Microstructure effects on the recrystallization of low-symmetry alpha-uranium

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HIGHLIGHTS

- We study the recrystallization behavior of uranium using EBSD.
- We are able to relate the recrystallization texture to measured characteristics of the deformed microstructure.
- High angle grain boundaries are recrystallization nucleation sites, but twin boundaries are not.

ARTICLE INFO

ABSTRACT

We employ electron backscatter diffraction (EBSD) to investigate microstructural evolution of uranium during recrystallization. To understand the relationship between microstructure and recrystallization, we use measures of intra-granular misorientation within grains and near grain boundaries in both deformed (non-recrystallized) uranium and recrystallizing uranium. The data show that the level of intra-granular misorientation depends on crystallographic orientation. However, contrary to expectation, this relationship does not significantly affect the recrystallization texture. Rather, the analysis suggests that recrystallization nucleation occurs along high angle grain boundaries in the deformed microstructure. Specifically, we show that the nucleation of recrystallized grains correlates well with the spatially heterogeneous distribution of high angle boundaries. Due to the inhomogeneous distribution of high angle boundaries, the recrystallized microstructure after long times exhibits clustered distributions of small and large grains. Finally, twin boundaries do not appear to act as recrystallization nucleation sites.

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1. Introduction

Similar to the processing of other metals, uranium components are commonly made by a series of mechanical and thermal processes. Mechanical processes such as forging, rolling, swaging, and forming are used to refine the starting cast microstructure and make metal forms for further processing to produce final parts. Thermal annealing processes are interspersed within the mechanical processes to produce finer, recrystallized grains and to soften and restore ductility [1] to a metal for further mechanical processing. The combination of mechanical and thermal processes has significant effects on the evolving microstructures; i.e. grain size, grain morphology, and texture. Significant progress has been made in understanding the microstructure evolution that accompanies deformation of uranium [2—6]. However, little work has

been done to understand the recrystallization behavior of uranium [7].

There is a large body of work examining the recrystallization behavior of other metals. The physics, phenomenology, and modeling of recrystallization is well documented in several review articles [1,8—11]. A vast majority of recrystallization work to date examines recrystallization in metals with face centered cubic (FCC) crystal structures. The recrystallization of body centered cubic (BCC) metals has also received considerable attention, and limited efforts have looked at the recrystallization behavior of hexagonal close packed (HCP) metals [12—15].

One principal aim of the aforementioned body of work is to relate the deformed microstructure to the recrystallized microstructure under subsequent annealing. As defined by Refs. [1], "recrystallization is the formation of a new grain structure in a deformed material by the formation and migration of high angle grain boundaries driven by the stored energy of deformation." Over

the years, several specific characteristics relating deformed and recrystallized microstructures have been shown. Recrystallization nuclei already exist in the deformed microstructure; i.e., recrystallized grains grow from orientations that are present, although they may not represent dominant deformation orientations, in the deformed microstructure [1,8,9]. Because recrystallization occurs through the motion of high angle grain boundaries, recrystallization nuclei are expected to be in close proximity to the high angle boundaries in the deformed microstructure [8,9]. Grain boundary character [16,17] has also been shown to influence grain boundary mobility, and thus, the rate of growth of nucleating grains [9,10]. In some metals, grain boundaries with certain character are more mobile and this dependence can have a significant influence on the evolving, recrystallizing microstructure. The driving force for recrystallization is the stored energy of deformation, primarily in the form of dislocations [9,10]. The driving force for recrystallization is expected to be higher in grains or regions of high dislocation content. A successful recrystallization nucleus is also expected to have an energetic advantage [10] in addition to sharing a high energy boundary. Finally, the recrystallized grain size depends on the grain size prior to deformation and the amount of deformation applied to the material [9,18].

As stated previously, many of the above relationships have been reported for deformed metals with cubic crystal structures. Recrystallization of deformed metals with crystal structures of lesser symmetry, such as orthorhombic uranium, has not received nearly as much attention and is comparatively less well understood. These metals deform via several modes of slip and twinning and the relative amounts of these modes are highly sensitive to grain orientation. Thus, interesting and potentially distinct relationships between orientation and the propensity to recrystallize could arise. In the present study, we use electron backscatter diffraction (EBSD) to statistically analyze the relationship between a few important microstructural characteristics to recrystallization in deformed uranium. EBSD is an excellent tool for this study allowing for statistically relevant comparisons of spatially correlated orientation information. In particular, with EBSD it is possible to partition recrystallized grains from the deformed microstructure [7]. Taking advantage of this capability, we show that the development of intra-granular misorientations depends on grain orientation. Interestingly, this relationship has an insignificant effect on recrystallization texture. Our analysis suggests that recrystallization nucleation occurs along high angle grain boundaries in the deformed microstructure, which does affect the evolving texture. In contrast, the twin boundaries do not appear to act as recrystallization nucleation sites.

