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EFFECT OF RECRYSTALLIZATION ANNEALING AND AGING TREATMENT ON MICROSTRUCTURE AND PROPERTIES OF HIGH STRAIN ROLLED Cu-Cr-Zr ALLOY

To obtain high strength and high electrical conductivity, precipitation-strengthened Cu-Cr-Zr alloys are treated by heat treatment and mechanical treatment. The effects of rolling temperature, recrystallization, and aging treatment on the microstructure evolution, mechanical properties, and electrical conductivity of Cu-Cr-Zr alloy were investigated in detail. The results indicated that the optimal comprehensive performance of tensile strength (595.9 MPa), percentage elongation (14.35%), hardness 202.60 HV, and electric conductivity (77.1% IACS) was obtained by cold rolling and aging at 450°C for 1 hour. It was found that the precipitates inhibit the formation of recrystallized grains. The improvement of tensile strength, hardness, and conductivity of the Cu-Cr-Zr alloy is mainly attributed to aging strengthening, and the advancement of elongation is primarily attributed to recrystallization. Mechanical properties and electrical conductivity of the alloy after aging treatment are improved, indicating that proper aging treatment is helpful in obtaining better properties of Cu-Cr-Zr alloy.

Keywords: Cu-Cr-Zr alloy; Recrystallization; Aging treatment; Microstructure; Mechanical properties; Electric conductivity

1. Introduction

In recent years, Cu-Cr-Zr alloys have been widely used in the electronic field due to their high strength and electrical conductivity [1-4]. The electronic package lead frame is one of the main parts of the integrated circuit. The role of the lead frame is to provide a channel for the electronic signals between the device and the circuit, as well as a fixture on the circuit board. Therefore, lead frame alloys are required to have high strength and conductivity. Up to now, the lead frame materials are dominated by Cu-Fe-P alloy [5,6] and Cu-Ni-Si alloy [7,8], which cannot meet the development needs. The Cu-Cr-Zr alloy has emerged as a focal point of research within the realm of copper alloys, owing to its exceptional combination of high strength and conductivity. Therefore, Cu-Cr-Zr alloy is the most promising candidate material for lead frames of integrated circuits [9-13].

Several studies have investigated the impact of heat treatment on the microstructure and properties of Cu-Cr-Zr alloys [14,15]. In addition, researchers have implemented innovative techniques to produce high-performance Cu-Cr-Zr alloys, including equal channel angular pressing (ECAP), rotary die forging,

and aging treatment following cryogenic rolling [16-18]. Pang et al. [19] studied 80% cold rolled Cu-Cr-Zr alloy aged at 450°C for 6 h, with a hardness of 175HV and an electrical conductivity of 81.1%IACS. Zhang et al. [20] obtained the desired combination of the tensile strength (690.13 MPa) and electrical conductivity (67% IACS) for Cu-1Cr-0.1Zr alloy by using Two passes of cry rolling and intermediate aging treatments. Huang et al. found that [17] Cu-Cr-Zr alloy with an ultimate tensile strength of 612 MPa, uniform elongation of 5%, and electrical conductivity of 84.7% was obtained by enhanced rotary forging and two-step peak aging. Fine grain strengthening, precipitation strengthening, and dislocation density strengthening can improve the strength, and aging treatment can improve the conductivity. In conclusion, remarkable achievements have been made in improving the strength and conductivity of Cu-Cr-Zr alloys. These studies offer fresh insights into the efficient fabrication of high-performance Cu-Cr-Zr alloys. Compared with equal channel Angle extrusion and ultra-low temperature forging, the thermal-mechanical treatment based on rolling aging is more easily used for the industrial production of Cu-Cr-Zr alloys. Therefore, clarifying the effect of rolling conditions and aging treatment on the microstructure

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and properties of Cu-Cr-Zr alloy is of great significance for the reasonable selection of process parameters and effective control of alloy properties.

This research investigated the impact of rolling temperature, short-time recrystallization, and aging treatment on the microstructure evolution, electrical conductivity, and mechanical properties of Cu-Cr-Zr alloys. The relationship among the process, microstructure, and mechanical properties was established by analyzing the relevant influence mechanism.

