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Version of record first published: 04 May 2012

To cite this article: H. Li, C.J. Boehlert, T.R. Bieler & M.A. Crimp (2012): Analysis of slip activity and heterogeneous deformation in tension and tension-creep of Ti-5Al-2.5Sn (wt %) using in-situ SEM experiments, Philosophical Magazine, 92:23, 2923-2946

To link to this article: http://dx.doi.org/10.1080/14786435.2012.682174
Analysis of slip activity and heterogeneous deformation in tension and tension-creep of Ti–5Al–2.5Sn (wt %) using in-situ SEM experiments

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(Received 14 November 2011; final version received 25 March 2012)

The deformation behavior of a Ti–5Al–2.5Sn (wt %) near-α alloy was investigated during in-situ deformation inside a scanning electron microscope. Tensile experiments were performed at 296 K and 728 K (≈0.4 $T_m$), while tensile-creep experiments were performed at 728 K and 763 K. Active deformation systems were identified using electron backscattered diffraction-based slip trace analysis. Both basal and prismatic slip systems were active during the tensile experiments. Basal slip was observed for grains clustered around high Schmid factor orientations, while prismatic slip exhibited less dependence on the crystallographic orientation. The tension-creep experiments revealed less slip but more development of grain boundary ledges than in the higher strain rate tensile experiments. Some of the grain boundary ledges evolved into grain boundary cracks, and grain boundaries oriented nearly perpendicular to the tensile axis formed ledges earlier in the deformation process. Grain boundaries with high misorientations also tended to form ledges earlier than those with lower misorientations. Most of the grain boundary cracks formed in association with grains displaying hard orientations, where the $c$-axis was nearly perpendicular to the tensile direction. For the tension-creep experiments, pronounced basal slip was observed in the lower-stress creep regime and the activity of prismatic slip increased with increasing creep stress and temperature.

Keywords: titanium alloy; tension; creep; dislocation slip; grain boundary sliding

1. Introduction

Titanium (Ti) and Ti alloys are often employed in structural applications in which weight reduction is important, including armor, portable electronic devices, biomedical devices, automobiles, and aerospace components [1]. A thorough understanding of the deformation mechanisms is required for the development of new Ti alloys with higher strengths. Researchers have investigated the intrinsic deformation mechanisms of Ti alloys in terms of slip and local crystal orientation. Examples of such studies under different loading conditions include uniaxial tension [2,3], four-point bending [4], micro-bending [5–7], fatigue [8,9], dwell fatigue [10–12]

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ISSN 1478–6435 print/ISSN 1478–6443 online
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http://dx.doi.org/10.1080/14786435.2012.682174
http://www.tandfonline.com
Experiments on high-purity, single-crystal Ti (hexagonal closed packed structure) have shown that the critical resolved shear stress (CRSS) for \{10\overline{1}0\} \{\overline{1}2\overline{1}0\} prismatic slip is much lower than that of other observed slip modes including \{0001\} \{\overline{1}2\overline{1}0\} basal slip, \{10\overline{1}1\} \{\overline{2}1\overline{1}3\} pyramidal \langle c+a \rangle slip and \{10\overline{1}1\} \{\overline{1}2\overline{1}0\} pyramidal slip [15]. More recently, Gong and Wilkinson [5] examined single-crystal commercially-pure (CP) Ti at room temperature (RT) using micro-bending experiments and found the CRSS values for prismatic slip (181 MPa) basal slip (209 MPa), and pyramidal \langle c+a \rangle slip (494 MPa). While there is no consensus on the precise CRSS ratio of these slip systems because alloy content and testing temperature affect the absolute CRSS values, the trends are consistent. Paton et al. [16] found the CRSS ratio for prism: basal: pyramidal \langle c+a \rangle to be 0.8:1:2.1 for Ti–6Al–4 V (wt %)\footnote{Paton et al. [16]} at 295 K. Bieler and Semiatin [17] found a ratio of 0.7:1:3 for the same alloy at temperatures between 1088 K and 1228 K. However, the CRSS of prismatic and basal slip was found to be nearly equal in a Ti–6.6Al single crystal at 300 K and 1000 K [3]. The RT CRSS of basal slip was found to be even lower than prismatic slip for Ti–6Al–4 V [2]. Regardless, prismatic slip alone is not sufficient to accommodate an arbitrary plastic strain, which according to the von Mises criterion requires five independent slip systems [18]. As a result, non-prismatic slip and/or deformation twinning are also necessary to achieve significant plastic deformation in polycrystalline Ti [19,20]. Besides dislocation slip and twinning, grain boundary sliding (GBS) is also a deformation mode. However, the role of GBS during deformation of Ti alloys at moderate-to-high temperatures is not thoroughly understood.

