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Role of geometrically necessary dislocations on mechanical properties of friction stir welded single-phase copper with medium stacking fault energy

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Abstract

The contribution of geometrically necessary dislocations (GNDs) and grain boundaries (GBs) on the mechanical properties of different zones in a friction stir welded pure copper joint is unveiled. This representative and simple medium stacking fault metal is selected to decouple the contributions of complex microstructure (precipitates, second phases and phase transformation) and focus only on GNDs and GBs. The GNDs density is much higher in the weld compared to the base material but similar in the thermomechanically affected zone (TMAZ) and the stir zone (SZ). Their difference is rather related to the dislocation distribution: in the grain interior in the SZ and near the GBs and grain interior in the TMAZ. In comparison to the TMAZ, the higher strength and elongation of the SZ results from the formation of finer grains and sharp-clean grain boundaries issued from continuous dynamic recrystallization.

Keywords: Friction stir welding; Electron backscattering diffraction (EBSD); Copper; Dislocation; Mechanical properties.

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1. Introduction

Plastic deformation in metals is related to its dislocation structure. Dislocations can be classified as geometrically necessary dislocations (GNDs) and statistically stored dislocations (SSDs) [1]. During plastic deformation, GNDs are formed and act as barriers to mobile dislocations, i.e. strain hardening of metals [2]. To understand the behavior of dislocations, different methods such as transmission electron microscopy (TEM), X-ray diffraction (XRD), and electron backscattering diffraction (EBSD) have been used [3, 4]. In TEM images, it is difficult and time consuming to separate the GNDs and SSDs. Hence, the measured dislocation density will then refer to the total dislocation density. EBSD allows to estimate only the GNDs, while XRD method allows to estimate the total dislocation density [4]. GNDs with a net non-zero Burgers vector, unlike SSDs with a net-zero Burgers vector, result in a local lattice curvature or misorientation in deformed materials [3]. Therefore, high resolution EBSD, in which the data acquisition is based on crystallographic orientation or lattice curvature, can be used for fast and accurate estimation of GNDs density [5, 6].

Friction stir welding (FSW) has attracted the attentions of industry because its solid state nature can avoid defects inherent to fusion welding processes [7]. Friction stir processing (FSP) based on FSW principles has also been developed for surface modification and composite production [8]. FSW/P is a thermomechanical process, in which the sharp gradients of strain, strain rate, and temperature result in a gradient microstructure from the base material (BM) to the stir zone (SZ). FSW/P may improve the mechanical properties of materials by activating dynamic restoration mechanisms, precipitation, phase transformation, etc. [7]. For example, in the case

of pure copper, low heat input FSW/P results in higher yield strength (σ_y) [9] and microhardness values [10] compared to BM. This strengthening has often been attributed to grain boundaries (GBs) and dislocations but without proofs of their contribution in different microstructural zones of BM, SZ, and thermomechanically affected zone (TMAZ) [9, 10]. This distribution should be unveiled to understand and consequently control the final mechanical properties. In this study, the contribution of GNDs and GBs in strengthening the different microstructural zones of a friction stir welded/processed (FSWed/Ped) pure copper plate is elucidated in details using EBSD and TEM techniques. Copper is selected as an appropriate “model” material as it eliminates the effects of other microstructural features such as precipitates, secondary phases, and phase transformation that could blur the importance of the GND and GB contributions.

2. Materials and methods

Pure copper plate with a medium stacking fault energy (SFE) of $\sim 80 \text{ mJ m}^{-2}$, and a thickness of 2 mm was FSWed at a rotational speed of 410 rpm, a traverse speed of 100 mm/min, and a plunge depth of 0.2 mm. AH13 steel tool with a shoulder diameter of 12 mm, a pin diameter of 3 mm and a pin length of 1.7 mm was used. The macrostructure of the joint was characterized using optical microscopy (OM-Tokyo, Japan). For orientation image microscopy (OIM), a field emission gun scanning electron microscope (Philips XL30 E-SEM, USA) equipped with EBSD detector was employed. The OM and OIM samples were electro polished and etched for 35 s at 10 °C and RT. For this aim, a solution of 250 mL H_3PO_4 , 250 mL ethanol, 50 mL propanol, 500 mL distilled water, and 3 g urea was utilized. A step size of 90 nm was used during EBSD

