Texture strengthening and anisotropic hardening of mill annealed Ti-6Al-4V alloy under equi-biaxial tension

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ABSTRACT

This work investigates the influence of equi-biaxial stress state on the deformation response of Ti-6Al-4V alloy in mill-annealed condition using a newly designed cruciform specimen geometry. The proposed geometry was validated using a non-contact strain measurement technique to obtain maximum plastic strain in the gage section. It is observed that the elasto-plastic response of Ti-6Al-4V alloy was profoundly influenced by the stress state and the loading orientation with respect to the rolling direction. The strengthening effect and the in-plane plastic anisotropic evolution observed under equi-biaxial stress state was ascribed to the presence of strong crystallographic texture in the as-received condition. The dominant deformation mechanisms operating under equi-biaxial stress state were deduced from the texture evolution studies.

1. Introduction

Titanium alloys are widely used in aerospace, marine, automobile and biomedical applications due to its high specific strength, moderate ductility, good fatigue strength, and corrosion resistance properties [1–3]. Among all the grades, Ti-6Al-4V alloy evinces interest, since a broad range of mechanical properties can be achieved by fine-tuning the degree and mode of deformation, annealing temperature and cooling rate during hot working and heat treatment respectively [4]. Furthermore, Ti-6Al-4V alloy has already replaced high strength steels, in several critical aerospace applications due to its superior performance coupled with minimal maintenance [3]. Some of the aerospace applications of Ti-6Al-4V alloy include compressor disks, blades in aircraft engines, forged rotor heads in helicopters, pressure vessels, storage tanks, and hydraulic tubings in space launch vehicles respectively [3].

In a conventional design approach, these structural components are designed based on the uniaxial mechanical properties alone, leading to an overly conservative structure. However, several components, for instance, spherical gas bottles, experience complex biaxial stress state during its service condition. Ti-6Al-4V alloy is widely used for the fabrication of gas bottles which store liquid helium for pressurizing liquid propellant in space launch vehicles. These spherical gas bottles are primarily produced by hot forming and electron beam welding of hemispherical domes, which experience the biaxial stress state, under pressurized condition [5]. Hence, it is essential to understand the deformation behavior of such materials under biaxial stress state, for the safe and reliable operations during its intended service. Moreover, the conventional design approach may not be sufficient for titanium-based hexagonal materials which are prone to develop crystallographic texture during processing and deformation [6]. In addition, the experimental data under the biaxial stress state can be utilized for improving the prediction capability and validation of various constitutive models proposed earlier to describe the deformation behavior as a function of stress state, strain rate and temperature respectively [7–13]. Furthermore, the advanced yield functions proposed in recent times which take tension-compression asymmetry into consideration requires yield data under equi-biaxial stress state for its calibration, and hence it is essential to test lower symmetric hexagonal materials under such complex stress states [14,15].

Several biaxial testing methodologies were utilized for evaluating the material properties under biaxial stress state such as the combination of axial compression/tension with internal pressure [16], hydraulic/viscous bulging technique [17], Stretch forming [18] and cruciform specimen technique [19]. Albeit the popularity of the hydraulic bulging technique for obtaining biaxial mechanical properties, this method suffers from certain inherent drawbacks such as (i) thickness gradient along the bulged portion of the sheet (ii) through-thickness stress gradient and (iii) presence of out-of-plane bending. On the contrary, the cruciform biaxial technique offers the possibility of testing materials
under in-plane configuration under any arbitrarily chosen load ratios along X- and Y- directions [19]. However, one of the drawbacks of the cruciform testing method is the non-availability of a universally accepted standard to study the elasto-plastic characteristics of the test material until fracture. The specimen geometry proposed by Deng et al., [20] was primarily designed for the construction of yield loci under various biaxial loading ratios, but not suitable for probing the material’s response until fracture. This was attributed to strain concentration build-up outside the gage section of the cruciform specimen such as cross-arm fillets (intersection of arms) and slit ends, respectively [21]. This precludes the possibility of capturing the complete plastic strain response inside the gage section under biaxial stress state. Hence, newer geometries were designed, with an intention to obtain the highest plastic strain in the central gage section of the cruciform specimen [22].

The design criteria for a valid cruciform specimen geometry are as follows (i) homogenous strain distribution in the gage section, (ii) failure initiation in the gage section, (iii) minimum shear strain in the gage section and (iv) minimum strain concentration outside the gage section.

In order to obtain a valid cruciform specimen geometry, finite element procedures were carried out by several researchers to obtain maximum homogenous stress/strain distribution in the gage section, such that failure occurs in the gage region [20–22]. Furthermore, to reduce the machining cost and the complexity of the design, asymmetric/one-sided cruciform specimen geometry in the thickness direction with a circular gage section was proposed to study the plastic characteristics of materials under biaxial stress state [23]. However, such asymmetric geometries may suffer from shear and bending stresses in the gage section of the cruciform specimen [21]. Zidane et al., [24] proposed a symmetric cruciform specimen geometry with two-step thickness reduction in order to obtain large plastic strain in the central gage section of the cruciform specimen. This specimen has been used extensively to obtain forming limits diagrams (FLD) under various complex strain states [24].

