Triple junction disclinations in severely deformed Cu-0.4%Mg alloys
Full length article

Triple junction disclinations in severely deformed Cu–0.4%Mg alloys

Siying Zhu\textsuperscript{a}, Andrey P. Jivkov\textsuperscript{a,∗}, Elijah Borodin\textsuperscript{a}, Anna Morozova\textsuperscript{b}

\textsuperscript{a} Department of Solids and Structures, School of Engineering, The University of Manchester, Manchester M13 9PL, UK
\textsuperscript{b} Belgorod State University, Belgorod, Russia

\begin{abstract}
Stress fields arising from triple junction disclinations (TJDs) play a significant role in the microstructure evolution during the plastic deformation of metals. The calculation of TJD strengths from grain orientation data was developed by Bollmann more than 50 years ago, but so far applied only to collections of a few grains. Developed here is a new methodology for calculating TJD strengths and the associated stress fields in large polycrystalline assemblies using experimental electron back-scattered diffraction (EBSD) maps. The methodology combines Bollmann’s approach with a representation of materials as cell complexes. It is computationally efficient and allows for obtaining the spatial distribution of TJD strengths from EBSD images containing thousands of grains. Analysed are the fraction, distribution, and strengths of TJDs within statistically representative microstructures of Cu–0.4%Mg alloy subjected to severe plastic deformation (SPD) by equal channel angular pressing. It is shown that the formation of low-angle grain boundaries (dislocation walls) during SPD leads to an increasing number of TJDs, whose spatial distribution is progressively more uniform and whose strength distribution remains nearly constant. This result suggests that the SPD reduces the internal stresses associated with disclinations in large regions of the material, as closely situated disclinations screen each other’s fields. Regions with high local stresses can be expected between sparsely distributed TJDs with the highest strengths. The average distance between such TJDs could be considered as a natural length scale in a material.
\end{abstract}

1. Introduction

Local residual stresses can affect significantly physical and mechanical properties of materials, such as fracture toughness, diffusivity, thermal and electrical conductivity, and optical properties [1–3]. An important way through which they affect the functional performance is by altering the behaviour of defects and interfaces in materials [4,5]. The quantification of local stresses and the identification of their sources are challenging with the current experimental and characterisation methods. In relatively pure alloys, such as the one considered in this work Cu–0.4%Mg alloy, subjected to severe plastic deformations at low to moderate temperatures, triple junction disclinations (TJDs) are the main source of high local stresses [6–12]. Triple junctions and triple lines will be used interchangeably in this paper. The possibility to characterise and govern the spatial distribution and strengths of TJDs, alongside other types of defects (grain and phase boundaries, dislocations, vacancies), could prove essential for the engineering of new functional materials and their applications [13–15].

Historically, the concept of disclination in the theory of elasticity was first proposed by Frank, who used the term to describe rotational Volterra cuts [16–18]. In practice, disclinations were observed and widely used in studies of liquid crystals [19]. The physical theory of disclinations was developed in the second half of the 20th century [6,20–22], following the successes of dislocation and crystal plasticity theories [6,14,23,24]. Disclinations have since been used in the description of mechanical behaviour of nanocrystalline (NC) materials [12,25–30], severe plastic deformations (SPD) of polycrystalline metals [8,31–35] and in the modelling of dynamic recrystallisation (DRX) [36]. Unlike dislocations, which break the translational symmetry of a crystalline lattice, disclinations break its rotational symmetry, i.e., they represent rotational distortions in the crystal lattice [4,25,37]. From the mathematical point of view, disclinations are different from dislocations: they are defined by a rank-3 tensor containing a rotation axis and an angle, whereas dislocations are defined by a vector. One of the main features of disclinations, making them of great importance for the analysis of residual local stresses, is the high elastic stress fields associated with them and the fact that these stresses decrease only as a logarithm of the distance [24,38,39]. This is to be compared with the dislocation stress fields which decrease inversely proportionally to the distance [18,40]. Such fields affect the surrounding material and can significantly alter its mechanical response [24,41,42]. In particular,

\begin{address}
\textsuperscript{a} Corresponding author.
E-mail addresses: siying.zhu@manchester.ac.uk (S. Zhu), andrey.jivkov@manchester.ac.uk (A.P. Jivkov), elijah.borodin@manchester.ac.uk (E. Borodin).
\end{address}

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disclination-generated local stress fields can affect dislocation motion, crack initiation and propagation [27,43]. At the same time, long-range stresses created by the disclination elastic fields can be effectively compensated by disclinations of the opposite “sign” through the formation of dipoles and quadruples configurations of disclinations [24,34,44,45].

