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2019

Kong, W., Li, X., Chen, B., Wang, C., Chu, H., Chen, Y., . . . Zhou, W. (2019). Microstructure and mechanical properties of diffusion bonded W/MA956 steel joints with a titanium interlayer by SPS. *Journal of Adhesion Science and Technology*, 33(17), 1847-1857.
doi:10.1080/01694243.2019.1615723

<https://hdl.handle.net/10356/144445>

<https://doi.org/10.1080/01694243.2019.1615723>

This is an Accepted Manuscript of an article published by Informa UK Limited, trading as Taylor and Francis in *Journal of Adhesion Science and Technology* on 13 May 2019, available online: <http://www.tandfonline.com/10.1080/01694243.2019.1615723>

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Microstructure and mechanical properties of diffusion bonded W/MA956 steel joints with a titanium interlayer by SPS

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Abstract

Diffusion bonding is an effective technique for joining dissimilar metals. In this paper, tungsten and MA956 steel were diffusion-bonded by Spark Plasma Sintering (SPS) technique with titanium (Ti) foil as an interlayer. The bonded joints were evaluated by metallographic analysis and mechanical tests, and the results reveal that all W/Ti/MA956 joints were well bonded by efficient SPS technique. Microstructure analysis showed that W-Ti solid solution formed at W/Ti interface; reaction phases at Ti/MA956 steel interface varied with the joining temperature, e.g. intermetallics phases FeTi for 850 °C, FeTi, Fe₂Ti and Cr₂Ti for 900 °C and 950 °C joining temperature. The peak value of microhardness occurred at the interfaces of Ti/MA956 steel owing to the formation of intermetallic compound. All specimens of shear testing fractured at the Ti/MA956 steel interface close to MA956, and the average shear strength of joints was 182 MPa, 228 MPa and 164 MPa bonded at 850 °C, 900 °C and 950 °C respectively.

Keywords: Diffusion bonding; SPS; W; MA956 steel; Metallographic analysis

Introduction

Experimental Advanced Superconducting Tokamak (EAST) realized the central electron temperature of 100 million degrees since that of 50 million degrees achieved in 2016, which was the first in the world up to now. The data obtained by EAST laid an important foundation for the development of clean nuclear fusion energy.

Nuclear fusion reactor test equipment, including EAST, ITER and DEMO, has been developed rapidly. The divertor serves as the vital part of the thermal fusion

reactor, and has the functions of collecting waste particles and impurities from the plasma and removing part of the reactor heat [1]. In the divertor design, owing to their good thermal conductivity and high resistance to radiation damage [2-7], tungsten (W) and oxide dispersion strengthened (ODS) steels were often selected as plasma facing material and structural material respectively. Hence, the joining of these two materials has been considered as one of the key technologies for a divertor [8-10].

In view of the huge differences in physical properties between W and MA956 ODS steel (UNS S67956), large residual stresses appear along the bonding interfaces. Traditional fusion welding is inapplicable to join W and MA956 steel. Brazing is also a preferred method to bond dissimilar metals, but the grain coarsening and phase transformation caused by too high brazing temperature may deteriorate the properties of steel.

As reported, solid-state diffusion bonding has been regarded as one of the most promising methods for joining tungsten and steels [11]. Consideration of the significant difference in physical properties of these two materials, such as the huge difference in their coefficients of thermal expansion (the CTE of W and MA956 is $4.5 \times 10^{-6} \text{ K}^{-1}$ and $11.1 \times 10^{-6} \text{ K}^{-1}$ (20°C), $5.2 \times 10^{-6} \text{ K}^{-1}$ and $14.6 \times 10^{-6} \text{ K}^{-1}$ (1000°C), respectively [12] [13]), direct bonding of W to ODS steel could cause a large residual stress in the joints. In order to improve the comprehensive properties of the diffusion bonded joint, an interlayer is often chosen to minimize the residual stress and decrease the formation of brittle metallic compounds. Zhong et al [11,14] investigated the diffusion bonding of W to steel with Ni interlayer and Ti interlayer to modify the joint

performance, Basuki and Aktaa et al [15, 16] bonded the W to EUROFER97 successfully by using a single Nb or V foil as interlayer. Wang et al [17] studied the effect of different thickness of Ti interlayer on the bonding quality of W/Ti/Steel HIP joint.

Spark Plasma Sintering (SPS) technique has been applied extensively to the joining of dissimilar materials as compared to the conventional diffusion bonding technology [18, 19]. Owing to its shorter holding time, faster heating and cooling rate, the grain growth of the substrate can be restrained by SPS technique. In this paper, efficient SPS diffusion bonding was adopted to join W and MA956 ODS steel with Ti interlayer. The interface microstructure, microhardness and mechanical properties of joints with Ti interlayer were analyzed.

