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Microstructure and properties of a nanocrystalline Cu-Al-NbC composite with high strength and good conductivity



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ABSTRACT

A combination of high tensile strength of 900 MPa and a good electrical conductivity of 50% International Annealed Copper Standard (IACS) was achieved in a nanocrystalline Cu-0.1 wt%Al (grain size: 96 nm) matrix composite reinforced with 6.4 vol% NbC nanoparticles (6.5 nm). The composite was produced by combining high energy mechanical milling (HEMM), sintering and powder compact extrusion. The composite was highly thermally stable that a higher electrical conductivity of 57% IACS was achieved, while maintaining a high tensile strength of 722 MPa and good ductility after annealing at 1000 °C for 5 h.

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1. Introduction

Engineering materials with excellent electrical conductivity and high microstructural stability at elevated temperatures were intensely researched over decades, among these, nanocrystalline (NC) copper reinforced with nanoparticles was demonstrated to be a promising candidate for applications such as welding electrodes, high voltage switches, combustion chamber liners and nozzle liners [1,2]. In related researches, oxide and carbide nanoparticles such as Al₂O₃, TiC, TaC and NbC were successfully introduced into NC copper matrix via high energy mechanical milling (HEMM) and thermomechanical powder consolidation [3-7]. However, nanoparticles dispersions in the Cu grains induce intensive electron scattering which inevitably results in degradation of electrical conductivity [6]. To achieve an optimal combination of electrical conductivity and strength, designing a microstructure with desired sizes of the Cu matrix grains and nanoparticles where the majority of the nanoparticles distribute at the grain boundaries was considered as an effective approach [8].

In view of the above discussion, this paper is to demonstrate that a high strength, good tensile ductility and high electrical conductivity could be achieved in a microstructure with nanocrystalline Cu-0.1 wt%Al matrix reinforced with NbC nanoparticles distributed at grain boundaries. HEMM, sintering and powder compact extrusion were performed to fabricate such a composite.

2. Experimental procedures

100 g of Cu-5 wt%Nb-0.1 wt%Al powder mixture consisting of gas atomized Cu, Nb and Al powder particles (purity: 99.9 wt%; particle sizes: Cu and Al, <45 µm; Nb, <75 µm) was milled with 1 wt% of stearic acid (SA) (purity: 99.9 wt%) in a QM-3SP4 planetary ball mill under argon protection, using 500 g of 440C stainless steel balls as milling medium. The powders were first mixed at a speed of 200 rpm for 2 h, and then milled at a speed of 500 rpm for 36 h with a 0.5 h interruption every 6 h. After screening through a -100 meshes sieve, the milled powder was die-pressed into a cylindrical compact with a pressure of 1300 MPa holding for 1 min. Subsequently, the powder compact was sintered in a vacuum furnace (pressure < 10^{-2} Pa) at 700 °C for 1 h, with a heating rate of 10 °C/min, followed by hot extrusion at 800 °C with an extrusion ratio of 25:1 to produce a rod with a diameter of 6 mm. Part of the as-extruded rod was annealed in the vacuum furnace at 1000 °C for 5 h.

The microstructure of powders and consolidated samples were characterized using X-ray diffractometry (XRD) (Panalytical, X'Pert PRO MPD) and transmission electron microcopy (TEM) (JEOL JEM2100F). The scanning speed of XRD measurement is 5° /min with a step size of 0.02° using *Cu* radiation. The TEM specimens





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were prepared by mechanical thinning to 50 μ m followed by ion milling (Gatan Model 691). Tensile tests of the rod samples were conducted on Zwick 100 tensile testing machine with a strain rate of 5 \times 10⁻⁴ s⁻¹, using dog-bone shaped tensile test specimens with a rectangular cross section of 1 \times 2 mm² and a gauge length of 15 mm. Prior to this, the electrical resistivity of the samples was measured by four-probe method using DC Resistance Meter (Tonghui TH 2515) and then converted into International Annealed Copper Standard (IACS). The fracture surfaces of the tensile tested specimens were observed by scanning electron microscopy (SEM) (NOVA 230, FEI USA).

3. Results and discussion

Fig. 1(a) and (b) shows the XRD patterns of powders and rod samples, respectively. As shown in Fig. 1(a), a small peak of Nb is evident in XRD pattern of as-milled powder, and after sintering, the peak of NbC instead of Nb appears, implying the in-situ formation of NbC by reaction of Nb and C. The reaction of Nb and C may take place at temperature higher than 700 °C with the decomposition of stearic acid during sintering, as suggested in our previous results [9]. As shown in Fig. 1(b), both Cu and NbC peaks remain in the XRD pattern of the as-extruded rod sample, the broadening



Fig. 1. XRD patterns of (a) the as-milled and heat-treated powders and (b) the as-extruded and annealed samples.



Fig. 2. TEM images showing the typical microstructure of the ex-extruded and annealed samples: (a) and (d) BF images, (b) and (e) DF images produced using NbC diffraction spots, and (e) and (f) SADPs of as-extruded and annealed samples respectively.

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