Bollmann [46] introduced triple line disclinations using the crystallographic orientations of the adjacent grains. The triple lines in polycrystalline materials are classified into U-lines that contain and L-lines that do not contain rotational incompatibility. The incompatibility at a U-line is referred to as TJD. Experimental evidence supporting Bollmann’s theory has been obtained through the identification of triple junctions exhibiting U and I crystallographic characteristics in high-purity Ni [47] and Cu-8.5%Al alloy [46]. It has been also reported that triple lines facilitate preferential corrosion relative to the adjacent grain boundaries in high-purity nickel and that these have been predominantly U-lines rather than L-lines [47]. The study of TJDs and their properties is closely intertwined with the investigation of grain boundary structures and should not be considered in isolation [48,49].

While Bollmann’s theory has been shown to be effective, it has been rarely used in the analysis of experimental data and in these rare cases, it has been limited to collections of a few grains. It is more common in crystal defect theories to represent rotational distortions by smooth and non-vanishing disclination density fields [43,50]. More recent studies have been focused on constructing such smooth disclination density fields using discrete orientation maps obtained from electron back-scattered diffraction (EBSD) measurements [51-54]. However, the EBSD maps are more suitable for implementing a different approach that combines a discrete representation of the microstructure with elements of Bollmann’s theory.

This paper presents a novel approach where the microstructure is represented by a polytopal cell complex (PCC) [55,56], which is a mathematics object studied in algebraic topology [57], and Bollmann’s theory is implemented to calculate the positions and strengths of TJDs given EBSD maps with a large number of grains. By overcoming the limitations of continuous models, this approach provides complementary information about the grain boundary network, including an opportunity to introduce a natural length scale defined by distances between disclinations with high strengths.

2. Microstructure representation

The experimental data used for the analyses in this work comprises 2D EBSD maps of Cu-0.4%Mg alloy subjected to ECAP processing [35,58]. Orientation measurements were performed using a Quanta 600 FEI SEM equipped with an EBSD detector and analysed using OIM Analysis 6 software for microstructure characterisation as described in [58,59]. In brief, the measurements were performed on samples taken after 1, 2, 4, and 8 ECAP passes. The samples were electro-polished at room temperature using an electrolyte, 75% HNO3 and 25% CH3OH, with a voltage of 10 V. The electron beam dimension, scanning rate, and cell size limited the scan step. Appropriate scanning steps were set in accordance with the empirical OIM Data Collection software tutorial and a common practice for obtaining EBSD data: a minimum 3 points reliably distinguish one grain [60]. Thus, the chosen step size proved reliable in the experimental results. The EBSD scan steps were 420 nm after 1 ECAP pass, 200 nm after two passes and 50 nm after four and eight passes.

A transmission electron microscope (TEM) JEOL JEM 2100 was used for the registration of TJDs. The misorientations between crystallites were analysed by the conventional TEM Kikuchi-line method using the converged-beam technique [44,61]. Foils for TEM investigations were prepared using an electrolyte of 75% HNO3 and 25% CH3OH at a temperature of -20 Celsius degrees using a Tenupol 5 twinjet polishing unit.

EBSD scans provide crystallographic orientations at pixels of imaged samples. Based on pixel orientations and on a user-prescribed misorientation threshold, the software determines boundaries separating regions with orientation differences larger than the threshold. Specifically for the data used here, the threshold was set to 2 degrees. Following the most commonly used classification, the boundaries between regions with misorientations between 2 and 15 degrees were classified as low-angle grain boundaries (LAGBs), while the boundaries between regions with misorientations larger than 15 degrees were classified as high-angle grain boundaries (HAGBs). Notably, these boundaries are just surface traces of LAGBs or HAGBs in the 3D microstructure and appear as fuzzy regions due to measurement noise.

A region surrounded by boundaries will be referred to as cell in this work. The reason for this naming convention is that the measured 2D regions are surface traces of 3D regions of unknown type — they could be grains or sub-grains. The term surface trace is used to describe the geometric object visible on the surface after making a cut through a volume of material. Each cell contains pixels with orientation differences smaller than the threshold. A single orientation for each cell is determined by the OIM software using an averaging procedure. The software also provides the barycentres of cells. In addition, it defines in a specific way a “grain” structure, where grains are regions surrounded by HAGBs and LAGBs containing one or more cells. This is done by the linear intercept method with orthogonal grids on the images using distances between HAGBs. The “grain” structure is determined by the barycentres and orientations of grains. Notably, the grain structure is algorithm-specific; it omits boundaries (HAGBs and LAGBs) that are inside “grains” and involves further averaging of crystallographic orientations within “grains”. The grain structure will be used in this work to illustrate the effect of these approximations on the calculated disclination strengths.

