JOURNAL OF MATERIALS SCIENCE **33** (1998) 5781 – 5787

Microstructure and mechanical properties of reaction-formed joints in reaction-bonded silicon carbide ceramics

M. SINGH
NYMA, Inc., Lewis Research Center Group, Cleveland, OH 44135
E-mail: msingh@lerc.nasa.gov

A reaction-bonded silicon carbide (RB-SiC) ceramic material (Carborundum's Cerastar RB-SiC) has been joined using a reaction forming approach. Microstructure and mechanical properties of three types of reaction-formed joints (350 μm, 50–55 μm, and 20–25 μm thick) have been evaluated. Thick ($\sim$350 μm) joints consist mainly of silicon with a small amount of silicon carbide. The flexural strength of thick joints is about 44 ± 2 MPa, and fracture always occurs at the joints. The microscopic examination of fracture surfaces of specimens with thick joints tested at room temperature revealed the failure mode to be typically brittle. Thin joints ($<$50–55 μm) consist of silicon carbide and silicon phases. The room and high temperature flexural strengths of thin ($<$50–55 μm) reaction-formed joints have been found to be at least equal to that of the bulk Cerastar RB-SiC materials because the flexure bars fracture away from the joint regions. In this case, the fracture origins appear to be inhomogeneities inside the parent material. This was always found to be the case for thin joints tested at temperatures up to 1350 °C in air. This observation suggests that the strength of Cerastar RB-SiC material containing a thin joint is not limited by the joint strength but by the strength of the bulk (parent) materials. *© 1998 Kluwer Academic Publishers*

1. Introduction

In recent years, there has been a surge of interest in the research, development, and testing of silicon carbide–based monolithic ceramic and composite components for a number of aerospace and ground-based systems. The engineering design often requires fabrication and manufacturing of complex shaped components, which are quite expensive. In many instances, it is much more economical to build up complex shapes by joining together geometrically simple shapes. The joints must have good mechanical strength and environmental stability comparable to the bulk materials, however. These joints must also retain their structural integrity at high temperatures. In addition, the joining technique should be robust, practical, and reliable. Thus, joining has been recognized as one of the enabling technologies for the successful use of silicon carbide–based ceramic components in high temperature applications.

Overviews of various joining techniques, in other words, 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 by diffusion bonding, metal brazing, brazing with oxides and oxynitrides, or diffusion welding [4–6]. These techniques require either high temperatures for processing or hot pressing (high pressures). The joints produced by these techniques have different thermal expansion coefficients than the parent materials have, which contributes to stress concentration in the joint area. Normally, the use temperatures for these joints are around 700 °C.

Ceramic joint interlayers have been developed as a means of obtaining high temperature joints [7–14]. These joint interlayers have been produced via preceramic polymers [8–11], *in situ* displacement reactions [12], and tape casting/reaction bonding [13–14] techniques. Joints produced by the preceramic polymer approach exhibit large amounts of porosity and poor mechanical properties. On the other hand, hot pressing or high temperature fixtures are needed for *in situ* displacement reactions. Because of the equipment required, this technique is not well-suited for joining large components. The tape casting/reaction bonding process has been able to produce joints with good strengths. This joining process has certain limitations, however. The joint thickness has to be controlled by the tape thickness, and these tapes cannot be applied effectively at desired places under certain joint configurations. In addition, joining pieces have to be held together at reaction bonding temperatures, which is not very practical for joining complex shape and large components.

The reaction-forming technique reported here is unique in terms of producing joints with tailorable microstructures. The formation of joints by this approach is attractive because the thermomechanical properties

0022–2461

of the joint interlayer can be tailored to be very close to those of the silicon carbide–base materials. In addition, high temperature fixturing is not needed to hold the parts at the infiltration temperature. A variety of silicon carbide–based ceramics and fiber-reinforced composites have been joined using this approach [15–20].

In this article, 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 with that of bulk Cerastar RB-SiC material.

2. Experimental

The Cerastar RB-SiC 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 × 3 cm silicon carbide pieces were machined from silicon carbide plates. These pieces were cleaned in acetone and dried.

