2.2 Low Activation Joining of SiC/SiC Composites for Fusion Applications — C. H. Henager, Jr., R. J. Kurtz (Pacific Northwest National Laboratory[1], 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 tested at Politecnico di Torino.

SUMMARY

The use of SiC composites in fusion environments likely requires joining of plates using reactive joining or brazing. One promising reactive joining method uses solid-state displacement reactions between Si and TiC to produce Ti_3SiC_2 + SiC. We continue to explore the processing envelope for this joint for the TITAN collaboration in order to produce optimal joints to undergo irradiation studies in HFIR. The TITAN collaboration has designed miniature torsion joints for preparation, testing, and irradiation in HFIR. PNNL synthesized 40 miniature torsion joints and several were tested for shear strength prior to irradiation testing in HFIR. The resulting tests indicated that 1) joint fixture alignment problems cause joint strengths to be lower than optimal, 2) that non-planar torsion test failures may limit the effectiveness of the miniature specimen design, and 3) that several joints that were well aligned had high shear strengths and promising mechanical properties. In summary, it appears that high joint strengths cause non-planar shear fracture and complicate strength analysis for miniature torsion specimens.

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].

[1] PNNL is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

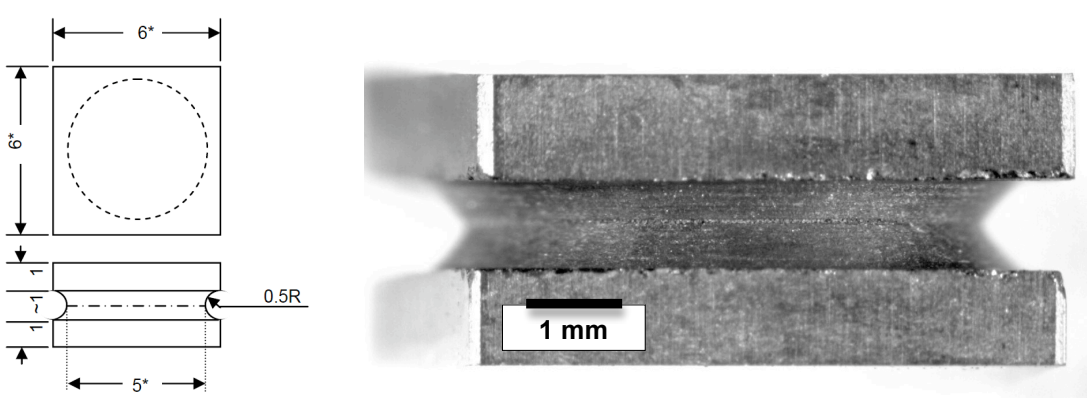
EXPERIMENTAL PROCEDURES

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. Two batches of specimens were prepared for this study, with the first batch using torsion samples with different dimensions as indicated in Figure 1. In both cases, the green joints were placed within a graphite 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.

The joints were sent to Prof. M. Ferraris of Politecnico di Torino, Torino, Italy for testing as part of the TITAN collaboration. The joints are tested in torsion using a test fixture as shown in Figure 3. The hourglass specimens are held in place using the square top and bottom specimen shapes with the joint joining the two half specimens as shown in Figure 1 along the circular center region. The test places the joint in pure shear and presumably tests the shear strength more accurately than other tests, such as notched shear tests with problematic bending moments and stress concentrations.

