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Microstructure, residual stresses and mechanical properties of diffusion bonded tungsten–steel joint using a V/Cu composite barrier interlayer

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ABSTRACT

Diffusion bonding is a preferred method to join W and steel for divertor applications. To minimize the residual stress induced by the large mismatch of thermal expansion coefficients and to inhibit the formation of brittle intermetallic phases, a V/Cu composite barrier interlayer was designed and examined to produce a joint between W and steel. The diffusion bonding was carried out at 1050 °C for 1 h under a 10 MPa pressure in vacuum. Metallographic analysis revealed excellent bonding at all of the joining interfaces. Neither intermetallic compounds nor other brittle phases were found in the bonded region. Nano-indentation test across the joint interfaces demonstrated the effect of solid solution strengthening in the diffusion zone. The strength of the joint was as high as 402 MPa and the failure occurred predominantly at the W substrate near the W/V interface due to the residual stress concentration.

Introduction

Tungsten (W) is a promising refractory material for fusion nuclear application and for its high resistance against sputtering and low tritium retention [1]. To apply W as a structural material, a joint to the basic structural materials, ferritic–martensitic high chromium steels, is foreseen in the current divertor design for demonstration reactor (DEMO) [2]. The method for realizing such kind of connection is restricted by a lot of problems related to the large differences in the physical properties between W and steel. For instance, the large difference of their melting temperature (3422 °C for W and ~1536 °C for steel depending on the alloying elements) do not make them possible to be joined by means of conventional fusion welding techniques. Furthermore, W and steel have a remarkable difference in their coefficients of thermal expansion (CTE, 4.5×10^{-6} K^{-1} for W and $12–14 \times 10^{-6}$ K^{-1} for steel at room temperature (RT)). Temperature changes induced during cooling from the joining temperature and during subsequent service can generate high internal stresses due to the CTE mismatch and lead to poor joint strength or failure [3,4].

Among the techniques developed for joining of W to steel, brazing [5–7] and diffusion bonding [8,9] are the most suitable methods for joining of W to steel. However, the process temperature of brazing is usually high enough to cause grains seriously coarsening in steel (e.g., EUROFER97 [8]), and consequently leads to property degradation. Diffusion bonding is an attractive method to join W with steel due to its tolerable bonding temperature and the joint could be used at high temperatures. In order to obtain a sound diffusion bonded joint, an interlayer inserted between substrates is often necessary to reduce the residual stress and to prevent the formation of intermetallic compounds [10–12]. Previous work deals with diffusion bonding of W to steel using a single metal foil as an interlayer, such as Ni [9,13], Nb [14], Ti [15] or V [16]. Though such sandwich design eases the stress concentration and suppresses direct reaction between both base metals, which promotes the formation of the brittle intermetallic phase FeW and metal carbides [8], the single metal interlayer itself may react with one or both of the base metals to form new brittle intermetallic phases or metal carbides, which might have a detrimental effect on mechanical properties of the bonded joint. Zhong et al. [9,13] tried Ni as an inserted material for diffusion bonding of W to steel. The maximum strength of 215 MPa was obtained for the sample bonded at 900 °C for 1 h and the strength decreased with increasing bonding temperature or holding time due to the higher volume fraction of Ni_4W intermetallic compound. Basuki and Aktaa [14] reported the bonding of tungsten and EUROFER97 using a Nb interlayer, and they found that the bonded sample showed a hard reaction layer consisted of niobium carbides (Nb_2C and Nb_6C_5) at the interface between EUROFER97 and Nb, which could weaken the joint interface. Thus the joint quality is still necessary to be improved.

The aim of the present work is to propose a new way to diffusion bonding of W to steel using V/Cu combination as an interlayer. The inserted materials between W and steel were chosen in order to: (1) prevent the formation of intermetallic compounds and other brittle phases between the base metals (W or steel) and interlayers (compatibility among different materials), and (2) reduce the residual stress in the joint. The refractory metal V was selected as an inserted material because the CTE of V is between that of W and steel which is expected to help mitigate the residual stress, as well as it forms a

