ACDAP REPORT 1
JUNE, 1971

TEST METHODS FOR ADVANCED COMPOSITES

ADVANCED COMPOSITES DATA AND ANALYSIS PROGRAM
Lockheed Georgia Company
Marietta, Georgia 30060

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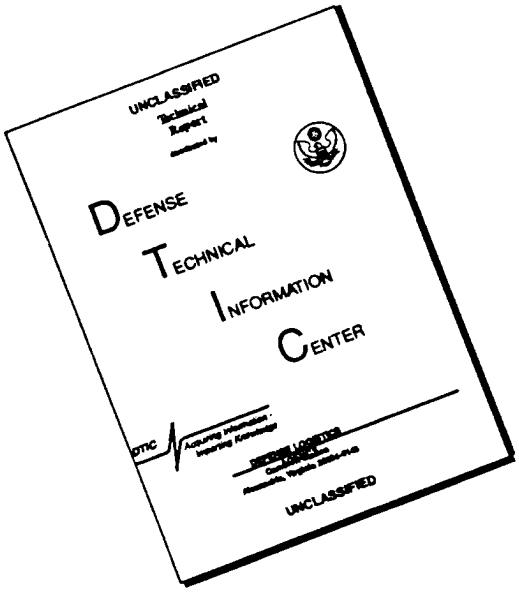

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A. L. Cunningham

FOREWORD

This report was prepared by the Lockheed-Georgia Company under Contract Number F33(615)-70-C-1486, Project No. 6169CW, for the Advanced Composites Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio. Mr. R. M. Neff (AFML/LC) is the Project Engineer for the Air Force and Mr. W. G. Jurevic is the Program Manager for the Lockheed-Georgia Company.

This report is one of a series of four state-of-the-art status reviews of selected facets of advanced composite technology to be compiled under Phase I of the Advanced Composites Data and Analysis Program.

Note - Program is terminated only 4 reports put-out.

ADVANCED COMPOSITES DATA AND ANALYSIS PROGRAM

Lockheed Georgia Company
Marietta, Georgia 30060

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ABSTRACT

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2 of 2

TEST METHODS

FOR

ADVANCED COMPOSITES

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A Division of Lockheed Aircraft Corporation
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ABSTRACT

A review of current state-of-the-art test methods for evaluation of selected physical and mechanical properties of advanced composites is presented. The report includes constitutive and composite materials acceptance tests, design and structural element tests, and the recommendation, where possible, of specific test methods as accepted standards for current industry-wide use.

1.0 INTRODUCTION

The increased use of advanced composites for aerospace structural applications has placed major emphasis on the development of mechanical property test methods and standards for materials evaluation and the establishment of reliable design data. The fundamental characteristics of filamentary composite materials, namely, anisotropy, high-strength, and high-modulus introduce a number of special problems associated with mechanical test methods and structural design procedures. The effective design of any structure is predicated on mechanical property data which accurately reflect the specific in-use environment. Currently, however, there are few accepted mechanical test standards for advanced composites. The basic difficulty relates directly to the inherent limitations in the use of small test samples which simulate the actual full-scale, end-item structural demands and performance.

During the course of the past several years, concerted efforts have been directed toward the development of reliable mechanical and related physical property test methods for advanced composites to establish accepted industry-wide standards. Such efforts, however, have been confined to a limited range of mechanical properties which provide the designer with insufficient data. Major problems classically associated with these material types are nonuniform introduction of load into test specimens and the relation of composite microstructure to test specimen size and symmetry. The specific method of test specimen fabrication is also of prime importance and should be representative of the fabrication method or process utilized for the structure of interest.

The objective of this report is two-fold; namely, (1) the presentation of a detailed review of currently utilized test methods for characterization and evaluation of advanced structural composites and their respective constituents; and (2) recommendation, where possible, of specific test methods as accepted standards for industry-wide use. Criteria governing selection and recommendation of candidate test methods are reliability and accuracy, simplicity of apparatus and procedure, ease of test specimen preparation, and low cost. The advanced composites treated include the boron-epoxy, and graphite-epoxy systems.

Test methods are developed under the following principal sections and in the sequence cited; namely, Materials Acceptance, Design, and Structural Element Tests. When a specific test can be applied under two or more sections, it is cross-referenced as

required. Test methods and procedures detailed herein are applicable to both boron and graphite-resin composites except where explicitly indicated.

The Materials Acceptance Test Section considers constitutive and composite test procedures for low temperature, advanced composites. Since major emphasis is directed toward resin matrix composites, certain physical property tests have been included, for example, density, and thermal expansion. Density assumes primary significance in relation to reinforcement filament, yarn and tow tensile properties, constitutive volume fractions, and composite void content. The significance of thermal expansion and its relation to uniaxial and multiaxial composites is emphasized. To date, limited studies have been conducted on thermal expansion behavior of composite materials[1-4]. This property, however, directly influences the dimensional stability of composites as well as their strength properties and mechanical stability in structural assemblies.

The Design Test Section treats mechanical and physical property test methods solely directed toward defining the engineering properties of composites and establishing reliable design data. This section also includes a review of environmental tests for low temperature composite materials to further establish performance data requisite to effective design.

The final section of the report is confined to a concise review of basic structural element tests utilized for evaluation of filamentary composite subcomponents and components.

This report summarizes state-of-the-art mechanical and physical property test methods currently applied in the detailed characterization of advanced composite materials and their constituents and contains recommended procedures to be followed when evaluating such materials. It is consistent with the prime objectives of this task to utilize fully all currently available technical literature treating mechanical and related physical property test methods either designed for or applicable to the characterization and evaluation of advanced filamentary composite materials. A summary of test methods recommended herein is given in Table 1-1.

TABLE 1-1 - SUMMARY OF RECOMMENDED TEST METHODS

Materials Acceptance Tests				Design Tests			
Test	Test Method	Page No.	References	Test	Test Method	Page No.	References
Fiber Density	Picnometric	10	13,14	Uniaxial Tension			
Resin Density	Liquid Displacement	9	11,35	Coupon	Coupon (IITRI)	74	65,66,78,79
Composite Density	Picnometric	54	13,14	Sandwich Beam	Sandwich Beam	97	
Thermal Exp. (Resin)	Dialatometer	39	35,36	Transverse	Coupon (IITRI)	77	65,66,78,79
Thermal Exp. (Composite)	Dialatometer	39	1,2,3,35,36,63	Biaxial Tension	Sandwich Crossbeam	103	64,79,83,84
Fiber Tensile Strength (Graphite)	Strand	21	23,24,25	Uniaxial Compression	Sandwich Beam	107	64
Fiber Tensile Strength (Boron)	Single Filament	28		Biaxial Compression	Sandwich Crossbeam	103	64,79,83,84
Fiber Modulus	Compliance	23	23,24,25,28	In-Plane Shear	Rail Shear	114	96,78
Resin Tensile Strength	Coupon	39	38,39,40,41	Impact	Izod	121	101,64
Resin Compressive Strength		41	43,44,45,38	Bearing Strength	Pin Bearing, Bulk Bearing	124	71,103
Resin Flexural Strength	Coupon (Four Point Loading)	41	46	Fatigue (Axial Loading)	Coupon (IITRI)	131	104
Prepreg Tack	Plate	49	49	Fatigue (Spectrum Loading)	Sandwich Beam	137	104
Prepreg Resin Flow	Platen	50	49	Creep	(IITRI-Type Coupon Specimen)	142	105,106
Prepreg Gel Time	Platen	51	49	Crack Propagation	Pulsating Tensile Stress	158	104
Prepreg Volatiles	Partial Ignition	51	49				
Prepreg Resin Content	Chemical Digestion	52	49,51				
Prepreg Shelf Life		53	49				
Composite Fiber V/O*	Ignition	55	38				
Composite Resin V/O	Resin Burn-out	56	38				
Composite Void V/O	Resin Burn-out	56	38,56				
Composite Tension	Coupon (IITRI)	74	65,66				
Composite Tension (Transverse)	Coupon (IITRI)	77	65,66				
Composite Compression	Coupon	83	66,67				
Composite Flexural	Coupon (4 Point)	41	46				
Composite Shear	Short Beam	91	66,67				

*High Modulus Graphite

2.0 MATERIAL ACCEPTANCE TESTS

2.1 GENERAL

Test methods described in this section are confined to a review of mechanical and physical property tests commonly utilized for quality control and acceptance of advanced filamentary composites and their constituent materials. The majority of test procedures discussed herein are translatable neither to the development nor establishment of design data for advanced composite structures.

In the development of constitutive test methods, emphasis has been largely limited to the evaluation of the mechanical and physical properties of the reinforcement; e.g., boron and graphite. Since the strain rate dependence of filament-reinforced epoxy is strongly influenced by the resin per se, it is apparent that a similar emphasis should be directed toward the detailed characterization of the matrix resin. Tensile, compressive, and shear properties of the epoxy should be determined at varying strain rates, for example, 0.005, 0.05, and 0.5 in/in/min. at both ambient and elevated temperatures. Thermal expansion characteristics of the resin is also of prime importance and should be determined at specified temperatures within the projected use temperature range.

In view of the increased availability and use of commercial monolayer composite prepreg tapes, an abbreviated treatment of resin prepreg evaluation and acceptance test procedures is included. With the availability of high quality commercial fiber-resin prepreg, greater emphasis is now placed on the required development of standardized, quality control and acceptance tests for advanced composites.

2.2 REINFORCEMENTS

2.2.1 Physical Properties

In any comprehensive summary of test methods applicable to characterization and analysis of filamentary composites, certain physical property tests should be included; namely, density and thermal expansion. Density assumes prime significance relative to its use in determining reinforcement yarn and tow tensile properties as well as composite constituent and void content volume fractions. Thermal expansion behavior of composites and their constituents must be considered in composite design and utilization since this property directly affects their dimensional and mechanical stability in structural assemblies.

2.2.1.1 Density

Density measurements may be performed by any convenient method which yields accurate and reproducible results. The most common techniques currently used for determining the density of filamentary reinforcements are described. The recommended test method for filamentary reinforcements including graphite and boron is the picnometric procedure based on the ASTM standard test for bituminous materials.

2.2.1.1.1 Density Gradient Column

This method involves immersion of segments of fibers or filaments into a density gradient tube[5,6]. The specific preparation and use of the gradient column is fully detailed in the ASTM Standard Method recommended for measurement of the density of rigid plastics[7].

The technique is based on hydrostatic equilibrium between a solid specimen and a liquid of identical density. A column of liquid exhibiting an approximately linear density gradient is maintained at a constant temperature. Calibrated density standards, in form of glass floats, are introduced into the column and a graphical representation of float position versus density is plotted. The graph should be plotted on a chart of sufficient dimension to permit a reading accuracy of ± 1mm height. The sample introduced into the column will descend until it reaches the level or height at which its density is equivalent to that of the liquid. With measurement of the specific height of the sample, its density can be read directly from the graph. A listing of candidate liquids, ranging in densities of 0.8 to 3.0 gm/cm^3 is shown in Table 2-1.

Fibrous specimens should be allowed, at least, two to three hours to reach their equilibrium position. If practical, it is advisable to allow a twelve-hour interval before recording sample density readings. Column densities should be checked each time the apparatus is used. Similarly, the graph should be replotted as required.

2.2.1.1.2 Sink-Float Method

A second, but related technique is the "Sink-Float" method which involves the use of two liquids of varying densities, for example, trichloroethylene and dibromoethane exhibiting densities of 1.60 gm/cm^3 and 2.089 gm/cm^3, respectively[8]. The test procedure and required density calculations are summarized herein.

TABLE 2-1 - LIQUID SYSTEMS FOR DENSITY GRADIENT COLUMNS[7]

SYSTEM	DENSITY RANGE g per ml
Methanol-Benzyl Alcohol	0.80 to 0.92
Isopropanol-Water	0.79 to 1.00
Isopropanol-Diethylene Glycol	0.79 to 1.11
Ethanol-Carbon Tetrachloride	0.79 to 1.59
Water-Sodium Bromide	1.00 to 1.41
Water-Calcium Nitrate	1.00 to 1.60
Zinc Chloride-Ethanol-Water	0.80 to 1.70
Carbon Tetrachloride - 1,3 - Dibromopropane	1.60 to 1.99
1,3-Dibromopropane-Ethylene Bromide	1.99 to 2.18
Ethylene Bromide-Bromoform	2.18 to 2.89
Carbon Tetrachloride-Bromoform	1.60 to 2.89
Trichloroethylene-Tetrabromoethane	1.45 to 2.875

1. Sample Preparation

   Dry approximately a two-inch sample in an aluminum dish for a minimum of two hours in an oven at 99° - 104°C.