scans. A scanning transmission electron microscope (STEM, Hitachi S-4800, Japan) characterized the dislocations. TEM lamellas from different zones were extracted using a dual beam FIB/SEM microscope (Lyra, Tescan, Czech Republic). Tensile samples with a gage length of 12 mm were cut from the different zones (Fig. 2a), and were tested using a strain rate of 1 mm/min at room temperature (RT).

To estimate the GND's density, the OIM data were analyzed using an appropriate optimization algorithm based on all the available components of the curvature tensor [11]. The crystal orientation measured by EBSD is represented by Euler angle notation (ϕ_1, ϕ, ϕ_2) of crystals within a diffracting volume [12]. A crystallographic orientation map is generated via continuous point by point scan over the surface of the sample by EBSD. Each pixel of the EBSD map contains information on the local orientation of the crystalline material. GNDs will induce local variations in the crystal orientation between neighboring measurements in the orientation map of EBSD as represented schematically in Fig. 1. P_1, P_2, P_3 and P_4 , are the typical neighboring points during EBSD scan. In Fig. 1, variations in orientation between neighboring points are due to the lattice curvature (ϕ), which, in turn, originate from GNDs.

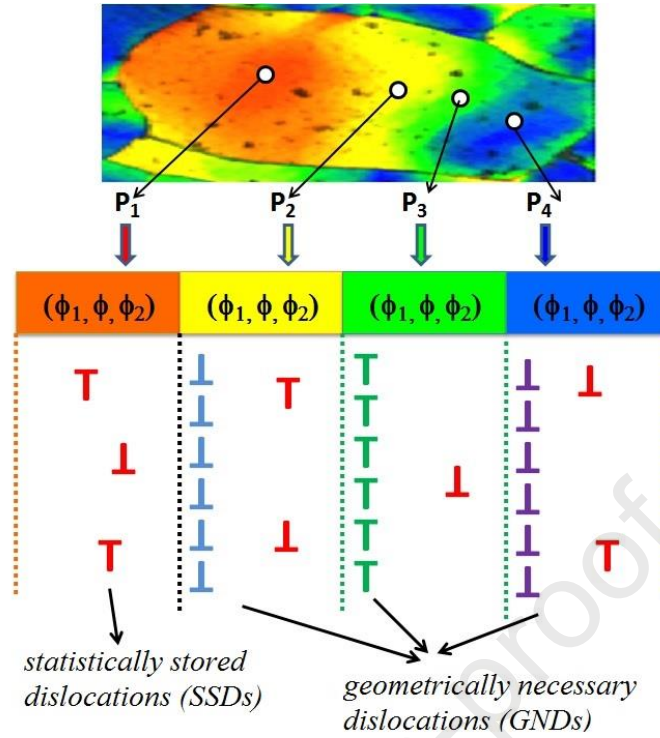


Fig.1. Schematic of geometrically necessary dislocations in EBSD orientation maps as a variation in crystal orientation (ϕ_1, ϕ, ϕ_2) due to the lattice curvature. The colors of grains refer to different crystallographic orientations.

