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Two-dimensional grain boundary sliding and mantle dislocation accommodation in ODS ferritic steel

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ABSTRACT

The mechanism governing grain boundary sliding (GBS) accommodated by dislocation and microstructural evolution in regions II/III and III was studied to understand superplasticity. Two-dimensional GBS that occurred during high-temperature shear in oxide dispersion strengthened ferritic steel exhibiting an elongated and aligned grain structure was analyzed using surface markers drawn by focused ion beams. In addition, the accommodating dislocation structure was evaluated by electron back-scattered diffraction and electron channeling contrast imaging. In the initial stage of deformation, GBS triggered dislocation slippage in "mantle" areas near grain boundaries. These mantles tended to appear around GBS-resistant areas such as curved boundaries and grain protrusions. Next, the mantle dislocations generated dislocation walls before forming low-angle boundaries (LABs) along {110} crystallographic planes via dynamic recovery at the core/mantle boundaries. Finally, secondary GBS or rigid rotation occurred at the newly formed LABs to compensate for the initial GBS and resulted in continuous dynamic recrystallization. These mantle dislocation activities and substructural evolution mechanisms were graphically modeled and validated by comparison with previous studies.
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1. Introduction

Evading the trade-off between material strength and formability presents a major challenge. These properties can become compatible by applying structural superplasticity, a state observed in fine and polycrystalline materials under particular conditions. Materials exhibiting high specific strength but low formability such as titanium or aluminum alloys also achieve extremely high plasticity, making them attractive for many fields such as the aerospace industry [1]. However, the mechanism governing superplasticity remains poorly understood, which precludes the prediction and control of microstructural evolution during superplastic forming.

Recently, Alabort et al. [2] have shown that microstructural evolution depended significantly on strain rates in a superplastic titanium alloy. According to their report, grains became finer via dynamic recrystallization at high strain rates but coarser via grain growth at slow rates. However, the underlying mechanism of these microstructural evolutions remains unclear, which limits the understanding of the fundamental principles of superplasticity.

The relative motion of grains with respect to one another along their boundary, or grain boundary sliding (GBS), and its accommodation have been widely believed to predominantly occur in the superplastic state. This accommodation, during which grains undergo deformation to relieve the GBS-induced stress concentration, is regarded as rate-controlling process of superplasticity. However, the existing mechanisms remain controversial. Ball and Hutchison [3] have suggested a superplasticity model of GBS accommodated by intragranular dislocation activities. Gifkins [4] has suggested that dislocation activities may be limited to narrow "mantle" areas along grain boundaries (core–mantle model). In contrast, Ashby and Verrall [5] have proposed a model of diffusion-accommodated GBS and grain switching. Numerous studies support either of these models [6–9] but there is no widespread agreement so far. Therefore, a consensus requires a careful study based on a direct mechanistic observation of superplasticity.

"Floating grains" represent a critical bottleneck during

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microstructural observations of superplasticity. Superplasticity and GBS have been evaluated by surface observation using line markers [10,11], which leave discontinuous gaps at grain boundaries when GBS occurs. However, some grains escape from the free surface during conventional tensile tests. These so-called "floating grains" do not need to undergo deformation by accommodation in the superplastic state because they do not interact with neighboring grains. They may behave in a manner irrelevant to their bulk counterparts, which may skew observations of superplasticity. Watanabe et al. [12] have detected different texture formations during superplasticity near the surface compared with in the bulk, emphasizing that surface observation may provide misleading results.

To avoid floating grains, Mayo and Nix [13], Rust and Todd [14], and Alabort et al. [15] have conducted shear tests, and deformed specimens within two dimensions because no floating grain would result from a truly two-dimensional deformation. This approach prevented the emergence of new grains from the subsurface at the free surface despite the presence of microscopic floating grains in certain parts. Rust et al. [14] investigated whether these microscopic grains presented any problem through careful experiments and discovered different phenomena at and beneath the surface even in the same grain. In particular, a grain-switching event actually occurred below the surface without appearing on the surface. This difference may be attributed to the absence of interaction between floating grains on the surface.

Even in a macroscopically two-dimensional deformation, out-of-plane grain movements have been unavoidable as long as the grains are equiaxed [13–15]. If an anisotropic microstructure comprising elongated and aligned grains were sheared perpendicularly to the longitudinal direction, grain movement would only occur in two dimensions (Fig. 1). This idea is similar to previous attempts by Muto et al. [16], who deformed a bundle of pencils acting as a two-dimensional model aggregate and evaluated their sliding behaviors. Based on this concept, two-dimensional GBS has been achieved and investigated in oxide dispersion strengthened (ODS) ferritic steel exhibiting an elongated and aligned grain structure [17–20]. Although this alloy does not make a superplastic elongation over hundreds of percent but a sigmoidal stress–strain rate relationship and two-dimensional GBS, which have been highly useful for interpreting an initial stage of superplasticity.

