



Influence of neutron irradiation on mechanical properties of vanadium/ceramics joints

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Abstract

Neutron irradiation effects on bend strength of V/MgO and V/Al₂O₃ joints, which had been bonded at appropriate conditions, were studied to evaluate ceramic/metal joint for fusion materials using three-point bend test and fractography. Bend strength of V/Al₂O₃ joints was lowered by neutron irradiation, but that of V/MgO joints was not changed by neutron irradiation. It was considered that reduction of the strength of V/Al₂O₃ joints was not caused by the weakening of the interface, but caused by the formation of residual stress crack during irradiation. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Vanadium (V)-alloys are considered as candidate structural materials for fusion application because of their high temperature strength, low-swelling property and low-induced radioactivity. When V-alloys are selected as structural materials of fusion reactor, liquid lithium (Li) will be used as coolant material because of high efficiency of heat exchange rate and its compatibility with V. On the other hand, arising MHD loss from liquid Li flowing in magnetic field will be important problem in the V–Li reactor system [1]. In order to decrease the MHD loss, it is considered to join or coat V-alloys with ceramics which have insulation and compatibility with the liquid Li environment. CaO, AlN, MgO, Al₂O₃ and MgAl₂O₄ are proposed as insulator coating materials. In selecting ceramic coating material, thermal expansion rate (Fig. 1) and integrity of joining in V-alloys are important parameters to obtain appropriate joining/coating materials.

When joining metals and ceramics using high temperature process, generally, residual stresses are generated near the metals/ceramics interface due to the difference in each thermal expansion rate. If the stress is so large as to form cracks in the ceramics, performance

of the joints drastically decreases. It is considered that a brittle reaction layer which forms at the interface during joining process also degrades joint integrity. Therefore, ceramic/metal joints without reaction layer and without residual stress in the interface will be the most appropriate joint.

Effects of neutron irradiation is one of many important issues in fusion reactor materials. Neutron irradiation produces displacement damage and enhanced diffusion in materials. Irradiation induced mixing at the interface, dimensional change due to swelling, and rearrangement of residual stresses may affect bond strength during irradiation.

In previous work, it was found that joints of V/Al₂O₃ and V/MgO could be produced with high bond strength and no reaction layer [2,3]. The purpose of this work is to study and discuss irradiation effects on the V/Al₂O₃ and V/MgO joints from a fundamental standpoint.

2. Experimental procedure

Short cylinders of V(99.9%), MgO(99.9%) and Al₂O₃(99.5%) which were 8.0 mm diameter and 3.0 mm thickness were used as bonding samples. Ceramics samples were supplied by Nikkato. Bonding surface of each sample was polished with diamond paste and grinding wheel and finished by buffing. Polished samples were joined in a sandwich configuration putting V

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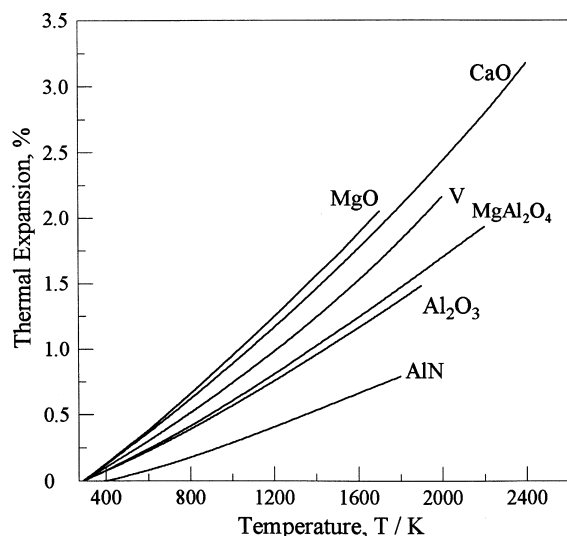


Fig. 1. Thermal expansion of vanadium and various ceramics.

