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Heat Treatment of CuFe2 and CuCr0.6 Alloys and the Effect of Precipitates on the Grain Refinement

A. Urbańczyk-Gucwa*, P. Amrogowicz, M. Jabłońska and K. Rodak
Silesian University of Technology, Faculty of Materials Science and Metallurgy,
Z. Krasińskiego 8, 40-019 Katowice, Poland

The results of microstructure and hardness investigations of the CuFe2 and CuCr0.6 alloys after solution and ageing treatment are presented in this paper. The variants of heat treatment as: solution at 1000 °C per 3 h and ageing treatment at 500 °C for 2 h and at 700 °C for 24 h were chosen for severe plastic deformation process realized by rolling with the cyclic movement of rolls method. The structure of CuFe2 and CuCr0.6 alloys was analysed using scanning transmission electron microscopy, and the quantitative studies of the substructure was performed with MET-ILO software, on the basis of images acquired on scanning transmission electron microscope.

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1. Introduction

Precipitation hardening copper alloys belong to a group of functional materials with a good combination of strength and electrical conductivity [1–5]. The effect of heat treatment in CuFe2 alloy, and CuCr0.6 alloy — especially influence of second phase particles on the evolution of deformation structures — has been systematically studied [4–7]. But this effect has not been extensively studied in the context of severe plastic deformation condition. From literature [1] it is known that metallurgical processes are very complicated and require high precision during melting and casting of CuFe2 and CuCr0.6 alloys, since susceptibility to oxidation processes is high. In order to reduce the oxygen content in the cast alloy and improve the mechanical properties, phosphorus is applied, which has a better chemical relation with oxygen than with copper. For this reason the dissolution of oxygen in the liquid copper is given [2].

The preferred temperature of solution for CuFe2 and CuCr0.6 alloys is 1000±10 °C [5–7]. Increase of solution temperature can cause oxidation of the Cu alloys and increase in thickness of oxide layer. In initial state of ageing the coherent γ-Fe precipitate particles first appear in a Cu matrix [8]. These nearly spherical γ-Fe particles can transform into α-Fe by plastic deformation or extraction process is possible during deformation [9]. In Cu–Cr system the equilibrium crystallographic structure of Cr is bcc, but in the early stage of precipitation it has been proposed that they could exhibit a metastable fcc structure [10]. The objective of the present study were to find the optimum heat treatment processing with completely different conditions, including solution heat treatment and ageing treatment, to obtain materials with different initial structures, which will be used for severe plastic deformation process (SPD). For this, the CuFe2 and CuCr0.6 alloy specimens were heat treated following various temperatures and times. Hardness and electrical conductivity values were characterized for the alloys specimens fabricated by various heat treatment.

2. Experimental procedure

In this study precipitation hardened copper alloys with addition of 2 wt% Fe (C19400) and 0.6 wt% Cr (C18200) were used, prepared by melting and alloying in open-air induction furnace. The alloys were prepared by casting into diameter of 50 mm mould. Ingots were hot rolled with using intermediate annealing at temperature 700 °C for about 10 min. The billets were hot forged at temperature of 750 °C and next rolled to strip sample $8 \times 8 \times 60$ mm^3. Samples were subjected to heat treatment by solution at 1000 °C per 1, 3, 6, and 12 h with water cooling. Next, on samples after treatment solution at 1000 °C for 3 h, there were performed ageing treatments at: 500, 600, 650, 700 °C for 2 h and at 700 °C for 24 h.

Microstructural observations of the alloys were carried out using scanning transmission electron microscope (STEM) Hitachi HD-2300A equipped with field emission type gun. The Vickers hardness was measured on an electrolytically polished surface of the samples by means of a Zwick hardness tester. A 1 kg load applied for 15 s was ensured for these measurements. The hardness values were taken as the average of a minimum of 10 measurements. The electrical conductivity was measured by means of Foerster Sigmatest 2.069. The microscope observations, hardness and electrical conductivity measurements were made in the middle of the sample in the transverse plane section. The quantitative studies of the substructure was performed with MET-ILO software, on the basis of images acquired on STEM microscope.