   Half fill a stoppered vial with trichloroethylene.

   Place vial in oven and cut three samples one to two mm long from the sample, into the vial.

   Place vial in vacuum oven and apply vacuum for five to ten minutes at 65°C (do not permit to boil).

2. Test Method

   Cool and start adding dibromoethane from a pipet in 2 ml increments.

   After each increment, shake vial and allow to set for a minimum of 30 minutes.

   As the fibers take longer to settle, add smaller increments of dibromoethane.

   When the fibers remain dispersed evenly in the liquid after at least 30 minutes, filter the mix through a frittered filter into a beaker.

   Weigh the pycnometer empty and fill with the liquid mixture.

   Adjust the temperature to 25°C, dry and weigh.

3. Calculations

$$\text{Density} = \frac{\text{Weight of liquid} \times \text{Density of water at 25°C}}{\text{Weight of water}}$$

2.2.1.1.3 Westphal (Mohr) Balance

An additional alternative to the density gradient column is the technique which utilizes the Westphal or Mohr density balance. The balance facilitates the direct reading of density by weighing a calibrated plummet immersed in a liquid having a specific gravity equal to that of the sample. The procedure as applied to fibrous materials is detailed below[9].

Fill a buret with orthodichlorobenzene (specific gravity 1.29) and another with tetrabromoethane (specific gravity 2.94), or with two alternative liquids covering a suitable specific gravity range.

Place a few short segments of fiber in a beaker immersed in a water bath controlled at 21°C. Run in some liquid from each of the two burets. Stir to mix the two liquids thoroughly, and observe whether the fibers float or sink in the mixture. (Note: Accurate temperature control is essential to prevent convection currents from invalidating this observation.) If they float, slowly add more of the liquid having the lower specific gravity with constant stirring until the fibers appear to remain suspended in the liquid mixture. If the fibers sink, adjust the specific gravity of the mixture by adding some of the liquid of higher specific gravity. When the mixture appears to have the same specific gravity as the fibers, make final adjustments by running in no more than a few drops of liquid, stir, and allow to sit still for at least one minute before deciding whether the fiber is sinking or floating.

Read from the burets the amount of the two liquids which were mixed to give a mixture of the correct specific gravity. Make up sufficient volume of this mixture using the same proportions of the two liquids, to fill the cylinder of the Westphal balance. Check to see that the specific gravity of this large volume of mixed liquids is correct by dropping in a few segments of the measured fiber to see if it remains at equilibrium.

Hang the calibrated plummet on the arm of the Westphal balance and bring up the cylinder containing the liquid of specific gravity equal to that of the fiber so that the plummet is completely immersed. Adjust the balance weights so that the pointer on the end of the arm lies at the center of the scale. The specific gravity of the liquid can then be read directly off the balance.

The specific gravity of the fiber is equal to the specific gravity of the liquid. Report this to the second decimal place.

2.2.1.1.4 Liquid Displacement (Archimedes Method)

The basic technique involved in this method is fully described in ASTM Standard Designation D-792[11]. The density sample is weighed in air and then immersed in a selected liquid medium and reweighed. The difference is the buoyant force which is converted to sample volume by dividing the sample weight in air by the calculated sample volume. Detailed treatment of this test method is presented in the Union Carbide Standard Testing Methods[12]. The immersed sample weight is determined by hanging a coil of yarn or fiber on a hook freely suspended in the container of liquid. The hook is attached to

the balance as depicted in Figure 2-1. Sample density is calculated using the following equation:

$$\rho = \frac{(S_3 - S_1)d_1}{(S_3 - S_1) - (S_4 - S_2)}$$

where

d_1 = density of immersion liquid

$(S_3 - S_1)$ = sample weighed in air

$(S_4 - S_2)$ = sample weight immersed

S_1 = weight of wire in air

S_2 = weight of wire in liquid

S_3 = weight of wire and yarn in air

S_4 = Weight of wire and yarn in liquid

2.2.1.1.5 Picnometric Method

This method is based on the ASTM Standard Test for specific gravity of semi-solid bituminous materials[13]. It has been modified, however, and applied as a technique for determining the density of fibers. The method, as practiced, is detailed below[14].

Standardization of a specific gravity bottle is required to prepare the test. Tare the empty gravity bottle and record weight as W_1. Fill with boiled, deionized, or distilled water and fit a thermometer into place. Place in a constant-temperature bath maintained at 25° ± 0.1°C (77°F) for a minimum of 30 minutes. Lift part way out of the bath to ascertain that the liquid level in the capillary side arm is exactly filled to the top. Wipe the outside of the specific gravity bottle with absorbent tissue, weigh, and record the bottle weight as W_2. Fill with cyclohexane and repeat temperature measurement for 30 minutes. Record the weight as W_3. Calculate the density of cyclohexane as follows:

$$\rho \text{ cyclohexane} = \frac{(W_3 - W_1)}{(W_2 - W_1)} (0.997)$$

where: 0.997 is density of water at 25°C.

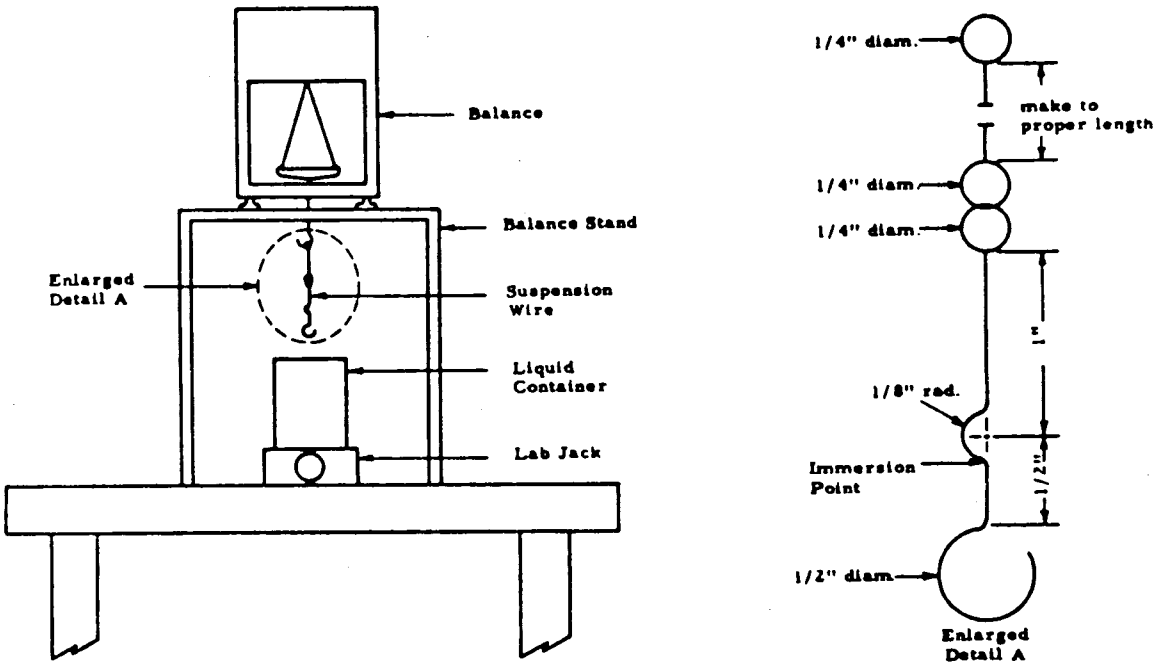


FIGURE 2-1. SCHEMATIC OF THE LIQUID DISPLACEMENT DENSITY[12] APPARATUS

Into a previously cleaned and dried specific gravity bottle, insert approximately 6 inches of carbon fiber tow. Weigh and record the tow weight at W_4. Introduce cyclohexane until the bottle is one-half filled. Place in vacuum desiccator and evacuate at 20 millimeters of mercury for 30 ± 5 minutes. Remove the bottle from the desiccator and fill the bottle with cyclohexane. Repeat the temperature measurement for 30 minutes and ascertain that the liquid level in the capillary is exactly filled to the top. Record weight as W_5. Calculate the density of the fiber tow as follows:

$$\text{Density of Carbon Fiber} = \frac{(W_4 - W_1)}{(W_3 - W_1) - (W_5 - W_4)} \rho \text{ cyclohexane}$$

This result is expressed in grams per cubic centimeter. Density in pounds per cubic inch may be obtained by multiplying grams per cubic centimeter by 0.03613. Perform the test and report the results individually from at least three determinations.

This method is recommended when precise fiber density measurements are desired. Measured density is accurate to the fourth decimal place. The test takes approximately one hour per sample.

2.2.1.2 Recommendation

The procedures described summarize the test techniques most commonly utilized for density measurements on a variety of fiber and filament types. Of the techniques treated, the liquid displacement (Archimedes Method) is the most rapid and exhibits good reproducibility. There are, however, several limitations and disadvantages to be considered; namely,

1. The liquid selected must penetrate and thoroughly wet the fibrous sample.
2. In the case of a porous material, the test result is dependent upon the degree of liquid penetration. This introduces a time-dependent element and can function to significantly reduce the accuracy of the test over a short time period.
3. The test method requires large samples. Graphite yarn samples as long as ten feet and corresponding tow samples of 18 to 20 inch lengths have been cited as a test requirement[15,16].

This method is more readily adaptable to density measurement of non-fibrous, non-porous solids as recommended in ASTM Standard Designation D-892[11].

The density gradient technique is considered one of the more accurate approaches to determining the density of fibrous materials. The test procedure and the required maintenance of the gradient column is time consuming and thus is not considered the most feasible method for quality control or material acceptance. The related "sink-float" procedure, similarly is time consuming and is not suggested for use as an acceptance test method. The picnometer procedure, based on ASTM standard test for bituminous materials, is recommended as the most feasible technique for use as a quality control or acceptance test method for filamentary reinforcements. This method provides for the complete removal of entrapped air or other gases from fiber surfaces and interfiber spaces in graphite yarn and tow and thus permits thorough penetration and wetting by the selected liquid. Since fiber density values are utilized in calculation of the effective cross-section area of yarn and tow for tensile property tests, and determination of constitutive and void volume fractions in composites, reproducible accuracy is required.

2.2.2 Mechanical Properties

Test methods employed for evaluation of the mechanical property characteristics of filamentary reinforcements cover a broad range of materials and thus must be carefully selected. Selection of the test method for a particular filament or fiber bundle is an important consideration. The numerical value obtained for the specific property measured can be dependent, to a significant extent, on the particular technique employed. No single test method is suitable or even applicable to all reinforcements.

While the testing of boron or other similar monofilament is conducted without difficulty, filamentary carbon and graphite reinforcements present a unique challenge in the development of mechanical property test methods. These materials are commonly used in two forms; namely, continuous yarn and tow. Tensile properties of the yarn are effectively determined using a modification of the ASTM standard procedure developed for glass fiber strands, yarn, and roving[17]. Tensile properties of graphite tow have been measured both by a single filament technique and an adaptation of the yarn strand test.

The strand test method eliminates the difficult problems classically associated with handling, measurement, and alignment of minute fibers, and is considerably more representative of composite properties. Table 2-2 summarizes single filament test results for Thornel 50 graphite yarn[18]. Test results are compared with those of the manufacturer. Tests conducted at other laboratories usually show lower strength averages with a wider range of individual values. The manufacturer's data, however, generally falls

TABLE 2-2 - AVERAGE GRAPHITE SINGLE FILAMENT PROPERTIES OF THORNEL 50[18]

Tested by AFML

Yarn A			Yarn B		
True Tensile Modulus ($psi \times 10^{-6}$)	Tensile Strength ($psi \times 10^{-3}$)	Density (g/cc)	True Tensile Modulus ($psi \times 10^{-6}$)	Tensile Strength ($psi \times 10^{-3}$)	Density (g/cc)
43	216		53	260	1.65
48	246		47	288	
52	250	1.63	45	233	
	226	1.61		262	1.65
	263			236	1.63
	260	1.57			
	173				
Avg 48	233	1.60	48	254	1.64

Quoted by Manufacturer

Yarn A			Yarn B		
47.4	262	1.62	55.4	319	1.62

within this range. It is also noted that current state-of-the-art fiber production and processing results in considerable property variability within a reel or between reels of material. Tensile tests on strands normally yield lower values than for single filament tests, but with reduced scatter. Tensile modulus values are essentially the same for both test techniques.

