3.1 DEVELOPMENT OF SiC JOINING TECHNOLOGIES FOR FUSION: TEST METHOD DEVELOPMENT - T. Koyanagi, J.O. Kiggans, J.M. Pryor, and Y. Katoh (Oak Ridge National Laboratory)

OBJECTIVE

This report presents a test method study on shear strength for ceramic joints toward development of a new ASTM C28 Test standard for the torsional shear strength of adhesive bonds for advanced ceramics. The effect of specimen dimensions on the apparent torsional shear strength was investigated utilizing brittle ceramic joints.

SUMMARY

Three different sizes of SiC to SiC brittle joint specimens were prepared for torsional shear tests; the machined hourglass test specimens have necks of 5, 10, or 15 mm diameter at the bonding plane. Uniformity of the bond quality was confirmed by acoustic scanning microscope prior to machining. Pure shear failure was observed for all the specimens, meaning that there was no obvious size effect on the fracture appearance. The size effect on the strength was apparently not clear due to the large scatter in strength values. The causes of the deviations and the possible methods to improve the testing are discussed.

PROGRESS AND STATUS

Introduction

The use of a reliable test for evaluation of bonding strength is a key to development of robust joining and integration technologies for ceramics materials. The lack of adequate standardized testing methods to evaluate the mechanical properties of ceramic joints is the motivation for the test method development in this work. Previous studies on evaluation of shear strength of ceramics joints revealed the usefulness of torsional testing to obtain pure shear strength, due to uniform stress state in bonding layer during testing [1]. For this reason, torsional tests were used in a post-irradiation experiment with various SiC joints, which provided understanding of irradiation tolerance of the SiC joints [2]. Because small-scale specimens are used in irradiation stability studies, specimen size effects on mechanical testing is an important consideration. There is limited space in a material test reactor, and the radiation exposure to personnel in post-irradiation testing needs to be minimized, making it a necessity to scale down the size of test specimens.

In this study, size effects on torsional shear strength of SiC to SiC brittle joints was investigated. The finding in this study will contribute to the test standard development for shear strength of ceramic joints in ASTM C28 on Advanced Ceramics.

Experimental Procedure

Plates of chemically vapor-deposited (CVD) SiC (99.9995 % pure, Dow Chemical, Marlborough, MA) were machined into 21 mm (w) × 31 mm (l) × 6 or 3 mm (t) blocks. The joining surface was perpendicular to the CVD growth direction. Commercial bonding slurry (Starfire AD-478, Starfire Systems Inc., Schenectady, NY) was used to fabricate SiC joints. The Starfire material is an adhesive in the form of a slurry. The composition is a proprietary mixture of SiC powder and polycarbosilane based SiC forming polymer (StarPCS^{TM}, Starfire Systems Inc., Schenectady, NY). A thin uniform layer of the adhesive was applied on each bonding surface of CVD SiC plates by spin coating with rotation speed of 4000 rpm, and then the plates were put together. The bonding layer was formed by hot pressing, according to the processing guidelines provided by the vender, except for use of vacuum as the furnace atmosphere.

The bonded plates were inspected by acoustic scanning microscopy with 10 or 15 MHz focused transducer, prior to machining into torsion specimens. The resolution limit of the images was 50 μm.

Figure 2 shows machined hourglass test specimens with a neck of 5, 10, or 15 mm diameter at the bonding plane. The specimen drawings and the IDs are shown in Fig. 1.

Figure 3 shows the setup for the torsion tests. Samples are mounted in a TestResources, Inc 160GT-125Nm torsion system with flexible couplers and sample grips. The flexible couplers were used to keep the alignment during testing. Aluminum tabs were installed at the square grip sections to obtain uniform stress distributions there. The rotation speed was ~0.10 deg/min. Nominal shear strength values (τ) in this work are given by following equation,

$$\tau = 16T/\pi d^3$$

where T is the applied torque and d is the specimen diameter of the neck. Further description of details of the test method can be found elsewhere [3]. All the torsion tests will be conducted at room temperature. The details of the fracture behavior will be investigated using a digital optical microscope (KEYENCE, VHX-1000).

