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Effect of different filler materials on the microstructure and mechanical properties of SiC–SiC joints joined by spark plasma sintering

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ABSTRACT

The SiC ceramic was joined via spark plasma sintering (SPS) using 4.5 wt% yttria partially stabilized zirconia (4.5YSZ) powder and 4.5YSZ+20 wt%Al_2O_3 powder mixture as fillers. The 4.5YSZ powder was synthesized by sol-gel method. The effects of different filler materials on the microstructure and mechanical properties of the SiC joints were investigated. Based on the X-ray diffraction and scanning electron microscope analyses, as well as three-point bend tests, the SiC joint with 4.5YSZ+20 wt%Al_2O_3 powder mixture as the filler material possesses higher flexural strength of 107.3 MPa at room temperature. The superior flexural strength of the SiC joint using the 4.5YSZ+20 wt%Al_2O_3 filler is attributed both to the carbothermic reaction and element diffusion at the SiC/4.5YSZ+20 wt%Al_2O_3 interface during the joining process.

1. Introduction

SiC ceramic is one of the most promising structural materials due to its attractive properties, such as high hardness, good oxidation resistance, low creep rate, superior corrosion resistance, high thermal conductivity and low thermal expansion coefficient (TEC: 4–6 × 10^{-6} K^{-1}) both at room temperature and at high temperatures [1,2]. Thus, SiC ceramic is highly suitable for high-temperature applications in the fields of aerospace, electronics, nuclear and transportation industries [3,4]. However, in most cases, SiC applications strongly depend on its joining, because the brittle nature and high rigidity of SiC makes it very difficult and expensive to manufacture with a large size and complicated shape [5–7]. Therefore, the development of joining materials and joining technologies for the fabrication of the SiC joints has been investigated intensively in recent years.

Currently, active metals/alloys, organic materials and inorganic materials are used as fillers for the SiC joining [8–13]. However, the mismatch in thermal expansion coefficients (TECs) between SiC ceramic and metal/alloys results in the generation of higher residual stress in the joints during the joining process. The SiC joints using metal/alloys as fillers exhibit inferior corrosion and oxidation resistance, thus can not be used above 700 K [14]. Additionally, the organic materials used as fillers for SiC joining can produce porosities and cracks due to their pyrolysis during the joining process, resulting in a reduction of flexural strength of the SiC joint. Therefore, the development of inorganic materials as fillers with high melting points and good oxidation resistance is necessary. Yttria partially stabilized zirconia (YSZ) (TEC: 6–10 × 10^{-6} K^{-1}) is considered as a candidate filler material for SiC joining, that possesses excellent strength (2.0 GPa) and fracture toughness (>20 MPa $m^{1/2}$) attributing to the stress-induced martensitic transformation of YSZ from tetragonal to monoclinic [15–17].

Except for the investigation of the candidate filler materials, the development of joining technologies for SiC joining has been investigated intensively. To date, SiC joining is commonly realized by brazing and diffusion bonding in last decades [18,19]. In addition, spark plasma sintering (SPS) as a new sintering method has drawn a great attention with its advantage of fast sintering, resulting in dense and fine-grained microstructure. Several studies on fabrication of SiC materials by SPS have been published in the last decade [20–26]. However, there is still a dearth of available

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literature on the SiC joining by SPS.

In this study, SPS was employed to join SiC using 4.5YSZ and 4.5YSZ+20 wt%Al_2O_3 powders as fillers, and the influence of different filler materials on the microstructure and mechanical properties of the SiC joints was evaluated.

2. Experimental procedures

In this work, 4.5YSZ (4.5 wt% Y_2O_3 partially stabilized ZrO_2) power and 4.5YSZ+20 wt%Al_2O_3 powder mixture were used as the fillers for SiC joining. 4.5YSZ powder was synthesized by sol-gel process using zirconium nitrate [$Zr(NO_3)_4 \cdot 5H_2O$] and yttrium nitrate [$Y(NO_3)_3 \cdot 6H_2O$] as the starting materials. 4.5YSZ+20 wt% Al_2O_3 powder mixture was prepared by ball-milling the synthesized 4.5YSZ and nano α-Al_2O_3 (~30 nm, Beijing DK nano technology Co. Ltd., Beijing, China) for 2 h. The commercial pressureless-sintered SiC ceramic rods (Φ 18 mm $\times$ 25 mm, Astek ceramic Co. Ltd., Shandong, China) with a bend strength of 220 MPa was used as the starting material to be joined, the surfaces of SiC rods were polished with diamond paste and ultrasonically cleaned in ethanol for 20 min.

A SiC rod, the fller and another SiC rod were loaded into a graphite die in a sandwich configuration as shown in Fig. 1a, and were joined in an SPS system (SPS-20T-10, Chen Hua Electric Furnace Co. Ltd., Shanghai, China) under vacuum (<6Pa). The heating rate was set to 150 °C/min, and the pressure was set to 40 MPa.

