Mechanical Testing

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Effect of thermomechanical processing on the mechanical properties of CuZn10 alloy

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Abstract: The effects of thermomechanical processing on the grain size, tensile strength, and strain rate of a CuZn10 alloy were investigated. Microstructures of thermomechanically treated samples were introduced using a scanning electron microscope. The relationships between the flow stress, reduction ratio, annealing temperatures, and strain rates were detected for determination of activation energies and optimized thermomechanical parameters. The effect of grain size on the mechanical properties was examined by tensile and microhardness tests. The typical fractographs of samples after tensile tests were examined using a scanning electron microscope. It was observed that the annealing temperature and aging time have significant effects on the optimum grain size. Mechanical properties of the thermomechanically treated samples were improved by strain rate, deformation ratio, annealing temperature, and aging time.

Keywords: CuZn10 alloy; hardness; microstructure; tensile strength; thermomechanical processing.

1 Introduction

CuZn10 is a copper alloy having 10 wt% zinc and reinforced by a solid solution, and it is suitable for bending, stamping, and other cold-forming processes. As the zinc rate increases, strength improves, but conductivity and ductility decrease. CuZn10 shows good durability to stress corrosion cracking. Brass is the most commonly used copper alloy, and it has economic advantages due to its high zinc content. These alloys are used in a wide variety of fields, such as architecture, deep-drawn products, jewelry, and mechanical and electrical engineering components. A maximum of 37.5 wt% Zn has a microstructure containing a cubic face-centered lattice (α-brass) [1–4]. The amount, kind, and dispersion of grain boundaries in the polycrystal metallic materials have a significant effect on their properties. The mechanical and chemical properties of various metallic materials can be improved by the grain boundary. Therefore, optimum grain boundary is important for manufacturing methods. Increasing the ratio of low-angle grain boundaries increases the effect of grain size on the mechanical strength [5–7].

The thermomechanical process (TMP) is used to obtain an appropriate grain size of metallic materials. The main variables for the thermomechanical properties are strain rate, annealing temperature, and annealing time. Optimization of grain boundaries can be accomplished by two types of TMPs such as strain annealing and strain-recrystallization types. For the annealing process, low strain deformation (2–7%) and low annealing regime at lower temperature are used. The strain-recrystallization process uses low or middle strains (5–30%) with low tempering time in the high-temperature TM application [8–12]. At the end of the TMPs, the microstructure change mechanism is still unknown and can be explained by the grain boundary phenomenon which is effected by twins during deformation and annealing [13, 14]. Tokita et al. [15] reported evolution in grain boundary distribution characteristics of 304 austenitic stainless steel samples during the TMP. The separation of random boundaries between clusters was accomplished by forming annealing twins as a result of inhibiting growing clusters during the TMP. The frequency of random area lattice boundaries increased, and a reduction in the boundary filtering occurred.

In this study, the effects of TMPs on the grain size, tensile strength, and strain ratio of CuZn10 alloy were investigated.

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2 Experimental methods

The CuZn10 alloy (90 wt% Cu and 10 wt% Zn) was first melted using an induction furnace. The 200 mm-thick cast ingot was cut into 40 mm-thick plates. The plates were hot-rolled at a temperature of 650 °C. Then, cold rolling was applied to the hot-rolled plate with a 50% reduction. The medium size grain samples were obtained by annealing at 580 °C, and these were defined as the base sample (BS). For the TMP, the BSs were cold-rolled at various reduction ratios of 0.4, 0.8, 1.2, 2.4, 3.6, 5.2, 10, 13, 15, 17, and 21%, respectively. For each reduction ratio, two samples were cut from the cold-rolled sheets and then annealed in the high-temperature annealing (650 °C for 10 min) and low-temperature annealing (450 °C for 7 h) regimes, respectively, and cooled in water rapidly after annealing. The experimental procedure of the samples is given Table 1. The microstructures of the thermomechanically processed samples were determined using a scanning electron microscope (SEM: ZEISS EVO LS10). The relationships between strength, reduction ratio, annealing temperatures, aging time, and strain rates were obtained. Microhardness tests were carried out using a QNESS Q10 M Brinell test device with a 10 s dwell time and 50 g test load at intervals of 0.5 mm. The activation energy and optimized thermomechanical parameters were determined. Tensile tests were performed for the samples obtained at strain rates of $\dot{\varepsilon} = 0.01\,s^{-1}$ and $\dot{\varepsilon} = 0.1\,s^{-1}$ by an Instron tensile test machine. The typical fractographs of samples after tensile tests were examined by SEM.