In the present work, modified specimen geometry with a square gage section, optimized using finite element simulations is proposed for 8 mm thick plates. Furthermore, the proposed geometry is validated experimentally for testing materials under biaxial stress state until fracture using non-contact strain measurement technique vis-à-vis strain homogeneity and attainment of maximum plastic strain in the gage section.

Biaxial deformation is comprehensively studied for other hcp materials such as magnesium and zirconium alloys [25,26], however, minimal information is available for titanium alloys. The biaxial yielding and hardening behavior of Ti-6Al-4V alloy at elevated temperatures using cruciform specimen geometry was reported by Merklein et al., [27], however, the microstructural and textural evolution upon deformation was not studied. It is worthwhile to mention that the texture evolution upon cold rolling/warm rolling [28,29], uniaxial hot compression [30], fatigue loading in the air and corrosive environment [6,31] and other severe plastic deformation [32] were well documented. However, studies concerning texture evolution upon equi-biaxial deformation of Ti-6Al-4V alloy have received little attention. Furthermore, to the best of our knowledge, the biaxial mechanical properties of Ti-6Al-4V obtained through cruciform specimen technique, and the influence of initial texture on biaxial mechanical properties has not been reported in the literature. Hence, the main objective of this research work is to study the microstructural and textural evolution of Ti-6Al-4V alloy under equi-biaxial stress state and its biaxial deformation response is reported using cruciform specimen geometry.

A stand-alone biaxial test rig of 250 kN capacity is designed to test high strength Ti-6Al-4V alloy until fracture at room temperature under equi-biaxial tension stress state. Ti-6Al-4V alloy, herein will be referred to as Ti64, is subjected to microstructural and textural characterization prior to testing. Quasi-static uniaxial tensile stress-strain responses at various tensile orientations and the subsequent texture evolution upon deformation are studied. A new cruciform specimen geometry is designed and proposed to obtain reliable data under equi-biaxial stress state. The non-contact strain measurement technique is utilized to capture the strain distribution within the gage section of the dog bone and cruciform specimens respectively. The obtained biaxial stress-strain response of Ti64 alloy is compared and contrasted with respect to uniaxial counterparts. The Knoop hardness yield loci constructed from the hardness measurements are compared with the biaxial yield data and the efficacy of the Knoop measurements in constructing the yield loci are outlined. The deformation mechanisms operating under equi-biaxial stress state are deduced from the observed texture transitions upon biaxial deformation.

2. Materials and Methods

2.1. As-received material

The Ti64 alloy used in the present investigation was supplied by Vikram Sarabhai Space Center, Trivandrum (India) in the form of a rolled plate. The alloy was heated to a temperature of 730 °C (α + β phase field) and soaked for 2 h, followed by furnace cooling until 565 °C and then subsequently cooled to room temperature in an argon atmosphere to obtain mill-annealed bi-modal microstructure.

2.2. Uniaxial and biaxial tensile testing

Uniaxial flat tensile specimens were prepared as per the ASTM E8 standard with the gage section length and width of 31.0 ± 0.1 and 6.6 ± 0.01 mm, respectively and the thickness of each specimen is around 2.5 mm. Specimens along three different orientations with a tensile axis parallel to the rolling direction (RD), transverse direction (TD) and 45° to RD (DD) were machined by electric-discharge machine (EDM) from the as-received 8 mm thick stock plates. The as-machined specimens were polished with SiC emery sheets to remove EDM affected surface layer prior to testing. Testing was carried out at room temperature at an initial strain rate of 3 × 10^-4 s^-1 (quasi-static) in an Instron 25 kN universal testing machine. Non-contact digital image correlation technique (DIC) was used to capture the spatial distribution of longitudinal and transverse strains (strain contour plots) in the gage section of test specimens. From the strain contour plots, the strain averaged over the entire gage section were extracted and used for plotting stress-strain curves at various tensile orientations with respect to the rolling direction. In addition, strain gages were pasted in the gage section of test specimens for the accurate estimation of the elastic modulus and Poisson’s ratio, respectively. Furthermore, strain hardening exponent (n) and the plastic anisotropic ratio (R-value) were calculated from the true plastic stress-strain curves in the strain range of 0.01 to 0.05, respectively. The range is chosen such that, the true-stress and true-strain pairs lies between the yield point and the maximum ultimate tensile point in the stress-strain curve. Elastic strains were subtracted from the total plastic strain, prior to the calculation of the strain hardening exponent and the plastic anisotropic ratio as per the recommendations of ASTM standard E646 and E517 respectively.

