Abstract. Three-dimensional electron backscatter diffraction (3D EBSD) has emerged as a powerful technique for generating 3D crystallographic information in reasonably large volumes of a microstructure. The technique uses a focused ion beam (FIB) as a high precision serial sectioning device for generating consecutive ion milled surfaces of a material, with each milled surface subsequently mapped by EBSD. The successive EBSD maps are combined using a suitable post-processing method to generate a crystallographic volume of the microstructure. The first part of this paper shows the usefulness of 3D EBSD for understanding the origin of various structural features associated with the plastic deformation of metals. The second part describes a new method for automatically identifying the various types of low and high angle boundaries found in deformed and annealed metals, particularly those associated with grains exhibiting subtle and gradual variations in orientation. We have adapted a 2D image segmentation technique, fast multiscale clustering, to 3D EBSD data using a novel variance function to accommodate quaternion data. This adaptation is capable of segmenting based on subtle and gradual variation as well as on sharp boundaries within the data. We demonstrate the excellent capabilities of this technique with application to 3D EBSD data sets generated from a range of cold rolled and annealed metals described in the paper.

1. Introduction
The ability to observe the structure of an opaque solid in three dimensions (3D) is important for understanding its true nature as it eliminates speculation about the spatial distribution of features associated with conventional 2D imaging techniques. This is an important step in exploring microstructure-property relationships relating to many metallurgical problems, since most practical materials have a complex, polycrystalline structure and most features affecting material properties are three dimensional. There are several 3D analysis techniques for determining the spatial distribution of microstructural features, each with varying resolution and specific capabilities [1]. For example, in situ 3D XRD has emerged as a powerful non-destructive 3D analysis tool for directly analysing microstructural changes subject to heating, loading etc. such as the time evolution of the size and shape of grains during recrystallization [2-5]. Schmidt et al. [2] used the technique to study recrystallization in deformed aluminium single crystals; the work showed that the orientation of new grains can be different to those present in the local deformation substructure as well as the growth fronts of the...
recrystallizing grains being highly irregular, which undermines the classic assumption of smooth and spherical growth of grains during recrystallization [2].

Over the past few years, considerable progress has been made in the development of another 3D technique, termed 3D electron backscatter diffraction (3D EBSD), for understanding various metallurgical phenomena [7-20]. Using a typical DualBeam™ or similar platform, sub-100 nm thick layers of a material can be milled sequentially by the focused ion beam (FIB) and each surface mapped by EBSD (figure 1a). The successive EBSD maps are then combined using a suitable post-processing method to generate crystallographic volumes of the microstructure (figure 1b). 3D EBSD is particularly suited to crystalline materials where the smallest resolvable crystallographic features are ~ 50 nm and encompasses many types of deformation and annealing phenomena.

The authors are using 3D EBSD as a tool for better understanding the geometric and crystallographic features of microstructures generated by deformation and annealing and phase transformation. In our particular 3D EBSD experiments, we achieve EBSD pattern indexing rates for each FIB-milled surface of greater than 85% using a 1nA FIB current for ion milling and 30kV SEM operating voltage. Since the reconstructed data represents a volume of the material, such information is useful for better understanding the mechanisms of formation of these features. For example, a significant structural feature associated with many types of plastically deformed metal is the characteristic subgrain/cellular structures that are generated.

Figure 1. (a) Sequential focused ion beam milling of a sample and concurrent EBSD mapping to generate (b) consecutive EBSD maps. (c) EBSD maps are combined to generate a volume of microstructure up to 100 × 100 × 100 µm$^3$ that can be interrogated further for investigating specific microstructural features (figure adapted from ref. [6]).

FIB/EBSD serial sectioning readily generates hundreds of consecutive layers of material with each EBSD map often containing over $10^6$ data points. Hence, a volumetric data set may easily contain $10^7$-$10^9$ points. While preliminary reconstruction of such a data set may be done using commercial software tools, they do not generate the accuracy needed for low angle grain boundary (LAGB) identification, skew correction, fitting of surfaces or local surface analysis needed for this particular problem. For example, while the misorientation thresholding capabilities of commercial software can be used to determine high angle grain boundaries (HAGBs), subgrain/cell features generated by plastic deformation usually have gradual boundaries with pixel-to-pixel misorientations of a few degrees or less. A novel algorithm for repeatable identification of subgrain boundaries that addresses all of these foregoing issues is outlined in section 3. The algorithm automatically generates both 2D and 3D EBSD data sets that can be scrutinised to further our understanding of the nature of the deformed and annealed states.
2. Application of 3D EBSD to investigating various boundaries in deformed metals and alloys

There are various microstructural features generated by plastic deformation including, for example, cells/subgrains, microbands, deformation bands, shear bands and deformation zones at coarse, non-deformable particles [21]. Such features can have a pronounced influence on recrystallization and subsequent texture development and, hence, on the mechanical and physical properties of metals and alloys [21]. They are often analysed by standard 2D techniques and TEM. This section illustrates how 3D EBSD can be used as a tool for better understanding some key microstructural features associated with cold deformed metals.