Table 1: Experimental procedure of the samples.

Sample number	Annealing temperature (°C)	Aging time (h)	$\dot{\varepsilon} = 0.01\,s^{-1}$ reduction ratio %	$\dot{\varepsilon} = 0.1\,s^{-1}$ reduction ratio %
1	450 °C	7 h	0	0
2	650 °C	10 min	0	0
3	450 °C	7 h	0.4	0.4
4	650 °C	10 min	0.4	0.4
5	450 °C	7 h	0.8	0.8
6	650 °C	10 min	0.8	0.8
7	450 °C	7 h	1.2	1.2
8	650 °C	10 min	1.2	1.2
9	450 °C	7 h	2.4	2.4
10	650 °C	10 min	2.4	2.4
11	450 °C	7 h	3.6	3.6
12	650 °C	10 min	3.6	3.6
13	450 °C	7 h	5.2	5.2
14	650 °C	10 min	5.2	5.2
15	450 °C	7 h	10	10
16	650 °C	10 min	10	10
17	450 °C	7 h	13	13
18	650 °C	10 min	13	13
19	450 °C	7 h	15	15
20	650 °C	10 min	15	15
21	450 °C	7 h	17	17
22	650 °C	10 min	17	17
23	450 °C	7 h	21	21
24	650 °C	10 min	21	21

3 Results and discussion

The microstructure of the BS is presented in Figure 1. The microstructure of the BS consists of grains with a medium size of approximately 20 µm. Annealing twins were determined in the parent sample. The shape and growth rate of the annealing twins together with stacking faults have a significant impact on the regeneration of low-angle grain boundaries [13, 14]. The effect of reduction ratio on the grain size for different annealing regimes are shown in Figure 2.

The grain size of the thermomechanically treated samples increased with a reduction ratio of 5% for low-temperature annealing and 10% for high-temperature annealing regimes. The increase in cold rolling reduction ratio over 10% decreased the grain size from 25 to 10 µm for low-temperature annealing (Figure 2). The most effective peak values were determined as ≅10% cold rolling reduction for the high-temperature annealing regime and ≅5% for the low-temperature annealing regime of the thermomechanically treated CuZn10 alloy. For each strain rate, the grain size of the samples formed by the low-temperature annealing regime of 450 °C for 7 h was higher than that of

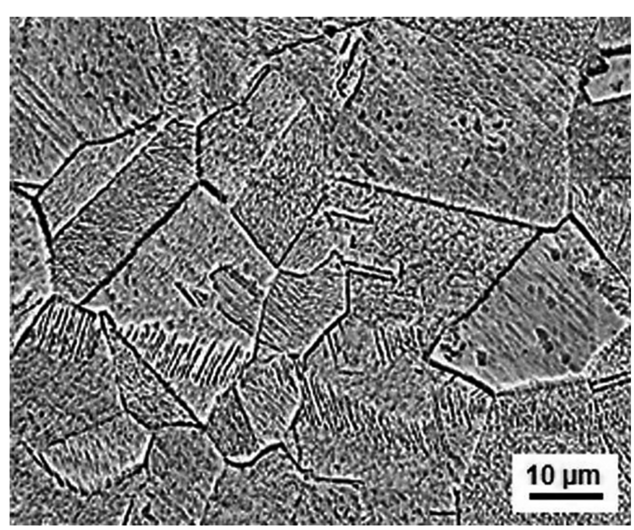

Figure 1: Microstructure of the BS.

