Final Draft
of the original manuscript:

Ghosh, A.; Brokmeier, H.-G.; Al-Hamdany, N.; Sinha, S.; Schell, N.; Gurao, N.:

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In: Materials Science and Engineering A 726 (2018) 143 - 153
First published online by Elsevier: April 22, 2018

DOI: 10.1016/j.msea.2018.04.036
https://dx.doi.org/10.1016/j.msea.2018.04.036

A synchrotron X-ray and electron backscatter diffraction based investigation on deformation and failure micro-mechanisms of monotonic and cyclic loading in titanium

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Abstract

Synchrotron X-ray diffraction technique has been used to estimate defect structure in terms of dislocation density, crystallite size and micro-strain in commercially pure titanium subjected to tension and cyclic deformation in stress and strain control mode. Statistical analysis of micro-texture data collected from electron backscatter diffraction approximately from the same region as that of synchrotron X-ray has been used to correlate orientation dependent micro-strain and dislocation density with deformation microstructure and micro-texture. Two different orientations, namely, A with prismatic-pyramidal and B with basal orientation along the loading axis has been considered. Weak initial texture yet significant anisotropy in hardening/softening response and failure mode for monotonic tension and cyclic loading paths has been observed.

Higher strain hardening response of orientation A during monotonic tensile deformation can be attributed to the evolution of lower micro-strain on basal orientation grains i.e, <0002>∥ND along with extensive multi-variant twinning that also restricts crack propagation and delays failure in stress control mode. On the other hand, in strain control mode, orientation B shows higher fatigue life due to the generation of lower micro-strain in the basal orientation grains and single variant twinning that can undergo detwinning easily is responsible for delayed crack nucleation and subsequent failure.

Keywords: Titanium; Texture; Dislocation density; Synchrotron X-ray; EBSD.

I. Introduction

Macroscopic plastic deformation of materials depends on the mode of loading as well as the strain path followed during deformation. Strain path involving tension-compression, tension-torsion and change in strain rate during quasi-static and dynamic loading shows different deformation behavior in materials. Also, there are different ways in which macroscopic deformation leading to failure in materials occurs for different loading paths [1]. For instance, failure during monotonic tensile loading occurs by necking due to localized stress overload, buckling during compression due to increase in friction force at the contact surface. While failure during torsion test shows no observable phenomenon like necking, rather formation of band of flow localization region leads to failure and hence material shows more ductility before failure during torsion test compared to tensile test. At the sub-microstructural level, the evolution of dislocation density with change in strain path dictates deformation behaviour and contributes to different behavior depending on monotonic, load reversal or cyclic mode of deformation. During monotonic loading, stored dislocation density decreases with increase in strain [2]. While in case of tension-compression cyclic loading, there is multiplication and annihilation of dislocations with overall increase in dislocation density with each cycle and all these affect failure too. The onset of necking in tension corresponds to the saturation dislocation density of statistically stored dislocation that can be stored in a grain and it is rational to assume that the saturation dislocation density can be achieved by large number of cycles leading to failure. However, unlike yielding and necking in tension which can be predicted and are design parameters for any material, failure in tension and more so in cyclic loading is stochastic in nature.

Nevertheless, the role of dislocation interactions is important, and we want to compare dislocation character and fracture features for differently loaded sample. Thus, the present paper is an attempt to bridge the gap in length scale and explain failure as a function of crystallographic texture that determines dislocation and twin activity. It requires characterization of the defect structure generated during deformation leading to failure. Synchrotron X-ray source provides statistical information of defects as well as deformation texture information due to its smaller wavelength [3-4]. Even techniques have been developed to carry out in-situ straining of materials accompanied by deformation texture measurement using neutron and synchrotron X-ray source [5].

