WC-Cu cermet materials: production and characterization

Flávio Diogo Gonçalves Guerreiro
flavio.guerreiro@tecnico.ulisboa.pt

Instituto Superior Técnico, Lisboa, Portugal

Abstract

Due to the similar properties between tungsten and tungsten carbide as well as to the extensive body on knowledge on WC cermets, WC-Cu cermets with 25, 50 and 75 % volume fraction of copper have been devised for thermal barriers. The composite materials have been prepared by hot pressing at temperatures ranging from 1173 to 1423 K with pressures of 22 to 47 MPa. These novel cermets were investigated by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy. The thermal conductivity was measured by laser flash tecnhique and the microhardness and geometric density were also evaluated. The consolidated materials consisted of homogeneous dispersion of WC particles in the Cu matrix without any evidence for oxide formation and with densifications of about 91-95%. No interfacial decohesion was detected in indented regions. The cermets thermal diffusivity is lower compared to copper or tungsten, as desirable for thermal barrier materials.

Keywords: WC-Cu cermet, hot pressing, thermal diffusivity, densification.

1. Introduction

The high melting point, high sputtering threshold and low tritium inventory turn tungsten into a potentially suitable material for plasma facing and structural components in the first wall of nuclear fusion reactors. However, a major disadvantage of current tungsten-grades is their relatively high ductile-to-brittle transition temperature [1]. Operation at higher temperatures is hence desirable to preserve the integrity of W components, such as the W tiles at the internal surface of the first wall. A CuCrZr alloy has been selected as heat sink material to remove heat from the plasma facing components due to its high conductivity, strength and microstructural stability [2]. However, the service temperature of this material is relatively low and, therefore, operation at higher temperatures demands thermal barriers between the plasma-facing W and the CuCrZr heat sink.

Metal Matrix Composite Materials are expected to introduce a transition from brittle (W) to ductile (CuCrZr) behavior if tailoring the properties of the material such as thermal conductivity and CTE from a ceramic-like to a metallic-like behavior by simply adjusting the proportions of the dispersed phase present in the material. For instance, W fiber-Cu and diamond-Cu composites have been proposed [REFs] as interlayer materials between the plasma facing and cooling materials. However, the W fiber-Cu composites suffers from thermal fatigue damage during thermal cycling and the thermal properties of diamond-Cu composite are expected to change in a fusion reactor due to the formation of a new carbon phase [REF].

A proposed strategy is the use of WC-Cu interlayer as thermal barrier, since Cu is a good electrical conductor [3] and the composition can be optimized for controls the thermal conductivity. Moreover tungsten carbide (WC) combines favorable properties, such as high hardness, plasticity and good wettability by molten metals [4].

The present work is focus in the production and charaterization of WC-Cu cermet materials using hot pressing. Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS) was used to characterize the microstructure of the WC-Cu cermets. The geometric density, microhardness and thermal diffusivity were also evaluated.

2. Experimental

The cermet composition series were produced by turbula blending (from 30 min to 90 min) commercially pure WC powder (diameter of 1 μm) with 99.9% nominal purity with Cu powder (diameter < 37 μm) with 99.99% nominal purity for variable volume fraction of Cu (25%, 50% and 75%) and subsequently consolidating the mixtures by hot pressing. In order to avoid oxidation, the mixtures were prepared inside a glove box in a controlled N_2 atmosphere. The WC-Cu powders were consolidated by hot pressing varying the temperature between 900 and 1150 ºC and using loads ranging from 22 to 47 MPa with an Idea Vulcan 70 VP hot pressing device. Table 1 evidences the mixtures prepared and the conditions for which highest densifications were obtained. The estimated heating rate is the same for all the cycles and approximately equal to 2 °C/s. The WC-Cu powder mixture was consolidated in graphite dies of 10 mm of length, 55 mm of width and 5 mm of thickness. Figure 1 shows schematically the thermal route followed during consolidation for the denser samples obtained for each composition.

