TUNGSTEN-STEEL COMPOSITES AND FGMS PRODUCED BY HOT PRESSING

Jiří MATĚJÍČEK¹, Hanna BOLDYRYEVA¹, Vlastimil BROŽEK¹, Elena ČIŽMÁROVÁ², Zdeněk PALA¹

¹Institute of Plasma Physics, Za Slovankou 3, 18200 Praha, Czech Republic; jmatejic@ipp.cas.cz
²Innovation Centre for Diagnostics And Application of Materials, Czech Technical University, Karlovo nám. 13, 12135 Praha, Czech Republic

Abstract

Tungsten-steel composites and FGMs are being developed for potential application in plasma facing components of fusion devices. In this study, uniform composites and graded layers produced from tungsten and steel powders by hot pressing were investigated. Formation of dense composites with uniform distribution and good bonding of the phases was observed. A thin layer of intermetallic phase Fe7W6 formed at the interfaces. Thermal and mechanical properties of the composites in the as-produced and annealed state were characterized.

Keywords:
FGM, composite, tungsten, steel, fusion reactor material

1. INTRODUCTION

Tungsten-steel composites and functionally graded materials (FGMs) are being developed for potential application in plasma facing components of fusion devices. In fusion devices, the materials can be subjected to complex loading – thermal, mechanical, chemical, electromagnetic, prospectively also in the presence of neutron irradiation [1]. The required ability to function in such extreme conditions poses serious challenges for materials development, with different requirements often calling for multi-material compounds. For example, for the ITER reactor, tungsten, beryllium and carbon fiber composites are planned as the plasma facing materials, according to different particle and heat fluxes expected in different regions of the plasma facing surface [2]. These are to be joined to copper-based heat sink or stainless steel construction material. For the next step device, DEMO, tungsten is foreseen as the main plasma facing material, to be joined to stainless steel as a heat sink and construction material [1,3]. When two or more materials are to be used, various joining issues come into play. These are namely the dissimilarity in thermal and mechanical properties, leading to stress concentration at the interface, physicochemical compatibility, such as lack of wetting or high mutual reactivity, neutronics problems with certain brazes, etc. [4]. Some of these issues can be solved by the use of interlayers or graded transitions between the two materials. It has been shown in specific tungsten/steel examples that the use of a graded interlayer can reduce the maximum strain by a factor of 3 [5] and that the reduction of maximum strain is larger the thicker is the graded layer [6]. Several techniques have been explored for the production of composites and graded layers, such as plasma spraying [7], pulse plasma sintering [8], resistance sintering [9], laser cladding etc. A brief assessment of different aspects of their applicability in plasma facing components of fusion reactors is provided in [6].

This paper is focused on the hot pressing technique, using tungsten and steel powders. Structure and selected properties of uniform composites with various compositions as well as graded layers are presented.
2. EXPERIMENTAL DETAILS

Tungsten-steel uniform composites and layered FGMs were produced by hot pressing powders in a graphite die at Bonar, a.s. (Šumperk, Czech Republic). The conditions were 2000 °C and 6 GPa, except where noted. Pellets of about 19 mm diameter and various thicknesses were formed, with different layer configurations as summarized in Tab.1. First, uniform composites were prepared from W + steel powder mixtures, consisting of nominally 0, 25, 50, 75 and 100 vol. % tungsten, to characterize the interaction and properties of the individual constituents. Thick and thin FGMs, consisting of 5 layers with the abovementioned compositions, were then produced. For the thin FGMs, 0.15 mm steel sheet partitions were used between powder layers, to facilitate planarity. Additionally, uniform composites with finer powder were produced, to observe the effects of particle size. The following powders were used: W 63-80 µm (Alldyne Powder Technologies, Huntsville, USA), W <50 µm (Osram Sylvania, Towanda, USA) and SS410 50-90 µm (Flame Spray Technologies, Duiven, Netherlands). The composition of the steel powder was as follows (wt%): Fe bal., Cr 12.3, Si 0.2, C 0.01, S 0.01. Selected samples were annealed in an inert atmosphere at 1000 °C for 4 h to observe possible formation of intermetallic phases and/or changes in properties.

