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Short communication

Electroplating assisted diffusion bonding of ZrC–SiC composite for full ceramic joints

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Abstract

Electroplated Ni interlayer was used in the diffusion bonding of ZrC–SiC composite. The interfacial reaction focused on Ni-SiC reaction system. Corresponding reaction products were identified to be Ni_2Si and graphite. An optimal Ni interlayer of 4 μm, which was precisely controlled by adjusting the plating time, was designed to ensure a reliable bonding for full ceramic joints. Shear strength of the electroplating assisted joints reaches 183 MPa and 45 MPa at room temperature and 800 °C, respectively, which are 9% and 67% higher than that of the traditional joints bonded with thicker Ni foil.

Keywords: Ceramic; Electroplating; Diffusion bonding; Microstructure

1. Introduction

ZrC–SiC composites (ZS hereafter) are important family members of ultrahigh-temperature ceramics (UHTCs). ZS composites have attracted increasing attention in recent years for their excellent properties [1–3]. To extend the practical application, it is essential to join ZS composites to themselves or to metals for complex components. However, the successful joining of ZS composite depends on the reliable interfacial bonding in a relatively short time and no degradation in operating temperature after the joining [4].

Diffusion bonding is a promising approach for high temperature joints [5,6]. However, direct diffusion bonding of ceramics seems impossible because the atomic diffusion is time-intensive for such materials [7]. Therefore, metal interlayers are usually used in ceramic joints to strengthen the interfacial reactions. Recently, a novel Ni/Nb/Ni multilayer-interlayer-based transient liquid phase diffusion bonding (TLP) was reported in the joining of alumina and carbides [8–10]. The TLP method enables rapid, reliable and reduced temperature bonding of ceramics while retaining the potential of high temperature application. Nevertheless, residual metal interlayers remain in the joints. Typically, since metals soften well below their melting temperatures and are easily oxidized, residual metal interlayers in the joints increase the likelihood of property degradation [11]. Therefore, the ideal ceramic joints are full ceramic joints.

In our earlier work [12], the ZrC–20 vol% SiC composite was diffusion bonded with Ni foil as the interlayer due to its relatively high melting point. But the residual Ni layer in the joint is unfavorable for its high temperature performance. Though the residual Ni layer can be removed at higher joining temperature or during longer holding time, the formed graphite will transform into large pieces of brittle flakes under such conditions.

An electroplating assisted diffusion bonding method was developed to improve the joining of ZS composite. The electroplated Ni interlayer, used to replace the traditional thick Ni foil, is aimed to be consumed during a shorter joining time. The reduction in joining time prevents the brittle flake graphite at the same time, which helps to get full ceramic joints with good mechanical properties.

2. Materials and methods

The ZrC–20 vol% SiC composite was sliced into 5 mm × 5 mm × 3 mm pieces for metallographic observation and 10 mm × 8 mm × 3 mm pieces for shear test, respectively. The ZS pieces were polished by diamond grinding discs up to 1200 grit and then ultrasonically cleaned in acetone for

