Materials Research Express

PAPER

Mechanical properties, microstructure, and texture evolution of copper sheets subjected to differential speed rolling process

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Keywords: copper sheet, asymmetric rolling, roll speed ratio, mechanical properties, EBSD, recrystallization

Abstract
Copper sheets were subjected to differential speed rolling (DSR) at roll speed ratios (RSRs) from 1 to 3. The effects of the RSR on the yield strength, uniform strain, microstructure, and texture evolution in the copper sheets were investigated. The grain refinement and uniformity were obviously enhanced, the yield strength and uniform strains increased slightly, and the shear texture components were further widely distributed when the RSR was increased up to 2. At RSR = 3, the yield strength decreased, with a distinct increase in the uniform strains due to dynamic recovery and recrystallization as a result of the steep reduction in the geometrically necessary dislocation (GND) density and the grain orientation spread (GOS), thereby increasing the work hardening coefficient (n) and decreasing the work hardening rate (\(\theta\)).

1. Introduction
Copper can be manufactured in different forms, but is most popularly used in sheet form, where copper sheets are used for welding fixtures, ground straps, plumbing fitting, power transmission, and heat exchanger. The strength, ductility, conductivity, and corrosion resistance of copper sheets must be enhanced to expand their application in targeted fields. The microstructure of copper sheets can be manipulated through grain refinement and retardation of texture development. The microstructure of the bulk material can be converted to a nanostructure by means of severe plastic deformation (SPD) methods (e.g., high pressure torsion, equal channel angular pressing, etc.) [1–5]. This affords materials with distinctive microstructure evolution and texture evolution compared to those fabricated by conventional plastic deformations. SPD induces an increase in the dislocation density leading to partial formation of a dynamically recovered and recrystallized microstructure. The SPD technique imposes a large plastic strain on bulk materials, leading to a non-porous structure, excellent mechanical properties (e.g., high strength, toughness), and appropriate dimensions for mechanical and physical testing [6, 7]. Sheet materials can be subjected to SPD techniques such as accumulative roll bonding (ARB) [8, 9], repetitive corrugation and straightening (RCS) [10], constrained groove rolling (CGR) [11], constrained groove pressing (CGP) [12] and differential speed rolling (DSR) [13].

In general, to achieve grain refinement in sheet materials, the rolling conditions, such as the deformation strain and speed, must be controlled, where fine grains are formed as a result of the strong shear strain presented by the large frictional force between materials and roll during rolling. Conventional rolling (CR) or symmetric rolling processes are limited to imposing additional shear strain at the centre of the sheets. On the other hand, in DSR, it is probable that additional shear strain may be applied to the centre of the sheet due to the difference between a peripheral speed of upper and lower rolls [14]. Exerting such an imposed shear strain on the sheets can induce texture evolution by enhancing the slip system [15]. For various rolling conditions, the author [16] reported that the roll speed ratio (RSR) had a greater impact on the microstructural evolution than other variables (e.g., lubrication, deformation strain, speed, etc.) [17]. However, there is a relative paucity of well-
controlled studies that seek to identify the mechanical properties and deformation characteristics of copper sheets deformed by DSR using a broad range of RSR.

The aim of this study is to examine the effects of the RSR on the microstructural and textural evolution, as well as the mechanical properties, of copper sheets during cold asymmetric rolling, and to determine the relationship between these properties based on electron backscatter diffraction (EBSD) data. The variation in the geometrically necessary dislocation (GND) density and the grain orientation spread (GOS) is analyzed in detail to examine the extent of dynamic recovery and recrystallization during differential speed rolling as a function of the RSR.

2. Materials and methods

Electrolytic tough pitch (ETP) copper sheets with more than 99.98% Cu and less than 0.02% total impurities were used for the study. The sheets were 2 mm thick, 50 mm in width, and 100 mm in length. The sheets were annealed at 400 °C for 1 h. The machine used for the rolling process had upper and lower rolls of 300 mm in diameter and 250 mm in width, and operated a round per minute (rpm) of 1 m min$^{-1}$. The roll speed ratio (RSR) was varied from 1:1 to 1:3; the speed of the lower roll was maintained at 1 rpm, and that of the upper roll varied with the RSR. The entire sample was inserted into the gap of 0.5 mm between the upper and lower rolls and rolled at room temperature. A lubricant-free rolling process was employed to intensify the shear imposed on the sheets during deformation.