X-ray line profile analysis (XRLPA) in conjunction with micro-texture analysis is very useful in investigating orientation dependent dislocation activity resulting in anisotropic deformation response [6]. There are different approaches followed for X-ray line profile analysis such as Variance method, Fourier analysis and Convolutional Multiple Whole Profile (CMWP) fitting method, depending on the microstructure feature dimension of concerned material. For instance, the evolution of dislocation during straining of fcc Cu alloy and Al alloy and bcc high entropy alloy under monotonic tensile and compression mode has been studied using variance approach for XRLPA of Synchrotron X-ray and TEM diffraction data [7-9]. On the other hand, XRLPA using CMWP approach for detailed characterization of dislocation of hcp commercially pure titanium and zirconium alloy subjected to various thermo-mechanical processing such as rolling, equal channel angular pressing etc. has been carried out [10-13]. A specific study on the cyclic deformation behavior has been carried out using Electron channeling contrast imaging (ECCI) images in combination with EBSD based strain indicators in Fe-Si steel [14]. Hence, an extensive literature survey reveals that there is no study reporting characterization of orientation dependent ratcheting or cyclic creep behavior using XRLPA.

Cyclic creep behavior is of immense importance for the purpose of improvement of service life of structural component material. In the past, detailed TEM analysis has been carried out on the cyclic creep behavior of polycrystalline copper explaining the phenomenon of acceleration and deceleration of steady state creep rate occurring due to "strain burst" involving disruption and rearrangement of dislocation cell structure [15]. Such organization of dislocation cell structure of saturated dislocation density occurs in the habit plane along the slip direction, which is responsible for strong anisotropy in fatigue life during ratcheting [16].

However, the materials considered so far have fcc crystal structure and slip is the dominant mode of deformation. Very few studies have been carried out on materials with hcp crystal structure which has also twinning as dominant mode of deformation. Hot rolled magnesium alloy showed in-plane anisotropy in ratcheting response [17]. However, reason behind the anisotropy in mechanical property of hcp system is not well explained in literature. This may be due to twinning phenomenon which occupies almost 50% of parent grain and induces change in orientation of twinned region too. Also, the difficulty in quantifying its effect arises from the numerous types of interactions, including primary twin-matrix interaction, primary twin-secondary twin interaction and most importantly dislocation interactions with all of these types of obstacles. Hence, the role of twinning and its effect on material behavior in polycrystalline titanium is very important which involves more complex interaction compared to magnesium and it has been the subject of most recent researches however, is still not fully understood.

Therefore, in the present paper correlation of defect structure with orientation dependent slip/twin activity during monotonic tension and cyclic loading in commercially pure titanium has been carried out. The statistical information of defect structure obtained from synchrotron X-ray diffraction in transmission mode has been combined with deformation texture information of from electron backscatter diffraction to correlate the observed anisotropy in tensile and cyclic deformation and failure in commercially pure titanium.

2. Experimental

Cold rolled and annealed plate of grade 2 commercially pure titanium was taken for the present investigation. Flat tensile and fatigue test specimen of ASTM standard E 606 were machined with loading axis parallel to rolling direction (RD) and transverse direction (TD) from normal direction (ND) and rolling direction RD plane, respectively. Fatigue tests were carried out in both stress control and strain control mode. In stress control mode, asymmetric stress cycle was applied which is also known as ratcheting test. The specimen dimension and specimen orientation are reported in [18]. Samples from normal plane and rolling plane were referred as A and B orientation respectively and the corresponding sample designation for tensile (T), stress control (R) and strain control (C) were listed in Table 1. Stress amplitude (σ_a=0.8σ_y) and mean stress (σ_m=0.4σ_y) was used for stress control mode and for strain control cyclic loading, strain amplitude of 0.83% was used. Deformation texture was measured in PETRA III@DESY/Hamburg using synchrotron X-ray radiation in transmission geometry at approximately 1mm away from fracture tip. The high energy beamline HEMS P07B with energy of 87 keV corresponding to a wavelength of 0.143155Å was used. In order to obtain sufficient local resolution required for deformation texture measurement 0.3 x 0.3 mm² slit was used. The experimental settings for this instrument required that the samples were cut into 15mm long and 1.5mm×1.5mm (thickness x width) rods. A rotation stage was used to obtain 37 images with PE XRD 1621 type solid state area detector every 5° (+90 to -90˚ span around vertical axes), generating a set of complete Debye-Scherrer rings for each image and allowing to gather information on all orientation planes in the same measurement. Exposure time for a single shot scan (only one image) was 3 sec per image by adding 5 frames and for texture measurement (37 images) fast scans with exposure time of 1 sec per image were carried out in 10 frames. Detector calibration was performed using Al_2O_3 powder NIST standard. Data evaluation includes corrections of primary intensity, absorption and exposed volume followed by pole figure data transfer in correct format using Fit 2D software. The data obtained from the pole figures were used to calculate the complete orientation distribution function (ODF) using Resmat software [19] and complete pole figures was recalculated. Also, the x-ray diffraction data were used for line profile analysis using variance method [20]. At about 1mm away from the fracture tip the specimen surface was electropolished using A3 electrolyte and subjected to micro-texture investigation using a field-emission gun scanning electron microscope (JSM-7100F FE-SEM) fitted with Oxford Channel 5 electron back scatter diffraction (EBSD) attachment. An area of 200 × 200 μm^2 was scanned with a step size of 0.5 μm.