The experimentally determined assemblies of cells and “grains” are not very convenient for the mathematical analysis of imaged microstructures because of the noise and curvatures of HAGBs and LAGBs. More convenient are representations based on tessellations of space, i.e., partitioning of spatial domains into assemblies of polytopes — polygons in 2D and polyhedrons in 3D [62], similarly to the ones employed by the widely used software for microstructure analysis in the MTEX project [63]. The methods for generating quasi-random non-periodic structures include Poisson–Voronoi, Laguerre–Voronoi, Controlled Poisson–Voronoi, and Weaire–Phelan tessellations. The method used in this study is an optimised Laguerre–Voronoi tessellation implemented in the open-source software Neper [64]. The barycentres of the experimentally characterised cells and “grains” after 1, 2, 4, and 8 ECAP passes are used as seed points for tessellations. The experimental cell or “grain” crystallographic orientations are assigned to the corresponding polygons of the assemblies. It is recognised that this process represents GBs that in reality maybe curved and have physical width with line segments in 2D. However, the loss of this geometric information does not affect the calculations of GB misorientations and TJ disclinations, as well as the statistical results obtained in the work.

During the ECAP passes, the average grain size of the initial coarse-grained Cu-0.4%Mg alloys decreases up to 10,000 times, from a few millimetres to 200–500 nm. While several complex processes at the nanoscale (e.g., self-diffusion, GB migration, and dislocation structure evolution) contribute to the microstructure evolution, it is well established that dynamic recrystallisation (DRX) is the main mechanism for microstructure changes at the polycrystalline scale. In particular, at low temperatures, typical for the considered ECAP pressing in copper alloys, the dominant mechanism is continuous DRX or CDRX. The observed in our experiments simultaneous growth in the number density of both LAGBs and HAGBs contained one or more cells. This is done by the linear intercept method with orthogonal grids on the images using distances between HAGBs. The “grain” structure is determined by the barycentres and orientations of grains. Notably, the grain structure is algorithm-specific; it omits boundaries (HAGBs and LAGBs) that are inside “grains” and involves further averaging of crystallographic orientations within “grains”. The grain structure will be used in this work to illustrate the effect of these approximations on the calculated disclination strengths.

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Fig. 1. Cell structure evolution in Cu–0.4%Mg alloy during ECAP: (a) pass 1, 100 μm×100 μm; (b) pass 2, 100 μm×100 μm; (c) pass 4, 50 μm×50 μm; (d) pass 8, 40 μm×40 μm. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

or “grains”, grain boundaries (GBs), and triple junctions (TJs) [67]. Notably, they are not exact replicas of the corresponding EBSD maps, but are topologically very similar: the numbers of cells or “grains” are the same, and the connectivity between them is very close to the experimental maps. A potential difference may come from the fact that the vertices in a Voronoi tessellation are meets of three edges (except possibly at domain corners), while the real microstructure might contain points where more than three edges meet. This situation is energetically unfavourable, and in alloys, such instances are very rare [68]. It is therefore accepted that the assemblies created by Voronoi tessellations are sufficiently representative for the analysis in the present work.

The assemblies created by Voronoi tessellations are geometric realisations of combinatorial structures referred to as cell complexes — collections of cells of different topological dimensions glued according to specific rules [57]. In particular, the assemblies considered here are 2-complexes, i.e., cell complexes of topological dimension 2. They are built of 2-cells (the polygons representing material cells or “grains”), 1-cells (the edges representing material GBs), and 0-cells (the vertices representing material TJs). The topology of a cell complex is uniquely described by a set of matrices encoding the connectivity between cells of different dimensions. These form the algebraic representation of a given cell complex. For convenience, the algebraic representation will be referred to as a polytopal cell complex (PCC). Algebraic representations provide tools for the topological analysis of cell complexes [57], and have been used recently in mathematical formulations of physical phenomena on discrete spaces [69,70]. They simplify significantly the calculations in this work. An in-house code, Voronoi PCC Analyser [71], is used to construct PCCs from tessellation data.

The binary classification of GBs into HAGBs and LAGBs leads to a classification of TJs into four types, based on the number of adjacent HAGBs: a junction of type \( p \) is a meet of \( p \) HAGBs, where \( p \in \{0,1,2,3\} \) [55,72–74]. The TJs however can be classified in a different manner. Lattice incompatibility at GBs can be represented by dislocation networks described by Bollmann’s O-lattice theory [75,76]. The branching of dislocation networks at a triple junction is then...
calculated from the networks of the three adjacent GBs [77,78]. Such calculation leads to two possibilities: either the dislocation networks at three GBs balance at their common TJ, in which case the TJ is referred to as \textit{I-junction}, or they are unbalanced, in which case the TJ is referred to as \textit{U-junction}. It has been shown that U-junctions can be interpreted as disclinations [77]. The calculation of TJ’s dislocation balance from the crystallographic orientations of the three adjacent grains/sub-grains can be performed using the concept of nearest neighbour relationships (NNRs). The mathematical formalism for determining NNRs using experimentally measured grain orientations is described in Bollmann’s original works [75,77,78]. A brief overview is offered in the next section.

