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1. Slip activity during low-stress cold creep deformation in a near-α titanium alloy

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Highlights of this paper:
1. For alloys with bimodal microstructure, susceptibility to cold creep increases with a coarser transformation product, i.e. coarser secondary α lath size and greater propensity to form α colonies. This is accompanied by increased slip localisation.
2. At holding stresses well below the 0.2% proof stress basal <a> slip is the dominant slip mode in the near-α titanium alloy TIMETAL®834 while when the stress level is raised to close to the proof stress prismatic <a> slip becomes dominant.
3. Deformation modelling suggests that neither the elastic anisotropy nor intragranular residual stresses can explain the early dominance of basal <a> slip.

Abstract

Near-α titanium alloys are known to be susceptible to cold dwell fatigue (CDF) debit, which has been linked to the occurrence of cold creep during high-load dwell times superimposed onto low cycle fatigue loading. In order to shed new light on the deformation mechanisms during cold dwell and to understand better the role of the microstructure, two different bimodal microstructures (fine and coarse transformation product) of TIMETAL®834 were investigated at stress levels below the 0.2% proof stress using a combination of grain orientation mapping and in-situ electron microscopy imaging. This enabled in-depth analysis of 2D slip patterns and slip system activity using High-Resolution Digital Image Correlation (HRDIC), showing that in both microstructures basal slip is initially the dominant slip mode before prismatic slip activity increases approaching the 0.2% proof stress. Comparing the two constituents in the bimodal microstructure, first slip bands are localised predominantly in primary α...
grains, indicating higher strength of secondary α colonies, particularly for finer transformation products. During 10-minute load holds at stresses below 0.2% proof stress, more plastic strain and longer connected slip traces across several grains were observed in the sample with coarse transformation product, indicating higher susceptibility to cold creep deformation.

Full-field crystal deformation modelling was utilised to determine local stresses in individual grains at the onset of plasticity and test the hypothesis that the dominance of basal slip at low-stress levels can be explained by the elastic anisotropy in Ti alloys. However, while consideration of elastic anisotropy increased resolved shear stress (RSS) values for basal slip relative to prismatic slip, it did not unambiguously explain the early activation of basal slip. Furthermore, thermal residual stresses at the crystal level, due to the anisotropy of coefficients of thermal expansion (CTE), were included in the simulation, which created a wider spread of the RSS data but did not preferentially promote high RSS values for grains well aligned for basal slip. In the absence of an unambiguous conclusion, it is hypothesised that basal slip might display lower critical resolved shear stress values than typically reported but high work hardening rates compared to prismatic slip.
1 Introduction

In near-α and α+β titanium alloys, including TIMETAL®834, creep deformation occurs readily at room temperature and stresses below the macroscopic yield point, often referred to as cold creep [1–7]. Because cold creep deformation can contribute to a reduction in cycles to failure (dwell-debit) for cold dwell fatigue (CDF) loading conditions [8], it is of interest to understand the mechanisms of cold creep deformation. CDF is cyclic loading with an additional load-hold applied at or close to the maximum applied stress [9,10], where a load-hold of 120 s is sufficient to lead to significant dwell-debits reducing the lifetime of the material by up to a factor of 18 [11]. It has been demonstrated that the susceptibility of cold creep [2,4,7] and CDF [12,13] increases with increasing primary α grain or colony size and is affected by microtexture. Crystal plasticity modelling studies suggest that these microstructural effects originate from the strong single-crystal anisotropy of α-Ti and differences in strain rate sensitivity for the different slip systems. [14] Therefore, dwell sensitivity seems to be affected by particular details of slip activity at the microstructural scale, for which there is a lack of consensus.

A number of previous studies on the slip activation at stress levels below the macroscopic yield point in different α+β titanium alloys (Ti-6Al-4V, Ti-6242, Ti-6246) revealed that basal <a> slip is the most dominant slip mode [15–20]. This dominance of basal <a> over prismatic <a> slip is unexpected, since studies in strongly textured material at slightly higher strains of 0.5% [22] suggest that prismatic <a> slip is more easily activated in α-Ti than basal <a> slip, as do more recent micro-pillar studies [14]. However, the reported critical resolved shear stress (CRSS) values for basal <a> and prismatic <a> slip in Ti-alloys cover a wide range and in some studies basal <a> slip is said to be more difficult, i.e. CRSS(Ba)>CRSS(Pr) [20,23–29] and in others easier i.e. CRSS(Ba)<CRSS(Pr) [18,19,30–34]. Tables reviewing these values have recently been published by Hémery et al. [19,21].

These discrepancies in CRSS are attributed to many factors including variations in chemical composition, in particular Al-concentration [21,24,25], the presence of α2-precipitates [32], lamellae spacing and orientation [23,26,35], and grain size [21,36]. Concerning the dominance of basal <a> slip at the onset of plasticity, some authors claim that basal <a> slip simply exhibits lower CRSS values than prismatic <a> slip [18,19,34], others have suggested that the presence of microtextured regions create stress heterogeneities due to elastic anisotropy [16] [34] that promote basal <a> slip. Thermal residual stresses, which develop during cooling from heat treatments as a result of anisotropic thermal expansion, maybe a further factor influencing early plastic deformation and activation of specific slip systems [37,38].

The work presented here aims to elucidate the effects of the microstructure on the slip activity in TIMETAL®834 during stress holds well below the yield stress (0.2% proof stress) and at room temperature. TIMETAL®834 (Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si-0.06C) is a near-α titanium-alloy developed for applications at elevated temperatures of up to 600°C, particularly for rotative-parts in aero-engine compressors [39–41]. Superior high temperature creep properties are achieved by using Nb and Mo as β-stabilisers instead of V, as e.g. in Ti-6Al-4V, as well as the addition of 0.35wt.% Si and 4wt.% Sn, while strength is increased by the formation of Ti3Al (α2) and silicide precipitates [41–47]. Typically, the best combination of high strength, creep and fatigue properties are achieved for bimodal microstructures with primary α volume fractions of ~15-20% [45,48,49].
Whilst previous work has shown that basal $<a>$ slip is the dominant slip system during initial plastic
deformation in Ti-6Al-4V, it is not clear if the same behaviour can be expected for cold creep deforma-
tion in TIMETAL®834. There is also no current experimental evidence of how the deformation is
distributed in the microstructure during cold dwell. Whereas previous work has shown that slip initiates
in the primary alpha grains, it is not known to what extent slip propagates through the secondary alpha
regions at stresses below yield, and with increasing stress level. Furthermore, although increasing the
cooling rate during processing increases the strength of the alloy by refining the secondary alpha [50],
it is not known how it affects the slip localisation and the partitioning of slip between primary and sec-
ondary alpha that appears to lead to the dwell debit seen in bimodal microstructures.

