On the mechanistic basis of fatigue crack nucleation in Ni superalloy containing inclusions using high resolution electron backscatter diffraction

Jun Jiang^{1}, Jie Yang², Tiantian Zhang¹, F.P.E. Dunne¹, T Ben Britton¹*

1. Department of Materials, Royal School of Mines, Imperial College London, Exhibition Road, SW7 2AZ, UK

2. Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, Beijing 100095, China

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Abstract

A series of interrupted three-point bend low-cycle fatigue tests were carried out on a powder metallurgy FHG96 nickel superalloy sample containing non-metallic inclusions. High resolution electron backscatter diffraction (HR-EBSD) was used to characterize the distribution and evolution of geometrically necessary dislocation (GND) density, residual stress and total dislocation density near a non-metallic inclusion. A systematic study of room temperature cyclic deformation is presented in which slip localisation, cyclic hardening, ratcheting and stabilisation occur, through to crack formation and microstructurally-sensitive propagation. Particular focus is brought to bear at the inclusion-matrix interface. Complex inhomogeneous deformation structures were directly observed from the first few loading cycles, and these structures were found not to vary significantly with increasing number of cycles. A clear link was observed between crack nucleation site and microstructurally-sensitive growth path and the spatially-resolved sites of extreme values of residual stress and GND density.

1. Introduction

High performance structural materials drive technology forward and enable us to travel the world, exchange cultures and ideas, and broaden collaboration and understanding. Material scientists and engineers enable progress through innovation in materials design and manufacture, delivering new technologies and making existing technologies more economical for both supplier and consumer. As our demands heighten so must understanding and prediction of materials performance improve, and in particular we must strive to obtain physical understanding and to enable prediction of materials performance at the microstructural level to generate change.

In this work, we focus on mechanistic understanding of fatigue in Ni superalloys used as structural materials in gas turbine applications. These alloys were first developed in the 1940s and evolutionary design since their invention has pushed their performance ever further and significantly improved fuel efficiency and thrust in a modern jet engines, enabling lower cost, more sustainable

- Corresponding author: jun.jiang@imperial.ac.uk

and safer transport of people and goods across the world. Well understood technical materials, with outstanding structural performance, like the Ni superalloys, are essential in delivery of successful jet engine technology, including blade and disk components [1].

Turbine disks in advanced aero-engine are subjected to very high cyclic stresses over long periods often at elevated temperatures ($\sim 550\text{--}750\text{ }^{\circ}\text{C}$) and their structural performance, as well as high resistance to creep and oxidation, are paramount. For Ni-based disk components, powder metallurgy manufacturing routes are often used to achieve more homogenous microstructures and chemical distributions. Of particular interest for this study, the second generation of FGH 96 powder metallurgy (PM) Ni superalloy was designed for damage tolerance.

Non-metallic inclusion is one of typical defects in Nickel-based PM superalloy. These inclusions are inevitable defects presenting in powder metallurgy alloy processing, as although extreme care has been taken contamination of powder cannot be avoided completely in melting, powder atomisation and handling, through to consolidation. It is found that non-metallic inclusions significantly degrade FGH96 low cycle fatigue life [2]. This motivates us to improve our understanding of this complex process, and we begin with low temperature fatigue crack nucleation utilising state of the art experimental techniques in order to improve fatigue life predictions, and deliver safer and more economical components.

Many recent reviews have outlined the importance of understanding of fatigue crack formation, and propagation mechanisms at the microstructure scale are critical to facilitate quantitative prediction of fatigue lifetime as a function of microstructure [2-7]. These reviews highlight the need to deliver new experimental insight into deformation processes and failure in fatigue to drive modelling that can accurately capture microstructurally-sensitive effects and use geometrically faithful models. These modelling efforts have focused at a range of length and timescales, using approaches such as molecular dynamic simulations [8, 9] up to the grain level using crystal plasticity finite element techniques [10-15]. This range of approaches necessitates ever increasing fidelity of experimental studies that span length and timescales, such as X-ray synchrotron [16] [17, 18] and high energy neutron diffraction [19]; as well as electron microscopy [20, 21] microstructurally-sensitive and physically based modelling approaches necessitate local measurements of defect content and residual stresses to improve the prediction of fatigue crack nucleation and short crack growth.

Modelling efforts using molecular dynamics [8] and crystal plasticity finite element [13] techniques lead to the conclusion that fatigue crack formation criteria should be closely linked to stored energy which, for materials that deform plastically, is related to stored dislocation density.

High resolution, cross-correlation based, electron backscatter diffraction (HR-EBSD) enables the evaluation of lattice curvature which may be linked to dislocation content using Nye's tensor [22] to calculate stored geometrically necessary dislocation (GND) densities. These GNDs, which are equivalent to a local accumulation of dislocations with the same sign, can be included as hardening terms in strain gradient plasticity, such as those proposed by Ashby [23], Fleck et al [24] and Gao et al [25]. Plastic strain gradients can play a critical role in understanding size effects present in most material systems and mechanical tests, either due to sample geometry or strain gradients developed because of heterogeneous microstructures, resulting in extra hardening effects [26] and can be incorporated into crystal plasticity finite element models [27-29].

