A statistical study of the relationship between plastic strain and lattice misorientation on the surface of a deformed Ni-based superalloy

A. Harte*, M. Atkinson, M. Preuss, J. Quinta da Fonseca

University of Manchester, Department of Materials, Sackville Street Building, Manchester, M1 3BB, UK

1. Introduction

The deformation response of an advanced polycrystalline alloy is determined by the deformation behaviour and interactions of the constituent phases and grains that make up its microstructure [1]. Studying these interactions is a powerful way of understanding the strengthening and damage mechanisms that control their performance, which in turn will help develop new, improved alloys and tailor microstructures for a given application. One of the prevailing ideas is that damage develops through the localisation of stresses and plastic strains at the microstructural scale, which leads eventually to failure, e.g. [2]. The local plastic strain can create an accumulation of defects (dislocations and vacancies) that lead to damage but can also produce stress concentrations, e.g. ahead of blocked slip bands [3], that cause damage. However, not all plastic strain creates defects and local plastic strain is difficult to measure at the microstructural scale, particularly in the bulk and post-mortem. Instead, researchers often use misorientation data from electron backscatter diffraction (EBSD) as a proxy for local strain and to link local deformation to damage.

EBSD can provide a quantification of local lattice distortion, measured as lattice misorientation [4,5]. The usefulness of this technique for measuring deformation has been demonstrated in the literature. For example, average map misorientation, calculated by reference to the mean orientation of each neighbour in the deformed state, correlates well with applied plastic strain during both quasistatic loading in tension [6-8] and in compression [7], as well as during creep [7,9,10], and geometrically necessary dislocation map averages have been shown to correlate to the global plastic strain in polycrystalline copper under tension [11] but not in single crystal nickel [12]. Since grain misorientation uses a reference point in the grain it can be readily measured and mapped using EBSD, both post-mortem and in the bulk, via sectioning. Average map misorientation correlates well with the applied macroscopic strain, but local lattice misorientation should not correlate spatially with plastic deformation. This is because lattice misorientation is produced by gradients in plastic strain [13] and therefore it is not proportional to the magnitude of plastic strain but rather to its heterogeneity. For example, slip band termination at a grain boundary will result in
local strain gradients and misorientation [14,15] and the extent of the misorientation will depend upon grain boundary character, dislocation slip type and the boundary condition associated with the free surface [16]. Therefore, lattice misorientation data is difficult to interpret without knowledge of the underlying local plastic phenomena. In metallic microstructures, deformation gradients and therefore lattice misorientation can be caused by the deformation incompatibility between different microstructural constituents, like different phases or grains of the same phase but with different crystallographic orientations, or by plastic instability within individual crystals [17]. This explains why mean grain misorientation correlates with macroscopic plastic strain. In a polycrystalline specimen hardening the strain gradients within individual grains should generally increase with increasing plastic strain. However, the correlation between plastic strain and local lattice rotation (as measured by misorientation) breaks down at the grain level, as demonstrated for copper deformed in tension to 5% [18]. Although covering hundreds of grains, in this copper study the EBSD misorientation measurements were performed at a very different spatial resolution to the strain measurements and therefore the origins of the scatter could not be studied in detail. Despite this, the EBSD measurements of lattice distortion are often used as a proxy for locating and estimating plastic strain levels [19–21] and have been used to identify different fracture modes [22] and crack initiation modes [23]. Lattice distortion has been correlated to local stress corrosion crack (SCC) initiation sites [24] and shown as localised along crack paths [22], suggesting a relationship between local deformation and crack initiation and propagation, respectively. Lattice distortion has been shown as a variable when surrounding different types of SCCs [25] and it has been used to quantify the effect of the annealing temperature on residual deformation and the resulting effect of this localised deformation on rates of corrosion [26].

In this paper we aim to make a direct comparison between surface lattice misorientation and plastic strain at high spatial resolution and over a wide field of view containing a large number of grains in an attempt to achieve measurements that are statistically representative of the surface studied. We carried out these measurements on a precipitation-strengthened Ni superalloy deformed to a small plastic strain of around 2%, a level of plastic strain that is relevant to the small deformations and lattice rotations experienced in fatigue testing and in service and typically found in previous attempts to use EBSD to quantify plastic strain. We used automated data acquisition to cover an area 1 × 0.5 mm with a spatial resolution of ~120 nm and developed automated data analysis routines for the manipulation of sub-grain scale data, making it possible to correlate directly HRDIC plastic strain information and EBSD lattice distortion information [27]. Because there are many different ways to manipulate EBSD data in order to obtain lattice distortion information [5,28–30], we calculated nine different measures of lattice misorientation from the same Hough-indexed EBSD data set. These values were compared to the local plastic strain, measured using HRDIC, both at the mean grain-to-grain variation scale and on the pixel-to-pixel sub-grain scale. Finally, we assessed the validity of global boundary condition orientation-based predictors of deformation, such as the Taylor factor and Schmid factor in a statistical manner by looking for correlations between these predictors and the actual plastic deformation and lattice misorientation measured on the grain scale. These calculations are easily performed in commercial EBSD post-processing software and often used to understand local plastic deformation at the microstructural scale [19–21]. However, we demonstrate that these predictors break down at the local level and cannot be used to predict the magnitude of local strain.