We investigate slip activation during 10-minute load-hold experiments, at three stress levels below the
0.2% proof stress, and on two different bimodal microstructures: one with coarse and one with fine
transformation product. SEM-based high-resolution digital image correlation (HRDIC) in combination
with crystallographic orientation mapping is used to accurately determine slip activity during creep
deformation across many grains, whilst also allowing to separate slip activity between primary $\alpha$ and
secondary $\alpha$ constituents [51]. Full-field polycrystalline deformation modelling is used to calculate the
resolved shear stresses of individual grains during elastic loading. This is the first study of this kind on
TIMETAL®834, the first to quantify the strain partitioning between the primary and different secondary
$\alpha$ in-situ during stress holds, and the first to critically evaluate the effects of elastic anisotropy and
thermal residual stresses on the slip activity measured using polycrystalline deformation modelling.
2 Experimental

2.1 Sample preparation

The near-α titanium alloy TIMETAL®834 was supplied by TIMET, UK. A bimodal microstructure with a primary α volume fraction of around 15% was produced by solution heat treating two blocks of material for two hours at 1015 °C, i.e. about 30 °C below the β transus temperature [52]. Subsequently, one of the blocks was cooled rapidly by oil quenching (10 – 30 K/s), leading to a fine transformation product. The other block was furnace cooled to room temperature at a rate of 1 K/s, resulting in a coarser transformation product. Subsequently, both blocks were annealed for two hours at 700 °C, which is known to lead to the precipitation of Ti₃Al (α₂), predominantly in the primary α grains [45,46].

Flat dog-bone specimens, with a gauge volume of 1 x 3 x 26 mm³ (t x w x l) were electrical discharge machined for uniaxial tensile testing. Subsequently, both sides of all samples were ground to #800 grit paper to remove deep scratches and oxides formed during machining. The samples were further ground followed by hand polishing in water-based colloidal silica solution (4:1) for 10 mins to mirror finish. To reveal the microstructure by optical microscopy, polished samples were etched with Kroll’s reagent (2 ml HF, 4 ml HNO₃, 94 ml H₂O). For mapping strain patterns by high-resolution digital image correlation (HRDIC), a gold speckle pattern was applied to the polished surface using the gold remodelling technique [47,53–55]. To produce the speckle pattern, a thin film of gold was deposited onto the sample surface, at a current of 40 mA and pressure of 0.5 mbar for 5 minutes, using an Edwards S150B sputter coater. The film was then remodelled in a water-vapour environment at 275 °C for four hours [53,55], producing isolated speckles, of which 99% have a diameter in the range of 28 to 150 nm.

2.2 Baseline characterisation

For each material condition, a 2 mm² area was characterised by means of optical bright-field microscopy. Segmentation of the primary α constituent was performed, using the image analysis software Fiji [56], based on the differences in grey-scale values between the two constituents. The ‘characterise particles’ function was chosen to determine the average size and volume fraction of primary α grains. The spacing of secondary α lamellae was calculated utilising the line intercept method for 30 secondary α colonies.

Crystallographic orientation mapping was performed using Electron Back Scattered Diffraction (EBSD) in an ‘FEI Quanta 650’ field emission gun scanning electron microscope (FEG-SEM) equipped with Oxford Instruments HKLNordlys EBSD detector at an acceleration voltage of 20 kV. An area of 250 x 250 µm² was analysed with a step size of 1 µm, which coincided with the area analysed later by HRDIC but at approximately twice the interrogation window size. A misorientation of 10° was chosen for grain boundary definition. Grains smaller than 10 µm² were not considered in any subsequent analysis.

Stress-strain curves were recorded for uniaxial tensile tests to determine the 0.2% proof stress (σ₀.2%) for both material conditions employing an Instron 5569 electromechanical tester with a 25 kN load cell.
at a constant strain rate of 0.005 1/s [57]. Strain was recorded using an axial extensometer with a
gauge length of 10 mm.

2.3 HRDIC – experimental

In-situ loading experiments were performed using a Deben 5 kN tensile stage placed inside an FEI
Quanta 650 FEG-SEM. This setup enables imaging specimens at load, avoiding possible strain rever-
sal during unloading and any possible issues with enhanced accumulation of plastic deformation due
to repeated loading and unloading [58]. Hence, this procedure was used to record images of the gold
patterned sample surface during a series of deformation steps to monitor the strain evolution during a
single loading process. As two material conditions with different yield strengths were compared, the
load steps were defined as the relative values of 70, 80 and 90% of $\sigma_{0.2}\%$ (Table 2). For each load
step, a stress hold of 10 minutes was applied. Afterwards, the stress was reduced by 5%, e.g. from 70
to 65% of $\sigma_{0.2}\%$ to avoid further plastic deformation during the imaging step, which took around 1.5
hours (Fig. 1). The strains measured in these experiments include the elastic and plastic strains in the
sample after loading and holding. Owing to the long acquisition time needed for each loading step, it
was not possible to measure the strain just after loading, and before creep occurred. To ensure that
the slip activity studied was relevant to the cold creep regime, polished samples were also loaded
using much faster optical dark field imaging. These tests showed that the plastic strain accumulation
during cold creep occurs by either further slip in the bands created during the initial loading, or by the
nucleation of new slip bands in the same grains, parallel to the slip bands already formed. These re-
results indicate that the analysis of the spatial distribution of slip and of slip activity is relevant to cold
creep deformation. The images obtained during of these tests are given in the supplementary material.

Images for the HRDIC analysis were recorded at an acceleration voltage of 10 kV with a scan speed
of 5 µs/pixel and 2 times frame averaging to reduce drift compared to a scan speed of 10 µs/pixel and
charging issues [59,60]. A Deben Centaurus photomultiplier detector PMD was utilised as it can be
manoeuvred in-between the grips of the micro-tester, allowing for a relatively low working distance of
11.7 mm. The imaged area of 250 x 250 µm² for HRDIC analysis was covered by recording 12 x 12
images (each frame being ~ 30 x 27 µm², 2048 x 1832 pixels) with an overlap of 20%, which were
then stitched together before processing using the ‘Grid/Collection stitching’ plug-in in Fiji [56,61].

2.4 HRDIC – data analysis

The displacement maps for each deformation step were computed by using LaVision’s DIC software
package DaVis version 8.4.0 [62]. The image of each load step was correlated directly to the unde-
formed sample, using an interrogation window size of 32 x 32 pixels, with no overlap between the
interrogation windows. This window size represents a good trade-off between high spatial resolution
(468 nm) and low noise [63] but provides a lower spatial resolution than typically achieved during ex-
situ experiments (147 nm [47]) while the error and noise are comparable.

Calculation of in-plane strain tensors by differentiation of the in-plane displacements determined by the
DIC software and subsequent data analysis was performed using in-house Python routines [64] and
the NumPy numerical library [65]. The deformation maps contain three components, which are the in-
plane shear strain ($\varepsilon_{xy}$), and the strain parallel ($\varepsilon_{xx}$) and perpendicular ($\varepsilon_{yy}$) to the loading direction. For
the data analysis, we used the effective shear strain ($\gamma_{\text{eff}}$) defined by equation (1), as it takes all in-plane shear components into account [53,66].

$$\gamma_{\text{eff}} = \sqrt{\left(\frac{\varepsilon_{xx} - \varepsilon_{xy}}{2}\right)^2 + \varepsilon_{xy}^2}$$  \hspace{1cm} (1) 

Image correlation of two images of the undeformed sample was used to determine the systematic error of the HRDID analysis. Between the two image acquisitions, the set-up process of the SEM was repeated without removing the sample from the microscope, as this is also not necessary during the in-situ experiment. The strain uncertainty was calculated to be approximately 0.15% for the average effective shear strain value of each deformation map.

BSE-SEM images of the undeformed samples, which are inherently perfectly aligned with the deformation maps, were used to generate separate strain maps for primary and secondary $\alpha$ constituents. A binary image of the constituents was created by selecting all primary $\alpha$ grains in the BSE-SEM images, using the ‘Freehand selection’ tool in Fiji [56], which was then used to mask out either of the two constituents in the effective shear strain maps and to calculate the average values, as well as the 99.5th percentiles.

