SPECIMEN SIZE EFFECT CONSIDERATIONS FOR IRRADIATION STUDIES OF SiC/SiC G. E. Youngblood, C. H. Henager, Jr., and R. H. Jones (Pacific Northwest National Laboratories)*

OBJECTIVE

The objective of this work is to examine current practice for comparing the mechanical and thermal properties of irradiated, continuous SiC fiber/SiC matrix composites (SiC/SiC) and to recommend test methodologies within the constraints of limited reactor test volumes.

SUMMARY

For characterization of the irradiation performance of SiC/SiC, limited available irradiation volume generally dictates that tests be conducted on a small number of relatively small specimens. Flexure testing of two groups of bars with different sizes cut from the same SiC/SiC plate suggested the following lower limits for flexure specimen number and size: Six samples at a minimum for each condition and a minimum bar size of 30 x 6.0 x 2.0 mm^3.

PROGRESS AND STATUS

Introduction

Continuous fiber ceramic composites (CFCC's) are currently under development for advanced aerospace or heat engine components. These applications for CFCC's will require optimum material behavior and a reliable means for physical and mechanical property characterization. For this reason, the ASTM Subcommittee C28.07 recently has set some standard methods for analyzing and testing CFCC's, i.e., tensile and shear strength as of December 1995.[1] Other test methods currently are being considered, i.e., flexural and compressive strength, tension-tension cyclic fatigue, creep and creep rupture under tensile loading, fracture toughness, tube strength and interfacial and thermal property testing.

A SiC fiber-reinforced/SiC matrix composite (SiC/SiC) is a CFCC that is particularly attractive for structural applications in fusion energy systems.[2] Unfortunately, there is a very limited amount of reactor test space available with high neutron fluences in which to conduct irradiation studies of SiC/SiC. Furthermore, smaller test volumes usually involve larger flux gradients and perhaps temperature gradients, each of which are additional incentives to use small specimen sizes. Within these constraints, the effect of specimen number and size reduction on the validity of the experimental measurements for irradiation studies of SiC/SiC is an important consideration.[3]

Mechanical property testing of irradiated SiC/SiC to date has emphasized the flexural method primarily because it uses a simple sample geometry that is compatible with SiC/SiC fabrication procedures.[4-5] Reactor space and ease of testing will likely make this geometry useful for fusion purposes. Therefore, this report will focus on the influence of the specimen number and size on the mechanical property values when determined by the flexural method.

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SiC/SiC Background

Figure 1 depicts a common SiC/SiC architecture, where the composite is made up of two-dimensional (2D), 0-90° plain weave cloth layers (plies) with a surrounding matrix of polycrystalline SiC deposited by chemical vapor infiltration (CVI). In this balanced 2D structure, the fibers make up about 40% of the volume with 20% in each principal direction. Obviously for this SiC/SiC architecture, care must be taken to prepare samples compatible with the principal directions, with the understanding that properties in other directions can be filled in with appropriate composite models.

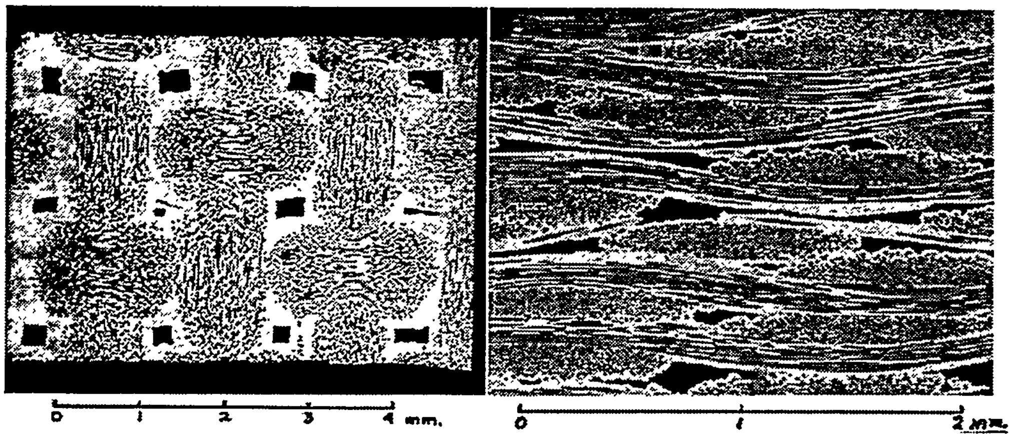

Figure 1. SiC/SiC 2D, 0-90° balanced weave architecture and macrostructural features.

