CHARACTERIZATION OF MESOSCOPIC CRYSTAL PLASTICITY FROM HIGH-RESOLUTION SURFACE DISPLACEMENT AND LATTICE ORIENTATION MAPPINGS

A thesis submitted to The University of Manchester for the degree of Doctor of Philosophy in the Faculty of Engineering and Physical Sciences

2013

Fabio Di Gioacchino

School of Materials
Contents

Contents ........................................................................................................................................... 2
List of figures ................................................................................................................................... 4
List of tables .................................................................................................................................. 12
List of symbols and abbreviations ................................................................................................. 13
Abstract .......................................................................................................................................... 15
Declaration .................................................................................................................................... 16
Copyright Statement ..................................................................................................................... 17
Acknowledgments .......................................................................................................................... 18
1 Introduction .................................................................................................................................. 19
  1.1 Thesis layout ............................................................................................................................. 20
2 Introduction to digital image correlation at high magnification .................................................. 23
  2.1 Basic principles of DIC ............................................................................................................ 23
    2.1.1 Correlation coefficient ...................................................................................................... 24
    2.1.2 Shape function and interpolation scheme ...................................................................... 25
    2.1.3 Sub pixel registration algorithm .................................................................................. 26
    2.1.4 Strain estimation and subsets overlap ........................................................................ 28
    2.1.5 In-plane, stereovision and volumetric DIC ................................................................. 30
  2.2 Pattern texture ........................................................................................................................ 31
    2.2.1 Speckles morphology .................................................................................................... 32
    2.2.2 Assessment of pattern quality ..................................................................................... 33
  2.3 DIC using SEM imaging ......................................................................................................... 35
    2.3.1 Source of error in SEM imaging .................................................................................. 36
    2.3.2 Pattern application methods for DIC of SEM images ................................................. 37
    2.3.3 Gold remodelling ......................................................................................................... 39
3 Introduction to electron backscatter diffraction and dislocation state characterization .......... 42
  3.1 Basic principles ....................................................................................................................... 42
    3.1.1 Diffraction patterns and indexing ................................................................................. 42
    3.1.2 Spatial resolution and accuracy .................................................................................... 43
    3.1.3 DIC of diffraction patterns: elastic strain and improved accuracy ............................. 44
  3.2 Lattice curvature and geometrically necessary dislocations ................................................. 46
    3.2.1 Nye tensor .................................................................................................................... 46
    3.2.2 Lattice curvature components from EBSD data .......................................................... 49
    3.2.3 Estimating the density of geometrically necessary dislocations ............................... 51
List of figures

Fig 1.1 Distinct scales of investigation in crystal plasticity [6]

Fig 2.1 Schematization of the DIC principle. (a) Division of the images to be correlated in small subsets. (b) Correlation of initial and deformed subsets to yield a displacement vector [11]

Fig 2.2 Displacement of subset pixels operated by second and third order shape functions [16]

Fig 2.3 Systematic errors of three sub-pixel registration algorithms for rigid body translation images over a 0–1 pixel displacement range with the 31 × 31 pixels subset (a) and the 61 × 61 pixels subset (b). Standard deviation of three sub-pixel registration algorithms for rigid body translation images over a 0–1 pixel displacement range with the 31 × 31 pixels subset (c) and the 61 × 61 pixels subset (d) [24]

Fig 2.4 ε_{yy} values calculated for a pattern deformed in compression along y (vertical direction). (a) 8 × 8 pixels window with 0% overlap (b) 16 × 16 window with 50% overlap

Fig. 2.5 Equivalent stereovision systems (a) Standard Stereovision (SSV) with two identical cameras imaging at positions symmetric about the specimen normal axis; (b) Moving Camera Stereovision (MCSV), position 1 and position 2 are the positions of Camera 1 and 2 in 1(a) respectively; (b.1) and (b.2) are individual representations for a camera in position 1 and 2, respectively; (c) Tilting Specimen Stereovision (TSSV), the images of the specimen in position 1 and 2 can be decoupled to give the same perspective as shown in (b.1) and (b.2) [38]

Fig. 2.6 Examples of stereo and volumetric DIC. (a) 3D displacement field computation associated with a stamping process [30]. (b) 3D rendering of a specimen of Hostun sand showing the grain detail [40]. (c) Discrete VDIC derived grain displacements (vertical component) viewed in three orthogonal slices through the volume for strain increment of about 7%. Grains coloured grey are those for which the image correlation was not successful [40]

Fig 2.7 Examples of patterns not suitable for DIC: (a) lamellae features. (b) gradual gray level variation. (c), (d) and (e) examples of suitable patterns [30]

Fig 2.8 Schematic representation of the aperture problem. The displacement of a needle type feature cannot be measured accurately along the direction normal to the feature
Fig 2.9 Speckle patterns used for numerical experiment: (a) Speckle Pattern A and its histogram, (b) Speckle Pattern B and its histogram, (c) Speckle Pattern C and its histogram and (d) Speckle Pattern D and its histogram [43]

Fig 2.10 Curves of the mean bias errors related to the imposed displacement for four speckle patterns. Results calculated with the subset of (a) 41 × 41 pixels and (b) 71 × 71 pixels [43]

Fig 2.11 (a) Schematic of a scanning electron microscope and imaging process (a) [44]. (b) Comparison between ideal and actual e-beam scanning process for SEM imaging [13]

Fig 2.12 Image distortion fields for Quanta 200 at 300× associated with specific imaging parameters [38]

Fig 2.13 (a) BE image of the 2 µm pitch gold microgrid pattern [54]. (b) BEI image of specimen surface after Au coating through a mesh [55, 56]. (c) SE image of the hafnium oxide speckle pattern created by e-beam lithography (also shown is a grid pattern) [58]. (d) BE image of FIB assisted Pt nano dots at 20\% coverage [59]. (e) BE image of sputtered Pt nanoparticles [57]. (f) SE image of Au nanoparticles [61]

Fig 2.14 Schematic of two patterning arrangements for metallic thin films [63]

Fig 3.1 (a) Diagram of a typical EBSD installation [65]. (b) Diffraction pattern and indexing of a GaAs lattice [67]

Fig 3.2 (a) and (b) Schematic showing the effect of a subgrain boundary on the EBSD pattern. (c) and (d) EBSD patterns from well-prepared surface and from a poorly prepared surface of zirconium [77]

Fig 3.3 A crystal lattice strained 11\% along the horizontal direction and a schematic overlay of the patterns with (red) and without strain (black) [77]. (b) Schematic diagram showing how a strain and rotation (exaggerated) act to alter a zone axis direction \( r \) shifting across the EBSD screen by \( q \) [81]

Fig 3.4 Variations in the finite lattice rotation tensor (R) and elastic (Green’s) strain tensor measured using HR-EBSD in deformed titanium. A slip band location is illustrated with a black dashed line, which terminates at the grain boundary. (Colour scale for the lattice rotation matrix is \( \pm 5 \times 10^{-2} \) for the off diagonal terms and \( 1 \pm 2.5 \times 10^{-3} \) for the leading diagonal. Color scale for the elastic strain tensor is in absolute strain measured. All maps are plotted with respect to the reference point for each grain. [82]

Fig 3.5 Representation of link between lattice curvature components \( \Delta \theta_{12} \) and \( \Delta \theta_{11} \) and Burgers vectors of dislocations piercing a volume of material

Fig 3.6 Crystal lattice rotations: (a) in-plane lattice rotation, \( \omega_{x} \) about the \( x_{3} \)-axis. (b) out-of-plane crystal lattice rotation, \( \omega_{out} \). (c) and (d) Lower bound GND density calculation (m\(^{-2}\)) on distinct plane strain slip systems with superimposed traces [74]
Fig 4.1 Representation of the relationship between spatial and Euler reference systems in commercial software for EBSD data acquisition [92]

Fig 4.2 Scanning electron micrograph of (a) an undeformed and (b) a 7.4% deformed OFHC copper specimen. Horizontal loading direction [48]

Fig 4.3 Cumulative strain maps for the deformation step from 0 to 7.4% macroscopic tensile strain. Plotted are the axial $\varepsilon_{xx}$, transverse $\varepsilon_{yy}$ and shear strains $\varepsilon_{xy}$ as well as the planar rotations $r_{xy}$ (loading is along the horizontal $x$ direction) [48]

Fig 4.4 (a) The local lattice rotation angle for the deformation step from 0 to 7.4% macroscopic tensile strain measured along two profiles L1 and L2. The vertical lines indicate the position of the grain boundary. (b) Distribution of the axial $\varepsilon_{xx}$, transverse $\varepsilon_{yy}$ and shear strains $\varepsilon_{xy}$ along the same profiles [48]

Fig 4.5 (a) Microstructure determined by EBSD analysis – Schmid factors (white = high value, black = low value). (b) Axial strain field $\varepsilon_{xx}$ (loading along the horizontal $x$ direction). (c) Superposition of both experimental results [54]

Fig 4.6 Comparison of the axial (horizontal) strain field $\varepsilon_{xx}$. (a) DIC measurements (b) simulation with experimental boundary conditions and (c) simulated with homogeneous strain boundary conditions [54]

Fig 4.7 (a) Maximum shear strain map of a Ni-based superalloy sample deformed in tension along the horizontal direction to 2%. (b) Maximum shear strain map with the grain boundaries overlaid [57]

Fig 4.8 (a) Schmid factor map of the region of interest and (b) maximum shear strain vs Schmid factor at a nominal stress of 1280 MPa [57]

Fig 4.9 Fig. 7 (a) inverse pole figure map (b) tensile strain in the $x$-direction (c) shear strain and (d) tensile strain in the $y$-direction [58]

Fig 4.10 (a) visible cracks and slip steps on the sample surface correlate with (b) high angle and twin boundaries. (c) principle and (d) shear strain maps. Schmid Factors for [111] slip systems in (e) $<110>$ or (f) $<112>$ slip directions [58]

Fig 4.11 Calculated axial strain field evolution for a tensile test on 1100Al. Iron intermetallic particle at the bottom right of the map

Fig 4.12 (a) Inverse pole figure map of the investigated area. (b) Strain map and overimposed grain boundaries and (111) plane traces (black lines)

Fig 5.1 Comparison between the length scales of investigation of distinct microscopy techniques

Fig 5.2 (a) Phase distributions in an idealized multiphase titanium microstructure. (b) Plot of total slip system activity for normalized by the average rate, so that the average becomes white on the indicated color map. Blue indicates zero slip activity, and values greater than or equal to twice the average are plotted in red [97]

Fig 5.3 Kinematic model of elastoplastic deformation of a single crystal
**Fig 5.4** Kinematic model of elastoplastic deformation of a single crystal emphasizing the lack of experimental methods available to measure $F^p$.

**Fig 7.1** Comparison between (a) random distribution of speckles and (b) homogeneous distribution. Possible subsets position highlighted in red.

**Fig 7.2** Illustration of a possible application of nanoscale speckle pattern for studying the micromechanisms of plastic deformation in crystals.

**Fig 7.3** Illustration of three possible nanoindenter positions for inducing deformation incompatibilities in a microvolume.

**Fig A.1** Schematization of identification process of grain boundary points in the EBSD map. The points were the black arrows start are those identified as a boundary points. The red arrow indicates mis-indexed points. The mis-indexed point in (a) can be identified by a second scan along a distinct direction (b).

---

**List of figures in Paper 1**

- **Fig 1** Apparatus used for the remodelling of the deposited gold layer.
- **Fig 2** Speckles patterns formed after 1 hour vapour exposure at 280°C using apparatus in Fig. 1(a) following distinct surface finish and gold film deposition. 0.25 micron diamond finish and 50 nm and 80 nm film thickness (a), (b). 0.05 micron aluminia finish for 80 nm film thickness (c). 10 minutes of 0.05 micron colloidal silica polishing with a 1:30 volumes diluted solution (pH≈7) for 30 nm 50 nm and 80 nm film, (d), (e), (f). 10 minutes of 0.05 microns colloidal silica polishing with a 1:10 diluted solution (pH≈8) for 50 nm and 80 nm film (g), (h). 20 minutes 0.05 microns colloidal silica polishing with a 1:10 diluted solution (pH≈8) for 80 nm film (i). Patterns most suitable for plastic strain mapping with sub-micron resolution are highlighted in red.
- **Fig 3** Diagram showing the change in pattern suitability for DIC as a function of gold film thickness and polishing time for an OPS solution diluted to reach a pH between 7 and 8.
- **Fig 4** Values of maximum shear strain induced by the spatial distortion associated with instabilities of the raster scanning process using $6 \times 6$ (a) and $4 \times 4$ (b) interrogation window sizes.
- **Fig 5** Average values of maximum shear strain and scatter induced by the spatial distortion associated with instabilities of the raster scanning process for different interrogation window sizes.
**Fig 6** Detail of the speckle pattern at 0% (a) and 5% (b) macroscopic strain. Displacement vector field after correlation with sub-micron resolution (c) and maximum shear strain mapping showing regular spaced bands to about 1 µm (d)

**Fig 7** Values of maximum shear strain calculated for 7% macroscopic elongation using different sub-region sizes: 9 × 9 µm² with 50% overlap (a) 216 × 216 nm² with 0% overlap (b)

**Fig 8** Values of maximum shear strain calculated for 1.5% macroscopic elongation (a) and 5% macroscopic elongation (b). Intersecting bands “I”, abrupt change in band direction “C”, hot spot of intense strain localization “H”

**Fig 9** Detail of the strain maps of the region of interest at 1.5%, 5% and 7% macroscopic strain in grey scale adjusted to highlight the distribution of bands

**Fig 10** Maximum shear strain as a function of the strain along the loading direction for the studied region. Max. shear strain for the whole region, blue dots. Max. shear strain for the “hot spot” areas indicated in Fig. 8 (b), remaining markers

**Fig 11** Values of maximum shear strain calculated for 7% macroscopic elongation in the deformed configuration. Grain boundaries and {111} planes traces obtained from lattice orientation data analysis acquired following deformation are superimposed. Traces of planes with the two highest Schmid factors are highlighted in red. Red dots indicate the grains where only partial correspondence is found between high Schmid factor traces and bands directions

**Fig 12** Values of maximum shear strain (a) and rotation angle (b) for an area at the top-right corner of the investigated region. Deformation schematized in (c), boundaries depicted using dashed lines

---

**List of figures in Paper 2**

**Fig 1** (a) Specimen geometry and highlight of the investigated region, units in millimeters; thickness 2mm. (b) Details of gold speckles pattern imaged using electron backscatter mode.

**Fig 2** Values of maximum shear strain for 5% macroscopic elongation. Strains are expected to induce an equivalent distribution of statistically stored dislocations.

**Fig 3** Lattice orientation map (EBSD map) of the investigated region in inverse pole figure (IPF) colors. Boundaries and deformation twins highlighted.

**Fig 4** Detail of the maximum shear strain map in Figure 2. Grain boundaries and {111} plane traces (in purple) are superimposed to evidence the crystallographic nature of microscale deformation.
Fig 5 Kernel average misorientation (KAM) results from the lattice orientation data of the investigated region using a 3×3 kernel size.

Fig 6 Orientation spread map of the investigated region, values in degrees.

Fig 7 Detail of a grain in the region of interest. (a) Maximum shear strain map. (b) Channeling contrast image. (c) KAM map. (d) Orientation spread map.

List of figures in Paper 3

Fig 1 Kinematic model of elastoplastic deformation of a single crystal emphasizing the lack of experimental methods available to measure \( P \).

Fig 2 Comparison between the length scales of investigation of distinct microscopy techniques.

Fig 3 Schematization of the link between displacement field and strain gradient induced lattice curvature in condition of single slip.

Fig 4 Schematization of the step needed to meet the alignment of the slip \( s \) with the grid of the DIC software.

Fig 5 Kinematic model of elastoplastic deformation of a single crystal extended for two consecutive deformation steps \((t_1, F_1)\) and \((t_2, F_2)\).

Fig 6 Schematization of the relationship between lattice rotations predicted by the deformation mapping and the lattice orientation measured after deformation.

Fig 7 Microstructural features of the investigated region. Grain boundaries are depicted in black and the grain numbered in sequential order. BCC lattice is colored in red.

Fig 8 Values of Curl following 6% macroscopic tensile strain along the horizontal direction. Primary bands (PB) and secondary bands (SB) are highlighted for grain 3 as example.

Fig 9 Details of deformation features observed in Fig 6. Examples of curved bands, (a) and (b). Values of curl in correspondence of second phase particles, (c), (d) and (e).

Fig 10 Details of the gold speckle pattern in the investigated region before (a) and after 6% macroscopic strain. Curl calculated from the displacement field obtained by correlating the image of the deformed pattern to the image of the pattern before deformation (c). Interpretation of the kinematic of deformation (d).

Fig 11 Values of -curl calculated for 6% macroscopic elongation in the deformed configuration. \{111\} planes traces obtained from lattice orientation data analysis acquired following deformation are superimposed. Traces of planes with the two highest Schmid factors are highlighted in red. Red dots indicate the grains where...
only partial correspondence is found between high Schmid factor traces and bands directions

**Fig 12** In-plane lattice rotations calculated for each grain in the investigated area from EBSD map acquired after deformation

**Fig 13** HDIC derived lattice rotations for regions crossed by primary bands at an <90° with respect to the loading direction; the remaining areas are obscured

**Fig 14** Graphs showing the variation of DIC predicted and EBSD measured lattice rotations for the Linescans 1 to 4 in Fig 12 and Fig 13

**Fig 15** DIC derived lattice rotations from HDIC measurements for regions crossed by primary bands at >90° with respect to the loading direction

**Fig 16** Graphs showing the variation of DIC predicted and EBSD measured lattice rotations for the Linescans 5 and 6 in Fig 12 and Fig 15

**Fig 17** Details of Fig. 13 showing the interaction between bands and grain boundaries resulting in lattice curvature. The letters follow the sequence started in Fig. 15

**Fig 18** Predicted lattice rotations using the proposed method and considering the direction of slip coinciding with primary bands (left) and secondary bands (right). The orientation gradients given by the predicted counter-rotations is observed in the measured orientation gradients

**Fig 19** Schematization of a possible mechanism leading to diverted microbands for DIC measurements described in the initial configuration

**Fig 20** Values of −Curl(μ′) the boundary region between grain 2 and 3 (a). Evidence of dislocation structures in the channeling contrast images that can be attributed to SSDs densities

**Fig 21** Mechanisms of deformation-induced lattice curvature observed for the investigated material

---

**List of figures in Paper 4**

**Fig 1** Transmission electron micrographs from sections parallel to the transverse plane from A1-0.8 Si. (a) and (b) after cold rolling to a reduction of ε=0.11. (c) after cold rolling to a reduction of ε=0.29. Images show alignment of cell walls along a {111} plane trace and high-density dislocation patterns aligned perpendicular to this plane at the particle (P). Courtesy of Humphreys FJ

**Fig 2** Kinematic model of elastoplastic deformation of a single crystal

**Fig 3** Schematization of the link between displacement field and lattice curvature in condition of single slip as theorized by strain gradient plasticity
Fig 4 Schematization of the convention used for plotting in-plane lattice rotations and associated net Burgers vector field.

Fig 5 Schematization of the step needed to meet the alignment of the slip $s$ with the grid of the DIC software.

Fig 6 Backscatter electron images of the investigated area at 0% (a), 10% (b) and 15% (c) macroscopic compression. Image acquired after 20% compression rotated to align the horizontal direction along the direction of slip (d). $O_{sd}$ represents the region of the matrix fixed with respect to the observer.

Fig 7 (a) Predicted lattice rotations induced by the accommodation of incompatible deformation between matrix and particle. The square highlights the region for which the lattice curvature is depicted in Fig. 7 (b).

Fig 8 (a) Derived values for the component of the tensor $F^p_{12}$ describing the shear given by lattice slip. (b) Measured values for the component of the deformation gradient tensor $F_{12}$.

Fig 9 Graph plotting the average values of the components $F^p_{12}$ and $F_{12}$ calculated for the areas highlighted in Fig. 8.

Fig 10 (a) Derived values of compression normal to the direction of slip $x_1$. (b) Values of stretch along the direction of slip $x_1$.

Fig 11 Values of $\varepsilon^p_{12(\text{MAX})}$ for the investigated area.

Fig 12 Net Burgers vector associated with in-plane lattice curvature at the particle matrix interface.

Fig 13 Details of net Burgers vector field associated with in-plane lattice curvature at the top and bottom sides of the particle.

Fig 14 Magnitude of the net Burgers vector associated with in-plane lattice curvature in gray scale and reduced range of values to evidence GNDs patterns. DP1 and DP2 locate the patterns shown Fig. 13.

Fig 15 Schematization of the interaction between hard particles embedded in a softer matrix shearing of a prescribed amount $\Gamma=4\gamma_0$ which is equally distributed in four slip planes. (a) Particles of dimension order of magnitude smaller than the slip spacing do not interfere with the slip. (b) Particle of dimension of the order of the band spacing cause it to interfere with the matrix shear deformation. The resistance to rotation induces strain gradients at the particle/matrix interface.

Fig 16 Schematization of the lattice slip distribution in the particle/matrix interface. (a) Intermediate configuration ($F^P$). (b) Deformed configuration following strain gradient induced lattice rotations.
List of tables

Table 1.1 Summary of patterning techniques for SEM-DIC [62]
Table 3.1 Summary of typical EBSD performance for different metals [25]. Quantities $\Lambda_A$ and $\Lambda_P$ describe the elliptical geometry of the interaction volume
Table 3.2 The dislocation basis used to describe the dislocation state in FCC crystals [90]
Table 4.1 A list of studies dedicated to large deformation mapping in metals at reduced length scales

List of tables in Paper 1

Table 2 A list of experimental parameters and observations from previous investigations dedicated to large deformation mapping in metals at reduced length scales
Table 2 Chemical composition of the 304L stainless steel in weight %
Table 3 SEM scan parameters used for image acquisition

List of tables in Paper 2

Table 1 Chemical composition of the 304L stainless steel in weight %

List of tables in Paper 3

Table 1 Chemical composition of the 304L stainless steel in weight %
Table 2 SEM scan parameters used for image acquisition
**List of symbols and abbreviations**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>DDD</td>
<td>Discrete Dislocations Dynamic</td>
</tr>
<tr>
<td>DIC</td>
<td>Digital Image Correlation</td>
</tr>
<tr>
<td>HDIC</td>
<td>High Resolution Digital Image Correlation</td>
</tr>
<tr>
<td>FEG-SEM</td>
<td>Field Emission Gun Secondary Electron Microscope</td>
</tr>
<tr>
<td>SEM</td>
<td>Secondary Electron Microscope</td>
</tr>
<tr>
<td>SEI</td>
<td>Secondary Electron Imaging</td>
</tr>
<tr>
<td>BEI</td>
<td>Backscatter Electron Imaging</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
</tr>
<tr>
<td>CCI</td>
<td>Channeling Contrast Imaging</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Backscatter Diffraction</td>
</tr>
<tr>
<td>ISDCC</td>
<td>Iterative Spatial Domain Cross-correlation</td>
</tr>
<tr>
<td>NR</td>
<td>Newton-Rapson</td>
</tr>
<tr>
<td>FFT</td>
<td>Fast Fourier Transformation</td>
</tr>
<tr>
<td>MSF</td>
<td>Mean Subset Fluctuation</td>
</tr>
<tr>
<td>FCC</td>
<td>Face Centered Cubic</td>
</tr>
<tr>
<td>BCC</td>
<td>Body Centered Cubic</td>
</tr>
<tr>
<td>KAM</td>
<td>Kernel Average Misorientation</td>
</tr>
<tr>
<td>GOS</td>
<td>Grain Orientation Spread</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite Element Method</td>
</tr>
<tr>
<td>CPFEM</td>
<td>Crystal Plasticity Finite Element Method</td>
</tr>
<tr>
<td>CPFFT</td>
<td>Fast Fourier Transformation-based Crystal Plasticity</td>
</tr>
<tr>
<td>GNDs</td>
<td>Geometrically Necessary Dislocations</td>
</tr>
<tr>
<td>SSDs</td>
<td>Statistically Stored Dislocations</td>
</tr>
<tr>
<td>SGP</td>
<td>Strain Gradient Plasticity</td>
</tr>
<tr>
<td>PDZ</td>
<td>Particle Deformation Zone</td>
</tr>
<tr>
<td>SCC</td>
<td>Stress-Corrosion Cracking</td>
</tr>
<tr>
<td>EAC</td>
<td>Environmental Assisted Cracking</td>
</tr>
<tr>
<td>IGSCC</td>
<td>Intragranular Stress-Corrosion Cracking</td>
</tr>
<tr>
<td>OPS</td>
<td>Colloidal silica solution</td>
</tr>
<tr>
<td>0</td>
<td>Order of magnitude</td>
</tr>
<tr>
<td>( \mathbf{u} )</td>
<td>Displacement vector</td>
</tr>
<tr>
<td>( c_{\text{ZNSSD}} )</td>
<td>Zero normalized sum-square difference correlation coefficient</td>
</tr>
<tr>
<td>( c_{\text{ZNCC}} )</td>
<td>Zero normalized cross-correlation coefficient</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>$f, g$</td>
<td>Gray levels images</td>
</tr>
<tr>
<td>$M$</td>
<td>Subset dimension</td>
</tr>
<tr>
<td>$d$</td>
<td>Approximation solution of the iteration</td>
</tr>
<tr>
<td>$d_0$</td>
<td>Solution of the previous interaction</td>
</tr>
<tr>
<td>$\nabla$</td>
<td>Gradient operator</td>
</tr>
<tr>
<td>$C$</td>
<td>Correlation coefficient</td>
</tr>
<tr>
<td>$S_f$</td>
<td>Mean subset fluctuation parameter</td>
</tr>
<tr>
<td>$R(\omega, r)$</td>
<td>Angle/axis rotation</td>
</tr>
<tr>
<td>$r$</td>
<td>Axis of rotation, lattice vector</td>
</tr>
<tr>
<td>$\omega, \theta, \varphi$</td>
<td>Angle of rotation</td>
</tr>
<tr>
<td>$\delta$</td>
<td>Misorientation angle error</td>
</tr>
<tr>
<td>$\beta$</td>
<td>Misorientation axis error</td>
</tr>
<tr>
<td>$Q$</td>
<td>Displacement of points in a crystal lattice</td>
</tr>
<tr>
<td>$q$</td>
<td>Projection of $Q$</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Unknown scalar</td>
</tr>
<tr>
<td>$q$</td>
<td>Quaternion</td>
</tr>
<tr>
<td>$m_s$</td>
<td>Quaternion describing a misorientation in sample coordinates</td>
</tr>
<tr>
<td>$K$</td>
<td>Lattice curvature tensor</td>
</tr>
<tr>
<td>$n$</td>
<td>Vector normal to surface</td>
</tr>
<tr>
<td>$\Pi$</td>
<td>Surface</td>
</tr>
<tr>
<td>$b$</td>
<td>Net Burgers vector</td>
</tr>
<tr>
<td>$b_{\text{par}}$</td>
<td>Partial net Burgers vector</td>
</tr>
<tr>
<td>$d$</td>
<td>Volume linear dimension</td>
</tr>
<tr>
<td>$G$</td>
<td>Geometrically necessary dislocation tensor (Nye tensor)</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>Slip system</td>
</tr>
<tr>
<td>$\rho_{GND}$</td>
<td>Geometrically necessary dislocation density</td>
</tr>
<tr>
<td>$s^\alpha$</td>
<td>Slip direction unit vector</td>
</tr>
<tr>
<td>$m^\alpha$</td>
<td>Normal to slip plane</td>
</tr>
<tr>
<td>$t_j^\alpha$</td>
<td>Dislocation line unit vector</td>
</tr>
<tr>
<td>$F$</td>
<td>Deformation gradient tensor</td>
</tr>
<tr>
<td>$F^e$</td>
<td>Elastic part of the deformation gradient tensor</td>
</tr>
<tr>
<td>$F^p$</td>
<td>Plastic part of the deformation gradient tensor</td>
</tr>
</tbody>
</table>
Abstract

CHARACTERIZATION OF MESOSCOPIC CRYSTAL PLASTICITY FROM HIGH-RESOLUTION SURFACE DISPLACEMENT AND LATTICE ROTATION MAPPINGS

Fabio Di Gioacchino

Doctorate of Philosophy, The University of Manchester, 2013

Being able to predict the evolution of plastic deformation at the microstructural scale is of paramount importance in the engineering of materials for advanced applications. However, this is not straightforward because of the multiscale nature of deformation heterogeneity, both in space and time\(^1\). The present thesis combines four related studies in a coherent work, which is aimed to develop experimental methods for studying crystal plasticity at the micro and mesoscale.

A novel methodology for gold remodelling is initially proposed and used to apply high-density speckle patterns on the surface of stainless steel specimens. The unique properties of the speckle pattern enabled plastic deformation mapping with submicron resolution using digital image correlation (HDIC). It was therefore possible to study the concomitant evolution of microbands and transgranular deformation bands in such alloy.

High-resolution deformation mapping also enabled comparison with high-resolution electron backscatter diffraction (EBSD) observations. The only partial correspondence of results proved the limits of EBSD in characterizing plastic deformation. The cause of such limitation is later identified in the reduced sensitivity to lattice slip of the EBSD technique. Hence, a novel method of HDIC data analysis is proposed to separate the contributions of lattice slip and lattice rotation from the deformation mapping. The method is adopted to characterize plasticity in austenitic stainless steel and at the plastic deformation zone (PDZ) around a silicon particle embedded in a softer aluminum matrix. Results show that the proposed experimental methodology has the unique capability of providing a complete description of the micro and mesoscale mechanics of crystal plasticity. HDIC therefore emerges as a key technique in the development of accurate physical-based multiscale crystal plasticity models.

---

Declaration
I, the author, declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.

Submission in Alternative Format
This thesis has been submitted in alternative format with permission from the Faculty of Engineering and Physical Science.

The author
The author obtained an MPhil in Mechanical Engineering at the University of Bologna (Italy) in 2007. He started his PhD course in Materials Science at The University of Manchester (UK) in September 2008 under the supervision of Dr. João Quinta da Fonseca.
Copyright Statement

i. The author of this thesis (including any appendices and/or schedules to this thesis) owns certain copyright or related rights in it (the “Copyright”) and s/he has given The University of Manchester certain rights to use such Copyright, including for administrative purposes.

ii. Copies of this thesis, either in full or in extracts and whether in hard or electronic copy, may be made only in accordance with the Copyright, Designs and Patents Act 1988 (as amended) and regulations issued under it or, where appropriate, in accordance with licensing agreements which the University has from time to time. This page must form part of any such copies made.

iii. The ownership of certain Copyright, patents, designs, trade marks and other intellectual property (the “Intellectual Property”) and any reproductions of copyright works in the thesis, for example graphs and tables (“Reproductions”), which may be described in this thesis, may not be owned by the author and may be owned by third parties. Such Intellectual Property and Reproductions cannot and must not be made available for use without the prior written permission of the owner(s) of the relevant Intellectual Property and/or Reproductions.

iv. Further information on the conditions under which disclosure, publication and commercialisation of this thesis, the Copyright and any Intellectual Property and/or Reproductions described in it may take place is available in the University IP Policy (see http://documents.manchester.ac.uk/DocuInfo.aspx?DocID=487), in any relevant Thesis restriction declarations deposited in the University Library, The University Library’s regulations (see http://www.manchester.ac.uk/library/aboutus/regulations) and in The University’s policy on Presentation of Theses.
Acknowledgments

I am truly indebted and thankful to Dr. Joao Quinta da Fonseca for his trust and guidance during the years of my PhD studies. I greatly profited from engaging in discussions with a person of such genius and wit.