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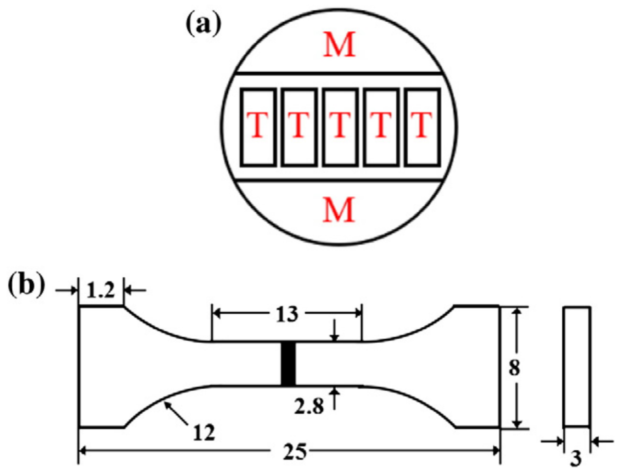

Fig. 1. (a) Sectioning outline on the top view of the diffusion bonded sample, M: specimens for microstructure and hardness investigations, T: tensile test specimens, and (b) schematic drawing for tensile testing (all dimensions are in mm).

continuous solid solution with W. Moreover, vanadium alloys (e.g., V–4Cr–4Ti alloy) are applicable in the fusion reactor due to their low induced activation, excellent thermal stress factor, high strength at elevated temperatures, and superior ductility at low temperatures [17]. However, V is not compatible with steel due to the formation of the brittle phase (V_2C or δ) with a very high hardness of about 14 GPa [16]. Cu could be considered as a potential candidate to be used as an inserted material between V and steel because Cu has a substantial solid solubility in V and does not form brittle phases with steel. Besides, Cu is a non-carbide forming element which can inhibit the diffusion of C atoms from steel to V [18]. In particular, it is a soft metal which deforms and accommodates the stresses caused by the mismatch of thermal expansion coefficients. Meanwhile, copper alloys (e.g., Cu–Cr–Zr alloy) have also been proposed as heat sink materials in high heat flux components due to their high thermal conductivity [19].

The present study reported diffusion bonding of tungsten to steel using a V/Cu composite barrier interlayer which can inhibit the formation of the hard and brittle intermetallic compounds. The strength of the joint was measured by tensile testing and the site of fracture was compared to the residual stress distribution estimated by a finite element method (FEM).

Experimental procedure

The base materials used in this study are a high-Cr ferritic steel (Fe–17Cr–0.1C in wt.%) and commercially available W (99.95 wt.% purity). The samples were cut into a size of Ø25 mm × 13 mm. The commercial Cu (99.9 wt.% purity, 0.1 mm thickness) and V (99.95 wt.% purity, 0.2 mm thickness) foils were selected as insert materials. The surfaces of the materials to be joined were polished and then ultrasonically cleaned in acetone.

The prepared materials, assembled in the structure of W/V/Cu/steel, were mounted in the bonding chamber. The diffusion bonding was carried out at 1050 °C for 1 h under a 10 MPa pressure in vacuum ($<10^{-3}$ Pa). The bonded samples were cooled to 400 °C at a rate of 5 $°C\cdot min^{-1}$ and followed by furnace cooling to RT in vacuum.

After bonding, the bonded sample was sectioned into small specimens for microstructure and mechanical property (hardness and tensile tests)