Structural observations were carried out on polished cross sections using an EVO MA15 scanning electron microscope (Carl Zeiss, Oberkochen, Germany). Energy-dispersive spectroscopy, integrated in the SEM, was used for local compositional analysis. Volume percentage of the tungsten and steel phases was determined by image analysis on SEM images in backscattered electron mode. Thermal conductivity was determined by the xenon flash method using an FL-3000 instrument (Anter Corp., Pittsburgh, USA) at 100 °C. Mechanical properties of the individual phases were characterized by instrumented indentation on a Nanotest instrument (Micromaterials Ltd., Wrexham, UK) with a 50 mN load and loading/hold/unloading times of 20/10/20 s. Phase composition of was determined from X-ray diffraction pattern measured by an X’Pert PRO MPD diffractometer (PANalytical B. V., Almelo, Netherlands) equipped with a copper anode X-ray tube and a fast RTMS (Real Time Multiple Strip) detector. The diffractometer was used in Bragg-Brentano geometry with the sample surface being positioned by a laser triangulation gauge. Structure refinement and quantitative phase analysis are results of Rietveld refinement procedure employing X’Pert HighScorePlus software.

<table>
<thead>
<tr>
<th>Label</th>
<th>Layer sequence</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S100</td>
<td>100% SS410</td>
<td>2.7</td>
</tr>
<tr>
<td>W25S</td>
<td>25% W + 75% SS410</td>
<td>2.6</td>
</tr>
<tr>
<td>W50S</td>
<td>50% W + 50% SS410</td>
<td>2.1</td>
</tr>
<tr>
<td>W75S</td>
<td>75% W + 25% SS410</td>
<td>2.5</td>
</tr>
<tr>
<td>W100</td>
<td>100% W</td>
<td>2.5</td>
</tr>
<tr>
<td>W50F</td>
<td>50% W fine + 50% SS410</td>
<td>2.9</td>
</tr>
<tr>
<td>W75F</td>
<td>75% W fine + 25% SS410</td>
<td>2.8</td>
</tr>
<tr>
<td>FGM1</td>
<td>100% SS410, 25% W, 50% W, 75% W, 100% W</td>
<td>7.8</td>
</tr>
<tr>
<td>FGM2</td>
<td>100% SS410, 25% W, 50% W, 75% W, 100% W fine</td>
<td>1.5</td>
</tr>
</tbody>
</table>

3. RESULTS AND DISCUSSION

3.1 Structural observations

Representative structures of the uniform W+SS410 composites are shown in Fig. 1. Relatively homogeneous mixing of the two constituents, essentially full density and good bonding can be observed. The particles are slightly elongated in the horizontal direction, as a result of the uniaxial pressure. Besides the original phases, a newly formed thin layer can be seen at the interfaces (shown in grey in the BE images). This layer has been identified as Fe7W6 by x-ray diffraction (see below) and its thickness slightly increases with increasing
W content. Detail of this interlayer is shown in Fig. 2a. For the most part, this phase appeared contiguous; isolated cracks across the thickness were occasionally observed. Moreover, in composites with a higher W content, a sort of “microcomposite” has formed on the steel side of the interface. Its detail is shown in Fig. 2b, where heavily intermixed, submicron grains are observed. In some cases, this microcomposite formed the entire particle, while in others, a steel core remained in the center. Detailed structure of the steel phase is also shown in Fig. 2a. On the right, rows of ultrafine pores (≤300 nm in size), formed likely at the original particle boundaries, can be seen. However, their occurrence was rather infrequent. In the center, a network of diffuse lighter “threads” can be seen, indicating a higher W content. Preferential diffusion paths were probably provided by the original particle boundaries as well. Detail of the tungsten phase is shown in Fig. 2c. At enhanced contrast, a very fine grain structure can be seen, as well as a row of submicron-size pores in the center. With finer W powder, similar structures were obtained, but with a more homogeneous distribution of the phases. In two samples, an isolated small region with a very fine structure was found, exhibiting intense mixing and higher carbon content. This may have resulted from local overheating, accompanied by ingress of carbon from the die.

A thick 5-layer FGM is shown in Fig. 3. A stepwise compositional transition from steel (bottom) to tungsten (top), with good planarity of the layers can be seen. A slight variation in composition within a single layer can be observed, probably as a result of manual pouring of the powders into the die. A thinner FGM was also attempted. To ensure good planarity and more precise control of the layer thicknesses, 0.15 mm stainless steel sheet partitions were used between each layer. As can be seen in Fig. 4, the planarity was preserved and the partition integrated well with the neighboring layers. This was also observed in other cases (bi-layers), not shown here.

Upon annealing, the structure of the composites was largely preserved, only the intermetallic layers have grown slightly. In some locations of the annealed W75S sample, two sub-layers within the intermetallic layer could be discerned. According to point-wise EDS, the composition corresponded roughly to Fe7W6 and Fe2W, but the sub-layers were too thin for definitive identification. Quantitative x-ray diffraction analysis has shown the following phase content in the annealed W75S sample (wt/vol%): W 79.3/65.6, Fe 12.4/25.4, Fe7W6 3.8/4.1, Fe2W2C 3.8/4.1, WC 0.7/0.7. The formation of the carbides was likely facilitated by enhanced diffusion of carbon from the graphite die through the molten steel. This is also supported by the localized EDS measurements, performed in several ~500x500 μm regions across diagonal sections of the W50 and W75 samples. Although only semi-quantitative, the results showed uniform carbon content across the sample thickness.

