JOINING OF SILICON CARBIDE-BASED CERAMICS BY REACTION FORMING METHOD*

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Introduction

Recently, there has been a surge of interest in the development and testing of silicon-based ceramics and composite components for a number of aerospace and ground based systems. The designs often require fabrication of complex shaped parts which can be quite expensive. One attractive way of achieving this goal is to build up complex shapes by joining together geometrically simple shapes. However, the joints should have good mechanical strength and environmental stability comparable to the bulk materials. These joints should also be able to maintain their structural integrity at high temperatures. In addition, the joining technique should be practical, reliable, and affordable. Thus, joining has been recognized as one of the enabling technologies for the successful utilization of silicon carbide based ceramic components in high temperature applications.

Overviews of various joining techniques, i.e., mechanical fastening, adhesive bonding, welding, brazing, and soldering have been provided in recent publications [1-3]. The majority of the techniques used today are based on the joining of monolithic ceramics with metals either by diffusion bonding, metal brazing, brazing with oxides and oxynitrides, or diffusion welding [4-6]. These techniques need either very high temperatures for processing or hot pressing (high pressures). The joints produced by these techniques have different thermal expansion coefficients than the ceramic materials, which creates a stress concentration in the joint area. The use temperatures for these joints are around 700 °C.

Ceramic joint interlayers have been developed as a means of obtaining high temperature joints [7-11]. These joint interlayers have been produced via pre-ceramic polymers [8-9], in-situ displacement reactions [10], and reaction bonding [11] techniques. Joints produced by the pre-ceramic polymer approach exhibit a large amounts of porosity and poor mechanical properties. On the other hand, hot pressing or high pressures are needed for in-situ displacement reactions and reaction bonding techniques. Due to the equipment required, these techniques are impractical for joining large or complex shaped components.

The reaction processing technique [12] reported here is unique in terms of producing joints with tailorable microstructures. The formation of joints by this approach is attractive since the

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thermomechanical properties of the joint interlayer can be tailored to be very close to those of the silicon carbide base materials. In this paper, the microstructure and mechanical properties of reaction formed joints in Cerastar RB-SiC material are presented. The high temperature flexural strength of joints has been measured up to 1350 °C in air. Scanning electron microscopy has been used to characterize the fracture surfaces. The flexural strength of joints has been compared to those of bulk Cerastar RB-SiC material.

Experimental Procedures

The Cerastar reaction-bonded silicon carbide (RB-SiC) materials used in this study were provided by Carborundum Co., Gardner, MA. These materials were fabricated by the reaction bonding of coarse and fine silicon carbide grains with silicon using a liquid silicon infiltration process. As processed samples were sectioned, mounted, and polished for metallographic studies. For joining studies, 6 cm x 3 cm size silicon carbide pieces were machined from SiC plates. These pieces were cleaned in acetone and dried. Joining is carried out by initially applying a carbonaceous mixture to the joint areas between the silicon carbide pieces. The specimen is then heated to 100 °C for 15-20 minutes. Next, the joint area is infiltrated with molten silicon at 1425 °C for 15 minutes. Molten silicon reacts with carbon to form silicon carbide, with a controllable amount of residual silicon phase in the joints.

Flexure bars were machined from the joined pieces, with joints in the middle of the flexure bars. Four-point flexural strength testing was carried out with MIL-STD-1942 (MR) configuration B specimens with 20 mm inner and 40 mm outer spans. Flexure tests were carried out at room temperature, 800, 1200, and 1350 °C in air. A number of Cerastar RB-SiC bars were heat treated at 1200 °C for 4 hrs. in air. For the as-machined and heat treated Cerastar RB-SiC materials, at least six to nine specimens were tested at room temperature, and three specimens were tested at each high temperature. Three joint thicknesses were investigated. For each joint thickness, at least three joined specimens were tested at room temperature while two were tested at high temperatures. After testing, fracture surfaces were examined by optical and scanning electron microscopy to identify the failure origins.

Results and Discussion

Microstructure

An optical micrograph of as received Cerastar RB-SiC material is shown in Fig. 6. This micrograph shows the distribution of coarse and fine silicon carbide grains (gray) in a silicon phase (white). There are pools of silicon and some porosity in this material. Microstructures of reaction formed joints are shown in Fig. 7 (a-c). In Fig. 7(a), the joint was very thick (~ 350 μm) and silicon rich. This joint will be referred to as Joint A. Two thinner joints, referred to as Joints B and C are shown in Fig. 7 (b) and (c). These joints contain silicon carbide and silicon phase. The joint thickness and composition have a strong influence on the room and high temperature properties of the joined materials.

