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ARTICLE

Study on Deformation Structure and Texture of Pure Zirconium with Large Grain Size Rolled at Liquid Nitrogen Temperature

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Abstract: The microstructure and the twinning behavior of the commercial pure zirconium with large grain sizes rolled uniformly at 77 K were studied. The deformation structures and the textures of samples with different rolling reductions were investigated by optical microscopy, scanning electron microscopy, electron backscattering diffraction (EBSD) and X-ray diffractometer. The EBSD results show that three types of twins, including $C_1\{11\bar{2}2\}<11\bar{2}\bar{3}>$ compressive twins, $T_1\{10\bar{1}2\}<10\bar{1}\bar{1}>$ and $T_2\{11\bar{2}1\}<11\bar{2}\bar{6}>$ tensile twins, occur during the rolling and the $C_1\{11\bar{2}2\}<11\bar{2}\bar{3}>$ compressive twins are the main twinning type. At the initial stage of the deformation, an amount of twins appear rapidly with only a few low-angle boundaries, indicating that twinning is favorable compared with dislocation slip. As the deformation reduction increases to 30%, however, a large number of low-angle boundaries are produced. It seems that the dislocation slip accommodated by twinning becomes the main deformation mechanism when the deformation reduction reaches approximately 30%. With the increase of the deformation no significant change on the texture type has been observed except slightly reduced intensity.

Key words: pure zirconium; liquid nitrogen temperature; rolling; texture; twinning

It is generally believed that zirconium, as a metal with hexagonal close-packed (hcp) crystal structure is deformed mainly by slipping and twinning[1]. Prismatic slip is generally considered to be the dominant deformation mode in Zr as it has the lowest value of critical resolved shear stresses (CRSSs)[2-7]. Pyramidal slip and the basal slip are always harder to be activated due to their higher CRSSs. It was reported[8] that with the decrease of the deformation temperature, such as to 77 K, the CRSS for slipping would increase dramatically and exceed that for twinning. As a result, twinning occurred readily at 77 K[9]. The three main twinning types of pure zirconium at liquid nitrogen temperature are $\{10\bar{1}2\}$, $\{10\bar{2}1\}$ and $\{10\bar{2}2\}$ while the type of $\{10\bar{1}1\}$ always turns up at higher temperature[9,10]. The key deformation modes include prismatic slip, two tensile twins, $T_1\{10\bar{1}2\}<10\bar{1}\bar{1}>$ and $T_2\{10\bar{2}1\}<10\bar{2}\bar{6}>$, and two compressive twins, $C_1\{10\bar{2}2\}<10\bar{2}\bar{3}>$ and $C_2\{10\bar{1}1\}<10\bar{1}\bar{2}>$, where compressive and tensile twins are named for their ability to accommodate strain during c-axis deformation[11-16]. Second generation compressive and tensile twins, which are within the first generation twins, can be observed in monocrystalline and polycrystalline zirconium sometimes.

The basal texture is commonly observed in hexagonal close packed metals during rolling. While the diverse basal pole textures caused by the different axial ratios show the slight difference. For zirconium sheets after rolling, the bimodal texture on the basal plane occurs that c-axis inclines to the transverse direction (TD). After studying the coarse grain size

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zirconium with random texture, which was deformed by cold rolling, Tenckhoff[6] confirmed that the compressive and tensile twinnings impel the basal pole texture in the early stage while the pyramidal slip plane with a (c+a) Burgers vector significantly contributes to the bimodal texture on the basal plane. At present, researches for commercially pure zirconium deformed at low temperature focus on the annealed pure zirconium with concentrated basal texture compressed in different orientations at liquid nitrogen temperature, discussing the twinning of pure zirconium, the mechanic performance testing, the change of the performance caused by twinning and the establishment of the twinning model. In the present paper, the deformation microstructure of the pure zirconium rolled at liquid nitrogen temperature was studied, whose initial grain size was large and initial texture was the bimodal texture. The authors aimed at exploring the deformation mechanics at low temperature and the deformation criterion influenced by twinning mechanics.

1 Experiment

The as-received zirconium plate with a thickness of 2.6 mm provided by Northwest Institute for Non-ferrous Metal Research was fully recrystallized and it consisted of equiaxed grains with an average grain size of 40 μm. The microstructure and texture of initial sheets shown in Fig.1 were tested by EBSD. The basal plate pole figure of initial texture showed that most grains were orientated along the normal direction (ND) with the spread of ±30° toward the transverse direction (TD).

