Incipient slip and long range plastic strain localization in microtextured Ti-6Al-4V titanium

McLean P. Echlin*, Jean Charles Stinville, Victoria M. Miller, William C. Lenthe, Tresa M. Pollock

Materials Department, University of California Santa Barbara, Santa Barbara, CA 93106-5050, USA

Abstract

High resolution scanning electron microscope digital image correlation (SEM DIC) was performed in situ during uniaxial loading on Ti-6Al-4V rolled titanium plate to determine the dependence of strain localization on microstructure and microtexture. Individual grains with preferred orientation for basal slip exhibited plastic localization along basal planes before macroscopic yielding. With additional strain, but still below macroscopic yielding, pyramidal and prismatic plastic activity was observed as slip bands transmitting across many grains and entire microtextured regions (MTRs). The localization of long range plastic strain occurred within MTRs that allowed for slip transmission across grains with low angle boundaries. The rolled titanium plate material having strong [0001] and [1010] texture components showed pyramidal and prismatic type slip extending across entire MTRs at strains well below macroscopic yielding. These strain localization processes occur much earlier in straining and over length scales much longer than observed with conventional slip offset imaging. The implications for properties are discussed.

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1. Introduction

Ti-6Al-4V is the most common wrought titanium alloy, used in industries such as aerospace, biomedical, and chemical processing and synthesis. Titanium alloys possess a wide range of microstructural conditions as a function of processing conditions, which result in a wide range of material properties [1,2]. The processing history of a Ti alloy is therefore of critical importance to the development of the microstructure and texture present in a final component and has implications for design strategies, which increasingly can leverage anisotropic material properties [3]. An example of texture control in titanium was recently demonstrated using additive manufacturing methods [4]. After a homogenization step in the β phase field, Ti-6Al-4V alloy hot working in the α + β field can result in equiaxed α grain microstructure, for sufficiently slow cooling rates [2]. The resulting equiaxed microstructure is often forged, producing extensive mm-scale bands of grains with preferred grain orientation, known as microtextured regions (MTRs) s or macrozones [3,5–9], which subsequent deformation steps can enhance or break apart. The slip activity within the MTRs affects the mechanical properties of Ti-6Al-4V, including fatigue properties [6], through the localization of plastic deformation, motivating a more detailed understanding of the initial slip processes.

Slip activity in the hcp α phase has been studied for titanium alloys using high resolution SEM surface slip identification correlated with EBSD and Schmid factor (SF) analysis [10]. Pyramidal (α) and (c + a), prismatic, and basal type slip were all identified as active during room temperature tensile loading. The transmission of slip has been shown to be influenced by partial or full adherence to the Burgers orientation relation (BOR) between α grains separated by the β phase [11]. However, the authors also note that dislocations still can cross-slip and transmit through the β phase. TEM analysis of titanium samples with a lath colony microstructure has revealed variability in dislocation transmission across the α-β boundary that is dependent on the presence of residual dislocations at the boundary, even when satisfying the BOR [12]. SEM-based surface slip trace analysis in strained samples showed a dependence on slip transmission between α and β requiring aligned slip planes with high Schmid factors for both phases [13,14]. Most current research utilizes SEM-based surface slip trace analysis to determine slip activity, which is dependent on the extrusion and/or intrusion of slip bands at the free surface. This approach has been
shown to provide incomplete information on the operative slip processes [15, 16]. Recently, resolution improvements in scanning electron microscope digital image correlation (SEM DIC) techniques [15–19] allow measurement of in-plane plastic strain distribution at the sub-micrometer scale, which is not resolved by conventional SEM imaging. In this study, \textit{in situ} tensile loading of Ti-6Al-4V specimens was performed to determine the slip system activity by high resolution SEM DIC. The extent of strain localization, the relation of the localization to the microstructure, and the relative activity of slip systems is described in the following sections.