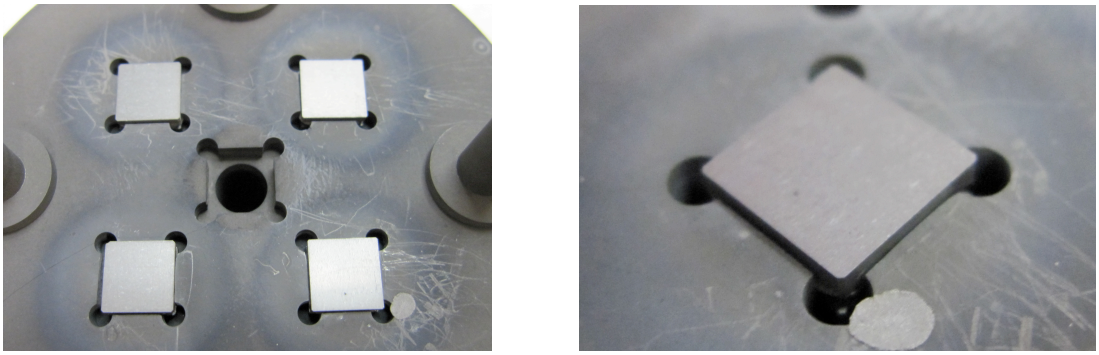
RESULTS

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.



(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.

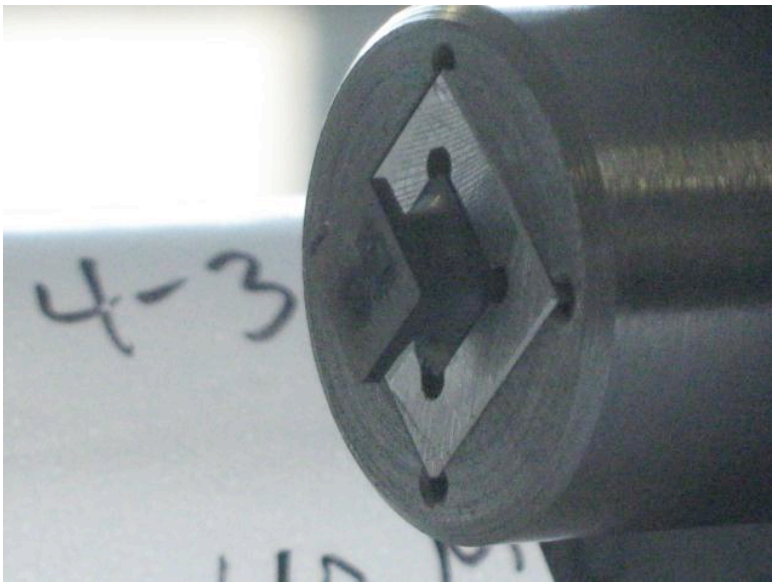


Figure 3. PNNL joint specimen 4-3 in the torsion test holder at Politecnico di Torino prior to testing. This particular specimen was one of the first batch that was taller, thus, it was almost 4-mm in height compared to 3-mm height shown in Figure 1.

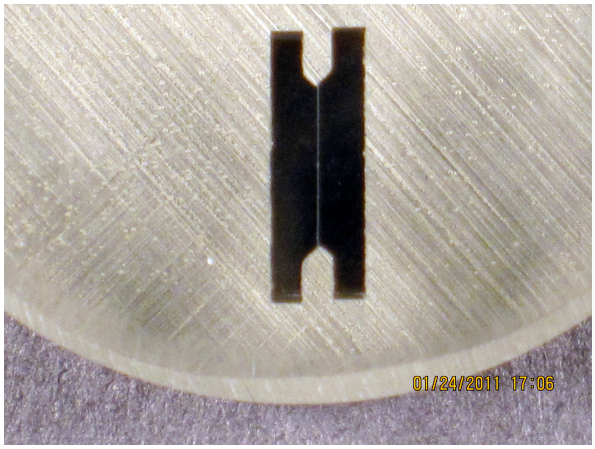


Figure 4. Sectioned 3-mm high joint mounted in epoxy for joint photomicrography and for checking joint alignment. The joint specimen can be seen to be slightly misaligned in this photo.

