Materials and Design 85 (2015) 296–308

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/jmad

Microstructural and textural evolution of commercially pure Zr sheet rolled at room and liquid nitrogen temperatures

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A R T I C L E   I N F O

Article history:
Received 10 December 2014
Received in revised form 15 June 2015
Accepted 16 June 2015
Available online 25 June 2015

Keywords:
Zr
Microstructure
Texture
Deformation mode
Rolling

A B S T R A C T

A commercially pure Zr sheet with a typical bimodal basal texture was rolled up to 70% reductions at room temperature (RT) and liquid nitrogen temperature (LNT) to follow its microstructural and textural evolution. Various features developed during rolling were interpreted largely based on analyses on active deformation modes by the use of electron backscatter diffraction (EBSD) technique. Results show that only dislocation slip is activated during RT rolling while both slip and twinning occur in LNT-rolled specimens. There are always some non-deforming grains after RT rolling because their orientations with the c-axes close to the normal direction (ND) of the Zr sheet are unfavorable for slip. The LNT rolling allows these non-deforming grains to be deformed by $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ compressive twinning and thus leads to more homogenous microstructures. The angle between basal pole peaks and the ND slightly decreases during RT rolling, which is attributed to the activity of $\{10\bar{1}1\}\langle11\bar{2}3\rangle$ pyramidal slip. Many twins initiated at the early stage of LNT rolling could help retard the centralization of bimodal basal texture. In addition, calculations in light of key structural parameters measured by the EBSD reveal that the contribution from grain refinement induced by twinning makes the LNT-rolled specimens harder than the RT-rolled specimens.

1. Introduction

Zirconium and its alloys are widely used for structural applications in chemical and nuclear industries thanks to their good mechanical properties, excellent corrosion resistance and low neutron absorption [1]. Rolling is frequently employed in order to produce tubes or plates/sheets, both of which comprise actually the main types of final Zr products namely channels in boiling water reactors, grids and cladding tubes in light water reactors. For tube reduction processes with the thickness-to-diameter reduction ratio higher than one, which are prevalent in fabricating Zircaloys as fuel cladding materials [2], the stress–strain condition is similar to that in plate/sheet rolling processes. As a result, an identical texture, i.e. the typical bimodal basal texture, is generally developed in both cases.

Textures in Zr alloys are important because first, the texture developed at an intermediate step will influence the ease of subsequent processing, and second, the texture in products has significant effects on their in-service performance such as creep, stress corrosion cracking and hydride formation [1]. To reach a thorough understanding on the texture development of Zr alloys, thorough knowledge on their deformation mechanisms is required. In a large temperature range, the easiest system in Zr is found to be prismatic slip on $\{10\bar{1}0\}$ planes and along $\langle11\bar{2}0\rangle$ directions [2,3]. Other slip systems like basal slip ($\{0002\}\langle11\bar{2}0\rangle$) and pyramidal slip ($\{10\bar{1}1\}\langle11\bar{2}3\rangle$) could be active in regions with high stress concentration or at elevated temperatures [4,5]. Additional importance may be attached to the pyramidal slip thanks to its ability to accommodate strain along c-axes [6]. As an alternative to slip, twinning can readily occur in Zr alloys under specific circumstances, especially relating to the loading directions relative to the bulk texture. Operable twinning modes are either $\{10\bar{1}2\}\langle10\bar{1}\bar{1}\rangle$ and $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$ types if the c-axis is subjected to a tensile stress, or $\{10\bar{1}1\}\langle10\bar{1}2\rangle$ and $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ types when a compressive stress is applied along the c-axis [2,7].

In recent years, there have been extensive experimental efforts trying to further quantitatively exploit the effects of initial texture [8–10], temperature [9,11,12], grain size [12], strain rate [9,13] and interstitial impurities [10] on deformation modes of polycrystalline Zr. Experimental results in these studies provided a large amount of reliable basic data that were well incorporated into various crystal plasticity models [14–16] developed to accurately predict textural evolution and mechanical response of Zr materials. It is to be noted that cross-rolled high-purity Zr plates with a strong c-axis fiber texture were preferably selected in those experiments to reduce the complexity and thus facilitate theoretical analyses. Clearly, the c-axis fiber texture acquired in

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http://dx.doi.org/10.1016/j.matdes.2015.06.088

laboratory is different from the bimodal basal texture often encountered in the commercially produced products.

On the other hand, active deformation modes can also determine microstructural homogeneity of Zr materials subjected to various plastic processes. For example, a heterogeneous microstructure, comprising deforming and non-deforming grains, was recently reported for room temperature rolled Zr-2 sheets [17] and Zr-4 tubes [18] with the typical bimodal basal texture. Main reasons for the heterogeneous microstructure were attributed to distinctly different slip activities inside both deforming and non-deforming grains. More homogenous microstructures relying on grain refinement may be realized by introducing dense twinning lamellae [9,12], which would unfortunately not occur in such textured Zr materials rolled at room temperature [17–21].

In the present work, a comprehensive study on microstructural and textural evolution was presented for a commercially pure (CP) Zr sheet, which initially had the typical bimodal basal texture and was subsequently rolled at room temperature (RT) and liquid nitrogen temperature (LNT) to various reductions. Specific deformation modes activated in specimens rolled at both temperatures are described using electron backscatter diffraction (EBSD) technique, which is capable of acquiring detailed crystallographic information on grain orientations. Effects of active deformation modes, especially the $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ compressive twinning, on microstructural and textural features are discussed. In addition, based on key structural parameters obtained also by the EBSD, contributions to hardness of the CP-Zr material from different strengthening mechanisms are quantitatively addressed. This work is expected to provide more direct implications for potential applications of cryogenic rolling to processing of typically textured Zr plates/tubes.