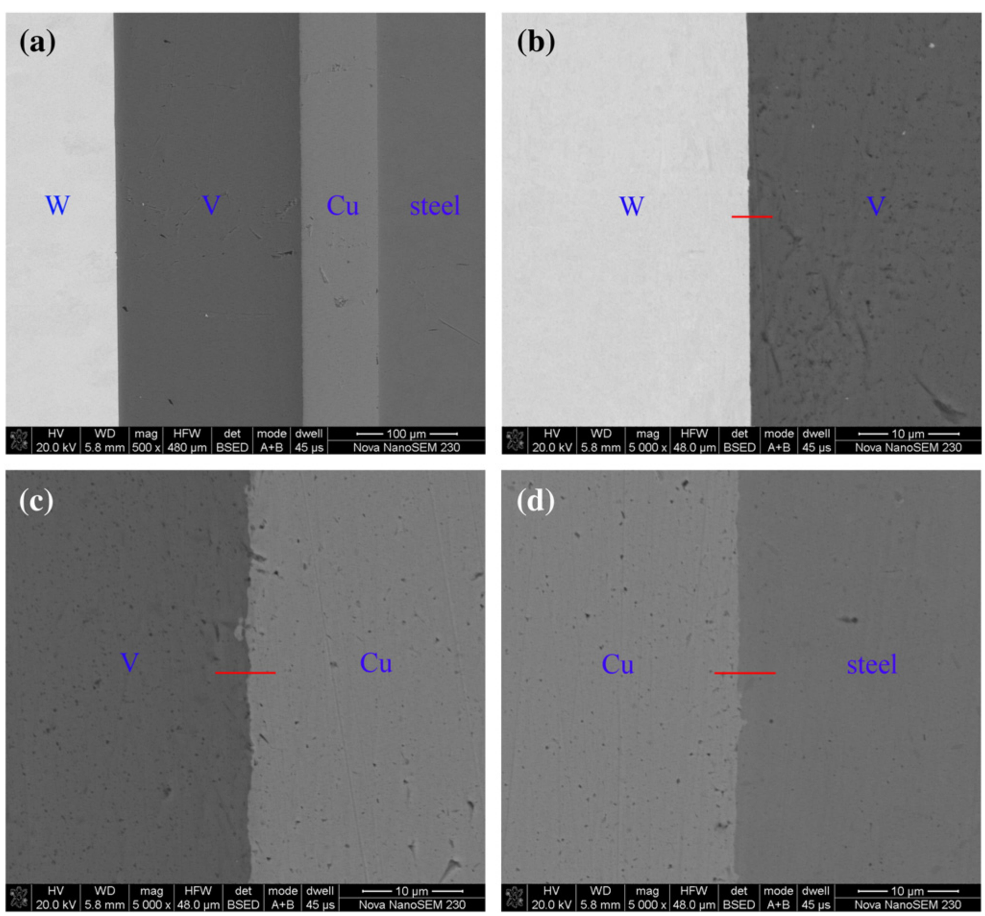

Fig. 2. SEM images of the cross-section of W/V/Cu/steel joint: (a) general view, (b) W/V, (c) V/Cu, and (d) Cu/steel interfaces.

