$\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ double twinning in a Zircaloy-4 alloy during rolling at ambient temperature

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In this paper, we report the formation of an atypical double twinning sequence in Zircaloy-4 during rolling at room temperature. The grain orientation analysis based on electron backscattering diffraction (EBSD) data reveal that this double twinning mode is of the $\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ type, namely, the secondary $\{10\bar{1}2\}$ twin generated within the primary $\{11\bar{2}1\}$ twin. Based on the combinative analysis of Schmid factor and local strain accommodation, the formation and variant selection mechanism of this double twinning are discussed.

Keywords: Zircaloy-4; Double twinning; Electron backscattering diffraction (EBSD); Schmid factor; Local strain accommodation

Zirconium alloys have long been used in nuclear reactors because of their good corrosion resistance and mechanical properties, combined with relatively low absorption cross-section of thermal neutrons [1]. Due to the limited slip systems in typical hexagonal close packed (hcp) structures, Zr and its alloys deform mainly by dislocation slip and/or twinning to satisfy the necessity of having five independent slip (twin) systems to accommodate polycrystal plasticity [2]. In Zr, the most commonly reported primary twin modes are $\{10\bar{1}2\} \langle \bar{1}011 \rangle$, $\{11\bar{2}1\} \langle \bar{1}\bar{1}26 \rangle$ and $\{11\bar{2}2\} \langle \bar{1}\bar{1}23 \rangle$ [3]. The former two are tensile twins (TTWs) because they are generated when the c-axis is elongated, whereas the latter is a compressive twin (CTW) formed by contraction of the c-axis [4-6]. These twins abruptly reorient the crystal lattice by 85.22° about the $\langle 11\bar{2}0 \rangle$ axis, 34.84° about the $\langle 10\bar{1}0 \rangle$ axis and 64.22° about the $\langle 10\bar{1}0 \rangle$ axis, respectively, and hence have a significant effect on the texture evolution [7] and the work hardening rate [4].

Under suitable conditions, secondary twins can be generated within the primary to form double twinning structures. For example, McCabe et al. [8] investigated the effect of loading direction on the domination twin modes in a cold-rolled high-purity Zr alloy at 77 K and found that the secondary $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ twinning increased with the third power of the primary $\{11\bar{2}2\}$ twin fraction for compression parallel to the primary c-axis texture, whereas the $\{10\bar{1}2\}$ - $\{11\bar{2}2\}$ double twins are generated only when the strain exceeds 17% in compression normal to the primary c-axis texture. Similar double twinning modes were also reported by Knezevic et al.

[7], who systematically investigated the temperature and rate dependence of slip, twinning and secondary twinning in high-purity α -Zr. They proposed that the $\{10\bar{1}2\} < \bar{1}011 >$ twin was the preferred tensile twin, either as a primary or secondary twin, depending on the sample orientation over a broad temperature and strain rate range. However, for other hcp crystals, such as Mg alloys, the most commonly observed double twinning is of the $\{10\bar{1}1\} - \{10\bar{1}2\}$ type [9, 10]. These modes can be simply denoted as CTW-TTW or TTW-CTW double twinning, depending on the twinning sequence. The formation of these double twins can be interpreted via the reorientation of the crystal within the primary twins; thus, deformation modes that are not active prior to twinning may be activated [11]. On the other hand, this reorientation might energetically hinder the formation of the secondary tensile (or compressive) twins inside the primary tensile (or compressive) twins, resulting in the absence of TTW-TTW or CTW-CTW double twinning.

However, a TTW-TTW double twinning has been reported in Mg alloys [11-13], which is consistent with the $\{10\bar{1}2\} - \{10\bar{1}2\}$ twinning sequence and is thus termed as double tensile twinning. The possible mechanisms of double tensile twinning have been well discussed under either single path loading [12, 13] or multiple path loading [11]. The observation of additional double twinning modes, including this $\{10\bar{1}2\} - \{10\bar{1}2\}$ double twinning, is not yet reported in zirconium and its alloys. In this paper, a type of $\{11\bar{2}1\} - \{10\bar{1}2\}$ double twinning is revealed and reported as a new twinning type during room temperature rolling in β -cooled Zircaloy-4. The formation of this sequence of double twinning is discussed based on local strain

accommodation effect and Schmid factor, which is expected to provide new insight into the twinning modes in Zr alloys.

