TEST METHODS FOR HOOP TENSILE STRENGTH OF CERAMIC COMPOSITE TUBES FOR LIGHT WATER NUCLEAR REACTOR APPLICATIONS

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ABSTRACT
The US DOE plans to replace conventional zirconium-alloy fuel rod tubes in light water reactors (LWR) with those consisting of ceramic matrix composites (CMC) thereby enhancing fuel performance and accident tolerance of LWRs. Silicon carbide fiber-reinforced silicon carbide-matrix (SiC/SiC) composites demonstrate tolerance to the irradiation and chemical environments of LWRs. Loss of gas tightness and mechanical integrity due to the build-up of internal gas pressure and the swelling of fuel pellets are among the anticipated failure modes for the LWR fuel cladding. Therefore, rigorous determination of the hoop tensile (or equivalent) strength properties is critically important for evaluation of SiC/SiC CMC fuel claddings. Because there are no commonly-accepted design methodologies for advanced composite tubular components, there are limited mechanical test standards for any properties of tubular ceramic composite components. Therefore, some current and proposed test methods for measuring tensile hoop strength of composite tubes are presented, discussed, and compared for application to CMCs. Proposed standard test methods are presented in terms of the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, and precision/bias.

KEYWORDS – hoop tensile strength, tubes, ceramic matrix composite, silicon carbide composite, nuclear fission.

INTRODUCTION
The US Department of Energy (US DOE) is currently exploring replacing conventional zirconium-alloy fuel rod tubes in light water reactors (LWR) with fuel rods consisting partly or entirely of ceramic matrix composites (CMC) thereby benefiting LWRs by enhancing fuel performance and accident tolerance. The specific CMC of interest for this application is silicon carbide continuous fiber-reinforced silicon carbide-matrix (SiC/SiC) composite because of the demonstrated tolerance of SiC/SiC CMC for the irradiation and chemical environment of LWRs. In particular, high strength at high temperatures and low chemical activity, including no exothermic reaction with water as zirconium demonstrates at elevated temperatures, were the primary reasons to select SiC/SiC CMCs for further LWR development. Additionally, the high temperature properties of SiC/SiC CMCs imply that the fuel system can retain its geometry and fuel protective function even during an accident. Removal of the exothermic zirconium and water reaction also increases the temperature at which the fuel can operate. Eliminating the generation of free hydrogen would also lower the type of risks created during an accident scenario.1,2

However, loss of gas tightness and mechanical integrity due to the build-up of internal gas pressure and the swelling of fuel pellets are among the anticipated failure modes for the LWR fuel cladding. Therefore, rigorous determination of the hoop tensile (or equivalent) strength properties is critically important upon evaluation of the SiC/SiC CMC fuel cladding. These CMCs consist of high-strength silicon carbide fibers in a high-temperature silicon carbide matrix. Such a composite structure provides high strength and high fracture resistance at elevated temperatures, in addition to their potentially higher resistance to neutron radiation than conventional material.
The ceramic reinforcement tows have high filament counts (500-2000) and are woven with large units cells, several millimeters in size. In a tube configuration, the composites can have a 1-D filament wound, 2-D laminate, or 3-D (weave or braid) construction depending on what tensile, shear, and hoop stresses are considered. The fiber architecture in the tubes can be geometrically tailored for highly anisotropic or uniform isotropic mechanical and thermal properties.

Tubular geometries for nuclear applications present challenges for both “makers” and “lookers” of SiC/SiC CMCs. For “makers”: how to make seamless tubes with multiple direction architectures; how to ensure integrity in the radial direction; and how to create uniform wall thickness and uniform/nonporous matrices. For “lookers”: how to build on decades of experience with consensus standards and databases for “flat” material forms; how to interpret information of tests of test specimen in component form; and how to adapt expertise at room temperature in ambient environments to conditions at high temperature in specific extreme-use environments.

However, not only are there no commonly-accepted design methodologies for tubular components comprised of advanced composites, at this time there are almost no mechanical test standards for any of these properties of tubular geometry ceramic composite components. In particular, for CMC tubes there is one standard for axial tensile strength that was currently being approved and published by ASTM as C1624-12 “Standard Test Method for Monotonic Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramic Tubular Test Specimens at Ambient Temperature.”