3. Results

3.1 Initial microstructure and texture

Figure 1(a) represents the initial microstructure of cold rolled and annealed plate of commercially pure titanium showing equiaxed alpha grains. The misorientation angle distribution shown in Figure 1(b) shows two peaks at about 30° and 75° corresponding to grains tilted away from ND towards TD in same and opposite direction, respectively. Figure 1(c) shows inverse pole figures (IPF) along loading direction for the two distinctly different orientations A and B considered for tensile and fatigue test. Sample A shows orientations near $<11\bar{2}0>$–$<10\bar{1}0>$ region of the IPF, while sample B shows high-intensity contours near crystallographic direction <0001> indicating clustering of grains near the basal orientation.

3.2 Tensile and cyclic deformation behavior

Figure 2(a) shows true stress vs true strain plot. It shows that yield strength (YS) of sample AT (262 ± 3 MPa) is lower compared to sample BT (298 ± 5 MPa). However, the strain hardening coefficient of sample AT (0.19) is higher compared to sample BT (0.13). Figure 2(b) and Figure 2(c) represents the number of cycles to failure for stress control and strain control specimen respectively. It shows slightly higher fatigue life for AR compared to BR while fatigue life of BC is almost double that of AC. Cyclic creep rate of AR has been found to be lower compared to BR under stress control mode [21]. Whereas in strain control mode, inspite of strong anisotropy in fatigue life, there is very less difference in stress amplitude observed [22].

3.3. Deformation microstructure and texture

The post deformation microstructure obtained for monotonic tension, stress control and strain control cyclic samples are shown in Figure 3. Orientation A shows deformation by formation of thin lamella of contraction twin $\{11\bar{2}2\}<\bar{1}\bar{1}23>$ and secondary extension twin formed within contraction twin in both the sample AT and sample AR. While orientation B shows formation of thick extension twin $\{10\bar{1}2\}<\bar{1}011>$ lamella in both BT and BR. The volume fraction and thickness of twin lamella in AR and BR are less than AT and BT, respectively due to lower maximum applied stress during stress control cyclic test. There are almost no twins in strain control samples AC and BC as they are loaded in the slip dominated regime of the stress- strain curve.

The deformation texture for monotonic tension and stress control cyclic samples are measured using synchrotron X-ray diffraction technique. A representative synchrotron X-ray diffraction intensity peak profile for BR derived from the Debye-Scherrer ring pattern formed on the image plate using the relation ($\theta = R_{hkl}/2L$, where θ is Bragg's diffraction angle, R is ring radius of (hkl) plane and L = 1000 mm is the camera length/source to detector distance) has been shown in Figure 4(a). The presence of varying intensity distribution along each Debye –Scherrer ring indicates presence of texture in the material. IPF plots shown in Figure 4(b) depict rotation of crystallographic direction $< 10\bar{1}0 >$ towards loading axis in sample AT and sample AR. While in sample BT and BR crystallographic direction near $< 11\bar{2}0 > - < 10\bar{1}0 >$ gets aligned along loading axis. Sample AC and BC will also show similar IPF and hence has not been reported here.