At present there is limited work that addresses the relationship between stored GND density in cyclically deformed materials and crack nucleation, likely due to the relatively recent renewed interest in GNDs and the development of measurement techniques. Littlewood et al [30] have used HR-EBSD to study the development of GND density in Ti-6Al-4V alloy subjected to load controlled dwell fatigue and four EBSD maps were acquired in different samples which were subject to 0, 1500, 3000, 4200 cycles respectively. Jiang et al [31] used HR-EBSD to study GND density distribution and evolution in cyclically deformed polycrystalline copper samples subjected to 0, 2, 200, 2000 cycles respectively. While these studies provide quantitative analysis of stored GND density and associated structure, no crack formation process was directly observed and comparison of the samples was statistical in nature as different samples and sampled areas were used. Biroasca et al [32] used Hough based EBSD approaches (which have ~100 less GND resolution) to quantitatively study GND density distributions near fatigue crack propagation paths in nickel superalloy subjected to thermo-mechanical fatigue. They found that local microstructural texture influences fatigue crack propagation and fatigue life. However the evolution of GND density and local texture was absent from their study which is required to link the development of GNDs to fatigue crack formation.

The EBSD-based curvature analysis of stored GND density does not enable measurement of statistically stored dislocations (SSDs), as they cause no change in net lattice curvature. SSDs may be closely bonded dipoles or multipoles and from earlier TEM studies on fatigued oxygen free high conductivity copper samples, it can be seen that reversible cyclic deformation generated high proportions of closely bonded ladders of dislocation dipoles in persistent slip bands (PSBs) which are potentially associated with crack formation sites [33-36].

While spatial resolution of SSDs is not currently possible with HR-EBSD based methods, recent work by Wilkinson et al. [37] includes quantitative estimation of the total dislocation (i.e. SSD and GND) content for a map (i.e. not spatially resolved, but still quantitative) through calculations based upon distributions of stress associated with the statistics of probing close to a dislocation core within a dislocated crystal, using an analysis based upon the work of Groma and colleagues [38, 39]. Wilkinson et al. [37] validated with discrete dislocation dynamics calculations and demonstrated that this technique works with HR-EBSD based residual stress maps assessing a series of experimental samples in Cu deformed in tension to increasing levels of plastic strain. The mathematical framework to calculate total dislocation density from HR-EBSD measurement can be found in appendix of this current work.

The main purpose of this current study is to provide new experimental insights into cyclic deformation in structural materials, with focus on room-temperature deformation of Ni-superalloys. We address correlations of dislocation content with fatigue crack formation, and their experimental study presented provides new information that is required to provide predictive capability in fatigue life, which encompasses both crack nucleation and microstructurally-sensitive crack growth, as well as crack propagation which all contribute to component life.

2. Method

2.1 Sample fabrication and preliminary characterisation

Figure 1 showing an image of PM Ni bar specimen with marked dimensions. The region of interest (ROI) indicates EBSD mapping area at the centre base of the bar.

A powder metallurgy (PM) Ni superalloy FGH96 bar-stock sample containing a non-metallic inclusion was supplied by AVIC-BIAM. A bending test sample with rectangular 12x3 mm² cross-section and 3 mm width was cut using electric discharge machining (as shown in Figure 1). It was annealed at 750 °C for 7 hours to relax internal stresses and reduce the stored dislocation content induced during manufacture. The sample was ground using 800-grit silicon carbide papers for 20 minutes at 20N force and this was followed with finer grinding (1200-, 2000-, and 4000-grit) for increasing durations with the same force. Final polishing was performed using oxide polishing suspension (OPS) for 40 minutes and high quality EBSD patterns were obtained for mapping (shown in Figure 2 (a) & (b)) and HR-EBSD analysis.

Figure 2 (a) EBSD measured pattern quality map overlaid with grain boundary skeleton map in which grain boundaries with larger than 10 degree misorientation angle tolerance are marked as black lines, Σ 3 twin boundary as red lines and Σ 9 twin boundaries as green lines, (b) Inverse pole figure (IPF) plotted with respect to the deformation axis (horizontal) showing the orientations and morphology of FGH96 microstructure.

Figure 3 (a) shows grain boundary misorientation angle histogram, and (b) the grain size distribution histogram.

Figure 4 showing convolution frequency (texture) of {001}, {111} and {011} pole figures (PF) of the EBSD map shown in Figure 2.

In general, the non-metallic inclusions present in this material vary significantly in terms of size and morphology. The maps in Figure 2 (a) and (b) are illustrative of the FGH96 Ni matrix microstructure and the associated inclusion. While this paper focuses on the behaviour of a particular inclusion, it remains indicative of deformation and failure in generic PM Ni superalloys with inclusions.

As with many FCC alloys, there are a significant number of annealing twins ($\Sigma 3^n$ 'special boundaries', where the $\Sigma 3$ coincident site lattice is a $60^\circ \langle 111 \rangle$ grain boundary) due to the low stacking fault energy in this material. The majority of grains in this cross-section contain at least one twin and many grains have several parallel twins present within them, as shown in the grain boundary plot in Figure 2(a) in which black lines correspond to high angle grain boundaries and red lines indicate $\Sigma 3$ boundaries. Furthermore, the grain boundary misorientation distribution histogram shown in Figure 3 (a) clearly shows that $\Sigma 3$ annealing twin boundaries account for approximately 60% of the total length of grain boundaries.