3.2 Properties

Thermal conductivities in the as-produced and annealed states are summarized in Tab. 2. As expected, the values for the terminal compositions are close to those for bulk materials found in the literature (164 W/m.K for W, 27 W/m.K for SS410 [10]). The trend of conductivity with composition is not linear; the values remain rather low for W content of up to 50% and only increase significantly for the higher W content. The value for the FGM1 was lower than expected, possibly due to the presence of carbon/carbide inclusions, as mentioned previously. After annealing, the values did not change considerably.

The results of mechanical characterization of the individual phases of the annealed W75S sample are summarized in Tab. 3. The Fe7W6 phase showed the highest hardness, while that of the microcomposite was between Fe7W6 and W. On the other hand, W phase had the highest Young's modulus, followed by the Fe7W6 and the microcomposite. The Fe7W6 phase exhibited the lowest ratio of plastic work to total work during the indentation, therefore the lowest ability to plastically deform (as could be expected from an intermetallic phase).
Fig. 1 Microstructures of the uniform W+SS410 composites: a) W25S, b) W50S and c) W75S. SEM-BE images; tungsten phase in light grey, steel phase in dark grey.

Fig. 2 Microstructural details of individual phases: a) W25S, b) W50F and c) W100.
Fig. 3 Overview of the thick graded structure (FGM1). Steel side at the bottom, tungsten side at the top.

Table 2 General characterization results. Tungsten content was determined by image analysis on the cross sections. Typical coefficient of variation of the thermal conductivity values is around 2%.

<table>
<thead>
<tr>
<th>Label</th>
<th>W content (vol%)</th>
<th>Thermal conductivity as-produced (W/m.K)</th>
<th>Thermal conductivity annealed (W/m.K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S100</td>
<td>0</td>
<td>24.8</td>
<td>26.9</td>
</tr>
<tr>
<td>W25S</td>
<td>27.3</td>
<td>17.7</td>
<td>20.5</td>
</tr>
<tr>
<td>W50S</td>
<td>46.6</td>
<td>25.7</td>
<td>24.7</td>
</tr>
<tr>
<td>W75S</td>
<td>68.4</td>
<td>78.6</td>
<td>70</td>
</tr>
<tr>
<td>W100</td>
<td>100</td>
<td>168.1</td>
<td></td>
</tr>
<tr>
<td>W50F</td>
<td>53.4</td>
<td>43.4</td>
<td></td>
</tr>
<tr>
<td>W75F</td>
<td>74.9</td>
<td>74.0</td>
<td></td>
</tr>
<tr>
<td>FGM1</td>
<td></td>
<td>16.3</td>
<td></td>
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</table>

Table 3 Results of the nanoindentation experiments on annealed W75S sample.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Hardness (GPa)</th>
<th>Young's modulus (GPa)</th>
<th>Plastic work (nJ)</th>
<th>Elastic work (nJ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>6.5 ± 0.3</td>
<td>396 ± 30</td>
<td>10.3 ± 0.4</td>
<td>1.5 ± 0.1</td>
</tr>
<tr>
<td>Fe7W6</td>
<td>13.7 ± 1.8</td>
<td>284 ± 27</td>
<td>5.7 ± 0.6</td>
<td>2.7 ± 0.2</td>
</tr>
<tr>
<td>Microcomposite</td>
<td>8.5 ± 0.4</td>
<td>244 ± 11</td>
<td>7.7 ± 0.3</td>
<td>2.5 ± 0.1</td>
</tr>
</tbody>
</table>

Fig. 4 Detail of the thin graded structure (FGM2). The sequence of layers, going up from the bottom, is the following: 100% W layer, stainless steel partition, 75% W layer, 50% W layer showing heavy mixing due to local overheating. Other layers are not shown, since they were disturbed due to a crack in the graphite die.
4. SUMMARY

Tungsten-steel composites and FGMs were produced by hot pressing. The composites exhibited full density, uniform mixing and good bonding between the tungsten and steel particles. Two examples of FGMs demonstrated the compositional gradation over different thicknesses. The formation of a thin Fe7W6 intermetallic layer at the interfaces was observed, in dependence on the tungsten content. Local mechanical characterization indicated this phase to be more brittle than the others, which may pose a problem to structural integrity upon exposure to high temperatures. Possible ways to inhibit the formation of this layer are being explored.

ACKNOWLEDGEMENT

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REFERENCES