3.4 Characterization of defect structure

3.4.1 X-ray line profile analysis

The experimentally obtained peak profile is assumed to be combination of Gaussian (G(2θ)) and Lorentzian (L(2θ)) function and the combination is represented by the pseudo-Voigt function (pV(2θ)) as in Eq. (1).

$$pV = I_0[\mu L + (1-\mu)G] \qquad (1)$$

Where, $L = [1 + \frac{(2\theta - 2\theta_0)^2}{w^2}]^{-1}$; $G = \exp[(-ln2)\frac{(2\theta - 2\theta_0)^2}{w^2}]$

w is full width at half maximum of fitted profile measured in radian unit and μ is the mixing fraction of Gaussian and Lorentzian profile so as to obtain integral breadth β in radian unit.

$$\beta = w(\pi\mu + (1-\mu)\sqrt{\frac{\pi}{ln 2}}).$$

$$k = 2\beta^{-1}\mu w^2$$

The instrumental corrected broadening corresponding to the diffraction peak is estimated as

$k\_net = k\_raw - k\_ref$, Al_20_3 powder used as reference.

The defect parameters crystallite size, micro-strain within the crystallite induced by dislocations and the dislocation density generating micro-strain are estimated using Eq. (2), (3) and (4), respectively.

$$< D >= \frac{0.9\lambda}{\pi^2 \, Cos\theta \, k\_net} \qquad (2)$$

Where <D> is crystallite size (Å), λ (A°) is the wavelength of X -ray radiation, θ (radian) is diffraction angle, k_{net} also in radian.

$$< \varepsilon >= \sqrt{\frac{w\_net\_modified}{4tan^2\theta}} \qquad (3)$$

Where <Ɛ> is micro-strain, $w\_net\_modified = |w\_net + 0.5\pi^2 k\_net * k\_ref|$

$$\rho = \left[\frac{\sqrt{12}<\varepsilon^2>^{\frac{1}{2}}}{<D>b}\right] \qquad (4)$$

Where b is the Burger's vector $\frac{a<11\bar{2}0>}{3}$ and the dislocation density ρ is defined as the length of the dislocation lines per unit volume of the crystal in m^{-2} unit.

It is important to understand the physical significance of crystallite size, micro-strain and dislocation density and their relationship with each other. Plastic deformation is accommodated by motion of dislocations and there is an increase in density of dislocations with increase in strain. Dislocations arrange themselves into low energy dislocation structures like Taylor lattice and cells which are coherently scattering domains also referred as crystallites. Micro-strain is caused by bending of the lattice due to geometrically necessary dislocations which are a fraction (mostly one fifth) of the total dislocation density. Evolution of higher micro-strain can lead to strain incompatibility between grains or sub-grains and lead to void nucleation in monotonic loading. The situation is more complex in cyclic loading with alternating tensile and compressive stress or strain but the evolution of micro-strain is paramount to decide initiation of failure. For hexagonal close packed material like titanium, stacking fault energy is also anisotropic with the stacking fault energy (SFE) of prism plane being lower (150 mJ/m^2) than the basal plane (300 mJ/m^2). This clearly indicates that it is easy for a dislocation to cross slip from basal to prismatic plane but difficult for a dislocation to cross slip from prismatic to basal plane [23]. Hence, it is important to monitor the dislocation density evolution on prismatic as well as basal planes.

