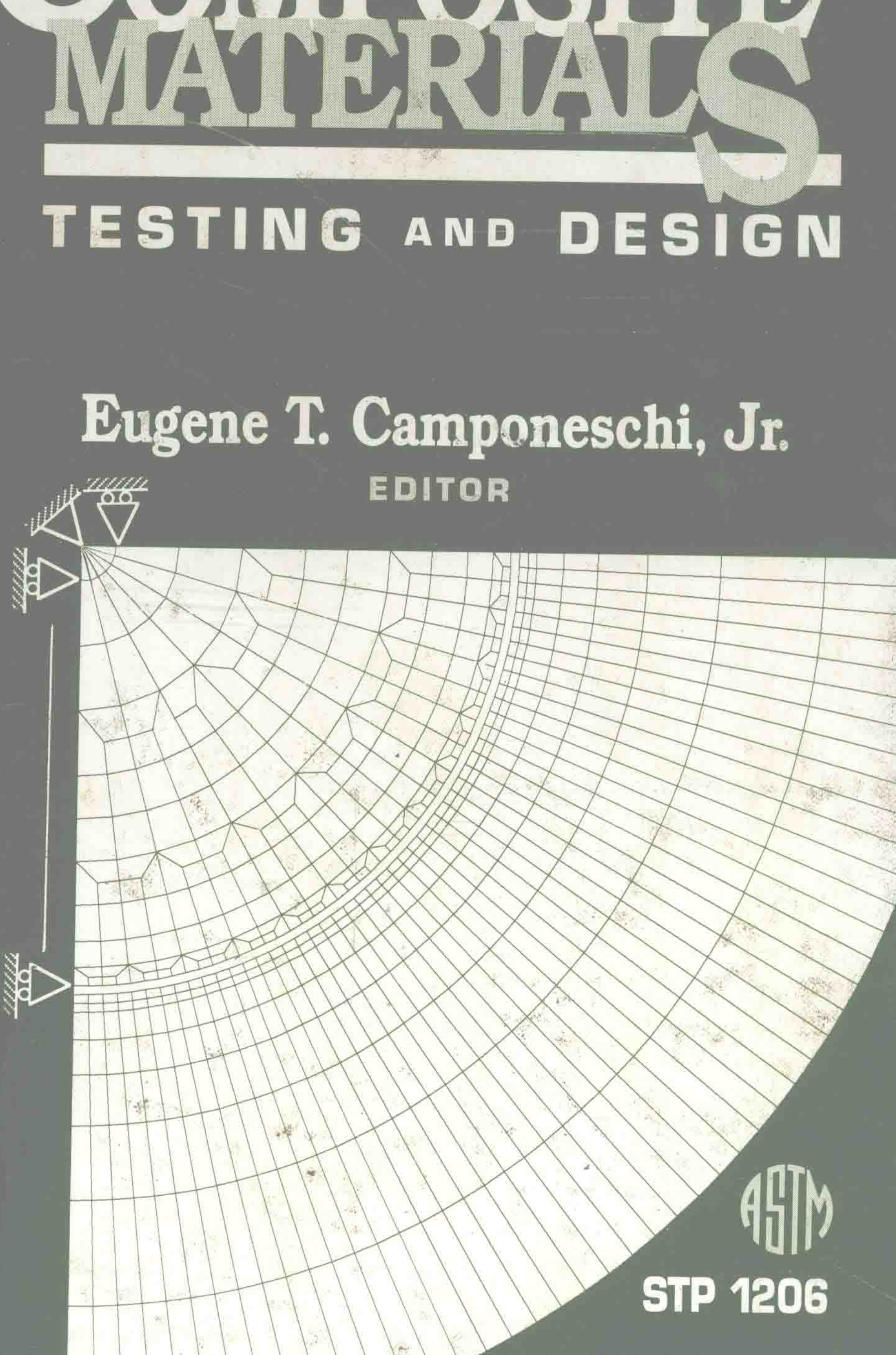
Eleventh Volume

COMPOSITE MATERIALS

TESTING AND DESIGN

Eugene T. Camponeschi, Jr.

