Original Article

Examination of high-temperature mechanisms and behavior under compression and processing maps of pure copper

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Abstract
The hot compression behavior of pure copper (commercial grade) was studied in a temperature range between 843 and 993 K and in a strain rate range between $10^{-3}$ and $10^{-1}$ s$^{-1}$. The activation energy for plastic flow was determined to be 215 kJ/mol, which is similar to the activation energy for lattice diffusion in copper ($=197$ kJ/mol). The stress exponent values associated with the plastic flow were 7–9, which are larger than the theoretical values associated with lattice-diffusion controlled dislocation climb creep (4.5–5). A comparison of the current data with the data obtained from the creep tests performed on the pure copper at low strain rates by other investigators in a similar temperature range revealed that the most of the data studied in this study belong to the regime where power-law breakdown occurs. Processing map constructed at a strain where steady-state plastic flow was observed showed that the power dissipation efficiency was low ($<21\%$) in the entire experimental ranges of strain rate and temperature. Discontinuous dynamic recrystallization (DDRX) associated with a single peak or multiple peaks in the stress-strain curves occurred during the first stage of plastic deformation. Dynamic recovery occurred after the DDRX activity ended, leading to the formation of a subgrains with low- and intermediate angle grain boundaries within the recrystallized grains with high-angle grain boundaries. Due to the formation of extensive substructure within DDRX grains, the fractions of high-angle grain boundaries measured after the compressive deformation were low ($<30\%$).

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1. Introduction

Forged copper with excellent formability, high electrical and thermal conductivities, nonmagnetic properties, nonsparking characteristics and high corrosion resistance has been widely used in manufacturing electrical and electronic components for aerospace, energy and defense applications.

The creep properties of pure copper have been extensively studied [1–8]. For example, Wang and Nix [3] studied the tensile creep behavior of pure Cu with a grain size of 310 μm in the temperature range between 726 and 993 K. They reported that the activation energy for creep measured in the strain rates below $10^{-4}$ s$^{-1}$ was 197 kJ/mol, which is equal to the activation energy for self-diffusion in copper ($=197$ kJ/mol [9]). Forging practices, however, require a large deformation at high...
strain rates under compression. Therefore, to find the optimum conditions for hot working of pure Cu and to understand the associated hot deformation mechanism under hot working, an analysis of its deformation behavior, post-deformation microstructures and processing maps at high strain rates of $10^{-3}$–$10^{-1}$ s$^{-1}$ under compression is essential.

There are several studies on the hot compressive deformation of pure copper. Gao et al. [10] studied the hot deformation behavior of coppers with different purities at temperatures from 523 to 773 K under strain rates of $10^{-4}$–$10^{-1}$ s$^{-1}$. The relationship between flow stress and strain rate could be expressed by a power-law function with stress exponents ($n$) of 7.3–8.3 and the activation energy for plastic flow was evaluated to be 210–245 kJ mol. Liu et al. [11] studied the hot compressive deformation behavior of pure copper in the high temperature range between 773 and 1073 K and in the strain rate range between $10^{-2}$ and $10$ s$^{-1}$. The activation energy for plastic flow was measured to be 185 kJ/mol, and the associated $n$ values were 5–7. The authors showed the presence of a flow instability region in the processing maps above $10^{-1}$ s$^{-1}$ in the temperature range between 773 and 970 K and claimed that the optimum hot deformation processing conditions were near 800–950 K and 0.05–2.5 s$^{-1}$ with power dissipation efficiencies of 28–40%. Huang et al. [12] studied the hot compressive deformation on the annealed pure copper in the temperature range between 673 and 1173 K and in the strain rate range between $10^{-3}$ and 1 s$^{-1}$. The average activation energy for plastic flow was 303.8 kJ/mol. In the processing maps, flow instability occurred at strain rates between 5 x $10^{-2}$ and 1 s$^{-1}$ in the temperature range between 673 and 1073 K.

The above reviewed results show that the stress exponents under hot compression at high strain rates are larger than those measured in creep at low strain rates, suggesting that a different deformation mechanism may operate at strain rates above $10^{-3}$ s$^{-1}$. In the present work, we systematically examined the hot compressive behavior of pure copper in the temperature range between 843 and 997 K and in the strain rate range between $10^{-3}$ and 10 s$^{-1}$ to understand the hot deformation mechanism and processing maps of pure copper at high strain rates for hot working. The obtained data were compared with the creep data, and the mutual correlation was examined. The microstructural evolution during compression deformation were also investigated.