Equi-biaxial tensile testing was carried out using a cruciform specimen (250 × 250 × 8 mm) and the geometry was optimized using numerical simulations to obtain maximum homogenous stress distribution in the gage section. The cruciform specimen with a two-step thickness reduction with square gage section (Fig.1.a), revealing homogenous stress distribution in the gage section, predicted using commercially available FEM package is shown in Fig.1b. In-house designed biaxial test rig of 250 kN capacity, equipped with four actuators (two per loading direction) was utilized to apply the desired biaxial tensile load along X- and Y- directions at a loading rate of 0.2 kN/s until failure. Four load cells (220 kN capacity, HBM, Germany) were placed in contact with the actuator to monitor and control the applied forces in X- and Y- directions. The cruciform specimens were fabricated by computer-numeric controlled (CNC) machining (Fig. 2), and the
Fig. 1. (a) Gage section of the cruciform specimen with two-step thickness reduction (b) stress distribution within the gage section predicted using FEM package.

Fig. 2. Biaxial cruciform specimen (a) Top view and (b) zoomed front view of the section X-X (dimensions shown in mm).
dimensional inspection was carried out to ensure uniformity in thickness at the gage section to prevent strain localization due to geometrical discontinuities. The X- and Y-loading directions in biaxial tensile tests corresponds to the rolling direction (RD) and transverse direction (TD) of the as-received plate.

Prior to testing, a stochastic isotropic speckle pattern was applied to the specimen surface using black and white aerosol spray paint for capturing strain distribution in the gage section of the cruciform test specimen using digital image correlation technique. A CCD camera (Point grey solutions, Canada) having 5 MP resolution and 15 frames per second (fps) capability was employed to capture the images of the spray-coated specimen surface during loading. LED lamps were used for the sample illumination and using the high-resolution lens (Edmund Optics, Singapore) the images of the gage section of the cruciform specimen (25 × 25 mm) were captured.

VIC 2D image correlation software (Correlated Solutions, Inc., USA) was used to obtain strain contour plots by correlating the image of the undeformed specimen with the images of the deformed specimen captured during loading. In addition, strain gages were pasted (gage factor - 2.12, TML strain gages, Japan) along the loading directions for the precise quantification of elastic strains under biaxial loading. Load cell and strain gage reading were recorded using Vishay micro measurements data acquisition system (USA) at a scanning rate of 5 data points per second until failure. A minimum of three specimens were tested under each condition, and representative stress-strain responses are presented. Experimental test results elucidating the validation of DIC strain measurements and the equi-biaxial tensile loading is included in the supplementary document.

2.3. Structural characterization of as-received and deformed specimens

The microstructures of the as-received and deformed specimens were examined using optical and scanning electron microscopy studies. Samples were metallographically polished with progressively finer grades of emery sheets, followed by fine diamond polishing (0.25 μm) to obtain mirror-finish surface condition, which is devoid of any scratches. Prior to optical and scanning electron microscopic investigations, the specimen surfaces were etched using a freshly prepared Kroll’s reagent (3 ml HF, 6 ml HNO₃ in 91 ml of water) to resolve the microstructural constituents. Transmission electron microscopy (TEM) was carried out using CM 12 120 kV (Philips) electron microscope to study the microstructural features of as-received samples. Prior to analysis, twin-jet electro-polishing (Struers, Tenupol) was carried out to achieve an electron transparent region in 80 μm thin lamella prepared by mechanical grinding using SiC sheets. The area fractions of all the microstructural constituents were evaluated from the representative optical and transmission electron micrographs using image analysis software. The fractographic analysis was carried out using Quanta 400 (USA) scanning electron microscope to understand the influence of stress state on macro/microscopic fracture modes.

X-ray macro-texture was measured on the RD-TD plane of as-received and deformed specimens using the Schulz back reflection technique. The diffracted intensities were collected continuously during the measurement scan when the sample is being rotated about the normal axis from 0° to 355° for various tilt angles (χ) ranging from 0° to 70° with a step size of about 5°. The crystal structure parameters (a = 2.9508 Å and c = 4.6855 Å) corresponding to the alpha-titanium hexagonal unit cell was used for the texture evaluation. The pole figures of the as-received and deformed specimens are represented in pole figures using sample frame of references such as RD, TD and ND respectively. The RD & TD corresponds to the rolling and the transverse directions whereas; the ND direction corresponds to the through-thickness direction of the plate. The orientation distribution function (ODF) was computed from the six experimentally measured incomplete pole figures (corresponding to alpha phase) such as (0002), (10T1), {10f2}, (10f2), {1120} and {10f3} for the quantitative estimation of texture components. Labotex 3.0 was used for the calculation of orientation distribution function and from the calculated orientation distribution function (ODF), complete pole figures were recalculated.

2.4. Knoop hardness yield loci (KHN) construction

The Knoop indentation hardness were measured by applying 300 g load on three orthogonal planes with indenter major axis parallel and perpendicular to the RD, TD, and ND of the sample-coordinate system [33]. All the samples were metallographically polished to obtain surface planarity, prior to hardness measurements to obtain impressions with a ratio of long diagonal to the short diagonal of 7:1 respectively. 20 indentations were performed on each plane, and the average value was used for the construction of the yield loci. The best-fit ellipse was drawn through six experimental points using least square regression analysis for the construction of Knoop hardness yield loci (KHN) in 2D stress-space. The calculation of stresses along the X- and Y-direction, from the Knoop hardness measurements is elaborated in the supplementary document. Each experimental point used for the construction of the Knoop hardness yield loci were measured within the standard deviation of 50 MPa.

