Short Communication

Influence of deformation temperature on the microstructure and thermal stability of HPT-consolidated Cu-1%Nb alloys

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A R T I C L E   I N F O

Article history:
Received 20 August 2019
Accepted 18 September 2019
Available online 10 October 2019

Keywords:
Cu–Nb alloys
Microstructure
HPT
Thermal stability
Electrical conductivity
Tensile strength

A B S T R A C T

Ultrafine grained Cu–Nb alloys with minimal additions of 1 wt.% Nb were prepared by using HPT at ambient temperature (20 °C) and elevated temperature (200 °C). The microstructure, thermal stability and properties were investigated. The results indicated that compared with the Cu-1%Nb alloy processed at ambient temperature, the sample processed at 200 °C shows higher density and higher thermal stability, which contributed to its higher electrical conductivity and tensile strength after annealing treatment.

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1. Introduction

Copper is widely used in electrical applications, due to their excellent electrical conductivity, high machinability and ductility, as well as high cost-effective. However, the relatively low intrinsic strength restricts its applications in some fields. A large amount of effort has been made to improve the mechanical property of the purity Cu without significant conductivity deterioration. One of the most useful methods is to add immiscibility elements (i.e. Ta [1,2], Cr [3], Fe [4], W [5], and Nb [6–9]) with Cu to form copper-based solid solution, which would precipitate dispersed nanoparticles after annealing treatment and therefore enhance its mechanical property by precipitation hardening. Among them, the element of Nb has been drawn extensive attention due to the fact that this element can enhance mechanical property of Cu significantly even compared with above elements [9]. For example, a compressive yield stress of as high as 1.6 GPa has been obtained in nanostructured Cu-10 at.% Nb alloy prepared by hot compaction of the mechanically alloyed powders [9]. More recently, Youssef et al. prepared nanostructured Cu-1 at.% Nb alloy by an in situ consolidation mechanical alloying technique and a true ultimate tensile strength of about 1308 MPa was obtained [10].

It should be noticed that the formation of a copper-based solid solution in Cu–Nb system cannot be achieved just by a conventional metal casting techniques or suitable heat treatment, due to their negligible mutual solid solubility [11]. Thus, non-equilibrium techniques such as mechanical alloying were usually used to process this immiscible system [11]. And
these mechanically alloyed powders usually require a further consolidation to evaluate their physical properties such as strength and electrical conductivity. However, the final products obtained by traditional consolidation methods usually have some problems such as porosity and incomplete particulate bonding, which ruin its ductility and strength [12]. As is well known, the high pressure torsion process is one of the attractive cold consolidation techniques, due to the fact that it could provide efficient powder consolidation and improve the particulate bonding [13].

However, up to now, there are few reports for the research of Cu–Nb alloys prepared by consolidation of the mixed powders by high pressure torsion (HPT) [5]. Additionally, previous study mainly focus on samples with high volume fraction of Nb [6–9], which showed high concentration of porosity after consolidation that contributed to the reported low electrical conductivity [9]. In the present study, a nearly full density of bulk ultrafine grained Cu–Nb alloys with minimal additions of 1 wt.% Nb (corresponding to about 0.72 at.% Nb) were prepared by using HPT at ambient temperature (20 °C) and elevated temperature (200 °C). The influence of deformation temperature on the microstructure, physical properties and thermal stability was studied.

2. Experimental

The high-purity Cu powder (99.9% purity, particle size 40–60 μm) and Nb powder (99.9% purity, particle diameter 45 μm) were mixed together with Nb content of 1 wt% using a planetary ball mill. The milling media consists of type 316 stainless steel balls with a powder-to-ball weight ratio of 1:15. After milling for 15 min, the milled Cu–Nb powder was consolidated by HPT with 1 revolution and a speed of 1 rpm under the pressures of 2.5 GPa in a “groove” with a diameter of 10 mm and 0.8 mm depth. The thickness of the samples after the prior process was approximately 0.9 mm. Then these Cu–Nb samples were respectively further deformed and consolidated by HPT at room temperature (about 20 °C) (CuNb-RT) and at elevated temperature (about 200 °C) (CuNb-ET) with another 15 revolutions, and the actual thickness were respectively reduced to 0.824 mm and 0.816 mm.

3. Results and discussion

Fig. 1 is a schematic diagram, which illustrates of HPT disc and positions for microhardness measurements, and locations for tensile testing specimens and EBSD. Fig. 2(a) is the DSC curves, the results show that there are two exothermic peaks in the HPT-processed alloys, whether they were processed at ambient temperature (CuNb-RT) or at elevated temperature (CuNb-ET). The onset of the first exothermic reaction begins at about 100 °C and the peak is visible at around 340 °C. The second maximum is achieved near 412 °C. Five annealing temperatures 250, 340, 390, 480 and 525 °C were chosen upon the DSC results, the evolution of electrical conductivity and the microhardness taken from the positions close to 1/2 radius of HPT-processed disks are shown in Fig. 2(b). The temperature of 250 and 340 °C was chosen in first onset of exothermal, the results show that the microhardness decreased but the electrical conductivity increased slightly compared with as deformed samples, which is supposed caused by recovery of crystal lattice defects and relaxation of grain boundaries. The temperature of 390, 480 and 525 °C was chosen in the second onset of exothermal. When annealed at 390 °C, the electrical conductivity and the microhardness were further increased simultaneous. Some increases in electrical conductivity were expected due to the fact that the density of defects would be further decreases. The microhardness should be decrease according to the above reasons, but the fact is quite the opposite. As we known, the extended solubility could be induced by deformation in Cu–Nb systems [6,14,15]. Therefore, the increase of hardness may be caused by precipitated phases derived from the decomposition of supersaturated solid solution after annealing at this temperature. As the annealing temperature increased to 480 °C and 525 °C, the electrical conductivity was further increased, while the microhardness begins to decline. This indicates that the process of grain grow up occurred. More details are show in Fig. 2(c), in which the micro-hardness along the radius before and after annealed at 480 °C for 1 h was measured. The results show that the microhardness is gradually ascending and reach steady state along the radius, and the hardness distribution shows no distinct different between CuNb-ET and CuNb-RT. While after annealing treatment, the microhardness of CuNb-ET is much higher than CuNb-RT. In addition, the tensile tests of CuNb-RT and CuNb-ET alloys after annealed at 480 °C were also implemented, and the typical tensile curves are shown in Fig. 2(d). The results also show that the tensile strength and ductility of annealed CuNb-ET were much higher than the annealed CuNb-RT alloys.