The density of GNDs can be quantified by calculating the number of GNDs needed to generate the detected lattice curvature. To locally extract the GNDs density (ρ_{GNDs}), the lattice curvature tensor obtained from Eq. (1) can be used [12]:

$$\begin{aligned}
 K_{11} &\cong \frac{\Delta\theta_1}{\Delta X_1}; & K_{12} &\cong \frac{\Delta\theta_1}{\Delta X_2} \\
 K_{21} &\cong \frac{\Delta\theta_2}{\Delta X_2}; & K_{22} &\cong \frac{\Delta\theta_2}{\Delta X_2} \\
 K_{31} &\cong \frac{\Delta\theta_3}{\Delta X_1}; & K_{32} &\cong \frac{\Delta\theta_3}{\Delta X_2}
 \end{aligned} \tag{1}$$

where K_{ij} is the component of the lattice curvature tensor between measured pixels, $\Delta\theta_i$ is the direction cosines determined by the unit vector $[uvw]$ defining the common axis of rotations about fixed axes, e.g. around the z and x axis which are commonly called Euler angles, and ΔX_i is the pixel separation distance. Assuming 18 independent dislocation types in face-

centered cubic (fcc) crystal structures of the $\{111\}\langle 110 \rangle$ types, and considering all GNDs are either pure edge dislocations or pure screw dislocations, 12 pure edge dislocations and 6 pure screw dislocations can be present in the fcc crystal structure. Furthermore, by considering the dislocations with opposite line vector and the same Burgers vector, the total number of possible dislocation modes that should be considered in the analysis amounts to 36. A lower bound of ρ_{GNDs} to create observed lattice curvature can thus be written as follows:

$$\begin{bmatrix} K_{11} \\ K_{21} \\ K_{31} \\ K_{12} \\ K_{22} \\ K_{32} \end{bmatrix} = \begin{bmatrix} \frac{1}{2}b_1^1l_1^1 & \frac{1}{2}b_1^2l_1^2 \dots & \frac{1}{2}b_1^6l_1^6 \\ \frac{1}{2}b_1^1l_2^1 & \frac{1}{2}b_1^2l_2^2 \dots & \frac{1}{2}b_1^6l_2^6 \\ \frac{1}{2}b_1^1l_3^1 & \frac{1}{2}b_1^2l_3^2 \dots & \frac{1}{2}b_1^6l_3^6 \\ \frac{1}{2}b_2^1l_1^1 & \frac{1}{2}b_2^2l_1^2 \dots & \frac{1}{2}b_2^6l_1^6 \\ \frac{1}{2}b_2^1l_2^1 & \frac{1}{2}b_2^2l_2^2 \dots & \frac{1}{2}b_2^6l_2^6 \\ \frac{1}{2}b_2^1l_3^1 & \frac{1}{2}b_2^2l_3^2 \dots & \frac{1}{2}b_2^6l_3^6 \end{bmatrix} \begin{bmatrix} \rho_G^1 \\ \rho_G^2 \\ \rho_G^3 \\ \cdot \\ \cdot \\ \cdot \\ \cdot \\ \cdot \\ \rho_G^6 \end{bmatrix} \quad (2)$$

where, l is the line vector and b is the Burgers vector for a given dislocation configuration. The simplex method was implemented in the optimization algorithm. The OIM grain identification map was generated using a threshold misorientation of 1.5 degrees and inserted in the algorithm. A sample calculation for the GNDs density is provided as supplementary material.

3. Results

Fig. 2a shows the macrostructure of the joint highlighting the BM, TMAZ and SZ. Figs. 2b provides the tensile curves of these weld zones showing that FSW/P caused a significant increase in σ_y in TMAZ and SZ compared to BM. In addition, the SZ displays a significant

increase in strength (σ_y), ultimate tensile strength (UTS) and elongation (EL) compared to TMAZ.

