Journal of Alloys and Compounds 742 (2018) 29–37

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Effect of pre-annealing deformation on thermally activated twin boundary migration in a zirconium alloy

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ARTICLE INFO

Article history:
Received 16 September 2017
Received in revised form
18 January 2018
Accepted 21 January 2018

Keywords:
Twin boundary migration
Annealing
Texture
Twinning
Zirconium alloy

ABSTRACT

Uniaxial compression along the rolling direction was conducted in a Zr-4 alloy sheet to induce twinning followed by annealing at various temperatures. The microstructure and texture before and after static annealing were investigated by electron backscatter diffraction (EBSD) to evaluate the possibility to tailor the recrystallized microstructure and texture of Zr alloys by pre-twinning and annealing. It is worth to note that microstructure and texture affect the performance of Zr alloys, for instance corrosion resistance, creep and delayed hydride cracking. During annealing, the twins show relative thermostability in the sample with a low pre-annealing deformation (5%), whereas obvious thermally activated twin boundary migrations start at a relative low temperature (650 °C) in the sample with a large pre-annealing deformation (20%). The texture induced by twinning was further strengthened during recrystallization annealing as a result of the increase in the twin area fractions. More coherency losses and a higher diving force in the sample with a larger pre-annealing deformation may help to induce twin boundary migration at lower annealing temperatures.

1. Introduction

Twinning is an important plastic deformation mode for zirconium (Zr) and its alloys as a result of their hexagonal-close-packed (HCP) structures, which are not as symmetrically distributed as cubic metals and cannot offer enough independent slip systems [1,2]. Four twinning modes, two tensile twinning and two compressive twinning, have been often observed in Zr alloys, where tensile and compressive twinning are named for their ability to accommodate the strain along the *c*-axis [3,4]. For tensile deformation along the c-axis, tensile twinning is more easily activated. Compressive twinning is initiated as when the c-axis experiences compression. The activation of twinning can cause large lattice rotations. For example {10–12}, tensile twinning rotates the c-axis by approximately 85° and {11–22} compressive twinning flips the c-axis by approximately 64°, leading to a distinct change in the crystalline texture of a Zr alloy [4,5].

Many studies have been carried out to investigate twinning in Zr alloys [6–10]. Pre-twinned Zr samples were reloaded at different temperatures to explore the role twinning has during hardening of Zr and dislocation/twin interactions [11,12]. It was found that twins influence the reload behavior by either reorienting the material for easy slip or by providing barriers to slip movement [11]. The effect of pre-stored dislocations on the onset and growth of {10–12} and {11–22} twins in pure Zr was investigated by Capolungo et al. [13]. A statistical analysis was carried out by Capolungo et al. [7] to expose correlations between {10–12} twinning activation process and grain size, crystallographic orientation, grain boundary length, and misorientation in high-purity polycrystalline Zr. A strong correlation was found between the activated twin variants and crystallographic orientation [7]. In-plane and through thickness compression operations were performed in a textured Zr sheet to study the contributions of twinning on the total compression plastic strain [14]. A set of thermal mechanical processes were proposed by Chung [15] to introduce a high volume fraction of twins in Zr. A statistical study on the influence of twin–twin junctions on the nucleation and growth of twins was conducted by Juan et al. [5]. The results indicated that twin–twin junctions hindered twin growth [5]. Higher strain rates were reported to cause higher twinning fractions in the Zr-1Nb alloy [16]. The effect of temperature and rate dependence of twinning and secondary twinning in high-purity Zr over a wide range of temperatures and strain rates (from 76 K to 673 K and 0.001 s^{-1} to 4500 s^{-1}) has also been investigated [17]. The results implied that all the studied twin

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https://doi.org/10.1016/j.jallcom.2018.01.288
0925-8388/

modes were rate insensitive and the strong influence of strain rate and temperature on twinning was due to the rate-sensitivity of the slip [17].

Annealing is an important process to tailor the microstructure and property of metals [18,19]. The recrystallization mechanisms and the texture evolution during primary recrystallization of low-alloyed Zr sheets can be quite different for samples with deformation textures and pre-deformed under different deformation paths [20]. During annealing of a Zr-2Hf alloy, in-situ HVEM investigations indicated that dense arrays of dislocations rearrange into a subgrain structure during recovery and subgrains growth was then at the origin of the formation of recrystallization nuclei [21]. In a 50% rolled Zr-2Hf alloy, ⟨10-10⟩//RD and ⟨11−20⟩//RD fiber texture components were equally present in the recrystallized material at all stages of primary recrystallization, thus showing equal nucleation capacity [22]. While, in subsequent grain growth stage, ⟨1−20⟩//RD fiber texture component was strengthened at the expense of ⟨10−10⟩//RD component [22]. Although many studies have been carried out to explore the recrystallization mechanism and texture evolution of Zr alloy during annealing, most of researches focused on microstructures deformed by dislocation slip [23,24]. As mentioned before, twinning is also an important deformation mechanism for HCP structured Zr alloy. However, few studies on the evolution of twins during annealing, especially during recrystallization annealing, are available in the literature. For magnesium alloys, which are also HCP metals, deformation twinning has a significant influence on recrystallization behavior [25]. Pre-stored dislocation and the twin size can affect the subsequent twin boundary migration during annealing, which has a remarkable effect on the microstructure and texture [26–28].