A schematic of the joining method is given in Fig. 1. The joining steps include the application of a carbonaceous mixture in the joint area and curing at 110–120 °C for 10–20 min. Silicon or a silicon alloy in tape, paste, or slurry form is applied in the joint region and heated up to 1250–1425 °C (depending on the type of the infiltrant) for 5–10 min. The molten silicon or silicon alloy reacts with carbon to form silicon carbide with controllable amounts of silicon and other phases as determined by the alloy composition. Joint thickness can be readily controlled in this process.

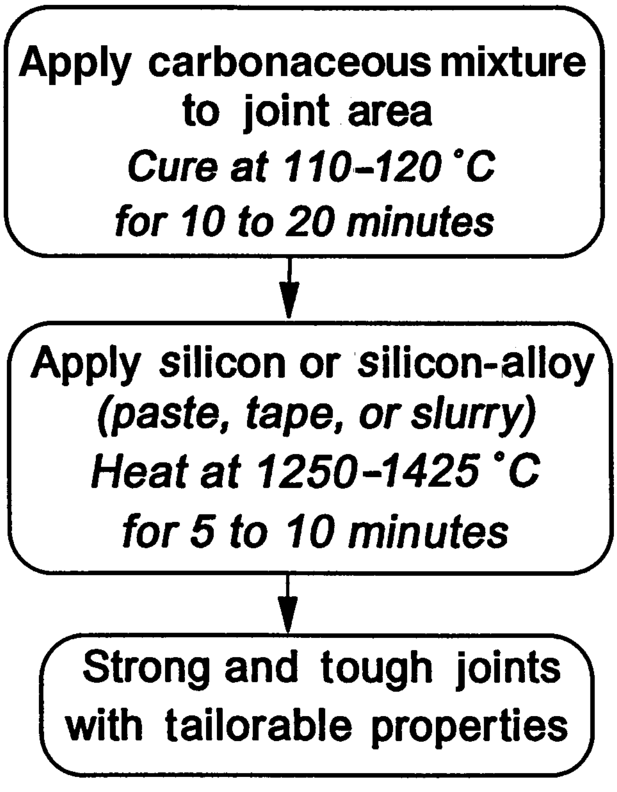


Figure 1 Schematic of the reaction-forming method for the joining of silicon carbide–based ceramics.

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 using MIL-STD-1942 (MR) configuration B specimens with 20-mm inner and 40-mm outer spans. Flexure tests were conducted at room temperature and at 800, 1200, and 1350 °C in air. A number of Cerastar RB-SiC bars were heat treated at 1200 °C for 4 h in air prior to testing. 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. A minimum of three joined specimens were tested at room temperature; two were tested at high temperatures. After testing, fracture surfaces were examined by optical and scanning electron microscopy to identify the failure origins.

3. Results and discussion

3.1. Microstructure

An optical micrograph of as-received Cerastar RB-SiC material is given in Fig. 2a. This micrograph shows a non-uniform distribution of coarse and fine silicon carbide grains (grey) in a silicon phase (white). There are large pools of silicon (Fig. 2b) and some porosity in this material.

Microstructures of reaction-formed joints are shown in Fig. 3. In Fig. 3a, the joint was very thick ($\sim$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. 3b and c. These joints contain silicon carbide and silicon phase. The joint thickness and composition have a strong influence on both the room and the high temperature properties of the joined materials.

3.2. Flexural strength and fractography

The room and high temperature flexural strengths of the as-machined and heat-treated Cerastar RB-SiC materials are shown in Fig. 4. 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 is one possible explanation. The flexural strengths of as-machined and heat-treated materials were similar at high temperatures and were comparable to the room temperature flexural strength of the heat-treated specimens.

The flexural strengths of three types of joined (as-machined) specimens are given in Fig. 5 along with the data for Cerastar RB-SiC specimens. The flexural strength of the thick joint A ($\sim$350 μm) is about 44 ± 2 MPa. In the 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 (Fig. 6a). Also, there is evidence of some porosity present at the fracture surface (Fig. 6b).