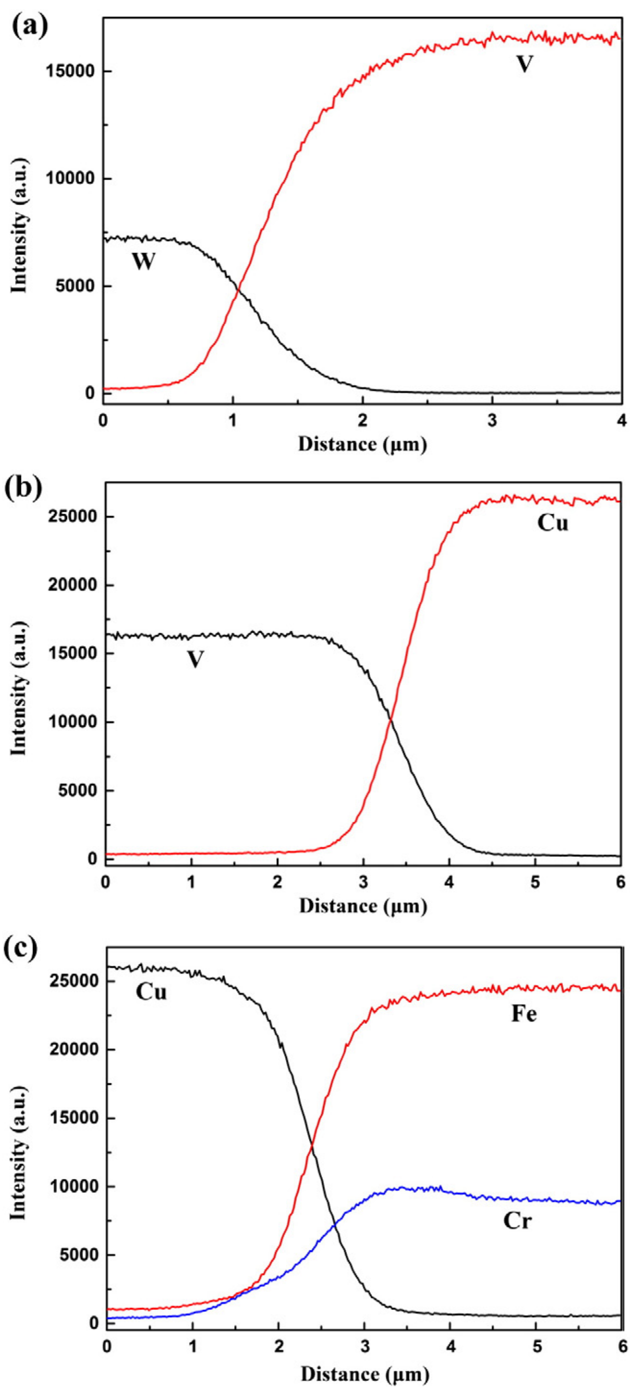

Fig. 3. Elemental concentration profiles across the: (a) W/V, (b) V/Cu, and (c) Cu/steel interfaces.

investigations (Fig. 1a). The microstructure and the chemical composition along the bond seams were analyzed using a field-emission scanning electron microscope (SEM) and electron probe microanalysis (EPMA). The phases in the joint interfaces were determined by X-ray diffraction (XRD, D/MAX-Rapid) with Cu Kalpha radiation. The local hardness distribution across the joint interface was examined by a nanohardness tester (VNHT) with a load of 5 mN. To determine the tensile properties of the bonded sample, transverse tensile test specimens were manufactured with a gauge length of 13 mm shown in Fig. 1b. The coupons were machined in such a way that the weld was positioned at the center of gauge length. Tensile tests were conducted by a tensile testing machine (Instron-3369) at RT with a crosshead speed of 1 mm/min. An average tensile test value was obtained by

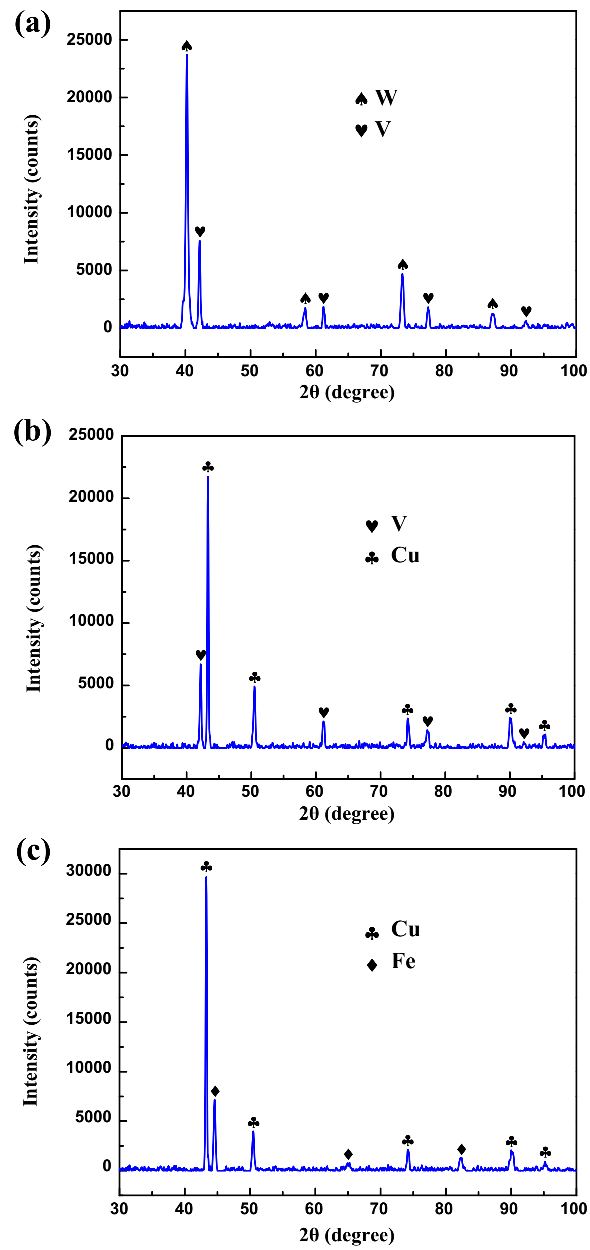

Fig. 4. X-ray diffraction patterns measured on the cross-section of (a) W/V, (b) V/Cu and (c) Cu/steel interfaces.