Several joints of both the 4-mm high and 3-mm high specimens were tested in Torino and the results of the 4-mm high specimen tests are shown in Figure 5. Two trends were observed after this more complete round of testing. One trend is that proper joint alignment and pressure application is required for this reaction joining process to obtain high strengths in the torsion measurement. The second is that most of the joined specimens fractured outside of the plane of the joint and, thus, do not give an accurate shear strength measurement. One joint that fractured mainly within the plane of the joint was specimen 1-3, which had the highest measured joint strength (See Figure 6). Other weaker joints were apparently measured accurately, such as specimen 6-1, which was measured at 3 MPa shear strength since it was not processed completely as shown in Figure 7. The remainder of the specimens fractured outside of the plane of the joint, either partly or completely, several of which are shown in Figure 8. These cannot be relied upon to give reliable data using this test specimen geometry. However, it may be that these joints exceed the shear strength of CVD SiC and planar joint failure cannot be expected to obtain.

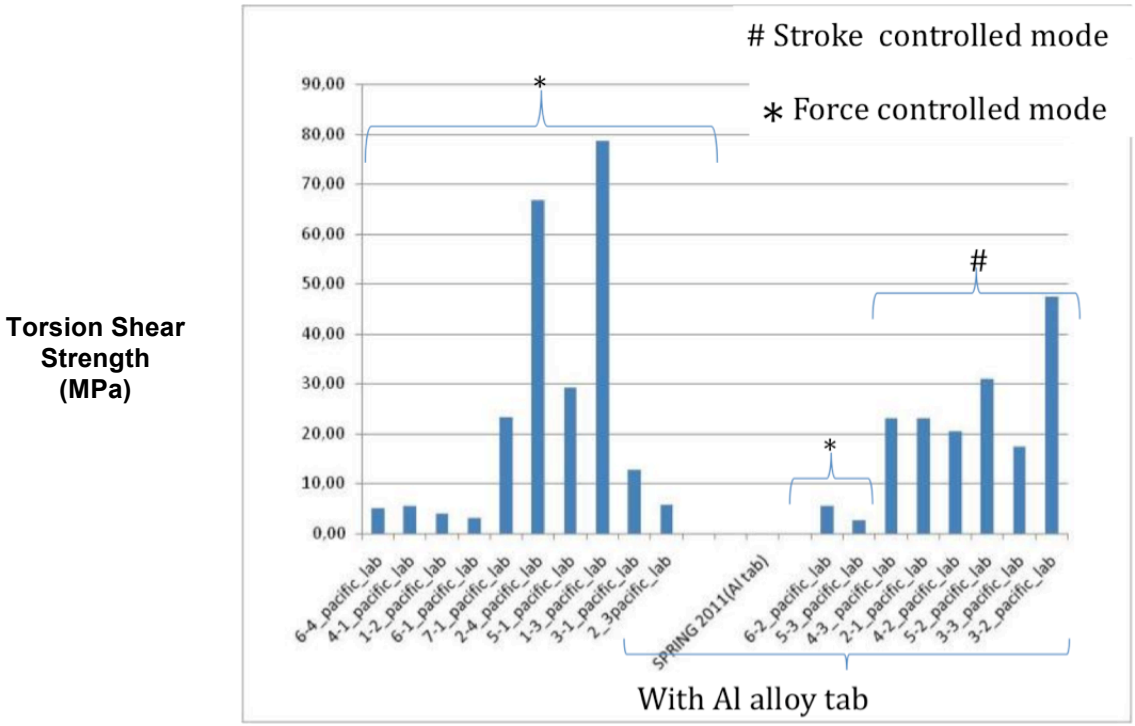
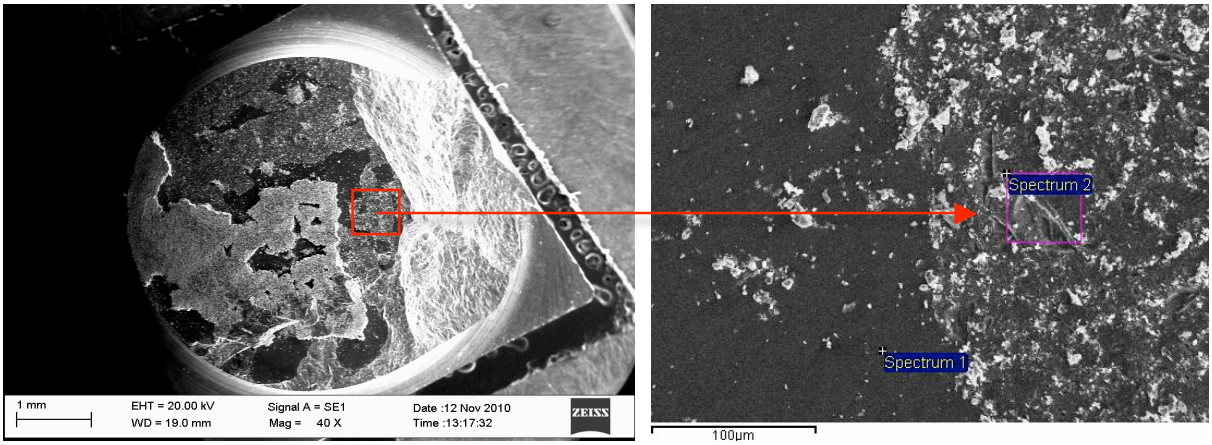


