Effects of stacking fault energy, strain rate and temperature on microstructure and strength of nanostructured Cu–Al alloys subjected to plastic deformation

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Abstract

Nanostructured Cu–Al alloys with different stacking fault energies (SFEs) corresponding to Al concentrations in a range of 0–4.5 wt.% are prepared by means of plastic deformation. Effects of SFE, strain rate and temperature on microstructure characteristics and strength have been systematically investigated in the Cu–Al alloys. It was found that the deformation occurs mainly by twinning at the nanoscale in all samples subjected to dynamic plastic deformation at liquid nitrogen temperature. In the quasi-static compression process at room temperature, dislocation slip dominates the plastic deformation when the SFE is higher than 50 mJ m$^{-2}$. With decreasing SFE, twinning becomes the dominant deformation mechanism. A map of deformation modes and corresponding strain-induced microstructures is drawn in the SFE-processing parameters space for the Cu–Al alloys. In both sets of deformation mode, twinning is obviously enhanced by decreasing the SFE, resulting in smaller twin/matrix (T/M) lamella thickness and grain sizes. Consequently, an obvious strength elevation is induced by the size effects of grains and T/M lamellae with lower SFEs.

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1. Introduction

Plastic deformation is one of the optimum approaches to fabricate nanocrystalline and ultrafine-grained metallic materials, which exhibit increased strength compared to their coarse-grained (CG) counterparts [1,2]. The microstructure and mechanical properties of materials processed by plastic deformation, such as cold rolling (CR) [3–5], wire drawing (WD) [6,7], equal-channel angular pressing (ECAP) [8–10] and high-pressure torsion (HPT) [11–13], have been intensively investigated over the past decades. Most investigations focused on materials with high or medium stacking fault energy (SFE), such as Al, Ni and Cu, where dislocation slip is usually the predominant deformation mechanism [3–13]. Consequently, grain refinement is induced by dislocation activities, tending to a saturation grain size in the ultrafine range with increasing plastic strain when a balance is reached between dislocation generation and annihilation. For example, a saturated grain size of 200–300 nm is often reached at large strains in pure Cu, corresponding to a saturated yield strength of ¬400 MPa [4–6,9,10,13]. These ultrafine grains could not be further refined by increasing plastic strain.

Nevertheless, it was found that nanosized grains (<100 nm) can be obtained in high-SFE materials (such as Cu) when strain rates are high enough or at low deformation temperatures, e.g. in ball milling [14], surface mechanical attrition treatment [15–17], cryogenic rolling [18], and dynamic plastic deformation (DPD) at cryogenic temperatures [19–21]. Strain rate and temperature are apparently two key processing parameters in determining the deformed microstructure of high-SFE materials. Recently, we systematically investigated the combined effects of strain rate ($\dot{\varepsilon}$) and temperature ($T$) on the microstructure and mechanical properties of Cu by using the
Zener-Hollomon parameter $\ln Z = \ln \dot{e} + \frac{Q}{RT}$, where $R$ is the gas constant and $Q$ is the activation energy for diffusion [22]. With increasing $Z$, i.e. increasing strain rate and/or decreasing temperature, the ultrafine-grained microstructure gives way to the mixed nanostructure of nanoscale twin bundles and nanosized grains. The formation of nanosized grains (below 100 nm) in Cu deformed at $\ln Z > 50$ was attributed to the refinement of coarse grains via nanoscale twinning and the subsequent fragmentation of the nanoscale twin/matrix (T/M) lamellae [21,23]. Nanoscale deformation twinning at high strain rates or low temperatures provides a novel way of grain refinement into the nanometer range [23,24].

