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Investigation on 316L/316L-50W/W plate functionally graded materials fabricated by spark plasma sintering

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ABSTRACT

Tungsten plate (W_p), tungsten powder (W) and stainless steel 316L (316L) were used to fabricate 316L/W composites, respectively. Due to the less pores for the interface of 316L/W_p, W_p was used to fabricate 316L-50W/W_p as well as 316L/316L-50W/W_p. All samples were sintered for 5 min under conditions of 1050 °C and 45.5 MPa by spark plasma sintering method. Morphology and elements distribution of the interfaces were investigated by optical microscope, SEM and EDS. The hardness distribution for 316L/316L-50W/W_p functionally graded materials was also studied. Results revealed that there were residual pores for the interface of 316L/W. Homogeneous interfaces with less pores could be obtained for 316L-50W/W_p and 316L/316L-50W/W_p with 316L-50W milled nearly 5 h. Longer milling time leads to more intermetallics, holes and micro-cracks at the interface, which is neither beneficial to obtain homogeneously distributed joints nor suitable to gain graded interfaces. The hardness analysis result gives the same conclusion as well.

1. Introduction

W has potential application for plasma facing materials of helium cooled divertor in fusion reactor due to its advantages of high thermal conductivity, low sputtering yield, high sputtering threshold, low tritium retention and low irradiation swelling [1–5]. For future fusion devices, including DEMO, reduced activated ferritic/martensitic (RAFM) steel and oxide dispersion strengthened (ODS) steel are usually used as structural materials [6]. However, the difference for coefficients of thermal expansion (CTEs) of W and steel (4×10^{-6}/K and 12–14 $\times 10^{-6}$/K) is large, which induces large thermal stresses and further leads to cracks generation and failure of the divertor. Thus the effective joint of W and structural materials is an important factor for the fusion reactor to run successfully.

Brazing with Cu, Co, Ni as solders [7,8] as well as vacuum diffusion brazing with Ta, Ti, Ni, Nb, V (whose CTEs are between those of W and steel) or their alloys [9–11] are mostly used methods for the joint of W and steel. However, solders are easily to be activated under high energy neutron flux [12]. Other techniques like hot isostatic pressing sintering [13,14] and spark plasma sintering (SPS) [15–18] have been used to study the joint of W and structural materials as well. Hirose found that the ferritic phase formed by SPS is good for the joint of W and F82H even without an artificial interlayer [16]. Wu's group studied the W/Fe diffusion bonding with Ti foil and Ti powder by SPS and found that the interface of samples with Ti foil as interlayer had better thermal stability [18].

Functional graded materials (FGMs) are materials with structures and properties changed gradually, which is beneficial to reduce thermal stresses especially for materials (for example W and steel) with large differences in CTE [19–21]. We mainly focus on the joint quality of W and steel at present. Thus stainless steel 316L (316L) was used in our study instead of RAFM or ODS steel since it is cheaper and the main element (Fe) is not changed. W/316L FGMs by mechanical alloying and SPS were obtained in our previous study with transition belts containing Fe_7W_6, Fe_3W_3C and Fe_2W [17].

Since the usual sintering temperature for W is too low to expel the residual holes and to obtain the high-density samples, W plate (W_p) was used in this work to prepare 316L/W_p at first in comparison with 316L/W (W powder). Then 316L/316L-50W/W_p FGMs were fabricated for better joint of W and 316L. Meanwhile, the effect of milling time on

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Table 1
The compositions of raw materials of 316L, W and W_p (wt%).

Item	C	O	Si	Mn	P	S	Ni	Cr	Mo	Fe	W
316L	0.028	–	0.74	0.3	0.027	0.007	12.25	17.32	2.24	rest	–
W	< 0.01	0.08	< 0.006	–	–	–	–	–	–[a]	0.01	rest
W_p	0.001		0.002	–	–	–	–	–	0.01	0.01	rest

[a] means too less to be detected, W means W powder and W_p means W plate.

Table 2
The designation of samples' names in the Figures.

Samples	Conditions and sample names		
	W powder (W)		W plate (W_p)
316L-W	1		2
	316L–50W		
Samples	As-mixed	5h-milled	10h-milled
316L-50W/W_p	3	4	5
316L/316L-50W/W_p	6	7	8

morphology and distribution of elements for 316L-50W/W_p as well as on the interface morphology and hardness of 316L/316L-50W/W_p FGMs were studied.

2. Experiment and materials

Commercially available W powder (average particle size of 20–45 μm), W_p (Φ10 mm x 2.5 mm) and 316L powder (particle size of 20–45 μm) were used in this study with compositions listed in Table 1. The designation of the samples' names is given in Table 2 for helping to identify the samples in Figures.

