

Study of rare earth elements on the physical and mechanical properties of a Cu–Fe–P–Cr alloy

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Abstract

The influence of rare earth elements (mischmetall, containing mainly La and Ce) on a high-strength and high-conductivity Cu–Fe–P–Cr alloy was investigated by optical microscope (OM), scanning electron microscope (SEM), transmission electron microscope (TEM), resistivity analysis, hardness analyses, and tensile tests, etc. Experimental results show that appropriate addition of rare earth elements (Re) can effectively improve the physical properties (electrical conductivity and temperature to softening) and the mechanical properties of the alloy by facilitating the precipitation of the strengthening phases, making the strengthening particles finer and well dispersed in the matrix, refining the matrix microstructure, etc. Excessive addition of Re can deteriorate the properties of the alloys. The results were discussed in relating to the metal purifying and to the strengthening mechanism in Cu–Fe–P alloy.

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1. Introduction

Copper-based alloys are widely used in many fields owing to their good combination of high thermal and electrical conductivities, high-strength and high resistance to softening, etc. In particular, Cu-based alloys with high performance are required in the field of electronic materials, such as substrate and lead frame in printed board, interconnectors and so on.

A number of commercial copper-based alloys have been developed, and the Cu–Fe–P alloy system is the most widely used because of their low cost, good formability and good combination of mechanical and physical properties [1,2]. In this alloy system, the high electrical conductivity is due to the very low solubility of the alloying elements such as Fe in Cu matrix, whereas the good mechanical properties are mainly attributed to the precipitation and particle-dispersion strengthening mechanisms.

Ageing treatment is an effective method for obtaining good combination of mechanical and physical properties of Cu–Fe–P alloys. During ageing treatment, the supersaturated solid solution decomposes into γ -Fe or Fe_3P [3,4], fine and well-dispersed γ -Fe or Fe_3P particles make great contribution to the strength,

meanwhile not do harm to the electrical conductivity of the alloy. Therefore, fine and well-dispersed strengthening particles in fine Cu matrix are desired for high-strength, high-conductivity Cu–Fe–P alloys.

However, for Cu–Fe–P alloys, the electrical conductivity and resistance to softening are usually not sufficient for their utilization in some fields such as lead frame and interconnectors [5]. In Cu–Fe–P alloys, the contents of alloying elements are usually limited, and previous work showed that the age hardening response of these alloys is limited [5]. Consequently, the mechanical and physical properties are usually not satisfactory by precipitation hardening solely due to the low volume fraction of precipitates. Besides the precipitation hardening, solution strengthening and cold working hardening are usually used to increase the strength of this alloy system, and these strengthening methods do little harm on the electrical conductivity. To develop copper-based high-strength and high-conductivity alloys, good combination of precipitation strengthening, solution strengthening and cold working hardening is indispensable [6].

Generally, trace element additions such as Mg are required to achieve optimum properties in these ternary alloys, and some other methods are often used to increase the strengthening effect [7]. Previous work showed that, a small addition of Cr to Cu–Fe–P alloys could give rise to a large precipitation

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hardening response [8]. In the solution treated condition, the supersaturation of Cr in Cu matrix creates a high degree of thermodynamic meta-stability, thus providing a high chemical potential for the precipitation reaction of Cr. Therefore, the increase of the strength by small addition of Cr is mainly derived from the distribution of nano-sized Cr rich precipitates in the Cu matrix due to the fine precipitate size in the peak-aged condition. Electrical conductivity is less influenced due to the low concentration of Cr in Cu solid solution after ageing [9].

It is well known that Re such as Ce, La, etc., due to its special physical and chemical characteristics, could effectively influence the nature of the melted alloy and therefore influence the properties of copper-based alloys. Until now, little works concerned the effect of Re on the microstructure and properties of Cu–Fe–P alloys. The aim of this work is mainly to investigate how the small addition of Re affects the morphology, mechanical and physical properties of a Cu–2.35%Fe–0.03%P–0.1%Cr (in weight) alloy.