2. Material and experimental methodology

2.1. Material and sample

The material studied was polycrystalline Ni-based superalloy RR1000 following uniaxial tension. RR1000 is a γ/γ’ alloy that was developed at Rolls Royce plc. for use as turbine disks in the rotational hot sections, has high tensile strength and retains good fatigue properties over its operational lifetime [31]. Its development is detailed in a comprehensive review elsewhere [32]. Here, we have applied heat treatments to obtain a unimodal distribution of γ’ size [33]. This was achieved through first applying a γ’ super-solvus solution heat treatment followed by an oil quench and a γ’ sub-solvus heat treatment with subsequent very slow cooling to promote growth of existing γ’ rather than nucleation of new one [33]. All heat treatments were performed in an Ar atmosphere. This procedure resulted in a unimodal size distribution of γ’ precipitates with diameter ~ 250 nm and a volume fraction ~ 40%. In this microstructure slip predominantly occurs by cutting of the γ’ particles [34] and therefore deformation heterogeneity between the two phases can be ignored, i.e. it can be treated as a single phase polycrystalline material for the purposes of this study. A characterisation of the grains after heat treatment is provided in Fig. 1, in which we show the average grain orientations and the grain size distribution (both post-deformation) in parts a) and b), respectively. The grain size was determined as the diameter of the equivalent circle of the grain area, and the mean grain size is ~20 μm. The inverse pole figure (IPF) scatter-plot in part a) is shown from the loading direction and demonstrates that the texture is random. Almost half (0.47) the grain boundaries in this microstructure are Σ3 annealing twin boundaries.

The tensile specimens were machined by electric discharge machining (EDM) to the geometry detailed in Fig. 1 part c). The EDM recast layer was removed by grinding and polishing with a final step of polishing with colloidal silica (0.06 μm) for ~10 min before pre-deformation orientation mapping. Then, a gold speckle pattern was applied to the sample surface to act as fiducial markers for the image correlation process. Images of the speckle pattern were obtained before and after uniaxial tensile deformation and the resulting displacement field was calculated. The gold particles were removed by
polishing with colloidal silica before post-deformation EBSD. The following contains details relating to this procedure. All displacement data, orientation data and in-house data manipulation procedures in python have been made open access and are available to the community [27,35].

2.2. Orientation mapping

Orientation mapping was performed in the region of interest both before and after deformation using a field emission gun (Zeiss Sigma) SEM equipped with an Oxford Instruments electron backscatter diffraction (EBSD) system, consisting of a NordlyNano detector and Aztec software version 3. In the non-deformed state the EBSD scans were obtained before gold remodelling and in the deformed state the gold layer was removed by ~60 s of polishing with colloidal silica. Scans were performed at an operating voltage of 30 kV and a probe current of ~7.8 nA using the 120 μm aperture in high current mode. An area of ~1 × 0.5 mm was scanned with a step size of 0.5 μm at an acquisition rate of ~35 Hz. The Oxford Instruments Aztec software was used for Hough-based indexing of diffraction patterns by comparison to theoretical patterns.

2.3. High resolution digital image correlation

2.3.1. Gold remodelling

The EBSD-induced carbon contamination was removed by a ~30 s polish with colloidal silica and a gold speckle was then applied to the sample surface using the gold remodelling technique [36]. A 25–40 nm thick gold layer was deposited onto the sample surface using an Edwards S150B sputter coater at a rate of 5–8 nm min⁻¹. Following this, the remodelling was performed in a water vapour environment for 3 h. This was achieved by placing the sample on a hotplate at 300 °C together with a beaker of water and a larger, inverted beaker to cover both the small beaker and the sample and topping up the water as necessary.

2.3.2. Image acquisition

Backscattered electron images of the gold speckle pattern were obtained before and after the deformation step using a FEI Magellan HR 400 LE-SEM. To maximise the spatial resolution, the microscope was operated at a voltage of 5 kV with a 2 kV stage bias and a probe current of 0.8 nA. A working distance of 4 mm was chosen to maximise the signal-to-noise ratio. A mosaic of 40 columns × 20 rows was used to collect 800 images with a 20% overlap, corresponding to a field of view of ~1 × 0.5 mm². Each image was of resolution 2048 × 1768 pixels and a pixel size of 14.6 nm. After the deformation step, great care was taken to ensure that the contrast and brightness of the post-deformation images closely represented those in the non-deformed state in order to minimise systematic errors.

2.3.3. Mechanical testing

The dogbone sample was deformed in uniaxial tension using a Zeiss–Kamrath 5 knm tension-compression microtester. The sample was deformed at a rate of 0.3 mm min⁻¹ to a global macroscopic uniaxial engineering strain of ~0.02. The engineering stress–strain curve is shown in Fig. 1 part d). After testing, the sample was removed from the tester and post-deformation images were obtained in the unloaded state according to Section 2.3.2.

2.3.4. Image correlation

The image mosaics were stitched together using the Grind/Collection stitching plugin for FIJI [37] and cropped into 8 separate areas for image correlation due to maximum image size limitations for the DIC calculations. The stitched images from the deformed and non-deformed state were then correlated using LaVision’s commercially available DIC software, DaViS 8 [LaVision, 2018] [38]. The systematic error was calculated by comparing two images in the non-deformed state. These images were from the same region but the sample was removed from the microscope and replaced before taking the second image. The two images were then correlated using decreasing interrogation window sizes to obtain the optimal interrogation window size [36]. We found that using a sub-window size of 8 × 8 pixels and no overlap resulted in the best compromise between spatial resolution, at around 120 nm, and the mean systematic error at approximately 0.01 effective strain (εэфф) over approximately 32 million data points. The image correlation results in full-field ut, uτ displacements on the x1, x2 plane with normal x3. The individual components of the displacement gradient tensor can then be found by the gradient of the displacement with respect to the macroscopic directions using Eq. (1).

\[
\frac{\partial u_x}{\partial x} = \left[\begin{array}{c}
\frac{\partial u_1}{\partial x_1} \\
\frac{\partial u_2}{\partial x_2}
\end{array}\right]
\]  \( (1) \)