Flexural Strength and Fractography

The room and high temperature flexural strengths of the as-machined and heat treated Cerastar

RB-SiC along with the joined materials are shown in Fig. 10. The average room temperature strengths of as-machined and heat treated Cerastar RB-SiC specimens were 157±11 MPa and 202±14 MPa, respectively. Thus, the flexural strength of heat treated bars is higher at room temperature. Healing of machining flaws by silica formation is one possible explanation. The flexural strength of the specimen containing thick joint A is about 44±2 MPa. In flexure bars with thick joints, fracture always occurred at the joints. In addition to being thick, this joint was also rich in silicon. The microscopic examination of fracture surfaces of specimens with thick joints tested at room temperature revealed the failure mode to be typically brittle. Specimens containing thinner joints, B and C, have flexural strengths comparable to those of bulk materials. In the flexure specimens with thinner joints, fracture always occurred in the bulk materials away from the joint. In this case, the fracture origins appear to be inhomogeneities inside the specimen. This observation indicates that the material strength is not limited by the joint strength but by the strength of the bulk materials. In addition, there is no significant loss in strength of materials with thin joints up to 1350 °C.

Conclusions

It has been demonstrated that the reaction forming approach can be used to produce strong joints in reaction bonded silicon carbide materials. Thin (SiC-rich) joints show no significant strength loss at high temperatures and have properties at least similar to the bulk parent material used in this study.

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OBJECTIVES

- To develop an affordable approach for the joining of silicon carbide-based materials.
- To characterize the microstructure and mechanical properties of reaction-formed joints.

Fig. 1

JOINING REQUIREMENTS

- Joint properties comparable to base materials.
  - Use temperature > 1200 °C
  - Good mechanical strength
  - Oxidation and corrosion resistance
  - Low CTE mismatch to minimize the residual stresses
  - Thermal shock resistance
- Leak tight joints.
- Practical, reliable, and affordable technique adaptable to in-field installation, service, and repair.

Fig. 2

Flow Chart for the Joining of Silicon Carbide-Based Ceramics by Reaction Forming Method (Schematic)

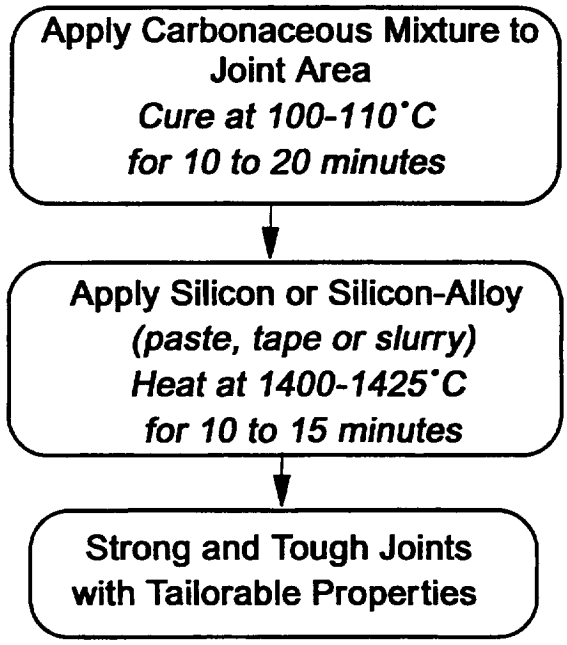


Fig. 3

EXPERIMENTAL PROCEDURES

- Material: Cerastar RB-SiC (Carborundum Co., Gardener, MA).
- Joining: As machined pcs. (6 cm x 3 cm x 0.8 cm) cleaned with acetone and dried.
- Butt joints formed between the pieces and three joint thicknesses evaluated.
- Flexure bars (50 mm x 4 mm x 3mm) were machined with joints in the middle of the bars.
- Monolithic specimens heat treated at 1200 °C for four hours to minimize surface machining flaws.

