

## Full length article

Local strain rate sensitivity of single  $\alpha$  phase within a dual-phase Ti alloyTea-Sung Jun<sup>\*</sup>, Zhen Zhang, Giorgio Sernicola, Fionn P.E. Dunne, T. Benjamin Britton

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## ABSTRACT

We have performed *in-situ* micropillar compression to investigate the local strain rate sensitivity of single  $\alpha$  phase in dual-phase Ti alloy, Ti–6Al–2Sn–4Zr–2Mo (wt%). Electron backscatter diffraction (EBSD) was used to identify two grains, anticipated to primarily activate  $\alpha$  slip on the basal and prismatic plane respectively. Comparative micropillars were fabricated within single  $\alpha$  laths and load-hold tests were conducted with variable strain rates (on the order of  $10^{-2}$  to  $10^{-4}$  s<sup>-1</sup>). Local strain rate sensitivity exponent (i.e.  $m$  value) is determined using two types of methods, constant strain rate method (CSR) and conventional stress relaxation method (SRM), showing similar rate sensitivity trends but one order higher magnitude in SRM. We thus propose a new approach to analyse the SRM data, resulting in satisfactory agreement with the CSR. Significant slip system dependent rate sensitivity is observed such that the prism slip has a strikingly higher  $m$  value than the basal. Fundamental mechanisms differing the rate sensitivity are discussed with regards to dislocation plasticity, where more resistance to move dislocations and hence higher hardening gradients are found in the basal slip. The impact of this finding for dwell fatigue deformation modes and the effectiveness of the present methodology for screening new alloy designs are discussed.

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## 1. Introduction

Titanium alloys are typically selected for structural load bearing components in the aeroengine industry due to their high strength-to-weight ratio, corrosion resistance and excellent mechanical properties. In service, these alloys are subjected to significant cyclic loading, with high thrust (i.e. stress) excursions during take-off, a load-hold during flight and unloading on landing. The implications of the load-hold have been systematically investigated by Qiu et al. [1] showing a pronounced holding effect on the fatigue life performance of dual-phase (i.e. HCP- $\alpha$  and BCC- $\beta$ ) titanium alloys, Ti–6Al–2Sn–4Zr– $x$ Mo ( $x = 2$  to 6, in wt%), where a significant hold at maximum load can reduce the number of cycles to failure by an order of magnitude or more when compared with simple ‘saw-tooth’ load-unload fatigue cycle. This is known as the dwell debit.

Dwell fatigue is clearly a time sensitive deformation mode, where simple evaluation of the critical resolved shear stress for individual slip systems is insufficient and therefore rate sensitive material properties should be considered [2,3]. Interestingly, some

dual-phase Ti alloys (e.g. Ti6242) are dwell sensitive, while others (e.g. Ti6246) are not. One potential factor explaining this dwell sensitivity is local load shedding [4–6]. This load shedding phenomenon is thought to occur during the load-hold, such that stress is shed from a ‘soft’ grain (i.e. deforming perpendicular to the  $c$  axis) to neighbour ‘hard’ grains (i.e. deforming parallel to the  $c$  axis), resulting in significantly high stresses towards the interface which may initiate fracture. This time dependent stress amplification plays a critical role in facet formation and the strain rate sensitivity (SRS) is thought to be an essential in understanding this load shedding phenomenon [7].

The concept of the SRS was well established with an application of uniaxial tension or compression testing approach [8–10]. Fundamentally, the SRS indicates the general relationship between a flow stress and a strain rate at constant strain rate and temperature, and this can be defined as follows:

$$\sigma = K\dot{\epsilon}^m \quad (1)$$

where  $\sigma$  is the true flow stress,  $\dot{\epsilon}$  the true strain rate,  $K$  a material constant, and  $m$  the strain rate sensitivity exponent. From a given stress-strain curve, the  $m$  value can be determined by the following

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equation:

$$m = d\ln\sigma/d\ln\dot{\epsilon} \quad (2)$$

Deformation in Ti alloys are in general more elastically and plastically anisotropic at the grain scale than other non HCP alloys [11–14], and it is therefore thought that strain rate sensitivity (SRS) is likely to be different with respect to grain orientations and the corresponding slip systems. Several authors have investigated the room temperature SRS of Ti alloys and determined the  $m$  values within a range of 0.007–0.04 for CP–Ti [15–19], 0.0185 for Ti–6Al and 0.01 for Ti6242 [20]. However, these studies cannot be simply compared owing to the use of materials with different grain sizes and/or testing methods, and more importantly the unspecified grain orientations and therefore corresponding slip systems.

Jun et al. [7] recently performed the comparative study to assess the local rate sensitivity of dual-phase, dwell sensitive Ti6242 and dwell insensitive Ti6246 using nanoindentation. The nano-indentation approach has been widely used to investigate the rate sensitivity of materials (particularly for nanocrystalline metals) and is insightful and relatively inexpensive to undertake [17,19,21,22]. They showed that similar rate sensitivities were found in hard and soft grain orientations of Ti6246, while a significant grain orientation dependence was observed in Ti6242. As the stress state around an indent is complex it was rather difficult to investigate the effect of individual phases and slip systems on the rate sensitivity, and therefore only averaged  $\alpha/\beta$  properties were extracted. Further investigation is therefore needed to establish whether  $\alpha$  type basal and prism slip of single  $\alpha$  phase have different strain rate sensitivity (similar to differing CRSS values) and relaxations of stress.

Precise mechanistic analysis using nanoindentation becomes highly complicated owing to the complex stress states existing near the indent and significant length scale contributions arisen from the indentation size effect for small indentation depths. In this study, we therefore adopted the micropillar compression technique [23,24] to isolating individual microstructural units and selecting particular slip systems for investigation.

For the SRS determination, we attempted to apply two well-known methods, the constant strain rate method (CSR) [5,15,20,25] and the stress relaxation method (SRM) [26–30], where the former uses the relations of flow stresses and a range of strain rates (see Equation (2)) and the latter of applied stress and a stress relaxation rate (see the Equation (3)).

$$m = d\ln\sigma/d\ln(-\dot{\sigma}) \quad (3)$$

The main purpose of this study is to investigate the local strain rate sensitivity of single  $\alpha$  phase in Ti6242 with a micropillar compression technique at differing strain rates. The focus is on how strain rate sensitivity varies with respect to individual slip systems (i.e.  $\alpha$  type basal and prism slip). This outlines an effective experimental methodology using a combination of SEM/EBSD, FIB and *in-situ* deformation with a nanoindenter (Alemnis) and discusses a potentially important contribution to the dwell fatigue problem, where particular slip systems are likely to enhance load shedding which leads to facet fatigue and ultimately can lead to component failure.

## 2. Materials and methods

A dual-phase Ti alloy, Ti6242 (Ti–6Al–2Sn–4Zr–2Mo in wt%), was supplied by IMR (Institute of Metal Research, China). The supplied material of as-forged bar with 20 mm diameter was sectioned perpendicular to the bar axis and metallographically prepared with SiC papers (up to 4000 grit) and then polished with ~50 nm OP–S (Oxide Polishing Suspensions) diluted with H<sub>2</sub>O by a

ratio 1:5 of OP–S:H<sub>2</sub>O. The Kroll's reagent (i.e. 2% HF, 10% HNO<sub>3</sub> and 88% H<sub>2</sub>O) was used for light etching for ~15sec. The samples were then finalised with the final etch and polish procedure, repeated 2–3 times until grain structure was clearly visible with polarized light microscopy.

Initial studies of the as-received material revealed complex lamellar structures, so that isolation of individual microstructure features would be complex. The sample was heat treated with conditions of holding at temperature of  $\beta$  transus +50 °C (i.e. 1040 °C) for 8 h and cooling down with a sufficiently slow rate of 1 °C/min [24]. This produced a colony-structured microstructure with large  $\alpha$ -lamella separated by thin  $\beta$ -ligaments, in clear prior- $\beta$  grain structures.

Electron backscatter diffraction (EBSD) was applied to characterise the microstructure and the crystal orientation of the sample surface using a Carl Zeiss Auriga CrossBeam FIB–SEM equipped with Bruker Esprit v1.9 software. EBSD maps were captured using a high current mode with an aperture size of 120  $\mu$ m and an accelerating voltage of 20 kV. Larger area map (1.6  $\times$  1.2 mm<sup>2</sup> with 1.2  $\mu$ m step size) taken was used to select grains for examination by considering Schmid factor calculations (performed based on [24]) to isolate grains for single slip.

