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A feasibility study of W-Cu composites production by high pressure compression of tungsten powder

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ABSTRACT

For manufacturing a heavy duty W-Cu composite, a porous tungsten skeleton is required; which later can be filled by molten copper via infiltration technique. The compression force usually up to 200 MPa can be provided by cold isostatic press (CIP) and the temperatures used for sintering the green compacts are more 2000 °C. However, in this research, high pressure within the range of 200 to 663 MPa was used to produce high density green specimens (60-80%) by CIP while sintering was carried out at a moderate temperature of 1550 °C. The tungsten skeletons were infiltrated with molten copper at 1300 °C.
The reduction of sintering temperature from over 2000 °C to 1550 °C for a highly densified W-skeleton not only resulted into a successful production of W-Cu composites but also the obtained physical and mechanical properties of these composites are comparable to those obtained for lower compaction pressures and sintering temperature higher than 2000 °C.

1. Introduction

Due to the high thermal and electrical conductivity of copper and the low coefficient of thermal expansion and high arc erosion resistance of tungsten, these elements are very good candidates for production of composites having suitable thermo-electrical and arc resistance properties [1–3]. Tungsten-copper composites are widely used for ultra-high voltage electric contacts, arc resistance electrodes, electrodes for electrical discharging machining and heat-sink materials for high density integrated circuits [1–3].These composites are also used in high temperature environments for their high melting points, high thermal shock resistance and high ablation resistance [4,5].

Composites with 10 to 40 wt% copper are commonly produced by infiltration technique; while at higher copper contents, a pure P/M route is used, so that the two powders are blended, pressed, and subsequently sintered in solid state [6].

The desired density of the sintered tungsten skeleton suitable for infiltration for some heavy duty applications is as high as about 80% of theoretical density [7–9]. In its typical production route, e.g. a green density of 62% of the theoretical density is obtained for a compact made from a powder with Fisher sub sieve size of 6.70 μm under a compaction pressure of 250 MPa. Extra densification takes place during the sintering. For such a densification it is necessary to sinter the W-compacts at a temperature as high as 2150 °C for about 4 hours [7].

Below 1900 °C usually little densification for tungsten compacts occurs, unless very long sintering time is applied [6]. Therefore adopting temperatures lower than 1900 °C for sintering the tungsten green compacts implies the use of very high consolidation forces. Meanwhile, since grain boundary diffusion is the predominant mechanism of material transport during the sintering of tungsten in the temperature range of 1100-1500 °C [10,11], sintering at this range of temperature can not lead to a suitable densification, unless an ultra-high pressure is used [12]. In this research, a medium sintering temperature of 1550 °C but

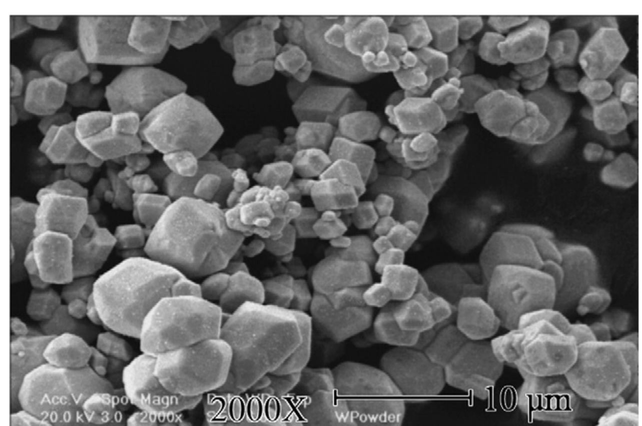

Fig. 1. Typical SEM microgragh of the initial tungsten powder.

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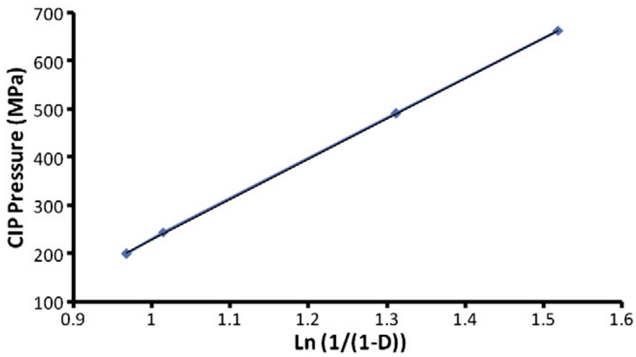

Fig. 2. Pressure of CIP compaction vs green density of the compacts.