EDITOR



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Composite Materials: Testing and Design— Eleventh Volume

Eugene T. Camponeschi, Jr., editor

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Peer Review Policy

Each paper published in this volume was evaluated by three peer reviewers. The authors addressed all of the reviewers' comments to the satisfaction of both the technical editor(s) and the ASTM Committee on Publications.

The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution to time and effort on behalf of ASTM.

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Foreword

This publication, *Composite Materials: Testing and Design—Eleventh Volume,* contains papers presented at the 11th Symposium on Composite Materials: Testing and Design, held in Pittsburgh, PA on 4–5 May 1992. The symposium was sponsored by ASTM Committee D–30 on High Modulus Fibers and Their Composites. Eugene T. Camponeschi, Jr., Carderock Division, Naval Surface Warfare Center (formerly the David Taylor Research Center), presided as symposium chairman and is editor of the resulting publication.

Contents

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Overview

The ASTM Eleventh Symposium on Composite Materials: Testing and Design was held on 4–5 May 1992 in Pittsburgh, Pennsylvania. As with the ten previous symposia of the same title, papers for the eleventh were solicited in the general area of testing and design of composite materials. Twenty-two of the twenty-nine papers presented at the symposium are included in this publication and fall into the three major categories of Materials Testing and Response, Design and Analysis, and Interlaminar Fracture and Strength.

Material Testing and Response

Twelve of the twenty-two papers in this volume fall in this category and are briefly described below.

Transverse Tension and Shear Properties

Four papers deal with the matrix dominated properties of transverse tension and shear. Lyon et al. present shear, transverse tension, and transverse compression data from unidirectional hoop-wound tubular specimens and cylindrical rods fabricated from wet-filament winding and prepreg systems. O'Brien and Salpekar discuss the influence of volume on the transverse tensile strength of carbon-reinforced composites and use transverse tensile strength data and Weibull statistics to predict the strength of 90° laminates loaded in three-point bending. Chatterjee et al. investigated the shear response of composite laminates using solid circular and rectangular bars, with Ho et al. investigating the same using V-notched beam specimens.

Compression Test Methods

In area of compression testing, two papers compare different compression test methods. Goeke discusses two test methods that can be used for evaluation of composite materials greater than 12 mm (0.5 in.) in thickness, and Daniels and Sandhu discuss six test methods and specimens for the evaluation of materials for more common specimen thicknesses (~2.5 mm [0.1 in.]).

Multiaxial Stress State Response

Crews and Naik evaluate the multiaxial ply strength of carbon-reinforced composites using an off-axis flexure test of unidirectional laminates. Wang and Socie present results from the multiaxial testing of fiberglass fabric-reinforced tubular specimens. In their work, loading is provided by varying combinations of tension, compression, internal pressure, and external pressure.

Other Properties

The four remaining papers in the category of Material Testing and Response address the following: deriving the elastic constants of nearly isotropic composites from modal vibration of a completely free plate (Ayorinde et al.), determining the high strain rate response (0/s to 3000/s) of composites using a high-strain rate drop tower and a split Hopkinson bar (Groves et al.), development of an elastic/viscoplastic constitutive model for in-plane, isothermal, axial tensile loading

(Gates), and the tension-tension thermomechanical fatigue response of titanium alumnide (Bartolotta and Verrilli).

Design and Analysis

This category contains six papers that range in level of focus from micromechanical analysis to the analysis of thick, laminated shells. At the micromechanical level, Naik and Crews used the classical Airy's stress function approach to compute fiber-matrix stresses for unidirectional carbon-reinforced composites under combined thermal and mechanical loading. At the level of laminate analysis, four papers are presented. The first two address the effect of holes in carbon-reinforced laminates with DiNicola and Fantle investigating the effect of clearance fit fasteners and Sorem et al. investigating the effect of hole interaction on stress concentrations. The other two papers on laminate level analysis address the design of laminates that exhibit optimum laminate extension-twisting coupling while considering laminate warping caused by residual thermal stresses (Armanios et al.), and the prediction of thermo-elastic properties of woven fabric composites in a deformed state such as those used for doubly curved surfaces (Laroche and Vu-Khanh). The final paper in this category is at the structural analysis level and deals with the behavior of thick-section composite shells subjected to hydrostatic pressure (Yuan).