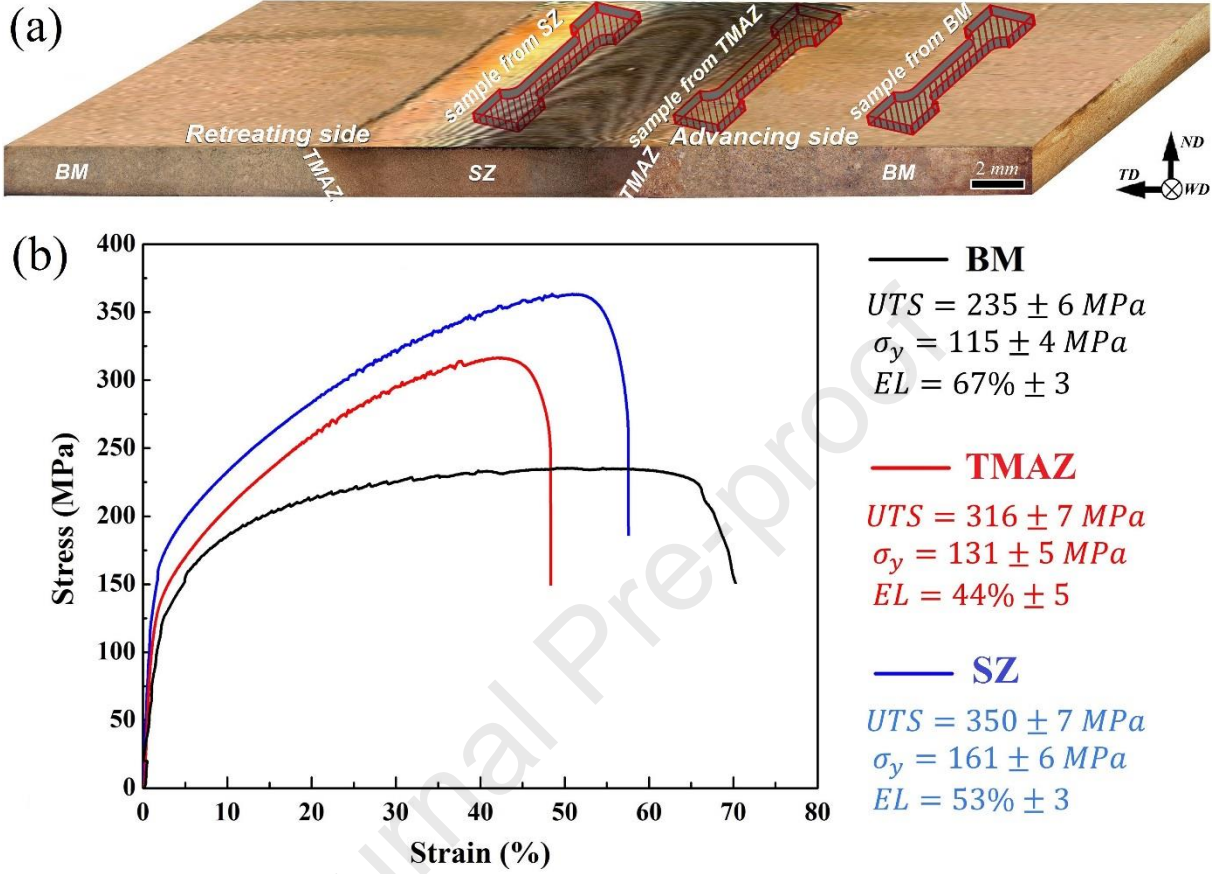


Fig. 2. (a) 3D macrostructure of the joint and tensile sample positioning. (b) Stress-strain tensile curves for different microstructural zones of the joint. The yield strength (σ_y), ultimate tensile strength (UTS) and total elongation (EL) are provided for each curve with their standard deviation.

Fig. 3 shows the Inverse pole figure (IPF) and GNDs maps of BM, TMAZ, and SZ. The average grain size of BM was equal to $15.3 \mu\text{m}$ (Fig. 3a₁) which was brought down to about $4.9 \mu\text{m}$ in SZ (Fig. 3c₁). TMAZ (Fig. 3b₁) is a transition zone with a bimodal grain structure containing deformed and recrystallized grains, which has an average grain size of $7.6 \mu\text{m}$. Note that the annealing twin boundaries were treated as high angle grain boundaries to refine the grain size and strengthen the alloy.