between ceramics. Heat treatment for joining was carried out with Ta-plate heater under compression stress in vacuum. The joining temperature and compression stress of V/Al₂O₃ joints and V/MgO joints were 1673 K/5.5 MPa and 1473 K/3.0 MPa, respectively [3,4]. After the joining, miniature bend test specimens were cut from the joined samples with thickness of 0.4–0.6 mm and a width of 1.0–1.4 mm (Fig. 2). Those specimens were irradiated in JMTR with neutron fluence of 2.2×10^{24} n/m² ($E_n > 1$ MeV) at 823 K in He filled capsule. After the irradiation, three-point bend tests were conducted at the interface of the joint to estimate the bond strength after neutron irradiation. Bend strength of the ceramics was measured using the ceramics part of the joining specimen. Bend strength was calculated by Eq. (1).

$$\sigma = 9.8 \times 3PL / (2bh^2), \quad (1)$$

where σ is the bend strength, P the load, L the fulcrums length, b the width and h the thickness. Fracture surface of the tested specimens was observed by a scanning electron microscope (SEM) to study fracture mode.

3. Results

Fig. 3 shows bend strength of V/Al₂O₃ interface and Al₂O₃ matrix before and after neutron irradiation. Bend strength of some unirradiated joints specimens were as high as that of unirradiated Al₂O₃ matrix, though there is scatter in the strength arising from specimen position in unirradiated joining specimens [3]. On the other hand, bend strength of the irradiated joining specimens decreases after irradiation, and the scatter of strength data also decreased. In the case of Al₂O₃ matrix, change of bend strength by the neutron irradiation was not observed [4,5].

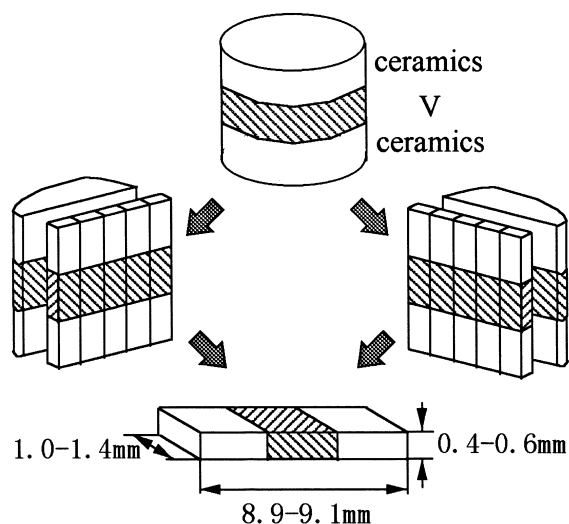


Fig. 2. A joined sample was cut in two equal parts. Miniature bend specimens for neutron irradiation were cut from the one, unirradiated specimens were done from the other.

Bend strength of V/MgO interface and MgO matrix before and after irradiation are shown in Fig. 4. Scatter of the strength was observed in both irradiated and unirradiated V/MgO joints, but change of bend strength by the irradiation was not observed. In the case of MgO matrix, bend strength was increased after irradiation. Maximum, minimum and average value of the bend strength of each joints and ceramics matrix were shown in Table 1.

Fractographs of V side of V/MgO and V/Al₂O₃ are shown in Fig. 5. Fig. 5(a) and (b) are micrographs of irradiated and unirradiated fracture surface of V/Al₂O₃ joints, Fig. 5(c) and (d) are that of V/MgO joints. These results show that irradiated V/Al₂O₃ joints did not fracture at the V/Al₂O₃ interface but at Al₂O₃ matrix. In the case of unirradiated joints, the fracture occurred in vicinity of the V/Al₂O₃ interface. It could be considered that Al₂O₃ matrix was weakened by neutron irradiation. But, the bend strength of Al₂O₃ matrix was kept after neutron irradiation (Fig. 3). The mechanism will be discussed in the next section. Fracture of both irradiated and unirradiated V/MgO joints occurred in MgO matrix in the vicinity of the V/MgO interface. Results of bend test and fractography indicate that the neutron irradiation conditions did not influence on mechanical properties of V/MgO joints.