The cloth layers are woven from untwisted yarns (tows) which contain 500 to 1000 individual SiC fibers with diameters of about 12 microns. The composite cell structure has a nominal repeat distance of 1-2 mm in the weave plane and about 0.3 mm ply-to-ply. The anisotropic interlayer macroscopic porosity appears correlated to the repeat cell structure. These macroscopic structural features will influence the mechanical properties, especially the interlaminar shear strength of SiC/SiC. Other physical properties, such as thermal conductivity, also will be particularly affected by the bundle geometry and the macroporosity. The shape and distribution of these macroscopic features suggest that a minimum number of repeat cells will set a practical lower limit on the specimen test size needed to be representative of this type of woven material.

In a SiC/SiC composite, the high strength fibers (~2 GPa) reinforce the matrix and can considerably improve the composite toughness and ultimate fracture strength over that of monolithic SiC. As stress levels increase, the matrix microcracks and debonds from the fibers, and the load is gradually transferred from the matrix to the stronger fibers. The mechanical properties now will be primarily determined by the nature of the matrix microcracking and by individual fiber cracking and pull-out. Due to the complex fiber-matrix interaction, these composites exhibit non-linear stress-strain behavior. The ultimate composite tensile strength is determined by gross fracture of the fiber bundles.

In Table 1, the room temperature ultimate strength and strain values and initial modulus values (% Std. Dev. in parenthesis), determined by three different test methods for a reference SiC/SiC, are compared.[6] The number of specimens and specimen dimensions used for each method also are listed. ASTM recommends that at least 10 specimens be tested for each condition to achieve statistically reliable average values.[7] To obtain a valid flexural strength, the composite must break or fail by tension or compression in the outer fibers, rather than by shear failure. Therefore, they also recommend a support span-to-depth ratio (S/d) of at least 32/1 and 16/1 for the 4-Pt and 3-Pt flexural methods, respectively. A high S/d ratio maintains the shear stresses low relative to the tensile or compressive stresses during flexural testing. Composite 2D SiC/SiC material can be relatively weak in interlaminar shear. The specimen dimensions listed in Table 1 meet ASTM specifications, but for the flexure tests the numbers of specimens are below the recommended level.

Table 1. Comparison of Tensile and Flexural Properties (at 23%C) of a Reference SiC/SiC*

Property	Tensile	4-Pt Flex	3-Pt Flex
Number	246	6	4
Dimensions, LxWxD (mm)	150x6x2	100x6x3	52x6x3
Ultimate Strength (MPa)	192 (9)	278 (7)	338 (6)
Initial Modulus (GPa)	228 (14)	180 (14)	140 (15)
Ultimate Strain (%)	0.22 (18)	0.22 (9)	0.30 (10)

*2D 0-90° balanced weave, Nicalon-CG fibers, 150 nm PyC-interphase, fabricated by Dupont Lanxide Composites, Inc., Newark, DE.

It is apparent from the data listed in Table 1 that measured property values depend upon the test method employed. The ultimate tensile strength and strain values are lower and the initial modulus is higher for the tensile test relative to either bend test, while a similar trend is observed going from the 4-Pt to the 3-Pt bend test. A lower strength from a tensile test relative to a bend test, and correspondingly from a 4-Pt to a 3-Pt bend test, reflects the differing stress distributions and volumes exposed to the maximum stresses within the specimens as well as non-linear flexure behavior of SiC/SiC.[8] These effects influence the initial modulus and elongation values as well.

The flexural test method should only be used for material development, quality control and component design flexural specifications. In this method, the flexural stress is computed from elastic beam theory with the simplifying assumptions that the composite is homogeneous and linearly elastic, which can be approximately valid for composites with the principal fiber direction coincident/transverse with the axis of the beam. Even though methods exist to determine tensile or compressive composite strengths from flexural data,[9] the methods can be expected to result in ambiguity where the composite structures are as anisotropic and inhomogeneous as the one illustrated in Figure 1. For this reason, the statistical uncertainties (listed in parenthesis in Table 1) probably reflect sample to sample variations in the macroscopic structural features (interlayer porosity, bundle weave pattern, etc.) rather than differences in microscopic flaw distributions.