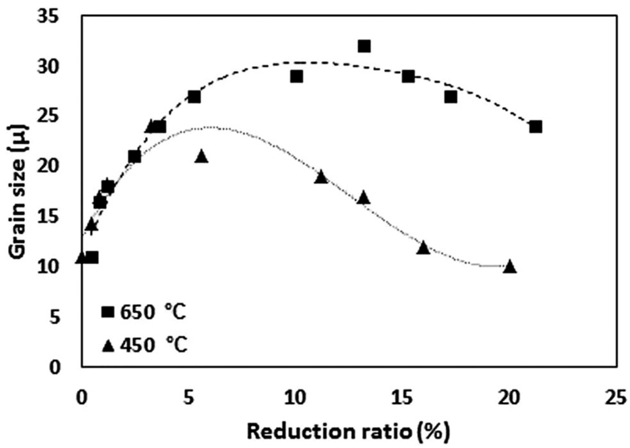

Figure 2: Effect of reduction ratio on the grain size for different annealing regimes.

the high-temperature annealing regime of 650 °C for 10 min. The effect of the annealing regime on the grain size was due to atomic diffusion and grain boundary migration [8–10]. The grain size of samples with TMP rised during 5–10% reduction and later declined until 20% reduction. The micrographs of TMP samples are shown in Figure 3. Low cold rolling samples exhibited microstructures with coarser grain sizes than the BS and other samples. It was observed from the micrographs that a 10% reduction ratio of the cold-rolled samples have approximately 20–25 µm grain size and a high-angle connection microstructure (Figure 3). The microstructure of the samples exhibited a finer grain size (10–15 µm) after the cold rolling reduction ratio was increased to more than 10%. For the effective grain size of the CuZn10 alloy, the thermomechanical

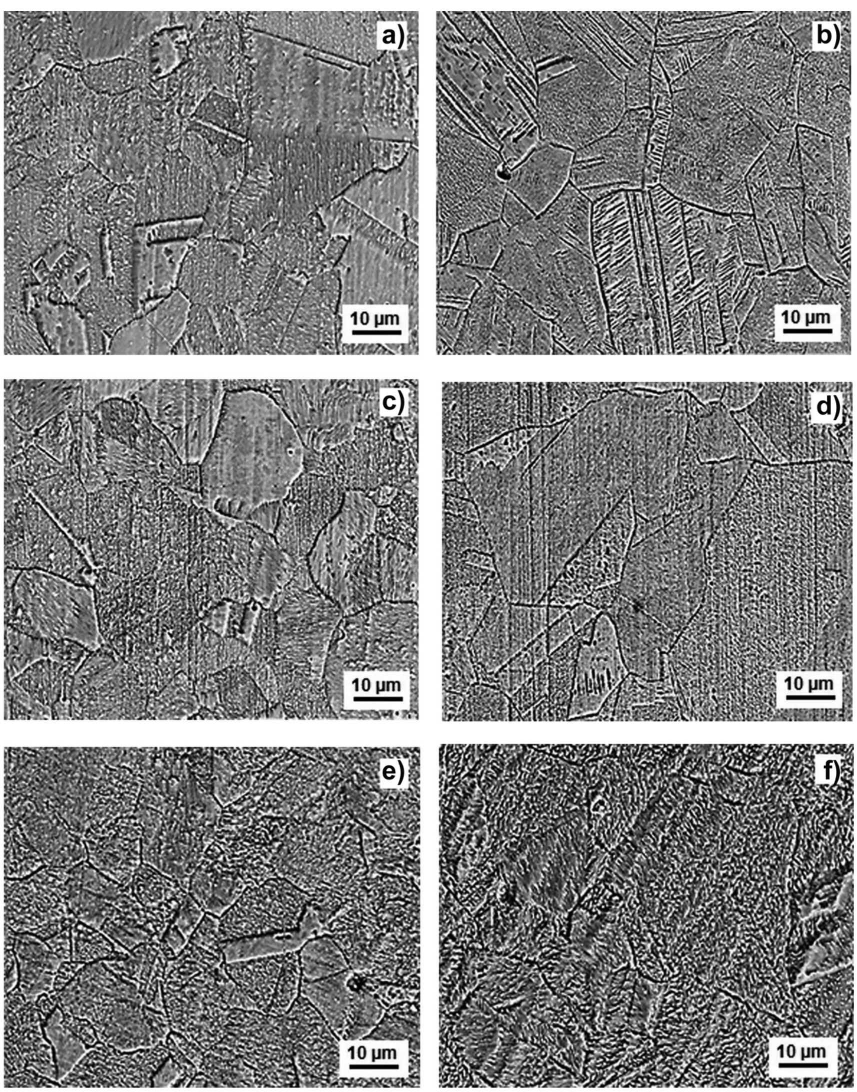

Figure 3: SEM micrographs of thermomechanically treated samples at a strain rate of 0.01 s^{-1}, a) sample 7, 450 °C, 7 h, b) sample 8, 650 °C, 10 min, c) sample 13, 450 °C, 7 h, d) sample 14, 650 °C, 10 min, e) sample 21, 450 °C, 7 h, f) sample 22, 650 °C, 10 min.

