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A nanoindentation investigation of local strain rate sensitivity in dual-phase Ti alloys

Tea-Sung Jun [a, *], David E.J. Armstrong [b], T. Benjamin Britton [a]

[a] Department of Materials, Royal School of Mines, Imperial College London, London, SW7 2AZ, UK
[b] Department of Materials, University of Oxford, Parks Road, Oxford, OX1 3PH, UK

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ABSTRACT

Using nanoindentation we have investigated the local strain rate sensitivity in dual-phase Ti alloys, Ti–6Al–2Sn–4Zr-xMo (x = 2 and 6), as strain rate sensitivity could be a potential factor causing cold dwell fatigue. Electron backscatter diffraction (EBSD) was used to select hard and soft grain orientations within each of the alloys. Nanoindentation based tests using the continuous stiffness measurement (CSM) method were performed with variable strain rates, on the order of 10^{-1} to $10^{-3}s^{-1}$. Local strain rate sensitivity is determined using a power law linking equivalent flow stress and equivalent plastic strain rate. Analysis of residual impressions using both a scanning electron microscope (SEM) and a focused ion beam (FIB) reveals local deformation around the indents and shows that nanoindentation tested structures containing both α and β phases within individual colonies. This indicates that the indentation results are derived from averaged α/β properties. The results show that a trend of local rate sensitivity in Ti6242 and Ti6246 is strikingly different; as similar rate sensitivities are found in Ti6246 regardless of grain orientation, whilst a grain orientation dependence is observed in Ti6242. These findings are important for understanding dwell fatigue deformation modes, and the methodology demonstrated can be used for screening new alloy designs and microstructures.

1. Introduction

Titanium alloys are used extensively in aerospace applications where fatigue is one of the most significant issues to contend with and manage. In particular, the nature of modern flying now includes some significant holding at high load for extended periods, for instance between take-off and cruise [1], in order to maximise fuel efficiency and reduce noise pollution. Further load-holds occur during thrust reversal to land on short run ways. These regimes are cyclic (once or twice per flight) and have a time sensitive load hold, leading to concern regarding rate sensitive deformation modes involved in dwell fatigue.

Cold dwell fatigue has been a critical issue in aero-engine industry for the last few decades [1,2]. Cold dwell susceptible alloys are those which suffer from a significant reduction in fatigue life due to the inclusion of a short load-hold (~120s) at low to moderate temperatures (up to ~200 °C). This can reduce the number of cycles to failure by a decade or more and this is called the “dwell debit”. In dual-phase Ti alloys (Ti–6Al–2Sn–4Zr-xMo), Mo content significantly affects dwell sensitivity, where Ti6242 is dwell sensitive and Ti6246 is not [3]. A chemical, structural or morphological effect has been thought of as a cause of dwell fatigue due to differences in β volume fraction and local chemistry between these alloys.

In practice, dwell fatigue failure is mitigated by use of dwell insensitive alloys and careful maintenance schedules, but management of this phenomena costs the aerospace industry significantly (~£100 m/year). It is therefore important to understand microstructural contributions towards the dwell process to enable more cost effective component management strategies and ultimately engineering new materials that are dwell insensitive.

Recently it has been demonstrated that dwell fatigue failure is dominated by local microstructure in many dual-phase Ti alloys, including the presence of a rogue grain combination [4]; where, during the load-hold, stress is shed (i.e. redistributed) from a ‘soft’ grain to a neighbour ‘hard’ grain in the Stroh model [2,5]. Therefore local regions of very high stress form across the interface [6,7]. Hard and soft grains here represent those grains that are poorly oriented for an applied deformation mode and appropriately oriented for easy slip (i.e. *a* type basal or prism slip) respectively. For titanium

* Corresponding author.
E-mail address: t.jun@imperial.ac.uk (T.-S. Jun).

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this is often described considering the angle between the c axis of the unit cell and principal loading axis, where applying a normal stress parallel to the c axis is a hard orientation and deforming perpendicular to the c axis is a soft orientation.

In dwell fatigue, time dependent stress amplification during this load shedding process at local microstructural regions near the interface is thought to play a predominant role in failure through facet formation [8]. Prior research indicates that the function of basal plane orientation is important in dwell fatigue for dual-phase Ti alloys [9–13]; yet fundamental deformation mechanisms within each hard and soft grain (i.e. contributions of individual slip systems) are still unknown.

One hypothesis on time sensitive dwell fatigue is that strain rate sensitivity (SRS) is a crucial factor governing the load shedding phenomenon. As titanium alloys are typically more elastically and plastically anisotropic at the grain scale [14–16], there are likely to be differences in the SRS of different grain orientations due to variable rate sensitivities of the different slip systems [17]. These slip systems are a dislocations on prismatic planes; a dislocations on basal planes; and $c+a$ slip systems on pyramidal slip planes. Each slip system has a different critical resolved shear stresses, where their ratio is likely to be ~3:4:9 [18,19]. Even though the $c+a$ deformation mode has a significantly higher CRSS value it is still likely to activate, as slip with a type dislocations only does not provide 5 independent deformation modes which are required to accommodate an arbitrary shape change.