2.1. Crystallographic nature of microbands in cold deformed metals and alloys

It is well-known that microbands (MBs) are generated in a wide range of deformed metals and alloys (Fe, Al, Cu, Ni etc.) in processes such as rolling and are recognizable by their characteristic lamellar appearance inclined at 25-35° to the rolling direction (RD) when viewing the normal direction (ND)-RD section (see e.g. figures 2c and d). In recent years there have been major efforts to determine the crystallographic nature of MBs with the aim of modelling their development during plastic straining and subsequent annealing. This has proven to be a challenge as shown by the recent controversy over the nature of their alignments, in particular for Al deformed in plane strain compression. The Risø group [22-24], based on detailed TEM observations, argue that subgrain boundaries or microbands lie mostly on the active \{111\} slip planes in fcc alloys. This is supported by the recent work using EBSD [25]. In contrast, the Manchester group [26-28], using EBSD microtexture analysis, asserts that the deformed microstructures are independent of crystallography and the MBs are aligned with the planes of maximum shear stress.

![Figure 2](image_url)

**Figure 2.** (a) Reasons for using Goss-oriented single crystals in investigations of plastic deformation of metals. (b) Plane strain compression (PSC) rig relative to the orientation of the Goss crystal. (c) 2D EBSD micrograph showing two non-coplanar sets of microbands. (d) Bright field TEM micrograph showing the dislocation structures associated with these microbands. (e) Standard method of FIB serial sectioning and EBSD mapping of our samples for 3D EBSD.
3D EBSD is being used for understanding the crystallographic nature of the surface of MBs in several types of material: cold rolled IF steel (BCC) [29-30]; plane strain compressed Ni (FCC) single crystals of the Goss orientation [31-33], and cold rolled commercial purity aluminium (FCC) [34]. These materials generate classic MBs. The actual analysis of individual MBs in these materials is challenging due to the subtle orientation gradients present in these features and the low angle nature of their boundaries. To address these challenges, an automated method has been developed for delineating the various types of boundaries found in deformed and annealed metals and alloys (see section 3). Serial sectioning and data reconstruction has enabled us to determine the general structure and surface topography of the MBs in these materials. The present section will focus on the formation of MBs in Ni single crystals (figure 2).

![Figure 3](image.png)

**Figure 3.** (a-c) EBSD micrographs of three orthogonal surfaces of the PSC Ni single crystal showing the \(1.5^\circ\) MB boundaries as black lines. (d) Plot of the MB boundary length as a function of misorientation showing that their boundary lengths with higher misorientations are short. In the TD section, the average MB length, for a given misorientation, is twice that of the ND section, after [32].

Figure 3 shows the three orthogonal sections of a 30% PSC Ni crystal, whereby MB traces are classically aligned in the transverse direction (TD) section at an acute angle from the rolling direction (RD), but appear wavy in the ND section. The latter observation may lead to the conclusion that microband boundaries are non-crystallographic. 3D-EBSD was used to reconstruct individual microbands in a deformed volume that was found to reveal significant new information about their structure [32]. Figure 4 shows the microband surfaces to be largely planar over large distances but frequently interrupted by local distortions and undulations due to interactions between intersecting, non-coplanar microbands (circled in figure 4). These findings are supported by detailed TEM analysis of general microband structure and interacting boundaries [31-33]. The combined EBSD/TEM investigation clearly reveals that MB boundaries are aligned close to an active \{111\} slip plane (i.e. they are crystallographic) but the bumps and distortions they contain are non-crystallographic in the sense that they deviate away from an active slip plane. The non-crystallographic features of microbands (as revealed by their wavy structure in the ND section, figure 3b) may be explained by the crystallographic oscillations of up to \(\pm 7.5^\circ\) towards RD that occur during plastic deformation. Such oscillations result in varying fractions of slip on a given \{111\} plane, thereby resulting in varying degrees of interaction.
between the two sets of non-coplanar microbands. These local and intense microband interactions result in their deviation away from their active slip planes.