I am also grateful to Dr. Fabio Scenini for his great support and to Mrs. Rebecca Sandala for her generosity.

I would like to acknowledge my research colleagues Dr. Kuveshni Govender, Mr. David Wright and Mr. David Gonzalez and all the guys from the D7 office and thank them for their friendship.

I am finally grateful to the examiners Prof. Angus Wilkinson and Prof. Michael Preuss for their valuable comments and suggestions for improvements to the present thesis.

This thesis is dedicated to my wife Johana, who is proud of me as much as I am of her.
1 Introduction

Extensive transmission electron microscopy (TEM) studies established that cold deformation of crystalline materials is accomplished by concurrent lattice slip and lattice rotation. Such mechanisms of plastic deformation occur heterogeneously at various length scales as distinct deformation features and dislocation structures can be detected at different magnifications. Examples are the formation dislocation cells and subgrains at the microscale or microbands and twins at grain scales [1-3]. The way cold deformation microstructure evolves during deformation is believed to govern the mechanical behavior of the material and also to affect its corrosion resistance [4, 5].

Modelling strategies appropriate for each scale span from atomistic simulation and discrete dislocations dynamics (DDD) to the continuum crystal plasticity and continuum plasticity, Fig. 1.1 [6]. Detailed reviews on the plethora of models developed over the years and their distinctive range of application can be found in Dowell (2008) [6]. Here, it is reported that authors have progressively moved towards a multiscale approach to account for the interaction between different length scales.

![Fig 1.1 Distinct scales of investigation in crystal plasticity [6]](image)

Predictions from multiscale modelling nevertheless suffer from inadequate experimental validation. This is because, unlike atomic scale simulations, which can be supported by TEM observations [7, 8], and unlike crystal plasticity models capable of predicting macroscopic phenomena such as texture, which can be validated by X-ray and neutron diffraction studies [9, 10], multiscale models require quantitative experimental observations that could bridge between intermediate scales. From Dowell (2008) [6]:
“The...“gulf” between atomistic simulation and continuum crystal plasticity models exists in part due to the lack of unambiguous data from observations of time-resolved dynamic formation of dislocations substructures, burst of strain associated with flow heterogeneity, etc.”

In practice, mapping techniques should be adopted which could span across submicron (O 10^{-7} m) and grain scales (O 10^{-5} m). Two candidate microscopy techniques are digital image correlation (DIC) and electron backscatter diffraction (EBSD). The present work is dedicated to the study of plastic deformation in crystals using these experimental techniques.

1.1 Thesis layout

The present thesis is written in an alternative format, which allows including documents submitted or in a format suitable for submission to peer-reviewed journals.

As for the present chapter (Chapter 1), the following three chapters, i.e. chapters 2 to 5, are introductive chapters.

Chapter 2 introduces the basic principles of DIC and SEM imaging. Also, it describes methods for high-density speckle pattern application that enable high-resolution DIC (HDIC).

Chapter 3 presents the basic principles of electron backscatter diffraction (EBSD). In particular, it describes how the technique can be used to extract the components of elastic strain and curvature of the crystal lattice.

Chapter 4 discusses the possibility of combining DIC and EBSD measurements to study plastic deformation at the microstructural scale. It also reviews the main results of previous related studies.

Chapter 5 gives a brief description of the basic principles in the continuum approach to crystal plasticity. Moreover, it emphasizes the lack of experimental methods available to describe plastic deformation at the mesoscopic scale. It also provides a list of aims and introduces the materials chosen for testing.

Chapter 6 covers the original contributions of the present work. These are presented as four papers positioned in a logical sequence to form a coherent study, which is also self-contained as it includes the description of the experimental and analytical methods used. The common line of the papers is the development of advanced experimental and analytical DIC methodologies for the characterization of crystal plasticity at the micro and mesoscale. Further submission or publication details are reported at the beginning of each paper.
**Paper 1** Plastic strain mapping with submicron resolution using digital image correlation

Abstract. Digital image correlation (DIC) of images obtained using scanning electron microscopy has been used to study, quantitatively, the plastic deformation of stainless steel at the microstructural scale. An artificial speckle pattern was generated by the remodelling of a deposited gold layer. A new experimental setup was shown to accelerate the remodelling process and promote the formation of finer nano-scale speckles with sizes ranging 30 nm to 150 nm and of similar spacing. The effects of surface preparation on speckle morphology are discussed. The high density of speckles enabled displacement mapping with resolution of one displacement vector each $0.2 \times 0.2 \ \mu m^2$ of surface area. It is shown that sub-micron resolution is necessary to capture the plastic deformation associated with the formation of slip bands in stainless steel, which are an important component of the deformation of these materials at the microscale. Electron backscatter diffraction (EBSD) was used to reconstruct the surface grain boundaries and enabled these deformation features to be linked to the microstructure.

**Paper 2** Understanding the limits of lattice orientation data analysis in environmental degradation studies

Abstract. Cold working can significantly increase the susceptibility of metals to environmentally assisted cracking. However, the reasons for this increased susceptibility are still unclear. This is due in part to the difficulty in quantifying and modelling plastic deformation at the required scale. Here, we use a new experimental procedure to study the local microstructural distribution of strain in 304L stainless steel. Digital image correlation was used to map strain at the microstructural level with sub-micron resolution. The results clearly show that a high degree of strain localization develops within individual grains, in the form of highly localized shear bands and micro-twinning. Electron backscatter diffraction was used to quantify the lattice orientation changes in the same area. Analysis of this data included the calculation of kernel average misorientation and of intragranular orientation spread following grain reconstruction. Comparisons of results clearly show that, in most cases, there is no evidence in the lattice orientation data analysis of the high levels of strain measured by DIC. This has important implications in the use of lattice orientation data in the study of the effects of plastic deformation on environment-assisted cracking.
1. Introduction

**Paper 3**  *Separating lattice rotation gradients from high-resolution deformation mapping. Insights on the microplasticity of austenitic stainless steel*

*Abstract.* High-resolution digital image correlation (HDIC) of FEG-SEM images is used to map the in-plane deformation of an aggregate of grains at the surface of a 304 austenitic stainless steel sample. Intergranular microbands appeared aligned with [111] plane traces obtained from the analysis of electron backscatter diffraction (EBSD) data. Such direction was thus taken as the main (in-plane) direction of local lattice slip. Hence, it was shown that it is possible to separate the contribution of lattice rotation from the local deformation mapping following deformation incompatibility arguments. To validate the proposed decomposition method, DIC-derived lattice rotations were compared with those measured by EBSD. Good agreement of results proved, for the first time, the existence of the link between certain gradients of slip and the lattice curvature. Results from the analysis of HDIC and EBSD measurements are further discussed to give new insights on the micromechanism of deformation in austenitic stainless steel.

**Paper 4**  *Characterization of crystal plasticity and dislocation density evolution at the particle deformation zone using high-resolution displacement mapping*

*Abstract.* High-resolution digital image correlation (HDIC) of FEG-SEM images is used to map the deformation of an aluminum matrix surrounding a hard silicon particle. A novel method for HDIC data analysis is used to separate the contributions to plastic deformation of lattice rotation and lattice slip. The mapping of lattice rotations is used to plot the net Burgers vector field describing the necessary dislocation state. The mapping given by lattice slip is instead used to characterize the distribution of statistically stored dislocations. Results provide unique insights on the kinematic of crystal plasticity at the particle deformation zone (PDZ). It is therefore demonstrated that HDIC is an essential experimental technique in the study of microplasticity.

**Chapter 7** contains concluding remarks that summarize the main results and contributions of the present work. Future investigations are also outlined.

**Appendix A** gives additional details on the way EBSD data were manipulated in the analysis.

**References** of the studies cited in Chapters 1-5 and 7 are arranged at the end of the document.
2 Introduction to digital image correlation at high magnification

2.1 Basic principles of DIC

Digital image correlation (DIC) is a computational technique that enables full-field displacement mapping of deforming surfaces by comparing pixel intensities of digital images acquired at different stages of deformation. The displacement mapping is achieved by dividing the images into smaller pixel subsets (sub-regions), which are individually correlated, Fig. 2.1 [11].

Fig 2.1 Schematization of the DIC principle. (a) Division of the images to be correlated in subsets. (b) Correlation of initial and deformed subsets to yield a displacement vector [11]

In order to measure the degree of correlation, a correlation coefficient must be predefined. Hence, a subset-matching algorithm is used to search the subset position in the deformed image giving the highest correlation. The differences in position of the correlated subsets yield the in-plane displacement vector, as depicted in Fig. 2.1. Performing this calculation for all subsets gives a displacement vector field that can be differentiated to give the components of the in-plane strain tensor [12, 13].
Each subset must show a unique set of features to be identifiable at different
deformation stages. Therefore, DIC usually requires the prior application of a speckle
pattern onto the surface of the sample. The pattern has to show certain morphology
and imaging properties; these will be discussed in section 2.2.

2.1.1 Correlation coefficient
The reference image $f$ and the “deformed” image $g$ can be described as data sets of
gray levels associated with pixel coordinates $f = f(x_i, y_j)$ and $g = g(x'_i, y'_j)$.
Considering square subsets of dimension $(2M + 1) \times (2M + 1)$ pixels and centered in
the point $P(x_0, y_0)$, zero normalized sum-square difference correlation coefficient $C_{ZNSSD}$ and zero normalized cross-correlation coefficient $C_{ZNCC}$ have been defined as:

$$
C_{ZNSSD} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ \frac{f(x_i, y_j) - f_m}{\Delta f} - \frac{g(x'_i, y'_j) - g_m}{\Delta g} \right]
$$

(2.1)

$$
C_{ZNCC} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ \frac{(f(x_i, y_j) - f_m) \times (g(x'_i, y'_j) - g_m)}{\Delta f \Delta g} \right]
$$

(2.2)

with:

$$f_m = \frac{1}{(2M+1)^2} \sum_{i=-M}^{M} \sum_{j=-M}^{M} f(x_i, y_j)
$$

$$\Delta f = \sqrt{\sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ f(x_i, y_j) - f_m \right]^2}
$$

$$g_m = \frac{1}{(2M+1)^2} \sum_{i=-M}^{M} \sum_{j=-M}^{M} g(x'_i, y'_j)
$$

$$\Delta g = \sqrt{\sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ g(x'_i, y'_j) - g_m \right]^2}
$$

The two coefficients are related as shown by Pan in [14]. From equations (2.1) and (2.2), it is evident that the values of both coefficients range in the interval $[0, 1]$. In particular, for increasing matching between the correlated images, $C_{ZNSSD} \rightarrow 0$ and $C_{ZNCC} \rightarrow 1$. The desirable property of these correlation measures with respect to alternative CC and SSD criteria is the insensitivity of the formers to the offset and change in scale of lightning [12 - 14]. It is in fact possible to consider a second deformed image $g'$ as:
\[ g'(x'_i, y'_j) = a \times g(x_i, y_j) + b \]  

(2.3)

where \( a \) represents the scale and \( b \) the offset of lighting.

Substituting (2.3) in (2.1) and (2.2) it is possible to show that equivalent values of \( C_{ZNCC} \) and \( C_{ZNSSD} \) will be obtained for both \( g'(x'_i, y'_j) \) and \( g(x_i, y_j) \). This indicates that such correlation coefficients can offer excellent imaging noise proof performance [14].

2.1.2 Shape function and interpolation scheme

Even with “well-behaving” speckle patterns, a low correlation measure can be induced by significant in-plane deformation of the area covered by the subset. Implementing so-called shape functions \( \xi = \xi(x_i, y_j) \) and \( \eta = \eta(x_i, y_j) \) can help improving the correlation measure and therefore the accuracy of DIC measurements [15]. These functions alter the position of the pixel whilst conserving their intensity, with \((i, j = -M: M)\):

\[ x'_i = x_i + \xi(x_i, y_j) \]  

(2.4)

\[ y'_i = y_i + \eta(x_i, y_j) \]  

(2.5)

First order shape function in (2.6) and (2.7) are commonly used because these suffice to describe translation, rigid rotation, shear and normal strains [16]:

\[ \xi(x_i, y_j) = u + u_x \Delta x + u_y \Delta y \]  

(2.6)

\[ \eta(x_i, y_j) = v + v_x \Delta x + v_y \Delta y \]  

(2.7)

with \( u \) and \( v \) being the displacement components of the pixels.

Fig. 2.2 shows examples of changes in pixels position associated with first and second order shape functions. Yet, higher order functions can be implemented at a cost of computational time to describe more complex deformation of the subset. It is evident from equations (2.4 – 2.7) that the new position of the pixels will generally locate within original pixels (sub-pixel location). Implementing a shape function therefore requires the use of interpolation methods to derive the intensity of these points with sub-pixel location. Different orders of interpolation can be used. Schreir et al. [17] and Knauss et al. [18] showed that higher orders interpolation schemes significantly improve the accuracy of displacement measurements.
1. Digital Image Correlation

2. Sub pixel registration algorithm

Similarly to the effect of a shape function, the actual displacement of speckles will in general correspond to decimals of pixels. It is therefore necessary to implement sub-pixel registration algorithms to increase the accuracy of displacement measurements. For the algorithm to converge toward a solution, it is usually required a less accurate (pixel level of accuracy) initial guess of the displacement. This can be achieved through different methods, the most common being the coarse-fine scheme (or nested searching scheme) and Fourier domain based methods. The first is an iterative method that uses a progressively finer subset size for correlation. The second method instead correlates the reference and deformed subsets in Fourier domain to take advantage of the computational speed associated with fast Fourier transformation (FFT). Further details of the latter methods can be found in the references given above.

Once the initial guess is made, different sub-pixel displacement algorithm can be used. A possibility is to extend the coarse-fine method to search steps of sub-pixel dimension; this would nevertheless lead to an exponential increase in computational time. Faster and most commonly used methods are the peak-finding algorithm and the iterative spatial domain cross-correlation (ISDCC) algorithm. As the name suggests, the first method is based on interpolation schemes to find the maximum (or minimum for SSD coefficient) position of the peak in the surface interpolated from values of correlation coefficient in neighboring points.
Again, various order interpolation schemes can be used [12]. However, the method does not consider the shape change of the deformed subset and therefore has limited accuracy [24, 26].

![Graphs showing systematic errors of three sub-pixel registration algorithms for rigid body translation images over a 0–1 pixel displacement range with the 31 × 31 pixels subset (a) and the 61 × 61 pixels subset (b). Standard deviation of three sub-pixel registration algorithms for rigid body translation images over a 0–1 pixel displacement range with the 31 × 31 pixels subset (c) and the 61 × 61 pixels subset (d) [24].](image)

The ISDCC method implements the Newton-Rapson (NR) iterative method to converge to the solution. As noted by Pan [24] the method has been proved to account for the deformation of the subset.
The NR method was originally developed by Bruck et al. [25] and defined in the form of:

$$
\mathbf{d} = \mathbf{d}_0 - \frac{\nabla \mathcal{C} (\mathbf{d}_0)}{\nabla^2 \mathcal{C} (\mathbf{d}_0)}
$$  \hspace{1cm} (2.8)

where \(\mathbf{d}\) is the approximation solution of the iteration, \(\mathbf{d}_0\) is the solution of the previous iteration (which start with the initial guess) and \(\nabla \mathcal{C} (\mathbf{d}_0)\) and \(\nabla^2 \mathcal{C} (\mathbf{d}_0)\) are the first (gradients) and second order derivation (Hessian matrix) of the correlation criteria \(\mathcal{C}\). Vendroux and Knuass [26] have later showed that the drawback of computational load can be greatly reduced by approximating the Hessian matrix.

Other algorithms have been proposed, namely: spatial gradient-based algorithms [27], genetic algorithms [28] and algorithms using FEM-based interpolation [29]. Within the plethora of suggested algorithm, Pan [24] showed that implementation of the NR method provides the highest accuracy and reliability in experimental use.

Fig. 2.3 compares results of sub-pixel registration accuracy of the correlation between computer-generated speckle patterns of known imposed deformation using different sub-pixel registration schemes.

### 2.1.4 Strain estimation and subsets overlap

As mentioned above, displacements can be differentiated to obtain strain components\(^2\). Yet, the numerical differentiation amplifies the noise contained in the computed displacement.

Three distinct components contribute to give the measured displacement, from [30]:

- the actual (mechanical) displacement
- a systematic error, which corresponds to the deterministic part of error induced by measurement conditions
- a random error, which corresponds to the non-deterministic part of error induced by measurement conditions

The systematic error is expected to equally affect the correlated images and therefore not to contribute to the noise in strain measurements [30]. Hence, experimental settings and imaging conditions need to be arranged in order to minimize the random error; refer to section 2.3.1 for discussion on the source of error in SEM imaging.

\(^2\) Displacement gradients can be directly obtained during the correlation process using NR-based algorithms as shown in [25].
The use of large subsets helps averaging out the random error to give more accurate measurements [31, 32]. Obviously, this will be achieved at the expenses of the spatial resolution. Hence, a variety of filtering techniques have been proposed to smooth the displacement field and reduce the noise of strain measurements without affecting the resolution; example includes the use of the penalty finite element method [33], pointwise local least-squares fitting technique [34] or moving least-squares (MLS) [35].

Post-processing of the displacement vectors is available in commercial DIC software [36, 37]. Also, DIC software gives the possibility to overlap subsets so that the overlapped areas are included in multiple correlations. The amount of overlap can be directly selected or it derives from reducing the distance between the centers of adjacent subsets (subset spacing). The operation increases the density of calculated vectors of an amount proportional to the overlap. For instance, a $8 \times 8$ pixels window with 0% overlap gives the same vector density as a $16 \times 16$ window with 50% overlap, Fig 2.4.

![Fig 2.4](image)

**Fig 2.4** $\varepsilon_{yy}$ values calculated for a pattern deformed in compression along $y$ (vertical direction). (a) $8 \times 8$ pixels window with 0% overlap (b) $16 \times 16$ window with 50% overlap

For a given density of displacement vectors, the redundant correlation improves the uniformity of the displacement field and therefore reduces the noise in strain mapping, Fig. 2.4. It is nevertheless important to find a compromise between reducing the effect of noise on the strain field and losing mechanical information due to excessive smoothing [30]. This is particularly true when the actual deformation is expected to be highly heterogeneous [12], i.e. when steep strain gradients are anticipated, as in the case of plastic deformation of crystals at the microstructural scale.
2.1.5 In-plane, stereovision and volumetric DIC

Two-dimensional (in-plane) deformation mapping is the basic application of DIC methods. For this application, the specimen is assumed to be planar, normal to the imaging source and deforming within the original object plane [13]. Multiple imaging sources can be used to obtain the out-of-plane component of the displacement, Fig. 2.5 (a). Digital image correlation is here used to determine point correspondences between the stereo images before and after deformation. Hence, the displacement field is computed using appropriate calibration parameters, perspective transformation and triangulation [30], Fig 2.6.

Recently, Zhu et al. [38] showed that it is possible to conduct stereovision DIC (SDIC) from SEM images acquired following compucentric tilting of the sample as shown in Fig. 2.5 (c). It should be nevertheless noted that, although SDIC yields a 3D displacement field, the latter describes the deformation of the surface of the specimen and not of its volume. Therefore, the components of the deformation gradient tensor $F_{ik}$, with $i=1,2,3$ and $x_3$ being the out-of-plane direction, cannot be resolved.

![Fig. 2.5](image)

**Fig. 2.5** Equivalent stereovision systems (a) Standard Stereovision (SSV) with two identical cameras imaging at positions symmetric about the specimen normal axis; (b) Moving Camera Stereovision (MCSV), position 1 and position 2 are the positions of Camera 1 and 2 in 1(a) respectively; (b.1) and (b.2) are individual representations for a camera in position 1 and 2, respectively; (c) Tilting Specimen Stereovision (TSSV), the images of the specimen in position 1 and 2 can be decoupled to give the same perspective as shown in (b.1) and (b.2) [38]

The full $F$ can be calculated from the three-dimensional displacement field obtained by correlating images acquired using volumetric imaging techniques such as (micro)tomography [39, 40]. Volumetric digital image correlation (VDIC) is nevertheless generally limited to the study of deformation in granulated or porous materials as these naturally provide the required speckle pattern, Fig 2.6 (b) and (c).
A comprehensive review of the experimental procedures and analytical methods used in stereovision and volumetric DIC is out of the scope of the present study and can be found in the referenced textbooks [13, 30].

![Image](image1.png)

**Fig. 2.6** Examples of stereo and volumetric DIC. (a) 3D displacement field computation associated with a stamping process [30]. (b) 3D rendering of a specimen of Hostun sand showing the grain detail [40]. (c) Discrete VDIC derived grain displacements (vertical component) viewed in three orthogonal slices through the volume for strain increment of about 7%. Grains coloured grey are those for which the image correlation was not successful [40].

### 2.2 Pattern texture

As mentioned above, each subset must show a unique set of features to be identifiable. In particular, gray levels in the image needs to exhibit a broad range of values to carry sufficient information for an accurate correlation measure [41]. It is also desirable to have strong contrast between adjacent pixels to be sensitive to small displacement amplitudes [30], unlike what it is seen for the pattern in Fig. 2.7 (b). Therefore, DIC usually requires the prior application of a speckle pattern onto the
surface of the sample that could be imaged in sufficient contrast and high signal to noise ratio [30].

![Examples of patterns not suitable for DIC: (a) lamellae features, (b) gradual gray level variation, (c), (d) and (e) examples of suitable patterns [30]]

**Fig 2.7** Examples of patterns not suitable for DIC: (a) lamellae features, (b) gradual gray level variation, (c), (d) and (e) examples of suitable patterns [30]

### 2.2.1 Speckles morphology

The morphology of the speckles may also affect the accuracy of the computed displacement field. This is the case of fusiform or lamellae features, as the one depicted in Fig. 2.7 (a), which can introduce ambiguity in the correlation (aperture problem). The elongated features can be idealized as straight lines; the latter could happen to be only partially covered by the reference subset, Fig. 2.8. Even though the displacement component normal to the line can be resolved, the one along the line cannot, i.e. a point on the line in the reference image can be matched to any arbitrary point on the line in the deformed image [13]. Pattern application methods giving equiaxed-shaped speckles are thus usually preferred; examples of suitable patterns are shown in Fig. 2.7 (c), (d) and (e).
2.2.2 Assessment of pattern quality

Recently, Pan et al. [42] and Hua et al. [43] have proposed ways to measure the “quality” of a speckle pattern based on evaluation of the gray levels, pixel size and distribution of the speckles. These measures help identifying the pattern that would give the most accurate displacement field independently on the subset size and the correlation algorithm used for DIC.

Fig. 2.9 and 2.10 reports results from Hua et al. [43]. The authors have defined a parameter $S_f$ termed mean subset fluctuation (MSF) as:

$$S_f = \frac{\sum_{p \in F} S_p}{H \cdot V} \quad \text{with} \quad S_p = \sum_{i=1}^{3} \sum_{j=1}^{3} |a_{ij} - \bar{a}|$$

(2.9)

where $a_{ij}$ is the gray value at each point $p$, $\bar{a}$ is the mean gray value of the subset, $F$ is the class of the points and the term $H \cdot V$ is the size of the speckle pattern. The MSF $S_f$ gives a measure of the mean flatness of the subsets: high values are obtained in the presence of steep intensity gradients [43].

The authors calculated the MSF for four distinct speckles patterns generated by spray painting, Fig. 2.9. These where translated by a known subpixel amount and correlated with the originals to estimate the associated error in displacement measurements. As shown in Fig. 2.10, the higher the MSF value, the higher the accuracy of DIC measurements. In particular, pattern (d) showing well-defined speckles gave the best error proof performance.
Fig 2.9 Speckle patterns used for numerical experiment: (a) Speckle Pattern A and its histogram, (b) Speckle Pattern B and its histogram, (c) Speckle Pattern C and its histogram and (d) Speckle Pattern D and its histogram [43]

Fig 2.10 Curves of the mean bias errors related to the imposed displacement for four speckle patterns. Results calculated with the subset of (a) 41 × 41 pixels and (b) 71 × 71 pixels [43]
2.3 DIC using SEM imaging

Intuitively, the resolution of measurements tends to increase with the density of features as smaller subsets can be adopted. The common combination of spray-painted speckle patterns and optical imaging is generally associated with a minimum subregion size of the order of tens to hundreds of square microns and therefore can only be applied for studying displacement mapping at macroscopic length scales. As discussed further in section 2.3.2, authors have explored different pattern application methods with the aim of producing microscopic patterns and increase measurement spatial resolution. The achieved miniaturization of the speckle pattern enabled the use of the high magnification capability of SEM imaging.

Fig. 2.11 (a) depicts the main components of a SEM system [44]. The electrons emitted by the electron gun at the top of the SEM column are focused into a beam through a series of electromagnetic lenses. The electron beam hits the sample and interacts within a teardrop-shaped volume of material (interaction volume) that can extend to about 50 nm to 5 μm into the sample surface [44].

![SEM layout and function](image)

**Fig 2.11** (a) Schematic of a scanning electron microscope and imaging process (a) [44]. (b) Comparison between ideal and actual e-beam scanning process for SEM imaging [13]

Most of the electrons interact anelastically with the electron cloud of atoms and are scattered with lower energy. These are collected by a secondary electron detector where the intensity of the current is converted into a signal that is subsequently digitalized. The energy absorbed by the atoms is dissipated as thermal energy or
emitted in the form of radiation, such as X-rays. The remaining electrons are scattered with higher energy and a part of these are imaged using a backscatter electron detector.

Due to the mechanism of interaction of the incident electron beam within the interaction volume, small variations of topography of the sample surface affect the number of emitted secondary electrons [45]. Hence, the secondary electron imaging (SEI) is mainly sensitive to the topography of the specimen.

On the other hand, the amount of electrons collected at the backscatter electron detector depends primarily on the atomic number of the material as the dimension of the electron cloud influences the probability of the electrons being scattered with high energies. Backscatter electron imaging (BEI) is therefore most sensitive to material composition.

### 2.3.1 Source of error in SEM imaging

SEM imaging is accomplished by the scan generator, which pilots the deflection coils to generate a raster scan of the electron beam Fig. 2.11 (a). In the ideal scanning concept, the beam hits the sample surface at points on a regular grid as in the left image in Fig. 2.11 (b). Yet, the accuracy of the scan generator and the inevitable instability of the electron beam introduce positional errors, Fig. 2.11 (b). The consequent variation of pixel intensity ultimately affects the correlation of images acquired at different times.

The effects of the beam drift can be estimated by correlating two images of a same region acquired without inducing deformation; any non-null value can then be regarded as random error and represents the source noise in DIC measurements. Sutton et al. [45, 46] showed that decreasing working distance and increasing dwell time (reducing scan speed) both help minimizing the spatial drift while improving the signal to noise ratio. The level of noise in strain measurements nevertheless remains in the order of $10^{-3}$ making the measurement sufficiently accurate only at moderate (>2%) imposed deformation.

A second source of error is associated with the spatial distortion induced by the scan, Fig. 2.12. For given imaging parameters, spatial distortion can be regarded as a systematic error and can be therefore corrected for as shown in [45, 46]. In these studies, the authors have developed computational methods to correct for both drift and spatial distortion and improve the accuracy of DIC measurement. Levels of standard deviation for strain measurement in the order of $10^{-5}$ obtained after correction suggests that SEM imaging can be used to map strain in the elastic regime [47]. Unfortunately, such a level of reliability cannot be generally achieved in the plastic deformation regime as deformation features such as slip bands are seen to affect the local correlation measure [48, 49], section 4.2.1.
2.3.2 Pattern application methods for DIC of SEM images

As mentioned above, numerous pattern application methods have been recently developed to enable DIC of SEM images acquired at high magnification [48 - 61]. Examples of high-density speckles patterns are shown in Fig 2.13. The pattern in Fig. 2.13 (a) is obtained by attaching a microgrid onto the surface of the specimen (microgrid method). The pitch of the grid determines the spatial resolution of the displacement field. The progressive miniaturization of commercially available grids to a pitch size of few microns has favored their use in microscale strain mapping [50 - 56]. Microgrids are also commonly used in template patterning techniques where the pattern is created through the deposition of a selected element, usually gold, through a template [55, 56], Fig 2.13 (b). Alternatively, it is possible to generate a bitmap image of the desired pattern and reproduced it using lithography techniques or by electron and focus ion beam (FIB) – assisted deposition of a selected element, usually gold or platinum, Fig. 2.13 (c) and (d) [58, 59]. Nevertheless, the deposition of micro or nanoparticles of various nature appears as the class of most versatile patterning techniques [57, 60, 61], examples are shown in Fig. 2.13 (e) and (f).
2. Digital Image Correlation

Fig 2.13 (a) BE image of the 2 µm pitch gold microgrid pattern [54]. (b) BEI image of specimen surface after Au coating through a mesh [55, 56]. (c) SE image of the hafnium oxide speckle pattern created by e-beam lithography (also shown is a grid pattern) [58]. (d) BE image of FIB assisted Pt nano dots at 20% coverage [59]. (e) BE image of sputtered Pt nanoparticles [57]. (f) SE image of Au nanoparticles [61].
A detailed review and critical discussion on the aforementioned pattern application methods can be found in [62]. Table 2, which is taken from the latter study, shows a qualitative comparison of such methods. Further discussion on the distribution of speckles in a pattern is given in Chapter 7.

