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INVESTIGATION OF INTERLAYER MATERIALS FOR THE MICROWAVE JOINING OF SiC

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Microwave joining of ceramics has the potential for increased speed and convenience [1,2]. Joints have been made in alumina, mullite and silicon nitride with flexure strength approaching, and in some cases exceeding, that of the as received material in a fraction of the time that is customarily required with conventional techniques [3,4]. This paper describes the initial results of investigations aimed at applying microwave joining to SiC and other carbide ceramics.

RATIONALE FOR THE JOINING APPROACH

Microstructural, mechanical and X-ray data reported in Refs. 3 and 4 suggest that microwave joining occurs through the filling of pores in the interfacial region by intergranular glassy phases. This mechanism is further supported by

conventional joining experiments in silicon nitride, in which the joining material is an oxynitride glass very similar to the silicon nitride intergranular phase [5,6]. These results suggest the following general approach to microwave joining: first investigate joining without interlayer materials, using the intergranular phases present in the material to be joined; if direct joining is not possible, then use as a joining interlayer a material that closely approximates the intergranular phases.

To implement this approach for SiC joining, two different avenues were followed. First, metallic braze joints were made using Si as the primary interlayer material. Second, attempts were made to initiate a combustion synthesis reaction [7] in the interfacial region to form a composite interlayer containing SiC or TiC.

PREPARATION OF SPECIMENS TO BE JOINED

The SiC specimens used for joining were disks of Carborundum Hexoloy™ approximately .952 cm in diameter and .635 cm in height, rough cut with a wafering saw, with no polishing.

Four different methods were used to prepare and apply interlayer materials. Three of these methods used blended powders of Si, C and Ti. The first method consisted of simply placing the powder mixture on top of one SiC disk and then pressing the second disk in place. In the second method, the powder was mixed with Nye* watch oil to form a slurry, which was applied to the top of the lower disk. In the third method, a cold pressed disk of the powder was made, using 1-Eicosene as a binder, and this disk was inserted in between the two SiC disks. In the fourth method, a thin layer of Si was plasma-sprayed onto one of the SiC disks.

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EXPERIMENTAL PROCEDURE

These microwave joining experiments, with the one exception noted below, were carried out using the apparatus described in Ref. 3. The samples were placed in the center of a TE_{103} rectangular microwave cavity and oriented with the electric field parallel to the disk axis. Coupling of microwave power from the 2.45 GHz magnetron was accomplished with a resonant rectangular iris. The dimensions of the iris were determined experimentally by requiring nearly critical coupling between the input waveguide and the cavity when the sample was within the desired joining temperature range.

The power incident on and reflected from the cavity was monitored with voltage probes inserted in appropriate positions through the broad wall of the waveguide. The temperature of the sample was measured through a shielded hole cut in the narrow wall of the cavity using a two-color IR pyrometer.

The SiC samples and interlayer materials were held in place for joining by two low loss alumina rods of .952 cm diameter. This sandwich of alumina-SiC-interlayer material-SiC-alumina was inserted through openings in the broad wall of the cavity, positioned so that the interlayer material was at the geometrical center of the cavity, and clamped in place with a pressure of 2-5 MPa provided by a hydraulic press. Microwave power was applied and the movable endwall of the cavity adjusted for minimum reflected power, as indicated by the voltage probes. The power was increased until the desired sample temperature was achieved, with the endwall readjusted as necessary to minimize reflected power during heating.

Joints were also made using a 6 kW magnetron and a 61 X 61 cm square multi-mode tunable cavity [8]. In this case, the sample consisted of a SiC disk with a plasma-sprayed Si coating on one side and a second (untreated) SiC disk which was placed on top of the coated side of the first disk. The two disks were

then placed inside a zirconia enclosure inside the multi-mode cavity and the power increased until the desired temperature was reached. The temperature was monitored through observation ports in the cavity and the zirconia enclosure using a two-color IR pyrometer.

DISCUSSION OF JOINING RESULTS

A series of joints was made using Si as the interlayer material. The joining temperature was approximately 1450°C, the joining time was 5-10 minutes, and the applied power was approximately 250 Watts. Figures 1-3 are SEM micrographs of sectioned joined specimens. While all of these joints have a homogeneous interlayer, the width of the joint varies considerably with the joining method. The joint made using Si powder (Fig. 1) is 50 micrometers in width. The joint made using an oil-based slurry made from the powder (Fig. 2) is 10 micrometers in width. The joint made from a SiC disk with a plasma-sprayed Si layer (Fig. 3) is less then 5 micrometers in width. A series of indentation measurements made on a line perpendicular to this last joint section showed no substantial variation in Knoop hardness across the joint interface.