**Figure 4.** 3D EBSD reconstruction of 25 MB boundaries in the PSC Ni crystal, showing their inclinations and curvatures when viewed from +TD (a) and -TD (b). (c) <111> pole figure showing the plot of the average normal of these boundaries (x symbols) showing their close proximity to the [111] pole of the Goss orientation ((110)[001]). The average normal of the flat segments of the MB boundaries are also plotted from their traces as N₁, N₂ and N₃ (in the shaded ranges), to show their close correspondence with the poles of the potential {111} slip planes, after [32].

2.2. Uncovering highly misoriented fragments at hotband grain boundaries in cold rolled IF free steel

3D EBSD was used to investigate the deformation heterogeneities that form in the vicinity of prior hotband grain boundaries in a 75% cold rolled IF steel [30]. This study focused on the actual structure of the boundary region between two near γ-fibre grains since it is generally reported for IF steels that nucleation of recrystallization takes place in the highly fragmented substructures of these γ-fibre grains [35-36]. Within the γ-fibre grains (<111>//ND), shear bands [36-37] and deformation bands [35, 38] form profusely and these are arguably the principal sites for recrystallization. Nevertheless, considerable nucleation also occurs at the prior HAGBs of the hot band [39].

Figure 5 shows a representative EBSD micrograph from one of the 45 FIB milled sections showing three deformed grains N, O and P. Grain N contains a parallel set of LAGBs inclined at an acute angle to RD. While the total orientation spread within this grain is large (~28°) (inset), the average orientation is close to one of the prominent γ-fibre orientations ([1 1 1][1 2 1]) of the BCC rolling texture. Grain P contains two sets of intersecting microbands at ±20° with RD and its average orientation (inset) is close to another prominent γ-fibre orientation (1 1 1)[0 1 1]. Within grain O, there is a very large orientation spread and the grain is highly fragmented, as revealed by the presence of numerous thick black lines in the EBSD micrograph.
**Figure 5.** (a) EBSD micrograph (and inset showing the corresponding <200> pole figure) showing the orientation spread of deformed grains N, O and P. Here, HAGBs (>15°) and LAGBs (2-15°) are plotted as thick and thin black lines, respectively, after [30].

**Figure 6.** (a) 3D reconstruction of the N/O grain boundary affected region showing the serrated appearance of the microband-containing surface corresponding to grain N and irregular nature of the HAGB corresponding to grain O. (b) 2D EBSD section of the N/O grain boundary and (inset) elongated fragment on the boundary (circled). (c) 3D reconstruction of the actual N/O boundary showing substantial crystal fragments. (d) <200> pole figure showing the orientations of the boundary fragments in (c) superimposed on the local spread in orientations of grains N, O and P (figures adapted from ref. [30]).
Figure 6a shows the 3D reconstruction containing the N/O boundary (yellow line) with figure 6b showing one of the EBSD sections of the reconstructed volume. A close inspection of this boundary (dashed white line) reveals the presence of a thin, elongated fragment (circled in the inset in figure 6b). The actual shape of this feature is almost impossible to reveal in a single 2D EBSD micrograph, and such features can be easily overlooked.

Figure 6c shows the N/O boundary extracted from the volumetric EBSD data set. The boundary contains several thin, elongated fragments (Q, R, S fragments are shown herein but there are others). While these fragments may have small and large orientation variations along their length and slicing directions (see figure 7), their central orientations are plotted in the <200> pole figure in figure 6d, and compared with the orientations of grains N, O and P. The identified fragments sit on the left side of the N/O boundary adjacent to grain O, but their orientations are neither close to any of the orientations within these adjoining deformed grains (figure 6d) nor are they close to the α- and γ-fibre orientations [30]. However, these fragments have an average orientation close to grain P (figure 6d). Hence, it is likely that these boundary fragments have originated from an earlier disintegration event of grain P leaving remnant orientations of this grain as a Q-R-S film on the N/O boundary. The evidence also suggests that grains O and P were actually a single grain that eventually fragmented during deformation. Hence, these fragments do not have orientations within the local orientation spread near the N/O boundary. The present findings are consistent with the 2D EBSD work of Ushioda et al. [40], who reported the occurrence of numerous thin fragments on γ/γ boundaries in their 75% cold rolled IF steel, and these fragments were also found to have different orientations to their adjoining grains.

Figure 7 shows a series of EBSD slices highlighting a thin grain boundary fragment spanning many EBSD slices. This particular fragment has a large orientation variation within the slice length (see relevant pole figure with slice 22 in figure 7a). Identical orientation plots from the subsequent slices reveal (in figures 7b-e) a gradual rotation in orientation in the slicing direction (compare the pole figures associated with slice 22 to 30).

**Figure 7.** Series of EBSD micrographs (with corresponding <002> pole figures) of a thin, elongated fragment at the N/O grain boundary showing a large orientation spread within the length of the fragment and along the slicing direction in slices 22, 24, 26, 28 and 30. In the EBSD maps, the yellow lines represent misorientation boundaries over 5°, after [30].