With the Table 1 data as a baseline, flexural tests were performed on a set of SiC/SiC bend bars with smaller than ASTM recommended dimensions for comparison. The dimensions were selected based on the dual practical consideration of a limited irradiation test space and a desire to retain a representative number of complete fiber bundle repeat cells in a test specimen. Currently, a second set of bars is being irradiated in the HFIR reactor as part of the U.S. DOE-Monbusho/Jupiter fusion materials program and will be evaluated later. The effect of using smaller and fewer samples for flexural measurements of unirradiated SiC/SiC will be evaluated in this progress report.

Experimental Procedure

The tested 2D 0-90° SiC/SiC composites were fabricated by CVI as before, but the fiber was Hi-Nicalon, an advanced SiC fiber. Hi-Nicalon has a much reduced oxygen content (<0.5 wt% O) and an improved thermal stability (to about 1300°C) compared to Nicalon-CG (~12 wt% O and 1100°C, respectively). In addition, the Hi-Nicalon fiber diameter uniformity is much improved over that of Nicalon-CG (about a 50% reduction in diameter variability). It is anticipated that SiC/SiC made with Hi-Nicalon will exhibit improved mechanical properties as well as improved irradiation performance. Improved mechanical properties is expected because the post-CVI fiber strength should be higher and improved irradiation performance is anticipated because the fiber is more crystalline.

Two size groups of flexural test bars (38.4 x 6.6 x 3.5 and 25.1 x 4.0 x 2.0 mm^3) were cut and machined from a 20 cm square by 3.5 mm thick plate. Although the overall dimensions differed, the S/d ratios were kept about the same for the two size groups. The bars had geometric densities in the range 2.52 ± 0.05 g/cm^3. For this size effect study, flexure tests were performed at 500°C in argon (7 bars per size group) to also provide a baseline for the later Jupiter irradiation tests.

The 4-Pt bend test procedures and fixtures generally were the same as used in previous studies.[5] The outer/inner pin spacings were 30/15 and 20/10 (mm) for the larger and smaller bars, respectively. For the 20/10 bars, the surface in compression had been machined to attain the 2 mm bar thickness. For both bar sizes, the tension surfaces were the as-received CVI-coated surfaces. The Instron cross-head speed was adjusted to maintain the outer surface strain rate at 1.1 x 10^{-3} s^{-1} to within 10% for each bar size, as recommended by ASTM.[7]

Results and Discussion

All of the seven bar samples in the 38 mm group appeared to fracture initially in tension between the inner load pins as required for valid flexural testing. Two of the bars in the 25 mm group appeared to fracture initially by shear directly under an inner load pin, and their data was invalidated. The other five bars in the 25 mm group appeared to fail in tension initially as required. The stress-strain curves appear slightly separated for the two size groups as shown in Figure 2. The valid average ultimate strength and strain values and the initial modulus values for the two groups are presented in Table 2 with standard deviations in percent given in parenthesis. The flexural strength and strain values measured for the Hi-Nicalon/SiC are considerably improved over the Table 1 baseline values for Nicalon-CG/SiC. The initial modulus determined for Hi-Nicalon/SiC is somewhat lower than determined for Nicalon-CG/SiC and may be due to the thicker carbon interphase used in the Hi-Nicalon composite.

Within experimental uncertainty, the initial modulus is the same for each size group, but above the matrix cracking stress of about 150-200 MPa, the 25 mm group exhibits a somewhat softer modulus than the 38 mm group. Since the S/d ratios were the same for both groups, and therefore the stress distributions based on LEFM were the same, the observed modulus difference above the matrix cracking stress likely was due to the difference in bar widths. The 38 mm and 25 mm groups contained about six and four fiber bundles across their widths, respectively. The bars containing more complete fiber bundles should statistically exhibit higher overall strength and more representative values. The somewhat larger uncertainties exhibited by the 25 mm group probably reflect the larger statistical variation in macroscopic structural features which would have a greater influence in the testing of smaller specimen volumes.

Table 2. Flexural Properties of SiC/SiC Calculated from Figure 2 Data (7 and 5 bars, respectively)*

Property	38 mm Group	25 mm Group
Ultimate Strength (MPa)	567 (3)	513 (10)
Initial Modulus (GPa)	107 (15)	108 (9)
Ultimate Strain(%)	0.83 (3)	0.86 (6)

* Fabricated by Dupont Lanxide, 2D 0-90° plain weave, Hi-Nicalon fiber, 1.2 μm C-interphase.