3. Results

3.1. Microstructural characterization of as-received rolled plates

The mill annealed microstructure consists of equiaxed (globularized) primary alpha of 15 μm in size, homogeneously distributed in the
transformed beta matrix as shown in Fig. 3a. Transformed beta is lamellar in nature, containing alpha (hcp) and beta (bcc) platelets of thickness 2.0 and 0.3 μm, respectively with a compositionally gradient α/β interface as evident from the representative TEM micrograph (Fig. 3b). In other words, the alpha phase exists in two-different morphological forms such as the equi-axed morphology and lamellar morphology, respectively. The mean area fractions of equi-axed alpha, lamellar alpha and beta phases are 36%, 55% and 9% respectively, with a standard deviation of about 1%. Such a bi-modal microstructure offered an optimal combination of strength and toughness in contrast to acicular martensitic alpha (distorted hcp structure) which forms at much higher cooling rates (10^4 to 10^6 K/s) through diffusionless phase transformation [34,35].

3.2. Uniaxial stress-strain response as a function of orientation

The uniaxial stress-strain response for various tensile orientations with respect to the rolling direction revealed marked anisotropy in yield strength, tensile strength, failure strain, and modulus as shown in Table 1 and Fig. 4. The yield strength corresponding to 0.2% offset strain, and its corresponding specific work (W) was calculated for the uniaxially tested tensile samples oriented along the rolling direction. The specific work is nothing but the area under the stress-strain curve until the onset of the plastic deformation, corresponding to that strain of 0.002 respectively. Subsequently, yield strength for other orientational strains corresponding to this equivalent work (W = 7.12 MPa) was computed on the basis of the plastic work equivalence principle.

Higher yield strength and ultimate tensile strength is observed for TD oriented samples in contrast to other tensile orientations. This was attributed to a weak transverse crystallographic texture component developed during processing and heat treatment, which is discussed at depth in the subsequent sections. The DIC technique was used to capture the spatial strain distribution along the loading direction and transverse to the loading direction (planar directions) within the gage section of the specimen. The strain in the thickness direction was calculated by assuming the constancy of volume condition and the plastic anisotropic parameter (R-value) was evaluated from the ratio of width strain to the thickness strain. It is worthwhile to mention that, higher R-value is observed for 45° oriented samples (DD) with concomitant higher elongation in that direction, in contrast to RD and TD oriented samples respectively (Table 1).

3.3. Uniaxial testing on biaxial cruciform specimens

The maximum attainable plastic strain in the gage section of the cruciform specimen is limited in previous geometries, due to premature failure outside the gage section such as cross-arm fillet and slit ends [21]. However, the present geometry is designed in such a way that, the failure occurs inside the gage portion of the cruciform geometry. For the experimental verification, the cruciform specimen was subjected to uniaxial loading (only one pair of the actuator was engaged), and the corresponding force-strain responses along the loading direction were extracted until failure. The failure strain (averaged over the entire gage section) extracted from the cruciform specimen, subjected to uniaxial loading closely matches with the failure strain obtained from the standard uniaxial tensile test specimens (Fig. 4 and 5a). Furthermore, the failure is initiated in the gage section, as shown in Fig. 5b (strain contour plot along the loading direction) and the corresponding photo-image of the failed specimen is shown as an inset in Fig. 5a. Hence, this geometry can be utilized for studying the equi-biaxial deformation behavior of Ti64 alloy until failure, without generating strain concentrations outside the gage section of the cruciform specimen.

The shear strain observed in the gage section was 0.0001, thus satisfying one of the design criteria of the valid cruciform geometry. The minimal shear strain is always beneficial in achieving the desired stress state in the gage section of the cruciform specimen. The experimentally measured ratio of transverse strain to the loading direction strain (−\( \varepsilon_{xx}/\varepsilon_{yy} \)) within the elastic regime, averaged over the entire gage section, upon uniaxial deformation of the cruciform specimen was around 0.35. This calculated strain ratio is slightly higher than the nominal Poisson’s ratio of this material, indicating that the portion of the applied load bypasses the gage section, resulting in load coupling between the adjacent arms of the cruciform specimen. In other words, the secondary stress component in the direction perpendicular to the principal loading direction was developed due to load sharing between the arms.

Nevertheless, the obtained stress state in the present geometry is nearly closer to the uniaxial stress state, justifying the uniqueness of the geometry in contrast to the previously proposed cruciform geometries [36,37]. Furthermore, it must be emphasized here that, from the uniaxial tensile tests on cruciform geometry, the load-bearing area of the cruciform specimen was calculated using the effective cross-sectional area method [38]. This method is used for the calculation of biaxial stresses, which is discussed briefly in the subsequent section.