Micropillar fabrication was performed using a FEI Helios Nanolab 600. An automated routine programmed by AutoScript™ was effectively used to make identical square cross-section pillars in the selected grains, which is likely to avoid pillar size effects [31,32]. A Ga<sup>+</sup> ion beam of 30 kV was used with a series of currents decreasing from 9.3 nA (rough milling) to 2.8 nA (medium milling) and finally to 0.92 nA (final milling). The fabrication time was about 17 min for 2  $\times$  2  $\times$  5.3  $\mu$ m<sup>3</sup> pillars.

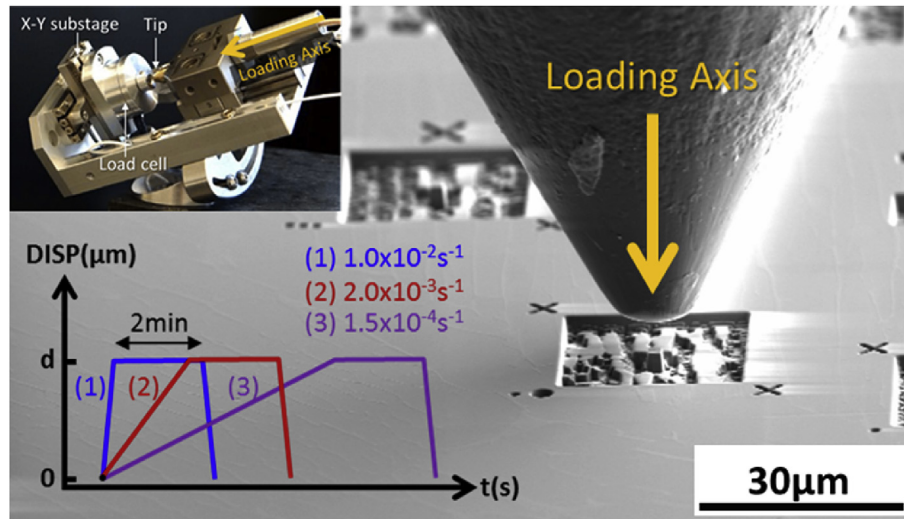
In order to isolate only  $\alpha$  phase within a pillar, each pillar was carefully machined within the  $\alpha$ -lamellar in between  $\beta$ -ligaments. The size of pillars was then measured based on the method shown in the Appendix.

*In-situ* micropillar compression tests were conducted at room temperature using an Alemnis nanoindentation platform (see Fig. 1) set in a SEM, where the load cell limit and resolution is 500 mN and 10  $\mu$ N, respectively. This platform has a pre-tilt angle of 30° with respect to the horizontal plane, and is actuated with a piezoelectric transducer, coupled through a linear spring to ensure that the displacement is in one axis only, and therefore operates in displacement control. The sample was mounted on top of a calibrated load cell, and carefully aligned to indent the pillars using a diamond flat punch indenter tip (10  $\mu$ m diameter, set on top of a 60° cone). This is likely to producing a contact misalignment angle between the top surface of a pillar and the indenter tip of less than a few degrees.

Load-hold experiments with variable strain rates, on the order of 10<sup>−2</sup> to 10<sup>−4</sup> s<sup>−1</sup>, were used in this study (see the schematic displacement–time graphs in Fig. 1). For each strain rate, the micropillars were near uniaxially compressed by the flat punch to a peak tip displacement of ~0.5  $\mu$ m (i.e. corresponding to ~0.1 strain) and held at this displacement for 2 min. The punch was then withdrawn to 1/10th of the maximum displacement and thus held to measure thermal drift, so as to correct the load–displacement results.

During the mechanical test, *in-situ* video was recorded at a low working distance using the secondary electron (SE) detector with an accelerating voltage of 5 kV. In the SEM, a reduced window with an effective frame rate of ~600 ms was used to improve the imaging quality. The videos were synchronised with the load–displacement–time data in a post-processing script written within Matlab, and these videos are provided in the supplementary section.

Post-mortem analysis of the deformed pillars was conducted by high resolution SEM imaging and engineering stress–strain determination (see the Appendix).



**Fig. 1.** SEM micrograph showing a flat punch tip (10  $\mu\text{m}$  dia.) and fabricated pillars within a trench, with inserts of Alemnis nanoindentation platform for in-situ compression test and the load-hold test conditions with various strain rates.

### 3. Results

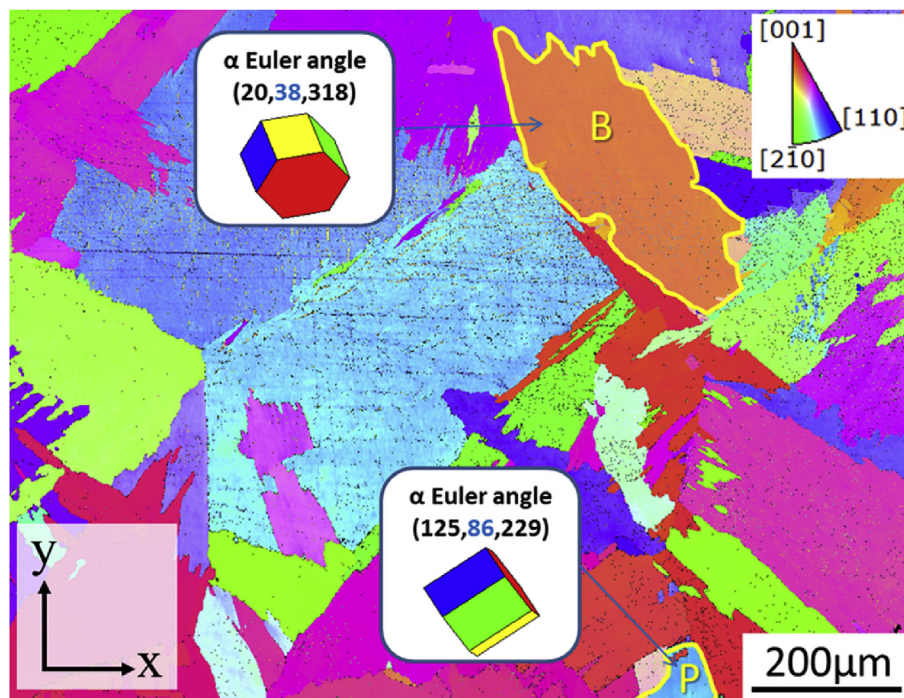
#### 3.1. Initial microstructural characterisation

The crystal orientation map is shown in Fig. 2 with inserts displaying the crystal orientations and the associated Euler angles ( $\phi_1$ ,  $\phi$ ,  $\phi_2$ ) of  $\alpha$  phase within two individual regions, labelled 'B' for the basal and 'P' for the prism slip systems. The orientations of each region indicate that the angle between the sample surface normal in Bunge convention and the c-axis of  $\alpha$  phase is  $38^\circ$  in 'B' and  $86^\circ$  in 'P'. The activated slip systems were then more accurately anticipated using Schmid factor calculation (see Table 1) such that  $a_1$  slip on the basal plane would be activated in the region 'B' and  $a_2$  slip on the prism plane in the region 'P'.

Fig. 3(a) and (b) show the secondary electron micrographs taken near the highlighted regions 'B' and 'P' respectively. It is seen that pillars were machined within each region isolated by individual microstructural units (i.e. colonies), which formed in different direction with respect to the sample coordinate system (x-y). In order to make the single (only)  $\alpha$  pillar a new pillar coordinate system (x'-y') was set near parallel to the colony direction (through in-plane rotation of x-y).

