2.2 Low Activation Joining of SiC/SiC Composites for Fusion Applications[1] – C. H. Henager, Jr., R. J. Kurtz (Pacific Northwest National Laboratory[2], Richland, WA 99336, USA), and M. Ferraris, (Politecnico di Torino, Torino, Italy)

OBJECTIVE

This work discusses the latest developments in TiC + Si displacement reaction joining at PNNL based on mechanical property test results from miniature torsion joints.

SUMMARY

The use of SiC composites in fusion environments may require joining of plates using reactive joining or brazing. One promising reactive joining method is the use of solid-state displacement reactions between Si and TiC to produce Ti_3SiC_2 + SiC. We continue to explore the processing envelope for this type of joint for the TITAN collaboration to produce the best possible joints to undergo irradiation studies in HFIR. The TITAN collaboration has designed miniature torsion joints for preparation, testing, and irradiation in HFIR. As part of that project PNNL synthesized 40 miniature torsion joints and several were tested for shear strength prior to irradiation testing in HFIR. The resulting tests indicated that joint fixture alignment problems cause joint strengths to be lower than optimal but that several joints that were well aligned had high shear strengths and promising mechanical properties. High joint strengths cause non-planar shear fracture and complicate strength analysis for these miniature torsion joints.

PROGRESS AND STATUS

Introduction

SiC is an excellent material for fusion reactor environments, including first wall plasma facing materials and breeder-blanket modules. It is low-activation, temperature-resistant, and radiation damage tolerant compared to most materials. In the form of woven or braided composites with high-strength SiC fibers it has the requisite mechanical, thermal, and electrical properties to be a useful and versatile material system for fusion applications, especially since microstructural tailoring during processing allows control over the physical properties of interest [1-6]. However, it is difficult to mechanically join large sections of such materials using conventional fasteners so the analog of welding is being pursued for these ceramic materials [2,4-15]. Such methods include metallic brazes [8,16], glass ceramics [7,17], preceramic polymers [15], and displacement reactions [2,6]. This paper reports on the current status of SiC and SiC-composite joining for fusion applications based on displacement reactions between Si and TiC. This has been used to produce bulk composite material consisting of SiC-Ti_3SiC_2, with small amounts of TiC determined by the phase equilibria conditions [18].

Experimental Procedures

Joints are made using a tape calendaring process using organic binders and plasticizers together with a mixture of TiC and Si powders, with 99.99% purity, average diameters less than 45 μm, and a TiC:Si ratio of 3:2. The flexible tapes were 200 μm thick and were cut to a disk shape and applied between two CVD SiC miniature torsion specimens as shown in Figure 1. These green joints were placed within a graphite joining fixture shown in Figure 2 that allows for alignment of the torsion joints and for applied compressive force during heating in an inert gas furnace enclosure. Joints were formed by heating the torsion coupons in argon to 1698K at 10K/min and holding for 2 hours at 40 MPa applied pressure. The resulting joints were dense and approximately 15 μm thick. They were prepared for optical and scanning electron microscopy using standard ceramographic cutting, grinding, and polishing methods. However, the SEM results have not yet been obtained for these joints. However, several prototype joints having slightly different total thickness dimensions were fabricated at PNNL and tested at ambient temperature in torsion

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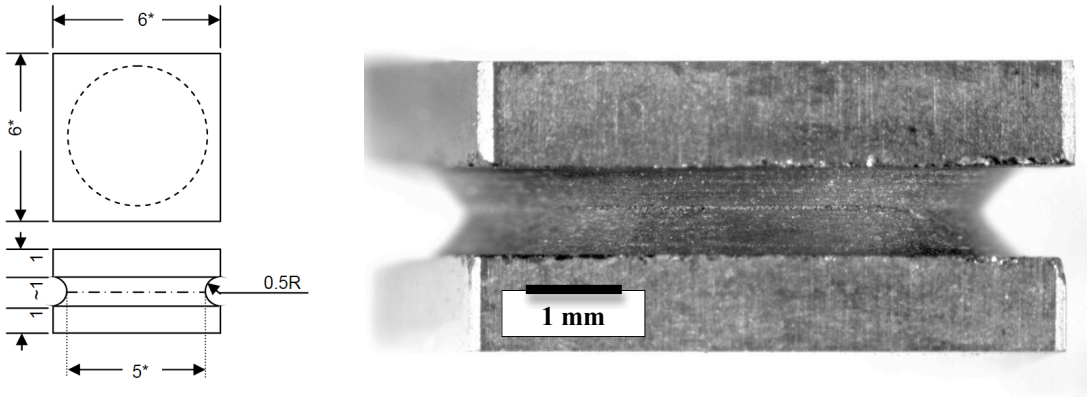
at the Politecnico di Torino, Torino, Italy by Dr. Ferraris' group and characterized there using SEM of the fracture surfaces.