3.4. Equi-biaxial tensile testing

Biaxial tensile tests (with two pairs of actuators engaged) were carried out using cruciform specimen geometry and the load curves along the two axes of the cruciform specimen for an equi-biaxial tensile test with the rolling direction along the X-axis is shown in Fig. 5a. The in-plane strains (averaged over the gage section) along the X- (RD) and Y- (TD) directions were extracted from the normal strain contour plots until fracture. However, for the sake of brevity, only principal strain

![Fig. 4. Uniaxial stress-strain responses as a function of tensile orientation to rolling direction (RD).](image-url)
contour plots corresponding to discrete load steps is shown in Fig. 6. During the elastic regime, homogenous strain distribution was observed, however beyond yielding strain builds up (as high as 0.17) in one of the corners of the gage section and eventually led to failure. The photograph of the failed cruciform specimen under equi-biaxial stress state is shown as an inset in Fig. 7a and its corresponding principal strain contour plot of the same specimen just prior to failure is shown in Fig. 6d. Though the crack propagation slightly deviated from the ideal 45° orientation with respect to the loading directions, failure occurred in the gage section.

Biaxial stress values along X- (RD) and Y- (TD) loading directions were calculated from the externally applied load, up to the small plastic strain of 0.01, using an effective cross-sectional area of 120.94 mm² [38]. However, to obtain biaxial stresses up to failure, inverse anisotropic FEM simulations are necessary for the accurate estimation of the biaxial stresses [38]. This is because, the change in apparent material constants during plastic deformation under the biaxial stress state affects the accuracy of calculation of effective cross-section for large plastic strain range. An increase in yield strength of about 19% (along RD) and 13% (along TD), is observed under equi-biaxial tension when compared to uniaxial properties along the RD and TD directions, respectively (Fig. 7a). Furthermore, severe reduction in ductility is observed under equi-biaxial loading condition as shown in Fig. 5a.

Strain gages were used to capture the elastic strain and the slope measured from the elastic portion of the biaxial stress-strain response is in good agreement with the calculated effective modulus using Eq. (1).

\[ E' = E/(1 - \nu) \]  

(1)

where \( E' \) is the effective modulus (slope of the stress-strain curve under biaxial condition).

The biaxial strain ratio (\( \tau \)), which is defined as the ratio of strain along the X- and Y- loading directions is 1.22, corroborating the anisotropic response of Ti64 alloy under equi-biaxial stress state. Furthermore, the biaxial strain ratio (\( \tau \)) monotonically increases until fracture, and the sharp increase in the final stage, prior to fracture is attributed to strain localization, as shown as an inset in Fig. 7b.

3.5. Texture evolution

Due to higher symmetry of the basal plane, (0002), X-ray pole figures corresponding to the alpha phase of the Ti64 alloy were plotted to reflect the crystallographic texture present in the material. The as-received material displayed strong basal (RD-split) and a weak transverse texture (c-axis parallel to TD) component as shown in Fig. 8a. The c-axis of the basal poles were tilted, with maxima occurring at ±20° away from the normal direction (ND) towards RD, whereas, in ideal basal texture, the c-axis of the basal plane perfectly coincides with the normal direction of the plate. The maximum pole intensity is 8.6 for the as-received Ti64 plates in rolled and mill-annealed condition.

Irrespective of the loading orientation, uniaxially failed samples did not reveal significant textural changes upon deformation (Figs. 8b and c). For uniaxially deformed samples along the diagonal direction, texture components were rotated by 45° along the ND direction, since the samples are extracted and tested along the 45° to the rolling direction. However, for biaxially deformed samples, dramatic texture evolution was observed in contrast to uniaxial condition with a significant reduction in basal pole intensity, as shown in Fig. 8d. New texture components were developed along the rim of the (0002)$_\alpha$ pole figure for the biaxially deformed samples.

4. Discussion

In α + β titanium alloys, the microstructure and texture play a dominant role in influencing the material properties and its performance during its service [6]. The Ti64 alloy investigated in the current study consists of equiaxed primary alpha dispersed in the transformed beta matrix, which is lamellar in nature. The lamellar structure offers hindrance to crack propagation resulting in improved fracture toughness, whereas, the equiaxed alpha possesses superior hardness, elastic modulus and good ductility in contrast to platelet/lamellar alpha [34,39]. Thus, through bimodal microstructure consisting of two different morphologies of hexagonal alpha (Fig. 3), the optimal combination of strength, ductility and fracture toughness is obtained. Furthermore, the degree and the type of crystallographic texture largely govern the deformation behavior and the concomitant plastic anisotropic mechanical properties [33].

The as-cast material normally shows weak texture upon cooling from the β-phase field. This is because, 12 crystallographic variants of alpha phase (hcp) is possible from one single orientation of beta (bcc)
Fig. 6. Strain evolution in gage section of the cruciform specimen at various load steps (a) 120 kN (b) 141 kN (c) 160 kN (d) 172 kN (First principal strain contour plots).