SFE is a crucial intrinsic parameter to determine plastic deformation mechanism in metals [25]. Deformation twinning is frequently observed in materials with low SFEs, such as Cu–Al alloys [26], Cu–Zn alloys [27–29] and austenite stainless steels [30], even those deformed at low strain rates and room temperature. Nanosized grains as small as ~10 nm have been achieved in Cu–Zn alloys [31,32] and Cu–Al alloys [26] subjected to plastic deformation, corresponding to significant enhancements in hardness and strength. Microstructure analysis in ECAP Cu–Al alloys [26] demonstrated that the nanoscale deformation twins in low-SFE materials play an important role in the grain refinement process, as manifested in Cu deformed at high $Z$. Clearly, decreasing the SFE may provide an alternative way to refine microstructures.

In this work, we systematically study the combined effects of SFE, strain rate and temperature on the microstructure and mechanical properties of deformed metals. Microstructure characteristics and the strength of Cu–Al alloys with a wide range of SFEs (78–12 mJ m$^{-2}$, corresponding to different Al concentrations) are studied with different processing parameters, including high strain rate (~$10^3$ s$^{-1}$) and cryogenic temperature (77 K), as well as low strain rate (~$10^{-1}$ s$^{-1}$) and room temperature (~293 K), respectively.

2. Experimental

2.1. Sample

The materials studied in the present work are pure Cu (purity 99.995 wt.%) and Cu–Al alloys with Al concentrations of 0.5, 0.75, 1.0, 1.5, 2.2 and 4.5 wt.%. The variation in SFE as a function of Al concentration is plotted in Fig. 1 according to the reported data in the literature on several compositions (Cu, 1.0% Al, 2.2% Al and 4.5% Al, respectively) [33,34]. Alloy samples were prepared from high-purity components (99.995 wt.% Cu and 99.999 wt.% Al) by means of vacuum induction melting. Before plastic deformation, cylindrical samples of Cu and Cu–Al alloys (9 mm in diameter and 12 mm in thickness) were annealed in a vacuum at different temperatures (973–1123 K) to obtain a homogeneous microstructure with similar grain sizes (~200 μm).

![Fig. 1. Variation in SFE values of Cu–Al alloys as a function of Al concentration [33,34].](image-url)

2.2. Plastic deformation

Plastic deformation of the samples at a high strain rate (~$10^3$ s$^{-1}$) was performed at liquid nitrogen temperature (LNT) on a DPD facility (hereafter referred to as LNT-DPD), the setup and processing parameters of which have been described in detail elsewhere [20,21]. The strain is defined as $\dot{e} = \ln(L_0/L_f)$, where $L_0$ and $L_f$ are the initial and the final thickness of the deformed sample, respectively. All samples were deformed to the same strain of $\dot{e} = 2.0$ except for sample Cu–4.5% Al, which was deformed to a strain of $\dot{e} = 1.7$ to prevent cracking at larger strains. Plastic deformation at a low strain rate (~$10^{-1}$ s$^{-1}$) was achieved by quasi-static compression (QSC) on an MTS servo-hydraulic testing machine at room temperature (RT, hereafter referred to as RT-QSC). The samples were deformed to the same strains as the DPD ones.

2.3. Microstructure characterization

Microstructures of the deformed samples were characterized by using transmission electron microscopy (TEM) on a JEOL 2010 high-resolution transmission electron microscope operating at 200 kV. Cross-sectional thin foils for TEM observations were prepared by means of mechanical grinding followed by double-jet electrolytic polishing in an electrolyte consisting of 25% alcohol, 25% phosphorus acid and 50% deionized water at ~−5 °C. The volume fraction of nanoscale T/M lamellae was statistically measured from a large number of TEM images for each sample.

2.4. Tensile tests

Uniaxial tensile tests were performed on an Instron 5848 Tester system operating at a strain rate of $6 \times 10^{-3}$ s$^{-1}$ at RT. A contactless MTS LX300 laser extensometer was used to measure the sample strain upon loading. The gauge section of dog-bone-shaped tensile specimens of 1 mm
thickness is 5 mm in length and 1 mm in width. More than four tensile tests were performed on each sample.