The test methods described are those which are currently used, and a considerable amount of data has been accumulated employing such methods. Several of the procedures, however, are subject to modifications as new materials or novel forms of existing materials become available. The strand test method is recommended for determination of the tensile properties of carbon or graphite yarn and tow. The single filament test is selected as the recommended acceptance test method for boron or similar monofilament reinforcements.

2.2.2.1 Tensile Strength and Modulus

2.2.2.1.1 Graphite

2.2.2.1.1.1 Single Filament Paper Tab

The single filament test method specific to small diameter graphite and carbon is largely confined to friable materials and is basically governed by filament diameter and the force required to initiate fracture. This test, in no manner, relates to the test method recommended for boron or similar monofilament reinforcements. Any fiber, however, the diameter of which does not exceed one mil and breaking load of which does not exceed a few hundred grams can be tested by mounting on a specially designed paper tab sectioned from a 3 x 5 inch file card[9, 14, 16]. The dimensions of the tab and the suggested fiber gage length is depicted in Figure 2-2. A straight line is drawn along the center of tab to allow alignment of the fiber parallel to the longitudinal edge of the tab. The fiber can be attached to the tab by any appropriate adhesive, for example, sealing wax or a drop of Eastman 910. The end of the tab containing the 1/4-inch circular hole is cut off. The sectioned portion is centered and mounted with cellulose acetate on a standard microscope slide. The fiber ends are sectioned and a cover galss is placed over the slide and the cellulose acetate permitted to dry for one hour. Fiber diameter is measured in micron units with a filar vernier or other optical measuring device.

Rubber-lined plastic clamps are used to grip the specimen. When grips are tightened

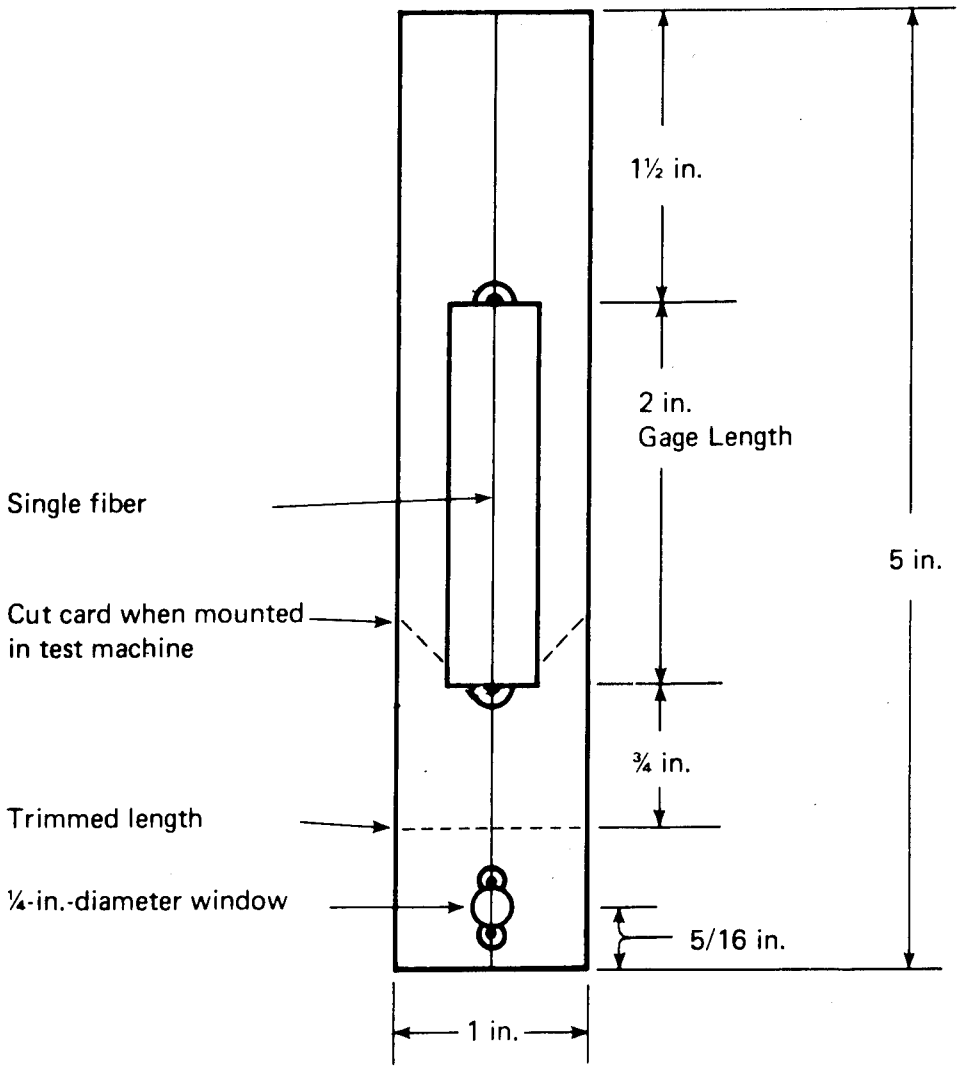


FIGURE 2-2. GRAPHITE SINGLE FILAMENT TAB TENSILE TEST SPECIMEN

and proper alignment is confirmed, the window frame is cut on both sides approximately 1/8-inch from the bottom of the window.

A Type A Instron Load Cell or equivalent (500-gram capacity) is used for the test. Tests are conducted at a cross head speed 0.05 in/min and a chart speed of 2.0 in/min.

Ultimate tensile strength is computed from the equation:

$$S_T = \frac{4P}{\pi D^2}$$

where P = Maximum load at failure
D = Fiber diameter
S_T = Ultimate fiber strength

Modulus of elasticity is obtained by extending the initial straight line portion of the load deflection curve and graphically determining the ratio of stress to corresponding strain. Modulus of elasticity is calculated from:

$$E_T = \frac{4 \Delta P L}{D^2 \Delta L}$$

where ΔP = applied load
L = specimen gage length
D = fiber diameter
ΔL = change in gage length of specimen at point where applied load is taken from chart
E_T = modulus of specimen

Modulus values should be corrected for machine compliance.

Although there are several highly accurate tensile test techniques and specialized test equipment adaptable to single filament graphite, the paper tab technique is recommended where graphite single filament tests are required[9, 19, 20]. The single filament method, however, is not recommended as a standardized material acceptance test. A minimum of fifteen tests should be conducted and the arithmetic mean of all values obtained to three significant figures. Calculate the estimated standard deviation and

report to three significant figures. Calculate the coefficient of variation and report in three significant figures as percent variation.

2.2.2.1.1.2 Strand Test

This method consists of impregnating graphite or carbon yarn with a selected resin binder. The impregnated yarn is cured to form strands which are bonded to metal tabs for testing[21,22]. The specimens are loaded to failure in an Instron Tensile Machine (Table Model Instron TM) or equivalent tension testing machine having controllable constant rate crosshead movement. Strand test results are representative of the actual tensile strength and Young's modulus achieved with graphite yarn-resin composites. Five tests should be conducted for each reinforcement sample selected. Estimated standard deviation and coefficient of variation should be determined for each set of test values generated.

$$A = \frac{g/m}{\rho}$$

where A = Area (m^2)

g/m = Fiber weight per unit length expressed as grams per meter

ρ = Fiber density expressed as g/cm^3

The area may be calculated in units of square inch:

$$A = \frac{g/m}{\rho} \; 0.00155 \text{ (conversion factor)}$$

Accuracy of the area determination is dependent upon the accuracy of the measured fiber density. Tensile tests are conducted at a crosshead speed of 0.5 in/min, a chart speed of 10 in/min at a 50-pound full scale load. A standard specimen gage length of 9.2 inches is used. The strand is preloaded 2 to 3 pounds and tested to failure. The load at failure is recorded in pounds to the nearest 1/4 pound.

Tensile strength of the yarn strand is calculated as follows:

$$S_T \quad \frac{P}{A}$$

where S_T = strand tensile strength (N/M^2)

P = load at failure (N)

A = effective cross-sectional area (M^2)

Tensile modulus is calculated using the following equation:

$$E_T = \frac{P \times L}{C \times A}$$

where A = effective cross-sectional area

L = strand gage length

C = compliance factor

For accurate modulus measurements, compliance corrections should be made. Compliance is the strain that occurs within the test sample gage length. This value is determined by calculating the apparent compliance for the sample tested and subtracting the system or machine compliance from this value. Apparent compliance is the strain indicated by the chart readout under the specified test parameters of crosshead speed, chart speed, and full scale loading. System compliance is simply defined as any deformation that occurs as a result of basic slippage, in the machine or in the gripping system, and stretch in the yarn sample. Procedure for determination of the correction factor for system compliance specific to yarn tests as detailed in the Standard Testing Methods of Union Carbide Corporation is outlined below[22].

- Select sample from a lot of yarn that has shown very uniform test parameters and prepare in same manner as regular test samples.
- Set measured gage lengths of 1, 3, 5, and 10 inches between the grips of testing machine.

- Test at least 3 samples at each gage length using the following conditions:

Gage Length, Inches	Cross Head Speed in/min	Chart Speed in/min	Chart Scale Lbs.
1	0.05	2.0	50
3	0.2	5.0	50
5	0.2	5.0	50
10	0.5	10.0	50

- Calculate the compliance for each using following formula:

$$\text{Compliance} = \frac{\text{Inches (from Chart)}}{\text{Full Chart Scale (lbs)}} \times \frac{\text{Cross Head Speed}}{\text{Chart Speed}}$$

- Calculate compliance and plot compliance versus gage length. The offset at the zero intercept shows the correction for the entire system including the machine, gripping devices, and yarn elongation. This value is subtracted from the apparent compliance as calculated for the sample as shown above.
- A compliance calibration check should be made at least weekly or at any time there is a change in an element of the system or test results indicate a possible equipment problem.

A strand test procedure suggested by the Naval Ordnance Laboratory and conducted by Aerojet General involved the use of an Instron Strain-Gage extensometer with a 2.54 cm (1 in.) gage length and 500 magnification for strain measurement[23,24,25]. The test method is applicable to both standard 2 ply yarn and the standard 10,000 filament tow. In accordance with the suggested procedure of NOL, lengths of single 50.8 cm (20 in.) yarn were impregnated with resin and cured. A tension of 35 grams was applied to the strand during the cure. At first, the ends of the strand were then bonded between cardboard tabs to facilitate mounting of the specimen in the test grips. Strain measurement was attempted through measurement of cross-head travel over 15.2-centimeter (six-inch) and 25.4-centimeter (ten-inch) gage lengths of the strand. Testing of the cardboard-mounted strands showed representative values for strength but low modulus values. This was attributed to minor slippage of the strand in the cardboard faces or the cardboard faces in the grips. Since slippage could be tolerated in the strength measurement but not in the strain measurement, the strain-gage extensometer was tried by mounting it directly on the strand, resulting in accurate modulus values but low strength values. The cardboard holders were then discarded, since they interfered with the extensometer

and the strands were clamped directly in the rubber-faced grips. Mounting the extensometer on the strands bent them and probably caused the low strengths. A pretensioning of 10% was then tried before the extensometer was attached; this procedure proved successful and resulted in good values for both moduli and strengths.

Test results of impregnated strands of six various graphite types, inclusive of standard 2-ply yarn and 10,000 filament tow, are shown in Figure 2-3. Room temperature tensile strength ranged from 13×10^8 N/M^2 (189,000 psi) to 21×10^8 N/M^2 (305,000 psi) with Morganite II exhibiting the highest strength. These values are consistently below the manufacturers specification values.

Hayes devised a strand test procedure specific to standard 10,000 filament graphite tow[25]. The method of tow strand preparation, test procedure, inclusive of determination of the compliance correction factor, and calculation of tensile strength and modulus is detailed below. The test procedure provides for use of a strain gage extensometer, if available, to record tow deformation during load.

DETAILED TENSILE TEST PROCEDURE - The 14 ± 1/4-inch specimens shall be loaded to failure in a testing machine (Instron, Model CD 1916 or equivalent) with rubber-faced grips. The specimen gage length shall be 10 inches and will be aligned in the direction of loading.