To investigate the mechanical properties of the copper sheets rolled at various RSR, a tension test with a video extensometer (Shimadzu AGS-X) was carried on at room temperature with a strain rate of 10$^{-3}$ sec$^{-1}$. The sample for the tension test was cut to fit the size and shape specified by the ASTM D638 standard, with a gauge length of 50 mm parallel to the rolling direction (RD). The microstructures and crystallographic orientations of the copper sheets at various RSR were characterized using the electron backscatter diffraction (EBSD) technique. The EBSD data were obtained for the longitudinal section of each sample in the planes in the normal direction (ND) and rolling direction (RD). Both planes of the samples were first mechanically ground, polished, and then electro-polished to remove the residual deformation on the surface layer to obtain an EBSD pattern with a high confidence index (CI). EBSD mapping was performed using a field-emission gun scanning electron microscope (Hitachi SU5000) with an EBSD acquisition camera (EDAX Velocity Super) and APEX software. An acquisition step size of 0.45 μm and area of 350 μm $\times$ 350 μm were applied for all samples. The raw data were interpreted using the orientation imaging microscopy analysis software provided by TSL OIM analyzer.

To calculate the strain rate with respect to the roll speed ratio, an approximate equation proposed by the authors [1, 18, 19] was used, based on the assumption that there was no frictional stress and that intense plastic strain was imposed on the entire sample during deformation.

$$\dot{\varepsilon} = \frac{\varepsilon}{t_{avg}} = \frac{\omega_{avg} \times \pi \times r}{15\sqrt{3} \times r (t_i - t_f)} \times \left[1 + \left(\frac{t_f^2}{t_f^2 - t_i^2} \tan\theta\right)\right]^{1/2} \times \ln\frac{t_i}{t_f}$$

Here, $t_i$ and $t_f$ indicate the thickness of the sample before and after the rolling process, respectively; $\theta$ is the apparent shear angle; $r$ is the radius of the roll; $\omega_{avg}$ is the average rpm of the upper and lower rolls. Table 1 lists the values of the parameters used in equation (1) and the calculated values of the strain rate corresponding to the roll speed ratios.

3. Results

The copper sheets rolled at various roll speed ratios were subjected to tension tests to investigate their mechanical properties; the results are shown in figure 1(a). The graphs indicate the true stress and strain curves.

| RSR | $t_i$ | $t_f$ | $\varepsilon$ | $\tan\theta$ | $\omega_{avg}$ | $\dot{\varepsilon}$ |
|-----|------|------|--------------|-------------|----------------|--------------|
| 1:1 | 2    | 1.03 | 0.76         | 0.081       | 49.3           | $\sim$ 3.1   |
| 2:1 | 2    | 0.78 | 1.08         | 0.09        | 73.9           | $\sim$ 5.9   |
| 3:1 | 2    | 0.62 | 1.35         | 0.096       | 98.6           | $\sim$ 9.2   |
until a neck begins to form in the engineering stress and strain curves. The as-received annealed copper sheets exhibited a yield stress (YS) of \( \sim 230 \, \text{MPa} \) and strain of \( \sim 0.2 \), and the sheets rolled at various RSR demonstrated a YS of more than 350 MPa and a strain of less than 0.02. Figure 1(b) shows the yield stress (YS) and ultimate tensile stress (UTS) for the copper sheets as a function of the RSR. The YS and UTS of the rolled copper sheets increased steeply with an increase in the RSR up to 2.0, followed by a sharp decrease when the roll speed ratio was increased to 3.0. The strength drop phenomenon has previously been documented for copper tubes hydrostatically extruded at a high extrusion ratio [20, 21].