2. Experimental procedures

The material used in this study was pure copper with a purity of 99.9% that was annealed at 1023 K for 10 h. Cylinder-shaped specimens with a diameter of 10 mm and a height of 12 mm were prepared for uniaxial compression tests. The hot compression tests were conducted in the temperature range between 843 and 993 K (0.62–0.73 $T_m$, where $T_m$ is the melting temperature of pure Cu (1356 K)) and in the strain rate range between $10^{-3}$ and $10^1$ s$^{-1}$ using a Gleeble 3500 thermomechanical simulator unit. To reduce the friction coefficient, both ends of the samples were lubricated by a Ni based lubricant. During the compression tests, the samples were heated to the test temperatures at a heating rate of 10 K/min and held for 5 min before the start of the compression test. The specimens were deformed to a total true strain of 1.2 to reach the steady flow-stress state. The adiabatic temperature rise was recorded for each compression specimen using a K-type thermocouple, and the flow stress due to deformation heat was corrected.

The microstructures after hot deformation were examined by electron back-scattering diffraction (EBSD). The microstructural observation was conducted at a location that was 1/3 away from the surface toward the center of the compressed sample. For the EBSD measurements in the field-emission scanning electron microscope (S-4300SE, HITACHI, Schaumburg, IL, USA), the samples were polished using a 1 μm diamond paste and then 0.04 μm colloidal silica after conventional polishing procedures were completed. The EBSD data were analyzed with the aid of TSL software (Version 7.0), which sets up a tolerance angle of 5° and a confidence index value >0.1. The EBSD step sizes was 0.2 μm.

3. Results

Fig. 1 shows the optical microstructure of the annealed copper. The microstructure is fully recrystallized, and many annealing twins are found within grains. The average grain size is 65.2 μm (measured without including the twin boundaries).

Figs. 2(a–e) show the true stress–true strain curves obtained from a series of compression tests carried out on the annealed Cu at different strain rates and temperatures. The true stress–true strain curves are characterized by multiple peaks or a single peak flow, followed by a steady state flow stress. When multiple peaks appear, stress reaches the peak, and a damped stress oscillation occurs during straining. The amplitude of the stress oscillation gradually decreases as deformation proceeds. The stress oscillation tends to increase as the strain rate decreases and temperature increases. Similar phenomena have been reported in the pure Cu by other investigators [13] and have been attributed to the occurrence of discontinuous dynamic recrystallization (DDRX) during deformation.

The relationship among strain rate, temperature and flow stress in pure metals and metallic alloys can be described by the sine hyperbolic Garofalo equation that considers the dependence of the steady-state strain rate on the flow stress.
and temperature for both low and high stresses, where power-law creep and power law breakdown (PLB) dominate plastic flow, respectively [14]:

\[ \dot{\varepsilon} = A \sinh(\alpha \rho) \exp \left( -\frac{Q_c}{RT} \right) \]  

(1)

where \( Q_c \) is the activation energy for plastic flow, \( A \) is the material constant, \( R \) is the gas constant, \( n \) is the stress exponent, and \( \alpha \) is the fitting parameter. At low stresses, Eq. (1) can be simplified into Eq. (2), which represents power-law creep. At high stresses, Eq. (1) can be simplified into Eq. (3), which represents power-law breakdown (PLB) where the strain rate is higher than that predicted by Eq. (2).

\[ \dot{\varepsilon} = A \alpha^n \exp \left( -\frac{Q_c}{RT} \right) \]  

(2)

\[ \dot{\varepsilon} = A \exp(\beta \sigma) \exp \left( -\frac{Q_c}{RT} \right) \]  

(3)

