Measurement of local plastic strain during uniaxial reversed loading of nickel alloy 625

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Abstract

The development and evolution of plastic strain localisation was measured at sub-micron resolution using high-resolution digital image correlation (HRDIC) during reverse loading of a solid solution strengthened nickel base superalloy. The deformation was applied in-situ, allowing measurements to be taken with load applied which will not include any reverse yielding that can occur during unloading due to the Bauschinger effect. The strain was found to be localised into crystallographic slip bands separated by lower strain channels. No large local deformation was seen when unloading the material following forward deformation. Analysis of the unload strains, at the grain level, showed that any non-linearity seen in the macroscopic unload curve is either due to elastic anisotropy or low magnitude plastic deformation. The slip band structures formed in forward loading did not change during the early stages of reversal, suggesting reverse deformation is accommodated by dislocations reversing their paths. After further reverse deformation, the slip band patterns appear to change in location and become more sharply defined.

Introduction

Reverse loading and other strain path changes often lead to differences in macroscopic flow behaviour from what would be seen after continued monotonic loading. Because this difference in macroscopic response originates from processes acting at the microstructural scale, measuring the deformation at the microstructural scale is a powerful way of understanding its origins, making it possible to assess the suitability of current work hardening models, and develop new ones for complex load cases.
The Bauschinger effect is the classic example where, following a load reversal, the yield and working hardening of an alloy occur at lower stresses than in forward loading [1] and a permanent reduction in strength is seen compared to continued forward loading [2]. The generation of internal backstresses during forward deformation is one explanation of the Bauschinger effect in polycrystals [3]. These backstresses resist continued forward deformation but aid yielding in some regions of the microstructure in the reverse direction, leading to the reduction of macroscopic flow stress seen. Internal stresses form in deformed metals due to the spatially heterogeneous distribution of plastic deformation in the microstructure. These heterogeneities arise from various microstructure features, leading to a hierarchy of the magnitudes of backstresses and therefore Bauschinger effect produced.

The largest magnitude backstresses are seen in dual phase materials with large contrast in mechanical properties between the phases, producing hard and soft regions [4–6] in the microstructure. Relatively harder and softer regions also exist, to a lesser degree, in single phase material due to the anisotropy of deformation in grains of different orientations [3,7,8] and even in single crystals due to the development of dislocation structures [9,10]. This deformation heterogeneity during load reversal has been studied using powder diffraction [5,11], but diffraction only provides averaged data over many grains in a sample, from which it is difficult to deconvolve the contributions from different levels of heterogeneity. In this work, we aimed to measure the spatial distribution of plastic strain, during load reversal, over hundreds of grains and with sub-micron spatial resolution.

Uniaxial reversed loading experiments are challenging because the buckling instability seen under compression must be suppressed, but the sample design must also allow loading in tension. Typically, modified cylindrical tensile specimens with reduced gauge length to diameter are used [12], however, such samples do not have a flat surface on which to measure spatially resolved deformation during the loading process. Some researchers avoid this issue entirely by applying bending deformation instead [10,13], although, uniaxial reversed loading of flat mechanical samples can be achieved by using an anti-buckling fixture. These fixtures suppress out of plane deformation of a flat mechanical specimen by applying a clamping force, either using flat plates [14,15] or a comb like arrangement of opposing metal fingers [16,17]. Here, we present a similar fixture adapted for use with an in-situ deformation stage, with provision made to image the deformed surface at any point of the loading process.
This new fixture was used to investigate the development of deformation heterogeneity during the uniaxial reversed loading of a solid solution strengthened nickel alloy. High-resolution digital image correlation (HRDIC) [18] was used to measure local deformation in-situ and over a large area containing hundreds of grains, after which the deformation data was analysed on a grain-by-grain basis, via correlation with an electron back-scatter map of the microstructure. Applying load in-situ allows local deformation to be measured under load, allowing the measurement of any local plasticity that may occur during unloading caused by backstresses generated in the microstructure. In this way, it becomes possible to determine whether measuring deformation in-situ is advantageous over measuring it ex-situ, which is more convenient and usually can give higher spatial resolution.

Methods

The material studied was nickel alloy 625, provided by Haynes International, Inc. in the form of hot rolled and mill annealed plate of 26 mm thickness. Alloy 625 is a solid solution strengthened nickel alloy with the nominal composition given in table 1. The received material consists of an FCC $\gamma$ matrix and a small fraction of carbides located at grain boundaries. No strengthening precipitate phase ($\gamma''$) is present. When measured using EBSD, the texture was found to be random. The average grain size of the received material is $7 \mu m$ with a standard deviation of $3 \mu m$. The microstructure contains annealing twins and when twin boundaries are excluded the grain size is $11 \mu m$ with a standard deviation of $5 \mu m$. The material is used in the received state and samples are taken from the central third of the rolled plate.