#### 3.2. Micropillar characterisation

The details of pillars machined in each region are shown with respect to the x'-y' coordinate system in Fig. 4. The contrast difference clearly shows that the only  $\alpha$  pillars were fabricated in

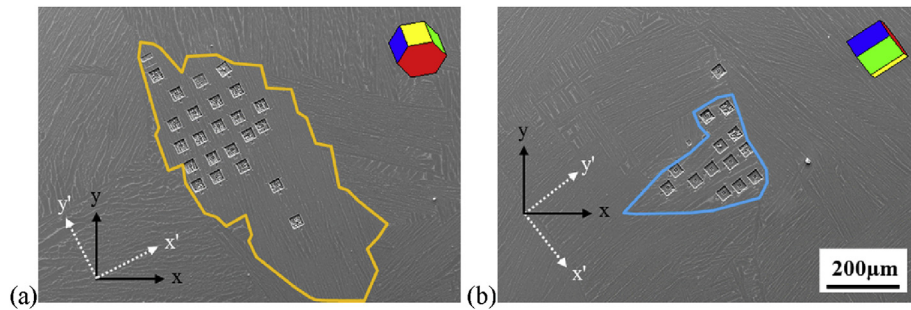


**Fig. 2.** EBSD derived inverse pole figure (IPF) map with inserts of unit cell structures of  $\alpha$  phase within regions 'B' and 'P', each of which has the primary slip system of  $a_1$  basal and  $a_2$  prism. The x-y in the small window indicates the sample coordinate system.

**Table 1**Schmid factors of HCP  $\alpha$  phase in regions 'B' and 'P' (30 slip systems in total).

|                                  | Slip system                                | B (basal)   | P (prism)   |                                  | Slip system                                | B (basal) | P (prism) |
|----------------------------------|--------------------------------------------|-------------|-------------|----------------------------------|--------------------------------------------|-----------|-----------|
| <i>a</i> Basal                   | (0001)[ $\bar{2}$ 110]                     | <b>0.46</b> | 0.02        | <i>c</i> + <i>a</i> Pyram. (1st) | ( $\bar{1}$ 011)[2 $\bar{1}$ 13]           | 0.26      | 0.08      |
|                                  | (0001)[1 $\bar{2}$ 10]                     | 0.36        | 0.05        |                                  | (0 $\bar{1}$ 11)[1 $\bar{1}$ 23]           | 0.13      | 0.40      |
|                                  | (0001)[11 $\bar{2}$ 0]                     | 0.10        | 0.07        |                                  | (1 $\bar{1}$ 01)[ $\bar{1}$ 2 $\bar{1}$ 3] | 0.14      | 0.06      |
| <i>a</i> Prism                   | (01 $\bar{1}$ 0)[ $\bar{2}$ 110]           | 0.11        | 0.31        |                                  | (10 $\bar{1}$ 1)[ $\bar{1}$ 123]           | 0.01      | 0.36      |
|                                  | (10 $\bar{1}$ 0)[ $\bar{1}$ 2 $\bar{1}$ 0] | 0.19        | <b>0.49</b> |                                  | (01 $\bar{1}$ 1)[ $\bar{1}$ 213]           | 0.23      | 0.32      |
|                                  | ( $\bar{1}$ 100)[ $\bar{1}$ 120]           | 0.08        | 0.19        |                                  | ( $\bar{1}$ 101)[2 $\bar{1}$ 13]           | 0.32      | 0.02      |
| <i>a</i> Pyram.                  | (10 $\bar{1}$ 1)[ $\bar{1}$ 2 $\bar{1}$ 0] | 0.01        | 0.41        |                                  | ( $\bar{1}$ 011)[1 $\bar{1}$ 23]           | 0.44      | 0.32      |
|                                  | (01 $\bar{1}$ 1)[ $\bar{2}$ 110]           | 0.32        | 0.26        |                                  | (0 $\bar{1}$ 11)[ $\bar{1}$ 2 $\bar{1}$ 3] | 0.19      | 0.25      |
|                                  | ( $\bar{1}$ 101)[ $\bar{1}$ 120]           | 0.12        | 0.13        |                                  | (1 $\bar{1}$ 01)[ $\bar{2}$ 113]           | 0.15      | 0.05      |
|                                  | ( $\bar{1}$ 011)[1 $\bar{2}$ 10]           | 0.34        | 0.45        | <i>c</i> + <i>a</i> Pyram. (2nd) | (11 $\bar{2}$ 2)[ $\bar{1}$ 123]           | 0.23      | 0.46      |
|                                  | (0 $\bar{1}$ 11)[2 $\bar{1}$ 10]           | 0.12        | 0.28        |                                  | ( $\bar{1}$ 2 $\bar{1}$ 2)[1 $\bar{2}$ 13] | 0.34      | 0.21      |
|                                  | (1 $\bar{1}$ 01)[1120]                     | 0.02        | 0.20        |                                  | ( $\bar{2}$ 112)[2 $\bar{1}$ 13]           | 0.33      | 0.04      |
| <i>c</i> + <i>a</i> Pyram. (1st) | (10 $\bar{1}$ 1)[ $\bar{2}$ 113]           | 0.02        | 0.15        |                                  | ( $\bar{1}$ 122)[11 $\bar{2}$ 3]           | 0.32      | 0.40      |
|                                  | (01 $\bar{1}$ 1)[ $\bar{1}$ 123]           | 0.40        | 0.46        |                                  | (1 $\bar{2}$ 12)[ $\bar{1}$ 2 $\bar{1}$ 3] | 0.03      | 0.17      |
|                                  | ( $\bar{1}$ 101)[1 $\bar{2}$ 13]           | 0.38        | 0.05        |                                  | (2 $\bar{1}$ 12)[ $\bar{2}$ 113]           | 0.07      | 0.06      |

The bold in each region indicates the highest Schmid factor among possible 30 slip systems, which enables us to anticipate the primary slip activity.



**Fig. 3.** Secondary electron micrographs showing fabricated pillars (centred in square boxes) within isolated morphologies of (a) region 'B' and (b) region 'P': The boxes were aligned with respect to the  $\alpha/\beta$  colony structures in each region, and thus a new coordinate system ( $x'$ - $y'$ ) was introduced.

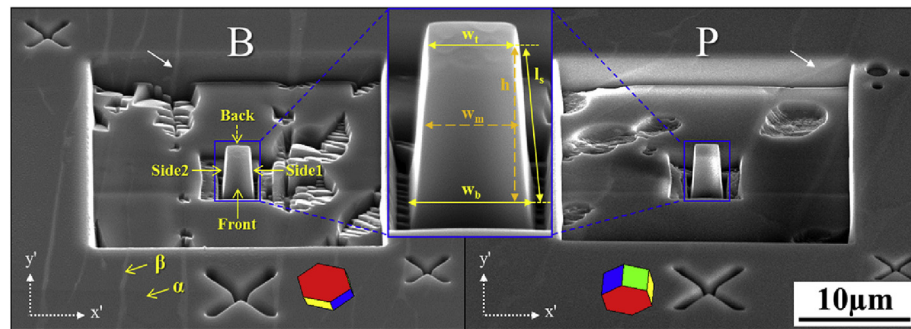
between  $\beta$  ligaments (i.e. thinner and brighter). Compared to the  $\alpha$  pillar fabrication in the region 'B', it was rather challenging in the region 'P' due to the highly slanted subsurface morphology of  $\beta$  ligaments.

A total of six pillars were considered in this study (i.e. pillars 1–3 for basal slip and pillar 4–6 for prism slip) and the dimensions of all pillars are very similar with the top width of  $\sim 2 \mu\text{m}$ , the taper angle of  $\sim 4.5^\circ$ , a height of  $\sim 5.3 \mu\text{m}$  and the aspect ratio of  $\sim 2.6:1$ . Note that the unit cells were matched with the micrographs (taken at tilting

angle of  $52^\circ$ ), by rotating  $27^\circ$  (CW) with respect to  $z$ -axis (out-of-plane direction) and tilting  $52^\circ$  with respect to  $x'$ -axis in the region 'B' and  $52^\circ$  (CCW) rotation plus  $52^\circ$  tilt for the region 'P'.