**Table 3.1** Summary of patterning techniques for SEM-DIC [62]

<table>
<thead>
<tr>
<th>Technique</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Focused ion beam patterning</td>
<td>Accurate control of pattern location</td>
<td>Expensive</td>
</tr>
<tr>
<td></td>
<td>Repeatable</td>
<td>Time consuming</td>
</tr>
<tr>
<td></td>
<td>Substrate independent</td>
<td>Ion beam surface damage</td>
</tr>
<tr>
<td>Template patterning</td>
<td>Fast</td>
<td>Filter must be held perfectly flat to the surface</td>
</tr>
<tr>
<td></td>
<td>Inexpensive</td>
<td>of the sample</td>
</tr>
<tr>
<td></td>
<td>Numerous samples can be patterned at once</td>
<td>Stock filter pore aspect ratio limits</td>
</tr>
<tr>
<td></td>
<td>Repeatable</td>
<td>the minimum feature size to 400 nm</td>
</tr>
<tr>
<td></td>
<td>Accurate control of pattern location</td>
<td>Low pore density in stock filters</td>
</tr>
<tr>
<td></td>
<td>Substrate independent</td>
<td>requires numerous applications</td>
</tr>
<tr>
<td>Nanoparticle patterning</td>
<td>Fast</td>
<td>Difficult to control pattern location</td>
</tr>
<tr>
<td></td>
<td>Inexpensive</td>
<td>Difficult to achieve repeatable results</td>
</tr>
<tr>
<td></td>
<td>Can be stock purchased with diameters ranging from 2 to 250 nm</td>
<td>Substrate dependent</td>
</tr>
<tr>
<td></td>
<td>Easily synthesized</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Broad distribution of speckle sizes</td>
<td></td>
</tr>
<tr>
<td>E-beam lithography</td>
<td>Accurate control of pattern location</td>
<td>Expensive</td>
</tr>
<tr>
<td></td>
<td>Repeatable</td>
<td>Time consuming</td>
</tr>
<tr>
<td></td>
<td>Easily scaled for different magnifications</td>
<td>Multi-step process</td>
</tr>
<tr>
<td></td>
<td>Suitable for high temperature</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Substrate independent</td>
<td></td>
</tr>
</tbody>
</table>

### 2.3.3 Gold remodelling

Vapor assisted remodelling of deposited gold is the pattern application method adopted in the present work to enable submicron resolution DIC measurements. The technique allows the application of nanoscale speckles in a high-density pattern following coalescence of the deposited gold particles. Remodelling on thin gold and silver films was first observed by Luo et al. [63] on glass substrates. Nanoparticles of the selected element were deposited by thermal evaporation of wires. These were observed to coalesce and form islands of the size of hundreds of nanometers when heated to relatively low temperature (60 - 120°C) and exposed to non-saturated vapor of a selected solvent.
time ranges from 5 h to 16 h

agarose-nanoclay composite prepared in our lab; (Fig. 5).

Fig. 4.

Fig. 2.14 Schematic of two patterning arrangements for metallic thin films [63]

Fig 2.15 SEM images of Au thin films patterned on different substrates at 120°C. The substrates are: (a) silicone rubber; (b) epoxy; (c) agarose-nanoclay composite; (d) Al wafer; (e) Al film coated on glass; (f) silicon wafer; (g) stainless steel wafer; (h) Cr film coated on glass; (i) Cr film (2.5 nm) coated on glass. The patterning time ranges from 5 h to 16 h [64]
The experimental set-up used by Luo is reported in Fig. 2.14. The authors further observed that the morphology of the speckles depended primarily on the exposure time and the initial film thickness. The choice of different solvents was instead seen to affect the rate of remodelling. Iodobenzene vapor resulted to be the most effective among those tested, yet water also showed the capability to induce remodelling. The observation that heating alone induced no remodeling suggested that the capillarity force of the solvent was driving the remodelling process [63].

Scrivens et al. [64] later used the same experimental settings to induce remodelling on metal substrates. The authors were also the first to propose the use of the speckle patterns for DIC studies. Examples of speckle patterns obtained in the study are reported in Fig. 2.15. In particular, the pattern obtained for stainless steel (Fig. 2.15 (g)) and aluminium (Fig. 2.15 (d)) can be considered for comparison with those obtained for such materials in Chapter 6, paper 1 and Chapter 6, paper 2.
3 Introduction to electron backscatter diffraction and dislocation state characterization

3.1 Basic principles

3.1.1 Diffraction patterns and indexing
Electron backscattered diffraction (EBSD) is based on the acquisition of diffraction patterns formed by the interference of backscatter electrons as described by Bragg’s law [65]. In order to increase the probability of having diffracted electrons, the sample is tilted to a small angle with the incident beam, usually 20°, i.e. 70° to the sample normal Fig. 3.1 (a) [66]. A phosphor screen positioned inside the SEM chamber reveals the diffraction pattern, also termed Kikuchi pattern. An example of a FCC pattern of GaAs is shown in Fig. 3.1 (b) [67]. The direction and position of the bands forming the pattern depends on the orientation of the crystallographic planes diffracting the electron beam. Therefore, diffraction patterns can be analyzed to extract the lattice orientation of the interaction volume. Each lattice orientation can be described as a rotation \( R(\theta, r) \) of an angle \( \theta \) about the axis \( r \) from a reference lattice orientation. In commercial software [68, 69], indexing of the diffraction patterns is performed using Hough transformation; further details can be found in [65, 66]. The process can be repeated automatically for all the points in a grid of a predefined pitch (step size) to produce a lattice orientation map.

![Diagram of a typical EBSD installation](image1.png)
![Diffraction pattern and indexing of a GaAs lattice](image2.png)

Fig 3.1 (a) Diagram of a typical EBSD installation [65]. (b) Diffraction pattern and indexing of a GaAs lattice [67]
3.1.2 Spatial resolution and accuracy

The dimension of the interaction volume determines the maximum spatial resolution of EBSD measurements. The former is linked to the beam size and enlarges by about three times following sample tilting. In addition, resolution is seen to depend on beam conditions and on the characteristic of the investigated material [70]. In practice, resolutions are in the order of tens of nanometers; values for certain SEM model/material combinations are reported in Table 3.1.

Table 3.1 Summary of typical EBSD performance for different metals [70]. Quantities $\Lambda_A$ and $\Lambda_P$ describe the elliptical geometry of the interaction volume

<table>
<thead>
<tr>
<th>Sample and microscope type</th>
<th>Spatial resolution (nm)</th>
<th>Angular precision (degrees)</th>
<th>Time/pattern (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\Lambda_A$</td>
<td>$\Lambda_P$</td>
<td>Beam scan</td>
</tr>
<tr>
<td>Aluminium W</td>
<td>60</td>
<td>180</td>
<td>0.2</td>
</tr>
<tr>
<td>FEG</td>
<td>20</td>
<td>60</td>
<td>0.2</td>
</tr>
<tr>
<td>Brass W</td>
<td>25</td>
<td>75</td>
<td>0.1-0.2</td>
</tr>
<tr>
<td>FEG</td>
<td>9</td>
<td>27</td>
<td>0.1-0.2</td>
</tr>
<tr>
<td>$\alpha$-iron W</td>
<td>30</td>
<td>90</td>
<td>0.1-0.2</td>
</tr>
<tr>
<td>FEG</td>
<td>10</td>
<td>30</td>
<td>0.1-0.2</td>
</tr>
</tbody>
</table>

The accuracy of lattice orientation measurements can be determined by acquiring a statistically relevant number of data points in an area where the lattice is expected to be homogeneously oriented, as in the case of a grain in an annealed material. The resulting “orientation noise” can be quantified in terms of the apparent misorientations between these data points [70]. The angular deviation $\delta$ from the average value is commonly found to be about 0.1° - 0.5° in modern EBSD systems [66].

As for the error in rotation angle $\delta$, there is error in the determination of the rotation axis $\beta$ [71, 72]. Bate [73] demonstrated that, in the case of small rotations $\omega$, the two errors can be related:

$$\beta = \tan^{-1} \frac{\delta}{\theta}.$$  \hspace{1cm} (3.1)

Relation (3.1) indicates that the error in the rotation axis $\beta$ tends to decrease for relatively high values of $\theta$. As a result of this, EBSD analysis based on the calculation of the misorientation between lattice of neighboring material points are generally less noise-affected when the imposed deformation induces steep lattice orientation gradients, i.e. strong lattice curvature. This is observed, for instance, in the deformation zone induced by indentations [74 - 76].
The advantage of having significant lattice curvature is compensated by the degradation of the Kikuchi bands associated to the dislocation content of the interaction volume. The presence of dislocations perturbs the orientation of the lattice planes so that Kikuchi patterns appear less well-defined and therefore less easy to index with accuracy, Fig. 3.2 [77]. Such deleterious effect makes sample polishing required to remove the cold work layer induced by sample machining and enable EBSD pattern acquisition [78]. In practice, this also limits the pattern quality and the indexing ratio that can be achieved for specific materials.

![Fig 3.2](a) and (b) Schematic showing the effect of a subgrain boundary on the EBSD pattern. (c) and (d) EBSD patterns from well-prepared surface and from a poorly prepared surface of zirconium [77]

### 3.1.3 DIC of diffraction patterns: elastic strain and improved accuracy

The variation of lattice spacing associated with elastic stretches manifests as changes in the width of the Kikuchi bands and, in particular, in the displacement of certain zone axes, which are formed by the intersection of such bands, Fig. 3.3 (a). Wilkinson [79 - 81] measured these shifts with respect to a (strain-free) reference pattern using DIC and provided a method to derive the elastic part of the deformation gradient tensor $^3F^e$.

Fig. 3.3 (b) shows the displacement $Q$ of a material point in the reference crystal

$^3F^e$ is the elastic part of the deformation gradient tensor $F = F^eF^p$ as defined in section 5.1.1
following an arbitrary strain and rigid body rotation of the lattice. Such displacement can be related to \( \mathbf{F}^e \) and to the position vectors of the point in the reference \( \mathbf{r} \) and deformed configuration \( \mathbf{r}' \):

\[
\mathbf{Q} = \mathbf{r}' - \mathbf{r} = (\mathbf{F}^e - \mathbf{I})\mathbf{r}
\]

(3.2)

However, EBSD can only measure the projection \( \mathbf{q} \) of \( \mathbf{Q} \) perpendicular to \( \mathbf{r} \), i.e:

\[
\mathbf{q} = \mathbf{Q} - \lambda \mathbf{r} = \mathbf{r}' - \mathbf{r} = (\mathbf{F}^e - \mathbf{I})\mathbf{r}
\]

(3.3)

where \( \lambda \) is an unknown scalar. Wilkinson showed that measuring \( \mathbf{q} \) for (at least) four widely spaced directions allows the values \( F_{ij}^e \) with \( i \neq j \) to be determined. The remaining components associated with hydrostatic strain of the lattice cannot be directly obtained since such strain component does not cause any shifts in the EBSD pattern [79, 80]. These can nevertheless be recovered by assuming that the out-of-plane stress component is zero and by knowing the elastic stiffness constants, see [79 - 81] for details. This gives full determination of the displacement gradient tensor (i.e., lattice strains and rotations) as in the example shown in Fig. 3.4, see caption for details [82].

Fig 3.3 A crystal lattice strained 11% along the horizontal direction and a schematic overlay of the patterns with (red) and without strain (black) [77]. (b) Schematic diagram showing how a strain and rotation (exaggerated) act to alter a zone axis direction \( \mathbf{r} \) shifting across the EBSD screen by \( \mathbf{q} \) [81]
The DIC of diffraction patterns allowed achieving an accuracy of $10^4$ to be obtained for elastic strain measurement and a reduction in the misorientation error (both in angle and axis) by two orders of magnitude [73, 79, 83]. In particular, the latter permitted an analogous improvement in the accuracy of Nye tensor calculation (and hence of GNDs density calculation) described below. Yet, it is noted that DIC on diffraction patterns can only be applied for similar pattern and therefore for small misorientation values [79].

Fig 3.4 Variations in the finite lattice rotation tensor ($R$) and elastic (Green’s) strain tensor ($\varepsilon$) measured using HR-EBSD in deformed titanium. A slip band location is illustrated with a black dashed line, which terminates at the grain boundary. (Colour scale for the lattice rotation matrix is $\pm 5 \times 10^2$ for the off diagonal terms and $1 \pm 2.5 \times 10^3$ for the leading diagonal. Color scale for the elastic strain tensor is in absolute strain measured. All maps are plotted with respect to the reference point for each grain. [82]

3.2 Lattice curvature and geometrically necessary dislocations

3.2.1 Nye tensor

In his pioneering work, Nye [84] demonstrated that the sum of the Burgers vector $\mathbf{b}$ of all the dislocations comprised in a volume of material is geometrically linked to the change in lattice orientation (lattice curvature)$^4$. Such dislocations are generally referred to as geometrically necessary dislocations (GNDs) [85]. The theory provides a continuum description of dislocations and operates at bigger scales than the scale

$^4$ Nye tacitly neglected the contribution to lattice curvature of the lattice elastic strain
of discrete dislocations individuated in Fig. 1.1, i.e. \( O > 10^{8} \text{m} \). Hence, common terms used to describe the nature of individual dislocations, such as edge and screw dislocation types, lose their meaning.

For an orthonormal reference system \((x_1, x_2, x_3)\), lattice curvature can be described by a second order tensor \( K \) with component \( k_{ij} \) \((i, j = 1,2,3)\) given by the lattice rotation \( \Delta \theta_i \) about the \( i \) axis following a displacement \( x_j \) along the \( j \) direction:

\[
\Delta \theta = K x
\]

The components of \( K \) and \( b \) can be related by geometric transformations operated by the Nye tensor \( G \), also referred as geometric dislocation density tensor:

\[
b = \int_{\Omega_i} G^T n d\Omega, \quad G = K^T - \delta K \tag{3.5a, 3.5b}
\]

Where \( \Omega_i \) are the surface of unit normal \( n_i \) confining the volume and \( \delta \) the Kroneker delta \([84]\).

The net Burgers vector \( b \) can be calculated by combining the system of equations in (3.4) and (3.5a, 3.5b) for a cube volume element. Considering the cube of dimension \( d \) and an orthonormal reference system as depicted in Fig. 3.4, it yields:

\[
b_1 = (-\Delta \theta_{22} - \Delta \theta_{33} + \Delta \theta_{21} + \Delta \theta_{31})d \tag{3.6a}
b_2 = (\Delta \theta_{12} - \Delta \theta_{33} - \Delta \theta_{11} + \Delta \theta_{32})d \tag{3.6b}
b_3 = (\Delta \theta_{13} + \Delta \theta_{23} - \Delta \theta_{11} - \Delta \theta_{22})d \tag{3.6c}
\]

In the EBSD mapping process, \( d \) would coincide with spatial resolution of the map, which is set as the step size.

As EBSD gathers lattice orientation at the surface of the sample, it is not possible to observe the variation of lattice orientation along the normal direction \( x_3 \) and therefore the components \( \Delta \theta_{13} \) cannot be calculated. Hence, only partial components of the net Burgers vector \( b_{i}^{\text{par}} \) can be derived:

\[
b_1^{\text{par}} = (-\Delta \theta_{22} + \Delta \theta_{21} + \Delta \theta_{31})d \tag{3.7a}
b_2^{\text{par}} = (\Delta \theta_{12} - \Delta \theta_{11} + \Delta \theta_{32})d \tag{3.7b}
b_3^{\text{par}} = (-\Delta \theta_{11} - \Delta \theta_{22})d \tag{3.7c}
\]
Fig 3.5 Representation of the link between lattice curvature components $\Delta \theta_{12}$ and $\Delta \theta_{11}$ and Burgers vectors of dislocations piercing a volume of material

The geometrical interpretation of equations (3.7) for $d = 1$ can be illustrated as follows. Let the Burgers vector of a dislocation to be generally oriented within the plane $\Pi_1$. The presence of this dislocation will cause a lattice curvature along the axes $x_2$ ($\theta_{12}$) and $x_3$ ($\theta_{13}$). As discussed above, unlike $\theta_{12}$, $\theta_{13}$ is not accessible by EBSD. Generally, Burgers vectors of dislocations piercing $\Pi_3$ will also have a component along $x_2$. The component would induce a lattice rotation $\theta_{32}$, i.e. about the axis $x_3$ along $x_2$. An additional contribution to $b_2$ will be given by dislocations accommodating the screw rotation (torsion) of the lattice $\theta_{11}$ along $x_1$ as shown in Fig. 3.5 (b). In particular, following the right hand convention, the Burgers vector will have opposite direction with respect to those obtained for $\theta_{12}$ and $\theta_{32}$, i.e. $-\theta_{11}$. Finally, these contributions can be summed to obtain the part of $b_2$ accessible by EBSD, i.e. $b_2^{par}$. An equivalent construction for an edge dislocation piercing the planes $\Pi_2$ and $\Pi_3$ would yield $b_2^{par}$.

Additionally, Fig. 3.5 (b) shows that the dislocations accommodating $\theta_{11}$ must also have components of the Burgers vector along $x_3$. Similarly, this must happen for the dislocations accommodating the component of lattice rotation $\theta_{22}$. The components of lattice curvature $\theta_{11}$ and $\theta_{22}$ are the only ones accessible by EBSD to describe $b_3$. These will therefore sum to give $b_3^{par}$ [84].
3.2.2 Lattice curvature components from EBSD data

In section 3.1.3 it was showed that is possible to perform DIC on diffraction patterns to obtain $\mathbf{F}^e$. Lattice curvature components can be then directly calculated as spatial variation of the components of the rotation tensor ($\mathbf{R}$ in Fig. 3.4). In the present work, lattice curvature components are calculated from the lattice orientation (EBSD) data gathered after indexing of the diffraction patterns.

Several ways to perform operation with rotation are available. In the present study, we use quaternion representation of rotations. An excellent treatment of the algebra of rotations using quaternion representation can be found in [86] as only expressions of interest are derived below.

Within the algebra of rotation, a quaternion can be conveniently expressed as the sum of a scalar $q_0 = a$ and an ordinary 3D vector $\mathbf{q} = q(a, b, c)$, that is $q = q_0 + \mathbf{q}$. The conjugate of a quaternion $q^*$ is therefore given by the quaternion with the sign of its vector part changed, $q^* = q_0 - \mathbf{q}$.

Using such representation, the definition of quaternion product $r = pq$ and quaternion dot product $p \cdot q$ take the following forms:

$$r_0 = p_0q_0 - \mathbf{p} \cdot \mathbf{q} \quad \text{(3.8)}$$
$$\mathbf{r} = p_0\mathbf{q} + p_0\mathbf{q} + \mathbf{p} \times \mathbf{q} \quad \text{(3.9)}$$

and:

$$p \cdot q = p_0q_0 + \mathbf{p} \cdot \mathbf{q} \quad \text{(3.10)}$$

In analogy with vector algebra, the magnitude of a quaternion is given by: $|q| = \sqrt{q \cdot q}$. It can be shown [86, 87] that the quaternion represents a rotation by an angle $\theta$ about the unit vector $\mathbf{n}$, if choosing $q$ as:

$$q = q(\theta, \mathbf{n}) = \cos \frac{\theta}{2} + \sin \frac{\theta}{2} \mathbf{n} \quad \text{(3.11)}$$

It is to note that $q$ is a unit quaternion, i.e. $|q| = 1$. It is also to observe that information on the axis of rotation is uniquely included in the vectorial part of the quaternion. As a result, the inverse rotation can be easily computed by changing the sign of $\mathbf{n}$, i.e. computed as the quaternion conjugate $q^*$. 

49
Performing operations of rotation using quaternions has to be preferred to the use of SO(3) matrixes\(^5\). The main advantages are related to the parameterization of quaternions, which allows treating rotations as vectors in a 4-dimensional space. For instance, build up of computational error during calculations of lattice misorientation will cause the matrix to lose their orthogonal property whilst quaternion to become of non-unit length [87]. This can be fixed by dividing the quaternion by its module \(|q|\) whereas fixing a non-orthogonal matrix demands more complex operations [87]. Moreover, the vectorial nature of the quaternion makes it straightforward to calculate the mean orientation within a set of lattice orientations, as for the calculation of grain average orientation [88] or to obtain random orientations for the use, for instance, in Monte Carlo simulation [87]. Finally, it allows intuitive geometric constructions as the one described by Bate et al. in [73] to derive the relation between the errors in determining the angle and axis of misorientation.

Performing a rotation \(q_1\) followed by a second rotation \(q_2\) is equivalent to a single rotation \(p\) obtained as the quaternion product \(q_1q_2\) defined as:

\[
p = q_1q_2 = \left(\begin{array}{c} q_1q_2^2 + q_1q_2 \\
-q_2q_1 + q_1q_2 - q_1 \times q_2 \end{array}\right)
\]

(3.12)

The conjugate \(q^* = (q_0, -\vec{q})\) of the unit quaternion \(q\) represents the inverse rotation. It is therefore possible to describe the difference between two rotations, i.e. the misorientation, in (sample) reference system as [88]:

\[
m_s = q_1^*q_2
\]

(3.13)

The expressions for the misorientation angle and axis derive from the definition of quaternion in (4):

\[
\theta = 2 \cos^{-1}(m_s^0) \quad \quad \quad \quad r = \frac{m_s}{\sin(\frac{\theta}{2})}
\]

(3.14a-3.14b)

The components of lattice rotation along the principal axes can be finally obtained from the component along \(x_3\) of the disorientation vector \(\theta_l = \theta r_l\), that is [89]:

\(^5\)Note: moving from matrix to quaternions it is important to notice that a rotation \(R^1\) followed by a rotation \(R^2\), which is given as \(R^2R^1\), is given as \(q^*q^2\) in quaternion algebra [88]
Fig. 3.6 (a) and (b) are taken from Kysvar et al. [74]. These show results of rotation decomposition from lattice orientation data gathered using EBSD for a copper specimen indented using a knife-shaped tip. It is evident how the lattice curvature is almost exclusively in plane.

\[ \theta_i = m_i \frac{2 \cos^{-1}(m_i^2)}{\sqrt{1-(m_i^2)^2}} \]  

(3.15)

3.2.3 Estimating the density of geometrically necessary dislocations

Since the early treatment on GNDs calculation by Arsenlis and Parks [90], numerous authors have been trying to characterize the actual GNDs content from EBSD measurements as in [74 - 76, 82, 91].

The dislocation density tensor \( \mathbf{G} \) can be in fact expressed in term of dislocation density \( \rho_{\alpha ND} \) on individual slip systems \( \alpha \) as:

\[ G_{ij} = \sum_{\alpha=1}^{N} \rho_{\alpha ND} b_{\alpha} s_{\alpha}^i t_{\alpha}^j \]  

(3.16)
where \( b^\alpha \) is the magnitude of the Burgers vector of the \( \alpha \)-th crystallographic slip system, \( s_i^\alpha \) is the unit vector along the slip direction, and \( t_f^\alpha \) is its unit tangent vector that indicates the line sense of the dislocation line [74, 90].

Expression (3.16) can be substitute into (3.5 (b)) to derive \( \rho_{\text{GNND}}^\alpha \) from lattice curvature values. Solving the system of equations implies the knowledge of all the possible dislocation types and the associated triplets \( (b^\alpha, s_i^\alpha, t_f^\alpha) \). The number of possible dislocation types would normally exceed the number of the nine Nye tensor components. The problem would thus remain undetermined as multiple solutions for the density of each dislocation type can be found.

The number of possible dislocation density types can be reduced assuming that slip occurs along principal planes and directions only. Yet, for instance, 12 distinct direction for edge dislocations and 6 for screw dislocation are possible in FCC crystals, Table 3.2, so that still an excess of 9 dislocation types exists.

**Table 3.2** The dislocation basis used to describe the dislocation state in FCC crystals [90]

<table>
<thead>
<tr>
<th>Density</th>
<th>( t )</th>
<th>( s )</th>
<th>( n )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \rho_1 )</td>
<td>( \hat{[112]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_2 )</td>
<td>( \hat{[121]} )</td>
<td>( \hat{[101]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_3 )</td>
<td>( \hat{[112]} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_4 )</td>
<td>( \hat{[121]} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_5 )</td>
<td>( \hat{[112]} )</td>
<td>( \hat{[101]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_6 )</td>
<td>( \hat{[121]} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_7 )</td>
<td>( \hat{[112]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_8 )</td>
<td>( \hat{[121]} )</td>
<td>( \hat{[101]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_9 )</td>
<td>( \hat{[121]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{10} )</td>
<td>( \hat{[100]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{11} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{12} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{13} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{14} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{15} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{16} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{17} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
<tr>
<td>( \rho_{18} )</td>
<td>( \hat{[011]} )</td>
<td>( \hat{[110]} )</td>
<td>( \hat{[111]} )</td>
</tr>
</tbody>
</table>

Optimization is usually performed by iterating the calculation over sets of nine dislocation types and then taking the set that yields the minimum amount of dislocation density as solution, Fig. 3.6 (c) and (d). The choice is justified by assuming the tendency of the lattice to store the minimum possible energy. Yet again, an approximated value for stored energy cannot be determined as the arrangement and dislocation line length of the dislocation remains unknown.
In the present study, plotting of the Burgers vector field is preferred over plotting of GNDs density. In particular, this is because the author believes that it is important to conserve the vectorial nature of $\mathbf{b}$, which would be otherwise lost in the calculation of the scalar value $\rho_{GND}^s$. 
4 Combining DIC and EBSD measurements

As discussed above, HDIC methods enable the mapping of in-plane deformation at the microstructural scale. On the other hand, EBSD allows the mapping of the residual lattice curvature and elastic strain. Therefore, comparing DIC and EBSD measurements appears as a powerful tool to study the kinetic of crystal plasticity and validate crystal plasticity models. This will be discussed in more details in Chapter 5.

In order to perform EBSD after straining, it is generally required to remove the applied speckle pattern and polish the surface to restore a microscopically flat surface as this improves the indexing of diffraction patterns. For a close one to one comparison of values, the resolution of the DIC and EBSD maps need to be similar. This means that DIC should be generally performed by choosing a subset size that matches the step size of the acquired EBSD map. Also, prior knowledge of the relationship between the reference systems used to describe the respective quantities is necessary as discussed next.

4.1 Reference system and superimposition

In both commercial DIC [36, 37] and EBSD software [68, 69], the spatial coordinates used to describe the position of points in the map coincide with the one used to index the components of a matrix. These are the spatial coordinates $X_{\text{spatial}}$ (columns) and $Y_{\text{spatial}}$ (rows) in Fig. 4.1. This particular choice resembles the row/column indexing of values in a matrix. For the DIC software, the spatial reference system is also the one used for describing the components of the displacement field. In contrast, Dasher et al. [92] were able to show that both EBSD software HKL and TLS have distinct reference frames to describe lattice orientations. These are positioned as shown in Fig. 4.1.

Once the relationship between the respective reference frames has been established, it is necessary to describe both DIC and EBSD values in the same configuration. For EBSD maps acquired after deformation, the latter will be the deformed configuration, which maps the material points from the deformed to the reference, undeformed configuration.

Fiducial markers such as indentations in the proximity of the investigated area can be used to facilitate the superimposition of DIC and EBSD values. The accuracy of the superimposition is yet hampered by the amplifying effect of sample tilting on the spatial distortion of SEM imaging described in section 3.3.1 [38].
4.2 Review of high resolution DIC and EBSD studies

A list of previous investigations dedicated to the study of microscale plastic deformation in metals is given in Table 4.1, which has been added at the end of the present section. Values are taken from the table reported in Chapter 6, paper 1. Yet, Table 4.1 includes the recent works of Carroll et al. [60] and Krammers and Dasy [61], which followed the publication of Chapter 6, paper 1.

The studies reviewed below have been selected because each adopts a distinct type of pattern application method. Only results and discussion related to DIC and EBSD measurements are reported.

4.2.1 Tatschl and Koledinik (2003)

Tatschl and Koledinik [48, 49] performed microscale strain mapping of polycrystalline copper deformed in tension to 7.4% and 14% macroscopic strain. The speckle pattern was applied by etching and imaged using secondary electrons, Fig. 4.2. A sub-region size of approximately $1.26 \times 1.26 \mu m^2$ can be calculated from the values of spatial resolution and interrogation window size reported by the authors.

Images acquired at 0% and 7.4% deformation are shown in Fig. 4.2 (a) and (b) respectively. The formation of slip bands is evident, markedly in grains 1, 8 and 11.
Fig 4.2 Scanning electron micrograph of (a) an undeformed and (b) a 7.4% deformed OFHC copper specimen. Horizontal loading direction [48]

Fig 4.3 Cumulative strain maps for the deformation step from 0 to 7.4% macroscopic tensile strain. Plotted are the axial $\varepsilon_{xx}$, transverse $\varepsilon_{yy}$ and shear strains $\varepsilon_{xy}$ as well as the planar rotations $r_{xy}$ (loading is along the horizontal x direction) [48]

The authors commented that, in correspondence of slip bands, the algorithm failed to give a sufficiently good correlation measure. Consequently, although strain mapping was performed using microscale subregions of dimension comparable with the
bands spacing, it was not possible to capture the intense shear localization expected in correspondence of slip bands, Fig. 4.3.

EBSD measurements were used to overlap grain boundaries onto the strain maps. Hence, two linescans were performed across the grain boundaries given by grains 1 and 2 and by grains 3 and 4. Values of lattice rotation angle were calculated with respect to the initial orientation of the respective grain. These were compared with the strain values along the same profiles as shown in Fig. 4.4. Some degree of correlation was observed between the value $\varepsilon_{xx}$ (loading direction) and $\varepsilon_{xy}$ and the misorientation angle, in particular at the grain boundary between grain 3 and 4.

**Fig 4.4** (a) The local lattice rotation angle for the deformation step from 0 to 7.4% macroscopic tensile strain measured along two profiles L1 and L2. The vertical lines indicate the position of the grain boundary. (b) Distribution of the axial $\varepsilon_{xx}$, transverse $\varepsilon_{yy}$ and shear strains $\varepsilon_{xy}$ along the same profiles [48]

### 4.2.2 Heripre et al. (2007)

Héripré et al. [54] investigated the plastic strain evolution of a zirconium alloy deformed in tension to 2.5% macroscopic deformation. A 2 µm pitch gold grid was imprinted onto the sample surface as to create the required pattern, which is shown in Fig. 2.13 (a).
Before applying the grid, a lattice orientation (EBSD) map was performed on the investigated area to allow the identification of the grain structure. Results of strain mapping showed transgranular deformation features in the form of bands at about 45° with respect to the horizontal loading direction.

Hence, strain values were superimposed to the Schmid factor map as shown in Fig. 4.5. The authors qualitatively observed that strain localization occurred preferentially at grain boundaries in particular in correspondence of those between high Schmid factor grains.

**Fig 4.5** (a) Microstructure determined by EBSD analysis – Schmid factors (white = high value, black = low value). (b) Axial strain field $\varepsilon_{xx}$ (loading along the horizontal x direction). (c) Superposition of both experimental results [54]

Also, strain values were compared with FEM simulations using elements obtained by meshing the grain boundary map. Fig. 4.6 shows a comparison between the strain along the tensile direction and two FEM calculations performed with different boundary conditions. In the first calculation, the displacements measured by DIC
were applied at each node at the margin of the mesh. In the second calculation, homogeneous strain boundary conditions were instead applied at each node of the mesh.

Bands of higher deformation oriented at 45° from tensile direction were observed in both simulations, yet the simulated strain appeared less localized than the actual one.

