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Transgranular dislocation activities and substructural evolutions accommodating two-dimensional grain boundary sliding in ODS ferritic steel

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ABSTRACT

Two-dimensional (2D) grain boundary sliding (GBS), which is useful for phenomenological understanding of superplastic and near-superplastic deformation, was achieved during a high-temperature shear test in oxide-dispersion-strengthened ferritic steel exhibiting anisotropic microstructure with largely elongated and aligned grains. In this study, 2D GBS, dislocation slip and subsequent microstructural evolutions were examined using surface markers drawn by focused ion beam and electron back-scattered diffraction analysis. In the near-superplastic state (region III), GBS was accommodated by transgranular dislocation activities initiating from grain protrusions or triple junctions into core areas, as described by the Ball–Hutchison model. The accommodation mechanisms were determined by the microstructural correlation between GBS-triggered stress concentration and available slip orientation and were closely related to the angle θ between GBS and dislocation slippage. When θ was small, GBS tended to be accommodated by a group motion of dislocations belonging to <111> {110} or <111> {112} slip systems (slip-band type). When θ was large, GBS tended to be accommodated by intragranular dislocation accumulation, which led to the development of sub-boundaries along {110} planes via dynamic recovery (sub-boundary type); this would be the origin of continuous dynamic recrystallization.

1. Introduction

Superplasticity, a phenomenon which causes extremely large deformation in fine and polycrystalline materials under particular conditions, is a strong tool for evading the trade-off properties between material strength and formability. Superplastic forming (SPF) has been in practical use for the aerospace industry [1]. Decreasing the forming time and temperature to improve the productivity is necessary for wider applications of SPF. One approach to address this issue is to extend the SPF window to lower-temperature and higher-strain-rate conditions rather than the optimum condition for superplasticity with enhanced strain-rate sensitivity m of 0.3–0.5 (region II) [2]. To ensure the reliability of this technique, it is essential to understand the deformation mechanisms and microstructural evolution processes in the near-superplastic state with moderate m below 0.3 (region II/III or III).

Continuous dynamic recrystallization (CDRX) and resultant grain refinement have been reported after superplastic or near-superplastic deformation under lower-temperature or higher-strain-rate conditions in many metals such as steels [3–5], aluminum alloys [6–8], and titanium alloys [9,10]. Understanding the CDRX mechanisms not only ensures the reliability of the non-optimum SPF techniques but also provides additional value; if microstructural evolutions can be predicted and controlled during superplasticity, the material strength may be improved by the grain refinement resulting from CDRX. To achieve such microstructural control during SPF, it is necessary to understand the relationships between CDRX and grain boundary sliding (GBS), the fundamental mechanism of superplasticity.

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GBS, the relative motion of grains with respect to one another along their boundary, has been generally believed to dominate superplasticity. In addition, GBS must be accommodated by another mechanism during which grains deform to relax the microscopic incompatibilities induced by GBS such as local stress concentration or cavitation. Several physical models of GBS coupled with accommodations have been proposed by earlier researchers and can be classified into two categories, GBS accommodated by diffusion [11,12] or dislocations [13–15]. Many surface studies have been conducted to evaluate GBS and to determine the mechanisms operative during superplasticity using scratch markers [16–18], or recently, using fine markers drawn by focused ion beam (FIB) [19–22]. These surface studies have characterized GBS and the local accommodation processes; however, only limited attention has been focused on dynamic microstructural evolutions, which include CDRX because of the following three issues:

(i) Method — GBS has been observed by surface markers as described above but there are few reports on GBS in bulk microstructure. On the other hand, microstructural evolutions have been examined by internal studies using transmission electron microscopy (TEM) or electron backscattered diffraction (EBSD) [3–9], which cannot directly characterize GBS.

(ii) Strain level — Surface markers are beneficial under small strain levels; surface studies of GBS have been reported within a strain range from 0.1 to 0.3 [16–22]. In contrast, microstructural evolutions generally become significant at larger strain levels. It is due to this inconsistency in strain levels that few reports have intentionally studied the role of GBS in microstructural developments.

(iii) Stereotype — The classical and representative models of GBS [11–15] have not considered such dynamic microstructural evolutions. As a result, the majority of GBS studies have insufficiently recognized CDRX as an important issue. In addition, CDRX studies [3–8] have also not recognized GBS as a major process during the microstructural development. As a result, the mechanism of CDRX has been constructed based only on dislocation creep.

