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Fabrication of W–Cu functionally graded material with improved mechanical strength

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ABSTRACT

W–Cu functionally graded material (FGM) is prepared by a novel method of high-gravity combustion synthesis and melt-infiltration in a short time. In the W–Cu FGM, W grains are micron-sized and partially sintered to form an incomplete net structure, the Cu grains surrounded by W are about 50 nm, and lots of dislocations exist in the Cu phase. The W-rich layer shows both better strength and plasticity than the commercial W–Cu composite with a similar composition prepared by a conventional infiltration method.

1. Introduction

Tungsten (W) is a promising plasma facing material (PFM) in the international thermonuclear experimental reactor (ITER) due to its high melting point, high sputtering resistance and low tritium retention [1,2]. For this fusion application, W is commonly joined with a heat sink material, such as copper (Cu), to improve the heat transfer performance [3]. Because of the large difference in the coefficient of thermal expansion ($\alpha_{Cu} \approx 4\alpha_W$), the joint of W and Cu endures high thermal stresses when exposed to high heat loads. Introducing a W–Cu functionally graded material (FGM) between W and Cu is regarded as an effective method to reduce the interfacial thermal stress with a relatively smooth spatial variation [4–6], which has been demonstrated by numerical simulations [7]. However, distinct differences in the melting point and the insolubility between W and Cu make it difficult to fabricate W–Cu FGMs. Many results about fabricating W–Cu composites have been reported, including laser sintering [8], infiltration method [9], resistance sintering [10], mechanical alloying [11], liquid-phase sintering [12] and plasma spraying [13]. All of the above processes are complex and require high energy input.

Now, we develop a new method of high-gravity combustion synthesis and melt-infiltration to fabricate W/Cu FGMs [14]. By combustion synthesis in high-gravity field, Zhao et al. have prepared ceramic-lined pipes, $TiC–TiB_2$ and Al_2O_3/ZrO_2 composite ceramics with high density (~98%) and high bending strength (1060 MPa) [15]. In this work, the conventional combustion synthesis, centrifugal method which is used to induce high-gravity, and melt-infiltration are combined for the first time by placing a designed graded W–Cu powder bed under the thermite for W–Cu FGMs preparation. It is expected that a high-gravity field would facilitate the melt-infiltration process, and thus, the prepared W–Cu FGM obtains a high density and improved mechanical properties. This new method is advantageous in terms of low-cost, simple operation and energy efficiency. The heat for producing Cu melt is provided from the exothermic aluminothermic reaction of $2Al + 3CuO = 3Cu + Al_2O_3$, and no external heating source is needed for the whole process.

2. Experimental procedure

Commercial powders of W, Cu and thermite (composed of CuO and Al) were used. Both the W and Cu powders had a purity >99.5% and sizes of ~3 μm. A commercial W–Cu composite containing 75 wt% W and 25 wt% Cu prepared by the conventional infiltration method was used for comparison. W–Cu blended powders with different chemical compositions of 90 wt%W–10 wt%Cu (W–10Cu), W–20Cu, W–30Cu, W–40Cu and W–50Cu were homogenized by rolling mill for 0.5 h. Each batch of ~20 g W–Cu blended powders was loaded into a graphite crucible of an inner diameter of 40 mm and a height of 55 mm. 200 g cold-pressed thermite compact with a porosity of nearly 50% was placed onto the W/Cu powders as illustrated in Fig. 1, where the direction of the arrow means the high-gravity direction. The graphite crucible was coated by carbon felt and placed in a steel vessel, and the vessel was mounted on a rotator. The high-gravity combustion synthesis was carried out in a closed reaction chamber, which was evacuated to a vacuum of

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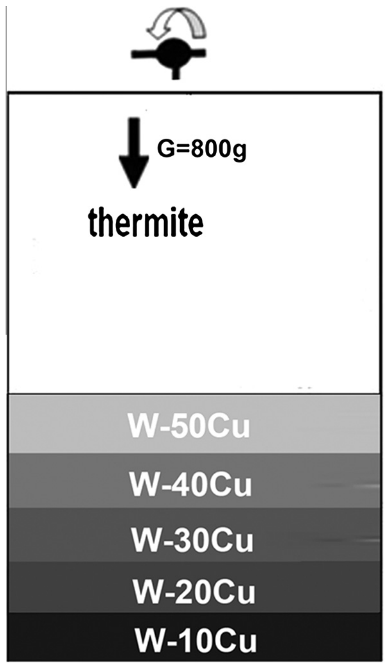

Fig. 1. Schematic diagram of fabricating W–Cu FGM by high-gravity combustion synthesis and melt-infiltration.

<100 Pa. The rotator was then started and a high-gravity field with an acceleration of G (G = 800 g, g means the gravitational acceleration) was induced by the centrifugal effect. By passing an electric current through a tungsten coil closely above the thermite, the combustion reaction was triggered. The rotator stopped in 5 min, and the W–Cu product was finally obtained.