Table 1: Consolidation parameters for each cermet composition using WC with 1 mm grain size.

Conditions	Composition %(V/V)		
	25WC-75Cu	50WC-50Cu	75WC-25Cu
Temperature (ºC)	900	1060	1150
Pressure (MPa)	22	37	47
Time (min)	5	5	6

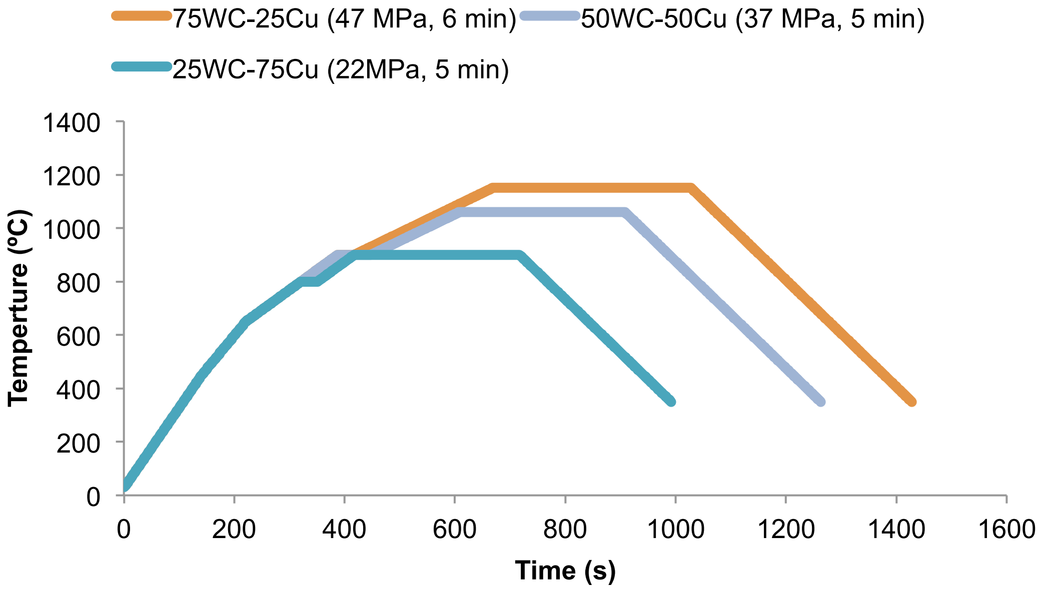

Figure 1- Hot pressing consolidation cycles for the three compositions cermets with higher densification.

The microstructures were observed with secondary and backscattered electron respectively, SE and BSE signals on polished surfaces, using a JEOL JSM-7001F field emission gun operated at an accelerating voltage of 20 keV. The instrument is equipped with an Oxford EDS system used for point analysis and X-ray map collection.

Thermal diffusivity measurements were performed with a flash instrument Flash Line 5000 Anter Corporation in the 100 ºC – 400 ºC temperature range.

The densification achieved was measured by the Archimedes method and the mechanical strength was evaluated by microhardness measurements using a Shimadzu HMV 2000 hardness tester with a Vickers indenter and a load of 19.6N applied for 15 s.

3. Results and discussion

3.1. Microstructure characterization

Figure 2 presents the microstructure of the consolidated cermets with WC with a 1-2 µm of grain size for three compositions 25WC-75Cu, 50WC-50Cu and 75WC-25Cu materials. The microstructures of all the cermets revealed particles of WC dispersed in a matrix of copper. Moreover the microstructures revealed that cooper melting occurred, which can be observed by the formation of cooper islands in Figure 2 (a). The maximum temperature of the cycles of the 25WC-75Cu and 50WC-50Cu materials are lower (900 ºC and 1060 ºC respectively) than the melting temperature of copper (1083 ºC), however a copper liquid phase was formed. The presence of impurities justifies the depression of the cooper melting temperature, since the Cu-O binary diagram [5] indicates that at 0,03 of wt.% of oxygen exist a eutectic between the two elements at 1065 ºC.

