In-situ investigation of the evolution of microstructure and texture during load reversal of Commercially pure titanium using Synchrotron X-ray diffraction

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Abstract

Understanding of the deformation micro-mechanism as a function of grain orientation during cyclic loading is of significant importance to have failure safe design of structural components made of commercially pure titanium (CP-Ti). The evolution of deformation microstructure and texture of commercially pure titanium samples with prismatic-pyramidal (orientation A) and near basal (orientation B) as initial texture along the loading direction has been investigated during load reversal at $\pm 8\%$ and $\pm 12\%$ strain using in-situ synchrotron X-ray diffraction. The synchrotron X-ray diffraction results have been further complemented with Elastic-Plastic Self-Consistent (EPSC) simulation of the texture data and ex-situ Electron backscatter diffraction (EBSD) scan taken at the same region. Orientation A showed partially reversible texture whereas

orientation B showed non-reversible texture with mechanical reversibility. The partial textural reversibility has been attributed to the prism to basal slip transition which toggles the c-axis between the normal and transverse direction during the tension-compression cycle. With an increase in strain from 8 to 12%, microstrain and dislocation density in the basal plane of orientation A decreases sharply. On the other hand, for the same level of strain in orientation B, microstrain increases but the dislocation density of basal plane shows insignificant change. The crystal orientation map obtained from ex-situ EBSD of deformed microstructure complements to the fact indicating, with increase in strain, deformation of basal oriented grains show non-Schmid behaviour of contraction twin propagation due to local strain incompatibility in orientation A. On the other hand, the prism oriented grains of orientation B have mostly deformed by extension twinning, which reorients them towards basal orientation. The deformation proceeds by lateral thickening of twins due to the lower elastic stiffness of the twinned region compared to the grain matrix. The extension twin boundaries get converted to contraction twin boundaries at higher strain during load reversal.

Keywords: CP-Ti; Load reversal; Synchrotron X-ray; EBSD; dislocation density; EPSC.

1. Introduction

Commercially pure titanium (CP-Ti) sheet is gaining attention in the electronic industry such as in the cover cases of notebook, mobile phone, computer, etc. [1]. For the production of such thin-walled structural components, press forming and stamping of CP-Ti sheets are carried out. However, due to low symmetry hexagonal crystal structure, rolled sheets of titanium exhibits strong anisotropic strain hardening behavior notably between the rolling direction (RD) and the transverse direction (TD) under monotonic [2] as well as cyclic [3] loading condition. In

titanium, the strain along <0002> crystallographic axis decides the slip/twin activity. The orientation of the <0002> crystallographic axis with respect to the loading direction is important in accommodating the strain in the different directions. This reduces the forming limit of CP-Ti and hence, the press forming process becomes difficult at room temperature. Although formability could be improved at elevated temperatures, however, a cost-effective manufacturing process would desire room temperature. Hence, the proper understanding of the effect of initial grain orientation on the evolution of deformation microstructure during load reversal at ambient temperature is necessary, in order to have stringent control over the forming parameters.

Load reversal often leads to tension-compression asymmetric deformation behaviour and this directionality of strain hardening is termed as Bauschinger effect [4]. The most widely explained theory of Bauschinger effect is through dislocation pile-ups at certain barriers such as grain boundaries, precipitate, etc. leading to lowering of stress required during compression compared to forward tension cycle. The following equation (1) explains the stress required to move dislocation pile-ups [5]

$$\tau_p = n\tau_a \dots\dots\dots (1)$$

Where n is the number of dislocations in a pile-up. The material doesn't undergo uniform internal stress over the entire cross-section even though applied stress τ_a is uniform in a crystalline material. So, local stress develops around dislocation pile-ups. These internal stress fields can be categorized into two ways one among those is short-range like tilt boundary dislocations, the second one spreads to long-range of the microstructure as for example dislocation cell, dislocation tangles, etc. Understanding of mechanisms behind plastic deformation, Bauschinger effect, spring back effect, cyclic deformation is explained by the concept of long-range internal stresses (LRIS) [5].

However, for materials having a lower number of slip systems, twinning plays a significant role in influencing the Bauschinger effect [6]. Yin et al. [7] have shown the detwinning phenomenon during load reversal of Mg responsible for the asymmetry in the hysteresis loop where yield stress in compression is lower than yield stress in tension. The deformation micro-mechanism of CP-Ti involves complex dislocation-dislocation interaction and dislocation/twin boundary interaction and is highly orientation-specific [8]. Hence, it is expected that the initial grain orientation will have a strong influence on load reversal deformation behaviour. A detailed microstructure analysis would require state of the art characterisation technique.