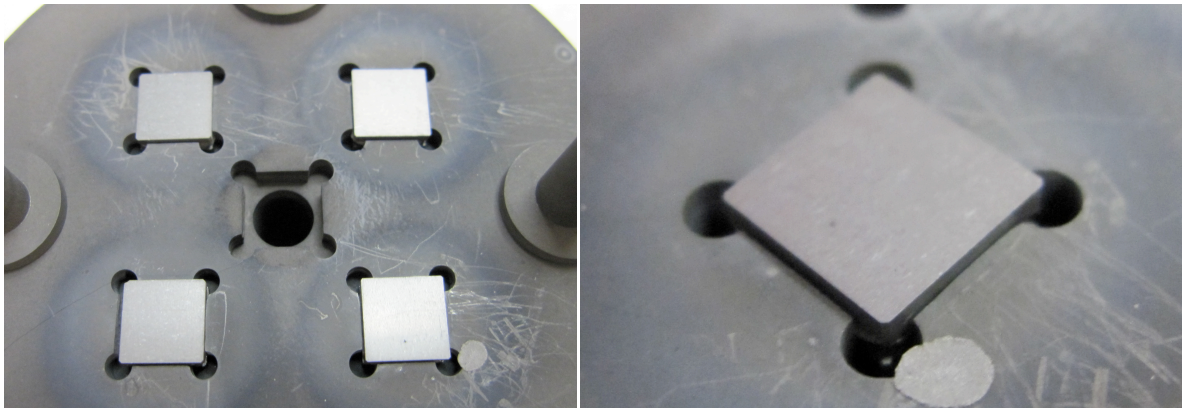
Results

Figure 1 shows the torsion specimen drawing and a fabricated joint at PNNL prior to any testing. Figure 2 shows the joints placed into the graphite alignment fixture prior to processing in argon at 1698K for 2 hours. Some slight misalignment can be observed in some of the processed joints as shown in Figure 3. An improved graphite alignment fixture has been designed based on these initial processing results, but many of the processed joints are nearly perfectly aligned as shown in Fig. 1b and these were sent to HFIR for irradiation testing and for pre- and post-test strength testing.



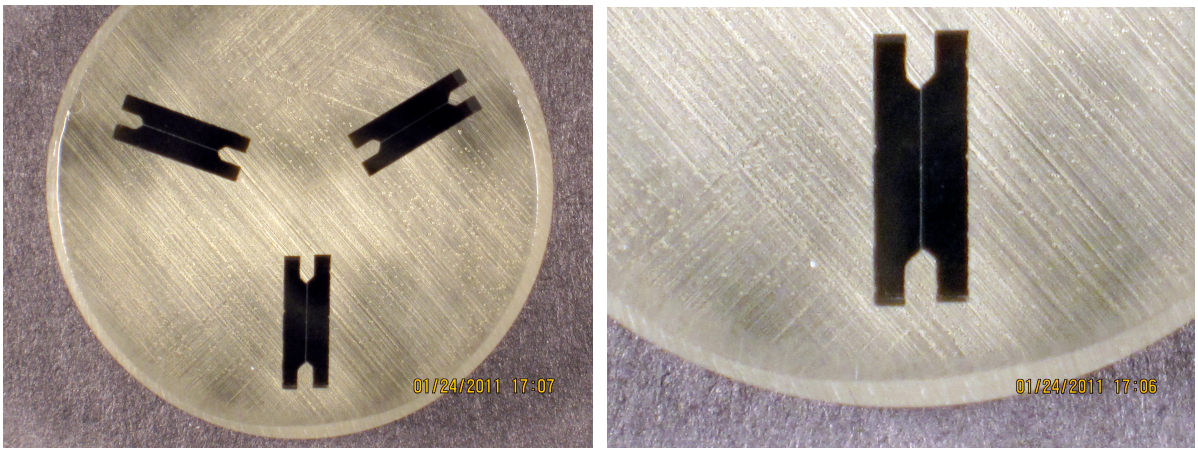
(a) (b)

Figure 1. (a) Drawing of miniature torsion joining specimen for HFIR irradiation. Dimensions are shown in mm in (a). Shown in (b) is a photomicrograph of a PNNL fabricated torsion specimen. The joint line is visible along the specimen horizontal centerline.