2. Experimental

Commercially available mill-annealed Titanium Ti-6V-4Al alloy rolled plate fabricated by Timet was investigated. The \(\alpha - \beta\) equiaxed microstructural condition was subjected to a heat treatment at 926 °C for 4 h followed by a 30 °C/h slow cool to homogenize. SEM and EBSD maps of the equiaxed microstructure are shown in Fig. 1(a–c). Tensile specimens were electrodischarge machined (EDM) from the plate material into flat dogbone samples of dimensions 11 mm \(\times\) 52 mm \(\times\) 1 mm, with a gauge cross-section of 1 mm by 3 mm and length of 10 mm. All surfaces of the samples were metallographically prepared with SiC to 1200 grit. Additionally, one of the 11 mm \(\times\) 52 mm faces was polished with a 6 \(\mu\)m diamond suspension and then chemo-mechanically polished in colloidal silica for 12 h.

Electron backscatter diffraction (EBSD) maps were collected from 2 mm \(\times\) 2 mm areas on the polished surface at 0.55 \(\mu\)m/pixel resolution with an EDAX Hikari XRD detector, as shown in Fig. 1(a). MTRs with different orientation are observed with 100–400 \(\mu\)m size along the ND direction that extend for millimeters along the TD and RD directions. The electron beam conditions used for EBSD mapping at 70° tilt before and after deforming the sample were a 30 kV beam energy, 6.55 nA beam current, with a 40 \(\mu\)m SEM aperture. The EBSD detector was operated using a 4 \(\times\) 4 camera binning mode with a 200 frames per second collection speed. A grain dilation routine was applied to the EBSD data to remove

![Fig. 1. Ti–6Al–4V alloy was investigated in the mill annealed \(\alpha - \beta\) condition, which is primarily composed of equiaxed \(\alpha\) grains that are strongly textured from rolling steps in the \(\alpha - \beta\) phase field. Microtextured regions (MTRs) are present at the scale of 100 \(\mu\)m-mm, as shown in (a), (b) Selected field of view (FOVs) were analyzed with subgrain resolution SEM digital image correlation (DIC), which exhibited MTRs of varied orientations and semi-randomly textured regions. The FOV in (b) is composed of many high resolution secondary electron SEM images which resolve the speckle pattern, (d), which has been etched into the microstructure as discussed in Section 2.](image1)

![Fig. 2. Mechanical testing of Ti-6Al-4V performed at room temperature loaded uniaxially and quasistatically resulted in the global stress-strain response for a sample with the loading direction oriented along the sample rolling direction (RD). SEM DIC measurements were made at the following strains: (a) 0.0065, (b) 0.0071, and (c) 0.0086 for the RD parallel to the loading direction samples, and at 0.0058, 0.0077, and 0.01 for samples with the loading direction parallel to the transverse rolling direction (TD).](image2)
misindexed points, primarily at or near the grain boundaries, for grains smaller than 5 pixels. Each grain, as determined by EBSD with a 5° tolerance, was also assigned an average orientation and used for the calculation of slip activity. Texture analysis using a harmonic series expansion with a series rank of 31 was performed and is represented in the inverse pole figure and pole figure plots.

Secondary electron SEM images collected with the electron beam normal to the surface (0° tilt) were made from selected areas in Fig. 1(b) within the EBSD mapped regions in Fig. 1(a). The field of view (FOV) in Fig. 1(b) is composed of many high resolution
images, one of which is shown in Fig. 1(c). The entire gauge section of the sample was speckle patterned for digital image correlation (DIC) measurements by chemically etching using Kroll’s solution at room temperature for 15 s. The alpha phase was preferentially etched, exposing 1–10 μm elevated regions of beta phase. The speckle pattern in Fig. 1(d) is composed of circular particles with diameter ranging between 50 and 250 nm with similar spacing between particles. This speckle pattern allows for in situ SEM DIC and simultaneous acquisition of EBSD information from the sample surface. The specific DIC parameters used to measure strain are discussed in Section 2.1. After DIC measurements were performed on the loaded samples, EBSD maps were again collected from the DIC measurement regions of interest. These EBSD maps were collected with the same parameters as described previously and were carefully aligned to the SEM DIC strain maps using affine transformation and alignment routines originally developed for merging 3-D multimodal datasets [20].