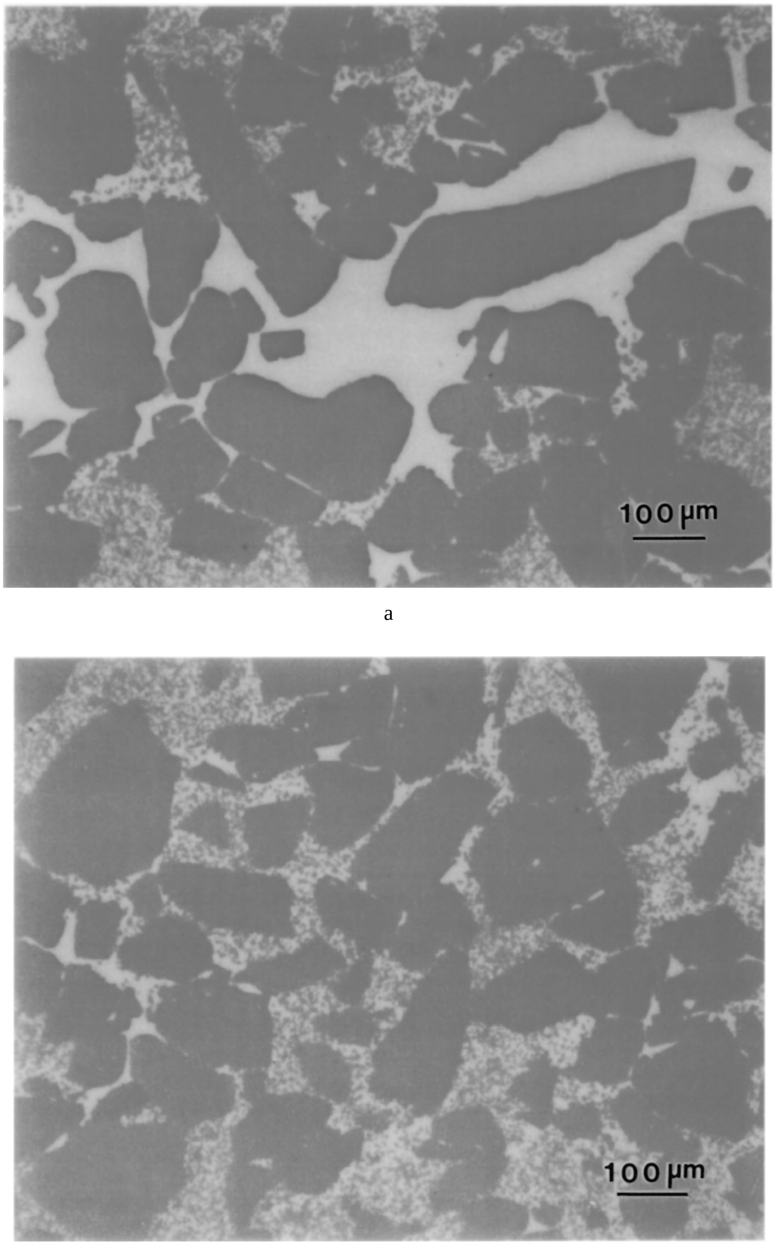


Figure 2 Optical micrographs of Cerastar RB-SiC showing a mixture of coarse and fine grain silicon carbide (grey) and residual silicon phase (white).