Reports of the deformation mechanism activation and evolution of Ti–5Al–2.5Sn are rare [21,22]. This near-\alpha alloy has approximately twice the yield and ultimate tensile strengths of CP Ti [23,24], but it is susceptible to cracking during forging and rolling used for breaking down the microstructure. This suggests that the activation of different slip systems may be altered with respect to CP Ti, as a result of alloying additions or the presence of the \beta phase. Unlike pure Ti, the deformation modes of Ti–5Al–2.5Sn, have not been well documented as a function of temperature. Such information may shed light on the underlying reasons for the susceptibility of Ti–5Al–2.5Sn to cracking during rolling and forging processes.

The objective of this investigation is to identify the activation of the different slip modes and GBS as a function of temperature and strain rate in Ti–5Al–2.5Sn. With that aim, \textit{in-situ} scanning electron microscope (SEM) constant strain rate tension and constant load creep experiments were performed in the temperature range from 296 K to 763 K. Microstructural examination of selected areas of specimen surfaces, both before and after testing, complemented with electron backscattered diffraction (EBSD), provided valuable quantitative information about the local deformation mechanisms prevalent in individual grains as a function of their orientations. A significant number of deformed grains were analyzed to obtain statistically significant information. In addition, the grain boundary deformation behavior, and in particular grain boundary ledge formation and boundary cracking, was examined by quantifying geometrical relationships between the loading axis and the grain boundaries as well as slip systems in neighboring grains.
2. Experimental methods

A forged Ti–5Al–2.5Sn near-α alloy was investigated. The bulk composition of the alloy was 92.4% Ti, 4.7% Al, 2.7% Sn, 0.2% Fe, and 0.1% Zn, as measured by inductively coupled plasma mass spectroscopy (ICP-MS). Flat dog-bone mechanical test samples with a 10 mm gage length were electrodischarge machined. The samples were sequentially polished using 9 μm, 6 μm, 3 μm and 1 μm diamond suspension and finished with 0.06 μm colloidal silica. A field emission Camscan 44FE SEM (Cambridge, UK), equipped with an EDAX-TSL (Mahwah, NJ, USA) EBSD system, was used to obtain EBSD orientation maps before and after deformation using either a 1 μm or 2 μm step size. X-ray diffraction (X’Pert Pro MRD, Philips, Eindhoven, Netherlands) was used to investigate the global texture of the material.

2.1. In-situ tensile experiments

Tensile tests were performed at 296 K and 728 K with a constant displacement rate of 0.004 mm/s (i.e. an approximate strain rate of \(10^{-3}\) s\(^{-1}\)) using an in-situ stage (described in [25]) inside a Carl Zeiss EVO LS25 SEM (Oberkochen, Germany). Displacement, time, and stress data were recorded during the tests using the MTESTW data acquisition and control software (Admet, Inc., Norwood, MA, USA). Backscattered electron (BSE) and secondary electron (SE) SEM images were acquired during the 296 K tensile experiments. The displacement was paused a few minutes while the images were acquired. During this pause, some stress relaxation of the specimens occurred. After imaging, straining continued at the same displacement rate. For the 728 K tensile experiments, a tungsten-based heating unit, powered by a constant voltage power supply, was used to heat the samples to the desired temperature, which was maintained for a minimum of 30 minutes before loading. The temperature was monitored using a thermocouple spot-welded to the side of the gage section. The 2 × 10\(^{-6}\) Torr vacuum environment inside the SEM chamber prevented significant oxidation and allowed imaging throughout the experiments. SE SEM images were acquired during the 728 K experiments. Strain was estimated using the measured gage length, specimen geometry changes, and the displacement data acquired during the experiments. The uniformity of strain was assessed by noting positions of microstructural features in low- and high-magnification SEM images. The samples were deformed to a minimum elongation of \(~3.5\)%, and none of the samples were taken to failure.