The steep orientation gradients near HAGBs, deformation-induced interfaces and grain boundary fragments generated in the cold rolled IF steel are all potential sites for nucleation of recrystallization [21]. The steep orientation gradient near the N/O boundary in figure 5 is likely to be an ideal site for recrystallization. The large width of this region within the reconstructed volume (see figure 6a) also
indicates that multiple nucleation events are likely to occur near a grain boundary face, as evidenced by the common occurrence of strings of grains nucleating along prior grain boundaries after deformation and subsequent annealing [30]. The high angle interface generated within grain O near the N/O boundary (figure 5) is also a potential site for recrystallization. Juul Jensen et al. [41], Hansen et al. [42] and Godfrey et al. [43] reported the early growth of subgrains at irregular boundary interfaces and concluded that local variations in texture (orientation) and microstructure (stored energy) play a major role in recrystallization.

Finally, but most interestingly, thin fragments left on the N/O boundary are bounded entirely by HAGBs and, hence, are also expected to be potent recrystallization nuclei. Despite these fragments not having a size advantage in the thickness direction over the average subgrain size of the deformation substructure (0.5 to 1.0 µm) in cold rolled steels [40,44-45]), their orientations are indeed found in low intensities in the recrystallization textures of IF steels. For example, the \{110\}<110> component (close to Q, R and S in figure 6d) is found in small intensities in the annealing textures of cold/warm rolled IF steels, despite these orientations not being identified in the rolling textures [36-38]. Therefore, it is possible that such orientations originate from these types of boundary fragments generated during rolling. Further, the discovery of such grain boundary fragments in the deformation microstructure provides a plausible explanation for the origin of recrystallized grains with orientations other than those found within the adjoining deformed grains in the vicinity of grain boundaries; this phenomenon has been commonly observed in texture data for many years and observed experimentally [21] but remained largely unexplained.

3. An automated method for reconstructing various types of boundaries and interfaces in 3D EBSD data sets

As discussed in section 2, plastic deformation generates complex deformation substructures that have a pronounced influence on subsequent recovery and recrystallization processes [21]. Unlike HAGBs, the variations between subgrain/cell structures are more subtle in magnitude (low total difference in orientation between subgrains) and spatially gradual (low point-to-point differences). This makes the automatic identification of their boundaries challenging and, hence, creates a significant obstacle to generating reliable data of the spatial distribution of these features within the overall deformation substructure. The ability to reliably segment a volumetric EBSD data set into substructure features is important for understanding the mechanisms of substructure formation by plastic deformation through to nucleation of recrystallization, and the overall relationship between microstructure and bulk material properties.

![Figure 8](image.png)

**Figure 8.** Misorientation thresholding of gradual boundaries in an EBSD data set containing microbands in a single grain of a commercial purity aluminium alloy loaded in uniaxial tension to a strain of 33%. (a) IPF colour map. The black points indicate locations of pixel-to-pixel misorientation greater than (b) 1° and (c) 2° (figure adapted from ref. [46]).

The identification of features in a typical EBSD data set may be achieved by grouping points that are not separated by a boundary with misorientation higher than a user-defined threshold. This is a common method in commercial software but fails when applied to subgrain/cell data since there is no threshold angle that identifies the boundaries of interest without being dominated by noise in the sample. As an
example, figure 8 shows thresholding on EBSD data from a deformed aluminium sample containing microbands. Noise also causes problems, as mis-indexed pixels are usually classified as boundaries. In addition, volumetric reconstructions require closed boundaries, something the pixel-to-pixel method does not guarantee.

An approach to solve this thresholding problem is to use an edge-preserving lowpass filter to smooth the data. Humphreys et al. [47] have implemented a Kuwahara filter, which reduces the noise in a data set by statistically analysing neighbourhoods surrounding every point. Each pixel is iteratively replaced with the average of the neighbourhood with the least variance. This technique is capable of reducing orientation noise by approximately a factor of ten, but it may also lead to an oversegmentation of microstructures. Noise and random differences in variation can compound over successive iterations, creating microstructural artifacts, such as boundaries that should not exist. Furthermore, the results are unpredictable over spatially gradual boundaries such as those present in a typical deformation microstructure.