The study of uniaxial reversed loading requires that both compression and tensile deformation be applied to a single sample. The flat tensile specimens used for HRDIC investigation are prone to buckling under compression loading, so a constraint fixture has been devised to prevent the buckling. The specimens were designed to preferentially buckle in the through-thickness direction ($z$ here) by using a gauge cross section with a large width to thickness ratio. Buckling in the $z$ direction was prevented by deforming

| Ni   | Co  | Fe  | Cr  | Mo  | Nb+Ta | Mn  | Si  | Al  | Ti  | C  |
|------|-----|-----|-----|-----|-------|-----|-----|-----|-----|----|
| Balance | 1   | 5   | 21  | 9   | 3.7   | 0.5 | 0.5 | 0.4 | 0.4 | 0.10 |

Table 1: Composition of Haynes 625 in weight percent.
the sample in a channel whose depth matched the thickness of the sample, constraining movement in the $z$ direction and preventing the specimen from bucking. The channel comprises of a lower block of 304 stainless steel which contains a trench for the specimen to deform inside. A lid is affixed to the lower section with bolts to fully enclose the specimen.

The design of the mechanical specimen and constraint fixture are shown in figure 1a. Part b) of the figure shows the profile of the specimen and the region which is covered by the channel has been highlighted in red. The cross-section of the gauge is $3.5 \times 0.76 \text{mm}^2$ with a parallel length of $9 \text{mm}$. To allow imaging of the specimen surface without removing it from the fixture, a $3 \text{mm}$ diameter tapered hole is located at the centre of the lid. The diameter of the hole is less than the width of the specimen, so the specimen is restrained along the entire length of the gauge section. When the constraining fixture is assembled, no parts protrude above the lid. This allows for a minimum distance between the specimen surface and detectors positioned above the lid.

The capability of the reverse loading constraint fixture was assessed using the commercial finite element method (FEM) modelling package Abaqus [19]. The arrangement of channel, specimen and lid were reproduced in the software. Four countersunk M3 bolts
were used to affix the lid and channel, which were also reproduced in the model using the fastener feature of Abaqus. Sliding friction was modelled between the surfaces of the specimen and channel using a coefficient of friction of 0.15, which is typical for sliding between lubricated metal surfaces. Isotropic elastic deformation was modelled for all the components, where appropriate values of Young’s modulus and Poisson’s ratio were used for each material. Plastic deformation was only allowed in the test specimen and was modelled with a yield stress of 400 MPa and simple isotropic hardening. A reverse loading cycle was simulated by applying displacement boundary conditions to nodes at both ends of the specimen, reproducing the action of the deformation stage.

The mechanical specimen was produced by wire electric discharge machining (EDM) to the geometry shown in figure 1b and a thickness of 1 mm. The preparation of the specimen surfaces for use with the constraint fixture followed standard metallographic methods. Both faces were successively ground from 600 to 4000 grit silicon carbide abrasive paper. During this stage, the thickness of the specimen was reduced to 0.76 mm to match the depth of the restraining channel. The material removal from subsequent polishing was assumed to be negligible. One of the specimen surfaces was polished further to allow the application of the HRDIC pattern. This included polishing with 1 and quarter micron diamond paste and finally with a concentrated 0.05 micron colloidal silica suspension. To reduce friction between the specimen and the restraining channel during testing, the internal surfaces of the channel were also polished to a 1 micron finish and a thin layer of vacuum safe lubricant was applied prior to testing. To measure the applied macroscopic strain, two micro-hardness indents were placed at the centre of the gauge 0.8 mm apart in the loading direction. The specimen was deformed using a Deben 2 kN deformation stage, which can apply both tensile and compressive load.

The HRDIC speckle pattern was produced following the procedure developed by Gioacchino and da Fonseca [18]. A detailed description of the technique and apertures used can be found in the reference. Briefly, this involved coating the surface with a 60 nm film of gold using an Edwards S150B sputter coater and then remodelling the film by heating to 290°C in a flow of water vapour for 5 hours.

An FEI Quanta 650 field emission gun scanning electron microscope (FEG-SEM) was used to image the HRDIC pattern. This microscope is equipped with large sample chamber and can accommodate the in-situ loading stage, allowing uninterrupted deforming and
imaging. Backscatter electron (BSE) imaging was used to image the speckle patterns during testing. This exploits the atomic number contrast between the gold pattern and nickel substrate, whilst minimising other unwanted sources of contrast such as surface contamination [20]. A Deben Centaurus scintillation BSE detector was used, which has a high detector efficiency allowing minimal scan speeds to be used while still achieving a good signal to noise ratio [21]. The imaging conditions used are summarised in table 2. Due to the thickness of the BSE detector and lid of the deformation channel, images had to be acquired at a relatively large working distance of 13.5 mm. However, the distance from specimen surface to detector was approximately 4 mm. A slight vibration of the stage was seen when imaging at high magnification, which had a detrimental affect on the achievable resolution. The affect of the vibration was minimised by averaging over 5 frames taken successively with a low dwell time, the frame averaging still gave a high signal to noise ratio while the influence of the vibrations was removed. This frame averaging approach has been used previously and shown to decrease correlation error [22].