*corresponding author; e-mail: Anna.urbanczyk-Gucwa@polsl.pl

3. Result and discussion

3.1. CuFe2 alloy

Figure 1a and 1b shows the microstructure of CuFe2 alloy after solution treatment at 1000 °C per 1 h and at 1000 °C per 3 h. Microstructures are characterized by the presence of undissolved equiaxial Fe precipitates in Cu matrix. Based on STEM/EDS investigations it has been shown that the precipitates are rich in P. Phosphorus was added as an antioxidant during casting. According with MET-ILO measurements, the average diameter of precipitates for solution treatment at 1000 °C per 1 h and at 1000 °C per 3 h for CuFe2 alloy were 0.5 µm and 0.27 µm respectively (Fig. 1c). The average distance of particles were 3.1 µm and 1.14 µm for CuFe2 alloy after solution treatment at 1000 °C per 1 h and at 1000 °C per 3 h (Fig. 1d). From the performed results it is known that increase of time solutioning results in dissolved precipitates in the matrix for the reason solution treatment at 1000 °C per 3 h were chosen for next ageing treatment.

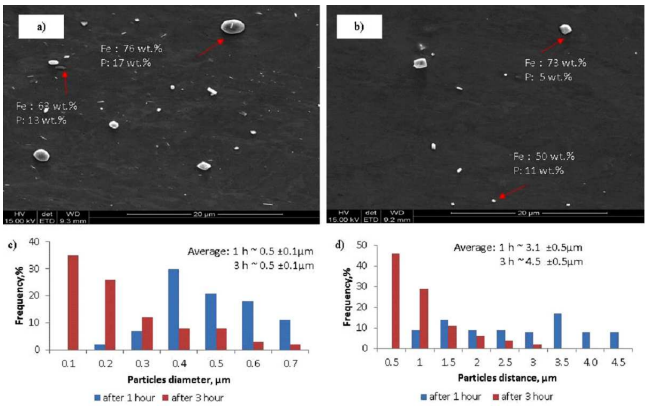

Fig. 1. Microstructure of CuFe2 alloy after solution treatment at 1000 °C per 1 h (a), after solution treatment at 1000 °C per 3 h (b), distribution of Fe particles diameter (c), distribution of Fe particles distance (d).

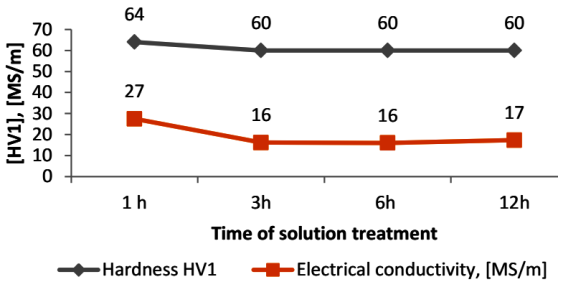

Fig. 2. Hardness and electrical conductivity obtained for CuFe2 alloy after solution treatment.

Figure 2 shows the results of hardness and electrical conductivity measurement after solution treatment. The obtained results of hardness measurements were comparable for 1, 3, 6, and 12 h of solution treatment and reached about 60 Hv1. A decrease of electrical conductivity from 27 MS/m to 17 MS/m were recorded with increase of solution treatment time (Fig. 2).

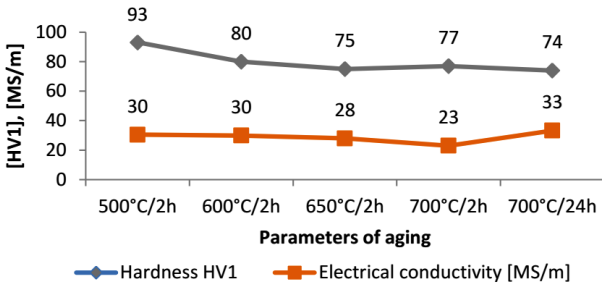

Fig. 3. Investigations results of hardness and electrical conductivity after ageing of CuFe2 alloy.