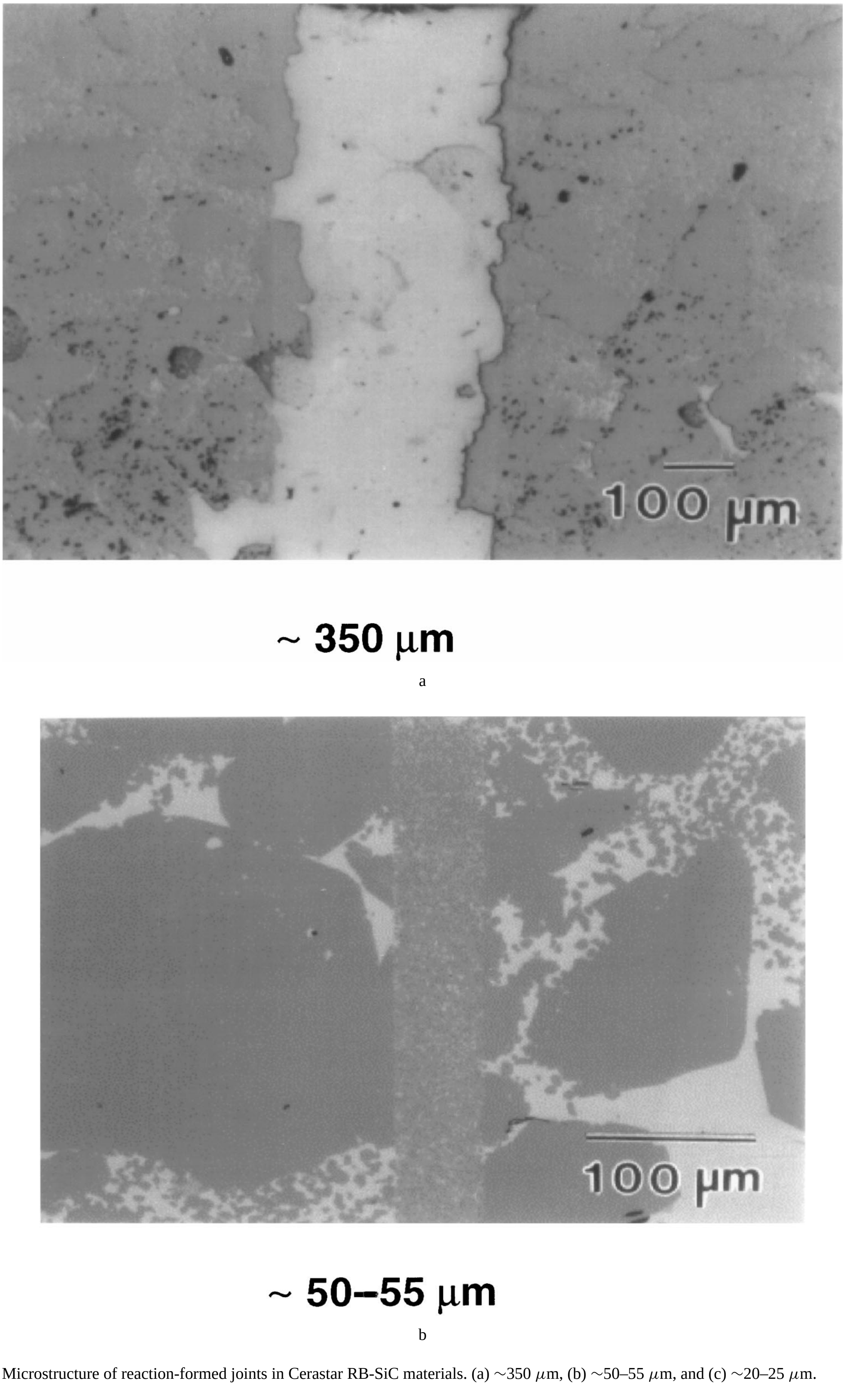
Specimens containing thinner joints, B ($\sim$50–55 μm) and C ($\sim$20–25 μm), have flexural strengths comparable to those of bulk materials. In the flexure specimens with thinner joints, fracture always occurred in the bulk materials away from the joint (Fig. 7a). In this case, the fracture origins appear to be inhomogeneities inside the specimen (Fig. 7b). This observation indicates that with this material, strength is not limited by the joint strength but by the strength of the bulk material. In addition, there is no significant loss in strength of materials with thin joints up to 1350 °C.

4. Summary of results

A reaction-forming method has been developed to join RB-SiC ceramic material. Three types of reaction-formed joints (350 μm, 50–55 μm, and 20–25 μm thick) have been fabricated and evaluated. The thick ($\sim$350 μm) joint consists mainly of silicon and has flexural strength 44 ± 2 MPa. The microscopic examination of fracture surfaces of specimens with thick joints tested at room temperature revealed that the fracture occurred at the joints and the failure mode was typically brittle. The room and high temperature flexural strengths of thin ($<$ 50–55 μm) reaction-formed joints have been found to be at least equal to that of the bulk Cerastar RB-SiC materials from room temperature up to 1350 °C in air. Thin joints ($<$ 50–55 μm) consist of silicon carbide and silicon phases. In the bars with thin joints, the fracture origins appear to be inhomogeneities within the parent material.