2. Experimental procedures

2.1. Material and process design

Alloy plates with a chemical composition of Cu-1.0Cr-0.5Zr-0.15Mg-0.05Si-0.2Al (wt.%) were solution-treated at 960°C for 1 h, followed by rapid water cooling. Subsequently, the alloys were cold rolled at room temperature with a rolling rate of 90%. After rolling, the alloys were subjected to short recrystallisation anneals of 0 min, 40 sec and 3 min, respectively, followed by ageing at 450°C for 1 h. The alloys were then subjected to a short recrystallisation anneal of 0 min, 40 sec and 3 min, respectively. In order to investigate the effect of rolling temperature on the Cu alloy, the sheets were hot rolled and then cold rolled at 780°C as shown in Fig. 1. The Cu-Cr-Zr alloy sheets were initially hot rolled at 780°C with a 60% reduction in thickness and then cold rolled at room temperature to a final thickness of 1 mm. After rolling, the Cu alloys were subjected to short recrystallisation anneals of 0 min, 40 sec and 3 min, respectively, followed by aging at 450°C for one hour.

2.2. Microstructure and properties characterization

According to the ASTM E8/E8M size standard, tensile samples with a rectangular cross-section of 1 mm × 8 mm and a gauge length of 20 mm were processed from the cold rolled sheet parallel to the rolling direction. The tensile test was carried out on the

electronic universal testing machine (WDW-20) to test the strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. A Sigma 2008 digital eddy current metal conductance meter was used to measure the conductivity of samples with dimensions of 30 mm (RD) × 20 mm (TD) × 1 mm (ND) and the sample surface (RD-TD) must be polished to a smooth finish. The conductivity value can be read on the instrument screen by placing the metal probe of the conductivity meter vertically flat on the sample surface (RD-TD) for at least 2 seconds. Vickers microhardness measurement was carried out on the rolling direction-normal direction (RD-ND) plane under an applied load of 50 g and a holding time of 15 s by the FM-700 Vickers microhardness tester. To reduce the potential for error, each sample was tested at least five times and the resulting values averaged. Tensile tests and conductivity measurements were conducted at room temperature.

3. Results and discussion

3.1. Effect of rolling temperature and heat treatment on microstructure evolution

Fig. 2 shows the microstructure of the recrystallized Cu-1.0Cr-0.5Zr alloy at 800°C for different durations. Fig. 2a-c represent the microstructure of the samples at 780°C Hot/Cold Rolling + Recrystallization 0min, the 40s, 3min, and Fig. 2d-f represent the microstructure of the samples at Cold Rolling + Recrystallization 0 min, 40 s, 3 min. The two distinct processing routes result in the formation of a deformed microstructure, characterized by the elongation of grains in the rolling direction. At the same time, many high-density dislocations and sub-grains are formed. In addition, some micron Cu grains, which include a grayish-white contrast during the rolling process, elongated along the rolling direction (Marked by a white dashed ellipse in Fig. 2a-f). Following the rolling treatment, the grains exhibit a range of shapes and sizes. The overall Cu grains display a striped pattern, with the majority of grains exhibiting an elongated morphology. The magnified microstructure is presented in Fig. 2c and f for reference. In the Cu-Cr-Zr alloy, the

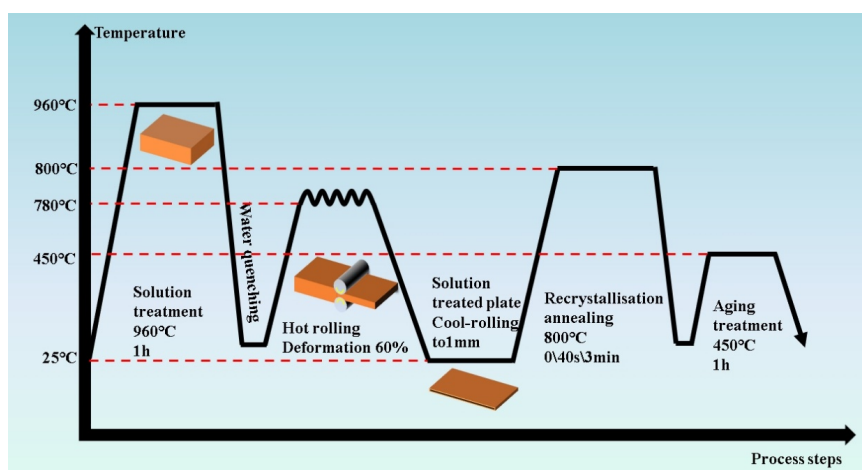


Fig. 1. Schematic illustration of thermo-mechanical treatment routes for Cu-1.0Cr-0.5Zr alloy plates