The tow tensile test shall be conducted at a crosshead speed of 0.1-inch per minute and a chart speed of 10 inches per minute. The specimen shall be preloaded 2 to 3 pounds. A 2-inch gage length extensometer is mounted on the center section of the specimen to record tow deformation during load for elastic modulus determination.

If an extensometer is not available for tensile testing of tow tensile specimens, the compliance test method could be used as an alternate method for determining fiber modulus. The method of conducting compliance tests is described below. All other test conditions remain the same. The load at failure shall be recorded in pounds to the nearest pound. The ultimate loads should be in the 150-300-pound range.

COMPLIANCE DETERMINATION - The compliance correction factor for a selected graphite fiber/resin shall be determined from load-deformation tests using test specimens having gage length varying from 1 to 10 inches.

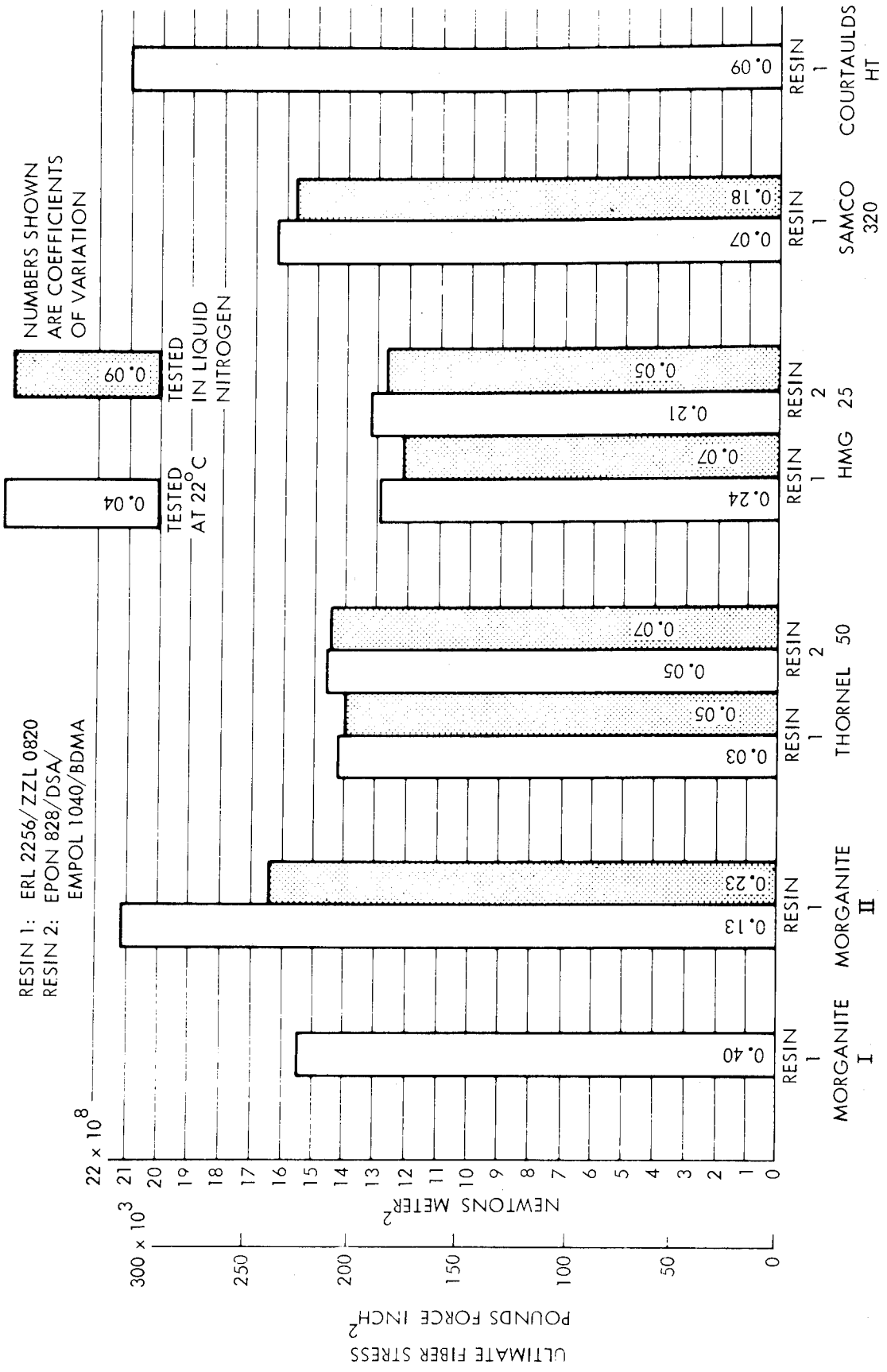


FIGURE 2-3. FIBER TENSILE STRENGTHS AVERAGE VALUES FROM RESIN-IMPREGNATED STRANDS TESTED AT 22°C AND IN LIQUID NITROGEN[23]

- Load specimens to 100-150 pounds using a crosshead speed of 0.1-inch per minute and a chart speed of 10 inches per minute to obtain load-deformation curve for each specimen gage length.
- Determine amount of deformation at 100-150 pounds load for each specimen gage length.
- Plot deformation versus gage length curve using a least squares fit from the load-deformation test data. Determine the machine compliance correction factor "K" for each fiber/resin system for fiber tow tensile modulus determination.

Calculations - Tensile modulus, ultimate tensile strength, and elongation shall be calculated as follows:

$$\text{Ultimate tensile strength} = \frac{(f)(L)(\rho)(2.54)^2}{W}$$

$$\text{Tensile modulus} = \frac{1000P}{A(D-K)} \text{ (compliance method)} = \frac{P}{A\epsilon} \text{ (extensometer method)}$$

$$\text{Elongation ultimate} = \frac{(E - K)(H.S.)\,0.100}{(C.S.)(G)} \text{ (compliance method)}$$

when
- f = Breaking force, pounds
- ρ = Material density, grams per cubic centimeter
- W = Weight of tow, grams (see note)
- L = Length of weighed tow, centimeters
- G = Gage length, inches
- P = Load, pounds
- A = Cross-sectional area, square inches
- D = Chart travel, inches
- K = Machine compliance correction factor, inches
- $C.S.$ = Chart speed, inches per minute
- $H.S.$ = Crosshead speed, inches per minute
- ϵ = Strain, inches per inch
- E = Chart elongation, inches

NOTE: Determined on representative sample taken adjacent to test sample.

2.2.2.1.1.3 Tow Test

A graphite tow tensile test procedure was developed by Morganite Research and Development, Ltd., to demonstrate that the single filament test technique yields mechanical property values which may be realized in practical composites[26]. The procedure also provided a useful check on the accuracy of the methods employed in the choice of a small sample of single fibers. The values quoted for Morganite graphite fibers were obtained by individual measurements in a random sample of approximately 40 single filaments. The tow test method, however, more closely simulates mechanical property measurements determined for the composite material per se, since the tow is impregnated with resin binder or matrix. It is on this basis that the above test method can be considered as an acceptance or quality control test procedure for graphite tow. The required preparation and testing of impregnated tow for determination of tensile properties is fully treated below. The test specimen geometry and loading tab configuration was modified to produce consistent failures within the gage length or central portion. The resin formulation was also modified to reduce the total time required for specimen preparation and curing[27].

Density of the tow selected for testing is determined and recorded. Tensile specimens are cut to a length of 17.8 centimeters (7 inches) and weighed. Record the results as the mass per unit length of tow.

The test method involves impregnating a sample of dry fiber which is nominally the size of a single tow with a liquid epoxy resin. The impregnated tow is then placed in an open faced mold and additional resin is cast around it to produce a dumbbell ended specimen suitable for gripping in a universal test machine. Figure 2-4 shows a typical fiber tow, the disassembled mold, a cast specimen in the mold and the finalized tensile coupon.

The resin system selected for this application was Shell Epon 815 mixed at a 4:1 volume ratio with triethylenetetramine catalyst. The formulation exhibits a room temperature pot life of 1/2 hour and cures rapidly within 1/4 hour at 125°C. A one-hour post cure at 125°C yielded improved but consistent properties.

A typical specimen fitted with an Instron strain gage extensometer of one inch gage length and mounted in a Table Model Instron Testing Machine is illustrated in Figure 2-5. The tensile test is conducted at a crosshead speed of 0.05/in/min. (0.127 cm/min),

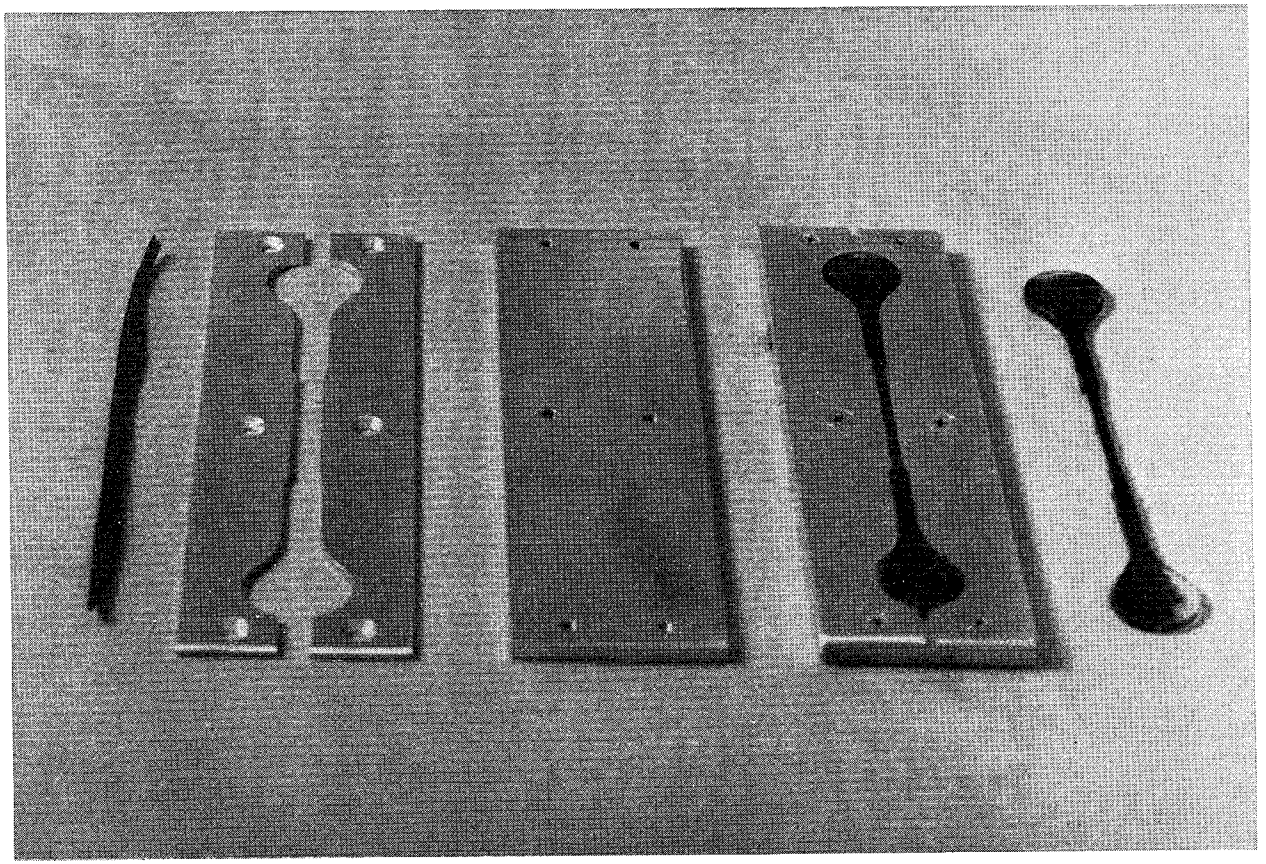


FIGURE 2-4. VIEW FROM LEFT TO RIGHT SHOWS TYPICAL TOW, DISASSEMBLED ALUMINUM MOLD, SPECIMEN AS CAST, AND SPECIMEN FOR TESTING[27]

