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Microstructure characteristic and its influence on the strength of SiC ceramic joints diffusion bonded by spark plasma sintering

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ABSTRACT

The interfacial microstructure evolution and shear strength of SiC joints for high temperature applications diffusion bonded by spark plasma sintering with a Ta-5W interlayer in the temperature range of $1500\,^{\circ}$ C to $1700\,^{\circ}$ C were investigated. The interfacial microstructure analysis indicated that (Ta,W)C phase formed initially and (Ta,W)-Si intermetallic compounds subsequently at SiC/Ta-5W interface. Bonding temperature had a significant effect on the reaction layer thickness, which increased with increasing the bonding temperature, and holding time also has an influence on reaction layer thickness. Calculation of diffusion kinetics for the SiC/Ta-5W interface showed that the diffusion constant was about two orders of magnitude larger than that obtained by hot-pressing bonding, and the activation energy was almost one-tenth that of hot-pressing bonding. Both the reaction layer thickness and the interfacial defects had a great effect on the robustness of the joint, and the maximum shear strength of 122 ± 15 MPa was obtained for the joint bonded at $1600\,^{\circ}$ C for 5 min.

1. Introduction

Silicon carbide (SiC) is one of the most important engineering ceramics used at high temperature and hostile environments due to its good thermal shock resistance, excellent high-temperature strength, and good chemical stability [1]. SiC also has low induced radioactivity under neutron irradiation [2]. SiC fiber-reinforced SiC matrix composites (SiC/SiC composites) exhibit higher fracture toughness and lower scatter of mechanical properties than SiC ceramic, although its mechanical strength is not as high as that of SiC. Owing to the attractive characterizations of SiC/SiC composites, it is expected to be utilized widely as high temperature components such as blanket structural material in advanced fusion reactors and fuel cladding material in fission reactors [3-5]. The manufacturing processes of SiC and its composites, nowadays, have been developed a lot and reached a high maturity level, but the integrate technology of components are relatively limited. Hence, one of the crucial issues for their applications is joining technique. It is widely accepted that joining technique suitable for SiC material may in principle provide a reference for joining of SiC/SiC composites [6], thus most of joining techniques were carried out on SiC bulk material instead of expensive SiC/SiC composites. Several joining technologies for SiC including metal-based brazing [7,8], glass-ceramic

joining [9], MAX-phase joining [10], polymer-derived SiC joining [11,12], solid state diffusion bonding [13], Si-C reaction joining [14,15], transient eutectic-phase joining [16], have been developed.

Spark plasma sintering (SPS) that developed in recent years is a high efficient fabrication method, since the electric filed can accelerate ion diffusion, thereby contributing to the migration of ions through the joining interface and integrating the materials in a short time [17]. Comparing with the conventional hot-pressing (HP) bonding [10,13,18], SPS bonding shows an advance in joining of ceramics which usually requires high bonding temperature and long holding time. For example, it needed 60 min for obtaining a sound SiC/Ti/SiC joint at 1400 °C by HP joining [19], while the bonding temperature and holding time could be decreased to less than 1000 °C and 5 min, respectively, by SPS joining [20]. Also, SPS could bond the ceramics without the aid of joining interlayers in a short time [21,22], which had not been reported for ceramics joining by HP to the best of our knowledge.

It is widely accepted that the initial surface roughness of specimen creates a series of interfacial voids which subsequently may act as a stress concentration site and causes the early failure for the solid-state diffusion bonded ceramic joints [23,24]. But if the voids eliminated by element diffusion through the interface to form reaction phases, the negative influence of voids on the mechanical properties of the joints

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H.X. Li et al. Ceramics International xxxx (xxxxx) xxxx—xxx