The microstructural changes during rolling deformation were observed using EBSD. Figure 2 shows the image quality (IQ) plus inverse pole figure (IPF) maps for the planes perpendicular to the normal direction (a–d) and transverse direction (e–h) as a function of the RSR. The IQ is defined as the sum of the detected peaks in the Hough transform, and describes the quality of the EBSD pattern. The contrast in the IQ maps arose from the strain, topography, and grain boundaries in the current study, where the ND plane (figures 2(a)–(d)) showed high contrast after applying the rolling process, irrespective of the RSR, whereas the TD planes (figures 2(e)–(h)) showed the lowest contrast at an RSR of 3.0.

Figures 3(a)–(c) shows the variation in the mean grain size and misorientation angles between neighboring grains in both the ND and TD planes as a function of the RSR. Figure 3(a) shows that the mean grain size in both planes decreased up to an RSR of 2, but increased when the RSR was further increased. By applying a copper sheet larger than the RSR of 1, the standard deviation of the grain size decreased, which shows that the rolling process over the RSR of 1, that is, the differential speed rolling (DSR) process, is effective for grain refinement with uniformity. The reduction in the grain size was more clearly observed in the TD plane than in the ND plane as a function of the RSR. The distributions of the misorientation angles across the grain boundaries in the ND and TD planes at different RSR are displayed in figures 3(b), (c). The low-angle boundaries below the misorientation angle of 10° are the most important components in the ND plane, irrespective of the RSR, and are the most important components in the TD planes rolled below the RSR of 2. However, at high angles, close to the 60° misorientation, there were over three times as many boundaries in the TD planes of the copper sheet rolled at the highest RSR than in the TD planes of the as-received and annealed copper sheets. A small peak also appeared around 38°–39° misorientation in the TD planes rolled at the highest RSR, which was not observed for the other samples. Some of these discrimination can be accounted for the presence of twin boundaries, which refer to coincident site lattice (CSL) boundaries. Here, the peaks at misorientation angles of 60° and 38.94° indicate the Σ3-type twin boundary rotating about \( \{111\} \) and Σ9-type twin boundary rotating about \( \{110\} \), respectively. The Σ3-type twin boundaries are also referred to as annealing twin boundaries, which indicate that heat was generated inside the TD planes by severe plastic deformation at the highest RSR. The misorientation angle is closely related to the degree of coherence between the crystal lattices on either side. A low misorientation angle (\( \langle 100 \rangle \)) is indicative of a sub-grain boundary, usually in the form of an array of dislocations with a degree of coherence between two adjacent lattices. A high-angle boundary (\( > 10^\circ \)) is usually an incoherent grain boundary; however, the twin boundary, although represented by high angles, has a degree of coherency across it, and the two crystal lattices are linked by a specific misorientation and rotation axis. Coincidence site lattice (CSL) boundaries are indicated by Σ, which is the reciprocal ratio of the coincident atomic sites to the atomic sites of any parent lattice. A high Σ value represents fewer coincident points in a unit area, and hence, a higher interface energy per unit area [22]. Thus, it is clear that the \( \{111\} \{112\} \) twin systems are activated in the interior of some grains of the TD plane at the highest RSR.

To investigate the dynamic recrystallization (DRX) behavior of the copper sheets during deformation, the variation in the grain orientation spread (GOS) with the grain size as a function of the RSR was determined from the EBSD data, as shown in figures 4(a), (b) for the ND plane and TD planes. The GOS parameter was employed to distinguish the DRXed grains from the deformed grains. As documented by Allain-Bonasso et al [23], the GOS of grain \( i \) is expressed as follows:
where \( J(i) \) is the number of pixels in grain \( i \), \( \omega_{ij} \) is the misorientation angle between the orientation of pixel \( j \) and the mean orientation of grain \( i \). The GOS value depends on the degree of deformation, which leads to grain distortion. The threshold value for the GOS to separate the DRXed grains from the un-recrystallized grains varied from to \( 1^\circ \)–\( 2^\circ \) in various materials [24–26]. In the ND planes, grains larger than \( 3 \mu m \) have a GOS value.
greater than 2°, indicating that dominate as the RSR increases. No significant differences were found between the ND plane and TD planes below an RSR of 2. However, at the highest RSR, the DRXed grains were dominant in the TD plane, regardless of the grain sizes. These grains plausibly contribute to reducing the yield stress in the copper sheet rolled at the highest RSR.

