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Direct solid-state diffusion bonding of zirconium carbide using a spark plasma sintering system



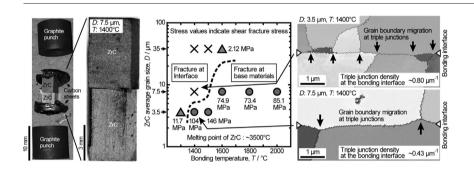
Kazuyuki Kohama*, Kazuhiro Ito

Joining and Welding Research Institute, Osaka University, 11-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan

HIGHLIGHTS

- ZrC-sintered materials with different grain sizes were diffusion-bonded using a spark plasma sintering system.
- The bonding temperature necessary to obtain high-strength ZrC-ZrC joints was reduced by reducing the ZrC grain size
- A smaller ZrC grain size enhanced grain boundary diffusion, which controlled the bonding process.
- The creation of wedges at bonding interface induced by grain boundary diffusion is responsible for increasing joint strength.

GRAPHICAL ABSTRACT



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ABSTRACT

The authors conducted direct solid-state diffusion bonding of zirconium carbide (ZrC)-sintered materials with different average grain sizes of 3.5, 7.5 and 35 μ m using a spark plasma sintering system. ZrC samples were bonded at 1300–2000 °C for 20 min with no defects or oxidation. Shear tests on the ZrC–ZrC joints at room temperature revealed that the bonding temperature to obtain joints with a strength at the bonding interface that is higher than the fracture strength of the base materials could be reduced by reducing the ZrC average grain size. Microstructure studies around the bonding interface showed that the bonding process was controlled by grain-boundary diffusion, and the dominant driving force was a lowering boundary energy at the intersection of the grain boundary and the bonding interface (i.e., triple junctions at the bonding interface). Grain boundary migration across the bonding interface at the triple junctions was suggested to increase joint strength. The higher density of the triple junctions derived from the reduced ZrC grain size is thought to enhance grain boundary migration and increase the driving force and diffusion paths for grain boundary diffusion, which results in the formation of joints with a higher bonding interface strength.

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1. Introduction

Zirconium carbide (ZrC) is a promising ceramic material for hightemperature applications because of its excellent physical properties that originate from its contained covalent and metallic bonds [1–6]. The strong covalent Zr–C bond provides an ultra-high melting point $(T_{\rm m})$ of ~3500 °C, a high hardness and a good thermochemical stability [7–9], and the metallic bond component provides a high electrical/thermal conductivity and good thermal-shock resistance.

As is the case with many ceramics, the use of ZrC in practical applications requires reliable joining methods that allow smaller and more simply produced ZrC components to be integrated into more complex structures. In general, conventional diffusion bonding and brazing with metal insert layers to reduce bonding temperature are used to join ceramics [10–16], but the joint high-temperature reliability is

^{*} Corresponding author.

E-mail address: kohama@jwri.osaka-u.ac.jp (K. Kohama).

decreased because of the presence of metal layers with a low $T_{\rm m}$ and a poor thermochemical stability compared with ceramics. Although transient liquid phase (TLP) bonding using multi-layered insert layers has been studied as a possible method to produce refractory joints [13,15, 17–23], its practical utilization is limited because it is costly. For example, ZrC-sintered materials were TLP-bonded at 1400 °C using Ni/Nb/Ni insert layers [15], in which the thinner Ni layers were incorporated with a part of the thicker Nb layer to form a eutectic liquid phase at the bonding temperature; these were then transformed into a refractory layer with a re-melt temperature of several hundred degrees above the bonding temperature by isothermal solidification. However, the process needed to be conducted under a high vacuum of $\sim 10^{-6} \, {\rm Pa}$ to prevent oxidation, and required in excess of 10 h for the isothermal solidification.

Direct diffusion bonding is considered to be a suitable joining method for ZrC as a low-cost process with high-temperature joint reliability. However, no reports exist on the formation of direct ZrC–ZrC joints, whereas other ceramics with lower $T_{\rm m}$ such as SiC and TiN have been joined directly [24–25]. A critical issue is that self-diffusion needs to be conducted at an extremely high bonding temperature of no less than $0.5T_{\rm m}$ (i.e., the recrystallization temperature), which corresponds to ~1600 °C for ZrC.

In this study, high-temperature processing was achieved using a spark plasma sintering (SPS) system [24–29]. In the SPS process, samples and surrounding graphite fixtures are Joule-heated by applying a pulsed direct current. The process can be conducted at an extremely high temperature with a rapid heating rate of more than 100 °C min⁻¹ in reducing atmosphere in low vacuum of several Pascals. From the viewpoint of materials science, the bonding temperature is expected to be reduced by reducing the grain size of polycrystalline ZrC because of enhanced grain boundary diffusion of Zr and/or C atoms. Thus, ZrC-sintered materials with different grain size were used for bonding, and the dependence of bonding strength of the ZrC–ZrC joints on bonding temperature and ZrC grain size was investigated.