The synchrotron is a very powerful tool for micro-texture measurement as X-rays of very fine spot size and high intensity is generated from very high speed moving electrons in the cyclic particle accelerator. Diffraction patterns of Synchrotron radiations are detected by Charge-couple Device (CCD) detectors and displayed in the form of Debye rings. The presence of texture is identified from intensity variation along with the rings. Also, due to the higher penetration depth and lower absorption, Synchrotron radiation give statistically reliable deformation texture and microstructure related information which cannot be obtained from conventional X-ray and transmission electron microscope (TEM).

Several researchers have reported the effect of initial orientation on monotonic and cyclic deformation behaviour of commercially pure titanium in the past [9-11]. Sinha et al. [9] extensively studied the effect of initial orientation on tensile behaviour from mechanical tensile tests as well as deformation behaviour through twinning from in-situ and ex-situ electron backscattered diffraction (EBSD). The characterisation of deformation heterogeneities near the crack region during tensile and fatigue of CP-Ti using EBSD has also been investigated [12-13]. However, EBSD is a technique used to map lattice misorientation across (2D plane) of a

crystalline sample. Crystal orientation maps, identification of twin boundaries and geometrically necessary dislocation density (GND) can be readily achieved from EBSD. The residual dislocation density and residual twin content assessed gives the projected information about the consequences of deformation history on the 2D plane. With proper calibration, synchrotron X-ray can be used to obtain dislocation density along the plane normal direction of dislocation cell structures developed during multiple slip deformation in polycrystals with increasing plastic strain. Thus, the combined information of EBSD and Synchrotron X-ray data will provide ample scope for obtaining 3D information of deformation at a much sub-structural level. Also, it will provide a much deeper insight into the study of twinning and its nucleation growth mechanism. In the present investigation, in-situ micro-focus synchrotron X-ray radiation and ex-situ electron backscatter diffraction have been combined to study 3D orientation information and deformation microstructure in order to understand the micro-mechanisms of deformation during the load reversal of CP-Ti plate.

2. Experimental

2.1. Material

The present investigation was carried out on grade 2 commercially pure titanium (CP-Ti) with the microstructure consisting of single-phase cold worked and annealed hexagonal close-packed α -titanium. The chemical composition of the material is given in Table 1

2.2. In-situ Load reversal testing

Load reversal tests were carried out on titanium samples obtained from two distinct orientations from the same block of cold-rolled and annealed titanium. In this particular experiment, Orientation A was obtained by machining along rolling direction (RD) in the normal direction

(ND) plane and Orientation B was machined along the transverse direction (TD) in RD plane by Electrical discharge machining (EDM) as shown in Figure 1. Orientation A and Orientation B deformed at different strain levels and designation has been reported in Table 2. The choice of the strain level is based on a previous investigation carried out by Sinha et al. [2]. The two different strain levels correspond to dominant deformation mechanisms comprising of slip + twin growth and twin saturated slip mechanism, respectively. Tensile specimens were made as per ASTM standard E606 specifications as shown in Figure 1a. Samples were tested using screw-driven tensile testing equipment in Desy labs, Germany. Load reversal tests were carried out at three different levels of loads in displacement controlled mode in tension followed with unloading in load controlled mode to zero stress. This was followed with compression in displacement controlled mode followed with unloading in load controlled mode to zero stress. The final step comprises reloading in tension in displacement controlled mode to the same level of strain as that of initial tension. The schematic of the stress-strain curve during load reversal at ± 8 and $\pm 12\%$ strain has been presented in Figure 1b and Figure 1c respectively.

2.3. Synchrotron X-ray Diffraction

In-situ Synchrotron X-ray diffraction was carried out to measure texture in various stages ranging from tension, compression and reload tension. PETRA III beamline P07 with the energy of 6.033 GeV machine was used to measure deformation texture exactly in the middle of the gauge section of the tensile specimen in transmission geometry as shown in Figure 2. This experiment requires specimen as bars with dimensions $15 \times 1.5 \times 1.5 \text{ mm}^3$. A beamline called HEMS P07B of Wavelength $0.143155 \text{ \AA}$ and energy of 87 KeV was used. A Slit system with a width of 0.7×0.7 and a beam size of 0.3 mm was used to obtain sufficient local information. Random sample orientation was taken for texture measurement. Counting time (exposure time)

is 0.2 sec per image for 10 images and 1 sec per image for 4 images. Diffraction data was captured in 37 frames in some cases, 19 frames in other cases to obtain information from all diffracting planes. Perkin - Elmer type digital solid-state area detector was used with a distance of 1000 mm from the surface of the sample. Single-shot measurements were taken. The Debye-Scherrer rings obtained have been converted to an intensity peak profile as shown in Figure 3 using Fit2D software [14] was used to analyse the data. The output of Fit2D was the input for Resmat software [15]. The data were used for the construction of 3D-Orientation distribution function, pole figures, inverse pole figures.