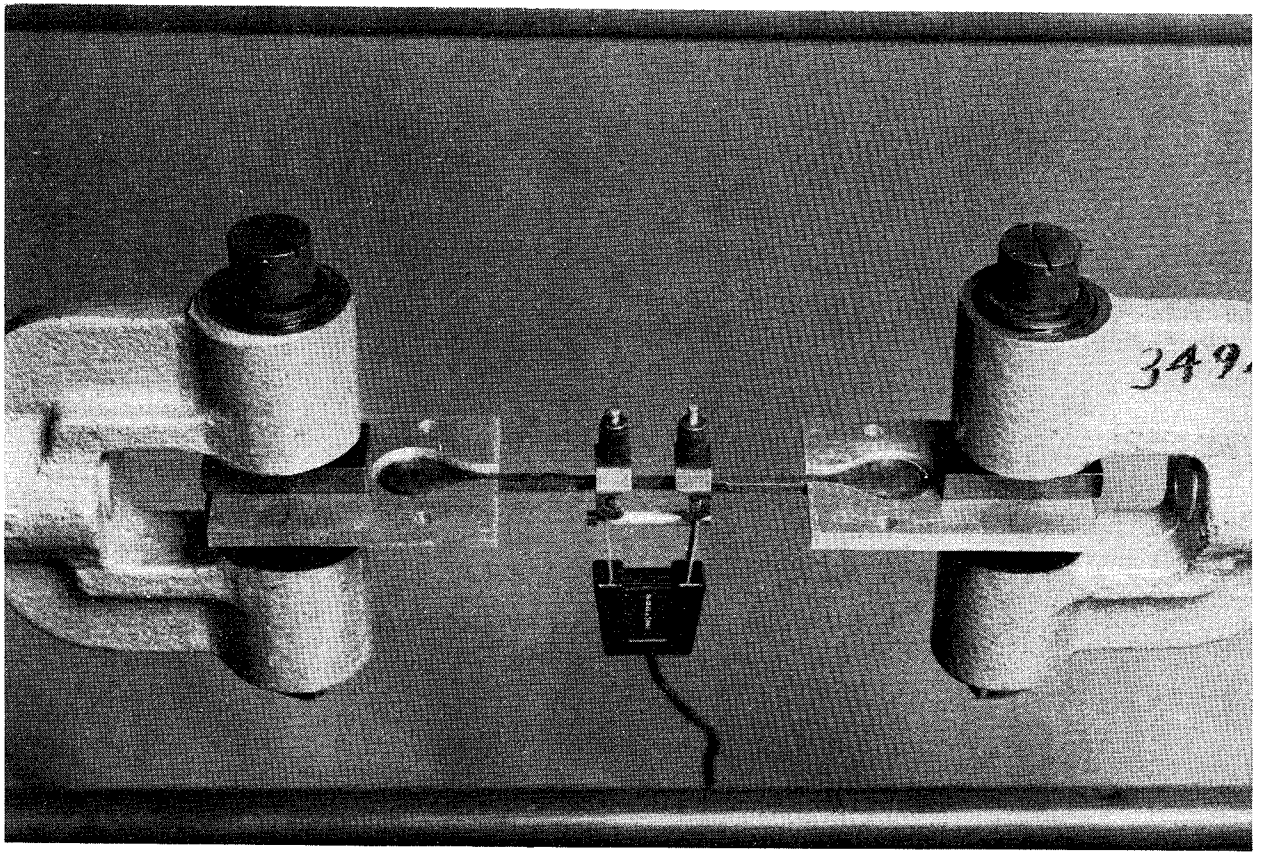


FIGURE 2-5. TOW TENSILE COUPON, WITH ATTACHED STRAIN GAGE EXTENSOMETER, AS MOUNTED IN SPECIALIZED FIXTURES FOR TENSILE TESTING[27]

a chart speed of 2 in/min (5.08 cm/min), and a full scale load of 400 lbs. (1778 N). Extension of the extensometer per pound of applied load is measured up to half of the expected specimen breaking load (i.e., approximately 100 to 150 lbs or 445 to 667N) and the extensometer is removed. The test is completed by loading the specimen to failure. Five specimens are used to characterize each batch of material.

Fiber tensile strength and modulus is calculated using the following equations:

$$S_T = \frac{P}{A_f K}$$

$$E_T = \frac{S_T}{\epsilon}$$

where S_T = Tensile strength psi (N/M^2)

A_f = Effective cross-sectional area of tow calculated using the mass/unit length and density of the tow:

$$A_f = \frac{W}{\rho l}$$

where W = Tow mass (grams)
ρ = Tow density (g/cm^3)
l = Tow length (cm)

K = Correction factor (taken from standard strength of materials text) to account for stress carried by the plastic and given by:

$$K = 1 + \left[\frac{(100 - V_f)}{V_f} \quad \left(\frac{E_R}{E_f}\right)\right]$$

where V_f = fiber volume fraction (%)
E_R = modulus of resin - psi (N/M^2)
E_f = modulus of fiber, psi (N/M^2)
E_T = modulus of tow
ϵ = extension of specimen per inch per pound load
P = load in lbs or N

V_f may be calculated by simply dividing the effective tow cross-sectional area by the specimen cross section:

$$V_f = \frac{A_f}{A_c} 10^2$$

where A_c = cross-sectional area of gage length section of tow tensile coupon

The modulus of the resin, as formulated and cured, was measured at 4×10^5 psi (2.759×10^9 N/M^2). A graph of the correction factor K versus filament volume fraction - V_f, can be plotted and utilized to facilitate rapid calculation of tow tensile properties.

While it is preferable to use the test on tow or roving, it may also be used on prepreg if the fiber content of the prepreg is accurately known. This was determined by resin extraction on samples removed from the prepreg tape in an area adjacent to the tensile specimen. It is also necessary to add additional catalyst to accelerate curing of the prepreg. This was best accomplished by painting it directly on the surface of the prepreg. The method was found to be successful in all cases where the fiber batt is completely impregnated throughout its thickness. However, in a few instances, the center fibers were dry and the specimens failed at low stresses since good fiber to fiber loading was not achieved. Erroneous results related to poor resin impregnation can be readily detected by the fibrous appearance of the fracture.

2.2.2.1.2 Boron

The tensile strength properties of boron and similar substrate filaments, e.g., silicon carbide (SiC) and silicon carbide-coated boron (BORSIC) are determined using the single filament test method. The following single filament test procedure is recommended employing a universal test machine capable of controlled crosshead speeds[28]. Test specimen length should be sufficient to engage fully the specimen grips based on a filament gage length of 1.00 inch. The filament can be gripped by any suitable method or rig which provides accurate alignment and prevents crushing of the filament during stress application. Breaking load of the filament should be measured via a calibrated load cell and accessory readout equipment capable of automatic recording. The strain rate employed should not exceed the response capabilities of the readout equipment.

Ten specimens should be selected for testing from a filament sample of 50 to 60 inches in length. Estimated standard deviation and coefficient of variation should be determined for each set of test values generated. Boron filament quality control tests, as practiced by R. D. Johnson, et al, involved the sampling of 10 to 12-foot lengths before and following the fabrication of each composite panel[29]. The selected filament was sectioned into 6-inch lengths and bonded at each extreme to aluminum tabs 3 inches long and 1/2 inch wide with Shell Company Epon 828 and 815 Resin (50% each) and a T-1 hardener (30 parts per 100 to provide fast cure). Ten filaments were tested at the following parameters; crosshead speed of 0.05 in/min, 5 in/min chart speed on a 10-pound load scale using an Instron or equivalent universal test machine. Properties of the boron filament (produced under AVCO prototype production conditions) are summarized in Table 2-2A.

The average tensile strengths are 500 ± 50 ksi (3.45×10^9 N/M^2 ± 3.45×10^5 N/M^2) with few exceptions, with a coefficient of variation of less than 10% in most instances.

Static tensile modulus of boron filament can be determined in accordance with the procedure as outlined for the graphite strand test inclusive of compliance corrections[21,25].

The effect of gage length on the tensile strength of boron as compared with that of E and S glass and SiC is illustrated in Figure 2-6. Similarity of the slope of boron and E glass curves suggests similar occurrence of flaws in both materials. Several investigators noted the same effect, and associated such flaws in the stressed filament with stress concentrations which cannot be relieved by plastic deformation[30,31]. Classically, no evidence of plastic deformation has been observed in boron filaments under tension.

2.2.2.2 Dynamic Modulus

The principal method for determining the elastic modulus of filaments, fibers, and fiber bundles involves the use of destructive tests. Unlike boron and similar substrate monofilaments, characterization of the mechanical properties of carbon and graphite fiber bundles require resin impregnation or embedding in a resin matrix or binder. Sources of errors related to testing of impregnated fiber bundles can arise from:

- Misalignment of fiber bundle with the principal loading axis.
- Misalignment of individual fibers within the bundle with respect to each other.
- Inability to determine number of broken or discontinuous fibers which terminate within the test section.

TABLE 2-2A - SUMMARY OF BORON FILAMENT* TENSILE TESTS[29]

Spool No.	Tensile Strength, KSI	Coefficient of Variation (%)
A-365	506	4.8
A-365	534	15.1
A-365	409	31.0
A-366	486	3.3
A-366	515	6.3
A-366	465	9.7
A-366	527	2.8
A-368	498	7.2
A-368	498	7.2
A-368	517	14.4
A-369	481	2.7
A-369	484	4.7
A-370	489	3.3
A-370	501	4.1
A-370	509	9.5

*Filament Diameter = 0.004 inch

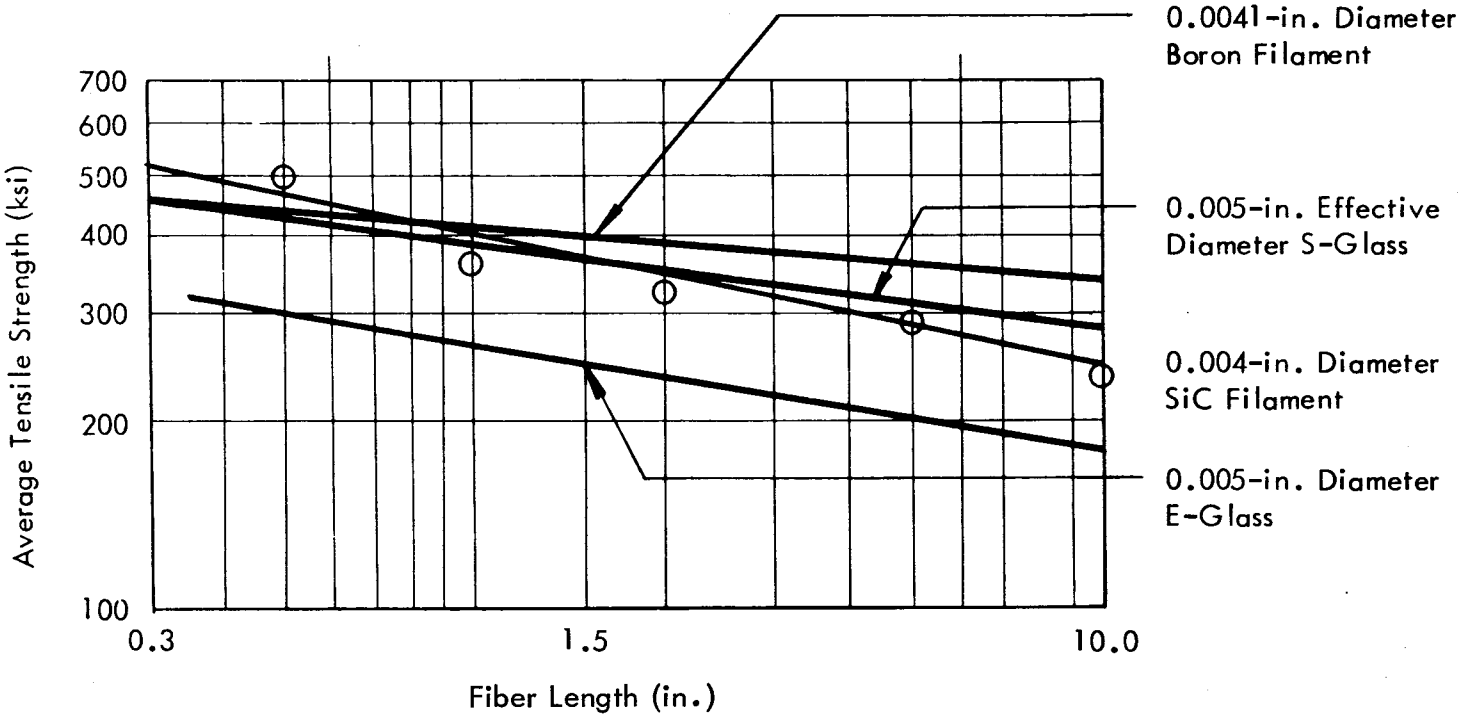


FIGURE 2-6. EFFECT OF SPECIMEN LENGTH ON THE TENSILE STRENGTH OF VARIOUS FILAMENTS[20]

A method developed for determining the modulus of both single filaments and bundles which is generally insensitive to the above error sources is based on the measurement of the velocity of a longitudinal wave propagated through the bundle. The product of the square of the velocity and fiber density is taken as Young's Modulus[32].

