#### Journal of Nuclear Materials 500 (2018) 280-294

Contents lists available at ScienceDirect

### Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

# Strength of SiC<sub>f</sub>-SiC<sub>m</sub> composite tube under uniaxial and multiaxial loading

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#### A R T I C L E I N F O

Article history: Received 19 December 2017 Accepted 1 January 2018 Available online 6 January 2018

Keywords: SiC-SiC composite Strength Fuel cladding Accident tolerant fuel

#### ABSTRACT

The authors report mechanical strength of nuclear grade silicon carbide fiber reinforced silicon carbide matrix composite ( $SiC_f-SiC_m$ ) tubing under several different stress states. The composite tubing was fabricated via a Chemical Vapor Infiltration (CVI) process, and is being evaluated for accident tolerant nuclear fuel cladding. Several experimental techniques were applied including uniaxial tension, elastomer insert burst test, open and closed end hydraulic bladder burst test, and torsion test. These tests provided critical stress and strain values at proportional limit and at ultimate failure points. Full field strain measurements using digital image correlation (DIC) were obtained in order to acquire quantitative information on localized deformation during application of stress. Based on the test results, a failure map was constructed for the SiC<sub>f</sub>-SiC<sub>m</sub> composites.

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#### 1. Introduction

Silicon Carbide (SiC) matrix, SiC fiber reinforced composites  $(SiC_f-SiC_m)$  have excellent high temperature strength and stability [1], and are being actively researched and implemented for high temperature load bearing components in many applications [2]. In nuclear power generation industry, replacing metal fuel cladding with SiC\_f-SiC\_m is being actively investigated due to the lack of a melting point (SiC sublimates at 2700 °C) [3], retention of strength at high temperatures, and a much lower reactivity with steam [4]. These attributes can significantly improve the tolerance of light water reactor (LWR) core to accident conditions. Understanding the stress-strain response of the composites is critical as the demonstrated strength and toughness is obtained through a mix of properly designed fiber architecture, carefully engineered fiber-matrix interphase layer, and advanced manufacturing processes that must be optimized based on feedback from mechanical testing.

During application of stress, SiC<sub>f</sub>-SiC<sub>m</sub> goes through several stages of deformation characterized by complex damage initiation and progression processes. At low loading, the material behaves linear-elastically without significant damage occurring in either the

[5]. In the last two decades a great amount of research effort was directed towards understanding the stress-strain response of nuclear grade  $SiC_f$ - $SiC_m$  composite tubes [6–11]. Nozawa et al. tested unidirectional composites produced by NITE method [12] and plain weave composites produce by CVI and NITE methods in tension, compression and shear with ranging fiber angles [6]. The PLS and UTS were reported. The results showed that at the same fiber angles CVI composites have tensile and shear strengths that are

matrix, interphase, or fiber. Once the stress reaches the matrix cracking stress, stress-strain curve starts to deviate from the linear

relationship; this threshold stress level is typically called Propor-

tional Limit Stress (PLS). At this point, the material starts to accu-

mulate irreversible damage in the form of matrix cracking. These

cracks typically propagate, especially as load increases, until they

are deflected at fiber/matrix interphase. This leads to the partial

debonding of the fiber from the matrix and load transfers onto the

fiber, which has a very large tensile strength relative to the matrix.

Further loading increases the matrix crack density, causing addi-

tional fiber-matrix debonding, and localized fiber breakage. Even-

tually, localized fiber breaks accumulate at a macro scale and the

Ultimate Tensile Strength (UTS) of the material is reached. A more

detailed discussion of stress-strain curve and failure behavior of

ceramic composites were given by Vagaggini, Domergue and Evans







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significantly greater than the other two composites fabricated with NITE process. Nozawa reported that the composite strength could be fitted with the Tsai-Wu failure criterion [6]. Rohmer et al. performed axial and hoop loading tests at ambient temperatures on braided SiCf-SiCm cladding tubes produced by CVI method [9]. Mechanical properties obtained by Rohmer were significantly lower than the values reported by Nozawa, however, the fiber type and sample geometries were different in these works. A 3D simulation model for an axial test of the composite had revealed stress concentration points where the fiber tows overlap each other. While the composites were braided and do not have a clear laminated structure, the tow-to-tow delamination can still happen. Rohmer also suggested that because of the stress concentration produced by the contacting tows of fiber, those locations could be the initiation sites for the delamination cracks. Jacobsen et al. applied C-ring test on nuclear grade claddings [8]. By cutting a slot out from a ring and compressing the resulting C shaped samples, material mechanical properties in the hoop direction were measured and compared to results obtained from the expending plug method. While these studies have provided key insight into the stress-strain response of SiC<sub>f</sub>-SiC<sub>m</sub> tubes, they do not represent a systematic investigation of SiCf-SiCm behavior under uniaxial and multi-axial stress.

This paper reports a comprehensive study of the strength of a nuclear-grade  $SiC_f-SiC_m$  composite tubing. Several techniques were applied to test the strength of the composite tube including: uni-axial tension, burst tests with elastomer insert, open-end burst, closed-end burst tests, and torsion tests. During these tests, load and strains were measured in order to construct a stress-strain curve. Full field strain maps were gathered using digital image correlation (DIC). Critical stress and strain values at PLS, and at UTS, were recorded. These test data provided strength of  $SiC_f-SiC_m$  materials under uniaxial and multi-axial stress state. A failure map was constructed based on the test results.