To correlate and compare the textures of the copper sheets rolled at RSR = 1, 2, and 3, the amended nomenclature and convention proposed by the authors [27] is used as a reference for the ideal simple shear of face-centered cubic (FCC) structured materials (see table 2). Figure 5(a) shows the ideal textures corresponding to Table 2 for the [111] pole figures (PFs), and figures 5(b), (c) shows the pole figures of the representative starting textures in the ND and TD planes of the as-received copper sheets with RSR = 0.

The [111] pole figures for the ND plane (figures 6(a)–(c)) and TD planes (figures 6(d)–(f) of the copper sheets rolled at RSR = 1, 2, and 3 closely match the representative projections of ideal simple shear texture components. For the ND plane of the rolled samples with RSR = 1, the [111] PFs (figure 6(a)) indicate that the original, mainly copper [112] {111}, texture of the as-received sample was transformed to closely correspond to that of the A1(111)[211] and A2(111)[2 1 1] orientations. For RSR = 2 (figure 6(b)), textural features were further spread along and parallel to the transverse direction with equivalent intensity, and the textural evolution was accompanied by the appearance of the A(111)[110] and Â(1 1 1)[1 1 0] orientations. For the sample with RSR = 3 (figure 6(c)), the trend was similar to that of the sample with RSR = 2, wherein a further spread of the central A1 and A2 components of the [111][112] orientation towards the A and Â components of the [111]
The results show the well-established effects of the RSR of the upper to lower roll on the mechanical properties, as mentioned in the literature. As shown in figure 5(d); however, the {111} PFs underwent evolution to form Λ1, Λ2, and Λ and A components with further spread, similar to the ND plane (figure 6(b)), when the RSR was increased from 1 to 2. When the RSR was further increased to 3, the {111} PFs indicate randomly spread textural features with half of the intensity compared to that of the samples with RSR = 1 and 2.

4. Discussion

The results show the well-established effects of the RSR of the upper to lower roll on the mechanical properties, microstructure, and textural evolution of copper sheets at room temperature. As mentioned in the literature [28], by using asymmetric or differential speed rolling (DSR) with RSR > 1, thin sheets can be produced more easily than via the symmetric or conventional rolling (CR) process, with RSR = 1, as characterized by the lower temperature and load in a magnesium alloy sheet with a hexagonal close-packed (hcp) structure, exhibiting only three distinct slip systems. The DSR process afford a sheet with microstructure refinement and peculiar texture formation due to the stronger shear stress and strain compared to those induced by the CR process [15, 28, 29]. Similar to those previous reports, in this study, a higher RSR afforded thinner copper sheets, refining the microstructure with smaller grains and uniformity, induced texture evolution, leading to a configuration with a wider spread due to the increase in the strain rate through the samples (table 1). By increasing the RSR, the YS and UTS of the copper sheets increased up to RSR = 2, and then decreased at higher RSR (figure 1(b)). EBSD analysis (figures 2 and 3(a)) indicates that this trend is related to the microstructure evolution because the mean grain size of the sample is inversely proportional to the YS and UTS; the mean grain size decreased as RSR increased up to 2, and but increased at even higher RSR. Perhaps the most interesting finding was a drop in the yield strength and an increase in the grain size of the sample subjected to the highest RSR of 3. This trend is in agreement with previous observations by Valiev et al and Dalla Torre et al [30, 31]. Dynamic recovery and recrystallization occur at the boundaries in the sample with RSR > 2, after which grain growth or coarsening occur in the microstructure of the copper sheet subjected to the highest RSR during deformation. This might be associated with the course of recovery mechanisms that decrease the volume fraction of cells, boundaries and the total dislocation density. In turn, this decrease might cause an increase in the mean free path of dislocations, triggered by a transformation from non-equilibrium boundaries to more equilibrium boundaries [31]. As shown in figure 3, the grain growth and sharp drop in the fraction of misorientation angles of less than 5° at the largest RSR provide evidence of recovery, leading to a decrease in the strength of the sheets. In contrast with the ND plane, the fraction of misorientations with a lower angle largely decreased in the TD plane. It is proposed herein that the presence of both Σ3-type and Σ9-type twin boundaries at RSR = 3 (figure 3(c)), the so-called coincidence site lattice (CSL) boundary, might demonstrate recovery because the CSL boundaries are simplified due to merging of closest-neighbor atoms by the heat generated during severe plastic deformation [20, 32]. In this study, the temperature of the surface of the copper sheets increased from ~15 °C for the as-received samples to ~55 °C for the sample rolled at RSR = 3. Such generated heat could increase the proportion of Σ3 boundaries, in agreement with previous studies relating to thermomechanical treatments consisting of repeated
cycles of cold work followed by very short annealing [33]. In general, the CSL boundaries are related to the stacking fault energy (SFE) of materials, and are easily formed in lower SFE materials (e.g., Cu), where the lower the SFE, the higher the Σ3 proportion. The steep increase in the fraction of Σ3 boundaries at RSR = 3 is associated with recrystallization, because the formation of special boundaries relies on combinations of strain and annealing [34, 35]. Such boundaries are supposed to occur for one of two reasons: to decrease the overall interfacial energy when the energy of the boundaries between a grain’s neighbors and its twin boundary is less than that of the boundaries between the neighbors and the grain itself [36], or to reorient grain boundaries to facilitate dislocation absorption and mobility during recrystallization [37].