Considering the EBSD crystal orientation maps shown in Figure 2, grains of varying sizes can be observed, and numerous twin boundaries complicate the grain size analysis. Considering all boundaries within the map, i.e. including the special boundaries, the grain size varies significantly from very small grains to those of $\sim 12\mu\text{m}$. The arithmetic mean grain diameter (area weighted, counting twins as separate grains) was determined to be $5.9\mu\text{m}$ as shown in Figure 3(b).

The EBSD map in Figure 2 (b) contains 2083 grains. From these maps, the orientation distribution function (ODF) was calculated in Espirit and pole figures representing the ODF is shown in Figure 4. This material had minimal preferred crystallographic orientation (texture) consistent with a powder metallurgy forming route.

2.2 Fatigue test methodology and mechanical loading history

Figure 5 showing three point bending low cycle fatigue test set up.

Figure 6, Cyclic load and displacement curves for the three-point bending test sample.

Figure 7 showing cyclic (incremental) displacement as a function of number of fatigue cycles

The matchstick sample was tested using a three point bending geometry with push-push fatigue capabilities. This geometry was selected to encourage cracking to occur in a small region containing the inclusion and limited the area of inspection with periodic electron microscopy-based characterisation.

The jig was designed and fabricated in-house from high strength stainless steel. The jig, shown in Figure 5, was mounted on a standard load frame. The sample is clamped in place between four stainless steel rollers and load was applied directly from the cross head through a rounded edge.

Low cycle fatigue (LCF) was conducted in load control, with a load amplitude of 550N to 5500N (load ratio = 0.1 and mean load = 2475N) at loading rate of 1000N/s. The displacement of the crosshead and load (from a load cell above the loading head) were recorded during tests.

The fatigue test was interrupted 16 times in total at 2 cycles, 20 cycles, 200 cycles, 400 cycles, and 800 cycles to 5200 cycles respectively. At each of these stages, the sample was removed from the testing rig and loaded into SEM to run an EBSD scan on the same ROI. The macroscopic cyclic load and displacement behaviour is shown in Figure 6.

As this fatigue test is load controlled and the mean load is not zero, significant ratcheting occurred as shown by the progressive development of displacement with increasing number of fatigue cycles in Figure 6. The cyclic displacement variation was extracted from Figure 6 and plotted in Figure 7 from which it can be seen that significant ratcheting strain and cyclic hardening occurred in the first 1000 cycles and was cyclically stabilised in subsequent cycles.

2.3 EBSD and HR-EBSD measurement

The sample was inserted into a Zeiss Auriga SEM and held in place mechanically, maintaining sample cleanliness and optimising multiple scanning attempts. A Bruker Quantax EBSD system was used to run EBSD scans on a selected ROI which includes the inclusion located at about $\sim 50\mu\text{m}$ from the base edge of the sample.

EBSD was performed with 20 keV acceleration voltage and a probe current of 16.8nA. The sample was inclined at 70° to maximise the quality of the resulting EBSD patterns. It was held at a working distance of 15 mm and the tilt of the EBSD camera¹ was optimised for this material, working distance and chamber design was set at 4.3° to produce diffraction patterns of very high, and near uniform, contrast across the field of view.

EBSD maps of 56.5 μm x 40.75 μm^2 with a step size of 0.25 μm were collected, with patterns captured at 2x2 binning (800x600 pixels) with 8 bits of greyscale intensity and no artificial gain, black level or background correction was applied. This is not the optimal setting for pattern capture, but it enables large, high spatial resolution map capture in a reasonable acquisition time of ~120ms per pattern (i.e. ~90 mins per map). Patterns were first analysed with commercially available indexing routes to obtain absolute crystal orientation using the Bruker Espirit software. EBSD maps were captured at 0, 2, 20, 200 to 5200 cycles, the latter with an increment of 400 cycles. This resulted in the capturing of 16 EBSD maps in total.

Subsequent analysis to calculate residual stress distributions and stored GND density was performed offline using the HR-EBSD analysis method described elsewhere [40-44]. Briefly, HR-EBSD involves direct pattern comparison of two or more diffraction patterns to determine the lattice rotation and residual elastic strain gradients between the crystals which are illuminated by the electron beam. Analysis of the rotation gradients in a map enables calculation of six lattice curvature components that can be related to the stored GND components.

EBSD patterns are taken from within one map and cross correlation analysis is performed between points within each grain with respect to a reference point of unknown strain. Measurement of absolute elastic strain and between maps is currently not possible. For this case, it requires a high precision of the geometry of the experimental system e.g. positions of sample to detector between maps need to be as precise as 1/20th pixel (i.e. 1 μm for this experimental set up). [45]. The direct pattern comparison to extract elastic strain and lattice rotation was developed initially by Wilkinson et al [40, 46] and markedly improved and optimised in the past few years especially using robust fitting [42], pattern remapping routines to calculate strains in the presence of larger lattice rotations [43]. Measurement of rotation gradients can be used to evaluate the net curvature and an estimate of GND content [41]. Measurement of residual stress variations within each grain across a map has recently been used to calculate total dislocation measurement [37]. Its capability has been extended significantly from qualitative measurement on single or a few grains [30, 47-50] to quantitative analysis in thousands of grains [51].