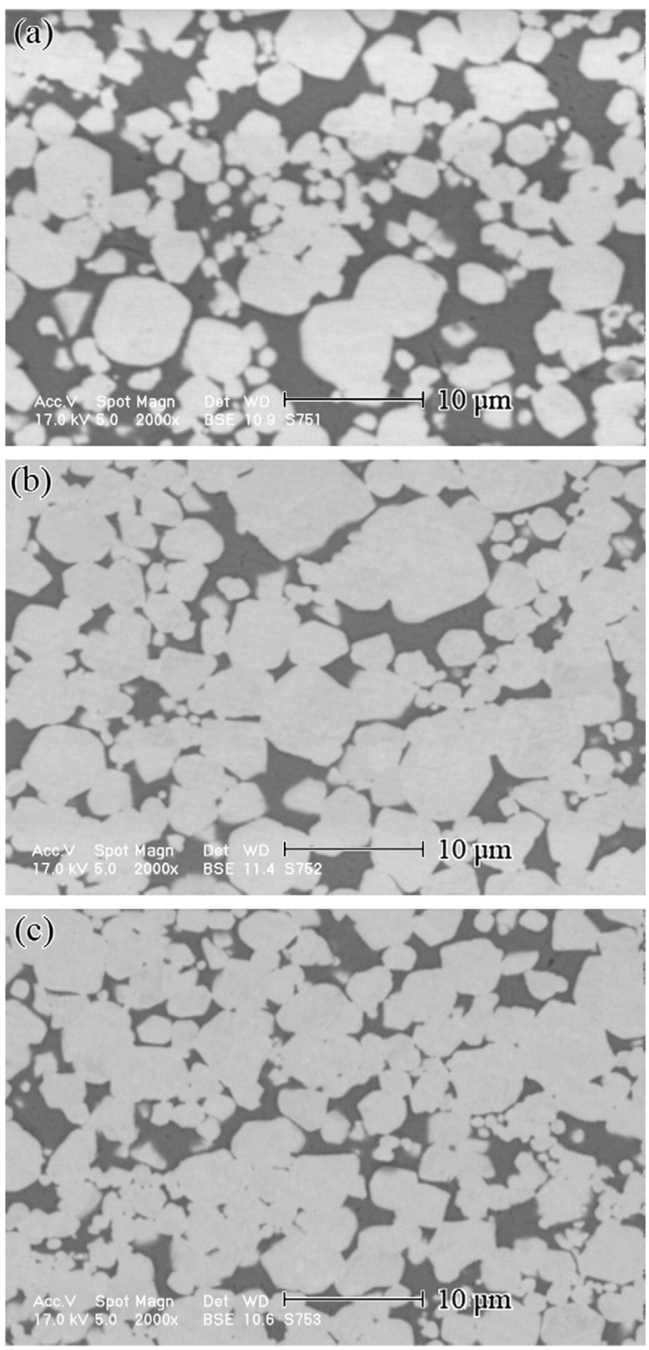

Fig. 3. Infiltrated green tungsten specimens without sintering compressed at: (a) 245 MPa, (b) 491 MPa and (c) 663 MPa.

high compaction forces were used to study the effects on the microstructure and consequently on the properties of the produced W-Cu composites.

2. Experimental procedure

Tungsten powder with Fisher sub sieve size of 6 μm was used as initial powder. As it is shown in Fig. 1, this powder has polygonal morphology. The powder was compacted in cylindrical rubber molds of 20 mm diameter and height of 20 mm. Compaction was carried out by cold isostatic pressing in a pressure range of 200 to 663 MPa. In addition, some longer specimens were produced at 663 MPa for tensile test. The tungsten compacts were sintered in a tube furnace for 4 hours at 1550 °C in a high-purity hydrogen atmosphere. Apparent density and porosity of sintered specimens were measured by Archimedes water immersion method according to ASTM B328 standard. All green and sintered specimens were subjected to pressureless infiltration of molten copper. For this purpose each individual tungsten skeleton and equivalent amount of electrical copper were put in a alumina crucible adjacent together and were heated up to 1300 °C in hydrogen atmosphere and were kept at this temperature for 1 hour. SEM and quantitative metallography were used for studying the detailed microstructure of specimens.

