Influence of Thermocyclic Treatment on the Anneal Hardening Effect of CuAl10 Alloy

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Abstract. This paper reports the results of investigations on the influence of the so called thermocyclic treatment on the anneal hardening effect of the copper-based CuAl10 alloy. The quenched samples were subjected to cold rolling of 20, 40 and 60% in reduction, followed by annealing below the recrystallization temperature. Anneal hardening effect was observed in the temperature range between 180 and 300^{o}C and was followed by an increase in the hardness and electrical conductivity. The exothermic heat effect was found in the same temperature range where the anneal hardening was detected. These investigations showed that the thermocyclic treatment increased the intensity of anneal hardening effect.

Introduction

The last few years have seen a major effort devoted to the exploration of copper-based alloys in the search for improvements of properties such as strength, conductivity, and stress retention at high temperature [1]. One of the processes which has the influence on the increase of mechanical characteristics is a so called thermocyclic treatment [2] consisting of alternating annealing and cooling of the supersaturated solid solution up to the recrystallization temperature of copper alloys. The mechanism responsible for the increase of strength of cold worked substitutional alloys known as anneal hardening was previously investigated in some copper-based systems [3,4,5-10]. 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 when producing spring materials for electro-mechanical devices.

The aim of this study is to assess the influence of the thermocyclic treatment on the anneal hardening effect of a copper-based alloy.

Experimental

A copper-based alloy containing 10at.%Al as a solute (CuAl10 alloy) was melted in a laboratory electro resistance furnace and cast into a copper mold with dimensions 100x100x30mm. Ingot mass of approximately 2 kg was protected with a graphite cover and homogenized at 850^{0}C for 24 h. Samples with dimensions 100x30x7mm were cut from the homogenized material and than cold rolled to the thickness of 5, 3.3 and 2.5mm. The samples were subjected to a thermomechanical treatment (TMT), i.e.: after solution annealing (at 700^{0}C for 1h followed by an ice-water quenching) samples were cold rolled with 20, 40 and 60% reduction, and the final thickness of all samples was the same, i.e.

2mm. In the next stage samples were annealed in the temperature range between 150 and 500^{0}C (in steps of 20^{0}C up to 300^{0}C, and 50^{0}C after 300^{0}C). Holding time at annealing temperatures was 30min. Following annealing, one set of samples was ice-water quenched (defined as thermocycled, TC, samples), whereas another set was air-cooled from annealing temperature (defined as annealed, AN, samples).

In order to compare some properties, an ingot of unalloyed copper (OFHC quality) was subjected to the same TMT as CuAl10 alloy.

Vickers hardness (applying load of 5kp) and electrical conductivity ("Sigmatest") were measured following each annealing. Five measurements were performed for each annealing temperature. Differential scanning calorimetry (DSC) was performed in the temperature range from 350^{0}C to the room temperature.

Results and discussion

Cold rolled samples. The hardness of samples increases with the degree of prior cold deformation due to the deformation strengthening (Fig. 1). Higher hardness was obtained for CuAl10 alloy, than for OFHC copper. According to maximum values of hardness of OFHC copper (126HV) and CuAl10 alloy (206HV) after 60 % deformation, it is obvious that higher deformation strengthening was achieved in CuAl10 alloy.

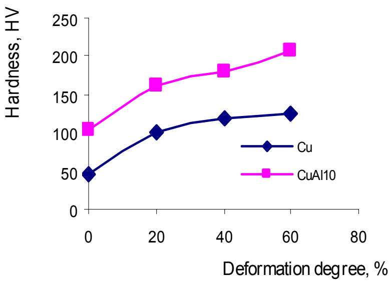

Fig. 1. *Dependence of hardness of cold rolled samples on deformation degree*

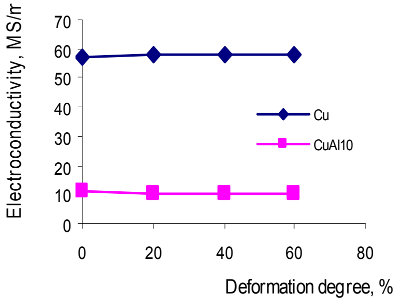

Fig. 2. *Dependence of electrical conductivity of cold rolled samples on deformation degree*

Fig. 2 shows the change of electrical conductivity after cold rolling. It can be seen that electrical conductivity of OFHC copper is higher than CuAl10 alloy. Fig. 2 also shows that electrical conductivity of alloy slowly decreases with the degree of prior deformation.

