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Hoop Tensile Strength and Fracture Behavior of Continuous Fiber Ceramic Composite (CFCC) Tubes from Ambient to Elevated Temperatures

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ABSTRACT: Presently, continuous-fiber ceramic composites (CFCCs) are considered leading candidate materials for many high-temperature applications, such as high-pressure heat exchangers, radiant burner tubes, and engine combustors. To adequately evaluate these materials in their cylindrical configurations, a hoop tension test is needed.

A hydrostatic pressurized test was developed to obtain the hoop tensile strength from ambient to elevated temperatures (>1500°C). The method allows only hydrostatic pressure to develop inside the cylinder to cause failure from a hoop tensile stress.

This test method evolved from testing monolithic ceramics to continuous-fiber ceramic matrix composite (CMC) tubes. The results of early hydrostatic tests are briefly reviewed. A highlight of one test identified fiber tow pull-out at 1000°C where the tube indicated localized aneurysm-type deformation. Another CFCC material system, evaluated at room temperature, exhibited fiber pull-out on the order of 5 to 7 mm. The circumferential elastic modulus was also obtained.

KEY WORDS: hoop tensile strength, pressurized cylindrical specimen, ceramic, continuous-fiber ceramic composite (CFCC), high temperature, mechanical properties, fracture behavior, diametral strain

Presently, continuous fiber ceramic composites (CFCCs) have been identified as useful materials for several severe environmental and industrial applications. These materials can be used as radiant burner tubes, high-pressure heat exchangers, and combustion chambers to increase thermal conversion efficiencies and conserve energy.

Flat coupon tensile tests have been used primarily for mechanical properties characterization of CFCCs to assess representative performance of cylindrical components. Other tests include flexural beams and tensile or compressive loading of C-ring or O-ring shaped specimens that are sectioned from tubular components. Unfortunately, the fracture behavior is at times biased by the specimen geometry and is not always representative of the actual component application. An example of a bias fracture behavior would be delamination from interlaminar shear stress caused by flexing a laminated composite component. To address this issue, an alternative test, such as an internally-pressurized cylinder, is used to assess the structural performance of tubular components.

Pressurized cylindrical tests have been demonstrated to (1) generate reliable test data for a potential design data base, (2) have a large tensile-test-gage-volume to specimen-volume ratio, (3) have a large tensile gage volume for Weibull analysis, (4) require minimal sample preparation, and (5) be a cost-effective specimen testing method. The main attribute of pressurized cylindrical tests is their ability to hydrostatically pressurize a right cylinder at elevated temperatures using a working fluid that exhibits viscoelastic behavior [*1,2*]. By eliminating gas pressurization and using a pressurizing media with plastic or viscoelastic behavior, the test technique virtually eliminates catastrophic explosion, and the failed test specimen is usually better suited for failure analysis.

From the theory of elasticity, the elastic-based hoop tensile stress profile $\sigma_t(r)$ is a function of the radius r and is given as [*3*]

$$\sigma_t(r) = r_i^2 p_i [1 + (r_o/r)^2]/(r_o^2 - r_i^2) \quad (1)$$

where r_o and r_i are the outer and inner cylindrical tube radii, respectively, and p_i is the internal pressure. The hoop tensile stress is maximum at $r = r_i$. A thin wall can be assumed if the ratio of the inner diameter D is greater than 20 times the wall thickness t. Equation 1 can then be simplified to the more familiar thin-walled pressure vessel form

$$\sigma_t = p_i D/2t \quad (2)$$

Hydrostatic pressurized tests of cylindrical tubes have been developed and used to obtain data from room temperature to 2070°C [*4–6*]. Common pressurizing media have been air, nitrogen, and argon. Oil and water have been primarily used for near ambient temperature. Elastomer solids, such as silicone rubber, have also been used [*4*]. The main pressurizing media used for high-temperature testing is gas; however, considerable care must be taken to limit damage to equipment and facilities from explosion when tube failure occurs. To address this issue, a high-temperature internal pressurization technique that minimizes the risk of explosion was developed at the University of Dayton Research Institute. This concept was demonstrated by testing a monolithic alumina tube by internally pressurizing the tube to failure at 1000°C in a vacuum

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chamber [1]. The technique uses an internal solid that deforms viscoelastically at the test temperatures of interest. The risk of explosion at failure is minimized by increasing the energy dissipation rate compared to that of a gas or liquid. The choice of deformable solids include metals, glasses, glass-ceramics, and ceramics. The attributes of pressurized testing of cylindrical tubes are large tensile-gage-volume to study Weibull scaling effects, efficient test-gage-volume to specimen-volume ratios that lower material consumption, no elaborate specimen alignment, reduced safety risks, and lower testing cost.