The imaging process was repeated automatically over a 4 × 4 grid using Oxford Instruments Aztec software package, taking approximately 26 min to acquire the 16 images. Each image in the grid had a 15 % overlap to its neighbours, to allow the images to stitched together into a single image. The stitching was performed using an algorithm that optimally positions the images to reduce the global error in all overlapping regions, which is implemented in the image manipulation software ImageJ [23, 24]. The complete mosaic was 7028 × 6110 pixel² in size, with one pixel corresponding to 13.3 nm. Images were collected at 6 points of a reverse load cycle, at strains of 0, −3.2%, −3.0 % (unloaded), −1.9%, 0.6% and 2.4%. These points are shown on a schematic stress strain curve in figure 2 and the strain steps will be referred to by the labels given in the figure throughout.

Surface displacement maps were calculated from the pattern images using a local DIC algorithm based on repeated cross-correlation between image sub-regions of successively smaller sizes, which is included in LaVision’s DaVis software package [25]. A final sub-

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### Table 2: Summary of imaging conditions used for HRDIC.

| Accelerating Voltage | Spot size | Working distance | Magnification | Field size         |
|----------------------|-----------|------------------|--------------|--------------------|
| 20 kV                | 3         | 13.5 mm          | 10000 x      | 27.2 × 23.8 µm²    |
| Field resolution     | Pixel size| Dwell time       | Averaged frames|
| 2048 × 1792          | 13.3 nm   | 5 µm             | 5            |
Figure 2: A schematic stress-strain curve of reverse loading, showing the points at which HRDIC data was collected. The error bars denote the difference between the average strain measured in the HRDIC region and the strain measured by the displacement of two micro-hardness indents, the value of both strain measurements is also given at each deformation step.
region size of $16 \times 16$ pixel$^2$ was used, corresponding to a spatial resolution of $0.21 \mu m$. The final DIC maps covered a region of approximately $85 \times 75 \mu m^2$ and 300 grains with an equivalent diameter greater than $2 \mu m$.

Following the HRDIC imaging an electron backscatter diffraction (EBSD) map was taken of the HRDIC region. To allow the collection of an EBSD map, the gold pattern was removed from the specimen by polishing with colloidal silica for 2 minutes. The EBSD map was acquired using an FEI Sirion FEG-SEM equipped with an Oxford Instruments EBSD system. EBSD was performed at an accelerating voltage of 20 kV with a 0.1 $\mu m$ step size and a 15 ms dwell time at each point. The EBSD map was collected post deformation, as attempts to collect an EBSD map before applying the HRDIC pattern lead to a speckle pattern with features too small to be resolved, with good contrast, during in-situ testing.

HRDIC and EBSD are two complimentary techniques for studying deformation of polycrystals, with HRDIC providing information on local deformation and EBSD on the crystallography of the underlying microstructure. Correlating data from both techniques requires that the map data from one source is registered to the other, which is not trivial due to differences in resolution and spatial distortions in the EBSD map. During EBSD acquisition, local sample charging can occur due the long dwell times and high beam currents required for collection of diffraction data. This charging can affect the beam position, leading to distortions in the map that turn the rectangular area selected for mapping into a trapezoid. Therefore a spatial transformation must be applied to the EBSD map to align it to the HRDIC data, which is assumed to be distortion free. The measured orientations do not need to be adjusted, however, as these only rely on the orientation of the sample relative to the fixed coordinates of the stage and EBSD camera position [26]. It is expected that slight variations in the position of the pattern centre will not influence the measured orientations significantly compared to uncertainty of traditional Hough transform indexing.

The transformation between datasets can be estimated by identifying homologous points (points at the same material location) in each of the datasets. The homologous points were manually selected on the stitched images of the DIC pattern, where grain contrast can be seen, and in the EBSD orientation map. Grain boundary triple points were the preferred locations to select, as they were easy to determine in both datasets. An affine transformation between the datasets was initially selected, being the simplest class
of transformations capable of mapping a rectangle to a trapezoid. An affine transform of a point with coordinates \((x, y)\) can be defined

\[
X = a_0x + a_1y + a_2 \\
Y = b_0x + b_1y + b_2,
\]

where \((X, Y)\) are the transformed coordinates and \(a_i, b_i\) are parameters of the transformation. This is a constant linear transformation which contains a translation, rotation and shear of the coordinates. The use of coordinates transformations that include higher order polynomial terms will also be investigated for registering the data maps, in the form,

\[
X = \sum_{i=0}^{n} \sum_{j=0}^{i} a_{ij}x^i y^j \\
Y = \sum_{i=0}^{n} \sum_{j=0}^{i} b_{ij}x^i y^j,
\]

where \(n\) is the order of the polynomial and \(a_{ij}, b_{ij}\) are parameters defining the transformation.

