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Influence of thermal cycling treatment on the anneal hardening effect of Cu–10Zn Alloy

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ABSTRACT

This paper reports the results of investigations on the influence of the so called thermal cycling treatment on the anneal hardening effect of the copper-based Cu–10 at.% Zn alloy. The quenched samples were subjected to cold rolling of 20, 40 and 60% in reduction, followed by annealing with and without thermocycling treatment below the recrystallization temperature. Anneal hardening effect was observed in both samples in the temperature range between 180 and 300 °C and was followed by an increase in the hardness and electrical conductivity. These investigations showed that the thermal cycling treatment some increased the intensity of anneal hardening effect.

1. Introduction

The last few years have seen a major effort devoted to the investigation of copper-based alloys in the search for improvements of properties such as strength, conductivity, and stress retention at high temperature [1–5]. One of the processes which has the influence on the increase of mechanical characteristics is a so called thermal cycling treatment [6–10] consisting of alternating annealing and cooling of the supersaturated solid solution up to the recrystallization temperature of copper alloys. Thermal cycling is a temperature modulation process developed to improve the performance, strength and longevity of a variety of materials. Also the strength properties of cold worked copper-base alloy systems are increased upon annealing up to the recrystallization temperature. This strengthening effect is termed anneal hardening and it was previously investigated in some copper-based alloy systems [11–18]. It was found that the amount of strengthening increases with both increasing degree of prior cold work and the concentration of substitutional elements. This strengthening effect could be applied to copper alloys in the production of spring materials for electromechanical devices [11,12].

The aim of this study is to know the influence of the thermal cycling treatment on the anneal hardening effect of a copper-based alloy. Also, the goal of these investigations is the improvement in properties by anneal hardening effect and by thermal cycling treatment, of copper-base alloy Cu–10 at.% Zn in comparison with pure copper.

2. Experimental

A copper-based alloy containing 10 at.% Zn as a solute (Cu–10Zn alloy) was melted in a laboratory electro resistance furnace and cast into a copper mold with dimensions 100 mm × 100 mm × 30 mm. Ingot mass of approximately 2 kg was protected with a graphite cover and homogenized at 850 °C for 24 h. Samples with dimensions 100 mm × 30 mm × 7 mm were cut from the homogenized material and then cold rolled to the thickness of 5, 3.3 and 2.5 mm. After that the samples were subjected to a thermomechanical treatment (TMT), i.e.: after solution annealing (at 700 °C for 1 h followed by an ice-water quenching) samples were subjected to a finally reduction of 20, 40 and 60% by cold rolling, and the final thickness of all samples was the same, i.e. 2 mm. In the next stage samples were annealed in the temperature range between 150 and 500 °C in 30 min intervals. Following annealing (heating), one set of samples was ice-water quenched (defined as thermal cycled, TC, samples). The heating and cooling rates and the temperature range for thermal cycling are 10 °C/s and 160–500 °C respectively. Thermal cycling was done in ice-water in the interval from 0 °C to annealing temperature. Specimens were subjected to such thermal cycling for 3 cycles annealing (10 min)–quenching—annealing (10 min)–quenching—annealing (10 min)–quenching. Samples photograph is given in Fig. 1.

In order to compare some properties, an ingot of unalloyed copper (OFHC quality) was subjected to the same TMT as Cu–10Zn alloy.

Vickers hardness (applying load of 50 N) and electrical conductivity ("Sigmatest") were measured following each annealing. Five different hardness and electrical conductivity measurements were recorded for each sample to minimize errors. The results were averaged for these measurements and reported as a single data set with its appropriate standard deviation which was typically in the order of ±1% for hardness, i.e. ±0.2% for electrical conductivity.

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Fig. 1. Samples photograph.

3. Results and discussion

3.1. Cold rolled samples

The hardness of samples increases with the degree of prior cold deformation due to the deformation strengthening (Fig. 2). Higher hardness was obtained for Cu–10Zn alloy, than for OFHC copper. According to maximum values of hardness after 60% deformation are 126 and 162 HV of OFHC copper and Cu–10Zn alloy respectively, it is obvious that higher deformation strengthening was achieved in Cu–10Zn alloy [19].

Fig. 3 shows the change of electrical conductivity after cold rolling. It can be seen that electrical conductivity of OFHC copper is higher than Cu–10Zn alloy. Fig. 3 also shows that electrical conductivity of alloy slowly decreases with the degree of prior deformation.

3.2. Anneal hardening effect dependence on thermal cycling treatment

Change of hardness of TC and AN samples as a function of annealing temperature is shown in Fig. 4. Values of hardness of TC samples are higher than those of AN samples.

For TC samples the hardness increases with increase of the degree of prior deformation [11–16]. This increase is about 30 HV (in comparison with the initial cold-rolled condition) for 60% deformation, and the anneal hardening effect is more pronounced than for 40% deformation (the hardness increase is about 28 HV) and for the deformation of 20% (the hardness increase is about 25 HV).