The methodology and application of the HR-EBSD technique have been well documented [37, 40-43, 46] and recently reviewed by Wilkinson et al [52] and Britton et al [53], hence it is just briefly described here. Forty 128 pixel x 128 pixel regions of interest were randomly placed across each EBSD pattern and comparison of ROIs between test and reference EBSD patterns enables the calculation of displacements corresponding to zone axes shifts. These shifts were used to estimate the lattice misorientation between test and reference. A remapping algorithm [43] was used to remap pixel intensities in the test pattern back towards the reference pattern and a second pass of cross correlation between new remapped ROIs and the original reference EBSD pattern ROIs was performed to calculate final deformation difference between test and reference. Results from the

¹ On-line adjustment of the camera tilt is available with this camera.

first and second pass cross correlation measurements were combined using a finite strain framework to calculate the residual elastic strain difference and total lattice rotation difference. As pattern shift methods cannot directly determine the volumetric strain changes, an out of plane traction free boundary condition was applied with anisotropic Hooke's law to calculate the stress tensor and separate the normal strain components [40]. As cross-correlation analysis was conducted in the Fourier domain, prior to the analysis, high-pass and low-pass filtering of [2, 9 cycles / ROI] (with gradual tails to reduce aliasing cut-off frequency and bandwidth) was applied to remove high and low frequency noise from EBSD patterns.

2.3.1 GND density estimation

The measured lattice rotation components are numerically differentiated spatially within each grain to calculate lattice curvatures. These are combined with Nye's theory [22] to calculate GND densities from elastic strain and rotation gradients. As EBSD can only measure these gradients within the 2D map, only five of nine Nye tensor components and one difference can be obtained [54]. From this, we calculate the GND densities assuming that 18 types of possible dislocations (12 edges and 6 screws) exist in FCC crystals [55]. This problem is underdetermined so a physically motivated L_1 minimisation scheme is used to calculate a set of dislocations with minimum line energy to support measured lattice curvature, ignoring elastic strain gradients as they are typically an order of magnitude smaller than the rotation gradients in metals [41]. This solution may not be unique, but it necessarily supports measured lattice curvatures. From this, a scalar value of the total stored GND density is calculated.

2.3.2 Total dislocation density

The total dislocation content in a mapped area can be estimated through analysis of the residual stress distributions. The detailed mathematical framework for total dislocation density estimation can be found in the appendix according to Wilkinson et al.[37]. Briefly in dislocated crystals the measured elastic stresses have some extreme values (tails) which do not follow a Gaussian normal distribution, unlike the majority of points towards the central region of the stress distribution. This follows work of Groma et al [38, 39] who made similar findings in their X-ray peak broadening measurement and proposed that these high stress magnitude points were generated from stress fields close to individual edge dislocations. Assuming that shear stress field are generated only from edge dislocations, the total dislocation density can hence be estimated using a probability distribution. It is found that the probability function comparing the total dislocation density with respect to the cube of shear stress tends to be very noisy. To reduce noise a 'restricted second moment of probability' is used by finding the integral over shear stress. This approach was validated by Wilkinson et al who have used 2D discrete dislocation dynamics [37] to calculate the probability functions from individual dislocations. The mathematics and assumptions for this analysis from Wilkinson et al's approach are summaries in the Appendix of this current work.

3. Results

3.1 SEM micrographs

Figure 8, captured SEM images showing the inclusion and the development of slip bands and fatigue cracking at 2, 20, 1200, 4800 and 5200 cycles. The contrast and brightness were adjusted to highlight slip lines and crack paths.

The non-metallic inclusion has irregular shape with size of $\sim 5\mu\text{m} \times 20\mu\text{m}$ as seen in Figure 8. After 2 cycles, clear out of plane slip traces can be observed around the inclusion. These slip lines are very grain dependant and in particular, when the slip lines cross twins, they are disrupted and restored. As the number of cycles increases, these slip bands became clearer and surfaces roughens.

In addition to local plasticity, a small crack was formed within the inclusion after 2 cycles (Figure 8). This crack grows with increasing number of cycles and after 4800 cycles it propagates into the Ni matrix. Broadly the crack propagates at $\sim 45^\circ$ from the macroscopic deformation axis.

Figure 9, Images obtained at the annealed, unloaded state showing (a) Crystal orientations with respect to the major deformation axis, (b) image quality overlaid with grain boundaries (high angle grain boundaries marked as black lines and twin boundaries as red lines) and. Figs (c) and (d) show the same data as for (a) and (b) but for the sample after 5200 cycles.

Figure 9(a) and (b) show the initial state of the sample's microstructure and grain boundary distribution. This region contains many annealing twins and the grain structure near the inclusion is similar to the parent matrix. Increasing numbers of cycles results in elongation of some grains (Figure 9(c) and (d)). Furthermore, internal structure forms within the Ni grains as indicated by patterning in the orientation map. The inclusion and fatigue crack formed can be identified by the unsolved points within the quality map (Figure 9(b) and (d)). The crack propagated through the Ni matrix and was broadly intergranular, but in some places it traversed a number of surface grains.