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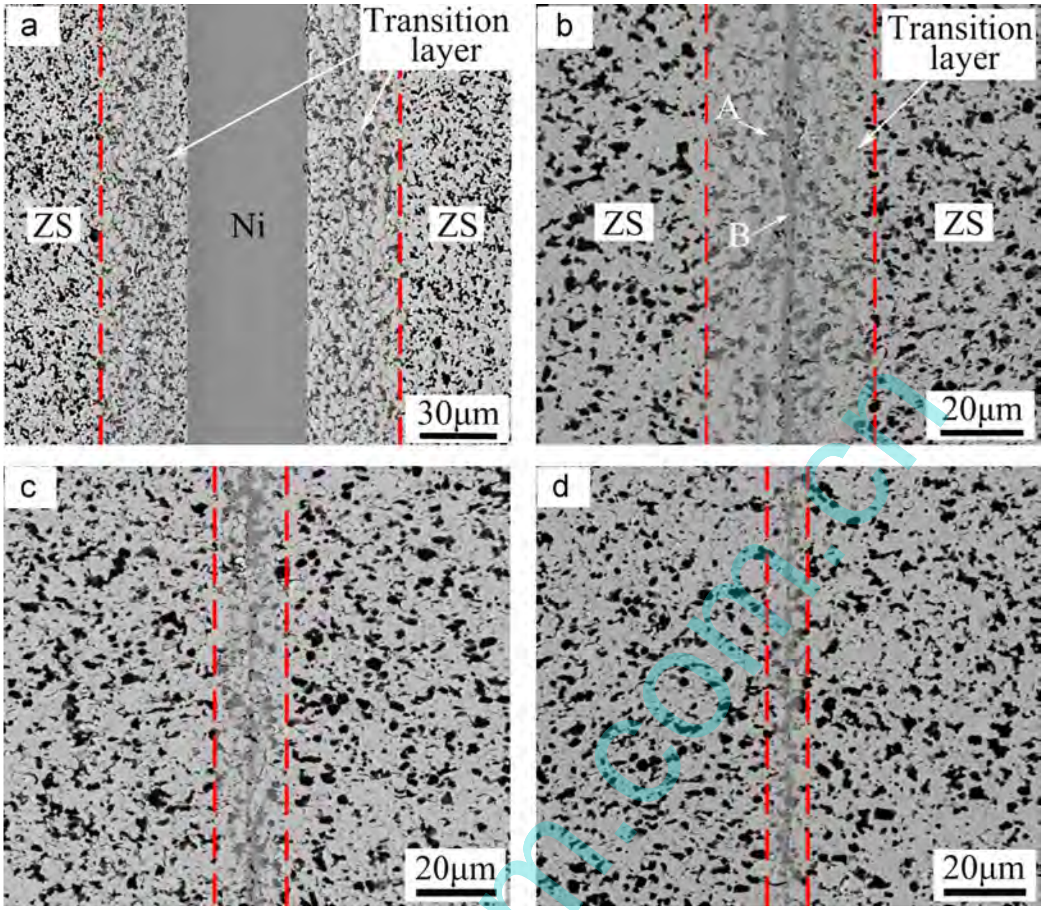

Fig. 1. Microstructures of the ZS joints bonded with different Ni layers: (a) 50 μm Ni foil, (b) 5 μm electroplated Ni layer, (c) 2 μm electroplated Ni layer and (d) 1 μm electroplated Ni layer.

10 min. Electroplating was performed in nickel sulfamic acid solution with a PH value of 4.5. A piece of pure nickel was used as the anode. Electroplating temperature and current density were controlled to be 45 °C and 0.01 A/mm^2, respectively. The thickness of the plated Ni interlayers ranges from 1 μm to 5 μm.

Diffusion bonding of the electroplated ZS composite pieces was conducted in a vacuum furnace with a vacuum of 1×10^{-3} Pa. The pressure applied on the samples was 10 MPa. The samples were heated to the joining temperature with a heating rate of 30 °C min^{-1}, isothermally held for a series of minutes, and then cooled to room temperature at a rate of 5 °C min^{-1}.

The roughness of the polished ZS composite surfaces was checked with AFM (CSPM5500, Being Nano-instruments, Ltd). The bonded joints were characterized by SEM (Quanta 200FEG, FEI Co. Ltd) equipped with energy dispersive X-ray spectroscopy (EDS, AMETEK). The TEM observation was performed with Tecnai G2 F30 (FEI Co. Ltd). Shear strength of the joints was tested using the Instron-1186 universal testing machine.

3. Results and discussion

ZS composite was first diffusion bonded with a Ni foil as the interlayer. The thickness of the used Ni foil is 50 μm. Corresponding joints are referred to as traditional joints. Fig. 1(a) shows the cross-section microstructure of the traditional joint bonded at 1000 °C for 30 min [12]. Meanwhile, Fig. 1(b)–(d) show the microstructure of the joints bonded with electroplated Ni interlayers. All the electroplating assisted ZS joints were bonded at 1100 °C but for different holding time. The holding time increased successively from 10 min to 30 min when the thickness of the plated Ni interlayer rose from 1 μm to 5 μm.

Table 1
EDS results of different points in the transition layer (at%).