### 3.3. Slip trace analysis

In Fig. 5(a) and (b), local slip activities were traced in pillars 1 and 4, which were chosen to represent the slip activity in regions 'B' and 'P' respectively. Note that the chosen regions 'B' and 'P' are



**Fig. 4.** SEM micrographs of a square-shape micropillar of only  $\alpha$  phase fabricated in regions 'B' and 'P': The micrographs were aligned with respect to the new coordinate system ( $x'$ - $y'$ ). The contrast difference clearly reveals the  $\alpha$  (darker) and  $\beta$  (brighter) phases on sample surface as well as subsurface. The  $\alpha$  pillars were carefully fabricated in between neighbouring  $\beta$  phases, which was more difficult for region 'P' due to the highly slanted subsurface  $\beta$  morphology (marked by white arrow). Four faces shown in blue rectangle are used for slip trace analysis. The pillar is magnified in the insert and each dimensions are used to calculate the pillar size, engineering strain and stress (see Appendix). Note that the new coordinate system and the unit cells are matched with the micrographs taken at the tilting angle of  $52^\circ$ . (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

preferentially oriented to activate the  $a$  type basal and prism slips with the respective Schmid factors of 0.46 and 0.49 (see Table 1).  $a$  directions are superimposed in the unit cells, where further tilt angle of  $5^\circ$  was added to both unit cells so as to consider the initial taper angle of the pillars. To get better slip trace the unit cell in Fig. 5(a) was rotated  $90^\circ$  with respect to  $z$ -axis to match with the pillar micrograph taken at Side1. The slip trace analysis and the recorded video (see the supplementary section) clearly show that  $a_1$  slip on the basal plane was activated in the region 'B' and  $a_2$  slip on the prism plane in the region 'P', which are well agreed with the anticipated slip systems by Schmid's law.

### 3.4. Local rate sensitivity analysis

This section describes the local strain rate sensitivities of two individual slip systems (i.e.  $a$  type basal and prism) derived from three types of method: the constant strain rate method (CSRSM), the conventional and newly proposed stress relaxation method (SRM).

#### 3.4.1. Constant strain rate method (CSRSM)

The engineering stress-strain curves measured at the various strain rates in the region 'B' are shown in Fig. 6(a). The stress increases ~ linearly up to 0.025 strain with some microplasticity at the stress level of around 650 MPa for the rates of  $1 \times 10^{-2} \text{ s}^{-1}$  and  $2 \times 10^{-3} \text{ s}^{-1}$ , and around 500 MPa for the rate of  $1.5 \times 10^{-4} \text{ s}^{-1}$ . Strain hardening is followed and the gradients are changed at ~0.05 strain, though slightly different behaviour is observed at the rate of  $2 \times 10^{-3} \text{ s}^{-1}$ .

In the region 'P', as shown in Fig. 6(b), linear stress increases are also found in the elastic regime up to 0.025 strain at ~ equally separated stress level of 500 MPa ( $1 \times 10^{-2} \text{ s}^{-1}$ ), 450 MPa ( $2 \times 10^{-3} \text{ s}^{-1}$ ) and 400 MPa ( $1.5 \times 10^{-4} \text{ s}^{-1}$ ). Hardening gradients at each strain rate are comparative and considerable softening & hardening phenomenon is observed during continued deformation at the rate of  $1.5 \times 10^{-4} \text{ s}^{-1}$ .

In order to determine the strain rate sensitivity exponent ( $m$

value), stresses at strains of 0.045, 0.060 and the end of strain in each slip system (i.e. 0.082 for basal and 0.095 for prism slip) were collected and plotted with respect to the strain rates in log–log form. As shown in Fig. 7(a) and (b), the SRS in the prism slip system (i.e.  $m$  values in 0.068–0.097) is significantly higher than that in the basal (i.e.  $m$  values in 0.024–0.033). The former has much broader  $m$  values that were affected by the softening region shown in Fig. 6(b).

Fig. 8 shows the SEM still images of pillars 1–3 (i.e. showing the basal slip activity) taken from the *in-situ* videos at the moment that the 2 min holding starts, and the corresponding stress-strain curves. The dashed, coloured lines superimposed on the images and the curves are linked each other, indicating the stage at which the individual slip band formed. The band spacing at the middle of each pillar is quite similar, but the band formation stage is rather different. In P1, the localised slip band (i.e. red, dashed line) is related to the steeper hardening gradient, and similar trend is observed in P2 and P3.

In the prism slip activity, as shown in P4–P6 (Fig. 9), the slip band formation is quite different with respect to the applied strain rates, but the hardening behaviour is similar in the tested pillars. At lower strain rates (i.e. P5 and P6), the double-slip (i.e. yellow, dashed line) was observed and this is ascertained as  $a_2$  prism slip (see Fig. 5(b)) which has the second highest Schmid factor among  $a$  slip systems as shown in Table 1. Similar hardening gradients are seen for each of these pillars, and it seems that in contrast to the previous basal slip activity the individual slip band does not change the hardening gradients. The *in-situ* video analysis indicates that the softening shown in P6 was formed when the second slip band (i.e. blue, dashed line) was forming.

#### 3.4.2. Stress relaxation method (SRM) - conventional

Fig. 10(a) and (b) show the stress–time curves obtained in regions 'B' and 'P' respectively during 2 min holding. The stress relaxation curves were then used to determine the SRS exponent,  $m$  value, based on the Equation (3) and the literature [26]. Note that

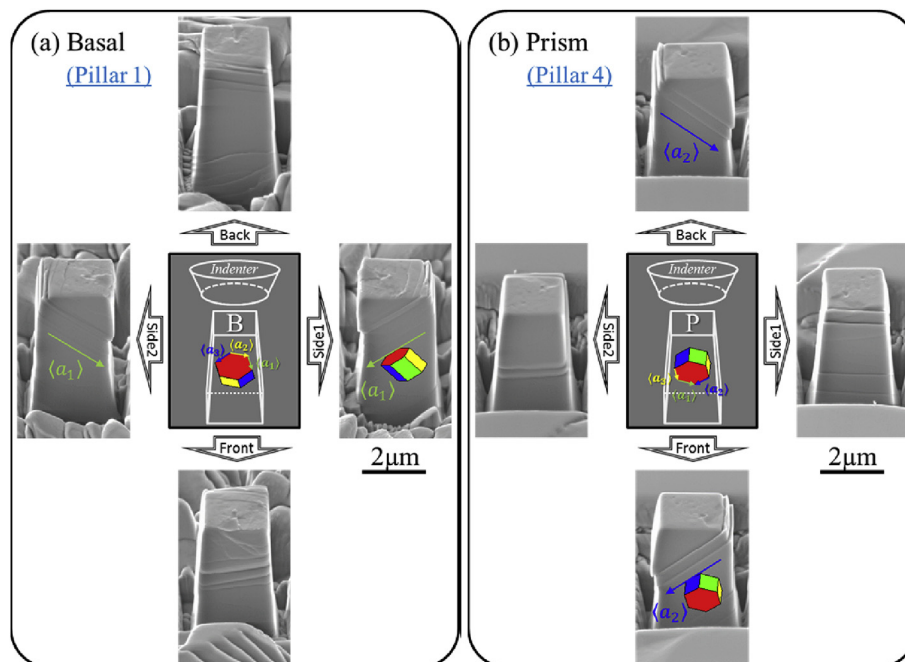
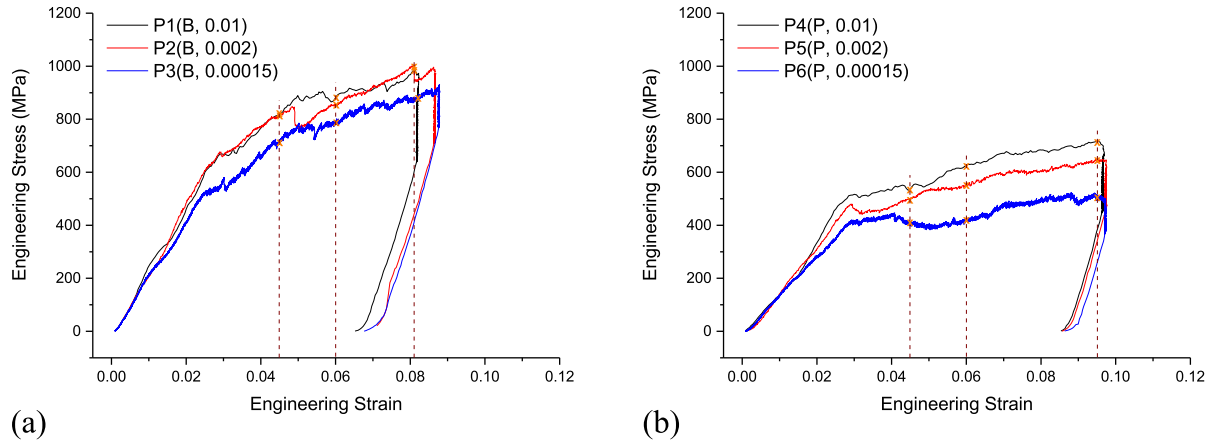
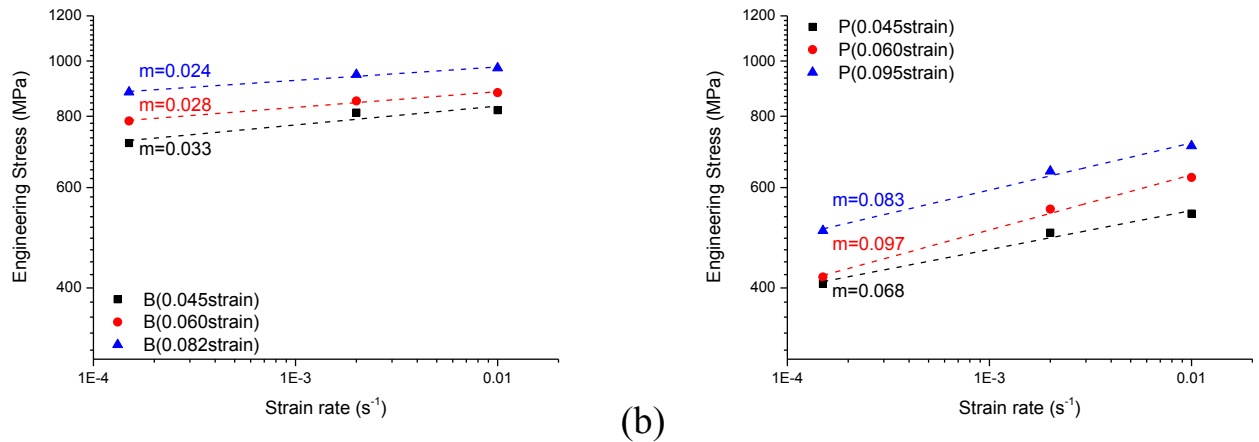