2.1. Digital image correlation

DIC measurements on the etched Ti-6Al-4V samples permitted examination of the dependence of initial straining on microstructural features and their relationship to the subsequent localization of strain within the microstructure. The speckle pattern produced by chemical etching generated random particle sizes and shapes, though mostly equiaxed, ranging from 50 to 250 nm with similar spacing between particles. This speckle pattern allows for in situ SEM DIC and simultaneous acquisition of EBSD information from the sample surface. The specific DIC parameters used to measure strain are discussed in Section 2.1. After DIC measurements were performed on the loaded samples, EBSD maps were again collected from the DIC measurement regions of interest. These EBSD maps were collected with the same parameters as described previously and were carefully aligned to the SEM DIC strain maps using affine transformation and alignment routines originally developed for merging 3-D multimodal datasets [20].

2.2. Mechanical testing

Mechanical testing was performed both ex situ and in situ to determine the macroscopic deformation response and the local strain states on the sample surface, respectively. All mechanical tests were performed at room temperature with strain rates of 10^{-3} to 10^{-4} on polished dogbone type samples with a 1 × 3 mm gauge section. An MTS instrument was used for macroscopic tensile testing in load control using an extensometer to measure displacement in the gauge section. In situ DIC experiments utilized a mechanical loading cell that was inserted into the SEM. Macroscopic strain was tracked by measuring displacements between fiducial marks on the sample surface in the SEM and also with an extensometer type strain gauge, which was monitored during loading. The stress-strain response of the Ti-6Al-4V alloy uniaxially
SEM DIC strain measurements were performed at macroscopic strains of 0.65, 0.71, and 0.86%, for samples that were loaded along the RD, as indicated on the stress-strain plot in Fig. 2. Samples were also loaded along the transverse rolling direction (TD) with SEM DIC measurements made at macroscopic strains of 0.58, 0.77, and 1.0%. The SEM DIC strain measurements collected at these macroscopic strains will be presented in Section 3.

3. Results

The extensive long range strain localization observed in this Ti-6Al-4V alloy required that large fields of view (500 μm × 500 μm) were collected at high spatial resolutions (0.067 μm step size and
0.472 by 0.472 \( \mu \text{m} \) subset size) with precise alignment of the imaging modalities (EBSD, secondary electron (SE)) in order to relate the strain localization to the microstructure.

3.1. Microstructure and mechanical response

To assess the anisotropy of the mechanical response due to the rolling process, regions were investigated from surfaces with the RD-ND and TD-ND directions in the imaging plane. The inverse pole figure (IPF) maps in Figs. 3 and 4 show the grain orientations with respect to the loading direction, which is parallel to RD or TD, respectively. The regions investigated show strong texture along the \( \{0001\}/C138 \) and \( \{010\}/C138 \) directions with respect to the sample RD, with maximum values of the orientation distribution function up to 20 times random. For the samples imaged in the RD-ND plane and the TD-ND plane, Figs. 3 and 4, a large fraction of the grains in the MTRs have orientations near the \( \{0001\}/C138 \) or \( \{010\}/C138 \). Three specific MTRs were studied, as shown in Fig. 3, one having predominant orientation near the \( \{0001\}/C138 \) (labeled as MTR 1), another having orientation near the \( \{0001\} \) (labeled as MTR 2), and a region with a more random orientation distribution (labeled as MTR 3).

Bulk Ti-6Al-4V samples were uniaxially strained in tension along the RD direction to produce the stress-strain plot shown in Fig. 2. The elastic modulus of the material loading along the sample RD was 116 GPa and had a 0.2% yield stress of 915 MPa. Flat specimens were machined, polished and etched from the same Ti-6Al-4V material and again uniaxially strained in tension along the specimen RD to the macroscopic strains shown in Fig. 2: (a) 0.65, (b) 0.71, and (c) 0.86, where SEM DIC measurements were made.

3.2. Strain localization at the surface

Strain localization was characterized using SEM DIC on surfaces of samples with RD-ND and TD-ND in the imaging plane, shown in Fig. 5 and Fig. 6, respectively. The regions were selected because they contained combined sub-regions with microtexture (MTRs) and with clusters of semi-random grain orientations.