4. Discussion

It has already been mentioned that the bend strength of V/Al₂O₃ joints decreased by neutron irradiation and fracture occurred in Al₂O₃ matrix. In spite of no change

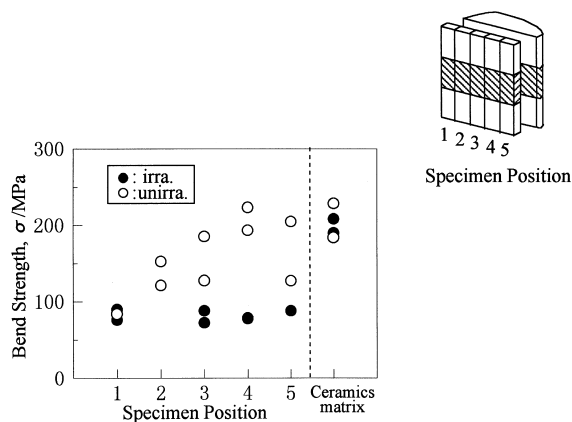


Fig. 3. Bend strength of V/Al₂O₃ joints and Al₂O₃ matrix irradiated at 823 K with neutron fluence of 2.2×10^{24} n/m² ($E_n > 1$ MeV). Right figure shows specimen position.

in the bend strength of Al₂O₃ matrix after irradiation, V/Al₂O₃ joints ruptured in the Al₂O₃ matrix, not in or near the interface. Fig. 6 shows the side surface structure of a ruptured specimen of irradiated V/Al₂O₃ joints. Fracture surface configuration from all at the direction (tensile, compression loaded surface and side surface) are consistent with residual stress crack direction generated during bonding process [2]. Previous work reported that residual stress crack generated above 1773 K in joining of V and Al₂O₃, and FEM analysis shows that residual tensile stress were concentrated at the edges of the interface [6]. Residual stress cracks were formed in the Al₂O₃ matrix in a lens like configuration parallel to the interface in the joints of V/Al₂O₃ because the thermal expansion rate of Al₂O₃ is smaller than that of V (Figs. 1 and 7). When bend tests were carried out on a specimen including such a crack, the bend strength will be lower, since tensile stresses act on tips of the cracks during the bend test (Fig. 7). Therefore, the decrease of bend strength might be attributed to the formation of residual stress crack in Al₂O₃ during the irradiation.

It is considered that residual stress cracks were formed at Al₂O₃ matrix during neutron irradiation, because the cracks did not exist before the irradiation. Though mechanism of the crack formation is not yet clarified, the related assumption is as follows: When hexagonal beryllium was irradiated with neutron, dis-

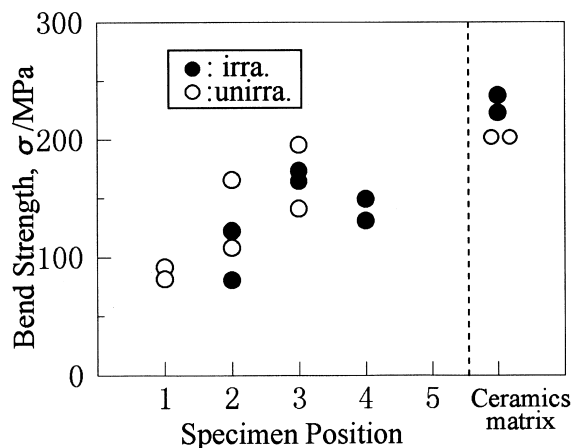


Fig. 4. Bend strength of MgO joints and MgO matrix irradiated at 823 K with neutron fluence of 2.2×10^{24} n/m² ($E_n > 1$ MeV).

location loops were formed predominantly on the basal plane, and so intergranular fracture was caused by the difference in grain growth between grain [7]. The dislocation loops were also predominantly formed on basal plane of Al₂O₃ and generated the stress at the grain boundaries [8]. Where the direction of the generated stress corresponds with that of the residual stress, micro-cracks may be formed at the grain boundaries.