Interlaminar Fracture and Strength

Four papers are included in this category. Three deal with delamination, and two of them focus on mixed-mode delamination and the mixed-mode bending (MMB) test method. This test method theoretically allows the evaluation of fracture toughness G_c under combinations of mixed-mode loading, from pure Mode I to pure Mode II. In the paper by Sriram et al., the MMB test is used to evaluate the fatigue response of two IM7 reinforced material systems. Reeder presents MMB data and introduces a bilinear failure criteria to characterize the data and account for different failure mechanisms observed on the delamination surfaces. The other paper on delamination considers the effect of various ply-drop configurations on delamination (Fish and Vizzini). The fourth paper in this category investigates curved beam specimens for the determination of interlaminar tension strength (Jackson and Martin).

Summary

In summary, the papers in this eleventh volume on Composite Materials: Testing and Design represent a significant contribution to the literature on testing, analysis, and design of composite materials. As with the previous ten volumes, the largest portion of the papers deals with testing and analysis of composites at the coupon level or below, with only a few papers dealing with design issues or analyses used specifically for a design. While this ratio is generally appropriate for the research-type symposia this STP covers, it is the editor's observation that earlier volumes of the same title (the First, Third, and Fifth) had a much higher percentage of papers describing testing and analysis with a specific design focus. It is also the editor's opinion that work on fundamental testing and analysis issues versus those with a design focus is a general trend in published literature at this time. While work to increase the level of understanding of fundamental mechanics issues is essential, the need to transition testing and analysis fundamentals into published design procedures is immediate.

In a time of defense conversion and the potential it provides for the composites industry, a large number of design engineers remain puzzled over the paradox between the thorough, fundamental understanding of testing and analysis procedures, and the lack of information on using these procedures in engineering design. It could be argued that it is difficult to establish simply used design procedures for complex, inhomogenous, anisotropic materials. While true, the testing, design, and analysis community can move further towards this end through the effective transition of design experience from the corporate knowledge base of the defense industry.

While design handbooks and manuals will be the eventual sources of such information for the design engineer, their publication is limited and far from complete. For the near term, engineers should consider the critical role they could play in the transition of research based knowledge to engineering design. A forum such as the ASTM series on Composite Materials: Testing and Design could specifically assist in this transition with future focus on (1) relating test data to working stress design allowables, (2) relating analysis results to design allowables through effective failure criteria, and (3) relating data, design allowables, and failure criteria to design factors of safety for safe yet efficient and cost-effective design.

A Final Note

Finally, the successful publication of this STP would not have been possible without the gracious assistance of many individuals, and I would like to thank the authors and reviewers for their contributions in this effort. For their help as my Symposia Session Chairmen, thanks go to Roger Crane, Scott Groves, Steve Lubowinski, and Kevin O'Brien. The talented staff at ASTM headquarters deserves a special thanks for flawlessly handling the mountains of paper and endless phone calls necessary for such a publication effort. The assistance and pleasant spirit of Therese Pravitz and Lynn Hanson was particularly appreciated.

Eugene T. Camponeschi, Jr.
Naval Surface Warfare Center
Carderock Division
Annapolis, MD 21402
Symposium chairman and editor

Material Testing and Response

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*R. E. Lyon,[1] D. L. Schumann,[2] and S. J. DeTeresa[2]**

Matrix-Dominated Mechanical Properties of a Fiber Composite Lamina

REFERENCE: Lyon, R. E., Schumann, D. L., and DeTeresa, S. J., "**Matrix-Dominated Mechanical Properties of a Fiber Composite Lamina,**" *Composite Materials: Testing and Design (Eleventh Volume), ASTM STP 1206,* E. T. Camponeschi, Jr., Ed., American Society for Testing and Materials, Philadelphia, 1993, pp. 7–22.

