Int. Journal of Refractory Metals and Hard Materials 42 (2014) 51–56

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Sintering behavior of W–30Cu composite powder prepared by electroless plating

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ARTICLE INFO

Article history:
Received 13 July 2013
Accepted 17 October 2013

Keywords:
Electroless plating
W–Cu composite
Sintering behavior

ABSTRACT

Powder metallurgy technique was employed to prepare W–30 wt.% Cu composite through a chemical procedure. This includes powder pre-treatment followed by deposition of electroless Cu plating on the surface of the pre-treated W powder. The composite powder and W–30Cu composite were characterized by X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). Cold compaction was carried out under pressures ranging from 200 MPa to 600 MPa while sintering at 850 °C, 1000 °C and 1200 °C. The relative density, hardness, compressive strength, and electrical conductivity of the sintered samples were investigated. The results show that the relative sintered density of the titled composites increased with the sintering temperature. However, in solid sintering, the relative density increased with pressure. At 1200 °C and 400 MPa, the liquid-sintered specimen exhibited optimum performance, with the relative density reaching as high as 95.04% and superior electrical conductivity of IACS 53.24%, which doubles the national average of 26.77%. The FE-SEM microstructure evaluation of the sintered compacts showed homogenous dispersion of Cu and W and a Cu network all over the structure.

1. Introduction

The properties of W–Cu composites, which are a combination of the high temperature strength of W and high thermal and electrical conductivities of Cu, make them appropriate candidates in a wide range of applications [1–3]. These materials can be used as arcing resistant electrodes, heavy-duty electronic contacts, heat sinks in high power microelectronic devices, welding electrodes for electro discharge machining, and divertor plates for fusion reactors [4,5]. The general method used for the fabrication of W–Cu composites is the infiltration of Cu into W skeleton. However, a homogeneous organizational structure with high weight percentage of W is hard to achieve in this process because of the difficulty in obtaining a uniform, porous W skeleton. The liquid-phase sintering of mixed powders is widely applied to fabricate the W–Cu composites. Unfortunately, W–Cu system exhibits mutual insolubility or negligible solubility, poor wettability, and high contact angle, such that its compacts show very poor sintering ability. Therefore, the fabrication of W–Cu composites with high relative densities and homogeneous microstructure is difficult [6–8]. In order to increase the wettability or decrease the contact angle of Cu and W for achieving full density, activators, such as Ni, Fe, and Co, have been used as the sintering aids for W–Cu composites. The challenge with this approach is that the sintering aids results in the deterioration of electrical and thermal conductivities of the composite materials [7–11]. The preparation of ultrafine nanosized particles by mechanical alloying [12,13] and activated sintering has been developed to improve the sintering strength and homogeneity of W–Cu composites. However, the mechanical alloying impurities, such as Fe and Co are introduced into the composites during severe milling from milling jar (stainless) and balls (cemented carbide or stainless). This also decreases the thermal and electrical conductivities of W–Cu composites. In recent years, a number of coating processes, such as electroless Ni [14,15] and Ni–P [16] plating have been developed essentially to address the above-mentioned drawbacks and improve the wettability of powder particles. The electroless Ni plating of metallic powders enhances composite homogeneity degree during pressing/sintering/infiltration. In this study therefore, W–30Cu composite powders were prepared by electroless plating and the W–30Cu composite obtained by conventional powder metallurgy method. The microstructure of the W–30Cu composites was observed by FESEM. The mechanical properties and electrical conductivity of the composites at different sintering conditions were also investigated.

2. Experimental

The W powder with Fisher particle size of 3 μm was activated by chemical activation pre-treatment. The surface of the activated W powder has some catalytic activity that is beneficial for coating with Cu by electroless plating. Activated W powders were put into

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http://dx.doi.org/10.1016/j.ijrmhm.2013.10.012

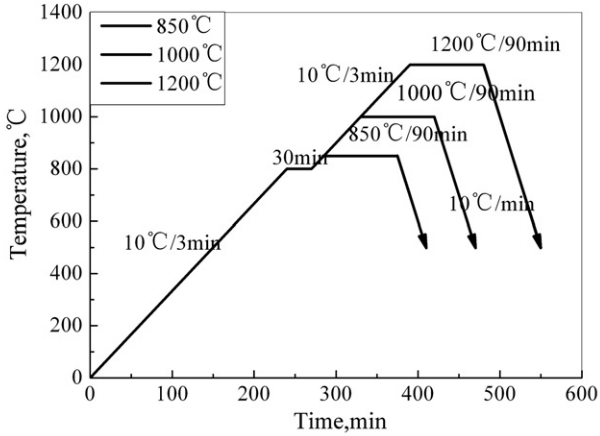

Fig. 1. Heating cycle for sintering.