The objectives of this paper are to review the early work of the UDRI hydrostatic pressurizing technique [1,2] and update other developments and results achieved using this testing technique. The review of the earlier work begins in the next section, Experimental Procedures.

The background of the second objective deals with the issue of obtaining an equivalent level of detection to determine the onset of matrix cracking for an internally-pressurized tube when compared to a flat coupon tensile test. By having the ability to measure the strain, the onset of matrix cracking can be detected and measured. The strain of a cylindrical-shaped component can be measured in the circumferential or diametrical direction. It can be shown that these two strain components ϵ_{cir} and ϵ_{dia}, are equivalent for homogeneous and isotropic material, as shown below. The circumferential strain ϵ_{cir} is expressed as

$$\epsilon_{cir} = \Delta c/c = \pi\Delta d/\pi d \quad (3)$$

or

$$\epsilon_{cir} = \epsilon_{dia} \quad (4)$$

where c is the circumference and d is the diameter. For a quasi-isotropic composite material $[0, 90, \pm 45°]_n$, these strain components, ϵ_{cir} and ϵ_{dia}, can be presently assumed to be the same for a wound or woven tube. In this assumption, the in-plane strain (circumferential direction) is detected in the diametral direction as diametral strain.

Circumferential strain is fairly easy to measure using strain gages on a smooth, clean surface. However, the surface preparation of continuous fiber-reinforced ceramic composites can be tedious and complex. Typically, as-densified components do not have the smooth surface that strain gages require. Surface preparation for strain gages require diamond grinding and lapping to obtain the relative smooth surface to bond gages. Surface grinding may cause an undue amount of subsurface damage to the matrix and fiber, as well as the fiber/matrix interface; that is, the damage from surface preparation cannot be readily ascertained. Radial strain is not technically feasible to measure because tiny gages would be required to meet the geometric constraint of the thin walled cylinders and the strain field distortion is large across the laminate layers of a composite.

Diametral expansion can be measured during internal pressurization. This method of measurement can be accomplished easily using extensometers, such as a linear variable displacement transducer (LVDT), a capacitive gage attached to a contact probe, or a strain gaged clip extensometer. Most of the methods to measure strain can be used, with appropriate modifications, for high-temperature measurements of a pressurized tube. In addition, the sensitivity of measuring strain is directly proportional to the diameter size; therefore, it is not favorable to have small tube diameters.

Experimental Procedures

Hoop Tensile Strength at Elevated Temperature

Two prototype tubes, used as display models from two manufacturers were tested. The first material was a Nicalon™ (Dow Corning, Midland, MI) braided tube with a matrix of Si-C-N formed from a polymer impregnation and pyrolysis (PIP) process. The braided tube used carbon-coated Nicalon™ fiber tow that was wound at a ±45° angle.

Two short tubular CMC test specimens were cut from the PIP tube (51-mm lengths), using an ordinary diamond cut-off saw with a water-based coolant. The wall thickness was 2.54 mm and the inside diameter was 28.6 mm. All dimensions were nominal. No further tube surface or end preparations were made.

The tube was filled with a solid that exhibited viscoelastic behavior at elevated temperatures and was sealed at both ends with ring seals and pistons. Details of the pressurizing process were reported earlier [1,2]. The sealed tube specimen was heated in a vacuum hot press unit using induction heating with graphite susceptors. The test temperature of 1000°C was selected because it is the typical upper use temperature of Nicalon™ fibers. The specimen was soaked at the test temperature for 15 min before testing. A hydraulic actuator was used to compressively load the pistons of the tube ends to generate the internal hydrostatic pressure. The load was applied monotonically until tube rupture. Only the failure load was recorded from the vacuum hot press unit and the specimen was allowed to cool before removal. Optical inspection and photomacrography were used for failure analysis.