predominant precipitate is the Cr phase. (Ref 13, 21, 22, 23). According to Fig. 2a-f, the rolled samples are composed of a Cu matrix and light gray granular Cr precipitates. The precipitates are uniformly distributed on the Cu matrix. Microscale precipitates were observed in the rolled samples and identified as the Cr phase by EDS analysis, as shown in the enlarged image in Fig. 2e. Following hot rolling, a notable increase was observed in both the quantity and size of the precipitated phases (Fig. 2a-c). In contrast, the cold-rolled alloy displayed a minimal amount of Cr precipitate, which was uniformly dispersed within the Cu matrix (Fig. 2d-e). Following a short thermal treatment, no significant changes were observed in the size or volume fraction of the precipitated phases. The volume fraction of the precipitated phase increases after hot rolling at 780°C, and the change in volume fraction is not obvious after short heat treatment. However, the alloy was cold-rolled at 25°C and then heat-treated. It was found that the amount of precipitated phase was few, and the size was small. Furthermore, the recovery and recrystallisation of the Cu-1.0Cr-0.5Zr alloy result in a reduction in hardness.

After aging at 450°C for 1 hour, fine black precipitates (Cu₃Zr) were found distributed along the grain boundaries of the alloy, accompanied by observations of incomplete recrystal-

lization grain formation. During the aging process, a significant portion of dislocations was recovered, and numerous precipitated phases were formed, resulting in an increase in the hardness of the alloy [15,24,25]. EDS analysis showed that the mass percentages of Cu and Zr are 69.47% and 30.43%, respectively, as shown in the enlarged image in Fig. 3e. The recrystallization temperature of Cu-Cr-Zr alloy ranges from 700°C to 900°C. In the study of this experiment, recrystallization grain formation was found only after aging at 450°C for 1 h. The recrystallization process of the alloy was delayed. When aging at 450°C, the precipitates occur before recrystallization, indicating that the precipitates strongly inhibit the recrystallization of Cu-1.0Cr-0.5Zr alloy.

3.2. Effect of rolling temperature and heat treatment on mechanical properties

Fig. 4 illustrates a notable increase in the hardness of the samples following a period of ageing at 450°C for one hour. The alloy exhibits a hardness of 163.53 HV after cold rolling at 25°C, which increases to 202.60 HV after aging at 450°C for 1 hour. Solid solution-treated alloys were hot-rolled at 780°C

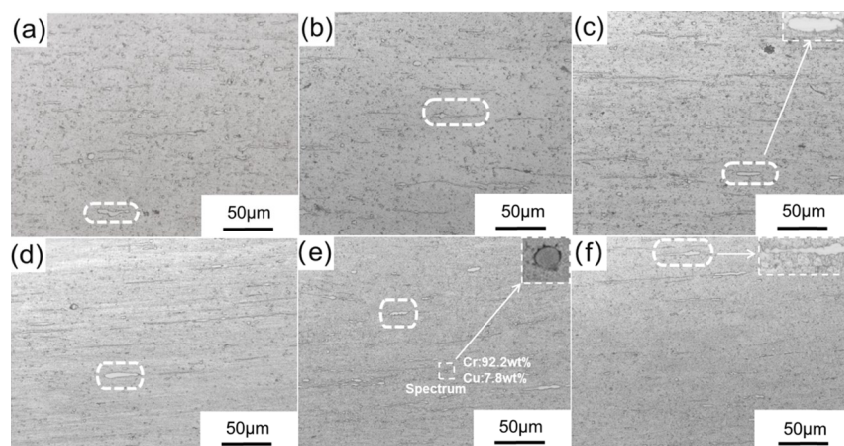


Fig. 2. Microstructure of Cu-1.0Cr-0.5Zr alloy rolled band after recrystallization annealing: (a)-(c) 780°C H/CR + Re 0 min, 40 s, 3 min; (d)-(f) 960°C St + CR + Re 0 min, 40 s, 3 min