ABSTRACT: Matrix-dominated mechanical properties of unidirectional fiber composite laminae were determined from hoop-wound tube specimens and cylindrical rods fabricated from both wet-filament winding and prepreg material systems. Longitudinal shear modulus and strength as well as transverse Young's modulus, transverse tensile strength, and transverse compressive strength were obtained from a thin-walled tube specimen using a new fixturing design. Lamina properties are presented for several carbon fiber/epoxy composite materials. Longitudinal shear moduli were measured for both tubes and rods in torsion. Results obtained in the linear-elastic regimes above and below the glass transition temperature (T_g) of the matrix phase were compared with micromechanics predictions. Although agreement between predicted and measured shear moduli was reasonable below T_g, for some composites large discrepancies were observed at temperatures above T_g of the neat matrix material.

KEYWORDS: fiber composite lamina properties, matrix-dominated lamina properties, torsion of tubes and rods, transverse properties, micromechanics

Background

A review of current static shear methods for composites [*1–4*] indicates a predominance of bending or uniaxial deformation tests requiring specimens cut from flat plates or prismatic bars, i.e., Iosipescu, panel shear, off-axis tension or compression, short beam shear, etc. None of these tests produces a state of uniform, simple shear in the specimen—making values for shear stiffness and strength determined by these methods potentially unreliable. For example, different correction factors have been proposed for reducing the modulus data obtained from Iosipescu tests on 0° versus 90° layups [*5*]. In contrast, torsion of thin-walled composite tubes is known [*1*] to produce a state of uniform shear if the ratio of the mean tube radius, R, to wall thickness, t, is greater than about 10, i.e., $R/t \geq 10$. The in-plane, longitudinal shear modulus, variously denoted G_L, $G_\parallel$, or G_{12}, can be calculated from the torsional rigidity of a unidirectional tube in which all of the fibers are oriented in the axial (0°) or transverse (90°) direction relative to the tube z-axis. In the subscripted notation, fiber direction is taken to be coincident with the 1–principle material direction while the 2,3 directions define the transverse plane perpendicular to the fibers in the assumed orthotropic

[1]Federal Aviation Administration, FAA Technical Center, Atlantic City International Airport, NJ 08405.
[2]University of California, Lawrence Livermore National Laboratory, Livermore, CA 94550.

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*To whom all correspondence should be sent.

where M_t is the applied torque and R the average of the inner and outer tube radii. The in-plane or axial shear strength, S, is calculated from Eq 1 with the measured failure torque and tube dimensions of hoop- or longitudinally-wound tubes, i.e., $S = \tau_{LT}^{\text{ultimate}}$.

Although torsion of thin-walled tubes is used as a calibration method for in-plane shear test methods, it is often claimed that specimens are expensive to fabricate and require sophisticated testing equipment [*3*]. However, hoop-wound tubes prepared by wet-filament or prepreg tow winding are relatively inexpensive to fabricate and are expected to have properties that are representative of individual lamina in multilayer filament-wound structures processed under similar conditions. Use of the same tube specimen and basic fixturing for transverse tension or compression testing would further improve test economics by providing much additional data at minimal cost. In this paper we present axial shear strength and stiffness data as well as transverse tensile/compressive strength and stiffness data for wet-filament wound and prepreg tape wrapped 90° tube specimens representing several materials systems, using a previously published test methodology [*4*]. Additionally, we compare the thin-walled tube results to those obtained from the torsion of unidirectional composite rods. Although this test method generates a nonhomogeneous stress/strain state, for transversely isotropic materials the distribution is simple and amenable to a straightforward determination of shear modulus from torque-twist data.

Materials

The physical properties of the matrix and fiber materials used in this study are listed in Tables 1 and 2. The properties of several of the filament-winding resins have been previously characterized in detail [*6–9*]. Resin pot life was determined from the time to doubling of the initial mix viscosity.

