusions and detrusions can form and disappear alternately on recrystallization boundaries during annealing thereby contributing to the stop-go motion of each boundary through the curvature-based dragging force, or, driving force.



Fig. 4 EBSP maps at the same surface area of the partially recrystallized microstructure of the ARB sample annealed at 220°C for: a) 10min; b) 10+10min.

4. Summary

The detailed structure in front of recrystallization boundaries and the migration behavior of the boundaries in heavily deformed pure nickel has been investigated using an ex-situ EBSP technique. The results show that locally many protrusions and detrusions can form and disappear alternately on a recrystallization boundary. Some of the protrusions/detrusions can provide a curvature-based dragging/driving force comparable to the driving force from the stored energy in the deformed structure. This curvature-based dragging/driving force may contribute to the stop-go motion of the boundaries.

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