2.4. Electron backscatter diffraction (EBSD)

Samples were prepared by cutting the gauge portion from the load reversal test specimens. The samples were polished till 3000 grid emery paper followed by electropolishing using Struers *Lectropol* electropolishing machine in A3 electrolyte at 30V for 15s. After electro polishing samples were characterized using JEOL Field Emission Gun Scanning Electron Microscope (FEG-SEM) with EBSD detector attachment at an operating voltage of 20kV and 12 μA probe current. An area of $300 \times 300 \mu\text{m}^2$ was scanned exactly in the middle of the gauge portion of the tensile specimen where Synchrotron diffraction was carried out at step sizes of 0.2, 0.25 and 0.4 μm respectively based on the resolution required. EBSD analysis was carried out using TSL software. Crystal orientation map, Grain Reference Orientation Deviation (GROD) map, Geometrically Necessary Dislocation density (GND) map, Pattern Quality map, Kernel Average Misorientation (KAM), Misorientation profile, Schmid Factor map, Elastic stiffness maps were generated using TSL software.

2.5. EPSC simulation procedure

In the present study, Elastic-Plastic Self-Consistent (EPSC) simulations [16, 17] of one-cycle tension-compression deformation has been carried out for samples to obtain the activity of slip and twin deformation modes during monotonic and cyclic loading. The list of initial orientations (Euler angles φ_1 , Φ , and φ_2) obtained from the Synchrotron X-ray data of the undeformed specimens has been used as the input texture for the EPSC simulations. The EPSC-4 model [18] predicts the elastoplastic response of a polycrystal from single-crystal elastic stiffness data and Critical Resolved Shear Stress (CRSS) and hardening data of operative slip and twin systems for the imposed strain. The hardening of various slip and twin systems is described by the following extended Voce law as given in equation 2.

$$\hat{\tau}_s = \tau_0^s + (\tau_1^s + \theta_1^s I) \{1 - \exp(\frac{\theta_0^s}{\tau_1^s})\} \dots\dots\dots(2)$$

The Voce hardening parameters (τ_0 , τ_1 , θ_0 , and θ_1) [2] and the elastic stiffness parameters [19] used as input into EPSC-4 to perform the simulation has been provided in Table 3. Five important operative slip and twin systems (prismatic, basal, and pyramidal $\langle c + a \rangle$ slip and extension and contraction twinning) have been considered in the simulation similar to investigations on CP-Ti carried out by few researchers earlier [20-21]. The hardening coefficients for interaction between different slip and twin systems are also shown in Table 3. The hardening coefficient of unity is used for interaction between primary and secondary slip systems as well as for slip/twin interaction. However, a slightly higher value of 1.5 is considered for twin-twin interaction because the occurrence of twinning is associated with hardening (mechanisms of twinning-induced hardening include Hall-Petch and Basinski hardening as well as the ability of twins to act as barriers for the propagation of dislocations or other twin lamellae [22-23]. It should be noted here that the effect of back stress was not incorporated in the present

load reversal simulations and, hence, no attempt has been made to match the experimental and simulated results. We only aim to show the difference between different orientations for load reversal.

3. Results

3.1. Initial microstructure and texture

The as-received cold-rolled and annealed plate of commercially pure titanium has been represented schematically in Figure 4a with two different orientations A and B of load reversal test specimen. It exhibits characteristic TD split basal texture as indicated from the 0002 pole figure shown in Figure 4b. Orientation A and Orientation B have a distinctly different texture as indicated from the crystal orientation map and the Inverse pole figure (IPF) contour plot (inset) shown in Figure 4c. Orientation A has crystallographic direction $\langle 10\bar{1}0 \rangle$ - $\langle 11\bar{2}0 \rangle$ parallel to RD while in orientation B crystallographic direction $\langle 0002 \rangle$ is parallel to RD.

3.2 Deformation texture

The experimental 0002 and $10\bar{1}0$ pole figure contour plot obtained from in-situ Synchrotron X-ray diffraction of tension-compression-reload tension and simulated final deformation texture of all the samples have been shown in Figure 5. It indicates a partial reversal of texture with mechanical reversibility in sample A8 and sample A12. The 0002 poles rotate towards the normal direction (ND) during the tensile cycle, then reverts to the loading direction (LD) during the compression cycle and again come back to ND during reload tension. While in sample B8 and sample B12, the deformation texture stabilizes to a non-reversible texture. The $10\bar{1}0$ poles initially oriented towards RD get splits along ND which points the 0002 poles towards RD. The experimental and simulated final texture matches to some extent, however, the pole positions are

slightly shifted in sample A8 and sample A12. However, the change in 0002 and $10\bar{1}0$ poles position in sample B8 and sample B12 could not be predicted in the simulated texture. It indicates the influence of local shear stress on texture is significant. EPSC simulation cannot take into account deformation by twinning accurately due to microstructural heterogeneity. Hence, the evolution of deformation microstructure with the increase in strain for two different orientations has been analysed further.