5. Conclusions

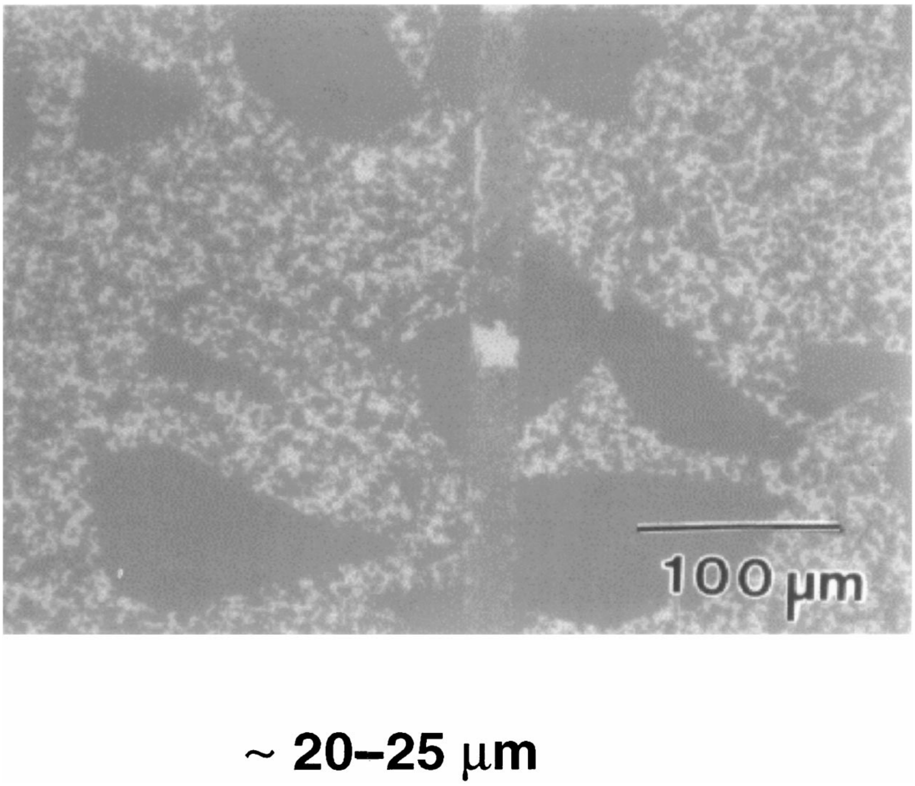
It has been demonstrated that the reaction-forming approach can be used to produce strong joints in RB-SiC



a

b

Figure 3 Microstructure of reaction-formed joints in Cerastar RB-SiC materials. (a) ∼350 μm, (b) ∼50–55 μm, and (c) ∼20–25 μm.



c

Figure 3 (*Continued.*)