Figure 5. Bar graph of joint strengths measured in MPa of torsion shear strength. The only high-strength specimen that fractured within the joint plane was Specimen 1-3, the one with the highest measured shear strength. Many of the specimens exhibiting low joint strengths were processed incompletely due to fixture alignment problems and did not receive the full applied joining pressure.

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 6. A mostly planar fracture in the joint region is observed that deviates from planarity at some point during fracture. Images at higher magnifications reveal that surface damage to the CVD SiC has occurred due to strong joint bonding. Both joint alignment and proper processing are observed to play a role in joint strength in these preliminary torsion shear tests. However, the lack of planar fractures for many joints that were processed correctly and that presumably had high strengths suggests that the miniature torsion specimen design needs to be modified to account for this. This will be the subject of future research and development.

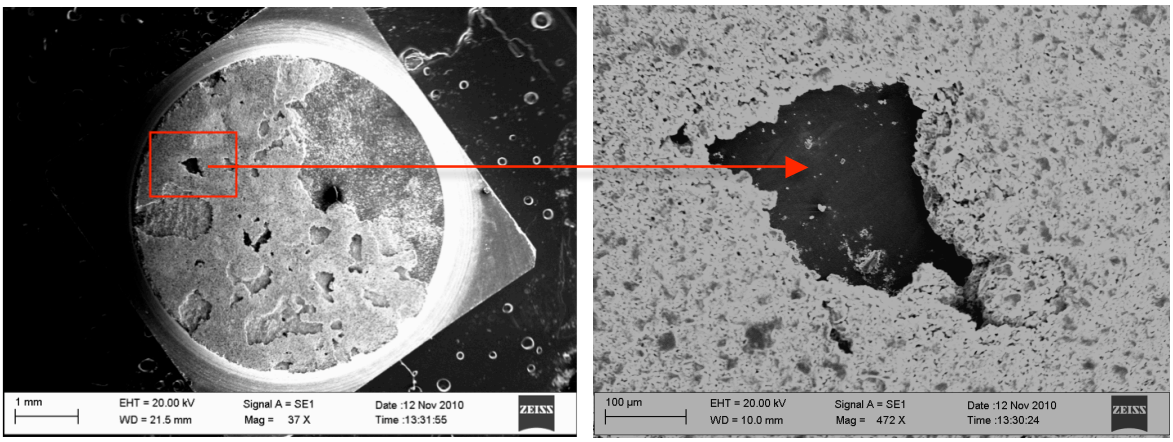
Some of the basis for the miniature torsion specimen design has been the use of epoxy joints between steel and SiC torsion specimens where the joint failure was definitely observed between the joining specimens in the plane of the joint as shown in Figure 9. Torsion strength data was obtained for at least 10 specimens for each of these materials and conditions and the data is shown in Figure 10. It is very consistent strength data and shows the advantages of this method when the joints are of moderate strength. One goal of the future test development will be to maintain the joint design overall so that the existing fixtures can be used but to explore ways to reduce the joint area. One suggestion is the drill a hole in the center of the joined

specimens so that the joined area changes from a circular region to an annulus with reduced joined area.