The authors have recently developed a robust boundary identification method, mathematically based on the fast multiscale clustering (FMC) image processing algorithm [46,48], which overcomes the major limitations of thresholding. The method can reliably reconstruct both 2D [46] and 3D [48] EBSD data sets to reveal the myriad boundaries found in deformed and annealed metals. Such boundaries can range from LAGBs through to HAGBs, the latter often associated with prior grain boundaries and those associated with nucleation of recrystallization. In our method, FMC was adapted to work with a quaternion representation of orientation data in 3D. The identification of features in a volumetric EBSD data set may be achieved by grouping points that are not separated by a boundary with a misorientation higher than a user-defined threshold. However, as noted above, this common method in commercial software is not applicable to subgrain data. Figure 9a-c shows an example of an artificially-constructed volume of orientation data and thresholding with two angles that illustrates this problem. We present a robust solution to segmenting 3D EBSD data, which identifies the actual boundary geometry, as shown in figure 9d.

![Figure 9](image_url)

**Figure 9.** (a) A constructed data set with 25 voxels per side and 9° corner-to-corner misorientation. The transition occurs linearly along the middle third of the cube diagonal. Noise with average misorientation of 0.5° is added to make the data more realistic. A 0.5° threshold angle finds many superfluous boundaries (b), whereas a 0.9° angle finds very few boundaries (c). In both cases noise dominates. (d) Segmentation using FMC produces two clusters with a diagonal boundary.

### 3.1. Fast multiscale clustering (FMC)

This segmentation technique was originally developed for 2D image processing [49]. The method creates a hierarchy of scales from groupings of data, starting with individual data points at the finest scale and iteratively coarsening to combine groups. At each scale in the hierarchy every group is then scored based on the similarity of points it contains and distinction from neighbouring groups. The
highest-scoring groups form the final segmentation of the data. Determining similarity of data requires a metric for measuring distance. For simple colour images, this can be a scalar difference between colours, but for application to EBSD data, the metric is based on misorientation. Hence, we have adapted FMC to work with a quaternion representation for orientation data. A detailed description of FMC and its modifications for both 2D and 3D EBSD data sets is provided elsewhere [46, 50].

At each scale in the hierarchy, groups inherit properties from the finer scale below, down to the individual data points [50]. This allows FMC to segment an entire data set with awareness of local and global trends. Another advantage is the freedom to decide neighbours of points, since the method does not depend on spatial distribution of the data. Thus FMC is not only applicable for 2D situations but easily applied in 3D. Furthermore, since there are fewer groups at coarser scales, FMC has a runtime linear with the number of data points, a necessity for methods applied to large data sets with more than two dimensions.

Misorientation is not useful on its own when comparing coarse groups containing many data points. Thus, Mahalanobis distance, where the distance between the average aggregated values is divided by the group’s variance, is used. This gives groups with low variation in orientation naturally less similarity to their neighbours than groups with high variance. We developed a novel variance aggregation method since the elements of quaternions are not linearly correlated [46, 48]. While this Mahalanobis distance lacks physical significance, it can be scaled at the aggregation step to give sense to closeness and farness, depending on the nature of the data set. The scaling parameter, $C_{\text{Maha}}$, determines the sensitivity of the segmentation [49]. FMC requires several other user-defined values, but fixed values for the rest have been effective for all data sets examined. Hence, FMC has the additional advantage that it is controlled with a single parameter for application to different data, analogous to the cut-off angle for a thresholding method.

![Figure 10](image)

**Figure 10.** Segmentation of the data in figure 9 with $C_{\text{Maha}}$ of: (a) 2.5, (b) 5, (c) 7.5, and (d) 10.

### 3.2. Determination of the optimal sensitivity parameter

The sensitivity of the segmentation, controlled by $C_{\text{Maha}}$, should be different based on the nature of the data. Boundaries between clusters in polycrystalline data should be on the distinct grain boundaries without consideration for variation within grains, but subgrain features are only captured with a more sensitive segmentation. There is no ground truth available for such a segmentation, so the user is responsible for deciding which segmentation to use. Thus, an automated method for determining the optimal value of $C_{\text{Maha}}$ has been developed, inspired by similar clustering problems in machine learning [51]. Oversegmentation is characterized by low marginal variance change from splitting existing clusters. Therefore, an “elbow” in a plot of a global variance measure as a function of $C_{\text{Maha}}$ indicates a shift from creating clusters that should be distinct towards erroneously breaking these clusters further. Once this elbow is identified, the segmentation created using the associated value of $C_{\text{Maha}}$ is selected as optimal. The sensitivity of the segmentation controlled with $C_{\text{Maha}}$ is demonstrated in figure 10.
3.3. Surface extraction

When analysing grain or subgrain volumes, it is often useful to isolate separate faces of an irregular feature. These faces may be defined as regions having similar normal vectors and, as the FMC method developed operates on orientations, only a small further modification is needed for application to normal vectors. With this modification, a second use of FMC identifies coherent faces from the set of points on the exterior of a cluster [48]. The voxel locations from a 3D segment are used as the input for a volume reconstruction using tetrahedra. The triangulated boundary surface is used to estimate local normal vectors, and these are used in the FMC process. As with the main use of FMC, the single $C_{Maha}$ parameter controls the sensitivity of the segmentation of the surface, and the same elbow criterion is used to find the optimal segmentation that separates distinct faces without oversegmenting. It is pertinent to note that it is a straightforward task to identify all of the large clusters in a volume and all of the largest faces on those clusters, making study of many subgrain surfaces possible.