This approach was also used to determine the constituent of each grain identified in the crystallographic orientation maps. In some cases, a primary $\alpha$ grain and an adjacent secondary $\alpha$ colony were found to have the same crystallographic orientation and therefore appeared as one grain in the orientation map. These grains were disregarded in further analysis. Therefore, the sum of primary $\alpha$ grains and secondary $\alpha$ colonies is lower than the total number of grains in Table 1.

Active slip systems were determined using slip trace analysis (STA) technique [63,67,68]. Initially, the orientation of a slip trace in the deformation maps is determined by fitting a line to it, which is then correlated to the directions of all possible slip systems based on the crystallographic orientation of the grain. The acceptance criterion for a possible slip system was $<5^\circ$ for the angle between the experimental and theoretical slip trace. Slip lines that did not match with any of the possible solutions were labelled as ‘no match’. Different slip systems may exhibit the same slip lines at the sample surface, resulting in ‘ambiguous’ solutions’ [51], in which case all possible solutions were reported. In these cases, relative displacement ratio (RDR) analysis can be used to determine the Burgers vector direction associated with the slip line [68,69]. If the Burgers vector matches with only one of the possible slip systems, an unambiguous solution can be reported. Other approaches that can be used for identifying active slip systems are the Heaviside-DIC technique [70] and a techniques that combines DIC with laser scanning confocal microscopy [71].

2.5 Polycrystal deformation modelling

Full-field polycrystal deformation modelling was used to determine local stresses in the microstructures analysed by HRDID. The CP-simulations were carried out on the Düsseldorf Advanced Material Simulation Kit (DAMASK) version 2.0.2 [72]. DAMASK utilises a Fast Fourier Transform (FFT) based approach to solving for mechanical equilibrium, which enables the direct input of the crystallographic orientation maps to define the simulation microstructure. As TIMETAL®834 has a very low $\beta$ volume
fraction, only α phase was included in the simulation and secondary α colonies were treated as single α grains [73].

The modelling work aimed to study the effects of elastic anisotropy and therefore only elastic deformation was simulated. It is well known that the deformation patterns predicted by crystal plasticity (CP) modelling at yield are different from those observed experimentally [30]. This is because slip initiation in practice occurs on a few slip bands while crystal plasticity models predict diffuse slip on several slip systems. Hence, plasticity in the model produces noticeable changes in the predicted local stress states, which are probably not representative of those in the actual material. In order to avoid any plasticity and stress redistributions in the model, the simulations were run up to very low-stress levels (1% of $\sigma_{0.2\%}$). Although this could also have been achieved by avoiding plasticity altogether in the simulations, some plasticity is needed when simulating the thermal residual stress development on cooling and therefore it was preferable to simulate plasticity but avoid its effects on loading. In the elastic regime, all local stresses scale linearly with the macroscopic stress. Therefore, the stress outputs were scaled to the stress applied at the first load step in the HRDIC experiment to enable direct comparison between experimental and modelling results. It should be kept in mind that the determined stress values can only be used for the discussion of initial slip activation, as further plastic deformation would lead to stress redistributions, which have not been considered in our simulations.

A 2.5D or quasi 3D microstructure [19,74] was created by extruding crystallographic orientation maps parallel to the plane normal. The crystallographic orientation maps were taken from the HRDIC areas, but the number of pixels was reduced by a factor of 2, from 520x520 to 260x260 pixels, to accelerate simulations and post-processing. As both the HRDIC experiment and the CP-simulations are linked to the same crystallographic orientation maps, it is trivial to link the datasets for post-processing and data analysis. The thickness of the extruded layer was 38 voxels (~36 µm), which is similar to the average grain diameter [74]. On top of the surface, a ‘buffer’ layer with a thickness of 5 voxels was added, which has a stiffness value that is around 3 orders of magnitude lower than the stiffness of titanium (Table A 1), representing the free sample surface in the HRDIC experiment [75]. On the other 5 sides, a ‘buffer’ layer of 5 voxels was added with isotropic properties and the same average stiffness as commercially pure titanium to produce the conditions of non-periodic material regions [76,77]. Except for the buffer layers, the elastic properties of commercially pure titanium were assigned to all grains (Table A 1) as they are assumed to be similar to TIMETAL®834.

The CP-simulation determined the full stress tensor at each point in the analysed area. In combination with the crystallographic orientations, resolved shear stress (RSS) values can be calculated for each slip system. Three calculation methods (equation (3)-(5)) were used to determine RSS values and the differences between them were evaluated. For the first method, the local stresses were assumed to be homogeneous throughout the sample and equal to the macroscopic stress. Therefore the effects of crystallographic orientations on stress distributions are not considered. RSS values were calculated by multiplying the applied stress ($\sigma_{xx}(applied)$) with the Schmid factor (SF) values of each grain (equation (1)).

$$\text{RSS} = \text{SF} \times \sigma_{xx}(applied)$$  \hspace{1cm} (3)
For the second method, local stresses in the loading direction ($\sigma_{xx}(\text{local})$) were multiplied with the Schmid factor (SF) of the grain, accounting for the effect of elastic anisotropy on local stresses.

$$\text{RSS} = \text{SF} \times \sigma_{xx}(\text{local}) \quad (4)$$

The third method used the full stress tensor for the calculation of RSS values, also taking into account stress components perpendicular to the loading directions and shear stresses. RSS values were determined by calculating the dot product of the slip direction ($\vec{b}$), the full stress tensor ($\sigma'$) and the slip plane normal ($\vec{n}$) (equation 5).

$$\text{RSS} = \vec{b} \cdot \sigma' \cdot \vec{n} = b_1n_1\sigma_{11} + b_1n_2\sigma_{12} + \cdots + b_3n_3\sigma_{33} \quad (5)$$

Some simulations also explored the consideration of thermal residual stresses present in the material at room temperature by setting the material to be stress-free at a temperature of 800 K, instantly cool to room temperature ($300 \, \text{K}, \Delta T = 500 \, \text{K}$) and solve the stress state in the material so that the average macroscopic stresses were equal to zero. While we used thermal expansion coefficients published by Pawar et al. [38] it should be pointed out that there is no consensus on its magnitude or direction [37,38,78–80].
3 Results

3.1 Baseline characterisation

Fig. 1a) and b) display the two bimodal microstructures that were investigated here. Due to the slower cooling rate for the coarse microstructure condition, the primary α size and volume fraction are slightly larger than for the fine microstructure condition, Table 1. The reduced cooling rate also led to an average lamellae spacing of 1.8 µm, compared to 1.1 µm for the faster cooling rate and also to wider secondary α colonies. The coarse microstructure condition displayed mainly colonies within the transformed β phase regions, whereas the fine microstructure condition exhibited a mixture of well-defined colonies as well as areas with a fine, basketweave type structure and more crystallographic variants (Fig. 1e-h).

The local texture of a material influences slip mode activity [81]. While Schmid factor calculations can only be an approximation in a polycrystalline aggregate, due to grain interactions driven by elastic and plastic anisotropy, the Schmid factor distributions shown in Fig. 2 still provide some indications in terms of how comparable the two microstructures are regarding local textures and which <a> type slip might be more likely. For both microstructures, the frequencies of Schmid factors larger than 0.45 is higher for prismatic <a> than for basal <a> slip (Fig. 2a&b) suggesting preferential activation of prismatic <a> slip in both cases if the CRSS for both slip systems is considered to be similar.