The affine transformation defined in equations 1 includes six unknown parameters, which require six equations, or three homologous points, to uniquely determine the transformation. It is desirable to use more than three homologous points to minimise the influence of the error in placement of any single point, leading to an overdetermined system. A least-squares fitting of equations 1 is therefore performed to find the optimal transformation parameters considering all the provided homologous points. The transformation functions included in the ‘skimage’ package [27] included in the Sci Py stack [28] were used to estimate the transformation between the two sets of homologous points. The performance of correcting the distortion in the EBSD map will be compared to higher order polynomial transformations (equation 2), which require more homologous points to uniquely define but could account for possible time dependence of beam drift when collecting the map. The alignment of HRDIC and EBSD data was performed using 11 homologous points which were manually identified.
Results

The FEM model was used to confirm the anti-buckling fixture prevents buckling when a specimen is compressed whilst still allowing uniaxial deformation. A reverse load cycle was simulated by first compressing the sample by 3% and then deforming by 6% in tension. When the anti-buckling fixture was excluded from the model, the specimen bows about its fixed ends instead of deforming in-plane. The simulation run with the fixture in place, on the other hand, did not show any buckling of the specimen. The spatial distributions of stress and strain are shown in figure 3, where both components in the loading direction (11) and the surface normal direction (33) are included for both compression and tension following compression deformation steps. Stress and strain are seen to be uniformly distributed in the gauge section of the specimen in all cases.

Volume preservation during plastic deformation implies that, for uniaxial deformation, transverse strains should be half the strain in the loading direction. After compressing, the average strain in the central two thirds of the gauge section is $-2.9\%$ in the loading direction and $1.4\%$ in the normal direction, confirming the specimen is deforming uniaxially and the fixture does not constrain the sample to plane strain deformation. A similar result is found for the predicted stresses in the sample normal direction, where no significant stresses are predicted as would be expected for a free surface. Stress concentrations of up to 60 MPa are seen around the tapered region but decay moving into the gauge length and are small compared to the stress in the loading direction, which has a peak of $-320$ MPa.

Surface strain maps collected using HRDIC at each deformation step are shown in figure 4, where the loading direction ($x$) is horizontal. The strain map for the step of compression and unload has been excluded as it is indistinguishable from the compression step when plotted in this manner. An effective shear strain, defined as

$$
\epsilon_{eff} = \sqrt{\left(\frac{\epsilon_{xx} - \epsilon_{yy}}{2}\right)^2 + \epsilon_{xy}^2},
$$

is plotted in the maps, where $\epsilon_{xx}$ is strain in the loading direction, $\epsilon_{yy}$ is in-plane transverse strain and $\epsilon_{xy}$ is in-plane shear strain. This effective shear strain measure assumes that all deformation has occurred by shear and combines in-plane shear strain with an estimate
Figure 3: Simulation prediction of the distribution of stress and strain in the gauge section of the reverse load mechanical specimen, produced using Abaqus [19]. Plots of stress and strain in both the loading direction (\(x, 11\)) and the sample normal direction (\(z, 33\)) are shown.

Strain is seen to be heterogeneously distributed for all deformation steps, with slip bands forming to accommodate the plastic deformation. Heterogeneity is also seen at a larger scale, where regions of comparable size to the grains display low deformation compared to the surrounding area. Examples of these regions are marked ‘a’ in the top left map of figure 4 and persist throughout all the deformation steps.

An estimation of the uncertainty for the HRDIC strain measurements was made by applying the DIC algorithm to two images acquired in the same area without deformation, where any deformation measured is attributed to random errors. The strain measured in the loading direction is distributed around 0 strain and the mean of the absolute value is 0.3\%, which is an estimate of the average uncertainty of the strain measured at each
Figure 4: Effective shear strain maps collected using HRDIC at the four distinct macroscopic strain steps, defined in figure 2. Intense slip localisations are marked by triangles and regions of low strain marked ‘a’ in the compression step (part a) and areas of high reverse deformation are marked with numbers in the full reversal step (part c). The loading direction is horizontal in the maps.
Table 3: Strain in loading direction measured using two methods: 1) the change in displacement between two micro-hardness indents placed either side of the HRDIC region 0.8 mm apart (macroscopic) and 2) the mean of the strain measurement using HRDIC region (average local).

| Strain step                  | Macroscopic strain (%) | Average local strain (%) |
|-----------------------------|------------------------|--------------------------|
| Compression (C)             | $-3.2 \pm 0.3$         | $-3.0$                   |
| Compression unload (CU)     | $-3.0 \pm 0.3$         | $-2.7$                   |
| Half reversal (HR)          | $-1.9 \pm 0.3$         | $-1.7$                   |
| Full reversal (FR)          | $0.6 \pm 0.3$          | $0.8$                    |
| Tension following reversal (TFR) | $2.4 \pm 0.3$         | $2.2$                    |

point in the calculated strain maps. This error is low in comparison to the localised strain formed in slip bands, where large strains of up to several percent are detected. However, the uncertainty is comparable to the elastic part of the strain, meaning we would expect that the elastic unloading cannot be measured at this spatial resolution.