3.2 Development of GND density

Figure 10 shows development of lower bound GND density maps near the inclusion with increasing number of fatigue cycles. The colour scale for all maps is the same in order to facilitate direct comparison. Blue lines indicate grain boundaries for which misorientation angle between two neighbouring points is larger than 10 degrees. White areas are rejected points in cross-correlation analysis as the displacement for these points cannot be reliably determined due to either poor image quality or large patterning variation compared to their reference points.

Lower bound GND density maps are shown for 10 of 16 observations in Figure 10. In the annealed state (Figure 10 (a)) there are initial GNDs present, but with a low density ($\sim 3.1 \times 10^{13} \text{m}^{-2}$). The GNDs are not preferentially located around the inclusion and are instead relatively homogeneously distributed across the map. Subsequent cycling results in a dramatic change in these maps, and the overall GND density increased drastically and inhomogeneous GND density patterning was formed within individual grains and can be seen in Figure 10 (b-j). Complex GND density networks are formed, connecting between stored GND locations across grains. This is most obvious as a band at 45 degrees to the horizontal axis (bottom left to top right). Furthermore, there are a few high GND density bands connected between some triple junctions and towards the tips of twins.

As the fatigue cycle number increased to 20 and 200 cycles in Figure 10 (c) and (d) respectively, GND density continues to increase leading to higher contrast between high and low GND density areas. It is very interesting to notice that the main frame of previously formed GND density structure at 2 cycles does not change significantly with further fatigue cycles. As more GNDs are generated with cycling, the GND density network became more complex and intense with increasing number of branches (GND bands with high density). More white areas (gaps) appeared in the 20 and 200 cycle maps that represent points of either low EBSD pattern quality or a highly distorted EBSD pattern compared to their reference patterns. The 45 degree GND density network is most noticeable at 2 cycles.

After ~ 400 cycles, qualitatively the structures seen within each GND map remain largely unchanged. However, quantitatively the maps show that the GND density appears to be reduced. It should be noted that observed GND density reduction was mainly caused by a carbon contamination layer build-up during re-scanning. Detailed measurement is included in section 3.6. Note that very high dislocation density points are challenging to measure, as the resultant EBSD patterns are poor and do not cross correlate well (seen in white within the GND maps).

After the fatigue short crack extends into the matrix (~4800 cycles, Figure 10 (I)) there is no significant alteration of the GND density structures, when compared with the map obtained after 4000 cycles in Figure 10 (h).

Figure 11 GND density distribution histogram plots at 0, 2,400,1200,2400,3600 and 4800 cycles on log10 scale. 40 bins were used with an interval of 0.125 on log 10 scale.

Figure 12 GND density error bar (red) plot showing the mean value and standard deviation of each map, together with total dislocation density, as a function of number of fatigue cycles.

Frequency histograms from these maps were calculated, and are shown in Figure 11. These reveal that stored GND density distributions can be described by a log-normal-like distribution and therefore geometric means and log-normal standard deviations are reported in Figure 12. The GND density rapidly increases up to ~200 cycles and then starts to decline. (The total dislocation density plot shown in Figure 12 will be discussed shortly.)

3.3 Development of residual stress

Figure 13 shows in-plane shear stress σ_{12} difference maps calculated by HR-EBSD within each grain by subtracting grain mean shear stress. σ_{12} maps at 0, 2, 20, 200, 400, 1600, 2800, 4000, 4800 and 5200 cycles are shown. Grain boundaries are marked as black lines. Dark blue areas are poor quality data which were filtered out during cross-correlation analysis.

In-plane shear stress difference maps are presented in Figure 13, where σ_{12} (intragranular stress) difference is calculated within each grain by subtracting from the point-wise shear stress the grain's mean shear stress value.

Initially small residual stress is found in the initial annealed, undeformed state. Further deformation results in a rapid rise of the intragranular residual stress magnitudes which are heterogeneously distributed across grains. Variations of the residual stresses are analysed using frequency histograms as shown in Figure 14. A significant difference can be seen between the undeformed and deformed states. Subsequent deformation results in a moderate systematic widening in the range of residual stress. At 4800 cycles where crack was formed, where the range of the residual stress is largest.

The tails of these distributions have been used to estimate the total dislocation content using Wilkinson et al's probability approach [37] and the results are presented in Figure 12. Total dislocation density increases pronouncedly from the annealed state to the deformed state. The density has a steady increase from 2 cycles to 1000 cycles and then it reaches a plateau with some small fluctuations until the crack was formed at 4800 cycles. Furthermore, compared to the total density estimate, it seems that the GND density accounts for less than 10% of the total dislocation density.

Figure 14 shows (a) histogram showing the development of normalized intragranular in-plane shear stress 12 distribution (along deformation axis) on selected maps at 0, 20, 400, 1200, 2400, 3600 and 4800 cycles respectively. 40 bins were used and note that the y axis (frequency) is a log scale to highlight the extreme stress 'tail' distribution, and (b) shows standard deviation from in plane shear stress 11 in Figure 9(a).

