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MICROWAVE JOINING OF SIC CERAMICS AND COMPOSITES

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MICROWAVE JOINING OF SiC CERAMICS AND COMPOSITES

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INTRODUCTION

Potential applications of SiC include components for advanced turbine engines [1], tube assemblies for radiant burners and petrochemical processing [2], and heat exchangers for high efficiency electric power generation systems [3]. Reliable methods for joining SiC are required in order to cost-effectively fabricate components for these applications from commercially available shapes and sizes. Joints are needed with strength approaching that of the base material under service conditions (e.g., $T > 1000^{\circ}\text{C}$ in a corrosive environment). It is possible to directly join SiC specimens by diffusion bonding, and a joint strength comparable to that of the base material has been achieved, but this required a joining temperature of 2000°C , plus constraints to avoid deformation [4]. Consequently, a great deal of recent work on joining of SiC has focused on forming interlayers composed exclusively or primarily of SiC in situ during the joining process through chemical reactions or decomposition of polymeric precursors [5-10].

Microwave heating was shown several years ago to be an effective means to join electrically insulating ceramics such as Al_2O_3 , mullite and Si_3N_4 , with joint strength and fracture toughness equal to or greater than the base material [11,12]. Recently, microwave diffusion bonding of small specimens of SiC has also been accomplished, with joint strength 71% of the base material [13]. As with the conventional method, a joining temperature of 2000°C was required; however, the joining time was reduced from one hour with conventional heating to five minutes with microwave heating. Other recent work on microwave joining of SiC has consisted of: (a) joining of reaction bonded SiC, which contains free silicon [14,15]; (b) joining with silicon-based braze interlayers [16]; and (c) microwave-

assisted decomposition of a polymer precursor to form a SiC-containing interlayer in situ [17].

This manuscript reports the results of microwave joining experiments performed using two different types of SiC materials. The first were on reaction bonded SiC, and produced joints with fracture toughness equal to or greater than that of the base material over an extended range of joining temperatures. The second were on continuous fiber-reinforced SiC/SiC composite materials, which were successfully joined with a commercial active brazing alloy, as well as by using a polymer precursor.

MATERIALS AND EXPERIMENTAL PROCEDURES

The SiC materials used in these experiments were reaction bonded SiC manufactured by Coors Ceramics Company of Golden, CO and chemical vapor infiltration (CVI) SiC/SiC composites manufactured by Dupont Lanxide Composites, Inc. of Newark, DE. For the reaction bonded SiC joining, no interlayer materials were used. For the SiC/SiC composite joining, the interlayer materials were Cusin-1 ABATM active brazing alloy manufactured by WESGO, Inc. of Belmont, CA or allyhydridopolycarbosilane (AHPCS) manufactured by Starfire Systems, Inc. of Watervliet, NY. SiC powders added to the AHPCS to form a slurry mixture were obtained from Performance Ceramics Company of Penninsula, OH.

Three different microwave joining apparatus were used to perform these experiments. The reaction bonded SiC joining was accomplished using a 6 kW maximum power CW microwave source operating at 2.45 GHz. Power was delivered via WR284 waveguide to a 2' x 2' x 2' rectangular multimode cavity applicator. The specimens were placed inside a cylindrical enclosure composed of high temperature alumina insulation which was lined on the inside with a thin layer of SiC to provide hybrid (microwave plus radiant) heating. This apparatus has been described previously [15]. The SiC/SiC composite brazing was accomplished using a 1 kW maximum power CW microwave source operating at 2.45 GHz. Power was delivered via WR284 waveguide to a TE₁₀₃ single mode cavity applicator that was sealed with pressure windows and fittings to allow filling with an inert gas. This apparatus has also been described previously [17]. The SiC/SiC composite joining using polymer precursor was accomplished using the 6 kW microwave source, together with a cylindrical cavity applicator approximately 16" in diameter and 16" long that contains O-ring vacuum seals as well as pumping and observation ports. During all joining experiments, input and reflected power were monitored with standard power meters and specimen temperature was monitored

using a two-color optical pyrometer, which was focused on the joint interface through observation holes or ports in the applicator and small holes made for this purpose in the insulated enclosure.