Use of new CMC materials in LWR applications requires mechanical test standards to support not only material development and property databases, but design codes and component specification documents, as well as Nuclear Regulatory Commission (NRC) regulations on nuclear design approval, certification, and licensing. In particular, the mechanical test standards for nuclear grade CMCs are necessary to provide for accurate and reliable data, based on well-defined test methods, detailed specimen preparation, comprehensive reporting requirements, and commonly-accepted terminology. The development and component design process using CMCs in LWR applications will be hampered and delayed, if appropriate CMC mechanical test standards are not available in a timely manner.
Fortunately the LWR applications of SiC/SiC CMCs builds on experience allowing nuclear applications to advance an existing mature specialized technology. For example, SiC/SiC CMC materials and structure technology were funded by the aerospace and defense industries/agencies. In addition, current evaluations and applications of SiC/SiC CMCs in fusion reactors (first wall) and tristructural-isotropic (TRISO) fuel forms that have established properties under extended neutron irradiation and at high temperatures as well as very hot steam environment. Growing, credible data bases for SiC/SiC CMCs now exist because of the evolution of consensus test methods and design codes. Finally, maturation of volume-scale manufacturing capability for all types of CMCs including SiC/SiC CMC adds to availability and understanding of these material.

Such professional organizations as ASME and ASTM have taken the lead in developing the codes, specifications, and test standards for CMCs in nuclear applications. ASTM Committee C28 on Advanced Ceramics has a particular focus on mechanical test standards for CMCs. Specifically, ASTM Subcommittee C28.07 has published eleven standards for CMCs (e.g., tensile, flexure, shear, compression, creep, fatigue, etc).

Mechanical testing of composite tube geometries is distinctly different from testing flat plates because of the differences in fiber architecture (weaves, braids, filament wound), stress conditions (hoop, torsion, and flexure stresses), gripping, bending stresses, gage section definition, and scaling issues.\(^5\) Because there are no commonly-accepted design methodologies for advanced composite tubular components, there are almost no mechanical test standards for any properties of tubular ceramic composite components.

Therefore, in this paper, some current and proposed test methods for measuring tensile hoop strength of composite tubes are presented, discussed, and compared for application to CMCs. Two proposed test methods are presented in terms of the following experimental issues -- test specimen geometries/preparation, test fixtures, test equipment, interferences, testing modes/procedures, data collection, calculations, reporting requirements, and precision/bias.

**HOOP TENSILE STRENGTH TEST OF TUBES**

A review of the literature for experimental and analytical methods applied to assessing behavior of tubes subjected to hoop tensile stress resulted in the following categories.

1) Mechanical loading methods applied to short sections of tubes
2) Viscoelastic loading methods applied to short and/or long sections of tubes
3) Pressure loading methods applied to long sections of tubes

Aspects of each category are discussed and illustrated in the following sub sections.

**Mechanical loading methods applied to short sections of tubes -** Longitudinally “short” sections of tubes are loaded transversely through split disk loading fixtures as illustrated in Fig. 2. This method has been standardized in ASTM D2290\(^6\) and previous work has shown that this developed test, compared to the quick burst test, induces a hoop (circumferential) stress, which is similar to the stress induced by internal pressure\(^7\). However, failures from these types of tests tend to initiate from the inner radius and edges of the short sections that may not be representative of failures of actual long tubes. Some pros and cons for this type of test are listed in Table 1.
Table 1 Some Pros and Cons for Mechanical Loading Methods Applied to Short Sections of Tubes

<table>
<thead>
<tr>
<th>Pros</th>
<th>Cons</th>
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<tbody>
<tr>
<td>- Simple fixtures</td>
<td>- Samples only part of tube</td>
</tr>
<tr>
<td>- Uses small sections of tube</td>
<td>- Edge effects</td>
</tr>
<tr>
<td>- Uses existing test machines</td>
<td>- Does not represent internal pressure loading</td>
</tr>
<tr>
<td>- Simple equations</td>
<td>- Limited to proof testing</td>
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Viscoelastic loading methods applied to short and/or long sections of tubes - This is a simple method to generate high-pressure conditions for pressure testing small-diameter tubes using the radial expansion of an axially-compressed viscoelastic insert. At room temperature, a piston is used to compress an elastomeric cylinder or plug inside a tube of material. The resulting Poisson’s expansion generates radial pressure along the inner wall of the tube sample. One of the attractive attributes of this test is that once the sample fails, the elastomer easily compresses and quickly lowers the stress and pressure in the system\textsuperscript{8-10}. Additionally, there are no high-pressure gases or fluids to contain. Also, the use of a solid material to generate the internal pressure removes the need for high-pressure seals. This method has been extended to high temperatures by using a glass insert material that behaves viscoelastically above its glass transition temperature\textsuperscript{10}. Some pros and cons for this type of test are listed in Table 2.

