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QUARTERLY TECHNICAL PROGRESS REPORT
SUBCONTRACT NUMBER 4008E0014-3G
RESEARCH ON MICROWAVE JOINING OF SiC
FM TECHNOLOGIES, INC.
APRIL 1 - JUNE 30, 1995

Abstract

The objectives of this research project are to identify optimum time-temperature profiles for the microwave joining of silicon carbide and to develop new microwave joining methods that can be applied to accomplish in situ formation of silicon carbide interlayers and to join larger samples required for industrial applications. Work during this reporting period was focused on investigation of the effect of specimen preparation on joining of SiC using polymer precursors to form SiC in situ at the interface. During this period, LANL also completed the evaluation of joints that were made by FMT using four different joining temperatures, as part of an effort to determine optimum joining temperature.

Summary of Work Performed

Task 1 - Optimization of time-temperature profile

During a previous performance period, specimens of Coors reaction bonded silicon carbide (RBSC) tubes with outer diameter of 3.49 cm (1.375 in), inner diameter of 2.54 cm (1 in) and length of 2.54 cm (1 in) were joined at four different joining temperatures: 1420°C, 1465°C, 1515°C and 1565°C. In each case the joining temperature was maintained to within 15°C for approximately 30 minutes. The joined specimens were sent to Los Alamos National Laboratory for mechanical evaluation. Test bend bars were machined from each specimen and Chevron notches machined into the joint interface, as indicated in Figure 1. Figure 2 shows the fracture toughness determined from 4-point flexure tests of the notched test bend bars. The data labeled "no joint" were derived from test bend bars machined from an as-received specimen of RBSC. The average fracture toughness was determined from measurements on 6-8 specimens and the standard deviation is indicated by the shorter bars in Figure 2. The joining temperatures for each specimen were as follows: specimen 1A at 1465°C; specimen 1B at 1515°C; specimen 2A at 1565°C; specimen 2B at 1420°C. Figure 2 demonstrates that the optimum joining temperature is likely between 1420°C and 1500°C, and perhaps very close to 1465°C. *Specimens joined near this optimum temperature have fracture toughness greater than the as received material.* The standard deviation of the fracture toughness values for the joined specimens was also smaller than that for the as received material, except for specimen 1B, which had one data point with a fracture toughness value far different from all others. Inspection of the fracture surface of this test bar is being performed to see if its failure was caused by a defect in the material or by the specimen preparation process, rather than by failure of the joint.