Hoop Tensile Stress versus Circumferential Strain at Ambient Temperature

The second prototype tube material (Textron Specialty Materials, Lowell, MA) was provided for room temperature evaluation to obtain the hoop tensile strength and to study the failure process. The tube's nominal dimensions were 90 mm (3.6 in.) inside diameter by 76 mm (3.0 in.) length by 2.9 mm (0.12 in.) wall thickness. The matrix was a reaction-bonded silicon nitride (RBSN) and the fiber reinforcement was SCS-6™ silicon carbide. The fiber architecture was a $[0°/\pm 45°]_r$ wound tube. The number of laminate layers was unknown.

The tube was hot wax mounted and sawn by a diamond wheel with a water-based coolant to create two tubular ring specimens with a final nominal length of 36 mm. The specimen wax was removed using a reagent. The specimens were baked for 2 h at 200°C and then air cooled to release the trapped moisture from the diamond machining operation.

A room-temperature test mandrel was designed and constructed to maintain a constant test gage length. The test mandrel was designed to pressurize the 20.3-mm central portion of the 36-mm tube length. Approximately 8 mm on each end of the tube length was not loaded under hydrostatic pressure to avoid edge effects. A solid media known to behave plastically at room temperature was used internally to hydrostatically pressurize the tube. The media in the reservoir was pressurized by loading a piston from a universal hydraulic testing machine (Riehle, SN: R-46343). The inside surface of the tube was lined with plastic food wrap to prevent the direct contact of pressurizing media to the inner tube surface. A strain gaged based extensometer was used to monitor the diametral expansion during loading. The loading rate was continually adjusted to compensate for leaks and to monotonically pressurize the tube to obtain failure within 1 min. After failure, the tube was removed from the mandrel for failure analysis.

Another prototype ceramic matrix composite (CMC) tube was provided by Textron. This tube had nominal dimensions of 88.9 mm (3.5 in.) inside diameter by 76.2 mm (3 in.) long by 1.8 mm (0.07 in.) wall thickness. The tube was cut into two specimens to obtain a nominal length of 38.1 mm (1.5 in.). Only one specimen was tested.

The pressurizing test mandrel used in a prior test was modified to maintain pressure better and prevent a potential pressure gradient from the pressure-supply reservoir to the specimen tube inner wall. The specimen was mounted on the test mandrel with a plastic film (Saran Wrap™) inside liner to minimize specimen contact with the pressurizing clay.

The Wheatstone bridge output of the extensometer and the force transducer were monitored by an *X-Y* plotter. The diametral extension of the tubular specimen was monitored to failure using a fully active resistive strain gaged clip extensometer. A video camera was also used to record the testing event.

Results and Discussion

High Temperature Hoop Tensile Strength of PIP-Processed SI-C-N/Nicalon Tube

Two tubular specimens were internally pressurized to failure at 1000°C in a vacuum chamber. The measured hoop tensile strengths were 182 and 237 MPa (26.4 and 34.5 ksi), respectively. The hoop tensile strengths assumed finite tube radii and thickness. Using the simplified equation for a thin-wall tube, the calculated values were 165 and 217 MPa (24.0 and 31.4 ksi), respectively. The simplified equation for thin-wall tubes typically yields conservative measurements when the ratio of the inner diameter to tube-wall thickness is much less than 10. The measured hoop tensile strengths in vacuum are comparable to those obtained from tensile tests of flat coupons of other CMCs. Comparable values found in the literature are presented in Table 1 [*1*].

Failure Analysis of PIP-Processed SI-C-N/Nicalon™ Tubes

The specimen with a measured strength of 182 MPa is shown in Fig. 1. Figure 1A shows the pressurized viscoelastic media that flowed radially out of fractured tube. The single failure path is parallel to the length of the tube. Figure 1B is the end view of the left side of the tube in Fig. 1A. The magnified view (Fig. 1B) shows the elliptical holes on each side of the fracture caused by the pullout of the 45° aligned fiber tows. The elliptical voids were found on both tested tubes where the fracture path intersected with the tube ends. These voids were found only at these sites.