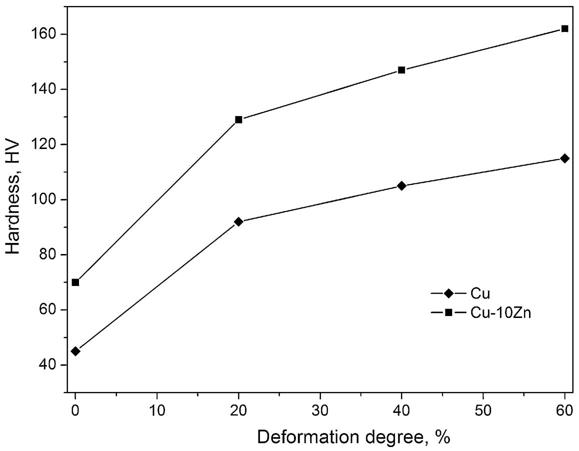

Fig. 2. Dependence of hardness of cold rolled samples on deformation degree.

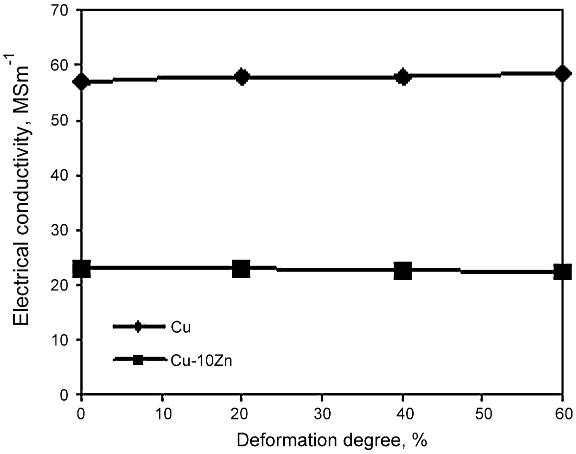

Fig. 3. Dependence of electrical conductivity of cold rolled samples on deformation degree.

For AN samples hardness also increases with the previous cold work [11–16], but at a slower rate than in the case of TC samples. The anneal hardening effect of 26 HV for 60% is somewhat higher than for the deformation of 40% (the hardness increase is about 24 HV) and for the deformation of 20% (the hardness increase is about 19 HV).

During thermal cycling process, materials are alternately cooled and heated due to optimized the molecular particulate structure. Anneal hardening effect has been investigated mainly in the copper-based alloys containing different alloying elements (Al, Ni, Au, Ga, Pd, Rh, Zn, Mn, Sb) [11,12,17,18]. The results of this papers tend to support the hypothesis that solute segregation to dislocation, analogous to the formation of Cottrell atmospheres in interstitial solid solutions, is primarily responsible for anneal hardening phenomenon [11,12].

Fig. 4 shows that the hardness of copper decrease after 200 °C for all applied deformation degrees. The maximum of hardness for both TC and AN set of samples is about 240 °C. After the maximum is reached hardness decreases slowly and at about 350 °C an abrupt decrease of hardness occurs. This decrease of hardness near 350 °C corresponds to the start of recrystallization. If the recrystallization temperature for the pure copper is reported to be around 240 °C [20] then it is obvious that the anneal hardening not only strengthens but also increases recrystallization temperature

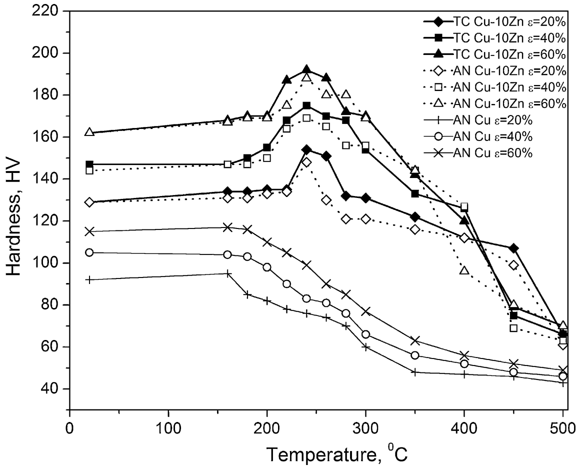

Fig. 4. Hardness of thermal cycled (TC) and annealed (AN) samples of Cu–10Zn alloy with annealing temperature.

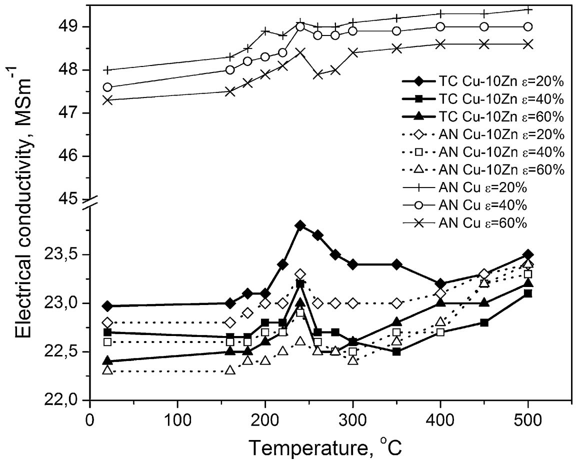

Fig. 5. Electrical conductivity of thermal cycled (TC) and annealed (AN) samples of Cu–10Zn alloy with annealing temperature.

of alloy Cu–10Zn at about 350 °C for both set of samples. The effect of Zn to increase the recrystallization temperature in comparison to pure copper was reported in previous papers [12,14].