Fig. 7. (a) Biaxial stress-strain response up to plastic strain of 0.01 (inset image: failed cruciform specimen); (b) Influence of stress state on the elastic behavior of Ti64 alloy (inset image: biaxial strain ratio evolution until fracture).
upon transformation during cooling, resulting in random texture [34]. However, due to limited number of slip systems in the hexagonal crystal structure, these alloys are prone to develop crystallographic texture during deformation and processing. Basal, transverse and basal/transverse textures are the commonly observed crystallographic textures in titanium alloys, which develops during the deformation. The texture type and its intensity depend upon the hot working temperature, degree and the mode of deformation imparted into the material during processing [6,40]. The observed texture components in the as-received Ti64 alloy was typical of a hot rolling deformation and depending upon the rolling temperature in the α + β phase field, basal splitting along TD or RD was reported. RD splitting was observed at higher rolling temperatures (900 °C) whereas, TD splitting was observed at a relatively lower temperature of 700 to 800 °C respectively [40].

Strong RD-split and the weak transverse texture component in the as-received condition (Fig. 8a) profoundly influenced the uniaxial mechanical properties as shown in Fig. 4. The variation in modulus, yield and tensile strength with respect to the tensile orientation can be explained on the basis of simplified Schmid (CRSS) analysis (i.e.) the basal plane orientation of crystallites with respect to the principal loading direction [41]. In textured titanium alloy, the yield strength is normally higher when the loading direction is parallel to the c-axis of the hexagonal crystal structure. This was attributed to the difficulty in accommodating the imposed deformation, due to limited number of slip systems in crystal c-axis direction (hard orientation). In the case of TD oriented tensile samples, the loading axis is nearly parallel to the hexagonal crystal c-axis resulting in higher yield strength, tensile strength and modulus in contrast to other orientations (Table 1 and Fig. 4). The normal anisotropy ($R_{N}$) and planar anisotropy (Δr) parameters computed from the R-values at various tensile orientations with respect to the rolling direction ($R_{00}$, $R_{45}$ and $R_{90}$) were 0.58 and −0.03 respectively.

Furthermore, to understand the anisotropic yield behavior of Ti64 alloy under various stress states, Knoop hardness yield loci was constructed from the Knoop hardness measurements on mutually perpendicular orthogonal planes (RD, TD and ND) along two different indenter orientations. Since the measured Knoop hardness number (KHN) is proportional to the octahedral shear stress; it can be converted in to yield stress in two principal directions without losing the anisotropic information (Eq. (2)). In other words, KHN is a graphical representation of resistance to plastic flow under plane stress condition, obtained using non-centrosymmetric Knoop indenter. Knoop-indentation based construction of yield loci also possesses certain inherent advantages such as small sample size requirement for its measurement, and it is non-destructive in nature.

$$\frac{\tau_8}{3} \alpha_i = \sqrt{\left(\frac{\sigma_{X}}{\sigma_i} - \sigma_{X} + \sigma_{Y} \right) \left(1 - \frac{\sigma_{i}^{2}}{\sigma_{X}}\right)}$$

(2)

where $\tau_8$ is the octahedral shear stress and $\alpha_i$ is the ratio of stresses in X-(RD) and Y-(TD) direction for six indenter orientations ($i = 1$ to 6).

Due to crystallographic texture in the as-received condition, Knoop indentation hardness varied with the direction of the indenter orientation with respect to the rolling direction on RD, TD and ND planes, resulting in asymmetric yield loci (Fig. 9). However, significant biaxial strengthening effect inferred from cruciform biaxial testing under equi-biaxial stress state was not reflected in the yield loci constructed from the Knoop hardness measurements. The ratio of equi-biaxial yield strength to uniaxial yield strength ($\sigma_{bi-RD}/\sigma_{uni-RD}$) evaluated from uniaxial and cruciform biaxial test results was around 1.19, whereas, from the Knoop hardness yield loci construction, this ratio was around 1.05.

Furthermore, the KHN yield loci of CP titanium and AA 7075-T6 alloy were included in Fig. 9 to demonstrate the efficacy of Knoop hardness measurements in capturing the equi-biaxial yield strength of materials with various degrees of anisotropy/texture in the as-received condition. In the case of strongly textured CP titanium, distortion in the first quadrant of the yield loci was evident, similar to the observed response of Ti64 alloy. However, this technique did not reflect the significant biaxial strengthening effect observed in CP titanium, under equi-biaxial condition [42]. The disagreement in measured values
between the Knoop hardness measurements and the cruciform equibiaxial tensile results may be attributed to localized deformation during Knoop indentation testing. Nevertheless, the texture induced distorted asymmetric yield loci was evidenced using the non-destructive Knoop hardness measurements. Furthermore, the root mean square error (RMSE) and the area of the ellipse were obtained from the best-fit regression analysis of six experimentally measured points in KHN yield loci (Table 2). The RMSE value is a measure of discrepancy between the experimental data points and the best-fit ellipse, respectively. The KHN loci did not accurately capture the ratio of uniaxial and biaxial yield strength of the material, nevertheless a good correlation exists between the RMSE value and the magnitude of anisotropic parameter (R-value), which is quantified from the uniaxial test results. When the anisotropy is lower in the as-received condition, good agreement is observed between the best fit ellipse and the experimental data points. Thus, the KHN yield loci can be used to reflect the degree of anisotropy in the material, which is manifested itself in the form of asymmetric loci as shown in Fig. 9.