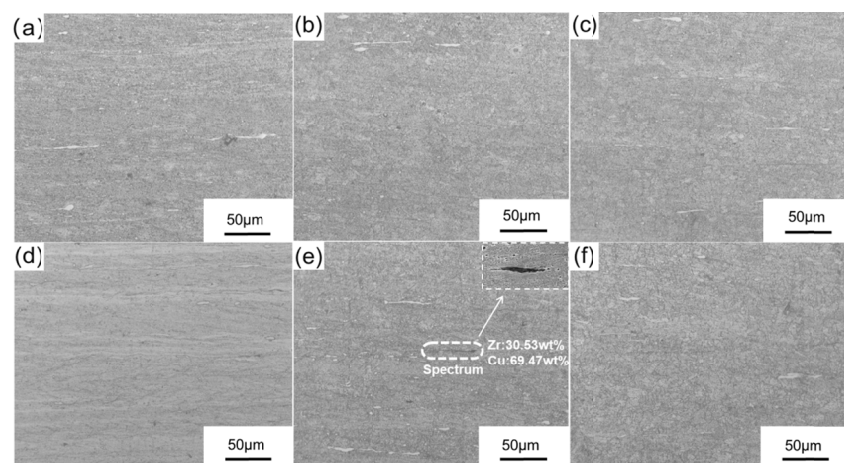


Fig. 3. Microstructure of Cu-1.0Cr-0.5Zr alloy after aging treatment: (a)-(c) 780°C H/CR + Re 0 min, 40 s, 3 min + aging; (d)-(f) 960°C St + CR + Re 0 min, 40 s, 3 min + aging

and cold-rolled to 1 mm to get a hardness of 128.54 HV. After aging treatment, the hardness increased from 128.54 HV to 144.68 HV. This increase is due to the segregation of solute atoms in the Cu matrix, which are precipitated to form a strengthened phase. These precipitates interact with dislocations, hindering the movement of dislocations and leading to an increase in hardness [14,17]. The hardness of the samples cold-rolled at 25°C is reduced to 113.54 HV by 40 s recrystallization heat treatment. The hardness of the alloy, which was hot rolled at 780°C and then cold rolled, was decreased to 89.65 HV after 40 s heat treatment. It can be observed that the hardness of the Cu-1.0Cr-0.5Zr alloy decreases significantly after a brief recrystallization process, which is attributed to the decrease of dislocation density caused by recrystallization annealing and the incomplete recrystallization of deformed grains. Rolling increases the hardness of the sample with the cold rolling deformation because the matrix forms many dislocations, so the dislocation occurs to a certain extent of blockage, resulting in the formation of a dislocation distortion zone and dislocation wall.

The stress-strain curves of the alloys that have been subjected to different heat treatment processes are shown in Fig. 5. The elongation of the specimen following cold rolling at 25°C is only 6%. The elongation of the sample after 40 s short recrystallization treatments is 14.35%, and its tensile strength is 502.45 MPa. After aging at 450°C for 1 h, the tensile strength is 595.9 MPa (Fig. 6b). The stress-strain curve in Fig. 5c also reflects the improved mechanical properties of the alloy. The alloy, which was hot rolled at 780°C and then cold rolled, exhibits an elongation of 5.02% and a tensile strength of 650.29 MPa, after a 40-second heat treatment, the elongation increased to 9.14%, while the tensile strength decreased to 476.2 MPa. After

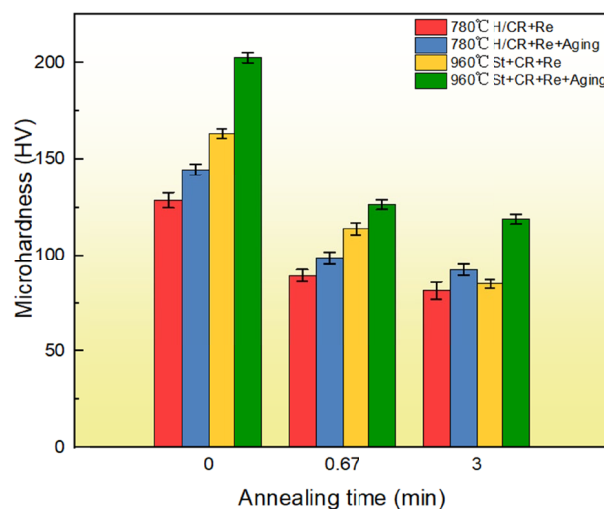


Fig. 4. The hardness of Cu-1.0Cr-0.5Zr alloy after recrystallization and aging treatment

the aging treatment, elongation and tensile strength increased to 12.39% and 525.98 MPa, respectively (Fig. 6a-b). This is attributed to the decomposition of the supersaturated solid solution during aging, as well as the pinning of dislocations and hindrance of grain boundary movement, which helps to improve tensile strength [26-28]. After recrystallization heat treatment for 3 min, the elongation of the alloy increased significantly and remained at about 35%, and the tensile strength also increased after aging treatment. When materials undergo severe plastic deformation, the dislocation density inside the alloy will continue to increase, leading to intensified mutual delivery during dislocation movement, resulting in dislocation entanglement and other obstacles, increasing the resistance during dislocation movement, and final-