(a) (b)

Figure 2. (a) Photomicrograph of graphite joining fixtures with 4 torsion joints ready for processing. The central hole in this photo allows an alumina-sheathed thermocouple to be placed close to the joints during processing. Shown in (b) is a close-up photomicrograph of a single joint held in place prior to processing. The torsion joints are 6-mm square and slightly less than 3-mm in height.



(a) (b)

Figure 3. (a) Three sectioned joints mounted in epoxy for joint photomicrography and for checking joint alignment. The vertically oriented joint in the lower portion of the photo can be seen to be slightly misaligned and this is seen more clearly in (b).

Several joints were tested in Torino and the results of those tests are shown in Figure 4. The focus here is the large discrepancy between the high strength and low strength joints. The main findings in this study are that well made joints, i.e., those that are aligned and fully dense have high strengths in torsion but otherwise do not, as shown in Figure 5. The highest torsion strengths measured here exceed the target strength of 50 MPa and agree reasonably well with strengths measured using other joint strength measurements [2]. Shear lap joint tests with similarly processed joints between Hexoloy SiC bars were measured at 50 MPa shear strength in this previous study. Additionally, optical photomicrography results on recently processed joints, shown in Figure 6, show similar dense regions with typical SiC/Ti_3SiC_2 interpenetrating microstructures with strong bonding to the CVD SiC coupon surfaces. Some residual porosity is observed due to binder gas release during joint processing.

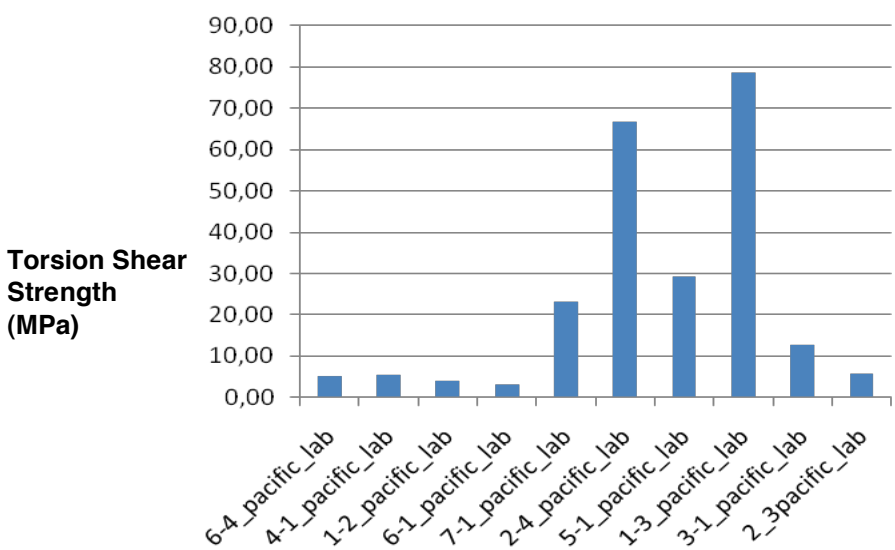
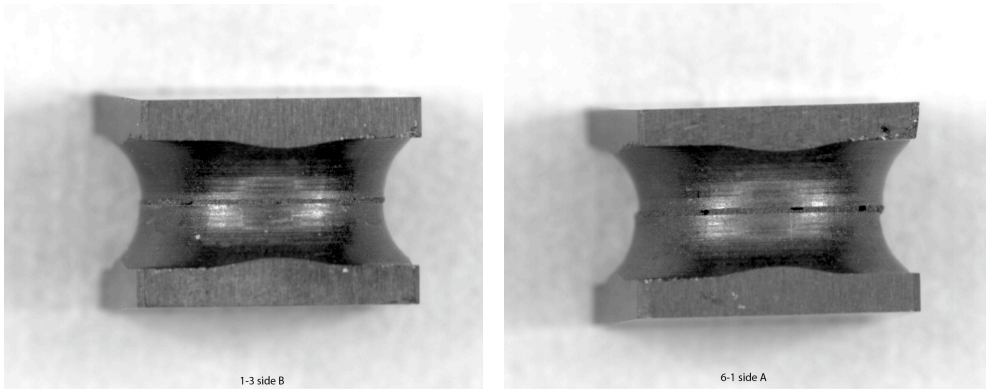
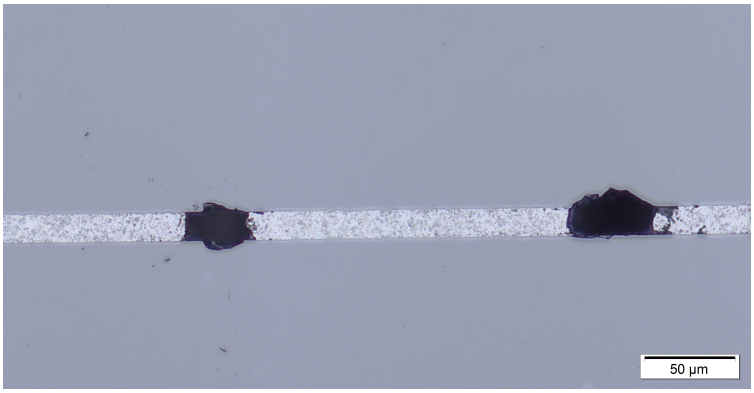