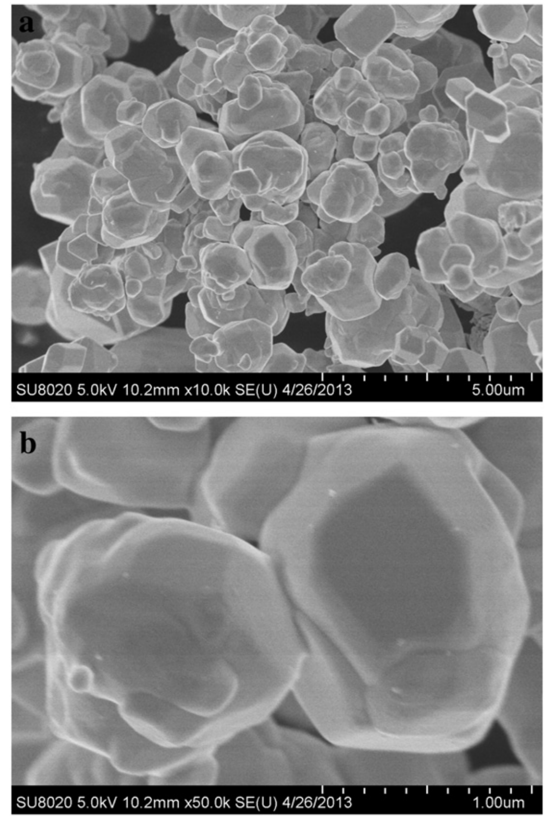

Fig. 3. FESEM photographs of the previous W powders.

the prepared Cu-plating solution for the electroless plating process. The plating solution consisted of copper sulfate, EDTA-2Na and 2,2′-bipyridine. During the electroless plating, the pH of the plating solution was adjusted from 11 to 13 with sodium hydroxide in order to control the rate of reaction. The amount of the copper sulfate in the plating solution was controlled to achieve about 30% Cu content for the W–Cu composite powders. The plating solution was continuously stirred to ensure a uniform dispersion of W powders during the reaction. The electroless plating was performed in a thermostat water bath at controlled temperature of 60 °C. After the electroless plating, W–30Cu composite powders were cleaned several times with de-ionized water until the residual liquid was clear. The composites were then dried in the oven at 50 °C. The prepared W–Cu composite powders were compacted into 32×8 mm dimension under various pressures of 200, 300, 400, 500, and 600 MPa.

The obtained green compacts were sintered in H_2 in a tubular sintering furnace at a heating rate of 10 °C/3 min. As depicted in Fig. 1, the samples were divided into three groups and subjected to sintering process at different temperature. The temperature of the first group was raised to 850 °C for 90 min for solid-phase sintering while those of the second and the third group were raised to 1000 °C, and 1200 °C respectively for 90 min, to conduct liquid-phase sintering. In order to facilitate solid phase sintering, the samples were first heated to 800 °C and kept at the temperature for 30 min. This is necessary to exclude any moisture content and discharge any captured gases in the pores.

W–30Cu composite powder was characterized by XRD and FE-SEM. The relative density of the sintered W–30Cu composite was determined by Archimedes principle. FE-SEM was employed to observe the specimen's microstructure. Vickers micro-hardness was measured on polished sections as the average of 10 readings, using MH-3L micro-hardness tester along the cross-sectional surface of the specimen, with a load of 100 gf holding for 15 s. Compression test was performed using MTS-809 Axial/Torsional Test System machine and the electrical resistivity measured using a four-wire method.

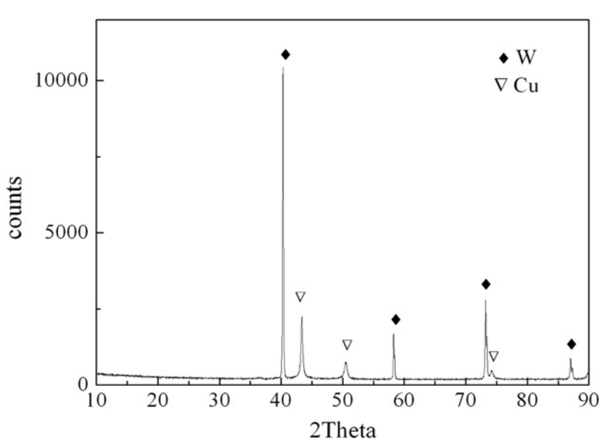

Fig. 2. XRD pattern of W–30Cu composite powder.

3. Results and discussion

3.1. W–30Cu composite powder

The XRD pattern of the W–30Cu composite powder is displayed in Fig. 2. The pattern reveals the absence of other element's diffraction peaks, which indicates that the powders consist of W and Cu phases. Therefore, it can be concluded that the electroless plating did not introduce impurities into the W–30Cu composite powder.

Fig. 3 presents the FESEM micrographs of the original W before pre-treatment. As can be seen in the figure, the surface of the W particles is clean and smooth and has a polygonal structure. On the other hand, Fig. 4 illustrates the FESEM photographs after the electroless plating of the investigated W–Cu composite powders. Inspection of Fig. 4(a), with the higher magnification morphology shown in Fig. 4(b), indicates that almost no Cu uncoated-W particles were developed. Cu coatings on the surface