The target W–Cu product was cut into several layers along the high-gravity direction, machined and polished for the following tests. The chemical composition was analyzed by portable X-ray fluorescence analyzer (XRF; X-MET5100). Analysis of ppm level can be achieved by the portable XRF and the sampling depth is dozens of micron for the metal. The microstructure was examined by scanning electron microscopy (SEM; S-3400, Hitachi, Japan) and transmission electron microscope (TEM; JEOL JEM-2100, Japan). The bending strength was tested by the three-point bending method with a span of 30 mm at a crosshead speed of 0.1 mm/min.

3. Results and discussion

By Archimedes principle, the relative density of the prepared W–Cu FGM is ~97%, and that of the commercial W–Cu composite is ~98%. Fig. 2a shows the SEM images of the five layers cut from the target W–Cu product along the high-gravity direction, where the bright phase is W and the dark phase is Cu. A gradient structure was observed. Since the microstructures of the target W–Cu FGM are similar for all the five layers, only the microstructure of the W-rich layer will be discussed in details later. In Fig. 2b, a SEM image of W-rich layer with a higher magnification is shown. It revealed that W particles partially sintered and formed an incomplete net structure, as the high temperature did not last long enough due to the fast cooling rate. For comparison, a typical microstructure of a commercial W–Cu composite produced by the traditional infiltration method is shown in Fig. 2c, in which W particles sufficiently sintered to form a complete net structure.

Fig. 2d–f is typical TEM photos of the W phase in the W-rich layer. The grain size of W ranged from 100 to 500 nm in Fig. 2e and 500 nm to 1 μm in Fig. 2f, respectively. There was no difference in the grain size of W in different parts of the W–Cu FGM. In other words, the grain size of W did not change with the W content. It can be concluded that W grains in the W–Cu FGM are micron-sized (0.1–1 μm), which was much smaller than that in the starting W powder (3 μm). During the solidification process of high-gravity combustion synthesis, high stress was easily generated in W grains due to the very high cooling rate, which was account for the transformation of the W grain size from original 3–0.1–1 μm. Fig. 2f revealed that the Cu grains surrounded by W phase were smaller than 50 nm. The high-resolution TEM photograph of the Cu grains surrounded by W phase is shown in Fig. 2g, where the phase analysis verified that the nano-crystals did belong to Cu phase. The mechanism of the formation of nano-crystalline Cu phase surrounded by W phase is interpreted as follows: the number of grains in unit volume Z is a function of the nucleation rate N and the crystal growth rate G ($Z = 0.9(N/G)^{3/4}$), so high nucleation rate and low crystal growth rate lead to a small grain size, where the high nucleation rate is the dominant factor [16]. The powder bed, considered as nucleating agents, effectively accelerated heterogeneous nucleation and caused grain refining. The powder bed can also be regarded as a cooling medium, especially as Cu powders absorbed considerable heat during melting. Moreover, the heat convection was dramatically increased by the high-gravity field. Therefore, liquid Cu was undercooled due to the high cooling rate which then leads to a high nucleation rate, and hence a small grain size. Then the viscosity of Cu melt was high so that nucleation rate was high and crystal growth rate was low [17–19]. Another reason why the nano-crystalline Cu was formed was that small nucleus crystallized in the melt could be fragmentized to several nuclei by impact and vibration induced by the relative macro flow of the Cu melt and W particles which was very intense in the high-gravity field. This mechanical increment effect dramatically increased the number of nuclei and refined the grain. Overall, the high gravity field and W particles played the leading role in the formation of nano-crystalline Cu grains surrounded by W phase.

Fig. 2h is a TEM image of the Cu phase not surrounded by W phase. There were a large amount of dislocations in the Cu phase, which were easy to be formed in the solidification process, for the cooling was fast. In the solidification process, the dislocations or other defects on crystal surface (including W particles' surface) directly grew into the solidified Cu crystals, and boundary atoms in different parts tended to form boundary dislocations because of different orientations. In addition, when the Cu melt was cooled quickly near the melting point, supersaturated voids were obtained, aggregated into a void cluster, and eventually transformed to a dislocation loop. Furthermore, W powders would introduce internal stresses in the solidified Cu phase, resulting in non-uniform shrinkages and the subsequent formation of dislocations. Because of the high cooling rate, the dislocations formed in the above procedures were difficult to be eliminated by diffusion and were eventually retained. To sum up, the dislocations in the Cu phase were generated by the combined effect of W powders and a rapid cooling rate introduced by the high-gravity field.

Three specimens with the size of 3 mm × 4 mm × 35 mm cut from the target W–Cu FGM along the high-gravity direction and a commercial 75 wt%W–25 wt%Cu composite were used in three-point bending tests. The mass fractions of W in the three FGM samples were about 45 wt%, 57 wt% and 70 wt%, respectively. Fig. 3a and b show the load–displacement curves and the bending strength of the three W–Cu FGM samples and the commercial W–Cu composite. The bending strength of the W-rich sample (W ≈ 75 wt%, 1127 MPa) was 15% higher than that of the commercial W–Cu composite (W ≈ 75 wt%, 982 MPa). The higher strength was attributed to the submicron level of W grains, the nano-crystalline Cu phase surrounded by W particles, and lots of dislocations in the Cu phase. There were a large amount of grain boundaries in the nano-crystalline Cu grains and micron-sized W phase which could hinder the dislocation motions. The dislocation intercross was easy because of the large amount of the dislocations, which further prevented the dislocation motions and improved the