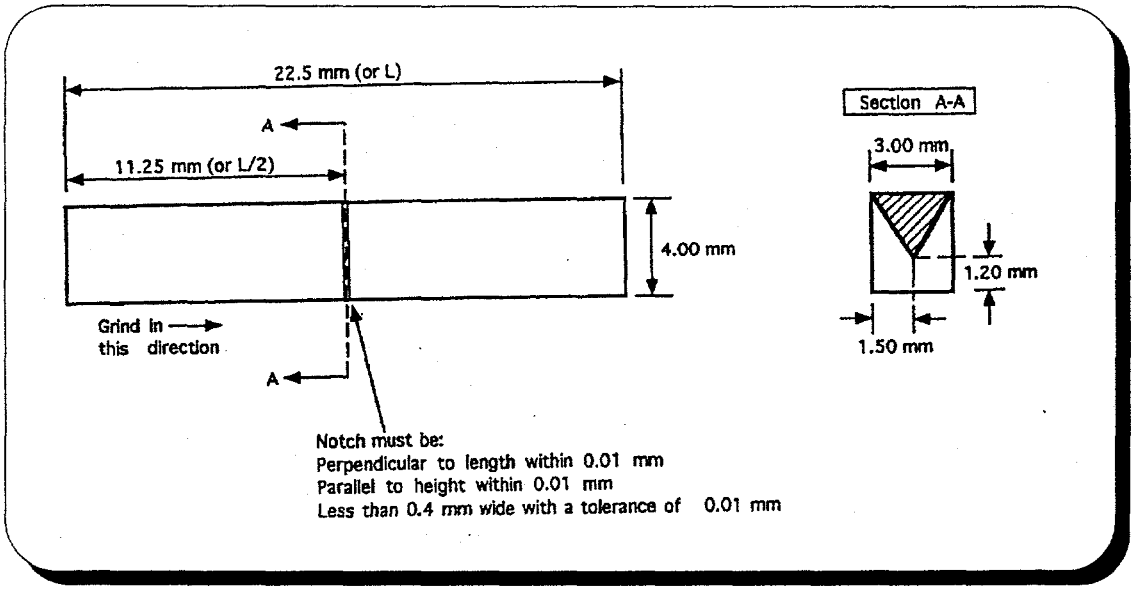

Figure 1: Schematic Illustration of Chevron Notched Fracture Toughness Specimen

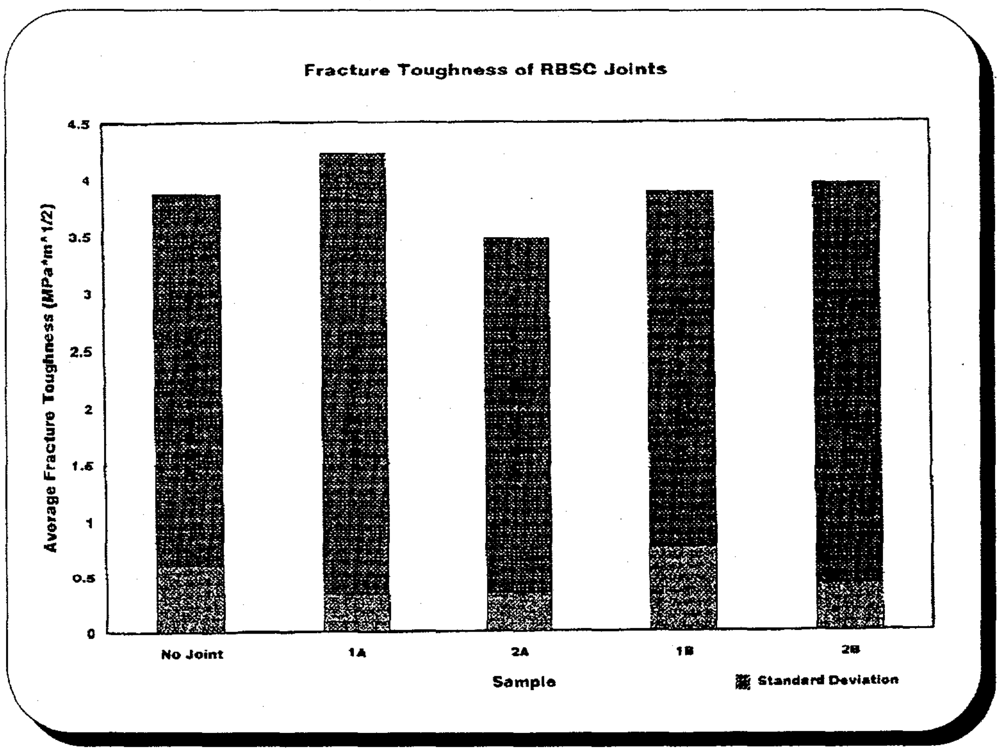

Figure 2: Fracture Toughness of RBSC Specimens (Joining Temperatures: Specimen 1A, 1465°C; Specimen 2A, 1565°C; Specimen 1B, 1515°C; Specimen 2B, 1420°C)

Task 3 - Joining of SiC using chemical precursors

A detailed evaluation was performed of the effect of surface preparation on the joining of sintered SiC, using microwave heating for the decomposition of polycarbosilane (PCS) to form SiC in situ at the interface. The specimens were Hexoloy™ sintered SiC rods 0.95 cm (0.375 in) in diameter and 0.5 cm (0.197 in) long, which were purchased from the Carborundum Company. Commercial PCS having a molecular weight of 1400 (manufactured by Nippon Carbon Corporation, Tokyo, Japan) was purchased from Dow Corning. Four sets of specimens were prepared using different surface treatments. All four sets of specimens were cut from as-received rods using a Buehler Low Speed Saw and a high concentration diamond blade. PCS was dissolved in hexane and applied to the surface to be joined. For the first set of specimens, the PCS solution was applied directly to the as-cut surface. For the second set, the surfaces to be joined were etched in 40% hydrofluoric acid before the PCS was applied. The joining surfaces of the other two sets of specimens were ground on a diamond wheel. One set of surfaces was then also etched before PCS application, while the other was not.

The specimens were placed in a TE_{103} single mode cavity applicator which had been modified under a previous task to allow processing under a mixed reducing atmosphere of 95% nitrogen and 5% hydrogen. Microwave power was coupled to the specimens using an adjustable iris and plunger and the specimens were heated to 1400-1450°C and held in this temperature range for 30 minutes. (Previous work has demonstrated that microwave heating of PCS to 1400°C provides a high degree of crystalline SiC.) These specimens either did not join or were weakly bonded. However, investigation of the adherence of SiC formed from the PCS on the sintered SiC surface indicated that the ground and etched surface was most favorable for joining. An additional set of specimens was then prepared using grinding and etching of the surface. In addition, a mixture of SiC powder and PCS was applied to the surface to be joined. This set of specimens was heated using the same conditions as above, resulting in a good joint.

Figures 3 and 4 are Scanning Electron Micrographs (SEMs) of the surface of the specimens which had the PCS applied to the as-cut surface and to the ground and etched surface, respectively. Both surfaces were scratched with a sharp metal point under a load of 820 grams in order to investigate the adherence of the SiC formed from the decomposition of the PCS. Comparison of the SEMs shows greatly enhanced wetting and spreading of the SiC formed from the PCS on the ground and etched surface. SEMs (not shown) of surfaces that were either ground or etched also showed improvement over the as-cut surface, but the combination of grinding and etching provided the best wetting and spreading. Figure 5 is a higher magnification of the micrograph shown in Figure 4, which indicates that larger flakes of SiC were peeled off by the metal point, but good coverage of the scratched area remained, suggesting that the smaller flakes were adherent. Figure 6 is an SEM of a cross-section of the specimen joined using the mixture of SiC and PCS, after grinding and etching of the surfaces to be joined. A continuous joint interlayer approximately 50-60 μm in thickness was formed. In addition, a combination of the SiC introduced at the interlayer and the SiC formed from the PCS completely filled the pores near the interface of the joined specimens.