practice showed that the optimal deformation condition was 5–10% cold rolling reduction at low- and high-temperature annealing regimes.

Tensile experiments were carried out on the BS and TMP sample for the strain rates $\dot{\varepsilon} = 0.01\,s^{-1}$ and $\dot{\varepsilon} = 0.1\,s^{-1}$. Figure 4 shows the difference between the tensile stress and the reduction ratio of the thermomechanically treated samples under different strain rates. It was observed that the low-temperature annealing regime was quite effective for increasing the tensile strength under a high strain rate ($\dot{\varepsilon} = 0.01\,s^{-1}$). The high-temperature annealing regime showed a sharp increase in the tensile strength under a high strain rate (Figure 4a). The relationship between the tensile strength and the strain rate of the samples was also compared for a low strain rate (Figure 4b). The TMP increased the tensile strength of the samples for a strain rate of $\dot{\varepsilon} = 0.1\,s^{-1}$ up to 290 MPa for the low-temperature annealing regime. Reducing the strain rate to $\varepsilon = 0.01\ s^{-1}$ decreased the effect of TMP on the tensile strength of the samples. There was a close correlation between the grain size and the strain rate.

The relationships among grain size, hardness, and reduction ratio for a strain rate of $\dot{\varepsilon} = 0.01\,s^{-1}$ and $\dot{\varepsilon} = 0.1\,s^{-1}$ are shown in Figure 5. The increase in the strain rate increased the grain size and hardness at all reduction ratios of the TMP. The strength of the thermomechanically processed samples depends on the cold rolling reduction ratio and the strain rate.

The relationship among the strength of samples, cold rolling temperature, and strain rate can be defined by Equations (1) and (2) as follows [15]:

$$\dot{\varepsilon} = A\left[\sinh(\alpha\sigma)\right]^{n}\sigma^{n1}\exp\left[-Q\cdot RT^{-1}\right] \tag{1}$$