4.2.3 Tschopp et al. (2010)

Tschopp et al. [57] made similar observations on deformed Ni-based superalloy. The pattern was here applied by deposition of nanoscale platinum particles and imaged using SEI; Fig. 2.13 (e) shows a detail of the obtained speckles pattern. Although the particles are of submicron dimension, the relatively low density of speckles limited the use of subsets to 7 × 7 µm². Similarly to the study of Héripré et al. [54], the maximum shear strain map reveals deformation bands at 45° with respect to the horizontal loading direction, Fig. 4.7 (a). Superimposition of the grain boundary map obtained from an EBSD scan on the same region revealed as well that most strain was localized in correspondence of grain boundaries, Fig. 4.7 (b).

![Fig 4.7](image)

(a) Maximum shear strain map of a Ni-based superalloy sample deformed in tension along the horizontal direction to 2%. (b) Maximum shear strain map with the grain boundaries overlaid [57]

Furthermore, the authors investigated the correlation between the Schmid factor and maximum shear strain, Fig. 4.8. They concluded that there is no decisive relationship between the two quantities in such alloy. The average maximum shear strain also showed a net increasing trend in maximum shear strain with increasing Schmid factor, although this trend was also considered to be minimal.
4. Combining DIC and EBSD

Fig 4.8 (a) Schmid factor map of the region of interest and (b) maximum shear strain vs Schmid factor at a nominal stress of 1280 MPa [57]

4.2.4 Walley et al. (2011)

Walley et al. [58] used DIC to study creep in a Ni-superalloy specimen deformed in tension to about 700°C. Hafnium oxide speckles were applied on the sample surface using lithography methods, Fig. 2.13 (c). The speckle pattern was reported to have good thermal stability and to provide excellent contrast for image acquisition using SE imaging at elevated temperature. The full-field displacement field was measured using subsets of $9 \times 9 \, \mu m^2$ with 95% subsets overlap.

As it was seen in the studies above, regions of localized high values of $\epsilon_{xx}$ and $\epsilon_{xy}$ were observed to correlate with grain boundaries, which were identified using EBSD, Fig 4.9. In particular, such regions were often seen in correspondence of annealing twin boundaries where cracks were found to develop during straining as shown in Fig. 4.10.

As observed in Tcshopp et al. [57] for the same material, there was not a clear correlation between Schmid factors and plastic strain localization, Fig. 4.10 (d) and (e).
Fig 4.9 Fig. 7 (a) inverse pole figure map (b) tensile strain in the \( x \)-direction (c) shear strain and (d) tensile strain in the \( y \)-direction [58]

Fig 4.10 (a) visible cracks and slip steps on the sample surface correlate with (b) high angle and twin boundaries. (c) principle and (d) shear strain maps. Schmid Factors for \( \{111\} \) slip systems in (e) \(<110>\) or (f) \(<112>\) slip directions [58]
4.2.5 Kammers and Dasy (2013)

Kammers and Dasy [61] recently developed a pattern application technique based on the rearrangement of deposited Au nano particles and used it to study the deformation of an aluminum specimen reinforced with iron intermetallic particles. The specimen was strained in tension to 9% elongation; Fig. 2.13 (f) shows the investigated area.

Combination of DIC and EBSD data revealed that strain was particularly localized in proximity of the iron intermetallic particle on the bottom right of map where values of axial strain reached 20 %, Fig. 4.11.

Fig 4.11 Calculated axial strain field evolution for a tensile test on 1100Al. Iron intermetallic particle at the bottom right of the map
Microbands were also evident in the strain map. Their crystallographic nature was confirmed by plotting \{111\} plane traces obtained from EBSD data as shown in Fig. 4.12.

Fig 4.12 (a) Inverse pole figure map of the investigated area. (b) Strain map and overimposed grain boundaries and \{111\} plane traces (black lines)
Table 4.1 A list of studies dedicated to large deformation mapping in metals at reduced length scales

<table>
<thead>
<tr>
<th>Pattern application method</th>
<th>Material</th>
<th>Imaging technique</th>
<th>Subset dimension / Grid dimension</th>
<th>Deformation bands observed in strain mapping</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microgrid methods</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ni based LIGA</td>
<td>SEM (BE)</td>
<td>21x21 µm²</td>
<td>Not expected (crack tip study)</td>
<td>[56]</td>
<td></td>
</tr>
<tr>
<td>Ni based LIGA</td>
<td>SEM (BE)</td>
<td>10x10 µm²</td>
<td>Not expected (crack tip study)</td>
<td>[55]</td>
<td></td>
</tr>
<tr>
<td>Zr and Ti</td>
<td>Optical/SEM (SE)</td>
<td>2x2 µm²</td>
<td>Yes</td>
<td>[54]</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>SEM (BE)</td>
<td>3x3 µm²</td>
<td>-</td>
<td>[53]</td>
<td></td>
</tr>
<tr>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>2.5x2.5 µm²</td>
<td>Yes</td>
<td>[52]</td>
<td></td>
</tr>
<tr>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>5x5 µm²</td>
<td>Yes</td>
<td>[51]</td>
<td></td>
</tr>
<tr>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>5x5 µm²</td>
<td>Yes</td>
<td>[50]</td>
<td></td>
</tr>
<tr>
<td>Alternative methods</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Electron beam lithography</td>
<td>Ni based Superalloy</td>
<td>SEM (SE)</td>
<td>9x9 µm² (95% overlap)</td>
<td>-</td>
<td>[58]</td>
</tr>
<tr>
<td>of hafnium oxide plus microgrid imprinting</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Platinum nanoparticle</td>
<td>Ni based Superalloy</td>
<td>SEM (SE)</td>
<td>7x7 µm² (95% overlap)</td>
<td>Yes</td>
<td>[57]</td>
</tr>
<tr>
<td>deposition</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Etching</td>
<td>Cu</td>
<td>SEM (SE)</td>
<td>≥1.26x1.26 µm²</td>
<td>-</td>
<td>[48]</td>
</tr>
<tr>
<td>Etching</td>
<td>Cu</td>
<td>SEM (SE)</td>
<td>≥1.26x1.26 µm²</td>
<td>-</td>
<td>[49]</td>
</tr>
<tr>
<td>Silicon powder</td>
<td>Ni based Superalloy</td>
<td>Optical</td>
<td>7x7 µm² (85% overlap)</td>
<td>Yes</td>
<td>[60]</td>
</tr>
<tr>
<td>Rearranging of gold</td>
<td>Al</td>
<td>SEM (SE)</td>
<td>0.9x0.9 µm² (95% overlap)</td>
<td>Yes</td>
<td>[61]</td>
</tr>
<tr>
<td>nanoparticles</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gold remodelling</td>
<td>Austenitic Steel</td>
<td>SEM (BE)</td>
<td>0.25x0.25 µm² (0% overlap)</td>
<td>Yes</td>
<td>Paper 1</td>
</tr>
</tbody>
</table>
5 Comments on the approach to mesoscale crystal plasticity

5.1 Crystal plasticity at the mesoscale

The schematization of different length scales of investigation in Fig. 1.1 can be reproduced to include deformation bands as observed in Héripré et al. [54] and Tschopp et al. [57], Fig 5.1. In particular, the central windows should be modified considering that the scale of polycrystal plasticity (O $10^{-5}$ m) individuated by McDowell in Fig. 1.1 is too big to resolve the presence of microbands of submicron spacing. Microbands are thus plotted in the window of dislocation patterns (O $10^{-7}$ m).

Notably, the two scales are characterized by a transition from crystallographic to non-crystallographic (macroscopic) deformation features. Therefore, these constitute what can be considered a mesoscale, which bridges atomistic and macroscopic scales. Accordingly, it is possible to develop a mesoscopic description of crystal plasticity.

**Fig 5.1** Comparison between the length scales of investigation of distinct microscopy techniques

Mesoscopic plasticity is greatly affected by the initial microstructure morphology, e.g. grain geometry, and heterogeneity, e.g. lattice orientation and multiple phases. It could be initially thought possible to reproduce microstructural features by extending atomistic and discrete dislocation dynamic simulation to the mesoscale.
This is nevertheless prohibitive due to the computational demand of simulating the motion of single atoms within microscale volumes [93]. As a result, the application of discrete simulation methods has been necessarily restricted to the study of defect nucleation and interaction [6 - 8]. It is also noted that any attempt to account for the discrete nature of plastic deformation is generally associated with a rise in degrees of freedom of the system, as shown in Fig. 1.1. This translates to an increased number of parameters and material properties that need to be measured experimentally, which contrasts with the challenge of developing accurate and yet simple physical based models.

A more realistic approach is implementing continuum crystal plasticity models into finite element analysis (CPFEM) or fast Fourier transformation-based (CPFFT) analysis [94]. Voronoi polyhedral construction [95] might be implemented to mimic grain geometry of most single-phase materials of interest. Alternatively, the actual 3D microstructure can be reconstructed using Contrast-enhanced SEM images or grain boundary EBSD plot following sectioning, Fig. 5.2 [96, 97]. Recently, non-destructive volumetric techniques such as diffraction contrast tomography (DCT) have been successfully used to obtain a realistic reproduction of the 3D grain structure [98, 99]. A review of the plethora of constitutive law and implementation of models is out of the scope of the present work. Further details can be found in [6, 100, 101].

![Fig 5.2](image)

Fig 5.2 (a) Phase distributions in an idealized multiphase titanium microstructure. (b) Plot of total slip system activity for normalized by the average rate, so that the average becomes white on the indicated color map. Blue indicates zero slip activity, and values greater than or equal to twice the average are plotted in red [97]
5.1.1 Elastoplastic model of crystal deformation

Crystal plasticity models are based on the kinematic representation of elastoplastic deformation of single crystal proposed by Asaro and Rice [102], which is represented in Fig. 5.3. Following Lee’s multiplicative decomposition of the deformation gradient \( \mathbf{F} = \mathbf{F}^e \mathbf{F}^p \) in its elastic \( \mathbf{F}^e \) and plastic part \( \mathbf{F}^p \) [103], \( \mathbf{F}^p \) maps the material point \( X \) in the initial configuration \( \mathcal{B}_0 \) to an intermediate (imaginary) stress-free configuration \( \mathcal{B}_l \), which is achieved by crystallographic slip only. The slip is assumed to leave the lattice vectors \( (s, m) \) unchanged and unrotated as in the isoclinic representation first proposed by Mandel [104].

\[
\mathbf{F}^e = \mathbf{F}^p \mathbf{F}^{-1} = \sum_{\alpha=1}^{N} \dot{\gamma}^\alpha m_{ij} \otimes s_{ij}
\]  

\( N \) is the number of possible active systems and \( \dot{\gamma}^\alpha \) is the slip system shearing rate for the \( \alpha \)-system.

Phenomenological or dislocation-based constitutive laws have been developed to describe, implicitly in the former and explicitly in the latter, the effect of dislocation population by linking hardening rules to \( \dot{\gamma}^\alpha \) (and so \( \mathbf{F}^p \)). Further details and examples of constitutive equations can be found in dedicated textbooks [101, 105].

Local crystal plasticity models proved capable of predicting the evolution of grain orientation distribution (texture) and the effect of the latter on yield strength and
work hardening. Such models nevertheless lack material length scales and therefore fail to predict important aspect of mesoscopic plasticity such as size effects or phenomena of microscale non-uniform plastic deformation showing characteristic wavelength [85]. Size effects include the well-documented Hall-Petch effect and the influence of particle dimensions on hardening in particle reinforced materials. Microscale non-uniform plastic deformation comprises the formation of dislocation cells and subgrains and the localization of slip in bands of distinct spacing. In particular, the latter is receiving increasing interest in the study of damage mechanics. For instance, the localization of slip and its interaction with the grain boundary is believed to play an important role on crack initiation and propagation in stress corrosion cracking (SCC) studies\(^6\) [106].

5.1.2 Strain gradient plasticity

Since the early discussion of Ashby [85], it became evident that it was possible to incorporate a material length scale into constitutive laws through functional of strain field. The physical foundation laid on the early treatment of deformation incompatibility pioneered by Nye [84], Bilby [107], Kondo [108] and Kroner [109].

Unlike the deformation gradient \( F \), which is the gradient of a vector field, i.e. \( \text{Curl} F = 0 \), \( F^P \) and \( F^e \) act as local deformation gradients and are generally incompatible, i.e. \( \text{Curl} F^P \neq 0 \) and \( \text{Curl} F^e \neq 0 \). Volume elements in the intermediate configuration can therefore detach or overlap. Cermelli and Gurtin [110] demonstrated that both the terms \( \text{Curl} F^P \) and \( \text{curl} F^{e-1} \), with \(( \text{Curl} F^P )_{ij} = e_{irs} \frac{\partial F^P_{js}}{\partial x_r} \) and \(( \text{curl} F^{e-1} )_{ij} = e_{irs} \frac{\partial F^{e-1}_{js}}{\partial x_r} \) are equivalent measures of the incompatibility of plastic deformation and that these quantities can be related to the dislocation state of the material in the form:

\[
G = (J^P / F^P) \text{Curl} F^P = J^e F^{e-1} \text{curl} F^{e-1}
\]

(5.2)

Where \( J^P = \det F^P \) and \( J^e = \det F^e \). The terms \((J^P / F^P)\) and \( J^e F^{e-1} \) refer to the mapping of the area \( \Pi \) between different configurations [110].

Combining expressions (3.5b) and (5.2), retrieves the relationship between deformation and lattice curvature. In summary, dislocations are conceived as lattice

\(^6\) The present study has been funded by Serco TCS (UK) as part of a research project aimed to understand the effect of cold work on the SCC susceptibility of on austenitic stainless steel
defects necessary to conserve the integrity of the crystal lattice between adjacent volumes of material deforming in a non-compatible fashion.

The characterization of the GND content varies depending on the length scale at which the deformation is characterized and therefore on the resolution of measurements.

Two adjacent volumes of material, A and B, can be in fact found to experience equivalent strain incompatibility of opposite nature, which gives \( \mathbf{b}_A = -\mathbf{b}_B \). As the necessary dislocation state of the volume \( A \cup B \) is also defined as the sum of the single Burgers vectors of the dislocations comprised in the volume, the necessary dislocation content will be null:

\[
\mathbf{b}_A + \mathbf{b}_B = \mathbf{b}_{AUB} = 0
\]  

(5.3)

Following \( \mathbf{b}_{AUB} = 0 \), the lattice curvature will also be zero.

This shows that there is a part of the dislocation density that cannot be described by simple geometrical arguments, as seen for GNDs [85, 90]. Nevertheless, it is reasonable to consider the density of such dislocations to be statistically linked to the extent of crystallographic slip, i.e. related to \( \mathbf{F}^p \). It is in fact plausible to assume that, the more a volume undergoes plastic deformation, the more its subvolumes are likely to experience incompatible deformation and therefore to store dislocations (GNDs) at smaller scales. Due to the their statistical nature at the scale of observation, such dislocations are commonly addressed as statistically stored dislocations (SSDs). The term coincides with the classic ideas of dislocations trapped in the lattice during plastic deformation. In brief:

\[
\rho_{SSDs} \leftrightarrow \mathbf{F}^p \quad \rho_{GNDs} \leftrightarrow \text{Curl} \mathbf{F}^p \leftrightarrow \text{curl} \mathbf{F}^{p-1}
\]  

(5.4)

Since the early success of Fleck on prediction of size effect on torsion of copper microwires [111], second order theories incorporating materials length scales have been developed and used in various applications, as in [111 - 116]. These theories link strain hardening with the evolution and interaction of SSDs and GNDs densities. Material length scales are in general associated microstructural features such as the grain size or the distance between particles in particle-reinforced materials [85, 111]. It is nevertheless noted that a multitude of distinct material length scales have been proposed and adopted for calculations over the years. Their definition and physical interpretation is in fact still subject to discussion and criticism; detailed discussion can be found in [117 - 119].
5.2 Comments on the experimental validation of crystal plasticity models

As a well-established and accessible technique, EBSD has been largely used to validate mesoscopic predictions of crystal plasticity models; examples can be found in [74, 120 - 123]. From the description of the EBSD technique given in Chapter 3, it is nevertheless evident that EBSD is only capable of measuring the elastic part of the deformation gradient tensor, i.e. lattice rotation and stretches. In other words, EBSD is insensitive to the part of plastic deformation that leaves the lattice unchanged. By definition, the latter is described by the plastic part of the deformation gradient tensor $F^p$.

As expressed by relations in (5.4), $F^p$ has a dual primary role in crystal plasticity. First, it can be associated to the evolution of SSDs and secondly, it describes the intermediate configuration where the deformation incompatibility originates. EBSD can only address the resultant of such incompatibility, which is manifest as lattice curvature. This same concept is expressed mathematically in (5.4). An infinite number of intermediate configurations $F^p$ can in fact be imagined that would yield the same value for $\text{Curl} F^p$ ($\leftrightarrow \text{curl} F^{e-1}$).

Fig 5.4 Kinematic model of elastoplastic deformation of a single crystal emphasizing the lack of experimental methods available to measure $F^p$

So far, $F^p$ has been accessible only through computational methods [101, 105]. Being an imaginary intermediate state, there are no direct microscopy techniques capable of isolating the contribution of crystallographic slip. An attractive indirect way of measuring $F^p$ is by applying $F^{e-1}$ calculated using EBSD to $F$ calculated using DIC, Fig. 5.4. Alternatively, it can be thought possible to develop a method for the decomposition of $F$ calculated using DIC measurements into $F^p$ and $F^e$. This would
be a significant achievement, as it would make DIC the most comprehensive technique for studying the kinematic of lattice deformation.

The elastic part $F^e$ measured by EBSD can be used to validate the results of the decomposition. In order to do so, it is first necessary to improve the resolution of DIC methods, which is in general orders of magnitude lower than the resolution of EBSD measurements, Fig. 1.1.

### 5.3 Aims and tested materials

Following the above discussion, the aim of the present work was to:

- develop a novel method for deformation mapping with submicron resolution (HDIC) and concurrently investigate the evolution of plastic deformation at the mesoscale in the tested material (Chapter 6, paper 1).
- compare HDIC and EBSD measurements at the same resolution to demonstrate the distinct abilities of these technique in characterizing plastic deformation (Chapter 6, paper 2).
- develop a method to separate lattice rotations from the deformation mapping obtained using HDIC. Validate the results by comparing HDIC derived lattice rotations to the actual values obtained by EBSD (Chapter 6, paper 3).
- use HDIC mapping and the proposed method to study mesoscale plasticity in well-characterized conditions of incompatible deformation. This is to prove the methodology capable alone of providing a comprehensive description of mesoscale crystal plasticity (Chapter 6, paper 4).

The materials chosen for testing were **304L austenitic stainless steel** and **silicon particle-containing aluminium**. From a micromechanics point of view, austenitic stainless steel is an interesting material to study because its low staking fault energy leads to strain localization at different length scales and also micro-twinning [124, 125]. This makes it an ideal material to study using sub-micron deformation mapping. The material is also known to produce high quality Kikuchi patterns after colloidal silica (OPS) polishing, which is a necessary prerequisite to enable efficient comparison of HDIC and EBSD measurements.

Silicon particle-containing aluminium is a versatile material for studying the deformation behavior of particle-reinforced materials [126].

Moreover, studying particle deformation zone (PDZ) is of particular interest as the soft-matrix/hard particle system represents an elementary setting for the development of incompatible plastic deformation. Unlike what is seen in the study of
deformation incompatibility at grain boundaries, information on the orientation relationship between the matrix and particle lattice is in fact not required as the latter is generally considered to be non-deformable. Moreover, the PDZ presents typical dislocations and lattice rotation patterns, which have been widely characterized by both experimental methods such as EBSD and TEM and computational simulations [126 - 128]. It therefore represents an ideal setting for testing the proposed HDIC analysis method.
6 Papers Section

THIS SPACE IS INTENTIONALLY LEFT BLANK
Paper 1
Published in the International Journal of Experimental Mechanics
Submitted: 3rd January 2012. Accepted: 25th September 2012

Comments: few typos and grammar mistakes spotted in the published version have been corrected. The term “randomly distributed” has been changed to “homogeneously distributed”, see discussion in Chapter 7, section 7.1.1.

Contributions of the author: the author of the present study has developed the high temperature remodelling technique described in the document. He also conducted the experiments and analysis that have been presented. The study was completed under the supervision of Dr. João Quinta da Fonseca
Plastic strain mapping with sub-micron resolution using digital image correlation

F. Di Gioacchino, J. Q. da Fonseca

Materials Science Centre, The University of Manchester, Manchester, UK

Corresponding author:
João Quinta da Fonseca
Materials Science Centre, The University of Manchester, Grosvenor Street, Manchester M1 7HS, UK
Joao.fonseca@manchester.ac.uk
Tel: 0044 306 4844
Fax: 0044 306 3586

Abstract

Digital image correlation (DIC) of images obtained using scanning electron microscopy has been used to study, quantitatively, the plastic deformation of stainless steel at the microstructural scale. An artificial speckle pattern was generated by the remodelling of a deposited gold layer. A new experimental setup was shown to accelerate the remodelling process and promote the formation of fine nano-scale speckles with sizes ranging 30 nm to 150 nm and of similar spacing. The effects of surface preparation on speckle morphology are discussed. The high density of speckles enabled displacement mapping with resolution of one displacement vector each $0.2 \times 0.2 \ \mu m^2$ of surface area. It is shown that sub-micron resolution is necessary to capture the plastic deformation associated with the formation of slip bands in stainless steel, which are an important component of the deformation of these materials at the microscale. Electron backscatter diffraction (EBSD) was used to reconstruct the surface grain boundaries and enabled these deformation features to be linked to the microstructure.

Keywords Strain localization, shear bands, slip bands, gold remodelling, austenitic steel.
Introduction

Understanding the evolution of deformation at microscale is an important part of the study of the deformation of polycrystalline materials. At this scale, microstructural heterogeneity in the form of grain boundaries, second phase particles and defect structures gives rise to non-uniform deformation which, by the generation of stress concentrations [1] or otherwise, can lead to damage such as (micro)void formation or (micro)cracking [2, 3].

One capable method for studying deformation at this scale is digital image correlation (DIC) of high magnification images acquired using a scanning electron microscope (SEM), [4, 5, 7, 9] and [10 - 20]. DIC is a computational technique which maps the displacement on the surface of the strained sample from images acquired at different stages of deformation. To achieve this, images are divided into subsets of a selected pixel size and shape, often referred to as interrogation window, which are individually cross-correlated to give a measurement of the local displacement. Performing this calculation for all subsets gives a displacement vector field that can be differentiated to give the components of the in-plane strain tensor [21, 22]. Subsets can be overlapped during the DIC analysis so that the overlapped areas are included in multiple correlations. This improves the uniformity of the displacement field and therefore reduces the noise in strain mapping [23]. Overlap also increases the number of calculated vectors per unit area, which nevertheless does not represent an increase on the actual resolution of the measurement, which is primarily determined by the dimensions of the area interrogation window, hereafter referred as sub-region. Each sub-region must show a unique set of features to be identifiable at different deformation stages. Therefore, DIC usually requires the prior application of a pattern onto the surface of the sample. The spatial resolution of measurements increases with the density of features. The smaller the features the smaller sub-region that can be used. This has led researchers to explore different methods for pattern application with the aim of producing microscopic patterns and increase measurement resolution. A list of previous investigations dedicated to the study of microscale plastic deformation in metals is given in Table 1. These are subdivided into three groups according to the pattern application methodology used, namely: fine paint decoration [6 - 12], microgrid methods [13 - 16] and alternative methods [17 - 22].
which dislocation structures such as cell walls and microbands (slip bands) form [24 - 26].

Table 4 A list of experimental parameters and observations from previous investigations dedicated to large deformation mapping in metals at reduced length scales

<table>
<thead>
<tr>
<th>Pattern application method</th>
<th>Material</th>
<th>Imaging technique</th>
<th>Subset dimension / Grid dimension</th>
<th>Deformation bands observed in strain mapping</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine paint/ ink decoration</td>
<td>Ti</td>
<td>Optical</td>
<td>6×6 μm²</td>
<td>Yes</td>
<td>[9]</td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>SEM (SE)</td>
<td>Not reported</td>
<td>-</td>
<td>[8]</td>
</tr>
<tr>
<td></td>
<td>Al-Mg</td>
<td>SEM (SE)</td>
<td>70×70 μm²</td>
<td>-</td>
<td>[7]</td>
</tr>
<tr>
<td></td>
<td>Al-Mg</td>
<td>Optical</td>
<td>268×268 μm²</td>
<td>-</td>
<td>[6]</td>
</tr>
<tr>
<td>Microgrid methods</td>
<td>Ni based LIGA</td>
<td>SEM (BE)</td>
<td>21×21 μm²</td>
<td>Not expected (crack tip study)</td>
<td>[16]</td>
</tr>
<tr>
<td></td>
<td>Ni based LIGA</td>
<td>SEM (BE)</td>
<td>10×10 μm²</td>
<td>Not expected (crack tip study)</td>
<td>[15]</td>
</tr>
<tr>
<td></td>
<td>Zr and Ti</td>
<td>Optical/ SEM (SE)</td>
<td>2×2 μm²</td>
<td>Yes</td>
<td>[14]</td>
</tr>
<tr>
<td></td>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>2.5×2.5 μm²</td>
<td>Yes</td>
<td>[13]</td>
</tr>
<tr>
<td></td>
<td>Cu</td>
<td>SEM (BE)</td>
<td>3×3 μm²</td>
<td>-</td>
<td>[12]</td>
</tr>
<tr>
<td></td>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>5×5 μm²</td>
<td>Yes</td>
<td>[11]</td>
</tr>
<tr>
<td></td>
<td>IF-Steel</td>
<td>SEM (SE)</td>
<td>5×5 μm²</td>
<td>Yes</td>
<td>[10]</td>
</tr>
<tr>
<td>Alternative methods</td>
<td>Ni based Superalloy</td>
<td>SEM (SE)</td>
<td>9×9 μm² (95% overlap)</td>
<td>-</td>
<td>[20]</td>
</tr>
<tr>
<td></td>
<td>Ni based Superalloy</td>
<td>SEM (SE)</td>
<td>7×7 μm² (95% overlap)</td>
<td>Yes</td>
<td>[19]</td>
</tr>
<tr>
<td></td>
<td>Cu</td>
<td>SEM (SE)</td>
<td>≥1.26×1.26 μm²</td>
<td>-</td>
<td>[18]</td>
</tr>
<tr>
<td></td>
<td>Cu</td>
<td>SEM (SE)</td>
<td>≥1.26×1.26 μm²</td>
<td>-</td>
<td>[17]</td>
</tr>
</tbody>
</table>
A promising pattern application method for producing the desired high-density speckles pattern is vapour-assisted remodelling of deposited gold films. The method can be used to produce nano-sized speckles, homogeneously distributed over the whole surface of the substrate and in a reproducible fashion. This has significant advantages over more elaborate and time-consuming techniques, such as lithography or ion-beam assisted deposition, where application of the pattern is necessarily limited to microscale pre-selected areas [20, 27]. Because of the relatively high atomic number, gold speckles appear brighter than most substrates of interest giving the desired high contrast in backscatter electron imaging (BEI). BEI also has the advantage of being less sensitive to the topographic features of the sample surface than secondary electron imaging (SEI) [15]. This is particularly important for strain measurements in metals at high magnifications as microscopic out-of-plane slip gives raise to sharp topographic staircase features (slip bands) that affect local pixel intensity in SEI creating, de facto, new patterns.

Developed by Luo [28], the remodelling method was first proposed by Scrivens [29] as a viable pattern application technique for digital image correlation studies and subsequently used by Sutton for the assessment of SEM image distortions and to measure averaged strains in aluminium using an imaging window of 25.6 × 22.1 µm² [5]. Yet, to the best of our knowledge, no DIC maps of plastic strain in metals using remodelling of deposited films as the pattern application method have been published.

Here, we propose alternative experimental settings for remodelling and describe how sample preparation can be controlled to obtain a variety of speckles patterns suitable for the DIC analysis with sub-regions ranging between approx. 100 × 100 nm² to 300 × 300 nm².

We then use the method to apply a speckle pattern on the surface of a 304L stainless steel specimen for tensile testing. This has enabled, for the first time, strain mapping with sub-micron spatial resolution. This is an exciting development, for it allows us to study the deformation fields at the scale of slip bands and other dislocation structures. By measuring strain at this scale, we can start bridging continuum mechanics with dislocation dynamics.

**Material**

304L stainless steel is used extensively in the power generation sector due to its excellent corrosion resistance and high strength. From a micromechanics point of view, it is an interesting material to study because its low staking fault energy leads to strain localization at different length scales and also micro-twinning. This makes it
an ideal material to study using sub-micron deformation mapping. The chemical composition of the 304L stainless steel studied is given in Table 2.

Table 2 Chemical composition of the 304L stainless steel in weight %

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>0.021</td>
<td>0.34</td>
<td>1.96</td>
<td>18.15</td>
<td>9.17</td>
<td>0.031</td>
<td>0.027</td>
</tr>
</tbody>
</table>

Experimental methods

Optimization of sample preparation and gold remodelling

Fig. 1 shows the experimental apparatus used to induce remodelling of the deposited gold layer and create the gold speckle patterns reported here. The remodelling set up relies on a common heat source for sample heating and vapour generation, which prevents condensation on the sample surface and the consequent clustering of undesired large gold islands. The hot plate allows uniform substrate temperatures of 300°C, whereas the large surface can be employed to accommodate several specimens for remodelling. Vapour generated at the central source flows to the bottom gap between the cover and the hot plate mantling the coated samples. Remodelling was investigated for 150°C to 300°C and exposures to water vapour for 60 to 90 minutes. Remodelling was observed only at temperatures exceeding 240°C. The patterns shown in this study were obtained after 1 hour exposure to 280°C with an average vapour flow of about 100ml/h.

Fig 1 Apparatus used for the remodelling of the deposited gold layer
Once favourable conditions of temperature and vapour flow are reached for remodelling, the morphology of the pattern depends on gold film thickness, as observed by Scrivens [29], but also on the roughness of the sample surface. Therefore, by varying these parameters, we can form a range of different patterns with distinct particles size and spacing. In order to separate the effects of sample preparation and gold film thickness on particle shape and spacing, a batch of square-shaped stainless steel samples were polished to different extents: one to 0.25 µm diamond finish, one to 0.05 µm alumina particle finish and another finished with 0.05 µm colloidal silica solution. The samples were then placed in an Edwards S150B sputter coater, where they were half covered with a piece of paper to avoid deposition of gold in the selected area and then coated with 50 nm gold film. After coating, the paper was moved 90 degrees with respect to its original position and another 30 nm gold film was deposited. This made possible to obtain three of the four quarters of the sample covered with 30, 50, and 80 nm films. These were distinguishable as areas of increasing opacity. Backscatter electron images of the patterns were acquired using a Sirion FEI scanning electron microscope (SEM) equipped with a Schottky Field Emission Gun (FEG). Imaging conditions were the same as those adopted during the tensile test, which are reported in Table 3. In addition, magnifications of 20000× and 50000× were used of to better characterize the morphology of the speckles. Assessment of the DIC systematic error associated to the pattern quality as described in [30] was not carried out. A single method was used to ensure the error level acceptable for the deformation experiments.