Experimental procedure

High-chromium ODS ferritic steel (MA956) and commercially available pure W were used as the base materials in this paper. The diameter and height of MA956 and W were 20 mm and 5 mm, shown in Fig. 1. A 40 μm thickness commercial Ti foil was used as an interlayer. The chemical compositions of the materials are given in Table 1. Prior to diffusion bonding, the joining surfaces were first ground on SiC abrasive paper, then polished by 5 μm diamond, and finally ultrasonically cleaned in alcohol for 20 min. After drying the samples and interlayer, the W and MA956 steel were stacked with Ti interlayer and placed in the middle of graphite mould.

The diffusion bonding experiment was carried out in SPS furnace with the vacuum of approximate 0.4 Pa. The joining temperature, holding time, and pressure

were 850-950 °C, 10 min, and 10 MPa, respectively. The thermal cycle of the bonding process was as follows: firstly heated to joining temperature with heating rate of 100 °C /min, held 20 minutes at joining temperature, and then cooled to 300 °C at a rate of 20 °C /min, finally cooled with furnace to room temperature.

After bonding, the bonded joints were cut into rectangular blocks ($\approx 10 \times 10 \times 10$ mm³). The surfaces of blocks were first ground by SiC abrasive paper, and then polished for microstructure observation. Vickers hardness was tested across the bonding interface by VTD401 tester with a dwelling time of 10 seconds and a load of 50 g. The specimen for shear strength test is 10 mm $\times$ 3 mm $\times$ 3 mm in size, which was assessed on CMT5150 Universal tester with a constant displacement rate of 0.5 mm/min, and the average value of three test results was calculated for each joining condition. Shear test mould and the macroscopic image of the fracture joint are shown in Fig. 2. The field-emission scanning electronic microscope (FE-SEM, Zeiss Sigma 300) equipped with energy dispersive spectroscopy (EDS, Oxford Inca Energy350) was used to analyze the microstructure, element distribution of the joint interface, and the fracture surface. X-ray diffraction (XRD, D/MAX2500V) was used to identify the formed phases.

Results and discussion

Microstructural Characterization

Microstructural examination was carried out through FE-SEM and EDS. The typical general view of SPS joint bonded at 850 °C is given in Fig. 3. It can be observed that W and MA956 steel were successfully bonded with Ti interlayer, No

obvious defects were observed along the joint bonding interfaces. Fig. 4 (a)-(c) display the joints microstructure bonded at 850 °C, 900 °C and 950 °C respectively. Although interdiffusion occurred at different bonding temperature, the diffusion zones of W/Ti interface were not wide enough to observe obviously. Micro-cracks were found at W side of W/Ti interface in the 950 °C SPS bonded joint shown in Fig. 4 (c), due to the large CTE mismatch between W and MA956 steel, and the residual stress of the joints develops with the joining temperature increasing [20]. Compared with the W/Ti interface, as a result of continuous atoms inter-diffusion and inter-reaction between Ti and MA956 steel, apparent diffusion zones can be observed at the Ti/MA956 steel interface.

EDS line-scan was used to analyze element distribution in the joints. Atom percentage of the detected zones was listed in Table 2. Fig. 4 (d)-(f) are the distribution of element concentration across the W/Ti interface and the Ti/MA956 steel interface bonded at 850-950 °C, and the profile curves reveal the diffusion paths of element W, Ti, Fe and Cr. The smooth variation of the profile curves indicated no intermetallic compounds formations but solid solution at W/Ti interface, as shown in Fig. 4 (e). However, Fig. 4 (d)-(f) show that the profile curves appear the “steps” at the Ti/MA956 steel interfaces, indicating the formation of different reaction regions. Moreover, the thickness of the reaction region at Ti/MA956 steel interface grows from 6.8 to 12.9 μm corresponding to the joining temperature from 850 to 950 °C.

Combined with the results of EDS in Table 2 and Fig.4 (a), Fig.4 (d), the Fe-Ti and the W-Ti binary phase diagram, the phases formed at the reaction zone were

readily analyzed. The atomic percent of W and Ti in region 1 is approximately 1:1, this confirmed the formation of the W-Ti solid solution in this region according to W-Ti binary phase diagram [21]. Region 2 is consisted of Ti (43.88 at. %), Fe (47.77 at. %) and Cr (8.35 at. %), corresponding to FeTi [15]. Hence the cross-section of SPS joint interface at 850 °C can be summarized as five regions: W base metal/W-Ti solid solution/Ti/FeTi phase /MA956 steel.

In Fig. 4 (b), 4 (e), region 3 adjacent to Ti was FeTi phase with average amount Ti (46.37 at. %), Fe (50.46 at. %), Cr (3.17 at. %); according to the Fe-Cr-Ti ternary phase diagram [22], region 4, containing Fe (60.34 at. %), Cr (10.69 at. %) and Ti (28.97 at. %), was suggested as Fe₂Ti and Cr₂Ti phase [23]. It is obvious that the increase of bonding temperature adds the variety of intermetallic compounds at the Ti/MA956 steel interface.