$$D_Q = \dot{\varepsilon}\exp\left[Q\cdot RT^{-1}\right] \tag{2}$$

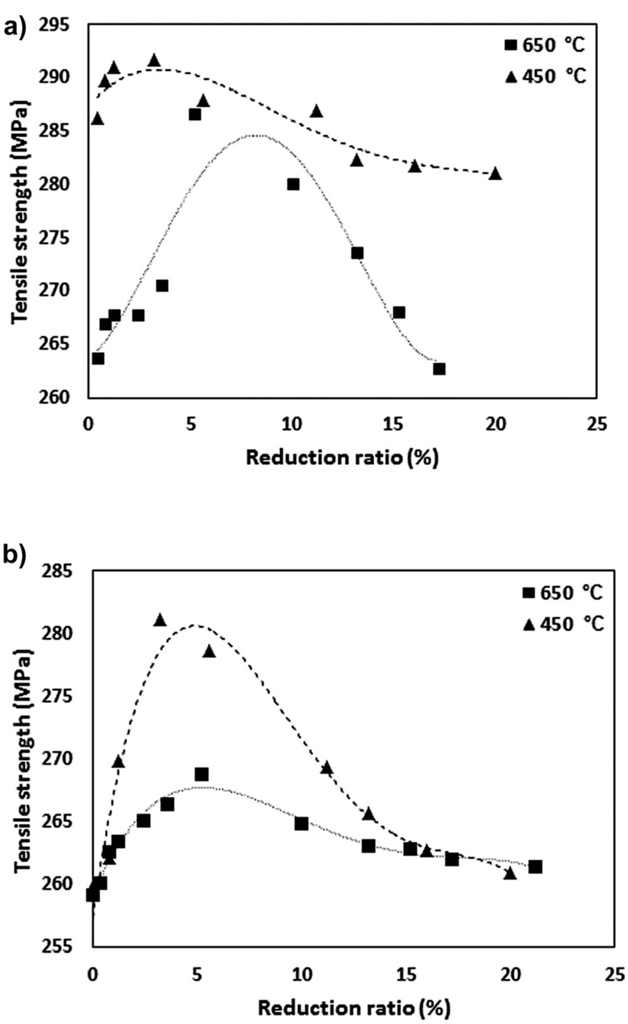

Figure 4: Tensile strength versus reduction ratio of thermomechanically treated samples, a) $\dot{\varepsilon} = 0.1\,s^{-1}$, b) $\dot{\varepsilon} = 0.01\,s^{-1}$.

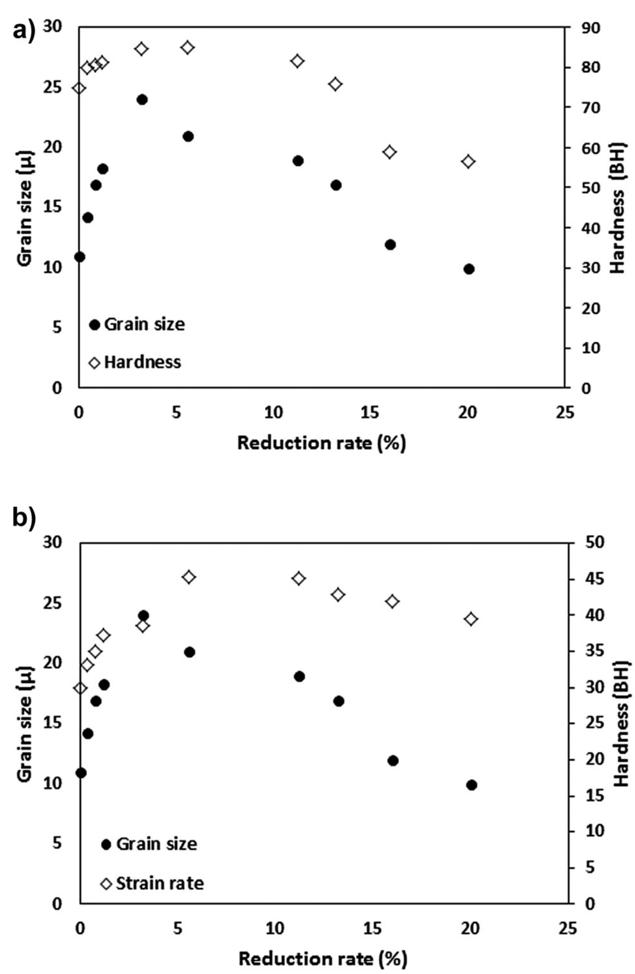

Figure 5: Comparison of the grain size of low-temperature annealed samples with brinell hardness and reduction rate (%), a) $\dot{\varepsilon} = 0.1\,s^{-1}$, b) $\dot{\varepsilon} = 0.01\,s^{-1}$.

D_Q relies on the lattice diffusion activation energy due to dislocation slip and escalation. σ is the strength (MPa); $\dot{\varepsilon}$ is the strain rate (s^{-1}); T is the temperature (K); Q represents the thermal activation energy (J mol^{-1}); A, α, n1, and n are the material constants; and R is the universal gas constant (8.314 J mol^{-1} K^{-1}). The activation energy of cold deformation could be calculated for different strain rates of the CuZn10 alloy. The activation energy of cold deformation for sample S13, which was deformed at a strain rate of $\dot{\varepsilon}$ = 0.01 s^{-1}, was calculated to be Q = 223.3 kJ mol^{-1}. The activation energy of cold deformation for sample S13 for a strain rate of $\dot{\varepsilon}$ = 0.1 s^{-1} was calculated to be Q = 340.6 kJ mol^{-1}. This result showed that the activation energy of deformation for CuZn10 alloy was significantly dependent on the strain rate.