3.3 Microstrain and dislocation density

A quantitative estimate of the lattice strain and the density of dislocations involved in tension-compression-reload tension have been obtained using in-situ synchrotron X-ray diffraction. The peak profile analysis based on the modified variance method [24] has been used for the calculation of microstrain and dislocation density. The broadening of X-ray diffraction peaks corresponding to prism $\{10\bar{1}0\}$ planes are mainly affected by the deformation along $\frac{1}{3}\langle 11\bar{2}0 \rangle$ (type $\langle a \rangle$ dislocations) whereas diffraction peaks corresponding to the basal $\{0002\}$ plane are more influenced by $\langle 0002 \rangle$ (type $\langle c \rangle$ dislocations) [25]. In the present investigation, the microstrain and dislocation density of prism $\{10\bar{1}0\}$ and basal $\{0002\}$ plane only has been estimated as shown in Figure 6, considering the initial texture of orientation A and orientation B. With the increase in strain from 8 to 12%, the microstrain and dislocation density of the prism plane during reload tension (indicated by blue stars) decreases in orientation A. Whereas, for the same level of strain in orientation B, the microstrain of the prism plane during reload tension increases slightly as compared to tension but the dislocation density (indicated by black dotted arrow) shows an appreciable increase. On the other hand, in the basal plane of orientation A with increase in strain from 8 to 12%, microstrain and dislocation density

(indicated by black dotted arrow) decreases sharply during tension and reload tension. Whereas, for the same level of strain in orientation B, microstrain (indicated by blue stars) increases but dislocation density (indicated by red dotted arrow) shows the insignificant change in the basal plane. The change in microstrain is not appreciable but the dislocation density of the prism plane decreases and basal plane increases during compression in both orientation A and orientation B. The observed change in microstrain and dislocation density is due to the contribution from $\langle a \rangle$ type dislocation produced by prism slip in case of prism plane but not from the $\langle a \rangle$ type dislocation produced by basal slip in case of basal plane. The change in microstrain in the basal plane could not be interpreted directly. The strain along the c-axis is accommodated by twinning and $\langle c+a \rangle$ pyramidal slip dislocation. However, the lattice strain arising due to twinning is very small. Thus, the insignificant change in dislocation density in the basal plane of orientation B could be due to twinning. But, the relatively less change in microstrain in the prism plane of orientation B with a significant increase in dislocation density is not well understood. The results indicate that the dislocation structure evolves with the load cycle, increase in strain and is different in the prism and basal oriented grains. In CP-Ti, plastic deformation proceeds through the simultaneous activity of a number of slip and twin systems. Thus, a thorough understanding of slip dislocation activity with an increase in strain and load cycle is required and has been calculated using EPSC simulation in the next section.

3.4 Relative slip/twin activity using EPSC simulation

The relative slip/twin activity during the individual tension-compression-reload tension cycle has been calculated using EPSC simulation as shown in Figure 7. It indicates deformation is governed by prism slip at lower strain and is taken over by basal slip in sample A8 at strain

(~0.04) and pyramidal slip in sample A12 at strain (~0.11) during initial tension. In sample A8, basal slip is taken over by pyramidal slip after compression to (~0.035) and reload tension whereas, sample A12 exhibits purely pyramidal slip activity during compression and reload tension. Sample B8 and sample B12 shows higher prism slip activity and is completely taken over by pyramidal slip beyond strain (~0.07) during initial tensile loading. It shows appreciable prism slip activity during initial compression and the deformation is dominated by pyramidal slip beyond compression strain (-0.01) and (0.07) in sample B8 and sample B12, respectively and during reload tension. Also, detwinning of extension twin has been predicted in sample B8 but not in sample B12. EPSC simulation can predict the slip dislocation activity with precision but not the twin activity which could be due to microstructure heterogeneity arising with the increase in strain. In order to get a complete picture, the characterisation of twins has been done using EBSD analysis.

3.5 Deformation microstructure and microtexture

The deformation microstructure of all the samples has been represented using an image quality map obtained from the SEM-EBSD scan in Figure 8. The colour contrast of indexed (grey region) and non-indexed (black region) indicates less deformed and highly deformed regions within the grains, respectively. Also, all the samples show the presence of deformation twins. Sample A8 has very thin twins. As sample A12 is highly deformed and some portion is unindexed it makes interpretation difficult. The twins in sample A12 have fragmented the large grains into smaller regions. The type of twin in each sample could be identified from band contrast map delineating the contraction (blue line) and extension (red line) twin boundaries in Figure 8 and their relative fractions could be obtained from the misorientation distribution profile