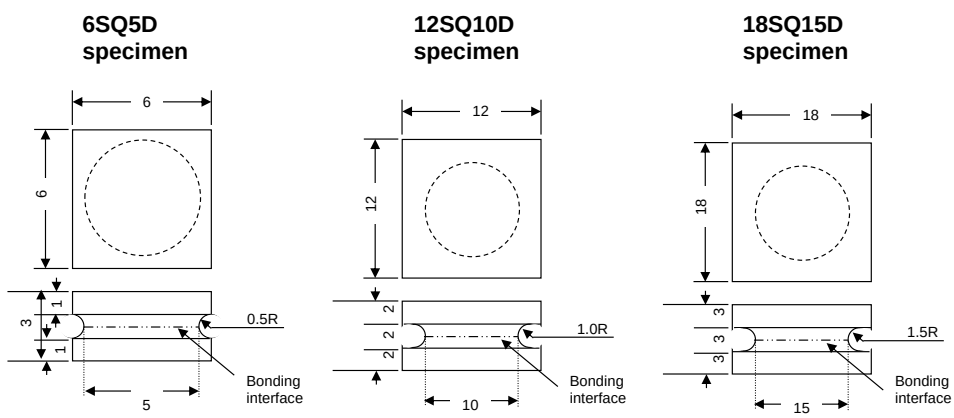

Figure 1. Drawings of three types of torsional shear specimens. Dimensions in mm.

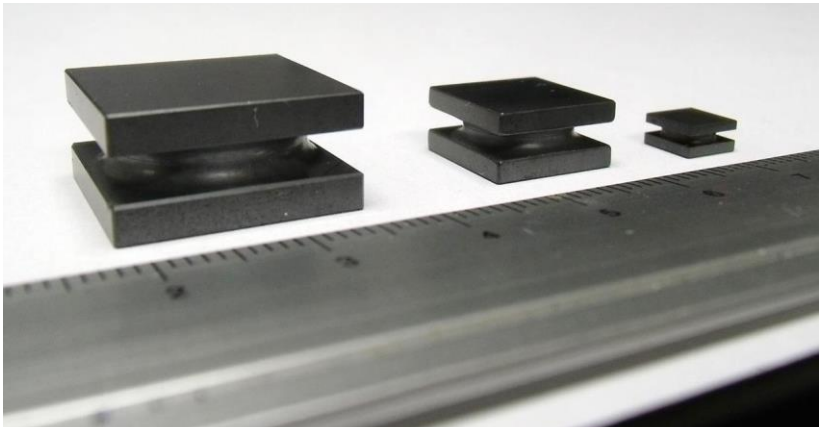

Figure 2. Three types of torsional test specimens. Scale is marked in mm and cm.

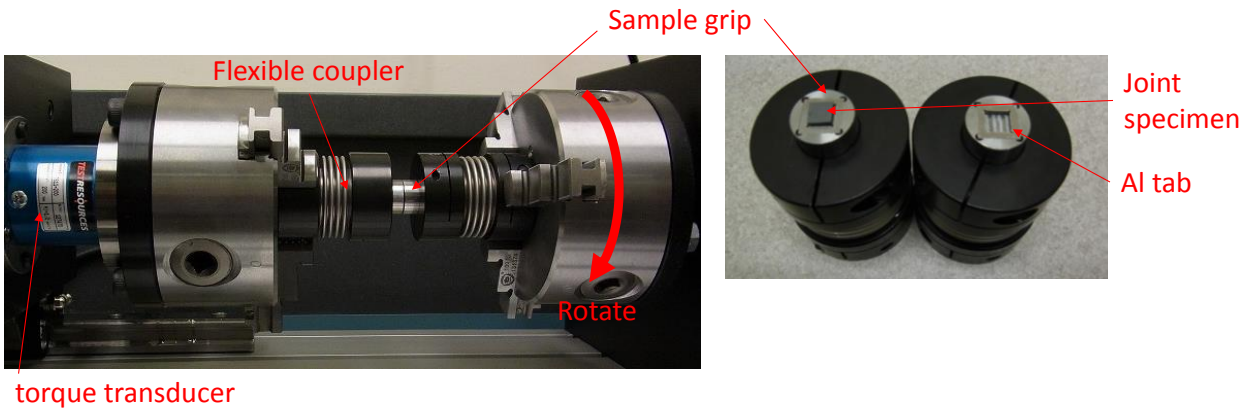

Figure 3. Appearance of torsional test system used to evaluate shear strength of joint specimens.

Results and Discussions

Images of the bonding plane taken using an acoustic scanning microscope are shown in Fig. 4. The image contrast shows amplitude change in the signal's gated area which is used to graphically display differences in the part's structure. Depending on the scan setup and type, this can show signal phase changes (an indicator of extreme acoustic impedance change) and/or reflectors showing the presence of defects such as pores and cracks. The left image shows significant contrast across the area, meaning that the bonding quality is non-uniform. On the other hand, contrast of the right image is uniform, showing absence of significant processing defects or non-uniformity in adhesion at this magnification. Only bonded plates with adequate uniformity confirmed by the ultrasonic imaging were machined into the torsional hourglass specimens used in this study.