The second high-temperature tested PIP-processed Si-C-N/Nicalon™ specimen, with a measured strength of 237 MPa, is shown in Fig. 2. In Fig. 2, the tube shows a slight diametral bulge. This deformation is visual evidence of high-temperature ductility. It can be hypothesized that crack arrest would have resulted had the tested tube been longer in length because of a decreasing stress intensity field. Though the strain before failure was not measured, high-temperature strain sensors could be incorporated.

Figure 2 is a typical fracture (seen on two tested specimens) in which individual fibers and fiber tows are exposed. The 45° aligned fiber architecture is clearly shown in Fig. 2B. The fiber tow pull-out length is estimated to be one half of the unit cell used for braiding. In Fig. 2, the fiber tow pull-out length is longer at the tube end. This was observed on both test specimens. It is believed that (a) plane stress conditions or (b) a reduction of shear coupling between the fiber and matrix near the tube end, or (c) both (a) and (b) are possible causes for the difference in fiber tow pull-out lengths. As indicated earlier, the second specimen tested at high temperature also exhibited localized elliptical holes (Fig. 3) where the fiber tows were pulled out of the Si-C-N matrix at the fracture site.

Room Temperature Hoop Tensile Strength of RBSN/SCS-6 Tubes

Two tubular ring specimens were pressurized to failure at room temperature and the results are summarized in Table 2. In the first test (Specimen 1074-1), since the internal pressure to cause failure was found to be questionably high, the measured hoop tensile strength value is also debatable. The uncertainty of this measured pressure is caused by a high back pressure at the supply reservoir, caused by a flow restriction from the undersized reservoir ports when a high loading rate is required on the reservoir. Conceivably, this back pressure is greater than the actual hydrostatic pressure on the specimen when there are leaks at the specimen seals. The leak at the specimen seals were expected because the test specimens were slightly noncircular.

Modifications were made to the test mandrel before conducting the second test to alleviate the back pressure by eliminating the flow restrictions. The viscosity of the pressurizing media was also increased to improve the sealing integrity. In the second test (Specimen 1072-2), the determined hoop tensile strength was 96.9 MPa (14.1 ksi).

TABLE 1—*Compilation of tensile strength for Nicalon™/SiC material systems found in the literature compared to the hoop tensile strength measurements using pressurized tubes* [1].

Process [Ref]	Test Method	Lay-Up Angle	UTS, MPa	Temperature, °C
PIP SiC [*1*]	pressurized tube	$[\pm 45°]_{2S}$	182.237	1000 vac
CVI[a] SiC [*7*]	flat coupon	$[\pm 45°]_{2S}$[b]	191.7 ± 2.1	RT
FCVI[c] SiC [*8*]	flat coupon	$[0/45/45/0]_3$[d]	175 ± 30	RT
FCVI SiC [*8*]	flat coupon	$[0/45/45/0]_3$[d]	180 ± 7	1000 air
ICVI[e] SiC [*8*]	flat coupon	$[0/45/45/0]_3$[d]	163 ± 35	RT
ICVI SiC [*8*]	flat coupon	$[0/45/45/0]_3$[d]	159 ± 18	1000 air

[a]Each lay-up angle refers to one ply of 2-D 8-harness satin weave of Nicalon™ fabric.
[b]Each lay-up angle refers to one ply of 2-D plain weave of Nicalon™ fabric.
[c]Chemical vapor infiltration.
[d]Forced chemical vapor infiltration.
[e]Isothermal chemical vapor infiltration.