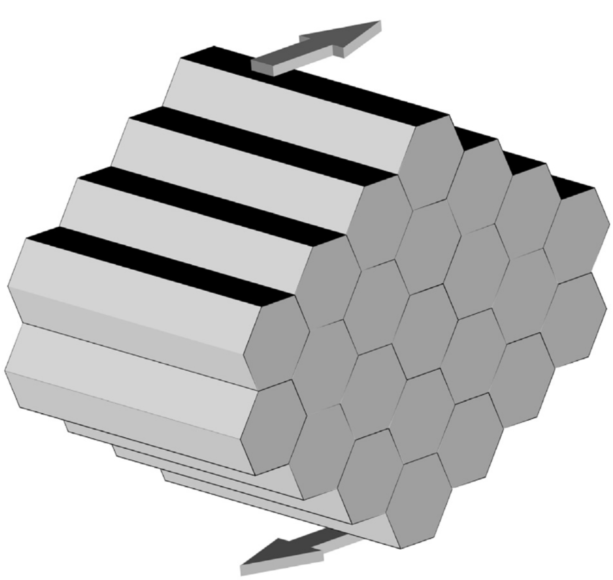

Fig. 1. Anisotropic microstructure composed of elongated and aligned grains for two-dimensional GBS.

In our former work [18], the deformed microstructures were evaluated by electron back-scattered diffraction (EBSD) in conjunction with surface microgrids drawn by focused ion beam (FIB) in regions II (optimum for GBS), II/III (border), and III (dislocation creep), respectively. Significant GBS was detected in regions II and II/III, where the density of geometrically necessary dislocations (GNDs) increased around the sliding boundaries. The GND density increased more significantly in region II/III than in region II, suggesting a larger contribution of dislocation activities in region II/III. On the other hand, non-dislocation mechanism, e.g. diffusion of matter, might result in more contribution in region II.

Local accommodations via dislocation activities have been observed in region II/III [19,20]. Dislocation slippage accommodated GBS in mantle areas near sliding boundaries only [19], which is consistent with the "core–mantle model" proposed by Gifkins [4], and produced new low-angle boundaries (LABs) cutting grain protrusions [20]. However, details of the dislocation accommodation and microstructural evolution have yet to be studied comprehensively.

In this study, the GBS-triggered mantle dislocation mechanism and its effects on microstructural evolution were unified and a comprehensive model was graphically proposed. Shear tests were conducted to achieve more refined evaluation than ever. Compared with conventional tensile tests that generated two GBS modes around 45° and 135° with respect to the tensile axis [18,19], shear tests produced only one GBS mode along the shear direction. This one-mode GBS is expected to facilitate a better characterization of GBS networks and analysis of accommodations such as intragranular dislocation activities.

2. Experimental

2.1. Material preparation

This study rests on the preparation of an anisotropic microstructure consisting of fine and elongated grains for two-dimensional GBS.

The alloy composition of ODS ferritic steel used here is shown in Table 1. Metal and Y_2O_3 powders were mechanically alloyed in an argon-gas atmosphere and hot-extruded at 1423 K into a round bar featuring dispersed nanosized Y_2O_3 particles. An almost identical material comprised spherical Y_2O_3 particles averaging 5 nm in diameter and occupying a volume fraction of 0.40 vol% [21]. The extruded sample was cold-rolled to achieve a reduction of 85% and annealed at 1423 K for 4 h before water quenching to create a recrystallized microstructure. The Y_2O_3 particles pinned grain boundaries and prevented their migration. Therefore, the grain sizes and shapes depended on the particle distribution. Fig. 2 shows inverse pole figure (IPF) maps obtained from EBSD analyses of an as-annealed sample. The recrystallized grains were largely elongated and aligned along the rolling direction (RD).

2.2. Specimen preparation

A schematic representation of a shear test specimen is shown in Fig. 3a. First, the as-annealed sample was cut into a tensile specimen with gauge width of 1.5 mm and thickness of 0.7 mm. Then, a

Table 1
Chemical composition of ODS ferritic steel.

Fe	Cr	C	W	Al	Zr	Ti	Y_2O_3
Bal.	15	0.03	2	3.8	0.32	0.12	0.35

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