The increase in activation energy was mainly due to the increase in twins at higher strain rates. The formation of twins and dislocations made cold rolling deformation difficult, and the activation energy of cold forming increased [15, 16]. The annealing process activated the recovery mechanism. The grain boundaries are rearranged by the dislocation motion, which affects the increase in the deformation activation energy [17]. The thermomechanically processed microstructure of the samples treated at different strain rates showed that the higher temperature annealing regime and high strain rate (ε = 0.1 s^{-1}) provided sufficient driving force for atomic diffusion.

This ensured grain boundary migration for recrystallization, which completely enlarged the grains and increased the tensile strength. The TMP showed that the subgrains were formed after cold rolling for a high strain rate, and grains were continuously distributed, especially during the low-temperature annealing regime.

The dislocation intensity increased during cold rolling. It dispersed by sliding along low-angle grain boundaries. Metals with FCC atomic structures have a high degree of symmetry with many slip systems. A slip was the main deformation mechanism during cold rolling. However, twinning could be proceeded under high strain rate cold-forming conditions [15]. Twinning was mainly dependent on the stacking fault energy. The intensity of twin formation during cold rolling had an impact on the formation of subgrains having high-angle grain boundaries during annealing [17–19]. Besides the grain size, low-angle grain boundaries were suitable for the slip. Therefore, plastic deformation occurred more easily. High-angle grain boundaries were unfavorable for dislocation movement. The formation of twins facilitated new sliding systems so that the dislocation could continue. During annealing, the favorable nucleation sites of the subgrains were mainly provided with high-angle grain boundaries of 10–15° [19, 20]. It was determined that the dislocation density of the thermomechanically treated samples for the annealing regime of 450 °C for 7 h was higher than 650 °C for 10 min. Furthermore, many subgrains with high strain rates occurred in these samples. This also showed that the low strain rate and high temperature delayed recrystallization. Figure 3b shows the microstructure of the thermomechanically processed

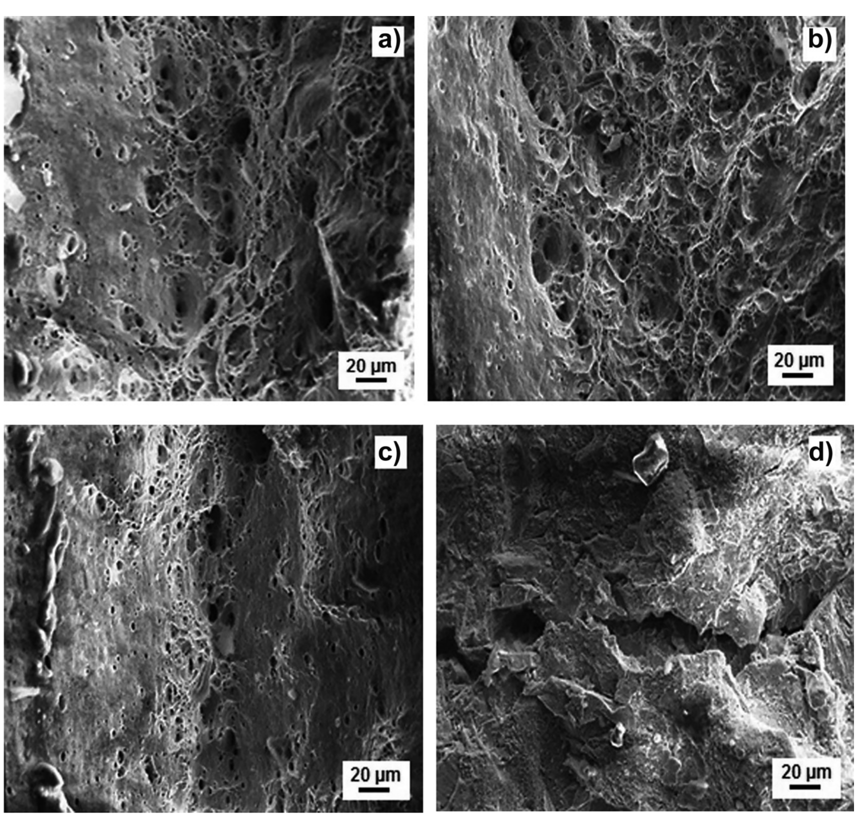

Figure 6: Fracture surface SEM micrographs of a) BS, b) S3, c) S7, d) S9 samples.