It is important to note that some spatial pre-processing must be carried prior to segmentation out since 3D EBSD data sets are collected in 2D sections generated by FIB. Measurement skew and instrument drift are corrected with a bilinear interpolation on each slice [52]. The data are then globally realigned, skewing the points off their regular grid. Points then may be interpolated to a rectangular grid for computational efficiency, but FMC can be performed with any spatial distribution of data points.

3.4. Automated reconstruction of complex boundaries in 3D EBSD data sets

We demonstrate the excellent capabilities of our method with application to 3D EBSD data sets generated from cold rolled aluminium containing well-defined microbands, channel-die plane strain compressed Goss-oriented nickel crystal containing microbands with very subtle changes in orientation, and cold rolled and partly recrystallized IF steel microstructure containing three magnitudes of boundary misorientations.

3.4.1. Microbands in cold rolled aluminium. We first apply FMC to segment microbands in a single grain of cold-rolled (22% reduction) commercial purity aluminium [34]. Figure 11 shows the EBSD data with inverse pole figure colouring, in which the microbands are visible, as well as its segmentation. The microband structure is captured by the FMC segmentation. The highlighted microbands in figure 11b are isolated in 3D in figure 11c to show the nature of the interior of the segmentation. This data set was also segmented with the Kuwahara filter method [47]. It was found that, while this type of filter partially aligns with the visible microband structure, no threshold value was found to be appropriate for

Figure 11. 3D reconstruction (1.1 million data points) of the cold rolled aluminium data set: (a) EBSD data using the IPF colour map of figure 8; (b) black and white lines show the boundaries of segments found with the optimal $C_{Maha}$ value of 4.4, and (c) eight selected clusters, indicated with white boundaries in (b), showing the underlying subgrain structure, including a boundary plane that extends through the entire data set (Scale is in micrometres), after [48].
the entire data set, which was evidenced by the presence of both significant oversegmentation and undersegmentation. This difficulty is overcome by FMC since its segments are determined with aggregate orientation and variance differences, not strictly local information.

The FMC surface extracting method was applied to extract microbands from the EBSD data set. Figure 12 shows the top microband of figure 11c, with the entire top and bottom surfaces identified. These surfaces can be used for further geometric and crystallographic analysis. FMC is a powerful tool for this application since it can form a full surface from similar components that are separated by a sharp step in the estimated normal vectors.

**Figure 12.** Two views of a cluster in the segmentation of cold rolled aluminium data. Colours indicate separate extracted surfaces. The top and bottom surfaces are isolated, as well as flat surfaces on the top and side from the boundary faces of the data set, after [48].

3.4.2. Microbands in deformed nickel single crystal. Microbands have been successfully isolated in a 3D EBSD data set from a channel die plane strain compressed (35% strain) Goss-oriented nickel single crystal [33]. The data have a standard deviation from the average orientation of only 0.7°. Figure 13 shows the segmented microbands. Identification of these subgrain features would not be attainable with conventional thresholding at any angle. The segmentation in figure 13b is performed with the optimal $C_{\text{Maha}}$. To show how even more subtle features may be identified, figure 13c shows a segmentation with a higher $C_{\text{Maha}}$ value. This comes with the consequence of oversegmenting the data overall, but specific features of interest may still be used for further analysis.

**Figure 13.** 3D reconstruction (> 1.3 million data points) of the nickel data set: (a) EBSD data using the IPF colour map of figure 8; (b) boundaries of segments found with the optimal $C_{\text{Maha}}$ value of 6, and (c) boundaries of segments found with a $C_{\text{Maha}}$ value of 7, showing the identification of more subtle features, after [48].