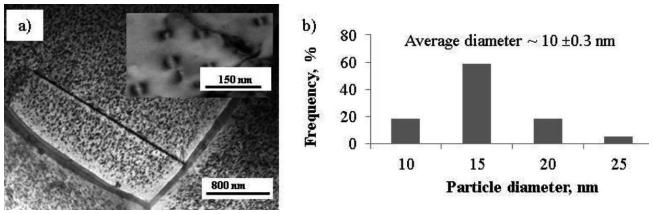

Fig. 4. Substructure of CuFe2 alloy after ageing in 500 °C/2 h (a), distribution of average diameter of coherent Fe precipitates (b).

Figure 3 shows the Vickers hardness and electrical conductivity for CuFe2 alloy after different parameters of ageing treatment. Hardness decrease with ageing time from 93 Hv1 (after 500 °C/2 h) to 74 Hv1 (after 700 °C/24 h). Maximal value of electrical conductivity about 33 MS/m was obtained after 700 °C/24 h of ageing. Two microstructure investigations were selected only extreme parameters of ageing treatment.

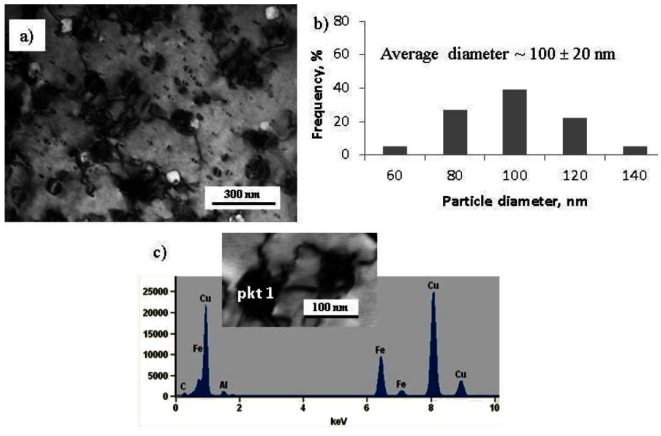

Fig. 5. Substructure of CuFe2 alloy after ageing in 700 °C/24 h (a), distribution of average diameter of noncoherent Fe precipitates (b), EDS spectra of precipitates (c).

Figure 4 and Fig. 5 show the microstructure of CuFe2 alloy after solution (1000 °C/3 h) and ageing treatment followed at 500 °C for 2 h and at 700 °C for 24 h. Ageing treatment at 500 °C for 2 h results in the formation of coherent Fe precipitates within the matrix (Fig. 4a). The size of these particles varied between 10 and 25 nm (Fig. 4b). With prolonged ageing temperature and time, the precipitates lost coherence with matrix (Fig. 5a) and amount of the Fe (Fig. 5c) particles within matrix decreases in number (spacing between particles increase) (Fig. 5b). The size of these particles varied between 60 and 140 nm (Fig. 5b).

3.2. CuCr0.6 alloy

Microstructure of CuCr0.6 alloy is characterized by the presence of undissolved equiaxial chromium precipitates in Cu matrix (Fig. 6a). Figure 6b,c shows the diameter and distance of neighbouring chromium particles. The average diameter of precipitates is about 0.75 µm and average distance of particles is about 2.75 µm.