where \( \beta = \omega \eta_1 \). The values of \( n_1, \beta, \alpha \) and \( Q_c \) were calculated at \( \varepsilon = 0.7 \) where the steady-flow stress state was obtained. To evaluate \( \alpha \), the experimental data at high stresses (for the determination of \( \beta \)) and low stresses (for the determination of \( n_1 \)) were used separately. Fig. 3(a) shows the plots of \( \ln \dot{\varepsilon} - \sigma \) for the determination of \( \beta \) at different temperatures at \( \varepsilon = 0.7 \). From a linear fit of the data points for high strain rates from \( 10^{-3} \) to \( 10^{-1} \) s\(^{-1} \), the \( \beta \) values were determined and are in the range of 0.124–0.15. The average \( \beta \) value is 0.125. In the analysis for determination of \( \beta \) value, the three data at high strain rates were used because appreciable deviation from linearity occurs when the data for lower strain rates are included. Fig. 3(b) shows the plots of \( \ln \dot{\varepsilon} - \ln \sigma \) for the pure Cu at \( \varepsilon = 0.7 \) for the determination of \( n_1 \) values at different temperatures from the linear fit of the data at low strain rates \( (10^3 - 10^1) \) s\(^{-1} \). The \( n_1 \) value gradually decreases from 9.06–8.58 as the temperature increases, and the average value is 8.76. The \( \alpha \) value was determined by using the average \( \beta \) and \( n_1 \) values \( (\alpha = \beta/n_1): 0.0142 \).

Once the \( \alpha \) is determined, the \( Q_c \) value in Eq. (1) can be calculated using Eq. (4), which is obtained by differentiating Eq. (1) with respect to the strain rate and temperature:

\[ Q_c = R \left[ \frac{\partial \ln[\sin h(\alpha \rho)\mid_{\ln(1/T)}]}{\partial (1/T)} \right] - \frac{\partial \ln \dot{\varepsilon}}{\partial \ln[\sin h(\alpha \rho)\mid_{\ln(1/T)}]} \]  

(4)

where \( \frac{\partial \ln[\sin h(\alpha \rho)\mid_{\ln(1/T)}]}{\partial (1/T)} \) is determined by measuring the average slopes of the curve fits in the plots of \( \ln \dot{\varepsilon} - \ln \sin h(\alpha \rho) \) at various temperatures (Fig. 3d). The \( Q_c \) value calculated by Eq. (4) is 215.1 kJ/mol. This value is reasonably close to the activation energy for self-diffusion in pure Cu (197 kJ/mol) [9]. The plots of \( \ln Z - \ln \sigma \) and \( \ln Z - \ln \sin h(\alpha \rho) \) with \( \alpha = 0.0142 \) MPa\(^{-1} \) and \( Q_c = 215.1 \) kJ/mol at \( \varepsilon = 0.7 \) (where \( Z \) is the Zener–Hollomon parameter, \( Z = \dot{\varepsilon} \exp \left( \frac{Q_c}{RT} \right) \) ) are shown in Fig. 4(a) and (b), respectively. A good correlation between the two parameters is observed in each curve, supporting the reliability of the \( Q_c \) and \( \alpha \) values calculated following the above procedures.

Fig. 5 shows the plot of \( \log \frac{1}{\dot{\varepsilon}} \) vs. \( \log (\rho/G) \) for the pure Cu. Here, \( D_t \) is the lattice diffusivity of pure Cu \( (= 2 \times 10^{-5}. \exp (-197000/RT) \) m\(^2\)/s) [9] and \( G \) is the temperature-dependent shear modulus of pure Cu \( (= 4.71 \times 10^4 - 16.77 \) T MPa) [9]. The creep data for pure Cu studied by Wang and Nix [3] are also included in the plot. The two data sets appear to be well correlated with a single curve where \( n_1 \approx 5 \) is associated at low stresses and \( n_1 \approx 7 \) is associated at high stresses. The transition of the \( n_1 \) value from 7 to \( \geq 7 \) occurs under \( \dot{\varepsilon} = 10^{13} \) m\(^{-2}\), where many metals show the onset of PLB [15,16]. This result
suggests that most of the data obtained from the compression tests belong to the regime where $D_L$-controlled PLB dominates deformation.

Processing maps, which are composed of a power dissipation map and an instability map, reveal the characteristics of the power dissipation by microstructural evolution and the regime where unstable plastic flow occurs [17]. The power dissipation map is plotted in terms of the efficiency of the power dissipation, $\eta$, which represents how efficiently the power is consumed by the microstructure change during plastic deformation [17]. The $\eta$ values were calculated using the strain-rate sensitivity exponent ($m = 1/n_1$) that was determined based on fits to the log $\sigma$ vs. log $\dot{\varepsilon}$ curves obtained from the true stress-true strain as a function of strain rate and temperature - at $\varepsilon = 0.7$ and 1.0 using a cubic spline (third-order polynomial):

$$\eta = \frac{2m}{m+1}$$

Fig. 3  (a) The plots of ln $\dot{\varepsilon} - \sigma$ for the determination of $\beta$ at various temperatures at $\varepsilon = 0.7$ and (b) the plots of ln $\dot{\varepsilon} - \ln \sigma$ at various temperatures for a $\Sigma = 0.7$, (c) the plots of ln $(\sinh(\alpha)) - 1/T$ at various strain rates and (d) the plots of ln $\dot{\varepsilon} - \ln (\sinh(\alpha))$ at various temperatures.