The phase analyses of the synthesized 4.5YSZ powder and the fracture surfaces of the joints were conducted by X-ray diffraction (XRD, Model Bruker advance-D8, Bruker, Germany) at a wavelength of 1.5406 Å. The phase transition temperatures of the synthesized 4.5YSZ powder were characterized through a thermogravimetric and differential scanning calorimetry (TG-DSC, Model STA 449PC, Netzsch, Germany) in air. The bend strengths of the joints (the flexural strength) at room temperature were measured by the three-point bend test with the test span of 30 mm and the displacement rate of 0.5 mm/min, using a universal mechanical testing machine (Model WDW-200, New Test Instrument Co. Ltd., Changchun, China). The schematic map for the bend strength test of the joints is shown in Fig. 1b. The microstructural observation of the joining zone was performed with a scanning electron microscope (SEM, Model JXA 840, JEOL, Japan). The composition of this zone was determined using an energy dispersive X-ray spectrometer (EDX, Model Inca, Oxford Instruments, United Kingdom).

3. Results and discussion

3.1. Synthesis of the 4.5YSZ powder

The TG-DSC curves of the 4.5YSZ gel powders measured at a heating rate of 10 °C/min in air are shown in Fig. 2. During the heating process, the first peak at 114.7 °C in the DSC curve is a broad endothermal peak accompanied with a weight loss of 6%, which is attributed to the dehydration of the 4.5YSZ gel powder. The second peak at 460.3 °C is a relatively sharp exothermal peak, which corresponds to the crystallization of 4.5YSZ gel powder from amorphous to tetragonal phase. Simultaneously, the weight loss of the 4.5YSZ gel powder in a temperature range of 200–500 °C in the TG curve is resulted from the oxidation of residual organic compounds, as well as the elimination of the chemisorbed water. The following small exothermal peak at 674.2 °C is assigned to the elimination of the residual carbon in the 4.5YSZ gel powder or/and the development of small amount monoclinic phase. A very broad endothermal valley observed from 800 to 1280 °C might be attributed to the growth of the crystallized 4.5YSZ grains as the temperature increases.

During the cooling process, there is neither endothermal peak nor exothermal peak in the DSC curve, indicating no phase transformation occurred. The phase transformation of ZrO_2 from tetragonal to monoclinic normally occurs at about 1000 °C [27]. The addition of 4.5 wt%Y_2O_3 into ZrO_2 makes 4.5YSZ to be tetragonal

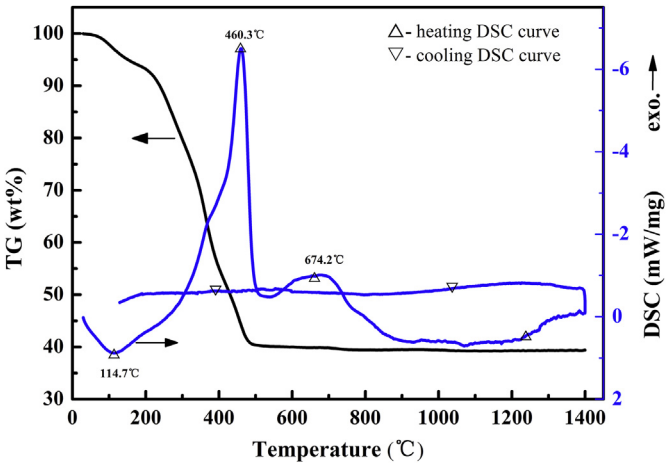

Fig. 2. TG-DSC curves of the 4.5YSZ gel powders.

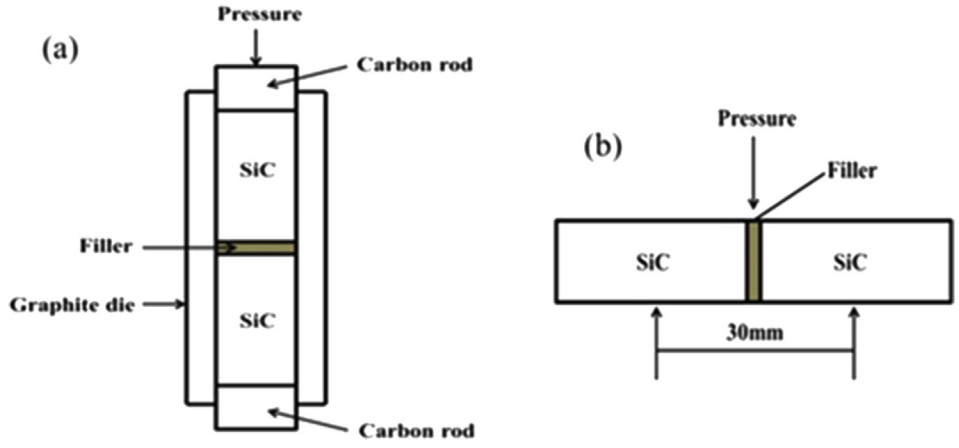

Fig. 1. Schematic setup for the joining process (a) and the bend strength tests of the joints (b).