TABLE 1—*Physical properties of matrix resin systems.*

Resin/ Hardener (mix ratio in parts by weight)	Initial Viscosity (Poise)	Pot Life (h)	Cure Cycle (°C/h)	Cured Density (g/cc)	Glass Transition Temp. (°C)	Young's Modulus (GPa)	Compressive Yield Strength (MPa)
DER332[a]/T403[b] (100/45)	8	3	60/4 + 95/2	1.15	85	2.9	83
DER332 + RD2[c]/MNDA[d] (90 + 10/27)	5	10	65/18 + 135/4	1.16	135	2.9	124
DER332 + RD2/DETDA[e] (90 + 10/26)	11	35	150/4	1.16	150	2.5	108
RD2/T403 (100/56)	0.7	11	60/24	1.15	−5	0.01	—
CY179[f]/ MTHPA[g] + CTBN[h] + 1MI[i] (100/100 + 10 + 1)	4	14	90/2 + 150/4	1.20	200	2.9	124
MY0510[j]/HY350[k] (100/ 56)	14	30	100/2 + 150/2 + 175/2	1.21	185	4.5	190
Hercules 8551-7A	(prepreg)	—	135/1 + 180/2	1.28	175	3.6	138

[a]Bisphenol A epoxy (Dow Chemical).
[b]Polyoxypropylene triamine (Texaco Chemical).
[c]Diglycidyl ether of butane diol (Ciba-Geigy Corp.).
[d]Menthane diamine (Rohm & Haas).
[e]Diethylene toluene diamine.
[f]Cycloaliphatic epoxy (Ciba Geigy).
[g]Methyl-tetrahydrophthalic anhydride (Anhydrides and Chemicals).
[h]Carboxy-terminated butadiene-nitrile rubber, (Hycar 1300x8 CTBN B. F. Goodrich).
[i]1-methylimidazole.
[j]Triglycidylether of p-aminophenol (Ciba-Geigy).
[k]Methylene bis-o-ethylaniline (Ciba-Geigy).

TABLE 2—*Physical properties of carbon fibers.*

Manufacturer	Fiber	Diameter (μm)	Density (g/cc)	Young's Modulus (GPa)	Tensile Strength (GPa)
Hercules	IM7	5	1.77	276	5.41
Toray	T-1000G	5	1.80	294	6.37
Hitco	Hitex 46-9A	6.1	1.81	317	6.46
Courtaulds	Grafil 33-500	7	1.83	228	3.79
BASF	Celion G30-500	7	1.78	234	3.79

Densities were measured by hydrostatic weighing in mineral oil. Glass transition temperatures (T_g) were obtained from dynamic mechanical analysis using a Rheometrics RMS-800 mechanical spectrometer, and Young's modulus (E_m) and compressive yield strengths (σ_c) were measured in static compression tests following ASTM D 695 (Test Method for Compressive Properties of Rigid Plastics) procedures using right circular cylinders conditioned according to ASTM D 618 (Method for Conditioning Plastics and Electrical Insulating Materials for Testing). To investigate the effect of drastic changes in matrix properties on lamina properties and the ability of currently accepted micromechanics models to predict such changes, a rubber matrix system, RD-2/T-403, was used in combination with T1000G fiber. This matrix has a T_g that is 25°C below room temperature and exhibits the typical low modulus (ca. 10 MPa) and elastic behavior of a rubber at temperatures well above this transition point.

The 8551-7A prepreg system is based on a toughened epoxy. It was fabricated into 12-in. (30.5-cm) wide tape by Hercules using their high-strength IM7 carbon fiber. The resin properties were measured using specimens supplied by the manufacturer.

The carbon fibers used in combination with both filament winding and prepreg resins are all based on poly(acrylonitrile) (PAN) precursor. With the exception of the Hitex 46-9A fiber which is only available as a 6000 (6k) filament tow, all fibers were supplied in 12k tows. According to manufacturer's data, the tensile moduli of all the fibers is in the ''intermediate'' range, but tensile strengths vary from intermediate (3.8 GPa) to high (6.5 GPa). Fibers were used with manufacturer's sizing.

