

Advancing analytical electron microscopy methodologies to characterise microstructural features in superalloys

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ABSTRACT

Electron backscatter diffraction (EBSD) generally links crystallographic orientation to the microstructure of crystalline materials. EBSD datasets are now commonly used to identify phases, grains, and their orientations using off-the-shelf software, although substantial additional information may be extracted. Due to the lack of commercially available software, advanced analyses are often done manually and provide only localised information, lacking statistical significance. Here we introduce novel automated methodologies for advanced analyses of microstructural features in Ni-based superalloys. Our methodologies provide additional insights into the characteristics of these features and their underlying physical phenomena. We showcase how to correct wrongly indexed γ/γ' interface artefacts in combined EBSD and energy-dispersive X-ray spectroscopy (EDS) measurements, how to classify recrystallised grains based on their location, how to assess and visualise grain boundary planes, and how to study the evolution of $\Sigma 3$ twins during hot deformation. We further demonstrate how phase fractions and grain sizes are more accurately determined in combined EBSD-EDS measurements. The classification of recrystallised grains into different groups enables individual analyses, facilitating the straightforward identification of the underlying recrystallisation mechanism. Our grain boundary plane analysis provides insights into the coherence of $\Sigma 3$ twins and the potential boundary planes of incoherent $\Sigma 3$ boundaries. The current paper is a tutorial-style guide for these methodologies. The algorithms are made freely available and, although demonstrated here on Ni-based superalloys, can also be applied to other systems.

1. Introduction

Electron backscatter diffraction (EBSD) is a well-established method for the analysis of crystalline materials. It links the crystallographic orientation to the microstructure, enabling the characterisation of phases, grains, crystal orientations, interfaces, and local strains [1]. The wide range of information that becomes easily accessible via EBSD makes it an attractive technique in materials science, in particular to study deformation and recrystallisation mechanisms in metals such as Ni-based superalloys [2]. Due to its popularity, EBSD has seen rapid developments in recent years [3]. Advances in detector technology and data processing led to increased resolution and faster acquisition rates of currently up to 6700 points per second [4]. The resulting large datasets contain a wide range of information beyond the standard microstructural features that are typically evaluated by off-the-shelf software. Most of this information is either discarded or only manually analysed due to the lack of automated software. However, manual analysis limits the

number of features that can be studied and, therefore, results may not provide quantitative information and lack statistical significance. For example, pole figures are often graphically inspected to establish the plane normal of individual interfaces [5]. Developments such as the five-parameter statistical stereological analysis [6] to analyse grain boundary (GB) planes or the variant graph approach [7] to reconstruct grains after solid state phase transformations demonstrate how automated methods contribute to advanced characterisation of microstructures. However, there is still a lack in advanced methodologies to study many additional microstructural features captured in EBSD datasets. These automated methodologies will be helpful to study the evolution of special GBs or recrystallised grains during deformation. In this paper, we develop advanced methodologies that provide important new insights into the microstructural evolution of wrought Ni-based superalloys, which remains challenging using standard EBSD software as outlined in more detail below.

The first challenge in EBSD characterisation of Ni-based superalloys

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is the differentiation between the face centred cubic (fcc) γ -Ni matrix and $L1_2$ (fcc ordered) γ' -Ni₃Al precipitates [8,9]. γ' precipitates provide high strength at elevated temperatures, allowing wrought Ni-based superalloys to be used as turbine disks in aircrafts or power-generation applications [10,11]. Complex thermo-mechanical processes promote the formation of multimodal γ' distributions ranging from 1–2 μm down to 50 nm [12–14]. Nanometre-sized secondary and tertiary γ' are precipitated within the γ matrix with a cube/cube orientation relationship and (semi)-coherent interfaces [15]. This makes these small precipitates indistinguishable from their surrounding matrix in EBSD data [9]. Micrometre-sized primary γ' form at the GBs of γ grains leading to duplex γ - γ' microstructures [14]. These primary γ' usually do not exhibit a cube/cube orientation relationship with the surrounding γ grains, which makes their orientation detectable via EBSD. Although the orientation of these primary γ' is detectable, their phase identity cannot be obtained. As phase differentiation in EBSD is based on diffraction and crystal structure only, it is impossible to differentiate between the (fcc) γ and (ordered fcc) γ' phases [9,16]. Energy dispersive X-ray spectroscopy (EDS) can provide the required complementary chemical information to distinguish primary γ' precipitates and the γ matrix. When EBSD and EDS are combined, the chemical spectrum of EDS is used to assign each data point to a phase and link it to the EBSD orientation information [16]. However, these techniques have different resolutions. EBSD collects information of backscatter electrons, which allows depth and spatial resolutions as low as 10 nm and 50 nm, respectively [16,17]. This is much smaller than the interaction volume of EDS, which is based on X-rays with an interaction volume of around 1 μm . A simulated graphical representation (using Casino v2.51 [18]) of the interaction volumes in Ni can be found in the supplementary figure S 1. Therefore, while a point contributes to the EBSD measurement alone, its surroundings will be contributing to the EDS signal. This can lead to γ/γ' interface artefacts due to an inaccurate assignment of phases in measurements close to interphase boundaries. We will present a methodology to correct these γ/γ' interface artefacts in combined EBSD-EDS measurements.

The second set of challenges is in the analysis of recrystallised grains in EBSD measurements of deformed samples. It is a common approach using off-the-shelf software to differentiate between different regions of the datasets, using simple threshold values. For example, the grain orientation spread (GOS) is a commonly used threshold to separate recrystallised from deformed grains [19,20]. However, dynamic recrystallisation (DRX) during hot deformation can occur via different mechanisms, even in the same sample. Continuous dynamic recrystallisation (CDRX) [21], discontinuous dynamic recrystallisation (DDRX) [22], and particle stimulated nucleation (PSN) [23] have all been observed in Ni-based superalloys. Differences between these mechanisms could be missed by classifying all recrystallised grains collectively, using a simple threshold. Commonly, misorientation line scans or pole figures are inspected to identify recrystallisation mechanism [24]. However, these methods allow for local analyses only and lack statistical significance. Furthermore, they cannot be automatically applied to large datasets, and regions of interests must be selected manually. This may lead to important features being overlooked. Here, we develop an automated grain classification methodology that categorises different recrystallised grains on basis of their locations. Emphasis is put on the nucleation of recrystallised grains at distorted $\Sigma 3$ annealing twins and the evolution of these twins during hot compression. In that sense, this paper is a companion paper to [25], where we have discussed the role of distorted $\Sigma 3$ twins in the nucleation of DRX grains. The current paper provides a detailed explanation of our developed methodology.

Thirdly, there are obstacles in the analysis of GB character in two-dimensional EBSD data. A full description of the GB character requires five macroscopic degrees of freedom (DOF) [26], which is usually only accessible through three-dimensional measurements such as 3D EBSD [27]. In two-dimensional EBSD, the inclination of the GB plane is unknown [26], resulting in only four out of these five parameters being determined. Commonly, misorientation angles of GBs are reported and

special GBs such as $\Sigma 3$ ($60^\circ(111)$) boundaries are identified. However, these descriptions use the misorientation (equivalent to three DOF) between adjacent crystals only, and do not include any information of the GB itself, despite many reports showing the critical role of the GB habit plane in determining properties of materials [28,29]. While the inclination of the GB plane is not known, the trace can be seen in two-dimensional EBSD [26]. Combining information of the GB trace (equivalent to one DOF) with the crystallographic misorientation of the neighbouring grains provides us with additional information of the GB character [30,31]. This can be used to obtain an estimate of the coherence of $\Sigma 3$ twins, which determines the properties of these interfaces [32,33]. Previously, such methods have either been manually applied to selected GBs only [34] or automated to entire datasets through the five-parameter statistical stereological analysis [6]. While the manual application of this method provides a good visual representation, it lacks statistical information. The opposite applies to the statistical stereological analysis where only heat maps are generated. We here propose an automated GB plane visualisation methodology that can be applied to large datasets and provide graphical and statistical information on the coherence of $\Sigma 3$ boundaries.

In summary, the aim of the current paper is to introduce our advanced methodologies to analyse EBSD datasets, using Ni-based superalloys as a case study. The developed methodologies may be applied to improve the analysis of phases (Section 3.1), recrystallised grains (Section 3.2), and GBs (Section 3.3). This paper is written in a tutorial-style to act as a guide through these developed methodologies which are implemented using the open-source MTEX toolbox [35] for Matlab. The algorithms and sample datasets are available under open-source license via <https://doi.org/10.5281/zenodo.7246613> [36]. Although the methodologies are here all applied on Ni-based superalloys, they are not limited to these materials, and the scripts have been written to allow application to other materials systems such as high entropy alloys, stainless steels, copper, or light metals.

2. Materials and experimental methods

The materials used to demonstrate the developed methodologies are two cast-and-wrought Ni-based superalloys. Their chemical compositions are shown in Table 1. Carbon and boron contents were determined using combustion analysis and spark spectrometry, respectively. X-ray fluorescence spectrometry was used to determine the chemistry for the other elements. The alloys were provided as billets by voestalpine BÖHLER Edelstahl GmbH & Co KG (Austria), were double remelted, homogenised, and underwent ingot breakdown by open die forging.

The as-received Alloy 1 was used in the section on ' γ/γ' interface artefact correction in combined EBSD-EDS measurements' (Section 3.1). The material used in the 'Classification of recrystallised grains' case study (Section 3.2.1) is Alloy 2, after undergoing hot compression (see below). The 'Twin boundary evolution during hot compression' (Section 3.3.1), and the 'Grain boundary plane visualisation' (Section 3.3.2) methodologies are demonstrated on Alloy 1 after a solutionising and ageing heat treatment. The solutionising consisted of heating with 5 $^\circ\text{C}/\text{min}$ to 1180 $^\circ\text{C}$, where it was held for 7 mins before water quenching. The ageing treatment comprised of quickly reheating samples to 1080 $^\circ\text{C}$ from where they were continuously cooled at 5 $^\circ\text{C}/\text{min}$ to room temperature. This heat treatment resulted in a comparable microstructure of Alloy 1 to the as-received Alloy 2.

Isothermal uniaxial hot compression experiments were conducted at 1050 $^\circ\text{C}$ and 1100 $^\circ\text{C}$ with a strain rate of 0.1 s^{-1} to 0.6 true strain in a Gleeble 3500GTC (Dynamic Systems Inc.) thermo-mechanical simulator. All specimens were immediately water quenched after deformation. A more detailed description of the hot compression experiments can be found in the companion paper [25].

All specimens were prepared following standard metallographic routines with a final surface finish achieved by polishing with 0.04 μm colloidal silica suspension. Specimens that were hot compressed were

Table 1

Chemistries of the Ni-based superalloys used in this study in wt.%.

	Cr	Co	Mo	W	Al	Ti	C	B	Zr	Ni
Alloy 1	17.4	14.6	2.9	1.3	2.5	5.1	0.04	0.04	0.03	bal.
Alloy 2	18.6	11.8	5.7	0.9	2.0	3.1	0.04	0.01	0.05	bal.

additionally vibro-polished using a 0.06 μm colloidal silica suspension for 15 hrs to minimise surface deformation.

Combined EBSD-EDS measurements were collected using the Zeiss AURIGA Cross-Beam field-emission scanning electron microscope (FE-SEM) equipped with an Oxford Instruments X-Max 20 mm² silicon drift EDS detector and Oxford NordlysF EBSD detector using a 6×6 binning. Both data were collected simultaneously using Oxford Instruments' Aztec 3.3 with the EDS-assisted phase indexing *TruPhase* enabled. A working distance of 9 mm and an acceleration voltage of 15 kV with an aperture of 60 μm were used. These parameters allowed a compromise between the quality of Kikuchi patterns and sufficient X-ray count while minimising the excited X-ray volume [9].