2. Experimental procedure

Polycrystalline ZrC-sintered materials with an approximately stoichiometric composition were produced by hot-pressing at about 2000 °C [30]. There were unintentional variations in ZrC average grain size among the sintered materials, and three types of materials with average grain size of 3.5, 7.5 and 35 µm were selected to be used for ZrC-ZrC bonding in this study. Small rectangular samples $(4 \text{ mm} \times 4 \text{ mm} \times 6 \text{ mm})$ were prepared using a low-speed wheel saw. To prepare the bonding surface and to adjust the sample length to between 5.95 and 6.00 mm, the bottom surface (4 mm \times 4 mm) was polished sequentially using 30-, 15-, 6-, 1- and 0.5-µm diamond lapping films. Solid-state diffusion bonding of the ZrC samples was conducted using an SPS system (Sumitomo Coal Mining Co., Ltd.). Fig. 1 shows a schematic illustration of the ZrC sample positions in the SPS chamber. Two ZrC samples in contact were held between upper and lower graphite punches in a cylindrical graphite die. A 1-mm-thick carbon sheet was placed between the sample and punch for impact absorption and to prevent reaction. The top surface of the upper punch was flush with that of the die, and a compressive stress was not applied initially to the ZrC samples but increased gradually with increasing temperature because of the thermal expansion difference between graphite and ZrC [31]. Prior to heating, the vacuum pressure in the SPS chamber was ~5 Pa. Samples were heated by applying a pulsed direct current to the graphite fixtures and samples. The bonding temperature was measured on the outer surface of the die using a radiation thermometer; this temperature varied from 1300 °C to 2000 °C. In preliminary experiments, die and sample temperatures were confirmed to be equal above 1000 °C. The heating time was ~4 min from room temperature to 700 °C, and 30 min from 700 °C to each bonding temperature. The holding time at the bonding temperature was 20 min. After bonding, samples were

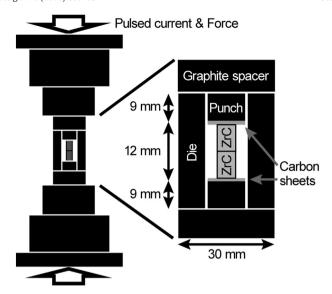


Fig. 1. Schematic illustration of ZrC sample position in SPS chamber.

cooled to less than ~150 °C in vacuum. The bonding strength of the ZrC–ZrC joints was evaluated by shear tests at room temperature using a Shimadzu Autograph (AG-10T). The crosshead speed was set to 0.1 mm min $^{-1}$. The microstructure around the bonding interface was studied by scanning electron microscopy (SEM) equipped with an electron backscattering diffraction (EBSD) analysis system.

3. Results and discussion

3.1. Appearance check and plastic deformation evaluation of ZrC-ZrC joints

ZrC samples were bonded at 1300–2000 °C for 20 min. Fig. 2 shows the ZrC–ZrC joint bonded at 1400 °C with an average grain size of 7.5 μ m. All other joints fabricated in this study were similar in appearance, and no defects such as voids or cracks at the interface were visible. X-ray diffraction spectra obtained for all joints indicated that they were not oxidized during bonding (spectra not shown). It is thought that sample oxidation was suppressed because of the reducing atmosphere generated by the heated graphite fixtures.

To clarify the effects of bonding temperature on joint deformation, the compressive plastic strain of the joints after bonding was evaluated as summarized in Fig. 3. The strain increased with increasing bonding temperature, which indicates that the ZrC samples were compressed by the graphite punches during bonding because of thermal expansion differences between graphite and ZrC. The strain value was as high as 4.6% for the joint bonded at 2000 °C, and less than 0.5% for those bonded at 1800 °C and lower. A lowering of the bonding temperature was essential for precision bonding of ZrC using the SPS system.

3.2. Dependence of ZrC–ZrC joint strength on bonding temperature and average ZrC grain size

Fig. 4(a) shows representative stress–displacement curves that were obtained by shear tests at room temperature for the ZrC–ZrC joints. The vertical and horizontal axes represent shear stress applied to the joints and displacement of the crosshead, respectively. For the joint bonded at 1400 °C with an average grain size of 3.5 μ m (T=1400 °C, $D=3.5 \mu$ m), the shear stress increased gradually as the displacement increased, and it reached a relatively high maximum value (σ_{max}) of 104 MPa as indicated by the solid black line arrow in Fig. 4(a). Fig. 4(b) is the corresponding photograph of the joint after the shear test. Fracture occurred in the base materials, which indicates that the shear strength of the bonding interface was larger than the shear fracture

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