There are no specimens ruptured at V/Al₂O₃ interface. The results showed the strength of interface is higher than that of ceramics matrix. Integrity of interface of V/Al₂O₃ joints which was irradiated at 693 K to a dose of 30 dpa were estimated using micro-indentation test and found to be the same after irradiation were reported [9]. Hence, it shows that degradation of the interface did not occur under this irradiation condition.

In the case of V/MgO joints, the bend strength and fracture mode were not changed by the irradiation. It is showed that integrity of V/MgO interface is not influenced by this irradiation conditions.

Since thermal expansion rate of MgO is larger than that of V, residual stress cracks in V/MgO joints formed perpendicular to the interface [3]. Such residual stress cracks do not largely influence on the bend strength, because of the direction of the crack propagation is

Table 1

Bend strength data of V/Al₂O₃ joints, V/MgO joints (maximum, minimum and average) and each ceramics matrix (average)

	Matrix		Joints					
	Unirra. average	Irra. average	Unirra.			Irra.		
			Max.	Min.	Average	Max.	Min.	Average
V/Al ₂ O ₃	208	199	223	84	158	90	73	82
V/MgO	204	230	196	81	130	173	80	137

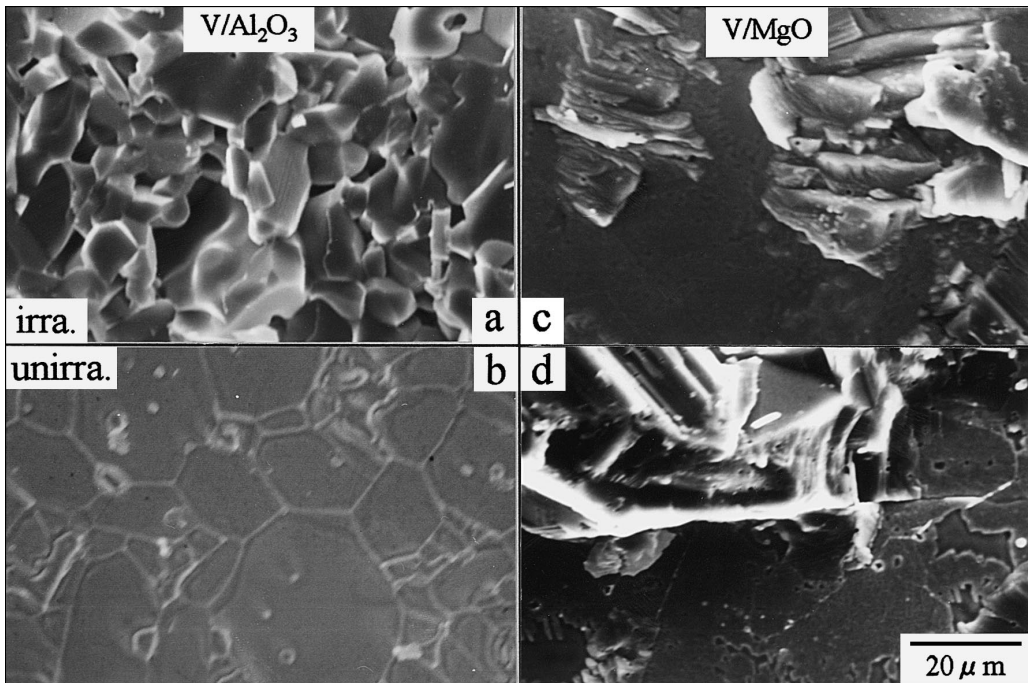


Fig. 5. SEM micro-photographs of fracture surface of V/Al₂O₃ joints and V/MgO joints tested at room temperature: (a) irradiated V/Al₂O₃ joints; (b) unirradiated V/Al₂O₃ joints; (c) irradiated V/MgO joints; (d) unirradiated V/MgO joints.