Uniaxial straining of 304 stainless steel: image acquisition procedure and correlation

A flat dog bone shaped specimen of 5 mm gauge length and 2 mm thickness was cut from the material in the solution annealed condition (1h at 1050°C and water quenched). The sample was polished with 10min OPS diluted to 1:10 volumes (pH≈8) and then gold coated with 50 nm gold film. This was later placed in the apparatus shown in Fig. 1 in order to induce remodelling of the gold film and create a speckles pattern of the type shown in Fig. 2 (g). Following the application of the gold speckle pattern, a series of micro-indents were created on the surface of the specimen to act as fiducial marks and allow quick identification of the area of interest and alignment of the sample following deformation.

Images of the region of interest were acquired using a Sirion FEI scanning electron microscope (SEM) equipped with a Schottky Field Emission Gun (FEG). Images were acquired at a magnification of 1000×, which corresponds to a field of view of about 100 × 100 µm² and a spatial resolution of 36 nm per pixel. For the reasons given
earlier, backscatter electron imaging was chosen, with high beam voltage and short working distance to enhance the backscatter signal. Contrast was kept high whilst brightness was gradually decreased until gold aggregates appear on a black background. This SEM system allows acquisition of extra high-definition images (XHD mode) with dimensions ranging from $2756 \times 1936$ to $4134 \times 2904$ pixels and with a linescan time between 40 to 240 msec. Image resolution of $2756 \times 1936$ pixels and linescan of 40 msec were used in this work; this limited single image acquisitions to approximately 80 seconds. The values used are summarized in Table 3.

The relatively low scan rate and a small working distance also work to minimize the spatial drift associated with positional errors in the raster scan of the electron beam [4]. These shifts in the beam position, usually of the order of fractions of a pixel, are mainly related to the operating conditions of the scan generator and depend on the SEM used. The effects of the drift can be estimated by correlating two images of a same region acquired without inducing deformation; any measured strain can then be regarded as systematic error and represent a good indicator of the noise in the strain measurements.

### Table 3 SEM scan parameters used for image acquisition

<table>
<thead>
<tr>
<th>Imaging mode</th>
<th>ACC. Voltage (kV)</th>
<th>Beam Current (nA)</th>
<th>Spot size</th>
<th>Aperture</th>
</tr>
</thead>
<tbody>
<tr>
<td>BEI</td>
<td>22</td>
<td>1.1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Working distance (mm)</td>
<td>Linescan time (msec)</td>
<td>Magnification</td>
<td>Image size (pixels)</td>
<td>Spatial resolution (nm/pixel)</td>
</tr>
<tr>
<td>5</td>
<td>40</td>
<td>1000×</td>
<td>2756 × 1936</td>
<td>36</td>
</tr>
</tbody>
</table>

Assessment of the systematic error was performed using different interrogation window sizes in order to identify the optimal sub-region size that would give the highest measurement resolution and acceptable noise.

The specimen was strained in uniaxial tension using a Zeiss-Kammrath micro-tester, at constant strain rate of $4\times 10^3$ s$^{-1}$ to three different elongation levels: 1.5%, 5% and 7%. At each stage, the sample was removed from the microtester and mounted on the SEM stage for image acquisition. Therefore, the strain maps were obtained in the unloaded state. The region was imaged keeping loading along the horizontal (x) direction of the imaging window.
In order to cover a larger area and include several grains in the measurement, a 300 × 300 µm² area was covered by acquisition of adjacent regions in a 3 × 3 mosaic. The images were slightly overlapped to enable seamless stitching into a larger map. Each set of images was then correlated with the undeformed set. All the DIC analysis and plotting was carried out using the commercially available software DaVis [31]. DIC produces maps of full-field in-plane displacement, \( u = u(x, y) \). However, when studying deformation at this scale, it is the distributions of strain that are of interest. The measurements allow the determination of the in-plane strain tensor, which cannot be easily visualized. Here, we chose to present the results as maps of maximum shear strain as defined by equation (1) below.

\[
\varepsilon_{xy(\text{max})} = \sqrt{\left(\frac{\varepsilon_{xx} - \varepsilon_{yy}}{2}\right)^2 + \varepsilon_{xy}^2}
\]

where \( \varepsilon_{xx} = \frac{\partial u_x}{\partial x} \) is the strain along the loading direction, \( \varepsilon_{yy} = \frac{\partial u_y}{\partial y} \) strain along normal in-plane direction and \( \varepsilon_{xy} = \left(\frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x}\right)/2 \) is in-plane shear.

Deformation in metals at room temperature takes place almost exclusively by the shearing of the lattice. Therefore maximum shear is a good scalar representation of local deformation at the sub-micron scale.

EBSD data acquisition and analysis

After image acquisition, the gold speckles were removed with few seconds of 0.25 µm diamond paste polishing for lattice orientation measurements using EBSD. In order to improve the indexing of lattice orientation, the sample surface was polished further using 0.05 µm colloidal silica solution for 20 minutes. This removed about 4 µm of material from the surface.

A lattice orientation map was then generated for the region of interest with a step size of 0.4 µm using HKL Channel 5 acquisition software [32]. Numerical Python [33] was used to plot grain boundaries and \{111\} planes traces. These were finally overlapped on top of the strain map calculated from the displacement mapping of the deformed configuration onto the undeformed one, i.e. obtained by inverting the order of correlation between images. This makes it possible to identify the position of deformation features in the deformed configuration allowing a more rigorous comparison with the microstructural features from the EBSD data.
Results: gold remodelling

Surface finish: diamond and alumina polishing

For the sample polished to 0.25 µm diamond finish, remodelling was observed only for 50 nm and 80 nm layers. The speckles produced have the required sub-micron size but they tend to form interconnected structures as can be seen in Fig. 2 (a) and (b).

Fig 2 Speckles patterns formed after 1 hour vapour exposure at 280°C using apparatus in Fig. 1(a) following distinct surface finish and gold film deposition. 0.25 micron diamond finish and 50 nm and 80 nm film thickness (a), (b). 0.05 micron aluminia finish for 80 nm film thickness (c). 10 minutes of 0.05 micron colloidal silica polishing with a 1:30 volumes diluted solution (pH≈7) for 30 nm 50 nm and 80 nm film, (d), (e), (f). 10 minutes of 0.05 microns colloidal silica polishing with a 1:10 diluted solution (pH≈8) for 50 nm and 80 nm film (g), (h). 20 minutes 0.05 microns colloidal silica polishing with a 1:10 diluted solution (pH≈8) for 80 nm film (i). Patterns most suitable for plastic strain mapping with sub-micron resolution are highlighted in red
With a 0.05 μm alumina particle finish, the pattern at 50 nm shows distinct but poorly defined speckles, while 80 nm shows irregularly agglomerated speckles, as shown in Fig. 2 (c). The pattern achieved using diamond polishing can be compared to that obtained previously on stainless steel (Fig. 5 (g) in Scrivens et al [29]). Speckles in Fig. 2 (a) and (b) appear significantly smaller in diameter; yet, these are less defined and difficult to image in sufficient contrast.

Surface finish: colloidal silica polishing

Colloidal silica solution (OPS) is prepared from an alkali-silicate solution which is partially neutralized. As a result, the solution adds a minor caustic effect to the polishing from the colloidal silica particles, which is beneficial for sample preparation for electron diffraction backscatter (EBSD) measurements [34]. The extent of corrosion depends primarily on the pH, which is controlled by the concentration of the solution, and on the polishing time. Both of these influence the morphology of the remodeled pattern. To investigate the effect of pH on the gold remodelling, the OPS solution used was diluted to different concentrations. Fig. 2 (d), (e) and (f) show the remodelling of 30 nm, 50 nm and 80 nm thick gold layer deposited after 10 minutes polishing using a 1:30 dilution. Whereas the original OPS solution had a pH of about 9 as measured using a pH paper indicator, the diluted solution showed a pH close to neutral. For the 30 nm thick layer, the backscatter signal is weak with speckles only distinguishable at high magnifications (50000×). Speckles dimension increases consistently with increasing deposition, as does the quality of the backscatter signal. In particular, the pattern obtained for the 80 nm thick layer appears suitable for DIC analysis with sub-regions of about 150×150 nm². For the imaging conditions and image acquisition resolution discussed later, this corresponds to magnifications ranging within 3000× and 5000×.

Polishing with a more concentrated solution of 1:10 volumes (pH=8) for 10 minutes increased the speckle spacing. This was balanced by the formation of bigger clusters as shown for 50 nm and 80 nm in Fig. 2 (g) and (h) respectively. These patterns allow a decrease in the magnification at which the images are acquired for DIC analysis (1000× - 3000×) while preserving sub-micron resolution with sub-regions of about 300 × 300 nm². Longer polishing times (>20 min), as with a more aggressive solution, have deleterious effects. The speckle spacing appears unaffected but the size of the particles diminishes significantly, Fig. 2 (i). This results in a low fraction of area covered by the speckles, which makes the pattern unsuitable for high resolution DIC. These observations have been summarized in a diagram shown in Fig. 3, which can be used as a reference for sample preparation of stainless steel substrates.
Fig 3 Diagram showing the change in pattern suitability for DIC as a function of gold film thickness and polishing time for an OPS solution diluted to reach a pH between 7 and 8.

Many materials of interest form thick oxide films at the temperature of gold remodelling. Oxidation can hinder the gold remodelling process. This has been observed in duplex steel and zirconium alloys. The effect on remodelling can be reduced by pre-oxidation of the sample at the temperature of remodelling prior to gold deposition. The passivation inhibits further oxide growth during remodelling, allowing gold particles to coalesce. The drawback of pre-oxidation is that a thick oxide film can introduce artifacts to the surface strain measurements. For such materials, gold remodelling should thus be performed in an inert environment in order to prevent oxidation.

**Results: strain mapping**

Assessment of background noise

For each measurement, two consecutive images were acquired at each deformation step. The cross-correlation of this pair of images was then used to provide an estimate of the systematic background error in strain measurements associated with the raster scanning process and detector noise.

Fig. 4 shows values of maximum shear strain of the central region of interest calculated, using $6 \times 6$ and $4 \times 4$ interrogation windows, which correspond to sub-regions sizes of $216 \times 216$ nm$^2$ and $144 \times 144$ nm$^2$ respectively. The dark regions at the
centre of the maps surrounded by high values of strain correspond to pre-existing microstructural features free from gold speckles. In both maps values appear higher at the left-down and right-up corners indicating the occurrence of more intense beam drift in these regions. Similarly, the background error is higher for the $4 \times 4$ window than for the $6 \times 6$ window.

**Fig 4** Values of maximum shear strain induced by the spatial distortion associated with instabilities of the raster scanning process using $6 \times 6$ (a) and $4 \times 4$ (b) interrogation window sizes.

**Fig 5** Average values of maximum shear strain and scatter induced by the spatial distortion associated with instabilities of the raster scanning process for different interrogation window sizes.

This trend is confirmed in Fig. 5 where average values of noise and the associated scatter are reported for different interrogation window sizes. Both quantities appear
to increase exponentially for decreasing windows sizes. This indicates that the drift effect tends to average out within increasing pixel areas. Following this analysis, a 6 × 6 sub-region size was chosen for DIC analysis as this yielded negligible level of noise with strain values within one to two orders of magnitude lower than those observed for deformation features. For this reason, no correction for the drift distortion as proposed in [5, 35] has been performed.

Sub-microscale resolution and microband detection

A small region of a speckle pattern image as exported from DaVis software at 0% and 5% macroscopic strain is shown in Fig. 6 (a) and (b). By looking at the movement of the features highlighted in red, it is possible to visibly detect significant compression along the direction transverse to the direction of the loading. Two immediate observations can be made: gold speckles stick to the surface and are easily distinguishable after plastic deformation and the intensity of the speckles and of the dark background is stable, i.e. no new image features appear during deformation. Correlation of the two images using an interrogation window of 6 × 6 square pixels corresponding to a sub-region dimension of 216 × 216 nm² gives the displacement vector depicted in Fig. 6 (c). About 100 displacement vectors are therefore available to describe the deformation of single 2 × 2 µm² areas. The displacement field indicates that the top-left region has moved down tangent to arrows depicted in blue. This gives the sharp shear band that appears in the calculated strain map, Fig. 6 (d). In addition, three bands with the same orientation but lower intensity also appear. These bands are regularly spaced with a pitch of about 1 µm. It is clear that sub-microscale resolution is needed to capture these features. In particular, as the sub-region dimension should be at least half the band spacing, a minimum sub-region size of 0.5 × 0.5 µm² is necessary.

Microscale vs. sub-microscale resolution

Before discussing results on strain measurements at the highest resolutions, it is instructive to calculate values of the area of interest at a lower resolution, simulating the use of alternative methods for pattern application such as grid imprinting and lithography used until now.
Fig 6 Detail of the speckle pattern at 0% (a) and 5% (b) macroscopic strain. Displacement vector field after correlation with sub-micron resolution (c) and maximum shear strain mapping showing regular spaced bands to about 1 µm (d).

Fig. 7 (a) shows values of maximum shear strain obtained for 7% strain with a sub-region size of 9 × 9 µm², and 50% overlap. Strain localization can be seen in the form of bands at about ±45° to the loading direction, i.e. along the directions of maximum resolved shear stress. The intersection of these bands forms a grid pattern with spacing of about 100 µm, which is about the same as the measured grain size. Results of strain for 1.5%, 5% and 7% obtained using sub-regions of 216 × 216 nm² are shown in Fig. 8 (a) and (b) and Fig. 7 (b) respectively.

As it can be seen, deformation in this material is highly localized, in the shape of clusters of shear bands. At 1.5% strain, these bands appear sharply defined with each group showing a distinct direction and spacing. Some intersect each other, as in region I in Fig. 8 (a). A small number of bands are particularly intense, with values exceeding 0.1. At 5%, the bands become better defined and are more intense, with values of strain exceeding 0.2. Bands can change direction abruptly, as in region C of Fig. 8 (b) or meet other cluster of bands. In the latter case, there are high levels of strain accumulation in the regions where the bands meet, forming hot spots of strain localization, labeled H. It is these that give rise to the periodic shear bands identified at lower magnifications. At 7% macroscopic strain, the values increase but strain appears localized in the same areas observed in the 5% map.
Fig 7 Values of maximum shear strain calculated for 7% macroscopic elongation using different sub-region sizes: $9 \times 9 \, \mu m^2$ with 50% overlap (a) $216 \times 216 \, nm^2$ with 0% overlap (b)

Fig 8 Values of maximum shear strain calculated for 1.5% macroscopic elongation (a) and 5% macroscopic elongation (b). Intersecting bands “I”, abrupt change in band direction “C”, hot spot of intense strain localization “H”

These changes can be better observed by looking at a smaller region, as shown in Fig. 9. A few intense bands can be seen in the 1.5% strain map, Fig. 9 (a). Yet, many of the bands are faint and only a few are clearly visible. At 5% strain, these bands darken as they approach the intensity of adjacent more prominent bands, Fig. 9 (b). This not observed in the 7% strain map, Fig. 9 (c), where the maximum intensity of bands is preserved whilst the overall shear strain increases. In Fig. 10, the average values of maximum shear strain for the region at 7% strain are plotted as a function of the strain component along the loading direction. The plots show that, as expected, shear strain increases linearly with applied axial strain. Also plotted are the maximum strain values calculated for the regions of intense localized deformation labeled H in
Fig 8 (b). As can be seen, the strain at these “hot spots” is also proportional to the strain along the loading direction, but can be twice as large as the mean shear strain of neighbouring regions.

**Fig 9** Detail of the strain maps of the region of interest at 1.5%, 5% and 7% macroscopic strain in grey scale adjusted to highlight the distribution of bands

**Fig 10** Maximum shear strain as a function of the strain along the loading direction for the studied region. Max. shear strain for the whole region, blue dots. Max. shear strain for the “hot spot” areas indicated in Fig. 8 (b), remaining markers

Relating local deformation to the microstructure

EBSD was used to determine the location of the grain boundaries and the orientation of the grains in the region of interest. The grain boundaries were then overlaid onto the 7% strain map in Fig. 11, revealing that the shear strain within a grain is made up of a smoothly varying “background” shear strain and distinct straight bands of highly localised shear. Each grain appears to contain a maximum of two distinct sets
of shear bands, each with characteristic alignment and spacing. Since slip is known to occur on the (111) planes, the alignment of these bands should coincide with the traces of the octahedral planes. The EBSD data was used to calculate the slip traces for 41 individual grains; the two traces for the planes with the highest Schmid factors are marked in red and the other (111) traces are marked with thin black lines. When overlaid onto the map, it can be seen that the traces more closely aligned with the bands are those corresponding to the systems with the highest Schmid factor. This is only untrue for three highlighted grains where the bands are instead oriented along other (111) traces. In many cases, the bands are not perfectly aligned with the slip plane traces. The misalignment can be as large as 20°.

![Image]

**Fig 11** Values of maximum shear strain calculated for 7% macroscopic elongation in the deformed configuration. Grain boundaries and (111) planes traces obtained from lattice orientation data analysis acquired following deformation are superimposed. Traces of planes with the two highest Schmid factors are highlighted in red. Red dots indicate the grains where only partial correspondence is found between high Schmid factor traces and bands directions.

Our results also show that the shear on the slip bands sometimes decreases as the bands approach grain boundaries. This is a consequence of the constraint imposed by the neighbouring grain. Highly localized deformation within one grain must give
rise to strain incompatibility at the grain boundary. Our maps show that, in these cases, the grains deform more homogeneously near grain boundaries, which leads to significant rotations of grain boundary regions as can be seen in Fig. 12. Values of maximum shear strain and in-plane rotation angle are compared for a grain boundary region at the top-right corner of the investigated region, showing clearly the narrowing of the bands as they approach the grain boundary and how the grains rotate on opposite directions to accommodate the misfit.

**Discussion**

**Gold remodelling**

Vapour-assisted remodelling at temperatures of about 300 °C proved advantageous with respect to remodelling at the lower temperature ranges previously investigated. Higher temperatures appear to accelerate the gold particle mobility and concurrently lead to the formation of smaller and yet well-defined speckle patterns. Moreover, it has been observed that sample preparation plays an important role on determining the characteristic of the pattern and its suitability for digital image correlation studies. For the stainless steel investigated in this work, only polishing using colloidal silica solution with pH close to neutral has been observed to produce the desired speckle pattern. The use of more aggressive solutions or extensive polishing time appears to inhibit remodelling. This can be explained by the higher corrosion rate with increased pH, which promotes the formation of nanoscale pits that affects the remodelling process.

**Gold pattern imaging**

As shown in Fig. 6, the applied gold speckles stick to the surface and conserve their shape during the plastic deformation of the substrate. This is essential to achieve high correlation measures and therefore reliable strain measurements in plastic regimes. Results also show that electron backscatter imaging is preferable for DIC mapping of plastic strain in metals with sub-micron resolution, as it is not significantly affected by slip bands and other topographic features generated during plastic deformation.
Using the gold remodelling technique we have been able, for the first time, to measure the plastic strain development at the sub-micron scale. Our analysis showed that, in 304 stainless steel, deformation is highly heterogeneous at the microstructural scale. The strain maps revealed that the spatial variation in strain can be divided into three different components, each with a characteristic wavelength: slip bands within grains, smooth variation across individual grains, and transgranular shear bands. The presence of transgranular slip bands and of smooth strain gradients within grains had been reported previously. Now, with gold remodelling, we can measure the strains generated by slip bands within grains.

Firstly, our experiments show that the slip bands are not exactly aligned with slip plane traces. Although some of this misalignment could be attributed to experimental error, the fact that it is quite large in some cases and that it varies from grain to grain suggests that the misalignment is real. Of course this does not mean that slip is not occurring in the expected {111} planes. Rather, the misalignment is a consequence of the fact that these bands are the mesoscale manifestation of slip and therefore, although they are ultimately composed of units of slip on {111} planes, they can happen on different {111} planes and can be co-planar or not. Therefore, although it is not surprising that the bands are closely aligned with slip plane traces, their actual alignment is affected by the stress state experienced by the individual grains, which can be significantly different from the far field applied stress.
Secondly, although units of slip happen preferentially on {111} planes with highest Schmid factor, the amount of strain in each slip band does not depend on its value, as it would be otherwise expected. Instead, the shear intensity varies with position in each grain, indicating not only that the stress state is different from the applied stress state but also that it varies with position within the grains. The origin of these intragranular fluctuations is the interaction between neighboring grains and is therefore non-local. It can be seen that these fluctuations combine to give the larger scale, transgranular shear bands that are particularly visible with the lower magnification analysis shown in Fig. 7 (a). It is in these transgranular bands that hot spots of intense strain localization appear, corresponding to specific grain boundaries and triple points. It appears therefore, that although the crystallographic nature of slip leads to strain localization within grains in the form of shear bands, the magnitude of this shear is not determined by crystallographic orientation alone. Rather, it depends on how the deformation is accommodated by the different neighbouring grains. In uniaxial tension, this leads to a quasi-periodic variation of transgranular shear bands at approximately 45° to the extension direction. This suggests that the different levels of strain heterogeneity are not completely independent. The larger wavelength transgranular bands are made up of regions within grains with the highest intragranular strains and the highest intensity of shear bands. This implies that the location of a strain “hot spot”, where the local strain is a maximum value, is not determined uniquely by the local crystallography and geometry but also by how the periodic transgranular strain heterogeneity develops. For example, although it would be tempting to state that regions marked H in Fig. 8 (b) are special because of their unique combination of orientation and even grain boundary properties, it is clear that they lie at the intersection of two transgranular slip bands, which seems to modulate the strain within slip bands in the grains and give rise to a strain maximum.

Conclusions

We have shown that it is possible to modify existing experimental settings for gold film remodelling to produce nanoscale aggregates of gold particles of sensibly smaller size and spacing those previously achieved and which can be used for DIC strain mapping with sub-micron resolution. The success of the gold remodelling depends primarily of the surface finish achieved prior to gold deposition. For the stainless steel studied here, a final polish with diluted OPS (pH=8) and sputtered film thickness of 50 nm gave speckles of 50 nm to 250 nm and a speckle density of
about 50 speckles/µm². This enabled DIC analysis using sub-regions of 216 × 216 nm² and a resolution of 21 vectors per square micron. We have also learnt that controlling imaging conditions is essential for reliable measurements of plastic strain at high magnifications.

Our experiments on stainless steel showed that sub-micron resolution is needed to capture the hierarchical nature of the deformation at the microstructural scale. In particular, we were able to measure the strain associated with slip bands within grains. The evolution of local strain distribution at 1.5%, 5% and 7% macroscopic strain suggests that the number of slip bands tends to saturate early on, after which the strain within each band increases at a higher rate than the average applied strain. EBSD orientation data revealed that the orientation of the intergranular slip bands is not strictly crystallographic. Instead, it seems to be affected by the constraint imposed by neighbouring grains and the slip planes can be misaligned by as much as 20° from the nearest slip plane trace. Furthermore, we have shown that the deformation within these intragranular bands appears to be modulated by longer range, transgranular deformation bands. This leads to the development of strain “hot spots”, where the local shear strain is a maximum. We have also shown that, at boundaries without high levels of strain localization, the constraint at grain boundaries leads to a narrowing of the slip bands as they approach the boundary and to increased local lattice rotation.

In conclusion, we have shown that sub-microscale surface strain mapping, made possible by gold remodelling, can provide new insights into the deformation of metals. In combination with EBSD, it enables the study of the deformation associated with deformation substructures. In particular, it enables the simultaneous study of the kinematics of deformation with the associated lattice deformation. This can provide invaluable information for the validation and development of new plasticity models that can account for the development of dislocation fields and provide a better description of the deformed state of the material. Work is underway to use this methodology to characterise the deformation of different alloys, both monolithic and dual phase, at different temperatures and strain rates.

**Acknowledgments**

The authors would like to thank Serco TCS for funding and Dr. Fabio Scenini, Mr. Michael Faulkner, and Mr. Ken Gyves for their useful comments on technical aspects of the present study.
References

4. Sutton MA, Li N, Joy DC et al. (2007) Scanning electron microscopy for quantitative small and large deformation measurements part I: SEM imaging at magnifications from 200 to 10,000. Exp Mech 47:775–787
5. Sutton MA, Li N, Garcia D et al. (2007) Scanning electron microscopy for quantitative small and large deformation measurements part II: experimental validation for magnifications from 200 to 10,000. Exp Mech 47:775–787
33. Python Programming Language. python.org Accessed on the 20th December 2011
Paper 2

Included in the proceeding of the 15th international conference on environmental degradation of materials in nuclear power systems – water reactors (2011)
Colorado, US

Comments: few typos and grammar mistakes spotted in the published version have been corrected. In particular, references 1, 2 and 5 have been added to the text.
Contributions of the author: the author of the present study conducted the experiments and analysis reported in the document. The study was completed under the supervision of Dr. João Quinta da Fonseca. Dr. Wright and Dr. Scenini have contributed to the writing of the document.
Understanding the limits of lattice orientation data analysis in environmental degradation studies

Materials Science Centre, The University of Manchester, Manchester, UK

Corresponding author:
Fabio Di Gioacchino
Materials Science Centre, The University of Manchester, Grosvenor Street, Manchester M1 7HS, UK
fabio.digioacchino@postgrad.manchester.ac.uk
Tel: 0044 07588395408

Abstract

Cold working can significantly increase the susceptibility of metals to environmentally assisted cracking. However, the reasons for this increase susceptibility are still unclear. This is due in part to the difficulty in quantifying and modelling plastic deformation at the required scale. Here, we use a new experimental procedure to study the local microstructural distribution of strain in 304L stainless steel. Digital image correlation was used to map strain at the microstructural level with sub-micron resolution. The results clearly show that a high degree of strain localization develops within individual grains, in the form of highly localized shear bands and micro-twinning. Electron backscatter diffraction was used to quantify the lattice orientation changes in the same area. Analysis of this data included the calculation of kernel average misorientation and of intragranular orientation spread following grain reconstruction. Comparisons of results clearly show that, in most cases, there is no evidence in the lattice orientation data analysis of the high levels of strain measured by DIC. This has important implications in the use of lattice orientation data in the study of the effects of plastic deformation on environment-assisted cracking.

Keywords: Digital image correlation, Electron backscatter diffraction, Strain localization, Statistically stored dislocations, Geometrically necessary dislocations, SCC
Introduction

It is well known that cold work increases the environment-assisted cracking (EAC) susceptibility of both stainless steels and nickel alloys. These alloys are widely used in nuclear power plants. The cold working of components can happen during processing and fabrication and is also often a direct consequence of welding. This makes it important to understand the process by which cold work can affect EAC susceptibility. Since the EAC process occurs at the microstructural scale, it is essential that the deformation of these materials be studied at the relevant scale. Recently, it has been proposed that lattice orientation data gathered using electron backscatter diffraction (EBSD) can be used to characterize the non-uniform plastic strain at the microstructural level during deformation [1 -5]. Yet, from the geometrical derivations of Nye [6] and the discussions of Ashby [7] and of Arsenlis and Parks [8], it is clear that only certain parts of the total dislocation density induces changes in lattice orientation (lattice curvature). These types of dislocations, which are necessarily stored for lattice compatibility, are called geometrically necessary dislocations (GNDs) and are associated with gradients of plastic strain. The remaining dislocations that are inevitably generated during crystallographic slip as a result of random nanometer scale heterogeneities, but which do not cause lattice distortion, are termed statistically stored dislocations (SSDs). According to literature [9], SSDs are expected to be predominant when plastic strain gradients are minimal, as in the case of uniformly deformed materials without hard particles or second phases. These dislocations contribute to the hardening of the material and are an important component of the effect of cold work on the environmental degradation of these materials [10-12].

Unlike GNDs, accumulation of SSDs does not alter the local lattice orientation. Thus, SSDs density cannot, in principle, be determined using the orientation data provided by electron backscatter diffraction methods [13]. Nevertheless, there is no reason why SSDs and the deformation associated with them are not relevant to EAC and therefore it is possible that, by relying solely on orientation data, an important component of cold work is ignored.

Digital image correlation (DIC) is a computational technique, which allows the measurement of strain at the surface of a strained material by cross-correlating images acquired during deformation [14]. Microscale resolution of strain can be achieved using high magnification system for image acquisition, such as scanning electron microscopes (SEM) [15]. Although there are several experimental challenges in high magnification image correlation, tailored experimental procedures have been refined over the past few years to achieve resolutions of about 20 displacement
vectors per square microns [16]. With such densities of displacement vectors, it is possible to accurately measure strain and strain gradients in micron sized regions of the material. As expected and shown in [16], the discontinuous nature of polycrystalline deformation at these scales becomes apparent. The distribution of SSDs can then be estimated by the simple assumption of proportionality with plastic strain, as suggested by numerous authors [17].

In this paper, we report results for 304L type stainless steel deformed to 5% in tension. The material is used in pressurized water reactor (PWR) and its susceptibility to stress corrosion cracking (SCC) in such environment has been assessed in previous studies [18]. Our results show a high degree of strain localization occurring at a microscale, which is thought to be relevant to EAC. Comparison with kernel average misorientation analysis (KAM) and orientation spread maps, which are widely used to estimate local variations of lattice orientation, confirms that a relevant part of deformation localization is not captured using EBSD data analysis.

**Materials and methods**

**Microstructure and Sample Geometry**

Chemical compositions and microstructural details of the 304L stainless steel used for our investigations are listed in Table 1. The material was tested in the solution annealed condition. The geometry of the specimens is depicted in Fig. 1(a) with the investigated region highlighted.

**Table 1** Chemical composition of the 304L Stainless Steel tested

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>0.021</td>
<td>0.34</td>
<td>1.96</td>
<td>18.15</td>
<td>9.17</td>
<td>0.031</td>
<td>0.027</td>
</tr>
</tbody>
</table>

**Gold Remodelling and Pattern Imaging**

The DIC technique requires images to show a sufficient amount of features, of the correct size and spacing, whose displacements during deformation is used to describe the underneath surface deformation. Therefore, the application of a speckle pattern onto the surface of interest is usually required. In this study, vapor assisted
remodelling of gold film has been used to produce the nano-sized speckle patterns necessary for sub-micron scale displacement resolution. Details on the remodelling process can be found in [19].

The samples were polished to a mirror finish using colloidal silica solution for 20 minutes and then gold coated for 5 minutes using an Edwards S150B sputter coater to achieve a nominal gold film thickness of 50 nm. The material was then placed in a purpose built apparatus, described in [16], where it was heated to 250°C and exposed to water vapor. Complete remodeling, i.e. clustering of gold particles to form nano-sized speckles, was observed after 90 minutes. Gold speckle size ranged between 20-100 nm with an average speckle distance of approximately 100 nm, Fig. 1(b).

![Image](image.png)

**Fig 1** (a) Specimen geometry and highlight of the investigated region, units in millimeters; thickness 2mm. (b) Details of gold speckles pattern imaged using electron backscatter mode.