The amount of deformation applied to the sample was measured using two methods: by the change in displacement of micro-hardness indents either side of the HRDIC region and by averaging the local strain measurements in the HRDIC region. Table 3 shows values from each method for each deformation step and the difference is also indicated by the error bars in figure 2. The uncertainty of the strain measured using the indents is estimated to be $\pm 0.3\%$, by observing that the microscope stage could be repeatably positioned within 3 μm of the centre of each indent. The uncertainty of the mean strain of the HRDIC maps is considered to be negligible, with a value of $8 \times 10^{-4}\%$ due to the large number of strain values used to calculate the average. The strains measured by the two methods agree to within these estimated uncertainties, suggesting the HRDIC region is large enough to be representative of bulk deformation measured over a distance of 0.8 mm.

The estimated transformation between the HRDIC and EBSD coordinate systems was used in figure 5 to overlay grain boundaries on the strain map from the final deformation step. The grain boundaries comprise of all points with greater than 10° misorientation to any neighbouring point in the EBSD map. A bitmap representation of the EBSD boundaries was then transformed to the HRDIC coordinate system for display. The DIC algorithm defines displacement vectors in the undeformed configuration, meaning only a single transformation needed to be defined and was valid for all deformation steps.
Figure 5: HRIDC strain map of the tension following reversal deformation step with grain boundaries shown as white lines. The grain boundaries were identified using EBSD and then a bitmap representation was transformed into the HRDIC reference frame.

Despite the HRDIC measurements being defined in the undeformed configuration and the EBSD mapping being acquired in the post deformation state, there is a good visual match between grain boundaries and deformation features and the influence of rigid body grain rotations is small at the low strains studied. Abrupt changes in slip bands orientation are seen to occur along grain boundaries, which has been reported in previous studies of grain boundary strain localisation where the orientation of active slip planes changes across a grain boundary [31, 32]. In all the grains measured, the orientation of the slip bands is found to agree with the interception of \{111\} slip planes with the sample surface (slip traces).

To assess the accuracy of the registration process between the EBSD and HRDIC datasets, the location of features that are present in both can be compared. The only features that can be seen in both datasets are grain boundaries, which are defined in EBSD by a critical misorientation and can be seen in the images of the speckle patterns as a difference in contrast between grains. Figure 6 shows a region on the stitched HRDIC speckle image, collected in the undeformed state, with EBSD grain boundaries overlaid.
Figure 6: Backscattered electron image of a portion of the HRDIC region. The grain boundaries are derived from an EBSD map of the same area and have been registered using an affine transformation. Different grain orientations produce different greyscale constant and the changes in constant are seen to agree with the registered grain boundaries.

using the calculated affine transformation between the datasets. The location of grain boundary lines can be seen to agree well with the changes in contrast between grains.

The local quality of fit around each homologous point can be quantified by calculating the distance between the manually selected point and the location of the corresponding transformed point. Figure 7a shows the mean, standard deviation and maximum of these distances for the 11 homologous points used to define the transformation. A comparison is made for the different transformations defined in equations (1 and 2), from the linear affine transform to a transformation defined by a fourth order polynomial. The plot shows that the uncertainty is at the sub-pixel level in the DIC map. There is also a trend that as the order of the transformation increases, the error in the positions decreases. However, the fourth order polynomial exactly matches all points, suggesting over fitting and that oscillations from higher order terms have allowed for this accuracy.
Figure 7b displays the same statistics of the distances but now with an additional erroneous homologous point included, which is placed approximately 8 pixels from the correct location in the EBSD map. The average and maximum distances increase for the affine and 2nd order polynomial transformations but no significant increase is seen for the 3rd order transform, again suggesting over fitting. Despite the affine and quadratic transforms displaying similar errors for the homologous points, the affine transformation was found to be the optimal choice as it matches the datasets better when outside the convex hull of the defined homologous points.

The registration of the two datasets allows information acquired from each to be used in combination. An example given already is defining grain boundaries in a HRDIC map. Automatically detecting grain boundary locations in the HRDIC images would prove challenging due to the small changes in contrast between grains (see figure 6). Neighbouring grains can also appear in the same shade if they have similar electron channelling alignment, making the boundary between them indistinguishable in an image. This is made worse in HRDIC images where the gold layer masks the contrast between grains. In contrast, grain boundaries can be simply and systematically defined in an EBSD map using a critical misorientation condition to define grains.

The combined datasets allow HRDIC deformation data to segmented into grains. Figure 8a displays histograms of grain average effective shear strain for the compression, full reversal and tension following reversal deformation steps. The compression step has a larger spread compared to the tension step with a higher maximum of 9.5% compared to 6%, suggesting larger intergranular heterogeneity in the compression step. However, some of the difference could be due to the compression step measurements being at higher level of macroscopic strain (−3.2% vs. 2.4%). Figure 8b then shows comparisons between the effective shear strain averaged over the entire HRDIC mapped region and the average and spread of the grain averaged values, with the spread shown by error bars at ±1 standard deviation. In all the deformation steps the map average is higher than the grain average and, since the grain average does not weight by grain size, implies that larger grains accommodate proportionally more of the deformation than the (apparently) smaller grains. Again, the larger spread in grain deformations is seen at the compression deformation step.