4. Discussion

During FSW/P of pure copper, continuous dynamic recrystallization (CDRX) rules the grain structure formation, which inherits from the deformation texture typical of the TMAZ [8]. In BM (Fig. 3a₂), no special dislocation substructure is observed and the overall ρ_{GNDs} is low confirming its initial full annealed state. In TMAZ (Fig. 3b₂) and SZ (Fig. 3c₂), GNDs are clearly heterogeneously distributed, and some of them are concentrated along the low angle grain boundaries (LAGBs), as shown in Fig. 3b₃ and b₄. During FSW/P, GNDs transform into the energetically favorable configurations, resulting in the formation of new LAGBs and dividing the grains into small subgrains. By assuming the lognormal distribution of the GNDs density described by Eq. (3) [13], the microstructure-averaged GNDs density (m_{GNDs}) can be calculated using Eq. (4) [14]:

$$f(\rho_{GNDs}|\mu, \sigma) = \frac{1}{\sigma \rho_{GND} \sqrt{2\pi}} \exp\left(\frac{-(\ln \rho_{GND} - \mu)^2}{2\sigma^2}\right) \quad (3)$$

$$m_{GND} = \exp(\mu) \quad (4)$$

where μ and σ are the location and scale parameter of lognormal distribution, respectively. Therefore, the ρ_{GNDs} in the BM, TMAZ, and SZ is estimated as $4.35 \times 10^{13} \text{ m}^{-2}$, $1.93 \times 10^{14} \text{ m}^{-2}$ and $1.90 \times 10^{14} \text{ m}^{-2}$, respectively. Unexpectedly, ρ_{GNDs} in TMAZ and SZ is almost equal. This behavior confirms the continuous nature of DRX. After the formation of CDRX grains they undergo further plastic deformation during grain structure formation, and hence the ρ_{GNDs} increases. The slightly lower ρ_{GNDs} in SZ compared to TMAZ may be due to the existence of clean-non-deformed CDRX grains and occurrence of static recovery (SRV) after the FSW/P tool passes [9].

