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ARTICLE

Characteristics of Nano-alumina Particles Dispersion Strengthened Copper Fabricated by Reaction Synthesis

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Abstract: Alumina dispersion strengthened copper (ADSC) composite was prepared by reaction synthesis (RS) process. Results show that nano-sized γ-Al_2O_3 particles with about 10 nm in size are homogeneously distributed in copper matrix. A coherent interface relationship with $(002)_{Cu}$ // $(\bar{1}\bar{3}3)_{\gamma\text{-}Al_2O_3}$, and $[110]_{Cu}$//$[011]_{\gamma\text{-}Al_2O_3}$ characterizes along the explored interfaces of the composites. All of the interfaces are clear and closely combined with each other. The composites have high properties, the tensile strength can reach 570 MPa and the yield strength can reach 533 MPa at room temperature. Meanwhile, the softening temperature is higher than 1173 K. The electrical conductivity of the sample is 85% IACS and the Rockwell hardness can reach 86 HRB.

Key words: composites; interface; dispersion strengthened copper; powder metallurgy; reaction synthesis

Copper and its alloys have the advantages of high thermal and electrical conductivity. They are widely used as conductors employed in electrical contact components, vacuum breakers in high voltage power supply appliances, very important materials in nuclear reactors, etc [1,2]. The key materials are usually made by solid solution hardening or precipitation strengthening copper alloys, such as Cu-Cr, Cu-Be and Cu-Cr-Zr alloys. However, they possess a fatal weakness, for being easy softening and degradation when temperature elevates. Thus, in order to meet these requirements, it is necessary to develop a copper alloy with high thermal conductivity, high electrical conductivity, especially excellent softening resistance at elevated temperature. Alumina dispersion strengthened copper (ADSC) alloy is a member of metal matrix composites using alumina ceramic particles uniformly distributed in the copper matrix as a strengthening phase. The alumina particles in copper can efficiently pin the dislocation and impede the movement of grain boundaries[3,4]. Owing to its superior advantages such as high strength, high hardness, excellent thermal and chemical stability at elevated temperature, ADSC alloys have been considered to be a suitable candidate material.

At present, the Al_2O_3 dispersoids can be added into copper matrix by ex-situ methods or in-situ methods [5]. The shortcoming of ex-situ method is that nano-sized Al_2O_3 dispersoids can't be dispersed in the copper matrix uniformly. Meanwhile, the bonding between the particles and the copper matrix is poor. In contrast, ADSC alloys can be produced by various in-situ processing methods with a homogeneous structure, such as internal oxidation (IO), reactive spray deposition (RSD) and reaction synthesis (RS), etc [5-8]. However, the traditional IO process always uses solid-phase cuprous oxide as oxygen source for selective oxidizing aluminum at elevated temperature in a gas shielded or sealed container [9], which involving some shortcomings need to be improved. For example, the coarse cuprous oxide can't be completely reduced to copper and remains in the copper matrix, which will seriously affect the performance of ADSC alloy. Besides, the traditional IO process is so complicated that the quality of the alloys is not easy to be controlled. RS method is performed by chemical reactions between additives and the alloyed powders with the help of external actions [10-13], which may be a new promising approach to produce ADSC alloy with high quality. If Al_2O_3 nano particles can be obtained and

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dispersed homogeneously inside the copper matrix, pinning effects to dislocations, sub-grains and grains can be acquired so as to increase the strength and the hardness of ADSC alloy at elevated temperature.

Recent years, a novel RS process to fabricate ADSC alloy has been explored. In the present paper, the phase structure of alumina particle, and the microstructure at the interface between Al_2O_3 dispersoid and the copper matrix were investigated. The softening temperature, Rockwell hardness, electrical conductivity and mechanical properties were also analyzed.

1 Experiment

The Cu-1.12wt%Al_2O_3 (0.6wt%Al) alloy was prepared by RS process. The Cu-Al_2O_3 powder compact was sintered at 1193 K for 60 min and hot-extruded at a 20:1 ratio to fabricate an alloy bar with 14 mm in diameter.

In order to evaluate the hot-softening resistance of the ADSC alloy and pure copper at elevated temperature, some specimens were annealed in a Muffle furnace under hydrogen protective atmosphere at different temperatures (473, 673, 873, 1073, and 1173 K) for 60 min. According to ASTM E10-01e1 standard test method, the hardnesses of the heat treated samples were measured at five different positions by HDI-1875 Rockwell hardness tester at room temperature, under load of 98 N and loading time was 10 s. Electrical conductivity (%IACS) was tested by a SIGMA-SCOPE-LSMP type eddy current electro conductive device with ten times. According to the ASTM E8M-04 standard testing method, the tensile properties of ADSC alloy specimens were measured with a MTS-810 type testing machine.