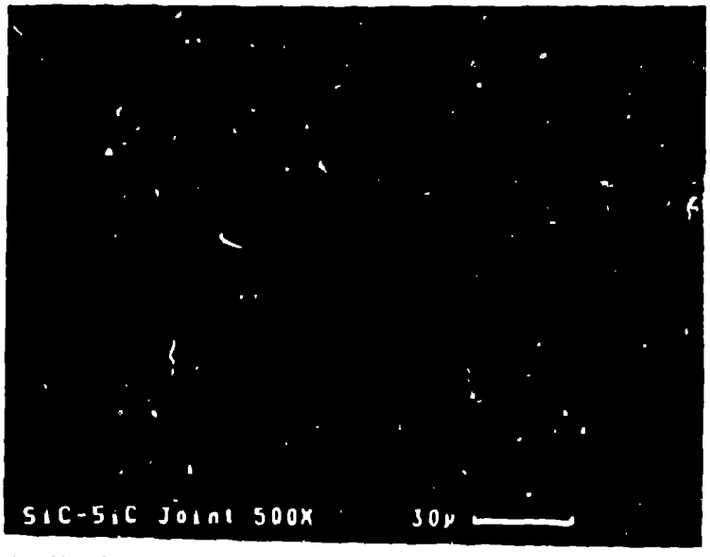

Figure 1 SEM micrograph of SiC joint made with Si powder.

Figure 2 SEM micrograph of SiC joint made with oil-based slurry of Si powder.

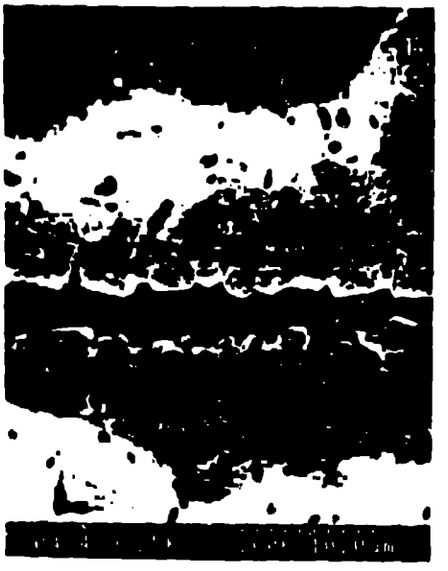

Figure 3 SEM micrograph of SiC joint made with plasma-sprayed Si coating.

As a prelude to joining experiments attempting to utilize combustion synthesis in the interfacial region, we used the multi-mode cavity to react cold pressed disks equal in size to the joining samples. A disk made from Si and C powders in stoichiometric ratio was heated first. X-ray diffraction data showed only Si and C peaks, indicating that no combustion synthesis reaction took place. Since the Ti-C reaction is much more energetically favorable than the Si-C reaction (Ref. 7), we then heated a cold pressed disk composed of 2% Ti and 98% stoichiometric Si:C powders. The X-ray diffraction data obtained from this disk clearly indicated the presence of SiC.

Based upon the above results, joints were attempted using powder mixtures of 4% and 10% Ti, with the balance being stoichiometric Si:C. These joints were attempted with all three of the methods described above, i.e., powder, oil-based slurry and cold pressed disk. No combustion reaction was observed during the heating of these materials.

An additional joining attempt was made using a cold pressed disk of about 1 mm in height made from a stoichiometric powder mixture of Ti and C, in an Eicosene binder. The assembly of SiC disk-Ti:C cold pressed disk-SiC disk was heated using the single mode cavity method described above. The Eicosene binder was first observed to vaporize; then as the power was increased a flame was observed to be ignited at the joining interface. When this flame extinguished, the temperature of the SiC disks rose rapidly to 1500°C and stabilized. This temperature was held for several minutes, and then the samples were cooled by gradually reducing the microwave power, over a period of approximately 15 minutes. Figures 4 and 5 are SEM micrographs of the sectioned joined specimens. Fig. 4 was taken at the outer edge of the specimen. Fig. 5 was taken in the interior of the sectioned specimen. The interlayer material in Fig. 4 is orange-white and appears to be an oxide. The interlayer material in Fig. 5 is dark gray and appears to be a carbide. X-ray analysis is in progress to identify these phases.

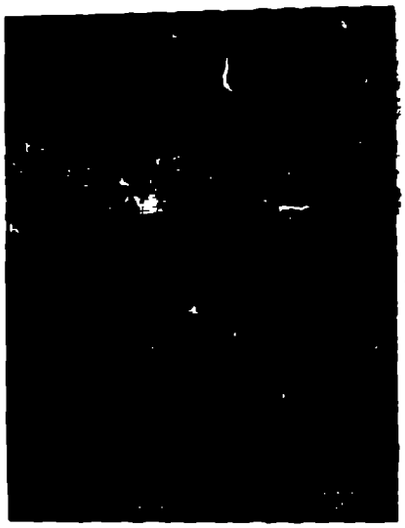

Figure 4 SEM micrograph of SiC joint made with cold pressed disk of Ti:C (outer edge).

Figure 5 SEM micrograph of SiC joint made with cold pressed disk of Ti:C (interior of sectioned specimen).

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