The remaining EBSD-only maps were collected using a Hikari Super system controlled by the EDAX TEAM™ V4.4.1 software that was installed in a JEOL 7001F FE-SEM with an acceleration voltage of 20 kV, probe current setting of '14' (SEM manufacturer internal parameter), and an 8×8 binning. Minimum grain confidence index thresholding was applied to these measurements, removing grains with a confidence index of < 0.1 .

Automated methodologies were developed to analyse the EBSD-EDS and EBSD data using the MTEX 5.7.0 toolbox [35] in Matlab R2019b. A minimum grain size of 5 pixels with a grain tolerance angle (θ) of 5° was used to detect grains. Unless otherwise indicated, the standard GB smoothing function, i.e. constrained Laplacian smoothing [37], in MTEX with 5 iterations was applied. The half quadratic filter with an alpha value of 0.25 was used to denoise orientation data. This filter was developed to preserve subgrain boundaries [38]. A GOS of 2° was used to differentiate between deformed and recrystallised grains. Grains were merged within the respective deformed or recrystallised regions along

$\Sigma 3$ $60^\circ\langle 111 \rangle$ boundaries with a tolerance angle of 8.66° based on the Brandon criterion [39] to remove twins.

3. Results and discussion

3.1. Phase analysis: γ/γ' interface artefact correction in combined EBSD-EDS measurements

The combined EBSD-EDS measurements of the as-received Alloy 1 with a step size of 0.1 μm are shown in Fig. 1. The microstructure consists of γ -grains with micrometre-sized primary γ' located at the GBs and secondary γ' inside the γ grains. There are also coherent tertiary γ' precipitates within the γ grains, but these particles are too small to be resolved here.

The band contrast map of the EBSD measurements (Fig. 1(a)) gives a good indication of the location of GBs. This band contrast map is overlaid with the qualitative EDS measurements of Cr (coloured in blue) and Ti (coloured in orange) in Fig. 1(b). The EDS measurements are used to differentiate the γ phase, which is high in Cr, from the γ' precipitates, which are high in Ti. The red arrows indicate regions that appear to belong to the primary γ' based on the EBSD band contrast map, but the measured EDS chemistry is that of the surrounding γ matrix. This is a result of the smaller resolution of ~ 50 nm for EBSD compared to ~ 1 μm for EDS [16]. When points close to the γ/γ' interface are mapped, the correct EBSD data are collected, while X-rays of the surrounding γ matrix also contribute to the EDS measurement. As the phase indexing is based on the EDS measurements, these points are wrongly assigned to the γ phase here. This leads to γ/γ' interface artefacts during the grain reconstruction. These γ/γ' interface artefacts are visible as γ regions

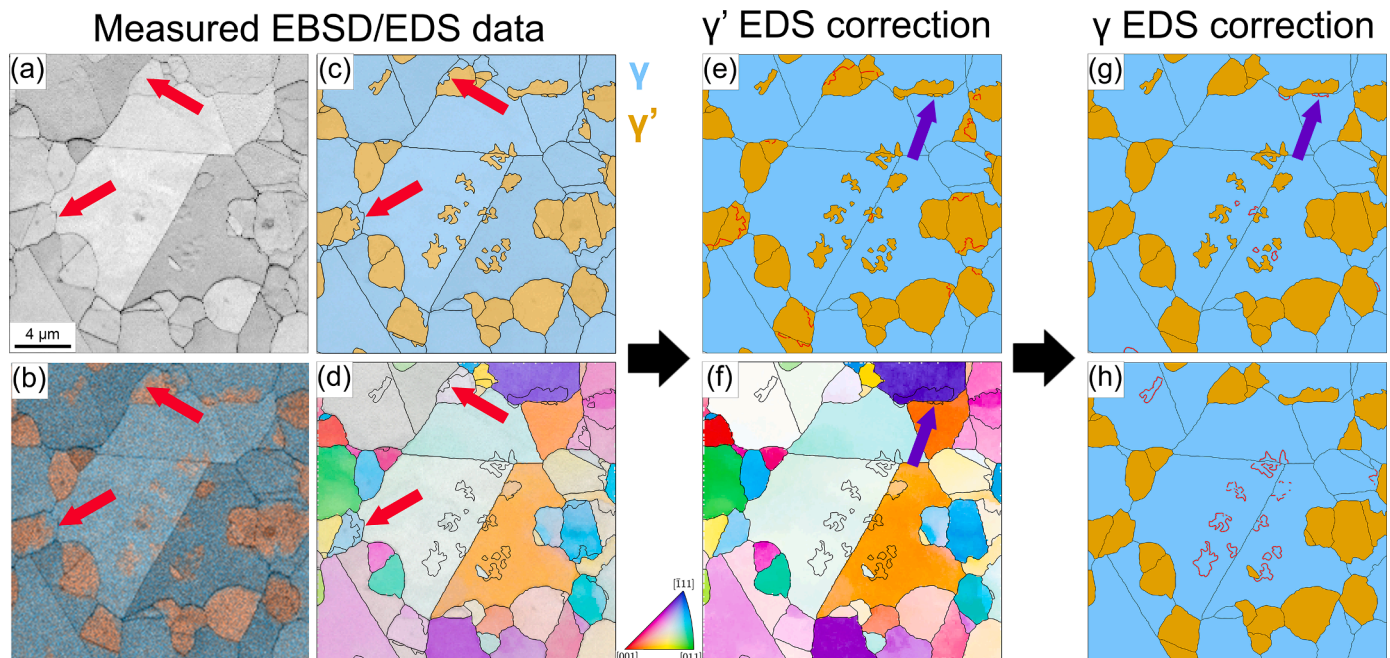


Fig. 1. Microstructure of Alloy 1 with γ grains as well as primary and secondary γ' collected with combined EBSD-EDS where GBs with misorientation $\theta > 5^\circ$ and interphase boundaries are marked in black. The measured and uncorrected (a) band contrast map, (b) EDS map with regions high in Cr in blue (γ) and regions high in Ti in orange (γ'), (c) phase map with γ in blue and γ' in orange, and (d) IPF map. After EDS correction of γ' : (e) phase map with changed interphase boundaries marked in red along which γ interface artefacts were reassigned to γ' , (f) IPF map after applying the correction. Phase maps after EDS correction of (g) the γ phase and (h) removal of secondary γ' with the changed interphase boundaries marked in red.

around primary γ' (Fig. 1(c), indicated by red arrows) that have the same orientation as the γ' (Fig. 1(d)).

We here propose a method to correct for these γ/γ' interface artefacts during post-processing of the collected EBSD-EDS data. To do this, we first find all interfaces between the precipitates (γ') and matrix (γ) with a misorientation angle less than a defined threshold (entered in degrees). This threshold ensures that artefacts are identified even if there is some misorientation between the phases, which could be e.g. due to noise associated with the EBSD measurements itself. Further, if there are orientation gradients within precipitates, this threshold has to be increased to account for them. Then, matrix grains along these interfaces that have an area below a defined datapoint limit are selected. This datapoint threshold is necessary as otherwise matrix γ grains that contain secondary γ' would be selected, as they share the same orientation per definition. The datapoint limit for a particular microstructure depends on the used step size, the size of the interface artefacts, and the size of the matrix grains. We change the phase identity of the selected matrix grains to the phase identity of the precipitate to remove the γ/γ' interface artefacts. Lastly, the grain reconstruction [40] is repeated

based on the corrected phase identities.

Fig. 1(e)-(f) show the EBSD maps after applying the correction for the γ/γ' interface artefacts. The misorientation angle threshold applied was 1° , and the datapoint limit for the matrix grains was 150 datapoints. The former phase boundaries between γ' precipitates and identified γ artefacts are indicated in red (Fig. 1(e)). The methodology correctly changes the phase identity of the selected grains from γ (Fig. 1(c)) to γ' (Fig. 1(e)). The collected EBSD orientation data remains unchanged, as can be seen by comparing the inverse pole figure (IPF) maps before (Fig. 1(d)) and after (Fig. 1(f)) the correction.

The same methodology can be applied to correct γ'/γ interface artefacts where regions of the γ -grains were wrongly indexed as γ' phase (Fig. 1(e)-(f), indicated by purple arrows). This simply requires the phases to be swapped in the above-described algorithm. The results are shown in Fig. 1(g). A boundary threshold angle of 1° and a limit of 15 datapoints were used in this case. Wrongly indexed γ' are now correctly assigned as being part of the γ grain. However, the algorithm also detects and removes smaller secondary γ' . This is due to these secondary γ' displaying the same orientation as their host γ grain and their smaller

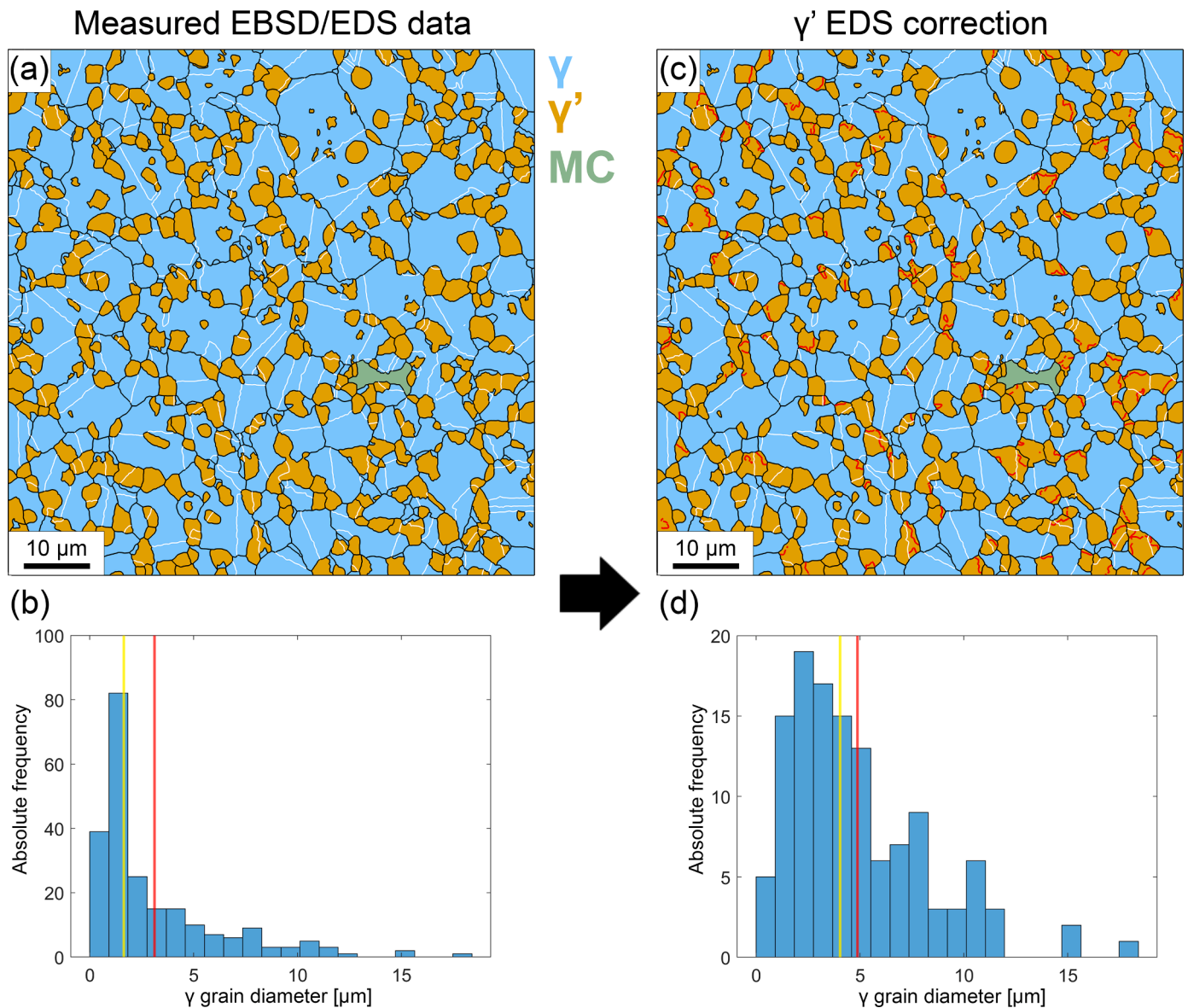


Fig. 2. Phase maps of Alloy 1 measured with combined EBSD-EDS (a) before and (c) after EDS correction. GBs with misorientation $\theta > 5^\circ$ and phase boundaries are marked in black, $\Sigma 3$ twin boundaries are indicated in white. The grain diameter distribution for γ grains (b) before and (d) after EDS correction; the yellow line and red line indicate the median and mean diameter, respectively.

size than the chosen limit of 15 datapoints. Therefore, our algorithm can also be used to remove secondary γ' , e.g. to analyse primary γ' only (Fig. 1(h)). This is achieved by using a higher limit of e.g. 150 datapoints. If there is interest in analysing secondary γ' , this would require minor changes to the algorithm to assign them to a separate partition instead of re-classifying them as γ matrix.