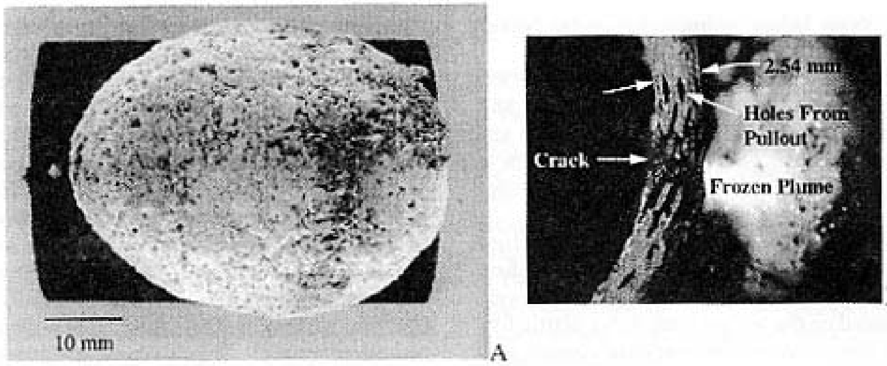

FIG. 1—*(A) Tube-side view of the frozen plume of the viscoelastic pressurizing media that has flowed out of the split open tube. (B) Magnified tube end view indicating the residual elliptical holes from the pulled-out fiber tows. The pressurized test temperature was 1000°C in vacuum.*

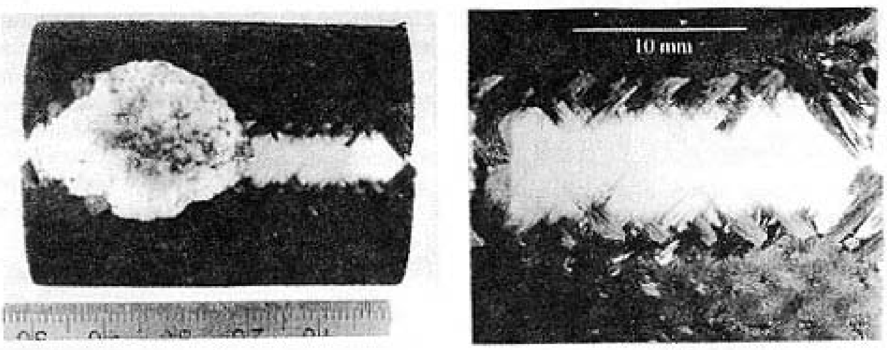

FIG. 2—*View of the split tube that failed at a hoop strength of 237 MPa. The pull-out length of the fiber tows are notably longer at the tube end (Fig. 2B is an enlarged view of 2A). The split tube also shows a slight bulge or aneurysm.*

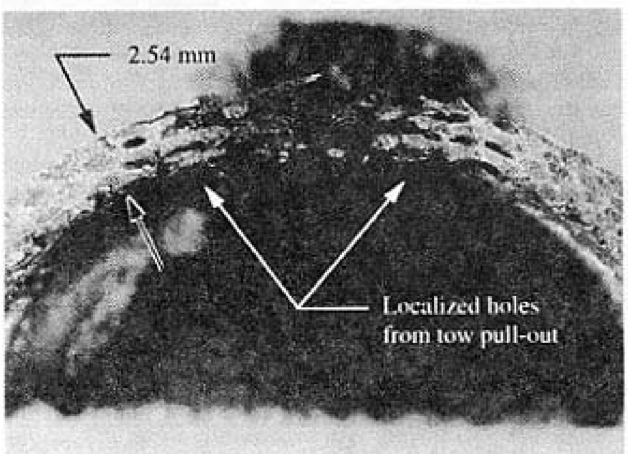

FIG. 3—*End view of the pulled-out 45° aligned fiber tows indicating elliptical holes in the Si-C-N matrix. Hoop strength is 237 MPa at 1000°C.*

TABLE 2—*Tabulation of room temperature hoop tensile test for RBSN/SCS-6 tubes.*

	Specimen Number	
	1074-1	1074-2
Tube length, mm	36.0 ± 0.5	36.6 ± 0.2
Max ID, mm	91.4	92.1
Min ID, mm	90.5	91.1
Mean thickness, mm	2.93	2.96
Max Pressure, MPa	≤13.7[a]	6.27
Hoop strength, MPa	≤213[a]	96.9

Failure Analysis

Specimen 1074-1 is shown in Fig. 4, and Specimen 1074-2 is shown in Fig. 5. As shown, the angle of the fracture plane for both test specimens is 45°. Both tubes exhibited one fracture. As expected, the amount of audible noise heard during loading increased as the pressure increased. For Specimen 1074-1 (Fig. 4), the quantity and length of the pulled out fibers is low because the fracture surfaces came in contact with one another after the specimen was removed from the test mandrel. After removal, the specimen was wedged open to prevent tube closure. For Specimen 1074-2 (Fig. 5), several pulled-out fibers were broken while removing the specimen from the test fixture. Some of the fibers in the pressurized gage section of the specimen were missing because the flowing pressurized media from the test mandrel broke the fibers.

