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Modeling and testing miniature torsion specimens for SiC joining development studies for fusion

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ABSTRACT

The international fusion community has designed a miniature torsion specimen for neutron irradiation studies of joined SiC and SiC/SiC composite materials. Miniature torsion joints based on this specimen design were fabricated using displacement reactions between Si and TiC to produce Ti_3SiC_2 + SiC joints with SiC and tested in torsion-shear prior to and after neutron irradiation. However, many miniature torsion specimens fail out-of-plane within the SiC specimen body, which makes it problematic to assign a shear strength value to the joints and makes it difficult to compare unirradiated and irradiated strengths to determine irradiation effects. Finite element elastic damage and elastic–plastic damage models of miniature torsion joints are developed that indicate shear fracture is more likely to occur within the body of the joined sample and cause out-of-plane failures for miniature torsion specimens when a certain modulus and strength ratio between the joint material and the joined material exists. The model results are compared and discussed with regard to unirradiated and irradiated test data for a variety of joint materials. The unirradiated data includes Ti_3SiC_2 + SiC/CVD-SiC joints with tailored joint moduli, and includes steel/epoxy and CVD-SiC/epoxy joints. The implications for joint data based on this sample design are discussed.

1. Introduction

Joining of SiC and SiC-composites has been identified as a critical technology for the use of these materials in either future fusion reactors or in fission power reactors [1–7]. The international fusion materials community is currently irradiating and testing several joint types and compositions in the High Flux Isotope Reactor (HFIR) reactor at Oak Ridge National Laboratory (ORNL) [1]. Pacific Northwest National Laboratory (PNNL) is working with Politecnico di Torino (POLITO) and ORNL using miniature torsion specimens, also referred to as torsion hourglass samples, that have been specifically designed for joint shear strength testing using small irradiation volumes (see Fig. 1) [8]. Ceramic joints and joint shear testing have been studied for many years and several shear joint tests have been designed for ceramics and ceramic composites [4,7,9–13]. These include shear lap tests [14], asymmetric 4-point bend tests [11,15], and double notch shear tests [16,17]. Each of these tests has some disadvantages; including stress concentrations leading to non-uniform shear stresses [17–19] that create large uncertainties in shear strength values. In addition, these test specimens are all quite large, or conversely are not miniature-type tests, whereas irradiation volumes are small and demand miniature specimen designs. The miniature torsion geometry, therefore, was designed to provide a test specimen consistent with small irradiation test volumes associated with in-reactor irradiation testing [3,8,20–24] and to provide a more consistent shear strength test. The experimental data for some joint configurations revealed excellent data reliability and reduced data scatter [21,24] for this specimen design. However, recent high-strength joints fabricated for SiC and SiC-composites have revealed that this test specimen design also has some problems [20,22,23], namely out-of-plane fractures that fail to provide simple shear strength values. Since the miniature torsion test appears to be reliable under certain conditions,

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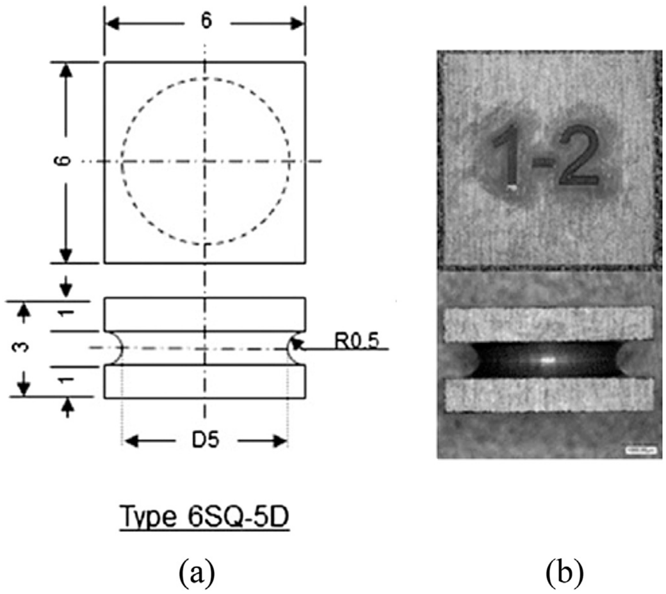

Fig. 1. The miniature torsion specimen geometry used in this study in schematic form in (a) and an optical image in (b) with dimensions in mm from Ref. [1].

but not others, and since this design is ideal for testing many joints in a small irradiation volume, a mechanical model of this joint was created to help understand the observed shear failures under a range of simulated conditions.