### Image Acquisition and Correlation

Following the application of the gold speckle pattern, a series of micro-indents were created on the surface of the specimen to act as fiducial marks. Images of the region were acquired using a Sirion FEI scanning electron microscope (SEM) using the backscatter imaging mode. The specimen was then strained, ex-situ, at a constant strain rate of $4 \times 10^{-3}$ s$^{-1}$ to 5% elongation using a Zeiss-Kammrath micro-tester, after which the sample was re-imaged in the SEM. Images (size 2576×1936 pixels) were acquired at a magnification of 2000×, which corresponds to a field of view of about 60×45 μm$^2$. In order to cover a larger area and include several grains in the measurement, a 150×120 μm$^2$ area was covered by acquisition of adjacent regions in a
3×3 grid. The images were slightly overlapped to enable seamless stitching into a larger map. As discussed in [16], BEI is preferred over secondary electron imaging (SEI) as the pixel intensities are less affected by the change in surface morphology during deformation. This improves the reliability of the displacements yielded by digital image correlation. DIC analysis was carried out using the commercially available software DaVis from LaVision, Germany [20]. The size of sub-region used in the analysis was 8×8 pixels yielding 6.4×6.4 displacement vectors per square micron.

**EBSD Orientation Mapping**

After image acquisition, the gold speckles were removed with few seconds of polishing using of 0.25 µm diamond paste for lattice orientation measurements. In order to improve the indexing of lattice orientation, the sample surface was polished using colloidal silica solution for 20 minutes. This removed approximately 5 to 10 µm of material. A lattice orientation map was then generated for the region of interest using EBSD. A step size of 0.13 µm was chosen to achieve resolution of lattice orientation map comparable with the one of the strain map.

**Results**

Strain From DIC and Further Characterization Using Lattice Orientation Data

A map of maximum shear strain following a macroscopic elongation of 5% is shown in Fig. 2. The strain map reveals groups of equally spaced shear bands with each group showing a distinct direction and spacing. The map is divided into distinct regions, which are delimited by either an abrupt change in the direction of the shear lines or a gradual fading of these lines.

An inverse pole figure (IPF) coloring map, representing the orientations of the grains in the region of interest, is shown in Fig. 3. The orientation map can be used to detect grain boundaries which, when overlaid onto the strain map, as in Fig. 4, reveals that individual grains have deformed distinctively.
**Fig 2** Values of maximum shear strain for 5% macroscopic elongation. Strains are expected to induce an equivalent distribution of statistically stored dislocations.

**Fig 3** Lattice orientation map (EBSD map) of the investigated region in inverse pole figure (IPF) colors. Boundaries and deformation twins highlighted.

The orientation data also allows the calculation of {111} plane traces, which can also be overlaid onto the strain map, Fig 4. Although the {111} plane traces are often
aligned with the shear bands detected using DIC, this is not always the case. This suggests that although the slip bands are usually caused by slip on a particular \{111\} plane, they are sometimes caused by multiple slip, occurring in a narrow region of the grain. The EBSD data also revealed that many of the more intense shear bands promote the formation of micro-twins (black bands in the grain boundary map, Fig. 4).

![Image of shear strain map](image.png)

**Fig 4** Detail of the maximum shear strain map in Fig. 2. Grain boundaries and \{111\} plane traces (in purple) are superimposed to evidence the crystallographic nature of microscale deformation.

Comparison with KAM Values

The KAM method is used to characterize local lattice distortion by averaging values of misorientation with respect to lattice orientation of adjacent regions. As perturbations of lattice orientation have been observed to be a result of plastic deformation, the method has been adopted to quantify the amount of local deformation induced during cold work and study the link with the enhanced corrosion susceptibility of the material [21, 22]. It is thus instructive to see how a KAM map correlates with the local shear map obtained by DIC. To do this, KAM values were calculated using kernel sizes of $3 \times 3$ finally plotted using commercially available software Channel 5 from HKL [23], Fig. 5. Grains are here evident, showing that highest KAM values are often found at grain boundaries. Band structures are
also visible at intragranular regions, yet, these are affected by noise making the bands poorly defined.

**Fig 5** Kernel average misorientation (KAM) results from the lattice orientation data of the investigated region using a 3×3 kernel size

**Fig 6** Orientation spread map of the investigated region, values in degrees
Comparison with Orientation Spread Map

The range of lattice orientations present within single grains can be revealed once average orientation values are obtained for each grain in the EBSD map. Thus, an orientation spread map can be generated by plotting, for each point, values of misorientation angle with respect to the average orientation of the grain the point belongs to. The advantage of this method is the possibility to visualize long-range curvature of the lattice within single grains. Tailored in-house coding has been used for our calculations; results are depicted in Fig. 6. Significant perturbation from the initial homogeneous lattice orientation is observed with spread reaching 10 degrees at certain grain boundary regions. Yet no fine and regularly spaced bands can be seen.

Discussion

Comparison of strain values in Fig. 4 with KAM and orientation spread values in Fig. 5 and Fig. 6 show opposite trends. Strain localization is predominant at intergranular regions (grain core) whilst it is in some case inhibited at grain boundaries. This can be justified by considering that the internal region of a grain tends to deform more freely than the respective more constrained boundary regions. Within a single grain, the high degree of strain localization is thus achieved as plastic deformation is ascribed to particular volumes of material. Here the intense shear can be expected to induce a proportionally relevant aggregate of lattice defects.

Conversely, as observed in previous studies [24], EBSD data analysis reveals that lattice curvature tends to be minimal in central regions of grains and accentuate in the proximity of grain boundaries. This can be explained if complex strain incompatibilities are to be generated at grain boundary regions only. Here multiple slip along preferential planes probably fails to guarantee lattice consistency so that lattice curvature develops as an additional deformation mechanism. As discussed earlier, lattice curvature requires accumulation of GNDs, which thus are expected to be stored at grain boundary regions. The small amounts of lattice distortion observed around the shear bands suggests instead that dislocation densities are here prevalently of SSDs type; this is shown in Fig. 7.

Electron channeling contrast imaging (ECCI) has been used for its sensitivity to lattice defects, details of the technique can be found in [25]. The presence of well-defined bands in the ECC image, Fig. 7(b), shows that the strain localization observed in Fig. 7(a) induces dislocation storage. As EBSD analysis reveals no
significant effect on the lattice curvature at shear bands, Fig. 7(c) and (d), it can be established that such dislocation accumulation is constituted by SSDs.

![Fig 7](image)

**Fig 7** Detail of a grain in the region of interest. (a) Maximum shear strain map. (b) Channeling contrast image. (c) KAM map. (d) Orientation spread map.

From the point of view of environmental degradation, it can be concluded that EBSD analysis is of limited utility in the study of transgranular processes while it can be regarded as a physically motivated method for studying the effect of plastic deformation on intergranular phenomena, e.g. stress corrosion cracking (SCC). Nevertheless, it is argued that the corrosion susceptibility of a grain boundary can be significantly affected by intense intragranular strain localization, as observed, for instance, in the spread of oxidation from grain boundaries to deformation bands in cold worked stainless steels tested in PWR [26]. Long-range stresses are in fact
expected to arise at boundary regions of two adjacent grains consistently with the level of incompatibility of shear deformation of such grains. However, since the lattice stretching associated with such stresses does not necessarily induce significant changes in lattice orientation it might not be detected by EBSD data analysis.

Conclusion

• The method for high magnification digital image correlation adopted in our previous study confirms that the material under study undergoes high level of strain localization during deformation. Overlaid of grain boundary map and \([111]\) plane traces onto the strain map confirms that lattice proprieties govern the deformation at sub-microscales.

• EBSD analysis failed to characterize the amount plastic deformation and strain localization observed experimentally.

• Comparison between KAM, orientation spread and shear strain maps suggests that SSDs are predominant with respect to GNDs in the intragranular regions of the tested material at a microscale. This makes EBSD analysis appears inappropriate for the characterization of transgranular processes.

• Although valid as a physically motivated method for studying the effect of plastic deformation on intergranular phenomena, e.g. stress corrosion cracking (SCC), it is argued that EBSD analysis might still miss important effects of deformation incompatibility on grain boundaries susceptibility.

• Alternative investigation strategies where EBSD is coupled with complementary techniques, as the one proposed in the present study, are thus highly desirable.

Acknowledgments

We would to acknowledge EPSRC and Serco for funding. We would also like to thank David Tice, Norman Platts, Kevin Mottershead, Samaneh Nouraei and Laura McIntyre for useful discussions and comments.
References

by Optical Correlation of Micrographs Acquired During Deformation”, *Journal of Microscopy*, 218 (2005), 9–21


Paper 3

Paper structured for submission to the Journal of the Mechanics and Physics of Solids

The results of this work were presented at the symposium “Microstructure Based Property Prediction and Small Scale Experimental Validation” held in October 2012 in Pittsburgh, US.

Contributions of the author: the author of the present study has developed the method to derive lattice rotation from DIC measurement described in the document. He also conducted the experiments and analysis that have been presented. The study was completed under the supervision of Dr. João Quinta da Fonseca.
Separating lattice rotations from the deformation mapping of crystals. Insights on the microplasticity of austenitic stainless steel

F. Di Gioacchino, J. Q. da Fonseca

Materials Science Centre, The University of Manchester, Manchester, UK

Corresponding author:
Fabio Di Gioacchino
Materials Science Centre, The University of Manchester, Grosvenor Street, Manchester M1 7HS, UK
fabio.digioacchino@postgrad.manchester.ac.uk
Tel: 0044 07588395408

Abstract

High-resolution digital image correlation (HDIC) of FEG-SEM images is used to map the in-plane deformation of an aggregate of grains at the surface of a 304 austenitic stainless steel sample. Intergranular microbands appeared aligned with [111] plane traces obtained from the analysis of electron backscatter diffraction (EBSD) data. Such direction was taken as the main (in-plane) direction of local lattice slip. Hence, it was shown that it is possible to separate the contribution of lattice rotation from the local deformation mapping following deformation incompatibility arguments. To validate the proposed decomposition method, DIC-derived lattice rotations were compared with those measured by EBSD. Good agreement of results proved, for the first time, the existence of the link between certain gradients of slip and the lattice curvature. Results from the analysis of HDIC and EBSD measurements are further discussed to give new insights on the micromechanism of deformation in austenitic stainless steel.

Keywords: crystal plasticity, strain gradient plasticity, digital image correlation, EBSD
1 Introduction

An increasing number of crystal plasticity models aim to accurately describe the evolution of plastic deformation at the micron and grainscale [1, 2]. This is because there are mounting evidence suggesting that material properties such as strain hardening and toughness are affected by the non-uniform plastic deformation developing at the microstructural (mesoscopic) scale. For instance, in the stainless steel tested here, localization of strain at microbands is believed to both promote hardening by deformation-induced twinning [3] and to assist damage by localization of stresses at grain boundaries [4 - 6].

Mapping techniques can provide experimental evidence to support model predictions on strain localization. As a well-established and accessible technique, electron backscatter diffraction (EBSD) has been widely used to map lattice rotation gradients, i.e. lattice curvature, to compare with the respective model predictions. Numerous applications can be found in the study of plastic deformation in polycrystalline aggregates [7, 8] as well as in microsized single crystals [9 - 11]. Interestingly, simulations tend to give realistic predictions when the lattice curvature is induced by the geometry of the imposed deformation, as in nanoindentation studies [9]. In contrast, predictions appear less accurate when the lattice curvature is promoted by microstructural heterogeneity [7]. This is likely to happen because the accuracy of lattice curvature predictions is strongly dependent on the accuracy of crystallographic slip predictions.

This is better pictured in the kinematic representation of elastoplastic deformation of single crystal proposed by Asaro and Rice [12], which is represented in Fig. 1.

Following Lee’s multiplicative decomposition of the deformation gradient $F = F^e F^p$ in its elastic $F^e$ and plastic part $F^p$ [13], $F^p$ maps the material point $X$ in the initial configuration $\mathcal{B}_0$ to an intermediate (imaginary) stress-free configuration $\mathcal{B}_i$, which is achieved by crystallographic slip only. The slip is assumed to leave the lattice vectors $(s, m)$ unchanged and unrotated [14]. It follows that the elastic part, which maps the materials points in intermediate configuration to the points $x$ in the current configuration $\mathcal{B}$, is given by elastic stretches and lattice rotations.

Diffraction patterns primarily reproduce rotations and, to some extent, elastic stretches of the crystal lattice [21, 22]. Hence, it is evident that EBSD is only capable of measuring the elastic part of the deformation gradient tensor, i.e. $F^e$. In other words, EBSD is insensitive to the part of lattice slip that leaves the lattice unchanged, i.e. $F^p$. 
The plastic part of the crystal deformation $F^p$ has a dual primary role in crystal plasticity. It is associated with the evolution of statistically stored dislocations (SSDs) [15] and, it describes the incompatibility of deformation as $\text{Curl}F^p$ that origins the lattice curvature [16 - 20].

So far, $F^p$ has been accessible only through computational methods [23, 24]. Being an imaginary intermediate state, it is hard to imagine microscopy techniques able of describing such state. Yet, it appears possible to obtain $F^p$ by applying the mapping $F^{e-1}$ calculated using EBSD to $F$ calculated using deformation mapping techniques such as digital image correlation (DIC), Fig. 1. DIC allows in-plane full-field displacement mapping by correlating images acquired at the surface of a strained sample for different stage of deformation [25, 26]. The displacement vector field can then be differentiated to derive the deformation gradient tensor $F$.

Alternatively, it can be though possible to develop methods for decomposing $F$ calculated from DIC measurements into $F^p$ and $F^e$.

In the present study, we aimed to develop a decomposition method built on the aforementioned arguments on deformation incompatibility. To validate the method, DIC-derived lattice rotations $F^e$ were compared with the actual measurements obtained by EBSD.

**Fig 1** Kinematic model of elastoplastic deformation of a single crystal emphasizing the lack of experimental methods available to measure $F^p$
1.1 Spatial resolutions of DIC and EBSD measurements

It is important to compare DIC and EBSD measurements at equivalent spatial resolutions. Fig. 2 modifies the schematization proposed by McDowell in [1] to highlight the difference in the scale of investigation of these techniques.

An EBSD scan can be performed with submicron resolution (O 10^{-7} m) and set to cover several grains in most material of interest (O 10^{-5} m) [21]. In contrast, current DIC methods allow mapping deformation with maximum resolution on the orders of microns (O 10^{-6} m) [27].

The resolution of DIC measurements primarily depends on the density of speckles of the pattern applied at surface of the material, which serves identifying distinct regions of the material during deformation [25, 27]. Recently, Di Gioacchino and Quinta da Fonseca were able to map plastic strain in austenitic stainless steel with sub-micron resolution of 0.21 × 0.21 µm^2 using gold remodelling as pattern application method [27]. Such method is therefore used in the present study to enable comparison of HDIC and EBSD lattice rotation (\( F^\theta \)) measurements.

**Fig 2** Comparison between the length scales of investigation of distinct microscopy techniques

2 In-plane lattice rotations from HDIC measurements

It is possible to identify a simple relation between the displacement vector field and the induced lattice curvature. For this purpose, we recall an intuitive 2D representation proposed by Fleck in [28].
Let three adjacent volume elements to shear following slip along the direction $X_1$ by an amount $\gamma_1 = \gamma_1(X_1)$ such that the intermediate configuration would look like depicted in Fig. 3 (b). The shear can be obtained by lattice slip carried by dipoles dislocations arranged as depicted. The component $\mathbf{F}^p_{12}$ varies according to the coordinate $X_1$ such that $\mathbf{F}^p_{12} = \gamma_1$. Joining the elements back together would require a rotation $\varphi_3$ with respect to the out-of-plane direction and a displacement $\mathbf{u} = \mathbf{u}(X_1, X_2, 0)$. For small rotations $\varphi_3 \equiv \sin(\varphi_3)$, it is

$$\varphi_3 = \mathbf{u}_{21} = \gamma_1 \quad \text{with} \quad X_1 \equiv s \quad (1)$$

Equivalent results would be obtained if the elements stretch along the $X_1$ direction of an amount that varies with $X_2$.

In order to plot the values of in-plane lattice rotations $\varphi_3$ in the current (deformed) configuration, the displacement field has to map the deformed configuration to the (initial) undeformed one, i.e. $\mathbf{u}' = \mathbf{u}'(x_1, x_2)$. Similarly to equation (1), it gives:

$$\varphi_3 = -\mathbf{u}'_{21} = \mathbf{F}^{pT}_{12} = -\gamma_1 \quad \text{with} \quad x_1 \equiv s \quad (2)$$

It is noted that joining the elements together has annihilated some dislocations and left some stored. These dislocations maintain lattice integrity and represent the so-called geometrically necessary dislocations (GNDs) [16]. Differentiating equation (2) gives:

$$b_1 = \varphi_{3,1} = -\mathbf{u}'_{2,11} = \mathbf{F}^{pT}_{12,1} = -\gamma_{1,1} \quad (3)$$

Expressions in (3) show the link between the dislocation state, the lattice curvature and the components of deformation $\text{Curl}\mathbf{F}^p$ and $\text{curl}\mathbf{F}^{e-1}$.

Fig. 3 illustrates the effect of the gradient of slip on the lattice rotation. In practice, the value of lattice rotation obtained as $\varphi_3 = -\mathbf{u}'_{2,1}$ with $x_1 \equiv s$ will also comprise the contributions of a grain based rigid body rotation given by the rotation of grains during deformation and of a rigid body rotation induced by sample mounting between deformation steps.
2.1 Identifying the direction of slip using microbands

The relations derived in the previous section require prior knowledge of the direction of lattice slip; this is, in fact, inherited in the decomposition of the deformation gradient tensor depicted in Fig. 1. Hence, in order to calculate $\phi_3$ in expression (2), it is necessary to first assume a direction of slip. At the microscale, it is reasonable to take the direction of microbands as the “mesoscopic” direction of slip as considered, for instance, in [29].

In the present study, the direction of microbands resolved by HDIC is therefore taken as the direction of slip $s$ (step 1 in Fig. 4).

In commercially available software for DIC analysis, as the one used here [30], correlation points lay on a grid with principal directions parallel to the sides of the correlated images. Hence, images can be rotated to make one of the two principal axes of the correlation grid coincide with the direction $s$ (step 2 in Fig. 4). The
unstrained image is thus correlated to the deformed one to obtain the displacement field $\mathbf{u}' = \mathbf{u}'(x_1, x_2)$.

**Fig 4** Schematization of the step needed to meet the alignment of the slip $s$ with the grid of the DIC software

2.2 The effect of multiple slip

In practice, the noise of SEM imaging limits plastic strain mapping measurements to increments of imposed deformation exceeding 1% [27]. During such deformation step, multiple slip systems are believed to be active for stainless steel as for other structural alloys [31]. This results in multiple groups of distinct microbands observed in the HDIC strain mapping.

Where two microbands meet, the volume element can be therefore imagined to have gone through (at least) two consecutive steps of deformation $(t_1, F_1)$ and $(t_2, F_2)$, Fig. 5. In this case, the proposed decomposition method cannot be applied. This is because choosing a direction $s_1$ for expression (2) will make the slip along the other direction $s_2$ affect the value of $\varphi_3$, Fig. 5.
Fig 5 Kinematic model of elastoplastic deformation of a single crystal extended for two consecutive deformation steps \((t_1, F_1)\) and \((t_2, F_2)\).

3 Measuring in-plane lattice rotations using EBSD

As discussed above, DIC allows measuring the displacement of material points at the surface of the strained sample. The displacement of material points along the normal to the sample surface is in fact not accessible, even in stereographic conditions (3D-DIC) [5]. It follows that only in-plane deformation can be described.

Similar to DIC, EBSD is a surface technique. Yet, measured lattice rotations occur around a general axis in the 3D space. It is nevertheless possible to obtain the component of the rotation with respect to the out-of-plane axis [32] and therefore enable comparison with the predicted in-plane lattice rotations.

The orientation of the crystalline lattice at each point of the EBSD map is described as the rotation \(\varphi\) along the axis \(r\) required to bring a common reference lattice to match the lattice at such point. Several ways to perform operation with rotation are available. In the present study, we use quaternion representation of rotations for the advantage in computational efficiency [33, 34]. Detailed description of quaternion algebra can be found elsewhere [35] as we report only the expressions used to derive the quantities of interest. The unit quaternion \(q = q(\varphi, r)\) describing the rotation \(\varphi\) around the rotation axis \(r\) can be constructed as:

\[
q = \begin{pmatrix} q_0 \\ q \end{pmatrix} = \begin{pmatrix} \cos(\frac{\varphi}{2}) \\ \sin(\frac{\varphi}{2})r \end{pmatrix}
\] (4)
The magnitude of $r$ is such to make $q$ a unit quaternion, i.e. $|q| = 1$. Performing a rotation $q_1$ followed by a second rotation $q_2$ is equivalent to a single rotation $p$ obtained as the quaternion product $q_1q_2$ defined as:

$$p = q_1q_2 = \left( q_1^0q_2^0 + q_1^1q_2^1q_1^2q_2^2 - q_1^2q_2^0 + q_1^0q_2^2 - q_1^1q_2^1 \times q_2^0 \right)$$ (5)

The conjugate $q^* = (q_0, -\vec{q})$ of the unit quaternion $q$ represents the inverse rotation. It is therefore possible to describe the difference between two rotations, i.e. the misorientation, in (sample) reference system as [32]:

$$m_s = q^*_1q_2$$ (6)

The expressions for the misorientation angle and axis derive from the definition of quaternion in (4):

$$\varphi = 2 \cos^{-1}(m_s^0) \quad \quad r = \frac{m_s}{\sin\left(\frac{\varphi}{2}\right)}$$ (7a-7b)

The in-plane component of lattice rotation can be finally obtained from the component along $x_3$ of the disorientation vector $\varphi_i = \varphi r_i$, that is [32]:

$$\varphi_3 = m_s^3 \frac{2 \cdot \cos^{-1}(m_s^0)}{\sqrt{1-(m_s^0)^2}}$$ (8)

3.1 Comparison of DIC and EBSD measured lattice rotations

Calculation of lattice rotations involves acquiring EBSD maps before and after deformation. Moreover, it requires the use of the deformation mapping to identify the position of the lattice points in the respective maps. However, this is no longer necessary when lattice points share a common orientation in the initial configuration. This is the case of materials deformed from an annealed state.

For these, a rigid body rotation $Q$, which brings the measured orientations to match the predicted lattice orientations, can be identified for individual grains in the
deformed configuration. This is depicted in Fig. 6 where $R_{PL}^e$ and $R_{EX}^e$ are the DIC derived and EBSD measured lattice rotations, respectively.

![Diagram of lattice orientation gradients](image)

**Fig 6** Schematicization of the relationship between lattice rotations predicted by the deformation mapping and the lattice orientation measured after deformation

In order to reveal lattice rotation gradients in the EBSD map, it is therefore possible to select an arbitrary lattice point to take as a new reference lattice orientation in each grain and use expression (8) to calculate the in-plane misorientation. Prior to this, it is necessary to divide lattice points according to the grain these belong by grain reconstruction. The symmetry of the lattice must also be considered in these calculations, as shown in [34].

### 4 Material and experimental methodology

#### 4.1 Material

The material chosen for this study is 304L stainless steel. From a micromechanics point of view, it is an interesting material to study because its low staking fault energy leads to strain localization at different length scales and also micro-twinning, which makes it an ideal material to study using sub-micron deformation mapping. The material is also known to produce high quality Kikuchi patterns after OPS polishing, which is a necessary prerequisite to enable efficient comparison of HDIC and EBSD measurements. The chemical composition is given in Table 1.
Table 1 Chemical composition of the 304L stainless steel in weight %

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>0.021</td>
<td>0.34</td>
<td>1.96</td>
<td>18.15</td>
<td>9.17</td>
<td>0.031</td>
<td>0.027</td>
</tr>
</tbody>
</table>

4.2 Pattern application for DIC and imaging

A flat dog bone shaped specimen of 5 mm gauge length and 2 mm thickness was cut from the material in the solution annealed condition (1h at 1050°C and water quenched). The nano-scale speckle pattern was applied using gold remodelling procedure described in details in [27]. In particular, the sample was polished using a diluted colloidal silica (OPS) solution for 10 minutes and gold coated with 50 nm gold film in order to induce a speckles pattern of the type shown in Fig. 2 (g) in [27] with remodelling. Following the application of the gold speckle pattern, a series of micro-indents were created on the surface of the specimen to act as fiducial marks and allow quick identification of the area of interest and alignment of the sample following deformation.

Table 2 SEM scan parameters used for image acquisition

<table>
<thead>
<tr>
<th>Imaging mode</th>
<th>ACC. Voltage (kV)</th>
<th>Beam Current (nA)</th>
<th>Working distance (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BEI</td>
<td>22</td>
<td>1.1</td>
<td>5</td>
</tr>
<tr>
<td>Linescan (msec)</td>
<td>Magnification</td>
<td>Image size (pixels)</td>
<td>Spatial resolution (nm)</td>
</tr>
<tr>
<td>40</td>
<td>2000×</td>
<td>2756 × 1936</td>
<td>≈21</td>
</tr>
</tbody>
</table>

Images of the region of interest were acquired using a Sirion FEI scanning electron microscope (SEM) equipped with a Schottky Field Emission Gun (FEG). Image acquisition was performed at resolution of 2756 × 1936 pixels and linescan of 40 msec, which limited single image acquisitions to approximately 80 seconds. Images were acquired at a magnification of 2000×, which corresponds to a field of view of about 60 × 45 µm² and a spatial resolution of 22 nm per pixel. For the reasons described in [27], backscatter electron imaging was chosen, with high beam voltage and a small working distance. The values used are summarized in Table 2.
4.3 Image acquisition procedure and correlation

The specimen was strained using a Zeiss-Kammrath micro-tester, at constant strain rate of $4 \times 10^{-3}$ s$^{-1}$ to final elongation level of 6%. At each stage, the sample was removed from the microtester and mounted on the SEM stage for image acquisition. The region was imaged keeping loading along the horizontal ($x$) direction of the imaging window.

In order to cover a larger area and include several grains in the measurement, image acquisition of adjacent regions in a $3 \times 3$ mosaic was performed. The images were slightly overlapped to enable seamless stitching into a larger map covering in total an area of $150 \times 120$ µm$^2$. The correlation was carried out using the commercially available software DaVis [30] with an interrogation windows of $8 \times 8$ pixels which corresponds to $0.17 \times 0.17$ µm$^2$ and a measurement density of about 34 displacement vectors per square micron. The images of the initial and deformed pattern were correlated to get both $\mathbf{u}(X_1, X_2)$ and $\mathbf{u}'(x_1, x_2)$.

The displacement data were then exported in a text file and analyzed using Numerical Python [36]. For preliminary observations on deformation features and comparison with EBSD data, we chose to calculate $\text{Curl}(\mathbf{u})$ and $-\text{Curl}(\mathbf{u}')$. The curl of a vector field is commonly taken to describe the rotational component of a vector flow with the sign of its value determined by the sense of the angle spanned. Following the right hand convention, and considering the reference system with in-plane direction normal to the surface, positive values were made to correspond to clock-wise sense and negative values to counter-clockwise sense.

Successively, once the direction of primary slip was individuated for each grain, the maps were rotated as showed in section 2.3 and expression (2) used to extract lattice rotations.

4.4 EBSD data acquisition and analysis

After image acquisition, the gold speckles were removed with few seconds of 0.25 µm diamond paste polishing for lattice orientation measurements using EBSD. In order to improve the indexing of lattice orientation, the sample surface was polished further using 0.05 µm colloidal silica solution for 20 minutes. This removed about 4 µm of material from the surface. A lattice orientation map was then generated for the region of interest using a step size of 0.13 µm to give a map with a resolution comparable to the strain map. The acquisition was performed using Channel 5 HKL software [37]. Again, the data were exported as a text file and analyzed using Numerical Python. In analogy to the convention of the DIC measurements, which
consider an out-of-plane direction, clockwise rotations taken positive and counterclockwise negative.

5. Results of DIC and EBSD mapping: preliminary observations

5.1 Microstructural features

EBSD data are used to highlight the microstructural features in the investigated region. In Fig. 7, grain boundaries are depicted in black whilst lattice region with BCC structures are colored in red. A ferrite particle is evident in grain 1. Deformation twins are clearly distinguishable as lamellae-shaped subgrains in several grains.

![Microstructural features of the investigated region.](image)

*Fig 7* Microstructural features of the investigated region. Grain boundaries are depicted in black and the grain numbered in sequential order. BCC lattice is colored in red

5.2 Deformation features: microbands

Values of $Curl(u)$ for 6% macroscopic strain are plotted in Fig. 8 with a scale between ±0.20. As can be seen, deformation in this material is highly localized with
contiguous regions showing up to 0.40 difference in values. Sharply defined bands appear clustered in groups of distinct direction and spacing. In particular, a single cluster of bands which extent across the grain can be identified for each grain. These are henceforth referred as primary bands (PB). In some cases, primary bands in adjacent grains appear connected at the boundaries as for grains 3-12-13-14 and the grains 6-7-8. The remaining bands are referred as secondary bands (SB), which, in contrast to primary bands, are seen to cover only limited portions of individual grains as evident in grain 2, 3, 4 and 11.

Fig 8 Values of Curl following 6% macroscopic tensile strain along the horizontal direction. Primary bands (PB) and secondary bands (SB) are highlighted for grain 3 as example

Individual bands can show distinctive traits. As also reported in [27], the trajectory of a band can divert slightly, as in the magnified regions in Fig 9 (a) and (b), or fade gradually, as seen for primary bands approaching grain boundaries in grain 2 and 4. Fading of bands is also observed in correspondence of the second phase particle in grain 1, Fig 9 (c), and in the regions of grain 4 and 7 in Fig 9 (d) and (e) respectively. Notably, there is no evidence of second phase particles in correspondence of these latter regions. Therefore, band fading might be here attributed to second phase particles right beneath the surface or alternatively, to particles which were later removed by polishing.
Fig 9 Details of deformation features observed in Fig 6. Examples of curved bands, (a) and (b). Values of curl in correspondence of second phase particles, (c), (d) and (e).

The sign of the curl observed at the bands can be associated to the characteristic of the imposed deformation. It is in fact seen that, whether primary or secondary, bands are blue colored (negative values) when found at $<90^\circ$ angles with respect to the tensile axis, whilst are red colored (positive curl) at $>90^\circ$ angles. Furthermore, regions between adjacent bands show values of curl of opposite sign with respect to the values at bands, i.e. blue bands appear on a red background whilst red bands appear on blue background.