Figure 4. Bar graph of joint strengths measured in MPa of torsion shear strength. The strengths are distributed basically in two groups: those less than 30 MPa and those greater than 60 MPa. The high strength joints are of interest for irradiation testing.

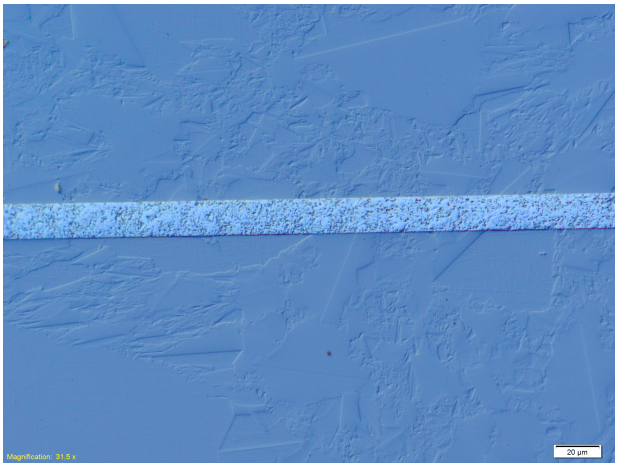


(a) (b)

Figure 5. Side-by-side comparisons of two processed joints tested in torsion at Politecnico di Torino. The joint 1-3 in (a) on the left is well aligned and is a thin, dense joint. The joint 6-1 in (b) on the right is not well aligned and the joint region is thick and not fully dense. The shear strengths were 79 MPa for joint 1-3 in (a) and 3 MPa for joint 6-1 in (b).



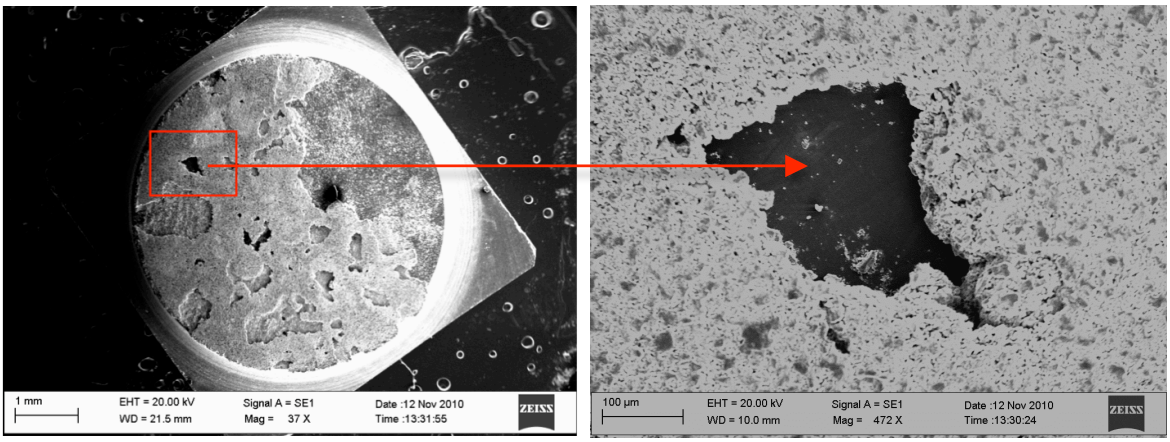
(a)



