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The effect of submicron-sized initial tungsten powders on microstructure and properties of infiltrated W-25 wt.% Cu alloys

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ABSTRACT

In the present work, several W-25 wt% Cu alloys have been prepared through combined processes of high-energy ball-milling, liquid-phase sintering and infiltration, using the precursors of industrial copper powders with an average particle size of 50 μm and tungsten powders with alternative average particle size of 8 μm, 800 nm, 600 nm or 400 nm. Microstructure characteristics, relative density, hardness and electrical conductivity of the WCu alloys were investigated to elucidate the effect of initial particle size of tungsten powders. EBSD was further utilized to reveal the orientation and grain size distribution in the WCu alloys prepared by 8 μm and 400 nm-sized tungsten powders. The results showed that the WCu alloy made by 400 nm-sized tungsten powders exhibited excellent homogeneity for both sintered tungsten powders and grains, together with the highest relative density of 98.9%, the highest hardness of 230 HB, and good electrical conductivity of 48.7% IACS. Moreover, it also showed highly improved arc erosion and mechanical wear resistances.

1. Introduction

With the rapid development of electrical power system for higher voltage and larger current, more rigorous requirements for circuit breaker, which is the core part of taking on and off the grid components, are put forward. As the heart of the circuit breaker, electrical contact not only endures the current with large capacity, but also experiences the high temperature induced by arc erosion during service [1–3]. Owing to the beneficial combination of good electrical and thermal conductivity of copper, with high melting point, high hardness, low thermal expansion coefficient as well as excellent arc erosion resistance of tungsten, WCu alloys as contact materials are widely used in various kinds of vacuum circuit breaker switches [4–8]. However, the inherent mutual immiscibility, and the large discrepancy in density and melting point between copper and tungsten, make the alloy difficult to prepare through traditional casting techniques. Currently available way takes the advantage of powder metallurgy [9–11], including infiltration of a pre-sintered tungsten skeleton with copper melt [12–14] and liquid phase sintering [15,16]. Consequently, the mechanical behaviors of the resultant powder metallurgy products are determined by the particle size of raw materials to some extent. Studies have shown that ultrafine/nanocomposite powders can significantly reduce the sintering activation energy of powders and promote excellent sintering process, superior to activated sintering at the expense of thermal and electrical conductivity due to the introduction of transition metals; however, the nano-sized powders with high surface energy usually make them aggregated during sintering, resulting in blind holes in the final products [17–20]. Considering that submicron-sized powders with the particle size between micron- and nano- scales may exhibit negotiated behaviors during preparation, thereby in the present work, submicron-sized tungsten powders with alternative average particle size of 800 nm, 600 nm or 400 nm have been utilized to fabricate WCu alloys, and the traditional micron-sized (8 μm) tungsten powders were also used for comparison. The resultant microstructure and properties including relative density, hardness and electrical conductivity were evaluated. Special attention was paid to two WCu alloys made by 8 μm and 400 nm -sized tungsten powders, the crystallographic misorientation, grain size distribution, arc erosion and mechanical wear behaviors of which were investigated and discussed with detailed comparison.

2. Experimental materials and methods

Tungsten powders (purity ≥ 99.9 wt.%) with average particle sizes of 8 μm, 800 nm, 600 nm and 400 nm, as well as copper powders (purity ≥ 99.9 wt.%) of 50 μm were used as raw materials. The WCu alloys prepared by tungsten powders with individually alternative average particle size were fabricated through the following processing technique: first, mechanical alloying of nominal amount of tungsten

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powders with 25 wt.% industrial copper powders was performed in a high energy ball mill using stainless steel container and balls with ball to powder weight ratio of 5:1 for 8 h. Subsequently, the composite powders were pressed into green compacts with the dimensions of φ 21 mm × 15 mm under the pressure of 340 MPa in a XTM-108-200T Hydraulic Press. Finally, the green compacts were sintered and infiltrated at 1350 °C for 40 min under H_2 atmosphere followed by furnace cooling.

The density of the WCu alloys was measured in accordance with Archimedes' principle, and the samples were wax-sealed before measurement to eliminate the errors from surface open pores. The hardness was measured by a Brinell Hardness Tester, and the average of 5 indents was used to ensure acquisition of a reasonable value. The electrical conductivity of alloys was measured by an Eddy Current Conductivity Meter. Typical secondary electron images of the specimens were obtained on a JEOL-6700F Field Emission Scanning Electron Microscope (SEM). The local crystallographic misorientation across the section of specimens was characterized by electron backscattering diffraction (EBSD) on a Zeiss-Merlin SEM with the acceleration voltage of 20 kV. The vacuum electrical breakdown tests were conducted in a vacuum arcing indoor chamber modified by a TDR-40A Single-crystal Furnace under the voltage of 8 kV, to simulate the electrical arc erosion process of the WCu alloys for circuit break. Considering the high-frequency opening and closing of WCu alloys in service, the friction and wear experiments (wear time of 180 min, wear radius of 8 mm, rotating speed of 80 r/min and loading of 500 g) were carried out on a HT-1000 Pin-on-Disk Tester with the wear pin and disk made of corresponding investigated alloys.