3.4.3. Recrystallizing grains in cold rolled and partly recrystallized IF steel. To further illustrate the capability of our method in identifying the broad range of misoriented boundaries that are often present in the same data set, the 3D EBSD data set of the partly recrystallized IF steel described earlier was analysed. Figure 14a shows the raw EBSD data of the reconstructed data set. Figures 14b and c reveal the subtle subgrain features, distinct grain boundaries, and a HAGB separating $\alpha$- and $\gamma$-fibre regions; such boundaries are almost impossible to delineate using conventional thresholding techniques. Further, the presence of the HAGB separating the regions does not affect the detection of boundaries with a...
smaller misorientation, even those adjacent to it. This is because variance comparisons are done between each neighbouring group, rather than with a global standard over the entire data set. Finally, figure 14d shows the volume of local stored energy using the subgrain method of Choi and Jin [53] for 2D EBSD data sets, which has been extended to 3D by modifying their method to use all neighbouring EBSD points in 3D. In conjunction with FMC, this will be a powerful method for scrutinising how variations in stored energy coupled with grain boundary character can influence the nucleation of recrystallization through to the early and latter stages of recrystallization in deformed metals.

![3D reconstruction](image)

**Figure 14.** 3D reconstruction (350,000 data points) of the partly recrystallized IF steel data set [54]: (a) EBSD data using the IPF colour map of figure 8; (b) segmentation with the optimal $C_{\text{Maha}}$ value of 3.4; (c) boundaries from the segmentation coloured by local misorientation, showing the HAGB between $\alpha$- and $\gamma$-fibre regions (arrowed), and (d) map of local stored energy computed with the so-called subgrain method described in [53] showing the high energy substructure surrounding the low energy recrystallizing grains (arrowed), whereby black indicates regions of low energy and white indicates regions of high energy in relative terms (Scale is in micrometres).

The foregoing examples illustrate the effectiveness of FMC as a tool for segmenting 3D EBSD data sets containing subtle and gradual variations in orientation. We have incorporated FMC modified to operate on orientation data into the free open source texture analysis software package MTEX [8], thereby allowing users to take advantage of its existing methods for importing and manipulating data. The ability of the method to segment data based on a wide range of boundary magnitudes with a linear runtime make FMC a robust method for both 2D and 3D segmentations.

### 4. Concluding summary

By combining FIB with FEGSEM in a DualBeam™ or similar platform, it is possible to sequentially mill sections of a material by FIB and map the crystallographic features of each newly created surface
by EBSD. This allows crystallographic volumes of microstructure to be generated and analyzed by this serial sectioning technique. 3D EBSD was used to investigate various phenomena associated with the plastic deformation of metals and alloys where new information it consistently revealed not clearly evident using standard 2D analysis techniques. For example, the structure of microbands in FCC Ni was highlighted and the new observation of grain boundary fragments that are potential sites for nucleation of recrystallization with orientations not present in the surrounding local substructure. We also illustrate an effective tool, based on fast multiscale clustering (FMC), for segmenting 3D EBSD data sets containing subtle and gradual variations in orientation. The ability of the method to segment data based on a wide range of boundary magnitudes with linear runtime make FMC a robust method for both 2D and 3D segmentations.

