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Preparation and properties of ultrafine-grained W-Cu composites reinforced with tungsten fibers

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ABSTRACT

In this work, ultrafine-grained W-Cu composites without and with reinforcement of tungsten fibers (W_f) were prepared by successive processes of mechanical alloying, cold pressing, sintering and infiltration. For W_f reinforced W-Cu composite, the introduction of W_f coated with magnetron sputtered Cr, improved the sinterability and contiguity of W_f with the neighboring W powders, eliminating porosity between W_f and the matrix and improving the interfacial strength. Further compressive tests demonstrated that, compared with conventional commercial W-Cu composite made of micron-sized tungsten powders, for the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f, the compressive strength increased dramatically by 122.9%, 43.6% and 91.3%, when deformed at room temperature, 300 °C and 500 °C, respectively. Besides, the electrical breakdown strength increased impressively by 118%, which can be ascribed to the arc dispersion from the increased fraction of grain boundaries in the ultrafine-grained structure free from blind holes.

1. Introduction

W-Cu composite, or W-Cu pseudo-alloy composed of immiscible W and Cu, exhibits good electrical conductivity, high strength as well as excellent resistance to arc erosion [1,2], making them widely used as electrical contact materials, throat linings for rocket tail nozzles, plasma facing materials and circuit chips [3,4]. Specifically, with rapid development of electrical power systems, the high-frequency breaking and connecting of the power grid under higher voltage requires higher properties of the contact materials [5]. In recent years, novel W-Cu composites made of nanosized W powders or added by reinforcements such as graphene or WC have been reported with better comprehensive properties. However, nanosized W powders are usually fabricated by chemical reactions such as co-precipitation, free drying, sol-gel procedure and nitridation-denitridation method [6,7], the drawbacks of which lie in the complicated processes and few quantities deficient for industrial products; besides, nanosized powders with high surface energy usually introduces agglomeration and arch bridge effect, leading to formation of blind holes and reduction of electrical conductivity [8]. In this work accordingly, submicron-sized W powders were utilized as initial feedstock, followed by further mechanical alloying, and tungsten fibers (W_f) were used as the reinforcements to further improve the mechanical properties of the composite. Besides, surface modification of W_f by magnetron sputtered Cr-coatings was also anticipated to improve the interfacial bonding strength.

2. Experimental

W_f networks as shown in Fig. 1(a) were immersed in 40% HF liquor for 15 min to remove the surface oxide, followed by rinse in alcohol and deionized water. The W_f were also evaluated by room temperature tensile test to present a high fracture strength of 3055.78 MPa (Fig. 1(b)). The surface-modified W_f networks with ~100 nm thick Cr-coatings were obtained by magnetron sputtering as confirmed in Fig. S1. W powders (W_p, 500 nm, purity >99.8 wt%) and 20 wt% Cu powders (Cu_p, 50–70 μm, purity >99.8 wt%) were milled at a rotation speed of 400 r/min with ball to powder weight ratio of 5:1 for 8 h in a KQM-YB/B planetary ball milling machine. The powders would be fractured and cold welded to further refine the size of the powder. Also, mutual solubility between W and Cu would occur and be extended by mechanical alloying [9]. The green body assembled with milled powders (Fig. 1(c, d)) and W_f were cold pressed into a 51 mm × 13 mm composite compact at 250 MPa under an XTM-40 forming hydraulic pressing machine, as illustrated in Fig. 1(e). The distance between W_f networks was 0.5 mm, and the content of the W_f