shown in Figure 9. It indicates that in sample A8, maximum fraction (~ 0.05) contraction twin type $\{11\bar{2}2\} < 11\bar{2}\bar{3} >$ with a misorientation angle of approximately 63° , while in sample A12 extension twins type $\{10\bar{1}2\} < \bar{1}011 >$ with misorientation angle of approximately 85° has also formed. Sample A12 shows an almost equal volume fraction of both contraction and extension twin. The extension twins have formed within the contraction twin in sample A12. While sample A8 and sample A12 has a higher fraction of low angle grain boundaries (~ 0.7) compared to twin boundary fraction (~ 0.05). This could be due to overriding slip dislocation leading to the conversion of twin boundaries into low angle grain boundaries at higher strain. However, the situation is exactly reverse in the case of sample B8 (~ 0.25) and sample B12 (~ 0.54) with low LAGB fraction. The maximum fraction of extension twin boundary is high in sample B8 (~ 0.26) and sample B12 has contraction twin (~ 0.1) and extension twin (~ 0.05) by re-twinning of contraction twin. The characteristic features of twins in different samples have been summarized in Table 4.

Thus, initial texture and percent strain both influences twin nucleation and twin growth mechanisms. Sample A8 and sample A12 showed a very low volume fraction of extension twin, however, the relative slip/twin activity plot has been shown in Figure 7, which depicts high extension twin activity at very low strain (~ 0.01) during tension cycle. In order to understand the underlying mechanism for twin nucleation, growth, and twin saturation, the factors controlling local stress-strain conditions within individual grains have been investigated.

The Schmid factor can determine the activity of a particular slip system, similar is the case with twin activity too. The Schmid factor map (Figure 8) for contraction twin shows that twins are present in grains with medium Schmid factor (yellow) for contraction twin in both Sample A8 (grains marked as 1, 2 and 3) and sample A12 (grains marked as 4 and 5). In contrast, in sample

B8 (grains marked as 6 and 7) and sample B12 (grains marked as 9,10 and 11) the extension and contraction twins respectively, are formed in grains having high Schmid factor (red) for both contraction and extension twin. Thus, twinning follows non-Schmid propagation behaviour of contraction twin in sample A8 and sample A12. The contraction twins with high Schmid factor for extension twins in sample A8 (grain marked as 12), A12 (grain marked as 4) and B12 (grain marked as 11) have re-twinned to form extension twin. Schmid factor can explain nucleation and propagation but not lateral thickening or growth. Lateral thickening is explained by the variation of elastic stiffness difference across the parent matrix and twin boundary. The underlying principle behind elastic stiffness calculation is the distortion of the parent grain and the boundary between parent grain and newly formed twins. Elastic stiffness of twinned region in sample A8 and sample A12 is medium (yellow) and high (red) respectively and that of the grain matrix is low (blue). In contrast, sample B8 has lower (blue) elastic stiffness of twins than that of parent grain (red). While sample B12 has twins of both high (red) and medium (yellow) elastic stiffness. Thus, the Schmid factor map and elastic stiffness constant map could explain the nature and twinning propensity in most cases but not in all (grain marked 6 in sample A12 and grain marked 12 in sample B12). Local stress state also plays an important role in twin nucleation apart from the Schmid factor [26-27]. The local stress state influences the evolution of dislocation density with an increase in strain and hence, twin nucleation and growth.

3.6 Local average misorientation and GND density

The crystal orientation map shown in Figure 10 indicates, most of the near basal (pink) and prism (blue) grains are twinned in sample A8 and sample A12. The twin free pyramidal (green) grains show colour gradient especially in sample A8 indicating local strain and local orientation

difference within individual grain due to dislocation activity. Sample A12 shows the extensive grain and twin fragmentation. In sample B8 grains with pyramidal (green), orientation is twinned and the twins have a basal (red) orientation. While in sample B12 grains predominantly aligned along prism (blue) orientation are twinned and the twins have near basal orientation.

Kernal average misorientation (KAM) maps of all the samples have been shown in Figure 10. The overall KAM value for sample A8 and sample A12 is higher compared to sample B8 and sample B12. Sample A8 and sample A12 shows homogeneous KAM distribution while that of sample B8 and sample B12 is heterogeneous, mostly concentrated near the grain and the twin boundaries.

The dislocation density includes contribution only from the geometrically necessary dislocations (GND) present at the grain boundary, twin boundary and at the dislocation cell boundary to maintain continuity between adjacent deforming regions. The statistically stored dislocations (SSD) produce insignificant lattice curvature and hence do not contribute to total dislocation density.