3. Results

3.1. Microstructure characterization

3.1.1. Microstructure of LNT-DPD Cu–Al alloys

Microstructures of LNT-DPD Cu–Al alloys with \( \leq 4.5\% \) Al are characterized by nanosized grains embedded with nanoscale twin bundles that are roughly perpendicular to the deformation direction, as shown in Fig. 2a. The sizes and volume fractions of the nanograins and the T/M lamella thickness vary with Al concentration. High-density dislocations exist at the twin boundaries (TBs; Fig. 2b), typical of deformation twins. Statistical measurements in the LNT-DPD Cu–1.0\% Al show that the volume fraction of the nanotwin bundles is about 26\%, with an average T/M lamella thickness of 25 nm (Fig. 2c), which is smaller than that of LNT-DPD Cu [22]. Nanosized grains in the LNT-DPD Cu–Al alloys (Fig. 2d), which are quite similar in morphology to those in the LNT-DPD Cu sample [22], are believed to be derived from three different processes, as investigated systematically before [22]. Most nanosized grains derived from nanoscale T/M lamellae are elongated, with an aspect ratio of 2–3 and an average transverse characteristic size of about 34 nm. In addition, some strongly elongated

Fig. 2. (a) A typical bright-field TEM image of the LNT-DPD Cu–1.0\% Al alloy. (b) A typical TEM image of nanoscale T/M lamella bundles. (c) A statistical distribution of T/M lamella thickness. (d) A typical TEM image of nanosized grains. (e) A statistical distribution of transverse grain size.
subgrains/grains with roughly parallel lamellar boundaries (with an average transverse size of about 106 nm and an aspect ratio of about 5–10) were observed in the LNT-DPD Cu–1.0% Al alloy, analogous to the microstructure resulting from dislocation structures in Cu processed by CR, WD and ECAP [5–7,9]. Their volume fraction is about 5–10%. This implies that 85–90 vol.% of the processed sample was twinned during the LNT-DPD processing.

In the LNT-DPD Cu–4.5% Al sample, the volume fraction of the T/M lamella bundles is comparable to that in the LNT-DPD Cu–1.0% Al, whereas the T/M lamella thickness is smaller, averaging ~12 nm. The grains that evolve from the nanoscale T/M lamellae are smaller, with an average transverse size of 23 nm. The volume fraction of the elongated subgrains/grains derived from dislocation structures remains unchanged relative to the LNT-DPD Cu–1.0% Al, but the average transverse size decreases to about 86 nm.

### 3.1.2. Microstructure of RT-QSC Cu–Al alloys

The microstructure of RT-QSC Cu–0.5% Al alloy consists of strongly elongated subgrains/grains with roughly parallel lamellar boundaries (Fig. 3a), typical of the microstructure in Cu processed by CR, WD and ECAP [5–7,9]. The average aspect ratio of these subgrains/grains is about 5–10. Statistical measurement of the transverse grain size between neighboring lamellar boundaries (Fig. 3b) indicates that it exhibits a normal logarithmic distribution in the range from 100 to 600 nm, with a mean value of ~166 nm, which is much smaller than that in RT-QSC Cu (~290 nm) [22]. No deformation twins are observed.

Few deformation twins are observed in the RT-QSC Cu–1.0% Al alloy, and their morphologies are similar to those in LNT-DPD samples. An average T/M lamella thickness of about 41 nm is measured. The volume fraction of the T/M lamella bundles is less than 5%, indicating that dislocation slip dominates plastic deformation. The predominant microstructure of RT-QSC Cu–1.0% Al is composed of elongated subgrains/grains similar to those in RT-QSC Cu–0.5% Al, with an average transverse grain size of about 160 nm.

Profuse nanoscale T/M lamellae are observed in RT-QSC Cu–4.5% Al alloy, as shown in Fig. 4a. Statistical results indicate that the volume fraction of T/M lamella bundles is 25% and the average T/M lamella thickness is 21 nm (Fig. 4b). Accompanying the increased tendency to twinning, the predominant microstructure in the RT-QSC Cu–4.5% Al alloy, as shown in Fig. 4c, is characterized by nanosized grains, the transverse size distribution of which is in range from a few to 120 nm (with an average of 32 nm; see Fig. 4d). Strongly elongated subgrains/grains derived from dislocation structures, with an average grain size of about 90 nm, are seldom observed. Clearly, the plastic deformation occurs mainly by deformation twinning in the Cu–4.5% Al sample during RT-QSC. It should be mentioned that twinning is always accompanied by dislocation slip, since twinning needs to be accommodated by dislocation slip and dislocation reaction [25].