Fig. 2 demonstrates the changes in γ' diameter measurements after correction of the EBSD-EDS γ/γ' interface artefacts. This map was collected over a larger area with a step size of $0.2\ \mu\text{m}$ on the same material condition as in Fig. 1. The parameters for the correction were 1.1° for the threshold and a limit of 40 datapoints. The lower datapoint limit compared to Fig. 1(a)–(f), where we used 150 datapoints, is due to the increase in step size from $0.1\ \mu\text{m}$ to $0.2\ \mu\text{m}$. At larger step sizes, the same grain or γ/γ' interface artefact will be composed of fewer datapoints. This illustrates that the datapoint limit must be chosen carefully depending on the data collection parameters. Fig. 2(a) and (c) show the phase map before and after the applied correction, respectively. $\Sigma 3$ twin boundaries within the Brandon criterion [39] are indicated in white. Phase boundaries that were removed by the correction are highlighted in red. Fig. 2(b) and (d) show the grain diameter distribution before and after the correction, respectively. The median of the distribution is indicated in yellow and the mean in red. These graphs clearly demonstrate how significantly the γ artefacts next to the primary γ' in the as-measured microstructure shift the distribution to smaller grain diameters, leading to an underestimation of the correct grain sizes.

Our methodology is designed to correct for EBSD-EDS γ/γ' interface artefacts. However, there are physical phenomena that may cause γ -shells with cube/cube orientation to be formed around primary γ' e.g., inverse precipitation [41], or heteroepitaxial recrystallisation [42]. Therefore, before applying the algorithm, the microstructure must be carefully studied to ensure that no such features would be eliminated. However, the same methodology may be adopted to analyse such γ -shells by assigning them to their own partition instead of removing

them.

Furthermore, the algorithm changes the assigned phase of wrongly indexed γ/γ' interface artefacts. It does not re-index the Kikuchi patterns but maintains the same orientation information as determined during EBSD data collection. This is not an issue in the case of Ni-based superalloys as demonstrated here because the crystallographic data used to index γ and γ' are identical. If the crystallographic structures are different, the corrected datapoints must be re-indexed using stored Kikuchi patterns. However, in the case of different crystal structures, EBSD alone would be sufficient to differentiate between phases [16].

3.2. Grain analysis

3.2.1. Classification of recrystallised grains

Some of the most noteworthy applications of EBSD are in studying the microstructures of hot deformed materials due to the ability to differentiate recrystallised and deformed grains using threshold values such as the GOS [19,20]. DRX itself is a complex phenomenon as there are several different mechanisms by which new DRX grains may nucleate. Using a threshold value alone does not allow a differentiation between DRX grains formed via different mechanisms. Here we propose a location-based classification methodology to separately analyse DRX grains that nucleate at different microstructural features (Fig. 3). To do this, we first identify all DRX grains by selecting grains that have a GOS below a certain threshold value (e.g. 2°). After that, DRX grains that share an interface with a selected second phase (e.g. MC carbides in our case study) are assigned to the ‘DRX at MC carbides’ partition (coloured red). DRX grains that are fully inside deformed grains, i.e. not touching any interfaces, are then assigned to ‘DRX inside grains’ partition (coloured yellow).

The deformed grains ($\text{GOS} > 2^\circ$) are then merged along $\Sigma 3$ ($60^\circ\langle 111 \rangle$) twin boundary segments within a maximum deviation of 8.66° as defined by the Brandon criterion [39]. This merging will cause

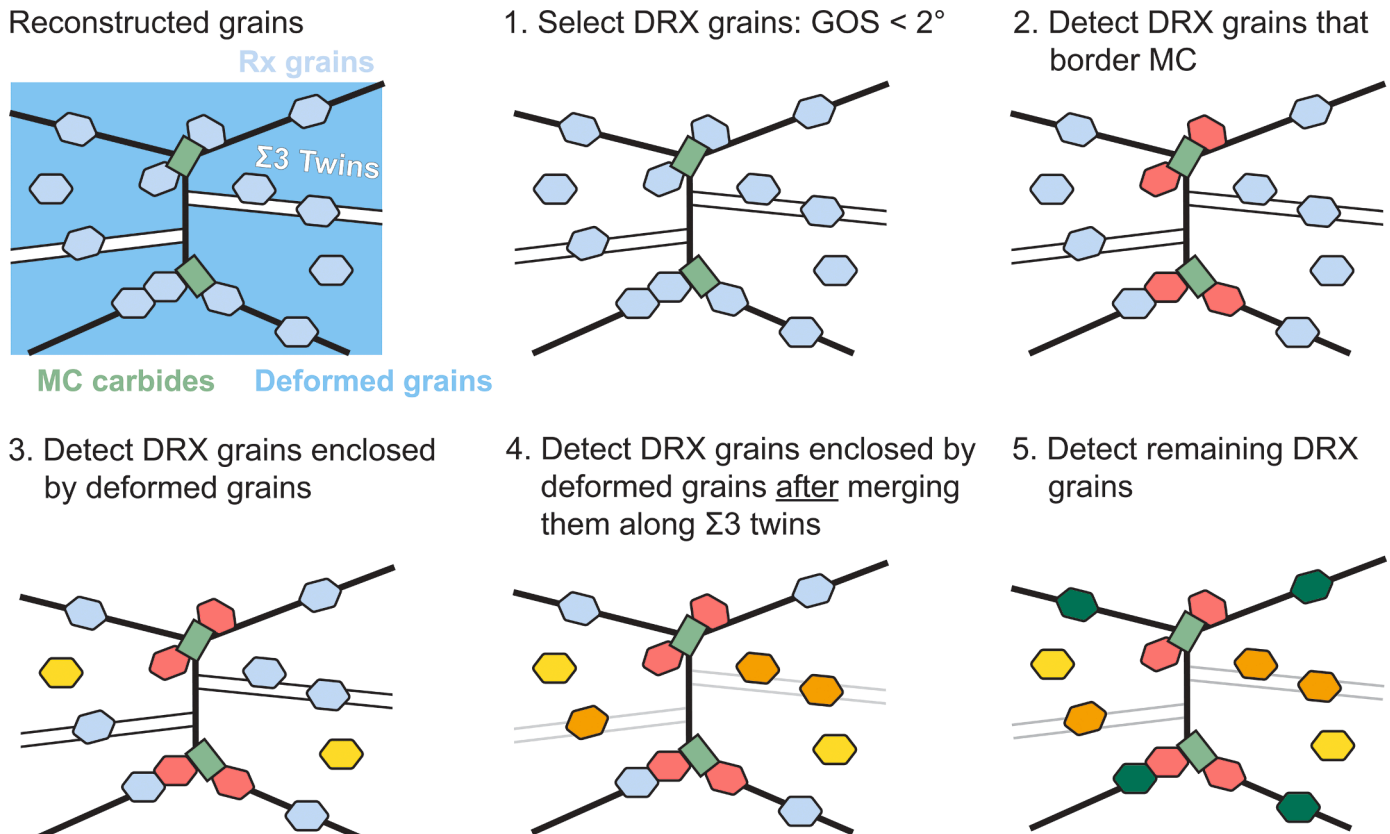


Fig. 3. A schematic outlining the criteria that are used to classify the recrystallised grains into groups depending on their location.

some DRX grains that were previously located at GBs to now be located inside the newly merged deformed grains. These recrystallised grains are then assigned to the ‘DRX at distorted twins’ partition (coloured orange). This method to detect ‘DRX at distorted twins’ has been chosen, because since these twins are distorted, many segments along these boundaries do not fulfil the $\Sigma 3$ condition anymore (Section 3.3.1). Therefore, if we are looking for DRX grains on $\Sigma 3$ boundaries between deformed grains directly, we would not be able to detect these DRX grains that nucleated at the most heavily distorted segments of these twins.

All remaining DRX grains that are located at GBs and do not fulfil any of the previously mentioned conditions are then assigned to the ‘DRX at random GBs’ partition (coloured green). Importantly, these criteria must be applied in this particular order. If multiple criteria are true, the grain is assigned to the first operation in the list that it fulfils. For example, a DRX grain that is located at a MC carbide and at a distorted twin between deformed grains will be assigned to ‘DRX at MC carbides’.

Fig. 4 shows EBSD measurements collected with a $0.2\ \mu\text{m}$ step size of Alloy 2 that was deformed at $1050\ ^\circ\text{C}$ at $0.1\ \text{s}^{-1}$ to 0.6 strain. The IPF map (Fig. 4(a)) reveals deformed grains with some recrystallised grains. The above-described location-based classification methodology was applied to the recrystallised grains, and the results are shown in Fig. 4(b). ‘DRX grains at MC carbides’ (coloured red) can be found along carbide stringers. DRX grains classified as ‘DRX inside grains’ (yellow), ‘DRX at random GBs’ (green) and ‘DRX at distorted twins’ (orange) are located as their name suggests. This illustrates that the algorithm successfully classifies recrystallised grains based on their locations.

The boundaries between merged deformed grains (i.e. distorted twins) are not shown, except the segments that still fulfil the $\Sigma 3$ condition (white). This results in gaps (indicated with an orange arrow) along these boundaries which makes it easier to differentiate ‘DRX at distorted twins’ from ‘DRX grains at random GBs’.