The microstructure of the as-prepared Cu-Al_2O_3 powder was investigated by JSM-7001F field emission scanning electron microscope (FE-SEM) attached with energy dispersive X-ray spectrometer (EDX). Meanwhile, the fracture surfaces of the tensile specimens were also observed. The morphology of alumina particles and the interface structure were investigated by a JEM-2010 type high resolution transmission electron microscopy (HRTEM) under an accelerated voltage of 200 kV. The TEM specimens of ADSC alloy were mechanically polished to 80 μm in thickness and cut into discs with 3 mm in diameter. Thin foil was obtained by an ion milling method.

2 Results and Discussion

Fig.1a shows the morphology of as-synthesized ADSC powder. Fig.1b offers a high resolution image of the ADSC powder, which manifests that many nano Al_2O_3 particles are uniformly dispersed in the copper matrix with sphere or nearly spherical shape. The EDX spectrum in Fig.1c confirms that only copper, oxygen and aluminum elements are presented in the ADSC sample. Since the copper is the matrix of the alloy, the second phase should be nano Al_2O_3 particle. Further confirmation can be examined by a transmission electron microscope.

Fig.2a is a representative HRTEM image and selected-area diffraction pattern of fine dispersoids (marked as II, taken along the [110] zone axis of copper) presented in the matrix of ADSC hot-extruded alloy. It can be seen that the second phase marked as I with a size of about 10 nm can only be indexed in the terms of fcc γ-Al_2O_3 phases. Fig.2b is the Inverse Fourier-filtered image taken from the region I in Fig.2a. The results indicate that the interfaces are bonded directly, and the inter-planar distance of white region is 0.181 nm. Corresponding to the (022) plane of the Cu matrix, the $(\bar{1}\bar{3}3)$ plane inter-planar distance of γ-Al_2O_3 phase is 0.181 nm too, the interface relationship is $(002)_{Cu}$ // $(\bar{1}\bar{3}3)_{\gamma\text{-}Al_2O_3}$, $[110]_{Cu}$//$[011]_{\gamma\text{-}Al_2O_3}$. In the light of crystallographic structure, it is easy to form a coherent phase boundary between copper matrix and γ-Al_2O_3 particles, and all the interfaces are clear and closely combined with each other. Such interfaces are beneficial to obtain a high electrical conductivity.

Fig.3 shows the curves of Rockwell hardness of as-synthesized ADSC alloy and pure copper at different annealing temperatures. It is demonstrated that the hardness of ADSC sample descends slightly from 86 HRB at room temperature to 72.6 HRB after annealing at 1173 K, and the hardness of pure copper decreases rapidly with the increase

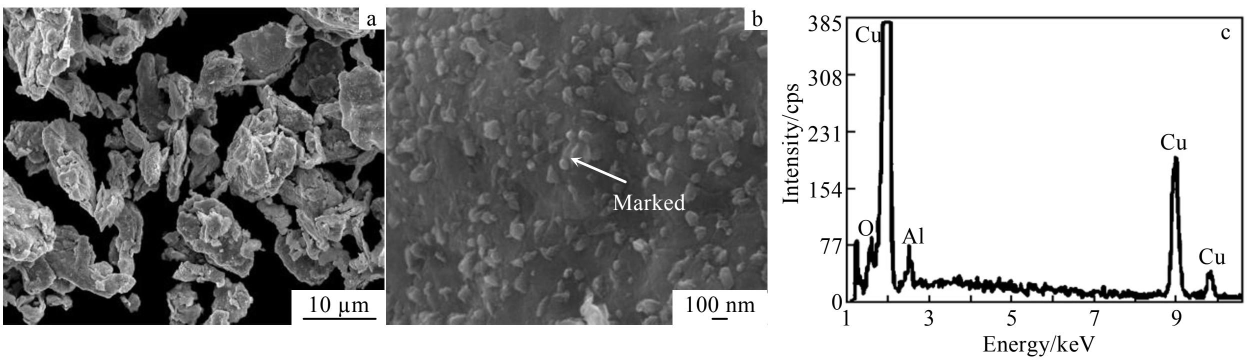

Fig.1 SEM images (a, b) and EDX spectrum (c) of the as-synthesized ADSC powder