#### 2. Materials and methods

The nuclear grade SiCf-SiCm composite tubing was fabricated at General Atomics (GA) from stoichiometric grade Hi-Nicalon Type S fiber using a chemical vapor infiltration (CVI) method. The details of the processing route was reported in Deck et al. [13]. A mandrel was used to define the inner diameter of the composite and fiber preform was constructed over the mandrel. Assume the reinforcing contribution of each fiber tow to a given direction of interest is defined as the cosine of the angle between the tow and the direction of interest, then the equivalent fiber tow counts in a given direction can be obtained by summing the contribution of all fiber tows. The ratio of equivalent fiber tow in the hoop to axial direction was 1:1.4, meaning an axial biased fiber architecture. Tubular fiber preforms were then coated with a thin, <250 nm pyrolytic carbon layer via chemical vapor deposition. The stoichiometric,  $\beta$ -SiC matrix was deposited via decomposition of methyltrichlorsilane under chemical vapor infiltration conditions. A total of fifteen 250 mm long specimens were fabricated within the batch and the batch was fabricated under a shared pressure, gas flow, and temperature regime which has previously been demonstrated to give uniform composite material [14]. Composites were infiltrated to 90% of theoretical density with a fiber volume fraction of 36%, in both cases this was found to be consistent within  $\pm 1\%$  across the batch of tubes. Nominal final specimen dimensions are: an inner diameter (ID) of 8.0 mm and an outer diameter (OD) of 10.2 mm as measured with calipers. Over a full length tube, nominal cylindricity was 0.73 mm at the ID and 0.77 mm at the OD, coaxiality was 0.04 mm, and straightness was 0.54 mm as measured using a Nikon XT H 225 X-ray computed tomography system (XCT) coupled with VGStudio Max analysis software; further details of how these XCT based measurements are performed can be found in Deck et al. [15].

For obtaining the axial strength of composites, an ASTM standard for this procedure has been established which dictates test setup and methodology [16]. Significant challenges exist in the gripping of these specimens in such a way that maintains alignment and ensures failure in the gauge region. Oak Ridge National Laboratory tested a series of tubes featuring tapered shoulders and the use of matching split copper collets with tapered cone to hold the specimen and maintain alignment [17]. During this testing, significant failure (~50%) occurred at/near the grips. Measured ultimate tensile strength of the composites was  $107 \pm 8$  MPa. GA has adopted an alternative gripping mechanism that uses epoxy and straight walled tubing. While failures can occur at/near the grips this gripping method results in 80-90% valid tests. This adhesive method was used for axial testing in the reported work.

An internal pressure burst test is a common test method for tubular structures that need to sustain high internal pressures during operations, such as nuclear fuel cladding. Various test methods had been developed to pressure load tubular specimens. Mosley et al. [18] used an elastomer insert method, also known as a polymer plug test, where a cylindrical elastomer is axially compressed inside a sample. An axial compression causes the elastomer insert to expand in the radial direction, therefore inducing pressure on the inner walls of a sample. It was noticed that the elastomer insert behaves like a hydraulic fluid up to 5% of axial compression. Through an analytical solution, stress profiles on the inside and the outside walls were derived. These stress profiles were compared to experimental results of mild steel tubes and matched within 5%. The method is relatively simple to implement on traditional mechanical testing machines, requires little time to set up and does not require any mechanical bonding to the test samples. These advantages make it a popular choice for an internal pressure test and it has evolved into an ASTM C1819-15 [19] standard for testing of ceramic composites. Ross [20] had compared experimental results from monolithic and composite ceramic tubes using the polymer insert method. However, buckling of the elastomer plug can occur when the ratio of plug length over plug diameter gets large. Due to its practicality, the elastomeric insert method is used herein as one of the methods for measuring the hoop strength of the composite tubing.

Another way of generating internal pressure is by pressurizing liquid inside of a tubular sample. This method often times requires some sort of seal between an experimental fixture and a test specimen. In works reported by Cain [21], Cohen [22], and Verrilli [23], similar fixtures were developed to test composite samples using pressurized oil. Two parallel plates were spaced just far enough for a test sample to fit in between. A soft material was placed on the inside wall of the sample to act as an oil seal. When the oil is pressurized, the seal is pressed against a specimen inducing stresses in the radial direction. A small amount of pressure is lost due to seal compression. All authors reported that their methods produced a uniform pressure on samples' walls. Verrilli [23] and Chuck [24] also extended the method to high temperature burst test. Verrrilli used a cooled contact block in between the hot sample and the hydraulic oil and Chuck used a pressurizing media that can be used at high temperature. In this work, a novel rubber bladder burst test method was implemented. A soft rubber tubing (bladder) was inserted into the tubular sample. Pressurized hydraulic oil was pumped into the rubber tubing, the expanding rubber tubing exerted a uniform controllable internal pressure on the sample tube. During pressurization, the hydraulic oil is contained within the rubber bladder without directly contacting the sample tube. The method can be applied to both dense and porous tubes. The bladder method eliminates the buckling issue associated

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