As expected, uniaxial tensile testing revealed a higher 0.2% proof stress (σ0.2%) value for the fine microstructure condition (934 MPa) compared to the coarse microstructure condition (905 MPa). These values were used to calculate the load steps for the in-situ loading experiment as a percentage of σ0.2% (Fig. 3, Table 2).

3.2 HRDIC results

3.2.1 Strain localisation

Since the images for the HRDIC analysis were acquired while being under load, the deformation maps show combined values of elastic and plastic strain. The amount of elastic strain has been estimated based on the modulus and the applied stress (Table 2). For the strain maps presented in Fig. 4a-e the difference between the theoretical elastic strain and the average of the recorded HRDIC maps are smaller than the uncertainty of the HRDIC analysis (Table 2). In these cases, the average plastic strains are too low to be quantified. Still, the 99th percentiles, see Table 2, can be used for all maps to analyse the heterogeneity of the strain distribution, as this parameter is sensitive to the local strain values in the slip bands. Fig. 4g and h highlight any grains that are not well aligned for either prismatic or basal <a> type slip, showing that the region of the fine microstructure does indeed show a few grains that display low Schmid factors for <a> type slip. This was not observed for the region in the coarse microstructure.

Both samples exhibit plastic deformation in the form of discrete slip bands at 70% of σ0.2% (Fig. 4a and b)), with all the slip bands being intragranular, located mainly in primary α grains and their length being equal to or less than the grain size in the EBSD maps. The slip band length, therefore, tends to be
longer in the coarser microstructure condition due to its larger grains. With increasing applied load, strain values in existing slip bands increase, new slip bands develop, and slip is activated in more grains (Fig. 4c-f). Table 2 and Fig. 5c and d show significantly higher values of the 99.5% percentile effective shear strain for the coarse microstructure compared to the fine microstructure condition when loaded to 80% and 90% of $\sigma_{0.2\%}$ highlighting a higher degree of slip localisation. For increasing average plastic strains, diffuse areas of slip start to develop in both conditions, particularly in the transformed $\beta$ phase, as the spacing between slip bands becomes narrower and gets close to the resolution limit (Fig. 5e-h). At 80% and 90% of $\sigma_{0.2\%}$, sharp slip along/near grain boundaries can be observed in a few areas in the coarse microstructure. Despite the lower absolute applied stress, the coarse microstructure condition exhibits higher average strain values and strain localisation at all load steps. For example, in the coarse microstructure condition around 0.9% plastic strain accumulated after 10 minutes holding at 90% of $\sigma_{0.2\%}$, indicating high susceptibility to cold creep. For the same loading condition, no average plastic strain accumulation was observed for fine microstructure.

### 3.2.2 Slip system activity

Slip trace analysis (Fig. 6a,b) shows that for both microstructures basal <a>-type slip is the dominant slip system at the first load step. At this load-step, the fine microstructure also displays some pyramidal but no prismatic <a> slip, while the coarse microstructure condition shows the opposite behaviour. It should be noted that there are ambiguous solutions for both microstructures. Hence, no-slip mode can be completely excluded based on this analysis. Interestingly, the fraction of pyramidal slip is always higher in the fine microstructure than in the coarse microstructure condition. With increasing load, the number of new grains found to deform by basal <a> slip reduces, while the number of grains deforming by the other two slip modes increases; hence the relative fraction of basal <a> slip decreases and the fraction of prismatic <a> slip, in particular, increases. At 90% of $\sigma_{0.2\%}$, the fraction of basal <a> and prismatic <a> slip is of a similar magnitude. It is also worth noting that in both microstructures at 70% and 80% of $\sigma_{0.2\%}$, no prismatic <a> slip was observed in primary $\alpha$ grains, while at 90% of $\sigma_{0.2\%}$ prismatic <a> slip was observed in both constituents.

### 3.2.3 Primary $\alpha$ grains and secondary $\alpha$ colonies

The fractions of deformed primary $\alpha$ grains and secondary $\alpha$ colonies are given as relative values weighted by area in Fig. 6c&d. Grains were considered as ‘deformed’ when at least one slip line was observed. For all loading steps and both microstructures, the fraction of deformed primary $\alpha$ grains is higher than the fraction of deformed secondary $\alpha$ colonies. This difference is more pronounced for the fine microstructure. Furthermore, the number of activated grains is always greater for the coarse microstructure condition. While the majority of primary $\alpha$ grains and secondary $\alpha$ colonies in the coarse microstructure were deformed plastically at 90% of $\sigma_{0.2\%}$, some primary $\alpha$ grains and a larger number of secondary $\alpha$ colonies stayed undeformed in the fine microstructure despite having high Schmid factors (> 0.4) for <a>-type slip.

To further investigate the slip trace orientation within secondary $\alpha$ lamellae a frequency distribution plot of the angle between slip trace and long direction of secondary $\alpha$ lamellae was generated. There is no clear correlation between slip traces angle and projected lamellae alignment. Angles between
slip traces and lamellae in the complete range of 0° to 90° were observed (Fig. A 3). In the fine micro-
structure, the average effective shear strain values, as well as the 99.5th percentiles, were identical for
both constituents at all load steps (Fig. 5a&c). Potential differences in strain distribution may not be
quantified for the fine microstructure, due to the low overall strain values. The average effective shear
strain values were also identical for the coarse microstructure (Fig. 5b). However, the 99.5th percentile
values (Fig. 5d) were higher in the primary $\alpha$ grains than in the secondary $\alpha$ colonies, which is particu-
larly apparent at 90% of $\sigma_{0.2}$%. For both material conditions, the segmented strain maps at the 90% of
$\sigma_{0.2}$% load step (Fig. 5e-h) show that plastic deformation in the primary $\alpha$ grains is mainly in the form of
discrete slip bands, while for secondary $\alpha$ lamellae, a combination of discrete slip bands and diffuse
slip can be observed. The coarse microstructure condition also displays wider slip trace spacing in the
primary $\alpha$ grains than in secondary $\alpha$ colonies.

3.3 Crystal plasticity modelling – RSS analysis

Fig. 7 presents calculated RSS maps utilising the three different methods described earlier. When
considering the first method using Schmid factors and assuming the same stress at each point in the
sample (equation 3), constant RSS values are calculated for each grain as there are no variations in
local stresses, Fig. 7a&b. As RSS values are linearly related to the Schmid factor values, average
RSS values are higher for prismatic $<a>$ than basal $<a>$ slip, with the same ratio between the slip sys-
tems as for the average SF values (Fig. 2a&b, Table 3). The differences in the frequency distributions
of the RSS values (Fig. 8a & b) and the Schmid factor values (Fig.2a&b) only arises from the different
bin-sizes.

In comparison, when elastic anisotropy and local stresses are considered (Fig. 7c&d), variations of
RSS values within grains are introduced. The average standard deviation of RSS values within single
grains is 5 MPa for both slip systems and microstructures. Locally, the relative changes in the RSS
maps are identical for all slip systems, as only the local stresses, but not the Schmid factor values,
have changed. The frequency distributions of RSS values (Fig. 8c&d) are less scattered for higher
than average values compared to the initial approach.