DRX grains that are classified by the developed methodology can then be analysed separately, providing insights into the underlying recrystallisation mechanisms. To illustrate this analysis, Fig. 5 shows EBSD scans of larger areas of Alloy 2 specimens deformed at $1050\ ^\circ\text{C}$

(Fig. 5(a)–(c)) and $1100\ ^\circ\text{C}$ (Fig. 5(d)–(f)) with $0.1\ \text{s}^{-1}$ to 0.6 strain, collected with a step size of $0.4\ \mu\text{m}$. These are new plots from data published in [25] where the physical phenomena are explained in more detail. The GOS map at $1050\ ^\circ\text{C}$ (Fig. 5(a)) reveals that the microstructure predominantly consists of deformed grains ($\text{GOS} > 2^\circ$), while at $1100\ ^\circ\text{C}$ (Fig. 5(d)) necklace-type recrystallisation [43] is evident. The DRX grains ($\text{GOS} < 2^\circ$) are successfully classified depending on their location (Fig. 5(b), (e)) into ‘at MC carbides’ (coloured red), ‘within grains’ (coloured yellow), ‘at distorted twins’ (coloured orange) and ‘at random GBs’ (coloured green). The GB misorientation angle distribution between these DRX grains and their adjacent deformed grains can be seen in Fig. 5(c) for $1050\ ^\circ\text{C}$ and (f) for $1100\ ^\circ\text{C}$. DRX grains that nucleated at random GBs (coloured green) follow the Mackenzie distribution [44] for randomly oriented grains (marked in dashed lines). This random distribution is expected for DDRX grains, which usually occur at random high angle GBs [45]. At $1050\ ^\circ\text{C}$, the distribution is shifted slightly towards lower angles. This suggests that these DDRX grains nucleate via strain induced boundary migration [24]. The grains nucleated next to MC carbides (coloured red) also follow a random distribution, as expected by grains nucleated via PSN [46]. DRX grains that nucleate at distorted twins share a mix of low angle and high angle boundaries with two adjacent grains. An advanced methodology to characterise these grains nucleated at distorted twins is described below (Section 3.2.2). DRX grains that are inside deformed grains without bordering any interfaces (coloured yellow) have a distribution shifted towards lower boundary angles. This suggests these grains formed by progressive subgrain rotation [24] or via shear localization [47].

However, Fig. 5 reveals some limitations of this kind of classification of DRX grains. The ‘DRX inside grains’ have a high proportion of GB segments centred around 60° misorientation angle that fulfil the $\Sigma 3$ condition, which cannot be explained by the subgrain rotation mechanism. Some of these ‘DRX inside grains’ are found along straight lines (Fig. 5(a), (b)), indicated by yellow arrows), which suggests that these DRX grains were located along former GBs. However, these boundaries were not detected as closed, continuous high angle GBs with misorientation angles $\theta > 5^\circ$ during grain reconstruction [40]. Potentially, the

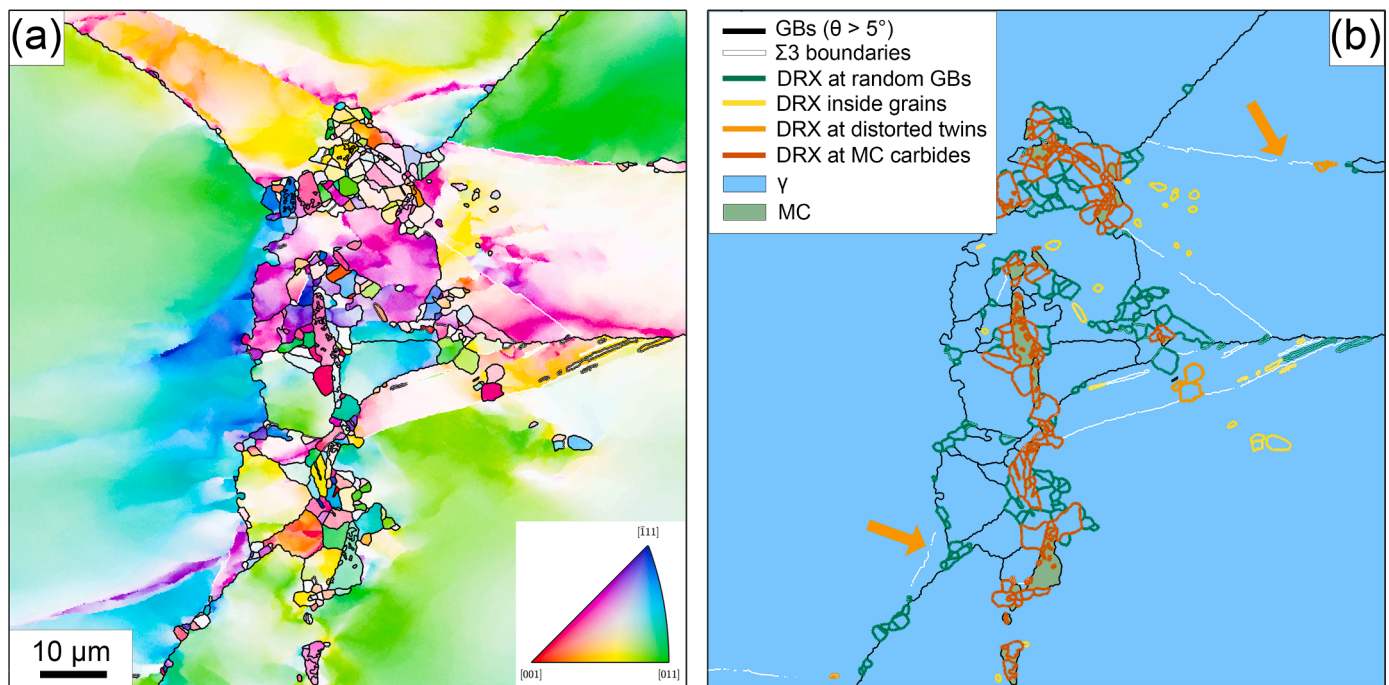


Fig. 4. Alloy 2 compressed at $1050\ ^\circ\text{C}$ with a $0.1\ \text{s}^{-1}$ strain rate to 0.6 strain; (a) IPF map in direction of deformation and (b) phase map overlaid with the results from the location-based grain classification methodology that categorised recrystallised grains. Random GBs with $\theta > 5^\circ$ are indicated in black, $\Sigma 3$ twin boundaries are marked in white. The orange arrows indicate examples of distorted twins.

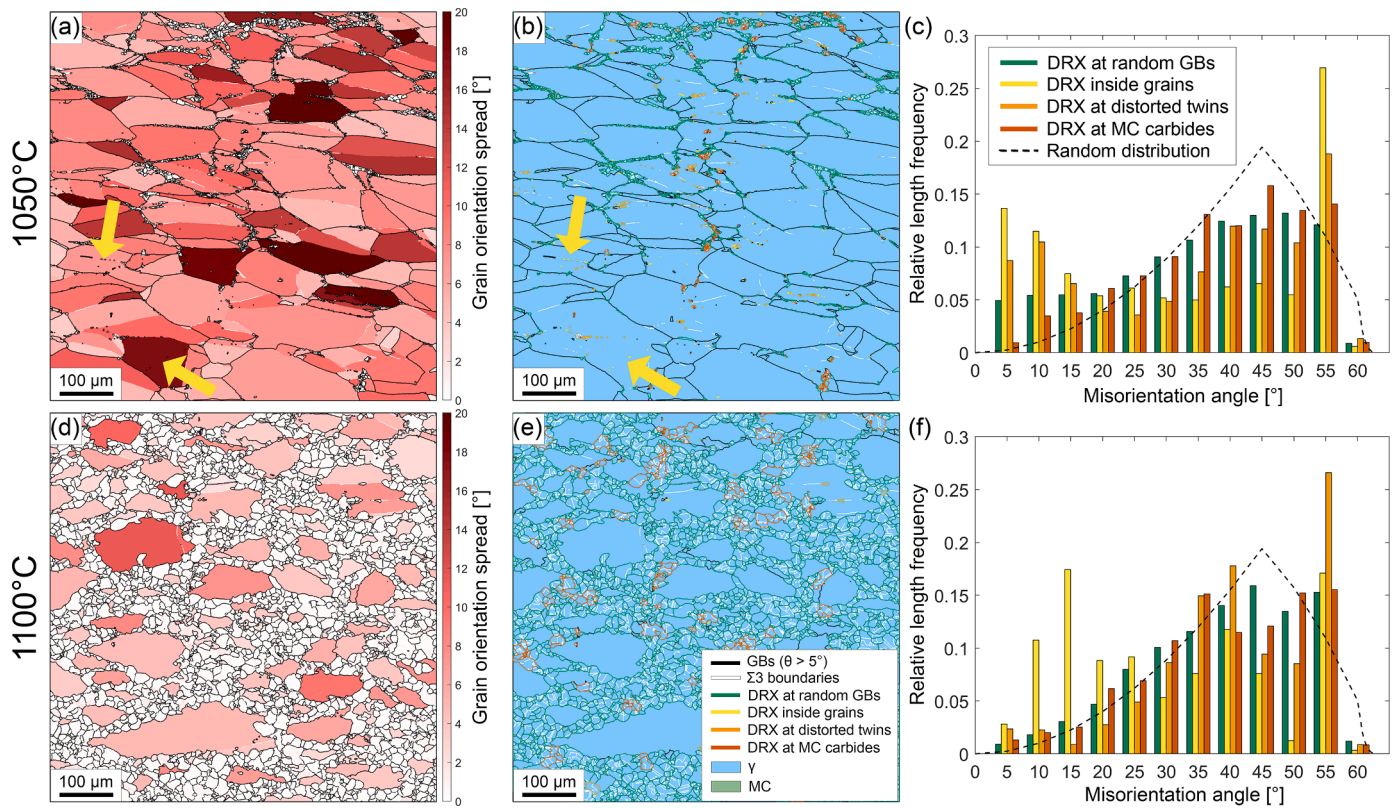


Fig. 5. Alloy 2 deformed at (a)–(c) 1050 °C and (d)–(f) 1100 °C with a 0.1 s^{-1} strain rate to 0.6 strain; (a) & (d) show the GOS before grains were merged along $\Sigma 3$ twins; (b) & (e) show the phase maps with the categorised DRX grains indicated in different colours; (c) & (f) show the misorientation angle distribution between the different categories of DRX grains and deformed grains (the order in the legend corresponds with the order in the histogram).

grains along these boundary segments deformed such that their misorientation was reduced to $< 5^\circ$. Therefore, the respective boundary segment was not detected and grains that nucleated there were wrongly assigned to the ‘DRX inside grains’ partition. The 60° misorientation angle is typical for DRX grains that nucleate at distorted twins (Section 3.2.2), suggesting that these boundaries are severely distorted twins.

Furthermore, there are some limitations in the detection of recrystallised grains that nucleated via PSN. Grains nucleated via PSN are found within the induced stress field of a particle and do not necessarily share an interface with the particle they nucleated from [46]. Without a shared interface, the here introduced location-based classification methodology will not assign these grains to ‘DRX at MC carbides’. However, in the example in Fig. 4, most grains located around carbide stringers are successfully identified. The identification worked in this case as the recrystallised γ grains are directly located next to the MC carbides, i.e. they share a common interface, and the overall volume fraction of recrystallised grains is low. Contrary, an additional limitation in highly recrystallised microstructures is that DRX grains that are classified as ‘DRX at MC carbides’ cannot be attributed to only PSN. In such cases, it is likely that recrystallised grains that nucleated at random GBs are neighbouring MC carbides and, thus, are assigned to ‘at MC carbides’ (Fig. 5(e)). This would lead to an overestimation of the ‘DRX grains at MC carbides’ volume fraction and an underestimation of the ‘DRX grains at random GBs’ one. This is more prevalent at higher deformation temperatures due to overall higher recrystallised fractions. Generally, we expect PSN to occur mostly in conditions with low degrees of recrystallisation where grains are small as otherwise DDRX grains (i.e. ‘DRX grains at random GBs’) would dominate the microstructure. Therefore, a possible solution would be to limit the ‘DRX grains at MC carbides’ partition to DRX nuclei by limiting the maximum size of grains in this partition only. Another solution would be to deactivate the detection of ‘DRX grains at MC carbides’ for microstructures where PSN

is not expected. These grains would then be automatically assigned to the ‘DRX grains at random GBs’ partition instead. This would prevent the overestimation of the ‘DRX grains at MC carbides’ volume fraction in highly recrystallised microstructures.

