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Deformation twinning and textural evolution of pure zirconium during rolling at low temperature

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Abstract: Industry pure zirconium sheets with a strong *c*-axis fiber texture were rolled to different strains at 77 K to investigate the twinning behavior and deformation mechanism. The microstructure and texture of the rolled specimens were characterized by scanning electron microscopy (SEM) together with electron backscatter diffraction (EBSD) techniques. The results show that the $\{10\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ compression twinning mode is the dominant deformation twin at low strains loaded along the *c*-axis, and the $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ tensile twinning generates as the second twin in $\{10\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twins. The selection of twinning modes is governed by Schmid factor (SF) due to the calculating of SF and the EBSD simulating of twinning distribution. The evolution of texture during rolling affected by twins with increase of the strain was explained.

Key words: zirconium; twinning; texture; rolling

1 Introduction

It is well known that the deformation mechanism of HCP metals is complex. The prismatic slip $\{10\bar{1}0\}\langle 1\bar{2}10\rangle$ can be observed at all temperatures [1]. However, other slip systems such as the pyramidal slip and basal slip must be present to provide five independent slip systems resulting in a general, small, homogeneous strain without change in volume [2,3]. In addition, the lack of slip systems in zirconium may operate twinning to coordinate slip, especially at a low temperature (77 K) [4]. Four types of twins are often observed in Zr at low temperatures including $\{10\bar{1}2\}$ tensile twins (T1), $\{11\bar{2}1\}$ tensile twins (T2) and $\{11\bar{2}2\}$ compression twins (C1) together with $\{10\bar{1}1\}$ compression twins (C2) at high temperatures [5,6]. Tensile twins are activated when there is a tensile strain along *c*-axis, while compression twins are activated when there is a compression strain along *c*-axis.

It is known that the operative twinning should have a small shear magnitude and a lower critical resolved shear stress (CRSS). The CRSS magnitudes of $\{10\bar{1}2\}$, $\{11\bar{2}1\}$ and $\{11\bar{2}2\}$ twins are $0.17\sigma_s$, $0.22\sigma_s$ and $0.23\sigma_s$, respectively [7]. And, the CRSS for slip system is much lower than twinning system at room temperature but higher at low temperatures. However, as the CRSS for slip is sensitive to the temperature but insensitive for twinning, to investigate the twinning behavior at low temperature is much more significant [8].

In general, twinning is more active at low temperatures and will cause the evolution of texture which affects the property of zirconium. Nevertheless, some basic scientific problems are not clear such as the number, type and mechanism of twins, and the interaction between twin and slip. The EBSD data can get the information of grains and twinning boundaries, thus, the different types of twins could be identified by measuring the Schmid factor of twinning. In this study, the pure zirconium under various strains rolled at low temperatures was characterized. Meanwhile, the texture evolution caused by the twinning was also discussed.

2 Experimental

An industry pure Zr sheet with a strong {0002} basal plane texture was chosen as a starting material. The average size of grains was about 30 μm. Rolling specimens with dimensions of 60 mm in the rolling direction (RD), 20 mm in the transverse direction (TD)

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and 2 mm in the normal direction (ND) were cut from the as-received sheet with the *c*-axis parallel to ND.

Samples were rolled to different strains of 5%, 10% and 15% in a two-high mill with 170 mm diameter rolls at a rolling speed of 0.2 m/s. During each pass, the thickness was reduced by 0.1 mm. Optical microscope (OM, Axiovert 40 MAT), X-ray Diffraction (XRD, Rigaku D/MAX 2500PC) and scanning electron microscope (SEM, FEI Nova 400 FEG-SEM) together with electron backscatter diffraction (EBSD, Oxford HKL Channel-5) were used to obtain the deformation microstructure, grain orientation and twinning boundaries. EBSD data were collected from 350 μm×350 μm scans on the cross section of RD−ND surface of each sample at a step size of 1 μm.

3 Results and discussion

Figure 1 shows the orientation structure and grain boundary maps for each sample. The orientation images are followed as ⟨0001⟩//ND, while the boundary structure maps illustrate both grain boundaries and twin boundaries.

The microstructure for samples shows that C1 and T1 types of twinning both generated in the grains with *c*-axis//ND. And the grains whose orientation preferred to induce T2 twinning are represented by white circles though the T2 twinning only grew under high strains. In addition, the grain orientation changes from randomly to orientatedly, grain numbers increase, and three types of twins (C1, T1 and T2) appear with the increasing rolling reduction.