The selected starting material is a hot-rolled and annealed Zircaloy-4 (Zr-1.5Sn-0.2Fe-0.1Cr in wt. %) plate of 4.5 mm thickness. Rectangular specimens with dimensions of 50, 16 and 3 mm along the RD, TD and ND were cut from the initial plate, where RD, TD and ND refer to the rolling, transverse and normal directions, respectively. Before the rolling process, the specimens were sealed in quartz tubes under vacuum and then β -solution treated at 1303 K for 2,400 s, followed by furnace cooling at an estimated cooling rate of 0.05 K/s [5]. Subsequently, the β -cooled specimens were rolled to different reductions using a rolling mill with a roller of 450 mm in diameter in one pass. Several small specimens were cut into 7×6 mm² (RD×TD) pieces from the center of the rolled plate.

The microstructures of the β -cooled and rolled specimens were characterized by electron channeling contrast (ECC) images and a fully automated electron back-scatter diffraction (EBSD) analysis system (Channel 5, HKL Technology-Oxford Instruments) in an FEI Nova 400 field emission gun scanning electron microscope (FEG-SEM). Before SEM-ECC and SEM-EBSD investigations, the specimens were mechanically ground using SiC paper and were then electro-polished in a mixed solution of 70% methanol, 20% butyl cellosolve and 10% perchloric acid at 243 K. To identify the twins in the rolled specimens, a mapping step of 0.5 μ m was chosen for the EBSD scanning under an accelerated voltage of 20 kV.

Fig. 1 presents the ECC images after β -cooling and rolling to a reduction of 10%.

As seen from Fig. 1a, the structure of the β -cooled specimen consists of numerous coarse plates typical of the lenticular structure in Zr when cooled from the β region at a lower rate [5]. Moreover, mutual parallel plates in Fig. 1a are generally in a similar orientation and constitute irregular grains of hundreds of micrometers. Some twin-like lamellae are found in Fig. 1a (indicated by black arrows), which have been reported to be tensile twins induced by intergranular thermal residual (ITR) stresses during cooling in our previous work [5]. Abundant twins are observed after rolling to 10% reduction (Fig. 1b), similar to earlier observations in commercial Zr alloys [3, 14-18].

To identify the twin modes, EBSD measurements are conducted on the as-rolled specimens. Due to the relatively large grain size and smaller mapping step size of 0.5 μm , each scanning area does not cover multiple grains in a limited mapping time. A typical scanning area inside one coarse grain is given in Fig. 2a, which exhibits multiple twins with lengths of tens to hundreds of microns. Fig. 2c shows the histogram of the misorientation angle distribution corresponding to Fig. 2a, where four peaks at approximately 5° , 35° , 60° and 85° are noted. The misorientation axes corresponding to the peaks at 35° and 85° are revealed in the insets of Fig. 2c to be $\langle 10\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$, respectively. These two angle/axis relationships correspond to the two tensile twinning modes in Zr, namely, the $\{11\bar{2}1\}$ (termed T_2) tensile twin (with boundary of $\sim 34.84^\circ / \langle 10\bar{1}0 \rangle$) and the $\{10\bar{1}2\}$ (termed T_1) tensile twin (with boundary of $\sim 85.22^\circ / \langle 11\bar{2}0 \rangle$) [7]. The misorientation axis related to the peak at 60° , which is also given in Fig. 2c, shows high Miller indices and does not belong to any established twinning modes in Zr. When different twinning variants interact with each