samples at 650 °C and 0.01 s^{-1}. As the annealing temperature increased, the dislocation density increased at grain boundaries or subgrain boundaries. During the TMP, the number of subgrains increased in the particles, and these were continuously collected in Figure 3a. This led to the thinning of the grains. Over time, dislocation intensity decreased. The movement of subgrain borders would be blocked by preventing the dislocation movement. Therefore, twinning occurred at high-angle grain boundaries, which may be due to the higher strain rate important for the formation and movement of dislocations. The tensile strength obtained for the low-temperature annealing regime was found to be significantly higher than the tensile strength obtained for the high-temperature annealing regime. It was thought that basically the intensity of the twins affected the tensile strength by reducing the path of dislocations, thus promoting hardening [20]. The typical fractographs of the BS, S3, S7, and S9 samples after tensile tests are shown in Figure 6. The fracture surface of the BS was predominantly cleavage, and the surfaces were very wide (Figure 6a). The fractured surfaces of samples S3, S7, and S9 in Figure 6b–d exhibited the mixed fracture mode of ductile fracture throughout the matrix. Fractures occurred in the form of crack initiation and propagation, and partially rapid fracture. The low tensile strength of the BS was due to large grain sizes. The presence of subgrains increased tension. Therefore, crack initiation and propagation easily occurred as a smooth surface. Thus, less energy was absorbed for the failure of the samples, resulting in a decrease in the tensile strength of the BS compared to the thermomechanically treated samples. Sample S7 exhibited the highest impact resistance value due to the smallest grain size.

4 Conclusions

The low-temperature annealing regime (450 °C for 7 h) yielded better results for the effective grain size of the CuZn10 alloy.

Increased tensile strength (290 MPa) was achieved by the low-temperature annealing regime.

The best reduction ratio for the formation of twins during cold rolling of the TMP was determined as 5–10%.

The increase in the strain rate ($\dot{\varepsilon} = 0.01\,s^{-1} \rightarrow \dot{\varepsilon} = 0.1\,s^{-1}$) increased the grain size and hardness for all reduction ratios of the TMP.

The optimal deformation conditions for grain boundary optimization were determined as a strain rate of 0.01 s^{-1} and cold rolling reduction of <10%.

The presence of subgrains increased the tensile strength.

The strain rate had a major effect on the TMP to increase the tensile strength.

The activation energy of the cold-rolled samples was increased by strain rates.

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References

[1] S. Nestorovic, D. Markovic, and I. Markovic, “Influence of thermal cycling treatment on the anneal hardening effect of Cu-10Zn alloy,” *J. Alloys Compd.*, vol. 489, no. 2, pp. 582–585, 2010, https://doi.org/10.1016/j.jallcom.2009.09.115.
[2] W. Chen, M. Wang, Z. Li et al., “A novel Cu-10Zn-1.5Ni-0.34Si alloy with excellent mechanical property through precipitation hardening,” *J. Mater. Eng. Perform.*, vol. 25, pp. 4624–4630, 2016, https://doi.org/10.1007/s11665-016-2354-3.
[3] S. Zhang, H. Zhu, Z. Hu, X. Zeng, and F. Zhong, “Selective laser melting of Cu-10Zn alloy powder using high laser power,” *Powder Technol.*, vol. 342, pp. 613–620, 2019, https://doi.org/10.1016/j.powtec.2018.10.002.
[4] Q. Liu and L. Cheng, “Structural evolution and electronic properties of Cu-Zn alloy clusters,” *J. Alloys Compd.*, vol. 771, pp. 762–768, 2019, https://doi.org/10.1016/j.jallcom.2018.08.033.
[5] L. Cai, X. Jiang, Y. Guo et al., “Friction and wear performance of a copper-based bond emery wheel for rail grinding,” *Mater. Test.*, vol. 62, no. 12, pp. 1205–1214, 2020, https://doi.org/10.3139/120.111594.
[6] T. Watanabe, S. Tsurekawa, X. Zhao, L. Zuo, and C. Esling, “A new challenge: grain boundary engineering for advanced materials by magnetic field application,” *J. Mater. Sci.*, vol. 41, pp. 7747–7759, 2006, https://doi.org/10.1007/s10853-006-0740-1.
[7] A. Das, “Grain boundary engineering: fatigue fracture,” *Phil. Mag.*, vol. 97, no. 11, pp. 867–916, 2017, https://doi.org/10.1080/14786435.2017.1285072.
[8] A. Telang, A. S. Gill, M. Kumar et al., “Iterative thermomechanical processing of alloy 600 for improved resistance to corrosion and stress corrosion cracking,” *Acta Mater.*, vol. 113, pp. 180–193, 2016, https://doi.org/10.1016/j.actamat.2016.05.009.
[9] C. K. Kaithwas, P. Bhuyan, S. K. Pradhan, and S. Mandal, “Microstructure evolution during low-strain thermo-mechanical processing and its repercussion on intergranular corrosion in