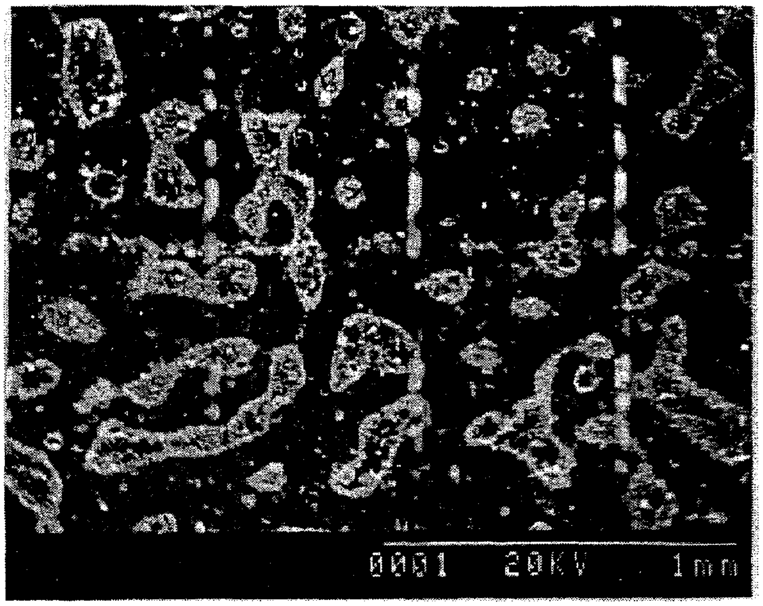

Figure 3: Scanning Electron Micrograph of As-cut SiC Surface After Application of Polycarbosilane and Microwave Heating to 1400-1450°C

Figure 4: Scanning Electron Micrograph of Ground and Etched SiC Surface After Application of Polycarbosilane and Microwave Heating to 1400-1450°C

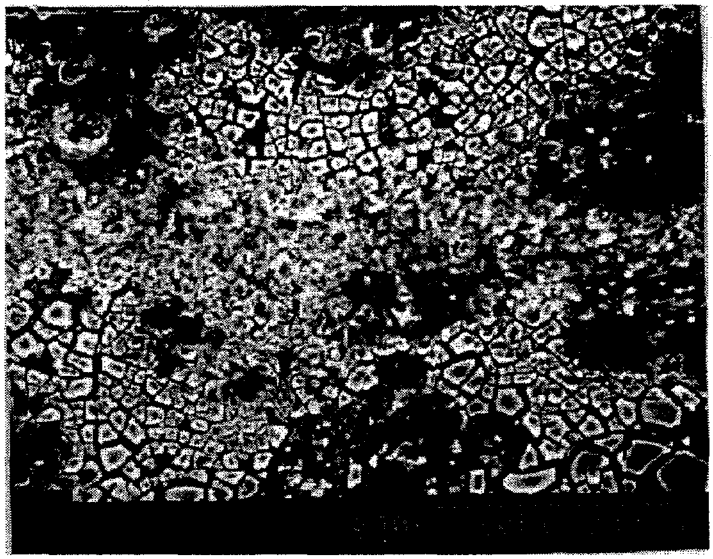

Figure 5: Higher Magnification of the SEM of Figure 4, Showing Adherence of SiC Formed From Decomposition of Polycarbosilane to the Ground and Etched SiC Surface

Figure 6: Cross-section of Sintered SiC Joined Using a Mixture of SiC and Polycarbosilane as the Interlayer Material

Presentations

Dr. Iftikhar Ahmad presented a paper describing the work on joining of SiC using PCS at the American Ceramic Society (ACers) Symposium on Microwave Processing during the ACerS Annual Meeting and Exposition, April 30-May 4, 1995 in Cincinnati, OH. Dr. Richard Silberglitt made a presentation on the work performed under this contract at the Advanced Industrial Materials (AIM) Program Annual Meeting, June 14-16, 1995, in Washington, D.C.

Publications

Two papers resulting from this work have been submitted for publication in the proceedings of the ACerS Microwave Symposium, which is currently in press as a volume of Ceramic Transactions. These papers are "Dynamic Model for Electromagnetic Field and Heating Patterns in Loaded Cylindrical Cavities," Y.L. Tian, W.M. Black, H.S. Sa'adaldin, I. Ahmad, and R. Silberglitt and "Microwave-Assisted Pyrolysis of SiC and Its Application to Joining," I. Ahmad, R. Silberglitt, T.A. Shan, Y.L. Tian, and R. Cozzens.