The amount and length of pulled-out fibers is evident in Figs. 4 and 5 in which a silhouette was made by back lighting the specimen. Both Figs. 4A and 5A indicate delamination of the laminate ply. From an end view of these tube specimens, cracks are aligned with the laminate plies (Fig. 6). One crack extended

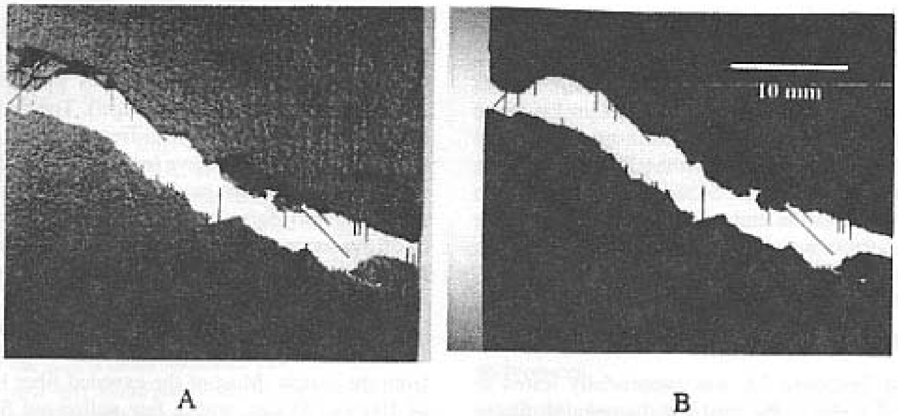

FIG. 4—*Optical fractograph of Specimen 1074-1 with a 45° initiate fracture plane. Fiber pull-out is predominately the 0° (circumferential) aligned fibers. The shadowgram (Fig. 4B) is shown to emphasize the fiber pull-out length.*

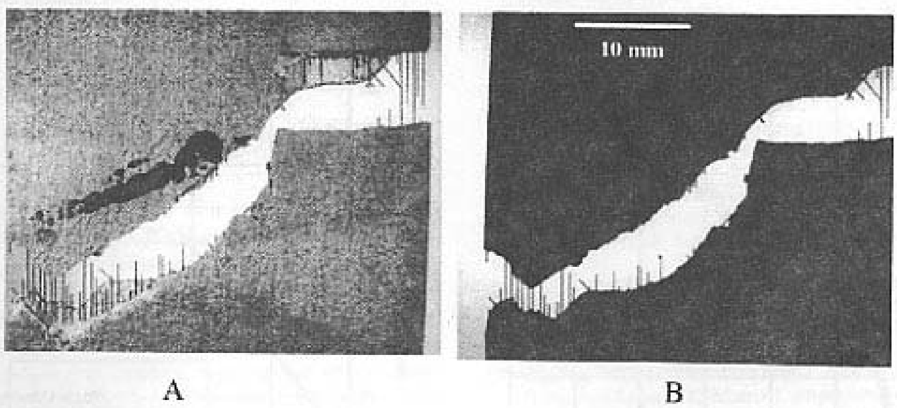

FIG. 5—*Optical fractograph of Specimen 1074-2 with a 45° fracture plane. Fiber pull-out is dominated by the pull-out of the 0° aligned fibers. The shadowgram (Fig. 5B) is shown to emphasize the length of fiber pull-out.*

