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Miniaturized fracture stress tests for thin-walled tubular SiC specimens

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Abstract

Two testing methods have been developed for miniaturized tubular specimens to evaluate the fracture stress of chemically vapor deposited (CVD) SiC coatings in nuclear fuel particles. In the first method hoop stress is applied to a thin-walled tubular specimen by internal pressurization using a polyurethane insert. The second method is a crushing technique, in which tubular specimen is fractured by diametrical compressive loading. Tubular SiC specimens with a wall thickness of about 100 μm and inner diameters of about 0.9 mm (SiC-A) and 1 mm (SiC-B) were extracted from surrogate nuclear fuels and tested using the two test methods. Mean fracture stresses of 239, 263, and 283 MPa were measured for SiC-A and SiC-B by internal pressurization, and SiC-A by diametrical loading, respectively. In addition, size effects in the fracture stress were investigated using tubular alumina specimens with various sizes. A significant size effect was found in the experimental data and was also predicted by the effective area-based scaling method.

1. Introduction

Carbon/carbide coated particle fuels have been developed for use in high temperature gas-cooled reactors [1–8]. The uranium dioxide (UO_2) or uranium carbide (UC) fuel kernels are coated with a porous pyrolytic carbon layer (buffer layer), and then with tri-isotropic (TRISO) coatings: inner pyrolytic carbon, silicon carbide (SiC), and outer pyrolytic carbon layers [1,2,6–8]. Among the TRISO layers, the SiC layer is the most important component for the structural integrity of fuel particles because it sustains most of the internal pressure caused by fission gas generation [3–5].

Since the diameter of the SiC coating is only about 0.9 mm and its thickness is usually less than 0.05 mm, no procedure for testing and evaluation of such a small component has been well established [9]. Further, it is known that the fracture strength of a ceramic material is dependent on the size and shape of the specimen [10–12]. In this study, therefore, testing and evaluation methods to produce the mechanical property data using miniature SiC specimens were developed and the size effects in small specimens were investigated. This paper introduces two testing and evaluation methods: an internal pressurization method and a diametrical loading method. Test results for chemically vapor deposited

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(CVD) SiC tubular specimens and for tubular alumina specimens are reported here.

2. Theoretical backgrounds for tubular specimens

2.1. Stress distribution under internal pressurization

Schematics of the two testing methods developed for tubular specimens are given in Fig. 1. Theoretical solutions to calculate fracture stresses using the two loading techniques are described here. If a tubular specimen is internally pressurized to a pressure, P, by compression of an elastomeric insert, the axi-symmetrical hoop stress is expressed as a function of the radial distance from the centerline of the tubular specimen, r [10,12–15] as:

$$\sigma(r) = \frac{r_i^2 P}{r_o^2 - r_i^2}\left(1 + \frac{r_o^2}{r^2}\right), \tag{1}$$

where r_i and r_o are the inner and outer radii of tubular specimen, respectively. The fracture stress, which is equivalent to the maximum stress when fracture occurs, is given as the stress at the inner surface ($r = r_i$):

$$\sigma_f = \frac{r_o^2 + r_i^2}{r_o^2 - r_i^2} P_f. \tag{2}$$

2.2. Stress distribution under diametrical loading

In crush test the tubular specimen is subjected to a diametrical compressive loading. The expression for stress distribution is derived from curved beam theory [16]. If a tube is subjected to a load per specimen length, L, applied through the centroidal axis on the outer surface of a tube, the resultant elastic hoop stress is the sum of the stress components caused by an axial load and by a bending load. The bending moment, M, at a point is given as a function of the angle from the horizontal line, θ:

$$M = \frac{1}{2} LR\left(\frac{2}{\pi(1+z)} - \cos\theta\right), \tag{3}$$

where

$$R = \frac{1}{2}(r_i + r_o),$$

$$z = \frac{R}{2c}\ln\left(\frac{R+c}{R-c}\right) - 1,$$

$$c = \frac{1}{2}(r_o - r_i).$$

The hoop stress at a point (y, θ) is expressed by [16]

$$\sigma = \frac{L\cos\theta}{2a} + \frac{M}{aR}\left(1 + \frac{1}{z}\frac{y}{R+y}\right), \tag{4}$$

where

$a = 2cl$,

l = the specimen length loaded, y = the distance from the mid-thickness position of the wall ($-c \leqslant y \leqslant c$).