### 3.1.3. Effects of SFE and deformation parameters on microstructures of Cu–Al alloys

With a systematic TEM characterization of Cu–Al alloys with various Al concentrations processed by LNT-DPD and RT-QSC, several microstructural features are obtained. These are summarized in Fig. 5. For the LNT-DPD samples, the volume fraction of nanoscale T/M lamella bundles (Vol_{T/M}) drops slightly with decreasing SFE. In contrast, an obvious increasing Vol_{T/M} trend with decreasing SFE is observed when the Cu–Al samples are processed by RT-QSC. No deformation twins were found in Cu–Al alloys with SFE > 50 mJ m\(^{-2}\). For SFE below 50 mJ m\(^{-2}\), Vol_{T/M} increases with decreasing SFE, tending to a saturated value of 26% in the samples with SFE < 25 mJ m\(^{-2}\). Balogh et al. [32] and Zhao et al. [31] qualitatively estimated the twin density (\(\beta\)), defined as the probability of finding a TB between any two neighboring \{1 1 1\} planes by means of X-ray diffraction analysis, in HPT Cu and Cu–Zn alloys (also included in Fig. 5a). The increasing trend of twin density is similar to that of Vol_{T/M} in the RT-QSC Cu–Al alloys. Published data on the Vol_{T/M} of CR Cu–30 wt.% Zn alloy (SFE \(\sim 14\) mJ m\(^{-2}\)) [29] and LNT-DPD Cu–32 wt.% Zn alloy (SFE \(\sim 13\) mJ m\(^{-2}\)) [35] are also

![Fig. 3](Image)

Fig. 3. A typical TEM image (a) and transversal grain size distribution (b) in the RT-QSC Cu–0.5% Al sample.
From Fig. 5b one may see that \( k \) in the LNT-DPD Cu–Al decreases drastically from \( /C_{24}46 \) to \( /C_{24}12 \) nm with decreasing SFE. A similar correlation was noticed in the RT-QSC samples: \( k \) decreases from \( /C_{24}41 \) nm (1.0% Al) to \( /C_{24}21 \) nm (4.5% Al). It is obvious that \( k \) in the LNT-DPD samples is smaller than that in the RT-QSC samples with the same SFE. The reduction in \( k \) with decreasing SFE and smaller \( k \) in the LNT-DPD samples can be explained in terms of the effects of SFE and processing parameters on the nucleation and growth kinetics of deformation twins, as discussed previously [36]. Literature data on \( k \) in a Cu–30 wt.% Zn alloy [29] and a Cu–32 wt.% Zn alloy [37] are also consistent with the present results.

**3.2. Tensile properties of deformed Cu and Cu–Al alloys**

Fig. 6 displays tensile engineering stress–strain curves of several Cu and Cu–Al alloys after LNT-DPD and RT-QSC treatments, in comparison with the CG Cu. It is apparent that the deformed Cu and Cu–Al alloys exhibit much enhanced strength but very limited uniform elongation (\( \approx 1\% \)) compared with the CG samples, analogous to other deformed nanostructured metals and alloys [5,7,26,40]. The limited uniform elongation results from insufficient strain hardening in the deformed samples, which causes the onset of early necking under tension [41].