other, boundaries with special angle/axis relationships are produced. Such misorientations induced by twinning interactions have been comprehensively investigated in Ti, whose c/a ratio is very close to Zr [19]. In Fig. 2a, most of the twins are identified as T_1 with only two variants of $(0\bar{1}12)[01\bar{1}1]$ and $(01\bar{1}2)[0\bar{1}11]$. These two variants share the same $\langle 11\bar{2}0 \rangle$ zone axis and produce boundaries with misorientation of $9.6^\circ/\langle 11\bar{2}0 \rangle$ once they interact. Therefore, the peak at 60° is believed to be induced by the interaction between the different T_1 and T_2 variants. The peak at approximately 5° corresponds to low-angle boundaries, which is produced by both dislocation accumulation and the co-zone T_1 variants interaction. Fig. 2b and d present the magnified maps circled by black lines in Fig. 2a, which are superimposed by hcp unit cells that reveal the orientation of the matrix and the tagged twins. To highlight the twin modes, the boundaries of T_1 and T_2 are colored in white and blue, respectively, in both Fig. 2b and d. The twins marked A, B and C are of the T_1 mode and their boundaries are in white, whereas the PT_2 in Fig. 2b and PT_2' in Fig. 2d represent the primary T_2 mode and they are plotted as blue boundaries. Another twin, termed ST_1 , is activated inside PT_2 . Clearer evidence is presented in Fig. 2d, where two mutual parallel twins are unambiguously seen inside the PT_2' . Both twins are highlighted as ST_1' because they are the same variant (same orientation). The length of the larger ST_1' shown in Fig. 2d is about $3\ \mu\text{m}$, which is six times of the mapping step, suggesting that the observation of ST_1' should not be an artifact of the EBSD scanning. As mentioned before, double twinning is a process of secondary twinning that occurs inside primary twins [13], and the observed secondary twins are

believed to be double twinning of the $\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ mode. To the authors' knowledge, this double twinning mode has never been reported in Zr alloys.

A detailed analysis of the twinning relationship in Fig. 2d based on the pole figures is performed in Fig. 3, in which the orientations of the matrix and all tagged twins are projected as different symbols. The $\{0001\}$ pole figure in Fig. 3a shows that the c-axis of the matrix is nearly perpendicular to ND, lying in the TD-RD region, as is typical in Zircalloys following cross-rolling. The c-axes of the twins, however, spread far from the matrix. From the $\langle 10\bar{1}2 \rangle$ and $\langle 10\bar{1}1 \rangle$ pole figures in Fig. 3b and 3c, one can see that the C twin is projected at the same site with the matrix in their habit planes and directions (the blue dotted circled region), whereas it is not for the ST_1' twins. The projected site of the ST_1' twins coincides with the PT_2' in their habit planes and directions, as shown by red dotted circle. Thus, it is confirmed that the ST_1' twin is a secondary twin activated within the primary PT_2' twin, i.e., double twinning.

From other EBSD patterns, additional 4 cases with similar structures are extracted and verified to be $\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ double twins, indicating their certain occurrence. Moreover, a common fact is that all the observed secondary twins are jointed with the primary $\{10\bar{1}2\}$ twins at grain boundaries (GBs) in the six cases, as seen in Fig. 2b and d where both secondary twins (i.e., the ST_1 and ST_1' twins) are interacted with the primary twins (A, B and C). It is thus believed that the activation of the secondary twins cannot be completely independent of the connecting primary twins. To understand this correlations, a geometrical factor (m') is employed here and

expressed as $m' = \cos\psi \cos\kappa$, where ψ and κ are the angles between the twin plane normal/directions of the two twinning systems. The geometrical factor (m') has been applied to explain the variant selection of the paired twins in Mg alloy [20-22], where a relatively high m' value indicates a better accommodation effect. Therefore, for the secondary twin connected with two primary twins, a higher m' value is chosen. For instance, it is seen in Fig. 2b that the secondary ST_1 connects with two primary twins (A and B) on both sides. The m' values between A, B and ST_1 are calculated to be 0.012 and 0.783, respectively. Accordingly, the strain accommodation effect between B and ST_1 is much better than that between A and ST_1 , and the value of 0.783 is presented. The m' values between the jointed twinning structures in the six cases are displayed in Table 1, where Case 1 and Case 2 represent the cases in Fig. 2b and d, respectively. It is clear that all the m' values in the six cases are very high, indicating good strain accommodation effects. It was reported that accommodation can occur when the misorientation of the GBs is within 20-40° [23]. In the present work, the GBs are of the T_2 twin boundaries (TBs) with a misorientation angle of 34.84° with respect to the matrix. This angle is within the range and may provide a good accommodation across the GBs. The above analysis indicates that the shear strains due to the connecting primary twins at T_2 TBs can be accommodated consistently by forming twins inside T_2 twins, generating the observed double twinning.