Importantly, the development in this work is based on the concept of “contours” around TJs, where any walk across the three adjacent grains in the absence of TJDs must be a closed loop [74]. Moreover, even high-order contours like the ones listed in the work [79] are also possible. Therefore, the provided methodology for calculating TJDs is geometrically precise and does not involve any approximations.

However, the elastic fields associated with TJDs may depend on further nano- and micro-scale factors, such as GBs’ curvatures in the vicinity of a TJD or non-equilibrium states of the adjacent GBs. These factors can be added in the future only to change the effect of TJDs on the local stress fields.

3. Bollmann’s triple junction disclinations

Fig. 3 shows a schematic of a TJ neighbourhood, considered with respect to a reference frame, denoted by $I$; this also represents the identity matrix/operator. In this section only, the areas referred to as cells in the rest of the paper will be referred to as grains, to avoid confusion with the crystallographic unit cells. The orientations of the three grains meeting at the TJ are given by the rotation matrices/operators $g_1$, $R_2$, and $g_3$. The misorientations between the grains, i.e., the rotations across the boundaries, are $R_1 = g_2 g_1^{-1}$, $R_2 = g_3 g_2^{-1}$, and $R_3 = g_1 g_3^{-1}$. The TJ cannot be characterised using these misorientations because $R_1 R_2 R_3 = I$, which is a consequence of taking the misorientations...
with respect to one reference system \( \mathbf{I} \). The TJ characterisation requires additional information about the dislocation structures at GBs.

Bollmann proposed a calculation of the dislocation structure at a GB using his O-lattice method \([75]\). The idea is to represent the misorientations between grains as rotations with respect to the crystal lattice coordinate system and calculate the minimal possible deviation between a unit cell in one grain and a rotated unit cell in the neighbouring grain. The minimal possible deviation determines the maximal spacing between dislocations and correspondingly defines the dislocation structure with minimal energy at the common boundary. The calculation of rotations with respect to the crystal lattice system uses the so-called structure matrix, \( \mathbf{S} \), which transforms operators defined in the orthonormal system \( \mathbf{I} \) into operators defined in the crystal lattice coordinate system.

The unit cell in face-centred cubic (FCC) crystals has eight corners \( C_{i,k} \), \( k = 1, 2, 3, 4, 5, 6, 7, 8 \), with crystal coordinates \((0 \ 0 \ 0)\), \((0 \ 0 \ 1)\), \((0 \ 1 \ 0)\), \((0 \ 0 \ 0)\), \((0 \ 1 \ 0)\), \((0 \ 1 \ 0)\), \((0 \ 1 \ 0)\), \((1 \ 1 \ 1)\), where the lattice constant \( a \) is taken as unity. These are referred to as external coordinates. Points inside a unit cell denoted by \( \mathbf{X} \), have crystal system coordinates \( 0 \leq x_i < 1 \ (i = 1, 2, 3) \), referred to as the internal coordinates. The structure matrix for FCC crystals is given by

\[
\mathbf{S} = \begin{pmatrix}
0.5 & -0.5 & 0 \\
0.5 & 0.5 & 0.5 \\
0 & 0 & 0.5
\end{pmatrix}, \quad \det(\mathbf{S}) = 1/4,
\]

and the metric tensor of the crystal lattice is \( \mathbf{G} = \mathbf{S}^T \mathbf{S} \). Using the structure matrix, the matrices/operators rotating unit cells between grains are given by:

\[
\mathbf{Q}_i = \mathbf{S}^{-1} \mathbf{R}_i \mathbf{S}, \quad i = 1, 2, 3.
\]

Let \( \mathbf{Q} \) be the rotation matrix for a given GB between grain A and grain B. Its column vectors are the crystal coordinates of the rotated unit vectors of A. In order to calculate the minimal deviation between the rotated unit cell and the unit cell in B, \( \mathbf{Q} \) is first decomposed into matrices with external and internal coordinates:

\[
\mathbf{Q} = \mathbf{M}_F + \mathbf{M}_I,
\]

where the components of \( \mathbf{M}_F \) are integers (positive, negative, or zero), while the components of \( \mathbf{M}_I \) are \( 0 \leq x_i < 1 \). Let \( \mathbf{X}_j \) denote the \( j \)-th column vector of \( \mathbf{M}_F \). Nearest-neighbours between the corners of a rotated unit cell of A and the corners of a unit cell of B are found by calculating the squared distances between \( \mathbf{X}_j \) and the corners \( \mathbf{C}_k \):

\[
D_{jk} = (\mathbf{X}_j - \mathbf{C}_k)^T \mathbf{G} (\mathbf{X}_j - \mathbf{C}_k),
\]