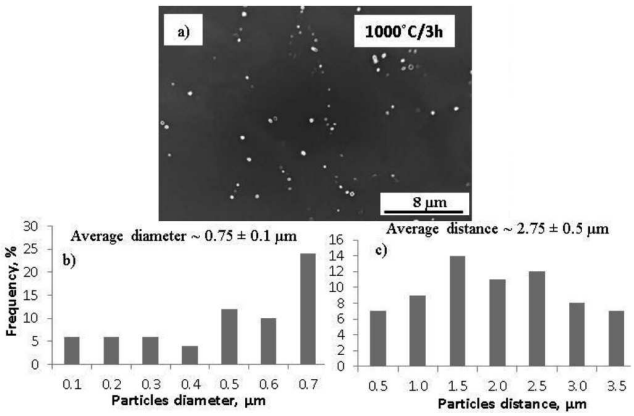

Fig. 6. Microstructure of CuCr0.6 alloy after solution treatment (a), distribution of the Cr particles diameter (b), distribution of the Cr particles distance (c).

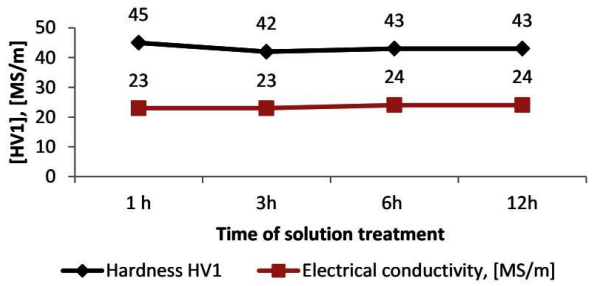

Fig. 7. Hardness and electrical conductivity obtained for CuCr0.6 alloy after solution treatment.

Hardness and electrical conductivity results for CuCr0.6 alloy after heat treatment are presented in Fig. 7 and Fig. 8. With increase time of solution treatment the changes in hardness of CuCr0.6 alloy are negligible and values are about 45 Hv1. Independently of the solution treatment time the electrical conductivity is about 23–24 MS/m.

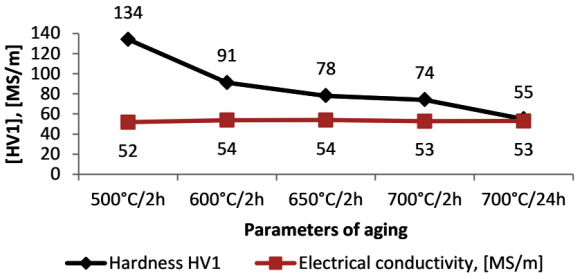

Fig. 8. Changes in hardness and electrical conductivity after ageing for CuCr0.6 alloy.

After ageing the hardness of CuCr0.6 alloy decreased from 134 Hv1 (after 500 °C/2 h) to 55 Hv1 (after 700 °C/24 h). Electrical conductivity slightly changed with parameters of ageing treatment and reached for example value about 52 MS/m (after 500 °C/2 h). The results of structural investigations on samples after solution (1000 °C/3 h) and ageing treatment followed at 500 °C for 2 h and at 700 °C for 24 h are presented in Fig. 9 and Fig. 10. Ageing treatment at 500 °C for 2 h result in the formation of coherent Cr precipitates within the matrix (Fig. 9a). The average diameter of these particles is 20 nm (Fig. 9b). With prolonged ageing temperature and time, the Cr precipitates (Fig. 10c) lost coherence with matrix (Fig. 10a) and amount of the particles within matrix decreases in number (spacing between particles increases). The average particles diameter is 270 nm (Fig. 10b).

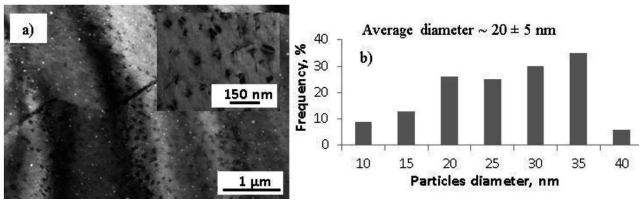

Fig. 9. Substructure of CuCr0.6 alloy after ageing in 500 °C/2 h (a), distribution of average diameter of coherent Cr precipitates (b).