It is convenient to visualise the local strain by combining the four components of the displacement gradient tensor into an effective strain, ϵэфф [1]. This is a shear-dominated term, which reflects the characteristic slip, and can be calculated using Eq. (2).

\[
\epsilon_\text{эфф} = \sqrt{\frac{1}{2} \left(\frac{\partial u_1}{\partial x_2} - \frac{\partial u_2}{\partial x_1}\right)^2 + \frac{1}{2} \left(\frac{\partial u_3}{\partial x_1} + \frac{\partial u_1}{\partial x_3}\right)^2}
\]  \( (2) \)

2.4. Data analysis routines

The EBSD orientation data and HRDIC displacement data was analysed using our own Python routines with NumPy for numerical computation [39] and Matplotlib for visualisation [40]. The routines are contained within the open source DefDAP library (Deformation Data Analysis in Python) [27]. The manipulation of orientations was performed with quaternion geometry, the details for which are well described elsewhere [41–43], and the grain-level misorientation was calculated in one of nine ways, as detailed in Table 1. All of these measures of misorientation calculate the pixel misorientation in the deformed state. One of the misorientation calculations considered is the kernel average misorientation (KAM). The KAM considers the local misorientation by the mean orientation difference to the pixels that surround it. The KAM calculation here was performed for a 3 × 3 array that surrounds the central pixel of interest and the misorientation was not allowed to exceed 5° so as to exclude high angle grain boundaries. The CMA measure of misorientation [30] can be thought of as a non-correlated KAM where the misorientation is calculated between each pixel and every other pixel in a grain and then the cumulative distribution of all the grain misorientations is plotted as a Weibull plot in order to determine the misorientation for which the cumulative distribution is equal to 0.632.

Seven of the measures of misorientation in Table 1 are variants of the grain reference orientation distribution (GROD) [29]. For the GROD the pixel misorientation is calculated for each pixel within a

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Lattice misorientation and GROD variant definitions.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lattice misorientation</td>
<td>Description</td>
</tr>
<tr>
<td>KAM</td>
<td>3 × 3 nearest neighbour kernel</td>
</tr>
<tr>
<td>CMA</td>
<td>Non-correlated KAM</td>
</tr>
<tr>
<td>GROD variants</td>
<td>Reference orientation</td>
</tr>
<tr>
<td>Mean</td>
<td>Average orientation</td>
</tr>
<tr>
<td>PQmax</td>
<td>Best Kikuchi pattern quality</td>
</tr>
<tr>
<td>Min KAM</td>
<td>Lowest kernel misorientation</td>
</tr>
<tr>
<td>Mean Min</td>
<td>Lowest GROD Mean</td>
</tr>
<tr>
<td>Mean Max</td>
<td>Highest GROD Mean</td>
</tr>
<tr>
<td>Original orientation</td>
<td>Pre-deformation orientation</td>
</tr>
<tr>
<td>Centroid</td>
<td>Euclidean grain centre</td>
</tr>
</tbody>
</table>
grain relative to a reference orientation. Those reference orientations may be in the deformed or the non-deformed state.

- For the GROD Mean the reference orientation is the mean orientation in the deformed state.
- For the GROD PQMax the reference orientation is that of the pixel in the deformed state with the best Kikuchi pattern value.
- For the GROD Min KAM the reference orientation is the pixel in the deformed state with the lowest $3 \times 3$ KAM value.
- For the GROD Mean Min and Max the reference orientation is the pixel in the deformed state with the minimum or maximum GROD Mean value, respectively (note that no pixel in the GROD Mean distribution will have a GROD Mean value of exactly zero).
- For the GROD Original the misorientation is calculated as the pixel orientation relative to the grain mean orientation in the non-deformed state.
- For the GROD Centroid the reference orientation is pixel in the deformed state with the greatest Euclidean distance from the grain boundary.

Note that while most misorientation functions explored here can be mapped at the sub-grain scale, the CMA cannot. The EBSD grain boundaries were defined as neighbouring pixels with a misorientation greater than 6° which successfully delineated individual grains and twins. The boundaries were then used to mask grains for both the EBSD and the HRDIC data. The EBSD grain boundaries were mapped onto the HRDIC data using manually selected homologous points (usually triple junctions that are obvious in both data sets) and an affine transform (resize, shear, rotation) via software implemented in Python. Single grain analysis of the HRDIC plastic strain data then becomes possible for hundreds of grains, as does its correlation to lattice misorientation data from EBSD. The sub-grain comparisons of strain to misorientation required smoothing of the strain data to obtain more of a continuum of strain, which was performed by applying a Gaussian kernel to the strain data using the convolution package within the Astropy library [44]. The sub-grain pixel-to-pixel correlations were performed by binning the grain strain data to the EBSD spatial resolution and performing a Spearman’s rank correlation test on the data using the stats package within the Scipy library [45].

![Image](image_url)

**Fig. 2.** The HRDIC effective strain ($\varepsilon_{\text{eff}}$) map at macroscopic $e_{\text{max}} \sim 0.02$, field of view 1 x 0.5 mm, spatial resolution 0.117 nm. The full resolution image can be found in the supplementary material. G1–G5 highlight grains with different slip character and the effect of this on HRDIC $\varepsilon_{\text{eff}}$ and EBSD lattice misorientation (GROD Mean). The region of interest (ROI) is used to exemplify different ways to calculate lattice misorientation in Fig. 3.
statistical significance of the Spearman’s rank was judged based on the value of the resulting coefficient in accordance with common practice [46], as significance tests produced unreasonably small p values due to the large number of data points in the correlations.