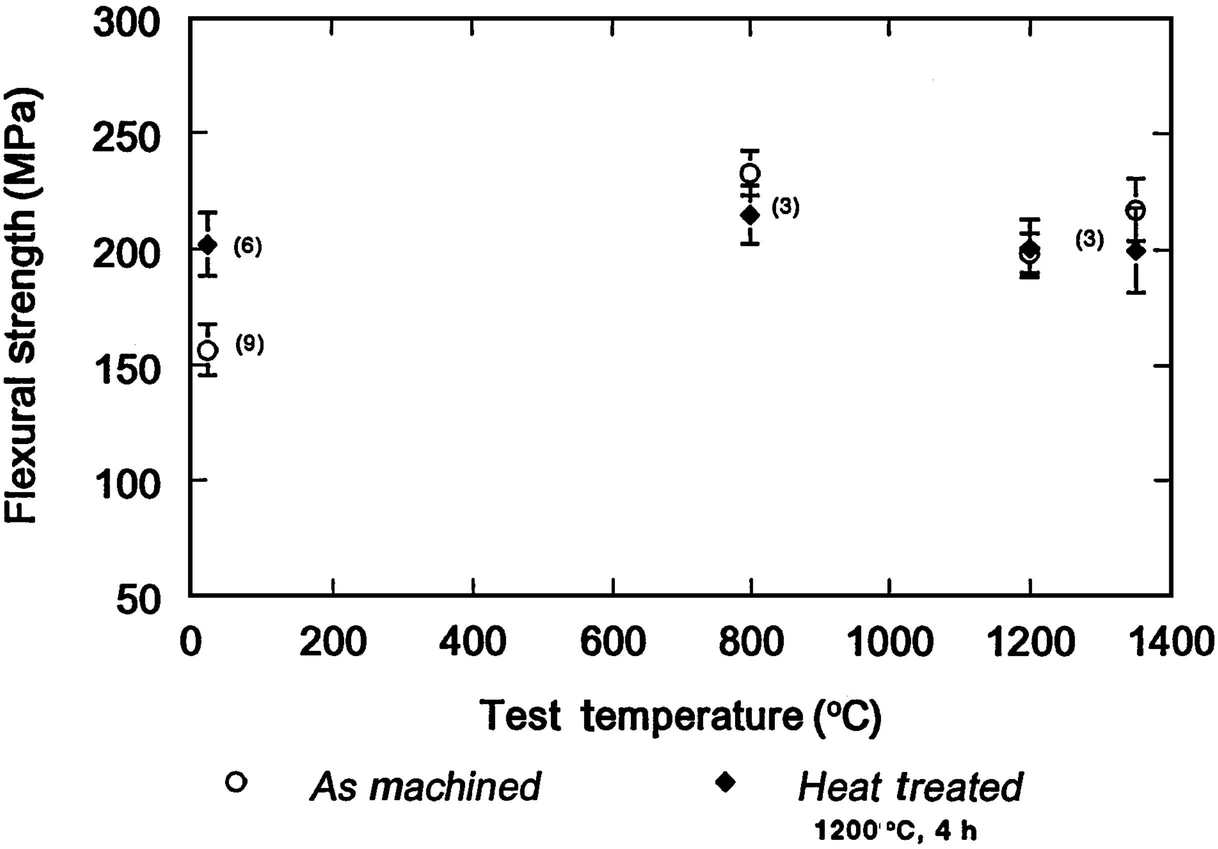


Figure 4 Flexural strengths of as-received Cerastar RB-SiC and heat-treated specimens as a function of temperature.