2. Experimental

The processing and experimental methods are nearly identical to those reported previously [7]. The previous study was one of the first successful uses of EBSD on uranium, and showed that recrystallized grains can be distinguished from non-recrystallized grains. A brief discussion of the manufacturing and specimen preparation is given here with details provided in Ref. [7]. The present study is based almost entirely on new EBSD data. Any experimental differences from the prior study and distinguishing details of the EBSD analysis will be highlighted.

A series of processing steps were used to produce a uranium plate including vacuum induction casting, hot upset forging (625 °C), and warm clock-rolling. The clock-rolling was performed at 300 °C in eight equivalent strain passes with rotations of 0°, 90°, 135°, 225°, 270°, 360°, 45° and 135° resulting in a final reduction of approximately 50%. The warm-rolling temperature is considerably below temperatures for which recrystallization is observed in

rolled uranium for similar levels of deformation, and dynamic recrystallization is not expected for this processing. Isothermal annealing experiments were performed in a quench dilatometer on 3.173 mm diameter, 10 mm long cylindrical samples with the axis of the sample along a consistent in-plane plate direction. Annealing experiments were performed for various times at 435 °C, 450 °C, and 475 °C under a vacuum of 10^{-5} Torr with a heating rate of 7.5 °C/s and quench rate of 10 °C/s to room temperature.

Axial sections cut from near the centers of the dilatometer samples were mounted and metallographically prepared. The EBSD preparation technique consists of polishing to a 1 μm diamond finish followed by two electropolishing steps. The first electropolishing step uses a room temperature, stirred solution of 45% ethanol, 27% ethylene glycol, and 27% phosphoric acid at 10 V for around 4 min. The second electropolishing step uses a room temperature solution of 5% phosphoric acid and 95% water at 5 V for 1–2 s.

Automated EBSD scans were performed at 25 kV in an FEI XL30 SEM equipped with TSL/EDAX data acquisition software. Regions 300 μm by 600 μm roughly at the center of the 3.175 mm diameter mounted specimens were scanned with a step size of 0.5 μm for microstructure analysis. In addition to the microstructure scans, the entire surfaces of the samples were scanned using a step size of 5 μm to generate "macro" textures. The orientation data was analyzed using TSL/EDAX Orientation Imaging Microscopy (OIM) Analysis 7.2 software. A minimal EBSD clean-up was performed on each of the microstructure scans using a neighbor confidence index correlation with a minimum confidence index of 10% of the average for the scan. Because samples were cut from round cross-sections, a priori alignment of samples in the microscope with a consistent orientation relative to the plate normal direction was not possible. Thus, in some maps, deformed grains are elongated vertically (Fig. 1a), and in other maps deformed grains are elongated horizontally (Fig. 1c) or at arbitrary angles. Texture symmetries are used for orientation alignments with observed grain shapes used for validation.

3. Results

We use EBSD to relate the deformed microstructure to recrystallization phenomena. Fig. 1 shows EBSD orientation maps of samples heat treated for different amounts of time at 450 °C. With these data, we quantitatively compare the characteristics of the as-deformed and recrystallizing microstructures. We pay special attention to local and grain level misorientation and characteristics of high-angle grain boundaries. These choices are based on the assumptions that 1) measures of misorientation correlate with the local defect densities that are expected to serve as a driving force for recrystallization and 2) the motion of high angle boundaries are the mechanism for recrystallization.

To this end, it is necessary to identify in EBSD a recrystallized grain from a non-recrystallized one. Partitioning the recrystallized grains from the non-recrystallized grains allows us to directly measure the recrystallized fraction as a function of time and temperature. As with previous studies [7,19], it is found that measures of internal grain misorientation work remarkably well for partitioning (separating) recrystallized grains from non-recrystallized grains. This is consistent with previous observations and theories of recrystallization where new, relatively defect-free grains form at the expense of non-recrystallized grains that contain large densities of dislocations. These higher dislocation densities result in higher levels of internal grain misorientation.

One measure we use is the Grain Orientation Spread (GOS). GOS is the average deviation between the orientation of each scan point in a given grain and the average grain orientation. Fig. 2a shows the