For calculations of RSS values based on the full stress tensor (equation 5), the RSS frequency distri-
butions appear less scattered for the complete range (Fig. 8e&f) and the spread of RSS values within
grains increases further (fine microstructure: 9 MPa; coarse microstructure: 8 MPa). The local changes
are different for basal $<a>$ and prismatic $<a>$ slip. In large parts of the analysed area, RSS values for
basal slip increase or at least stay constant, while RSS values for prismatic slip decrease in many
areas (Fig. 7e&f) compared to the calculations assuming homogeneous axial stresses. This brings the
average RSS values of both slip systems further together. The highest RSS values (95th percentile)
are similar for both slip systems in the fine microstructure condition, while for the coarse microstructure
condition, the maximum RSS values for basal $<a>$ slip are 27 MPa higher than for prismatic $<a>$ slip
(Table 3). Overall, using more detailed calculations reduces the difference in average RSS values
between basal $<a>$ and prismatic $<a>$ slip, and further increases the spread of RSS values within
grains (Table 3). Regarding the 95th percentile, RSS values increase for basal $<a>$ slip and decrease
for prismatic $<a>$ slip.
3.4 Crystal plasticity – correlation to experimental data

The average RSS values of each grain, based on the simulations using the full stress tensor, are presented in respect of their orientation in IPF-plots, Fig. 9a,b,e&f. To highlight regions of the highest Schmid factors for the respective slip modes within the IPF-plot, contour lines have been added. In addition, Fig. 9c, d highlight the grains that do display basal <a> slip traces after loading to 70% of \( \sigma_{0.2\%} \).

In terms of grain averages, the maximum RSS values are approximately 20 MPa (fine microstructure) to 30 MPa (coarse microstructure) higher for basal <a> than for prismatic <a> slip. Generally, RSS values appear to be positively correlated to Schmid factor values, for both material conditions and both slip systems. Fig. 9c&d shows that at 70% of \( \sigma_{0.2\%} \) the declination angles from the loading direction of all deformed grains in the fine microstructure are distributed symmetrically around 45°, with declination angles ranging from 39° to 57°. For the coarse microstructure, a shift towards the 0001 pole is observed. The deformed grains have declination angles as low as 26°, correlating to a Schmid factor of approximately 0.38.

3.5 Effect of thermal residual stresses

Fig. 8a&b show the frequency distribution of the calculated thermal residual stresses along the c-axis for both material conditions. On average there is compressive stress of just over 10 MPa along the c-axis for both microstructure conditions. Compressive residual stresses parallel to the c-axis were expected, as the thermal expansion coefficient is lower in this direction [37]. However, there are grains with compressive as well as tensile stresses along the c-axis, while there are also large numbers of grains that have no or low tensile stresses along the c-axis. For the simulated temperature difference \( (\Delta T = 500 \text{ K}) \), compressive stresses reach up to 125 MPa, around 50 MPa higher than the calculated maximum tensile stresses.

In Fig. 8c&d, the frequency distributions of basal and prismatic RSS values have been plotted for a simulation combining thermal residual stresses (\( \Delta T = 500 \text{ K} \)) and uniaxial loading (70% of \( \sigma_{0.2\%} \)). As the thermal residual stresses along the c-axis can be of either nature, there is no uniform change in RSS values and local RSS values might increase or decrease, leading to a broadening of the main peak at around 275 to 300 MPa. As a result, maximum RSS values, particularly for prismatic <a> slip, are higher in Fig. 8c&d compared to the calculations that do not consider thermal residual stresses, Fig.9e&f. Overall, the average RSS values for prismatic <a> slip decrease slightly, whilst almost no change is observed for basal <a> slip.
4 Discussion

4.1 Effect of microstructure on strain localisation

By separating the transformed β phase, i.e. secondary α, from the primary α grains it was possible to identify and quantify the strain localisation in the two constituents. It was observed that the initial slip traces form more readily in primary α grains, implying that the transformed β phase has higher strength, particularly for the material with a finer transformation product. Finer secondary α leads to a stronger Hall-Petch type strengthening [82,83] and it is also likely to further constrain the deformation of the primary α constituent, resulting in higher macroscopic strength. It is interesting that in the case of the coarse microstructure condition, primary α grains and secondary α colonies are of a similar size. Due to the higher Al concentration and presence of α₂-precipitates in primary α, due to the element partitioning effect, one might expect primary α grains to have a higher strength than secondary α colonies [21,24,43,47]. However, we observe the deformation occurring first in primary alpha, suggesting that the α/β transformed structure, despite the low volume fraction of β-phase and often being made up of similarly oriented laths, achieves additional strengthening from the presence of thin β ligaments, presumably by hindering the transfer of slip across α lath boundaries [84] [85].

In primary α grains, strain was observed to be more localised in discrete slip bands compared to the more diffuse slip bands observed in the secondary α colonies, particularly in the coarse microstructure, which could be explained by enhanced α₂ precipitation in primary α grains, which is known to promote slip planarity [47,82,86]. Lunt et al. reported that reduced levels of α₂ in the transformed β phase results in more diffuse and wavy slip in secondary α, which agrees well with the current observations [47].

4.2 Cold creep susceptibility

In the present case, the cold creep susceptibility is higher for the sample with the coarse transformation product compared to the fine transformation product when subjected to a 10-minute load-hold at stress levels below the 0.2% proof stress. This has been expected, due to the smaller structural unit size in fine transformation products, as seen in Fig. 1e-h, comparably to literature that correlated smaller grain size to lower susceptibility to cold creep [2,4,7,13].

As demonstrated in Fig.6c-d, in the case of the fine microstructure condition, slip at low-stress levels is localised mainly in primary α grains, which is to be expected as they are considerably larger than any single secondary α lamellae. Furthermore, the primary α grains are separated from each other by plastically undeformed secondary α, preventing the link-up/transfer of slip between grains and limiting the maximum slip length to the size of single primary α grains. This will result in fewer dislocation pile-up at grain boundaries and thus reduces local stress concentrations [8,87]. On the other hand, in the coarser transformation product, slip is activated readily in both constituents even at the low-stress
levels. For the coarse microstructure condition, the slightly larger grain size and higher volume fraction of primary α grains compared to the fine microstructure condition increase the probability of neighbouring primary α grains being connected. If these grains have similar crystallographic orientations, the transfer of slip and formation of interconnected slip bands is more likely, as has previously reported by Echlin et al. [88]. In contrast, a shorter distance between obstacles will result in reduced creep, fitting well with the observation that a finer microstructure, where distances between phase boundaries are shorter, exhibits a lower creep rate.