3. Results and Discussion

The relation between the compacting pressure and the green density of the compacts is roughly expressed by Heckles equation (Eq. (1)) [13].

$$P = \frac{\ln[1/(1-D)]-A}{K} \tag{1}$$

Where D is the fraction of theoretical density, P the applied pressure, K proportionality constant related to yield strength of metal and A is a material dependent constant. Using the above relation for plotting P versus Ln[1/(1-D)], one can obtain K and A values, which

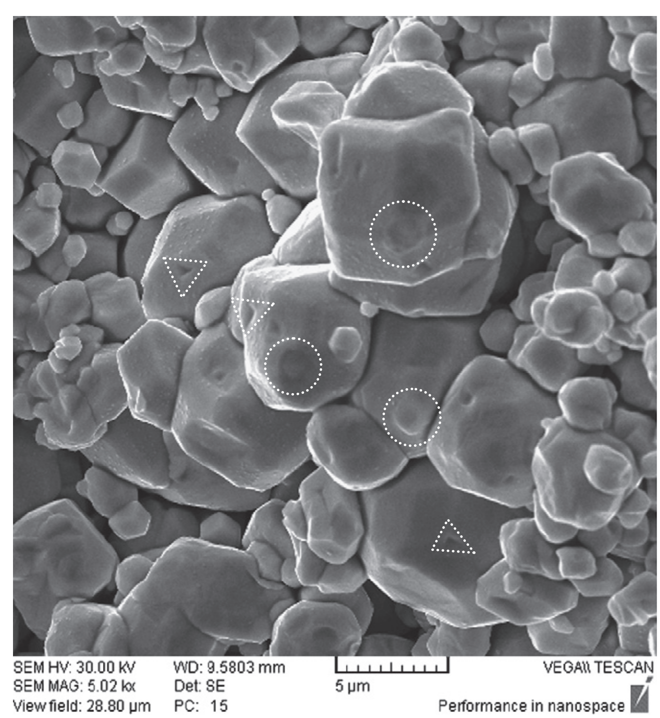

Fig. 4. SE-SEM micrograph of fracture surface of specimen compacted at 663 MPa; circle: example of tips flattening, triangle: example of tips penetrations.

according to Fig. 2 are 9.84×10^{-4} MPa^{-1} and 0.79 respectively in this work.

According to Radomysei'skii, the phenomena that occur during consolidation of the powder may be separated into several sequential stages: slip of non-deformed particles, deformation in the contact regions, and particle extrusion in the inter-connected pores [14].

Microstructural variation of green specimens due to pressure increase, illustrated in Fig. 3 proves that no particle fractured and no major plastic deformation of tungsten powder occurred. SEM micrograph of fracture surface of green specimen after compression at 663 MPa is shown in Fig. 4. As a result of high compression pressure, tungsten particles have undergone plastic deformation at contact regions. Particles that had had close contact with each other has deformed in these two ways: When two quite round surfaces are adjacent, particles' surface gets flat at contact regions. On the other hand, when the sharp tip of a particle especially small one is adjacent to a fairly round or flat surface, sharp tip can penetrate. These two phenomena are illustrated in Fig. 4. Therefore consolidation mechanism of tungsten powder is slip of non-deformed particles and deformation in the contact regions. In other words, the compaction pressures used in this research were not large enough to cause entire plastic deformation or fracturing of tungsten particles.