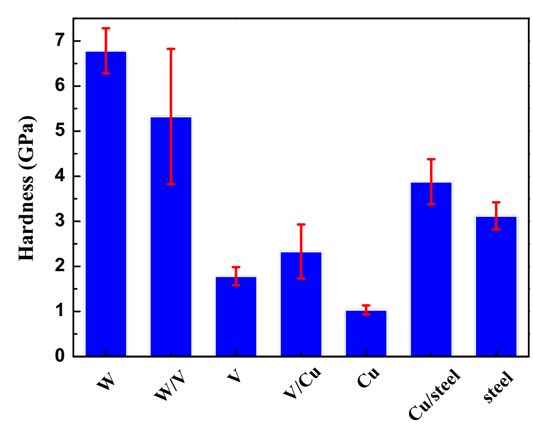

Fig. 5. Nanohardness of the substrates (W, steel), interlayers (V, Cu), and diffusion zones at the joint interfaces (W/V, V/Cu and Cu/steel).

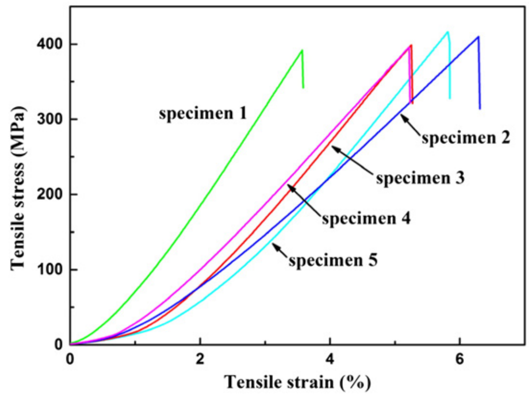

Fig. 6. Tensile curve of tensile specimens tested at RT.

testing five specimens. Finally, evaluations of the joint fracture surfaces were carried out by SEM combined with energy dispersive spectroscopy (EDS).

Results and discussion

Microstructure characterization

Fig. 2a shows the general view of the transition joint bonded. It has been observed that diffusion bonding of W to steel was successfully done using a V/Cu composite interlayer. The diffusion zone is free from cracks or discontinuities and the bond line is clearly visible. The higher magnification micrographs showing the detailed microstructures at W/V, V/Cu and Cu/steel interfaces are presented in Fig. 2b–d, respectively. Although interdiffusion occurred, neither intermediate phases nor reaction products were observed at all of the interfaces within the resolution limit of SEM. In addition, it should be noted that, compared with the W/V interface, a corrugated deformation pattern occurred in both V/Cu and Cu/steel interfaces, which might be attributed to the plastic deformation of Cu at V/Cu and Cu/steel interfaces under compression stress and high joining temperature. This was related to the following facts: (1) the softer nature of Cu in comparison with W, V and steel and (2) the proximity of the joining temperature to its melting point. This corrugated interface could produce a sound and robust interface by blocking the interstices [20,21].

In order to determine the elemental distribution and migration behavior, EPMA line-scan was done for the elements of interest in the joint. Fig. 3 is the elemental EPMA concentration profiles across the W/V, V/Cu and Cu/steel interfaces, respectively. The penetration curves of the chemical species exhibit a gradual change in concentration profiles, indicating the absence of intermetallic compounds in the diffusion zones but solid solution formation. The X-ray diffraction studies confirmed the absence of intermetallic compounds in the cross-section of the W/V, V/Cu and Cu/steel interfaces and are given in Fig. 4. The results are consistent with the W–V, V–Cu, Cu–Fe and Cu–Cr phase diagrams [22] which exhibit the good compatibility for W and V, V and Cu, Cu and steel.

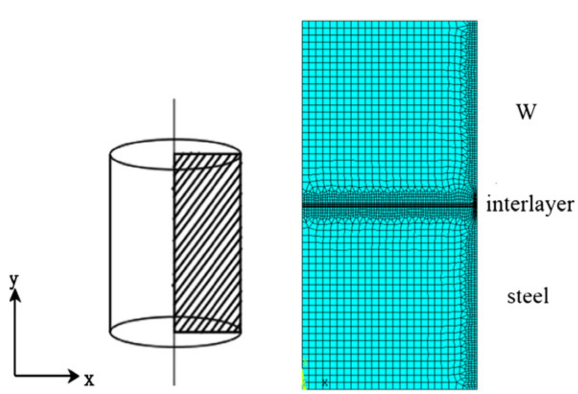

Fig. 7. Schematic representation of the model geometry resulted from the geometric simplifications (left) and the mesh configuration (right) used in the FEM calculation.