3. Results and discussions

3.1. Microstructure

Fig. 1 presents the SEM micrographs and W-W contiguity values ($C_{W\text{-}W}$) [21–23] of the WCu alloys prepared by different particle-sized tungsten powders. With limited sintering necks or poor contiguity between tungsten particles, the WCu alloy made by industrial micron-sized tungsten powders (Fig. 3(a)) exhibited rather coarser and inhomogeneous feature with evident enrichment of copper. This encounter agrees with previous dilatometry results showing that the particle rearrangement was limited for large particle-sized tungsten powders [24]. Comparatively, the WCu alloys made by submicron-sized powders (Fig. 3(b-d)) presented uniformly bi-continuous microstructure for both tungsten and copper phases, with progressively improved contiguity between tungsten powders. The excellent tungsten contiguity or more sintering necks would result in improved density, hardness and strength, while with certain reduction of electric conductivity due to relative damage of contiguity for copper.

3.2. Relative density, hardness and electrical conductivity

The change of the relative density, hardness and electrical conductivity of the WCu alloys with different initial tungsten particle sizes are shown in Fig. 2. It is obvious that the relative density and hardness of the WCu alloys increased with the decrease of tungsten particle size. It has been proposed that smaller particles lead to greater capillary forces, facilitating more condensed parts through particle rearrangement [24]. Accordingly, the highest relative density of 98.9% for the WCu alloy prepared by the starting tungsten powders of 400 nm can be attributed to the sufficient rearrangement of tungsten particles in copper melt. In general, it is directly related that the hardness increases with increasing density, and microstructural homogeneity can be characterized by quantitative analysis of the hardness values for the composites [25]. Compared with the WCu alloy prepared by industrial tungsten powders, the hardness of the WCu alloy made by 400 nm tungsten powders has increased by 84%, with a slight sacrifice of electrical conductivity by 11.6%. Nevertheless, the value of electrical conductivity of the WCu alloy prepared by 400 nm tungsten powders reached 48.7% IACS (as percent of the International Annealed Copper Standard), which is still much higher than the national standard (GB/T8320-2003: 42%).

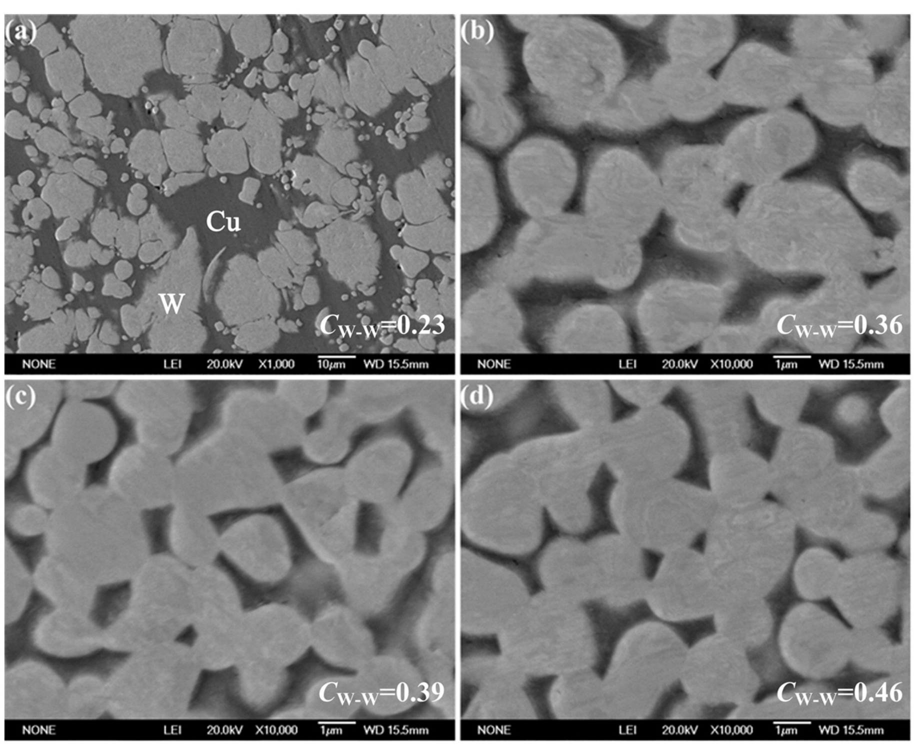

Fig. 1. SEM micrographs with inserted W-W contiguity values of the WCu alloys prepared from an initial tungsten particle size of (a) 8 μm, (b) 800 nm, (c) 600 nm, and (d) 400 nm.