Fig. 4  The plots of (a) ln $Z - \ln \sigma$ and (b) ln $Z - \ln \sinh(\alpha)$ at $\varepsilon = 0.7$ (where $Z$ is the Zener–Hollomon parameter, $Z = \dot{\varepsilon} \exp \left( \frac{Q_c}{RT} \right)$) in which $\dot{\varepsilon} = 0.0142$ MPa$^{-1}$ and $Q_c = 215.1$ kJ/mol.
The contour values in the power dissipation maps represent the $\eta$ values by percentage. The $\eta$ values extracted from the power dissipation map are plotted as a function of strain rate at 843 and 993 K (Fig. 6c). There are two important findings from Fig. 6(a–c). First, the $\eta$ values are small as 8–21%, and its variation with temperature and strain rate is small over the entire experimental range. The recorded $\eta$ values suggest that dynamic recovery (DRV) is dominant in the steady state deformation. This is because DRV is typically associated with $\eta$ values of 20–30%, while dynamic recrystallization (DRX) is associated with higher $\eta$ values of 35–45% [18]. Second, the flow instability regime does not form anywhere in the processing map at $\varepsilon=0.7$, but a small instability domain locally forms in the temperature range of 843–903 K above the strain rate of $10^{-1}$ s$^{-1}$ at $\varepsilon=1.0$.

Fig. 7(a–d) show the inverse pole figure and grain boundary maps of pure Cu after compressive deformation at 843 K and 993 K at two strain rates of $10^{-3}$ and $10^{-1}$ s$^{-1}$. The average grain sizes determined by EBSD are presented as a function of Zener-Holloman parameter in Fig. 8(a). As shown in Fig. 7(a–d), in all cases, the microstructures have fully recrystallized DRX grains, but substructures with low and intermediate angle grain boundaries developed in the grain interiors. The grain size decreases from 125.7–16.5 $\mu$m as the Z value increases (i.e., as the strain rate increases and temperature decreases). Due to the presence of many subgrains within the recrystallized grains, the fractions of high-angle boundaries are low (less than 0.3). The fraction of high-angle grain boundaries (HAGBs) tends to increase from 0.08 to 0.3 as the Z value increases.

**Fig. 5** – The plot of $\log \frac{\varepsilon}{\varepsilon_0}$ vs. $\log (\sigma/G)$ for the pure Cu.

In constructing the flow instability map, Prasad’s criterion [17] was used, and according to it, the instability parameter $\xi$ can be expressed as follows:

$$\xi = \frac{d\ln(m/(m+1))}{d\ln\varepsilon} + m < 0$$

(6)

When $\xi$ becomes negative, deformation in the material is predicted to be unstable due to flow localization or cracking.

The power dissipation maps for the pure Cu constructed at $\varepsilon=0.7$ and 1.0 are shown in Fig. 6(a) and (b), respectively.

**Fig. 6** – (a) The power dissipation map for the pure Cu constructed at (a) $\varepsilon=0.7$ and (b) $\varepsilon=1.0$. (c) The $\eta$ values of the pure Cu plotted as a function of strain rate at 843 and 993 K at $\varepsilon=0.7$. 
Fig. 7 – The inverse pole figure and grain boundary maps and the inverse pole figures of pure Cu after compressive deformation at 843 K at (a) \(10^{-3}\) and (b) \(10^{-1}\) s\(^{-1}\) and at 993 K at (c) \(10^{-3}\) and (d) \(10\) s\(^{-1}\). In the EBSD maps, low-angle grain boundaries \((2^\circ \leq \theta < 5^\circ)\), intermediate-angle grain boundaries \((5^\circ \leq \theta < 15^\circ)\) and high-angle grain boundaries \((15^\circ \leq \theta)\), are represented by light blue, blue and black colors, respectively.

(Fig. 8b). This is because the DDRX grain size decreases with increasing Z value.