It is also worthwhile to mention that the equibiaxial plastic response of Ti64 alloy differed quite significantly from the uniaxial counterparts as evident from anisotropic hardening under equibiaxial condition (Fig. 5a and 7a). Hence, in order to understand the operating deformation mechanisms as a function of stress state, fractography and X-ray texture evolution studies were carried out on failed samples. The representative fractographic images under uniaxial and equiaxial stress state are shown in Fig. 10. The fracture surface of the uniaxially failed samples is rough and transgranular in nature with conical dimples of 10 μm in size, which is reminiscent of a ductile fracture (Fig. 10b). The fracture surface of the biaxially failed samples revealed shallower dimples and the orientation of the fracture surface was 45° to...
the loading directions. Finer dimples with an average size of about 5 μm were observed in biaxially failed samples (Fig. 10e), exemplifying the influence of stress state on the fracture behavior of Ti-6Al-4V alloy. Furthermore, the microscopic glide feature, which is an interwoven pattern of glide plane de-cohesion steps were frequently observed in fractographs of uniaxially failed specimens [43]. The microscopic serpentine glide feature, which is observed on the inner walls of the dimples, was attributed to wavy slip/cross-slip on multiple set of intersecting planes, resulting in ripple or step-like features as shown in Fig. 10c. Such features were not observed in the fractographs of biaxially failed samples suggesting limited deformation and less pronounced dislocation activity under equi-biaxial stress state, prior to final rupture.

Based on the bulk X-ray texture analysis of as-received samples, most of the basal oriented grains are distributed in the RD-TD plane with crystal c-axis parallel to the ND direction. Hence, ⟨c + a⟩ slip and/or compressive twinning may likely to get activated to accommodate strain in the thickness direction during biaxial loading. Compression twins are anticipated since the c-axis of near-basal textured Ti64 alloy (Fig. 8a) will experience the compression state of stress during in-plane biaxial loading (c-axis is nearly parallel to ND/thickness direction). It is already reported in the literature that, (10T1) compression twins played a prominent role in enhancing the stretch formability of strongly textured (basal) coarse-grained magnesium alloy under the biaxial stress state [25]. Hence, the microstructures of the deformed specimens were examined adjacent to the fracture surface, to study if compression twins were operational during the deformation.

Uniaxially deformed samples revealed an elongated primary alpha along the loading direction, whereas, the biaxially deformed specimens retained equi-axed microstructure with a marginal increase in the size of the primary alpha as shown in Fig. 11. However, irrespective of the stress state, the deformed specimens did not reveal any mechanical twins. Twinning in Ti64 alloy depends upon the interstitial content (oxygen), temperature, stress and strain rate imposed during the deformation [32,44–46]. Furthermore, the reported twinned volume fraction in Ti64 alloy was always much lesser than unalloyed titanium after an equivalent degree of rolling reduction due to increased resistance to twinning deformation as a result of aluminum addition [47].

In the current scenario, since there was no microstructural evidence of operative twinning mechanisms, high energy ⟨c + a⟩ slip system should get activated to accommodate the strain in the thickness direction during loading [41]. However, the activation of ⟨c + a⟩ pyramidal slip system is often difficult due to higher resolved shear stress of this slip system in contrast to prismatic slip. Owing to the lower symmetry of hexagonal crystal structure, deformation mechanisms, hardening phenomenon and texture evolution were strongly linked to each other. Hence, the texture evolution of biaxially deformed samples was examined to understand the operating deformations mechanisms under equi-biaxial stress state.

The texture evolution of the alpha phase is only considered upon deformation, since the volume fraction of the beta phase is less than 10% in the present investigated material. Moreover, texture in beta phase plays a dominant role in influencing the anisotropy of the material only during high-temperature deformation [48]. Orthorhombic sample symmetry was imposed for the as-received rolled plates. The intensity distribution along the ND-RD section of the (0002), and (1120), pole figures were plotted (Fig. 12) and this intensity plot does not correspond to any specific orientation line. Basal poles are tilted from the ND towards the rolling direction (RD) with maxima occurring at 20° along the ND-RD section for the as-received samples. In a similar context, the intensity distribution along the ND-RD section of the (0002), and (1120), pole figures of the biaxially deformed samples were shown. It is observed that the maximum pole intensity of the (0002), pole figure decreased with a concomitant increase in the intensity of the (1120), poles upon biaxial deformation (Fig. 12). This was attributed to the rotation of most of the basal oriented grains to 120° orientation resulted in the weakening of the basal texture upon biaxial deformation. The Φ2 sections revealing the important texture components upon uniaxial and biaxial deformations are shown in Fig. 13. Such a dramatic change in the texture or lattice re-orientation upon biaxial deformation is possible, only if twinning and/or pyramidal slip system (c + a) were operative during the deformation. In a similar context, compressive twinning in other low symmerty aggregates such as zirconium also leads to the alignment of the basal pole along the rim of the (0002) pole figure [49]. However, compression twins were not observed in the biaxially deformed samples (Fig. 11).