Points	Zr	Si	C	Ni	Possible phases
A	2	14	58	26	Ni-Silicide + C
B	3	34	–	63	Ni-Silicide

The formed transition layers in the joints confirm the interfacial reaction between Ni and ZS composite. As shown in Fig. 1(a), two transition layers distribute at each side of the residual Ni layer. The thickness of each transition layer is 30–40 μm. For the electroplating assisted ZS joints, a gray layer located at the interface, which was labeled as C in Fig. 1 (b). According to the EDS results (Table 1), the gray layer belongs to a kind of Ni–Si compound. However, the volume of

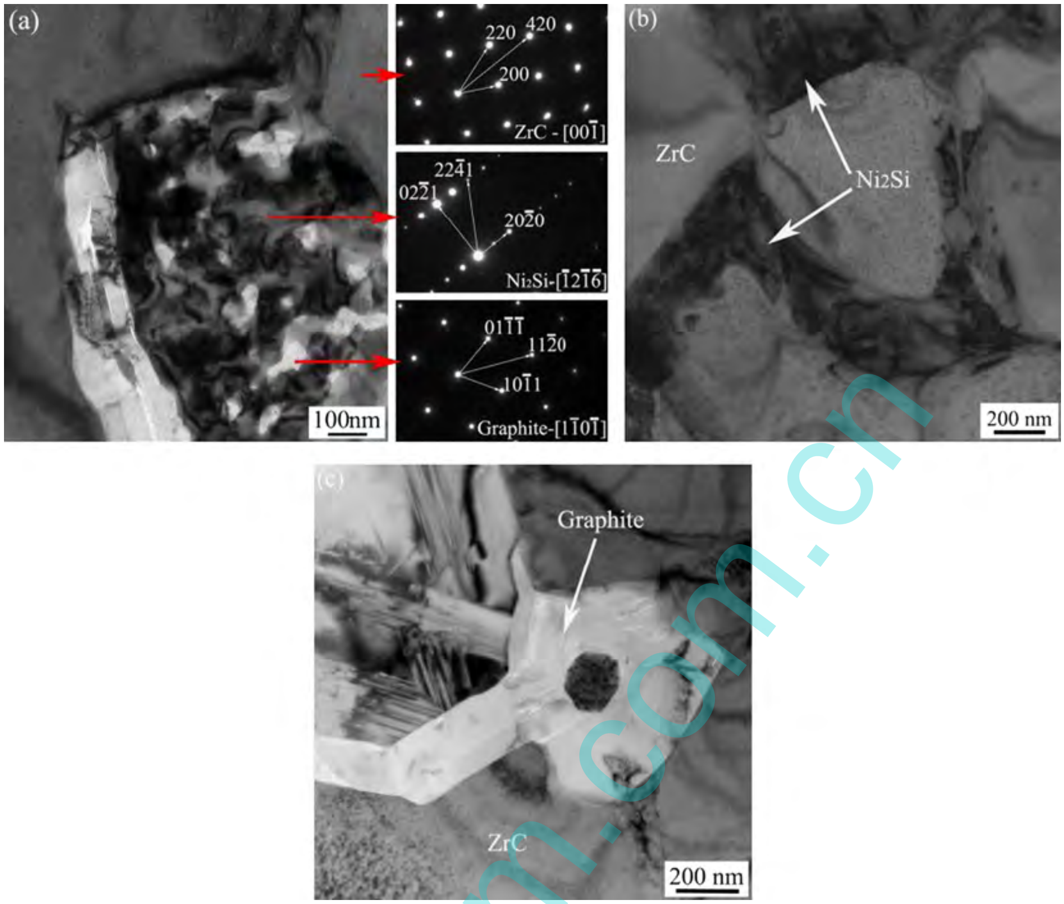

Fig. 2. Bright field image of the transition layer and corresponding SAED patterns of different joints: (a) 1100 °C/30 min, (b) and (c) 1200 °C/60 min.

the Ni–Si compound decreased when thinner Ni interlayer was used, seen in Fig. 1(c)–(d). In other words, the electroplated Ni interlayer has been consumed during the interfacial reaction and transformed into Ni-Silicide. No residual Ni interlayers were found in the electroplating assisted joints, which is the critical difference from the traditional joints.