The smallest of these distances determines the nearest neighbours \( \mathbf{X}_j \) and \( \mathbf{C}_k \). The vector \( \mathbf{C}_k \) is added to the \( j \)-th column of \( \mathbf{M}_F \) to produce a modified matrix of external coordinates \( \mathbf{M}_F \), and a transformation matrix \( \mathbf{M} = \mathbf{M}_F + \mathbf{M}_I \). This matrix must be unimodular, i.e., \( \det(\mathbf{M}) = \pm 1 \), because the volume of the rotated unit cell must be preserved. In rare cases, the selection of the smallest values \( D_{jk} \) leads to \( \det(\mathbf{M}) \in \{0, 2\} \). In such cases, the unit cell collapses to zero volume or expands to double volume, neither of which is physically meaningful. It is therefore practical to select a larger value of \( D_{jk} \) which gives \( \det(\mathbf{M}) = \pm 1 \). The inverse of the unimodular transformation matrix \( \mathbf{M} \) is a matrix \( \mathbf{U} \) describing the dislocation structure at the selected GB; \( \mathbf{U} = \mathbf{M}^{-1} \) is also unimodular.

Let \( \mathbf{U}_1, \mathbf{U}_2, \) and \( \mathbf{U}_3 \) describe the dislocation structures at the GBs calculated from the transitions between grains; these correspond to the rotations \( \mathbf{R}_1, \mathbf{R}_2, \) and \( \mathbf{R}_3 \) in Fig. 3. The balance of dislocation structures at the TJ is represented by the matrix

\[
\mathbf{T}_{123} = \mathbf{U}_1 \mathbf{U}_2 \mathbf{U}_3.
\]

There are two possible cases for this matrix: (1) \( \mathbf{T}_{123} = \mathbf{I} \), in which case the dislocation structures of the three GB balance at the TJ, indicating no disclination; and (2) \( \mathbf{T}_{123} \neq \mathbf{I} \), in which case the dislocation structures of the three GB do not balance at the TJ indicating disclination. TJs with \( \mathbf{T}_{123} = \mathbf{I} \) are referred to as \( I \)-junctions, while TJs with \( \mathbf{T}_{123} \neq \mathbf{I} \) are referred to as \( U \)-junctions. The disclinations at \( U \)-junctions will be referred to as triple junction disclinations (TJD).

At this point, the concept of TJD is purely geometrical. Energy associated with disclinations could be defined similarly to the energy associated with dislocations, where the Burgers vector \( \mathbf{b} \) and the shear modulus \( \mu \) define the energy of a dislocation line as \( \mu \mathbf{b}^2 \). The matrix \( \mathbf{T}_{123} \) can be considered to be an analogue of the Burgers vector. It is therefore proposed that the energy associated with a TJ with matrix \( \mathbf{T}_{123} \) is proportional to the parameter

\[
P = \det[(\mathbf{T}_{123} - \mathbf{I})^T (\mathbf{T}_{123} - \mathbf{I})],
\]

which corresponds to the factor \( \mathbf{b}^2 \) in the formulation of the energy of a dislocation line \([78]\). The determinant is taken with respect to the
The numbers of cells, "grains", and triple junctions are given with respect to a 50 μm × 50 μm region. They are obtained by re-scaling the numbers counted in the imaged regions, whose areas are given in the captions of Figs. 1 and 2. This is done to facilitate comparison between microstructures after different passes. Further, it should be noted that the domains imaged after certain passes for the cell and the “grain” structures are different and they do not contain the same material as the domains imaged prior to these passes. This might explain the result that the number of “grains” and their average sizes are not increasing monotonically with passes. However, this result might also be due to the unreliability of the algorithm defining “grains”. Nevertheless, the number of features available for analysis is sufficiently large to consider the results for TJDs as representative. The characterisation reveals several general trends. First, the number of cells in the cell structure increases and the average cell size decreases with passes. This shows that consecutive ECAP passes generate new boundaries (dislocation walls) that refine the cells. Second, the number of “grains” in the “grain” structure increases and the average grain size decreases with passes. This shows that the SPD process also converts LAGBs into HAGBs by cell rotations so that the algorithm characterises smaller regions as “grains”. Third, the fraction of TJs in the cell structure classified as disclinations increases insignificantly, and the maximum strength amongst TJDs remains constant. This maximum disclination strength is achieved after one ECAP pass, or perhaps existed prior to the SPD process, suggesting that the generation of new dislocation walls and cell rotations in the subsequent ECAP passes proceeds in such a way as to maintain the maximum strength, albeit with an increasing number of TJDs. Fourth, the fraction of TJs in the “grain” structure classified as disclinations remains approximately constant, slightly larger than that in the cell structure, while the maximum strength amongst TJDs increases significantly. These observations show the very strong effect of the dislocation walls. If some of the LAGBs forming the cell structure are not accounted for, as in the “grain” structure, the calculated maximum disclination strengths could be three or more times larger. Therefore, “grain” structures constructed by algorithms, such as the one used for this work, should be considered approximations that may not be appropriate for all characterisation purposes.