Shear Strength Evaluation

The strength and reliability of W/steel SPS joints are vital for the applications. Intermetallic compounds in diffusion zone have a significant effect on the properties of dissimilar material joint [24]. Shear strength of SPS joints obtained with different joining temperature were presented in Fig. 5. When joining temperature rose from 850-900 °C, higher strength joints were achieved. The average shear strength of the joint was increased from 182 MPa (850 °C) to 228 MPa (900 °C) and reached the maximum value at 900 °C. Due to the low bonding temperature at 850 °C restricted the interdiffusion of elements at interfaces, and thin diffusion zone was obtained. Up to 900 °C joining temperature, the increase of plastic deformation at the mating

surface promoted the contact quality and increased the width of the diffusion zone. In addition, the extent of interdiffusion of chemical elements was enhanced evidently at higher temperature. Although the diffusion zone became wider and a better plastic deformation happened at the mating surface, the lowest shear strength of 164MPa was obtained when the joining temperature was risen to 950 °C, owing to the formation of large amount of brittle intermetallics made the joint more brittle.

Compared with the results in ref. [14] and ref. [17], the higher shear strength was achieved in our experiment at the optimal SPS bonding parameters (228MPa at 900 °C, 20min).

Fracture surface analysis

In order to study the failure mechanism of joints, the fracture surfaces were surveyed after shear tests. From the fracture surfaces morphology in Fig.6 and the results of EDS analysis in Table 3, the specimens all fractured at the interfaces of Ti/MA956 steel. The fracture behavior mainly depended on the amount of the formed intermetallic phases (FeTi, Fe₂Ti and Cr₂Ti). Fig. 6 (a) shows fracture surface morphology of the joint at 850 °C, which is a basically facet type. Because of the slow diffusion rate of elements in the base metal at low temperature, the thickness of interfacial diffusion layer at Ti/MA956 steel interface is relatively thin. On the basis of EDS analysis, the dark region 1 of the fracture surface attached on the MA956 side is Ti (89.37 at. %). The atomic percent of region 2 is Ti (41.29 at. %), Fe (49.36 at. %) and Cr (11.35 at. %), the phase is suggested to be FeTi.

Transgranular brittle fracture with the appearance of obvious river patterns morphology occurred in the fracture surfaces of the joints (bonded at 900 °C) at W

side. Combining with Fig. 6 (b) and the EDS results shown in Table 3, the average composition of the cleavage facets (region 3) is Fe (10.87 at. %), Cr (3.25 at. %), Ti (85.88 at. %), corresponding to Ti phase; the gray area (region 4) was detected and FeTi phase was found with the atomic percent of Fe (49.49 at. %), Cr (7.51 at. %) and Ti (the balance); the bright area (region 5) contains Fe (57.57 at. %), Cr (11.97 at. %) and Ti (30.46 at. %), corresponding to Fe₂Ti and Cr₂Ti phases. Fracture morphology and intermetallics of joint at 950 °C were similar to that of at 900 °C. However, the amounts of the reaction formed phases (Fe₂Ti and Cr₂Ti phases) increased with the joining temperature rising from 900 to 950 °C. Based on the above-mentioned analyses of fracture surfaces, it could be inferred that the fracture mainly occurred at the Ti/MA956 steel diffusion zone where intermetallics FeTi, Fe₂Ti and Cr₂Ti were concentrated.

Fig. 7 shows the XRD pattern of the fracture surface on MA956 side of the joint bonded at 950 °C, which indicated that the reaction formed phases including FeTi, Fe₂Ti and Cr₂Ti were formed at the interface of Ti/MA956 steel. This was in good agreement with the previous work [14].

Microhardness test

Fig. 8 shows the hardness variation across profiles of the interfaces of joint bonded at 950 °C. The test points began with the W substrate and ended at MA956 steel substrate close to the interface of Ti/MA956 steel. At the W/Ti interface, it is observed that hardness value of W substrate is approximately 416 HV, while Ti interlayer has the hardness value of about 290 HV. Compare to the farther location in

Ti interlayer, the hardness value of W-Ti diffusion zone increases slightly and reaches 350 HV for the formation of W-Ti solid solution. Hardness value of MA956 substrate is below 200 HV, peak value 613 HV is located at diffusion zone between Ti and MA956, which suggests the formation of intermetallics in this region.

Conclusions

W and MA956 steel was connected with Ti interlayer using the SPS method. The optimal bonding parameter was 900 °C, 20 min, 10 MPa. W-Ti solid solution formed at W/Ti interfaces, the hardness value of W-Ti diffusion zone is between W and Ti, which can alleviate the joint thermal stress. Intermetallics such as FeTi for 850 °C, FeTi, Fe₂Ti and Cr₂Ti for 900 °C and 950 °C can be detected at the interface of Ti/MA956 steel, where the peak value of the hardness is located. The shear strength of the joint increased firstly and then decreased with the joining temperature rising, and reached the maximum value of 228 MPa at the bonding temperature of 900 °C. The joints all fractured at the Ti/MA956 steel interface in shear strength test.

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