It is obvious that, with increasing Al concentrations, the strengths of both the LNT-DPD and the RT-QSC samples
are much enhanced, while the uniform elongation is insensitive. For the same alloy, the strength of the LNT-DPD sample is apparently higher than that of the RT-QSC one. The variation in yield strength with SFE of the deformed samples is summarized in Fig. 7, together with literature data for Cu, Cu–Zn and Cu–Al alloys processed by other deformation methods [26,40]. As shown in Fig. 7, Al concentration has a rather weak influence on strength for the CG Cu–Al solid solution, meaning that the solution hardening effect of Al is negligible in CG Cu. For the LNT-DPD samples, strength increases monotonically with decreasing SFE. For the RT-QSC Cu–Al alloys, strength also increases with decreasing SFE, but the rate of increase becomes larger when SFE exceeds 50 mJ m\(^{-2}\).

It is interesting to see that strength difference between the LNT-DPD and the RT-QSC samples increases with decreasing SFE, reaches a maximum at SFE \(\sim 50\) mJ m\(^{-2}\) (with 1.0 wt.% Al) and then diminishes when SFE gets smaller (see Fig. 8). In Cu–4.5% Al, the two strengths become comparable. The variation in yield strength with SFE in these samples can be understood in terms of their microstructure characteristics.

4. Discussion

4.1. Deformation mechanism

Understanding the combined effects of SFE and processing parameters on the deformation mechanism is vital for explaining the variation in microstructure and corresponding properties. Hence, it is essential to determine quantitatively the volume fractions of twinned regions during the processing. The measured results are summarized in Fig. 9.

For the Cu and Cu–Al alloys processed by LNT-DPD, deformation occurs mainly by twinning due to suppression of dislocation activities by the high strain rate and low temperature. Statistical measurements from plenty of TEM images indicated that about 85–95 vol.% of the LNT-DPD Cu and Cu–Al alloys is twinned during plastic deformation, as shown in Fig. 9. The twinned regions consist of...
two parts. One part is the remaining nanoscale T/M lamella bundles; the other part is those regions in which the T/M lamellae have been transformed into nanosized grains via fragmentation or shear banding of the T/M lamellae in subsequent deformation \[21,23\]. Measurements showed that about 55–65 vol.% of the deformed sample corresponds to nanosized grains derived from nanoscale T/M lamellae. Accordingly, the volume fraction of the remaining nanoscale T/M lamellae is about 30\%, as indicated in Fig. 5a. Only a small fraction of grains (5–15\%) are deformed via dislocation cells (DCs) in the LNT-DPD samples, which may originate from those grains whose orientations are not favorable for twinning during the deformation. Similar microstructures were formed in the LNT-DPD samples with varied Al concentrations, although their SFEs are different.

However, a distinctly different scenario is seen in the RT-QSC Cu and Cu–Al alloys. It is known that the primary deformation mode at RT and at low strain rates is dislocation slip in materials with high SFEs \[42\], and the corresponding strain-induced grain refinement is realized via dislocation activities. At the initial stage of deformation, plenty of cell boundaries and/or low-angle grain boundaries (GBs) are formed in the original coarse grains as a result of dislocation manipulation. With increasing strain, dislocation cells or subgrains transform into the strongly elongated subgrains/grains, with increased misorientations across the boundaries due to increasing dislocation interactions. As shown in Fig. 9, grain refinement of Cu and Cu–Al alloys with SFE > 50 mJ m\(^{-2}\) is predominantly by this mechanism, and the microstructure is characterized by strongly elongated subgrains/grains derived from dislocation structures. In fact, some deformation twins are formed inside the original coarse grains with preferable crystallographic orientations for twinning in the RT-QSC Cu–1.0% Al with SFE of \(~50\) mJ m\(^{-2}\). As the volume fraction of the twinned region is small (\(~5\)%), the influence of twinning on the grain refinement mechanism and microstructure is negligible.

With decreasing SFE, e.g. in the RT-QSC Cu–1.5% Al, deformation twinning becomes more pronounced in accommodating plastic strain, about 55 vol.% of the sample being deformed via twinning. Hence, the contribution of deformation twinning to the grain refinement becomes more significant, resulting in an obvious drop in the average grain size (see Fig. 9). The statistical results showed that the majority of twinned regions are transformed into nanosized grains (about 40\% out of 55\%), indicating that deformation twinning and its subsequent evolution play a vital role in grain refinement of the RT-QSC Cu–1.5% Al alloy. Clearly, the grain refinement in RT-QSC Cu–Al alloys with SFEs in a range of 50–25 mJ m\(^{-2}\) is achieved via two competing mechanisms: dislocation subdivision and nanoscale T/M lamella subdivision. The contribution of the T/M lamella subdivision gradually increases with decreasing SFE.