The dependence between grain orientation and the strain seen at unload can also be
Figure 7: Statistics of the distances between the homologous points in the HRDIC map and the EBSD homologous points transformed into the HRDIC reference frame. This distance measures how well the transformation fits the points which define it. In a) only points at consistent locations between the two data maps are included. In part b) an extra spurious point is included to determine how errors in the placement of homologous point affect the transformations. For reference 1 EBSD pixel is equal to 0.1 µm.
studied using the combined datasets. Figure 9a shows a contoured inverse pole figure (IPF) plot of grain averaged unload strain measured between the compression step with the load applied (step C) and the compression step after unloading the specimen (step CU). The orientation of each grain was calculated as the mean of all the orientation measurements in the grain, using a quaternion averaging approach [33]. This averaging operation was performed on grains defined in the EBSD reference frame and only the final grain orientations were passed to the grains defined in the HRDIC reference frame, by way of a mapping between the grains defined in each frame. The mapping between grains was calculated by considering the overlapping areas of grain in each of the grain maps, making use of the transformation between the reference frames. The issue of large uncertainty of the HRDIC strain measurements (0.3%) compared to the small unload strains was resolved by averaging over the many data points in each grain and ignoring grains containing fewer than 100 data points. An orientation dependence is seen for the unload strains, where grains with compression axis aligned close to a [001] crystal direction deforming by larger strains when unloaded than for grains with compression axis close to a [111] direction.
Figure 9: Strain accumulated during unloading as a function of load axis for a) HRDIC measured strain averaged over each grain, b) expected strain based on orientation and single crystal elastic anisotropy and c) the difference between the strain in parts a) and part b).

Discussion

Heterogeneous plastic deformation at the sub-micron level is seen in all of the calculated strain maps. Deformation is arranged in slip bands, found to align with \{111\} slip traces when plotted from grain orientations measured by EBSD. Slip bands are seen generally to form in only one orientation in a single grain, although some grains do show slip bands in two or more orientations. Inter-granular heterogeneity can also be seen, with single grains and regions encompassing multiple grains displaying low deformation, labelled 'a' in figure 4a.

The error of the in-situ HRDIC measurements (±0.3 %) is similar to that reported for ex-situ HRDIC measurements using the gold remodelling pattern technique. The spatial resolution achieved with the in-situ loading of 210 nm, however, is lower than previous ex-situ studies, which report resolutions between 50 nm to 170 nm [29, 30, 34–36]. The spatial resolution and error are both linked to the density and size of speckle features in the pattern but images of the pattern must also have sufficient contrast between the
speckles and background. Although the same patterning technique was used here, the smallest features in the pattern could not be resolved with good contrast with the in-situ imaging, leaving only the larger features with lower density. Hence, to keep the error to an acceptable level, the resolution of the measurements was reduced compared to previous ex-situ measurements.

The strain patterning is similar to that seen previously in austenitic stainless steel [18,29], with slip bands formed on \{111\} planes and regions of relatively high and low slip activity. However, despite the comparable resolution (210 nm and 170 nm), the slip bands appear to be less distinct in the strain maps presented here. This could be attributed to a number of reasons, including the smaller grain and compressive deformation used here. Both alloys have FCC crystal structure but differences in stacking fault energy or existence of short range order will influence the character of slip [37]. Prior annealing could also be a factor, with mill-annealed material used here compared to solution treated stainless steel.

Particularly intense strain concentrations are formed during the forward compression step, marked using triangles in figure 4a. These concentrations form in straight lines and local strains up to 0.14 are seen, which is 4 to 5 times the macroscopic strain applied. Comparing the locations to the grain boundaries grain boundaries shown in figure 5, the shear concentrations all lie along straight twin boundaries. In all of the cases identified, the intense slip bands form on the same \{111\} plane as the twin boundary. In some cases the localisation encompass the entire twin, where individual slip bands cannot be observed, meaning either bands are too closely spaced to be resolved or that the entire twin is deforming.

The strain concentrations have been reported to form due to the difference in elastic properties on either side of the twin boundary due to elastic anisotropy, Which causes local stress concentrations that enhance glide at the boundary. This leads to shear strain concentrations at twin boundaries, as the boundary is parallel to a slip plane [38]. Twin boundary strain concentrations have been seen previously in \gamma^{'} containing Ni alloys loaded both monotonically and in fatigue [31,39].

It is unclear if elastic anisotropy could lead to such large concentrations at the strain of $-3.2\%$ measured here, well within the domain of plastic deformation within which plastic anisotropy would be expected to dominate the material response [40].
trations have also been linked to the formation of fatigue cracks, which form preferentially at longer sections of twin boundary with strain concentrated along the length [39].