2.2. Tensile-creep experiments

For the in-situ tensile-creep experiments, the specimens were heated and monitored using the same configuration and conditions described above. A constant load rate of 5 N/s was used to bring the samples to the desired loads. The temperature was controlled within ±10 K of the target temperature for the duration of the experiments. SE SEM images were acquired without pausing the experiments. EBSD maps were acquired before the experiments on the same areas that were imaged in-situ during the experiments. The tensile-creep samples were taken to a minimum elongation of \(\sim 15.3\)%, and none of the samples were taken to failure.
2.3. **Slip trace analysis**

Slip trace analysis was accomplished by inputting the Euler angles from the EBSD orientation measurements from a given grain and all the possible slip systems into a Matlab\textsuperscript{TM} code that provided a visualization of the plane trace, slip plane, Burgers vector, and unit cell for each slip system. The Schmid factors for these deformation systems were determined based on the uniaxial tension global stress state. A further description of the slip trace analysis technique is provided in [4] and [26].

3. **Results**

Table 1 lists the testing conditions of the specimens studied. The global strains were measured using the displacement data recorded by the MTESTW software, while the local strains were measured by measuring relative displacements of obvious microstructural features on in-situ collected images acquired before and after deformation. In some cases, the local strains were different than the global strains due to inhomogeneous deformation. The strains reported in the rest of this paper are local strains unless otherwise specified. Because the $\alpha$ phase dominated the microstructure (less than 1\% of the total volume consisted of the $\beta$ phase), the slip analysis in this study focused on the $\alpha$ phase. The interested reader is referred to [27] for information regarding the slip transfer behavior between the $\alpha$ and $\beta$ phases in this material.

It is noted that the observed slip traces were a result of dislocation motion. There are several reasons why some grains may not develop observable slip lines; including the Burgers vectors were parallel to the sample surface, the slip was diffuse and was not constrained to well-defined slip bands, or the magnitude of slip was small and the slip bands were not well developed.

3.1. **Microstructure**

The as-received Ti–5Al–2.5Sn alloy exhibited the near-$\alpha$ (hexagonal close packed) microstructure shown in Figure 1. The body centered cubic (bcc) $\beta$ phase was observed at some of the equiaxed $\alpha$-phase grain boundaries and consisted of less than 1\% of the specimen volume. The average $\alpha$ grain size was $\sim 45\, \mu m$. Figure 2 shows the $\{0001\}$ and $\{10\bar{1}1\}$ pole figures from the undeformed materials measured using
X-ray diffraction (XRD). The X-ray pole figures show that the material exhibited a weak fiber texture, where the $c$-axis was about $30^\circ$ from the sheet normal direction. This texture results in a large fraction of the grains being oriented with high Schmid factors for basal slip.

### 3.2. 296 K tension

The engineering stress versus displacement curve for the 296 K tensile test and corresponding *in-situ* BSE SEM images are illustrated in Figure 3. Slip traces were first observed (see Figure 3c) at a global stress of 690 MPa, which was just above the approximate yield stress (YS) $\sim 660$ MPa. Most of the slip traces were identified to be basal slip systems with Schmid factors greater than 0.37 (blue lines in Figure 3c). A few prismatic slip traces with high Schmid factors (red lines) were also observed. After $\sim 3.5\%$ deformation, 93% of the $\alpha$ grains in the observed region exhibited slip traces (see Table 2). Multiple slip traces within a single grain were commonly observed. However, wavy slip traces were rare, indicating that cross-slip was not

![Figure 1](image1.png)

Figure 1. (a) SE SEM and (b) BSE SEM photomicrographs of the as-received forged Ti–5Al–2.5Sn alloy.

![Figure 2](image2.png)

Figure 2. (a) $\{0001\}$ and (b) $\{10\bar{1}1\}$ pole figures of the as-forged Ti–5Al–2.5Sn alloy measured using XRD.
Figure 3. (a) Engineering stress versus displacement curve of 296 K tension test, where arrows indicate when the test was interrupted for imaging and the letters b, c and d indicate when the BSE SEM photomicrographs (b) 0 MPa, (c) 690 MPa (when slip bands were first observed, ~0.5% strain), the color-coded planes traces for prismatic slip (red) and basal slip (blue) are labelled along with their Schmid factors, and (d) 762 MPa (~3.5% strain) were acquired. The loading direction was horizontal.

Table 2. Deformation behavior summary of tested specimens.