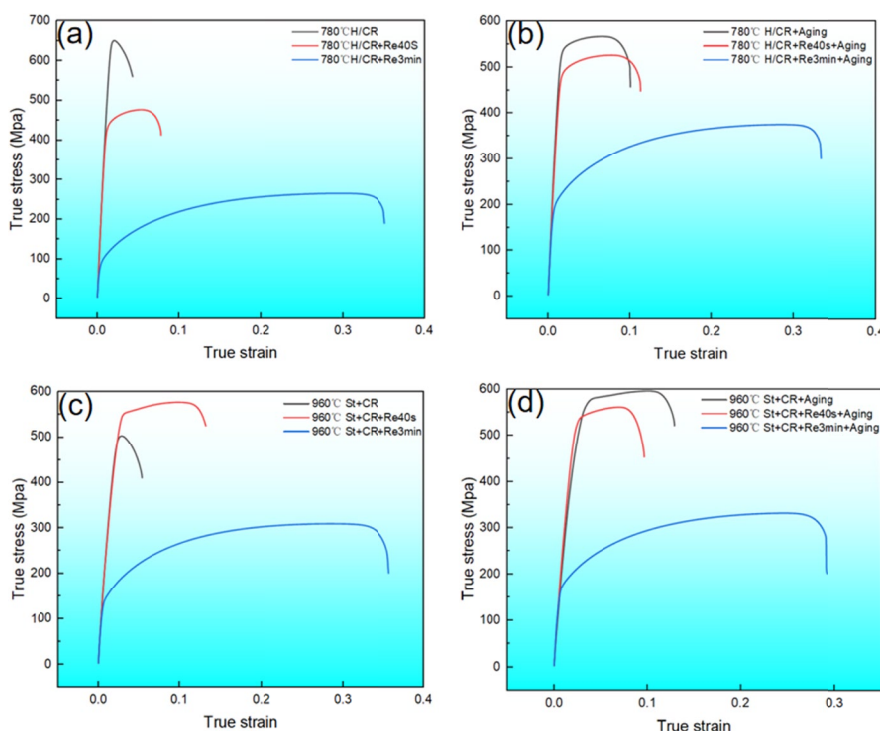


Fig. 5. Stress-strain curves of Cr-1.0Cr-0.5Zr alloys treated by different processes: (a) 780°C H/CR + Re 0 min, 40 s, 3 min; (b) 780°C H/CR + Re 0 min, 40 s, 3 min + aging; (c) 960°C St + CR + Re 0 min, 40 s, 3 min; (d) 960°C St + CR + Re 0 min, 40 s, 3 min + aging

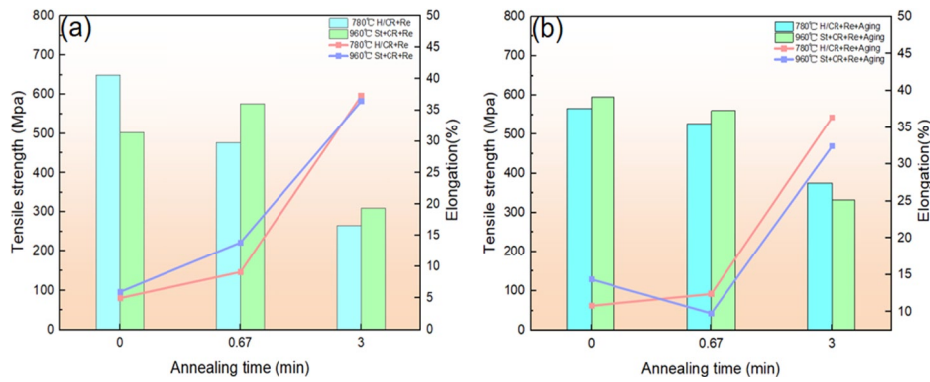


Fig. 6. Tensile strength and elongation of Cu-1.0Cr-0.5Zr alloy by different processes: (a) without aging; (b) aging. Bar graph–Tensile strength Line graph–Elongation

ly improving the strength of the alloy. After recrystallization heat treatment, dislocation motion was promoted, dislocation density was reduced, and the elongation of the alloy was increased. As annealing time increased, dislocation density decreased significantly, resulting in a substantial improvement in elongation.