Thermocycled (TC) and annealed (AN) samples of CuAl10 alloy. Change of hardness of TC and AN samples as a function of annealing temperature is shown in Fig. 3. 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. 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 24 HV) and for the deformation of 20% (the hardness increase is about 20 HV).

For AN samples hardness also increases with the previous cold work, but at a slower rate than in the case of TC samples. The anneal hardening effect of 19 HV for 60% deformation is somewhat higher than for the deformation of 40% (the hardness increase is about 16 HV) and for the deformation of 20% (the hardness increase is about 15 HV).

Anneal hardening effect has been investigated mainly in the copper-based alloys containing Al, Ni, Au, Ga, Pd, Rh and Zn [4]. The results of this paper 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. [3,4].

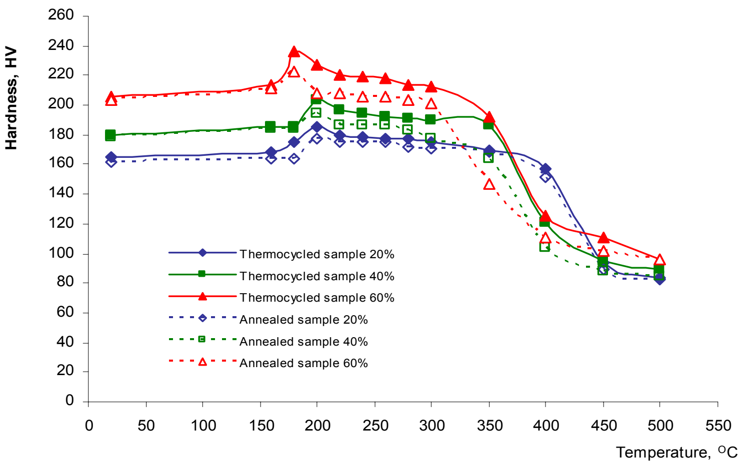

Fig. 3. *Hardness of thermocycled (TC) and annealed (AN) samples of CuAl10 alloy with annealing temperature*

The maximum of hardness for both TC and AN set of samples is reached between 170 and 200^{0}C (higher degree of deformation shifts the maximum of hardness to lower annealing temperature, especially in the case of TC samples). After the maximum is reached hardness decreases slowly and at about 350^{0}C an abrupt decrease of hardness occurs. This decrease of hardness near 350^{0}C corresponds to the start of recrystallization. If the recrystallization temperature for the pure copper is reported to be around 200^{0}C [5] then it is obvious that the anneal hardening not only strengthens CuAl10 alloy but also increases recrystallization temperature to about 350^{0}C. for both set of samples. The effect of Al to increase the recrystallization temperature in comparison to pure copper was reported in previous papers [8,9].

Fig. 4 shows that the electrical conductivity of TC an AN samples remains unchanged up to 200^{0}C and then starts to increase due to the anneal hardening effect. The slow increase above 350^{0}C is probably due to recovery and recrystallization. Bader et al. [3] obtained the similar results by electrical resistivity measurements.

Figure 5 shows the change of hardness with time during annealing at 200 ^{0}C. For both TC and AN samples hardness increases due to anneal hardening effect up to 150min, then slowly decreases with annealing time. After 150min of annealing the maximum of hardness of TC samples increases after prior deformation of 20, 40, 60% for 25, 26 and 31 HV, respectively. The values of hardness of AN samples are somewhat lower. After 5h 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.

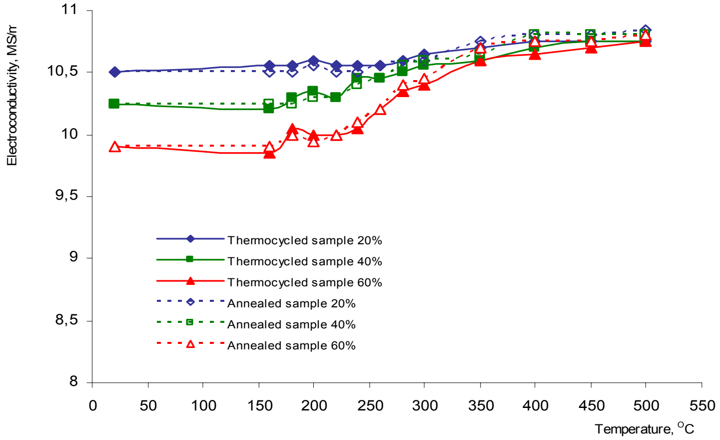

Fig. 4. *Electrical conductivity of thermocycled (CT) and annealed (AN) samples of CuAl10 alloy with annealing temperature*

Fig. 6 shows the change of electrical conductivity of TC and AN samples with time during annealing at 200^0C. Generally, electrical conductivity increases during time of annealing showing a relatively small maximum after 150min. Conductivity of AN samples is somewhat lower.