Table 1
Material properties employed in FEM.

Property	Temperature (°C)	Elastic modulus (GPa)	Poisson ratio	Thermal expansion coefficient (10^{-6}/°C^{-1})	Yield strength (MPa)
W[a]	20	398	0.28	4.7	1360
	200	397	0.28	4.7	1154
	400	394	0.28	4.9	948
	600	390	0.29	5.0	765
	700	386	0.29	5.1	682
	900	378	0.29	5.2	532
	1050	370	0.30	5.3	433
Steel[b]	20	217	0.31	10.4	500
	200	207	0.31	11.2	453
	400	197	0.31	11.9	402
	600	178	0.31	12.5	194
	700	161	0.31	12.6	100
	900	156	0.31	12.8	21
	1050	130	0.31	12.9	15
V[b]	20	128	0.37	7.8	380
	1050	110	0.37	10.0	100
Cu[b]	20	130	0.35	17.2	78
	1050	–	0.35	20.3	–

[a] Data from [3,26].
[b] Data from the TMA experiment.

Mechanical properties

The micro-mechanical properties of ceramic/metal and metal/metal joint interfaces can be evaluated by nano-indentation test, which is a useful technique to evaluate the mechanical properties of either films or small volumes of materials. The hardness was evaluated on the polished cross-section of the joint at the W/V, V/Cu and Cu/steel interfaces. The hardness of the substrates (W, steel), interlayer (V, Cu), and diffusion zones at W/V, V/Cu and Cu/steel are all presented in Fig. 5. The spread of hardness values for diffusion bonded joint, which was experimentally observed by most authors [23–25], was probably credited to the effect of residual stress [26] and surface roughness [27] of the cross sectional joint. The measured hardness for W is about 6.8 GPa which is similar to that previously observed by Zhong et al. [9]. The hardness values of the V and Cu were comparable with those reported by Basuki and Aktaa [16] and Sabetghadam et al. [21], respectively. The hardness of steel used in this study is similar to EUROFER97 steel [14,16]. It can be noted that substantial changes were observed at the interfaces which were directly related to the composition and thickness of diffusion zones generated. The hardness values (~5.3 GPa) tested in the W/V diffusion zone is associated with the migration of W into V. Likewise, the diffusion zone of V/Cu shows a higher hardness as compared to the V or Cu due to the formation of a (V, Cu) solid solution. In the Cu/steel diffusion zone, a higher hardness is noted in comparison with the Cu or steel, according to the evidence of Fe or Cr penetration into the Cu interlayer and Cu penetration into the steel. The region-dependent hardness observed is ascribed to the solid solution strengthening effect which is related to the interdiffusion process.

The strength and reliability of W/steel joint are of importance for its applications. For the dissimilar material joints, usually the intermetallics and residual stress significantly affect joint properties [25,28,29]. The W/steel joint possesses a relatively high strength of ~402 MPa (Fig. 6) and the fracture predominantly occurs at the W substrate near the W/V interface during tensile testing, indicating that all of the interfaces have high interfacial strength. The fluctuation and deviation of tensile curve may be attributed to the following reasons: (i) the spread on