<table>
<thead>
<tr>
<th>Test condition</th>
<th>Number of grains analyzed</th>
<th>(%) of grains showing slip traces</th>
<th>Dislocation slip</th>
<th>Grain boundary ledge/sliding</th>
</tr>
</thead>
<tbody>
<tr>
<td>296 K tension</td>
<td>~147</td>
<td>~93%</td>
<td>42% basal, 50% prismatic</td>
<td>Not prevalent</td>
</tr>
<tr>
<td>728 K tension</td>
<td>~130</td>
<td>~94%</td>
<td>42% basal, 47% prismatic</td>
<td>Not prevalent</td>
</tr>
<tr>
<td>728 K, 300 MPa creep</td>
<td>~140</td>
<td>~86%</td>
<td>43% basal, 46% prismatic</td>
<td>Prevalent</td>
</tr>
<tr>
<td>728 K, 250 MPa creep</td>
<td>~440</td>
<td>~10%</td>
<td>78% basal, 16% prismatic</td>
<td>Prevalent</td>
</tr>
<tr>
<td>763 K, 250 MPa creep</td>
<td>~64</td>
<td>~67%</td>
<td>59% basal, 35% prismatic</td>
<td>Prevalent</td>
</tr>
<tr>
<td>763 K, 200 MPa creep</td>
<td>~278</td>
<td>~15%</td>
<td>74% basal, 24% prismatic</td>
<td>Prevalent</td>
</tr>
</tbody>
</table>
prevalent in the 296 K tension deformation. Two tensile $\{10\bar{1}2\}$ $\{\bar{1}011\}$ T1 twins were also observed.

The slip-trace technique was used to identify a total of 204 active slip systems in 147 $\alpha$ grains, including the deformed grains shown in Figure 3. In the 147 grains analyzed, 85 distinct basal slip systems and 101 distinct prismatic slip systems were identified (some grains exhibited multiple prismatic slip systems), with the remainder of the 204 active systems attributed to pyramidal and pyramidal $\{c + a\}$ slip systems. These observations indicated that basal and prismatic slip systems are the dominant deformation modes at 296 K. Figure 4 shows a histogram of the basal and prismatic slip distribution with respect to the Schmid factor. About 80% of the activated slip systems in these 147 grains exhibited Schmid factors, based upon the global stress tensor, greater than 0.3 for both basal and prismatic slip. As shown in Figure 4, the activation of basal slip is more prevalent in grains with higher Schmid factors for basal slip, and basal slip in grains with Schmid factors below 0.27 for basal slip was not observed. However, prismatic slip occurred in grains with smaller Schmid factors, even less than 0.1, suggesting that prismatic slip was more easily activated than basal slip at RT. For the prismatic slip systems with small Schmid factors (smaller than 0.15), the slip traces were located near the grain boundaries or triple points, where the stress concentrations were expected to be high, suggesting that the activation of these slip systems were assisted by local heterogeneous stress concentrations.

3.3. 728 K tension

Figure 5 shows the stress versus displacement curve and corresponding sequential SE SEM images taken during the 728 K tensile test. Slip traces were first observed at a stress level of 301 MPa and most of the slip traces were characterized to be basal slip systems. The YS (~330 MPa) was lower than that observed at 296 K (~660 MPa), which was expected based on previous findings [24].

About 94% (122 out of 130) of the grains exhibited slip traces (see Table 2). Most of the slipped grains exhibited basal or prismatic slip. Only 2 out of 122 slipped
Figure 5. (a) Engineering stress versus displacement curve of 728 K tension test, where arrows indicate when the test was interrupted for imaging and letters b, c and d indicate when the SE SEM photomicrographs (b) before loading, (c) 301 MPa (in the elastic regime), the color-coded plane traces for prismatic slip (red) and basal slip (blue) are labelled along with their Schmid factors, and (d) 426 MPa (~9% strain) were acquired. Grain G in (d) is the cross-slipped grain analyzed in Figure 7. The loading direction was horizontal.

Figure 6. A histogram of the 728 K tension test Schmid factor distribution for basal and prismatic slip systems.
grains failed to exhibit either basal or prismatic slip and these two grains exhibited pyramidal \(c+a\) slip. These observations suggest that basal and prismatic slip systems are the main deformation modes at 728 K. 192 active slip systems were identified in 122 grains, with the distribution histogram shown in Figure 6. Both basal and prismatic slip systems were observed for global Schmid factors as low as 0.15. This is in contrast with the observation at 296 K that no basal slip was observed for systems with Schmid factors below 0.27. Prismatic slip was observed in a wide Schmid factor range at 728 K, similar to the observations at 296 K. This is consistent with the fact that the CRSS for basal slip decreases more rapidly with higher temperatures compared to prismatic slip [3].