Geometrically necessary dislocation (GND) density has been calculated from lattice curvature obtained from EBSD data following the method given in reference [28-29] using equation 3 [30] given below. GND density maps have been generated using TSL software and shown in Figure 10.

$$\rho^{\text{GND}} (\text{m}^{-2}) = \frac{\theta(\text{rad})}{x(\mu\text{m}) \times 10^{-6} \times b} \dots\dots\dots (3)$$

Where θ = misorientation, x = step size, b = burgers vector in m

Sample A8 shows GND density is very low in twin free grains but high within the twinned region. Sample A12 shows high GND density in the fragmented grain region due to more strain

or deformation. However, the situation is exactly reverse in the case of sample B8 and sample B12. Khosravani et al. [31] have reported low GND density within twins of magnesium alloy. Thus, the evolution of local average misorientation and GND density within the grain and the twinned region also depends on the initial texture and could be correlated with the lattice strain generated due to slip and twin dislocation activity.

4. Discussion

The above results indicate that the initial texture has a strong influence on the evolution of deformation microstructure and micro-texture during load reversal. In the following discussion, the orientation dependence of the evolution of deformation texture, microstrain due to energetically stable dislocation configuration and twin features with mechanical reversibility will be elucidated.

4.1 Effect of initial orientation on textural reversibility

From the above deformation texture result, it is well understood that initial orientation has a significant influence on textural reversibility with load reversal. It has been observed that in addition to operative slip mode, the nature and the volume fraction of twin also plays a significant role in the evolution of deformation texture. As indicated from the slip/twin activity curve shown in Figure 7, it is clear that with increase in strain, deformation mode changes from slip + twin at lower strain (8% in sample A8 and sample B8) to slip following twin saturation at higher strain (12% in sample A12 and sample B12). However, with mechanical reversibility, partial textural reversibility in sample A8 and sample A12 has been observed. On the other hand, sample B8 and sample B12 showed an evolution of non-reversible stable texture different from the initial texture. In Orientation A, deformation takes place via prism slip initially upto ~ 0.04

strain and by basal slip activity in the strain range of 0.04-0.11 during forward tension. However, during the compression cycle there is initial prism slip activity as indicated from the EPSC simulation. The basal slip occurs via tilting and the prism slip via rotation of the $\langle 0002 \rangle$ crystallographic axis according to Wang et al. [32]. Thus, mechanical reversibility toggles the $\langle 0002 \rangle$ crystallographic axis between the normal and the loading direction during the tension-compression cycle, which is responsible for partial textural reversibility observed in orientation A. On the other hand, the formation of non-reversible texture in case of orientation B could be attributed to dominant prism slip activity which causes rotation of the $\langle 0002 \rangle$ crystallographic axis permanently away from the loading direction.

Very interestingly, in the evolution of deformation texture, two different modes of twinning are taken as the intermediate deformation paths during prism to basal slip transition in orientation A and from prism to pyramidal slip transition in orientation B as indicated from the crystal orientation map. In orientation A, the prism to basal slip transition occurs via contraction twin. The EPSC simulation results show that prism activity ceases beyond ~ 0.07 strain. Twinning requires some slip activity in order to have twinning partial dislocations. As the prism activity ceases, thus twinning also ceases due to a reduction in twinning partial dislocations in the grain matrix. The crystal orientation map shows the presence of extension twin boundary in sample B8. However, in sample B8 prism slip is active during compression upto strain of $\sim (-0.001)$ which can cause detwinning of the extension twin. Sample B12(12%) has been subjected to higher strain as compared to B8(8%) during forward tension. Therefore, in sample B12 deformation occurs by pyramidal activity during forward loading beyond ~ 0.07 strain. There is less prism activity only upto ~ 0.06 strain during compression in sample B12 as compared to B8. Thus, there is insufficient twinning partial in the grain matrix to cause detwinning in sample B12.

during compression. Since, detwinning is a phenomenon of thinning of the twinned region via reorientation to parent grain as described by Wang et al. [33]. Therefore, detwinning also requires twinning partial dislocations for twin nucleation [34] and twin growth would depend on the local stress state at the twin front [35]. Rather, sample B12 showed the conversion of the existing extension twin boundaries into contraction twin boundaries due to pyramidal slip activity.

4.2 Evolution of dislocation density during load reversal

Load reversal causes asymmetric strain hardening behavior known as the Bauschinger effect. Bauschinger effect occurs due to the generation of backstresses at the pile-up dislocation. However, Bauschinger parameters are influenced by the density of dislocation which depends on the active slip/twin system of the crystal structure, crystal orientation and the amount of prestrain. But immobile dislocations do not contribute to backstress during pile-up. The number of immobile dislocations will increase with the increase in pre-strain due to the interaction of more dislocations. With increase in pre-strain, backstress will increase upto a certain limit and then saturates or decreases depending on the number of mobile dislocations. The microstrain generated due to lattice distortion and the mobile dislocation density involved in maintaining continuity between adjoining deforming regions will also depend on the backstress generated in the microstructure. The concept of dislocation density in hcp twinned microstructures is not very well understood as reported by Khosravani et al [31]. Also, the contribution of twin/matrix dislocation in long-range internal stress is small and hence can be neglected [4].

