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Stacking faults observed in $\{10\bar{1}2\}$ extension twins in a compressed high Sn content Zr alloy

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ABSTRACT

'Anomalous' stacking faults within $\{10\bar{1}2\}$ extension twins were extensively observed in a high Sn content Zr alloy 'Excel' after uniaxial compression testing. TEM characterization on these stacking faults shows that there are three types of stacking faults: basal, prismatic, and pyramidal stacking faults. Basal stacking faults usually cross a twin and have either both ends or at least one end connected to a twin boundary. A high-density of pyramidal or prismatic stacking faults are observed close to a twin boundary, and some discrete occurrences of these types of stacking faults are observed detached from the twin boundary.

In hcp metals, the relative energies of the stacking faults (SFs) on the prismatic and basal planes are thought to determine the selection of atomic planes on which $1/3\langle 11\bar{2}0\rangle$ dislocations prefer to glide [1–3]. For instance, in pure Zr and Ti where the prismatic plane stacking fault energy (SFE) is lower than the basal plane SFE, the primary slip mode observed is prismatic; however, in Mg and Be where the SFE on the basal plane is much lower, basal slip is much easier [4]. In addition, recent molecular dynamics (MD) modeling work has shown that SFs also play an important role in the propagation of $1/3\langle 11\bar{2}0\rangle$ screw dislocations gliding on the prismatic and pyramidal planes in hcp metals (Zr, Ti) [5–8].

SFs have not been frequently observed in the un-twinned matrix in deformed hcp metals, especially in the case of Ti and Zr due to the high SFE [9]. However, deformation induced SFs have been reported in the un-twinned matrix of pure Mg and Mg alloys, especially in alloys where the alloying elements can substantially reduce the energies of SFs [10–13]. The existence of those SFs in the deformed microstructures in Mg-Gd-Y and Mg-Y alloys have been suggested to be responsible for the improved mechanical properties of such Mg alloys [11,13]. In addition to those SFs in the un-twinned matrix, 'non-equilibrium' or 'anomalous' basal SFs have been extensively and exclusively observed within $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ extension twins in a variety of hcp metals including Zr [9], Ti [9,14], Mg [10,12,14,15], and Co [14]. $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ is a common twinning mode widely occurring in hcp metals when the $\langle c\rangle$ axis tends to extend during plastic deformation [16]. It has conventionally been thought that $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twinning causes a twinning shear calculated as $(3-\gamma^2)/(\sqrt{3}\gamma)$, where γ is the c/a ratio [17]. However, benefiting from high-resolution transmission electron microscopy (HR-TEM) and high-resolution scanning transmission electron microscopy (HR-STEM), more and more anomalous phenomena have been observed related to this type of twinning, including observation of no well-defined misorientation angle [15,18] and the absence of shear strain [19]. Li and Zhang [20] have briefly reviewed these phenomena and linked them to the proposed argument that $\{10\bar{1}2\}$ twinning, at least in Mg, actually can only cause zero shear strain and hence the growth of $\{10\bar{1}2\}$ twinning is solely accomplished by atomic shuffling, with no twinning dislocations involved [21]. MD modeling was carried out to understand the presence of SFs within $\{10\bar{1}2\}$ extension twins [14]. It was found that the formation of basal SFs arises due to the migration of the incoherent twin boundary (TB). When the structure of the initial TB is incoherent, basal SFs are nucleated with one end of the SF being locked at the moving TB. The basal SFs subsequently grow along with the moving TB, resulting in SFs with a large width that may cross the entire twin [14]. The simulation also showed that pyramidal SF can also form within the twin, and would intersect with the basal SF [14]. However, pyramidal SFs within twins have not been widely observed experimentally in the TEM. In this paper, we report the observation and determination of three different types of SFs (basal, prismatic, and pyramidal) in $\{10\bar{1}2\}$ twins produced in a uniaxially compressed high Sn content dual phase Zr alloy.