The typical microstructures of the CuNb-RT and CuNb-ET as well as additionally annealing at 480 °C for 1 h are shown in Fig. 3, and the resulting structural parameters like distribution of grain size and misorientation are summarized in Fig. 3(e–h) and (i–l). Both of these two kinds of samples were characterized by uniform ultrafine structures with an average
Fig. 2 – (a) The enthalpy release rate during linear heating of CuNb-RT and CuNb-ET; (b) the evolution of the microhardness and electrical conductivity as a function of the annealing temperature in the sample of CuNb-RT and CuNb-ET; (c) the micro-hardness along the radius in the sample of CuNb-RT and CuNb-ET before and after annealed at 480 °C; (d) engineering stress–engineering strain curve of CuNb-RT and CuNb-ET after annealing at 480 °C for 1 h.

grain size of 0.40 μm and 0.36 μm and consisted of a large fraction of high-angle grain boundaries with the value of 67.15% and 68.10%, respectively. As is well known, the grain refinement process in copper [16] or copper alloys [17] includes: (i) the formation of dislocation cells in original grains; (ii) transformation of dislocation cells walls into sub-boundaries with small misorientations, which would separate the initial grains into individual subgrains; (iii) the evolution of low-angle sub-boundaries into highly misorientation grain boundaries. Therefore, there is reason to believe that the high fraction of high-angle boundaries contained in the deformed Cu–Nb alloys was introduced during the process of HPT. After annealing at 480 °C for 1 h, the sample of CuNb-ET showed excellent thermal stability, and the distribution of grain size and the misorientations were little changed. Nevertheless, the sample of CuNb-RT showed poor thermal stability, and its average grain size was significantly increased to 2.20 μm after annealing treatment. In addition, the distribution of misorientations also changed significantly and the number fraction of the large-angle grain boundaries decreased to 28.12%, which indicates that the number of deformation-induced high-angle boundaries reduced by grain boundary migration and merging during annealing treatment. It should be noted that the DSC peaks of the CuNb-ET are lower than that of the CuNb-RT (Fig. 2(a)), which indicates that compared with the sample of CuNb-ET, the sample of CuNb-RT stored more energy, which maybe one of the reasons that make a contribution for its poor thermal stability. In addition, considering the very low thermal stability of the HPT-processed pure copper (as low as 50 °C) [18], the effect of the segregation of Nb atoms and nanoparticles pinning at grain boundaries and triple points in the present HPT-processed Cu–Nb discs must be taken into consideration. As is well known, the nucleation of precipitate is more likely to occur at the sites containing large amount of crystal defects, such as dislocations and grain boundaries. It is reasonable to believe that compared with the sample of CuNb-RT, the segregation concentration of Nb elements on grain boundaries in CuNb-ET is higher due to the fact that the diffusion coefficient increases with the increases of temperature, which would enhance its thermal stability.

It interesting found that the electrical conductivity, tensile strength as well as ductility of CuNb-RT is relatively higher than CuNb-RT, whether with annealing treatment or not, as shown in Fig. 2(b) and (d). This phenomenon may be caused by the different densities of these two kinds of samples. In details, the density of the CuNb-ET is approximately 8.92 g/cm³, which is higher than the sample of CuNb-RT (8.89 g/cm³). As is well known, the density is in positive correlation with amount of porosities to some extent, so the sample processed at room temperature (CuNb-RT) has a higher porosity, which could act as the insulation sites and have a negative effect on electrical conductivity and tensile properties [19].
Fig. 3 – Typical microstructures (a) of CuNb-RT; (b) additionally annealing at 480 °C for 1 h of CuNb-RT; (c) CuNb-ET; (d) additionally annealing at 480 °C for 1 h of CuNb-ET; (e–h) corresponding grain size distributions and (i–l) misorientation distributions, respectively.

4. Conclusion

1. The ultrafine grained Cu-1%Nb are synthesized by HPT at ambient temperature (20 °C) and elevated temperature (200 °C).
2. Compared with the Cu-1%Nb alloy processed at ambient temperature, the sample processed at 200 °C shows higher thermal stability. And an excellent combination of high electrical conductivity of 66.4 IACS% and high tensile strength of 904 MPa has been obtained after annealed at 480 °C for 1 h.

Conflict of interests

The authors declare no conflicts of interest.

Acknowledgments

This research was supported by the National Natural Science Foundation of China (U1810109) and China Scholarship Council.

References