(a) (b)

Figure 6. SEM micrograph in (a) reveals mostly planar fracture surface for specimen 1-3, which exhibited 80 MPa shear strength. The 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.



(a) (b)

Figure 7. SEM micrograph in (a) reveals a planar fracture surface for specimen 6-1, which had a 3 MPa shear strength. The photo in (b) reveals regions of unbonded SiC and porous joint material, which is evidence of a poorly processed joint due to low joining pressure.

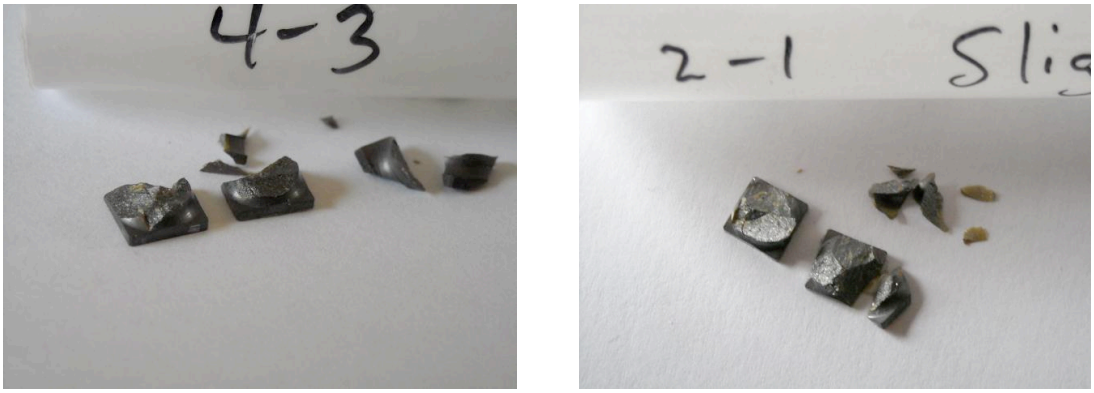


Figure 8. Photographs of fractured specimens exhibiting non-planar fracture from torsion testing. These fractures do not provide accurate shear strength data even though they suggest that the joints are strong.