According to CDRX studies [3–7], its mechanism is not due to discontinuous nuclear growth but by substructural development as follows. First, low-angle boundaries (LABs) form via dislocation rearrangement, i.e. dynamic recovery, and separate the original grain into subgrains. Next, the misorientation angles of the LABs increase by dislocation absorption. Finally, the subgrains transform into recrystallized grains as the LABs evolve into high-angle boundaries (HABs). These studies appear to describe the microstructural evolution processes well based on dislocation structures observed by TEM and EBSD but have almost ignored GBS without any confirmation of its absence despite these studies often being conducted in micrograin alloys with grain sizes below 10 μm. It should be carefully reconsidered whether GBS plays an important role in the CDRX process.

Recently, some of the most advanced studies [10,23] have shown strong relationships between GBS and CDRX using cutting-edge microscopy. Alabort et al. [10] applied *in situ* scanning electron microscopy (SEM) to examine superplastic and near-superplastic flows in an $\alpha+\beta$ titanium alloy and clearly showed subgrain developments accommodating GBS in region II/III. Ando et al. [23] applied a pinpoint FIB/TEM method and provided microstructural evidence of GBS and subgrain development around the sliding boundaries in a magnesium alloy. These studies have provided new insight into the mechanism linking GBS and dynamic microstructural evolutions; specifically, GBS triggered dislocation activities and these dislocations finally led to CDRX.

More direct and detailed evidence is desired to obtain a deeper understanding of the dislocation dynamics connecting GBS and CDRX from the following two viewpoints:

(i) Trigger of dislocations — There are two potential models of GBS–dislocation interactions in superplasticity, the Ball–Hutchison model [13] and the “core–mantle” model of Gifkins [15]. It should be determined which mechanism is truly operative.

(ii) Rearrangement of dislocations into sub-boundaries — CDRX has been generally considered as a random and homogeneous phenomenon in dislocation creep [3–7]. If GBS truly controls CDRX, however, there may be a rule for dislocation rearrangement.

An answer to the above questions was partly provided in our latest report using oxide-dispersion-strengthened (ODS) ferritic steel exhibiting two-dimensional (2D) GBS [24]. In region II/III, GBS was accommodated by mantle dislocation slippage near the sliding boundaries, obeying the “core–mantle” model [15], followed by CDRX of mantle areas. In region III, moreover, transgranular dislocation accommodation such as the Ball–Hutchison model [13] was also confirmed.

The objective of the present study is to reveal the mechanisms of the latter one, transgranular dislocation accommodation and substructural evolution in region III. In this study, ODS ferritic steel was also used as a 2D model aggregate. This alloy has a unique microstructure composed of anisotropic grains elongated and aligned in the rolling direction (RD), as illustrated in Fig. 1a. This alloy does not show superplastic elongation over hundreds of percent but 2D GBS within the RD plane [21,22,24,25], which is beneficial for understanding the mechanism in an early stage of superplasticity from the following two viewpoints:

(i) Simplicity — It is difficult to track three-dimensional grain movement during superplasticity. In contrast, 2D GBS makes the phenomenological understandings much easier as pointed out by Muto et al. [26].

(ii) Absence of floating grains — Some surface grains escape from the free surface during tensile superplasticity, termed “floating grains” [27]. They have no interaction with their neighbors, possibly behaving in a manner irrelevant to the bulk deformation. Therefore, surface observation of GBS may lead to a misleading interpretation of superplasticity. Such floating grains can be beneficially restricted by using 2D GBS. In the present studies, 2D grain movement is attributed to the anisotropic microstructure composed of elongated grains in ODS ferritic steel, unlike earlier studies, which have attempted to achieve 2D superplastic behavior through macroscopic torsion [27] or shear [10,20]; in their studies, some floating grains were unavoidable in the microscopic scale of individual grains.

2. Experimental

2.1. Material preparation

Table 1 lists the alloy composition of the ODS ferritic steel used in the present study. A recrystallized sheet with 0.7-mm thickness was fabricated via mechanical alloying in an argon atmosphere, hot extrusion into a round bar at 1423 K, cold rolling to 85% reduction, and heat treatment at 1423 K for 4 h. Fig. 1b, c, and d show backscattered electron (BSE) images of the recrystallized