To further investigate the generation of the secondary twins, a modified Schmid factor for rolling is expressed here as $SF_{\text{rolling}} = 0.5 \times (\cos\phi_{RD} \times \cos\lambda_{RD} - \cos\phi_{ND} \times$

$\cos\lambda_{ND}$), where $\varphi_{(RD, ND)}$ are the angles between the twinning plane normal and the rolling or normal direction, and $\lambda_{(RD, ND)}$ are the angles between twinning shear direction and the rolling or normal direction. More detailed descriptions about the equation can be found elsewhere [22, 24]. The calculated SF_{rolling} values of the six potential secondary T_1 twins in each case are presented in Table 1, where the SF_{rolling} ranks of the active twins are also listed. It is seen that only in two cases (Case 1 and Case 5) the active secondary T_1 twin selects the variant with the highest SF_{rolling} value, whereas in other four cases the variant with the second highest SF_{rolling} value is selected. However, it should be addressed that in most cases (except Case 5) the difference between the first and second highest SF_{rolling} values is not noticeable, which is too small to actually distinguish between the two variants. In fact non Schmid behavior has been reported many times in literature [9, 25, 26] and has led to the development of additional methods such as accommodation strain analysis [25, 27]. In view of this, geometric compatibility factor is further analyzed to better understand the variant selection of the secondary T_1 twins. Here, we take Case 1 and Case 2 as the examples. Table 2 lists the SF_{rolling} and m' values (related to the connecting primary twins) of the six potential secondary T_1 variants in Case 1 and Case 2. The results indicate that there are two variants (V_5 and V_6) which have relatively high SF_{rolling} values in both cases. For Case 1, the active secondary twin selects the variant (V_6) with the highest SF_{rolling} . Note that the m' value of this variant is also the highest one. For Case 2, however, the selected variant (V_5) is the one with second highest SF_{rolling} and the variant (V_6) with the highest SF_{rolling} is not selected. To note,

the m' values of these two variants are quite different, being 0.783 and -0.002 for V_5 and V_6 , respectively. The active secondary twin in Case 2 is indeed the one (V_5) with the highest m' value. The same phenomenon is also observed in other four cases, where the active secondary twins always select the variants with the highest m' values. The above analyses indicate that the local strain accommodation effect plays a crucial role in the variant selection of the secondary T_1 twins.

Based on the above analysis, the formation of the secondary T_1 twins are believed to accommodate the strain incompatibility caused by the other twins, but this process can also be accomplished by active slipping. In the present work, profuse dislocation slip behavior is observed in the matrix and even in the twins (grey lines in Fig. 2b and d), which is consistent with the observed peak of the misorientation angle at approximately 5° in Fig. 2c. Jain et al. [12] investigated the compression of AZ80 magnesium alloy at 77 K and found the occurrence of $\{10\bar{1}2\}$ - $\{10\bar{1}2\}$ double twinning to be due to the difficulty of non-basal slip at such low test temperatures. Conversely, one possible reason for the rare $\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ double twins in this work is the active slip behavior during room temperature rolling. One can also deduce that this double twinning may largely contribute to the plastic deformation of coarse-grained zirconium when tested at low temperature because of the unenergetic slip behavior and more twinning interactions.

In summary, an atypical double twinning sequence consistent with $\{11\bar{2}1\}$ - $\{10\bar{1}2\}$ has been observed in a β -cooled Zr-4 alloy rolled at room temperature. Local strain accommodation and Schmid factor are combined to

understand the formation and variant selection of this double twinning. The results show that the local strain accommodation effect plays a crucial role in the variant selection of the secondary T_1 twins, although their SF_{rolling} values are relatively high. The local strain incompatibility associated with the interacting primary twins contributes to the activation of the secondary $\{10\bar{1}2\}$ twin inside the primary $\{11\bar{2}1\}$ twin. Atypical twinning may also originate in zirconium at suitable conditions. However, more evidence for this double twinning mode is needed, along with detailed underlying mechanisms in zirconium and its alloys.

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Figure Captions

Figure 1. ECC maps showing the structure of (a) β -cooled specimen and (b) following 10% reduction. Black arrows in (a) indicate twin-like lamellae.