The material used in this study is a Zr-Excel alloy plate with 14 mm thickness manufactured by warm rolling. The nominal composition of the alloy is 3.5 wt% Sn, 0.8 wt%Nb, 0.8 wt% Mo, 1100 wppm O, 1000 wppm Fe and ~20 wppm H. This alloy is a dual phase (~90% hcp α + ~10% bcc β) material, however the majority hcp α phase is the

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focus of this paper; more details of this alloy can be found in our earlier paper [22]. Cylindrical samples cut from the plate with the axial direction parallel to the original rolling direction (RD) were subsequently compressed to ~8% strain. The texture evolution, as measured by *in situ* time-of-flight neutron diffraction, during the compression test is illustrated by the inverse pole figures in the compressive axis direction, as shown in Fig. 1. Before compression, the samples have a texture with the $\langle 10\bar{1}0\rangle$ directions aligned parallel or close to the axial direction. The density around the $\langle 0001\rangle$ pole is close to 0 multiples of random distribution (mrd). After compression test, the maximum density around the $\langle 0001\rangle$ pole increases to 2.5 mrd while the maximum density around the $\langle 10\bar{1}0\rangle$ pole decreases by 1 mrd. This type of sharp change in the texture is typically caused by the common $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ extension twinning as previously reported in Zr alloys and other hcp metals [23, 24] under this sense of loading relative to the texture.

Standard 3 mm discs were made with the disc normal either parallel to the longitudinal or transverse directions of the compressed samples, termed 'longitudinal' and 'transverse' samples respectively. The discs were ground by sand paper down to 100 μm and twin-jet electropolished with 10% perchloric in methanol at −40 °C in a TenuPol 5 electropolisher for TEM observation.

As has been suggested by the evolution of the texture, $\{10\bar{1}2\}$ extension twins (determined by ~85° lattice rotation around the $\langle 11\bar{2}0\rangle$ direction) are extensively observed in the grains with their $\langle 10\bar{1}0\rangle$ directions aligned close to the compressive axis (*i.e.*, 'prism grains'). In these prism grains, the parent grain [0001] direction experiences tensile strains/stresses due to Poisson effects. Fig. 2 shows an example of the $\{10\bar{1}2\}$ deformation twin in a prism grain in the longitudinal sample. The twin is re-oriented with the [0001] direction parallel to the loading axis. At higher magnification, some 'stacking fault' like features parallel to the basal plane are observed in the twin under $\mathbf{g} = \mathbf{0002}$ ($\mathbf{Z}$ ~ $[11\bar{2}0]$) two beam bright field (BF) condition. This observation is not an unusual case, as similar features are also observed in many other twins examined, see Fig. 3(a). These features exhibit fringe contrast and become wider once the twin is tilted away from basal plane edge-on condition to $\mathbf{g} = \mathbf{01\bar{1}1}$ ($\mathbf{Z}$ ~ $[11\bar{2}0]$) as shown in Fig. 3(b) and (c). The spacing of the fringes changes with diffraction deviation parameter (s_g); that is, the larger the s_g, the smaller the spacing is. This characteristic of these features confirms that these features are actually basal stacking faults.

When observation was made in the transverse sample, SFs were also observed. Fig. 4 shows the SFs observed in the transverse sample. Fig. 4(a) and (b) are taken under the same **g** vector but different $\mathbf{s_g}$. This shows that the spacing of the fringes changes with the $\mathbf{s_g}$. The fringes of the stacking fault A in Fig. 4(a) are parallel to the $[\bar{2}110]$ direction, however, the width of the SF contrast decrease from 27 nm to 9 nm when the orientation of the twin is tilted from the $(01\bar{1}0)$ plane edge-on condition to $(10\bar{1}0)$ plane edge-on condition. This means that the stacking fault A is not on the $(01\bar{1}0)$ prism plane. Since the tilting tends to make the $(01\bar{1}1)$ pyramidal plane move toward the edge-on condition, it can be determined that the stacking fault A is on the $(01\bar{1}1)$ pyramidal plane. Stacking fault B is edge-on when the $(01\bar{1}0)$ plane is edge-on and shows fringe contrast when the $(01\bar{1}0)$ plane is inclined. Therefore, the stacking fault B is on the $(01\bar{1}0)$ plane. All the pyramidal SFs observed in the twins are determined to be on $\{01\bar{1}1\}$, no SFs on the second order pyramidal planes are observed.

More frequently, those pyramidal and prismatic SFs were observed close or attached to TB as shown in Fig. 4(d–f). It should be noted that 5 SFs (2 pyramidal SFs and 3 prismatic SFs) on different atomic planes connect to each other and form an open irregular shape (highlighted by red lines in Fig. 4(e)), with the opening pointing toward the TB.

