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Effect of initial orientation on dynamic recrystallization of a zirconium alloy during hot deformation



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ABSTRACT

The present study investigated the influence of the initial orientation on the dynamic recrystallization (DRX) behavior of a Zr-1Sn-0.3Nb alloy. A hot-rolled and annealed Zr sheet was compressed along two directions: along the normal direction so that the grains' $\langle c \rangle$ -axis was nearly parallel to the loading direction (0° sample) and along the transverse direction so that the grains' <c>-axis was nearly perpendicular to the loading direction (90° sample). The samples were compressed at 700 $^{\circ}$ C at a strain rate of 1 s⁻¹. The microstructures at different strains were characterized by the electron backscatter diffraction (EBSD) technique, and conventional dislocation analysis using transmission electron microscope (TEM) was performed. A threshold value of grain orientation spread (GOS) equaled to 5° was used to distinguish the dynamically recrystallized grains from the deformed matrix. The results revealed that the DRX behavior strongly depended on the initial orientation. The Schmid factor analysis and TEM observation confirmed that pyramidal $\langle c + a \rangle$ slip operated from the first stage of deformation in the 0° sample but not in the 90° sample. For the 0° sample, in the early and medium stages of deformation, due to the high stored energy caused by the operation of pyramidal $\langle c + a \rangle$ slip, DDRX was mainly contributed to the formation of new fine grains. However, in the later stage of deformation, the DRX mechanism changed from DDRX to CDRX. In the 90° sample, although DDRX featured by grain boundary bulging occurred, the main DRX mechanism was CDRX in the whole deformation processing. Moreover, texture induced hardening in the early stage of deformation hides the softening induced by DDRX.

1. Introduction

Zr alloys are widely used in nuclear reactors because of their excellent mechanical properties, good corrosion resistance, and low neutron absorption [1,2]. The plastic deformation mechanisms observed in Zr and its alloys include slip and twinning. Because the critical resolved shear stress (CRSS) for the activation of $\{10\overline{1}0\}\langle 11\overline{2}0\rangle$ prismatic slip is the lowest, this slip is the most easily activated mode. Slip systems in the $\langle a \rangle$ -direction also include the $\{0001\}\langle 11\overline{2}0 \rangle$ basal slip and $\{10\overline{1}1\}\langle 11\overline{2}0\rangle$ pyramidal $\langle a \rangle$ slip. Two pyramidal slip systems in the $\langle c + a \rangle$ -direction— $\{10\overline{1}1\} \langle 11\overline{2}3 \rangle$ and $\{11\overline{2}1\} \langle 11\overline{2}3 \rangle$ pyramidal $\langle c + a \rangle$ slips—cause plastic deformation along the <c>-axis of the grains. Depending on the strain applied along the $\langle c \rangle$ -axis of a particular crystal, twinning observed in Zr includes $\{10\overline{1}2\}\langle\overline{1}011\rangle$ and $\{11\overline{2}1\}\langle11\overline{2}6\rangle$ tension

twinning or $\{11\overline{2}2\}\langle 11\overline{2}3\rangle$ and $\{10\overline{1}1\}\langle 10\overline{1}2\rangle$ compression twinning [3-9]. Zr is a hexagonal close-packed (HCP) metal; then, owing to the anisotropy of the HCP structure, the deformation mechanisms of Zr alloys depend on the crystallographic texture [8-10]. A previous work reported that the onset of different slip or twinning systems depends on the angle X_B between the tensile direction and the basal plane: for $X_B < 35^\circ$, the first-order prismatic slip is responsible for yielding, whereas for $X_B > 35^\circ$, crystal deformation is dominated by prismatic slip and $\{11\overline{2}1\}$ twinning at liquid nitrogen temperature [7]. Tomé et al. found that in the case of compressive deformation of pure Zr along or perpendicular to the $\langle c \rangle$ -axis of the grains, the flow stress variation and strain hardening behaviors are caused by different activations of the slip and twinning systems in the two loading directions [8,9].

In general, only 5-10% of deformation energies are absorbed by a

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deformed metal, and these absorbed energies are mainly stored as a dislocation structure which generates during deformation. So that the magnitude of stored energy is orientation dependent. The amount of stored energy has a significant influence on the nucleation and growth of new grains during recrystallization, which can further affect microstructure evolution and grain texture. Hadadzadeh et al. [11] claimed that the dynamic recrystallization (DRX) mechanism for an extruded ZK60 magnesium alloy deformed along the extrusion direction is discontinuous DRX (DDRX) and along the radial direction is continuous DRX (CDRX). Galiyev et al. [12,13] confirmed that the DRX mechanisms in Mg are governed by the operating slip and twinning systems, which change with temperature: examples of these mechanisms include bulging of original grain boundaries and growth of subgrains (at 573-723 K) and CDRX (at 473-523 K). Dudamell et al. [14] found that DRX is promoted by the operation of $\langle c + a \rangle$ slip along with cross-slip and climb in an AZ31 magnesium sheet deformed at dynamic strain rates ($\sim 10^3 \text{ s}^{-1}$) at 250 °C. Srinivasarao et al. [15] pointed out that DDRX can be activated more easily when all three slip systems (basal, prismatic, and pyramidal $\langle c + a \rangle$ slips) are activated in the AZ31 magnesium alloy. Honniball et al. [10] reported that the temperature and initial texture have a great influence on the grain breakup in Zircaloy-4. And Gerspach et al. [16] indicated that the recrystallization texture is influenced by the grain fragmentation.