Another aspect is the error of the pyrometer in the temperature reading, since the pyrometer reads the temperature at the surface of the graphite mold and such temperature is not exactly the same of the materials being consolidated, and consequently can induce an error of the temperature measure. Once the cooper liquid is formed it tends to fill the smallest pores (due to capillarity effects) in order to minimize the total energy of the system leaving pores at the sites occupied by the copper particles prior melting [6]. However, the high pressure applied (47 MPa) when consolidating the 75WC-25Cu materials may be responsible for the formation of the copper patches by forcing the liquid copper to fill existing large pores (FIGURE (e)).

The starting WC powders have a grain size 1-2 µm and no grain growth was observed. However, the 50WC-50Cu and 75WC-25Cu cermets exhibit large aggregates with medium size of ~21 µm and ~37 µm, respectively. The overall faceted shape and the grain contrast in the slab-like WC aggregates found in 50WC-50Cu samples (FIGURE (d)) suggests aggregation along preferred orientations and coalescence of the WC particles [16]. It seems that, as it occurs in the WC-Co system, in WC submicron sized powders there is a preferred grain boundary orientation between WC grains [17] which makes grain growth by coalescence easier. The microstructure of the 75WC-25Cu sample evidenced the coalescence of WC particles more compact than those observed in the previous samples, as the applied pressure used to consolidate the these materials was higher (47 MPa). Such particles in 75WC-25Cu samples possessed a mosaic structure typical of polycrystalline aggregates (FIGURE (f)). These larger particles possessed a mosaic structure typically of polycrystalline aggregates.