The variation of crystallite size, micro-strain and dislocation density for tensile (T), stress control (R) and strain control (C) cyclic samples for basal $\langle 0002 \rangle$ and prism $\langle 10\bar{1}0 \rangle$ planes are plotted in Figure 5. Figure 5(a) shows that crystallite size of basal orientation is higher than prism orientation in both tensile and stress control cyclic samples. The crystallite size of basal orientation in sample AT is lower than sample BT and the corresponding crystallite size is equal for prism orientation. The crystallite size of sample AR is highest for both basal and prism orientation. High crystallite size is an indication for lower lattice distortion. Figure 5(b) shows that micro-strain within crystallite of basal orientation is lower in sample AT than sample BT and the micro-strain is equal for prism orientation. While micro-strain within crystallite of sample AR is higher compared to sample BR for basal orientation and is lower for prism orientation. Higher micro-strain within the crystallite indicates higher lattice deformation induced by dislocation. Figure 5(c) reveals that dislocation density within crystallite of basal orientation is slightly higher in sample AT compared to BT and is equal for prism orientation. In sample AR dislocation density is lower than sample BR. The trend observed in samples AC and BC are similar to that in AR and BR. However, the lower value of crystallite size, micro-strain and higher dislocation density of prism orientation in strain control (C) compared to stress control (R) is due to predominant prism slip activity under imposed strain amplitude in strain control mode, whereas in stress control mode twin activity is dominant.

3.4.2 Electron backscatter diffraction

Crystal orientation map developed selectively for $\langle 0002\rangle \parallel$ND and $\langle 10\bar{1}0\rangle \parallel$ND grain orientation for tensile, stress control and strain control specimens are shown in Figure 6. It shows that for orientation A deformation of grains by twinning occurs in both $\langle 0002\rangle \parallel$ND and $\langle 10\bar{1}0\rangle \parallel$ND grain orientation in both AT and AR. Whereas in case of orientation B it is $\langle 10\bar{1}0\rangle \parallel$ND oriented grains which undergoes twinning in both BT and BR. In orientation A twinning occurs in both the grain and the micro-strain is accommodated by $\langle 0002\rangle \parallel$ND thus provides better stress distribution condition while in orientation B, $\langle 0002\rangle \parallel$ND is hard orientation and micro-strain is accommodated by $\langle 10\bar{1}0\rangle \parallel$ND. Sample AC and BC show only colour gradient within basal and prism orientation indicating slip activity.

The local average misorientation (LAM) and grain reference orientation deviation (GROD) value determined from EBSD data of $\langle 10\bar{1}0\rangle \parallel$ND and $\langle 0002\rangle \parallel$ND orientations are given in Table 2. LAM value for $\langle 0002\rangle \parallel$ND orientation have been found to be higher for AT compared to BT while the LAM value for both $\langle 10\bar{1}0\rangle \parallel$ND and $\langle 0002\rangle \parallel$ND have been found to be higher for BR compared to AR. GROD value of AT is much higher than BT for both the orientations $\langle 10\bar{1}$

0>∥ND and <0002>∥ND while GROD value of <0002>∥ND is lower for AR compared to BR. No significant difference in LAM and GROD value for respective orientations has been observed in case of sample AC and BC. GROD gives the measure of long range (grain level) distortion, while LAM is the measure of short range (pixel level) distortion. Since polycrystal deformation is characterized by strain compatibility between individual grains, high GROD indirectly indicates overall higher deformation of a grain, while high LAM value can be correlated with high average local deformation. The fraction of extension and contraction twins for each loading condition has also been reported in the Table 2.

3.5 Fractographs for failure analysis

Representative fractographs of tensile, stress control and strain control specimen for two different orientations A and B are shown in Figure 7, Figure 8, and Figure 9, respectively. Due to the presence of high stress intensity regions in the microstructure, the fracture surface of tensile specimen shows crack nucleation site. Appreciable amount of ruptured dimples and voids of varying size and shape are observed in the region of overload. Fracture surface is more fibrous in orientation A indicative of ductile failure. A small yet distinct region of crack initiation and early microscopic crack growth is observed in fractographs of stress control samples AR and BR. The crack propagation region has been found to be larger in AR compared to BR indicative of its higher fatigue life compared to BR. Also, the region within the stable crack propagation of sample AR shows presence of striations or “furrow” marks indicating better resistance to crack propagation and delayed failure [24]. The fast crack growth region consists of granular fracture feature in sample AR and is relatively smooth in case of BR referring to less resistance to failure in BR. Comparing fracture surface of sample AC and BC indicates that sample BC has larger crack initiation and stable crack growth region compared to AC indicating delayed crack initiation and hence higher fatigue life of BC. Both AC and BC show presence of shallow striations in the region of stable crack growth.