3.3. Effect of rolling temperature and heat treatment on fracture morphology

Fig. 7 shows that the alloy undergoes a plastic fracture mechanism, characterized by severe plastic deformation and pronounced necking before fracture. There are many dimples in the fracture of the sample and larger dimples in the fracture of the sample after a short recrystallization heat treatment (Fig. 7a-b). With the increase of annealing time, the size and quantity of dimples increase, and the small number of large dimples in uneven

distribution on the cross-section. The macro fracture morphology of the alloy after cold rolling at 25°C is shown in Fig. 7d, with many shallow but small dimples and dense distribution in Fig. 7e, large dimples can be observed on the fracture surface. Fig. 7a shows the fracture morphology of the cold-rolled alloy after hot rolling at 780°C, with small dimples observed on the cross-section. This highlights the underlying mechanism explaining why the tensile strength of the latter is higher than that of the former (Fig. 5a-c). The macroscopic fracture of the alloy cold-rolled at 25°C after 3min heat treatments showed that there was a secondary crack in the core of the sample throughout the whole sample (Fig. 7g). As shown in Figs. 7h and 7i, the cracks are connected by dimples of varying sizes, which are enlarged in the central part of the image. New dimples appeared in the inner wall of the dimple, and the dimple characteristics changed gradually from tear type to equiaxed type (Fig. 7i). All the tensile fractures are dimple-shaped ductile fractures, which belong to

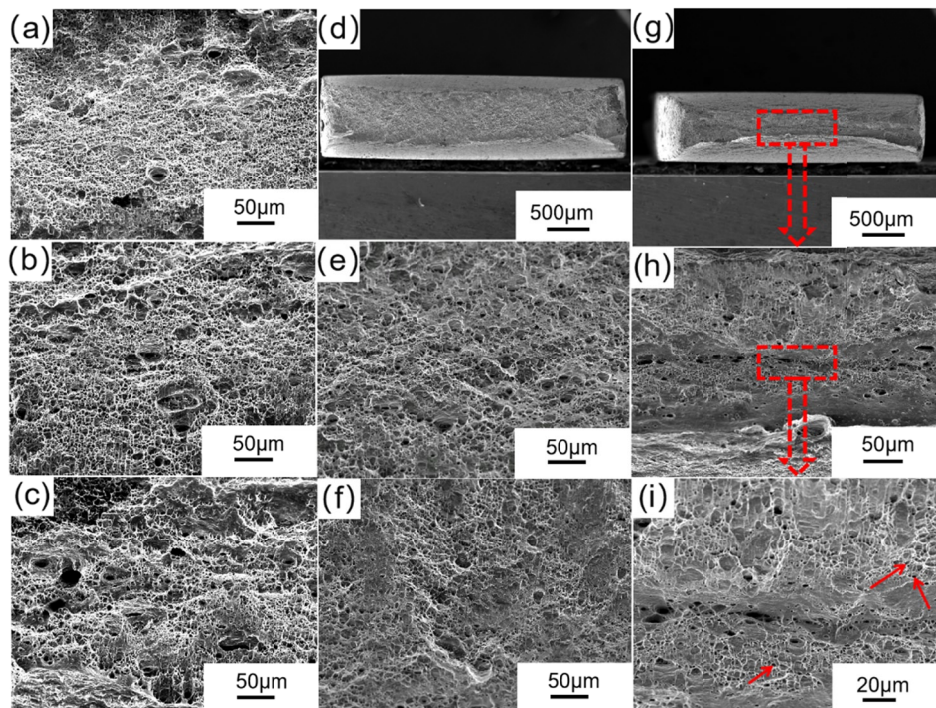


Fig. 7. The fracture morphology of Cu-1.0Cr-0.5Zr alloy after recrystallization: (a)-(c) 780°C H/CR + Re 0 min, 40 s, 3 min; (e),(f),(h) 960°C St + CR + Re 0 min, 40 s, 3 min; d is the macro fracture morphology of e

the typical micropore aggregation type. The bonding strength between the precipitated phases and the matrix in the alloy is exceptionally high, which delays the nucleation and growth of micro-pores until after necking. The fracture mechanism of Cu-Cr-Zr alloy is mainly that the material necked first, the cavity formed at the second phase particle grew, expanded, and polymerized, and joined with each other, resulting in fracture. The final fracture region is the dimple region.

