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Triple junctions network as the key pattern for characterisation of grain structure evolution in metals

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HIGHLIGHTS

• Microstructures represented by combinatorial complexes and maps to properties
• Configurational entropy introduced as complexity measure of property map
• Entropy evolution related to measurable macroscopic behaviour
• Results demonstrate qualitatively different modes of dynamic recrystallization.

ABSTRACT

Describing dynamic recrystallisation is challenging with existing material characterisation tools, which are typically based on grain boundary character distribution. This is one barrier to further developments in grain boundary engineering. We consider the network of triple junctions in copper alloys as the sub-structure that governs continuous dynamic recrystallisation and propose one descriptor of this sub-structure, referred to as the structural entropy. With the limited available characterisation data we demonstrate that the proposed descriptor correlates well with the evolution of the microstructure during severe plastic deformation. Importantly, our descriptor allows for elucidating micro-localisation features in copper alloys observed in some recent experiments.

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1. Introduction

Severe plastic deformation (SPD) of copper [1–3], aluminium [4–6], magnesium [7], titanium [8,9] and other alloys is presently an expanding research area [10–13]. The interest stems from increasing in size and broadening in scope industrial applications of the process [8,12,14]. SPD is a top-down approach for producing ultrafine-grained (UFG) and even nanocrystalline (NC) materials, where the initial coarse-grained structure is refined via a series of plastic deformation passes [15]. The first passes generate predominantly new dislocation structures, while later passes, typically after accumulated plastic strain of 2, lead to grain refinement down to sub-micron level. While there are a number of experimental works describing the SPD technology
and reporting on microstructural changes, such as grain size, texture, dislocation density, etc., the theoretical formulation of structure evolution during SPD is poorly developed [3,16,17]. The majority of theoretical works [9,18–20] use a continuum framework, which does not allow to link the description of plastic flow (a continuous parameter) to the complex evolution of the material microstructure (a finite discrete geometry).

The current mechanistic understanding is that the changes in the microstructure during SPD can be considered as a continuous dynamic recrystallisation (CDRX) process [4,21]. CDRX proceeds by: (1) division of the initial coarse grains into sub-grains (dislocation cells) [21]; and (2) formation of a new ultrafine-grained structure by the sub-grains. The ultrafine-grained structure is characterised by a large fraction of high angle grain boundaries (HAGBs) [3,6,22], producing a significant strain hardening effect. Although the dislocation activity can be considered as continuous at a grain level, the CDRX is essentially a process of discrete and finite changes in a polycrystalline assembly [16]. Its description, therefore, requires new non-continuum approaches. These are expected to produce advanced theoretical and computational methods, required for further developments in the area of SPD and related grain boundary engineering (GBE) problems [23–25]. As one example, we mention the industrial need for manufacturing of copper and aluminium electrical wires with superior physical and mechanical characteristics [26].

In this work, we consider discrete structures formed by HAGBs, as well as triple junctions (TJs), to formulate a combinatorial descriptor which correlates with experimental observations of microstructure evolution during SPD. This is the necessary first step to a mechanistic theory for GBE of SPD processed materials.

2. Fraction of HAGBs and TJs network

The evolution of HAGBs in copper alloys subjected to SPD has been investigated and reported in a number of recent works [2,3,17,22,27]. Based on the experimental evidence, the following relation between the HAGBs fraction, \( p \), and the accumulated plastic strain, \( \varepsilon \), has been proposed [17]:

\[
p = \frac{\sqrt{A} \exp (A \varepsilon)}{\sqrt{1 + B \exp (2A \varepsilon)}} + C \ln (1 + \varepsilon) - \sqrt{\frac{A}{1 + B}}
\]  

(1)

where \( A, B, C \) are (dimensionless) material parameters. The derivation of Eq. (1) in [17], uses a statistical mechanics approach. The fraction of HAGBs \( p \) is considered as an order parameter, for which the Landau formalism can be applied [28]. The parameters \( A, B, \) and \( C \) are coefficients in the expansion \( dp/d\varepsilon = Ap - Bp^2 + Cp^6 \), and are related to the expansion of free energy \( F(p) \) in powers of \( p \). Their values are determined by the best possible fit of Eq. (1) to experimental data.