Experimental Methods

Sample Preparation

Unidirectional, hoop-wound (90°) composite tubes were produced by both wet-filament winding and by circumferential wrapping of prepreg tape. Wet-filament winding of hoop-wound tube specimens was performed on a McCLean-Anderson W90, 6-axis filament winding machine. The epoxy impregnated tow was passed through a wire-drawing die having an orifice diameter calculated to control wet resin pickup to approximately 45% by volume. The impregnated tow was wound onto a 2.54-cm diameter by 1-m long polished, cylindrical, aluminum mandrel that had been treated with a release coating (Release-All RA-100, Airtech Co.). Tow tension during winding was maintained at $\approx$10 N at the mandrel. A bandwidth setting of 2.79 mm was used, resulting in an actual wrap angle of $\pm$88.7° (i.e., $\pm$1.3° off of the tube long axis normal). Tubes were traversed 16 times to a final outside diameter of 3 cm, corresponding to a wall thickness of $\approx$2 mm. After winding, wet tubes were rotated while cured in a circulating air oven according to the schedules listed in Table 1. This fabrication procedure was found to produce high-quality tubes containing $\leq$1% voids. After curing, the aluminum mandrel was easily removed from the inside of the hoop-wound tube by placing the assembly in a freezer to use the differential thermal expansion between winding, wet tubes were rotated while cured in a circulating air oven according to the schedules listed in Table 1. This fabrication procedure was found to produce high-quality tubes containing ≤1% voids. After curing, the aluminum mandrel was easily removed from the inside of the hoop-wound tube by placing the assembly in a freezer to use the differential thermal expansion between the aluminum and the fiber-dominated hoop direction of the composite. No visible transverse cracking was noted on any of the tube specimens from either the mandrel removal process or the z-axis mismatch in thermal expansion.

Prepreg tape wrapping of 90° unidirectional tubes was contracted to Advanced Reinforced Tubing Co., Carson City, NV. The commercial 12-in. (30.5-cm) wide Hercules IM7/8551-7A prepreg tape was circumferentially wrapped onto 2.54-cm-diameter steel mandrels, and the manufacturer's recommended autoclave cure cycle was followed. The resulting unidirectional composite tubes were 1 m long and, in contrast to wet-filament wound tubes, contained noticeable fiber waviness as a result of buckling during the autoclave compaction process.

Cylindrical composite rods having unidirectional fiber orientation were fabricated from the filament winding resin systems and several of the PAN-based fibers listed in Table 2. Spools of dried fiber were vacuum-impregnated and then drawn through circular wire-drawing dies to remove most of the excess resin. The removal of the remaining excess resin and the shaping of the rods into right circular cylinders was achieved by first threading one or two impregnated strands through poly(ethylene) (PE) heat-shrink tubing and then collapsing the tube around the strands using a standard heat gun. Rods were cured under minimal tension in the PE tubing according to the schedules given in Table 1. After cure, the PE tubing was carefully cut away using sharp single edge razor blades. No visible damage of the cured composite rod occurred during this cutting step. Rods produced in this manner were of both uniform dimensions and shape. Diameters were approximately 1 mm and varied by less than 2.5% over 20-cm lengths. Some control over the final volume fraction of fiber in the rods could be attained by using different size PE tubes and by threading more than one strand in a tube. Fiber volume fractions ranged from 50 to 70% and void volume fractions were essentially negligible.

Test Specimen Design and Fixturing

Preliminary investigations of various gripping configurations for determining the torsional shear strength of hoop-wound graphite/epoxy tubes showed that approximately 50% of shear failures occurred at the grips when straight-walled tubes were gripped using internal or external mechanical pressure from expanding or contracting collets. Moreover, the specimens were prone to slipping except at the highest mechanical pressures. This problem was partially eliminated by switching to an adhesive bond to grip the tube, whereby a urethane adhesive was used to bond a close fitting, 25-mm-diameter aluminum plug with a flatted shaft to the inside of the straight-walled composite tube. However, the process of adhesive bonding was slow and messy—each specimen requiring one hour in a precision alignment fixture to set the adhesive. In addition, a significant amount of machining was required to fabricate the aluminum plugs with the 1.25-cm-diameter flatted shaft for gripping in the torsion chucks of the testing machine. Due to these constraints it was decided to investigate direct machining of the composite tube to obtain a test specimen with a reduced gage section, in the hope that failures could be made to occur exclusively in the gage section region of high stress.