3.2.2. Recrystallisation mechanism at distorted twins

The GB misorientation angle distribution of recrystallised grains that nucleated at distorted twins (Fig. 5(c) & (f), coloured orange) show that these DRX grains share some low misorientation angle GB segments with deformed grains and other segments with high angles (around 60°). The example of a DRX grain that nucleated at a distorted twin (Fig. 6) suggests that each DRX grain has a lower angle GB to one deformed grain, and a higher angle GB with $\Sigma 3$ misorientation to the other. Additionally, all three grains share a close to common $\langle 111 \rangle$ direction within $< 8^\circ$ deviation. We further analysed these data to evaluate if all DRX grains at distorted twins in the microstructure share similar characteristics. Here we describe the developed methodology. The detailed results of this analysis are published elsewhere [25].

The individual steps of this automated methodology are illustrated in Fig. 7. First, all recrystallised grains are classified depending on their location (described in Section 3.2.1). Then, only grains that nucleated at distorted twins are selected. We exclude all grains that have more than two deformed neighbours (i.e. are located at triple junctions). For each of the selected recrystallised grains, we then calculate the mean orientations along these boundaries. These mean orientations are used to calculate a mean misorientation between the DRX grain and both deformed grains. Additionally, the mean misorientation between the deformed grains is calculated (coloured red). The boundaries are then assigned to two different variables based on these mean misorientations between the DRX and deformed grains. One variable contains all GBs of all selected grains with higher mean misorientation angle (coloured blue), the other one contains all GBs with the lower mean angle

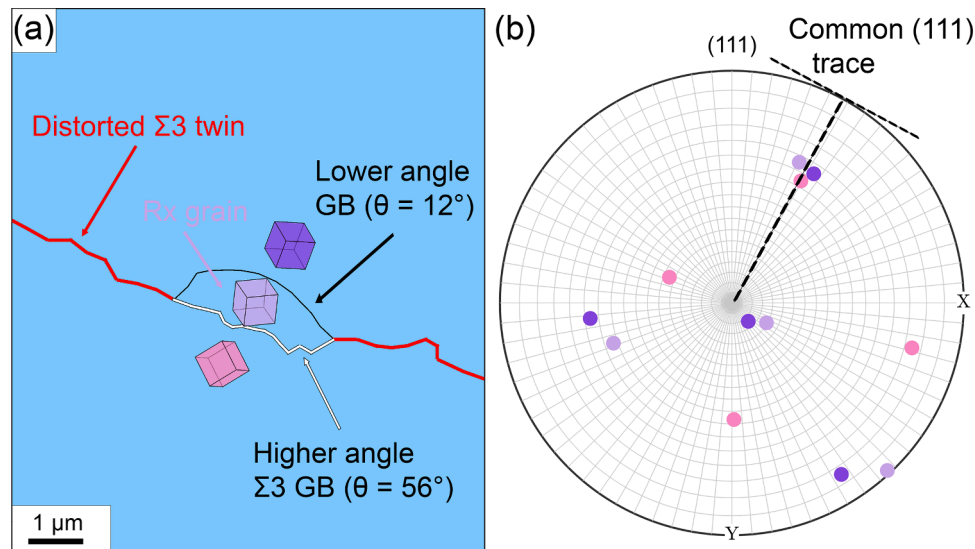


Fig. 6. Graphical example of a DRX grain that nucleated at a distorted twin; (a) the boundary map of a DRX grain between two deformed grains along a distorted twin boundary; the cubes represent the crystal orientation of the grains and (b) their orientations are indicated in the respective colours in the (111) pole figure. (reproduced after modification from [25] with permission).

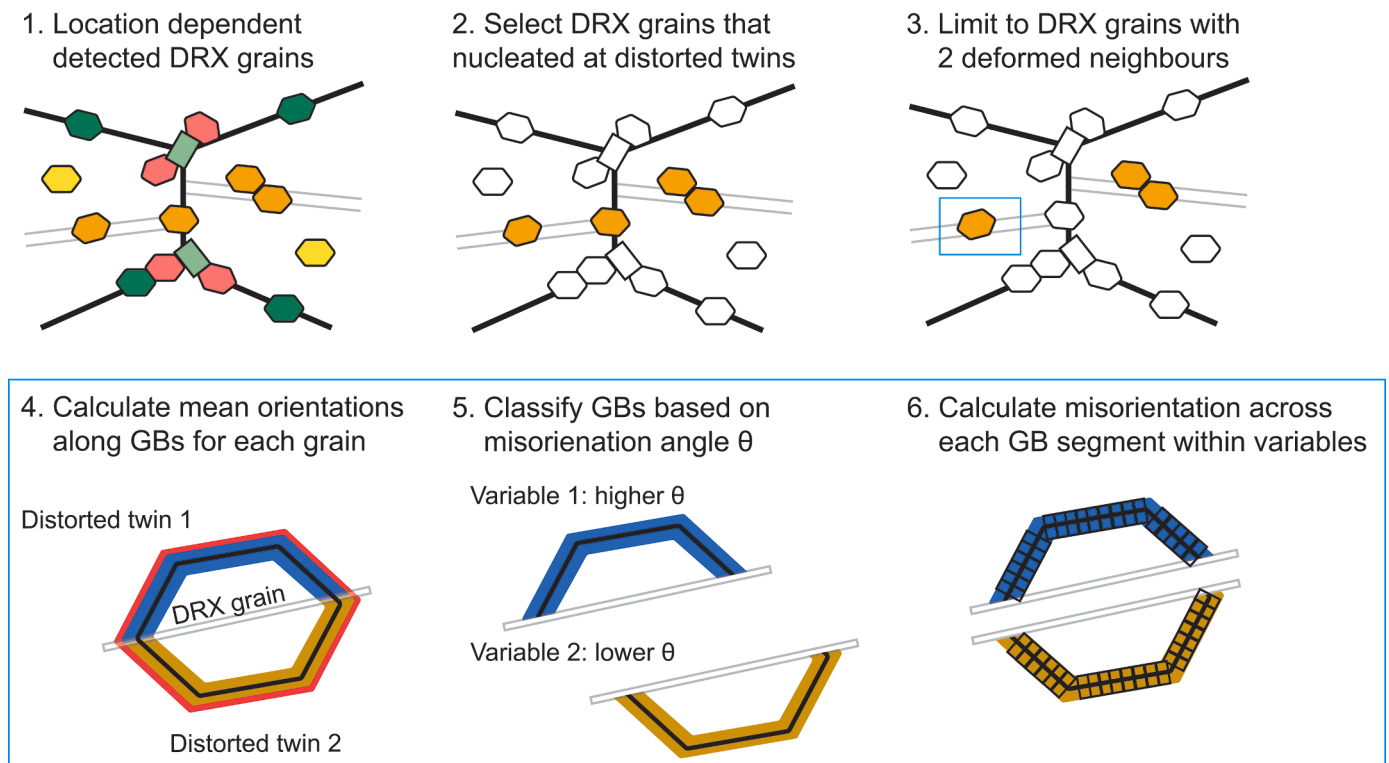


Fig. 7. Schematic of the methodology used to analyse the GB misorientation of recrystallised grains that nucleated at distorted twins.

(coloured yellow). Finally, the misorientation across each GB segment within both variables is calculated and will be used for the following analysis.

We aim to answer two questions with this analysis: (i) if all DRX grains at distorted twins have one lower and one higher angle GB to their respective deformed neighbours and (ii) if all these DRX grains share a close to common $\langle 111 \rangle$ direction with both deformed grains. We calculate the deviation of their closest $\langle 111 \rangle$ direction for each GB segment by considering the crystal orientations $\mathbf{o}_{1,i}$ and $\mathbf{o}_{2,i}$ for grain 1 and 2 along the selected GB segment i . Importantly, orientations in the

MTX convention are 3×3 matrices that transform crystal coordinates to specimen coordinates (opposite of the Bunge convention) [48]. The misorientation matrix $\mathbf{g}_i^{1 \rightarrow 2}$ that transforms $\mathbf{o}_{1,i}$ into $\mathbf{o}_{2,i}$ can be described in the following way:

$$\mathbf{g}_i^{1 \rightarrow 2} = \mathbf{o}_{2,i}^{-1} \mathbf{o}_{1,i} \quad (1)$$

The $\{111\}$ planes or $\langle 111 \rangle$ directions of orientation 1 can be transformed into the reference frame of orientation 2 via the calculated misorientation:

$$\mathbf{m}_{111,i}^{1 \rightarrow 2} = \mathbf{g}_i^{1 \rightarrow 2} \mathbf{m}_{111} \quad (2)$$

with $\mathbf{m}_{111}$ being all the symmetrical equivalent $\{111\}$ Miller planes or $\langle 111 \rangle$ directions. Then, the angle between all transformed $\mathbf{m}_{111,i}^{1 \rightarrow 2}$ and $\mathbf{m}_{111}$ of the new reference frame are calculated:

$$a_{\min,i} = \min \left(\cos^{-1} \left(\frac{\mathbf{m}_{111,i}^{1 \rightarrow 2} \cdot \mathbf{m}_{111}}{|\mathbf{m}_{111,i}^{1 \rightarrow 2}| |\mathbf{m}_{111}|} \right) \right) \quad (3)$$

The smallest calculated angle $a_{\min,i}$ represents the closest $\langle 111 \rangle$ directions or $\{111\}$ planes for the considered GB segment i .

An example of this analysis applied to solutionised and aged Alloy 1 deformed at 1050 °C with a 0.1 s⁻¹ strain rate to 0.6 strain is shown in Fig. 8. The misorientations for the GB segments between recrystallised and deformed grains are coloured in blue for the higher angle GBs, and yellow for the lower ones. The mean misorientations between the deformed grains along these GBs are indicated in red. An ideal $\Sigma 3$ boundary is expected to have a misorientation angle (Fig. 8(a)) of 60° and a common $\langle 111 \rangle$ rotation axis, thus a deviation of 0° between the closest $\langle 111 \rangle$ directions (Fig. 8(b)). The distribution for randomly oriented grains is shown as dashed lines. The random distribution for the GB misorientations is the Mackenzie distribution [44]. The random distribution for the closest $\langle 111 \rangle$ directions is simulated using Eqs. (1)–(3) with randomly generated orientations as input.

Distorted twins that provided nucleation sites for DRX grains generally deviate from the $\Sigma 3$ misorientation (coloured red). The higher angle GB (coloured blue) between the deformed and DRX grains have a misorientation angle distribution centred around 60° with shared $\langle 111 \rangle$ directions. The lower angle GBs spread around a 10° misorientation angle, and their $\langle 111 \rangle$ directions are closer than expected from a random distribution. This suggests that DRX grains at distorted twins nucleate via twinning phenomena and share a close to common $\langle 111 \rangle$ with their neighbouring deformed grains. These results confirm the observation of Fig. 6. An in-depth analysis of the contribution of distorted twins to DRX can be found in [25].

3.3. Grain boundaries

3.3.1. Twin boundary evolution during hot compression

The developed automated methodologies do not only enable characterisation of recrystallised grains that nucleated at distorted twins (chapter 3.2.2) but also provide insights on the evolution of $\Sigma 3$ (60° $\langle 111 \rangle$) annealing twins during hot deformation. Hot deformation may cause annealing twins to deviate from their original $\Sigma 3$ misorientation, which are referred to as distorted twins. Here, we define distorted twins as GBs between deformed grains that have at least one $\Sigma 3$ GB segment that still fulfils the Brandon criterion (max. 8.66° deviation [39]). These distorted twins are automatically detected following the methodology outlined in Fig. 9. First, the grains are reconstructed using the standard MTEX grain detection algorithm [40]. Deformed grains are identified by having a GOS above a specified threshold (here GOS > 2°). Then, GB segments between deformed grains that fulfil the $\Sigma 3$ misorientation within the Brandon criterion [39] are selected. Finally, entire GBs between deformed grains with at least one $\Sigma 3$ segment are classified as distorted twins.