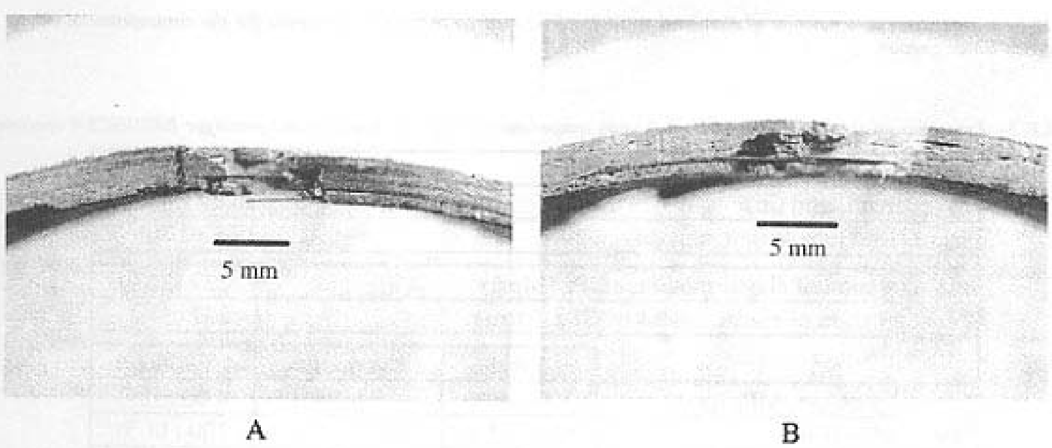

FIG. 6—*End views of Specimen 1074-1 and 1074-2, respectively, in which a delamination ply crack is aligned with the laminate plies.*

approximately 12 mm in length. From optical inspection, few elliptical holes are evident on the end of the tube. These holes were created by the pullout of the ±45° aligned fibers. It appears that delamination of the layers is principally found on the plane of the fiber's center line. The delamination crack may be caused by the reduced net cross-sectional area of the RBSN matrix to accommodate the large diameter of the SCS-6 filaments. Because delamination was detected at the end of the specimen, it is not certain whether edge effects were minimized or if they were an artifact after failure.

Hoop Tensile Stress-Diametral Strain of RBSN/SCS-6 Tube

The cylindrical test Specimen 2A was successfully tested to failure. The resulting *X-Y* plot of the load and diametral displacement was digitized and analyzed to obtain the resulting hoop tensile stress-diametral strain plot (Fig. 7). The physical dimensions and summary of the mechanical properties of Specimen 2A are given in Table 3. The yield strength (using the deviation from linearity as the criteria) was determined to be 84.8 MPa (12.3 ksi) at a yield strain of 0.031%. The ultimate hoop tensile strength was determined to be 162 MPa (23.6 ksi) at a corresponding strain of 0.202%. The failure strength was determined to be 100 MPa (14.5 ksi) with a strain to failure of 0.280%.

The range of the elastic modulus was determined to be from 227 to 237 GPa (32.9 to 34.4 Mpsi). The range of the moduli was dictated by the choice of data from the linear portion of the hoop stress-diametral strain curve (enlarged view of linear elastic portion of Fig. 7). The lower modulus was determined from the third to the fourteenth data point. The higher modulus was determined from the second to the eighth data point. The straight lines from the linear regression for the elastic modulus are shown as solid and dashed lines.

The tube failed at a 45° angle plane (parallel with the 45° angle fiber lay-up). Optical inspection indicated short fibers extending from the matrix. Most of the exposed fiber length is on the order of 100 to 200 μm, with a few pulled-out fibers on the order of 500 to 700 μm in length. Optical inspection also indicated a single 10+ mm long fiber, aligned at 45°, where this fiber could have been the main contributor to failure as the strength-limiting flaw. Long (10+ mm) secondary cracks seen to emanate from the primary crack. This would suggest that the addition of another layer of 0° aligned fibers may be beneficial to decrease the likelihood of a 45° angle shear plane failure.