3. Results

3.1. The spatial relationship between strain and misorientation at the mesoscale and mean grain scale

The effective strain (\(\varepsilon_{\text{eff}}\)) map at \(\varepsilon_{xx} \approx 0.02\) is shown in Fig. 2 on a log scale. In this map the tensile axis is horizontal, the field of view is \(\sim 1\) mm by \(0.5\) mm and the spatial resolution is \(\sim 120\) nm. The supplementary material contains a full resolution image and a link to a video overview of this data set. Five grains are highlighted in the figure to demonstrate different slip characteristics, G1-G5. The \(\varepsilon_{\text{eff}}\) data and the EBSD lattice misorientation data, \(\phi\), based on GROD Mean (G Mean) are shown together for each grain G1-G5. We observe discrete, localised slip bands that are contained in individual grains, sometimes of a single slip plane (G1) and sometimes of two or three slip planes (G2, G3, respectively), but always on {111} (confirmed by slip trace analysis utilising grain orientation data from EBSD). We also observe cross slip (G4) and more diffuse strain within grains (G5), the latter possibly discrete but if so then beyond the resolution of the strain data. Importantly, we observe diffuse strain bands that span many grains at \(\sim \pm 45^\circ\) to the tensile axis that cannot be attributed to single grain behaviour. Grains with complicated slip geometries (G4) often show correspondingly complex distributions of lattice misorientation and grains with diffuse slip (G5) can result in unexpected misorientation distributions, such as misorientation banding that does not correspond to plastic slip bands (G5).

To investigate the impact of calculating misorientation (\(\phi\)) in different ways, we have taken the EBSD data from the region of interest (ROI) in Fig. 2 and plotted nine different lattice misorientation measures, each calculated from the same EBSD data set, which we display in Fig. 3. The name of each calculation is given above the figure and these names relate to the descriptors detailed in Table 1. There are significant differences in the magnitude and spatial

Fig. 3. The ROI sub-region in Fig. 2 is compared here to the EBSD misorientation data, calculated nine different ways.
which is primarily due to their lack of range: the GROD Mean Max and Centroid functions do not cover the low strain range and the PQ Max does not cover the high strain values.

In Fig. 3 we show how the misorientation is calculated affects the magnitude and localization of lattice misorientation within grains. We know that the map average misorientation is positively correlated to the macroscopic strain. To test whether this relationship holds at the grain level, we have taken the mean of the $\epsilon_{\text{eff}}$ values within a grain ($\bar{\epsilon}_{\text{eff}}^f$) and plotted it against the mean of the misorientation values within that grain ($\bar{\phi}$) for each of the nine calculations for misorientation. In Fig. 5 we plot these grain mean correlations for ~600 grains for each of the nine methods. Most correlations are positive, but some have a higher gradient than others and all of the data have significant scatter. In most of the correlations between $\epsilon_{\text{eff}}$ and $\phi$ the larger grains show a higher $\phi$ for a given $\epsilon_{\text{eff}}$. However, in the correlation between strain and grain average $3 \times 3$ KAM in Fig. 3(i) the largest grains have the smallest mean misorientations. Of all of the $\phi$ measures, the KAM is the most sensitive to step size and grain size because the kernel size must be defined with respect to these length scales. Grain distribution measurements that do not require a kernel, such as GROD, are much less sensitive to step size [8,28,30].

We can obtain values that reflect the sensitivity and reliability of each of the $\epsilon_{\text{eff}} - \bar{\phi}$ correlations by performing a simple linear regression analysis. We define the sensitivity of $\bar{\phi}$ to changes in $\epsilon_{\text{eff}}$ by calculating the gradient of the $\epsilon_{\text{eff}}$ - $\bar{\phi}$ correlation ($m$) and we define the reliability of $\bar{\phi}$ as an indicator of $\epsilon_{\text{eff}}$ by calculating the correlation coefficient ($R$) and the standard error of the moving mean (SE). In this way, the $\epsilon_{\text{eff}} - \bar{\phi}$ correlations with a high $m$, high $R$ and low SE are the most sensitive and reliable correlations. While a significance test can be performed for linear regression, the resulting probability $p$ values are very sensitive to the sample size and, in this case, results in unrealistically low values. Instead, we use the concept that a value of $R > 0.3$ indicates a low but detectable correlation [46]. In Fig. 6 part a) we show these three correlation descriptors for each of the nine correlations and include a dotted green line to show the $R = 0.3$ limit for detectable correlation. Only the $\epsilon_{\text{eff}} - \bar{\phi}$ correlations using the GROD KAM Min, GROD PQ Max and the $3 \times 3$ KAM result in non-detectable correlations, which might be expected due to the sensitivity of the KAM to the grain/step/kernel size and the randomness with which the pixel with the highest pattern quality might be selected. The other $\epsilon_{\text{eff}} - \bar{\phi}$ correlations, and especially where $\bar{\phi}$ is taken from the GROD Mean, KAM and CMA, give sensitive and reliable correlations. While the $\bar{\phi}$ taken from the GROD Mean gives the most sensitive correlation, it has a large standard error and is not as reliable. To answer the question of which is the best grain mean correlation in Fig. 5, we have combined these correlation descriptors as $Rm$ and $Rm/SE$, the latter giving more weight to the standard error, which is also included in the calculation of $R$. In Fig. 6 part b) show how $Rm$ and $Rm/SE$ vary for each of the nine $\epsilon_{\text{eff}} - \bar{\phi}$ correlations. The greater the value of this combined statistic, the better $\bar{\phi}$ acts as a predictor of $\epsilon_{\text{eff}}$. If considering $Rm/SE$, the best predictors of grain mean strain are the GROD Mean and the CMA; note that the GROD Mean Min and GROD Centroid have similar values to the GROD Mean due to their similarity in spatial distribution in the strain. However, if considering $Rm$ then the best measure of $\bar{\phi}$ is the GROD Mean Max, largely due to its steep correlation gradient. The GROD Mean Max is calculated using a reference orientation that is the most severely rotated away from the mean orientation, which therefore highlights misorientation heterogeneity: the GROD Mean Max may indeed be the most suitable indicator of strain field because the $\epsilon_{\text{eff}}$ distribution is highly heterogeneous.