Fig. 10 shows distorted twins that are detected by the developed methodology. The IPF map (Fig. 10(a)) shows that the deformed grains have developed $\alpha\langle 110 \rangle$ deformation texture. Between these deformed grains, only a few short segments still fulfil the $\Sigma 3$ misorientation (Fig. 10(b), coloured white). The $\Sigma 3$ segments are used to reconstruct the distorted twins (coloured red) between deformed grains. These distorted twin boundaries can now be analysed separately. The GB angle distribution of the $\Sigma 3$ segments within distorted twins (Fig. 10(c), coloured blue) is spread around a 60° misorientation angle. The distorted twin boundaries (coloured red) are also centred around 60° but deviate further when compared to their $\Sigma 3$ segments. If the identified distorted twins were random GBs, we would expect the GB angles to follow the Mackenzie distribution [44] for randomly oriented grains (indicated with dashed lines). This confirms that the developed methodology successfully identifies $\Sigma 3$ annealing twins that were distorted during hot compression. Additionally, we show that the distortion of twins (Fig. 10(d), coloured red) is accompanied with a rotation axis shift from $\langle 111 \rangle$ to $\langle 110 \rangle$. Previously, this has been shown on selected GBs only [49,50] but no statistically relevant information probing a large quantity of analysed boundaries was provided. A detailed evaluation of the phenomenon has

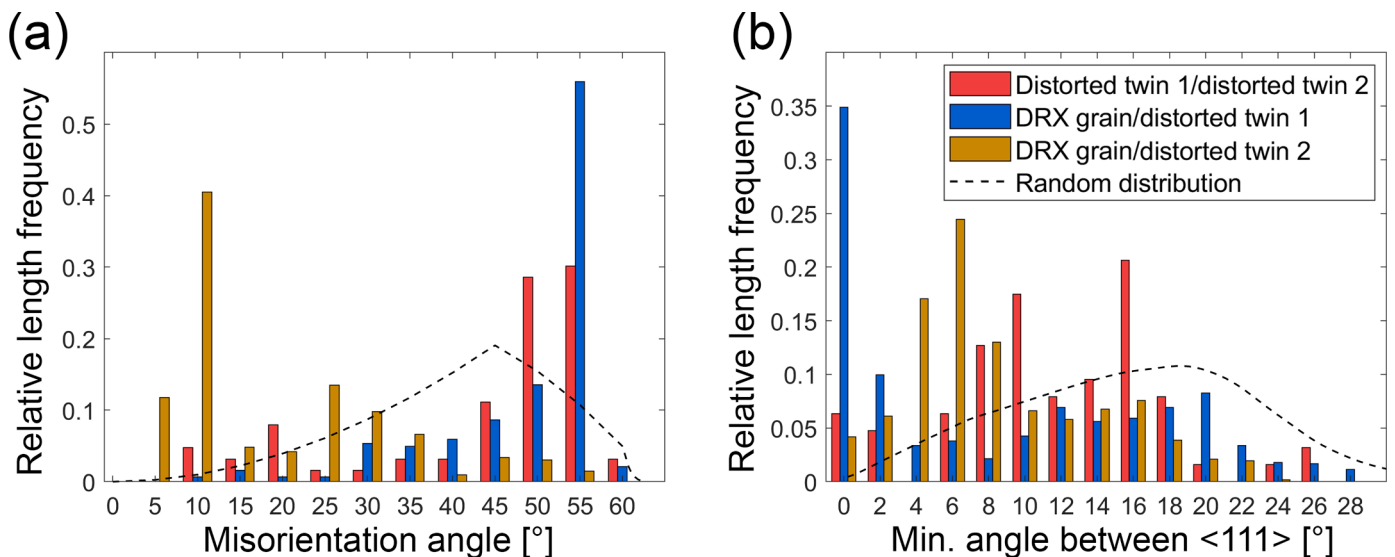


Fig. 8. Results of the GB misorientation analysis of DRX grains that nucleated at distorted twins in solutionised and aged Alloy 1 deformed at 1050 °C with a 0.1 s⁻¹ strain rate to 0.6 strain. (a) shows the misorientation angle distribution and (b) the minimum (min.) angle between the closest $\langle 111 \rangle$ crystal directions. The GBs between DRX grains and deformed grains with higher misorientation angle ('1') are shown in blue, the ones with lower misorientation angle ('2') in yellow. The mean misorientations between both distorted twins at the position of the DRX grain are shown in red. The dashed lines indicate the distribution expected for fully random microstructures.

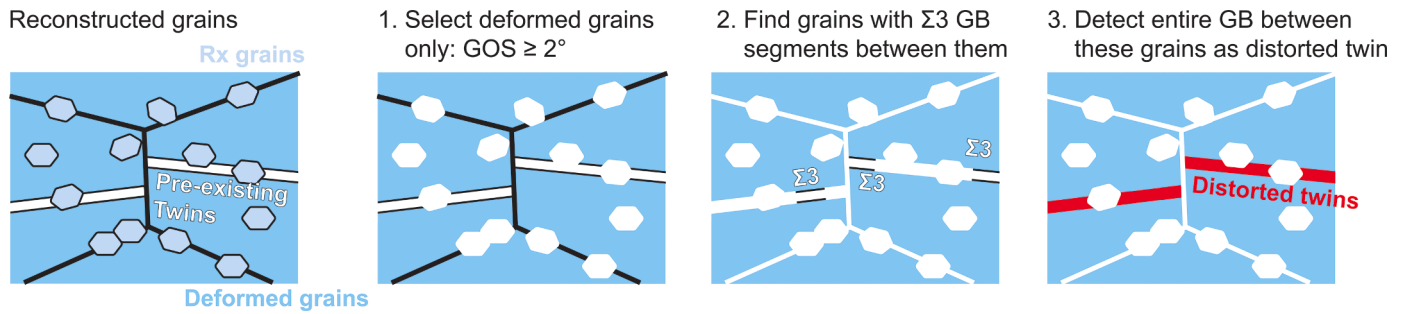


Fig. 9. Schematic of the procedure of detection of distorted $\Sigma 3$ twin boundaries.

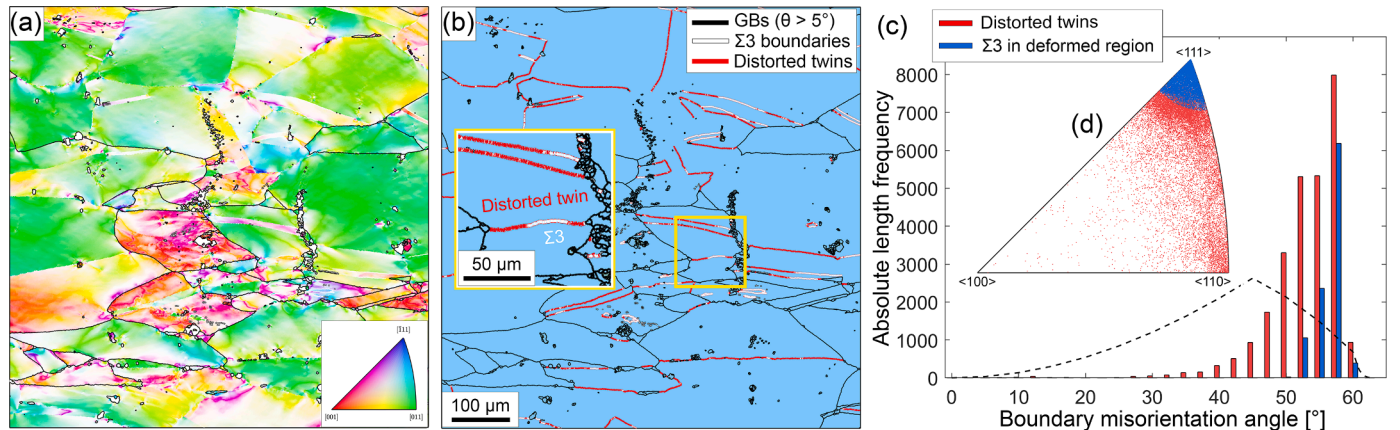


Fig. 10. EBSD maps of solutionised and aged Alloy 1 deformed at 1050 °C with a 0.1 s^{-1} strain rate to 0.6 strain; (a) IPF map in compression direction; (b) boundary map with random GBs with misorientation $\theta > 5^\circ$ (black), $\Sigma 3$ boundaries (white) and detected distorted twins (red); (c) boundary misorientation angle distribution and (d) fundamental sector projection of the misorientations for distorted twins (red) and $\Sigma 3$ boundaries between deformed grains (blue); the random GB distribution is indicated with dashed lines.

been published elsewhere [25].

As the methodology depends on identifying remaining $\Sigma 3$ segments between deformed grains, a potential limitation of the algorithm is in microstructures where $\Sigma 3$ segments are heavily distorted and/or only few of these $\Sigma 3$ segments remain. This could be the case in severely deformed microstructures, where twins are distorted and deviate too far from the $\Sigma 3$ misorientation. The methodology also cannot obtain statistically significant data on distorted twins in highly recrystallised microstructures, where most distorted twins are replaced with recrystallised grains.

3.3.2. Grain boundary plane visualisation

The GB plane plays a critical role in determining the properties of GBs [30,31]. Although a full description of the GB character requires three-dimensional boundary information [26], two-dimensional data may be able to provide an assessments of the GB plane. This information gives us insights into the coherence of $\Sigma 3$ twin boundaries. Here, we introduce a GB plane visualisation methodology to define the planes GB segments terminate on by combining information of the GB misorientation with the GB trace [30,31]. However, EBSD data are recorded on either a two-dimensional hexagonal or cube grid. Therefore, the real GB traces are not readily accessible in the as-recorded data [30]. Therefore, sufficient GB smoothing must be applied to remove the effects of the grid on the reconstructed GBs [40]. The impacts of smoothing are illustrated in the supplementary figure S 2. Here we use 100 iterations of the standard MTEX GB smoothing algorithm. This algorithm smooths the segments connecting triple junctions but leaves the location of these triple junctions unchanged. The GB plane visualisation methodology was applied on solutionised and aged Alloy 1 as an example.

After GB smoothing, we identify $\Sigma 3$ boundaries that fulfil the 60°

$\langle 111 \rangle$ misorientation (Fig. 11(a)) within 8.66° tolerance defined by the Brandon criterion [39]. This itself does not allow any conclusion on the GB plane and, thus, includes coherent and incoherent $\Sigma 3$ segments. The coherence of a $\Sigma 3$ GB segment is then evaluated by identifying the direction of its $\langle 111 \rangle$ misorientation axis in specimen coordinates, which is perpendicular to the trace of their shared $\langle 111 \rangle$ plane (Fig. 11(b)). If the trace of the common $\langle 111 \rangle$ plane and the direction of the GB segment are parallel within a threshold of 10° [31], the segment is classified as coherent (coloured white). In other words, for coherent $\Sigma 3$ twist boundaries, the shared $\langle 111 \rangle$ misorientation axis is normal within 10° to the GB segment (or parallel to the GB plane normal). If this is not the case, then the segment is classified as incoherent $\Sigma 3$ boundary (coloured red).

This GB plane visualisation methodology was applied to a $1600 \times 1600 \mu\text{m}^2$ EBSD map with step size of $3 \mu\text{m}$ (Fig. 12). The grains that are used to illustrate the methodology in Fig. 11 are highlighted in the same colours (purple) and the same methodology has been automatically applied to the recorded GB network here. Long, straight $\Sigma 3$ twin boundaries are observed to be coherent (coloured white). Steps within these boundaries and the twin fronts are of incoherent character (coloured red). The results are in agreement with what is expected for these boundaries based on the literature [51].