Nanoindentation has been previously used to study the mechanical response of single grains in titanium alloys. Quasistatic nanoindentation, combined with TEM of Ti–7Al, reveals that indentation into different grain orientations activates different slip systems [20]. In particular indentation into the basal plane activated both $c+a$ type dislocations and a type dislocations. The a type dislocations were of opposite sign to the a component of the $c+a$ dislocations to enable local deformation and support the local curvature around the indent. This supports a prior demonstration [21] that indentation into grains of Ti–6Al–4V revealed clearly that these mechanisms also happen in dual-phase microstructures and in particular that $c+a$ type dislocations are geometrically required if twinning does not activate. This change in plastic deformation mechanism, when indenting into grains of different crystallographic orientation, results in significant anisotropic hardness measurements. Additionally indentation, combined with EBSD, has been used to systematically study grains of different orientations and confirmed that the anisotropic mechanical performance is consistently observed across a range of Ti alloys [14,16,22,23]. Furthermore, experimental data has been fitted with crystal plasticity models has enabled extraction of the critical resolved shear stress for different dislocation types [14,16].

The nanoindentation approach is insightful and relatively inexpensive to undertake, but precise mechanistic analysis is fraught with complications, as the stress state around the indention is complex and there are likely to be significant lengthscale contributions due to the indentation size effect for small indentation depths. Often these effects are considered for relatively isotropic materials, yet in HCP alloys ignoring the effect of orientation is likely to be problematic. This does not diminish from the potential use of nanoindentation to elucidate mechanisms and highlight opportunities for further study.

Moving beyond hardness (i.e. strength) and considering strain rate sensitivity is now possible with indentation, as new experimental approaches have been developed. Typically nanoindentation is used for exploring a deformation resistance (i.e. hardness and modulus of elasticity) on a local scale [24], and use of the continuous stiffness measurement (CSM) approach [25] enables continuous measurement of hardness with depth during indentation, through measurement of the phase change of an applied oscillation of the tip displacement and the measured response of the material. Recent work has highlighted that this CSM approach can be very valuable in performing variable strain rate testing and therefore accessing the strain rate sensitivity at a local scale, such as exploring the SRS of nanocrystalline and ultrafine-grained metals [26–30].

In practice, a power law constitutive equation is typically used to define the indentation-based SRS exponent (i.e. m value) as follows [28,31–36]:

$$m_{nanoindentation} = \frac{d(\ln H)}{d(\ln \dot{\varepsilon}_{indentation})} \tag{1}$$

with further following relations, $\dot{\varepsilon}_{indentation} = \dot{h}/h$ and $H=P/A$, where h is the indentation depth, $\dot{h}$ the penetration rate, P the applied load, A the project area for indent (e.g. $24.5h^2$ for perfect Berkovich tip) and H hardness.

The SRS at the nanoscale [36,37] and macroscale [38–41] of Ti alloys have been determined using either uniaxial tensile/compression or nanoindentation-based test with different methods (e.g. constant strain rate and strain rate jump test). Observed m values were within a range of 0.007–0.04 for CP-Ti [36–39,41], 0.0185 for Ti–6Al and 0.01 for Ti6242 [40], where the m value of metallic materials is in general lower than 0.1 at room temperature [42]. Direct comparison across alloys from these studies is not possible as microstructural features, such as grain size, and testing method were not similar.

Within this work, we use the CSM technique due to its fairly-straightforward experimental setup and data analysis. We note that there are some significant limitations that must be considered with this approach [28,43], such as: (i) indentation data are sensitive to microstructure; (ii) thermal drift may play a prominent role at lower strain rates (i.e. longer testing time); and (iii) significant experimental error can be occurred in soft metallic materials (e.g. single crystal) with a large E/H ratio. Recently several authors have reported [28,44–46] using a modified CSM method to study SRS effects on hardness by utilising step changes in load rate in a single indentation. This has the advantage that a single indentation can be utilized to study the effect of rate sensitivity on hardness and this can also overcome issues with environmental instabilities leading to thermal drift artefacts in very slow strain rate indentations. However, while the modified CSM method has been shown to work well on monocrystalline metals, there are issues with interpretation of the data on coarser grained materials. This is especially important when there are significant indentation size effects [47] that lead to an increased hardness as a function of indentation depth, superimposed on-top of any change in hardness due to strain rate changes. Secondly the hardness change measured is by definition taken at points where differing volumes of plastic deformation have occurred. In nanocrystalline materials this is not of concern as the 'length scale effects' are linked to the nano-grain sizes, rather than the indentation volume. However in other microstructures this can lead to comparing hardness changes which are controlled by both sampling differing microstructures, e.g. due to heterogeneity or length-scale effects under the indenter impression, as well as strain rate changes.

While nanoindentation approaches can clearly measure different rate sensitivities during deformation, care must be taken in interpretation, as comparison with more conventional engineering measures of strain rate sensitivities is difficult because the local strain field around the indenter during each test is rather complex [14,48]. Here, we describe rate sensitivity (i.e. equivalent to SRS obtained from uniaxial tensile or compression test) as the material response to the variable indentation rate. We use