The fractions of disclinations with different strengths (probability density) after the four passes are shown in Figs. 5 and 6, for the cell and the “grain” structures respectively. To improve readability, the x-axis represents the square root of the strengths, and the y-axis is in logarithmic scale. Further, the numbers of disclinations with every measured strength are given at the end of the corresponding bars. These numbers are given with respect to a 50 μm × 50 μm region as in Table 2. Fig. 5 shows that the distribution of strengths in the cell structure is largely unaffected by the ECAP process. The almost linear relation between the probability density (in log scale) and the (square-root of) strengths suggests that the distributions of strengths follow a power law which is an invariant of the microstructure evolution. The new disclinations generated with the ECAP passes are almost exclusively of low strengths (P ≤ 16); the number of higher-strength disclinations (P > 16) remains nearly constant. Fig. 6 shows that the distribution of strengths in the “grain” structure may vary significantly during the ECAP process, particularly in the region of medium strengths (16 ≤ 𝑃 < 100).

### Table 1

Axes and rotation angles for adjacent cells in Cu–0.4%Mg alloy after 1 ECAP pass.

<table>
<thead>
<tr>
<th>Measured points</th>
<th>Angle, °</th>
<th>Axis</th>
<th>[u v w]</th>
<th>[u v w]</th>
</tr>
</thead>
<tbody>
<tr>
<td>2–3</td>
<td>8.86</td>
<td>-1</td>
<td>-0.1723</td>
<td>-0.1522</td>
</tr>
<tr>
<td>4–5</td>
<td>6.25</td>
<td>1</td>
<td>0.1243</td>
<td>0.1470</td>
</tr>
<tr>
<td>6–1</td>
<td>3.41</td>
<td>2</td>
<td>0.7016</td>
<td>0.6198</td>
</tr>
<tr>
<td>TJD</td>
<td>7.2</td>
<td>1</td>
<td>0.6536</td>
<td>0.6146</td>
</tr>
</tbody>
</table>

**Axes and rotation angles for adjacent cells in Cu–0.4%Mg alloy after 1 ECAP pass.**

**Fig. 4.** Cell structure of Cu–0.4%Mg alloy after 1 ECAP pass: TEM image of a disclination localised at a TJ.

<table>
<thead>
<tr>
<th>Structure</th>
<th>History</th>
<th>𝑑 (μm)</th>
<th>𝑁_{cell}</th>
<th>𝑁_{TJD}</th>
<th>(f_{TJD})</th>
<th>(P_{\text{cell}})</th>
<th>(P_{\text{TJD}})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell</td>
<td>pass1</td>
<td>0.8326</td>
<td>2029</td>
<td>4058</td>
<td>0.6681</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass2</td>
<td>0.8568</td>
<td>2424</td>
<td>4848</td>
<td>0.6718</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass4</td>
<td>0.5296</td>
<td>8203</td>
<td>16408</td>
<td>0.6741</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass8</td>
<td>0.3997</td>
<td>13170</td>
<td>26344</td>
<td>0.6773</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td>“Grain”</td>
<td>pass1</td>
<td>1.1522</td>
<td>1500</td>
<td>3001</td>
<td>0.7137</td>
<td>81</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass2</td>
<td>0.9227</td>
<td>2111</td>
<td>4224</td>
<td>0.7562</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass4</td>
<td>0.3785</td>
<td>13778</td>
<td>27558</td>
<td>0.7448</td>
<td>196</td>
<td></td>
</tr>
<tr>
<td></td>
<td>pass8</td>
<td>0.4885</td>
<td>9865</td>
<td>19732</td>
<td>0.7533</td>
<td>100</td>
<td></td>
</tr>
</tbody>
</table>

**Elements of the cell and “grain” structures after ECAP passes: average cell or “grain” size 𝑑, number of cells or “grains” \(N_{cell}\), number of TJs \(N_{TJD}\), fraction of TJs classified as disclinations \(f_{TJD}\), maximum disclination strength \(P_{\text{cell}}\), \(P_{\text{TJD}}\) are re-scaled from the numbers counted in the imaged areas to a common area of 50 μm × 50 μm.**
There is no clear linear or other monotonic between the probability density and the strengths. These differences from the cell structures illustrate again the effect of omitting internal boundaries in the construction of a "grain" structure.