Fig. 1 shows experimentally measured relations between \( p \) and \( \varepsilon \) for several copper alloys with different levels of purity processed by equal-channel angular pressing (ECAP) [15]. It can be seen that the fraction of HAGBs at a given plastic strain level increases with increasing impurities. The figure shows also two plots of Eq. (1): a dashed curve using \( A = 1, B = 2 \) and \( C = 0.2 \), obtained by fitting the equation to the experimental data for nearly pure copper; and a solid curve using \( A = 1.5, B = 0.65 \) and \( C = 0.15 \), obtained by fitting the equation to the experimental data for Cu-2%Co alloy, i.e. with the highest fraction of impurities. Similar curves can be produced for the other copper alloys processed by ECAP shown in the figure. Based on the data for a number of aluminium and magnesium alloys [6], processed by high-pressure torsion (HPT), similar functional relation between \( p \) and \( \varepsilon \) is expected for all copper alloys processed by HPT.

Our approach is based on a combinatorial representation of a polycrystalline assembly, which is used to construct appropriate descriptors for sub-structures that correlate with measurable characteristics. This means that the present development does not involve length scales. To put modelling into perspective, consider 2-dimensional electron backscatter diffraction (EBSD) maps [29], widely used for microstructure characterisation of metals after SPD. We construct structures topologically similar to EBSD maps using the well-known Voronoi tessellation [30]: an area is partitioned into non-overlapping polygons, which represent grains with random crystal orientation distribution [31,32]. This approach has numerous successful applications in many areas of science [33,34]. In the present work we do not use crystal

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**Fig. 1.** Fraction of high-angle grain boundaries as a function of accumulated plastic strain for copper alloys processed by ECAP. Symbols depict experimental data; curves calculated by Eq. (1).
orientations as a starting point, but rather a binary classification of the grain boundaries as HAGBs and low angle grain boundaries (LAGBs), which can also be considered as dislocation cell boundaries. This classification is represented by a map from the edges of the Voronoi diagram to the values 0 for LAGBs and 1 for HAGBs. The topological similarity between an EBSD map and a Voronoi diagram in terms of grain shapes distribution allows for considering a correspondence between the evolution of material’s microstructure and changes in the classification map on the Voronoi diagram. In particular, the process of gradually increasing the number of HAGBs can be modelled as a kinetic process on the edges, which leads to transitions from 0 to 1 in the classification map, i.e. conversions of LAGBs into HAGBs.

We will use the following terminology: a grain is a region fully enclosed by HAGBs, and a cell is a region enclosed by at least one LAGB. Thus, in the absence of HAGBs, all Voronoi polygons are cells. CDRX process converts LAGBs into HAGBs, leading to emergence of grains, until eventually all Voronoi polygons become grains. Fig. 2 shows an example for such evolution of grain boundaries structure, where the conversion of LAGBs (shown with dashed lines) to HAGBs (shown with solid lines) is random. Our simulations start with a fraction of HAGBs of approximately 0.05, which corresponds to the experimental value at accumulated plastic strain of about 1 (see Fig. 1). The HAGBs fraction is increased until the maximal possible fraction according to Eq. (1) is reached.

At this point, we note that the network of triple junctions (TJs) contains richer information about the microstructure than the fraction of HAGBs, but its use in the context of SPD has been limited. The binary classification of grain boundaries adopted here defines four types of TJs. These will be denoted by $J_i$ ($i = 0,1,2,3$), where $i$ stands for the number of adjacent HAGBs. Based on previous experimental observations [3], the network of TJs was considered in the works [16,17] with TJ types controlling the conversion of LAGBs into HAGBs. However, the conversion criterion adopted there was not physically underpinned, but required calibration with the experimentally measured evolutions of TJ fractions. This issue is addressed properly in the present work.

We use combinatorial techniques to analyse changes of material sub-structures, such as the sub-structure of HAGBs or the sub-structure of particular TJs type, with changes in the classification map. The Voronoi diagram is considered as a discrete (combinatorial) cell complex [35–37], where each Voronoi polygon is a face (2-cell), each grain boundary is an edge (1-cell), and each triple junction is a vertex (0-cell). A set of combinatorial operators, incidence matrices (boundary operators) and adjacency matrices [35], provide full description of the cell complex structure. The boundary operators allow for calculating topological invariants of any required sub-structure; a capability not used in the present work, but subject of ongoing investigations. Here, the adjacency matrices are only used to calculate the fractions of different TJ types for given distribution of HAGBs, which is sufficient for the purposes of the work.