According to the distribution of misorientation angle, the quantitative analysis of the twins is performed. The different twin mode area fractions are shown in Table 1 and Fig. 2.

When the strain is under 5%, the low angle grain boundaries increase slowly, and the number of C1 is the largest in those grains with ⟨0001⟩//ND. And that kind of C1 takes up 70% of the total number of twinning boundaries, while 20% of T1 generates in C1 as the second twinning. This reveals that under low strain deformation there is little slip in material and the deformation is finished almost by C1 twinning. As the strain increases to 10%, the number of low angle grain boundaries grows faster. The number of C1 is still the largest. However, the number of T1 is two times before, and T2 appears in grains whose *c*-axis is far away from ND. Finally, when the strain is up to 15%, the slip increases, and the deformation mainly depends on the twinning coordinated with the slip. The number of C1 and T1 stabilizes, but starts to grow across the grains. Because C2 twinning almost exists in high temperature deformation or where the stress concentration is high [9], the C2 twinning is ignored during this experiment.

Thus, during the whole process of deformation, the order of twins generated is C1 followed by T1 which was generated in C1. With the rolling reduction increasing, the T2 appears in grains with the *c*-axis far away from ND. Finally, in the heterogeneous deformed grains, the C2 twinning generates to keep zirconium holding a good ductility characteristic during the process of macro homogeneous deformation at low temperatures [10,11].

The generation of twins or slips depends on the critical resolved shear stresses (CRSS) [12] and the orientation of materials which is indicated by Schmid factor (SF) [13,14]. However, the CRSS is to be simulated clearly [15] except using the visco−plastic self-consistent (VPSC) [16] or Taylor-type model [17]. Therefore, the SF is commonly used to predict the activated slip systems or twinning modes in metals.

Figure 3 shows the SF of different types of twinning. The initial orientation of grains is favorable for C1 twinning while T1 twinning is difficult to generate due to its low SF value. In 5% strain sample, the number of C1 compression twinning is the largest though the SF of this type of twinning decreases. As shown in Fig. 3(b), the SF of T1 twinning is changed by the formation of the C1 twinning, which leads the T1 easily to form in C1 twinning [10].

With the reduction up to 10%, the intensive stress makes the T2 twinning meet its CRSS and grains are refined by twins. At that moment, T2 increases and also the dislocation appears to relax the stress.

As the reduction is larger than 15%, the SF of each twin is low. In the further rolling, the deformation pattern is as the same as the room temperature depending on the various slips. It agrees well with the result of KASCHNER et al [6] who said that twinning only increased heavily in small deformation when compressing samples along *c*-axis direction by ODF quantitative analysis, and was saturated at 20% reduction. The low angle grain boundary distribution shown in Fig. 2 also implies that during the rolling process at low temperatures, the deformed grains obey slip→ twinning→slip rule constantly in order to keep the low angel grain boundary distribution presenting as low as 5% reduction and ascending in 15%. Due to this complex deformation mechanism the multiple slip systems emerge in large strain which allows zirconium exhibiting good ductility even rolled to large deformation at a low temperature (77 K) without fracture existence.

In short, when deforming the material into various strains at low temperatures, the twins generate following