Although the measured differences in mechanical property values weren't large between the two groups, the 38 x 6.6 x 3.5 mm^3 bar size data appears to be more representative and reproducible than the 25 x 4 x 2 mm^3 bar size data. If reactor test volume restricts flexure bar sizes below the 38 mm group size, based on this work a recommended lower limit bar size would be 30 x 6.0 x 2.0 mm^3, which maintains the 2/1 flexure pin spacing with S/d = 10. During a flexure test, the maximum shear stress (τ) that builds up along the neutral plane is given by $\tau = 0.75P/bd$ where P is the load and b and d are the bar width and depth, respectively. To prevent premature failure by shear rather than failure by tension, the shear stress can be reduced by increasing the bar width which compensates for decreasing the bar depth to 2.0 mm. Also, a 6.0 mm bar width would contain at least five full fiber

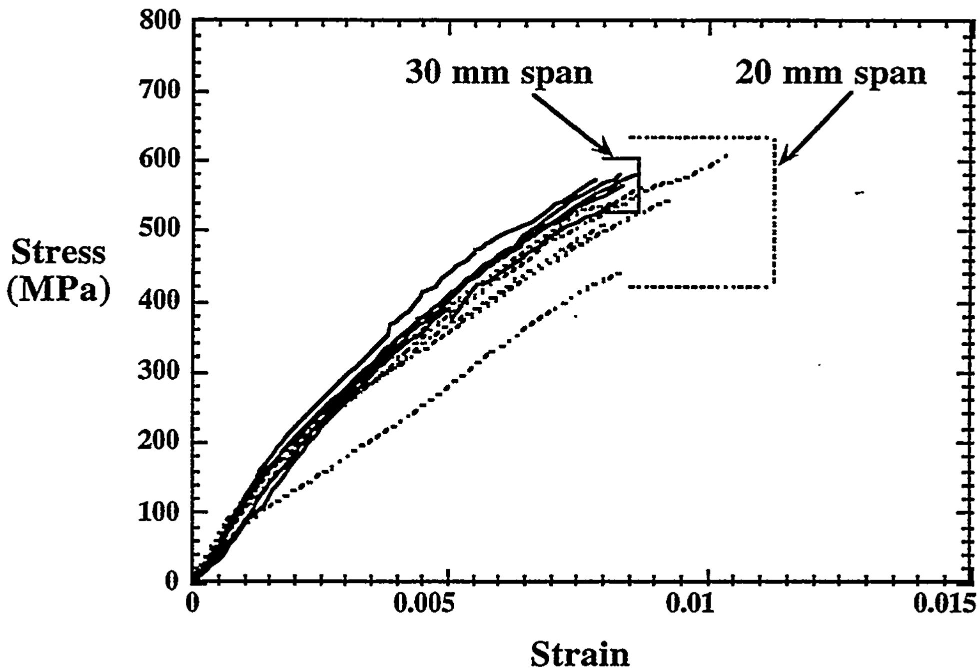

Figure 2. Stress-Strain Curves from 4-Pt Flexure Tests for Two Bar Sizes of 2D 0-90° SiC/SiC.

bundles across its width, given the current 2D 0/90° composite architecture and therefore is more representative of the composite material than a narrower bar. If bar depths are reduced, they should only be reduced from one surface and that surface should be the compression surface. A slight increase in length from 25 to 30 mm would allow a 5 mm outer pin overhang, which is recommended by ASTM specifications.[7] Finally, at a minimum, at least six samples for each condition should be tested to achieve statistically representative (± 10%) flexure property values.

CONCLUSIONS

1. The flexural ultimate strength and strain values measured for a Hi-Nicalon/SiC composite were considerably higher than baseline values representative of a Nicalon-CG/SiC reference composite.

2. For reliable and reproducible flexure testing of typical 2D 0-90° SiC/SiC, the following recommendations are proposed:

- minimum sample dimension of 30 x 6.0 x 2.0 mm^3.
- span to depth ratio of 10.
- 2/1 flex pin spacing.
- minimum width of 6 mm.
- test a minimum of 6 samples.
- reduce thickness from one side only - compressive surface.

FUTURE WORK

The size effect analysis for flexure bars will be repeated for a set of bars currently being irradiated in the HFIR reactor as part of the Jupiter fusion energy materials test program.

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