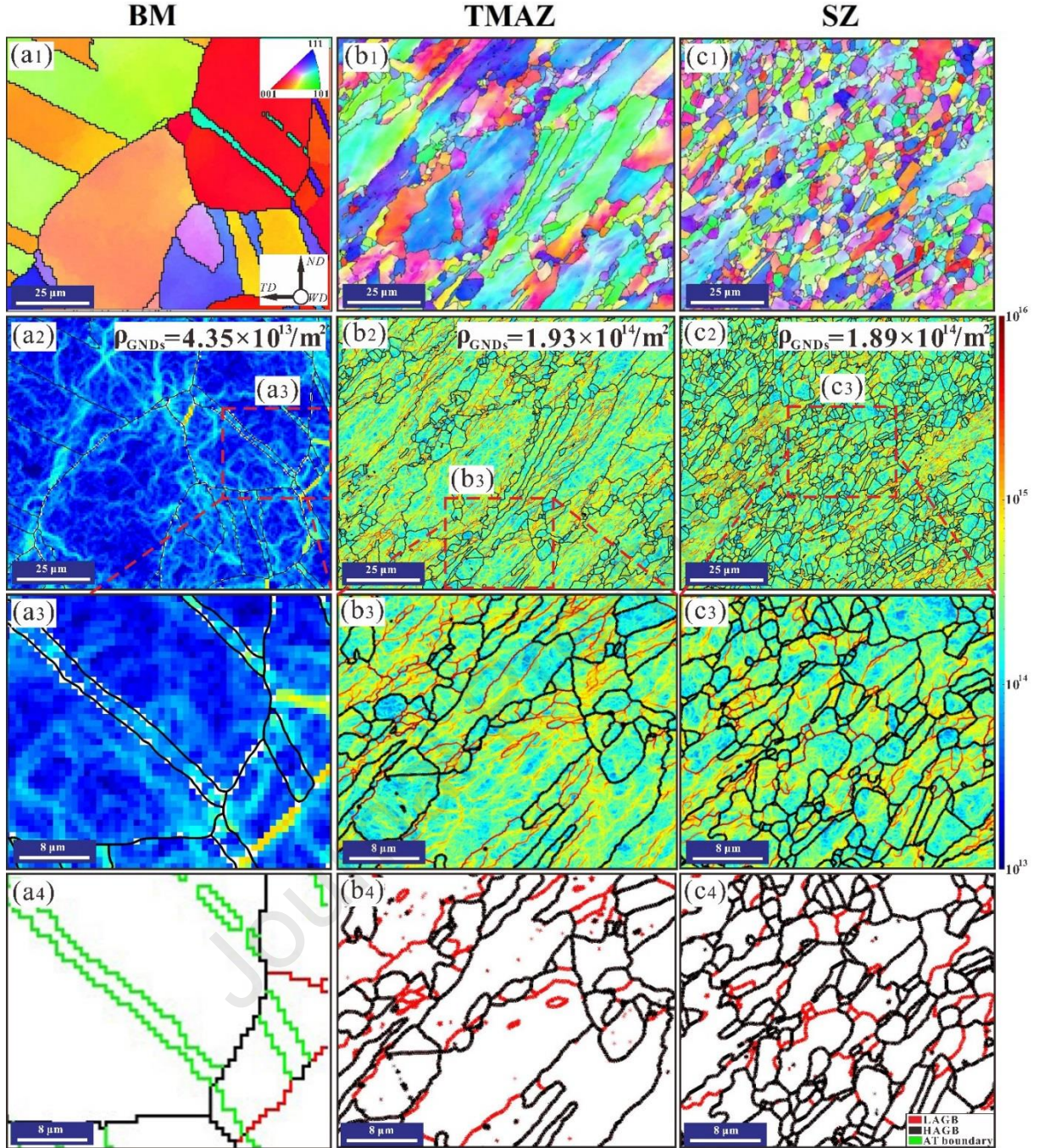


Fig. 3. IPF, GNDs density and grain boundary maps of different zones, respectively for: (a₁, a₂, a₃, a₄) BM, (b₁, b₂, b₃, b₄) TMAZ, and (c₁, c₂, c₃, c₄) SZ. Color scale indicates GND density in lines/m². Black lines denote high angle grain boundaries (HAGBs); red and green lines in Figs. (a₄), (b₄) and (c₄) indicate low angle grain boundaries (LAGBs) and annealing twin (AT) boundaries. The average calculated GND densities are superimposed on the GND maps (a₂, b₂, and c₂). 15° misorientation criterion was used to distinguish LAGBs and HAGBs, and the minimum grain size is set to 3 μm.

Fig. 4 shows the variations of GNDs near grain boundaries (GBs) of TMAZ and SZ highlighted by TEM. In TMAZ, GNDs appear as rather wide bands (white arrows) and dislocation cells (red arrows) forming geometrically necessary boundaries (GNBs) (Fig. 4a). While in SZ, the GNBs appear as sharp lines (blue arrows) with low ρ_{GNDs} (Fig. 4b). In addition, dislocation cells are formed inside the SZ's grains (red arrows), a sign of static recovery [9]. CDRX occurs by subsequent transformation of GNBs to LAGBs and finally to HAGBs. In fact, the gradual increase in misorientation during CDRX happens by absorption of dislocations to LAGBs, and hence it reduces the ρ_{GNDs} at GBs. Thus, the ρ_{GNDs} of SZ corresponds to the grain interior, while in the TMAZ it corresponds to both the GBs and grain interior.

σ_y is the sum of friction stress (σ_0), grain size strengthening (σ_{GBs}), solid solution strengthening (σ_{SS}), precipitation strengthening (σ_P), and dislocation strengthening (σ_{Dis}):

$$\sigma_y = \sigma_0 + \sigma_{GBs} + \sigma_{SS} + \sigma_P + \sigma_{Dis} \quad (5)$$