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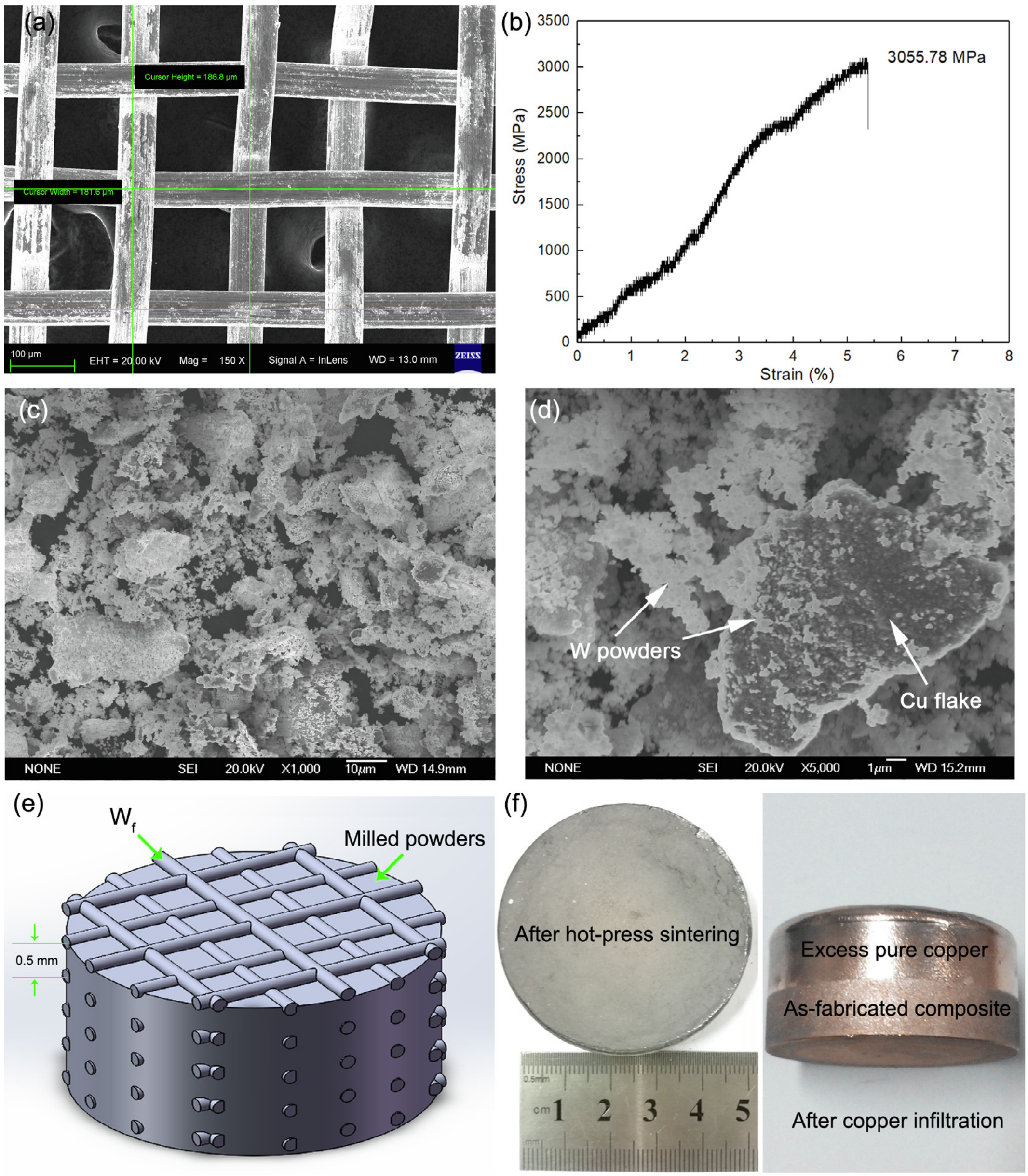

Fig. 1. SEM image of introduced W_f network (a), tensile stress-strain curve of the W_f (b), mixed powders after milling (c, d), schematic architecture of W_f reinforced green body (e) and sample views after hot-press sintering or final step of infiltration. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

networks in the composite green body was calculated to be 3.3 wt %. Also, the milled powders without W_f were cold pressed into a green compact for comparison.