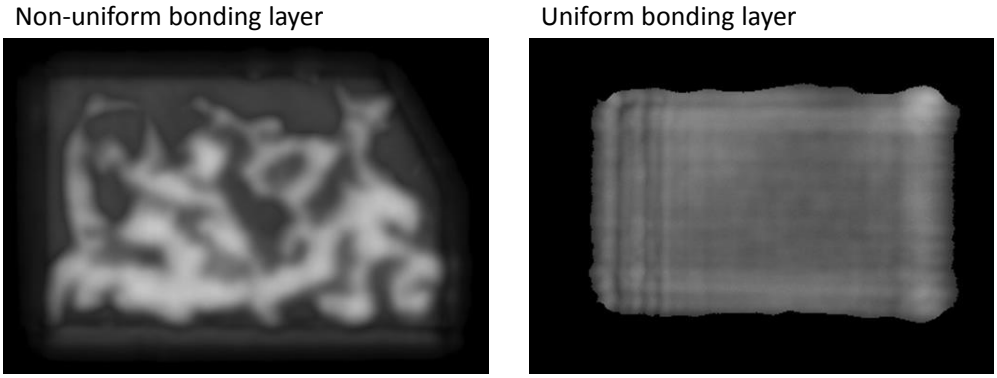

Figure 4. Acoustic images of SiC bonding plates with non-uniform and uniform qualities.
The plate size was ~20 mm × ~30 mm.

Figure 5 shows a typical secondary electron image of the polished cross-section of the joint. The typical thickness of the bonding layer was ~10 μm. No processing defects such as cracks were observed in the CVD SiC substrates. Comparison of polished surface and the surface of the torsional test specimen for this SiC joint is shown in Fig. 6. No obvious pore larger than 100 μm was found in the polished surface. However, large pores were clearly found on the surface of the torsion specimen. The bonding layer and the CVD SiC substrate near the joint interface was locally damaged, likely during machining of the torsion specimen.

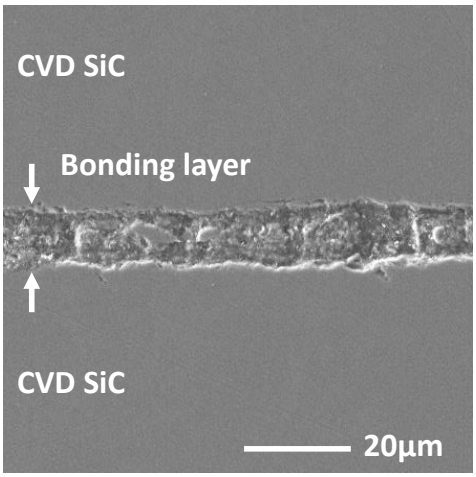

Figure 5. Cross-sectional secondary electron image of the SiC joint.

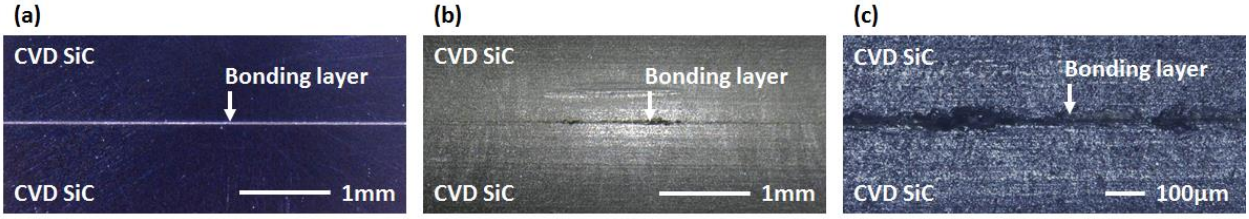

Figure 6. Optical micrographs of the SiC joint: polished surface (a), and surface of the torsion specimen (b) and (c).

Results of torsional shear tests are shown in Fig. 7. The graph shows both average strengths and individual data for each specimen tested. The error bars indicate ± one standard deviation. Similar average shear strengths of ~10 MPa were obtained for the three different size specimens. The scatter in shear strength appeared to be very significant, e.g., more than 30% of the average strength for all cases, making detailed discussion of size effect on the strength difficult. Optical micrographs of torsional tested specimens are shown in Fig. 8. It is important to note that all the tested specimens exhibited shear failure at the bonding plane as shown in the images, showing the lack of specimen size effect on the fracture behavior of the joints.