The friction stress is given by $\sigma_0 = \frac{2G}{1-\nu} e^{-\frac{2\pi d_I}{b(1-\nu)}}$ [13, 15], where $G = 45.4$ GPa is the shear modulus of copper [16], $\nu=0.34$ is the Poisson's ratio [17], d_I is the interplanar spacing of the primary slip ($d_{\{111\}}=0.2084$ nm for face-centered cubic (FCC) copper), $b = 0.256$ nm is the magnitude of Burgers vector [16], respectively. Considering the activation of $\{111\}<110>$ slip in FCC crystal during plastic deformation, the friction stress is calculated as 10.09 MPa for pure copper. In addition, σ_{SS} and $\sigma_P \approx 0$ (due to use of pure copper in this study), and thus σ_y can be calculated as follows:

$$\sigma_y = 10.09 + \sigma_{GBs} + \sigma_{Dis} \quad (6)$$

The strength contribution from grain boundaries and dislocations can be described using the classic Hall-Petch [18] and Taylor relationship [13]:

$$\sigma_{GBs} = \frac{k_y}{\sqrt{d_g}} \quad (7)$$

$$\sigma_{Dis} = M\alpha Gb\sqrt{\rho_{total}} \xrightarrow{\rho_{GNDs} \gg \rho_{SSDs}} \sigma_{Dis} = M\alpha Gb\sqrt{\rho_{GNDs}} \quad (8)$$

where $k_y = 0.12 \text{ MPa}\cdot\text{m}^{1/2}$ is the strengthening coefficient [19], d_g is the average grain size of samples, M is the Taylor factor derived from the EBSD measurements to be 2.87, 2.93, and 3.06 for BM, TMAZ, and SZ, respectively. α is an empirical constant related to the dislocation structures [13, 16]. For the SZ and TMAZ samples, dislocation structures were formed in the microstructures, thus α is 0.19 [16]; whereas, $\alpha = 0.3$ for the full annealed BM sample without dislocation cells in the microstructure [13]. G and b has been defined in Eq. (5). Since in deformed metals and alloys, GNDs have a considerably key role on strength [20], the total dislocation density is assumed equal to ρ_{GNDs} in Eq. (8). Using Eq. (8), measured ρ_{GNDs} from EBSD data (Fig. 3) and σ_y (Fig. 2), σ_{Dis} and σ_{GBs} may be calculated and are summarized in Table 1. For comparison, the estimated yield strength (σ_e) resulting from different strengthening mechanisms were also calculated and listed in Table 1, which gives a comparable value to the experimental one, illustrating the microstructure analysis carried out in the present work is sufficiently reliable.

