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Cryo-rolling enhanced inhomogeneous deformation and recrystallization grain growth of a zirconium alloy

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ABSTRACT

This work focuses on the influence of cryo-rolling on subsequent recrystallization of hexagonal Zr-4 alloy. With initial recrystallized microstructure, the as-received Zr-4 sheets were rolled at liquid nitrogen temperature (LNT) and room temperature (RT). For each rolling temperature, two levels of strain, 20% and 40%, were applied. Then, samples, including as-received and rolled sheets, were annealed at 700 °C for 3 min–5 min. Microstructures of the deformed and annealed Zr-4 samples were characterized in detail by electron backscatter diffraction (EBSD) method. After rolling, no twin lamella is observed in Zr-4 sample. It is found that the intensity of the heterogeneous deformation in cryo-rolling is more severe than that in RT-rolling. After annealing, the recrystallized average grain size of Zr-4 decreases with the increases of the applied strain at both deformation temperatures. For a given rolling reduction, the recrystallized average grain size increases with decreasing deformation temperature. The texture orientation of Zr-4 was not remarkably changed during the rolling deformation and annealing process.

1. Introduction

With low probability of neutron capture and excellent corrosion resistant in water or vapor environment, zirconium alloys have been widely used in nuclear industry [1–4]. Zirconium alloys are often used as structural components in reactors, for example cladding tube, space grid or channel box. The mechanical property of zirconium alloy is thus one of the important factors. Many researches have been conducted on the microstructure and texture evolution of zirconium alloys during thermal or mechanical processing in order to improve mechanical properties [5–10]. As an important part of the fabrication route of zirconium alloy component, annealing may enable different extent of microstructural changes [11–13]. Many studies have been performed to investigate the annealing related microstructure or texture changes [14–16]. Zhu et al. [14,15] studied the primary recrystallization mechanisms and texture changes of Zr-2Hf alloy during annealing by in-situ high voltage electron microscopy, electron back-scattered diffraction and X-rays. Hiwarkar et al. [17] reported that the deformed microstructure of zirconium generally were consisted of fragmented and non-fragmented grains. The fragmented grains had higher stored energy and caused the early recrystallization through preferred nucleation during annealing. In the annealing of Zr-4 after moderate deformation, Kumar et al. [16] found that three distinct stages of annealing were observed. Stage I caused changes in the misorientations of the non-fragmented grains. In stage II, discontinuous recrystallization and grain coarsening consumed the fragmented regions. Stage III created recovery-induced grain refinement of the larger non-fragmented grains. Jedrychowski et al. [18] believed that strain induced boundary migration was the main recrystallization mechanism in the annealing of an alpha zirconium after moderate plastic deformation. However, compared with FCC or BCC metals, much work need to be done to further clarify the annealing related mechanisms of zirconium alloys.

To improve the mechanical properties, many new plastic deformation processes, for example accumulative roll bonding [19], high pressure torsion [20] and cryo-rolling [21,22], have been applied to zirconium alloys. The microstructures processed by these new plastic deformation techniques and their correlations with the mechanical strength were generally studied in detail [23,24]. However, rarely attention has been paid on the effect of

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these new plastic deformation techniques on the subsequent annealing behaviors of zirconium alloys. As a massive and continuous processing method, cryo-rolling has great potential application in zirconium alloys. In this work, a nuclear grade Zr-4 sheet was rolled to different reductions using cryo-rolling and room temperature rolling followed by annealing for different time. The deformed and annealed microstructures have been examined to investigate the effect of cryo-rolling on the subsequent recrystallization behaviors.

2. Experiments

Nuclear grade Zr-4 alloy, with nominal chemical composition Zr-1.4%Sn-0.2%Fe-0.1%Cr, was used as the experimental material in this work. With thickness of 2.2 mm, the as-received sheet provided by State Nuclear Baoti Zirconium Industry was fabricated through rolling and annealing. The as-received sheet has a recrystallized microstructure. The initial average grain size is 2.9 μm.

The as-received sheet was rolled at two different temperatures, namely liquid nitrogen temperature (LNT) and room temperature (RT). Rolling reduction of 20% and 40% were imposed. Therefore, four types of rolling were conducted. In the following, deformed specimens are named after the rolling temperature and reduction and denoted as LNT-20%, LNT-40%, RT-20% and RT-40%. The cryo-rolling in the current work was performed by immersing sheet specimens in liquid nitrogen for more than 30 min before rolling. The total reduction was achieved by a series of passes. In each rolling pass, 4% reduction was imposed. Before every rolling pass, the specimen was immersed again in liquid nitrogen for more than 15 min to allow sufficient cooling. The RT-rolling was also performed with 4% thickness reduction per pass.