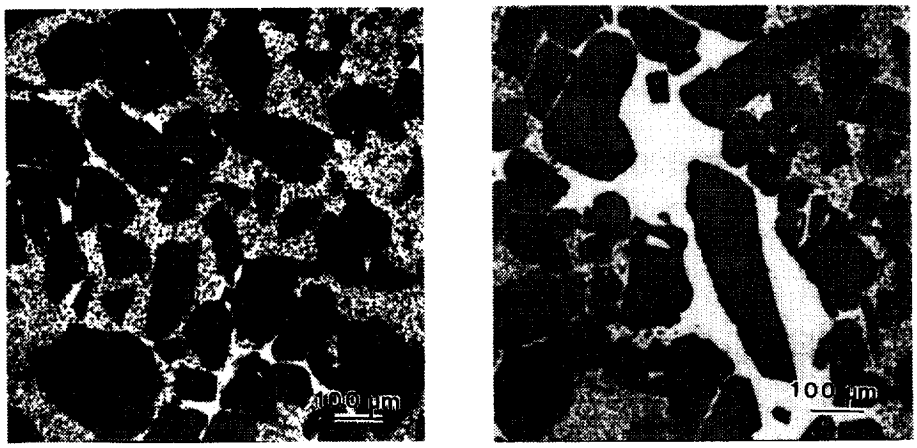
Fig. 4

CHARACTERIZATION

- Microstructure (Optical microscopy)
- Four-point flexure tests (40/20 span)
- Loading rate : 0.5 mm/min
- Test temperatures (25, 800, 1200, 1350 °C), Air
- Fractography (SEM, Optical microscopy)

Fig. 5

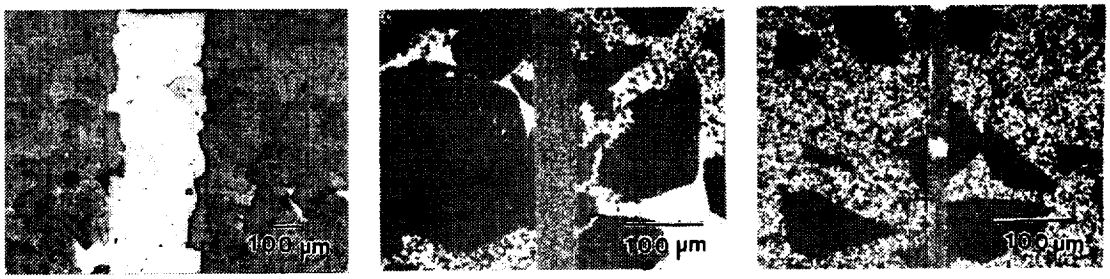
Microstructure of As-Received Cerastar RB-SiC



- Fabricated by the reaction bonding of coarse α-SiC grains with silicon phase.
- Uneven distribution of both phases.

Fig. 6

Microstructures of Reaction Formed Joints in Cerastar RB-SiC



Joint A (~350 µm) Joint B (~50-55 µm) Joint C (~20-25 µm)

Fig. 7

Flexural Strength of Cerastar RB-SiC Ceramics as a Function of Temperature

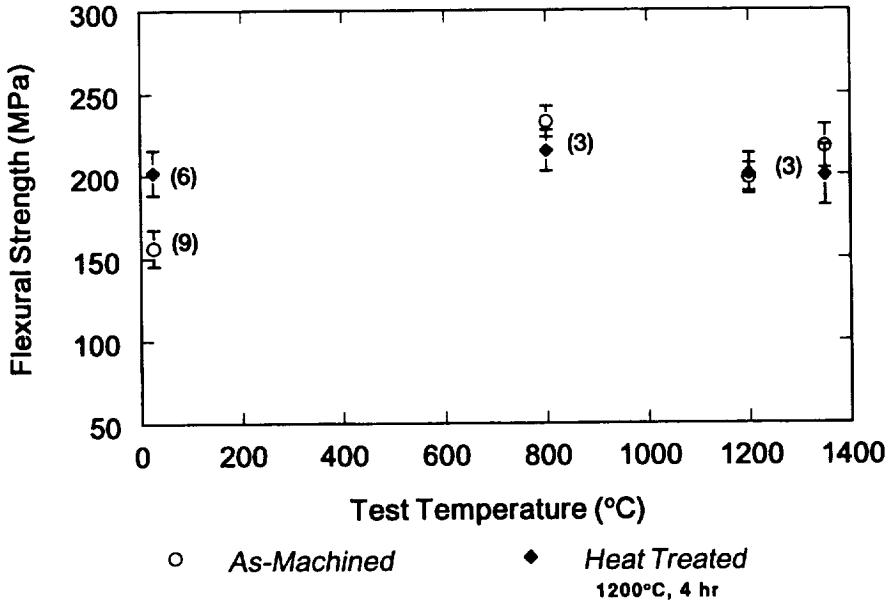


Fig. 8

Flexural Strength of As-Received and Joined Cerastar RB-SiC Ceramics at Room Temperature