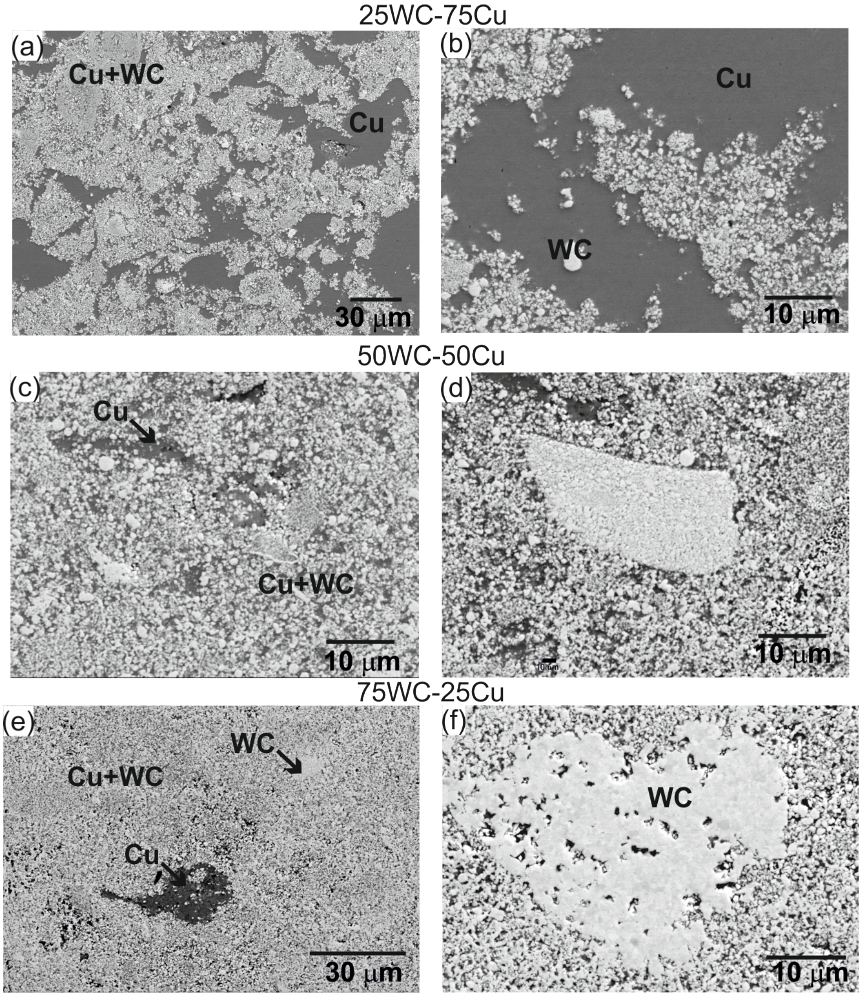

Figure 2 - SEM images showing the microstructure of WC-Cu cermet with different compositions (a) and (b) 25WC-75Cu, (c) and (d) 50WC-50Cu and (e) and (f) 75WC-25Cu consolidated by hot pressing.

3.2 Density and microhardness

The relative density values obtained for the consolidated materials are presented in Table 2. The consolidated materials present densifications above 90 %, the highest density achieved for the 75WC-25Cu is probably due to the high pressure in combination with the high temperature and small amount of copper.

Table 2: Densification values of the cermets for WC.

Property	**Composition %(V/V)**		
	25WC-75Cu @900 ºC, 22 MPa, 5 min	50WC-50Cu @1060 ºC, 37MPa, 5 min	75WC-25Cu @1150 ºC, 47MPa, 6 min
Densification (%)	91	92	95

The hardness results for the three composition cermets are presented in Table 3. The addition of 75 vol. % of WC particles to a copper matrix increases the Vickers hardness of 87 MPa for pure copper [3] to about 463 MPa for the 75WC-25Cu cermet. The increase in copper content (and decrease of WC) causes the hardness to decrease in comparison to the 75WC-25Cu cermet. Figure 4 shows in detail the Vickers indentation resulting from a load of 19.614 N in the 25WC-75Cu and 75WC-25Cu cermets. The micrographs of the high load indentations (HV2) of 25WC-25Cu composition show some level of deformation in the copper matrix at the vicinity of the indentations (black arrows in Figure 4 (a)). No decohesion between the WC particles and the copper matrix was observed. However the indented microstruture of 75WC-25Cu sample revealed the intergranular fracture of the WC grains. The same behaviour was observed for the 50WC-50Cu cermet.

Table 3:Mean HV2 hardness values of the cermets and the respective standard deviation

Property	**Composition %(V/V)**		
	75WC-25Cu @1173 K, 47 MPa, 6 min	50WC-50Cu @1333 K, 37 MPa, 5 min	25WC-75Cu @1423 K, 22 MPa, 5 min
HV2 (MPa)	463	395	139
σ_2 (MPa)	225	50	20

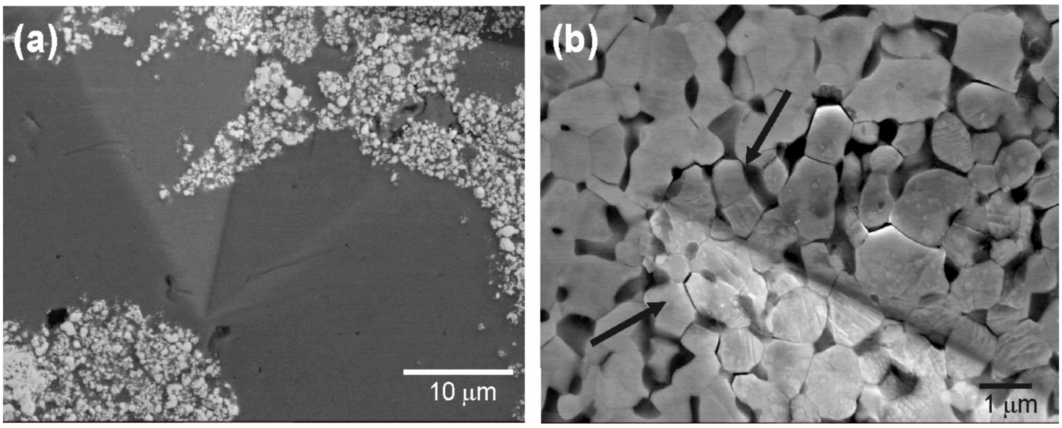

Figure 4 - Micrograph showing a detail of the HV2 indentation for (a) 25WC-75Cu and (b) 75WC-25Cu cermets.