### 4.3 Slip activation mechanisms below the 0.2% proof stress

The experimental work has shown that for both material conditions, basal <a> slip was the dominant slip mode at low-stress levels (70 and 80% of 0.2%) under cold creep loading conditions. Similar observations have been reported in the literature for different near-α and α+β Ti-alloys, in particular Ti-6Al-4V, Ti-6242 and Ti-6246 [18,32,88–90]. While local texture strongly influences strain localisation at higher strain levels, the initial activation of slip systems is expected to be mainly influenced by the orientation of each grain and not the local grain neighbourhood [31,34]. With increasing stress levels, but still below 0.2%, both microstructure conditions have shown that prismatic <a> slip starts to become the dominant slip mode. This is in good agreement with previous studies where deformation studies well beyond the proof stress have always shown dominance of prismatic <a> slip [29,91]. It is noticeable that the sample with the finer transformation product exhibits a higher frequency of pyramidal slip at all load steps, with most of it being localised in the transformed β phase. Due to the higher CRSS value, pyramidal slip would be expected to be found in hard grains, respectively grains in which activation of basal or prismatic slip is not easily possible. However, grains in which <c+a> type slip is localised are at various orientations, indicating that activation of pyramidal slip is not only related to the magnitude of the RSS value, but also the stress state in the grains. Note that the slip trace analysis cannot distinguish between pyramidal <a> and <c+a> slip. RDR technique would generally be able to distinguish the two slip systems. However, this was not possible in this specific case due to low strain magnitude and small number of slip lines at the low applied stress levels. However, as pyramidal <a> is very rare, it is assumed here to be pyramidal <c+a> slip. As the fine transformation product displays no activation of slip during the early loading stage, high stresses are achieved in this constituent making activation of pyramidal <c+a> slip, which has a higher CRSS value [87] than basal and prismatic <a> slip, more likely. In addition, the fine transformation product provides a hardening increment that increases the effective CRSS values in polycrystals for all slip systems equally and therefore reduces the relative difference between them [92].

**Hypothesis 1:** The elastic anisotropy of the α crystal structure is responsible for early basal <a> slip:

A possible hypothesis to explain the prevalence of basal <a> slip during the onset of plasticity is that the elastic strain heterogeneity dominates over plastic anisotropy and that the intrinsic c-axis inclina-
tion of grains well aligned to basal \langle a \rangle slip towards the loading direction results in comparatively high stresses in such grains during tensile loading. This idea is in principle supported in Fig. 9d for the coarse microstructure condition where grains with evidence of early basal \langle a \rangle slip are not evenly distributed around the highest Schmid factor value but instead the distribution is tilted towards the 0001 pole. However, the same is not observed for the fine microstructure condition in Fig. 9c. Equally, the CP-simulations did not provide unambiguous support for the hypothesis. While the consideration of elastic anisotropy increased average RSS values for basal \langle a \rangle slip relative to the values for prismatic \langle a \rangle slip, this change was small, and the average values of the whole region of interest were still highest for prismatic \langle a \rangle slip. Regarding the highest, local RSS values (using the 95th percentile) the picture changed with values for basal \langle a \rangle slip being higher for the coarse microstructure condition, but RSS values of the two slip systems were almost identical for the fine microstructure condition. Regardless of the grain averaged RSS values, the maximum values for basal \langle a \rangle slip were higher than for prismatic \langle a \rangle slip in both microstructures. However, the studied regions contain primary \alpha grains well aligned for prismatic \langle a \rangle slip that did not show any signs of plasticity, despite having RSS values higher than the grains that deformed early by basal \langle a \rangle slip.

A further step was taken by simulating the thermal residual stresses assuming a temperature difference of 500 K, not considering the temperature dependence of the thermal expansion coefficients [37] and not allowing for stress relaxations during cooling. While the magnitude of the thermal residual stresses is an overestimation [37], the results clearly show that the thermal residual stresses increase RSS values for prismatic \langle a \rangle slip relative to basal \langle a \rangle slip, which stands in contrast to the observation of basal \langle a \rangle slip being activated before prismatic \langle a \rangle slip at the onset of plasticity.

To further explore the effect of the declination angle between the loading direction and c-axis, Fig. 11 is presented. It clearly shows that due to elastic anisotropy, axial stresses in the loading direction indeed increase for lower declination angles and are up to 20% higher than the macroscopically applied stress. Based on the higher axial stresses in grains well aligned for basal \langle a \rangle slip, the increase of the calculated RSS values for basal \langle a \rangle slip becomes apparent when considering elastic anisotropy. It also explains the principle that grains deformed by basal \langle a \rangle slip in the coarse microstructure on average have declination angles smaller than 45° (Fig. 9d). On the other hand, this effect can again not be observed for the fine microstructure condition. Due to the fine morphology and high strength of the transformed β phase, deformation is almost entirely confined to primary α grains. At the same time, only one primary α grain and otherwise secondary α colonies had declination angles in the range that resulted in basal \langle a \rangle slip activity in the coarse microstructure. Therefore, there is the possibility that the fine microstructure condition lacked suitably oriented primary α grains in the analysed region. There was one primary α grain that had its c-axis almost parallel to the loading direction, though the basal slip related Schmid factor was low for this particular case. Overall, the effects of elastic anisotropy are clearly visible; however, the data and analysis presented here do not unambiguously explain
the dominance of basal \( \langle a \rangle \) slip during the onset of plasticity while assuming similar CRSS values for basal and prismatic \( \langle a \rangle \) slip.

**Hypothesis 2: Basal \( \langle a \rangle \) slip is more likely to result in out-of-plane shear than prismatic \( \langle a \rangle \) slip:**
Considering that the work is purely based on surface observations, it is also interesting to explore the direction of shear in respect to the surface. We assume that slip at a free surface is easier to initiate than in the bulk material, as there is no constraint in the out-of-plane direction, making slip easier for Burgers vectors with a larger out-of-plane component. Fig. 10 shows the frequency distribution for the angle between Burgers vectors and sample surface of grains deformed by basal slip (23 grains) in the material with coarse microstructure together with all grains that have RSS values higher than the calculated average value. This translates to an additional 99 grains well aligned for basal \( \langle a \rangle \) slip and 97 grains well aligned for prismatic \( \langle a \rangle \) slip. Firstly, for grains well aligned for basal \( \langle a \rangle \) slip, the associated Burgers vectors have a larger out-of-plane component than for grains well aligned for prismatic \( \langle a \rangle \) slip. Secondly, grains that have deformed by basal \( \langle a \rangle \) slip tend to have a greater out-of-plane Burgers vector direction than grains well aligned for basal slip, which have not deformed. As a maximum Schmid factor for basal \( \langle a \rangle \) slip is achieved for grains with a declination angle of 45° one would expect an equal distribution around this angle for grains deformed by basal \( \langle a \rangle \) slip. It might be assumed that as plasticity evolves that the out-of-plane components become less important though this would not explain the dominance of prismatic \( \langle a \rangle \) slip at this stage if one assumes a relatively equal CRSS value for the two \( \langle a \rangle \) slip systems.

**Hypothesis 3: CRSS of basal \( \langle a \rangle \) slip is lower than CRSS of prismatic \( \langle a \rangle \) slip**
A number of recent studies have reported CRSS values for basal being lower than for prismatic \( \langle a \rangle \) slip [18,30–32]. However, a lower CRSS value for basal \( \langle a \rangle \) slip does not explain the changeover from dominant basal to prismatic \( \langle a \rangle \) slip as plasticity proceeds. Here the work by Hutchinson et al. might provide an explanation [92]. Firstly, their theoretical consideration concludes that CRSS values recorded on single crystals do not directly reflect the applied shear stresses that are necessary to activate different slip modes in polycrystalline materials. They state that most hardening contributions are not proportional to the CRSSs but are additional to them. Therefore, if basal \( \langle a \rangle \) slip is activated due to having the lowest CRSS value, the relative difference of the CRSS values of the basal and prismatic \( \langle a \rangle \) slip would decrease in a polycrystalline material with increasing strain levels and the fraction of all slip systems would converge.