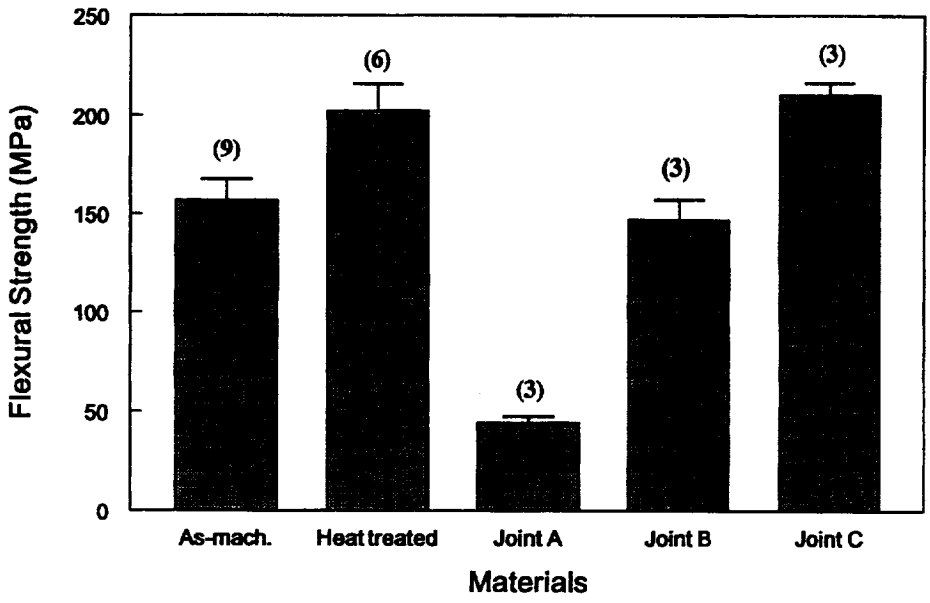


Fig. 9

Flexural Strength of As-Received and Joined Cerastar RB-SiC Ceramics as a Function of Temperature

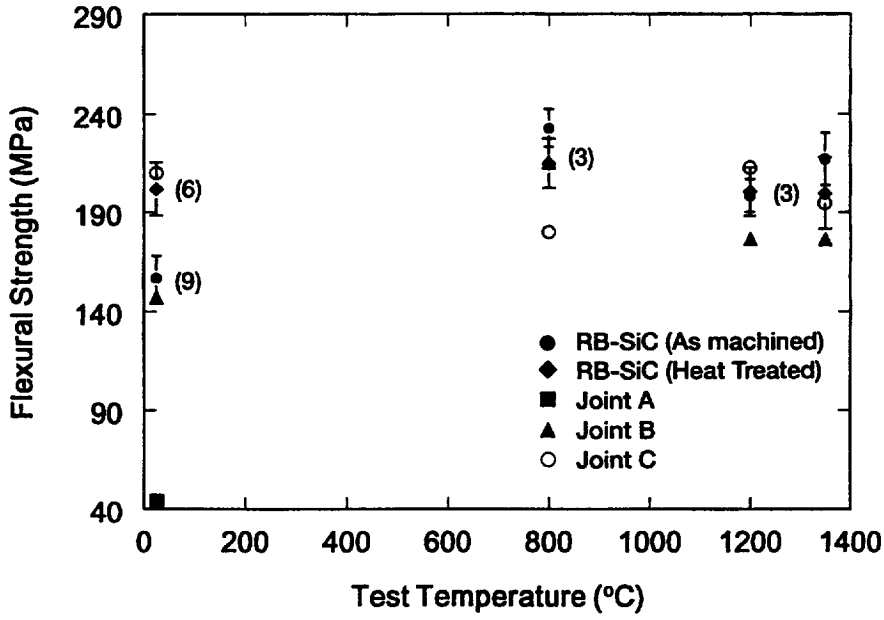
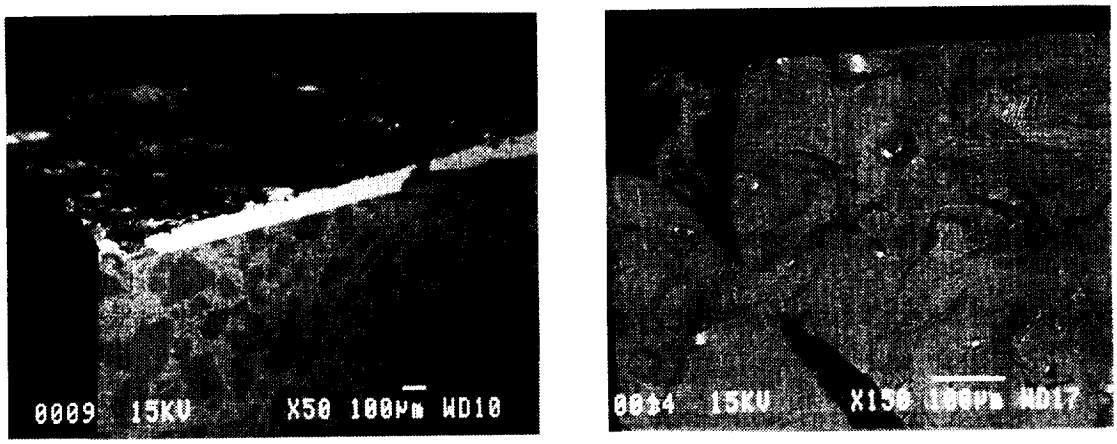