A further analysis is applied to estimate the possible boundary planes of the incoherent $\Sigma 3$ segments. When $\mathbf{m}_j$ are the symmetrical Miller indices of the selected planes of interest j , then these can be transformed from crystal into specimen coordinates $\mathbf{m}_j^s$ via:

$$\mathbf{m}_j^s = \mathbf{o}_{1,i} \mathbf{m}_j \quad (4)$$

with $\mathbf{o}_{1,i}$ being the orientation of one grain along the incoherent $\Sigma 3$

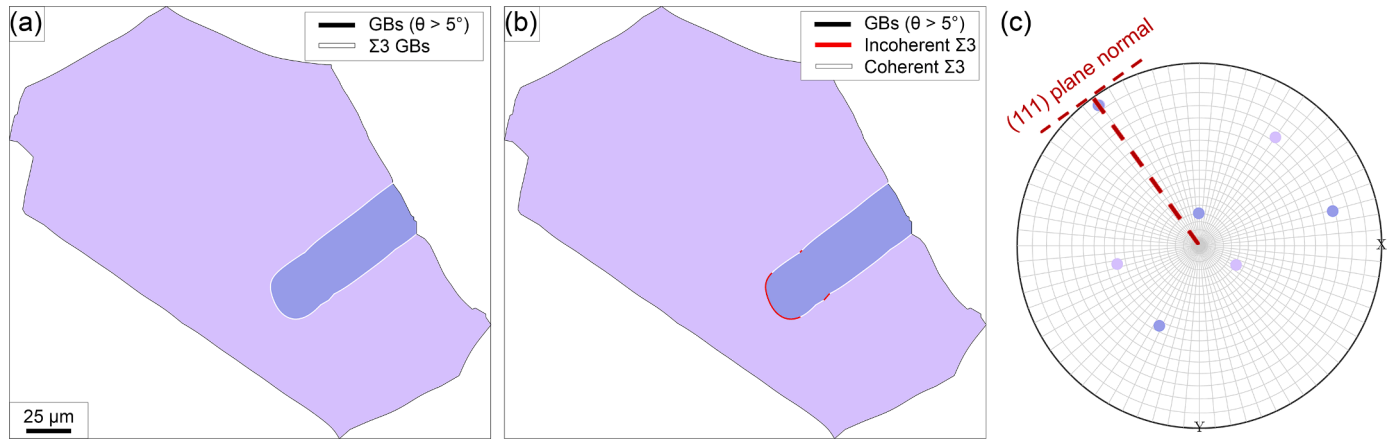


Fig. 11. (a) Identified $\Sigma 3$ GBs (coloured white) within the Brandon criterion; (b) coherent (coloured white) and incoherent $\Sigma 3$ (coloured red) boundaries differentiated by measuring angles between GB traces and (c) common (111) plane trace.

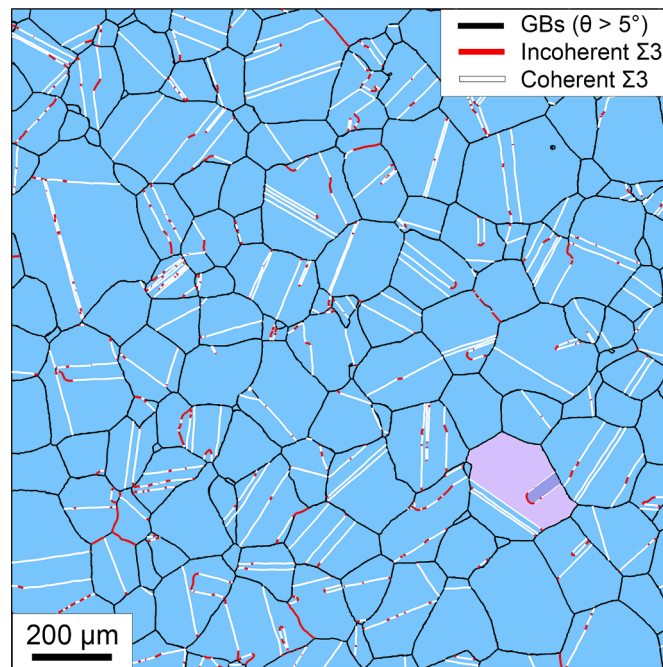


Fig. 12. Phase boundary map with random GBs with misorientation $\theta > 5^\circ$ (coloured black), incoherent $\Sigma 3$ (coloured red) and coherent $\Sigma 3$ boundaries (coloured white) of solutionised and aged Alloy 1. The grains that are used to illustrate the methodology in the other figures of this section are marked in purple.

boundary segment i .

Based on two-dimensional EBSD, we cannot identify the correct boundary plane. However, only planes with a plane trace parallel to the trace of the boundary segment $\mathbf{d}_i$ can possibly provide the correct boundary plane [52], therefore we can exclude the impossible planes. The plane trace is perpendicular to the direction of the miller index of the plane $\mathbf{m}_j^s$ in specimen coordinates. Therefore, the angle $\alpha_{min,i}^j$ between direction of the boundary segment $\mathbf{d}_i$ and the closest trace of the $\mathbf{m}_j^s$ plane is defined by:

$$\alpha_{min,i}^j = \min \left(90^\circ - \cos^{-1} \left(\frac{|\mathbf{d}_i \cdot \mathbf{m}_j^s|}{|\mathbf{d}_i| |\mathbf{m}_j^s|} \right) \right) \quad (5)$$

The common boundary planes of interest $\mathbf{m}_j$ for incoherent $\Sigma 3$ segments are the {112}, {122} and {110} planes [28,53,54]. Fig. 13(a)-(c)

show the results of the above-described analysis on a selected grain. The colour scheme indicates the smallest angle between the boundary segment and the traces of (a) {112}, (b) {122}, and (c) {110} planes. The pole figures of the respective planes for the same grain are presented in Fig. 13(d)-(f). For some segments, one plane shows a distinctly lower angle than the other planes (indicated by a yellow arrows), while for others, two planes have traces close to parallel (indicated by red arrows). In the first case (yellow arrow), it is likely that the {122} is the boundary plane, while in the latter case the {112} and {122} planes are both possible boundary planes.

Furthermore, to enable more straightforward visualisation and analysis, we can assign the GB segment i the boundary plane with the lowest trace deviation:

$$a_{min,i} = \min \left(a_{min,i}^1, a_{min,i}^0, \dots, a_{min,i}^j \right) \quad (6)$$

Importantly, by doing this, it is not immediately apparent if there is another boundary plane with a close to parallel trace. The same procedure is also applied to the orientation $\mathbf{o}_{2,i}$ of the other grain bordering the segment i . Therefore, this can lead to dissimilar results for both grains along a boundary segment as these grains have different crystallographic orientations and might terminate on different planes.

The assigned boundary planes are shown in Fig. 14. Fig. 14(a)-(b) show the boundary planes that terminate on that GB segment for the respective purple coloured grain. The coherent (111) segments are the same segments in both grains. This is due to the shared (111) trace being used for identifying these boundaries. Furthermore, some of the incoherent boundary planes are the same in both grains (an example is indicated by a yellow arrow). This is the case for symmetric GBs. While the methodology is here applied on incoherent $\Sigma 3$ segments, it can theoretically be applied to other types of GBs. Additionally, the GB plane visualisation methodology can be adopted to include any planes of interests.

Based on the averaged results of three $1600 \times 1600 \mu\text{m}^2$ maps collected with a step size of $3 \mu\text{m}$, 45 ± 4 length-% of all boundaries have a $\Sigma 3$ misorientation and 40 ± 4 length-% of all boundaries are coherent $\Sigma 3$. To evaluate the performance of our GB plane visualisation methodology, we applied the well-known five-parameter statistical stereological analysis developed by Rohrer et al. [6] to the same dataset. It results in similar fractions of total $\Sigma 3$ boundaries of 45 ± 4 length-% and coherent $\Sigma 3$ of 41 ± 4 length-%. It has previously been demonstrated that the statistical stereological analysis can accurately identify the boundary planes for different misorientations when compared to three-dimensional methods [55]. This suggests that our GB plane visualisation methodology can successfully differentiate between coherent and incoherent $\Sigma 3$ boundaries within the limitations inherent to

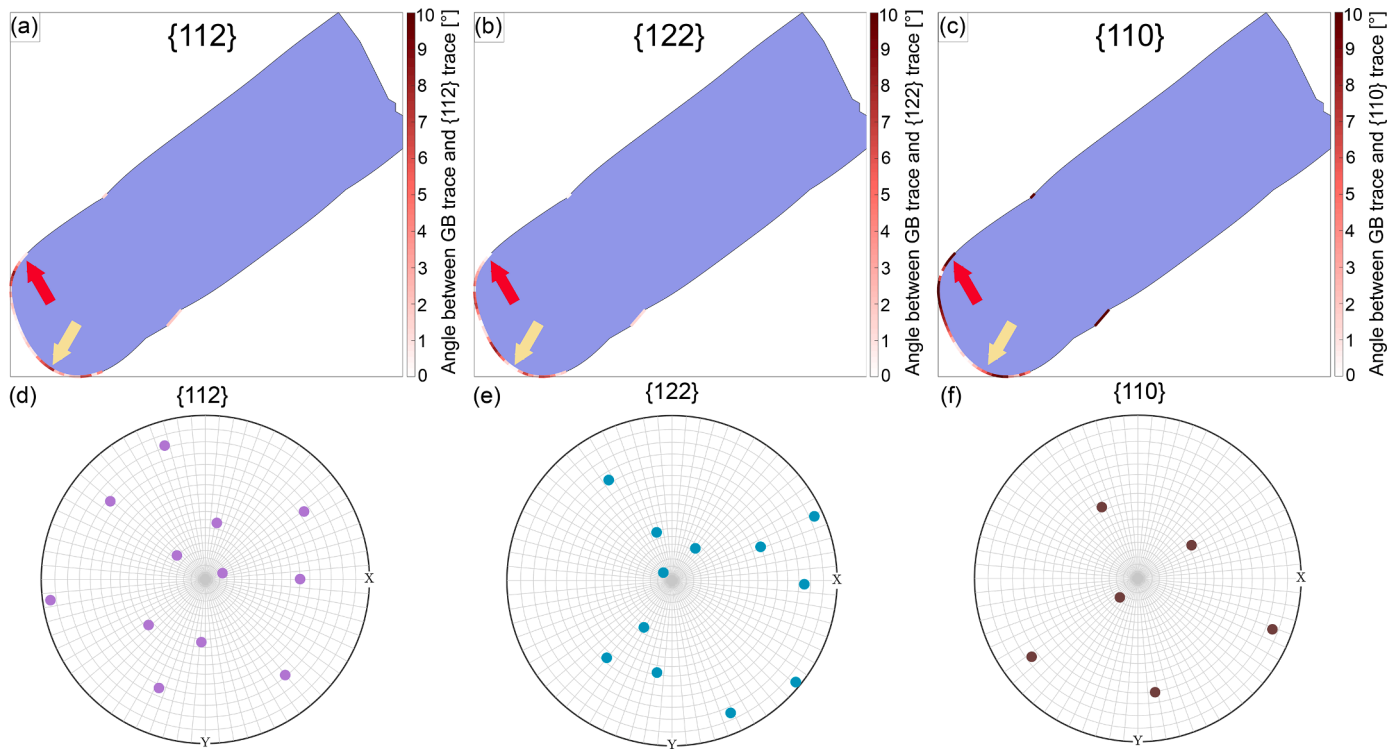


Fig. 13. Angle of the traces of selected GB planes and the GB segments for (a) {112}, (b) {122} and (c) {110}; (d-e) respective pole figures that show the GB planes.