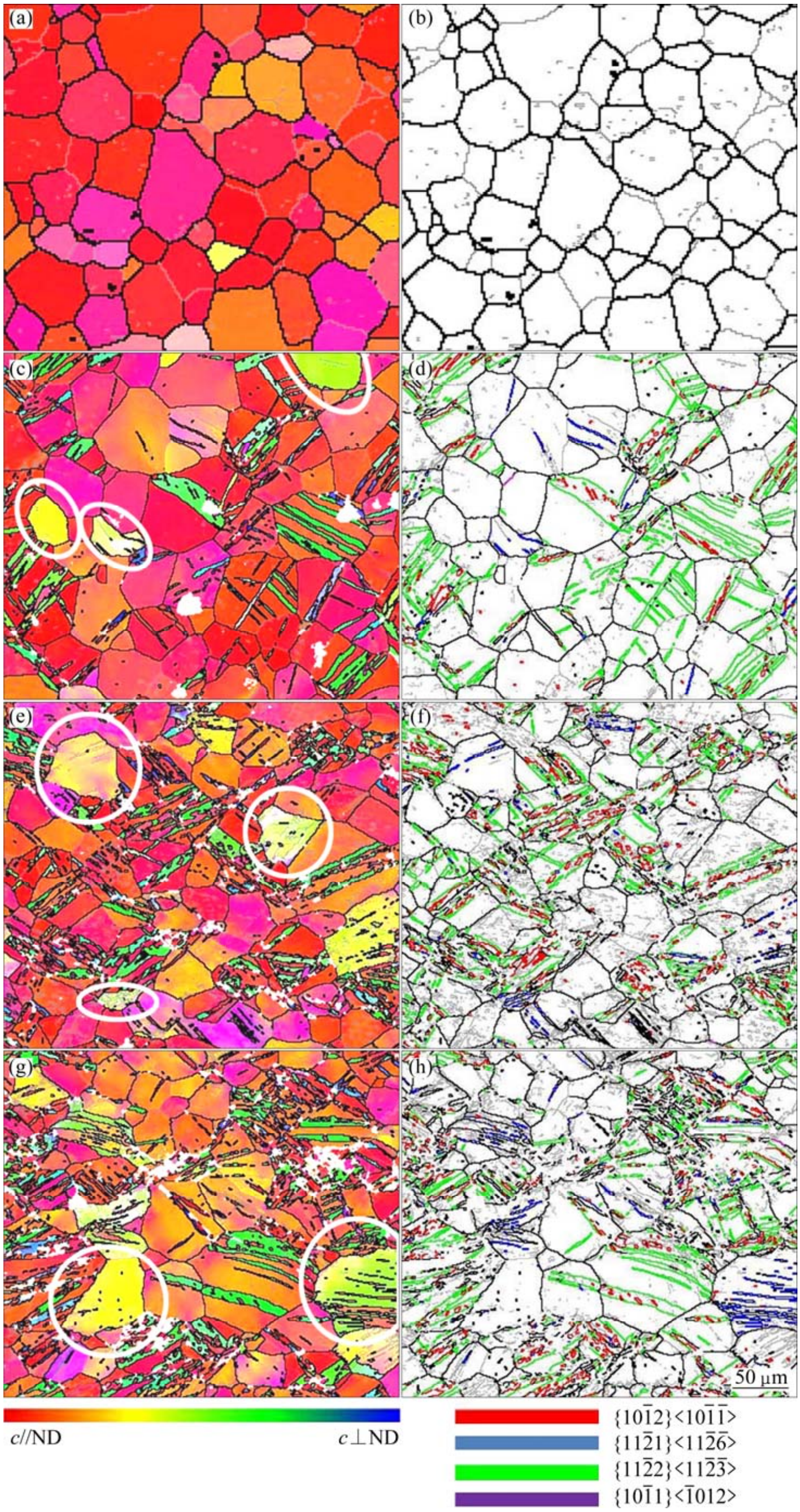

Fig. 1 Orientation structure and grain boundary maps under different rolling reductions rolled at 77 K: (a), (b) As-received; (c), (d) 5%; (e), (f) 10%; (g), (h) 15%