With SFE below 25 mJ m\(^{-2}\), deformation occurs mainly by twinning, and the final grains are mainly derived from the nanoscale T/M lamellae in the RT-QSC Cu–Al alloys, the microstructure of which is similar to that in the LNT-DPD samples. Qu et al. \[26\] found that the microstructure
of an ECAP Cu–5 at.% Al alloy consists of equiaxed and strongly elongated grains, and is less homogeneous than that of a Cu–8 at.% Al alloy in which the grains are nearly equiaxed. It is obvious that the equiaxed grains and strongly elongated grains correspond to those derived from T/M lamellae with a short aspect ratio and those derived from DCs with a large aspect ratio, respectively. Hence, our observed dependence of the microstructure on the SFE in Cu–Al alloys provides a reasonable explanation for their results.

Our results clearly demonstrate that the deformation mode is dependent upon the SFE of deformed materials. This may be explained in terms of the twinning stress dependence on SFE, which has been discussed by Christian and Mahajan [25]. Generally, twinning stress decreases while the stress for slip increases with decreasing SFE for copper-based alloys [25,34]. Hence, the twinning stress will be less than the stress for slip at a certain SFE in Cu–Al alloys, meaning that twinning preferentially takes place below the critical SFE. Mechanical twins can be found in the RT-QSC Cu–1.0% Al alloy with an SFE of 50 mJ m\(^{-2}\). As the SFE further decreases, more twinning tends to occur due to lower twinning stress compared with the flow stress, as seen in Fig. 9a. It should be noted that both the yield stress and the flow stress are not as sensitive to the SFE as the twinning stress for Cu–Al alloys in the present results and in the literature [34].

The effect of processing parameters on the deformation mode of Cu alloys can also be clarified from Fig. 9. For the SFE range of 50–78 mJ m\(^{-2}\), a pronounced effect of processing parameters was found in controlling the deformation mode: dislocation slip dominates the RT-QSC process while deformation twinning governs the LNT-DPD process. For SFE < 25 mJ m\(^{-2}\), deformation occurs mainly by twinning in both processes. In the SFE range of 25–50 mJ m\(^{-2}\), both slip and twinning occur, and the effect of the processing parameters diminishes with decreasing SFE.

Consequently, one may say that decreasing SFE has a similar effect as increasing strain rate or decreasing temperature in promoting deformation twinning in the plastic deformation of Cu–Al alloys, which facilitates the refinement of grains into the nanoscale.

4.2. Grain size

An obvious SFE dependence of \(D\) is observed in the Cu–Al alloys, consistent with previous investigations [26,31,32] that decreasing SFE leads to smaller grains in the HPT Cu–Zn alloys and ECAP Cu–Al alloys. To reveal the underlying mechanism responsible for this dependence, we performed statistical grain size measurements by distinguishing grains derived via different refinement mechanisms, i.e. those from dislocation structures and those from the nanoscale T/M lamellae, as indicated in Fig. 10. It is seen that for grains derived from DCs in the RT-QSC samples, their sizes decrease dramatically from about 300 to 90 nm with decreasing SFE. For the grains derived from the nanoscale T/M lamellae in the RT-QSC Cu–Al alloys, the size decreases from about 76 to 32 nm when the SFE decreases from 37 to 12 mJ m\(^{-2}\), as shown in Fig. 10a. In the LNT-DPD samples with decreasing SFE, the sizes of the grains derived from dislocation structures showed a slight decreasing trend from about 120 to 90 nm, while that from T/M lamellae also drops from about 50 to 25 nm.