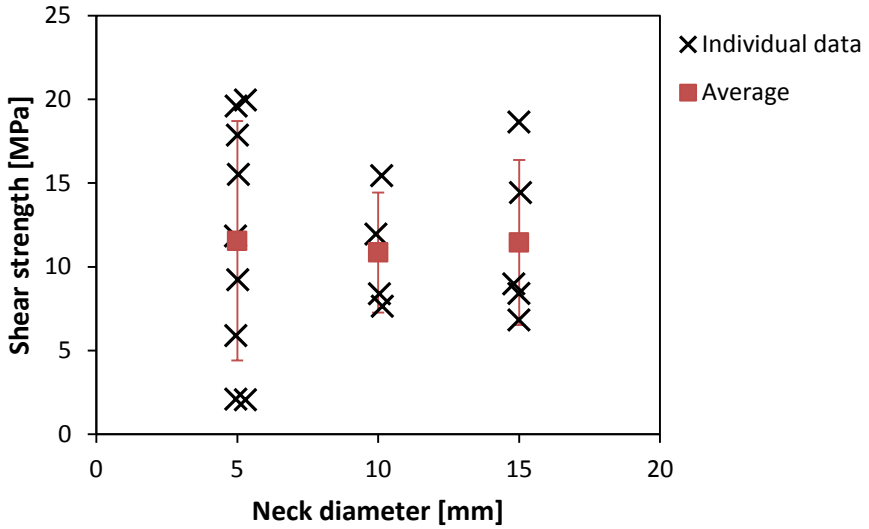

Figure 7. Torsional shear strength of SiC joints with different specimen sizes.

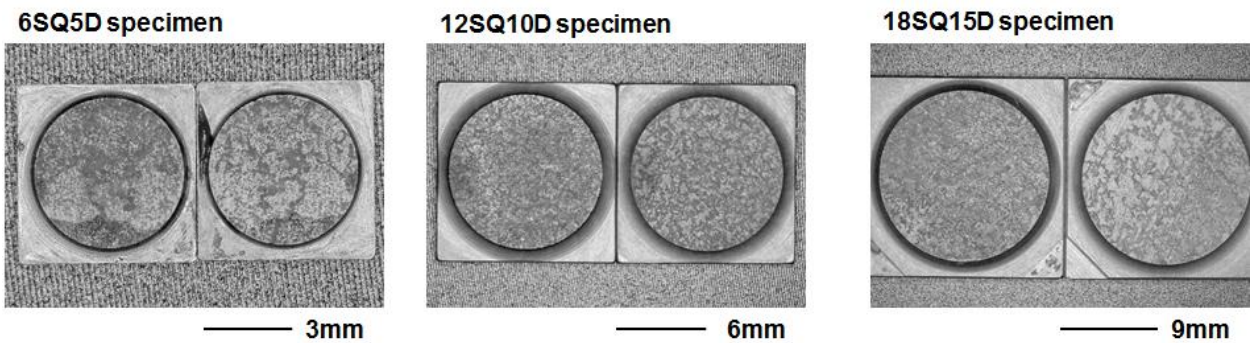

Figure 8. Typical fracture appearances of torsional tested specimens. The specimen IDs are shown in Fig. 1, and scale marks under each photo indicate sizes for the tested specimens.

Again, the large scatter in the torsional shear strength make detailed discussion on the specimen size effect a challenge. Such scatter, particularly for small specimen dimensions, may be at least partially attributed to the damage within bonding layer during machining in the torsion specimen surface, as shown in Fig. 6. The maximum shear stress is applied at the circumference of the bonding layer during the testing in the case that the joint is ideally uniform and the bonding layer has similar mechanical properties to the SiC substrate, according to the stress analysis [3]. Ferraris et al. also reported large data scatter in the torsional tests due to the surface defects [4].

In previous works [1, 2, 4, 5], two types of joint specimens were used. One was the specimen bonded prior to machining (Type A), which is same procedure as used to prepare the specimens in this work. The other was for the specimens bonded following machining (Type B). Ideally, machining defects on the surface of bonding layer can be avoidable by use of the specimen Type B. The one standard deviation for torsional shear strengths for the specimen Type A varied from 2 to 43% of the average strength [2], likely depending on the surface conditions and material uniformity. In the Type B specimens, unexpected up to 28% of large deviations of the strength were also observed [1, 2, 4, 5]. Based on the current knowledge, use of the specimen Type B might not significantly reduce the data scatter in the torsion test. Investigation of the size effect based on fracture statistics is one possible solution.

References

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[5] M. Ferraris, M. Salvo, V. Casalegno, A. Ventrella, Int. J. Appl. Ceram. Technol., 9 [4] (2012) 795–807.