With the increase in strain from 8 to 12% in sample A8 to sample A12, the decrease in microstrain and dislocation density of the slipped region of the prism oriented grains is due to the

decrease in prism slip activity during reload tension. Since, the peak broadening of the basal plane is due to $\langle c \rangle$ type dislocation and hence, is not affected by basal slip activity but by twinning. The sharp decrease in dislocation density of the slipped region of the basal oriented grains during tension and reload tension could be due to extensive grain and twin fragmentation by pyramidal slip in sample A12. On the other hand, in sample B8 and sample B12, grains have prism ($//ND$) and pyramidal ($//ND$) orientation. The prism slip activity is relatively high compared to orientation A. However, the slipped regions of the prism oriented grains show less change in microstrain inspite of an appreciable increase in dislocation density with the increase in strain. The twins formed in the prism oriented grains during tensile loading have the basal orientation as depicted from the crystal orientation map shown in Figure 10. The dislocations within the twinned basal regions are sessile and do not contribute to slip. Thus, the dislocation density within the twinned basal region showed no change. But the microstrain increases due to the lateral thickening of extension twin boundaries and conversion of extension twin boundaries into contraction twin boundaries with the increase in strain as indicated in Figure 8. From the slip/twin activity plot shown in Figure 7, it is clear that the pyramidal slip activity is higher during compression and reload tension. However, screw dislocations of $\langle c+a \rangle$ type can move to the next slip planes by double cross slip followed by dislocation annihilation, which leads to the decrease in the density of $\langle c+a \rangle$ type dislocation [36].

As evident from the misorientation distribution plot shown in Figure 9, sample A8 and sample A12 has higher LAGB fraction indicating the formation of dislocation cell structure during load reversal. EPSC simulation result showed higher basal activity in sample A8 and sample A12. Cross slip of type $\langle a \rangle$ basal slip dislocations in the basal plane could be the reason for the same [37]. Cross slip leads to the formation of a 3D dislocation cell structure. These dislocation cell

structures are the regions of low dislocation density with a well-defined cell wall acting as the barrier to pile-up dislocation. On the other hand, for the same level of strain, sample B8 and sample B12 show lower LAGB fraction. The low LAGB fraction is due to appreciable prism slip and extension twin which can easily detwin and exerts less potential dislocation barrier for prism slip to continue. However, this is not the case with sample B12, due to straining in the twin saturated regime of deformation, it exhibits relatively higher pyramidal activity (as indicated from Figure 7) by cross slip resulting in slightly higher LAGB fraction of sample B12 compared to sample B8. Therefore, it is clear that the dislocation structure is strongly influenced in the twin active regime of deformation and is highly orientation-specific too.

4.3 Effect of orientation on Twinning/detwinning

Normal stresses play a key role in twin nucleation but shear stresses play a role in twin propagation [34, 38-39]. Extension twin nucleation may be attributed to local stress concentrations due to dislocation slip while contraction twins may be formed due to strain incompatibilities as reported by Galindo-Nava et al. [40]. Bao et al. [41] have studied the deformation behaviour of CP-Ti through the twinning mechanism and reported that contraction twins nucleate and propagate till they intersect some boundary during initial deformation and further deformation takes place due to repeated nucleation. Extension twins cause initial deformation by repeated nucleation and further deformation due to lateral thickening [41]. Twin nucleation occurs by the co-operative movement of partial dislocations on parallel twin planes [39]. In hcp metals, movement of zonal dislocations precedes twin nucleation and synchronous movement of these dislocations can cause higher contraction twinning.

The high KAM and high GND density within the twins compared to the grain matrix indicates high local strain incompatibility at the twin tip in sample A8 and sample A12 shown in Figure 10. This makes the energetically favourable condition for contraction twin nucleation and hence lengthening of contraction twin in sample A8 and twin fragmentation at the relatively higher strain in sample A12. Contraction twins have high critical resolved stress value and hence contraction twins can nucleate and propagate easily in the grain matrix having high local stress state. The high value of Kernel average misorientation (KAM) and geometrically necessary dislocation (GND) density indicates local strain incompatibility. Thus, the regions of high local stress concentration in the grain matrix, which fulfills the energy criteria required for contraction twin formation and its propagation in grain matrix with low Schmid factor for contraction twin.

As contraction twins have high Critical Resolved Shear Stress (CRSS), they exhibit low lateral growth rate and hence strain accommodation via lateral thickening of contraction twin is limited. With the increase in strain during forward tension, harder pyramidal slip is activated beyond 0.11 strain. The pyramidal slips dominate the deformation mechanism during compression and reload tension as indicated from the EPSC simulation result shown in Figure 7. As the applied cyclic strain in A12 ($\pm 12\%$) is higher compared to A8 ($\pm 8\%$), therefore, deformation occurs mostly by activity of harder pyramidal slip which causes more grain and twin fragmentation in A12 compared to A8.