Fig. 10

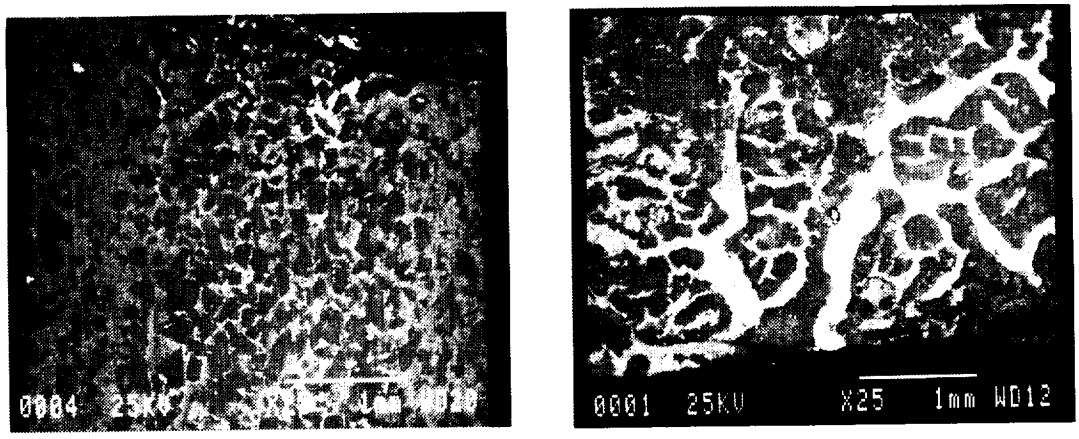
Fractographs Showing the Failure Behavior in Thick (~350 μm) Joints



- Flexure bars always fracture at thick joints because of their poor mechanical strength.

Fig. 11

Fractographs Showing the Failure Behavior in Thin (~ 50 μm) Joints



- Joined flexure bars fail away from the joint regions.

Fig. 12

SUMMARY OF RESULTS

- A reaction based joining approach for silicon carbide materials has been developed.
- The thickness of the reaction formed joints can be tailored.
- Thin joints (< 60 μm) have good room and high temperature properties.
- Failure of flexure bars with thin joints occurs away from the joint regions.

Fig. 13

CONCLUSIONS

- A reaction based joining approach for silicon carbide materials has been developed. Using this approach, joints with tailorable thickness and good room and high temperature properties can be produced.

FOCUS OF FUTURE RESEARCH

- Joining of other types of silicon carbide (Hexoloy-SA, RFSC) and characterization of high temperature thermomechanical properties.
- Joining of fiber reinforced composites (C/SiC and SiC/SiC) and characterization of joint properties.

Fig. 14