Figure 2 Illustration of split disk mechanical loading method applied to short section tube (from Ref 7)

Figure 3 Illustration of elastomeric insert applied (viscoelastic loading) applied to short section of tube (from Ref 8)
Table 2  Some Pros and Cons for Viscoelastic Loading Methods Applied to Short and/or Long Sections of Tubes

<table>
<thead>
<tr>
<th>Pros</th>
<th>Cons</th>
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<tbody>
<tr>
<td>- Simple fixtures</td>
<td>- May be complex stress states</td>
</tr>
<tr>
<td>- Uses short or long sections of tube</td>
<td>- Friction effects for rough surfaces</td>
</tr>
<tr>
<td>- Uses existing test machines</td>
<td>- May be force/pressure limited</td>
</tr>
<tr>
<td>- Has been extended to high temp</td>
<td>- May be limited to material selection</td>
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Pressure loading methods applied to short or long sections of tubes - This is a conceptually simple but experimentally-challenging method for pressure testing tubes using internal pressurization. Pressure can be applied using either gas or liquid. In addition, the pressurized medium can be applied directly to the tube or through an internal bladder. Finally, end caps can be attached to the side walls of the tube or to each other. Experimental conditions that have produced consistent hoop stresses in composite tubes include the following: pressurized liquid to minimize explosive failures upon rupture; internal bladders to prevent leakage through the composite tube walls; and end caps attached to each other and not to the tube walls to eliminate axial stresses. ASTM D1599 uses this method for "plastic" pipes. Using tubes with proper length to diameter ratios, end effects are eliminated and the hoop tensile stresses and strains are uniform in the gage section, thereby resulting in measure of the material properties of the tubular material during testing. With proper configuration, these test methods can be extended to elevated temperature. Some pros and cons for this type of test are listed in Table 3.

Various studies of homogenous, isotropic, linear elastic materials such as low carbon steel test materials using methods from each of the three techniques have indicated that the elastic response and the strength behavior determined from hoop stresses and strains are comparable regardless of test method. However, the complexity of the non homogeneous, anisotropic, nonlinear elastic behaviour of fibre-reinforced composites has led to differences in results from the different methods. It should be noted that the potential biaxiality and even triaxiality of the stress states may limit interpretation of these tests methods for "pure" hoop behavior. Therefore, the most appropriate test methods for ceramic composites at room temperature are those that result in hoop stresses that arise from internal pressurization. The evolving standardized test method for hoop tensile strength of ceramic composites described in the following sections reflects this conclusion.
Table 3 Some Pros and Cons for Pressure Loading Methods Applied to Short or Long Sections of Tubes

<table>
<thead>
<tr>
<th>Pros</th>
<th>Cons</th>
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<tbody>
<tr>
<td>- Real internal pressure loading</td>
<td>- May require internal bladders for non impervious materials</td>
</tr>
<tr>
<td>- Uses short or long sections of tube</td>
<td>- Stress state may be biaxial</td>
</tr>
<tr>
<td>- Simple equations</td>
<td>- May require special equipment</td>
</tr>
<tr>
<td>- Related directly to applications</td>
<td>- May be high-temp problematic</td>
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**SCOPE AND APPLICATION**

A working group of ASTM Subcommittee C28.07 on CMC tube testing is developing two draft test methods: 1) “Hoop Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramic Composite Tubular Test Specimens at Ambient Temperature” and 2) “Hoop Tensile Strength of Continuous Fiber-Reinforced Advanced Ceramic Composite Tubular Test Specimens at Ambient Temperature Using Elastomeric Inserts.”

The first proposed test method has wide applicability with the test results intended for design data generation as well as model verification. The second proposed test method has limited applicability with the test results intended for material down selection / screening.