At relatively low strains such as 0.65%, shown in Fig. 5, strain localized along individual slip planes within individual \( \alpha \) grains. However, upon further loading the regions of strain localization extended through the neighboring \( \beta \) grains, and into the adjacent \( \alpha \) grains (d.f.h). Slip band localization is not visible in any of the secondary electron images (c.e.g) until 4.8% macroscopic applied strain (i).

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through the β grains, the dark regions in the strain maps in Fig. 9(d–f, h), between the α grains. Only at strains beyond 1.0% do slip traces (shear or extrusions) begin to become visible in the high resolution secondary electron SEM images without using DIC. Out of plane extrusions do correlate with slip band formation on the surface of material at large macroscopic strains, imaged by secondary electron SEM, however in-plane shearing within grains is not resolved, as demonstrated by a comparison of Fig. 9(c and g) to Fig. 9(f and h). The images in Fig. 10(c) show in-plane strain measured using the secondary SEM images for unstrained (Fig. 10(a)) and 1.0% strained (Fig. 10(b)) Ti-6Al-4V specimen. Incipient slip is not easily observable without DIC analysis, as is apparent from Fig. 10(b). Readers may wish to refer to the associated movie in the Supplemental Material, where slip bands are shown to extend up to a β grain as observed in the micrograph macroscopically strained to 1.0% in Fig. 10(e). However, transmission across the β grain into the adjacent α grain is only resolved in the SEM DIC strain measurement, shown in the inset region of Fig. 9(f, h), and not in the secondary electron SEM image. The strain localization events connect α grains with different active slip systems despite the presence of β grains, which will be discussed in detail presently. The movies provided in the Supplemental Material, which refer to Fig. 10(d and e), indicate significant shearing in the β grain.

Supplementary video related to this article can be found at http://dx.doi.org/10.1016/j.actamat.2016.04.057.

The SEM DIC strain measurements were aligned with the EBSD maps in order to determine types of active slip systems on a grain-by-grain basis and to compare with the location of the MTRs. The type of slip system activity in each grain was determined by comparing the angle of the localization band measured using SEM DIC to the angle of the calculated slip trace on the surface, resolved using the EBSD grain orientation information, as plotted in Figs. 11 and 12 for the measurements in the RD-ND and TD-ND planes. A small proportion of the grains (<5%) had surface slip traces that were indistinguishable between the pyramidal and prismatic slip systems and were excluded from the analysis. At large deformations however, the ability to discern the independent slip system type may become challenging due to grain rotations, relative order of slip system activation, and microscope distortions. Especially at large macroscopic strains, emerging methods for the identification of slip systems [25] may become necessary.

The orientations of the grains that exhibited strain localization as a result of basal, prismatic, and pyramidal type slip are plotted with the calculated Schmid Factor (SF) contours on the inverse pole figures at each strain increment for both sample orientations, shown in Figs. 11 and 12. Only the pyramidal (α) type SF is shown on the contour plots; however (c + α) type is considered in more detail in Fig. 13. The orientations of grains plotted for each strain increment correspond to additional localization events since the previous strain increment.

Initially at low strains of 0.65% and 0.55%, the grains oriented such that they have a high SF for basal slip exhibit early strain localization, shown as the blue points in Figs. 11 and 12. However, with increased strains of 0.71% and 0.77%, the proportion of grains oriented with high SFs for both prismatic slip and pyramidal slip become greater, as evident by the grain orientations marked with red (prismatic) and green (pyramidal) points which show non-basal shear localization. The grains observed to have pyramidal slip have a Burgers vector of either (α) or (c + α) type. The large number of grains with orientations near the [0001] such as those in the MTR region labeled 2, also shown by the pole figures (PF) in Figs. 3 and 4, preferentially exhibit pyramidal type slip. These grains have a high SF for pyramidal slip (c + α) type, when compared to pyramidal slip (α) type SFs for near [0001] orientations. The slip system activity of a near [0001] grain is shown in detail in Fig 13, where the surface slip traces are calculated for each possible slip system. The strain map in Fig. 13 shows that the direction of the slip trace closely matches 1st and 2nd order (c + α) type pyramidal slip traces and (α) type pyramidal slip. The maximum SF for both 1st and 2nd order (c + α) type slip are greater than 0.45 for near [0001] orientations, whereas the SF for (α) type pyramidal slip is below 0.2.
MTRs with orientation near (10T0) such as those labeled 1, predominantly show prismatic type slip. The MTRs with more random orientations, such as those labeled 3, show activation of all three slip systems with some bias toward the basal slip. All SEM DIC strain measurements were performed below macroscopic yielding in the Ti-6Al-4V samples. Localized plasticity is observed by SEM DIC as shown in Figs. 5 and 6, which is associated with slip activity. Additionally, elastic strains can be resolved in the SEM DIC measurements. In reference to the horizontal loading direction the elastic modulus of individual grains has been calculated, Fig. 14(b). The high modulus regions (i.e. MTR 2) arise from the texturing.