Obtained second particles during various heat treatments parameters are different in dimension, distance between them and crystal lattice fit. These particles have a significant effect on grain refinement (Fig. 11). Obtained result of hardness and electrical conductivity are close to the results presented in literature [3–6]. In samples after ageing treatment at 500 °C/2 h, which next were deformed by severe plastic deformation by using RCMR method at total effective strain $\varepsilon_{ft} \approx 5$ the presence of dislocation inside new grains/subgrains and inside grain/subgrain boundaries should be attributed to

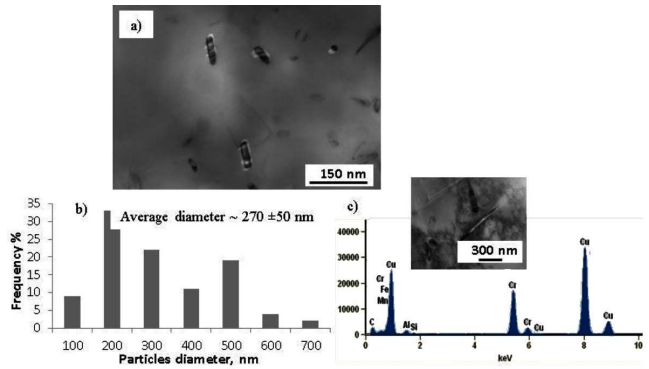

Fig. 10. Substrucrure of CuCr0.6 alloy after ageing in 700 °C/24 h (a), distribution of average diameter of non-coherent Cr precipitates (b), EDS spectra of precipitates (c).

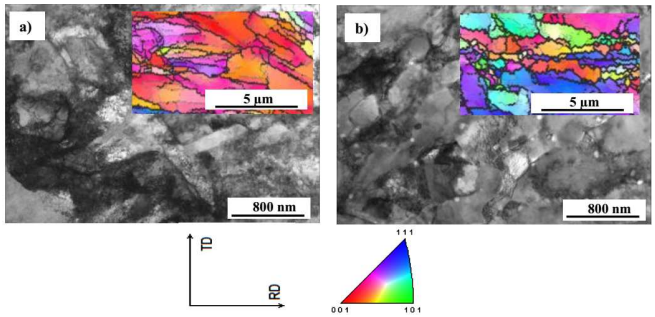

Fig. 11. STEM micrographs and EBSD maps in sample after ageing treatment at 500 °C/2 h with the next SPD deformation (a), in sample after ageing treatment at 700 °C/24 h with the next SPD deformation (b).

the strong deformation and mainly by increase the rate of dislocation generation. It should be taken into account that the high amount of dislocation produced in the first passes of RCMR thanks to fine precipitation can strongly accelerate the formation of numerous deformation band. During next passes, the coherent precipitates inhibit the long-range migration of dislocation and, less dynamic boundary migration occurs (Fig. 11a). Therefore, it is possible to obtain much finer deformation structures. In samples after ageing treatment at 500 °C/2 h, which next were deformed by severe plastic deformation at $\varepsilon_{ft} \approx 5$ the grain/subgrain boundaries are strongly pinned by fine precipitates with 100 nm dimension (Fig. 11b). In a result the structure recovery/ recrystallization is in large extent suppressed because these particles constitute additional barriers to the movement of grains.

4. Summary

1. The average diameter of precipitates during ageing increases from 10 nm to 100 nm with increase of temperature from 500 °C/2 h to 700 °C/24 h for CuFe2 alloy. The average diameter of precipitates during ageing increases from 20 nm to 270 nm with increase of temperature from 500 °C/2 h to 700 °C/24 h for CuCr0.6 alloy.

2. After ageing at 500 °C/2 h coherent precipitates are observed in both alloys.

3. Presence of second-phase particles has a significant effect on formation of UFG structure during RCMR deformation:

   — Existence of fine coherent particles (10 nm in size) is effective in dislocation pinning and increase of dislocation density.

   — The particles with the average dimension 100 nm were effective in hindering grain growth.

Acknowledgments

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