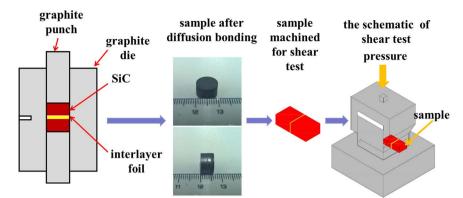
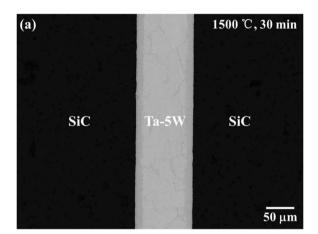
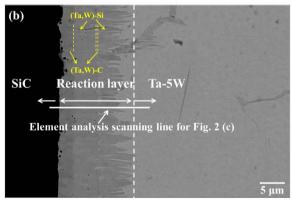


Fig. 1. Schematic of samples preparation for shear strength test.





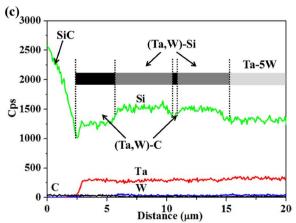


Fig. 2. Back scattered electron (BSE) micrographs of cross-section of SiC/Ta-5W/SiC joint bonded at 1500 °C for 30 min (a) low magnification image, (b) high magnification and (c) the distribution curves of Ta, W, Si and C of line-scanning in (b).

may be insignificant [25]. Although the interfacial voids elimination models have been well investigated for ceramic/metal joints based on HP bonding [13,19], rare information aiming to the interfacial voids evolution and its effects on the joints diffusion bonded by SPS have been reported. Considering that the elimination of interfacial voids depends strongly on the diffusion constant at a given bonding temperature, it is interesting to make a comparison of diffusion constants obtained by HP and SPS.

Residual stresses in ceramics joints are typically thermal in nature and are the result of the difference in coefficient of thermal expansion (CTE) between the interlayer and the being joined bulk ceramics. Thermal residual stresses can be generated while cooling the joints from bonding temperature to room temperature because of the CTE mismatch between the reaction phases formed in interlayer and the bulk ceramics. Although it is not possible to eliminate the residual stresses that arise during processing, it is possible to minimize their negative effect by selecting an interlayer having a CTE close to that of being joined ceramics and by carefully controlling the interfacial reactions. In this work, tantalum (Ta) alloyed with 5 wt.% tungsten (W) (hereinafter referred to as Ta-5W) foil having a CTE (Ta: $6.62 \times 10^{-6} \, \text{K}^{-1}$ [26]) closes to that of SiC (4.02 \times 10⁻⁶ K⁻¹ [5]) were selected as interlayer to join SiC ceramic by SPS. Moreover, the high melting points and low induced radioactivity of Ta and W make the SiC/Ta-5W/SiC joint suitable for fusion applications [27]. Therefore, Ta-5W foil was selected as interlayer materials to join SiC ceramic and the effects of bonding temperature and holding time on the interfacial microstructure and shear strength of the joints were investigated. The mechanism of interfacial voids evolution was also discussed and the comparison was made for the diffusion constants obtained by HP and SPS. The activation energy and diffusion constant were calculated by diffusion kinetics by taking the change of reaction layer thickness into consideration.

2. Experimental

The commercial available pressureless sintered $\alpha\text{-SiC}$ (> 99.9%, Astek ceramic company, China) was machined in dish shaped samples with diameter of 9.8 mm and the height of 3 mm. The surfaces of these specimens were polished up to 1 μm finish and then these specimens were ultrasonically cleaned in acetone for 10 min dried in air before assembly. The cold-rolled Ta-5W foils (> 99.9%, 100 μm) were polished and then cleaned in acetone for 10 min.

The SiC/Ta-5W/SiC sandwich were assembled in a graphite die for SPS joining (Labox-350 spark plasma sintering system, Japan), as shown in Fig. 1. The SPS diffusion bonding processes was conducted with a heating rate of 50 °C/min in Ar atmosphere and pulse-mode direct current (pulse 40 ms, pause 7 ms) was applied for heating. In order to reduce the residual stress caused by CTE mismatch in the joints, a low cooling rate was set when the holding time was finished.

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