3.3 Thermal diffusivity

Figure 5 presents the thermal diffusivity of the consolidated WC-Cu cermet together with literature values for pure Cu [3], W [7] and WC [8]. Since 75WC-25Cu cermets present a high content of WC, the thermal behavior of the materials is close to the one of pure WC. On the other hand, the high content of copper seems not to cause a significant increase in the thermal diffusivity of the cermets since both the 25WC-75Cu and 50WC-50Cu samples present the same thermal dependence. This similar behavior between 25WC-75Cu and 50WC-50Cu should be associated with densification, which is similar for both (91% and 92% respectively).

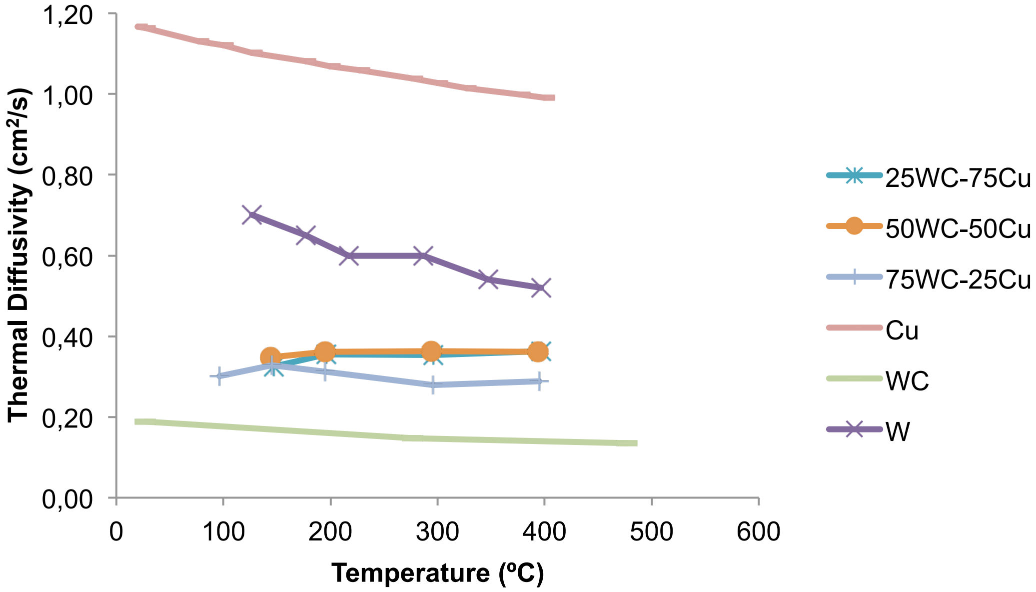

Figure 5 – Thermal diffusivity of samples: 75WC-25Cu, 50WC-50Cu and 25WC-75Cu cermets. For comparison the curves for Cu [7], W [8] and WC [9] are also presented.

4. Conclusions

The WC-Cu cermets with 25, 50 and 75 % volume fraction of copper have been devised for thermal barriers. The materials have been prepared by hot pressing at temperatures ranging from 1173 to 1423 K with pressures of 22 to 47 MPa. These cermets were investigated by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy. The consolidated materials consisted of dispersion of WC particles in the Cu matrix without any evidence for oxide formation and with densifications of about 91-95%. No interfacial decohesion was detected in indented regions. The cermets thermal diffusivity is lower than that of copper or tungsten, as desirable for thermal barrier materials.

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