Figure 2. (a) Inverse pole figure (IPF) map in the RD-ND plane following 10% reduction. Grain boundaries in (a) are colored in black ($>15^\circ$) and grey ($>2^\circ$) lines. Two black circled areas in (a) extracted for a detailed analysis in magnified IPF maps are presented in (b) and (d). The boundaries in (b) and (d) are represented as follows: white lines (misorientation of $85^\circ \pm 5^\circ / \langle 11\bar{2}0 \rangle$) indicate the boundaries of the primary $\{10\bar{1}2\}$ twins (A, B in (b) and C in (d)) and secondary $\{10\bar{1}2\}$ twins (ST_1 in (b) and ST_1' in (d)), blue lines (misorientation of $34^\circ \pm 5^\circ / \langle 10\bar{1}0 \rangle$) indicate the boundaries of the primary $\{11\bar{2}1\}$ twins (PT_2 in (b) and PT_2' in (d)) and the others are the same as those in (a). The hcp unit cells are also shown. (c) Misorientation angle distribution histogram and rotation axes corresponding to (a).

Figure 3. Orientation relationships of the tagged twins and the matrix in Figure 2d measured by EBSD: (a) $\{0001\}$ pole figure; (b) $\{10\bar{1}2\}$ pole figure; (c) $\{10\bar{1}1\}$ pole figure. The symbols in (a)-(c) indicate the experimentally projected sites of both the twins and the matrix by EBSD. Blue and red dotted circles represent the coincidence sites of the symbols.

Table Caption

Table 1. The m' values of the connected two twins and the SF_{rolling} values of the six potential variants of the secondary T_1 twins in the six cases. V1-V6 represent the twinning systems consistent with reference [22].

Table 2. The SF_{rolling} and m' values of the six potential variants of the secondary T_1 twins in Case 1 and Case 2.

Figure(s)

Figure(s)

Figure(s)

Table 1

	m'	SF _{rolling}						SF _{rolling} rank
		V1	V2	V3	V4	V5	V6	
Case 1	0.795	0.076	0.062	-0.231	-0.240	0.172	0.176	1
Case 2	0.783	0.100	0.083	-0.225	-0.240	0.160	0.163	2
Case 3	0.81	-0.032	-0.024	-0.199	-0.194	0.231	0.228	2
Case 4	0.804	-0.016	-0.007	-0.206	-0.197	0.226	0.225	2
Case 5	0.715	0.109	0.115	-0.049	0.003	0.242	0.289	1
Case 6	0.801	0.099	0.084	-0.242	-0.250	0.142	0.149	2

Table 2

		V1	V2	V3	V4	V5	V6
Case 1	SF _{rolling}	0.076	0.062	-0.231	-0.240	0.172	0.176
	m'	0.359	-0.058	-0.043	0.324	0.011	0.795
Case 2	SF _{rolling}	0.100	0.083	-0.225	-0.240	0.160	0.163
	m'	-0.008	0.278	0.377	-0.122	0.783	-0.002

(a) Inverse pole figure (IPF) map in the RD-ND plane of following 10% reduction.

Grain boundaries in (a) are colored in black ($>15^\circ$) and grey ($>2^\circ$) lines. Two black circled areas in (a) extracted for a detailed analysis in magnified IPF maps are presented in (b) and (d).

Boundaries in (b) and (d) are represented as follows: white lines indicate boundaries with misorientation of $85^\circ \pm 5^\circ <11\bar{2}0>$, blue lines indicate boundaries with misorientation of $34^\circ \pm 5^\circ <10\bar{1}0>$ and others are the same as those in (a). The hcp unit cells are also shown. (c) misorientation angle distribution histogram and rotation axes corresponding to (a).

The angle/axis relationships related

to the peaks at 35° and 85° correspond to the two tensile twinning modes in Zr,

namely, the $\{11\bar{2}1\}$ tensile twin ($\sim 34.84^\circ <10\bar{1}0>$) and the $\{10\bar{1}2\}$ tensile twin

($\sim 85.22^\circ <11\bar{2}0>$). The misorientation axis related to the peak at 60° has high Miller

indices and does not belong to any established twinning modes in Zr and is produced

by twinning interaction.