### 3.2. Correlations between plastic strain and lattice misorientation at the sub-grain scale

To approach the problem of what measures of misorientation can tell us about damage mechanisms, we must study the spatial...
relationship between strain and misorientation within the grain. We do this here through a pixel-by-pixel comparison within each grain between $\varepsilon_{\text{eff}}$ and $\phi$. In correlating the mean grain values $\varepsilon_{\text{eff}}$ and $\phi$, the GROD Mean Max and GROD Mean give the most sensitive and reliable correlations and so we will use these measures for the sub-grain correlations. We will also use the KAM because it is itself spatially resolved due to its kernel averaging and it is so frequently used in the literature [8]. Within grains the plastic strain ($\varepsilon_{\text{pl}}$) is highly localised in slip bands but the misorientation varies smoothly. We have performed a smoothing and coarsening function to the $\varepsilon_{\text{eff}}$ data, resulting in a continuum of strain ($\varepsilon_{\text{C}}$) in an attempt to improve the pixel-to-pixel comparisons. This was achieved by applying a 2D Gaussian filter to the $\varepsilon_{\text{eff}}$ over a square kernel of $11 \times 11$ pixels. We have also calculated the gradient of this coarsened strain ($\nabla \varepsilon_{\text{C}}$) as the root of the sum of the squares of the gradient in the horizontal and vertical directions.

Although some authors have reported no correlation between the plastic strain and the lattice misorientation at the sub-grain scale [8], we see strong spatial correlations in some cases, depending on how the data is treated and the slip characteristics in a particular grain. Some grains deform relatively homogeneously and an example of such a grain is shown in Fig. 7: in this grain the slip bands are evenly spaced and of similar intensity but they fade or change in character as they approach the grain boundary. The strain component inserts in Fig. 7 show that the strain is reasonably homogeneous when smoothed and the gradient of that strain field is highest at the grain boundaries. For this analysis we use the Spearman’s rank ($\rho$) as a measure of correlation strength, which, unlike Pearson coefficient, is non-parametric and does not assume normal distribution nor linearity, only monotonic differences. The pixel-to-pixel correlations for this grain show that the spatial correlation is strongest and positive between the strain gradient and the KAM ($\rho = 0.64$), closely followed by the GROD Mean ($\rho = 0.62$). Therefore, it is the interaction of the slip with the microstructural grain boundaries that govern the relationship between plastic strain and lattice misorientation in this grain. In some grains the strongest correlation is a negative correlation, especially between the coarsened strain and the GROD Mean or KAM. This negative correlation occurs largely because the misorientation is positively correlated to the strain gradient. Some grains deform more heterogeneously and an example of such a grain is shown in Fig. 8; in this grain the slip bands bifurcate in the grain centroid in the vicinity of a penetrating twin. The bifurcation is associated with greater values of strain in the grain centroid, which is highlighted in the coarsened strain map. The pixel-to-pixel correlations for this grain show that the spatial correlation is strongest and positive between the coarsened strain and the GROD Mean Max ($\rho = 0.75$). For this grain, the measure of misorientation that creates the greatest amount of grain-interior misorientation heterogeneity is the best indicator of local plastic strain. In other grains there can be large amount of strain in one region that fades towards a grain boundary, associated with a high GROD Mean at the boundary, and no strain in another region of the grain; in this case the GROD Mean Max will be strongly but inversely correlated to the strain.
We have performed the sub-grain pixel-to-pixel analysis for 613 grains and show the distribution of the Spearman’s rank for each of the nine correlations ($r_{eff}, r_{eff}^C$ and $\nabla r_{eff}$, correlated each to GROD Mean, KAM and GROD Mean Max) in Fig. 9. Correlations are stronger as the Spearman’s rank approaches ±1 and the positions of $\rho = \pm 0.3$ are shown by vertical lines to indicate where a correlation becomes detectable. It is immediately clear that the correlations between the $r_{eff}$ and misorientation are the poorest with a high fraction of grains showing $\rho \rightarrow 0$. The widest distribution and therefore the best sub-grain spatial correlation is between $r_{eff}^C$ and GROD Mean Max but there are also some strong positive correlations between $\nabla r_{eff}$ and GROD Mean and the KAM. Each of the 613 grains has a maximum positive correlation $\rho \rightarrow 1$ and a minimum inverse correlation $\rho \rightarrow -1$. The distribution of these maximum positive and inverse correlations is shown in Fig. 10a, together with guide lines for the $\rho = \pm 0.3$ condition for a detectable spatial correlation. From this histogram we find that the positive correlation is stronger than the inverse correlation in 60.2% of grains and that a total of 87.9% of grains have a detectable correlation ($\rho > 0.3$, $\rho < -0.3$) between one of the measure pairs: 52.2% positive correlations and 35.7% inverse correlations. We show which correlation pair gave the strongest positive and inverse correlation in Fig. 10b as a bar chart for each of the nine pairs. From this bar chart it is clear that the strongest sub-grain pixel-to-pixel correlations are most common between the coarsened strain and the GROD Mean Max. While Fig. 8 is an example of this positive correlation, the bar chart in Fig. 10b shows that the correlation can be both positive and inverse with almost equal probability. Therefore, while GROD Mean Max might give a good indication of the magnitude of strain and its heterogeneity within a grain, it cannot be relied upon for the spatial location of strain; for a positive spatial correlation the one should use the GROD Mean or the KAM to infer the spatial location of the gradient of plastic deformation.