The microstructure and properties of the transition layer are different from those of the ZS composite. Especially, the formed graphite prefers to transform into brittle flake graphite. The longer joining time or higher temperature, the larger pieces of flake graphite. Therefore, a thinner Ni interlayer is more favorable since it not only reduces the reaction products, but also cuts down the joining time. According to the experimental results, the plated Ni interlayer with a thickness of 1 μm is sufficient for sound ZS joints without voids. And the transition layer was successfully controlled to be less than 10 μm, as shown in Fig. 1(d).

Fig. 2 shows the bright field image of the formed transition layer. The interfacial reactions took place mainly in Ni-SiC reaction system. The Ni-SiC reactions offen give Ni silicides [13,14]. Among all the possible Ni silicides, Ni_2Si is the most stable product due to its highly negative ΔG value (-46.5 KJ mol^{-1} at 1100 °C). According to the selected area electron diffraction (SAED) patterns, the interfacial reaction products were confirmed to be Ni_2Si and graphite, which is in agreement with the theoretical analysis. No Zr–Ni compounds or other products were detected in the joints.

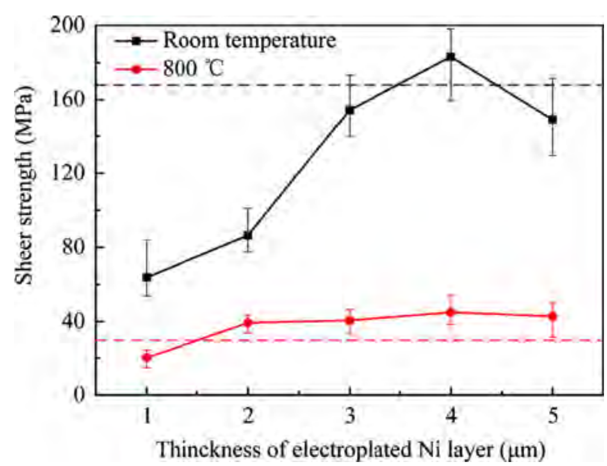

Fig. 3. Shear strength of the diffusion bonded ZS joints.

At higher joining temperatuer or during longer holding time, Ni_2Si and graphite separated from each other. A portion of Ni_2Si spred along the ZrC boundaries (Fig. 2(b)), while graphite was leaved behind and transiformed into lamellas, seen in Fig. 2(c). In the pure SiC joints diffusion bonded with Ni foil, interfacial reaction often leads to the periodic bands of graphite and Ni silides [15]. The formed graphite layers increase the brittleness of the joints. Nevertheness, the volume of ZrC accountes for 80% in the ZS composite used in this study. the ZrC particles serve as the framework and the reaction products distributed dispersively, which is benificial to the joints.

Mechanical properties of the diffusion bonded joints were evaluated by shear test at both room temperature and 800 °C. The average strength was evaluated from five test values, as shown in Fig. 3. For the electroplating assisted joints, the room temperature shear strength increases to the maximum and then decreases when the Ni interlayers increase from 1 μm to 5 μm. The maximal value reaches 183 MPa when the Ni interlayer is 4 μm. The high temperature shear strength (800 °C) reaches the maximum of 45 MPa at the same point. Shear strength of the traditional joints was also tested for comparison, shown as the two dash lines. The maximal values are 168 MPa at room temperature and 27 MPa at 800 °C, respectively.

The maximal shear strength of the electroplating assisted joints is 9% higher than that of the traditional joints at room temperature and 67% higher at 800 °C. Since the electroplating assisted joints avoid the CTE mismatch between ZS composite and the residual metal interlayer, and large pieces of brittle flake graphite are also prevented in a relatively short joining time, these joints possess better mechanical properties.

4. Conclusions

In conclusion, electroplating assisted diffusion bonding was used in the joining of ZS composite. Full ceramic joints were obtained by adjusting the thickness of the plated Ni interlayer and corresponding joining process. The interfacial reaction products were identified as Ni_2Si and graphite. The maximal shear strength of the electroplating assisted joint reaches 183 MPa at room temperature and 45 MPa at 800 °C, respectively, which are 9% and 67% higher than that of the traditional joints.

Acknowledgements

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