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Effect of loading direction on the deformation and annealing behavior of a zirconium alloy

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ABSTRACT

Zr alloys are used as cladding materials in nuclear reactor, and new alloys and process are continuously developing. This work focuses on the influence of loading direction on deformation and subsequent annealing behavior of hexagonal Zr-4 alloy. As-received Zr-4 sheets with recrystallized microstructure and strong crystallographic texture were compressed along the normal direction (ND) and rolling direction (RD) at room temperature. Different strain levels were applied and subsequent annealing at various temperatures was carried out. Microstructures and textures were characterized by electron backscatter diffraction (EBSD) method. For a same strain level, the fraction of low angle misorientation is higher after ND compression than RD compression, indicating higher stored dislocation density. During annealing, obvious recrystallization causes grain growth in ND compressed specimens when the annealing temperature is higher than 650 °C, while no significant recrystallization happens in RD compressed specimens, even annealed at 700 °C. Visco-plastic self-consistent (VPSC) simulations are used to evaluate the type of stored dislocations, their density and the stored energy, and explain the different annealing behaviors after ND compression and RD compression.

1. Introduction

At room temperature and bellow ~882 °C, zirconium (Zr) have hexagonal close-packed (HCP) structure, characterized by an insufficient number of independent slip systems activated [1]. Prismatic slip ({10−10} < −12 to 10 >) is well accepted as the primary deformation mode in HCP Zr alloys [2]. However, prismatic slip can not accommodate deformation along the *c-axis* (normal direction of basal plane). Pyramidal < c+a > slip ({10−11} < −1 to 123 >) and twinning ({10−12} tensile twinning and {11−22} compressive twinning) are also some important deformation modes in Zr alloys although they have much higher critical resolved shear stress (CRSS) than prismatic slip [3–5]. The relative contribution of these deformation modes strongly depend on crystal orientation, temperature and strain rate [6–8]. As results of the low symmetry of HCP structure, highly textured Zr alloys generally show strong anisotropy. When loaded along different directions or different stress states, the relative activity of the deformation modes changes, thus displaying anisotropic yielding, plastic flow, *etc*… [9–11]. Crystal plasticity modeling is a useful method to investigate the relative contribution of various deformation modes, which has been applied in many studies [12–14]. Generally, a set of parameters have to be determined in a crystal plasticity model, for example in Visco-plastic self-consistent (VPSC) model [15]. The parameter values for a specific material are identified by using the starting texture measurement and fitting several true stress-strain curves.

Annealing is an important procedure in the fabrication of Zr alloys components, which allows different microstructural and mechanical changes resulting from recrystallization or recovery [16–18]. Many studies have been performed to investigate the annealing related microstructure or texture changes [19–21]. Zhu *et al.* [19,20] investigated the primary recrystallization mechanisms and texture changes of Zr-2Hf alloy during annealing. Hiwarkar *et al.* [22] reported that non-homogenous deformation can cause early recrystallization by preferred nucleation during annealing. For moderately deformed Zr, Jedrychowski *et al.* [23] stated that strain induced boundary migration was the main recrystallization mechanism during annealing. Isaenkova *et al.* [24,25] found that twinned regions show a reduced tendency to recrystallize compared with un-twinned regions, thus leading to a decrease of the intensity of the twin-induced texture component after annealing. On the opposite, our previous studies [26,27] showed that twins tend to grow during annealing of Zr alloy after moderate deformation. The applied strain in Isaenkova's study (~50%) [24,25]and our experiment (≤20%) [26,27] are different, which may affect the recrystallization mechanisms.

However, few studies explored the effect of loading path to

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subsequent recrystallization of Zr alloys. Athreya *et al.* [28,29] reported that the deformation mode, namely rolling or torsion, has significant influence on the deformation and recrystallization behavior of titanium. Different contributions of the deformation modes (prismatic slip, pyramidal slip or twinning) in rolling and torsion were supposed to be the underlying mechanism [28]. For Zr alloys with relatively strong texture, different deformation modes may be activated during loading along different directions, which may affect the subsequent recrystallization. In this work, to reveal the effect of loading direction on the deformation and recrystallization behavior of Zr alloy, a Zr alloy sheet with an initial relatively strong crystallographic texture was first compressed in two different directions, and then annealed at various temperatures. Microstructures and textures before and after annealing were characterized by electron backscatter diffraction (EBSD) method. Crystal plasticity modeling was carried out to evaluate the relative activity of deformation modes.

2. Experiments

A Zr alloy Zircaloy-4 (Zr-4), with a nominal chemical composition of Zr-1.4%Sn-0.2%Fe-0.1%Cr, was used as experimental material in this work. The as-received Zr-4 sheet (with a thickness of 2.2 mm) was industrially fabricated by rolling and annealing. The as-received Zr-4 sheet has a recrystallized microstructure, which is free from sub-boundary and twin lamella, as observable in Fig. 1(a). The initial average grain size is ~3.6 µm, measured by linear intercept method. The starting texture is a typical basal texture, in which the *c-axis* is mainly parallel to normal direction (ND) with an spread of ~20°. Another two texture components < 10–10 > //rolling direction (RD) and < 11–20 > //RD can also be characterized in the as-received Zr-4 sheet, as shown in Fig. 1(b).