These findings can be easily interpreted by looking at the displacement of the gold speckles in correspondence of a single microband. Fig. 10 shows details of the speckle pattern in grain 3 before (a) and after deformation (b). The position of selected speckles is highlighted by squares and circles and tracked during deformation. It can be seen that the speckles in the red squares have moved up along direction $s$ with respect to the speckles in white squares spanning an angle in a counter clockwise sense. Conversely, the displacement of the speckles in the circles with respect to their initial position has spanned an angle in a clockwise sense. The correlation between the two images yields the $Curl (\mathbf{u})$ map in Fig. 10 (c).
Fig 10 Details of the gold speckle pattern in the investigated region before (a) and after 6% macroscopic strain. Curl calculated from the displacement field obtained by correlating the image of the deformed pattern to the image of the pattern before deformation (c). Interpretation of the kinematic of deformation (d).

As expected, a band of negative value is detected forming an acute <90° angle with the loading direction in correspondence of the squares whilst the adjacent regions show positive values. The kinematic observed is consistent with the region contributing to the accommodation of the imposed deformation, Fig. 10 (d).

Relevant to the discussion on deformation incompatibility presented below is the link between the sense of shear of primary bands and the one of secondary bands. For grains where the latter are observed, secondary bands have opposite sign with respect to primary bands, and consequently such do the regions of material between the bands. Following the arguments described above, this indicates that the regions of the grain covered by primary and secondary bands rotate in opposite senses.

5.3 Comparison between deformation and microstructural features

Fig. 11 shows values of $-\text{curl } (\mathbf{u}')$ for 6% macroscopic strain between ±0.20. As discussed earlier, describing the deformation in the deformed configuration enable
the comparison of EBSD data acquired after deformation. As expected, values are of equivalent sign and intensity to those of $\text{curl } (u')$ in Fig. 8. Yet, deformation features are now observed in their current (deformed) position.

- Curl $(u')$

![Image showing values of $-\text{curl}$ calculated for 6% macroscopic elongation in the deformed configuration. (111) planes traces obtained from lattice orientation data analysis acquired following deformation are superimposed. Traces of planes with the two highest Schmid factors are highlighted in red. Black dots indicate the grains where only partial correspondence is found between high Schmid factor traces and bands directions.]

**Fig 11** Values of $-\text{curl}$ calculated for 6% macroscopic elongation in the deformed configuration. (111) planes traces obtained from lattice orientation data analysis acquired following deformation are superimposed. Traces of planes with the two highest Schmid factors are highlighted in red. Black dots indicate the grains where only partial correspondence is found between high Schmid factor traces and bands directions.

Similarly to the analysis done in previous work [27], (111) planes traces are plotted using numerical python and superimposed on the strain map. In particular, the traces associated with the planes having the two maximum values of Schmid factor are plotted in a thicker red line. The bands appear aligned with the latter traces confirming both the crystallographic nature of the bands and the geometric link of the sense of shearing with the direction of the imposed deformation. The alignment is achieved following a $7^\circ$ clockwise rotation of the EBSD map, which indicates that the EBSD map was acquired along such orientation with respect to acquisition scan of the DIC images. As observed in [27], only for a small fraction of grains highlighted using block dots, the bands match (111) planes traces that are not associated with the
highest Schmid factor planes. Again, these appear more likely to be found in small grains. This is plausible if considered that the deformation imposed on smaller grains might diverge more readily from the macroscopic imposed deformation than in bigger grains.

6 Results: comparison between deformation derived and measured lattice rotations

6.1 EBSD measured lattice rotations

Fig. 12 shows the in-plane lattice rotations $\varphi_3$ calculated from EBSD data as described in section 3. Values of the rotation angle are plotted in the same range as the values of curl in Fig. 8 and Fig. 11, i.e. between ±0.20 radians, which corresponds to about ±11°. Also, equivalent convention for the sign is adopted: positive (red) values represent clockwise rotations and negative (blue) counter-clockwise rotations. Grain boundaries including deformation twins are preserved. It is evident that no sharp lattice rotation gradients are present in correspondence of the microbands observed in the strain maps.

![Fig 12](image.jpg)

**Fig 12** In-plane lattice rotations calculated for each grain in the investigated area from EBSD map acquired after deformation
On the contrary, long-range gradients of lattice rotation are detected, which develop within each grain (intragranular rotation gradients) and also across adjacent grains (transgranular rotation gradients). This is evident, for instance, in the column-shaped feature crossing grains 12, 13 and 3. Linescans have been performed along the plotted arrows to compare the values with the DIC derived lattice rotations given in the next section.

6.2 HDIC derived lattice rotations

In the present section, we follow the procedure of DIC analysis described in section 2 to derive in-plane lattice rotations $\varphi_3$ in the regions where primary bands are found. The direction of PB is thus used as direction of slip. Regions are analyzed separately according to the angle formed by primary bands with the loading direction. Fig. 13 shows results for the grains having blue (negative values) primary bands in Fig. 8 and Fig. 11, which, as observed, are those at $<90^\circ$ with respect to the loading direction. The remaining grains are outlined.

![Diagram showing HDIC derived lattice rotations](image)

**Fig 13** HDIC derived lattice rotations for regions crossed by primary bands at an $<90^\circ$ with respect to the loading direction; the remaining areas are outlined.

As seen in the measured gradients of rotations, values in correspondence of primary bands are consistent with the rotation predicted for the surrounding lattice giving rise to long-range gradients. The values are mostly on the positive range testifying
the grains have rotated clockwise. Secondary bands are nevertheless visible. This can be attributed to the effect of multiple slip described in section 2.4. Notably, smooth changing values are also found in correspondence of microbands that have been seen to induce twinning, as shown in Fig. 7. This is indeed expected as the change in lattice orientation that defines twinning is not an actual lattice rotation but a result of pure crystallographic slip [3, 6].

Although the column-shaped feature individuated in Fig. 13 is not visible, gradients of predicted lattice rotations are seen to agree with the measured rotations gradients. To verify the agreement of DIC derived and EBSD measured lattice rotations, line scans have been performed in same regions scanned in Fig. 12. In particular, a scan is done across the deformation field affected by the second phase particle (Linescan 3) and across grain 13 (Linescan 4), where the most intense lattice curvature is observed in both HDIC and EBSD measurements.

![Graphs showing the variation of DIC and EBSD measured lattice rotations for the Linescans 1 to 4 in Fig 12 and Fig 13](image)
The value of $\varphi_3$ at each point is calculated as the difference with respect to the value at the initial point of the scan. Although the column feature appeared smeared in the DIC-derived lattice rotation map, as shown in the graphs in Fig. 14 (a) and (b), the scans reveal the same trends for lattice rotations with analogous change in magnitude and sense of rotation, Fig. 14. In-plane lattice rotation $\varphi_3$ is seen to vary as much as 0.011 radians, i.e. about 6°, within a 15 $\mu$m distance in Linescan 4.

**Fig 15** DIC derived lattice rotations from HDIC measurements for regions crossed by primary bands at $>90^\circ$ with respect to the loading direction

**Fig 16** Graphs showing the variation of DIC and EBSD measured lattice rotations for the Linescans 5 and 6 in Fig 12 and Fig 15
Fig. 15 shows results of the analysis for the grains having red primary bands in the regions obscured in Fig. 13. The values are here in mostly on the negative range testifying the grains have rotated counterclockwise. Again primary bands are not observed and only the secondary bands remain visible. As seen for the other cases, new secondary bands systems are now distinguishable.

Graphs in Fig. 16 compare the predicted and measured lattice rotations for the Linescans 5 and 6. Again, a same trend of in-plane lattice rotations values is visible across the scans.

6.3 Microband - grain boundary interaction

The possibility of analyzing each grain separately provides a way to visualize the source of deformation incompatibility, i.e. to identify the mechanisms leading to lattice curvature. Example of regions of interest are labeled using lettering in Fig. 15 and in Fig. 17, with the latter showing magnified regions of Fig. 13.

Fig 17 Details of Fig. 13 showing the interaction between bands and grain boundaries resulting in lattice curvature. The letters follow the sequence started in Fig. 15.
It is possible to observe that primary bands in regions A-D and secondary bands in E and F induce lattice rotations in the adjacent grains. In particular, the sharp rotation gradients observed in both measured and predicted lattice orientation for grain 13 are evidently a result a primary band from grain 1 hitting the grain 13 in D. Moreover, it is evident that the induced lattice curvature is related to the deformation imposed by the hitting bands. The rotational component described by the values of curl at these bands is in fact of the same nature (sign) than the lattice rotation observed in the affected regions.

6.4 HDIC analysis using secondary bands as direction of slip

As for the primary bands, secondary bands can also be taken as direction of slip. The secondary slip in grain 12 are taken as example, Fig 18.

![Diagram](image)

**Fig 18** (a) strain map. (b) and (c) HDIC derived lattice rotations using taking the direction of slip parallel to the primary bands (left) and secondary bands (right). (d) EBSD measured lattice orientations
The −$\nabla \times (\mathbf{u}')$ map shows that the interior of the grain shear through primary bands at an acute <90° angle with respect to the loading (horizontal) direction. Within the same deformation step, the outskirt is also shearing along secondary bands with common direction, Fig. 18 (a). It is possible to calculate \( \varphi_3 \) for \( s = s_{PB} \) and \( s = s_{SB} \) to study the predicted orientation gradients for the two regions. Results are shown in Fig. 18 (b) and (c) respectively. As seen above, the inner region covered by primary bands alone is seen to rotate clockwise (positive values) whilst the outskirt is seen to rotate counter-clockwise (negative values). If we image to combine the predicted lattice rotation for the secondary region with the primary regions we see that the combined lattice rotation agree with those calculated from EBSD data, Fig. 18 (d).

7 Discussion on preliminary deformation mapping

7.1 Trajectory of microbands

Results showed that microbands are well-aligned to the \{111\} planes traces in the reference configuration. Yet, at higher magnifications, bands can present microsteps or appear curved. A possible interpretation given in [27] assumes crystallographic slip to be shared by distinct \{111\} slip systems across the length of the band as a result of a complex distribution of intragranular stresses. An additional mechanism can be imagined if considered that slip along two different crystallographic planes cannot happen simultaneously but only sequentially, as depicted in Fig. 19.

Fig 19 Schematization of a possible mechanism leading to diverted microbands for DIC measurements described in the initial configuration
Gold speckles in the initial configuration \((t_0)\) shear along the direction \(s_1\) to the deformed configuration \((t_1)\) and subsequently along a secondary direction \(s_2\) to a second deformed configuration \((t_2)\). Measuring strain by correlation of the deformed pattern at \(t_2\) with the initial pattern allow identifying the regions that have sheared. As schematized in the bottom-left image of Fig. 19, the blue band will divert its course in correspondence of the red band forming a step of amount proportional to the shear along \(s_1\) at \(t_1\). Both mechanisms are believed to coexist during deformation. For instance, the former mechanism is believed responsible for the curved bands in Fig 9 (b), whereas the second might be the cause of the microband diversion in Fig 9 (a).

7.2 Dislocation types at microbands

Besides the change in lattice orientation following twinning, no bands are evident in the EBSD lattice rotation map. This can be justified considering that plastic deformation at microbands is mostly compatible at the length scale associated with the resolution of HDIC and EBSD measurements, i.e. \(\text{Curl} \mathbf{F}^P = 0\). This is confirmed by the fact that bands chosen as the direction of slip disappear in the lattice rotation maps. Here the presence of GNDs is limited to those associated with long-range rotation gradients. If lattice defects are present at microbands, as suggested by the channeling contrast imaging (CCI) shown in Fig. 20, these are expected to be primary SSDs at the length scale of DIC and EBSD measurements.

**Fig 20** Values of \(-\text{Curl}(\mathbf{u}')\) in the boundary region between grain 2 and 3 (a). Evidence of dislocation structures in the channeling contrast images that can be attributed to SSDs densities.
8 Discussion on the results of HDIC analysis

8.1 Validity of the decomposition method

Expression (2) appears as the simplest possible expression to obtain lattice rotations from high-resolution deformation mapping. By taking only the component of the displacement vector normal to the direction of slip, the effect of stretch along the slip direction is implicitly assumed negligible. In a 2D representation, this can nevertheless be accounted for using straightforward geometrical arguments. This might be the cause of the only partial correspondence of DIC derived and EBSD-measured lattice curvature values observed for grains 3 and 13 and indicated as the column feature in lattice orientation scans in Fig. 14 (a) and (b). More significant source of errors probably derives from the 3D deformation of the sample surface and from the subsequent removal of a micro layer of material to enable EBSD measurements. Further studies are underway to verify this in more details.

Regardless of this, the good agreement between DIC derived and EBSD measured lattice rotations suffices to provide a first direct experimental evidence of the link between certain deformation gradient and the lattice curvature. This is an important experimental observation as this link is the common basis of strain gradient plasticity theories. Furthermore, the validity of the decomposition proves that the proposed HDIC method can be used to separate the contributions to deformation of lattice slip and lattice rotation. As initially discussed, this gives DIC the ability to provide unique information in support of the development of crystal plasticity models.

8.2 Compatible deformation of adjacent grains

It has been evidenced that, in certain grain boundary regions, cluster of primary microbands from the adjacent grains meet forming a continuous transgranular microbands. These regions are also those that showed more readily continuity of predicted lattice rotations. This observation is consistent with the fact that, if no strain gradients are generated, no lattice curvature is required.

Joining of microbands will therefore be favored as this maximizes the degree of compatible deformation. On a 3D representation, such degree of mismatch can be derived by information on the lattice orientation of the adjacent grains [5].
8.3 Accommodation mechanisms of incompatible deformation

EBSD results evidence a complex variation of lattice rotation within single grains and across neighboring grains, which is commonly observed in deformed polycrystals. Two competing accommodation mechanisms of incompatible deformation can be identified. The first has been described in Fig. 3. The shear at microbands can encounter microstructural barriers, such as second phases and more readily grain boundary region, which oppose to slip. Fading of band is an intuitive evidence of such slip inhibition, Fig. 21 (a). The momentum associated to the microband shear is transformed to lattice rotation by the presence of lattice regions which oppose to slip, Fig. 21 (b). Therefore, the sense of rotation of the lattice will follow the sense of shear at the band, as confirmed by experimental observations.

**Fig 21** Mechanisms of deformation-induced lattice curvature observed for the investigated material

The induced lattice curvature can extend across the grain boundary. In the extreme case, the incompatible deformation would be accommodated solely by curvature of the lattice in the adjacent grain; this would explain the “burst” of lattice curvature induced by the bands hitting grain boundaries in Fig. 13 and 17.

In the second mechanism, incompatible deformation is accommodated by the activation of secondary slip Fig. 21 (c). An equivalent incompatible deformation between the adjacent grains could be accommodated by the lattice rotation that
follows the activation of secondary slip. Moreover, secondary bands are required to be of opposite nature with respect to primary bands, as depicted in Fig. 21 (d). This was as well observed experimentally. Although the two mechanisms lead to an equivalent GNDs content, the two micro-mechanisms might lead to different level of stored energy. Comparing the lattice rotation gradients in grain 4 and grain 12 provides evidence of this. In the right-side boundary in grain 4, where fading of primary bands is observed, there is a smooth variation in lattice orientation. In contrast, at the outskirt of grain 12, where secondary bands were found, there is a sharp variation in lattice orientation, as evidenced in Fig 18 (d). Hence, lattice curvature is, in case of activation of secondary slip, accomplished in a more limited region. In this latter case, GNDs are therefore more likely to arrange as to lower the elastic energy stored by the distorted lattice. If the contribution to the energy stored by SSDs at secondary bands is neglected, the activation of secondary systems is expected to be favored. Regardless of this, it is believed that both accommodation mechanisms contribute to lowering the level of stresses induced by the incompatible deformation of adjacent grains [5]. The ability of the material to allow the progression of such mechanisms has therefore important implications in the study of strain hardening and damage in such alloy.

8 Conclusions

Testing crystal plasticity models prediction on the evolution of microscale plastic deformation solely by comparison with EBSD measurements appears inappropriate. This is because infinite numbers of plastic deformation paths can be imagined that would induce equivalent lattice curvature. Hence, it was envisaged a more widespread use of deformation mapping techniques such as digital image correlation (DIC). In the present study, we used high-resolution DIC (HDIC) to study the deformation of an austenitic stainless steel at the microstructural scale. Microbands appeared aligned with {111} plane traces obtained from the analysis of electron backscatter diffraction (EBSD) data. The direction of microbands was therefore taken as the main (in-plane) direction of local lattice slip. It was shown that, in regions covered by a single group of bands, which are therefore considered to experience single slip, it is possible to separate the contribution of lattice rotation from the local deformation mapping. To validate the proposed decomposition method, DIC-derived lattice rotations were compared with those measured by EBSD.
Good agreement of results gave, for the first time, direct evidenced of the link between certain non-homogeneous deformation and lattice curvature. We were therefore able to provide unique insights on the kinematic of plastic deformation in austenitic stainless steel. In particular, two competing mechanisms accommodating incompatible deformation between adjacent grains were identified and quantitatively described: lattice curvature and secondary slip activation. Although GNDs are stored as a result of the latter mechanisms, results suggested that the majority of dislocations in a polycrystalline aggregates are stored as SSDs as predicted by prominent authors [28, 38, 39]. In conclusion, we proposed a novel approach to the analysis of HDIC measurements that promises to establish DIC as an essential and unique tool in the study of mesoscopic crystal plasticity.

Acknowledgments

The authors would like to thank Serco TCS for funding. The authors are grateful to Prof. Angus Wilkinson and Prof. Michael Preuss for their valuable comments and discussions.

References


143
33. Salamin E (1979) Application of quaternions to computation with rotations. Working paper available online
35. Altmann SL (1986), Rotations, quaternions and double groups. Dover publications
Paper 4

Paper structured for submission to the International Journal of Plasticity
The results of this work were presented at the symposium “Microstructure Based Property Prediction and Small Scale Experimental Validation” held in October 2012 in Pittsburgh, US.

Contributions of the author: the author of the present study has developed the method to derive lattice rotation from DIC measurement and has performed the analysis that has been presented. PhD student Mr. Ko L.C.L. conducted the experiments. The study was completed under the supervision of Dr. João Quinta da Fonseca.
Characterization of crystal plasticity and dislocation density evolution from high-resolution displacement mapping: particle deformation zone

F. Di Gioacchino, L. C. L. Ko, J. Q. da Fonseca

Materials Science Centre, The University of Manchester, Manchester, UK

Corresponding author:
João Quinta da Fonseca
Materials Science Centre, The University of Manchester, Grosvenor Street, Manchester M1 7HS, UK
Joao.fonseca@manchester.ac.uk
Tel: 0044 306 4844, Fax: 0044 306 3586

Abstract

High-resolution digital image correlation (HDIC) of FEG-SEM images is used to map the deformation of an aluminum matrix surrounding a hard silicon particle. A novel method for HDIC data analysis is used to separate the contributions to plastic deformation of lattice rotation and lattice slip. The mapping of lattice rotations is used to plot the net Burgers vector field describing the necessary dislocation state. The mapping given by lattice slip is used to characterize the distribution of statistically stored dislocations. Results provide unique insights on the kinematic of crystal plasticity at the particle deformation zone (PDZ). It is therefore demonstrated that HDIC is an essential experimental technique in the study of microplasticity.

Keywords: geometrically necessary dislocations, statistically stored dislocations, particle reinforced materials, HDIC

1 Introduction

Dislocation structures in deformed crystals have been widely characterized by transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD) observations. However, there is little quantitative information on the plastic deformation associated with their formation.
Recently, digital image correlation (DIC) of images acquired using SEM has been successfully used to measure plastic strain in metals with microscale resolutions [1 - 13]. The authors of these studies were able to observe transgranular deformation bands [3 – 5, 7, 12] and yet addressed no evidence of plastic deformation that could be related to the formation of discrete dislocation structures.

The size of dislocation cells and subgrains or the spacing between slip bands are usually seen to be in the order of fractions of a micron to few microns [14 - 16]. It is therefore not surprising that multiple measurements per square micron are needed to capture the evolution of such microstructural features.

The resolution of DIC measurement primarily depends on the density of speckle in the pattern applied on sample surface, which is required to make each subregion of the surface identifiable during deformation. More details on the DIC technique can be found elsewhere [17]. Di Gioacchino and Quinta da Fonseca used remodelling of gold films to produce dense nano-scale patterns on an austenitic stainless steel substrate [18]. This enabled plastic strain mapping with sub-micron resolution (HDIC). The same authors proposed a simple method to separate the contributions to deformation of lattice slip and lattice rotation from HDIC measurements [19]. In the present work, HDIC is used to map the deformation of an aluminium matrix around a hard silicon particle and the aforementioned method adopted to study the evolution of dislocation structures.

Studying particle deformation zone (PDZ) is of particular interest as the soft-matrix/hard particle system represents an elementary setting for the development of incompatible plastic deformation. Unlike what is seen in the study of deformation incompatibility at grain boundaries, information on the orientation relationship between the matrix and particle lattice is in fact not required as the latter is generally considered to be non-deformable. [20, 21].

Since the early work of Ashby [22] and Brown [23] on particle-reinforced materials, several elementary dislocation arrangements have been imagined that would accommodate prescribed incompatible deformations developing at the PDZ. Recent TEM and EBSD studies have nevertheless revealed unexpected lattice orientation gradients (lattice curvature) and more complex dislocation patterns evolving around hard particles. In particular, Humphreys and co-workers [24 - 26], Konrad et al. [27], Xu et al. [28] and Karamched and Wilkinson [29] have reported plumes of distinctive texture departing from the matrix-particle interface. Example of TEM micrographs of Al-Si cold rolled to different amount of reduction is shown in Fig. 1 as reported in the work of Humphreys et al. [25]. The authors observed alignment of dislocation cells along a specific \{111\} plane trace, Fig. 1 (c), and dislocation patterns with a width of
about 2-3 µm extending along the direction normal to such trace, Fig. 1 (a), (b) and (c).

Fig 1. Transmission electron micrographs from sections parallel to the transverse plane from Al-0.8 Si. (a) and (b) after cold rolling to a reduction of $\varepsilon = 0.11$. (c) after cold rolling to a reduction of $\varepsilon = 0.29$. Images show alignment of cell walls along a {111} plane trace and high-density dislocation patterns aligned perpendicular to this plane at the particle (P). Courtesy of Humphreys FJ

1.1 Dislocation state from plastic deformation incompatibility

In order to understand the kinematic of formation of such dislocation patterns, it is required to establish a link between the dislocation state of the lattice and its deformation.

Pioneer studies by Nye [30], Bilby [31], Kondo [32] and Kroner [33] have been dedicated to mathematically describe the dislocation state as a function of plastic deformation.

Several expressions have since been proposed [34 - 36]. These are based on the kinematic representation of elastoplastic deformation of single crystal proposed by Asaro and Rice [37], which is represented in Fig. 2. Following Lee’s multiplicative decomposition of the deformation gradient $\mathbf{F} = \mathbf{F}^e \mathbf{F}^p$ in its elastic $\mathbf{F}^e$ and plastic part
\( \mathbf{F}^p \) [38], \( \mathbf{F}^p \) maps the material point \( X \) in the initial configuration \( \mathcal{B}_0 \) to an intermediate (imaginary) stress-free configuration \( \mathcal{B}_i \), which is achieved by crystallographic slip only. The slip is assumed to leave the lattice vectors \((s,m)\) unchanged and unrotated [39]. It follows that the elastic part, which maps the material points in intermediate configuration to the points \( x \) in the current configuration \( \mathcal{B} \), is given by elastic stretches and lattice rotations.

Fig 2 Kinematic model of elastoplastic deformation of a single crystal

Unlike the deformation gradient \( \mathbf{F} \), which is the gradient of a vector field, i.e. \( \text{Curl}\mathbf{F} = 0 \), \( \mathbf{F}^p \) and \( \mathbf{F}^e \) act as local deformation gradients and are generally incompatible, i.e. \( \text{Curl}\mathbf{F}^p \neq 0 \) and \( \text{Curl}\mathbf{F}^e \neq 0 \). Volume elements in the intermediate configuration can therefore detach or compenetrates. Cermelli and Gurtin [36] demonstrated that both the terms \( \text{Curl}\mathbf{F}^p \) and \( \text{curl}\mathbf{F}^{e-1} \), with \( (\text{Curl}\mathbf{F}^p)_{ij} = e_{irs} \frac{\partial F^p_{is}}{\partial x_r} \) and \( (\text{curl}\mathbf{F}^{e})_{ij} = e_{irs} \frac{\partial F^e_{is}}{\partial x_r} \), are equivalent measures of the incompatibility of plastic deformation and that these quantities can be related to the dislocation state of the material in the form:

\[
\mathbf{G} = (\mathbf{J}^p / \mathbf{F}^p)\text{Curl}\mathbf{F}^p = \mathbf{J}^e\mathbf{F}^{e-1}\text{curl}\mathbf{F}^{e-1} \tag{1}
\]

Where \( \mathbf{J}^p = \det \mathbf{F}^p \) and \( \mathbf{J}^e = \det \mathbf{F}^e \). The terms \((\mathbf{J}^p / \mathbf{F}^p)\) and \( \mathbf{J}^e\mathbf{F}^{e-1} \) refer to the mapping of the area \( \Pi \) between different configurations [36].
The tensor \( \mathbf{G} \) is termed geometric dislocation tensor as the quantity \( \mathbf{G}^\top \mathbf{n} \) provides a measure of the local Burgers vector in the intermediate configuration given by the dislocations piercing the plane \( \Pi \) with unit normal \( \mathbf{n} \):

\[
\mathbf{b}_{\text{net}} = \int_\Pi \mathbf{G}^\top \mathbf{n} \, d\Pi
\]  

(2)

Because of their necessary nature, these dislocations are termed geometrically necessary dislocations (GNDs).

For a rigid-plastic material, the displacement of material points given by elastic stretches of the lattice can be neglected. The mapping \( \mathbf{F}^\varepsilon \) would therefore describe a lattice rotation and take the form of a rotation matrix \( \mathbf{R}^\varepsilon \); \( \mathbf{R}^{\varepsilon^{-1}} \) when described in the deformed configuration [36].

1.2 The length scale and SSDs

The measure of strain incompatibility depends on the dimension of the volume element for which quantities in expression (1) are measured. Adjacent volumes of material, A and B, could in fact experience equivalent strain incompatibility of opposite nature, that is \( \mathbf{b}_A = -\mathbf{b}_B \). As the necessary dislocation state of the volume \( A \cup B \) is also defined as the sum of the single Burgers vectors of the dislocations comprised in the volume, it follows that its necessary dislocation content would be null:

\[
\mathbf{b}_A + \mathbf{b}_B = \mathbf{b}_{AUB} = 0
\]  

(3)

It is therefore clear that the characterization of the GNDs content varies depending on the length scale at which the deformation is characterized and therefore on the resolution of the DIC measurements.

A volume can therefore appear to deform “homogeneously” at a certain scale of investigation, yet its parts might be deforming in a non-compatible fashion with consequent storage of dislocations, which are GNDs at the smaller scale. As expressed by (3), the lattice curvature would appear to be zero at the investigated scale. This prevents the characterization of the dislocation content by simple geometrical arguments, as seen for GNDs [40, 41]. Nevertheless, it is reasonable to consider the density of such dislocations to be proportional to the extent of crystallographic slip, i.e. related to \( \mathbf{F}^\varepsilon \). It is in fact plausible to assume that the more a volume undergoes plastic deformation, the more its subvolumes are likely to
experience incompatible deformation and therefore to contain dislocations (GNDs) at smaller scales. Due to their statistical nature at the scale of observation, these dislocations are commonly referred as statistically stored dislocations (SSDs).

2 Characterization of crystal plasticity from high-resolution displacement mapping

DIC allows measuring the displacement of material points at the surface of the strained sample; therefore, the technique can only be used to derive the in-plane components of the quantities in expression (1). It follows that it is only possible to obtain a partial measure of the in-plane component of \( b \), will be hereafter indicated as \( b_{\text{par}} \) \cite{30}. This is the part associated with the in-plane component of lattice curvature.

The following section summarizes the description of the method of HDIC data analysis proposed in \cite{19} used for deriving the in-plane components of \( F^e \) (\( R^e \)) and \( F^p \). The discussion is extended to include the characterization of SSDs.

2.1 Calculation of \( F^e \)

Under certain assumptions, it is possible to identify a simple relation between the displacement vector field and expected lattice curvature. For this purpose, we recall an intuitive 2D representation proposed by Fleck in \cite{42}.

Let three adjacent volume elements to shear following slip along the direction \( X_1 \) by an amount \( \gamma_1 = \gamma_1(X_1) \) such that the intermediate configuration would look like depicted in Fig. 3 (b). The shear can be obtained by lattice slip carried by dipoles dislocations arranged as depicted. The component \( F^p_{12} \) varies according to the coordinate \( X_1 \) such that \( F^p_{12} = \gamma_1 \). Joining the elements back together would require a rotation \( \varphi_3 \) with respect to the out-of-plane direction and a displacement \( u = u(X_1, X_2, 0) \) such that:

\[
\varphi_3 = u_{Z1} = \gamma X_1 \quad \text{with} \quad X_1 \equiv s
\]

Therefore, the rotation of each element expressed by the rotation matrix in Fig. 3 coincides with a lattice rotation. Equivalent results would be obtained if the elements stretch along the \( X_1 \) direction of an amount that varies with \( X_2 \).
If we now want to compare the lattice orientation predicted by DIC analysis to the actual lattice orientation as would be measured after deformation, we have to consider that the volume elements displace as shown in Fig. 3(c). In order to plot the lattice orientation in their current position, it is required to calculate the displacement field that maps the deformed configuration to the undeformed one, i.e. $\mathbf{u}' = \mathbf{u}'(x_1, x_2)$. In analogy with expression (4), it gives:

$$\varphi_3 = -u'_{2,1} \quad \text{with } x_1 \equiv s \quad (5)$$

At the elements interface some dislocations of opposite nature will annihilate whilst other will remain stored and accommodate the lattice curvature. Differentiating equation (5) along the principal directions gives the accessible components of the net Burgers vector $b^\text{par}$:

$$b^\text{par}_1 = \varphi_{3,1} = -u'_{2,11} \quad b^\text{par}_2 = \varphi_{3,2} = -u'_{2,12} \quad \text{with } x_1 \equiv s. \quad (6)$$

In the present study, dislocations will be schematized following the Burgers circuit convention as shown in Fig. 4.