Elastic strains measured below 0.8% require different visualization parameters than those required for plasticity. The $\varepsilon_{xx}$ strain field has been measured at 0.71% macroscopic strain in Fig. 14 which includes both the elastic and plastic strain fields. An average variation in strain can be observed between the MTRs labeled 1 and 2, as a result of elastic strain contributions. The (10T0) oriented grains (labeled MTR 1 - blue in the IPF map) have higher average $\varepsilon_{xx}$ total strain per grain, by roughly 0.15%, in relation to the (0001) oriented grains (labeled MTR 2 - red in the IPF map). The spatial relation of the diffuse elastic strain with respect to the MTRs is shown in Fig. 14, where the elastic modulus, (b), of the average orientation of the grains is shown in comparison to a $\varepsilon_{xx}$ strain map, (c), for the same microstructural region at 0.71% macroscopic tensile strain. The MTR labeled 2 has high elastic modulus and shows low elastic strains, compared with the MTR labeled 1, which shows the opposite trend.

4. Discussion

The SEM DIC strain measurements show extensive localized plastic straining below the 0.2% yield strength of the material, as shown in Figs. 5 and 6. Comparison of the calculated surface slip trace based on the grain orientations to the measured strain localization on the surface indicates the type of slip activity present up to the onset of macroscopic yielding, in Figs. 11 and 12. In the rolled equiaxed Ti-6Al-4V material tested here, basal type oriented grains preferentially exhibited strain localization upon loading. These basal oriented grains and the basal slip they produce is isolated from the surrounding MTRs, as shown by the strain localization within single grains in the lowest macroscopic strain
measurements at 0.65% and 0.58%, for samples loaded in tension along the RD and TD, respectively. With continued loading, but still well below macroscopic yielding, pyramidal and prismatic slip develop with long range localization across MTRs containing grains with similar orientations. The material tested has strong $\langle \frac{1}{2} 0001 \rangle/C138$ and $\langle \frac{1}{2} 10 \rangle/C138$ texture components, shown in Figs. 3 and 4, which predisposes the material for early pyramidal type slip. The SF for 1st and 2nd order $\langle c+a \rangle$ type slip is near 0.5 for $\langle 0001 \rangle$ grain orientations, however (a) type pyramidal slip also has a high SF compared to prismatic and basal near the $\langle 0001 \rangle$. The surface slip trace analysis employed here cannot distinguish between 1st and 2nd order $\langle c+a \rangle$ and (a) pyramidal type slip.

Individual MTRs were observed to have differing mechanical response, depending on the net orientation of the MTR. MTRs with primarily $[10 \bar{1} 0]$ orientation and labeled as MTR 1 in the IPF maps show mostly prismatic type slip, while the $\langle 0001 \rangle$ orientation MTRs labeled as MTR 2 show mostly pyramidal. The regions with more random orientation, labeled MTR 3, show contributions from all three slip systems with some bias toward the basal slip.

In addition to the SFs of each slip system, the relative critical resolved shear stress (CRSS) values will play a role in determining which slip systems are active. Reported CRSS values for all slip modes from the literature [10, 26–33] are shown in Table 1. Pyramidal $\langle c+a \rangle$ slip in titanium alloys is most commonly reported to be 1st order [26], though in many cases the Burgers vector is determined but the plane is not confirmed. Generally, all three
modes of \(\{a\}\)-type slip are soft relative to \(\{c+a\}\) slip, with the prismatic \(\{a\}\) to pyramidal \(\{c+a\}\) CRSS ratio falling between 1:1.6 and 1:3.9 for the considered alloys. Of the \(\{a\}\)-type slip modes, prismatic \(\{a\}\) slip typically being only slightly harder. The CRSS ratio between pyramidal \(\{c+a\}\) and pyramidal \(\{a\}\) slip is nearly the inverse of the ratio between the SFs, indicating that either type of slip may be occurring in the \(\{0001\}\)-oriented grains.