We have shown that while the GROD Mean and KAM measures of misorientation are the most reliable indicators of local strain gradients, the GROD Mean Max gives the strongest local positive and inverse correlations due to its inherent measure of heterogeneity. The suitability of one particular EBSD-based measure of misorientation in representing the distribution plastic deformation depends upon the number and relative strength of slip modes active in that grain and their interaction with microstructural features such as grain boundaries. Relative slip activity is affected by crystal orientations and constraint and will be different at the surface and in the bulk.

### 3.3. The influence of grain orientation on grain deformation

Since the relative slip activity is affected by orientation, it is plausible that grain orientation correlates with measures of local strain and misorientation. To assess the influence of grain orientation on the magnitude of plastic strain, we have plotted the grain mean strain on a colour scale within an IPF (loading direction) based on the scatter orientations in Fig. 1 part a). The texture in this material is random and we have smoothed the scatter plots by local linear interpolations to produce the grain mean $\varepsilon_{eff}$ IPF in Fig. 11. The IPF plot suggests that the grain mean $\varepsilon_{eff}$ is independent of the orientation of the grain. Since grain mean $\varepsilon_{eff}$ is positively correlated to the $\varepsilon_{eff}$, Fig. 5, we might also expect the misorientation IPFs to be random. To confirm this we have plotted the $\varepsilon_{eff}$ IPFs (loading direction) in Fig. 12 for each of the nine measures of $\varepsilon_{eff}$, and, indeed, there is no obvious correlation between the orientation of a grain and the magnitude of $\varepsilon_{eff}$. This is an important result because orientation-based predictors of deformation based on the global boundary condition, such as the Taylor factor or the Schmid factor, are often used for the explanation of observed localized deformation [48–52] but we find that on the surface of this alloy and after a strain of 0.02 there is no relationship between the orientation of a grain and its magnitude of deformation. To demonstrate this explicitly, we have calculated the Taylor factor and Schmid factor for each grain in its original, non-deformed orientation and plotted this on the IPFs (loading direction) in Fig. 13 parts a) and b), respectively.

The Taylor factor of a grain is a prediction of the amount of work required to deform that grain; all grains are assumed to undergo the same strain and so a higher Taylor factor indicates that a greater amount of work should be necessary to deform that grain [53,54]. Therefore, grains with a higher Taylor factor are often considered harder than grains with a lower Taylor factor, and would be expected to have lower values of strain. Similarly, a higher Schmid factor corresponds to a higher resolved shear stress for slip and therefore a grain with a higher maximum Schmid factor is often considered softer than a grain with a lower maximum Schmid factor. As Fig. 13 shows, however, the distribution of Taylor factor and Schmid factor in Fig. 13 shows no resemblance to the strain distribution in Fig. 11 nor any of the nine misorientation distributions in Fig. 12. To solidify this point, in Fig. 14 we plot the grain mean HRDIC plastic effective strain (data point positions) against the Taylor factor in part a) and the Schmid factor in part b) as calculated by the grain’s original, non-deformed orientation, where the colour and size of the data points relate to the GROD Mean misorientation. By plotting these values for 590 grains we are able to show definitively that, at a macroscopic axial strain of $\varepsilon_{xx} \sim 0.02$, there is no relationship between the Taylor factor as calculated using macroscopic or local boundary conditions (see Supplementary material for the latter) and the amount of plastic strain or lattice misorientation. There is also only a weak relationship between Schmid factor and effective strain: grains with low Schmid factor tend to deform less and while grains with a higher strain tend to have a higher Schmid factor, grains with a high Schmid factor do not necessarily undergo large strain.

### 4. Discussion

The global average misorientation has been shown to correlate linearly with macroscopic plastic strain [6,7,9,55] up to strains of $\varepsilon_{xx}$
The HRDIC measurements reveal that the magnitude and localization of the lattice misorientation within a grain depends on not only the magnitude of the plastic strain, but also the number and relative strength of the slip modes active in that grain and the interaction of those slip modes with microstructural features such as grain boundaries. We therefore argue that the local deformation characteristics of plastic strain and lattice rotation emerge from the complex interaction between grains during deformation and cannot be determined by simple orientation-based predictors of plasticity relative to the global deformation tensor, such as the Taylor factor or Schmid factor. This non-crystallographic strain localization is especially evident when we consider the strain bands at the mesoscale that span many grains in Fig. 2 at $\pm 45^\circ$ to the loading direction, which must arise from effects that relate to deformation compatibility and the interactions between many grains. These bands of high strain, which are also predicted by crystal plasticity finite element models [56], cross many grains with different orientations and are consistent with a lack of correlation between local strain and crystal orientation at the grain level. Further, the suitability of the lattice misorientation to describe the plastic strain depends upon the way in which the misorientation is calculated.

4.1. Orientation-based predictors of deformation

The discrete slip bands observed within grains are thought to arise in this alloy due to the low stacking fault energy of Ni-based...
superalloys and the resulting dissociation of dislocations into partials, which cannot cross-slip easily [58]. Moreover, the local shearing of the γ’ strengthening precipitates results in glide plane softening [59] and so locally the shear bands contain high strain (\(\varepsilon_{\text{eff}} \approx 0.4\)). The high spatial resolution employed in the present work allows us to measure discrete plastic events and so we are able to validate or contest traditional assumptions employed in continuum theories of plasticity.