Reaction Bonded SiC Joining

The specimens were tubes with outer diameter of 1.375", inner diameter of 1", and length of 1" with polished ends. They were placed inside the insulated enclosure in the rectangular multimode applicator with the tube axis vertical and a joining pressure of approximately 0.23 MPa was applied via a SiC tube inserted through a hole in the top of the insulation. No interlayer material was used. Using microwave input power ranging from 2 to 3.5 kW, the joining temperature was reached in 35-45 minutes and the specimens were held (to within 15°C) at this temperature for approximately 30 minutes. Specimens were joined at four different joining temperatures above the (1410°C) melting point of silicon: 1420°C, 1465°C, 1515°C, and 1565°C.

Brazing of SiC/SiC Composites

The specimens were 1" diameter disks approximately 0.125" thick which were prepared for joining by grinding the surfaces to be joined on a 240 and then a 600 grit diamond wheel. The brazing foil was 1" in diameter and approximately 0.002" thick. The specimens were placed inside a block of alumina insulation at the maximum electric field position of the applicator with the electric field perpendicular to the disk cross-section and held in place with mullite push rods. The applicator was purged and filled with a gas mixture consisting of 95% N₂, 25% H₂. Using 600-800 Watts of input microwave power, the specimens were raised to a joining temperature of approximately 900°C, which was held for 30 minutes. (The liquidus temperature of the brazing alloy is 806°C.)

Joining of SiC/SiC Composites Using Polymer Precursor

The specimens were 0.5" x 0.5" x 0.125" plates, which were prepared for joining by grinding the surfaces to be joined on a 240 and then a 600 grit diamond wheel. The precursor was mixed with a fine SiC powder to form a viscous paste that was applied to both of the surfaces to be joined. The specimens were then squeezed together by hand. Prior to inserting the specimens into the applicator, the hybrid heating enclosure was placed inside the applicator on a stand consisting of ceramic bricks, with alumina insulating blocks above and below the enclosure. This assembly was baked at approximately 1000°C for one and one half hours using the microwave power source, with the vacuum valve opened periodically to remove moisture. Microwave power was then turned off, and the applicator was evacuated to a pressure of 6×10^{-3} torr and then back-filled with argon. The

applicator was then opened briefly to allow insertion of the specimens, which were placed approximately in the center of the hybrid heating enclosure. The applicator was then closed, evacuated to a pressure of approximately 1×10^{-2} torr, and back-filled with argon. Using 2-3 kW of microwave input power, the specimens were brought from ambient temperature to 700°C in 1 hour and 15 minutes, and then to the joining temperature of 1100°C in an additional 15 minutes. The specimens were held at 1100°C for 30 minutes.

RESULTS AND DISCUSSION

Reaction Bonded SiC

Test bend bars oriented along the tube axis and containing the joint in the center were machined from each joined specimen. Each specimen provided 6-8 test bars. Microscopic examination of the joint interfaces revealed a continuous silicon interlayer approximately $5\text{-}10\ \mu\text{m}$ thick, consistent with previous work [14,15]. Chevron notches were machined into the joint interface of each test bar, as indicated in Figure 1. The bars were then tested to failure in 4-point flexure, and the fracture toughness was computed from the load to failure data. Figure 2 is a plot of the average fracture toughness of each joined specimen, versus joining temperature. The fracture toughness of the base material was determined by machining identical test bend bars out of an unjoined rod of the same material. Figure 2 shows that the average fracture toughness of the specimens joined in the range of $1420\text{-}1515^{\circ}\text{C}$ is equal to or greater than that of the base material.

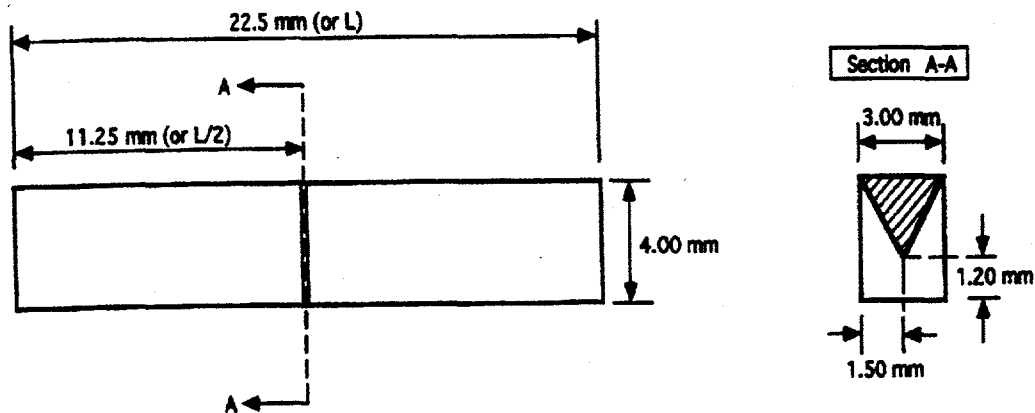


Figure 1: Test bend bar machined from joined reaction bonded SiC tubes.