3.4 Identification of crack sites

Figure 15 shows GND density map (a) and grain normalised in-plane shear stress map (b) at 2 cycles. The dark blue lines represent grain boundaries and note that the colour scale is changed from Figure 10 and Figure 13 in order to highlight the hot and cold spots. The top 5 percent of (c) GND density and (d) the grain normalised in-plane shear stress maps at cycle 2 are selected and plotted as red dots and overlaid with the image quality map in which the crack path is clearly marked.

Qualitative inspection of GND and residual stress maps in Figure 10 and Figure 13 reveals that the magnitudes of GND density and residual stress vary with number of fatigue cycles but the deformation structure formed in the first few cycles does not change significantly. Qualitative comparison between the residual stress and stored dislocation density heterogeneities are shown in

Figure 15 (a & b) and show that these are often co-located. To explore this further, the top 5% of highest GND density points in Figure 15 (c) and points of top 5% absolute in plane shear stress points are overlaid on EBSD image quality maps and shown in Figure 15 (d).

These hot spots broadly trace out the path of microstructure-sensitive crack propagation (Figure 8). The GND density map is more clustered around the crack path than the residual stress hot spot map; however we note that the residual stress hot spots are formed not only from sampling near dislocation cores (as used for the total dislocation density estimates), but in addition from where stresses may be large enough to encourage local separation of the Ni matrix.

3.5 Further studies – post test

A larger area surrounding the mapped region was examined with a finer step size and patterns were again captured. This map, given in Figure 16 (a), shows that the region which has been repeatedly mapped, outlined in black, demonstrates a dramatic reduction in the measurement of stored GND density as compared with the neighbouring region. Maps of stress as shown in Figure 16 (b) do not show this ‘picture frame’ effect. While this certainly looks dramatic, closer examination of the region within the picture frame reveals that dislocation substructure is clearly observed and it is similar to the features seen after only 2 cycles (in Figure 10). It is also noted that there is a general GND density reduction after re-scanning compared to Figure 10 (j).

Figure 16, (a) showing GND density map at 5200 cycles with 0.125μm step size. (b) showing corresponding in plane shear stress map.

Figure 17, (a) SEM image showing PM Ni sample surface with carbon coated region and non-coated region. The boundary was marked by red dash line. A ROI is selected to run EBSD map highlighted as a red square and showing in (b). Inverse pole figure was generated to with respecting to deformation axis (horizontal). (d) Estimated GND density map at log10 scale m⁻². White regions represent rejected points from HR-EBSD analysis mainly due to low quality EBSD pattern images. (e) Column averaged GND density line profile across GND density map shown in (d). Arithmetic mean was used to calculate averaged log10 GND for each column and non-determined points were excluded.

To further study the effects of surface contamination due to electron beam re-scanning, a ~5-10nm thin carbon layer was directly coated on one side of the sample. Hence, a clear contrast between coated and uncoated regions can be seen in Figure 17 (a) and its boundary is marked as a red dashed line. A ROI of 300 μm x300 μm at the coating boundary was selected (as shown in Figure 17 (a) and (b)) to run an EBSD map with step size of 1 μm. The microstructure of the ROI is shown in Figure 17 (c) and corresponding GND density was calculated and shown in Figure 17(d). The column averaged GND density was plotted in Figure 17(e).

As seen in Figure 17 (d) and (e), significantly higher GND density areas are developed on the uncoated region (left hand side). The 5-10 nm carbon layer leads to a significant GND density decrease (right hand side).

4. Discussion

Microstructurally sensitive fatigue crack nucleation, centred upon a non-metallic inclusion in a Ni superalloy polycrystal sample, has been explored using interrupted three point bend loading, with interruptions enabling observation within the SEM using both EBSD and HR-EBSD. The sample was fabricated and loaded to encourage crack formation around the metallic inclusion to restrict requirements on mapping, and this proved insightful and revealed the formation of a microstructurally-sensitive crack whose path correlated with areas of high GND density and local stress hot spots.

The sample was subjected to (zero to zero) load-controlled LCF, with $R=0$ such that a significant non-zero mean stress applied during the cycling. As a result, significant strain ratcheting (progressive accumulation of plastic strain) with increasing number of cycles is observed in Figure 7 (a). From the macroscopic response in Figure 7 (b), it can be seen that significant cyclic hardening occurred during the first 200 cycles after which the cyclic loop became stable for subsequent cycles. As shown in Figure 11, as a result of the progressively increasing total dislocation and GND densities, the material response showed cyclic hardening. More dislocations were generated and stored in the sample during cyclic loading, increasing the opposition for mobile dislocation slip and consequently increasing hardening. Once the cyclically stable state had been achieved, although further cycles led to an increase in (ratchet) displacement, dislocation density remained approximately constant, implying that an equilibrium condition of dislocation generation and annihilation had been reached.

The observed HR-EBSD measured dislocation evolution trend agrees well with Huang et al's [19] neutron diffraction study in which in-situ neutron diffraction was carried out on Nickel superalloy subjected to LCF at room temperature. However, in their study, only grain-averaged dislocation density could be estimated from measuring the peak profile variation (peak position and bordering) and the dislocation structure established within grains was not accessible.