In a previous study [17] on the effects of initial texture on the mechanical behaviors of Zr alloys, we found that the fraction of recrystallized grains having low internal misorientation is higher when the $\langle c \rangle$ -axis of the grains is normal to the compression direction. However, the previous study did not investigate in detail the DRX mechanisms involved in samples with different initial orientations. On the other hand, a few studies have thus far focused on the DRX behavior of Zr alloys, and several DRX mechanisms have been proposed in these studies. Sarkar et al. [18] observed that DRX occurs during the high temperature deformation of Zr-1Nb alloy and that the dynamically recrystallized grains are formed along the boundaries of deformed grains. Chakravartty et al. [19] indicated that the DRX process in five Zr-alloys is controlled by nucleation, i.e. the DRX mechanism is DDRX. Hayes et al. [20] claimed that dislocation climbing is the restoration mechanism for Zr during creep in the five-power-law regime based on creep data over a wide range of stresses (0.1 to 115 MPa) and temperatures (300-850 °C). Perez-Prado et al. [21], who conducted a detailed study (tension, torsion, and creep tests performed at temperatures of 400–800 °C) on dynamic restoration in α -Zr, reported the occurrence of geometric dynamic recrystallization (GDRX). Further, Chauvy et al. [22] reported the occurrence of CDRX during high-temperature deformation of Zircaloy-4. Logé et al. [23] reported that DDRX occurs at high strain rates and dynamic recovery (DRV) occurs at lower strain rates in Zircaloy-4 deformed at temperatures higher than 600 °C. Results suggest the existence of a relationship between deformation mechanisms and DRX mechanisms; however, further investigation is required to confirm this relationship. Therefore, in the present work, in order to investigate the DRX in a Zr alloy, samples of a Zr alloy with two different initial orientations were compressed to different strains at a strain rate of 1 s⁻¹, and their microstructures were characterized using the electron backscatter diffraction (EBSD) technique. Moreover, with the aim of identifying the dislocation types in the two samples, conventional dislocation analyses using transmission electron microscope (TEM) were performed.

2. Materials and Methods

A hot-rolled and annealed Zr-1Sn-0.3Nb alloy (1.06Sn-0.36Nb-0.3Fe-0.1Cr-0.13O and balance Zr, in wt%) plate was used in this work. Two types of cylindrical samples, 6 mm in height and 6 mm in diameter, were cut from the plate: the axial direction of one sample was parallel to the plate normal direction (ND) and that of the other sample was parallel to the plate transverse direction (TD) (see Fig. 1); these two



Fig. 1. Method of sampling from original sheet.

samples are hereafter referred to as the 0° sample and the 90° sample, respectively. Fig. 2 shows the microstructure and {0001} pole figure of the initial plate along the ND (0° sample) and TD (90° sample). The Zr alloy plate exhibited a bimodal texture with {0001} poles lying in the TD-ND plane, and mostly tilting at approximately 20°–30° away from the ND (see Fig. 2). In the 0° sample and 90° sample, the $\langle c \rangle$ -axis of the grains was nearly parallel and nearly perpendicular, respectively, to the loading direction of the sample.

Compression experiments were performed at 700 °C in vacuum at a strain rate of 1 s⁻¹, by using a Gleeble-3500 thermal simulator. In order to obtain more recrystallized grains at high strain rate (1 s^{-1}) , the deformation temperature was selected as high as possible. Moreover, at 700 °C, considering the adiabatic temperature rise, the samples' temperatures during deformation were still lower than the phase transition temperature (787 °C) of this Zr alloy. A tantalum sheet was added between the sample and compression dies as lubricant to minimize friction. The specimens were heated to 700 °C at 5 °C/s, and then held for 180 s at that temperature before the deformation. After completion of the compression experiments, the samples were immediately quenched by a water jet to freeze the deformed microstructure. The deformed samples were cut along the compression axis for microstructure observation via EBSD. Sample preparation for EBSD investigation consisted on standard mechanical polishing followed by electro-polishing with a solution of 10% perchloric acid, 20% 2-Butoxy ethanol and 70% Methanol at a voltage of 20 V for 12 s at -30 °C. The microstructure observation was carried out at the center of the sample's vertical section by using a Tescan Mira 3-XMU scanning electron microscope (SEM) equipped with a backscatter electron detector and an EBSD analysis system (AZtec, Oxford Instruments). Moreover, EBSD maps were measured on 139.7 $\mu m \times 139.7 \, \mu m$ areas with a step size of 0.1 $\mu m.$ TEM observation was performed on FEI F20 operated at 200 kV. The deformed specimens were firstly sliced across the mid-plane along the compression axis, and these slices were mechanically polished to a thickness of 55 μ m, then punched into Φ 3 mm discs and finally doublejet electrolytically polished with a solution of 10% perchloric acid and 90% ethanol at a voltage of 30 V at -30 °C.

The grain size and area were automatically calculated from the EBSD maps, measured by circle equivalent diameter with Channel 5 software. For this calculation, a grain was defined as a region completely bounded by boundaries with a misorientation angle larger than 15°. In addition, if the number of the points in a grain was less than ten, that grain was discarded. Identification of recrystallized grains was also performed using the Channel 5 software based on the grain orientation spread (GOS) value of a grain. Several studies have chosen a threshold value of 1–2° for GOS to distinguish the dynamically recrystallized grains successfully [24,25]. However, during the process of DRX, some grains with high GOS values were the recrystallized grains that formed at lower strain but undergo deformation at higher strain [26]. Therefore, recrystallized grains were defined as individual grains with a measured GOS \leq 5° in this study. The recrystallized fraction could subsequently be calculated.

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