Subsequently, under hydrogen atmosphere, the three samples were firstly hot-press sintered under a pressure of 5 MPa for 50 min at 970 °C, then held at 1350 °C for 2 h, and finally cooled to room temperature with furnace cooling (Fig. 1(f)). After that, liquid infiltration route of molten pure Cu block into the sintered performs was carried out at 1350 °C for 2 h in hydrogen atmosphere followed by furnace cooling (Fig. 1(f)). Densities of the three ultrafine-grained W-Cu composites were measured according to Archimedes principle, and electrical conductivity was measured by an Eddy Current Conductivity Meter. Details of wear resistance, compressive and electrical breakdown tests can be found in our previously reported work [10]. Secondary electron images were obtained using Zeiss Merlin or JEOL-6700F scanning electron microscopes (SEM).

3. Results analysis and discussion

Fig. 2(a) shows the cross-section view of the ultrafine-grained W-Cu composite reinforced with uncoated W_f. It can be seen that W_f and tungsten particles (W_p) exhibited gray contrast, while Cu presented darker contrast; meanwhile, blind holes with black contrast were also detected. The blind holes indicated the constraining grain rearrangement during sintering. Apparently, Cu aggregation can be easily found in the sample, especially at the vicinity of interface between W_f and W_p. This is considered to form due to the deficient sintering between W_f and W_p, and subsequent filling of the interstice by infiltrated Cu, which would deteriorate the strengthening anticipation of W_f reinforced W_p architecture [11]. Studies have shown that trace addition of transitional elements such as Cr, Fe and Ni into W-Cu composites effectively increased the formation of sintering necks and improved their wettability among W_p [12]. Therefore, W_f modified by magnetron sputtered Cr-