The maximum stress occurs at the inner surface of the tube ($y = -c$, $\theta = 90°$) or at the inside of the point where load is applied ($y = +c$, $\theta = 90°$). When fracture occurs at a load, L_f, the fracture stress is given by

$$\sigma_f = \frac{L_f}{\pi a(1+z)}\left(1 - \frac{1}{z}\frac{c}{R-c}\right). \tag{5}$$

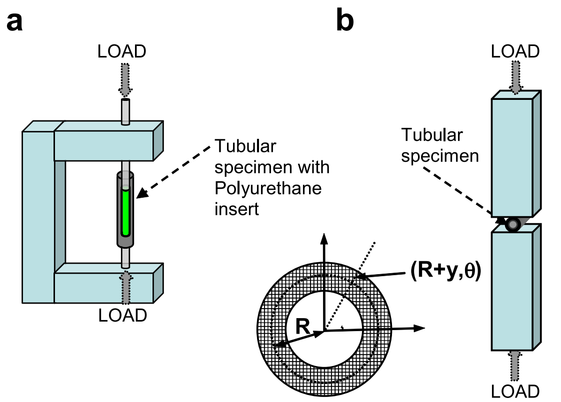

Fig. 1. Schematics of two loading methods for tubular specimens in fracture strength tests: (a) internal pressurization using elastomeric insert and (b) diametrical loading (coordinate system ($R + y$, θ) is displayed).

2.3. Statistical analysis and size effect

Ceramic materials usually fail in a brittle mode without plastic deformation and their tensile fracture strengths are dependent on the inherent flaws and microstructural characteristics that produce stress concentrations [17–20]. The Weibull model (or the weakest link model) is the most widely used model to account for the statistical behavior of fracture stress. The model states that fracture can occur at the most critical flaw by the lowest stress [17,18]. The flaw dependence of strength also leads to a size and geometry dependence of fracture strength because a larger stressed volume or area yields a higher probability of failure at a given maximum stress [10–12,18–20].

The probability of failure, F, is described by a two-parameter equation [10,18,20]:

$$F = 1 - e^{-\int (\frac{\sigma}{\sigma_0})^m \mathrm{d}V}, \tag{6}$$

where m is the Weibull modulus (or the shape parameter); σ_0 is the characteristic strength (or the scale parameter); and σ is the stress given as a function of position. For a set of fracture stress data, the probability of failure at a given fracture stress can be estimated from the ratio, $i/(n+1)$, where n is the total number of data and i is the rank order of a fracture stress measurement.

When the stress distribution is known for a specimen geometry, the maximum stress at fracture, $\sigma_{\max}$, can be measured from strength tests. In Eq. (6) the volume integral of the normalized stress distribution function is called the effective volume, V_E, which is a key parameter when failure occurs in a volume. If the fracture events predominantly occur at the surface, the volume integral in the exponent of Eq. (6) should be replaced by the area integral for the same normalized stress term, which is called the effective area, S_E. For two specimens having different sizes or shapes, 1 and 2, the ratio between their fracture stresses (maximum stresses at fracture) for a probability of failure can be correlated with the ratio or effective volumes or of effective areas, respectively:

$$\frac{\sigma^1_{\max}}{\sigma^2_{\max}} = \left(\frac{V^2_E}{V^1_E}\right)^{1/m}, \tag{7}$$

$$\frac{\sigma^1_{\max}}{\sigma^2_{\max}} = \left(\frac{S^2_E}{S^1_E}\right)^{1/m}. \tag{8}$$

3. Experimental

Tubular SiC specimens were obtained from rod-type surrogate fuels in which the pyrolytic carbon and silicon carbide layers were deposited on graphite rods using the CVD processes. In the process, graphite rods with two different nominal diameters, about 0.9 mm and 1.0 mm, and with a length of 6 mm are coated in a fluidized bed using a vertical, high temperature furnace. The SiC layer is deposited on the pyrolytic carbon layer by decomposition of methyltrichlorosilane (CH_3SiCl_3) in a hydrogen carrier gas (H_2) at about 1500 °C. This coating process produced the same structure of SiC layer as that produced by the TRISO fuel coating process. To extract the SiC tubular shells from the coated rods, both ends of the coated cylinders were removed by grinding. Then the carbon coating and the graphite substrate were burned off by baking the cylinders in air at 700 °C for 4–6 h.

Two types of tubular SiC specimens having slightly different sizes were produced for strength tests. One of the specimen types was 0.95 μm thick SiC tubular specimen with an inner diameter of 0.89 mm and a length of 5.27 mm; the other type was a slightly thicker and larger tubular specimen with a wall-thickness of 100 μm, an inner diameter of 1.02 mm, and a length of about 5.83 mm. The nominal dimensions of SiC specimens are listed in Table 1, along with other statistical parameters. Note that these nominal dimensions were used in calculations.