Fig. 5. Slip trace analyses of the deformed micropillars in (a) region 'B' (rep. by pillar 1) and (b) region 'P' (rep. by pillar 4), showing the activated slip system of  $a_1$  dislocation on the basal plane and  $a_2$  dislocation on the prism plane, respectively.



**Fig. 6.** Engineering stress-strain curves observed in (a) region 'B' and (b) region 'P': Three strain rates of  $1 \times 10^{-2} \text{s}^{-1}$  (0.01),  $2 \times 10^{-3} \text{s}^{-1}$  (0.002) and  $1.5 \times 10^{-4} \text{s}^{-1}$  (0.00015) were applied for individual slip systems in each region.



**Fig. 7.** Flow stress vs. strain rate (log–log form) in (a) region 'B' and (b) region 'P': The stress values were taken at three different strains (i.e. 4.5%, 6.0% and ~end of strains in each region). A slope of each fitted line indicates the strain rate sensitivity exponent,  $m$  value.

each curve was reproduced to simplified plots to improve the data analysis.

Fig. 11(a) shows a logarithmic plot of stress relaxation rate ( $-d\sigma/dt$ ) against applied stress for pillar 1 (and similar analysis is presented for other pillars in the [supplementary section](#)). The  $m$  value was then determined using inverse of the slope of each plot. Resultant SRS exponents are displayed in Fig. 11(b), showing similar SRS trend to that found in the CSRM analysis. Quantitative agreement was not found as this SRM analysis provides one order of magnitude higher than the CSRM. This may be due to the nature of stress relaxation test, where the load-time record is affected by both the plastic properties of the specimen (i.e. pillar) and the elastic properties of the testing machine, specimen and specimen mountings [27]. Further procedures are required to extract the exact material dependent rate sensitivity, but instead of calibrating further properties we propose a new methodology for analysing this SRM data in the following section.

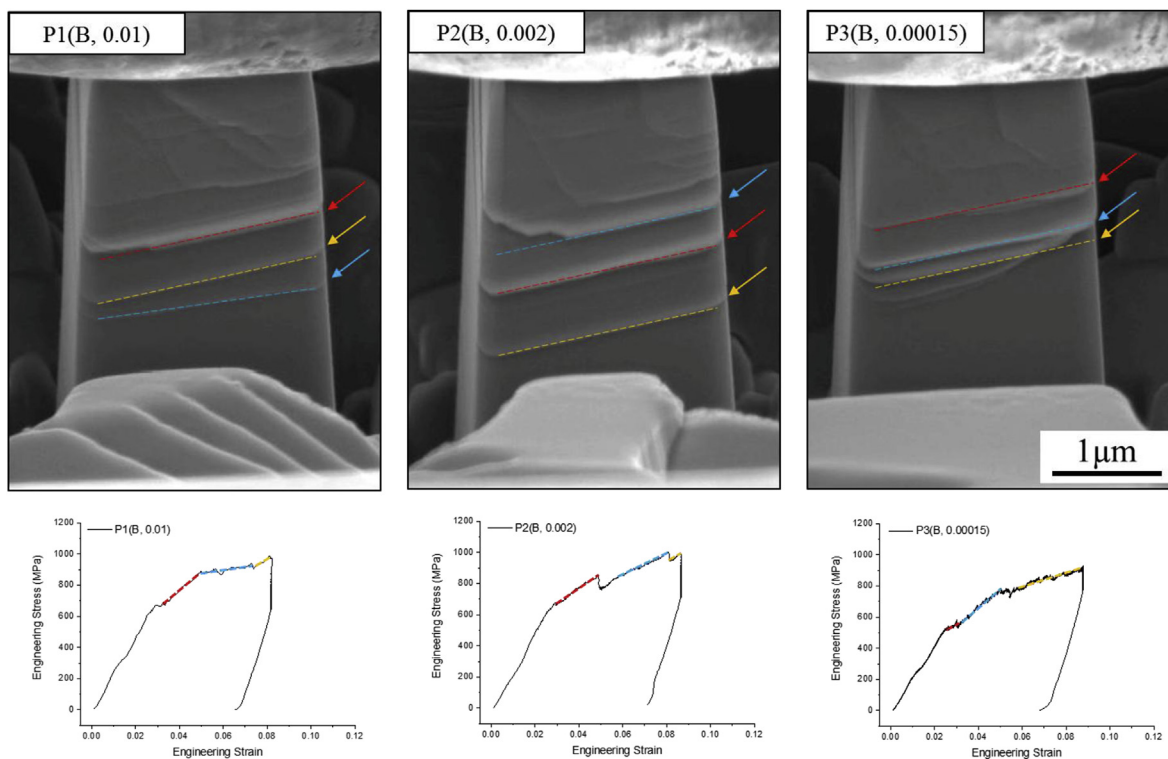
### 3.4.3. Stress relaxation method (SRM) – newly proposed

In this methodology, we first assumed that stress relaxation mechanisms of pillars tested for individual slip systems are consistent regardless of the applied strain rates. This was confirmed by *in-situ* videos, showing that neither a movement of existing slip bands nor a formation of new slip bands was observed. The rate