Recently, Isaenkova et al. [29,30] studied the texture evolution of pure Zr single crystals during deformation and annealing by using X-ray diffraction (XRD). They reported that twinning induced texture was enhanced up to 53% rolling deformation. During the recrystallization annealing of ~50% rolled pure Zr single crystal, the twinned regions were characterized by a reduced tendency to recrystallize, thus leading to a decrease of twinned texture intensity [29,30]. But microstructure evolution was not provided in their results. In this work, we investigate the evolution of twins during annealing for a Zr alloy using twins induced by pre-compression followed by annealing at various temperatures. As a first step, we focus on tensile twinning in this work. The microstructure and texture of Zr samples before and after annealing were characterized by electron backscatter diffraction (EBSD). The motivation of this study is to explore a possible method for texture tailoring of Zr alloys by pre-twinning and annealing. Texture is generally accepted to have a significant effect on the performances of Zr alloys [31,32].

2. Experiments

A Zr alloy (Zr-4), with a nominal chemical composition of Zr-1.4%Sn-0.2%Fe-0.1%Cr, was used as the experimental material in this work. The as-received Zr-4 sheet (with a thickness of 2.2 mm) was provided by the State Nuclear Baoti Zirconium Industry and was fabricated through rolling and annealing. The as-received Zr-4 sheet has a recrystallized microstructure.

To induce twinning, the as-received Zr-4 sheet was compressed at room temperature along the rolling direction (RD). Nominal compression strains of 5%, 10% and 20% were imposed. Thereafter, the deformed Zr-4 samples were annealed at 650 °C and 700 °C for 2 h. A 2 h annealing time was selected based on our previous research experience, that the recrystallization can be achieved during this time period [33]. For the 20% deformed sample, an extra annealing round at 600 °C for 2 h was conducted. Here, the specimens are named after the compression strain and annealing temperatures. For example, the specimen with a 10% compressive deformation that was annealed at 650 °C for 2 h is denoted as 10%-650.

The microstructure and texture evolution before and after annealing was characterized by a field emission gun scanning electron microscope (FEI Nova 400) equipped with an electron backscatter diffraction (EBSD) detector and commercial available analysis system (Channel 5, HKL Technology-Oxford Instruments). The EBSD technique was very good at revealing microstructural characteristics related to local crystallographic orientation. In this work, EBSD data for each specimen were collected using a step size of 0.3 μm. The area for microstructural characterization was located at the center of the transverse direction (TD)-normal direction (ND) plane. 100 μm × 100 μm and 200 μm × 200 μm were scanned for the deformed and annealed samples, respectively. To avoid the effect of insufficient grain number on the texture measurement, pole figures for some samples were also measured using X-ray diffraction (XRD, Rigaku D/max-2500PC).

3. Results

3.1. As-received and deformed microstructure

The as-received and deformed microstructures are presented using orientation imaging and boundaries distribution maps. The grains in the orientation imaging maps are colored according to the orientation of sample's RD in a standard triangle of crystal stereographic projection, as indicated in Fig. 1(a). For convenience, the orientation imaging maps are often called IPF maps in literatures (e.g. Refs. [34,35]) since the coloring rule is closely related to an inverse pole figure. In the IPF maps and boundaries distribution maps, the grain boundaries, with misorientation angles higher than 10°, are represented with dark solid lines. The sub-grain boundaries, or sub-boundaries, with misorientation angles higher than 2° and less than 10°, are depicted by gray solid lines. In the boundaries distribution maps, the {10−12} tensile twin boundaries are represented with red solid lines. Twin boundaries are determined by the characteristic rotation angle and rotation axis pair, in which a maximum rotation angle deviation of 5° was used if no special emphasis is given. Fig. 1(a) indicates that the microstructure of the as-received Zr-4 sample is composed of equiaxed grains and is almost free of sub-boundary and twin boundaries, indicating a recrystallized state. The {0001} pole figure of the as-received Zr-4 sample is also presented in Fig. 1(a), which shows that the c-axis is mainly orientated along the ND with an inclination of ~30° with respect to the TD. This is typical TD-split basal texture, which is very common in Zr alloy sheet produced using a rolling process [4,36].

After the 5% compression along the RD, some twins were activated, as indicated in Fig. 1(b). All lamellar twins are identified as {10−12} tensile twins according to their rotation angle and axis. During RD compression, the loading tends to cause tensile deformation along the ND (c-axis), leading to the activation of tensile twinning instead of compressive twinning. The {10−12} tensile twinning rotates the c-axis by approximately 85° [4], which aligns the c-axis with the RD in lamellar twins, causing the lamellar twins to appear as a red color (parallel with RD) in the IPF map. Since the color of the initial grains (before deformation) are far from red, the red grains or lamellar twins in the IPF map can be roughly treated as the twin induced microstructure. As the compression strain increases, the area fraction of the twins increases, as shown in Fig. 1(c)−(d). More than one lamellar twin can be observed in a single grain. Moreover, the twin size increases by growth or merging with other twins as the deformation increases. Some twins consume the entire grain in the 20% deformation sample (Fig. 1(d)). In addition, more sub-boundaries are observed as the deformation