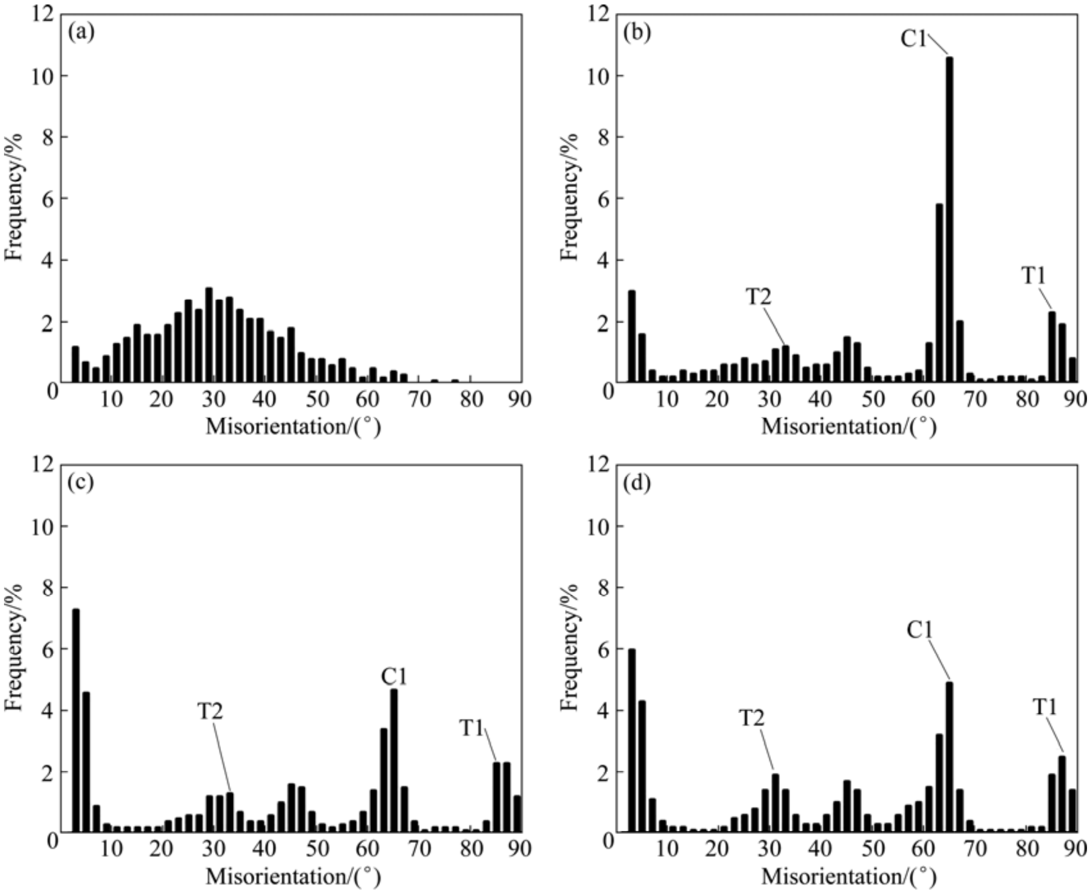

Fig. 2 Misorientation angle distributions of rolled samples under as-received (a), 5% (b), 10% (c) and 15% (d) rolling reduction

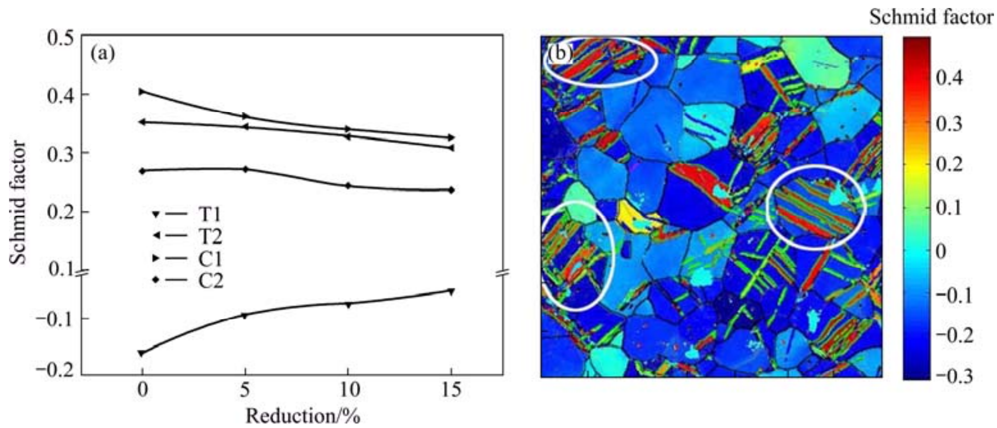

Fig. 3 Schmid factor (a) and SF distribution (b) of T1 at 5% reduction

Table 1 Area fraction of different types of twins

Type of twin	Misorientation angle/axis	Area fraction/%		
		5%	10%	15%
T1 $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$	85.22°$\langle 11\bar{2}0\rangle$	2.1	5.2	7.1
T2 $\{11\bar{2}1\}\langle 11\overline{26}\rangle$	34.84°$\langle 10\bar{1}0\rangle$	0.35	0.92	2.5
C1 $\{11\bar{2}2\}\langle 11\overline{23}\rangle$	64.22°$\langle 10\bar{1}0\rangle$	14.2	16.2	18.8

the steps of C1→T1→T2→C2. Moreover, at the same time there should be some prismatic slip and pyramidal slip in a large deformation, resulting in good ductility of pure zirconium.