The elastic field created by a single TJD can be expressed by the Airy stress function [18]:

\[
\Psi_D(r) = D \cdot \omega(P) \left( r^2 \ln \frac{r}{R} - \frac{r^2}{2} \right),
\]

\( P \leq 81 \).
where $D = \mu / 2\pi(1 - \nu)$, with $\mu$ being the shear modulus and $\nu$ the Poisson ratio of the material, $\omega(P)$ is a function of the disclination strength $P$, $r$ is the distance from the disclination axis and $R > r$ is the screening radius considered as the average distance between disclinations of similar strength. The stress components are derived from the Airy stress function:

$$
\sigma^{D}_{rr} = \frac{1}{r} \frac{\partial \psi^{D}}{\partial r} + \frac{1}{r^2} \frac{\partial^2 \psi^{D}}{\partial \varphi^2} = D \cdot \omega(P) \ln \frac{r}{R},
$$

$$
\sigma^{D}_{\theta\theta} = \frac{\partial^2 \psi^{D}}{\partial r^2} = D \cdot \omega(P) \left( \ln \frac{r}{R} + 1 \right),
$$

$$
\sigma^{D}_{r\theta} = -\frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial \psi^{D}}{\partial \varphi} \right) = 0.
$$

These show that disclinations are sources of significant elastic stresses, which decrease as logarithms of a distance. The stress field created by a disclination can be effectively screened only by disclinations of similar strengths and opposite signs. Effectively, this leads to relatively low average stresses throughout the material volume, but with considerable local stresses near disclinations with stress gradients that depend on the average distances between disclinations of similar strengths and opposite signs. The method for calculating disclination strengths proposed here does not provide signs of disclination. As a first approximation, one can assume that disclinations of similar strength have opposite signs, and therefore screen each other, when the distance between them is less than the distance to any other disclination of the same strength.

The analysis of the disclination strengths is geometrically rigorous for the detection of the crystalline lattice incompatibilities as the sources of elastic stresses. However, the full characterisation of the local residual stresses can be performed only after taking into account further mechanisms that might relax the stresses due to disclinations. Examples of such mechanisms are the creation of non-equilibrium GBs during SPD, nano-scale curvature, and facetting of GBs in the vicinity of their TJs, redistribution of alloying elements, nano-pores, and others. The effect of these mechanisms on local stress relaxation and their statistical significance require considerable future experimental and theoretical efforts.

Figs. 7 and 8 show the spatial distributions and strengths of disclinations in the cell and ”grain” structures, respectively, after 1, 2, 4, and 8 ECAP passes. The cell structure after 1 pass contains non-uniformly distributed TJDs (shown by side bars) with a random distribution of a few TJDs of maximal strength (largest dots). After subsequent ECAP passes, the TJDs distribution becomes more uniform (shown by side bars) without noticeable changes in the number of TJDs of maximal strength (largest dots) but with a substantial increase of total disclination strengths after 4 and 8 passes (side bars). Considering the relatively uniform distribution of TJDs after several passes, it can be considered that those of lower strengths screen closely situated ones of similar strengths, so that the average stresses are small in large parts.
of the domains. However, the disclinations of higher strengths may be responsible for elevated local stresses when situated sufficiently apart as those encountered after pass 4.

The “grain” structure after 1 pass has larger grains resulting in a sparse and non-uniform distribution of TJDs. Because of the sparse distribution and relatively high strength of some TJDs, it could be inferred that this structure, if it were the realistic one, would have inner regions with significant stresses. This situation does not change noticeably after 2 passes, but after 4 passes, when the smallest grain sizes are approached, the distribution of TJDs becomes nearly uniform as in the cell structure. Despite the presence of TJDs with significantly higher strength after 4 and 8 passes, the total strengths in the regions shown by the side bars are smaller than those in the cell structure, which shows again the strong effect of omitting many boundaries in the construction of the “grain” structure. As for the cell structure, the nearly uniform TJD distribution could be an argument that the average stresses are small in large parts of the domains and that the disclinations of higher strengths may be responsible for elevated local stresses when situated sufficiently apart.