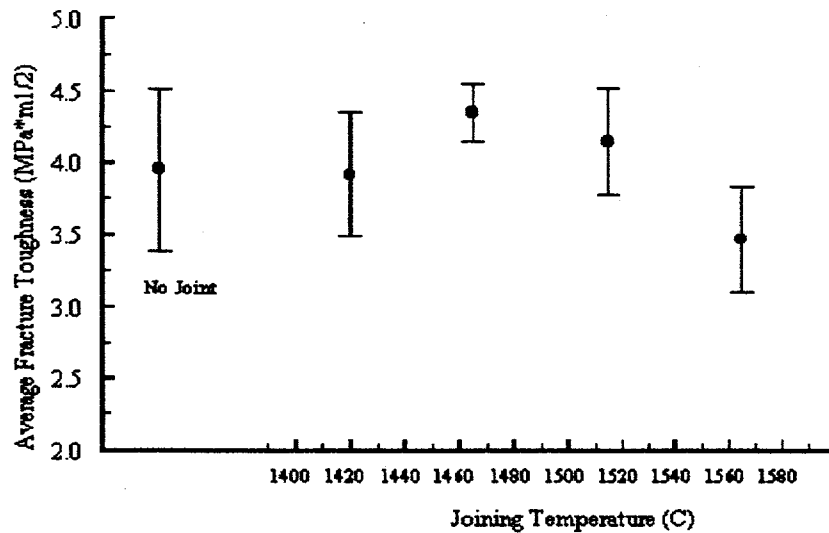


Figure 2: Average fracture toughness of joined reaction bonded SiC tubes.

Brazed SiC/SiC Composite

The specimen was joined when removed from the applicator. The region near the outer edge of the specimen fell apart during sectioning, but the remainder of the specimen remained intact and appeared to have a homogeneous metallic interlayer. An optical micrograph of the sectioned joint is shown in Figure 3. It appears from this micrograph that the braze alloy wet the ceramic composite and spread into the pores in the SiC matrix.

SiC/SiC Composites Joined Using Polymer Precursor

The joined specimens were sectioned, polished and examined with a Scanning Electron Microscope (SEM). A typical SEM micrograph is shown in Figure 4, with the joint interface vertical and approximately in the center of the figure. From Figure 4 it is evident that the joint contains some bonded regions with no visible interface, some bonded regions with a continuously bonded interface, and some bonded regions with remnant porosity. However, the remnant porosity at the joint is similar to porosity elsewhere in the specimen, except that there is substantial pore filling by the interlayer material in the porosity at the joint. Figure 5 is an SEM micrograph that shows the filling of porosity at the interface with a two-phase mixture consisting of SiC formed from polymer decomposition and SiC powder that was added to the polymer to form the slurry used for joining.

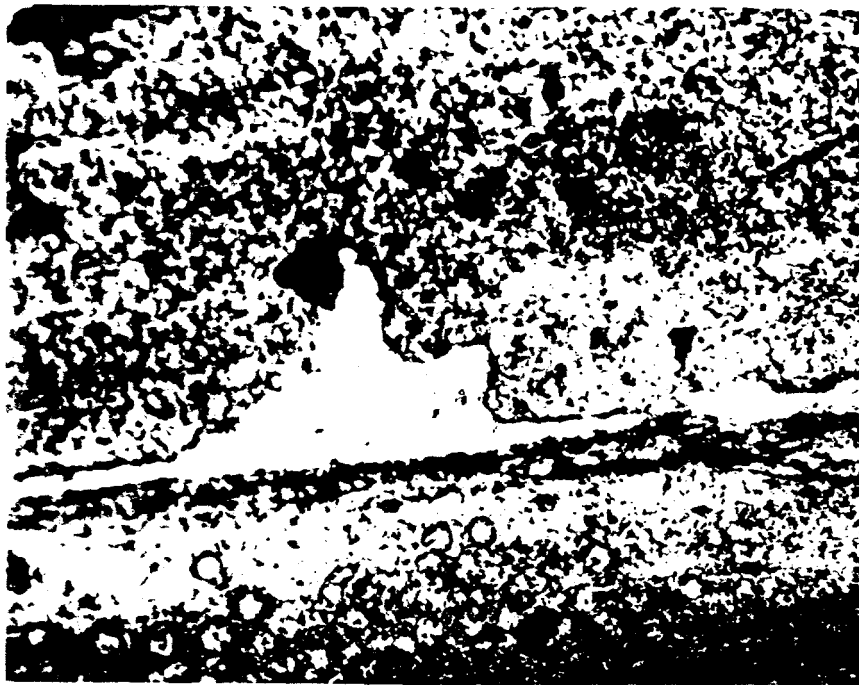


Figure 3: Optical micrograph (200 X) of SiC/SiC joint made using braze alloy.