The misorientations measured along the arrows marked in Fig. 7(a–d) are shown in Fig. 9(a–d). In all cases, the point-to-point misorientations are 2–5° and the point-to-origin misorientations are less than 10–15°. Negligible point-to-origin misorientation gradients are present within grains, indicating the homogeneous formation of subboundaries in the grain interior during compressive deformation, which gives rise to defined and separated regions inside the grains. The inverse pole figures shown in Fig. 7(a–d) indicate that hot compression induces an apparent, though weak, \((110)\) fiber
texture. It is known that deformation twinning and dislocation slip lead to the formation of ⟨110⟩ fiber texture in copper [19].

4. Discussion

The appearance of multiple peaks in the stress-strain curves in metals has been attributed to multiple instances of DDRX during deformation [13]. Whether the flow curve has multiple peaks or a single peak depends on the relative grain size ($D_o/D_s$), where $D_o$ is the initial grain size and $D_s$ is the stable dynamic grain size established at large strains [13]. When $D_o > 2D_s$, grain refinement occurs and a single peak flow appears, but when $D_o < 2D_s$, grain coarsening occurs and multiple peak flow appears. As shown in Fig. 8(a), the grain size ($D_s$) decreases as the Z value increases. Condition with $D_o > 2D_s$ corresponds to the deformation at 843 K and 10 s$^{-1}$, and condition with $D_o < 2D_s$ corresponds to deformation at the 998 K and 10$^3$ s$^{-1}$ and 843 K and 10$^3$ s$^{-1}$, indicating that the multiple peak criterion based on $D_o = 2D_s$ agrees with the current experimental observation.

During dislocation climb creep, it has been shown that subgrains form as a result of climb of gliding dislocations created by deformation, and the subgrain size decreases as flow stress increases [15,20]. During PLB, dislocations also form subgrains, but it is proposed that they are imperfect sink for
excess vacancies and thus do not have the ability to sweep out any excess vacancies that are created during dislocation glide or climb [15,20]. When the rate of vacancy production exceeds the rate of vacancy annihilation, the rate of creep is expected to be higher than that predicted by the power-law creep equation since excess vacancies accelerates creep process by increasing atomic diffusivity.

In the stress-strain curves (Fig. 2a–e), peaks do not appear after a strain of ~0.6 at all conditions, indicating that the microstructures observed by EBSD in Fig. 7(a–d) represent those developed after the DDRX activity ended. DRV occurred and as a result, subgrains were extensively developed in the interiors of recrystallized grains. Many low-angle grain boundaries are observed to have transformed into intermediate-angle grain boundaries in grain interiors (Fig. 7), which are necessary for the nucleation of continuous dynamic recrystallization (CDRX) [21]. During CDRX, low-angle grain boundaries transform into HAGBs by three ways [20]: (1) by a homogeneous increase in the misorientation (HIM) of low-angle grain boundaries, (2) by a progressive lattice rotation near the grain boundaries, and (3) by the formation of micro średni parts. The GB maps and misorientation angle profiles shown in Figs. 7(a–d) and 9(a–d) suggest that CDRX by HIM will occur when compressive strain is further imposed beyond ε = 1.2. This speculation is based on the observation that there is no gradient in the point-to-origin misorientations and the intermediate angle grain boundaries evolved from low angle grain boundaries are homogeneously distributed in the interiors of grains. The occurrence of CDRX during hot compressive deformation has been recently observed in Al-Mg alloys [22] and high entropy alloys [23].

5. Conclusions

The hot compression behavior of pure copper (commercial grade) was studied in the temperature range between 843 and 993 K and in the strain rate range between 10^{-3} and 10 s^{-1}, and the following results were observed.

1. The appearance of a single peak or multiple peaks during compressive deformation could be explained with the multiple peak criterion based on the relative grain size (Dp/Dl).
2. The activation energy for plastic flow was measured to be 215 kJ/mol, indicating that the lattice diffusion controlled plastic flow occurred during the compressive deformation.
3. The stress exponent values associated with plastic flow were 7–9 over the entire range of experimental conditions. Comparison with the current data with the data obtained from the creep tests performed on the pure Cu at low strain rates suggests that most of the data obtained from the current compression tests belong to the Dl-controlled PLB regime of which onset occurs around Dl = 10^{13}.
4. Processing maps constructed at strains where a steady state was attained indicated that the power dissipation efficiency was ≤21% over the entire experimental conditions, suggesting that DRV dominantly occurred after DDRX.
5. CDRX by HIM is anticipated to occur at large strains beyond a strain of 1.2.

Conflict of interest

The authors declare no conflicts of interest.

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