It is interesting to compare the binary classification of TJs as I-junctions and U-junctions with the tertiary classification as $J_0$, $J_1$, $J_2$, $J_3$ dependent on the number of HAGBs meeting at a junction. For example, Bollmann [77] and Fortier et al. [82] suggested that TJs of LAGBs, i.e., those classified as $J_0$, are not disclinations. Differently, Randle [83] provided evidence that all TJ types from the tertiary classification may be TJDs. Table 3 lists the fractions of junctions of each type classified as disclinations. It is clear, that the results support Randle’s report — TJDs appear at all TJ types, slightly less at $J_0$ and $J_1$ than at $J_2$ and $J_3$. The trend that can be observed is that disclinations at $J_2$ and $J_3$ have typically higher strengths. On the other side, in the “grain” structure, the fractions of junctions of types $J_0$ and $J_1$ that are disclinations are negligible and the maximum strengths amongst these are low. This supports Bollmann’s suggestion but is obviously derived from an approximation of the real microstructure. The junctions of types $J_2$ and $J_3$ are substantially more likely to be disclinations with very high strengths.

5. Conclusions

The microstructure of Cu–0.4%Mg alloy is significantly refined by the ECAP process into increasingly smaller crystallites. It exhibits higher stress concentrations and dislocation density, resulting in substantial strengthening of the alloy. Disclinations contribute to the formation of residual stress fields, which in turn affect the behaviour of other defects and ultimately the macroscopic behaviour of the nanostructured material.

Proposed in this paper was a new methodology for the quantification of triple junction disclinations suitable for the characterisation of microstructures imaged by EBSD. It combines a discrete description of the microstructure as a polytopal complex onto which the experimental
data is mapped, with a procedure proposed by Bollmann for calculating dislocation structures at grain boundaries and disclinations at triple junctions as unbalanced dislocation structures. The characteristic of this unbalance was used here to suggest a measure of disclination strength, which could be seen as an analogue of the Burgers vector of dislocations (more precisely its square). The methodology was applied to the analysis of 2D EBSD data from an ECAP-processed Cu-0.4% Mg alloy. This data accurately captures the structure of grain boundaries and triple lines. Notably, the methodology can be applied to 3D EBSD data without modifications.

The results suggest that the evolution of the grain boundary structure during the continuous dynamic recrystallisation at severe plastic deformation proceeds in such a way that the stress fields created by disclinations remain nearly constant. A comparison of the disclinations in the cell (where all measured boundaries were included) and the “grain” (where internal boundaries were omitted by an algorithm) structures revealed the key role of dislocation walls in reducing the strengths of disclinations (several times) and maintaining lower strengths during the SPD process. Considering the results obtained with the cell structure, it was shown that the ECAP process altered the microstructure so that the number of triple junction disclinations increased and their spatial distribution became more uniform while the distribution of their strengths remained almost constant. This suggests that by increasing the density of disclinations, the uniformity of their spatial distribution, and maintaining their strengths, the ECAP effectively reduced the average stresses in large parts of the material, except in a limited number of places where disclinations of higher strengths remained at larger distances. This observation suggests that the distances between sparsely distributed disclinations of high strengths can be used to define a characteristic length scale within the material, a topic of ongoing work. Finally, it was shown that all types of TJUs from the standard classification based on adjacent HAGBs can be disclinations. 

These findings in this work enhance our understanding of material microstructural behaviour and mechanical properties. The main disadvantage of the proposed methodology is the lack of distinction between positive and negative disclinations which limits it to the quantification of disclination strengths and their spatial distribution without reliable calculations of the local elastic fields and residual stresses. This is also a subject of ongoing research.

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.

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References


Table 3

Relation between the TJ types classified by the number of adjacent HAGBs, the fractions of TJUs, and the maximum disclination strength $P_{max}$ in TJs of the given type obtained by the cell and the “grain” structures after 1, 2, 4, and 8 ECAP passes.

<table>
<thead>
<tr>
<th>Structure</th>
<th>TJ</th>
<th>$f_{12B}$ Pmax 1</th>
<th>$f_{12B}$ Pmax 2</th>
<th>$f_{12B}$ Pmax 4</th>
<th>$f_{12B}$ Pmax 8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell structure</td>
<td>$J_2$</td>
<td>0.2604 4</td>
<td>0.2605 4</td>
<td>0.2605 4</td>
<td>0.2605 4</td>
</tr>
<tr>
<td></td>
<td>$J_1$</td>
<td>0.1295 81</td>
<td>0.1271 64</td>
<td>0.1051 144</td>
<td>0.1318 64</td>
</tr>
<tr>
<td>“Grain” structure</td>
<td>$J_2$</td>
<td>0.0354 49</td>
<td>0.0296 4</td>
<td>0.0310 121</td>
<td>0.0297 4</td>
</tr>
<tr>
<td></td>
<td>$J_1$</td>
<td>0.2307 81</td>
<td>0.2224 36</td>
<td>0.2141 196</td>
<td>0.2580 100</td>
</tr>
</tbody>
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