It is of interest to note that as the sample became cyclically stable after about 200 cycles, the total dislocation density also reached a stable state. Although the dislocation density shows small fluctuations with further cycles, no considerable increase or decrease was observed to occur. However the GND density showed a different tendency to that for the total dislocation density with

cycles, shown in

Figure 11 and Figure 12, which indicates that after cyclic hardening, the GND density apparently decreases continuously with increasing fatigue cycles. However, a concern was raised when a larger size of map was acquired at 5200 cycles.

As highlighted in Figure 16, a clear 'picture frame' is shown for the ROI scanned for 16 times, and seems to indicate a very different GND density compared to points outside of the ROI. Figure 16 indicates that repeated EBSD preparation and scanning over the same area can reduce the apparent stored GND density in that area. We suspected the effect might be due to a surface carbon contamination layer built up by the electron beam hitting hydrocarbons and other contaminants

within the chamber and adhering them to the free surface. To prove this hypothesis, a part of the sample was coated with a 5-10nm carbon layer and an EBSD map was captured at the coating boundary such that clear coated and uncoated areas can be seen (as shown in Figure 17 (a) and (b)). Figure 17 (d) and (e), this 5-10nm carbon layer led to a significant decrease in GND density as suspected. The carbon layer led to electron beam scattering and diffusion before generation of the diffraction pattern. It seems that the diffused electrons travelled larger distances than the step size, and the EBSD pattern was generated from a larger area over which the GND density would be averaged. Hence, it is argued that the observed GND density drop shown in Figure 12 is simply an artificial artefact rather than a genuine observation of GND density decrease. Despite this problem, structures within the GND maps seem consistent (and evolving locally) and indicate the microstructurally-sensitive accumulation of GNDs which may affect crack nucleation and microstructure-sensitive crack growth.

The GND density distributions observed, and their evolutions, can be described with a $\sim$ log-normal

distribution as shown in

Figure 11. This is consistent with related work by Jiang et al [31, 51] for measurements of GND density distributions in deformed copper under tension and cyclic loading. Testing precisely for a log-normal distribution, compared with a skew or Rayleigh distribution (as suggested by Pantleon [56]) is difficult. Consistent forms of these distributions between tension and cyclic deformation suggest that the GND density accumulation is broadly similar with monotonic or cyclic deformation. Of course it is likely that other defects, such as spatially resolved SSDs, in the form of dipoles and persistent slip bands [33, 35], as well as point defects, may be different and this is not tested here.

As the mean load is non-zero and tensile ratcheting occurs during cycling. It is interesting to observe the residual stress magnitudes increasing systematically with increasing fatigue cycles as shown in Figure 14 (a) and (b). This implies that cyclic deformation leads to residual stress 'accumulation'. Furthermore, the development of the intricate patterning of the residual stress fields is generally smoother than the dislocation bands (due to elastic fields extending further than accumulated dislocation networks). The form of the patterning starts early in the cyclic deformation process and develops with increasing numbers of cycles implying that residual stress patterning was determined in the first few cycles, likely due to the elastic/plastic mismatch between grain orientations and

matrix and inclusion. The patterning did not alter by increasing cyclic deformation and it remained the magnitude which did vary with fatigue cycles.

Inspection of Figure 10 reveals complex, microstructurally-sensitive dislocation networks are readily formed. Generally GND density hot spots tend to form bands which can link across several grains and accumulate around triple junctions and the tips of twin boundaries. Compared to monotonic and cyclic loading in copper in Jiang et al [31, 51, 57], very similar dislocation networks and hot spot accumulation trends are formed in the cyclically deformed nickel. The form and structure of the bands here follow a 45 degree angle to the macroscopic stress axis and coincide with the inclusion. The band is co-located with regions of high residual stress (as shown in Figure 10), and both the GND band and that for high stress may be strong indicators for the microstructurally-sensitive crack path.

In order to understand the relationship between GND density hot spots and microstructure, preliminary work to identify GND density hot spots was carried out by Jiang et al [47]. However, more quantitative maps with representative sampled volumes are required in order to provide more conclusive findings. In related studies, the complementary technique of crystal plasticity modelling has been demonstrated to capture GND density and residual stress distribution near non-metallic inclusions, generated during heat treatment in Ni single and polycrystals [58]. In addition, recent work using this approach has enabled the microstructural sites of crack nucleation, for a range of differing microstructures in steel, to be identified correctly and predicted [59]. The experimental technique utilised in this work, namely high-resolution EBSD, provides the detailed microstructure-level information with which good predictive models, including those for fatigue crack nucleation, may be developed.

Dislocation density, and particularly high GND density, has been shown in this study, to be indicative of both the location of crack formation and of subsequent growth path. In connection with the former, independent experimental and crystal plasticity computational studies in ferritic steel samples have indicated the importance of stored energy density (is Jm^{-2}) in determining both the location of crack nucleation, and in predicting cycles to nucleation [14]. The stored energy, it is argued, accrues by virtue of the establishment of sessile dislocation structures of both geometrically necessary and statistically stored types, both of which have been incorporated in determination of stored energy in dislocation-based crystal plasticity models. The stored energy as the mechanistic basis for defect nucleation has led to some success in capturing observed crack nucleation sites, and cycles to defect nucleation, and the present experimental observations support the hypothesis.