Fig. 8 illustrates the fracture morphology after aging. In the macroscopic fracture, secondary cracks were observed in the core of the sample, connected by dimples of different sizes, and showed obvious necking characteristics. Microscopic fracture analysis reveals that the fracture originated from the central coarse-grained region, with a direction nearly perpendicular to the tensile axis. The main microscopic features include intergranular secondary cracks, surrounded by shallow, fine dimples. The secondary crack partitions the section into two distinct regions, resulting in the formation of two separate free surfaces within the sample. Several discrete fine cracks parallel to the secondary crack are also observed (Fig. 8a-c Solid red arrows). The microscopic fracture analysis reveals that the elongated crack initiated along the grain boundary, forming two shear planes with small parabolic dimples distributed across the surface (Fig. 8f, red box). Dimples of different sizes are present on the cross-section. A serpentine slip line is observed on one side wall of the large dimples, indicative of an intergranular or delaminated fracture, followed by zonal tearing. Compared to the alloy without cracks, the tensile strength and elongation in the stress-strain curve shown in Fig. 5d are higher than those in Fig. 5b. This indicates that the secondary crack prevents the increase of tensile strength and elongation of the alloy. Fig. 8g-i show the microscopic fracture morphology of cold-rolled alloy after aging. It can be observed that there are

large dimples distributed unevenly on the fracture. After aging treatment, the tensile strength and elongation of the sample have been significantly improved (Fig. 5c-d). This mechanism plays a crucial role in enhancing tensile strength and elongation.

3.4 Effect of rolling temperature and heat treatment on conductivity

By comparing the electrical conductivity of samples before and after the aging process (Fig. 9a), it was found that the electrical conductivity of all samples was improved after aging treatment at 450°C for 1 h. The cold-rolled alloy was recrystallized for 40 seconds and the conductivity of the alloy increased most significantly from 19.4 MS/m to 47 MS/m, reaching a peak value of 57 MS/m after ageing. It can be seen from Fig. 9b that the conductivity of the cold-rolled alloy after the 40 s recrystallization treatment showed an increasing trend, and Conductivity increased from 33.4%IACS to 81%IACS. During the recrystallization process, the defect density of the crystal was significantly reduced, resulting in a rapid increase in the alloy's conductivity. The cold-rolled alloy without recrystallization annealing showed the largest increase in electrical conductivity after aging treatment, and the electrical conductivity increased from 19.4 MS/m to 44.7 MS/m. As can be seen from Fig. 9b, the conductivity of cold-rolled alloy increases from 33.4%IACS to 77.1%IACS after aging treatment. During aging treatment, the solute atoms in the solid solution gradually diffused out to the matrix, resulting in the dissolution of the matrix. The number of solute atoms in the matrix continuously decreased, which reduced the scattering of electrons [29,30]. Cold rolling is carried out after hot rolling at 780°C, and the conductivity of the alloy is 42.9 MS/m. After

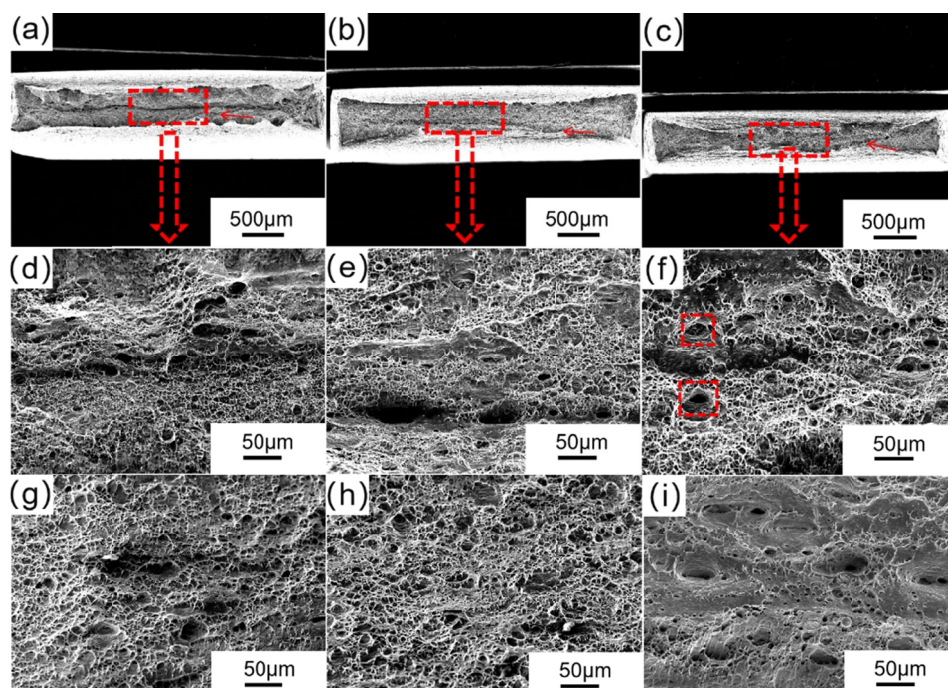


Fig. 8. The fracture morphology of Cu-1.0Cr-0.5Zr alloy after aging treatment: (a)-(f) 780°C H/CR + Re 0 min, 40 s, 3 min + aging; (g)-(i) 960°C St + CR + Re 0 min, 40 s, 3 min + aging