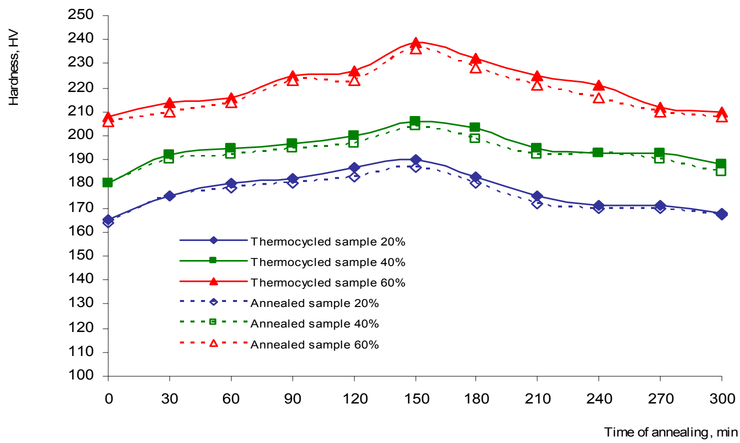

Fig.5. *Hardness of samples after thermocycling (CT) and annealing (AN) as a function of time of annealing at 200^0C*

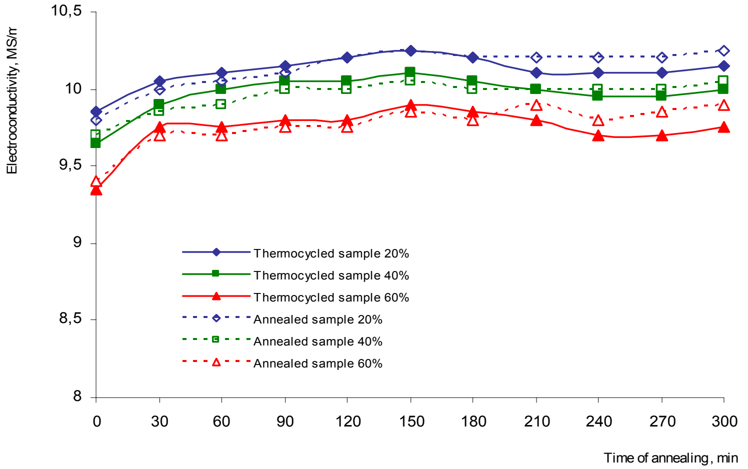

Fig.6. *Electrical conductivity of samples after thermocycling (CT) and annealing (AN) as a function of time of annealing at 200^{0}C*

In the previous investigation of anneal hardening of a CuAl alloy it was shown that the major decrease in electrical resistivity during annealing cannot be explained by short range ordering [11]. 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.

Figure 7 shows the exothermic heat effect of cold-rolled samples of CuAl10 alloy upon heating detected by DSC analysis. Heat effect exists in the range between 210 and 300^{0}C, i.e. in the same range where the anneal hardening effect was attained (see Fig. 3).

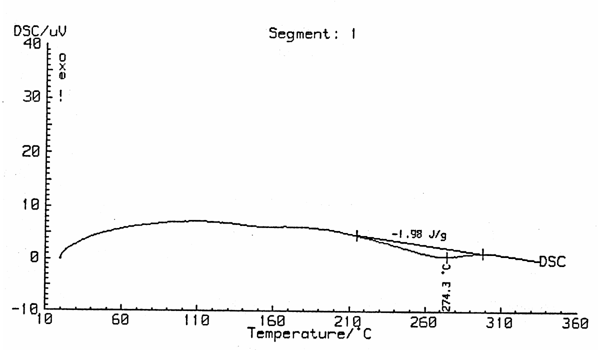

Fig.7. *DSC of CuAl10 alloy cold rolled with 60%*

X-ray diffraction analysis showed that lattice parameters of the previously cold worked CuAl alloy 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 [10].

Conclusions

- Aluminium as alloying element was found to have a pronounced effect on the increase of the recrystallization temperature of the cold rolled CuAl10 alloy.

- The anneal hardening effect was attained below recrystallization temperature in the temperature range between 180 and 300^{0}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.
- Thermocycling threatment (CT) has more pronounced influence on anneal hardening than annealing (AN) treatment.
- The exothermic heat effect was found in the same temperature range where the anneal hardening effect was detected.

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