Zaefferer [46] reported that the beta (bcc) phase deformation or
grain boundary sliding would have resulted in accommodating the deformation (up to 5%) along the c-axis during the biaxial expansion of 1 mm thick Ti64 sheets. However, grain boundary sliding will play a significant role only at high-temperature super-plastic deformations [30]. Moreover, the volume fraction of the beta phase is less than 10% to accommodate the imposed c-axis deformation during biaxial expansion. Based on the evidence of observed texture transition, it is concluded that the $<c+a>$ deformation mechanism was activated during the biaxial deformation of Ti64 alloy, to accommodate the imposed deformation in the thickness direction. In addition to reduced dislocation activity, i.e. the absence of serpentine glide features (Fig. 10) under equi-biaxial stress state, less probable pyramidal $<c+a>$ slip system is activated during biaxial loading. The $<c+a>$ slip system is a secondary slip system which needs higher critical resolved shear stress (CRSS) for its activation in contrast to $<a>$ slip [6]. However, due to initial RD-split texture of the as-received material coupled with equi-biaxial tensile loading resulted in the activation of $<c+a>$ slip system, to accommodate the imposed deformation. Furthermore, the distinct deformation behavior and texture development of Ti64 alloy under equi-biaxial stress state implies the importance of experimental biaxial tensile testing.

5. Conclusions

The equi-biaxial tensile tests of Ti64 alloy were carried out using cruciform specimen geometry, and the following conclusions were drawn.

1. The proposed cruciform geometry was validated experimentally using digital image correlation technique and the strain homogeneity was witnessed in the gage section. Failure initiated in the gage section as evidenced through principal strain contour plots, thus addressing one of the key concerns of the previously proposed geometries. Furthermore, the failure strain extracted from the uniaxial tensile tests on cruciform specimen geometry was in close agreement with the failure strain obtained from the standard uniaxial dog bone specimen geometry.

2. The as-received Ti64 alloy with a bi-modal microstructure exhibited a weak transverse texture component and a strong RD-split-basal texture component which profoundly influenced the mechanical properties of Ti64 alloy, as evidenced through uniaxial and biaxial stress-strain responses. Significant improvement in yield strength with a reduction in ductility was observed under the equi-biaxial condition in contrast to uniaxial properties. The effective ductility under biaxial condition was 0.05 in contrast to uniaxial tensile ductility of 0.10 respectively, with a reduction of about 50% under equi-biaxial condition.

3. The reduction in ductility under biaxial condition was further corroborated by the reduction in dimple size and absence of serpentine glide features in fractographs of biaxially failed samples in contrast to pronounced serpentine glide features observed in uniaxial tests.

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**Fig. 11.** Microstructure of deformed Ti64 specimens as a function of stress state (a) uniaxial DD (b) biaxially deformed.

**Fig. 12.** Intensity distribution along the RD-ND section from the recalculated pole figures (a) $\{0002\}_\alpha$ and (b) $\{11\bar{2}0\}_\alpha$ pole figure.
to uniaxial condition.

4. The increased deformation constraint due to bi-directional loading coupled with difficulty to accommodate c-axis deformation due to strong RD-split basal texture, resulted in increased yield strength with a reduction in ductility under biaxial stress state. The biaxial anisotropic ratio ($R_b$) at the onset of plastic deformation was 1.22, which implies the anisotropic strain response of Ti64 alloy under equi-biaxial stress state.

5. Though the Knoop hardness (KHN) yield loci technique reflected the asymmetric yield behavior of Ti64 alloy, the ratio of uniaxial yield strength to equi-biaxial yield strength calculated from the KHN measurements was not in good agreement with the ratio of yield strengths estimated from uniaxial and equi-biaxial tensile tests. The observed asymmetric yield locus was a manifestation of crystallographic texture in the as-received condition.

6. The compression twins were not observed in the deformed specimens and hence the contribution of twinning mechanism during equi-biaxial deformation can be disregarded. However, based on the texture evolution studies, it is concluded that the $\langle c + a \rangle$ slip system was activated to accommodate the imposed deformation in the c-axis direction upon biaxial deformation.

Data availability statement

Data will be made available upon request.

Declaration of competing interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of the article titled “Texture strengthening and anisotropic hardening of mill annealed Ti-6Al-4V alloy under equi-biaxial tension” that was submitted for peer-review and publication in materials characterization (Elsevier) journal.

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Appendix A. Supplementary data

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References