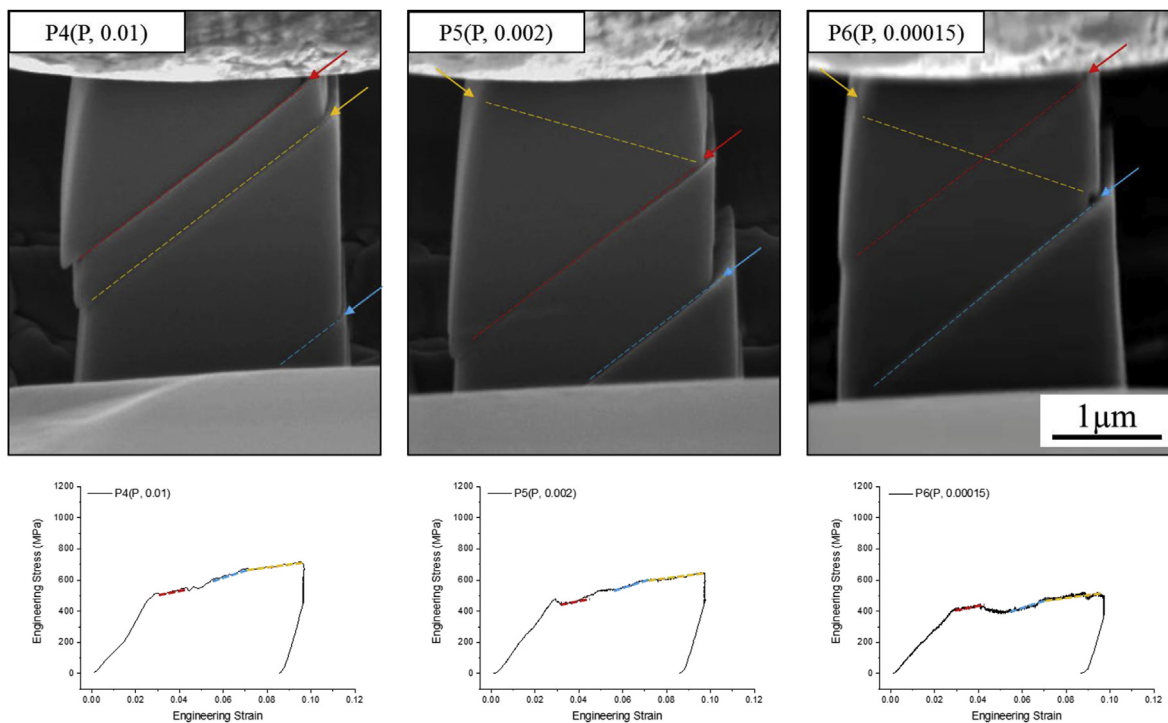
sensitivity analysis was similarly performed to the CSRM, such that from the stress–time curves (see Fig. 10) stresses at times of 30 s, 1 and 2 min were collected and plotted with respect to the strain rates in log–log form. The resultant plots of relaxation stress against strain rate for basal and prism slips are shown in Fig. 12(a) and (b) respectively. The  $m$  values determined for the prism slip (0.054–0.063) were higher than those for the basal (0.021–0.042), as similarly obtained from the CSRM.

## 4. Discussion

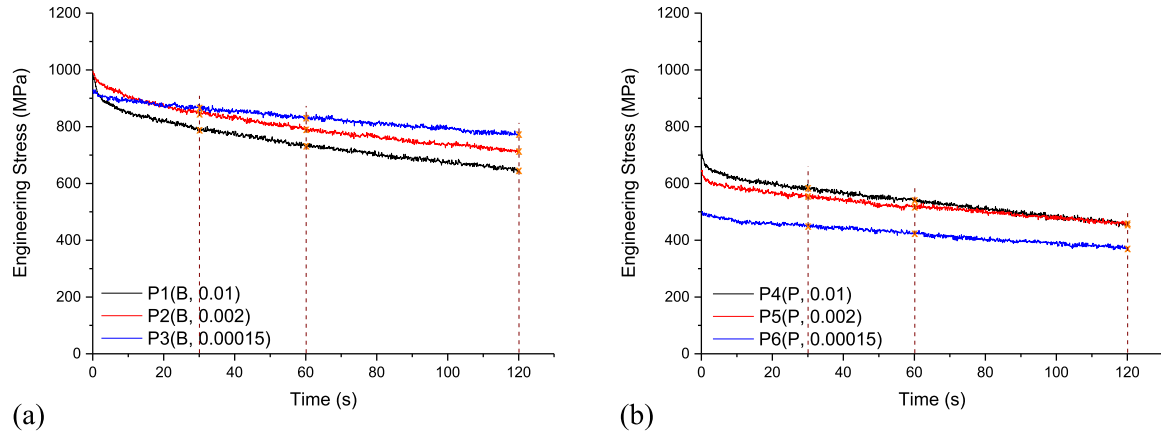
The strain rate sensitivity of deformation in Ti alloys is essential in understanding the time dependent nature of dwell fatigue as the dwell (i.e. hold) time affects significantly the plastic strain accumulation, resulting in reducing the dwell debit by one order of magnitude or more [1]. Our previous work using nanoindentation shows the significance of this rate sensitivity issue such that local rate sensitivity of a cluster of similarly oriented  $\alpha$ -phase lath connected with  $\beta$ -ligaments in the dwell-sensitive Ti6242 and the dwell-insensitive Ti6246 is strikingly different [7]: Ti6242 has higher rate sensitivity ( $m = 0.039$ ) for the hard grain orientation as compared to the soft grain orientation ( $m = 0.005$ ), while comparable rate sensitivities ( $m = 0.025$ ) were observed for the hard and soft grain orientations in Ti6246. Following this observation we



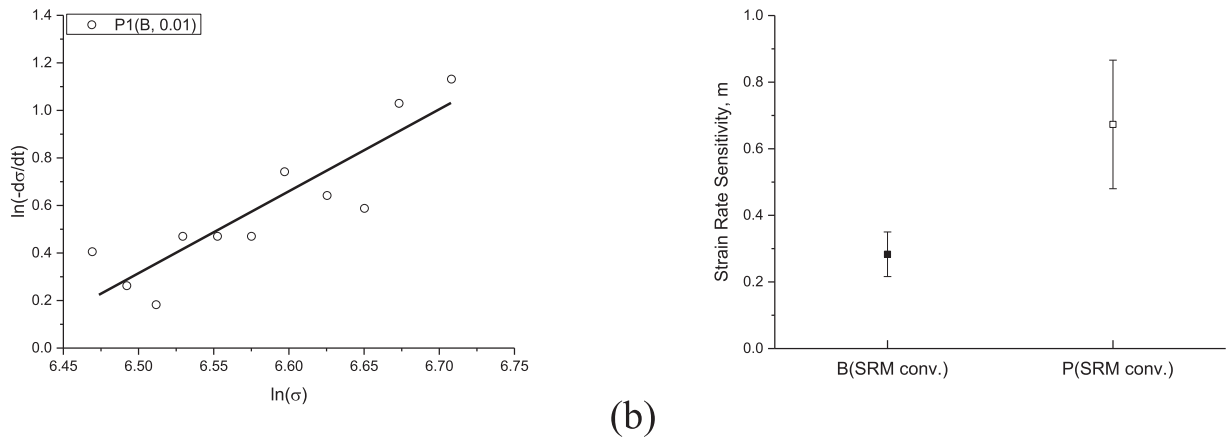
**Fig. 8.** In region 'B', (above) SEM still images captured from in-situ videos at the beginning of holding stage and (below) the associated engineering stress-strain curves: Slip bands were similarly formed in all three pillars compressed by different strain rates, but different hardening behaviours were observed. The dash lines in different colours superimposed on SEM images correspond to those on the curves, indicating at which stage the individual slip band forms. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



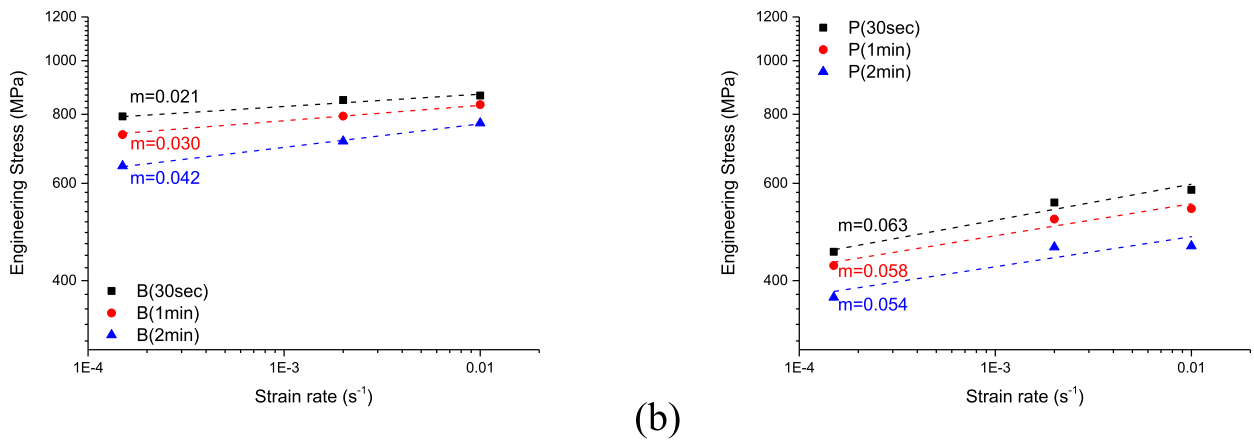
**Fig. 9.** In region 'P', (above) SEM still images captured from in-situ videos at the beginning of holding stage and (below) the associated engineering stress-strain curves: Slip bands were differently formed in each pillar, but similar hardening behaviours were observed. The dash lines in different colours superimposed on SEM images correspond to those on the curves, indicating at which stage the individual slip band forms. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 10.** Engineering stress–time curves observed in (a) region 'B' and (b) region 'P', during the holding stage (i.e. 2 min).