2. Experimental

The as-received CP-Zr material (impurities in wt.% less than: 0.2 Sn, 0.07 Fe, 0.02 Cr, 0.005 Ni, 0.006 C, 0.003 N, 0.12 O) was fully recrystallized with a thickness of 3 mm. Different rolling reductions (up to 70%) were performed for the sheet by 5% per pass at two different temperatures, namely room temperature (RT) and liquid nitrogen temperature (LNT). The strain rates during these rolling processes were estimated to be between 3.9 and 7.1 s^{-1}. The LNT rolling in the present work was performed by immersing sheet specimens in liquid nitrogen for more than 15 min to allow sufficient cooling before every subsequent rolling pass. In the following, each specimen is named after the rolling temperature and reduction. For example, the specimen rolled at liquid nitrogen temperature to 30% reduction is hereinafter designated as LNT-30% specimen.

Microstructural evolution of the rolled specimens was followed by employing a field emission gun scanning electron microscope (FEI Nova 400) equipped with a back scattered electron (BSE) detector and an electron backscatter diffraction (EBSD) analysis system (Channel 5, HKL Technology-Oxford Instruments). The BSE detector was used to acquire electron channeling contrast (ECC) images for directly observing grain morphologies [22,23]. The EBSD technique, however, was expected to further reveal microstructural characteristics related to local crystallographic orientations. For example, EBSD is able to identify twinning types and quantitatively determine the proportion of each type. In this work, EBSD data for each specimen were collected at a step size of 0.5 μm with a scanning area of 200×200 μm^2 on the surface determined by rolling direction (RD) and normal direction (ND). With respect to the macroscopic texture of the specimens during rolling, X-ray diffraction (XRD) measurements were performed on their rolling surface using a Rigaku D/MAX-2500 X-ray diffractometer with CuK_α radiation. Preparation procedures of specimen surfaces for ECC, EBSD and XRD examinations were presented in detail in our previous work [24–26]. In addition, micro-hardness measurements were performed for all specimens using a Vickers indentation machine (EVERONE MH-5L) at a load of 490 mN. The micro-hardness of each specimen was determined as the average value of 10 measurements.

3. Results

3.1. Microstructure and texture of the as-received materials

Fig. 1a is an ECC image of the specimen cut from the as-received CP-Zr sheet. One can see that the initial microstructure of the experimental material is composed of equiaxed grains with relatively uniform size, indicating complete recrystallization. Also, the contrast for each grain is found to be clear and uniform, evidencing their crystal integrity. Using these ECC images, the recrystallized grain size is measured by the linear intercept method to be 9.6 μm on average. To reveal more features of the initial microstructure, an EBSD measurement is performed and the results are shown in Figs. 1b–d. Fig. 1b is an orientation imaging map according to a reference inverse pole figure (IPF) shown as the standard triangle (the inset in Fig. 2b). From the IPF map, it can be noticed that the majority of the initial grains are colored red, suggesting that many of their c-axes are close to the ND. The misorientation angle distribution (MAD) histogram corresponding to Fig. 1b is displayed in Fig. 1c. To facilitate discussion in the following text, grain boundaries with misorientations lower and higher than 15° are separated into low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs), respectively. The lower bound for LAGBs is selected to be 2° according to the angular resolution limit of the EBSD technique. Clearly, the fraction of HAGBs (81.3%) is overwhelmingly larger than that of LAGBs (18.7%) in the as-received CP-Zr specimen (see Fig. 1c). This can be well rationalized by the fact that sluggish LAGBs are annexed by HAGBs with high mobility during recrystallization. Compared with the MAD for the ideal random case (dashed line in Fig. 1c), the MAD of the as-received CP-Zr specimen shows a considerable concentration at around 20–40° along with a reduced fraction for misorientations larger than 40°. Indeed, such a MAD characteristic is very common in rolled and recrystallized Zr sheet materials and relates to their specific texture component [27,28]. Fig. 1d is a grain boundary (GB) map in which LAGBs, HAGBs and some special boundaries are separated by different colors. $64 \pm 2°/\langle 10\bar{1}0\rangle$, $85 \pm 2°/\langle 11\bar{2}0\rangle$ and $34 \pm 2°/\langle 10\bar{1}0\rangle$ correspond to boundaries of three types of most common twins in Zr, namely $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$s compressive twinning, $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twinning and $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$ tensile twinning, respectively. None of the twin boundaries are clearly evident in Fig. 1d indicating the absence of these twins in the as-received specimen. Fig. 1d is also able to clearly present the predominance of HAGBs over LAGBs, consistent with that revealed in Fig. 1c.

Texture measured by XRD for the as-received CP-Zr sheet is presented in Fig. 2. A bimodal basal texture can be seen in the {0002} pole figure with each maximum density tilted around 30° from the ND toward the transverse direction (TD). Another textural feature revealed by the $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ pole figures is that the $\langle 11\bar{2}0\rangle$ directions of most grains are parallel to the RD. These textural features suggest a typical texture which is widely reported in rolled and fully recrystallized Zr and Zr alloys [1,2].

3.2. Microstructural evolution during rolling

Microstructural evolution of the CP-Zr sheet during rolling at room temperature is shown in Fig. 3. The contrasts inside some grains are not uniform as noted in the ECC image of RT-5% which is different from the initial microstructure (Fig. 1a). This implies that the 5% rolling may have initiated the change of local orientations in these grain interiors. Since no twins are found, the change of local orientations should be attributed to grain subdivision caused by dislocation slip. As the rolling reduction increases, deformation occurs in more grains and when the reduction reaches 30%, it appears that grains begin to be