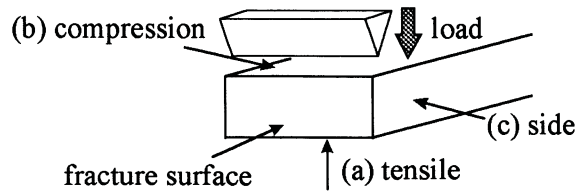
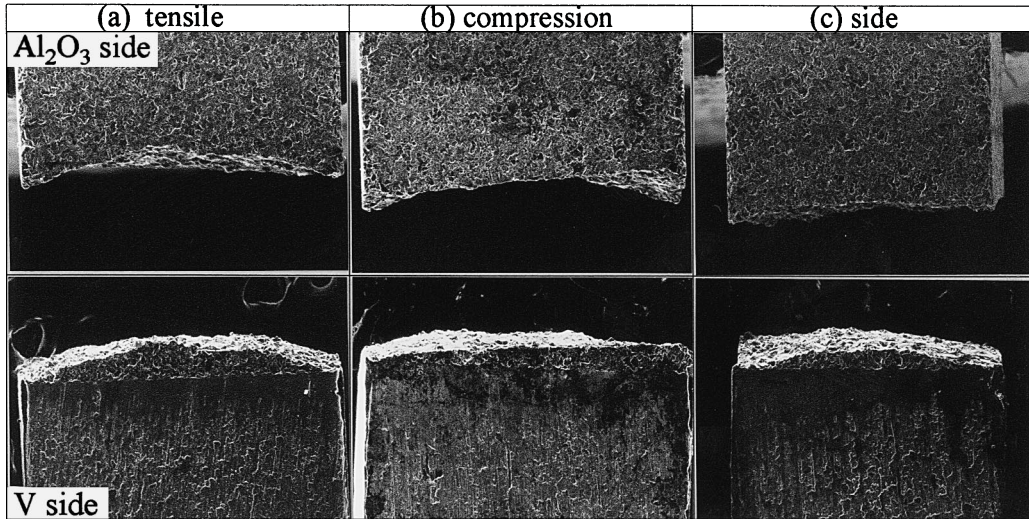


Fig. 6. SEM micro-photographs of side surface of ruptured specimens: (a) loaded tensile surface; (b) loaded compression surface; (c) side surface.

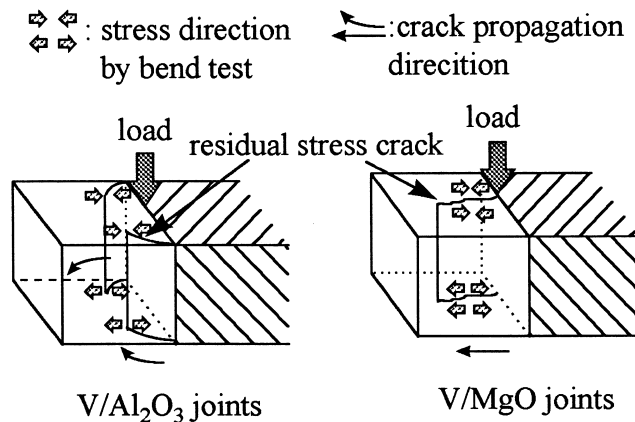


Fig. 7. Difference in residual stress crack propagation direction between V/Al₂O₃ joints and V/MgO joints.

parallel to that of stress which was generated by bend test (Fig. 7).

5. Summary

Integrity of the V/Al₂O₃ and V/MgO joints after neutron irradiation was evaluated by three point bend test and fractography. Bend strength of V/Al₂O₃ joints decreased due to the presence of residual stress cracks formed during neutron irradiation. However, it is important to notice that the V/Al₂O₃ interface maintained its integrity even after irradiation. Bend strength of V/MgO joints did not change after the neutron irradiation because the integrity of the interface remained stable even after irradiation and the influence of residual stress crack was not large.

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