Taylor theory [53], after von Mises [60], assumes that all grains undergo the same shape change by activating a minimum of five slip systems. While it has been noted that different parts of a grain may exhibit single slip Sachs-like behaviour, it is generally accepted that a grain will meet this five slip system criteria overall [61]. However, we observe many grains in which only one slip plane is active, incurring likely a single slip system but a maximum of three. The limited number of slip systems observed is in part due to the plane stress state experienced at the surface as is predicted by full field crystal plasticity models [62] but also because, unlike the core assumption of the full constraints of Taylor theory, the grains deform differently from one another and from the macroscopic deformation. This is not new observation, and other crystal plasticity models, be it self-consistent or full-field, account for this. Nevertheless, orientation-based

---

**Fig. 8.** The pixel-to-pixel correlations within a single grain in which the strain is relatively heterogeneous. Data arranged as a table; strain data at the top, horizontal, and misorientation data on the left, vertical. The correlations in parts a), b) and c) correlate the GROD Mean to the effective strain, the coarsened strain and the gradient of the strain, respectively. Similarly, parts d), e) and f) correlate the KAM to these measures of strain and parts g), h) and i) correlate the GROD Mean Max to these measures of strain. The Spearman’s rank (\(\rho\)) is displayed in each part.

**Fig. 9.** The frequency distribution of the Spearman’s rank (\(\rho\)) for each of the nine sub-grain pixel-to-pixel correlations.
correlations produces the maximum positive and inverse Spearman’s rank. A bar chart showing the fraction of grains in which each of the nine pixel-to-pixel correlation pairs. A histogram showing the fraction of grains that demonstrate the associated range in the absolute maximum positive and inverse Spearman’s rank.

Likewise, there may be significant plastic strain but on only one slip system. We therefore conclude that the concept of “hard” and “soft grains” based on their orientation [73] is not relevant to FCC γ/γ’ Ni-based superalloys at small global plastic deformation. The effect of grain size has been suggested as more significant than that of orientation [64] and as important for fatigue crack initiation sites [74]. In Fig. 5 the colour and size of the grain mean strain-misorientation correlation data points relate to the grain size. We observe a positive correlation between the lattice misorientation and the grain size, but this relationship is less clear for the plastic strain. Interestingly, the misorientation as measured by the grain mean KAM has an inverse relationship with grain size. This is because, in this material, many grains contain relatively large regions of single slip bands, resulting in almost no local lattice misorientation within the grain interior. Larger grains, therefore, have larger areas that correspond to these regions, which results in lower mean KAM values. Importantly, because the KAM is a very local measure of deformation it is step-size dependent and as the number of steps in a grain is proportional to the grain size, the KAM is also proportional to the grain size. The GROD variants and the CMA measure do not suffer from this issue [28,30].

4.2. Sub-grain deformation localisation

Through specific examples of grain plasticity and lattice misorientation in Fig. 2, Fig. 7 and Fig. 8 we have shown that the lattice misorientation within a grain does not only depend on the magnitude of the strain but also on the slip characteristics specific to that grain. This implies that a grain may experience low but heterogeneous plastic strain and that this would result in a large lattice misorientation. Likewise, there may be significant plastic strain but on only one slip system, resulting in grain lattice rotation but low lattice misorientation within the grain. There can be significant misorientation where there is very little plastic strain, especially in the vicinity of grain boundaries. This is because the slip bands fade before the boundary, resulting in a sharp change in the plastic strain gradient that requires an associated lattice reorientation in order to maintain deformation.
compatibility with both the grain centroid and the neighbouring grain. In terms of plastic strain, previous lower-resolution DIC studies on the proximity of plastic strain to grain boundaries have demonstrated a wider range in strain values closer to grain boundaries [68,75], but that the mean plastic strain is not significantly different at the grain boundary relative to its centroid. Analysis that details the change in strain along individual slip traces towards the grain boundary is necessary to explore this further [76].

The correlation that we observe between the strain gradient and the misorientation, especially at grain boundaries, is supported by reports of misorientation localisation in the literature [69,75,77–80]. This relationship is in agreement with Ashby’s explanation of GND pile up at grain boundaries that results in local lattice misorientation [13] and back-stresses that contribute to local slip resistance [81]. However, there are ways in which lattice misorientation can occur at grain boundaries other than that according to Ashby, and these deformation modes have been well described in HRDIC-EBSD sub-grain comparisons in an austenitic stainless steel [17]. For instance, slip band impingement on a grain boundary can cause lattice misorientation in the neighbouring grain close to the boundary [14,15], which is especially prevalent if slip transmission is not favourable [14]. Further, special types of boundaries, such as the Σ3 annealing twin boundaries, and their interaction with high angle grain boundaries are known to be important in strain localisation that develop towards fatigue crack initiation sites [74]. Whilst many of the grains studied here exhibit a strong spatial correlation between the strain gradient and the KAM or GROD Mean, most do not. Therefore, it is likely that the phenomena of slip band fading, slip band impingement on grain boundaries and grain break up are all active throughout this material. Grain neighbourhoods, how they deform and how they interact with longer-range localisations like shear bands all determine the strain localisations that are often used to explain localised failure.