4. Discussion

Considering initial TD-split basal texture of commercially pure titanium as depicted from Figure 1, it is clear that orientation A has high Schmid factor for prismatic slip system and orientation B has high Schmid factor for basal slip system with respect to the loading axis. However, basal slip has higher critical resolved stress compared to prismatic slip in titanium due to less than ideal c/a ratio. Therefore, resolved shear stress on prismatic plane is higher in orientation A compared to orientation B, which can activate prism slip at lower applied stress leading to lower yield strength in orientation A compared to orientation B during monotonic tensile loading. Beyond yield point, twinning operates at moderate strain followed with pyramidal and basal activity at larger strain. The basal activity being higher in orientation A compared to orientation B as shown by Sinha et al. [18] using Elastic plastic self consistent (EPSC) simulation is one of the reason for higher strain hardening and ductility of orientation A compared to orientation B. The other reason is formation of thin multi variant contraction twin during plastic deformation of AT [25]. Strain control (C) and stress control (R) cyclic behavior is basically governed by the active deformation modes at low and high strain respectively. Hence, stress control sample shows analogous anisotropy in deformation behavior as that of tensile samples i.e, higher strain hardening and ratcheting life of orientation A while poor fatigue life under strain control mode. The underlying micro-mechanism for such anisotropic deformation and failure behavior will be discussed in the following sections.

4.1 Anisotropy in defect parameter and mis-orientation parameter

In titanium, deformation proceeds via <a> type prismatic slip, <a> type basal slip, $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$contraction twin, $\{10\bar{1}2\}\langle\bar{1}011\rangle$extension twins and <c+a> type pyramidal slip. The sample with prismatic-pyramidal texture deforms mainly by slip mode followed by twinning at latter stage. During deformation by slip sub-grain boundaries are formed which are the store house of dislocations. Higher the number of sub-grain boundaries smaller will be the crystallite size causing more broadening of diffraction peaks indicating more deformation. Therefore, orientation A shows lower crystallite size in tension and stress control cycle. The observed higher crystallite size of basal orientation in both tensile and stress control samples from Figure 5(a) is due to deformation by twin mode mainly which is also evident from crystal orientation map shown in Figure 6(a-b) and Figure 6(d-e). So, the orientation dependent anisotropy in crystallite size is because of relative twinning propensity in both basal and prism orientation in AT and preferentially in prism orientation in BT due to its unfavorable orientation for slip with respect to the loading axis.

Higher micro-strain within the crystallite indicates higher lattice deformation induced by dislocations. Therefore, higher micro-strain and dislocation density generation within the crystallite of prism and basal orientation is obvious due to favorable prism slip activity in orientation A, causing higher strain hardening in AT. While, higher micro-strain from lower dislocation density observed in basal orientation in BT is responsible for lower strain hardening observed in BT during tensile deformation shown in Figure 2(a). The low value of GROD reported in Table 2 also indicates less overall deformation of grains with basal orientation due to low dislocation density within basal orientation of BT. However, during stress controlled cyclic loading lower micro-strain occurs in prism orientation from higher dislocation density, which is responsible for slow creep rate in AR compared to BR leading to higher ratcheting life in AR compared to BR. The lower LAM value of AR compared to BR also confirms the same fact.

In strain control samples AC and BC, due to deformation in slip active zone, it shows high dislocation density, low crystallite size and high micro-strain within the crystallite. This is also reflected in very low GROD values given in Table 2 indicating predominant intra-granular distortion only. The increase in dislocation density for strain control samples compared to stress control sample has also been observed in AISI 316L [15] and pure copper [16].