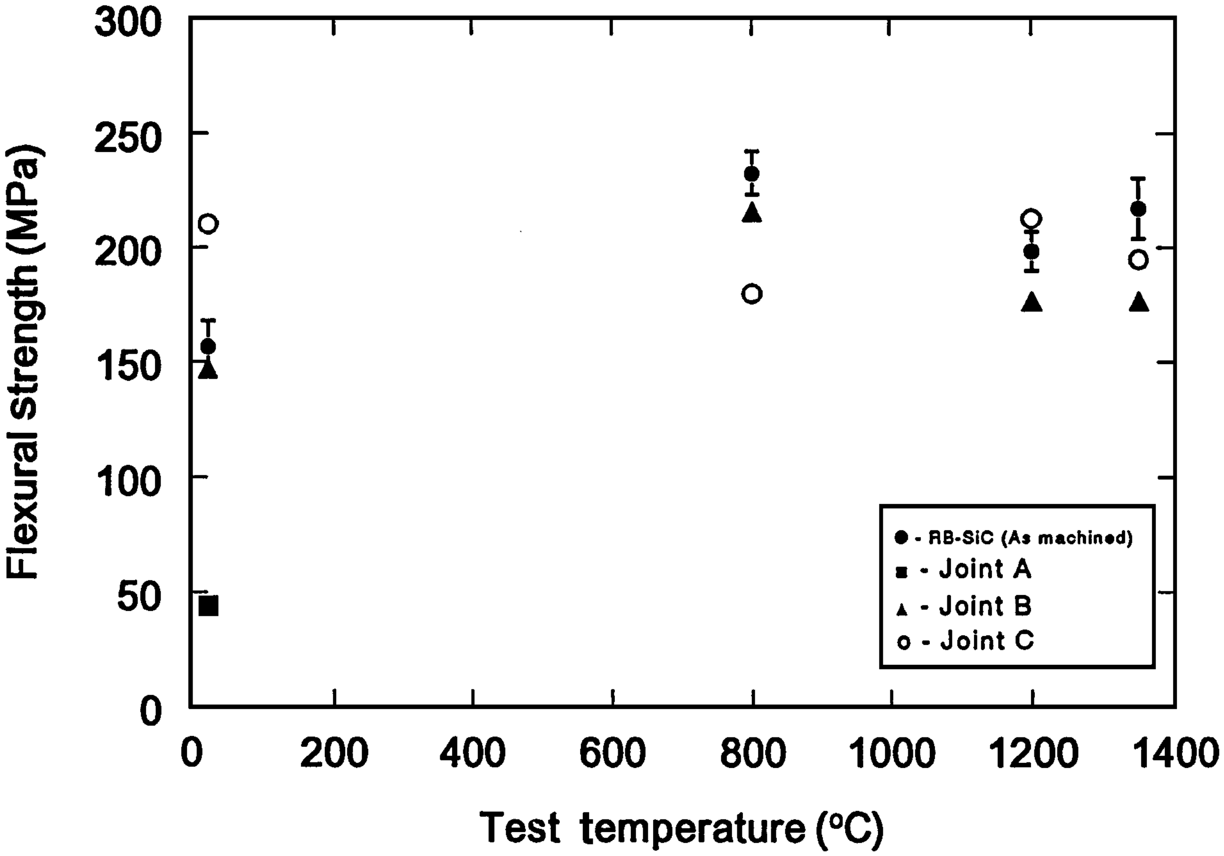
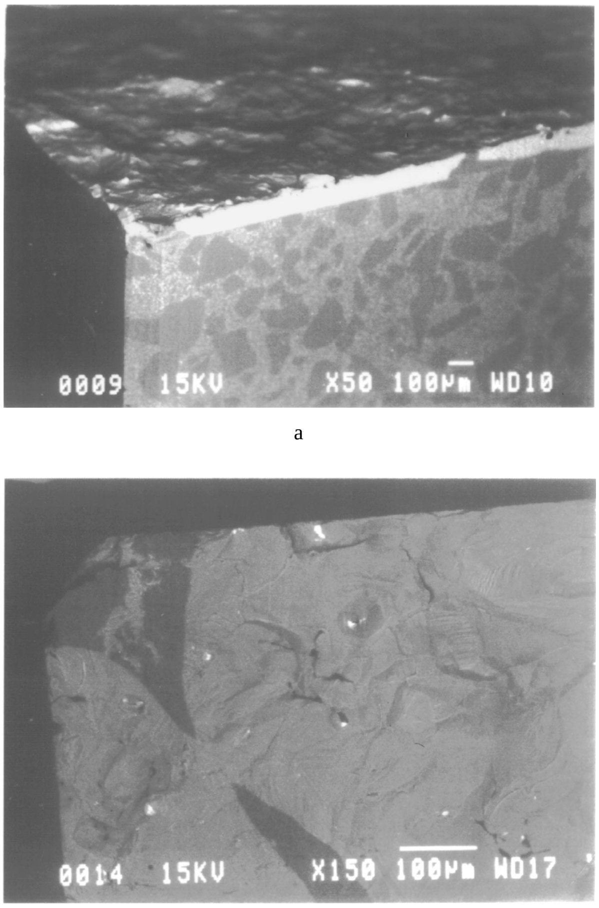


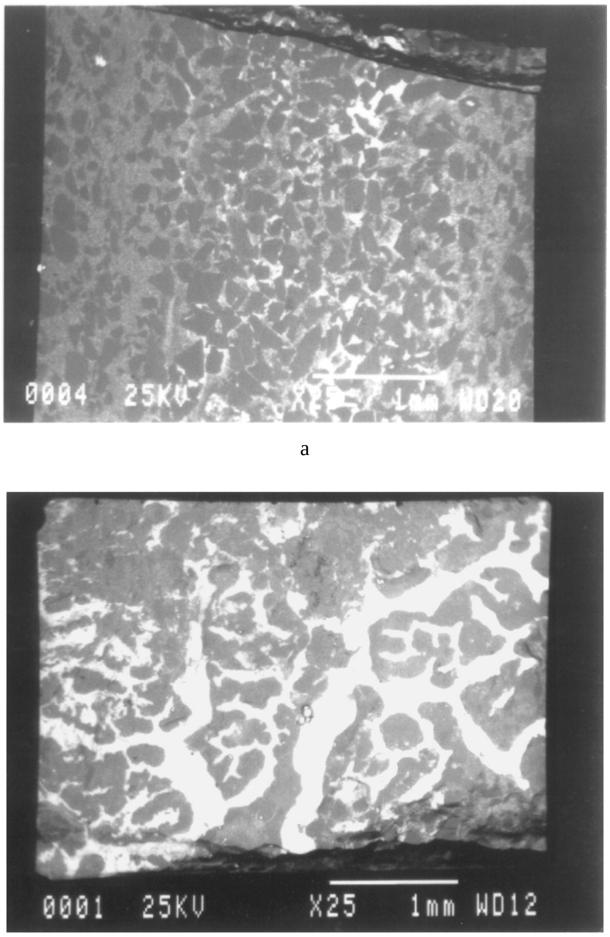
Figure 5 Flexural strengths of joined Cerastar RB-SiC specimens as a function of temperature.