5. Conclusion

Load controlled low cycle fatigue testing was conducted on an annealed FGH96 nickel superalloy containing non-metallic inclusions. An inclusion near the maximum central tensile strain area was selected and EBSD mapped 16 times from crack nucleation through to fatigue crack propagation into surrounding grains. HR-EBSD was used to calculate maps of GND density, and residual stress, as well as an estimate of the total dislocation density. The development and distribution of these quantities as a function of number of fatigue cycle were evaluated and the relationships between residual stress, high dislocation density and crack path assessed. The following conclusions are presented.

1. The fatigue crack first nucleated within the non-metallic inclusion after 2 cycles and was found to be constrained within the inclusion for the first 4800 cycles. It began to propagate out of the inclusion and into the Ni matrix after 4800 cycles.
2. Significant cyclic hardening took place while increases in GND density and total dislocation density occurred in the first 200 cycles until a stable state was achieved. From this point, plastic ratcheting continued but the measured dislocation density did not continue to increase with increasing number of cycles.
3. A significant increase of residual stress was observed in the first 400 cycles, which was found to continue to increase with increasing numbers of cycles.
4. Qualitative assessment showed that an intricate microstructurally-sensitive GND network is formed after 2 cycles. The GND network coincided with the inclusion and was concentrated along a 45 degree band to the primary deformation axis.
5. Residual stress distributions were formed in the first two cycles and were found not to alter with further cyclic deformation. However, the magnitudes of the stresses were found to increase with fatigue cycles.
6. The GND density distributions in the cyclically deformed sample were observed to be similar to those in independent tests on monotonically deformed samples, both of which may be described by a log-normal function.
7. The initial locations of hot spots of GND densities and residual stresses at two loading cycles were found to coincide with the observed fatigue crack path formed at 4800 cycles, lending credibility to the hypothesis that local stored energy rate provides the mechanistic basis for defect nucleation and microstructurally-sensitive crack growth in fatigue.
8. Re-scanning the same region of the EBSD map led to an accumulated layer of carbon contamination that diffuses back scattered electrons to a relatively large distance. This affected GND density and caused a rather significant GND density drop. Hence, care must be taken when in-situ deformation tests are to be conducted where the HR-EBSD technique is to be used to evaluate GND density at the same ROI for a number of times.

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Appendix:

Estimation of total dislocation by the restricted second moment of probability

Figure 18, schematically illustrating that stress field is dominated by a single edge dislocation toward the patch centre.

Imagining that a material can be partitioned into a series of patches with area of A_0 as shown in Figure 18, stress increases significantly toward to the dislocation in the centre. Assuming all dislocations is edge type; the stress near a single edge dislocation with dislocation line along the x_3 axis and Burgers vector magnitude b along the x_1 axis can be expressed as:

$$\sigma_{ij} = \frac{DK_{ij}}{r} \quad \text{Equation 1}$$

Where the r is the radial distant from the dislocation core and θ is the angle from x_1 axis constant D is a constant given in terms of the shear modulus G and Poisson ratio ν by

$$D = \frac{Gb}{2\pi(1-\nu)} \quad \text{Equation 2}$$

The K_{ij} is the angular dependence of the stress. For shear stress σ_{12} , K_{12} is given by

$$K_{12} = \cos(\theta) \cos(\theta) \quad \text{Equation 3}$$

The probability of high stress near dislocation can be established from the fraction area of the patch as shown in Figure 18

$$P = \frac{dA}{A_0} = \frac{rd\theta dr}{A_0} \quad \text{Equation 4}$$

Combining Equation 5 and Equation 6 leads to

$$P(\sigma, \theta) = \frac{D^2 K^2(\theta) d\sigma}{A_0 \sigma^3} \quad \text{Equation 5}$$

Integrating around an annulus radius r with width dr allows the angular variation to be averaged, giving the probability as a function of stress:

$$P(\sigma) = \frac{D^2 \langle K^2 \rangle(\theta) d\sigma}{A_o \sigma^3} \quad \text{Equation 6}$$

Where

$$\langle K^2 \rangle = \int_0^{2\pi} K^2(\theta) d\theta \rightarrow \langle K^2 \rangle = \pi/2 \quad \text{Equation 7}$$

From equation 6 we can see that the probability of single dislocation within a patch area A_o is proportional to σ^{-3} . As dislocation density ρ_0 is given by $1/A_o$, dislocation density is hence proportional to σ^3 . As the experimental results are rather noisy, here we follow Groma's approach and use the restricted second moment v_2 of probability

$$\begin{aligned} v_2(\sigma) &= \int_{-\sigma}^{+\sigma} P(\sigma) \sigma^2 d\sigma \rightarrow \frac{D^2 \langle K^2 \rangle}{A_o} \ln(\sigma) = D^2 \langle K^2 \rangle \rho_0 \ln \frac{\sigma}{\sigma'} \\ &= \frac{(Gb)^2}{8\pi(1-\nu)^2} \rho_0 \ln \frac{\sigma}{\sigma'} \end{aligned} \quad \text{Equation 8}$$

Therefore total dislocation density ρ_0 can be estimated by determining the gradient of v_2 as a function of $\ln \sigma_{12}$.