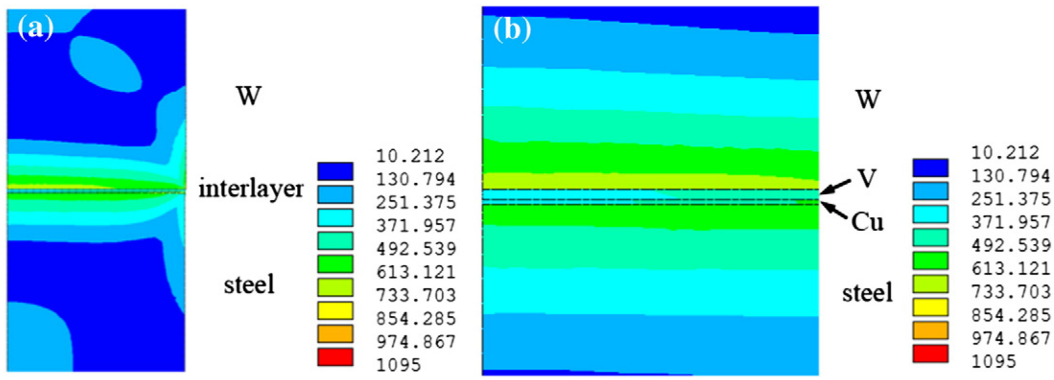

Fig. 8. Distribution of residual stress in W/V/Cu/steel joint. (a) von Mises stress distribution and (b) magnification of near the interface.

the strength level of the W itself, owing to its brittle nature and sensitivity of impurity distribution; and (ii) the differences in the magnitude and distribution of residual stress induced by CTE mismatch in the joint. Because the strength of the bonded specimens is effectively limited by the strength of the W, residual stresses are responsible for reducing the strength of the bond below the intrinsic strength of the W, which is consistent with the previous observation by Zhong et al. [9].

Once the bonding was obtained, stresses were induced in the joint during quickly cooling from the bonding temperature to RT due to the large difference in thermal shrinkage between W and steel. These thermal stresses can strongly influence the strength and failure characteristics of the joint during subsequent mechanical loading. So FEM was applied to determine the magnitude and distribution of the residual stresses in the joint.

Residual stress distribution

FEM calculations were made to obtain the residual stresses which developed in the bonded sample as it was cooled from the bonding temperature to RT (from 1050 °C to 20 °C). The FEM model is established for a 2-D axisymmetrical thermal elastic–plastic stress analysis to the joint. Due to the axial symmetry of the joint (Fig. 7), an arbitrary meridian plane is selected to analyze the stress in the joint with four-node axisymmetrical elements. The division of the finite element mesh is shown in Fig. 7. The material properties employed are shown in Table 1, according to the thermomechanical analysis (TMA) experiments and some references [3,30].

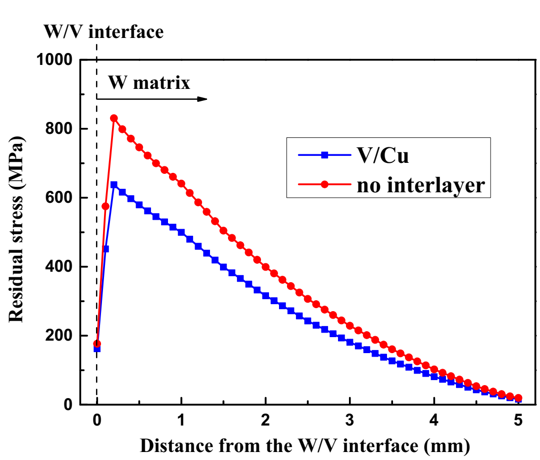

Fig. 9. Residual stress profile in the W substrate as a function of distance from the W/V interface.

Fig. 8 shows the contour plots of the von Mises stress distribution for the W/V/Cu/steel joint. It can be seen that the maximum residual stress located adjacent to the joining interface in the W substrate. Accordingly, it can be concluded that the residual stress is much more detrimental for W than for steel. To analyze the stress distribution in W in detail, Fig. 9 presents the stress distribution along the axis of symmetry in the W substrate as a function of distance from the W/V interface. The stress profiles confirmed that the residual stress in the W side was maximum in the vicinity of the interface; it might induce fracturing in W near the interface, which was consistent with the experimental result.

Additional FEM analysis was performed, comparing a V/Cu interlayer with no interlayer. It was verified that residual stress acting on the W substrate was reduced by using a V/Cu interlayer (Fig. 9), which could be attributed to the dual-effect of V and Cu. When cooling from the bonding temperature, the V interlayer would transfer some residual stress from W and the soft Cu interlayer would relieve the residual stress by plastic deformation. This suggests that the design of a V/Cu composite interlayer between W and steel relieved some residual stress, thus enhancing the joint properties.