The grain sizes derived from dislocation structures are controlled by the dimensions of the dislocation cells. An empirical relationship between the cell size (\(d_{\text{cell}}\)) and the applied stress (\(\tau\)) has been found [43]: \(d_{\text{cell}} = \frac{K\tau}{G}\), where \(K\) is a constant with a value close to 10, \(G\) is the shear modulus, \(b\) is the Burgers vector. This relationship applies to materials in which the applied shear stress is well in excess of the friction stress. It is apparent that, with an increment of the applied shear stress, the cell size decreases. With a decreasing SFE, the shear stress applied to the samples increases substantially due to the increase in strength of the RT-QSC sample, resulting in smaller dislocation cell sizes and eventually smaller grain sizes. For the LNT-DPD Cu–Al sample, the majority volume of which is deformed via nanoscale twinning, the effective shear stress applied on the untwinned volume is very high so that the resultant grain sizes are small. Plotting the measured grain sizes against the applied shear stress (approximated by the yield strength of the processed samples, as in Fig. 11) shows that...
the data from the LNT-DPD and RT-QSC samples fall on the same line. This is clear evidence that the highly elongated grains in the two sets of samples originate from the same grain refinement mechanism. Further decreasing SFE seems to lead to a saturated grain size of about 90 nm in both the RT-QSC and LNT-DPD samples. The limit of the dislocation cell size can be estimated when a maximum shear stress is applied. Taking the maximum shear stress as \( \frac{G}{30} \), then a minimum cell size of \( d_{DC} = 300b \) is obtained based on the relationship between the cell size and the applied stress. For Cu, \( d_{DC} \) is about 90 nm, which is consistent with the experimental observations reported in the literature [22]. Hence, the grain sizes derived from dislocation cells are usually in the submicron-sized regime, as seen in Fig. 10.

Grains derived from nanoscale T/M lamellae are always in the nanometer regime (<100 nm), much smaller than those derived from dislocation structures. The size of these grains is determined by their formation mechanisms. Two possible mechanisms have been proposed [21]: (i) fragmentation of nanoscale T/M lamellae and (ii) shear banding within the nanoscale T/M lamella bundles. The grain refinement process of mechanism (i) includes the following steps: (a) the formation of nanoscale-thick twins divide the original coarse grains into twin/matrix lamellae; (b) the development of dislocation walls inside the twin/matrix lamellae further subdivide these lamellae into equiaxed nanosized blocks; (c) these preferentially oriented blocks evolve into randomly oriented nanosized grains. Detailed analysis has been documented in Ref. [44]. With mechanism (i) grain sizes are very close to the original T/M lamella thickness (\( \lambda \)) since the grains are derived from the break-up of nanoscale-thick twin/matrix lamellae [21,44]. For mechanism (ii) nanosized grains are formed inside shear bands of the twin/matrix lamellae, involving detwinning and formation of dislocation boundaries, as discussed in detail in Refs. [23,45]. The sizes of the grains are slightly larger than \( \lambda \) due to grain coarsening induced by high stress and a transient temperature rise within the shear band [21,35,45]. With both mechanisms, grain sizes are correlated with and close to the T/M lamella thickness. Comparison of the SFE dependence of grain sizes from the T/M lamellae and the SFE dependence of \( \lambda \), as shown in Fig. 10, shows their consistency. Consequently, we may reasonably attribute the decreasing grain size with decreasing SFE to the SFE effect on the T/M lamella thickness.

### 4.3. Deformation and microstructure map

With the structure information presented above, we may summarize the deformation mode and microstructure characteristics of the Cu–Al alloys in an SFE-processing parameter (ln \( Z \)) space, as displayed in Fig. 12. The literature results from different sources [6,7,26–28,31,32,35,45–54] are also included. Three regions can be differentiated according to the deformation mode and microstructure:

- **Region I**: Deformation is dominated by dislocation slip and microstructures are characterized by ultrafine grains derived from dislocation cell (DC) structures with sizes of 100–300 nm. In region II, dislocation slip and deformation twinning compete with each other, forming a mixed microstructure of nanoscale T/M lamellae, nanosized grains derived from T/M lamellae and ultrafine grains from DCs. The proportion of nanoscale T/M lamellae and nanosized grains increases with decreasing SFE or increasing ln \( Z \).
- **Region III**: Deformation occurs mainly by deformation twinning. The microstructure is mainly composed of nanoscale twins and nanosized grains, and \( \lambda \) and \( D \) decrease.