SEM micrographs of sintered and infiltrated specimens are shown in Figs. 5 and 6 respectively. According to these micrographs, tungsten particles have formed fairly wide necks and also changed from faceted polygonal shape observed in the initial powder (Fig. 1) to non-faceted

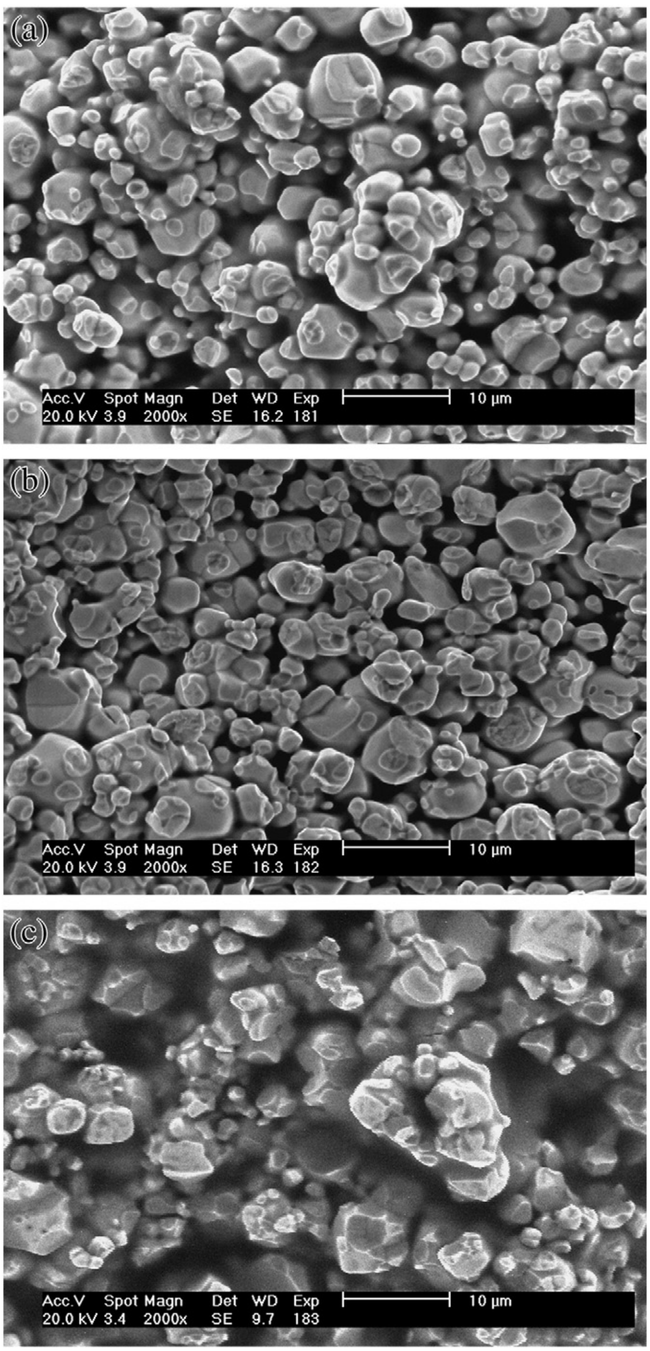

Fig. 5. SE-SEM micrographs of fracture surface of specimens compacted at (a) 245 MPa, (b) 491 MPa and (c) 663 MPa and sintered at 1550 °C.