Complementary to this argument, it is interesting to note that higher work hardening rates have been reported for basal \( \langle a \rangle \) slip compared to prismatic \( \langle a \rangle \) slip [93]. Such increased hardening rate for the basal \( \langle a \rangle \) slip system might be due to cross-slip onto prismatic and pyramidal planes, resulting in complex interactions between dislocations and reducing dislocation mobility [94]. Therefore, after the initial dominance of basal \( \langle a \rangle \) slip, the accumulative CRSS and large hardening contribution may re-
duce basal \(<a>\) slip activity at the expense of prismatic \(<a>\) slip activity. Besides, recent work on exploring strain rate sensitivity of individual slip systems in Ti has highlighted that basal \(<a>\) slip exhibits a greater strain rate sensitivity than prismatic \(<a>\) slip [85,93,95]. As strain rate sensitivity and strain hardening are closely linked, these observations further confirm that basal \(<a>\) slip strain hardens more easily than prismatic \(<a>\) slip.

**Summary of hypotheses**

While during this study, hypothesis 1 seemed initially the most likely, only the coarse microstructure condition provided support for it. There was evidence that grains well aligned for basal \(<a>\) slip tend to display a greater out-of-plane shear strain component than grains well aligned for prismatic \(<a>\) slip in the present case. However, the clear change of slip system dominance as plasticity proceeds might be difficult to explain with hypothesis 2. This leaves hypothesis 3 as a possible candidate highlighting the issue around the uncertainty of CRSS values for basal and prismatic \(<a>\) slip.

**4.4 Implications**

The work has demonstrated that basal \(<a>\) slip is the dominant slip mode in the near-\(\alpha\) Ti-alloy studied here for stresses below macroscopic yield and under cold creep loading conditions. It seems likely that this is an observation generally applicable to Ti-alloys since it has been observed previously for other alloys [15–20]. It highlights the importance basal \(<a>\) slip may have for deformation mechanisms associated with triggering CDF. Furthermore, transformed \(\beta\) phase regions obtained in a bimodal microstructure can effectively reduce the slip length that develops during the onset of plasticity if they are sufficiently strengthened by having a fine lath structure and a basketweave structure.

Recent studies on CDF have reported that the normal of facets formed during CDF loading is tilted approximately 30° from the loading direction [96], while in earlier work the plane normal of facets were reported to be parallel to the loading direction [10,97]. Interestingly, we have observed early activation of basal \(<a>\) slip in grains with a declination angle of the basal plane normal as low as 26° in respect to the loading direction. Generally, facets have been reported close to parallel to the (0002)-plane [10,97,98]. Hence, a hypothesis to develop from the work is that the damage associated with the shear along basal planes, that have a normal comparatively parallel to the loading direction, might be enabling decohesion and facet formation [99]. It seems that such a process would not require neighbouring hard grains or any ‘rogue grain’ combinations [8,100,101], which are often considered to be a requirement for load shedding and CDF [102]. Finally, these slip bands have formed during initial loading and load hold, in the absence of cyclic loading indicating that the location of facets might be predefined by the initial, local plastic deformation during the first load cycle, while cyclic loading will be required for opening the facets and crack propagation.
5 Conclusion

The present work has investigated the deformation mechanisms during cold creep deformation for applied stresses below the 0.2% proof stress for the near-\(\alpha\) Ti-alloy TIMETAL®834. It has been shown that secondary \(\alpha\) colonies, particularly for fine transformation products, are stronger than primary \(\alpha\) grains and that slip is more localised in discrete slip bands in primary \(\alpha\) grains. Finer transformation product constrains plastic deformation mainly to primary \(\alpha\) grains and reduces the slip length, leading to lower susceptibility to cold creep deformation in material with fine transformation product.

For cold creep loading conditions, basal \(<\alpha>\) slip is the dominant slip mode during initial plastic deformation, independently of the material condition, suggesting the importance of this slip mode on the deformation mechanisms associated with cold dwell fatigue.

The effects of the elastic anisotropy of \(\alpha\)-Ti and the anisotropy of the thermal expansion coefficient on the elastic strain distribution and slip activity were evaluated using polycrystalline deformation modelling. It was demonstrated that the consideration of elastic anisotropy increases RSS for basal slip compared to the values for prismatic slip, but none of the results of the simulations unambiguously explained the dominance of basal \(<\alpha>\) slip at low applied stresses. Other hypotheses were explored highlighting the need to create greater confidence in the relative CRSS values for basal \(<\alpha>\) and prismatic \(<\alpha>\) slip considering the contradicting data reported in the literature.

Acknowledgement

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Figures

Fig. 1: (a & b) Optical micrographs of the microstructure and (c & d) crystallographic orientation maps for material with (a & c) fine and (b & d) coarse microstructure. (e - f) Area containing transformed β phase at higher magnification, displayed with (e & f) Euler colours and (g & h) band contrast.
Fig. 2: Relative frequency distribution of (a & b) Schmid factors for basal (black) and prismatic (red) slip, and (c & d) angles between c-axis and loading direction for all grains in the HRDIC area.

Fig. 3: (a) The solid lines show stress-strain curves for material with fine (black) and coarse (red) microstructure. The absolute stresses relating to 70, 80 and 90% of the 0.2% proof stress are indicated by dashed lines. (b) The schematic shows the load profile of the DIC experiment, indicating the stress-holding and imaging steps.
**Fig. 4:** Maps showing effective shear strain maps at 70% (a & b), 80% (c & d) and 90% (e & f) of the 0.2% proof stress, and (g & h) maximum Schmid factors obtained from EBSD (basal and prismatic slip) for both materials with fine (a, c, e, g) and coarse (b, d, f, h) microstructure.
Fig. 5: Plots of average values (a & b) and the 99.5th percentile (c & d) of effective shear strain at all load steps, separately for the primary $\alpha$ and secondary $\alpha$ constituents. The strain maps (e - h) show normalised effective shear strain values, separately for the primary $\alpha$ (e & f) and secondary $\alpha$ (g & h) constituents.
Fig. 6: (a & b) Relative frequencies of, both, unambiguously and ambiguously identified slip systems as a fraction of all observed slip systems. (c & d) Relative frequencies of primary α grains (magenta) and secondary α colonies (light blue) that have plastically deformed in the HRDIC experiment at the specific load steps for (a & c) fine and (b & d) coarse microstructure.
Fig. 5: Maps of resolved shear stress RSS on (a, c & e) basal and (b, d & f) prismatic slip systems for the material with fine microstructure. The simulations are for loading to 70% of 0.2% proof stress of initially stress-free material. The RSS values have been calculated according to three different approaches. Additionally, maps showing the difference between the calculation methods are displayed.
Fig. 6: Histograms of resolved shear stresses (RSS) from crystal plasticity simulations, both for basal and prismatic slip. RSS values have been calculated (a & b) from SF – homogeneous stress, (c & d) from SF – local axial stresses, and (e & f) with full stress tensor. The simulations are for loading to 70% of 0.2% proof stress of initially stress-free material, both for (a, c & e) fine and (b, d & f) coarse microstructure.
**Fig. 7**: Resolved Shear Stress (RSS) values for (a – d) basal and (e & f) prismatic slip for (a, c & e) fine microstructure and (b, d & f) coarse microstructure. (c & d) Grains in which basal slip has been identified at 70% of \( \sigma_{0.2\%} \) in the HRDIC experiment are plotted separately.
Fig. 8: Results from CP-simulation: (a & b) Relative frequency distribution of thermal residual stress along the c-axis, for cooling from 800 K to room temperature. (c & d) Relative frequency distribution of RSS values for material with thermal residual stresses loaded to 70% of 0.2% proof stress.
**Fig. 9**: Grain average axial stresses normalised by the macroscopic axial stress for **a**) fine microstructure and **b**) Coarse microstructure.