![Fig 3 Schematization of the link between displacement field and lattice curvature in condition of single slip as theorized by strain gradient plasticity](image)
Fig 4 Schematization of the convention used for plotting in-plane lattice rotations and associated net Burgers vector field

Calculation of $F^p$ and SSDs description

Once the rotation matrix $R^e$ has been obtained using expression (5), it can be applied to the deformation gradient tensor $F$ to calculate $F^p$:

$$F^p = F^{-1}e F = R^{eT}F$$  \hspace{1cm} (7)

From the assumption of $x_1 \equiv s$, it follows that $F_{21}^p = 0$.
As indicated, there are no geometrical arguments to characterize the content of SSDs.
Here, we assume the SSDs content to be proportional to the local amount of maximum plastic shear strain $\varepsilon^{p}_{12(MAX)}$, which evaluated from the local plastic strain tensor $\varepsilon^p$:

$$\varepsilon^{p}_{12(MAX)} = \sqrt{\left(\frac{\varepsilon^{p}_{11} - \varepsilon^{p}_{22}}{2}\right)^2 + \varepsilon^{p}_{12}^2}$$  \hspace{1cm} (8)
2. 2 Identifying the direction of slip using microbands

In order to calculate $\varphi_3$ in expression (2), it is necessary to first assume a direction of slip. At the microscale, is reasonable to take the direction of microbands as the “mesoscopic” direction of slip $s$ as shown in [19], (step 1 in Fig. 5). In commercially available software for DIC analysis, as the one used here [43], correlation points lay on a grid with principal directions parallel to the sides of the correlated images. Hence, images can be rotated to make one of the two principal axes of the correlation grid coincide with the direction $s$ (step 2 in Fig. 5). The unstrained image is thus correlated to the deformed one to obtain the displacement field $\mathbf{u'} = \mathbf{u'}(x_1, x_2)$.

![Fig 5 Schematization of the step needed to meet the alignment of the slip $s$ with the grid of the DIC software](image)

3 Application to study PDZ: material and experimental methods

The material studied is a high purity Al-Si alloy. In order to produce micro-sized equiaxed particles as those observed in [25] and reported in Fig. 1, the alloy was annealed at 490°C for 120 hours and slow cooled at 1°C/hr to room temperature. A cube shape sample of 5 mm dimension was then machined for compression testing. A side was polished using 1:10 diluted OPS solution for 10 minutes and then coated with 50 nm layer of gold using a Edwards S150B sputter coater. Hence, the gold layer was remodeled following the gold remodelling procedure described in details in [18]; this produced a nano-scale high-density speckle pattern and enable HDIC. A
series of micro-indents were created on the pattern to act as fiducial marks and allow quick identification of the region. The sample was then placed in the SEM stage for image acquisition of the undeformed pattern. In such region, a particle of about 10 \( \mu m \) diameter was identified and chosen for image acquisition. Images were obtained using a scanning electron microscope (FEI Sirion FEGSEM) in backscattered electron imaging (BEI), details of imaging conditions are reported in table 1. The sample was removed and the compressed with the loading direction parallel to the side of interest. The image acquisition was repeated at different stages of deformation.

**Table 1** SEM scan parameters used for image acquisition

<table>
<thead>
<tr>
<th>Imaging mode</th>
<th>ACC. Voltage (kV)</th>
<th>Beam Current (nA)</th>
<th>Working distance (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BEI</td>
<td>22</td>
<td>1.1</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Linescan (msec)</th>
<th>Magnification</th>
<th>Image size (pixels)</th>
<th>Spatial resolution (nm/pixel)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>2000×</td>
<td>4134 × 2904</td>
<td>( \cong 10 )</td>
</tr>
</tbody>
</table>

Images of the speckle pattern surrounding the hard particle acquired after 0%, 10% and 15% macroscopic compression are reported as processed by the DIC software in Fig. 6 (a), (b) and (c). The gold particles appear bright in the dark background of the metal surface. Bands of marked pixel intensity variation are visible in the deformed pattern. Although backscatter electron imaging is less sensitive to topographic features than secondary electron imaging, it can be seen that these bands are clearly a result of sharp out-of-plane displacement gradients developing during deformation. If these images were correlated with the undeformed image, the change in the pixel intensity of the pattern would locally lower the correlation measure and lead to unreliable displacement mapping. In the present study, correlation is thus performed between the deformed pattern in Fig. 6 (b) and (c). Excellent correlation is achieved as the change in pixel intensity due to out-of-plane deformation is less pronounced. Preliminary results on the analysis of the displacement field (step 1 in Fig. 5) indicate that the bands have a strong shear component associated with their formation. For this reason, their direction is taken as the main direction of lattice slip. As depicted in Fig. 5 step 2, both images are rotated to make one of the principal directions (\( x_1 \)) coincide with the direction of slip \( s \). In particular, whilst the deformed pattern is rotated accordingly, the DIC software is used to rotate the initial pattern as to maintain a region \( O_m \) away from the particle fixed during deformation.
An interrogation window of $32 \times 32$ pixels was adopted for calculation, which corresponded to a spatial resolution of $0.3 \times 0.3 \, \mu m^2$. Numerical Python [44] was used to develop routines for the analysis of the displacement vector field and for plotting the results.

![Backscatter electron images of the investigated area at 0% (a), 10% (b) and 15% (c) macroscopic compression.](image)

**Fig 6** Backscatter electron images of the investigated area at 0% (a), 10% (b) and 15% (c) macroscopic compression. Image acquired after 20% compression rotated to align the horizontal direction along the direction of slip (d). $O_M$ represents the region of the matrix fixed with respect to the observer.

**4 Results**

**4.1 Mapping of in-plane lattice rotations**

Fig. 7 shows the derived in-plane component of lattice rotation following deformation from 10% to 15% compression as seen by an observer fixed with the lattice in $O_M$. The map is intentionally left rotated as to keep the slip direction parallel.
to the horizontal direction. Positive values in red color range describe clockwise lattice rotation whilst negative values in the blue range describe counter-clockwise rotations. The latter coincides with the sense of shear of the aluminium matrix.

Two sets of plumes are identifiable. These show point symmetry with respect to the particle center and extend perpendicular to the slip direction. In particular, the red plumes given by clockwise lattice rotations spread vertically from the region at the sides of the particle labeled C in Fig. 7 (a). Here, compressive stresses are expected according to the sense of shear depicted in figure using white arrows [41]. Lattice rotation reaches about 4° close to the particle and decreases gradually towards the edges of the plumes. The blue plumes originate instead at the top and bottom sides of the particle, i.e. at the particle/matrix interface parallel to the direction of shear. Here, counter-clockwise lattice rotations of about 4° are calculated. As seen for the red plumes, values decrease gradually away from the particle. A counter-clockwise rotation of 2.1° is also measured for the particle.

In Fig. 7 (b), results are used to outline the in-plane lattice curvature induced by the presence of the particle following the 5% compression stage. The dots delineate the interface between red and blue plumes where the no lattice rotation should be measured by the observer. The schematization gives a first glimpse on the link between the shear and the nature of the plumes.

Another area of particular intense lattice rotation highlighted is evident at the bottom-left corner of the map in Fig. 7 (a); this feature is not investigated further in the present study.

![Fig 7(a) Predicted lattice rotations induced by the accommodation of incompatible deformation between matrix and particle. The square highlights the region for which the lattice curvature is depicted in Fig. 7(b)](image)
4.2 Calculation of $\mathbf{F}^p$ and estimation of SSDs distribution

Once $\mathbf{F}$ is calculated and $\mathbf{F}^e$ obtained from expression (5), $\mathbf{F}^p$ can be derived from expression (7).

Values of the component $\mathbf{F}^p_{12}$ are shown in Fig. 8 (a). As discussed, this describes the shear of the volume in the intermediate configuration due to lattice slip only. The presence of microbands of lattice slip is revealed. Positive values indicate that the sense of shear given by lattice slip is equal to the sense of shear strain calculated from $\mathbf{F}$, again depicted in the map using white arrows. The most intense bands reaching values of 0.1 appear in correspondence of the deformation features identified in Fig. 6. In particular, two intense bands are seen to travel along the top and bottom edges of the particle. Additional bands of lower intensity are visible which are not evident in the correlated images. Band spacing also appears to vary. Although the evaluation of the actual spacing requires information on lattice orientation, this is expected in the order of few microns.

Average values for the rectangular subregions in Fig. 8 have been calculated to better describe the variation of lattice slip across the PDZ. Values are reported in the graph in Fig. 9. These indicate that the lattice slip increases at the sides of the particle along the slip direction whilst drops in correspondence of the particle.

It is instructive to compare the values of $\mathbf{F}^p_{12}$ with those of $\mathbf{F}_{12}$, which can be given by rigid rotation of the volume as well as shear. In contrast to what observed for $\mathbf{F}^p_{12}$, average values of $\mathbf{F}_{12}$ calculated for the same subregions are constant and equal to about 0.05, Fig. 9. If the component were given only by lattice rotation, the value would indicate a counter-clockwise rotation of about 2.8 degrees, which is about 0.6 degrees higher than observed particle rotation. This indicates that the particle oppose to the rotation imposed by the matrix deformation. The magnified region shows that more intense bands at the top and bottom of the particle balance low values in correspondence and at the sides of the particle.

The component of compressive strain along the direction normal to slip for volumes in the intermediate configuration $\mathbf{F}^p_{22}$, show similar band features to those described for $\mathbf{F}^p_{12}$, Fig. 10 (b). The apparent in-plane compression is nevertheless believed to result from the out-of-plane slip.

Plotting the strain component along the slip direction $\mathbf{F}^p_{11}$ gives indication of the actual distribution of $\mathbf{F}^p_{22}$ values, Fig. 10 (c). Values appear more homogeneously distributed as bands are not visible. High values are nevertheless observed at the top and bottom edges of the particle.
Fig 8 (a) Derived values for the component of the tensor $F_{12}^p$ describing the shear given by lattice slip. (b) Measured values for the component of the deformation gradient tensor $F_{12}$.

Fig 9 Graph plotting the average values of the components $F_{12}^p$ and $F_{12}$ calculated for the areas highlighted in Fig. 8.
Fig 10 (a) Derived values of compression normal to the direction of slip $x_1$. (b) Values of stretch along the direction of slip $x_1$

Expression (8) is used to obtain information on the distribution of SSDs, Fig. 11. As expected, the highest density of SSDs can be found in correspondence of the slip bands as these are narrow regions where plastic deformation accumulates.

Fig 11 Values of $\varepsilon_{12}^{p,\text{MAX}}$ for the investigated area
4.3 Net Burgers vector field in the particle deformation zone

Fig. 12 shows the result of the net Burgers vector field at the particle matrix interface calculated as shown in Fig. 4 using expressions in (6). Each vector represents the sum of the partial Burgers vectors associated with in-plane lattice curvature of each dislocation within unit areas of about $0.5 \times 0.5 \ \mu m^2$. Vector scaling is intentionally set as labeled to make vectors visible.

The Burgers vector flux in the proximity of the particle presents several distinct patterns. Most notably, Burgers vectors located along the sides of the particle show a common direction towards the centre of the particle and a magnitude which increases from about $1 \cdot 10^{-3} \ \mu m$ in A and C to $1 \cdot 10^{-2} \ \mu m$ in B and D. In addition the field is seen to diverge from the center area in B and D. This indicates that GNDs of opposite nature are here present within a short range.

Finally, a less ordered layer of Burgers vectors can be detected above the particle in correspondence to the slip band observed first in Fig. 8.
Details of net Burgers vector field associated with in-plane lattice curvature at the top and bottom sides of the particle

4.4 Net Burgers vector field at plumes and dislocation cell structures

The Burgers vector field in correspondence of the plumes described before represents a further area of interest. Fig. 13 shows the field at the top and bottom region of the particle. Group of most intense Burgers vector can be identified forming column features of about 2 µm width. These have been evidenced within red dashed contours and appear to be slightly curved following the sense of shear. The enclosed Burgers vectors form a uniform flux, which appears normal to the dashed fronts. The magnitude and similar direction of the net Burgers vectors indicates that the two regions contain high GNDs dislocation density. Vertically oriented features of aligned Burgers vectors as the one highlighted in Fig. 13 have been found in other regions across the field. These forms a net with the GNDs left by plastic deformation at microbands, as evidenced by plotting the magnitude of the Burgers vectors with a threshold of $6 \cdot 10^{-4}$ µm, Fig. 14. Such net resemble the cells structure observed in the TEM images in Fig. 1.
The value $1 \cdot 10^{-4} \mu m$ is the order of magnitude of the noise in the net Burgers vector calculation. Evidently, the latter depends on the noise DIC measurements, which is amplified by the two differentiation steps given by relation (5) and (6). It has been demonstrated that the noise has a strong dependence on imaging conditions and that it decreases with larger interrogation windows sizes [17]. Moreover, it is to be considered that deformation features, such as slip lines, are particularly evident at high magnification, so that the correlation measure and therefore the accuracy of HDIC measurements cannot be easily addressed. A systematic study of noise is beyond the scope of the present work.

![Diagram](image)

**Fig 14** Magnitude of the net Burgers vector associated with in-plane lattice curvature in gray scale and reduced range of values to evidence GNDs patterns. DP1 and DP2 locate the patterns shown Fig. 13

### 5 Discussion

The results provide a complete description of the kinematic of deformation that the aluminum matrix experiences in the proximity of harder silicon particles. This is because HDIC measurements can be analyzed to extract information not only on lattice rotation and therefore on GNDs content, which can be otherwise straightforwardly measured using EBSD, but also to measure lattice slip. Observation of the discretization of lattice slip gives a first argument for discussion. The spacing between slip bands can explain the development of lattice curvature at
the particle/matrix interface for increasing particle dimension as observed experimentally for the investigated material [25 - 27]. In these studies, the lattice is seen to rotate only for particles with diameters of the order of microns, i.e. for particle of diameter equal or bigger than the slip band spacing observed in the present work. Particles of such dimension can be expected to perturb slip and induce slip gradients and lattice curvature. Conversely, in the presence of nanoscale particles, slip bands can distribute such as to minimize the interaction with the particles, Fig. 15 (a), see caption for description. The low dislocation density observed around the latter [25] can be attributed to the need of accommodating different types of matrix deformation, such as long-range elastic lattice stretch. Results of the present study also show that the particle rotates less than the rotation associated with the extent of shear of the matrix, i.e. the particle oppose to the rotation. As a result, gradients of slip along the slip direction appears to develop at the compressive sides of the particle, Fig. 15 (b). Such deformation incompatibility is accommodated by lattice curvature.

Fig 15 Schematization of the interaction between hard particles embedded in a softer matrix shearing of a prescribed amount \( \Gamma = 4\gamma_0 \) which is equally distributed in four slip planes. (a) Particles of dimension order of magnitude smaller than the slip spacing do not interfere with the slip. (b) Particle of dimension of the order of the band spacing cause it to interfere with the matrix shear deformation.

Fig. 16 (a) shows a representation of the intermediate configuration at the right side of the particle from the values of \( \mathbf{F}_{12}^P \) in Fig. 8 (a). As for the example depicted in Fig. 3, it is possible to join the volumes together and predict the nature of lattice rotation, Fig. 16 (b). The matrix at the particle interface and the particle will be seen to respectively rotate in a clockwise and counter clockwise sense. Furthermore, the increase in slip gradient towards the edge of the particle will make the magnitude of the net Burgers vectors increase accordingly as described in Fig. 12.
GNDs can be here imagined to harden the matrix therefore extending the volume of non-deformable material along the direction normal to the shear sense. This can explain the evolution of the plumes and the dislocation patterns described previously in the TEM micrographs reported in Fig. 1.

Fig 16 Schematization of the lattice slip distribution in the particle/matrix interface. (a) Intermediate configuration ($P^P$). (b) Deformed configuration following strain gradient induced lattice rotations

6 Conclusions

In the present study, we used the method for HDIC data analysis proposed in [25] to characterize the crystal plasticity of an aluminium matrix around a non-deformable silicon particle of microscopic dimension. Once the direction of slip has been individuated, the deformation gradient tensor was decomposed in its elastic part and plastic part to the in-plane component of lattice rotation and lattice slip, respectively. Results of lattice rotation mapping and the derived the Burgers vector field around the particle showed to agree with EBSD and TEM observations in previous studies. The analysis of HDIC measurements also demonstrated that, unlike the latter microscopy techniques, DIC has the unique ability of providing access to the intermediate relaxed configuration of lattice deformation, which is given by lattice slip.

For the investigated PDZ, we were able to show only using kinematic arguments that the hard silicon particle opposes to the rotation exerted by the shearing of the aluminum matrix. Such resistance was identified as the mechanism inducing the peculiar plume-shaped lattice curvature and dislocation arrangement observed in early microscopy studies.
It was finally suggested that the extent of lattice curvature as a function of particle dimension (size effect) observed for this material can be attributed to the localization of strain at microbands. The present and future HDIC observations might thus help to better understand the nature of material length scale in strain gradient plasticity.

Acknowledgments

The authors would like to thank Serco TCS for funding. The authors are grateful to Prof. Angus Wilkinson and Prof. Michael Preuss for their valuable comments and discussions.

References


7 Concluding remarks and future work

In recent years it has become evident that the mechanical behavior of polycrystalline materials cannot be solely explained through arguments on the motion of dislocations found in dislocation theory textbooks. Observations on localization of plastic deformation across multiple length scales suggest that there exists a link between microstructural heterogeneity and multi-range stress fields, which therefore has a crucial role on determining material properties. The mechanisms of such interplay are not fully understood and mostly subject to speculation.

The efforts in developing increasingly sophisticated microstructure-based crystal plasticity models have not been paralleled by drives to improve relevant experimental characterization methods. Most predictions on the mesoscale evolution of plastic deformation have been solely supported by comparison of the predicted deformation-induced lattice curvature with corresponding electron backscatter diffraction (EBSD) measurements.

Treatments on micromechanics of crystal plasticity consider lattice curvature as a deformation mechanism required to accommodate the incompatibility in lattice slip. As discussed in more details in Chapter 4 and Chapter 5, paper 3, such incompatibility is expressed as \( \text{Curl} \mathbf{F}^p \), with \( \mathbf{F}^p \) being the intermediate configuration given by crystallographic slip only. Hence, an infinite number of plastic deformation paths and intermediate configurations can be imagined, which would give rise to equivalent strain incompatibility and consequently lattice curvature. Existing crystal plasticity models can thus be calibrated to provide good prediction on the progress of lattice rotation and yet still fail to appropriately describe the kinematic of plastic deformation.

Deformation mapping has the obvious advantage of being able to directly describe and quantify deformation. Yet, limited use of full-field displacement mapping techniques has been made to validate predictions on micro and grain scale (mesoscopic) deformation. This is possibly due to the experimental difficulties faced when trying to achieve sufficiently high-resolution measurements.

7.1 Concluding remarks

7.1.1 Speckles distribution in gold remodelling

Gold remodelling has an evident disadvantage over alternative pattern application methods because it requires high temperature exposure of the material substrate. Although remodelling could be performed in inert or reducing environment to avoid
the deleterious effect of the oxide grow, high temperature might still affect the integrity and the microstructural characteristics of the specimen. Others pattern application methods appears advantageous, in particular, the deposition nano-particles of heavy element such as gold or platinum. Such particles are usually seen to stick to the surface and form speckles that give good signal to noise ratio, in particular, when imaged using backscatter electrons. It is therefore not clear why studies using pattern of comparable speckle density have instead used subsets of microscopic dimension and a relevant amount of subsets overlap.

The author of the present study believes that an important factor affecting the correlation measure and the accuracy of strain measurements is given by the spatial distribution of the speckles. This aspect has not been investigated enough in the literature.

Particles deposited using sputtering methods distribute randomly on the surface of the specimen. A random distribution yet results in are of the specimen not covered by speckles and other areas where accumulation of the particles have formed larger speckles which affect the correlation as described in Fig. 2.8. The remodelling of thin film gives homogeneously distributed speckles. Given a same speckle density, this would allow both the use of smaller subsets, i.e. a higher spatial resolution, and more accuracy of DIC measurements. Therefore, pattern application methods based on nanoscale rearrangement processes such as remodelling of thin films are expected to give the highest resolution and the lowest noise in strain values and should thus be preferred.

![Random distribution](image1.png) ![Homogeneous distribution](image2.png)

**Fig 7.1** Comparison between (a) random distribution of speckles and (b) homogeneous distribution. Possible subsets position highlighted in red
7.1.2 Advances in DIC methods and crystal plasticity characterization

An important contribution of the present work is having refined pattern application methods for digital image correlation (DIC) analysis at high magnifications. The experimental methodology has been described in details in the technical paper presented in Chapter 6, paper 1. Performing deformation mapping with submicron resolution does not just give the advantage of more detailed observations. It most importantly gives access to a new scale of observation in crystal plasticity. In particular, the latter is the scale of crystallographic deformation features, i.e. features that can be directly related to the local orientation of the lattice.

The advantage of this has been shown in Chapter 6, paper 3 and paper 4 where crystallographic microbands have been used to identify the direction of lattice slip. This allowed the development of methods for separating lattice slip and lattice rotation in deformed crystals. It also allowed proving, for the first time experimentally, the existence of a link between certain gradients of slip and the lattice curvature.

It is finally noted that further miniaturization of speckles would become beneficial only at the scale of individual dislocations, as this represents the next smaller scale of observation, Fig 1.1. The need for atomic scale patterns might require the development of novel pattern application methods and the adoption of alternative imaging techniques such as TEM or atomic force microscopy (AFM).

7.1.3 The development of incompatible deformation in polycrystals

As expected, HDIC results on stainless steel show that the difference in lattice orientation of grains has a central role in the evolution of deformation incompatibility.

Similar to the distinct length-scale of non-uniform deformation described in Chapter 6, paper 1, three different scales of deformation incompatibility can be identified.

A first submicron scale can be associated to the microstructural heterogeneity, which causes local fluctuation in the intensity of lattice slip in correspondence of microbands.

The second is the scale of individual grains where deformation incompatibility results from the deformation mismatch with respect to surrounding grains. This therefore includes constrains exerted at grain boundary regions where slip is generally inhibited, as discussed in Chapter 6, paper 3. For the tested stainless steel, this results in smooth variations in the intensity of lattice slip along the length of microbands (band fading) or in the activation of secondary slip at grain boundary regions.
The third is a macroscopic scale of deformation incompatibility that can be imagined to result from non-uniform imposed deformation, such as bending. Hence, the uniform imposed deformation associated with a tensile test is believed not to induce long-range strain incompatibility. The transgranular deformation bands observed for stainless steel deformed in tension in Chapter 6, paper 1 are therefore expected not to induce long-range “lattice curvature”, i.e. relevant GNDs density when this is calculated for large volumes. Regardless of this, transgranular deformation bands are seen to greatly influence the extent of incompatible deformation developing at smaller scales. Grains located along these bands deform more and therefore are more likely to develop significant strain incompatibility. This has important implications in the study of damage mechanics. For instance, it indicates that the hot spots of localized deformation observed for stainless steel in Chapter 6, paper 3 cannot be predicted a priori simply from the orientation relationship of adjacent grains, as otherwise suggested in previous studies.

7.1.4 Comments on size effects and material length scale
As mentioned in Chapter 5, size effects have been modeled by introducing material length scales in the constitutive laws. These are usually associated with microstructural features such as grain size, distance between particles in particle-reinforced materials, etc. The underlining idea is that, for an equivalent imposed deformation, the smaller the length scale, the “steeper” is the strain gradient and therefore the higher would be the density of stored GNDs. Results from the study of a particle deformation zone (PDZ) presented in Chapter 6, paper 4 suggest otherwise. Here, the material length scale was individuated in the characteristic microband spacing. Interestingly, such length scale is not clearly associated with specific microstructural features. Moreover, strain localization at microbands is not itself expected to induce lattice curvature. This is because slip gradients develop normal to the direction of lattice slip and are therefore associated to compatible deformation. As seen for the tested materials, deformation incompatibility rises only when microstructural heterogeneity perturbs such discretization of slip. Further investigations are thus envisaged to clarify the mechanisms of microbands formation. As discussed in the following, this requires having a better understanding of the local stress state.
7.2 Including boundary conditions: proposed work

In the present work, studies were limited to the description of the kinematic of plastic deformation with no information on the forces involved and therefore on the local stress state. This is because the length scale of the deformed specimens was several orders of magnitude bigger than that the dimension of the investigated areas. Hence, the deformed volume should be small enough to be entirely covered by the HDIC mapping.

It appears attractive to implement the HDIC into existing experimental procedures developed to plastically deform mesoscale volumes [129] and microvolumes of metals and ceramics [130 - 134]. It can be imagined, for instance, to use a focussed ion beam (FIB) to mill a parallelepiped volume at the surface of the sample and then apply the high-density speckle pattern using gold remodelling. The macroscopic dimension of the sample would facilitate its handling during the deposition and remodelling steps.

Fig 7.2 Illustration of a possible application of nanoscale speckle pattern for studying the micromechanisms of plastic deformation in crystals

Fig. 7.2 shows a possible scenario of the milled volume covered by the pattern in Fig. 2 (f) in Chapter 6, paper 1. The flat surfaces would facilitate DIC data processing without the need of prior geometric transformation. The volume can be deformed by an indenter similarly to the procedure used for compressing micropillars in [131] and
7. Conclusion

the flow stress recorded. During the test, images of the speckle pattern can be acquired along multiple sides of the volume to allow a 3D reconstruction of the deformation mapping, Fig. 7.2. After image acquisition the focused ion beam could be used to remove the gold pattern and enable the acquisition of EBSD data.

Alternative loading configurations can be also imagined as those proposed in Fig. 7.3. These would be expected to induce distinct degrees of incompatible deformation and possibly distinct evolution of lattice slip and lattice rotation. By comparing the latter with the evolution of the flow-stress, it may be possible to isolate the effect of these on the strain hardening/softening. Investigations can be repeated for different lattice orientations and for different volume dimension to obtain insights on size effects. The observation should be sided with CPFEM or CPFFT analysis where boundary conditions can be easily reproduced. Hence, the capability of existing models to predict mesoscopic evolution of crystal plasticity could be tested with unprecedented accuracy. This would possibly spark the development of new class of crystal plasticity models, which promises to give unprecedented insight and understanding of the physics of deformation of crystalline materials.

Fig 7.3 Illustration of three possible nanoindenter positions for inducing deformation incompatibilities in a microvolume
Appendix A

Whilst Python [135] routines for processing DIC data can be developed in a straightforward manner, additional programming skills are needed to develop Python routines for the analysis of EBSD data\(^7\).
The EBSD acquisition software [68] adopted in the present study uses Euler angles to describe the lattice orientation. Hence, it is convenient to generate a second .txt file where each set of Euler angles is replaced by the components of the corresponding quaternion; this avoids repeating the conversion in subsequent calculations. The function reading the new file was designed to build a dictionary of quaternions with keys being the coordinates of the respective points in the map. Following the above discussion, each quaternion was represented as a list of two elements: a scalar value and a numpy.array object describing the vectorial part:

\[ q = [q_0, \text{numpy.array}[q_1, q_2, q_3]] \] \hfill (C1)

Plotting of grain boundaries and \{111\} plane traces

In paper 1, EBSD analysis was used to superimpose grain boundary and \{111\} plane traces on the strain map. In particular, the \{111\} plane traces associated with the maximum value of Schmid factor were highlighted using distinct colors.
The algorithm used to detect grain boundaries is based on the point-to-point calculation of lattice misorientation. When the angle of misorientation exceeds a predefined value, the point is potentially part of a grain boundary. Due to the symmetry of the face-centered cubic (FCC) lattice of stainless steel, the quaternion is multiplied by each of the 24 quaternions describing the symmetry rotations and the misorientation angle calculation repeated. If the minimum angle of misorientation (also referred as disorientation) still exceeds the predefined threshold, the point will be part of a grain boundary. The quaternions describing the 24 symmetry rotations are easily derivable from relation (3.11); a list of these can be found in [88].
The point-to-point scanning process has to be repeated along the principal directions of the EBSD map. This is because consecutive points of a grain boundary oriented parallel to the direction of the scanning will not be both detected as points of a grain boundary, see Fig. A.1.

\(^7\) Excellent introductions to Python language and data plotting can be found in [136, 137].
Fig A.1 Schematization of identification process of grain boundary points in the EBSD map. The points were the black arrows start are those identified as a boundary points. The red arrow indicates mis-indexed points. The mis-indexed point in (a) can be identified by a second scan along a distinct direction (b).

Once the grain boundary matrix was obtained, this was plotted using matplotlib library [117]. Points within the plotted map were manually selected using the ginput function, which returns the point coordinates. The latter were taken as key for the dictionary to retrieve the associated quaternion. The quaternion is used to rotate the (111) vectors and identify the position of the {111} planes in the sample reference. Subsequently, the set of four vectors describing the intersection (trace) of {111} planes and the plan of the map {001} can be easily calculated as the vectorial product of the rotated (111) vectors and the (001) vector. Before plotting the traces, the quaternion is used to identify the position of the rotated slip direction (110) for the respective {111} plane to enable the calculation Schmid factor using the well-known relation. Therefore, the two planes yielding the maximum values of Schmid factor can be individuated. Knowing the relationship between spatial (sample) and Euler coordinates, it is possible to plot the traces onto the grain boundary map and highlight those associated with the maximum Schmid factor planes.

**Grain reconstruction**

Grain orientation spread type analysis showed in Chapter 6, paper 2 and paper 3 are based on the misorientation calculation executed on a grain-to-grain basis. This requires prior grain reconstruction, which means grouping points of the EBSD map...
according to the grain these belongs. This can be achieved by performing a recursive flood fill type algorithm on the grain boundary map previously obtained. The flood fill algorithm adopted in the analysis presented here is designed to scan the map for points that do not belong to a grain boundary. Once the coordinates of a non-grain boundary point are obtained, the algorithm starts building a kernel that expands through the grain while gathering the coordinates of points in a list. The flood fill function thus returns a nested list where the elements are the lists of points within single grains.

In Chapter 6, paper 2, the misorientation calculation at each point is done automatically with respect to the parent grain average orientation. The latter is calculated as the average of the components of the $N$ quaternions for individual grain [115]:

$$ q_i = b_i / |b_i| \quad \text{with} \quad b_i = \left( \frac{1}{N} \right) \sum_{k=1}^{N} q_i^k $$

(C2)

In Chapter 6, paper 3, the calculation of misorientation is instead performed with respect to the lattice orientation of a manually selected point within each grain. Lastly, it is noted that, in both cases, it is necessary to consider lattice symmetry as described above.
References

deformation of steel grains. Comparison with polycrystal models predictions.
Mat Sci Eng A234–236:853–856
Scripta Mater. 42:1053–1058
behaviour of single crystals from the local response of polycrystals. Acta Mater
51: 5477–5488
plasticity in polycrystals. Mat Sci Eng A342:152–168
polycrystalline OFHC copper. Int J Plast 19:1355–1376
measurements and polycrystal finite element calculations for micromechanical
digital image correlation technique and scanning electron microscope imaging. J
Strain Anal Eng Des 43:719–728
strain behavior in polycrystals through in-situ scanning electron microscope
tensile experiments. Metall Mater Trans A 40:2363–2368
characterizing strain localization during deformation at elevated temperatures.
Exp Mech 52:405–416
59. Winiarski B, Shajer GS, Withers PJ (2011) Surface decoration for improving the
accuracy of displacement measurements by digital image correlation in SEM. Exp
Mec 52:793–804
60. Carroll JD et al. (2013) On the interactions between strain accumulation,
microstructure, and fatigue crack behavior. Int J Fract in press
improved digital image correlation in a scanning electron microscope. Exp Mec
in press


References