The development of wavy slip lines, indicative of cross slip, was first observed at a strain level of \(\varepsilon=2.8\%\). The development of cross slip in grain G in Figure 5(d) is shown in a series of SE SEM images in Figure 7. Trace analysis of the slip lines indicated that slip in the \(\overline{2}1\overline{1}0\) direction on (0001) basal, (01\(\overline{1}0\) prism, and (01\(\overline{1}1\) pyramidal planes was involved in the cross-slip with the common screw dislocation Burgers vector of \(\overline{2}1\overline{1}0\). These three slip systems exhibited relatively high Schmid factors of 0.3, 0.32, and 0.42, respectively.

3.4. 728 K, 250 MPa creep
Sequential SE SEM images taken during the 728 K, 250 MPa tensile-creep experiment are shown in Figure 8. The grain boundaries were locations where visible offsets were noticed. These offsets were typically observed out of the plane of the image (i.e. vertical) and were characterized as ledges once they were measured to be more than 2 \(\mu\)m in length. These grain boundary ledges formed at strains less than
0.5%, prior to any slip traces becoming evident, as shown in Figure 8b. These ledges continued to form with increasing strain, while only a few grains developed observable slip traces (see Table 2). After ~16.5% strain, ~10% of the total number of observed grains exhibited slip traces, suggesting that dislocation slip did not
contribute to the strain as much as GBS. Some of the observed grain boundary ledges eventually evolved into grain boundary cracks, as illustrated in Figure 9. Figure 10 shows a higher magnification SE SEM image that shows the details of GBS, which was evidenced by the protrusion of the grain labeled P and the recess of the grain labeled R.

Figure 9. SE SEM photomicrograph illustrating grain boundary cracking during the 728 K, 250 MPa tensile-creep experiment. The total estimated global strain was \(~13\%\). The loading direction was horizontal.

Figure 10. SE SEM photomicrograph of a grain boundary ledge which displaced approximately 16 \(\mu\)m and was evidenced by the protrusion in the grain labeled P and recess in the grain labeled R for the 728 K, 250 MPa creep experiment. The total estimated global strain was \(~13\%\). The loading direction was horizontal.
Slip trace analysis indicated that the majority of the grains that exhibited slip traces deformed by basal slip (see Figure 11). 45 slip systems were identified in the 44 grains exhibiting slip traces, with 35 basal slip system traces observed (35/45 ≈ 78%). In the 35 basal slip systems, 32 basal slip system traces observed were activated with Schmid factors greater than 0.35. Three basal slip systems were activated with relatively small Schmid factors (0.15–0.3) in three separate grains in which the Schmid factors of prismatic slip systems were less than 0.05. This suggests that the activation of these three basal slip systems were the result of strain accommodation. Prismatic slip only comprised a small fraction (7/45 ≈ 16%) of the identified slip systems, and all had Schmid factors greater than 0.4. This is in contrast to the tension tests where a large fraction of prismatic slip was observed (50% at 296 K and 47% at

Figure 11. A histogram of the Schmid factor distribution for basal and prismatic slip systems for the 728 K, 250 MPa creep experiment.

Figure 12. The y-axis represents the normalized fraction of grain boundaries for 26 grains that exhibited ledges at less than 0.5% strain during 728 K, 250 MPa creep experiments. The x-axis represents the angular range for either the angle between the grain boundary and the tensile-creep axis or the grain boundary misorientation angle. The plot for the total grain boundary misorientation includes all the grain boundaries in the microstructural patch analyzed (>1000), where only 26 of the grain boundaries exhibited ledges.
728 K), and prismatic slip was activated over a relatively large Schmid factor range. Compared with the higher strain rate tensile experiments, GBS and basal slip systems were much more favored during creep.

Figure 13. SE SEM photomicrographs taken during the 728 K, 300 MPa creep experiment: (a) before loading; (b) <0.5% strain, 8 min; (c) ~4% strain, 9 h; (d) ~9% strain, 24 h; (e) ~21.4% strain, 32 h. The loading direction was horizontal. The vertical lines evident in (a) and (b) were imaging artifacts.
The misorientation angles between grain pairs on either side of a given grain boundary ledge, as well as the angle between the boundary trace and the loading direction, were analyzed for 26 boundaries where ledges formed at less than 0.5% strain (Figure 8b) and the data are presented in Figure 12. The y-axis represents the normalized fraction of the number of observations in a particular angular range. There was a strong correlation between the boundary trace angles and the ledge formation. Ledges tended to form at boundaries orientated perpendicular or nearly perpendicular to the loading direction. More than 90% of the ledges formed at grain boundaries where the angles between the boundary and the loading axis were greater than 60°. Moreover, compared with the total grain boundary misorientation distribution, which included more than 1000 grain boundaries in the microstructural patch analyzed, ledges also tended to form at boundaries with large misorientation angles (greater than 30°). Of the 26 ledges that formed at less than 0.5% strain, 23 ledges formed at boundaries with high misorientation angles and large angles between the boundary traces and the loading axis (>60°). Only one formed at a low angle boundary and this boundary was orientated 75° to the loading direction. Two boundaries were observed to form ledges at boundaries with trace angles from 30° to 60° and these two boundaries had misorientation angles of 75° and 70°. Thus, the misorientation angle and the angle between the boundary and the loading axis were both associated with the formation of grain boundary ledges.