2. Experimental procedures

To prepare Cu–2.35%Fe–0.03%P–0.1%Cr alloys containing different volume fractions of rare earth elements, small pieces of electrolytic copper, Cu–25 wt%Fe master-alloy, Cu–P master-alloy, Cu–25 wt%Cr master-alloy and Re (mischmetall, mainly containing La and Ce), weighed in an appropriate ratio, were melted together in a graphite crucible in an induction furnace. In this work, 0.1%Cr was added to the alloys to improve the strength and the resistance to softening by leading to an increased volume fraction of precipitates as indicated above. The addition of 0.03%P is mainly used to deoxidize while melting and to form a small fraction of strengthening phase Fe₃P. High purity nitrogen was used as a protective atmosphere in the induction unit. The compositions of the ingots analyzed by ICP are given in Table 1. The four ingots weighing approximately 15 kg, respectively, were homogenized at 950 °C for 2 h, and then hot-rolled to 6 mm. The hot-rolled plates were solution treated at 930 °C for 70 min in an electric resistance furnace full of nitrogen atmosphere, followed by water quenching. The solution and quenching treated plates were cold-rolled to a thickness of 1 mm, and then cleaned and aged at different temperature for 2 h, followed by air cooling. The aged samples were cut into small pieces of desired sizes for mechanical and physical property analyses.

A Nikon Epiphot-200 optical microscope was used to observe the microstructure of the studied alloys. The samples were metallographically polished and then electrolytically etched in

concentrated phosphoric acid. For TEM studies, discs of 3 mm in diameter were punched from aged pieces, ground to about 120–150 µm, and then double jet thinned at room temperature in etching solution HNO₃:CH₃OH = 1:3, electric parameters 20 V, 120 mA. Prior to loading in TEM, the jet-thinned discs were ion-milled in Gaton Doumill for about half an hour. The thin foil specimens were examined in JEM 3010-HRTEM equipped with EDXS at 300 kV.

To determine tensile properties, samples of 12.5 mm gauge length were machined out of the aged plates. Tensile strength tests were carried out on CSS 44100 tensile machine at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ at room temperature. Vickers micro-hardness measurements were determined on HVS-1000 digital micro-hardometer with the loading of 100 g and loading time of 20 s.

Electrical conductivity testing was carried out on the samples with the gauge length of 100 mm. The same equipment and testing technique were used for all testing samples. All measurement was carried out at a uniform temperature of 23 °C. Prior to testing, the surface of each specimen was polished with 1200 grit SiC paper to remove the oxide. The dimensions for calculating the cross-sectional areas were carefully measured with a digital vernier to the nearest 0.01 mm.

For each examination, at least 3 samples were used to ensure the results were reproducible and with little scatter.

3. Results and discussion

3.1. Microstructure

Despite the same melting and casting processes, the four tested ingots show different optic microstructures. Fig. 1 shows the as-cast microstructure of the alloys with different Re volume fractions: Fig. 1(a) is the alloy without Re, Fig. 1(b) with 0.1%Re, Fig. 1(c) with 0.5%Re, and Fig. 1(d), 0.8%Re. It can be observed that, the four as-cast microstructures are all almost equiaxed, but the grains of the alloys with certain Re are more homogeneous, and the grain boundaries are much cleaner compared with Fig. 1(a). With the increase of Re, the microstructure becomes finer: the average grain size of Fig. 1(a) is more than 150 µm, and that of the alloy with 0.8%Re is less than 40 µm. These results confirm that the addition of Re can considerably refine the as-cast microstructure of the Cu–Fe–P–Cr alloys.

Fig. 2 is the bright field TEM microstructure of the four studied alloys aged at the same conditions (at 525 °C for 2 h). Fig. 2(a) is the Cu–Fe–P–Cr alloy without Re, Fig. 1(b) with 0.1%Re, Fig. 1(c) with 0.5%Re, and Fig. 1(d), 0.8%Re. Fig. 3 is a typical EDXS spectrum of the alloys with Re. It can be seen from Fig. 3 that the alloys contain Fe, Cr, P, and the SEM analyses indicate that most of the precipitates are γ-Fe, with a little volume fraction of Fe₃P particles. It can be observed from Fig. 2 that, with the increase of Re, the volume fraction of precipitates increase substantially, and the dispersion of precipitates in matrix becomes more homogeneous. It can also be observed that, the alloy without Re presents coarser precipitates, and with the increase of rare elements, the precipitates become finer and well distributed in the matrix. These results indicate

Table 1
Composition of the ingots

	Elements				
	Fe	P	Cr	Re	Cu
Ingot 1	2.35	0.030	0.098	–	Balance
Ingot 2	2.34	0.035	0.096	0.10	Balance
Ingot 3	2.36	0.033	0.103	0.51	Balance
Ingot 4	2.35	0.031	0.099	0.81	Balance

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