Besides the SFs with well-defined fringes, some 'anomalous' features with a straight trace but no well-defined fringes even after being tilted to different orientations are also observed, as shown in the area circled by a dashed line in Fig. 4(a). These anomalous features are observed more frequently than the SFs with well-defined fringes within the $\{10\bar{1}2\}$ twins. The features are obviously different from the contrast caused by dislocations observed within $\{10\bar{1}2\}$ twins and dislocation slip traces. The origin of the 'anomalous' contrast is unclear. However, such features were also observed by us (exclusively) in the extension twins in ~10% uniaxially compressed Zr-2.5Nb alloy rod, which has a similar microstructure to the Zr-Excel samples; no pyramidal or prismatic SF with well-defined fringes were observed in that case (see Supplemental materials for Zr-2.5Nb data).

The SFs on the basal planes usually cross the twin and have either both ends or a least one end connected to the TB(s). The basal SFs observed here are actually consistent with the basal SFs within $\{10\bar{1}2\}$ twins previously observed in other hcp metals [9,10,12,14,15]. Song and Gray [9] postulated that the reason for the formation of basal SFs in $\{10\bar{1}2\}$ twins in hcp metals is that the $\{10\bar{1}2\}$ twinning cannot be accomplished by a pure homogeneous shear process along the twinning direction. In hcp metals, basal planes stack in a sequence of ABAB. When $\{10\bar{1}2\}$ twinning occurs, matrix-A plane can transform to twin-A plane by a shear strain, while an atomic shuffling is required to transform matrix-B to twin-B plane. The atoms on the matrix-B plane can shuffle to the normal position (twin-B plane) or stacking fault position (twin-C) position [9]. Once the stacking fault forms, the stacking order becomes ABABA|CACA. As has been pointed out by Song and Gray [9], the stacking fault is different from the three well established types of stacking faults, which include I_1 (intrinsic), I_2 (formed by dislocation dissociation), and E (extrinsic). Although there is a $1/3\langle 10\bar{1}0\rangle$ displacement between the twin-B and twin-C plane, twin-A plane is not displaced as seen from the stacking sequence. This stacking fault is not accomplished by the gliding of a partial dislocation, and only involves the displacement of the B plane, therefore it was named a 'partial stacking fault' (PSF) [9]. The atomistic modeling performed by Zhang et al. [14] has confirmed the basal partial stacking fault proposed by Song

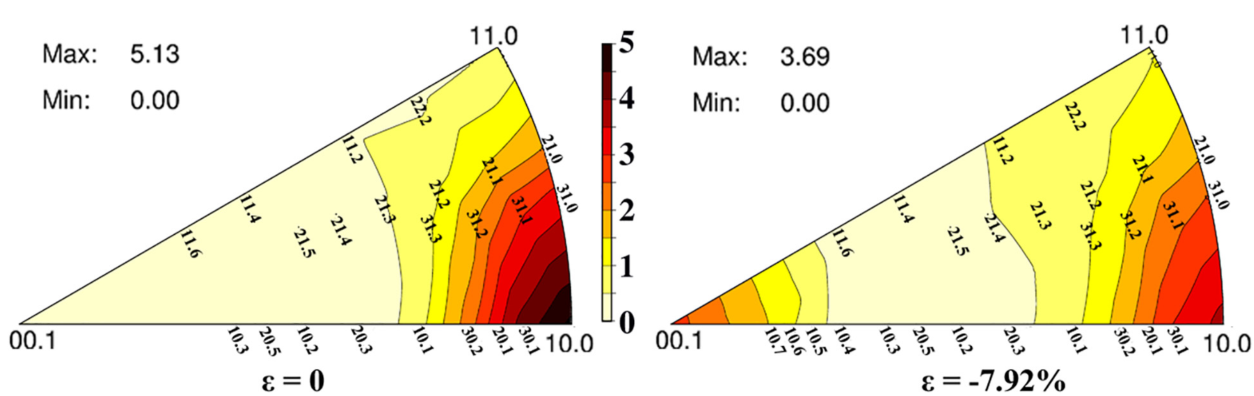

Fig. 1. The evolution of the inverse pole figure of the α phase in the compressive axis direction.