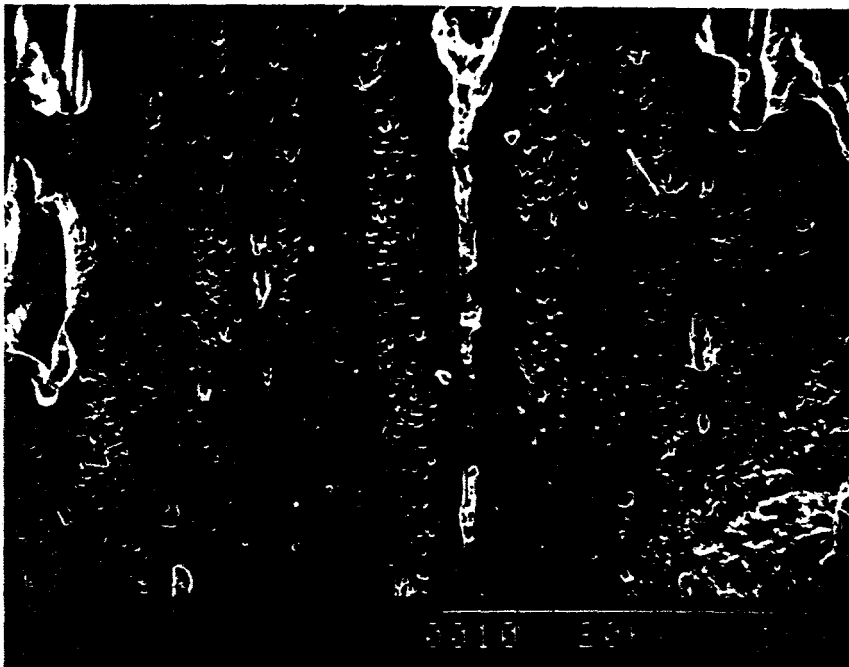


Figure 4: SEM micrograph of SiC/SiC joint made using polymer precursor.

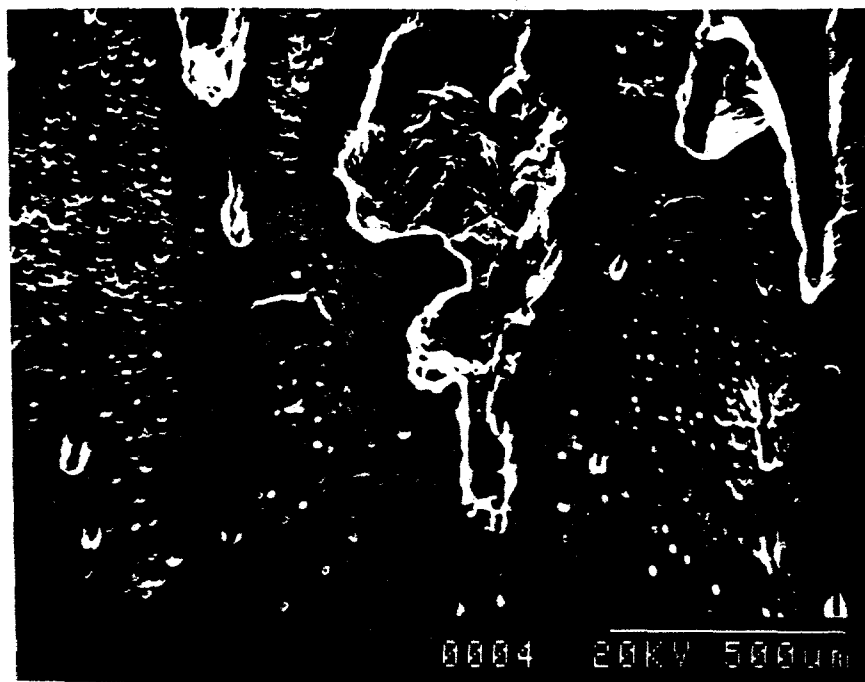


Figure 5: SEM micrograph of SiC/SiC joint made using polymer precursor.

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