For compressions, specimens with size of 2.2 mm (ND) × 4.0 mm (RD) × 5.0 mm (TD, transverse direction) were cut from the as-received sheet. The uniaxial compression tests were carried out at room temperature on an AG-X50kN machine. To reduce the effect of friction, Teflon was used between the specimen and tension machine. Compressions were along two different directions, ND and RD. Nominal strain of 5% and 10% were imposed in each compression direction. ND compressions were performed at a constant speed of 0.3 mm/min (initial strain rate of $2.3\times10^{-3}\ s^{-1}$), while RD compressions were conducted at a constant speed of 0.5 mm/min (initial strain rate of $2.1\times10^{-3}\ s^{-1}$). Each compression was repeated 4 times. During compression, the displacement and the loading force were recorded to evaluate true stress and true strain. Assuming that the compressive deformation is uniform at a small range of strain (≤10%), the true stress and true strain can be calculated with following two formulas:

$$\sigma = F/A = FL/A_0L_0 = F(L_0{-}\Delta L)/A_0L_0$$
$$\varepsilon = ln(L/L_0) = ln((L_0{-}\Delta L)/L_0)$$

where σ and ε are the true stress and true strain, F is the loading force, A and L are the instantaneous cross area and height of the specimen, A_0 and L_0 are the initial cross area and height of the specimen, ΔL is the displacement. We use the absolute value of the true stress and strain in following discussions.

For convenience, deformed specimens are named after the loading direction and reduction and denoted as ND-5%, ND-10%, RD-5% and RD-10%. The deformed specimens were then annealed for 2 h at 600 °C, 650 °C and 700 °C, respectively. A 2 h annealing time was used, based on our previous research experience, so that recrystallization can be achieved during this time period [30]. In the following, annealed specimens are named after the loading direction, reduction and annealing temperature. For example, ND-5% sample annealed at 650 °C is denoted as ND-5%-650.

Microstructure and texture evolution during compression and annealing were characterized by a field emission gun scanning electron microscope (TESCAN MIRA3) equipped with an electron backscatter diffraction (EBSD) detector and commercial available analysis system (Channel 5, *HKL* Technology-Oxford Instruments). EBSD data for each specimen were collected with a step size of 0.5 µm. The area for microstructure characterization was located at the center of plane RD-ND.

3. Experimental results

3.1. Deformed state

Orientation imaging maps of deformed specimens are presented in Fig. 2. The grains in Fig. 2 are colored according to a reference inverse pole figure (IPF) shown in the standard triangle. Grain boundaries, with misorientation angle higher than 10°, are represented with dark solid lines. While subgrain boundaries, or sub-boundaries, with misorientation angle higher than 2° and lower than 10°, are depicted by gray solid lines.

In ND-5% specimen, color gradients, which actually indicate orientation gradients, exist in many grains, as marked with white circles

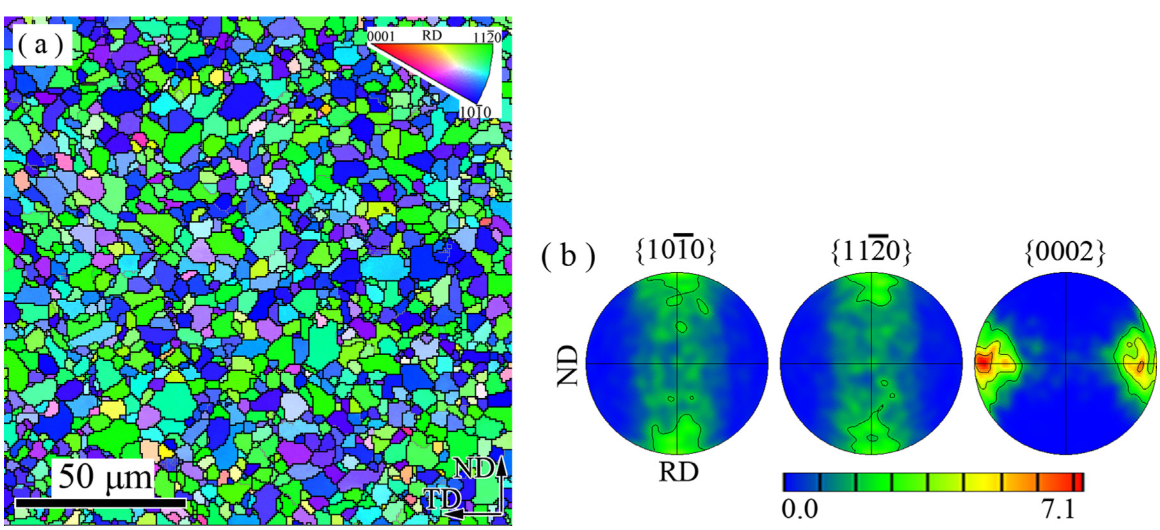

Fig. 1. Starting (a) microstructure and (b) texture of Zr-4 alloy.