4.3. The suitability of different lattice misorientation calculations as a measure of local plasticity

Lattice misorientation is often used to calculate the density of geometrically necessary dislocations as a measure of residual plastic deformation [82] and also as a validation for full field models of crystal plasticity [63,70,83–87]. However, the different measures of lattice misorientation shown in Fig. 3 demonstrate the wide range of magnitude and spatial variation that is possible from the same EBSD data set. A deviation in the spatial variation of lattice misorientation has been shown for a number of GROD variants [29] to highlight that the GROD reference orientation as the original orientation results in a lattice misorientation that contains both the rigid body rotations and the rotations that arise from plastic strain, whereas the other GROD variants only describe the latter. In Fig. 5 and Fig. 6 part a) we show the significance of this difference; the GROD Original measure of misorientation is poorly correlated to the in-plane measure of
deformation and this is due to the three dimensional nature of the rigid body and plastic rotations. In Fig. 6 part a) we show that the GROD Mean and the CMA give the strongest correlation to the magnitude of grain mean plastic strain and that the GROD Mean Max gives the most sensitive correlation and is more indicative of heterogeneity.

It has been shown that calculating the misorientation from the GROD Mean is much less sensitive to EBSD step size than using the mean KAM value [8,28,30]. The CMA as a measure of grain misorientation cannot be mapped the sub-grain scale, but it is even less sensitive to step size than the GROD Mean and more sensitive to changes in global plastic deformation [30]. This improved sensitivity is reflected in the statistical correlation analysis at the grain scale in Fig. 6. In Fig. 4 we show the distributions of plastic strain and misorientation for the whole field-of-view, ~1 x 0.5 mm. Whilst the GROD Mean and the KAM distributions have the lowest Kullback–Leibler deviations and therefore best represent the distribution of the plastic strain, the similarity in distributions is predominantly in the low strain and low misorientation regime. The high strain regime, which is thought of as important for crack initiation and localised degradation mechanisms [74], is best represented by the GROD Mean Max. We must therefore conclude that strain localisation is difficult to quantify using only a single EBSD lattice misorientation measure and so any interpretation of these data sets for such a purpose should be done with caution. This is especially the case when local lattice misorientations or GND densities are used exclusively to explain local failure mechanisms. As our results show, slip bands produce almost no lattice misorientation within grains, and yet corrosion and cracking occur preferentially along slip bands in stainless steel [25,88].

4.4. The spatial distribution of slip and lattice misorientation

One of the observations here is the difference in the spatial distribution of slip and lattice misorientation when sampled at the same spatial resolution in the pixel-to-pixel sub-grain analyses. In this alloy, slip is highly localised in slip bands and therefore discrete in nature. Conversely, the misorientation shown here varies smoothly at the same scale and slip bands can only be seen in EBSD studies with very high spatial and angular resolution [15]. For the majority of grains, slip bands carry most of the strain and yet produce almost no local misorientation. This difference in the nature of spatial distribution implies that the dislocations producing the strain are different from those created to accommodate the deformation gradients. In Ashby’s terminology, dislocations stored along slip bands are statistically stored dislocations (SSDs), whereas the dislocations producing misorientations are geometrically stored dislocations (GNDs). If slip were homogeneous, then GNDs could be assumed to have originated as SSDs that collect at regions of strain heterogeneity. But since slip occurs in bands, most of the GNDs produced are not involved in slip, although their development is affected by the slip activity. This is very different from how most continuum mechanics based crystal plasticity frameworks model polycrystalline deformation. In these models slip is assumed to be homogeneous and lattice rotation continuity is not enforced [89–91]. Given that the slip band spacing is of the order of 1 μm, this difference between how deformation is modelled and how it occurs in practice might limit the ability of these modelling approaches to predict damage nucleation or the detailed descriptions of the deformed state needed to model annealing. Certainly, there is currently no way to model different degrees of slip localisation, which as recent experiments on a proton irradiated zirconium alloy show [92], can produce very different patterns of lattice misorientation.

5. Conclusions

At the sub-grain scale, the lattice misorientation is the result of the number, strength and spatial distribution of slip modes within the grain and their interaction with microstructural features such as grain boundaries. Further, the way in which the misorientation is calculated has a drastic effect on the magnitude and spatial distribution of the lattice curvature. We find that the strongest spatial correlation between plastic strain and misorientation within a grain is obtained when the plastic strain is coarsened to smooth out the strain in the slip bands and the misorientation is calculated using the GROD Mean Max to reflect heterogeneity. However, in practise, this correlation can be positive or inverse with equal probability and so it is
recommended to also use the GROD Mean or KAM to identify the region of highest strain gradient.

The Taylor factor and the Schmid factor cannot be used as a predictor of the plastic strain of a grain or its lattice misorientation. This is because of grain interaction effects at the microscale, such as slip band fading towards grain boundaries, slip impingement on boundaries from neighbouring grains and the interaction of slip bands with annealing twins, all of which do not depend on grain orientation alone, and further because of mesoscale deformation banding, which is not crystallographic.

The wide scatter in the positive correlation between the plastic strain in a grain and its lattice misorientation does not correlate with its orientation: local grain interactions and the development transgranular bands of high strain erase any relationship between local plastic strain and crystallographic orientation.

The spatial distribution of slip and lattice misorientation are very different: slip is localised in sharp bands whereas misorientation varies smoothly over the same scale. This is very different from how deformation is modelled in continuum crystal plasticity models, which could have implications for their applicability to model damage and the deformed state at the microstructural scale.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors would like to acknowledge funding for their time from EPSRC [EP/K005420/1, EP/M005607/1, EP/M003737/1]. We would also like to thank the following people for useful discussions: Philippa Reed, Fabrice Pierron and Rong Jiang from the University of Sheffield, Philippa Reed, Fabrice Pierron and Rong Jiang from the University of Sheffield, Philippa Reed, Fabrice Pierron and Rong Jiang from the University of Sheffield, Philippa Reed, Fabrice Pierron and Rong Jiang from the University of Sheffield.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.actamat.2020.05.029.

References


A. Harte et al. / Acta Materialia 195 (2020) 555–570