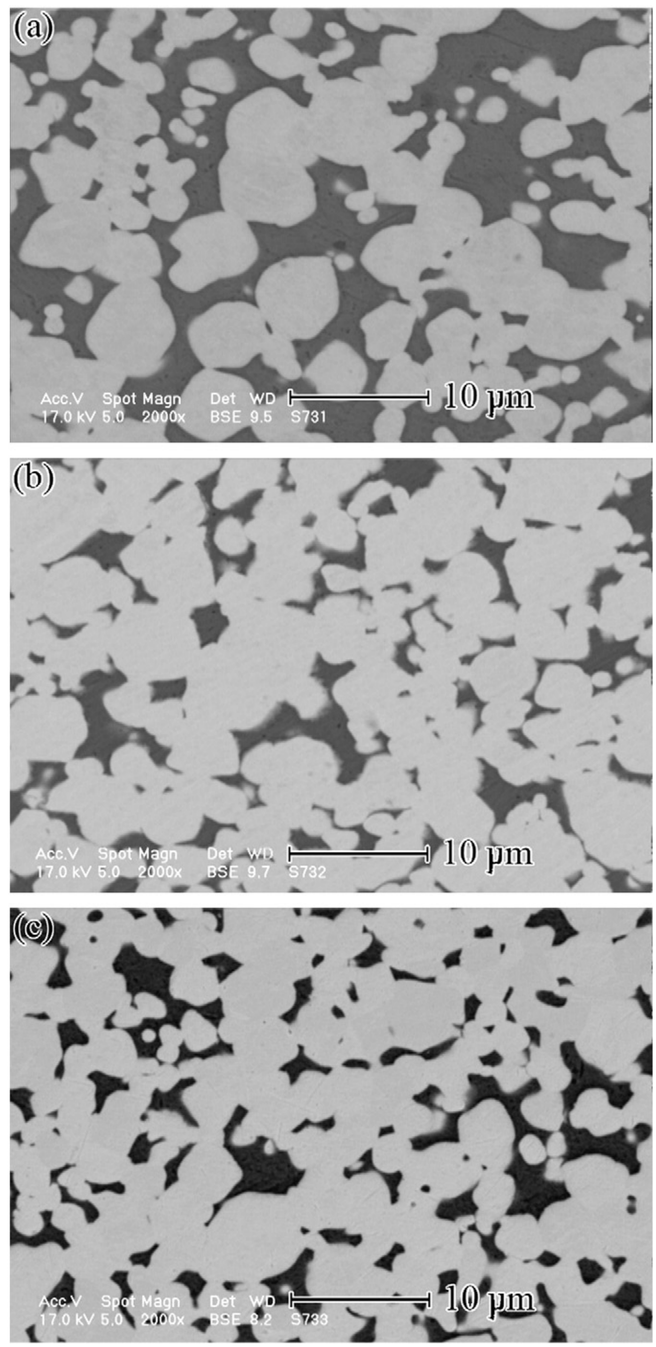

Fig. 6. BS-SEM micrographs of infiltrated tungsten specimens after sintering at 1550 °C for 4 hours compressed at: (a) 245 MPa, (b) 491 MPa and (c) 663 MPa.

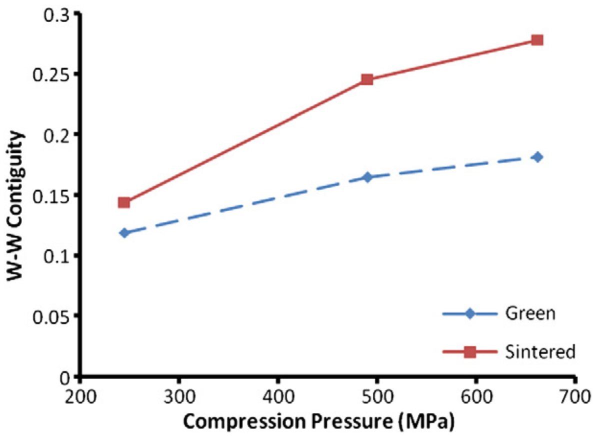

Fig. 7. W-W contiguity change vs. compression pressure for green and sintered specimens.

globular shapes. This is an indication of the fact that surface diffusion sintering mechanism was active and led to neck formation and removal of the facets during sintering of the W-particles.

Contiguity of tungsten particles is the ratio of W-W interface to entire interfaces of W-W and W-Cu. Tungsten contiguity is proportional to neck size ratio and expresses sintering progress. Fig. 7 illustrates the contiguity variation for green and sintered specimens vs. compression pressure. Contiguity increases with compression pressure. Moreover, sintering produces a supplementary effect on contiguity.

Since sintering was carried out at a relatively low temperature (i.e. 1550 °C), the only active atomistic mass transport mechanisms for tungsten can be surface and grain boundary diffusions [15]. Surface diffusion produces neck growth without a change in particle spacing. In other words, since mass flow originates and terminates at particle surface, no shrinkage or densification can be expected. Due to compression, tungsten particles undergo plastic deformation only at contact regions and the entire surface remains fairly unchanged. Therefore surface diffusion causes transportation of equal mass for all specimens irrespective to green density amount. This mass covers more surface and form wider necks in closely packed specimens, so neck growth and contiguity increase will be proportional to compression pressure (see Fig. 7).