**Strain during unload**

The strains were measured in individual grains during the unloading from step C to step CU and the trends between the strain on unload and crystal orientation are consistent with the elastic anisotropy of this nickel alloy. The elastic modulus in the [001] crystal direction is less than half that in the close-packed [111] direction, hence larger elastic strains would be expected in the [001] direction than the [111] direction. This dependence can be seen in the measured strains, shown in 9a.

The single crystal elastic constants of alloy 625 have previously been measured for alloy 625 [41] and can be used to calculate the expected elastic strain for each grain orientation in the mapped region if it were deformed in isolation. To calculate the expected elastic strain, the grains are assumed to be in a state of uniaxial stress equal to the macroscopic stress, which was measured as $-480 \text{ MPa}$ when the unload commenced. These predicted elastic strains are plotted as a function of crystal orientation on a contoured IPF plot in figure 9b.

The elastic strains calculated from the crystal compliances display a more pronounced orientation dependence than for the measured strains at unload. Figure 9c shows the difference between the strain measured at unload and the calculated elastic strain. The difference in the strain values range between $\pm 0.15\%$ but are broadly less than $\pm 0.1\%$. In the central region of the IPF the strain values agree well and discrepancies form towards the [001] and [111] crystal directions, where the extreme values for unload strain are expected from the single crystal elastic anisotropy. The measured strains at unload are more isotropic than the single crystal elastic strains due to the intergranular constraint present in polycrystals [7]. The deviation from the expected elastic anisotropy means internal stresses between neighbouring grains must be present and these stresses will lead to early yielding in the reverse direction, possibly during the unloading.

Residual strains induced by plastic deformation have been measured previously using neutron diffraction [11, 12, 42] in austenitic stainless steel, which has similar elastic anisotropy properties to nickel. When unloaded following compressive plastic deformation, the residual strains are found to be positive in the [111] and [101] crystal directions.
and negative in the [001] direction [12]. These residual strains are in qualitative agreement with the difference between the strains at unload and expected elastic strains shown in figure 9c.

When a polycrystal material is unloaded, the grains deform collaboratively to remain compatible but the differences in elastic modulus in the loading direction for the grains leads to internal stresses. If these stresses are sufficiently large, plastic deformation will occur locally to reduce the stress. The small non-linearity in the macroscopic curve [43], the neutron diffraction measurement and our own HRDIC measurements, all suggest that the amount of plastic strain on reversal is only a small fraction of the unload strain, which is dominated by elastic relaxation, and therefore below the resolution of the HRDIC technique. If this strain were to be concentrated in a few slip bands, it might have been large enough to measure on unloading. However, no sharp strain features were found in strain maps corresponding to the unloading, which is dominated by the random uncertainty of the HRDIC method and the systematic error caused by stitching multiple images, shown in figure 10. If there is any change in the strain within slip bands on unloading it is too small to detect in this case, suggesting it is distributed equally through all slip bands. The implication is that measurements of plastic strains made ex-situ, on unloaded samples, will be essentially identical from those made in-situ and under load.

**Strain reversibility**

Following the unload and subsequent application of reverse deformation, no differences are seen in the strain patterning for the half reversal deformation step (figure 4 b) compared to the initial compression. At this step the net macroscopic strain is still compressive. The magnitude of strain in the slip bands has reduced but their locations remain unchanged from the first deformation step, suggesting that, at least initially, slip is reversing along the paths formed during forward deformation. This agrees with experiments of bending and straightening single crystal cantilever beams [10], where local misorientation caused by the bending was removed by the straightening and attributed to the remobilisation of geometrically necessary dislocations (GNDs) during straightening of the beam. This reversible slip explains the transient softening component of the Bauschinger effect, in which yield occurs at reduced stress with a lower rate of work hardening over a larger strain range for reversal compared to forward loading. Assuming that a fraction of the forward
Figure 10: Strain map showing the strain measured during unloading of the sample. No distinct features can be seen in the strain values which have a large contribution from the random uncertainty of the HRDIC method and the systematic error caused by stitching multiple images.
plastic deformation is reversible has been used to successfully describe the Bauschinger effect seen in macroscopic flow curves [44]. At the micro-scale, this reversible strain is the dislocations in slip bands backing off and moving away from obstacles that arrested their forward path.

When further reverse strain is applied to a net tensile macroscopic strain, the strain pattern begins to differ from the patterns formed during the forward compression loading. The strain maps at steps FR and TFR (figure 4 c-d) were both taken at net tensile strain and slip bands are seen along the same slip planes as forward loading, but the bands appear more distinct with fewer regions of diffuse slip. Two examples are labelled in figure 4 c of distinct slip bands formed following the strain reversal, with high strains compared to other bands in the strain map. These distinct bands have formed in regions of diffuse slip in the compression deformation step, so present as a change in the slip behaviour caused by the load reversal. The intense strain localisations seen in forward loading along twin boundaries, highlighted with triangles in 4 a, are also not seen following the load reversal.