Fracture characteristics

Fig. 10 shows a typical fracture surface on the W side of the joint. Fracture occurs in the brittle mode and is characterized by faceted grains. Furthermore, some phases on the fracture surface were identified as containing W and V by EDS, which indicated that the fracture took place predominantly in the W substrate, and some in both the W/V interface and V interlayer. The residual stress at the W/V interface was relatively high though it was not the maximum one. The joining interfaces, which are considered to be weak due to the presence of reaction areas, were also prone to rupture under the influence of the residual stress. Therefore, the failure behavior of the joint during tensile testing can be explicated that the crack initiated in the W substrate near the W/V interface due to the residual stress concentration and then propagated rapidly along the W grain boundaries and partly into the W/V interface and V interlayer.

In the previous attempt, Ni [9,13], Nb [14], Ti [15] and V [16] have been used separately as interlayer materials to produce the W/steel transition joints and achieved the joint strength of about 215, 272, 113 and 207 MPa at RT, respectively. Compared with them, the W/V/Cu/steel joint with a higher strength was produced in this work, which can be attributed to the beneficial effects on reducing the residual stresses and preventing the brittle intermetallic compound formation in the joint by the design of a V/Cu composite interlayer.

Conclusions

Due to the technological interest for fusion applications, using a V/Cu interlayer for joining of W to steel has been developed. Such interlayer

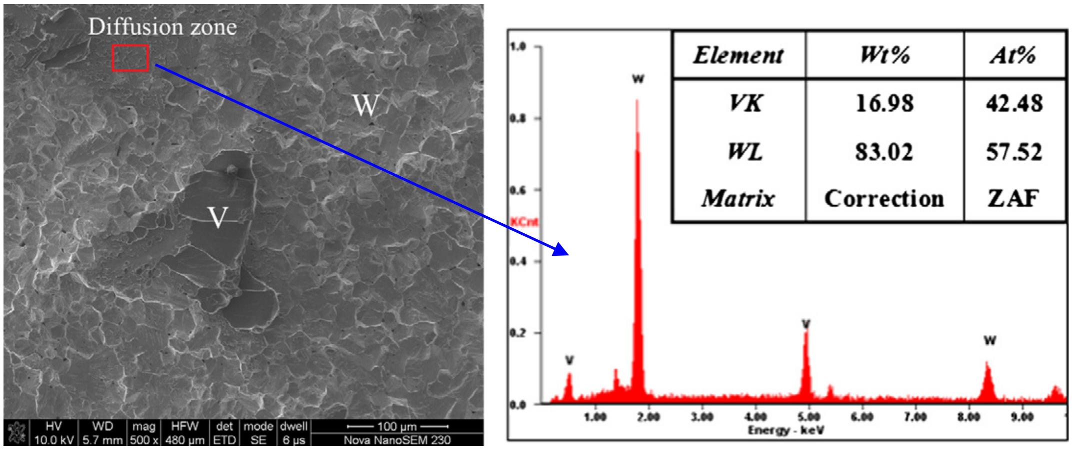

Fig. 10. Representative fractured surfaces on the W side.

was designed to prevent the formation of the hard and brittle intermetallic compounds and to reduce the residual stress in the joint. Microstructure, residual stresses, and tensile strength of the joint and hardness near the interfaces were investigated. The following conclusions were drawn:

(1) The proposed V/Cu composite interlayer was successful for diffusion bonding of W to steel. The capability of forming a strong W/steel joint using a V/Cu interlayer has been demonstrated by both microstructural examination and mechanical evaluation.
(2) The interface of W/V/Cu/steel was free from intermetallic compounds and other brittle phases, and the observed high hardness at both W/V, V/Cu and Cu/steel interfaces is ascribed to the formation of solid solution phases.
(3) The strength of the joint was as high as 402 MPa, which is clearly higher than that of the joint obtained by using a single metal interlayer in the previous work. The surface fracture analyses revealed a brittle fracture in the W substrate near the W/V interface.
(4) The FEM resulted in a satisfactory description of the residual stress distribution in W/steel joint. The results through FEM simulation showed that the maximum stress occurred at the W substrate adjacent to the joining interface. These results are in good agreement with the fractographic observation of the W/steel joint. The FEM simulation also suggested that the V/Cu interlayer reduced the stress level, when compared with that obtained without interlayer.

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