3. Structural entropy and informativeness

We use the concept of configurational entropy [38] in alloys to underpin a proposal for conversion criterion based on TJ types. We note, that Frary and Schuh [39] used a similar concept to show that different values of the entropy correspond to distinct local textures or patterns in the TJ network. They proposed to use the microstructure configuration with maximum configurational entropy as a reference configuration, and to characterise any given configuration by comparing its entropy to the entropy of the reference state. Our approach is different, as we are interested in the evolution, rather than in snapshots of different configurations. We define a structural entropy characterising the state of the whole system, which is functionally closer to the Shannon’s information entropy, but analogous to that of configurational entropy, in the following way:

![Fig. 2. Illustration of possible microstructure configurations: (a) no grains, all polygons are cells; (b) grains and cells coexist; (c) no cells, all polygons are grains.](image-url)
corresponding changes in the fractions of TJ types. The LAGB, whose appearance of any special configuration with lower entropy indicates larger complexities. Since information entropy is typically measured in bits, the base of the logarithm in Eq. (2) is 2. Entropy values determined with this base can be converted to any other required base. This will not affect the results presented here, since relations between entropies of different configurations will be preserved.

We denote by $S_{\text{max}}(p)$ the entropy calculated with Eq. (2) with experimentally measured fractions of triple junction at a given fraction of HAGBs, $p$. In the Voronoi complex we simulate the structural evolution by converting one LAGB to HAGB per step.

One type of simulations uses conversions of randomly selected LAGB. In this case, the resulting entropy, denoted by $S_{\text{rand}}(p)$, is calculated after the conversion of LAGB to HAGB by taking into account the corresponding changes in the fractions of different TJ types (the relation between TJs and HAGBs is shown in Fig. 2). We note that $S_{\text{rand}}(p)$ can also be calculated analytically by using the functions $j_i(p)$ in [41] and discussed in [16] for the case of CDRX.

A second type of simulations uses a newly proposed constrained conversion, based on the maximum entropy production principle (MEPP), which governs the evolution of non-equilibrium dissipative systems [42]. Here, we assume that the value of entropy is proportional to the power of disclinations [43] in the triple junctions network, or equivalently to the stress intensity of their elastic fields. Physically, this means the conventional assumption that the most probable state of the system corresponds to a maximal diversity of its elements. The appearance of any special configuration with lower entropy indicates the action of some directed forces accompanying the process. In this type of simulations the conversion is performed after visiting all available LAGBs and calculating the potential entropy increase due to their potential conversion. This is again done by considering the corresponding changes in the fractions of TJ types. The LAGB, whose conversion to HAGB will result in maximal entropy increase is converted into HAGB. The resulting entropy evolution calculated after each conversion step is denoted by $S_{\text{max}}(p)$.

Our calculations of $S_{\text{max}}(p)$ and $S_{\text{rand}}(p)$, together with the calculations of $S_{\text{max}}(p)$ performed with the experimental values of TJs fractions reported in [3], are shown in Fig. 3. It can be seen that both the random and the MEPP-controlled conversions provide trends in good agreement with the experimental data. This suggests that the structural entropy based on TJs fractions is a viable macroscopic characteristic of the system. Both conversion approaches reach maximum entropy at HAGBs fraction of 0.5, where the TJs fractions are equal to 0.25. The MEPP-controlled conversion gives larger entropy at any $p$, with consistently higher entropy growth rate than the random conversion.

Fig. 3b shows the evolution of $S_{\text{max}}$ as a function of the accumulated plastic strain obtained with Eq. (1) for pure copper ($A = 1, B = 2$ and $C = 0.2$) and Cu-2%Co alloy ($A = 1.5, B = 0.65$ and $C = 0.15$). It can be seen clearly that for pure copper the structural entropy attains maximum at $\varepsilon \approx 6$, however it increases sharply at the beginning of the process reaching 85% of the maximum entropy already at accumulated strain $\varepsilon \approx 2$. Importantly in this case (ECAP processed pure copper), the structural entropy remains large across a wide accumulated strain range. This is typically observed in experiments. The entropy behaviour shows that a high diversity of TJ types is maintained. In contrast, in the Cu-2%Co alloy, the maximum entropy is attained already at $\varepsilon \approx 1.7$ and the entropy decreases gradually to the initial value at $\varepsilon \approx 12$. This behaviour indicates earlier appearance of individual ultrafine grains and more intensive recrystallisation in Cu-2%Co alloy. Notably, the maximum entropy appears within the range of critical strains between 1.5 and 4 [16], where the CDRX process in copper alloys is found to produce a new ultra-fine grain structure with a significant number of new HAGBs [3,16,44].