4.2. Role of slip dislocation and twins in failure

At dislocation level, failure occurs due to saturation of stored dislocation density for slip or exhaustion of dislocation induced twinning phenomenon in individual grains [2]. The dislocation density accumulated in AT and BT at ultimate tensile strength (UTS) may be considered as the saturation dislocation density of statistically stored dislocation in individual grains. Although the deformation behavior of tensile and stress control samples are same and the stress control conditions are much below the UTS, failure in stress control samples occur at much lower total dislocation density compared to tensile sample, as observed from Figure 5(c). While in AC and BC, which are loaded under strain control mode failure occurs only after cumulative accumulation of dislocations in each cycle almost equal to the saturated dislocation density of tensile samples is reached. This may be due to the applied non-zero mean stress in stress control loading which reduces the stored dislocation density of individual grains, and hence, leads to much lower fatigue life compared to strain control fatigue samples [21]. This early saturation of dislocation density in AR and BR has been attributed to the formation of well organized dislocation cell structure during stress controlled cyclic loading as described in case of SS 316 [26].

The formation of thin twins and their intersection as observed from Figure 3, contribute to higher strain hardening and higher percent elongation in AT, while deformation by lateral thickening of twins in basal orientation causes lower strain hardening and lower percent elongation in BT. The difference in strain hardening is attributed not only to different twin morphology but also to multiple variant and single variant nature in AT and BT, respectively [25]. The observed twin feature is also responsible for higher fatigue life in AR compared to BR due to better local stress relaxation in the former sample [21]. However, the situation gets reversed in case of strain control sample i.e, BC showing higher fatigue life compared to AC. The corresponding microstructure for AC and BC do not show twins as they are taken from region away from fracture tip and their presence is evident from region closer to fracture tip as shown by Sinha et al [22]. The presence of multiple twin variant in AC has less detwinning tendency in low cycle fatigue and increases local backstress leading to lower fatigue life. On the other hand, single variant twin in basal orientation causes easy detwinning and higher low cycle fatigue life in BC. The phenomenon of detwinning during load reversal has been shown using in-situ EBSD experiment [22]. The role of slip/twin activity and its effect on failure mechanism in different load path can be summerised in Figure 10.

4.3 Failure mechanism in monotonic tension and cyclic loading

Fractographic analysis reveals that failure during monotonic tensile and stress control cycle occurs via similar mechanism with very small crack initiation stage followed by rapid crack propagation along transgranular path as observed from Figure 7. On the other hand, during fatigue there is formation of large crack initiation site and stable crack growth region Figure 8 and Figure 9. In hcp metals, fracture features depend highly on the grain orientation due to different dislocation structure leading to anisotropic properties [27]. AR shows granular fracture feature in the fast crack growth region. This is due to weak basal texture, grains of different orientations are prevalent along plane normal. Thus, the propagating crack faces resistance to follow transgranular path throughout the microstructure, and it is forced to follow intergranular path for neighbouring hard grains with different orientation [28]. For the same texture characteristic in BR, most grains are with prism plane along normal direction, resulting in easy crack propagation along transgranular path through coalescence of micro-voids mechanism which produces fibrous fracture feature.

Strain control mode has been identified as low strain deformation. In orientation A, strain hardening occurs with increase in number of cycles. This leads to easy crack nucleation in softer grains with preferential prism slip orientation and the stress intensity at the crack tip increases with increasing in hardening of the surrounding matrix with increase in cycle number leading to rapid propagation of crack, resulting in shorter fatigue life of AC. While in sample BC harder prism slip orientation causes delayed crack nucleation and the propagation of nucleated crack is also slow due to less hardening of surrounding matrix with increase in cycle number thus showing longer fatigue life.

V. Conclusions

The dislocation induced micro-strain generation within crystallite influences both tensile and cyclic deformation behavior in commercially pure titanium. Tensile and stress control cyclic behavior are governed by slip and twinning activity in high strain regime while strain control cyclic behavior is governed by slip activity in low strain regime in commercially pure titanium. Thus, the observed anisotropy in tensile and cyclic deformation and failure behavior has been interpreted from the orientation dependent dislocation activity as follows.