At total strains between 0.7% and 0.9%, strain localization continues with the longest band spanning across 21 grains (258 \(\mu\)m), as shown in Figs. 5 and 6. Long range strain localization was even observed for many non-basal slip configurations, as enabled by the

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### Table 1

<table>
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<tr>
<th>Material</th>
<th>Critical resolved shear stresses (MPa)</th>
<th>CRSS normalized to prismatic</th>
</tr>
</thead>
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<td>Prism.</td>
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banded MTRs containing many grains with similar orientations. The MTRs are 100–400 μm measured along the ND direction, while extending millimeters in the TD or RD directions. In other engineering materials such as polycrystalline superalloys and stainless steel, slip is localized within 2–4 grains (roughly 88 μm) [16] and 2–4 grains (roughly 100 μm) [18], when loaded to strains near the onset of yielding. Furthermore, long bands of shear (ε_y) are also shown to develop in MTRs at strains between 0.7% and 0.9%, with slip transmission between grains of similar orientations. The length of the concentrated shear band is dependent on the orientation of the MTR and the ability of slip to transmit between grains of similar orientation. This enables the propagation of slip across a MTR, based on the preferred orientation of the region. In addition to the plastic localization processes observed here, the overall heterogeneity of the straining process is also influenced by the elastic properties of the material.

The elastic anisotropy of the hcp α phase [34, 35] is apparent in Fig. 14(b), with high contrast between the MTR regions that predominantly have high or low elastic modulus. The average strain in the MTRs scales with the elastic modulus as shown in the strain measurements for the same field of view in Fig. 14(c). Within MTR 2 a small number of grains exhibit orientations that are far from the predominant basal orientation of the MTR. They are spatially located within the MTR and are constrained by the deformation of the MTR and therefore are more likely to develop plasticity. For example, the grains in MTR 2 that show strain localization first (non-red in IPF) are softer oriented and constrained by the predominantly basal oriented grains (red in IPF) that are deforming in unison, resulting in enhanced plasticity in the former. Furthermore, grains that had localized strain at small deformation initiated long range plastic strain localization in nearby grains that extended across entire MTRs having implications for crack initiation and fatique. Basal oriented MTRs that contain grains with dissimilar orientations (such as MTR 2), and high elastic anisotropy, have been shown to initiate cracks during low cycle fatigue [6]. Also in low cycle fatigue, a high frequency of crack initiation events and subsequent coalescence has been observed in basal and prismatic MTRs, but not in orientations that are less favorably oriented for slip [36]. Furthermore, at larger lengthscales, the preferential grain orientation distribution intrinsic to MTRs arranges the structure of rolled components such that adjacent MTRs will also be strained non-uniformly.

5. Conclusions

The experiments performed using SEM DIC provide evidence that long range strain localization occurs across MTRs at strains below macroscopic yield conditions. The relationship between the orientation of the MTR and the loading condition determines the activity of the slip system type, however pyramidal and prismatic slip are easily activated in these rolled materials due to preferential (0001) and [10T0] texture. Pyramidal slip was observed in [0001] oriented MTRs, likely to be (c + a) type because of high SF for these grains. The bands of localized strain developed via prismatic slip spanning as many as 21 grains (258 μm), and have been shown to originate from some grains showing early basal type slip in MTRs with [0001] grain orientations. In fatigue conditions where the applied macroscopic strain is often below the yield strength, the basal oriented grains will exhibit slip band localization in the first cycle. Basal slip was observed to initiate at the lowest strains, however the long range strain localization at elevated strain (still below macroscopic yielding) was primarily pyramidal and prismatic type. Furthermore, elastic anisotropy influenced the overall distribution of strain within MTRs with different preferred orientations.

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