It has been shown in [16,17] that a process with random conversion of LAGBs to HAGBs does not yield an evolution of TJs fractions in agreement with the experimental results [3]. Using the MEPP-controlled conversion, we calculate the evolution of TJ fractions and compare these to the experimental data in Fig. 4. It can be seen that the predicted evolutions of $J_0$ and $J_2$ are very close to the experimental data, while the evolutions of $J_1$ and $J_3$ are slightly different, but follow the same trends. It should be noted, that the experimentally determined TJ fractions are based on rather limited number of measurements [3], so it cannot be fully established whether the actual evolution is controlled by MEPP defined in this work. It can be concluded, however, that the evolution of the material microstructure, measured by the evolution of TJs, follows...
the average HAGBs fraction EBSD maps for a series of accumulated plastic strains will give \( J_0^\text{exp}, J_1^\text{exp} \) and \( J_2^\text{exp}, J_3^\text{exp} \) with the corresponding curves \( J_0, J_1, J_2 \) and \( J_3 \) obtained with MEPP-controlled conversion of LAGBs to HAGBs.

Based on the results presented in Fig. 3a and Fig. 4, we propose a measure of informativeness \( i(p) \) of the deformation process

\[
i(p) = \frac{S_{\text{max}}(p) - S_{\text{rnd}}(p)}{S_{\text{max}}(p) - S_{\text{fl}}(p)}, \tag{3}
\]

where \( S_{\text{fl}}, S_{\text{max}} \) and \( S_{\text{rn}} \) are defined previously. This function measures the deviation of the conversion process from purely random one, reflecting the significance of various microstructural factors, such as stress concentrators, on the conversion in the real material. A deformation with purely random conversion will give \( S_{\text{max}}(p) = S_{\text{rn}}(p) \) and \( i(p) = 0 \), while a deformation with purely MEPP-controlled conversion will give \( S_{\text{fl}} = S_{\text{rn}} \) with \( i(p) \) approaching 1. Other values of \( i(p) \) indicate the presence of additional constraints on the evolution. Of particular interest is the value of \( i(p) \) at maximum entropy attained at \( p = 0.5 \). From the data shown in Fig. 3a this is estimated to be \( i(0.5) \approx 0.5 \). This shows a deviation from MEPP-controlled and random conversions which could be related to manifestation of micro-structural peculiarities, such as micro-localisation phenomena reported in \([27,45]\).

The introduction of \( i(p) \) allows for defining a procedure for material characterisation based on experimental data: (1) Analyse sufficient EBSD maps for a series of accumulated plastic strains \( \varepsilon \) to determine the average HAGBs fraction \( p(\varepsilon) \) and TJ fractions \( j(\varepsilon) \); (2) Calculate the material structural entropy \( S_{\text{max}} \), as well as \( S_{\text{fl}} \) and \( S_{\text{rn}} \), by Eq. 2 close the point \( p = 0.5 \); (3) Find \( i(p) \) by Eq. 3 and characterise the deformation process accordingly to estimate the level of constraint imposed by microstructural factors.

4. Conclusions

We have presented a study of microstructure evolution in alloys undergoing SPD at the polycrystalline scale. The fraction of HAGBs can be considered as one state parameter, but experimental observations show that this alone cannot be responsible for the peculiarities of the CDRX process: the fraction of HAGBs cannot differentiate between purely random/homogeneous distribution and a heterogeneous distribution containing different clusters \([16,17]\). The structure of the TJs network appears to contain important additional information. Structural entropy based on TJs, similar to the configurational entropy in alloys and to the Shannon information entropy, has been introduced as an additional state parameter. Together with the fraction of HAGBs, it characterises the state of the entire polycrystalline assembly, but unlike the HAGBs fraction it is able to reflect the inhomogeneity effects, such as micro-localisation during SPD in copper alloys, which has been shown by the correct representation of the evolution of TJ types \([3]\). The work used a limited set of data for validation, as measurements of TJ types is not common practice after EBSD. The proposed model requires further experimental investigations of TJs network evolution in various alloys for stronger validation, including from 3D EBSD maps, where the full connectivity matrix should provide a richer information about the topological structure of the grain system. Work on 3D microstructures is ongoing and will be reported in the near future.

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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