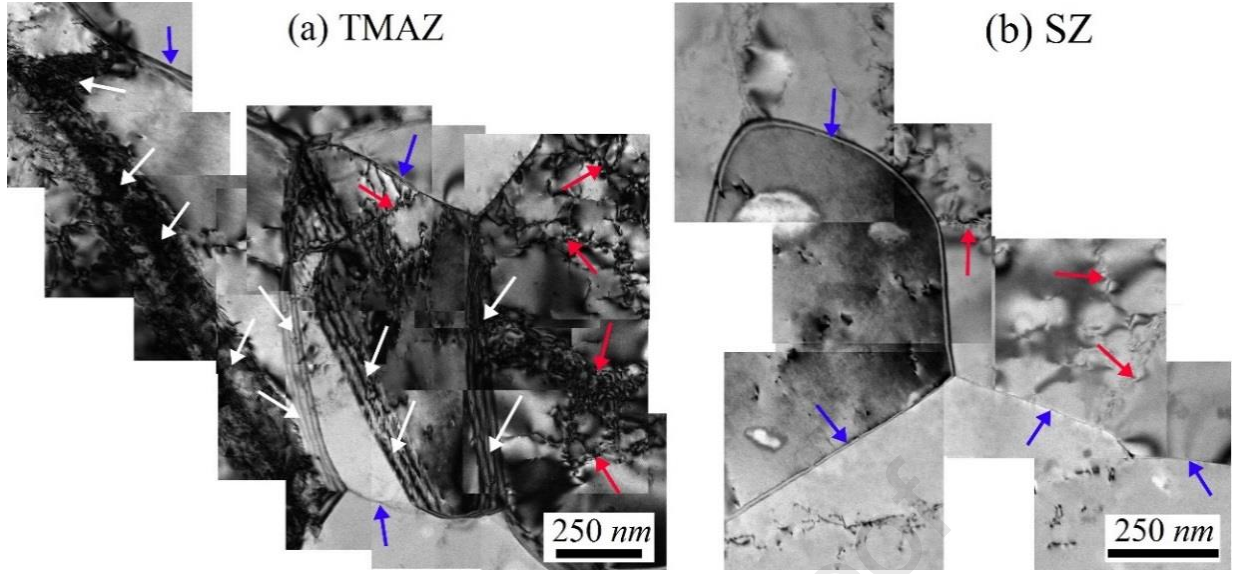


Fig. 4. TEM images of dislocation structures near the grain boundaries in: (a) TMAZ, and (b) SZ. White, red, and blue arrows refer to wide GNBs, dislocation cells, and sharp GBs, respectively.

The mechanical properties disclosed in Figs. 2b are explained as follows:

- (1) The higher σ_y and UTS of TMAZ and SZ compared to BM is due to their higher ρ_{GNDs} and finer grain size (in the case of SZ) (Fig. 3).
- (2) ρ_{GNDs} is similar in the TMAZ and SZ, leading to a similar contribution to strengthening of 87.77-93.12 MPa (Table 1).
- (3) GBs are more effective in strengthening the SZ (54.11 MPa) compared to the TMAZ (43.59 MPa) due to its finer grains formed by CDRX. The dominating strengthening mechanism in the TMAZ is the GNDs while it is the GBs in the SZ (Figs. 3b₂, 3c₂, and 4).
- (4) The higher elongation of SZ compared to TMAZ (Fig. 2b) is due to sharp and clean GBs of SZ (Fig. 4b) compared to those of TMAZ (Fig. 4a) in conjunction with

small grain sizes (larger surface of GBs), which helps to store more dislocations during tensile loading.

Table 1.

Strength contributions of friction stress (σ_0), grain size (σ_{GBs}) and geometrically necessary dislocations (σ_{GNDs}) of the different zones of the joint. The experimental and estimated yield strength are also provided.

<i>Zone</i>	σ_y (MPa) (Experimental)	σ_e (MPa) (Estimated)	σ_0 (MPa)	σ_{GBs} (MPa)	σ_{GNDs} (MPa)
<i>BM</i>	115	106.75	10.09	30.70	65.96
<i>TMAZ</i>	131	141.45	10.09	43.59	87.77
<i>SZ</i>	161	157.32	10.09	54.11	93.12

5. Conclusion

(1) The microstructural zones in the gradient macrostructure of an FSWed pure copper joint reveal different mechanical properties. These differences in geometrically necessary dislocations (GNDs) density and grain boundaries (GBs) size and sharpness have been thoroughly quantified by orientation image microscopy in EBSD and observed by TEM analysis.

(2) GNDs are increasing the strength of both the thermomechanically affected zone (TMAZ) and stir zone (SZ), leading to a higher σ_y and UTS compared to base material (BM), with a significant contribution of 88-93 MPa. This contribution is similar in the SZ and TMAZ but the GNDs distribution is different in both zones.

(3) The GND density in the SZ corresponds to the grain interior, while in the TMAZ it corresponds to both the GBs and grain interior.

(4) GBs are the dominant contribution to strengthening in SZ, which causes a synergic increase in strength and elongation in SZ in comparison to TMAZ. This superior macromechanical properties is due to occurrence of continuous dynamics recrystallization (CDRX), which produces finer grains and clean GBs.

(5) The outcome of this study helps to understand the strengthening mechanisms in single phase metals with high to medium stacking fault energy (SFE) undergoing CDRX during FSW/P. This can be considered as a basis for future investigations on other metals with lower SFEs, precipitates, secondary phases, and phase transformations.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time but may be requested to authors.

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Declaration of interests

☒ 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.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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