The deformed specimens were then annealed at 700 °C for 3–5 min. In the following, annealed specimens are named after the rolling temperature, reduction and annealing time. For example, if a LNT-20% sample was annealed for 5 min, it is denoted as LNT-20%-A5. The annealing treatments were performed in a furnace filled of inert gas nitrogen. After annealing, the specimens were naturally cooled down in atmospheric environment. For the sake of comparison, the as-received sheet was also annealed.

Microstructure evolution during rolling deformation and annealing was characterized by a field emission gun scanning electron microscope (FEI Nova 400) equipped with an electron backscatter diffraction (EBSD) detector and analysis system (Channel 5, HKL Technology-Oxford Instruments). The EBSD technique was good at revealing microstructural characteristics related to local crystallographic orientations. In this work, EBSD data for each specimen were collected at a step size of 0.5 μm. The area for microstructure characterization was located at the center of plane rolling direction (TD)-normal direction (ND).

3. Results and discussions

3.1. As-received and deformed microstructure

Fig. 1 shows orientation imaging maps of the as-received and deformed specimens. The grains in Fig. 1 are colored according to a reference inverse pole figure (IPF) shown in the standard triangle. The orientation imaging maps are therefore denoted as IPF maps. Grain boundaries, with misorientation angle higher than 10°, are represented with dark solid line in the IPF maps. While subgrain boundaries, or subboundaries, with misorientation angle higher than 2° and lower than 10°, are depicted by gray solid line, as shown in Fig. 1(b). The black areas in the IPF maps represent the material points that are not indexed by EBSD measurement. The misorientation angle distribution (MAD) histograms corresponding to Fig. 1 are presented in Fig. 2.

Fig. 1(a) displays the initial microstructure of the as-received sample, which is composed of equiaxed grains and is free of subboundary, indicating recrystallized state. From the IPF map, it can be found that the majority of the grains are colored red, suggesting that their c-axes are close to the ND, which is very common in zirconium sheet produced by rolling process [1,25]. Fig. 2(a) indicates that the MAD of the as-received specimen shows considerable concentration at around 20–40°. In addition, compared with the high angle misorientation (>15°), the low angle misorientation (<15°) account for very low fraction. Indeed, the profile of the MAD of the as-received specimen is very common for rolled and recrystallized Zr sheet materials [26,27].

Fig. 1(b)–(e) show the microstructure of Zr-4 sheet after cryo-rolling and RT-rolling. It can be seen that most of the grains are still colored red, which means that the texture has not been remarkably changed during rolling. In contrast to the as-received microstructure, it is found that the grains are elongated after rolling deformation. Moreover, many subboundaries have appeared in the deformed grains, which are attributed to grain subdivision caused by dislocation slip and accumulation. Compared with RT-rolling, the cryo-rolling shows more severe non-uniform distribution of subboundaries. For example in LNT-20%, many grains have very few subboundaries, as indicated by N (non-deformed) in Fig. 2(b), while in some other grains a high density of subboundaries can be found, as shown in the marked circle area. In RT-20%, there are a lot of subboundaries in almost every grain although the densities may be different in different grains. The non-uniformly distributed subboundaries in the observed area indicate heterogeneous plastic deformation, which may be related to grain's initial orientation and have been reported by several studies [14,16]. However, compared with RT-rolling, the phenomenon that cryo-rolling can enhance the inhomogeneous plastic deformation has seldom been reported before and will be discussed in next subsection.

It is worth to note that no twin lamella was observed in the rolled Zr-4 samples in the current study, which is different from our previous work [26,28]. A lot of twins, including compressive and tensile twinning, were activated in the LNT rolling of zirconium or zirconium alloy [26,28]. The stress to activate twinning may be very dependant of the grain size [29]. The smaller the grain size, the higher the stress for activating twinning according to the results by Yu [29]. Compared with our former work (average grain size larger than 50 μm), the initial average grain size is only 2.9 μm in this study, which is too small to activate twinning (a very high stress would be needed).

Fig. 2(b)-(e) display the MAD histograms corresponding to Fig. 1(b)–(e). It is found that the low angle misorientations (<15°) account for the majority in deformed Zr-4 samples, which is caused by the dislocation slip and agrees well with the observed subboundaries in the IPF maps. Additionally, at a given rolling reduction, the fraction of low angle misorientations in cryo-rolled samples is a little higher than that after RT-rolling. For example, the fraction of low angle misorientations in LNT-20% and RT-20% is 59.2% and 54.1%, respectively. With increase of the rolling reduction, the fraction of low angle misorientations increases. Since low angle misorientations are mainly the result of dislocation activity, the higher fraction of low angle misorientations is supposed to indicate a higher density of dislocation. Indeed, cryo-rolling is often used as severe plastic deformation method due to the fact the recovery (mainly dynamic recovery) would be suppressed, thus leading to high density of dislocation [30]. Based on the fraction of low angle misorientation, it is extrapolated that the storage energy of the deformed samples are ordered as: LNT-40% > RT-40% > LNT-20% > RT-20%.