**Fig. 11.** (a) Stress relaxation rate vs. applied stress (log–log form) for pillar 1: The slope of the plot indicates  $1/m$  and the rest of pillars were also similarly analysed like this, (b) comparison of strain rate sensitivity between basal and prism slip systems: The conventional SRM analysis shows quite similar SRS trend but different magnitude (one order of magnitude difference) to that in the CSRM analysis.



**Fig. 12.** Relaxation stress vs. strain rate (log–log form) in (a) region 'B' and (b) region 'P': The stress values were taken at three different times (i.e. 30 s, 1 and 2 min). A slope of each fitted line indicates the strain rate sensitivity exponent,  $m$  value.

have investigated the local strain rate sensitivity of 'single  $\alpha$  phase' in dual-phase, dwell sensitive Ti6242, using *in-situ* micropillar compression technique within the present manuscript.

One of the crucial issues on micropillar compression is the experimental setup, where a contact misalignment between the

top surface of the pillar and the flat punch is likely to affect the load–displacement response and change the stress state within the pillar [33]. As slip progress the pillar accommodates misalignment and therefore this is less likely influence measurement of rate sensitivity study. The indenter system used, alignment and FIB

milling have been optimised to reduce alignment error (as evidenced through correct prediction of slip activity by Schmid factor analysis) indicating that misalignment is less than a few degrees.

The most significant observation within the present study is that, the rate sensitivity of single  $\alpha$  phase for  $a$  type prism slip activity is strikingly higher than that for  $a$  type basal slip. This trend was attained by three different SRS analyses (see section 3.4) and Fig. 13 shows the comparison of the CSRM and the newly-proposed SRM, where the conventional SRM was not included due to its higher order of magnitude. Note that this new SRM is a comparative analysis of relaxation curves so that pillars tested for individual slip systems have similar testing factors influencing the stress relaxation (e.g. elastic property of a testing machine), which are likely to be negligible. Good agreement is found between the analyses for the basal slips while there is a discrepancy in the prism slips. The main cause for this discrepancy is due to the considerable softening occurred at the strain rate of  $1.5 \times 10^{-4} \text{ s}^{-1}$  in the CSRM (see Fig. 6(b)) which increases the  $m$  value.

To the authors' knowledge, this study reports for the first time the strain rate sensitivity with respect to individual slip systems of single  $\alpha$  phase in Ti alloy. The resultant  $m$  values are 0.024–0.033 (basal) and 0.068–0.097 (prism) for the CSRM vs. 0.021–0.042 (basal) and 0.054–0.063 (prism) for the new SRM. Although numerous research on strain rate sensitivity have been performed (e.g. Refs. [21,34]), it is thought that interpreting the  $m$  value and correlating to physical phenomenon is still an open question. However, the key aspect of the present study is to explore a relative difference of SRS by fixing the pillar geometry (see Fig. 4) and testing methodology (see Fig. 1), and only altering the crystal orientation and the corresponding slip system (see Fig. 2). Careful slip trace analysis shows that the activated slip systems in regions 'B' and 'P' are confidently the  $a$  slip on the basal plane and  $a$  slip on the prism plane respectively (Fig. 5).

We can now argue that the dwell sensitive Ti6242 has different rate sensitivity for  $a$  type basal and prism slips. It is necessary to revisit our findings to figure out the fundamental mechanisms of the slip system dependent rate sensitivity. Quasistatic micropillar compression was performed in the present study, where understanding size effect and the associated mechanisms is a major research field for which this technique has been used. Several hypotheses have been introduced to elucidate the size effect with

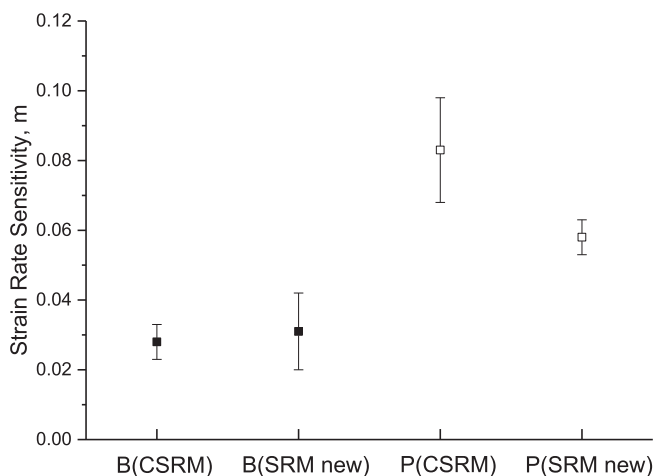
proposed mechanisms involving source exhaustion [35], source truncation [36], dislocation starvation [37] and so on. Similar to FCC and BCC single crystals HCP Ti also shows the significant size effect, resulting in a linear relationship between a flow stress and a pillar diameter with a slope of  $-0.5$  (i.e. flow stress increases with decreasing pillar size) [38]. This effect is typically important for sub-micron sized pillars and may also be for micron sized pillars. However, we have controlled this variable in this comparative rate sensitivity study of individual slip systems by using a consistent size (i.e.  $2 \mu\text{m}$ ) for all pillars and hence argue that the rate sensitivity difference is unlikely related to the size effect.

As shown in Fig. 6, yield strength ( $\sigma_Y$ ) and the associated critical resolved shear stress (CRSS) are apparently influenced by the applied strain rates. Yield points determined by proportionality limit provide that  $\sigma_Y$  & CRSS for basal slip varies between 620 & 285 MPa ( $1 \times 10^{-2} \text{ s}^{-1}$ ) and 500 & 230 MPa ( $1.5 \times 10^{-4} \text{ s}^{-1}$ ), and those for prism slip does between 470 & 230 MPa ( $1 \times 10^{-2} \text{ s}^{-1}$ ) and 400 & 200 MPa ( $1.5 \times 10^{-4} \text{ s}^{-1}$ ). It can be deduced that strength is a sampling of the 'strength vs strain rate' space, referring to the fact that strength and strain rate sensitivity are coupled and consequently this is important for time dependent deformation mechanisms.