Fig. 5 shows that the electrical conductivity of TC and AN samples remains unchanged up to 200 °C and then starts to increase due to the anneal hardening effect. The slow increase above 350 °C is probably due to recovery and recrystallization. Bader et al. [11] obtained the similar results by electrical resistivity measurements.

Fig. 6 shows the change of hardness with time during annealing at 240 °C. For both TC and AN samples hardness increases due to anneal hardening effect up to 180 min, then slowly decreases with annealing time. After 180 min of annealing the maximum of hardness of TC samples increases after prior deformation of 20, 40 and 60% for 21, 24 and 28 HV, respectively. The values of hardness of AN samples are somewhat lower. After 5 h of annealing the values of hardness are higher compared to the cold-rolled condition for both set of samples which implies that the recrystallization does not occur. This may be explained by the fact that anneal hardening effect shifts the onset of recrystallization to higher temperatures [11,12].

Fig. 7 shows the change of electrical conductivity of TC and AN samples with time during annealing at 240 °C. Generally, electrical conductivity increases during time of annealing showing a relatively small maximum after 180 min. Conductivity of AN samples is somewhat lower.

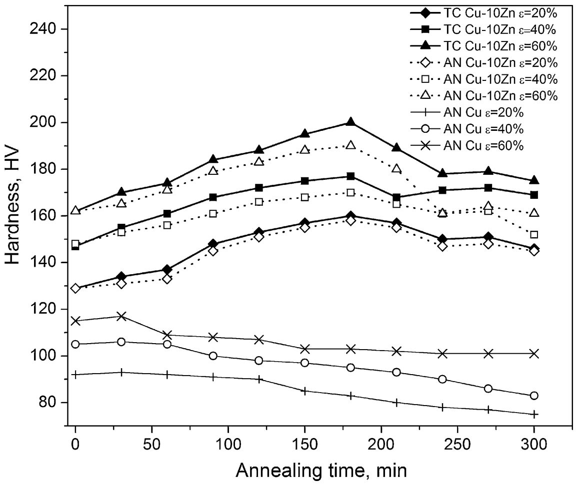

Fig. 6. Hardness of samples after thermal cycling (TC) and annealing (AN) as a function of time of annealing at 240 °C.

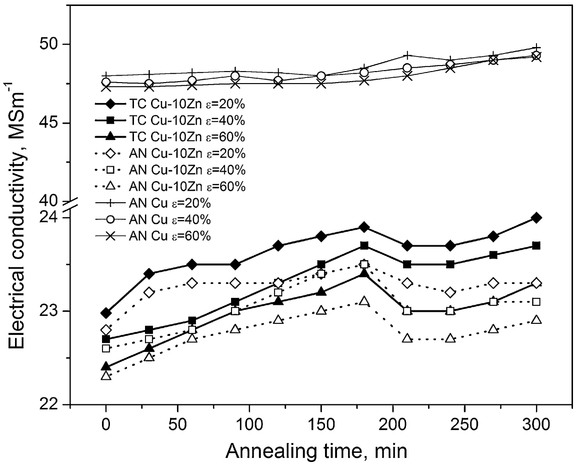

Fig. 7. Electrical conductivity of samples after thermal cycling (TC) and annealing (AN) as a function of time of annealing at 240 °C.

In the previous investigation of anneal hardening of copper-based alloys [12] it was shown that the major decrease in electrical resistivity during annealing cannot be explained by short range ordering. It was concluded that the segregation of solute atoms at dislocations is the only consistent interpretation for the major portion of the change in resistivity. X-ray diffraction analysis [11,12] showed that lattice parameters of the previously cold worked Cu–Al alloys was changed during subsequent annealing. This led to the conclusion that the solute clustering at dislocations should be one of the major causes of the observed effects caused by anneal hardening.

4. Conclusions

The obtained results can be summarized as follows:

- Zinc as alloying element was found to have a pronounced effect on the increase of the recrystallization temperature of the cold rolled Cu–10Zn alloy.
- The anneal hardening effect was attained below recrystallization temperature in the temperature range between 180 and 300 °C and was followed by an increase in hardness and electrical conductivity.
- The amount of strengthening increases with increasing the degree of the prior cold work and the maximum of hardness was established after 60% of deformation.
- Thermal cycling treatment (TC) has more pronounced influence on anneal hardening than only annealing (AN) treatment.

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