316L/W (Sample 1) and 316L/W_p (Sample 2) were prepared from 316L with W and W_p, respectively. 316L-50W for 316L-50W/W_p and 316L/316L-50W/W_p was obtained as follows: 1) W and 316L powders (316L50%-W50%) were weighed and mixed according to the volume ratio of 1:1; 2) The blended powders were mixed or milled with a high-energy planetary ball milling system in an Ar-filled glove box with designed milling parameters listed in Table 3, which are the same as our previous work [17]. The milling jars and balls are both made of WC and the diameter of the balls is 1.5 mm. For SPS process, the powder was loaded into a graphite mold with a diameter of 10 mm after being dried in a vacuum dryer, which was sintered under conditions of 1050 °C, 45.5 MPa and 5 min in vacuum. For all samples, W or W_p was put at the bottom. Three 316L-50W/W_p (Samples 3–5) and three 316L/316L-50W/W_p FGMs (Samples 6–8) were obtained. The obtained circular samples were cut into halves, whose cross sections were ground and polished for microstructure and hardness characterization.

The microstructure characterization of the above mentioned powders and sintered samples was carried out in a field-emission scanning electron microscope (FE-SEM) and optical microscope as well. Composition analysis of the specific area was done by energy dispersive X-ray spectrometer (EDS) attached to FE-SEM. The phase analysis was performed by X-ray diffraction (XRD). Vickers hardness test for sintered composites was carried out with an indent force of 100 g (0.98 N) and a dwelling time of 10 s.

Table 3
The mechanical alloying parameters for 316L and W powders.

316L-50W	Rotating Speed (rpm)	Ball-to-powder Ratio (mass ratio)	Milling Time (hour)
As-mixed	100	1:1	8
5h-milled	200	3:1	5
10h-milled	200	3:1	10

3. Results and discussion

3.1. Interface morphology for 316L/W and 316L/W_p

As stated in [18], the interface of a W/steel joint sample with Ti foil as interlayer had better thermal stability than that with Ti powder. W plate was tested in this work since the sintering temperature is too low for W powder to obtain high-density samples due to its high melting point. 316L/W (Sample 1) and 316L/W_p (Sample 2) were prepared at first for interface characterization and comparison.

Morphology for samples 1 and 2 prepared from 316L with W and W_p was shown in Fig. 1(a) and (b), respectively. Both samples have 316L and W joined successfully with clear interfaces. It seems that there are many residual pores near the interface of 316L and W and the interface is not regular. There are much fewer pores for the interface of 316L and W_p, which is more homogeneous with obvious interface. This phenomenon is probably due to the low SPS sintering temperature for W compared to its high melting point.

It is much clearer from SEM micrograph and EDS patterns as shown in Fig. 2. The dots shown in the lines indicate the start positions of line scanning of SEM. There are many holes near the interface of 316L/W and the transition region is about 2 μm in length from EDS analysis. The transition region for 316L/W_p is about 2.5 μm in length with similar variation trend for W content. In all, the interface has less pores and is more homogeneous for 316L/W_p compared to that of 316L/W.

3.2. Morphology of 316L/316L-50W/W_p FGMs

According to the above results, W_p was used as an end material and was put at the bottom always for the following study. 316L-50W obtained with 316L and W mixed or milled for different time was used as interlayer to fabricate 316L/316L-50W/W_p FGMs.

3.2.1. The effect of milling time on morphology of 316L and W

SEM morphology for the as-mixed 316L and W is shown in Fig. 3(a) with corresponding EDS pattern shown in Fig. 3(b). It shows that most W and 316L particles keep their original shapes after the mixing process: W particles have angular surfaces while 316L particles show smooth surfaces. For the powders being milled for 5 h, the SEM morphology is shown in Fig. 3(c). It indicates that most 316L particles have plastic deformation and changed to plate-like shapes, while W particles are less angular and some 316L fragments attached to W particles. Fig. 3(d) shows the corresponding EDS pattern, which indicates that 316L fragments were just attached to the surface of W particles. SEM morphology for the 10 h-milled powders is shown in Fig. 3(e), which shows that almost all 316L particles changed to plates due to the severe deformation. The corresponding EDS analysis indicates that 316L fragments were embedded into W particles due to the high contents for elements from 316L. The impurities of O may be from the atmosphere during milling process, while C could be probably from WC balls and jars as well.

3.2.2. The effect of milling time on interface morphology of 316L-50W/W_p samples

SEM images of the interface between W_p and the as-mixed 316L-50W (Sample 3) as well as those milled for 5 h and 10 h (Samples 4 and