a

b

Figure 6 Fractographs showing the failure behavior in thick (~350 μm) joints.



a

b

Figure 7 Fractographs showing the failure behavior in thin (~20–25 μm) joints.

materials. Thin (silicon carbide–rich) joints show no significant strength loss at high temperatures and have properties similar to the bulk parent material used in this study.

Acknowledgements

The author would like to thank Mr Steve Cogoli of Carborundum Co. for materials and Dr S. C. Farmer and Ms Pat Dickerson for their help in fractography. The technical assistance of Mr R. F. Dacek in this work is greatly appreciated.

References

1. R. W. MESSLER, JR., in "Joining of advanced materials" (Butterworth-Heinemann, Boston, MA, 1993).
2. M. M. SCHWARTZ, "Ceramic joining" (ASM International, Materials Park, OH, 1990).
3. C. H. BATES, M. R. FOLEY, G. A. ROSSI, G. J. SANDBERG, and F. J. WU, *Ceramic Bulletin* **69** (1990) 350–356.
4. O. M. AKSELSEN, *J. Mater. Sci.* **27** (1992) 569–579.
5. *Idem, ibid.* **27** (1992) 1989–2000.
6. T. J. MOORE, *J. Amer. Ceram. Soc.* **68** (1985) C151–153.
7. T. ISEKI, K. ARAKAW, and H. SUZUKI, *J. Mater. Sci. Lett.* **15** (1980) 1049–1050.
8. S. YAZIMA, K. OKAMURA, T. SHISHIDO, Y. HASEGAWA, and T. MATSUZAWA, *Ceram. Bull.* **60** (1981) 253.
9. I. E. ANDERSON, S. IJADI-MAGHSOODI, O. UNAL, M. NOSRATI, and W. E. BUSTAMANTE, in "Ceramic Joining," edited by I. Reimanis, C. H. Henager, Jr., and A. P. Tomsia. (The American Ceramic Society, Westerville, OH, 1997) pp. 25–40.
10. R. A. SILBERGLITT, I. AHMAD, W. M. BLACK, and J. D. KATZ, *MRS Bull.* (November 1993) 47–50.
11. I. AHMAD and R. SILBERGLITT, *Mater. Res. Soc. Proc.* **314** (1993) 119–130.
12. C. H. HENAGER, JR., and R. H. JONES, in "Ceramic Joining," edited by I. Reimanis, C.H. Henager, Jr., and A.P. Tomsia (The American Ceramic Society, Westerville, OH, 1997) pp. 117–126.
13. B. H. RABIN and G. A. MOORE, *J. Mater. Synth. Process.* **1** (1993) 195–201.
14. *Idem, Mater. Res. Soc. Proc.* **314** (1993) 197–203.
15. M. SINGH, *Technology* **2006** (1996).
16. M. SINGH, J. D. KISER, and S. C. FARMER, *Ceram. Eng. Sci. Proc.* **18** (1997) 161–166.
17. M. SINGH, *Scripta Materialia* **37** (1997) 1151–1154.
18. M. SINGH and J. D. KISER, in "Physics & Process Modeling and other Propulsion R&T Conference," NASA CP-10193, **5** (1997) 1–10.
19. M. SINGH, *J. Thermal Technology* (September 1997) 91–93.
20. *Idem, J. Mater. Sci. Lett.* **17** (1998) 459.

Received 15 January
and accepted 7 February 1998