3.5. 728 K, 300 MPa creep

Figure 13 shows sequential SE SEM images acquired for the 728 K, 300 MPa creep test. Like the 250 MPa creep test, grain boundary ledges were observed early in the experiment (<0.5%), however, more slip traces were observed. After ~21.4% strain, ~86% of the total number of observed grains exhibited slip traces (see Table 2). This is significantly greater than the ~10% value exhibited by the 728 K, 250 MPa creep sample. It is expected that more slip activity would be present at 300 MPa, which is
close to the measured YS (~330 MPa) at 728 K. Wavy slip lines associated with cross-slip were observed at ~4% strain (see Figure 13c).

The result from this test was quite similar to the tensile experiment (Figure 6) where prismatic slip systems were activated over the entire Schmid factor range of 0 ~ 0.5. Basal and prismatic slip systems were almost equally active, as shown in

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Figure 15. SE SEM photomicrographs taken during the 763 K, 250 MPa creep experiment: (a) before loading; (b) ~0.7% strain, 8 h; (c) ~5.5% strain, 23 h; (d) ~8% strain, 34 h; (e) ~12% strain, 56 h; (f) ~15.3% strain, 59 h. The loading direction was horizontal.
146 slip systems were identified in the 121 grains that exhibited slip traces. In the 146 active slip systems, 63 were basal slip systems and 67 were prismatic. Pyramidal and pyramidal \((c + a)\) only comprised a small portion of the identified slip systems. Most of the basal and prismatic slip systems were activated with relatively large Schmid factors \((> 0.3)\). Compared to the creep sample deformed at 250 MPa, prismatic slip was more active and observed over a larger Schmid factor range \((0.15 \sim 0.5)\) for the creep sample deformed at 300 MPa. The pronounced prismatic activity in the 300 MPa experiment indicated that prismatic slip becomes easier to activate with increasing stress.

3.6. 763 K, 250 MPa creep

Figure 15 shows in-situ SE SEM images acquired during the 763 K, 250 MPa creep experiment. At \(\sim 5.5\%\) strain, grain boundary ledges and slip traces were observed. With increasing strain, more grain boundary ledges formed and more grains exhibited slip traces. The slip traces were planar and cross slip was not prevalent.

After \(\sim 15.3\%\) strain, 43 out of 64 grains \((\sim 67\%)\) showed evidence for 44 activated slip systems (see Table 2). About 24 \((24/44 \approx 59\%)\) of the activated slip systems exhibited basal slip and 17 \((17/44 \approx 39\%)\) displayed prismatic slip. The trace analysis showed that all of the activated slip systems had Schmid factors greater than 0.3 (with one exception for a prism slip system with a Schmid factor of 0.23), as shown in Figure 16. At 763 K, more prismatic slip and less basal slip was activated than in the sample creep tested at 728 K and 250 MPa. Thus, temperature plays an important role in determining which slip systems are activated.

3.7. 763 K, 200 MPa creep

To compare the effect of stress on the deformation mode at 763 K, a lower stress \((200 \text{ MPa})\) creep test was performed. Figure 17 shows the sequential SE SEM images
acquired for this test. Some grain boundary ledges formed at less than 0.5% strain. Later in the deformation, almost all the grain boundaries formed ledges. Several grains exhibited slip traces at ~2.9% strain. However, the extent of slip activity was much less than at the higher-stresses. The grains elongated in the loading direction, and grain boundary cracking was evident.