alloy 600H," *Mater. Charact.*, vol. 145, pp. 582–593, 2018, https://doi.org/10.1016/j.matchar.2018.09.019.
[10] D. L. Engelberg, R. C. Newman, and T. J. Marrow, "Effect of thermomechanical process history on grain boundary control in an austenitic stainless steel," *Scr. Mater.*, vol. 59, no. 5, pp. 554–557, 2008, https://doi.org/10.1016/j.scriptamat.2008.05.012.
[11] A. Telang, A. S. Gill, K. Zweiacker, C. Liu, J. M. Wiezorek, and V. K. Vasudevan, "Effect of thermo-mechanical processing on sensitization and corrosion in alloy 600 studied by SEM-and TEM-based diffraction and orientation imaging techniques," *J. Nucl. Mater.*, vol. 505, pp. 276–288, 2018, https://doi.org/10.1016/j.jnucmat.2017.07.053.
[12] M. Kumar, W. E. King, and A. J. Schwartz, "Modifications to the microstructural topology in f.c.c. materials through thermomechanical processing," *Acta Mater.*, vol. 48, no. 9, pp. 2081–2091, 2000, https://doi.org/10.1016/S1359-6454(00)00045-8.
[13] C. Cayron, "Quantification of multiple twinning in face centred cubic materials," *Acta Mater.*, vol. 59, no. 1, pp. 252–262, 2011, https://doi.org/10.1016/j.actamat.2010.09.029.
[14] V. Randle, "Twinning-related grain boundary engineering," *Acta Mater.*, vol. 52, no. 14, pp. 4067–4081, 2004, https://doi.org/10.1016/j.actamat.2004.05.031.
[15] S. Tokita, H. Kokawa, Y. S. Sato, and H. T. Fujii, "In situ EBSD observation of grain boundary character distribution evolution during thermomechanical process used for grain boundary engineering of 304 austenitic stainless steel," *Mater. Charact.*, vol. 131, pp. 31–38, 2017, https://doi.org/10.1016/j.matchar.2017.06.032.
[16] M. Kumar, A. J. Schwartz, W. E. King, "Microstructural evolution during grain boundary engineering of low to medium stacking fault energy fcc materials," *Acta Mater.*, vol. 50, no. 10, pp. 2599–2612, 2002, https://doi.org/10.1016/S1359-6454(02)00090-3.
[17] C. Zener, J. H. Hollomon, "Effect of strain-rate upon the plastic flow of steel," *J. Appl. Phys.*, vol. 15, no. 1, pp. 22–27, 1944, https://doi.org/10.1063/1.1707363.
[18] C. A. Schuh, M. Kumar, W. E. King, "Analysis of grain boundary networks and their evolution during grain boundary engineering," *Acta Mater.*, vol. 51, no. 3, pp. 687–700, 2003, https://doi.org/10.1016/S1359-6454(02)00447-0.
[19] V. Randle and M. Coleman, "A study of low-strain and medium-strain grain boundary engineering," *Acta Mater.*, vol. 57, no. 11, pp. 3410–3421, 2009, https://doi.org/10.1016/j.actamat.2009.04.002.
[20] M. Shimada, H. Kokawa, Z. J. Wang, Y. S. Sato, I. Karibe, "Optimization of grain boundary character distribution for intergranular corrosion resistant 304 stainless steel by twin-induced grain boundary engineering," *Acta Mater.*, vol. 50, no. 9, pp. 2331–2341, 2002, https://doi.org/10.1016/S1359-6454(02)00064-2.

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