In the two test methods, a ceramic composite tube/cylinder or tube/cylinder section with a defined gage section and a known wall thickness is selected to be the test specimen. The test specimen is inserted into the appropriate test fixture assembly is subject to one of the following monotonic loadings depending on the test method:

1) Direct internal hydrostatic pressure produced from hydraulic fluid
or
2) Indirect pressure produced by axial loading of an elastomeric insert

Either pressure or axial load is recorded along with hoop displacement/strain in the gage section. Results include hoop tensile stress/strain, ultimate hoop tensile strength, fracture hoop tensile strength and proportional limit hoop tensile stress along with corresponding strain, and elastic constants. The test method addresses test equipment, interferences, gripping and coupling methods, testing modes and procedures, tubular test specimen geometries, test specimen preparation and conditioning, data collection, calculations, reporting requirements and precision/bias. The methods are applicable to a wide range of CMC tubes with 1-D filament, 2-D laminate, and 3-D weave and braid architectures. In addition, the test methods reference test procedures and fixturing from research work done on CMC tubes as well as test procedures and research on PMC tubes.

**EXPERIMENTAL FACTORS**

CMCs generally exhibit “graceful” failure from a cumulative damage process, unlike monolithic advanced ceramics that fracture catastrophically from a single dominant flaw. The testing of CMC (both flats and tubes) has a range of different material and experimental factors that interact and must be controlled and managed (See Fig. 5). These factors must be managed and understood to produce consistent, representative failures in the gage section of test specimens. Tubular test specimens with cylindrical geometries provide particular challenges in the areas of gage section geometry, loading and bonding failures, extraneous “parasitic” stresses (including biaxial and triaxial stresses), and out-of-gage failures.
TEST SPECIMEN GEOMETRIES

Test Specimen Size -- CMC tubes are fabricated in a wide range of geometries and sizes, across a spectrum of fiber-matrix-architecture combinations. It is not practical to define a single test specimen geometry that is universally applicable. The selection and definition of a test specimen geometry depends on the purpose of the testing effort. With that consideration, the test method is generally applicable to tubes with outer diameters \((D_o)\) of 10 to 150 mm and wall thicknesses \((t)\) of 1 to 25 mm, where the ratio of the outer diameter to wall thickness is commonly \(D_o/t = 5\) to 30. Tube sections may vary depending on the type of test (e.g., 25 mm to 1000 mm). In many cases, the wall thickness is defined by the number of plies and fiber-reinforcement architecture, particularly for woven and braided configurations.

Gage Section Geometry -- Tubular test specimens are classified into two groups -- straight-sided and contoured gage-section, as shown in Figures 6 and 7. Contoured gage-section test specimens are distinctive in having gage sections with thinner wall thicknesses than in the grip sections.

Although straight-sided test specimens are easier to fabricate and are commonly used, tubular test specimens with contoured gage sections are preferred to promote failures in the uniformly-stressed gage section.

Experience has shown that successful tests can be maximized by using consistent ranges of relative gage section dimensions, as follows:

\[
2 < \frac{L_o}{D_o} < 3 \quad \text{and} \quad 15 < \frac{L_o}{t} < 30
\]

where \(L_o\) is the defined gage length, \(D_o\) is the outer diameter in the gage section, and \(t\) is the wall thickness in the gage section of the tube. Deviations from the recommended geometries may be necessary depending upon the particular composite tube geometry being evaluated.
Figure 8 Illustrations of test setups for i) Insert testing and ii) Hydraulic testing of tubes (from Ref. 9)

TEST EQUIPMENT AND PROCEDURES
Test setup, Force and Strain Measurement, Data Acquisition -- The test method can use a standard load frame with a hydraulic or screw drive loading mechanism and standard force transducers for controlled axial loading for the elastomer insert test method. Guidance is given regarding type, composition and properties of the elastomeric insert material. However, a source of controlled-pressure hydraulic fluid is necessary for the internal pressure, hydraulic test method. Primary strain measurement can be measured by strain gages and/or string extensometers in the “gage section.” If required, an environmental test chamber may be used to control humidity and ambient temperature. Data collection should be done with a minimum of 50-Hz response and an accuracy of ±0.1 % for all data.