Figure 17. SE SEM photomicrographs taken during the 763 K, 200 MPa creep experiment: (a) before loading; (b) <0.5% strain, 9 h; (c) ~2.9% strain, 24 h; (d) ~8.3% strain, 30 h; (e) ~21.2% strain, 70 h; (f) ~24.8% strain, 94 h. The loading direction was horizontal. Note that ledges were first apparent in (b) and grew larger with increased deformation.
After ~24.8% strain, about 15% of the observed grains (43 out of 278) exhibited slip, and 46 slip systems were identified in the 43 grains after ~24.8% strain. 34 out of 46 slip systems were found to be basal slip systems and 11 were prismatic (see Figure 18). Only one pyramidal slip system was observed, with a Schmid factor 0.48. Similar to the 250 MPa creep tests performed at 728 K and 763 K, basal slip was dominant and activated in the Schmid factor range of 0.25–0.5. At 200 MPa, dislocation slip was not prevalent and grain boundary ledge formation and cracking appeared to be the preferred way to accommodate the strain. After ~24.8% strain, cracking was observed at triple points and grain boundaries (see Figure 19).

Figure 18. A histogram of the Schmid factor distribution of basal and prismatic slip systems for the 763 K, 200 MPa creep experiment.

Figure 19. SEM SE photomicrograph illustrating grain boundary cracking during the 763 K, 200 MPa creep experiment (~24.8% strain). The loading direction was horizontal.
4. Discussion

4.1. Deformation mechanisms

Dislocation slip and GBS were the two major deformation mechanisms observed in this study (see Table 2). At least 93% of the α grains exhibited slip traces in the 296 K and 728 K tension experiments, while the percentage of grains that displayed slip traces was smaller in the elevated-temperature creep experiments. During the creep experiments, grain boundary sliding was more active, as evidenced by the observed grain boundary ledges (see Figures 9 and 10). The highest-stress creep experiment (728 K, 300 MPa) appeared to be near the transition between these two processes as the percentage of grains which exhibited slip traces was similar to that observed in the tension experiments (i.e. 86% in the 728 K, 300 MPa creep sample and ~94% in the 728 K tension sample, see Table 2). The lower-stress creep experiments, for which a significantly lower fraction of grains exhibited slip traces (see Table 2), revealed more extensive grain boundary ledge formation compared with the higher-stress creep experiments. This is consistent with the work of Lüthy et al. [28], who indicated on deformation mechanism maps that grain boundary sliding tended to be the dominant deformation mechanism, relative to dislocation glide and power law creep, at lower stresses.

In addition to the change in the dominant deformation mechanisms with applied stress and temperature, the relative activation of different slip systems also changed with test condition. During the tensile tests, the observed basal and prismatic slip systems were almost equal in number, but prismatic slip was activated within a larger Schmid factor range. This could be explained by the fact that the prismatic slip systems with low Schmid factors usually activated later in the deformation process and were found to be localized at the grain boundaries and triple points, suggesting these low Schmid factor systems were activated under local stress concentrations (non-global Schmid) to accommodate the local strain. Nevertheless, the relative activation of basal and prismatic slip systems was affected by the deformation temperature and the applied stress level. As the stress level decreased, the overall amount of observed dislocation slip activity decreased, but the amount of basal slip system activation increased relative to the observation of prismatic slip. This is consistent with the work of Williams et al. [3] who found that basal slip became more dominant with higher temperature deformation, and this observation was rationalized based on a decreasing CRSS for basal slip with increasing temperature.

4.2. Deformation behavior compared with CP Ti and other titanium alloys

Compared to CP Ti [4], basal slip was more active in Ti–5Al–2.5Sn. Extensive basal slip has also been observed in Ti–6Al–4 V [2] and Ti–6Al [29]. These observations, along with the current work, are consistent with prior work that showed that as the c/a ratio increases with increasing Al content [30], the basal plane becomes more closely packed and thus more favorable for slip, while the prismatic planes become less closely packed and less favorable for slip. Increases in Al content also result in a decrease in the stacking fault energy on the basal planes, and as a consequence dislocation movement is increasingly restricted to the basal planes [31,32].
In the present study, only two twins were observed during the 296 K deformation and these were T1 twins: (10\(\overline{1}\)2)/(\(\overline{1}\)011). Previous work found that twinning in CP Ti frequently occurs in hard orientation grains [4]. Twinning has also been observed frequently in Ti–1.4Al, Ti–2.9Al, and Ti–5Al single crystals, yet twinning was not observed for Ti–6.6Al single crystals [3]. Twinning was also not observed in a

Figure 20. Unit triangle plots of grains which exhibited basal and prismatic slip for the two tension and four tension-creep specimens with Schmid factors contours overlaid.

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separate deformation study of Ti–6Al at RT [29], however, twinning has been observed at RT in Ti–1.6V and Ti–0.4Mn [33,34]. Williams et al. [3] found that twinning can be restricted by the ordering of Al in the α phase, which is consistent with the lack of deformation twinning observed in the studied alloy. Williams et al. [3] also found that twinning activation decreased with increasing temperature. Thus the lack of twining observed in the present study, especially at elevated temperatures, is not unexpected.