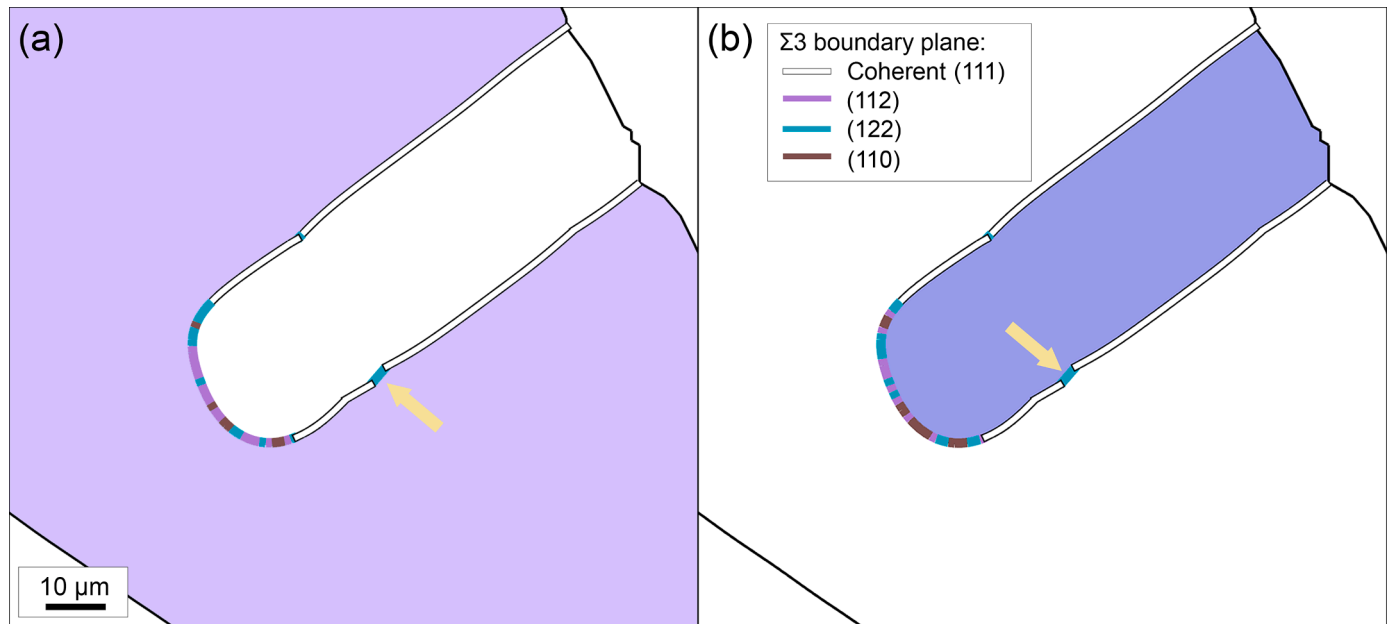


Fig. 14. GB planes with the smallest trace deviation for incoherent $\Sigma 3$ segments for (a) grain 1 (coloured light purple) and (b) grain 2 (coloured dark purple).

two-dimensional datasets. The main result of the five-parameter statistical stereological method is the GB plane distribution (see e.g., Fig. 15). Although this provides results with statistical significance, it does not graphically show results for individual GB segments. Therefore, the here introduced GB plane visualisation methodology provides helpful complementary information to link specific microstructural features to certain boundary planes.

As the GB inclination is not directly accessible via two-dimensional data, the exact plane normal cannot be determined from these data alone [52]. A full description of all five macroscopic parameters requires

three-dimensional measurement methods, which is connected to additional experimental efforts. However, the two-dimensional boundary trace of $\Sigma 3$ boundaries allows an estimate of the GB plane with high probability. Randle and Davies [31] found that for just 10% of $\Sigma 3$ boundaries two-dimensional data are not sufficient to determine if they have a (111) boundary plane.

3.4. Assessment of achievements and outlook

The here introduced methodologies can be helpful in the advanced

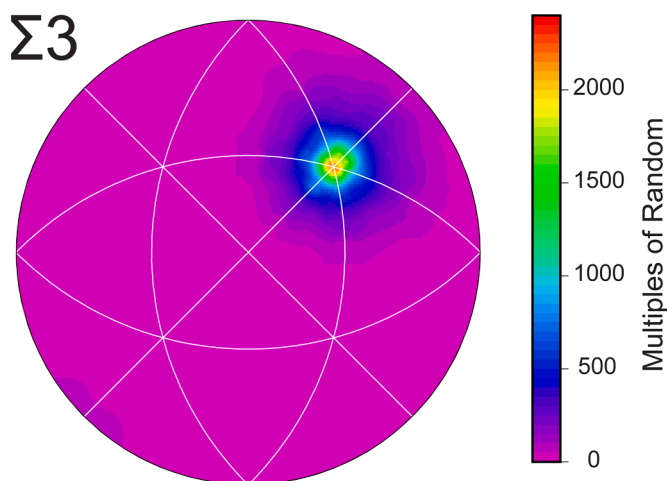


Fig. 15. GB plane distribution for $\Sigma 3$ boundaries of determined by the five-parameter statistical stereological analysis of solutionised and aged Alloy 1.

characterisation of Ni-based superalloys and other materials after deformation. There are challenges in the analysis of as-deformed microstructures due to different simultaneously acting phenomena. These may include, for example, substructure development via dynamic recovery [45] and the formation of new grains via different recrystallisation mechanisms such as CDRX [21], DDRX [22], PSN [23], and heteroepitaxial recrystallisation [42]. Additionally, these phenomena often occur over different length scales, i.e. DRX nucleation and subgrain formation in the range of a few hundred nanometres [56] whereas texture development may span over millimetres [57]. EBSD is a powerful tool to distinguish between many of these phenomena. However, previously, most of such analyses were done (semi-)manually. Although many of these previous analyses provide graphical results that are helpful aids in the visualisation of a certain mechanism, they lack statistical significance as they are difficult to quantify. Moreover, any manual method is highly time consuming for the operator. The here introduced automated methods overcome both problems. They enable fast, systematic analyses and can be implemented as standardised data evaluation procedures. Although EBSD and crystallographic characterisation techniques have been continuously improved over the years, further efforts in data mining are still needed to make the most of the information gained [3]. Advanced automated methodologies are especially needed considering the ongoing improvements in EBSD acquisition technology. Faster acquisition rates allow the collection of increasingly large datasets, and this requires automated procedures to allow for efficient data processing, and in-depth analysis of the collected microstructures.

The challenges in handling large datasets become more prevalent in 3D characterisation methods such as 3D EBSD, where multiple layers of EBSD maps are collected [58]. Such three-dimensional datasets provide valuable information that are not accessible from two-dimensional data alone. This additional complexity makes it challenging to analyse these datasets via manual methods only. Here, many challenges can be overcome by developing automated methodologies. This has recently been demonstrated with algorithms for improved 3D EBSD reconstruction and γ/γ' phase differentiation [59], or with procedures to analyse variant selection in 3D EBSD maps of titanium alloys [60]. The advanced methodologies introduced here may also be extended to be applied to 3D EBSD or transmission Kikuchi diffraction (TKD) data. This will enable in-depth analysis of recrystallised grains and provide further insights into the underlying mechanism. The here introduced methodology to determine the boundary planes would further benefit from three-dimensional data, as the complete GB inclination can be determined. This would allow to accurately assess the GB plane of boundaries.

The methodologies developed here can be applicable to many other materials systems. To provide such an outlook, it is helpful to separate the methodologies introduced here from the algorithm that implements them (accessible via [36]). This allows us to identify the following three cases: materials systems the methodologies can be applied to (i) without changes to the algorithm, (ii) with slight modifications of the algorithm, or (iii) where the methodology itself needs to be updated:

To address case (i), the interface artefact correction in combined EBSD-EDS measurements (Section 3.1) can be readily applied to any materials systems, as long as both phases have the same crystal structure but different chemistries. For example, it can be used to correct artefacts in body centred cubic high entropy alloys that precipitate a B2 ordered phase [61]. Additionally, the other developed methodologies can be applied without changes to fcc metals with low-to-medium stacking fault energy (SFE), such as stainless steel [62] or Cu [63]. As their dominant DRX mechanism is DDRX [45], the methodology to classify recrystallised grains (Section 3.2.1) can be used without modification. Further, coherent twins in these materials systems also have a $\Sigma 3$ misorientation with a (111) habit plane, thus the introduced grain boundary plane analysis (Section 3.3.2) can differentiate between coherent and incoherent twins. This also enables the analysis of distorted twins (Section 3.3.1) in these materials. For example, distorted twins have been identified in various stainless steels [49,64]. However, these results were based on the analysis of selected boundaries only and they come to ambiguous conclusions if a rotation axis shift from $\langle 111 \rangle$ to $\langle 110 \rangle$ occurs. The here developed methodology could be helpful to establish if the distortion in some systems is not accompanied by a rotation axis shift. This will provide a more comprehensive understanding of the evolution of twins during hot compression in these materials systems.

Examples for (ii) materials systems where the same methodology can be applied with minor changes of the algorithm are hexagonal close packed systems like many Mg alloys [65]. Coherent twins in these systems have a different misorientation and habit plane. However, stereological methods have been successfully applied to these systems [66]. Thus, the methodology to identify coherent twins via GB plane analysis (Section 3.3.2) should work, but the twin identity must be updated in the algorithm.

Lastly (iii), materials systems where our methodologies cannot be directly applied are for example fcc materials with high SFE such as Al [63]. The dominant DRX mechanism in these systems is CDRX [45], which has implications for the methodology that classifies recrystallised grains based on their location (Section 3.2.1). If the fraction of CDRX grains is sufficiently high, they may not be fully surrounded by deformed grains, thus, not be assigned to the 'inside grains' partition, but to the 'at random GBs' one instead. This may cause CDRX grains to be separated into two different categories. However, as DDRX at random GBs is usually not found in these systems, the 'at random GBs' partition could be simply removed. This demonstrates how the introduced methodologies can be adopted for application in a wide range of materials systems.

4. Summary

We developed automated methodologies to analyse large EBSD datasets. The algorithms are implemented using the Matlab toolbox MTEX and are available under open-source license via <https://doi.org/10.5281/zenodo.7246613> [36]. Although the algorithms have been implemented for Ni-based superalloys, they can be applied to other alloying systems such as high entropy alloys, stainless steels, copper, or light metals. Our new methodologies can be applied to analyse following microstructural features, providing statistically significant data:

- Wrongly indexed datapoints (γ/γ' interface artefacts) in combined EBSD-EDS measurements, due to the different interaction volumes, can be re-assigned to the correct phase.

- Recrystallised grains can be classified into different groups based on their location, i.e., at random GBs, at $\Sigma 3$ boundaries, inside grains, or adjacent to second phase particles. This allows to analyse the properties of each group individually and provide in-depth insights on the present nucleation mechanisms.
- The evolution of special GBs such as $\Sigma 3$ twin boundaries during hot deformation can be studied.
- The boundary planes of $\Sigma 3$ boundaries can be analysed and visualised, which allows differentiation between coherent and incoherent twins. For incoherent twins, likely alternative boundary planes can be identified.

CRedit authorship contribution statement

B. Schulz: Conceptualization, Methodology, Formal analysis, Investigation, Software, Writing – original draft, Writing – review & editing, Visualization, Project administration. **N. Haghdadi:** Methodology, Writing – review & editing. **T. Leitner:** Conceptualization, Resources, Writing – review & editing, Funding acquisition. **M. Hafok:** Conceptualization, Resources, Writing – review & editing, Funding acquisition. **S. Primig:** Conceptualization, Supervision, Resources, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

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

Data availability

The here introduced methodologies and example data are openly available under GPL-3.0-or-later on GitHub and archived in Zenodo at <https://doi.org/10.5281/zenodo.7246613>

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.ultramic.2023.113699](https://doi.org/10.1016/j.ultramic.2023.113699).

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