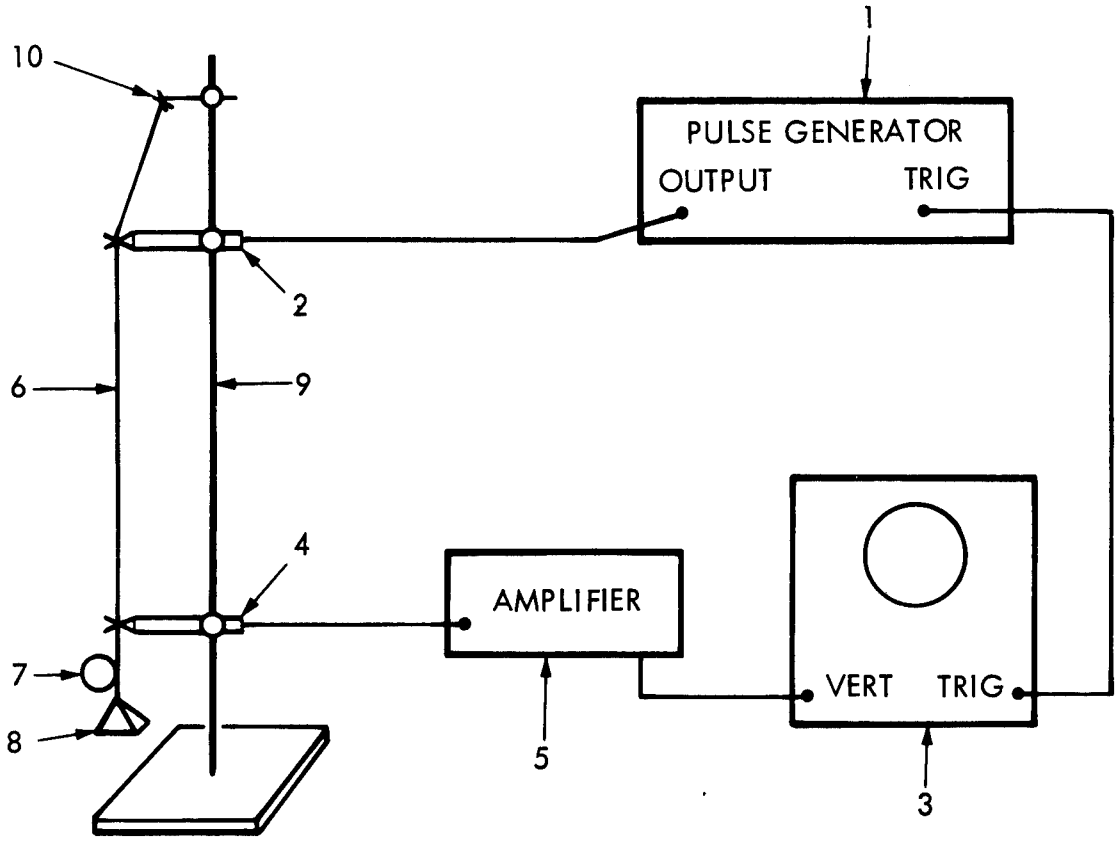
$$E = \rho C^2$$

where E = Young's Modulus
C = Longitudinal Wave Velocity
ρ = Fiber Density (g/cm^3)

The square of velocity is numerically equal to the specific modulus. The accuracy in determining modulus depends on the accuracy of the density and velocity measurements. Density measurements can be accurately determined using the recommended test method discussed in the previous section of this report. Velocity measurements with errors of less than 0.2% can be obtained first by measuring the transit time of a pulse over a number of path lengths and subjecting the resulting data to a linear regression analysis to obtain velocity.

A schematic of the test apparatus is shown in Figure 2-7. As indicated, a pulse generator at (1) is used to excite a transmitting transducer (2) and to trigger an oscilloscope with a calibrated internal-delay generator with a required resolution of 0.05 microseconds. The transducer is a cantilever-mounted crystal with a natural frequency of approximately 0.5 Mhz with a V groove in the free end. The width of the pulse is selected to couple with the resonant mode of vibration of the crystal. Since the second pulse is positioned in time to best reinforce this oscillation, it is also of the same width as the first pulse. The pulse height (voltage) required is determined by the attenuation of the signal of the fiber.

The signal introduced into the specimen is a dampened sine wave, since the crystal will continue to oscillate after being pulsed. As this train of longitudinal waves passes along the specimen, the signal is attenuated, and the inertia of the receiving transducer distorts the signal so that the leading wave is not clearly recognizable. However, subsequent waves are more clearly visible in the received signal. The receiving transducer (4) is similar to the transmitting crystal. Some amplification of the received signal is required before most oscilloscope amplifiers are able to display the signal. Although



1. Pulse Generator
2. Transmitting Transducer
3. Oscilloscope
4. Receiving Transducer
5. Amplifiers
6. Specimen
7. Roller
8. Weight
9. Support
10. Clamp

FIGURE 2-7. DYNAMIC MODULUS TEST APPARATUS[32]

this method was developed for graphite fibers, it has been successfully used with boron filament and a variety of other fibrous materials. Preparation of both graphite and boron reinforcements is detailed below.

2.2.2.2.1 Graphite Fibers

The as-received material is in the form of either two-ply yarn or tow of various lengths, with about 10,000 fibers in cross-section. As a result of the low breaking stresses of high modulus fiber and the tendency of the tow to develop twists, an entire tow is an unsuitable sample, since the signal path would be uncertain and some material is non-linear at low loads (E = f (load)). Approximately one tenth of the material can be removed from the tow by locating a group of fibers which tend to cling to one another; groups of this type separate easily from the tow. A section about 1/2 meter long is used to obtain a test length of about 20 cm; the additional material is required for clamping and loading. The final set of fibers is gripped at each end with a piece of cellophane tape. One end is fixed in the clamp, as shown in Figure 2-7, and a small weight is attached to the free end. The sample is then located over the transducers (separate transducers to maximum desired reading position), and the received signal is observed. In almost every case, some loose and discontinuous fibers exist in the test portion of the specimen. A light air jet across the bundle will cause this type fiber either to blow loose or blow out. The loose fibers are cut free and pulled out of the test section. If a suitable scope trace is not yet obtained, increasing the weight on the specimen will help. The weight used is quite critical in higher-modulus materials, where the greatest non-linearity exists.

2.2.2.2.2 Boron Filaments

Boron or similar substrate filament may be gripped with plastic tape and clamped over the transducers so that the transducers exert only a small force against the fiber. The specimen is weighed and the roller (7) is positioned to maintain the degree of contact. Excessive pressure will result in bending, thus changing the path length and possibly breaking the fiber.

Another method of dynamically determining the modulus of elasticity of filament and fibrous materials involves the "vibrating reed" technique[33]. A single filament specimen fixed at one end and free at the other, is caused to oscillate as a vibrating reed by coupling mechanical energy at sonic frequencies into the fixed specimen end. The

source frequency is varied until specimen resonance is observed (i.e., a maximum of vibration amplitude). Young's modulus is a function of the square of this frequency and of the sample geometry and density as shown by the equation:

$$E = \frac{L^4 \omega^2 \rho_o}{M^4 K^2}$$

where L = specimen length, cm

ω = resonant frequency, radians/sec

ρ_o = specimen density, g/cm^3

M = constant, 1.8751 for fundamental resonant frequency

K = specimen radius/2, cm

Specimen length, which ranges from 1.9 to 2.3 cm, is measured with a traveling stage microscope to an estimated accuracy of 0.02 cm. The diameter is measured at a minimum of four points along its length with a 100X microscope using a calibrated eyepiece reticle. The estimated precision is ±.0001 inch (0.00025 cm). The resonant frequency of the specimen is read directly from the calibrated dial of an audio oscillator to an estimated accuracy of ±5%. The approximate range of resonant frequency required is 100 to 500 cps. The apparatus can be modified to accommodata a small electric resistance furnace for elevated temperature modulus determinations. Figure 2-8 illustrates results of elevated temperature measurements of the modulus of boron and SiC filaments[20].

2.2.2.3 Recommendations

Test methods employed for evaluation of the mechanical properties of filamentary reinforcements include single filament and multifilament techniques. The strand test is recommended for determination of the tensile properties of fiber bundles in form of carbon or graphite yarn and tow. The single filament test is selected as the recommended materials acceptance test method for boron and similar monofilament reinforcements. Tests for both reinforcement types can be conducted on an Instron Universal test machine or equivalent. The suggested minimal number of tests, in ach case, is five. The arithmetic mean for each set of values obtained should be reported. It is further recommended that both strand and single filament tensile modulus be determined employing the compliance technique, and that test parameters, namely crosshead, speed, chart speed and load rate, should be assigned for both yarn and tow strands.

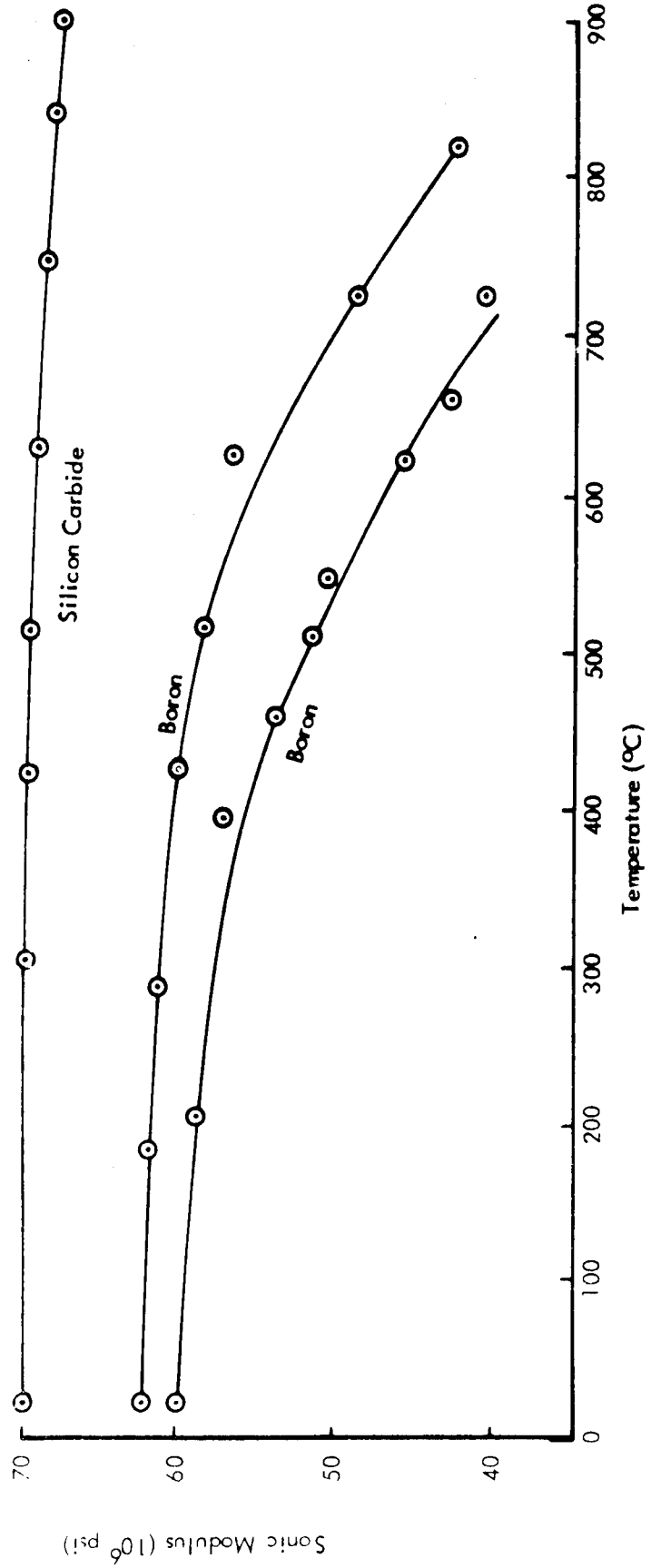


FIGURE 2-8. SONIC MODULUS OF SILICON CARBIDE AND BORON VERSUS TEMPERATURE IN AIR[20]

A statistical interpretation and analysis of test results for the recent ASTM round robin on graphite fiber strands showed significant differences for different participating laboratories[34]. The differences were sufficiently great that the testing laboratories (11) could not be considered equivalent. The analysis was limited to seven laboratories which conducted complete sets of data, and included both high strength tow strand and high strength yarn strand. The results of the interpretation yielded the following findings:

- Lack of uniformly similar strength results among testing laboratories indicated use of different test crosshead speeds. The high strength values were gained at lower crosshead speeds than the lower or reduced strength values. For strength measurements, therefore, a specified crosshead speed should be used.
- The specific effect of impregnation of prepregging of strands by the participating laboratories appeared to be nil. Although the interpretation indicated that the prepreggers cannot be considered equivalent, the error generated is small.
- For modulus measurements among the participating laboratories, differences between the moduli reported were significant. Laboratories which make compliance corrections for machine stiffness may measure higher average modulus values than those which do not make such corrections. Compliance corrections should, therefore, be made for all modulus determinations to reduce the variance.