The size effect on fracture strength was investigated using tubular alumina specimens of 12 different sizes, 4 diameters × 3 lengths for each diameter. The inner (outer) diameters of the tube specimens were 0.51(0.79), 1.02(1.98), 2.39(3.19), and 6.35(9.50) mm, and the specimen lengths were 1 to 3 times their outer diameters for the largest three

Table 1
Specimen dimensions and statistical parameters for tubular silicon carbide specimens

Specimen	Loading method	Specimen dimensions, mm			# of tests	Insert length, % of L	Statistical parameters			
		I.D.	O.D.	L			Mean fracture stress, MPa	Weibull modulus (m)	Effective volume (V_E), $\times 10^{-10}$ m^3	Effective area (S_E), $\times 10^{-6}$ m^2
SiC-A	Internal	0.89	1.08	5.27	17	50	238.8	9.7	3.10	7.40
SiC-B	Pressurization	1.02	1.22	5.83	21	50	263.2	5.0	6.36	9.35
SiC-A	Diametrical loading	0.89	1.08	5.27	15	–	282.6	4.9	0.19	3.82

Table 2
Specimen dimensions and statistical parameters for tubular alumina specimens

Specimen	Loading method	Specimen dimensions, mm			# of tests	Insert length, % of L	Statistical parameters			
		I.D.	O.D.	L			Mean fracture stress, MPa	Weibull modulus (m)	Effective volume (V_E), $\times 10^{-10}$ m^3	Effective area (S_E), $\times 10^{-6}$ m^2
0.51L	Internal pressurization	0.51	0.79	2.36	24	60	326.0	10.3	0.627	2.27
0.51E		0.51	0.79	3.15	25	50	359.1	10.7	0.696	2.52
0.51F		0.51	0.79	4.72	22	30	389.1	9.6	0.627	2.27
1.02SM		1.02	1.98	1.98	21	70	296.7	9.1	2.25	4.42
1.02M		1.02	1.98	3.96	24	50	318.5	12.8	3.21	6.32
1.02LM		1.02	1.98	5.94	21	70	267.3	10.1	6.75	13.3
2.39SM		2.39	3.18	3.18	21	60	258.0	9.6	18.4	14.3
2.39MM		2.39	3.18	6.35	27	50	251.9	11.7	30.7	23.8
2.39LMM		2.39	3.18	9.53	15	15	302.8	9.5	13.8	10.7
2.39LM		2.39	3.18	9.53	22	40	269.2	11.6	36.9	18.6
6.35SM		6.35	9.5	9.5	21	50	260.0	11.3	328	94.7
6.35MM		6.35	9.5	19.0	18	60	235.7	13.4	787	227
6.35LM		6.35	9.5	28.5	16	60	229.3	10.5	1180	341
1.02S	Diametrical loading	1.02	1.98	1.98	21	–	345.0	9.8	0.180	0.883
2.39S		2.39	3.18	3.18	21	–	343.8	5.9	0.505	3.43
6.35S		6.35	9.5	9.5	18	–	248.2	6.8	16.4	27.0

diameters and 3 to 5 times the outer diameter for the smallest diameter. Dimensions, number of specimens, and other related parameters of the tubular specimens are listed in Table 2.

In the internal pressurization test a polyurethane insert in the form of cylindrical rod is inserted into a tubular specimen and is axially compressed by pistons (flat-ended pins) to internally pressurize the specimen for fracture by the induced tensile hoop stress, Fig. 1(a). The plastic inserts were made by casting from liquid polyurethane mixtures or by punching from solid polyurethane rod and then cut to appropriate lengths, typically 50–70% of the specimen lengths. (Note that the use of the limited insert lengths was for convenient loading and to avoid edge effects.) The maximum stress at fracture, which occurs at the inner surface (Eq. (2)), was calculated and used as the fracture stress of the specimen [13–15].

In the diametrical loading method, on the other hand, a tubular specimen is compressed by diametrical loading on the outer surfaces using two flat-ended loading columns, as illustrated in Fig. 1(b). The highest tensile stress is calculated at the two inner surfaces below the contacts with the end faces of two loading columns. The load at fracture was recorded to calculate the fracture stress for the specimen using Eq. (5). Crosshead speed was about 0.01 mm/s for both test methods, which were performed at room temperature.

4. Results and discussion

4.1. Fracture stress of SiC and statistical characteristics

The Weibull plots for fracture stress (variable x) from the two versions of SiC specimens are displayed in Fig. 2. The two testing methods were applied to the specimen SiC-A. The fracture stress by the diametrical loading method was about 18% higher than that by the internal pressurization method; the mean

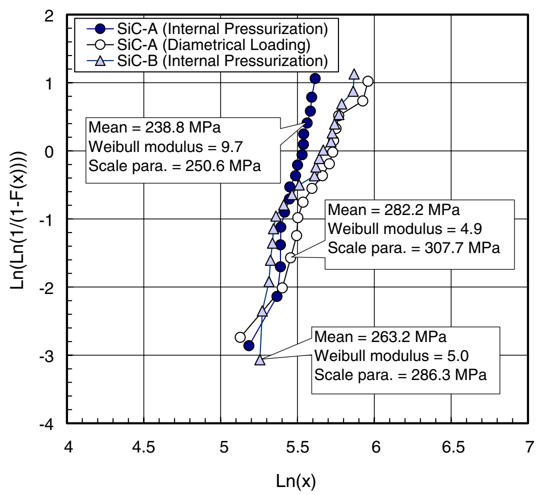

Fig. 2. Comparison of fracture stress data for CVD SiC tubular specimens (x = fracture stress).