Grain boundary diffusion is a bulk transport mechanism and contributes in densification [15]. Grain boundaries form in the sinter bonds between individual particles due to misaligned crystals. As illustrated in Fig. 8, sintering of green compacts at 1550 °C led to slight densifications (<2%), and by increasing the green density, densification through sintering even decreased. As mentioned above, by increasing compression pressure extensive contacts formed between particles and diffusion paths for grain boundary diffusion became longer so that the densification proceeded more slowly. However, the

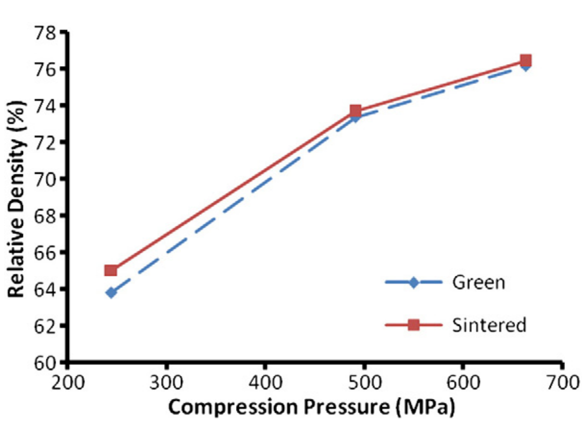

Fig. 8. Density vs. compression pressure for green and sintered specimens.

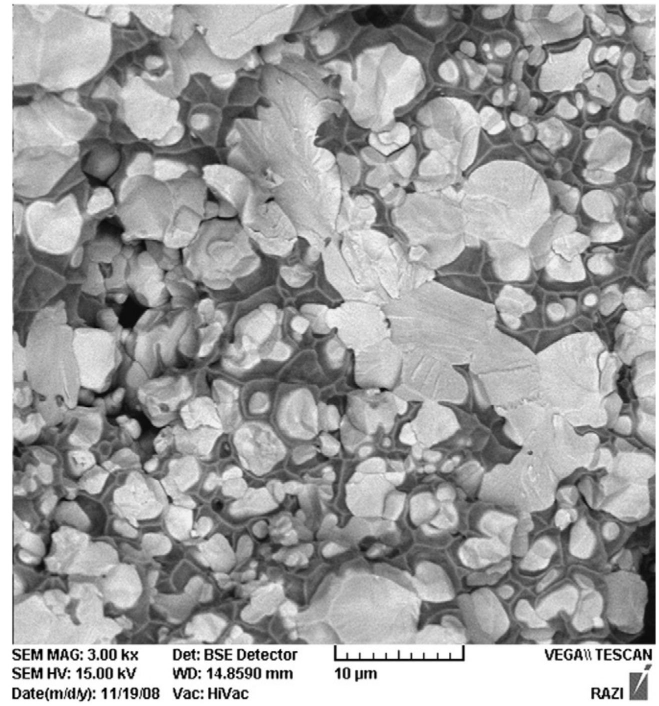

Fig. 9. Typical BS-SEM micrograph of a fracture surface of infilterated specimen after compression at 663 MPa and sintering at 1550 °C for 4 h.

negligible amount of increase in the relative densities demonstrates that the sintering temperature was not high enough to activate volume diffusion to accelerate densification of tungsten compacts.

A few long specimens prepared by compacting the W-powder at 663 MPa; sintered at 1550 °C for 4 hours and then infiltrated by copper melt at 1300 °C. These specimens underwent tensile tests. The mean ultimate tensile strength obtained was 605 MPa. This strength is even more than the strength of conventionally produced composites at high sintering temperature and low compression pressure [6,7].

Fracture surface of tensile specimen is shown in Fig. 9. Tungsten particles mainly have demonstrated transgranular brittle fracture. The absence of interface fracture of the particles (i.e. necks) is an indication of the fact that these interfaces are strong enough to withstand crack propagation.

4. Conclusions

1. The results of this research show that it is possible to produce tungsten copper composite with a density as high as 17.2 gr/cm^3 by applying high pressure for compaction of W-particles and using moderate sintering temperature lower than the temperatures used conventionally. By this way, high temperatures (e.g. 2200 °C) are not needed for sintering of tungsten compacts.
2. Tungsten copper composites produced by a compaction pressure of 663 MPa, sintering of the tungsten skeleton at 1550 °C for 4 hours and copper infiltration at 1300 °C have comparable and even better strength than those composites produced conventionally.

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