The as-received pure zirconium plate was rolled on LG300 rolling mill. Samples with different reductions, 5%~40%, were produced by deformation-controlling rolling which were rolled by 5% reduction of initial sheet in each rolling pass. The samples were cooled in the liquid nitrogen for enough time to reach the deformation temperature before rolling. The microstructure was observed by optical microscope. Deformed samples were polished conventionally and etched by an acidic solution (10 mL HF, 45 mL HNO_3 and 45 mL H_2O) for optical metallographic examination after mechanical lapping. The samples were anodized with 5% H_2SO_4 before observing. The RD (rolling direction)-ND plates of samples were examined using FEI NOVA 400 field emission scanning electron microscopy with EBSD detector. EBSD data were collected at a 0.5 μm step size scan of 200 μm × 200 μm area. Prior to EBSD examination, samples were ground and electrochemically polished in a mixed solution (70 mL CH_3OH, 20 mL $C_6H_{14}O_2$ and 10 mL $HClO_4$). Channel 5 software was used to process the result, rebuild the microstructure and get information of twins. The RD-TD plates of samples were examined using D/Max-2500PC X-ray diffractometer in order to get the information of texture in macroscopic way.

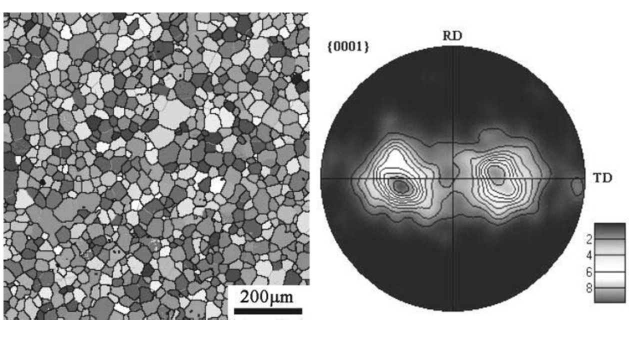

Fig.1 Structure and texture of the initial pure zirconium sheet

2 Results and Discussion

2.1 Metallographic experiment

Fig.2a shows an optical microstructure of the as-received pure Zr. The as-received sample was annealed, and the grains are equiaxed with the size about 40 μm. The microstructure characteristics of the specimen are twin-free and uniformed. The prominent difference on twins appears with the increase of the deformation reduction the quantity of the twins, the twinning appearance and the distribution of the twins. At the

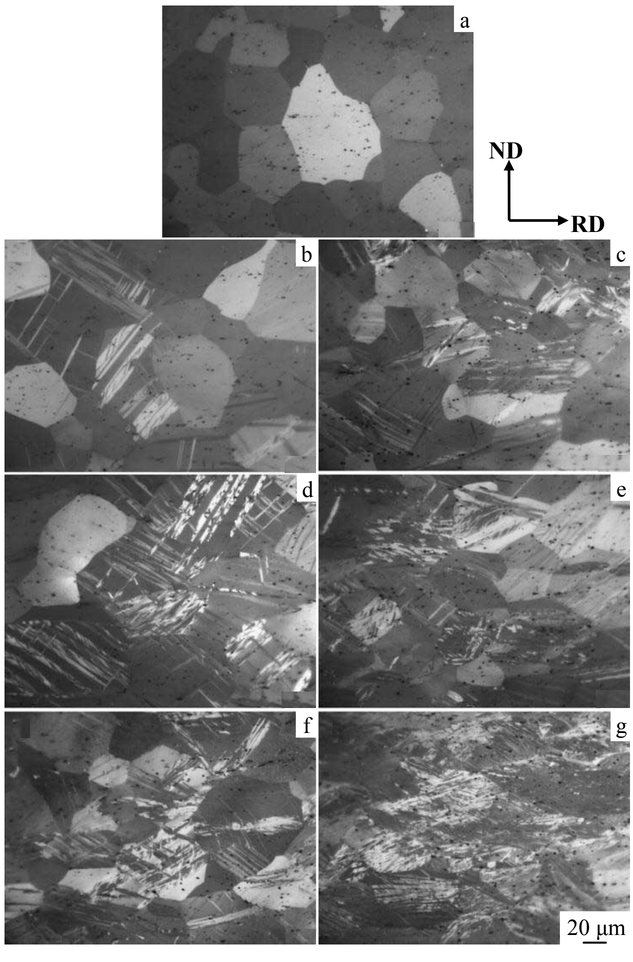

Fig.2 Optical microstructures of pure Zr at different deformation reductions: (a) as-received, (b) 5%, (c) 10%, (d) 15%, (e) 20%, (f) 30%, and (g) 40%