On the other hand, low KAM and moderate GND density at the grain and the twin boundary indicates non-uniform strain due to the formation of extension twin of higher misorientation angle with the grain matrix in sample B8. The evolution of KAM is more homogeneous even though the KAM value is high in sample A8 and sample A12. This explains the non-Schmid

propagation behaviour of contraction twin as observed in Figure 8. Non-Schmid propagation behaviour is more prevalent in contraction twinning because of twin shear which is more for contraction twinning than extension twinning. So, the formation of contraction twins in sample A8 and A12 is easier which reorient by small angle than extension twins which reorient by large angles even though in a particular grain Schmid factor is more for extension twins than contraction twins. The heterogeneous distribution of KAM at the twin and the grain matrix interface in sample B8 and sample B12, indicates higher number of twin nucleation sites at the twin boundaries. However, due to the activation of pyramidal slip, the grain matrix remains twin unsaturated in sample B8 and B12. The higher number of twin nucleation sites and the local stress state promote detwinning phenomenon during the compression cycle in the twin unsaturated grain matrix of sample B8. This is because detwinning occurs by the nucleation and glide of twin dislocations at the twin boundaries. The gliding of twin dislocations depends on the resolved shear stress of the twin or the grain matrix. Usually, all the mobile dislocations become sessile once the twin is formed. Thus, the resolved shear stress of the grain matrix unsaturated of twins during forward loading causes detwinning during compression and shrinks the twin thickness. On the other hand, sample B12 shows more contraction twin boundaries which could be due to the nucleation of contraction twin at the pre-existing extension twin boundary followed by its coalescence during unloading from tensile strain as proposed by Jiang et al. [42].

Elastic stiffness compatibility is a prerequisite for twin growth because there is a volume change during the transformation of parent matrix or grain into twins. If there is too much variation in elastic stiffness between parent matrix and grain for example if the twin formed is elastically stiffer than parent grain, the resultant grain after reorientation is harder at the twin region. In this case, deformation is difficult so the growth of the twin is unfavourable because it is transforming

more volume of material from soft to hard region. This situation leads to more nucleation but less growth or lateral thickening. If the elastic stiffness of the twin is almost the same or lower than the grain matrix, then it will result in substantially favourable lateral growth.

Elastic stiffness of the twinned region in sample A8 and sample A12 has medium (orange) and high (red) elastic stiffness respectively, results in limited lateral growth. Multivariant twins formed in sample A12. Since the lateral growth of twins in an elastically harder parent grain matrix is energetically favourable as it involves lowering of elastic strain energy of dislocations from harder grain matrix to softer twinned region. Thus, sample A8 and A12 exhibit restricted thickening and hence thin twins. In sample B8, elastic stiffness of twins is lower (blue) than that of parent grain (red) which results in laterally thick twins. In sample B12 elastic stiffness of twins and parent grain are almost similar so both extension and contraction twins are laterally thick. Second variant twins in B12 are much thicker (yellow) due to less difference in elastic stiffness between twins and parent grains.

GND density is also influenced by the nucleation of twins like at grain boundaries and intersection between grain boundary and twin boundary. Usually, twins formed at grain boundaries have less GND density compared to twins formed at the intersection of grain boundary and twin boundary. Highly deformed A12 sample has laterally grown twins in some grains with high GND density. At very high strain levels, deformation occurs due to pyramidal slip when twinning saturates [43]. Hence dislocation slip assisted twinning mechanisms are responsible for high GND densities inside the twins and at the twin boundaries.

Summary and conclusions

- The Initial texture of the CP-Ti plate has a strong influence on deformation texture during load reversal. Mechanical reversibility accompanied with partial textural reversibility in orientation A but a non-reversible texture evolution in orientation B.
- As initial orientation has a strong influence on the operative slip/twin mode of deformation mechanism. Thus, with the increase in strain from 8 to 12%, microstrain of prism plane decreases due to lower prism slip activity and prism to basal slip transition occurs. On the other hand, for the same level of strain, prism plane has higher microstrain due to more prism slip activity and prism to pyramidal slip transition occurs in orientation B.
- The prism to basal slip transition in orientation A occurs via non-Schmid propagation of contraction twin in orientation A. Whereas, in orientation B lateral thickening of extension twin is the intermediate step in prism to pyramidal slip transition. As Schmid factor is a necessary condition for the nucleation of a twin but not sufficient for all twin formation. Local strain incompatibility at the twin front provides the driving force for the non-Schmid behaviour of contraction twin propagation. While elastic stiffness difference between twin and the parent grain causes lateral thickening of extension twin.

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Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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