To confirm the occurrence of dynamic recovery and recrystallization during severe plastic deformation (RSR = 3), it is useful to measure the geometrically necessary dislocation (GND) density, which is termed the extent of the lattice curvature. The GND, develops as a result of non-uniform strain at the crystal scale, accommodated by some dislocations during plastic deformation [38–40]. Dislocation annihilation and rearrangement, (constituting the recovery phenomenon), occur to a great extent prior to recrystallization, after which, the GND density decreases by reducing the amount of lattice curvature. EBSD is an advantageous technique for estimating the GND density based on analysis of the intergranular orientations. Based on the strain gradient model proposed by Gao et al, Kubin and Mortensen considered the case of simple torsion and related the misorientation angle θ to the GND density using the following equation [41, 42]:

$$\rho_{\text{GND}} = \frac{\alpha \theta}{b x}$$

(3)

here $\alpha = 2$ for tilt boundaries (low-angle grain boundaries), $\theta$ is the local misorientation angle, $x$ corresponds to the EBSD scan step used to compute the kernel average misorientation (KAM) map, and $b$ is the magnitude of the Burgers vector. Using the EBSD data, the average KAM value of all measurement points was calculated for the first five neighbors. The results are presented as a function of the absolute kernel radius in figure 7(a) for the ND plane and in figure 7(b) for the TD plane, of the as-received sample and the samples rolled with RSR = 1–3. The slope of the linear evolution (figure 7(c)) is the misorientation per unit length $d\theta/dx$ and can be used in equation (3) to replace $\theta/x$ [43]. Figure 7(d) shows the plot of the average GND density versus the RSR (from 0 to 3) for the ND and TD planes of the samples. The GND density of the as-received sample increased from $\sim 1.1 \times 10^{14} \text{ m}^{-2}$ to $\sim 2.0 \times 10^{14} \text{ m}^{-2}$ in the rolling process with RSR = 1; however, the GND density did not increase when the RSR was further increased. In the case of the TD plane, the GND density decreased significantly to $\sim 0.2 \times 10^{14} \text{ m}^{-2}$. This phenomenon indicates that dynamic recovery leading to dislocation annihilation or rearrangement occurred in the sample rolled at RSR = 1, irrespective of the increasing strain rate (table 1). Furthermore, the most compelling finding was that the GND density of the TD plane was greater than that of the ND plane up to RSR = 2. This difference indicates that the TD plane of the copper sheets was subjected to more strain than the ND plane during deformation. Up to RSR = 2 (figure 3(c)), large numbers of low-angle grain boundaries (LAGBs) persisted in the microstructure of the TD plane. These LAGBs form upon the coalescence of cells that accumulate misorientation as the RSR increases. As a result, these regions are rich in GNDs, which provide the strain energy required to drive recrystallization. This phenomenon can be explained by the large reduction in the GND density in the TD plane of the sample rolled at RSR = 3. This postulate is
further supported by the lowest GOS value (figure 4) of the microstructure in the copper sheets with RSR = 3, which considers the overall grain size.