To quantify the differences in the slip behaviour between forward and reverse deformation, the reversibility of the strain formed in forward loading can be considered. A reversibility parameter is calculated at each point of the HRDIC region by summing normalised strain in the loading direction for two deformation steps, the forward step and a
reversal step. The strain is normalised by the absolute mean value of the map, to account for the difference in macroscopic strain at each step (table 3). Values of this reversibility parameter close to zero are interpreted as roughly equal, but opposite, intensity of slip at the point, where the intensity is relative to the average in the HRDIC region. Negative values imply higher slip intensity in the forward direction and positive values imply higher slip intensity in the reverse direction.

Histograms of the reversibility parameter are included as figure 11, with a plot for small reverse deformation to a net compressive strain (step C to HR) and a plot for larger reverse strains to a net tensile strain (steps C to TFR). If forward plastic deformation were to perfectly reverse, the distribution would resemble a delta function or a narrow peak centred at 0. The distribution for the half reversal step (step C to HR) has a small spread of values around 0, suggesting at this point of the reverse cycle the plastic strain tends to reverse along the same paths formed in the forward loading. When further reverse strain is applied in the tension following reversal step (step C to TFR), the distribution has a much larger spread in values, meaning the strain localisation patterns have changed as the reverse strain increased and slip is no longer reversing along forward paths.

Orowan’s explained the reduction of reverse yield stress [45,46] as dislocations backing away from obstacles along forward paths in to material with relatively fewer obstacles, that have already been overcome during forward deformation. The shear strain in the slip bands initially reduces, suggesting a mechanism similar to Orowan and the dislocations initially reverse along their forward paths. As further reverse strain is applied, the dislocations in the slip bands will encounter obstacles that have not been overcome in forward deformation or that developed during the forward deformation, such as forest dislocation tangles from slip on other crystal planes. This reduces the population of dislocations that have an easy reverse path and the amount of shear strain that can be accommodated by the reversal of dislocation path. After the initial easy reversal, the increase of obstacle density at the slip bands will make it unfavourable for reverse deformation to be accommodated on these slip bands. This leads to changes in the strain localisation patterns after the full reversal step, as new slip bands preferentially form in material adjacent to the slip bands formed during forward deformation. Similar was seen from tracking dislocation content in single cantilever beams [10], where GND content did reverse but the statistically stored dislocations remained and will affect slip localisation on reversal past the unstrained
Observing the reversal behaviour of individual slip bands directly in the strain maps has not been possible at the spatial resolution that was achieved. The resolution was not sufficient to identify individual slip bands in all grains, which is most apparent in the map of the compression step shown in figure 4a. An example of the behaviour of slip bands during reversed deformation is, however, shown in figure 12. The strain data shown in this figure was processed using a smaller DIC sub-region size of $12 \times 12$ pixel$^2$, which increases the spatial resolution to 160 nm at the expense of a higher average uncertainty of 0.7%. The increase in resolution shows more definition to the slip bands for the grain in figure 4, while the uncertainty is not unreasonably large when restricting attention to the large configuration.
strain localisation.

Figure 12a shows strain maps centred on a single grain where the character of the slip bands is seen to differ between the compression step and the tension following reversal step. The slip bands in the tension step are more sharply defined than in the compression step, which can be seen clearly in a line profile of strain taken perpendicular to the slip bands (figure 12b). Higher peaks in strain are seen in the tension step compared to the compression step but the location of the peaks also changes after the reversal of loading direction. Vertical dashed lines marking the peak locations in the tension step do not consistently align with the peaks formed in the compression step and, in some cases, peaks in the tension step have formed at strain minima in the compression step. This suggests that new slip bands are forming to accommodate reverse deformation, rather than being accommodated by the bands formed during forward deformation. Further work is required to confirm this behaviour, possibly using ex-situ DIC to achieve a higher spatial resolution to clearly distinguish individual slip bands in more grains.

Conclusion

The development of microstructure informed hardening models of reverse loading relies on the measurement of local deformation to calibrate the models against and test if the correct behaviour is predicted. We have successfully carried out in-situ load reversal tests in the SEM and measured local plastic deformation at several points of the load cycle. When load was removed from the specimen following forward straining, no large strain features were seen in the local strain maps and any non-linearity seen in unload stress-strain curves is produced by either elastic anisotropy or low magnitude plastic deformation.

We found that the achievable spatial resolution of HRDIC was compromised by the in-situ loading apparatus and, since no local plasticity was seen during unload, it can be concluded that ex-situ loading is appropriate for these tests.

Overall, it was found that reversibility is different for different grains, and that this difference increases as the reverse strain is increased. Strain was localised into slip bands aligned with crystallographic slip traces, which do not change significantly during early stages of reverse loading, and the reverse plastic strain is accommodated by strain in existing bands. This is consistent with the reduced yield strength of the Bauschinger
effect being due to easy dislocation travel along previous slip paths.

At larger reverse strains, slip bands become more sharply defined and new bands form at different locations. Further work at higher spatial resolution is required to investigate this behaviour in more detail.

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