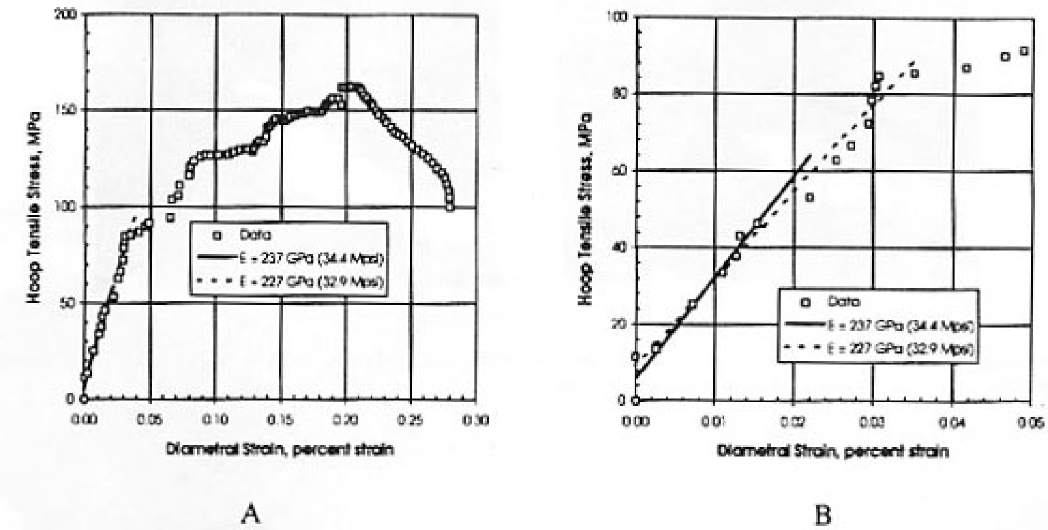

FIG. 7—*(A) Hoop tensile stress as a function of diametral strain for Specimen 2A to obtain an estimate for the circumferential elastic modulus. (B) Enlarged view of the linear portion.*

TABLE 3—*Tabulation of specimen dimensions and room temperature properties of the second prototype RBSN/SCS-6 specimen.*

Inside diameter, mm (in)			90.2 (3.55)
Tube length, mm (in)			36.2 (1.43)
Pressurized tube length, mm (in)			17.8 (0.70)
1st Regression of elastic modulus, GPa (Mpsi)			227 (32.9)
2nd Regression of elastic modulus, GPa (Mpsi)			237 (34.4)
	Yield	Maximum	Failure
Internal pressure, MPa (psi)	3.44 (499)	6.59 (956)	4.06 (589)
Stress, MPa (ksi)	84.8 (12.3)	162 (23.6)	100 (14.5)
Strain, % strain	0.031	0.202	0.280

Summary

High Temperature Hoop Tensile Strength Testing of PIP-Processed Si-C-N/Nicalon™ Tubes

The prototype braided Nicalon™ fiber CMC tubular components using the PIP process to make the Si-C-N matrix were tested to failure successfully at a high temperature using a pressurizing media that exhibited viscoelastic behavior at elevated temperatures. The measured hoop tensile strengths were 182 and 237 MPa (26.4 and 34.5 ksi), respectively. The strength measurements were comparable to flat tensile coupon tests (found in the literature), however, the tube test gage volume in tension is considerably larger.

Failure analysis of the tested tubes indicated fiber tow pull-out. The pulled-out fiber tow left localized holes in the matrix. The pull-out length of the longer fiber tows, localized at the tube ends, indicated edge effects where the fiber tow was not as well constrained. Since ductility was indicated by the observed aneurysm-type bulge of the tube, ductility can be engineered into structural ceramics.

By internally pressurizing tubes, the failure mode represents real components—combustion chambers, heat exchangers, or even large flat panels. The tubular test specimen configuration allows a greater test gage volume to be subjected to a tensile load to support design data generation.

Room Temperature Hoop Tensile Strength Testing of RBSN/SCS-6 Tubes

The first prototyped RBSN/SCS-6 tube indicated a minimum hoop tensile strength of 96.9 MPa (14.0 ksi) at room temperature. Optical fractographic analysis determined extensive fiber pull-out, sometimes exceeding 5 mm. Fiber pull-out was mostly dominated by the 0° aligned fibers as compared to the 45° aligned fibers.

The second RBSN/SCS-6 tube was tested to failure at room temperature and monitored to obtain a hoop tensile stress versus diametral strain plot. The yield and maximum tensile strength were measured as 84.8 and 163 MPa (12.3 ksi and 23.6 ksi), respectively. The elastic modulus from the hoop tensile stress versus diametral strain gave a range of 227 to 237 GPa (32.9 to 34.4 Mpsi). Optical analysis indicates mostly short fiber pull-out (100 to 200 μm) on the 45° shear fracture plane.

Acknowledgments

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