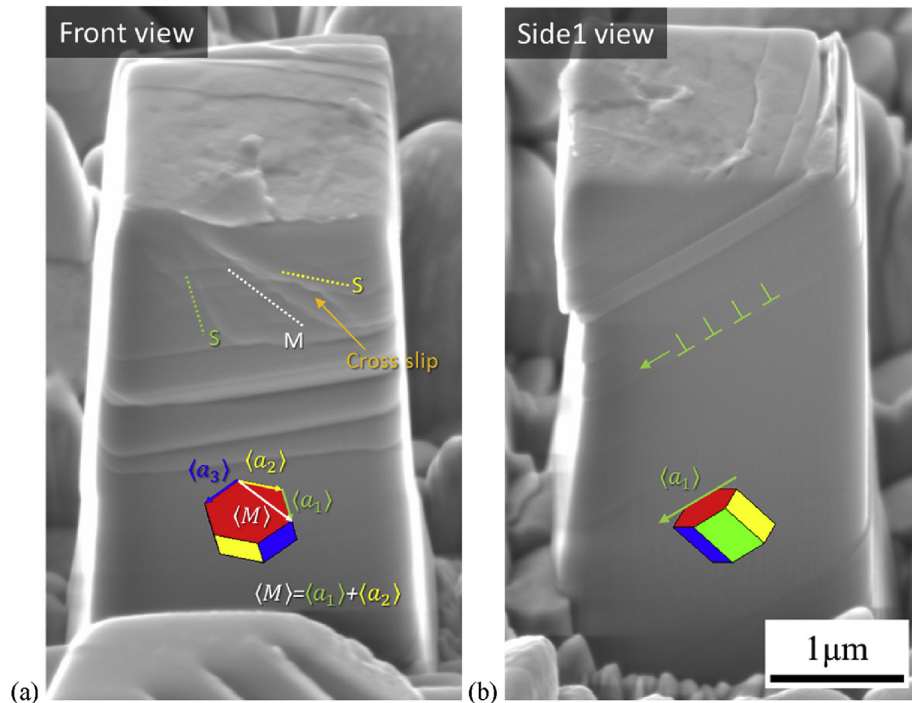
Two methods, CSRM and SRM, may involve different types of dislocation plasticity [39,40]. Fundamental rate sensitivity mechanisms for the CSRM is likely to be elucidated by dislocation hardening while a shedding of load due to room temperature creep is main driving force to evolve dislocation motion and stress relaxation in the SRM. As more experimental observations are available for the CSRM (i.e. inter-relationship between  $\sigma$ - $\epsilon$  curves, *in-situ* video and slip trace analysis) further discussion on the rate sensitivity mechanisms is carried out with the CSRM.

Stress-strain curves in Fig. 6 shows that both basal and prism slips undergo strain hardening with different hardening gradients, where the hardening is likely to be observed in small-scale micropillar of single crystals mainly due to size effects [41–44]. Still images captured during compression tests shown in Figs. 8 and 9 may have some clues elucidating the different strain hardening behaviour we observed between basal and prism slips. It is apparently seen that the steeper hardening gradient is associated with localised slip band formation in the basal slip, whereas no slip band localisation and variation of the hardening gradient were observed in the prism slip. Details of this phenomenon can be linked to the dislocation motion, which is analogised by Fig. 5.

In the prism slip, 'sharp' slip steps indicate that edge dislocations were easily moved through  $a$  direction on one prism plane. This is in disagreement with previous finding by Sun et al. [38] showing the cross slip of screw dislocations in a prism slip of single crystal titanium, probably due to the change of dislocation structure after heavy deformation (0.2–0.25 strains). On the other hand, more complicated slip bands were formed in the basal slip with 'broad' and 'wavy' slip steps. Two magnified micrographs are reproduced in Fig. 14 with inserts showing the unit cells, the associated Burgers vectors and the geometry of dislocations. It is seen from the Side 1 view that edge dislocations were glided through  $a_1$  direction on basal plane. In the Front view, 'wavy' slip bands are found near the top of the pillar, revealing that screw dislocations were formed perpendicular to  $a_1$  and  $a_2$  directions. Moreover, mixed dislocations  $M$  and cross slip (marked by the orange arrow) are observed. This demonstrates that in the basal slip there are more complex dislocation–dislocation interactions, which is likely to interrupt free movements of dislocations and make dislocation annihilation through the sample surface difficult. Consequently, increased stresses are required to activate dislocation sources and move dislocations, resulting in higher hardening gradients.



**Fig. 13.** Comparison of strain rate sensitivity between the CSRM and the newly-proposed SRM: Two methods show good agreement, though in the prism slip there is a discrepancy which mainly caused by considerable softening (see Fig. 6(b)). Note that the uncertainties were measured by standard deviation of  $m$  values which were determined using three strains for the CSRM and three times for the SRM new.



**Fig. 14.** Magnified micrographs of (a) the Front view and (b) the Side1 view from the slip trace analysis of the basal slip (see Fig. 5(a)): In (a), dot lines marked with S and M indicate screw and mixed dislocation respectively. Orange arrow points out the region where cross slip formed. In (b), pillar failure mode indicate the glide of edge dislocations through  $a_1$  direction. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

Phenomenological observation between both slips was the difference of the hardening mechanisms, though correlating this to the rate sensitivity mechanisms is yet possible within this study. The next steps could be: (i) developing rate-dependent crystal plasticity model with consideration of self-diffusion energy [45] which is known to play a prominent role in creep [46] and may differ across individual slip systems; (ii) TEM [47] or TKD analysis [48] to examine dislocation structures and verify how the rate sensitivity mechanisms is controlled by dislocation hardening mechanisms.

The rate sensitivity difference observed here is unlikely to be observed by macro-scale testing. Neeraj et al. [20] investigated the room temperature creep behaviour in single-phase Ti–6Al and dual-phase, polycrystalline Ti6242. The authors argued that abnormally low strain hardening exponents of Ti alloys are the most crucial factor affecting the room temperature creep while their SRS exponents are similar to other metals and thus have no significant effect. This results were disagreed with early works by Hatch et al. [49] and Odegard et al. [50], suggesting that high SRS of Ti alloys can rationalise the room temperature creep. There are still ongoing arguments on this low temperature creep behaviour, but given the early and present works we hypothesize that SRS at macroscopic level (i.e. structural rate sensitivity) is different from that at microscopic level (i.e. micro-intrinsic rate sensitivity). This has significant impact on dwell fatigue as our current state of knowledge suggests that dwell facets are likely to be initiated at highly localised region by pile up of either basal or prism slip on a soft-oriented grain (the Stroh's hypothesis) [5,51,52]. The presence of a rate sensitive mechanism may potentially alter the structural unit size, the effective slip length and the number of dislocation in a pile-up, required for the Stroh model of load shedding and stress amplification during dwell leading to facet formation and failure.

In this work we have exploited new micropillar testing strategies to isolate the performance of individual microstructural units

(colonies) and explored the slip system dependent rate sensitivity of single  $\alpha$  phase in complex colony-structured dual-phase Ti alloy. Complementary SEM/EBSD, FIB and *in-situ* deformation effectively enables pillar compression of target phase in preferentially orientated regions to activate target slip systems and therefore as fair a test as we can manage, given the complexity of the deformation field involved.

## 5. Conclusions

In this study, we have investigated the local strain rate sensitivity of single  $\alpha$  phase in dual-phase Ti6242 using *in-situ* micropillar compression with the aid of EBSD, SEM, FIB and Alemn timer. The rate sensitivity of targeted  $a$  type basal and prism slips was analysed using three types of methods: the constant strain rate method and conventional and newly-proposed stress relaxation methods. A significant slip system dependent rate sensitivity was observed as the prism slip activity has around 2–3 times higher  $m$  value than the basal. The nature of the difference was discussed with regards to strain hardening and further dislocation plasticity, where there are more resistance to dislocation motion and more complicated dislocation–dislocation interaction in the basal slip, resulting in slip localisation and consequently higher hardening gradient.

The observed slip system dependent rate sensitivity is likely to have a significant impact when considering the dwell fatigue and the Stroh model of load shedding and stress amplification during dwell leading to facet formation and failure.

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## Appendix. Determination of pillar size, engineering stress and strain

The dimension of each pillar (see Fig. 4) was carefully measured after fabrication. The pillar widths ( $w_t$  and  $w_b$ ) and the side length ( $l_s$ ) were measured, where the taper angle ( $\theta_t$ ) and a height ( $h$ ) are then defined by the following equations:

$$\theta_t = \sin^{-1} \left( \frac{w_b - w_t}{2l_s} \right) \quad \text{and} \quad h = l_s \cos \theta_t$$

As the pillars are flat faced, slip trace analysis can be easily performed and the slip angle ( $\theta_s$ ) can be expressed as follows:

$$\theta_s = \tan^{-1} \left( \frac{\tan \theta'}{\sin \varphi} \right)$$

where  $\theta'$  is an angle on any pillar faces after tilting and  $\varphi$  a tilting angle. Note that a tilting angle of  $52^\circ$  was predominantly used in this study.

Engineering stresses and strains were then calculated by dividing the applied load by the cross-section area at the mid-height of each pillar and the displacement by the height, respectively. Note that the uncertainties associated with the cross-sectional area and the height of the pillars were  $\sim 2\%$  and  $\sim 1\%$  respectively and the magnification of the microscope was verified using a standard sample.

## Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.actamat.2016.01.057>.

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