2.3 MATRIX MATERIALS

2.3.1 General

Advanced filamentary composites treated herein consist largely of epoxy resin matrices. This section will, therefore, be confined to a review of pertinent physical and mechanical property test methods for use as evaluational and acceptance tests for structural plastic materials.

The more common epoxy resins used in aerospace applications include the conventional epoxy; namely, diglycidyl ether of bisphenol A, and the novolacs which are glycidyl ethers of novolacs. Other types used to a lesser extent include glycidyl ethers of various phenols or bisphenols, cycloaliphatics based on diepoxide carboxylates or adipates, and resins based on cyclopentyl ethers. The conventional epoxy is frequently blended with a solid polyfunctional resin, for example, tetraglycidyl ether of tetraphenylethane.

Generally, most resin formulations used in aerospace applications employ elevated temperature curing systems. Optimum cure cycles for systems are determined empirically. Variables which must be considered include the concentration of curing agents, catalysts, time-temperature schedule for B-staging, initial cure and postcure. Normally, each resin property is optimized independently and the general practice is to establish the conditions which yield the desired combination of properties.

Properties yielded from cast resin samples are utilized in some instances for comparison or optimization of resin systems and for use in estimation of composite properties via micromechanics analysis; however, projected evaluation based on cast resin properties is no insurance that similar performances can be attained in a composite. Other salient factors such as cure cycle, void content, resin distribution, resin shrinkage and processing conditions can substantially affect the results. Verification by testing of composites is, therefore, essential. Since the strain rate dependence of high modulus filament reinforced epoxy is strongly influenced by the resin matrix, a detailed characterization should be conducted. The characterization should include thermal expansion and the classic mechanical properties, e.g., tensile, flexure, compression, and shear.

2.3.2 Physical

2.3.2.1 Density

The density test method most appropriate to and recommended for structural plastics is the liquid displacement (Archimedes method) technique[11,34]. Accordingly, the test sample should consist of single pieces of the material of any size and symmetry which can be readily prepared. A sample weight of 1 to 5 grams is considered convenient.

Details of the test procedure and calculation of density values are cited in Section 2.2.1.1.4.

2.3.2.2 Thermal Expansion

The coefficient of thermal expansion for epoxy resins is dependent upon the resin type, curing agent, and amount of the specific curing agent. Within the temperature range of -65°F (-54°C) to 220°F (104°C), thermal expansion varies from 20 to 60 x 10^{-6} in/in°F.

The standard thermal expansion test method is recommended and is detailed in ASTM D-696-70(1970) and the Structural Plastics Applications Handbook[35,36]. The test specimen may have either a round or square cross section and should readily fit into the outer tube of a dilatometer without excessive play. Specimen length may range from 2 to 4 inches[35,36]. Ends of the specimen must be sectioned perpendicular to the axis of the specimen. Conditioning of the sample resin should conform to ASTM D618[37]. The test equipment required includes a dilatometer, calibrated scale or caliper, and a controlled temperature bath. The thermal expansion behavior of two resin systems are illustrated in Figure 2-9. Four samples of each resin material were used to determine thermal expansion values[38]. These values were determined within a temperature range of -80° to 450°F (-62° to 232°C).

2.3.2.3 Thermal Stability

Thermal stability of epoxy resins is generally evaluated by thermogravimetric analysis (TGA), differential thermal analysis (DTA), or other related techniques. Conventional epoxy and epoxy novolacs subjected to TGA show rapid decomposition at approximately 572°F (299°C) in vacuum and in oxygen atmospheres. Total decomposition upon reaching 1652°F (898°C) varies with the resin type and curing agent, ranging from 50 to 80 percent with epoxy novolacs showing the lower losses[39].

2.3.3 Mechanical Properties

2.3.3.1 Tension

Tensile properties of the selected epoxy resin system should be determined at room temperature and at assigned elevated temperatures near the projected or anticipated maximum use temperature of the resin.

In view of the influence of the resin on the strain behavior of high-modulus, resin-matrix composites, it is advisable, if possible, to conduct both room and elevated temperature tests at varying strain rates. AVCO conducted such tests at strain rates of .005, 0.05, and 0.5 in/in/min both at room temperature and 350°F (177°C)[37]. Four specimens were used for each condition and 10-minute soak periods were employed for the elevated temperature tests. Tensile coupon configuration and specifications should conform to that given by ASTM Specification D-638 and Federal Test Method Standard No. 406[40,41]. Conditioning of the test specimens should conform to ASTM D-618[37].

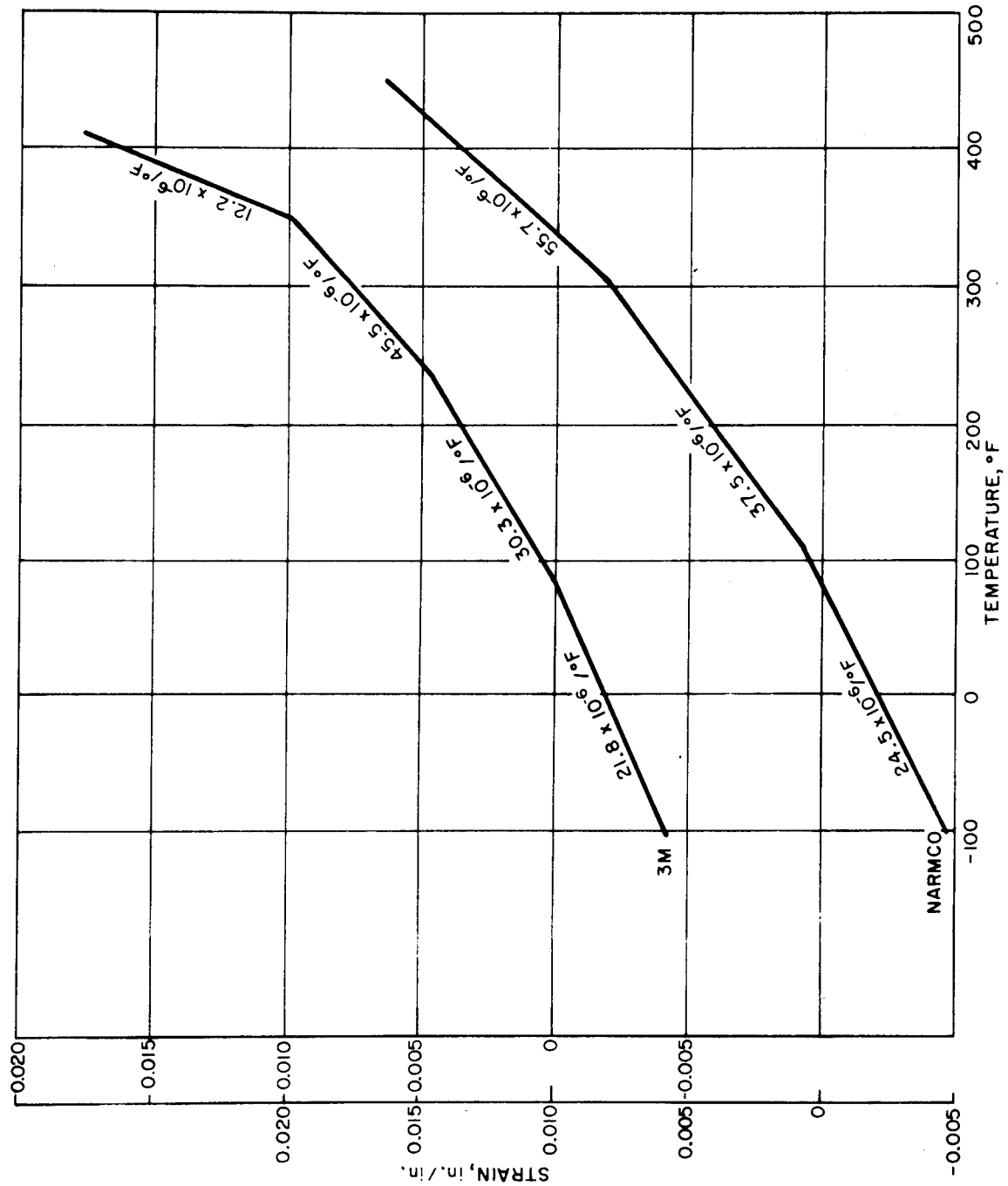


FIGURE 2-9. THERMAL EXPANSION BEHAVIOR FOR 3M AND NARMCO RESINS[38]

Typical room temperature stress-strain diagrams for various epoxy resins systems are illustrated in Figure 2-10. Figures 2-11 and 2-12 show the values of two resin systems at room and elevated temperatures, respectively, and at strain rates of 0.005, 0.05 in/in/min. Generally, both resin systems exhibit approximately equal strengths. At elevated temperatures, both resin strengths indicated an increase with increasing strain. Similarly, the moduli of both systems at both room and elevated temperatures increased with strain rate. Strain-to-failure values approximately doubled going from room temperature to the assigned elevated temperature of 350°F (177°C). The effects summarized above indicate the influence which the resin matrix can exert in a composite system at increasing strain rates. For quality control and material acceptance test procedures, however, a uniform strain rate should be adopted. Time-rate effects which relate to specific service applications can be examined by matching the test parameters.

2.3.3.2 Compression

The compression test for rigid plastics as detailed by ASTM Specification D695-69 is recommended as the evaluational or material acceptance test method[43]. Federal Test Method 1021[44] generally conforms to this ASTM specification. Specimen design recommended by the above specifications used a 1/R ratio of 16 to 1. The dimensions permit determination of elastic modulus and offset yield stress. Compression tests can be conducted on any universal testing machine haveing controlled crosshead motion. Tests should be conducted at a crosshead speed of 0.05 in/min below the yield point. Rates of 0.20 to 0.25 in/min may be used beyond this point if desired.

Results of compression tests conducted on two resin systems at room temperature and 350°F (177°C) and at three different strain rates; namely, 0.005 in., 0.05 in., and 0.5 in. are shown in Figures 2-13 and 2-14[44]. An analysis of these results shows, in general, that values of ultimate stress and modulus were reduced by approximately 50 percent as a function of temperature increase from room temperature to 350°F (177°C), while percent strain-to-failure increased nearly threefold. Translation of these effects to the composite couple and their relation to structural design and application of resin matrix composites will be discussed in the section treating design tests.