The reduction in the GOS and GND density during the asymmetric rolling process (RSR > 1) was accompanied by, texture evolution, with further spread of the configuration and lower intensity (figure 6), especially at RSR = 3. Thus, it can be concluded that asymmetric rolling methods are more efficient in activating various slip systems than symmetric rolling methods (RSR = 1) because the shear texture components (see table 2) were more obvious in the sample rolled at RSR ≥ 1. Consequently, it was possible to obtain a copper sheet with a microstructure consisting of distributed grains with greater uniformity. To investigate the relationship between the mechanical properties and microstructure evolution, including the texture evolution, the hardening exponent \( n \) and strain hardening rate \( \theta \) were calculated, as shown in figure 1(a). The work hardening exponent, or \( n \)-value, of a material is a measure of how quickly the material gains strength during deformation. The \( n \)-value was determined from the slope of the true stress versus true strain curve in figure 1(a) and, plotted on a logarithmic scale (figure 8(a)). The relationship between stress and strain can be expressed by equation (4):

\[
\sigma = k \epsilon^n
\]  

(4)

where \( \sigma \) and \( \epsilon \) are the true stress and strain, respectively, and \( k \) is constants. As can be seen in figure 8(a), \( n \)-value increased with an increase in the RSR, which indicates that the copper sheets fabricated with higher RSR show good formability because they can work sufficiently hard in critical areas to better distribute the strains over other areas, thus reducing the local buildup of strains [44]. The increase in the uniform plastic elongation at higher RSR (figure 1(a)) can be explained by the less pronounced decrease in the work-hardening rate \( \theta \) as the stress and strain increase. The normalized work-hardening rate \( \theta \) can be defined as equation (5):

\[
\theta = \frac{\partial \sigma}{\partial \epsilon}
\]  

(5)

Figure 8(b) shows the work-hardening rate curves corresponding to figure 1(a). The overall work-hardening rate clearly decreased with increasing RSR. The less pronounced decrease in the work-hardening rate of the samples subjected to a higher RSR indicates that work hardening due to the accumulation of dislocations was regained, which consequently leads to larger plastic deformation owing to the presence of dynamically recovered and recrystallized grains (figure 7(d)) [45, 46].

5. Conclusion

The effects of the rolling speed ratio (RSR) of upper to lower roll on the mechanical properties, microstructure, and texture evolution of an electrolytic tough pitch (ETP) copper sheet were assessed using tension tests and EBSD analysis. Significantly, microstructure and texture development during deformation was influenced by RSR, attributed to the reduced yield strength and increase in the strains until necking occurred at the largest RSR due to dynamic recovery and recrystallization. The results of the study led to the following conclusions.

1. The strain rate imposed on the copper sheet increased with the RSR, and thinner copper sheets consisting of microstructures with smaller grains were easily fabricated, which induced greater strength and a more refined microstructure. Furthermore, at the largest RSR of 3, the yield strength decreased and the uniform strain increased.

2. The microstructure and texture of the ND and TD planes in the rolled samples evolved differently with increasing RSR. In both planes, the grain refinement in the microstructure improved as a function of the RSR; furthermore, more fine recrystallized grains were observed in the TD plane than in the ND plane. For the
copper sheet rolled at RSR = 3, the TD plane contained a large fraction of the special boundaries of the Σ3 and Σ9 types, which influence the texture intensity. Moreover, the shear components were more expansively distributed in the texture configuration, thereby affording a sheet with larger uniform strains.

3. With increasing RSR, dynamic recovery (DRV) and recrystallization (DRX) actively occur during deformation, which is supported by the fact that the GND density and GOS did not increase with the RSR; in addition, the GND density and GOS decreased significantly at the largest RSR of 3. DRV and DRX in the copper sheets promote an increase in the work-hardening coefficient and a decrease in the work-hardening rate during uniaxial tensile deformation.

Acknowledgments

This study was supported by a Grant from the Multiannual R&D Program (PIC21017) funded by the Ministry of Trade, Industry and Energy, Korea.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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