4.3. Role of crystallographic texture in slip system activation

The extensive activation of basal slip observed in this study may in part be a result of the crystallographic texture (see Section 3.1 and Figure 2), leading to a larger than random number of grains with large Schmid factors for basal slip. This crystallographic texture may also play a role in the observation that prismatic slip contributes less to the strain than basal slip with decreasing stress levels. Figure 20 shows stereographic triangles with the grain orientations plotted for basal and prismatic slip for six different test conditions. The stereographic triangles show Schmid factor contours for the two slip system types, while the line AB on each of the triangles represents orientations where the Schmid factors for basal and prismatic slip were equal. For both the 296 K and 728 K tension experiments where multiple slip systems were common, basal slip was generally confined to grains with high Schmid factors for this type of slip (see Figures 20a and c). In contrast, prismatic slip was activated over the entire spectrum of Schmid factors (see Figure 20b and d). For those grains that exhibited prismatic slip with low Schmid factors (<0.2), usually another slip system with a high Schmid factor was activated and dominant in the same grain. Thus, it appears that the local stress state varied and secondary or tertiary prismatic slip systems with low Schmid factors were activated, indicating that Schmid’s law was not sufficient to quantify such multiple slip activations. In contrast, during the creep experiments (especially 728 K, 250 MPa; 763 K, 250 MPa and 763 K, 200 MPa), the deformed grains usually exhibited one active slip system and both basal and prismatic slip systems were activated in high Schmid factor domains (see Figures 20e, f and i–l), implying that both slip systems have a large dependence on orientation. Thus, the Schmid factor was an effective parameter to describe the activation of slip systems for the single slip grains in creep.

4.4. Cracking with respect to local crystal orientation

Cracks were found at both grain boundaries and triple points in all four of the creep-tested specimens. For cracks that formed at grain boundaries, most were observed in association with grain boundaries that had at least one of the two neighboring grains in a hard orientation, where the c-axis was within 30° of the loading direction, as shown in Figure 21a. Likewise, cracks that formed at triple points were also found to form in association with at least one grain having a c-axis oriented within 30° of the loading axis, as shown in Figure 21b. Because the hard-oriented grains were more difficult to deform than the surrounding grains, incompatibilities were expected to develop at the boundaries, leading to large local stress gradients and cracking. Grain
boundary cracking associated with hard-oriented grains has also been observed in hot deformation of Ti–6Al–4 V and analyzed using self-consistent models [35,36].

5. Summary and conclusions
The deformation mechanisms of Ti–5Al–2.5Sn were studied using in-situ tensile and tensile-creep experiments performed inside a SEM. Basal and prismatic slip systems were dominant for the 296 K and 728 K tension experiments. Twinning was rarely observed (and only at 296 K) and this was likely due to the Al content present in the alloy. Prismatic slip was activated within a wider Schmid factor range than basal slip and this was explained by its tendency to be confined to localized reasons, such as triple points and grain boundaries, where stress concentration are likely. With increased temperature, basal slip systems became easier to activate, implying that the CRSS for basal slip decreases with increasing temperature. During creep performed at temperatures between 728 K and 763 K and applied stresses in the range 200–300 MPa, slip was observed to a significantly lesser extent than for the higher-stress tension experiments. Instead GBS was dominant. Grain boundaries with larger misorientations and orientations nearly perpendicular to the loading axis tended to form ledges earlier in the deformation process. Some ledges eventually evolved to grain boundary cracks. Most of the grain boundary cracks formed in association with grains displaying hard orientations, where the \( c \)-axis was nearly perpendicular to the tensile direction. The transition from GBS to dislocation slip during creep appeared to be around an applied creep stress close to the YS, based on the observation of a significantly increased number of slip traces in the highest-stress creep experiment. During creep, prismatic slip was much less active than basal slip. However, the prismatic slip activity was increased by both increased creep stress and temperature.

Acknowledgments
This research was supported by the US Department of Energy, Office of Basic Energy Science through grant No. DE-FG02-09ER46637. The authors are grateful to J. Seal, L. Wang, Y. Yang and Z. Chen of Michigan State University for their intellectual discussions and
assistance with sample preparation. The authors are grateful to T. Van Daam of Pratt & Whitney Rocketdyne for providing the alloy used in this study.

Note
1. Henceforth, all alloy compositions are given in weight percent.

References