Test Procedures -- Generally, a test mode is used to avoid “run-away” tests that sometimes occur in force-control tests. Test mode rates are chosen so as to produce test specimen failures in 5-50 s. Failure within one minute or less should be sufficient to minimize slow-crack growth (SCG) effects. If slow crack growth is observed (e.g. under slow test mode rates), subsequent tests can be accelerated to reduce or eliminate slow crack growth. The test specimen is tested in hoop tension to fracture. The test specimen is retrieved for failure analysis and post-test dimensional measurement. A minimum of five valid tests is required for the purposes of estimating a mean. A greater number of tests may be necessary, if estimates regarding the form of the strength distribution are required. Fractography is suggested if the failure mode and fracture location are of interest.

CALCULATION, REPORTING, PRECISION AND BIAS
Calculations -- Using the measured force and or/pressure data along with the measured strain and/or deformation data as well as the test specimen dimensions, the resulting hoop stress-strain curve for each test specimen is determined. Calculation of the hoop stress is dependent on test method as shown in Fig. 9. Calculations for the elastomeric insert method may need to account for friction effects between the insert and the walls of the tubular test specimen. From the stress-stain curve, the following hoop tensile properties are determined: i) ultimate hoop tensile strength and corresponding strain, ii) fracture hoop tensile strength and corresponding strain, iii) proportional limit hoop tensile stress and corresponding strain, iv) elastic modulus in the circumferential direction, v) modulus of toughness.
**Figure 9** Calculation of hoop stress depending on pressurization method

\[
\sigma_\theta = P \frac{2r_i^2}{r_o^2 - r_i^2} \quad \text{and} \quad \varepsilon_\theta = \text{measured directly [at outer radius for internal pressure]}
\]

where: \( P = \text{pressure} \)

[internal hydraulic pressurization]

or

\( P = f (F_{\text{axial}}, A_{\text{insert}}, \text{Elastic constants, stiffnesses}) \)

[elastomeric insert loading]

Reporting -- The test methods provide detailed lists of reporting requirements for test identification, material and test specimen description, equipment and test parameters, and test results (statistical summary and individual test data).

Precision -- CMCs have probabilistic strength distributions, based on the inherent variability in the composite: fibers, matrix, porosity, fiber interface coatings, fiber architecture and alignment, anisotropy, and inherent surface and volume flaws. This variability occurs spatially within and between test specimens. Data variation also develops from experimental variability in test specimen dimensions, volume/size effects, extraneous bending stresses, temperature and humidity effects and the accuracy and precision of transducers and sensors.

Once the test methods are drafted, vetted and balloted, ASTM Committee C28 is planning interlaboratory testing programs per ASTM Practice E691 to determine the precision (repeatability and reproducibility) for a range of ceramic composites, considering different compositions, fiber architectures, and specimen geometries.

**CURRENT STATUS AND FUTURE WORK**

The draft standard test methods for hoop tensile behavior of CMC tubes are scheduled for first rounds of consensus ballots at subcommittee and main committee levels in late Spring 2013. If balloting is successful, publication of the full consensus standard test methods is expected for Fall 2013 or Spring 2014. Once the standards have been published, a round-robin interlaboratory testing program will be organized and executed, given available material, funding, and participating laboratories.

With sufficient interest and participation within the CMC community, new mechanical test standards for CMC tubes are planned for axial tensile strength, torsional shear strength, and flexural strength.

**CONCLUSIONS**

There is a real need for a comprehensive and detailed consensus test standard for hoop tensile testing of CMC tubes. This need is based on the certification and qualification requirements for CMC tubes in nuclear fission reactors. Test standards for tubes are needed because tests on flat composite panels are not representative of the architecture and geometry of composite tubes, with their 2-D and 3-D fiber architectures. The proposed ASTM standard test methods for hoop tensile testing of CMC tubes will be comprehensive and detailed, providing strong procedural documents using the conventional ASTM format. These new standard test methods will be applicable to 1-D, 2-D, and 3-D CMC tubes with diameters up to 150 mm and wall thicknesses up to 25 mm. The test methods will
address the following experimental issues -- test specimen geometries and preparation, different loading methods, test equipment, interferences (material, specimen, parasitic stresses, test conditions, etc), testing modes and procedures, data collection, calculations, reporting requirements, and precision/bias.

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