(b)

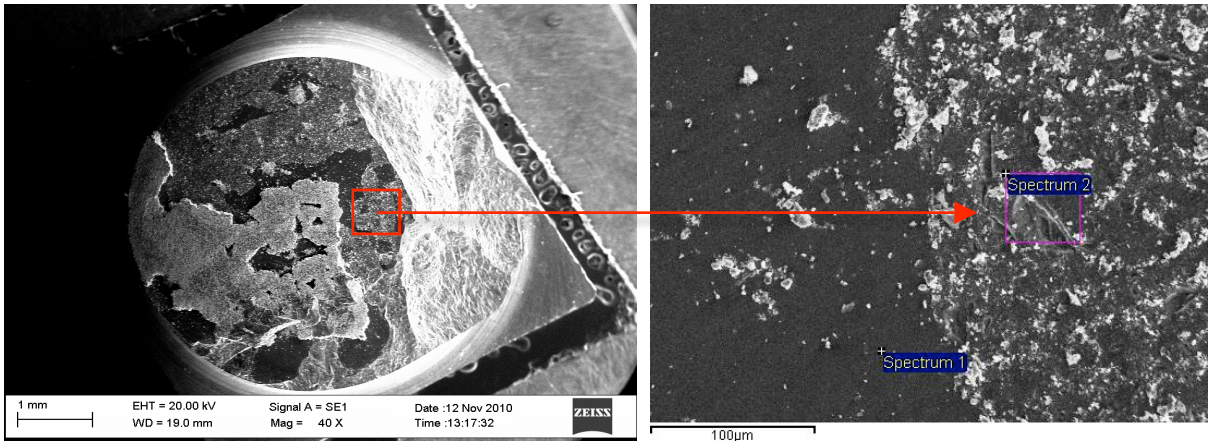
Figure 6. Photomicrographs of a polished cross-section torsion joint as seen in Figure 3. Shown in (a) is a bright-field reflected image of a joint region showing dense joint region and porosity in the joint. The porosity is thought to arise from gas release from the binder during joint processing. In (b) is a differential interference contrast (DIC) image under conditions that reveal the CVD SiC grain structure and the joint interpenetrating two-phase structure is also highlighted by this technique.

Several of the fractured joints were examined at Politecnico via SEM. Specimen 6-1, a low strength joint (3 MPa) fractured along the torsion joint in shear as shown in Figure 7. Fractography reveals that this joint was weak due to poor bonding to the CVD SiC surface and due to the porosity of the joint. However, joint 1-3, which was a high strength joint (79 MPa) is imaged in Figure 8. A non-planar fracture in the joint region is observed and at higher magnifications it can be seen that surface damage to the CVD SiC has occurred due to strong joint bonding. Further, although these images do not reveal that explicitly, the joining materials imaged on the joint fracture surface are dense compared to those seen in Figure 7. Therefore, both joint alignment and proper processing are observed to play a role in joint strength in these preliminary torsion shear tests. Recent joints sent to ORNL for HFIR irradiation testing were handpicked for alignment and much care was taken in proper processing conditions for those joints to ensure that the proper joining pressure was applied during processing. Joint regions should be less than 20 μm in thickness as shown in Figure 6 to ensure proper densification has occurred.



(a) (b)

Figure 7. Low magnification SEM micrograph in (a) reveals a planar fracture surface for specimen 6-1, which had a 3 MPa shear strength measurement. The higher magnification photo in (b) indicates that regions of unbonded SiC are observed and that the joint material is highly porous.



(a) (b)

Figure 8. Low magnification SEM micrograph in (a) reveals a non-planar fracture surface for specimen 1-3, which had a 79 MPa shear strength. The higher magnification photo in (b) indicates that regions of severe surface damage in the CVD SiC can be observed where the fracture path was presumably located at the joint/SiC interface. In addition, although it is not shown here, the joint material was observed to be denser on the fracture surface of 1-3 as compared to 6-1.

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