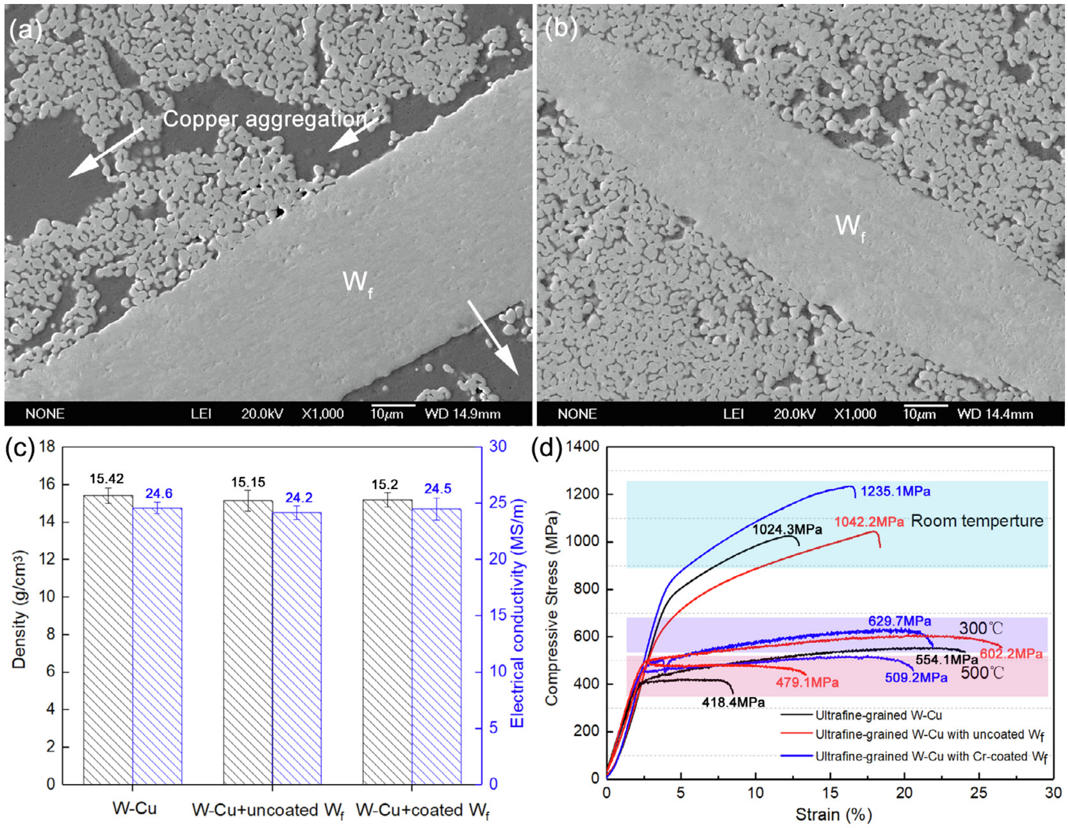

Fig. 2. SEM images of the ultrafine-grained W-Cu composites reinforced with uncoated W_f (a) and Cr-coated W_f (b), comparison of density and electrical conductivity (c), compressive stress-strain curves (d) of the three composites.

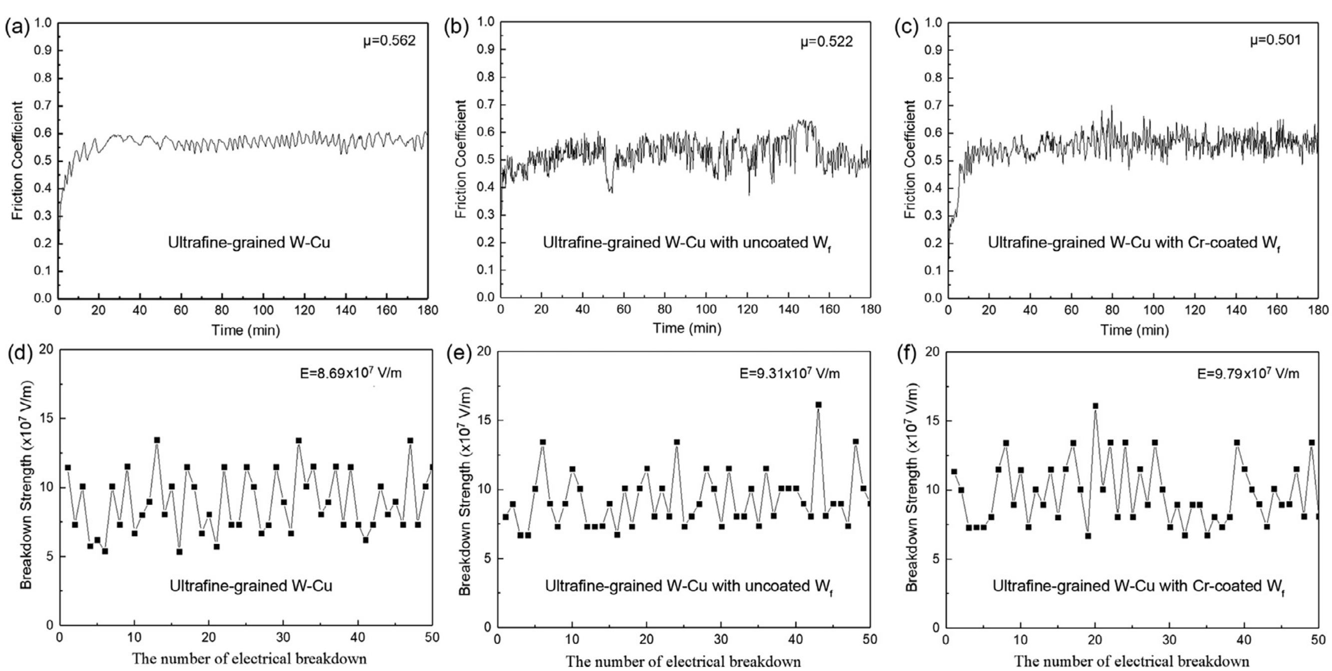

Fig. 3. Friction coefficient curves (a–c) as well as the relationship between electrical breakdown strength and breakdown times (d–f) of the three composites.

coatings were introduced to ameliorate the interfacial state between W_f and W_p [13–15]. It can be seen from Fig. 2(b) that Cu aggregation and blind holes have been dramatically decreased for the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f. As shown in Fig. 2(c), there is no significant difference in electrical conductivity for the three composite, as the continuity of the dominant Cu phase for electron transportation was not disrupted. The values are higher than the national standard of 24.02

MS/m according to GB/T8320-2003. However, the introduction of W_f slightly reduced the density of the final composites, since W_f hindered the rearrangement of powders during sintering.