**Fig. 10**: Relative frequency distributions of angles between Burgers vectors and the free sample surface for grains with RSS values higher than the mean value of the complete sample, regarding basal and prismatic slip separately, as well as the grains which unambiguously deformed by basal slip in the
HRDIC experiment in the ‘coarse microstructure’. Burgers vectors have been determined using the RDR technique.

Fig. 11: (a) BSE-SEM image of coarse microstructure in undeformed condition and (b) effective shear strain map at 90% of $\sigma_{0.2\%}$, which is a sub-set of Fig. 4 (f).
8 Tables

Table 1: Lamella spacing, primary $\alpha$ volume fraction and the average size of primary $\alpha$ grains of the complete sample determined by optical microscopy. The number and size of primary $\alpha$ grains and secondary $\alpha$ colonies in the DIC area is based on crystallographic orientation mapping.

<table>
<thead>
<tr>
<th></th>
<th>Fine microstructure</th>
<th>Coarse microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Grain size analysis using optical microscopy</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lamella spacing</td>
<td>$1.1 \pm 0.3 \mu m$</td>
<td>$1.8 \pm 0.4 \mu m$</td>
</tr>
<tr>
<td>Volume fraction of $\alpha$(P)</td>
<td>$18.2 \pm 0.7 %$</td>
<td>$23.1 \pm 0.3 %$</td>
</tr>
<tr>
<td>Average grain size of $\alpha$(P)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>area diameter *</td>
<td>$379 \pm 18 \mu m^2$</td>
<td>$469 \pm 11 \mu m^2$</td>
</tr>
<tr>
<td></td>
<td>$22 \pm 1 \mu m$</td>
<td>$25 \pm 1 \mu m$</td>
</tr>
</tbody>
</table>

**Summary of grain statistics in DIC area**

<table>
<thead>
<tr>
<th></th>
<th>Fine microstructure</th>
<th>Coarse microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>total number of grains</td>
<td>298</td>
<td>175</td>
</tr>
<tr>
<td>average grain size</td>
<td>$199 \mu m^3$</td>
<td>$309 \mu m^3$</td>
</tr>
<tr>
<td>number of $\alpha$(P) grains</td>
<td>25</td>
<td>21</td>
</tr>
<tr>
<td>number of $\alpha$(S) colonies</td>
<td>235</td>
<td>128</td>
</tr>
</tbody>
</table>

$\alpha$(P) → primary $\alpha$

$\alpha$(S) → secondary $\alpha$

* assuming spherical grains

Table 2: Summary of results of the mechanical baseline characterisation, the applied load steps in the HRDIC experiment, as well as the mean values and 99th percentiles of the effective shear strain determined by HRDIC. The elastic strain response has been calculated based on the applied stress in each load step and Young’s modulus of the bulk material.
Table 3: RSS values, mean and 95th percentile values of the complete maps, for basal and prismatic slip, using three different calculation methods.

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<tr>
<th>Microstructure</th>
<th>Yield stress $\sigma_y$ [MPa]</th>
<th>Young’s modulus [GPa]</th>
<th>Load step [% of $\sigma_y$]</th>
<th>Load $[\text{MPa}]$</th>
<th>Elastic strain [%]</th>
<th>Effective shear strain Mean [%]</th>
<th>99th PCTL [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine</td>
<td>934</td>
<td>117.2 ± 2.3</td>
<td>70</td>
<td>654</td>
<td>0.5</td>
<td>0.4</td>
<td>0.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>80</td>
<td>747</td>
<td>0.6</td>
<td>0.5</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td>841</td>
<td>0.7</td>
<td>0.8</td>
<td>3.1</td>
</tr>
<tr>
<td>Coarse</td>
<td>905</td>
<td>117.2 ± 2.3</td>
<td>70</td>
<td>634</td>
<td>0.5</td>
<td>0.5</td>
<td>1.0</td>
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<tr>
<td></td>
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<td>80</td>
<td>724</td>
<td>0.6</td>
<td>0.7</td>
<td>2.6</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td>815</td>
<td>0.7</td>
<td>1.6</td>
<td>8.3</td>
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<table>
<thead>
<tr>
<th>RSS values [MPa]</th>
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<th>SF - local axial stress</th>
<th>Full Stress Tensor</th>
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<tr>
<td>Microstructure</td>
<td>Slip system</td>
<td>mean 95th PCTL</td>
<td>mean 95th PCTL</td>
</tr>
<tr>
<td>Fine</td>
<td>Basal Prismatic</td>
<td>180 315</td>
<td>181 319</td>
</tr>
<tr>
<td></td>
<td></td>
<td>227 323</td>
<td>223 322</td>
</tr>
<tr>
<td>Coarse</td>
<td>Basal Prismatic</td>
<td>193 313</td>
<td>196 320</td>
</tr>
<tr>
<td></td>
<td></td>
<td>209 310</td>
<td>205 304</td>
</tr>
</tbody>
</table>

PCTL: percentile
Appendix

Figure A 1: Plastic strain as a function of time for a sample with coarse microstructure under static loading at 80%, 85% and 90% of $\sigma_{0.2\%}$.

Figure A 2: Optical dark field images of a sample with coarser secondary $\alpha$ lath size loaded to 70% of $\sigma_{0.2\%}$, directly after applying the load (0 minutes), and after holding the load for 5 and 10 minutes. As recording an image only takes ~1 second, there was no need for reducing the load for the imaging as it was done in the HR-DIC experiment.
**Fig. A 3**: Absolute frequency distribution of angles between slip line and lamellae in deformed secondary α colonies in (a) fine and (b) coarse microstructure.
Fig. A 4: Maps of resolved shear stress RSS on basal and prismatic slip systems for the material with coarse microstructure. The simulations are for loading to 70% of 0.2% proof stress of initially stress-free material. The RSS values have been calculated according to three different approaches. Additionally, maps showing the difference between the calculation methods are displayed.

Table A 1: Material elastic, plastic and thermal properties used for crystal plasticity simulation [103].

<table>
<thead>
<tr>
<th>Material</th>
<th>Application</th>
<th>Elastic properties [GPa]</th>
<th></th>
<th>Plastic properties [MPa]</th>
<th></th>
<th>Thermal properties [10⁻⁶ m/(mK)]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>c11  c12  c13  c33  c44</td>
<td>n_{&lt;a&gt;}</td>
<td>n_{&lt;a&gt;}  t_{&lt;a&gt;}</td>
<td>n_{&lt;a&gt;}  t_{&lt;a&gt;}</td>
<td>n_{&lt;a&gt;}  t_{&lt;a&gt;}</td>
</tr>
<tr>
<td>Anisotropic Ti Bulk</td>
<td>160 90 66 182 47</td>
<td>3 349 568</td>
<td>3 150 1502</td>
<td>6 1107 3420</td>
<td>9.5</td>
<td>5.6</td>
</tr>
<tr>
<td>Anisotropic Ti Buffer</td>
<td>176 91</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Free surface Buffer</td>
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<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

- $n_{<a>}$ Number of slip systems
- $t_1$ initial slip resistances
- $t_2$ saturation slip resistances
- $\alpha$ Coefficient of thermal expansion