Specimens 10 cm in length were cut from the 1-m-long composite tube for subsequent machining. Reduced diameter gage sections were ground from the mid-section of the specimens using a lathe equipped with a counter-rotating diamond grinding wheel. Using a double pass grinding operation, approximately 5 to 10 specimens/hour could be fabricated. Outside gage section diameter was held to 2.692 ± 0.005 cm resulting in a nominal wall thickness, t = 0.762 mm, and an acceptable radius to thickness ratio of R/t = 17. A gage section length of 3.175 cm was obtained with the specified 1.3-cm radius. A centered 6.35-mm (¼-in.) hole was drilled 1.27 cm in from the end of the specimen in both grip sections for fixturing. Surface roughness on the ground gage

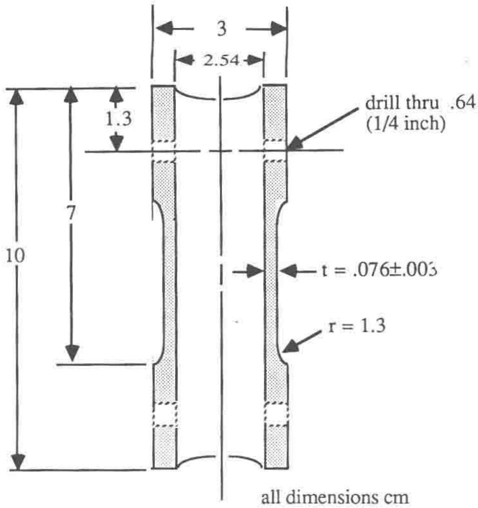

FIG. 1—*Cross-section of hoop-wound composite tube torsional shear and transverse tension/compression specimen.*

shafts as shown in Fig. 2. The drawing illustrates how the drilled composite tube is captured between the internal plug and the steel, semicircular, split collars. External pressure is supplied by the 6.35-mm (¼-in.) diameter bolt which connects the split collars through the composite specimen and plug at each end. In practice, it was found that knurling of the inner surface of the steel collars (or sand paper) was required to prevent slipping of the specimen in the grips during both torsion and tension testing.

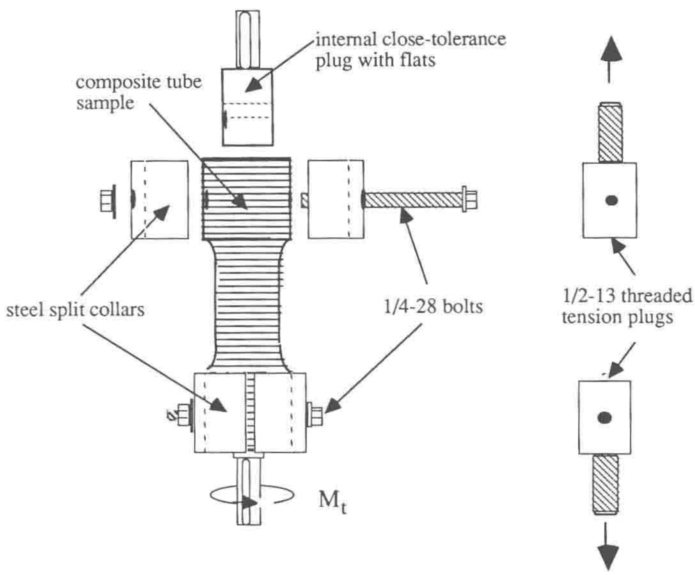

FIG. 2—*Fixturing for torsional shear and transverse tension.*

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