As W-Cu composites utilized as electrical contacts usually endure transient high temperature and contact press during service [16], therefore, compressive stress-strain curves of the three composites along radial direction under various temperatures were further verified as shown in Fig. 2(d). The results showed that, with the increase of temperature, the increased softening rate resulted in an obvious decrease of stress endurance. Compared with the conventional commercial W-Cu composite made of micron-sized tungsten powders [10], all the three composites reported here exhibited improved compressive strength at any definite temperature. Especially, for the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f, the compressive strength increased dramatically by 122.9%, 43.6% and 91.3%, respectively when deformed at room temperature, 300 °C and 500 °C. Meanwhile, this composite exhibited high plastic deformation strain of over 13% at all temperatures. The superior compressive properties can be ascribed to its excellent interfacial bonding state between W_f and the surrounding matrix, which ensured a sufficient load transfer across the interfaces.

Considering the breaking and connecting of the frictional switch between contacts, wear resistance test was conducted. Compared with the ultrafine-grained W-Cu composite (Fig. 3(a)), the friction coefficient curves of the ultrafine-grained W-Cu reinforced with W_f (Fig. 3(b, c)) exhibited more fluctuated serrations due to the presence of W_f during stable wear period. All the three composites behaved higher wear resistance due to grain refining effect [17], compared with the friction coefficient (~0.72) of the conventional commercial W-Cu composite made of micron-sized tungsten powders [10]. To simulate the arc erosion process of W-Cu composites used as electrical contact materials, electrical breakdown tests were carried out for 50 times for each composite. In comparison with the conventional commercial W-Cu composite made of micron-sized tungsten powders (4.50×10^7 V/m [10]), all of the three composites exhibited much higher breakdown strength. Especially, the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f increased dramatically by 118%, which can be ascribed to the arc dispersion from the increased fraction of grain boundaries in the ultrafine-grained structure free from blind holes [17,18], which provides useful clues regarding the design of novel W-Cu composites and is transferrable to other refractory metals for property improvement.

4. Conclusions

In conclusion, three novel ultrafine-grained W-Cu composites without and with reinforcement of tungsten fibers (W_f) have been prepared by successive processes of mechanical alloying, pre-assembling, cold press, hot-press sintering and infiltration. The main investigations can be summarized as follows:

(1) For W_f reinforced W-Cu composites, the introduction of W_f coated with Cr layer by magnetron sputtering, improves the sinterability and contiguity of W_f with the neighboring W powders, eliminating porosity between W_f and the matrix and improving the interfacial strength.
(2) Compared with the conventional commercial W-Cu composite, the ultrafine-grained W-Cu composite, as well as the ultrafine-grained W-Cu composites reinforced with uncoated and Cr-coated W_f, exhibited superior properties including compressive strength, wear resistance and arc erosion resistance.
(3) Compared with the conventional commercial W-Cu composite, for the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f especially, the compressive strength increased dramatically by 122.9%, 43.6% and 91.3%, respectively when deformed at room temperature, 300 °C and 500 °C, due to its excellent interfacial bonding state between W_f and the surrounding matrix, which ensured a sufficient load transfer across the interfaces.
(4) Compared with the conventional commercial W-Cu composite, for the ultrafine-grained W-Cu composite reinforced with Cr-coated W_f, the friction coefficient and the breakdown strength decreased by 30.4% and increased by 118%, respectively, providing useful clues regarding the design of novel W-Cu composites.

Conflict of interests

The authors declared that they have no conflicts of interest to this work.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2019.01.146.

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