Figure 4 shows the basal pole figure of different reduction samples and texture evolution sketch map. During the rolling deformation, the concentrated *c*-axis of basal texture orientates from ND towards TD and then

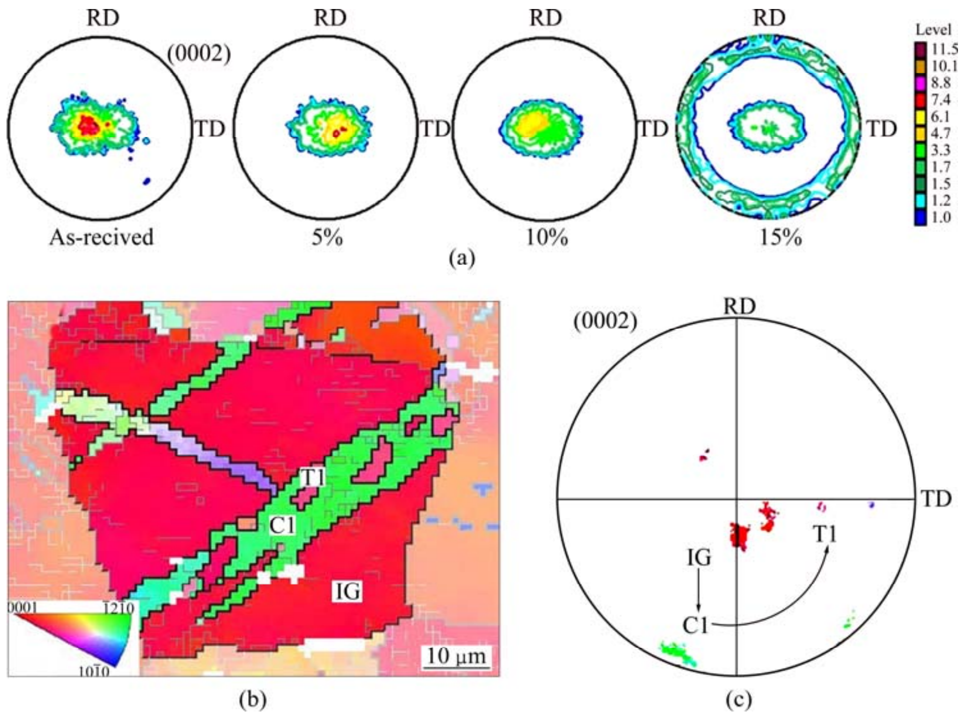

Fig. 4 Pole figures of as-received, 5%, 10% and 15% samples (a), texture evolution sketch map (b) and (c) (IG, C1, and T1 stand for initial grain, C1 twinning and T1 twinning, respectively)

results in bimodal texture together with that distributing far away from ND.

To investigate the effect of different types of twinning on the texture, certain single grains which have the second twinning are calculated. In Fig. 4(b), the initial grain is marked by IG, C1 type of twinning is marked by C1 and the second grain is marked by T1. After the grain twinning (twined by C1), the (0002) basal pole figure rotates from the IG spot (Fig. 4(b)) to the C1 spot, and at that moment the grain's orientation facilitates for the T1 twinning. Then due to the second twinning of T1, the grains rotate 85.22° relative to the C1 position. During the whole process, the frequency of C1 increases slowly but T1 increases fast. That is why in 15% reduction sample the *c*-axis changes from ND towards TD, forming bimodal texture.

4 Conclusions

1) Under rolling strain less than 5% at 77 K, the major deformation mode is C1 $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twinning. The twins adjust grain to slip through changing the SF of grains.

2) As the reduction grows up to 15%, the deformation mechanism is twinning coordinated by slip. With the increase of strain, the second twin T1 $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ is induced in the first twin C1 accompanying with T2 $\{11\bar{2}1\}\langle 11\bar{2}\bar{6}\rangle$, but C2 $\{10\bar{1}1\}\langle \bar{1}012\rangle$ is very difficult to generate consistently.

3) The basal texture changes from ND towards TD mainly due to the effect of C1 and T1 twinning modes.

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低温轧制纯锆的形变孪晶及织构演变

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摘 要：通过在 77 K 温度下进行不同变形量的低温轧制实验，研究了具有强烈单轴织构的工业纯锆板材在低温轧制变形条件下的孪生行为及变形机理。采用扫描电镜(SEM)和电子背散射衍衬(EBSD)分析和表征了变形材料的微观组织和织构。结果表明，在沿 c 轴加载的低应变条件下 $\{10\bar{2}2\}\langle 11\bar{2}3\rangle$ 压缩孪生是主要的变形机制，同时在 $\{10\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ 压缩孪晶中产生了二次孪晶($\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ 拉伸孪晶)以协调变形。施密特因子计算及孪晶分布的 EBSD 模拟结果表明，在低温变形条件下的孪生模式的选择是由施密特因子的数值大小决定的。探讨并解释了轧制过程中随着应变量增加由孪生所导致的织构演变。

关键词：锆；孪生；织构；轧制

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