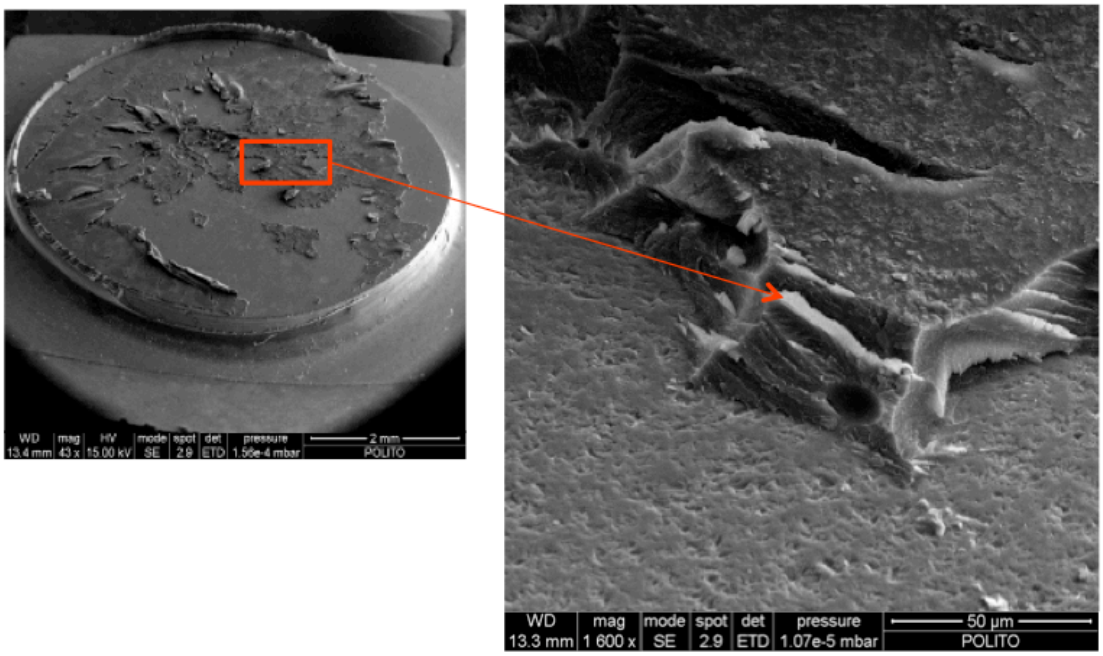


Figure 9. SiC/epoxy joint showing the in-plane fracture path and surface fracture morphology after torsion strength testing at Politecnico di Torino.

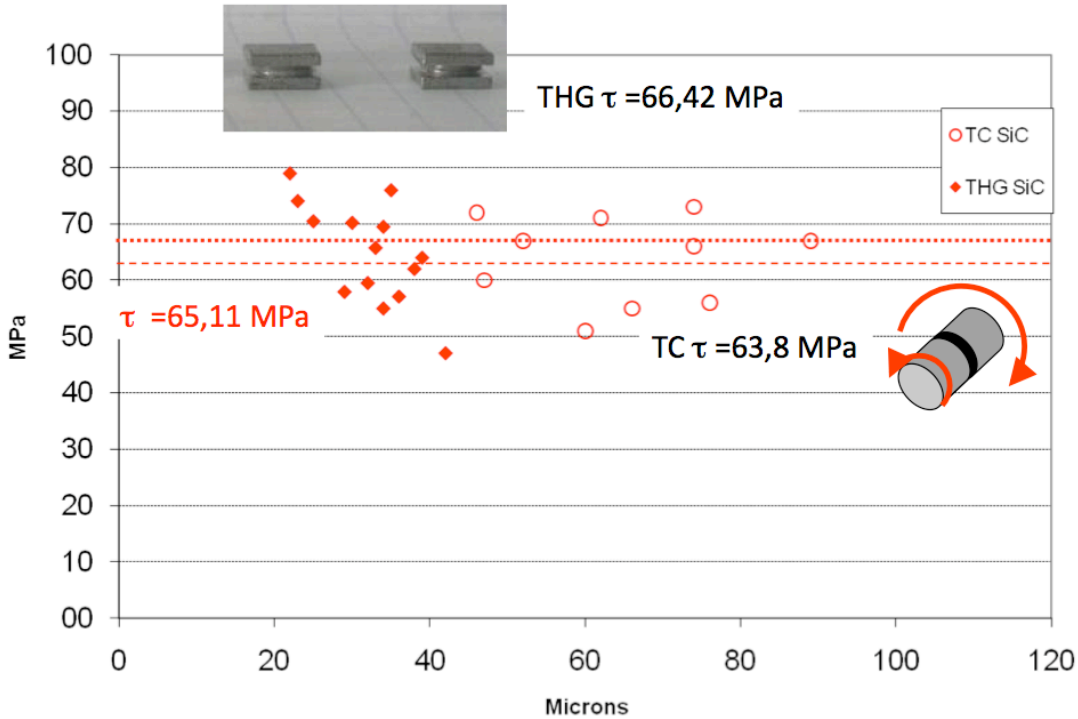


Figure 10. Comparison of two torsion test methods with epoxy joints using torsion strength data obtained at Politecnico di Torino. A torsion cylinder test (TC) is compared to the torsion hourglass (THG) specimen referred to in this report as the miniature torsion test. The results are consistent and comparable.

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