![Fig. 12. A map of deformation mode and strain-induced microstructures in the SFE-processing parameters (ln \( Z \)) space for the Cu–Al alloys, Ultrafine grain (UFG); nanoscale twin (NT); nanosized grain (NG).](image-url)
with decreasing SFE or increasing ln Z. It is apparent from this map that decreasing SFE and increasing ln Z play a similar role in enhancing the tendency to deformation twinning and to achieve the mixed nanostructure of nanoscale T/M lamellae and nanosized grains.

4.4. Strength

The observed variations in yield strength with SFE in the two sets of samples can be reasonably attributed to the grain size effect as well as the T/M lamella thickness effect. Refining grains (increasing GB areas) induces strengthening, as described by the classical Hall–Petch relation. Similarly, increasing TB densities may also increase the strength of metals, and the twin (thickness) size effect on strength was found to be roughly identical to the grain size effect in Cu [55,56]. Therefore, we may use the effective grain size ($D_{\text{eff}}$) to analyze the strength variation. Here, $D_{\text{eff}}$ is defined as the average value of the grain size ($D$) and T/M lamella thickness ($\lambda$) in the sample, i.e., $D_{\text{eff}} = D(1 - \lambda V_T/M) + \lambda V_T/M$. Clearly, such an effective size includes the overall boundary density of both GBs and TBs. Plotting the measured yield strengths as a function of $D_{\text{eff}}^{1/2}$, as shown in Fig. 13, shows that all the yield strength data from the two sets of samples, together with the literature data on Cu processed by severe plastic deformation [9,13,57,58], fall approximately on one straight line. This implies that increasing densities of TBs and GBs contribute equally to strength, consistent with the literature [56,59].

It is noted that the yield strength values for the Cu–Al alloys are higher than the traditional Hall–Petch line derived from CG pure Cu [60]. The difference may be attributed mainly to the dislocation hardening, though also to the slight solution hardening effect. In deformed ultrafine-grained or nanograined metals, a high density of dislocations exists in the refined grains in various configurations (especially in the submicron-sized grains), which provides additional hardening to the material. This phenomenon has been observed and discussed in deformed pure Cu [61].

5. Conclusions

The effects of SFE, strain rate and deformation temperature on microstructure characteristics and strength have been investigated in nanostructured Cu–Al alloys with Al concentrations in a range of 0–4.5 wt.%, prepared by means of plastic deformation.

(1) In the LNT-DPD process, nanoscale deformation twinning dominates the plastic deformation process in each sample, in which overall twinned regions constitute about 90 vol.%. With decreasing SFE, the twinning tendency is enhanced, and is accompanied by a decreasing T/M lamella thickness and a reduction in grain sizes; hence, an increasing strength results.

(2) In the RT-QSC process, plastic deformation is governed by dislocation slip when the SFE is above 50 mJ m$^{-2}$. With decreasing SFE, twinning was found in deformed materials. When the SFE is below 25 mJ m$^{-2}$, deformation occurs mainly by twinning, and the resultant microstructures are comparable to those of LNT-DPD samples of the same composition. Twinning is obviously enhanced by decreasing SFE, resulting in smaller T/M lamella thickness and grain sizes. Consequently, an obvious strength elevation is induced by the size effects of grains and T/M lamellae with decreasing SFE.

(3) A map of the deformation modes and corresponding strain-induced microstructures is drawn in the SFE–processing parameters space for the Cu–Al alloys.

(4) The increasing strength with decreasing SFE in both sets of Cu–Al samples can be attributed to the increasing density of GBs as well as TBs.

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