The PNNL joints, which are synthesized using displacement reactions between TiC and Si, are observed to fail out-of-plane, or in the base SiC material, during torsion testing [1], similar to what others had observed for high-strength joints [20,22,23]. Most of the types of joints reported in Ref. [1] exhibited out-of-plane or base material failures, including the glass ceramic joints from POLITO. This type of failure within the base material, while encouraging with regard to joint strength since it implies that the joint is as strong or stronger compared to the base material, does not allow accurate comparisons between types of joints, tailored joints, or failed joints. In particular, post-irradiation joint testing reported in Ref. [1] clearly showed some issues related to the shear strength measurement of several different strong joints. Some observations were consistent with irradiation-induced joint damage and, in the case of the PNNL displacement reaction joint and of glass-ceramic (CA) joints, a fracture mode change from base material to in-plane of the joint was observed that could be interpreted with an appropriate model [1].

Therefore, this study was undertaken to determine 1) if some simple modifications to the miniature torsion specimen could be used to address this problem and 2) if a mechanics-based model could better quantify the joint failure response. The first step was to modify the specimen geometry to reduce the joined surface area of the torsion samples in order to understand the effects of fracture initiation on the outer annulus versus an inner annulus and the complex effect of total joint surface area on fracture. The second step was to create a finite element model of the miniature torsion specimen with parameters that could be varied over the wide range of tested materials from POLITO [3,20,21,24]. This study will also use the model results to discuss the recent data set obtained at ORNL using the HFIR test reactor and the pre- and post-irradiation test results from several joints [1].

2. Experimental

2.1. Miniature torsion specimen and modifications

The standard miniature torsion specimen (Fig. 1) was used for the majority of the tests reported here, designated as full-bonded joints and referred to as torsion hourglass samples (THGs). Reduced area annular joints were made by dimpling one of the surfaces with either a 2.3 or 3.1-mm diameter diamond slurry drill and are referred to as reduced-area torsion hourglass samples (RATHGs).[1] These joined samples were fabricated at PNNL (see below) and tested at POLITO. Separately, THG joints were fabricated for HFIR testing and those joints have been irradiated and tested at ORNL and reported in Ref. [1]. No RATHG specimens were prepared for HFIR testing.

2.2. Joint synthesis

2.2.1. Ti_3SiC_2+SiC joints

Strong joints between miniature torsion halves of chemical vapor deposited (CVD) SiC (CVD-SiC) were made using solid-state displacement reaction joining methods discussed previously [2,25]. Fully dense joints are processed in pure argon at 1425 °C (1698 K) for 2 h at either 30 or 40 MPa of applied pressure using tape cast powders of TiC+Si as a bond layer between the CVD-SiC halves. Joints were observed to consist of a dual-phase interpenetrating microstructure with SiC-platelets interpenetrating Ti_3SiC_2 particles with about 40% SiC by area fraction analysis [2]. The joints are strongly bonded at the CVD-SiC/Ti_3SiC_2+SiC interface due to the in-growth of SiC from the CVD-SiC during the displacement reaction processing, which is explained by the CVD-SiC surfaces being favorable nucleation sites for the SiC-phase produced during the displacement reaction. Additionally, joining pressures of 5, 10, and 20 MPa were also used to produce porous joint test samples in comparison to the fully dense joints from the higher synthesis pressures of 30 and 40 MPa. THG joints were made as shown in Fig. 1. A circular dimple of 2.3 or 3.1-mm diameter was placed in one of the joint halves to create the RATHG samples as shown in Fig. 2. These joints, both the reduced joining-pressure joints and the RATHG joints, were created to help understand in more detail test difficulties that were occurring with the miniature torsion joint specimens when joint strength is high [1,20,25].

2.2.2. Adhesive epoxy joints

As discussed in Refs. [21,24] THG joints were made using Araldite AV119 epoxy between CVD-SiC and between a Type 316-grade stainless steel. AV119 was used to bond these materials after acetone and ultrasonic cleaned surfaces were prepared. The epoxy was cured for 1 h at 130 °C (403 K) [24]. The torsion tests for these epoxy-joined THG samples were performed in the same manner as all the other torsion testing. In addition, simple compressions tests were performed [21] on cylinders of cured AV119 epoxy to establish the mechanical properties of this toughened adhesive material [26,27].

2.3. Ti_3SiC_2+SiC joint microstructures and porosity

Representative joints synthesized at each of the five joining pressures were cross-sectioned and examined using optical (OM) and scanning electron microscopy (SEM). Representative SEM images of these joints are shown in Fig. 3 for each of the applied joining pressures. The porosity content as a function of joining

[1] RATHG is the same as torsion-ring hourglass samples (TRHG) in Ref. [21].