- The observed anisotropy in tensile behavior gets subdued in stress control cyclic deformation while it gets amplified in strain control cyclic deformation.
- Evolution of comparable micro-strain with dislocation density in basal orientation grains i.e, $\langle 0002\rangle \parallel ND$ along with extensive multi-variant twin formation causes higher strain hardening in orientation A (AT) compared to orientation B (BT).
- Lower micro-strain generation in prism orientation grains i.e, $\langle 10\bar{1}0\rangle \parallel ND$ due to mean stress effect causes low steady state creep and hence higher fatigue life in orientation A (AR) compared to orientation B (BR) during stress control cyclic loading.
- Higher micro-strain but lower dislocation density generated in basal orientation grain in orientation A (AC) causes early crack nucleation and lower its fatigue life compared to orientation B (BC) in strain control cyclic loading.

Acknowledgement

The authors would like to thank Department of Science and Technology for funding through INSPIRE faculty fellowship of Dr. Atasi Ghosh (research grant no. DST/INSPIRE/04/2016/001217). The authors would also like to thank Department of Science and Technology, Government of India and the synchrotron X-ray facility in DESY (Deutsches Elektronen-Synchrotron) lab at Hamburg, Germany for providing access to synchrotron through DST-DESY project. NPG thanks funding through Indian National Science Academy project for Young Scientist to carry out this work. The authors thank Ms. Reshma Sonkusare, Mr. Mrityunjay Kumar and Mr. Vivek Kumar Sahoo, research scholars at IIT Kanpur for their help in experimental data collection and analysis.

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Table

Table 1. Sample designation of tensile, stress control and strain control samples for A and B orientation.

Orientation	Tensile	Stress control	Strain control
A	AT	AR	AC
B	BT	BR	BC

Table 2. Grain and twin boundary characteristic of <10$\bar{1}$0> and <0002> orientation.

Sample	LAM (°)		GROD (°)		Extension twin (%)	Contraction twin (%)
	<10$\bar{1}$0>	<0002>	<10$\bar{1}$0>	<0002>		
AT	0.58	0.61	4.06	4.45	9.4	18.5
BT	0.54	0.49	1.83	1.99	9.0	6.3
AR	0.59	0.56	3.44	3.49	0.89	4.84
BR	0.76	0.86	4.26	3.79	4.39	2.04
AC	0.54	0.49	0.45	0.85	0.02	0.012
BC	0.51	0.58	0.60	0.49	0.01	0.015

Figure captions

Figure 1: (a) Initial grain microstructure of cold rolled and annealed CP-Titanium, (b) Misorientation angle distribution and (c) Inverse pole figure for orientation A and orientation B along loading axis.

Figure 2: (a) True stress vs true strain plot and number of cycles to failure for (b) stress control cycle (c) strain control cycle samples.

Figure 3: Deformation microstructure delineated with contraction and extension twin boundary after (a) tensile, (b) stress control and (c) strain control cyclic loading.

Figure 4: (a) Representative synchrotron diffraction intensity peak profile of BR sample derived from the Debye-Scherrer ring pattern (inset) using Fit2D software and (b) Inverse pole figure contour plots along loading axis for orientation A and orientation B.

Figure 5: Variation of (a) crystallite size, (b) micro-strain, and (c) Dislocation density for tensile (T), stress control (R) and strain control (C) samples.

Figure 6: Crystal orientation map of $\langle 0002\rangle \parallel$ND and $\langle 10\bar{1}0\rangle \parallel$ND grain orientation for (a) AT, (b) AR, (c) AC, (d) BT, (e) BR, and (f) BC.

Figure 7: Scanning electron micrograph of fracture surface (magnified view of region within yellow dotted circle) showing presence of microvoids in AT and fibrous fracture surface BT.

Figure 8: Scanning electron micrograph of fracture surface (magnified view of region inside (middle) and outside (right) the blue dotted curve) showing granular feature in AR and smooth fracture feature in AC.

Figure 9: Scanning electron micrograph of fracture surface (magnified view of region inside (middle) and outside (right) the blue dotted line) showing presence of ductile fracture feature in BR and striation marks within fatigue zone of BC.

Figure 10: Schematic representation of slip/twin activity in basal plane for crystal orientation with prismatic-pyramidal loading axis under different loading path.