values were 282.6 and 238.8 MPa, respectively. The fracture stress depends on the effective volume or area [10–12]. In the tubular CVD SiC specimens, the inner surface is assumed to be the most probable crack initiation site not only because the maximum stress occurs at the inner surface but also because the inner surface consists of dimples of a few μm, which originate from the rough surface of the pyrocarbon substrate.

While the difference between the mean fracture stresses by different loading techniques is not significant, the Weibull modulus by the internal pressurization method (9.7) was almost two times higher than that by the diametrical loading (or crush) method (4.9). The stress distribution in the diametrical loading is highly localized at the inner surface of tubular specimen beneath the contact with loading column. Crack initiation events under a highly localized stress may be more sensitively influenced by flaw distribution characteristics than under a uniformly distributed stress. The measured fracture stress shows larger scatter, or lower Weibull modulus, when the stress distribution is so highly localized that the size of the maximum stress region reaching the fracture stress is comparable with the size or mean distance between the flaws; i.e., only sampling a small volume of the tube for the crush test, Table 2. This crack initiation issue should be pursued in future study for the CVD SiC material. For SiC-B specimens, the internal pressurization method gave a similar mean fracture stress of 263.2 MPa, and the Weibull modulus and scale parameter were 5 and 286.3 MPa, respectively.

4.2. Size effect in the fracture stress of alumina

The mean fracture stresses for tubular alumina specimens are listed in Table 2, along with other Weibull parameters. A significant size effect is found in the fracture stress: the fracture stresses for the specimens with the largest diameter (I.D. = 6.35 mm) are in the range 220–260 MPa, while those with smallest diameter (I.D. = 0.51 mm) are in the range 320–390 MPa. Such a size effect on strength is commonly found in brittle materials such as ceramics [10–12]. Also, the values for the Weibull modulus m were in the range 5.9–13.4, with averages of 10.78 for internal pressurization, and 7.47 for diametrical loading. The influence of variability in the manufacturing process of alumina tubes was ignored in this study.

The average of these two values, 9.13, was used to calculate the other parameters such as effective volume and effective area. Using these parameters, the fracture stresses for different size specimens were predicted from the mean fracture stress of 2.39LM specimens, 269.2 MPa, using Eqs. (7) and (8). Fig. 3 shows the size effects and comparisons between the predicted and measured fracture stresses. In the effective area-based prediction all the values are within the bounds given by the measurement mean values ±2 × standard deviation. In general, the effective area-based method predicted the size effect on fracture stress more correctly than the effective volume-based method, indicating that cracking starts at the inner surface of tubular specimens.

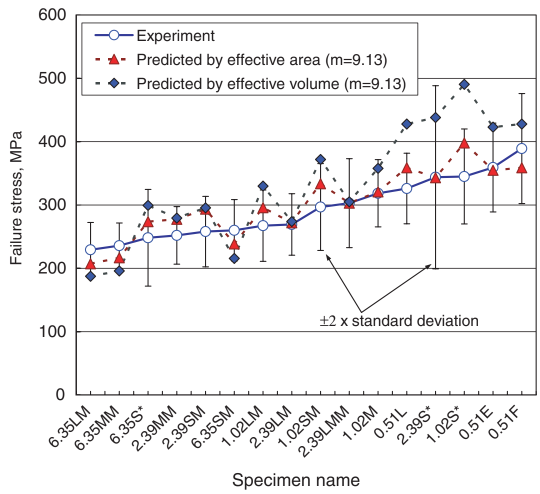

Fig. 3. Size effects in the fracture hoop stress of alumina specimens. The effective area-based predictions agree better with the experimental measurements than the effective volume-based predictions.

5. Summary

(1) Two testing methods using internal pressurization and diametrical loading have been applied to evaluate the fracture stress of miniature tubular specimens.
(2) Tubular SiC specimens with a wall thickness of about 100 μm and inner diameters of about 0.9 mm (SiC-A) and 1 mm (SiC-B) were tested using the two methods. The mean values of fracture stresses 239, 263, and 283 MPa were measured for SiC-A and SiC-B by internal pressurization, and SiC-A by diametrical loading, respectively.
(3) Fracture stress results displayed a significant size effect in tubular alumina specimens; the mean fracture stress of the smallest specimens

was about 70% higher than that of the largest specimens. A Weibull analysis predicted a similar size effect on the fracture stress when the effect area-based method was applied.

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