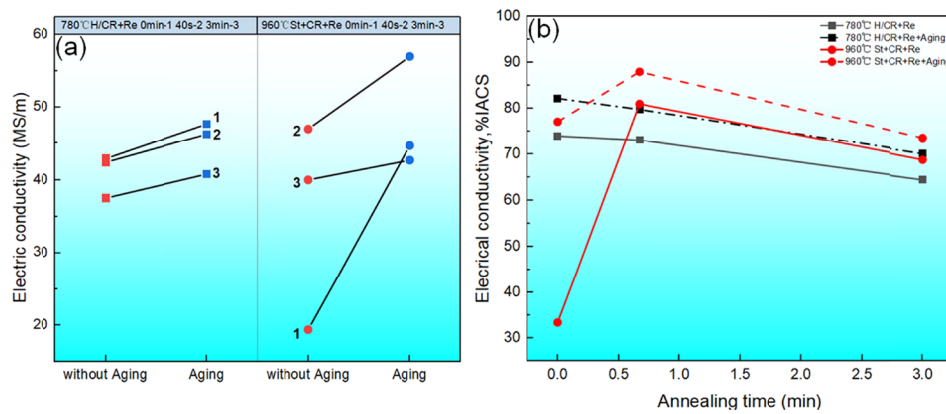


Fig. 9. The conductivity of Cu-1.0Cr-0.5Zr alloy by different processes: (a) electrical conductivity;(b) electrical conductivity ratio

40 s and 3 min recrystallization, the conductivity of the alloy is 42.4 MS/m and 37.5 MS/m, respectively. It can be seen from Fig. 9b that the conductivity shows a downward trend. This is because the recrystallization temperature is too high, so that the grain boundaries of the new grains after recrystallization hinder the electron movement. The conductivity of the recrystallized samples was improved by aging treatment.

As the amount of undercutting increases during the rolling process, the grain structure of the Cu-Cr-Zr alloy becomes elongated, leading to the generation of a large number of internal dislocations, grain boundaries, and internal stresses. This, in turn, increases electron scattering and hinders the movement of electrons, thereby reducing the electrical conductivity of the alloy. The recrystallization annealing process helps to alleviate the dislocations and internal stresses accumulated in the Cu-Cr-Zr alloy, while promoting grain regrowth. This reduces the number of grain boundaries and sharpens their structure, effectively minimizing electron scattering and enhancing the alloy's electrical conductivity. Furthermore, the aging treatment facilitates the formation and redistribution of precipitated phases within the Cu-Cr-Zr alloy. During this process, fine precipitates form and distribute uniformly, refining and optimizing the microstructure to create strengthening phases that, to some extent, reduce electron flow resistance, thereby improving the electrical conductivity of the alloy.

4. Conclusions

In this study, to obtain a better match between electrical conductivity and mechanical properties, the solid-solution Cu-1.0Cr-0.5Zr alloy was rolled at different temperatures, followed by short-time crystallization and aging treatment. The microstructure, mechanical properties, and electrical conductivity of the samples at each stage were studied carefully. The main conclusions and findings can be summarized as follows:

1) The precipitates containing Cr inhibit the formation of recrystallized grains. In addition, the precipitates could improve the dispersion-strengthening effect, thus enhancing the tensile properties of the Cu-1.0Cr-0.5Zr alloy.

- 2) The optimal combination process (960°CSt + CR + Re 0 min + aging) has a tensile strength of 595.9 MPa, elongation of 14.35%, hardness of 202.60 HV, and conductivity of 77.1% IACS. Compared with before aging treatment, the tensile strength, elongation, hardness, and conductivity were successively increased by 93.45 MPa, 8.35%, 39.7 HV, and 43.7%IACS.
- 3) The aging strengthening mechanism improves the tensile strength, hardness, and electrical conductivity of Cu-1.0Cr-0.5Zr alloy, and the primary strengthening mechanism for elongation is recrystallization strengthening.

Acknowledgments

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