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References

[1] Möbus G and Inkson BJ 2007 Materials Today 10 18
[2] Schmidt S, Nielsen SF, Gundlach C, Margulies L, Huang X and Juul Jensen D 2004 Science 305 229
[3] Larsen AW, Poulsen H F, Margulies L, Gundlach C, Xing Q, Huang X and Juul Jensen D 2005 Scripta Mater. 53 553
[4] Schmidt S, Olsen U L, Poulsen H F, Sørensen H O, Lauridsen E M, Margulies L, Maurice C and Juul Jensen D 2008 Scripta Mater. 59 491
[5] West S S, Schmidt S, Sørensen H O, Winther G, Poulsen H F, Margulies L, Gundlach C and Juul Jensen D 2009 Scripta Mater. 61 875
[6] www.edax.com/Products/EBSD
[7] Xu W, Ferry M, Mateescu N, Cairney JM and Humphreys FJ 2007 Mater. Charact. 58 961
[8] Xu W, Ferry M, Cairney J M and Humphreys F J 2007 Acta Mater. 55 5157
[9] Konrad J, Zaefferer S and Raabe D 2006 Acta Mater. 54, 1369
[10] Mateescu N, Ferry M, Xu W and Cairney J M 2007 Mater. Chem. Phys. 106 142
[11] Zaafarani Z, Raabe D, Singh RN, Roters F and Zaefferer S 2006 Acta Mater. 54 1863
[12] Calcagnotto M, Ponge D, Demir E and Raabe D 2010 Mater. Sci. Eng. A 527 2738
[13] Robin L, Laws K J, Kurniawan G, Xu W, Privat K and Ferry M 2010 Metall. Mater. Trans A 41 1691
[14] Rowenhorst D J, Gupta A, Feng C R and Spanos G 2006 Scripta Mater. 55 11
[15] Lewis AC and Geltmacher A B 2006 Scripta Mater. 55 81
[16] Lin FX, Godfrey A, Juul Jensen D and Winther G 2010 Mater. Charact. 61 1203
[17] Bachmann F, Hielscher R and Schaeben H 2011 Ultramicroscopy 111 1720
[18] Groeber M, Ghosh S, Uchic M D and Dimiduk DM 2008 Acta Mater. 56 1257
[19] Xu W, Ferry M and Humphreys F J 2009 Scripta Mater. 60 862
[20] Xu W, Quadir M Z, Ferry M and Humphreys F J 2009 Metall. Mater. Trans A 40 1547
[21] Humphreys F J and Hatherly M 2004 *Recrystallization and Related Annealing Phenomena* (UK: Elsevier)
[22] Godfrey A, Juul Jensen D and Hansen N 1998 Acta Mater. **46** 823
[23] Winther G, Huang X and Hansen N 2000 Acta Mater. **48** 2187
[24] Winther G, Huang X, Godfrey A and Hansen N 2004 Acta Mater. **52** 4437
[25] Lin F X, Godfrey A and Winther G 2009 Scripta Mater. **61** 237
[26] Hurley P J and Humphreys F J 2003 Acta Mater. **51** 1087
[27] Hurley P J, Bate P S and Humphreys F J: Acta Mater. 2003 **51** 4737
[28] Humphreys F J and Bate P S 2007 Acta Mater. **55** 5630
[29] Quadir M Z, Mateescu N, Bassman L, Xu W and Ferry M 2007 Scripta Mater. **57** 977
[30] Afrin N, Quadir M Z and Ferry M 2015 Metall. Mater. Trans A (DOI: 10.1007/s11661-015-2878-4)
[31] Afrin N, Quadir M Z and Ferry M 2014 Metall. Mater. Trans B **45** 345
[32] Afrin N, Quadir M Z, Xu W and Ferry M 2012 Acta Mater. **60** 6288
[33] Afrin N, Quadir M Z, Bassman L, Albou A, Driver J H and Ferry M 2011 Scripta Mater. **64** 221
[34] George C, Soe B, King K, Quadir M Z, Ferry M and Bassman L 2013 Mater. Charact. **79** 15
[35] Duggan B J and Tse Y Y 2004 Acta Mater. **52** 387
[36] Barnett M R 1998 ISIJ Int. **38** 78
[37] Barnett M R and Jonas J J 1997 ISIJ Int. **37** 697
[38] Quadir M Z and Duggan B J 2006 Acta Mater. **54** 4337
[39] Quadir M Z 2003 PhD thesis *The University of Hong Kong*
[40] Ushioda K, Nakanishi S, Morikawa T, Higashida K, Suwa Y and Murakami K 2013 Mater. Sci. Forum **753** 58
[41] Juul Jensen D, Lin F X, Zhang Y B and Zhang Y H 2013 Mater. Sci. Forum **753** 37
[42] Hansen N and Juul Jensen D 2011 Mater. Sci. Tech. **27** 1229
[43] Godfrey A, Juul Jensen D, Hansen N 2001 Acta Mater. **49** 2429
[44] Dillamore I L, Smith C J E and Watson T W 1967 Metal Sci. **1** 49
[45] Goodenow R H 1966 Trans ASM **59** 804
[46] McMahon C, Soe B, Loeb A, Vemulkar A, Ferry M and Bassman L 2013 Ultramicroscopy **133** 16
[47] Humphreys F, Bate P and Hurley P 2001 J. Microsc. **201** 50
[48] Loeb A, Ferry M and Bassman L 2015 Ultramicroscopy (to be submitted)
[49] Kushnir D, Galun M, Brandt A, 2006 Pattern Recogn. **39** 1876
[50] Sharon E, Meirav G, Sharon D, Basri R and Brandt A 2006 Nature **442** 810
[51] Sugar C A, Lenert L A, Olshen R A 1999 Technical Report. Stanford University
[52] Soe B, McMahon C, Golay D, Quadir M Z, Ferry M and Bassman L 2012 1st Int. Congress on 3D Materials Science (Seven Springs, Pennsylvania, USA, 8-12 July 2012) eds M De Graef, H F Poulsen, A Lewis, J Simmons and G Spanos, pp 189-194
[53] Choi S and Jin Y 2004 Mater. Sci. Eng. A **371** 149
[54] Loeb A, Soe B, McMahon C, Ferry M and Bassman L 2014 2nd Int. Congress on 3D Materials Science (Annecy, France, 29 June-2 July 2014) eds D Bernard, J-Y Buffiere, T Pollock, H F Poulsen, A Rollett and M Uchic, pp 9-14