2.3.3.3 Flexure

The four point loading flexure test procedure as detailed in ASTM Specification D651-70 is recommended as the acceptance or evaluational test method for epoxy resin

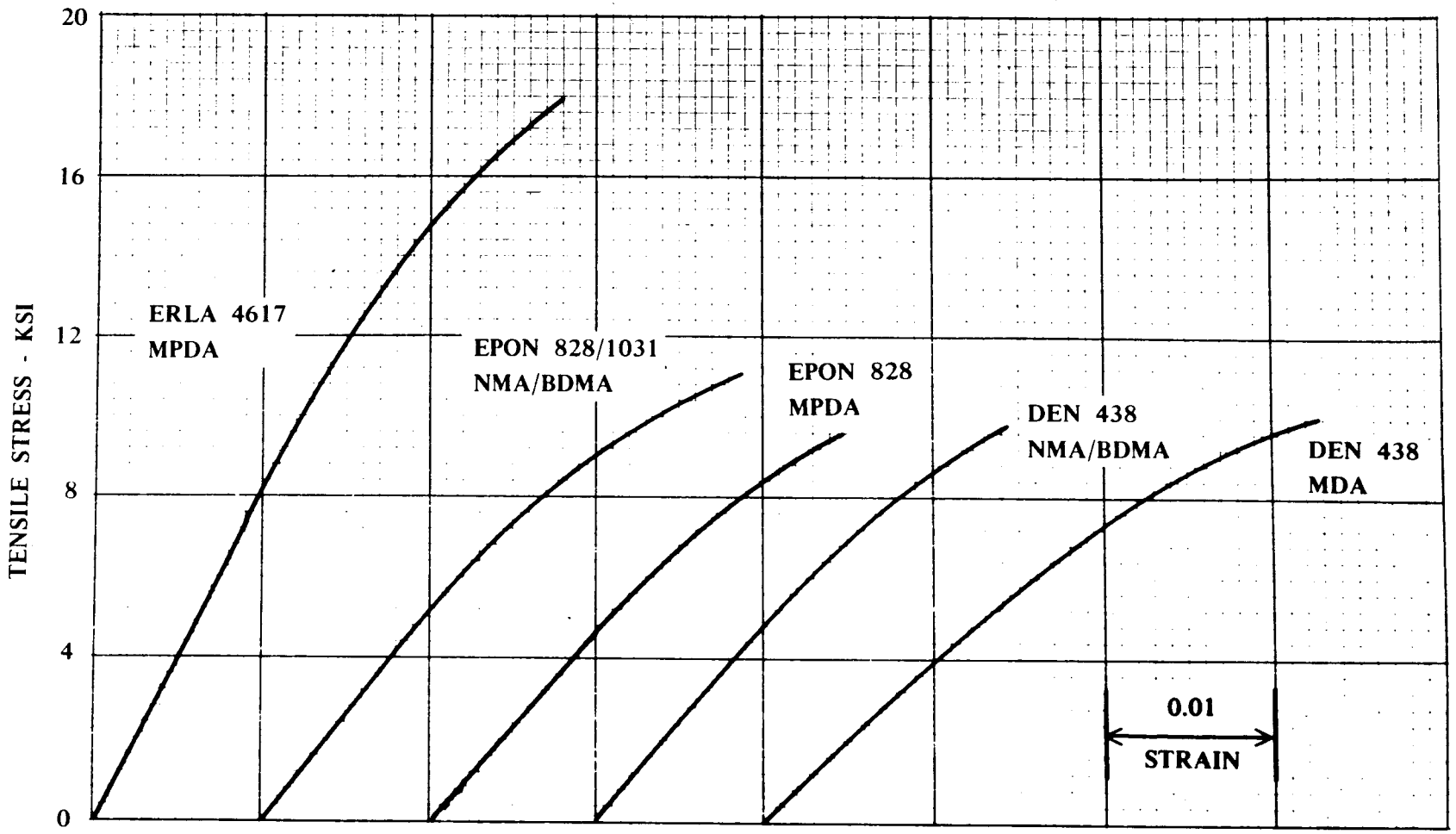


FIGURE 2-10. TYPICAL TENSILE STRESS-STRAIN FOR EPOXY RESINS[42]

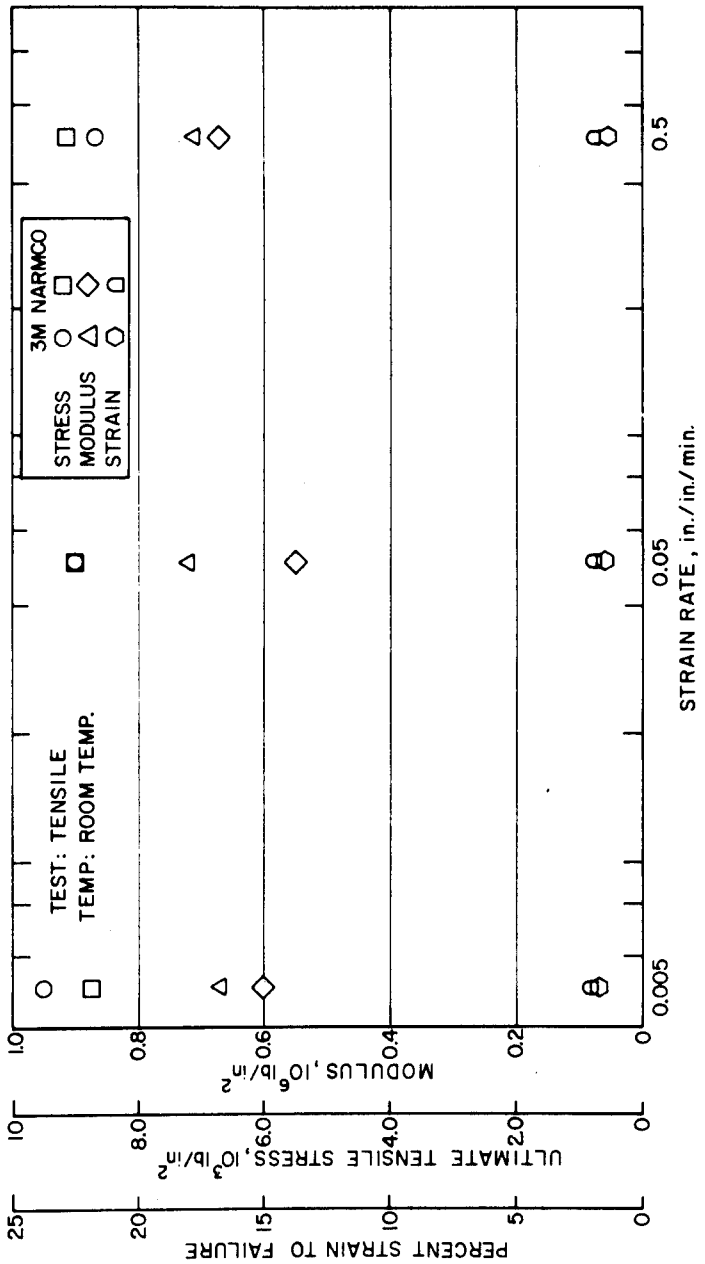


FIGURE 2-11. TENSILE DATA FOR NARMCO AND 3M RESIN SYSTEMS, 73°F[38]

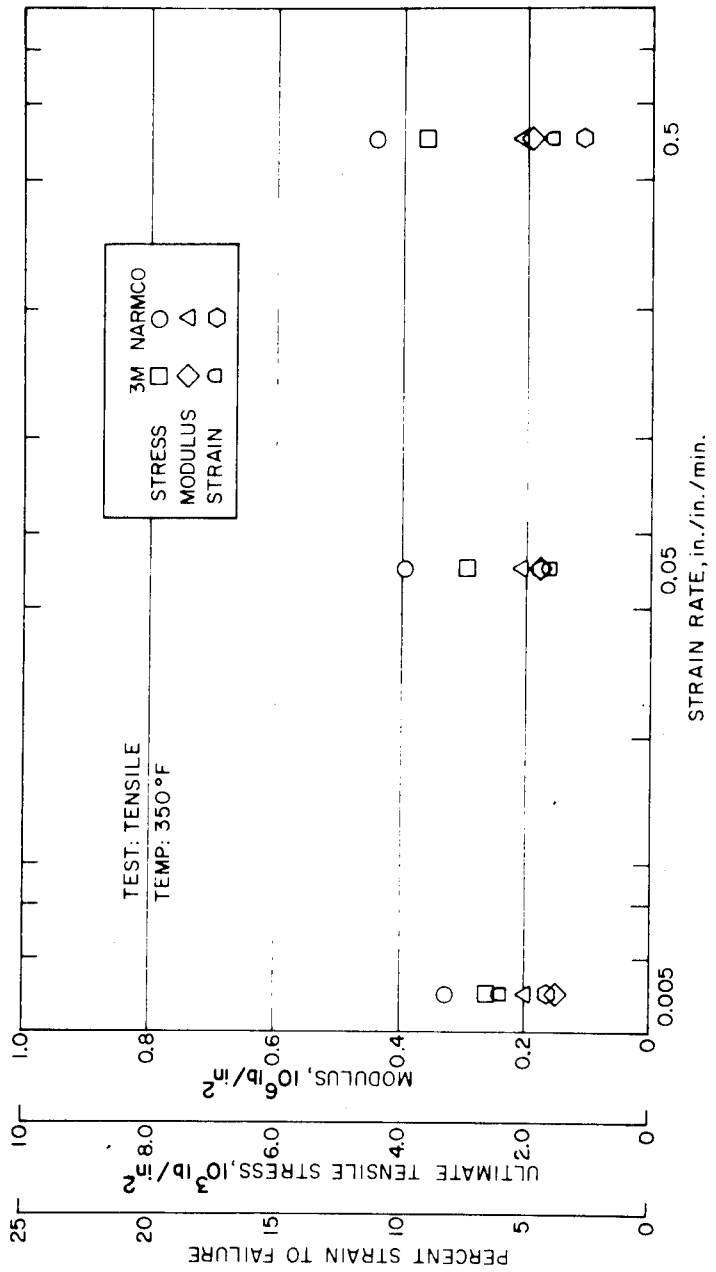


FIGURE 2-12. TENSILE DATA FOR 3M AND NARMCO RESIN SYSTEMS, 350°F[38]

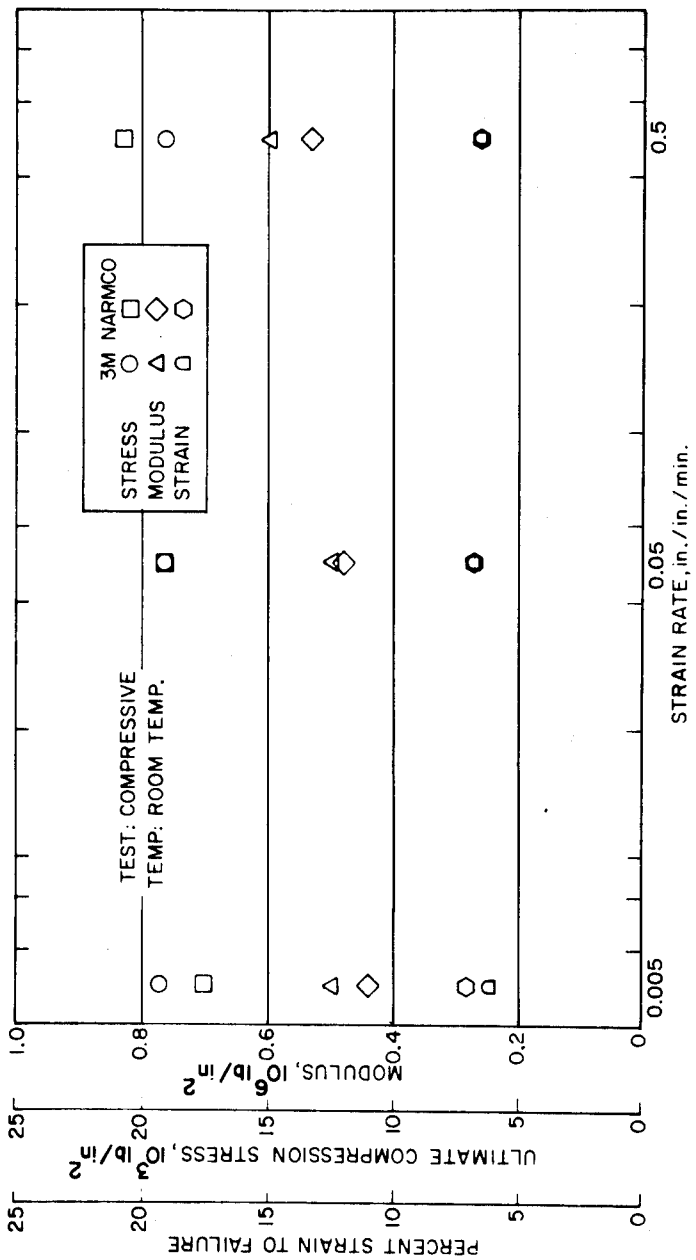


FIGURE 2-13. COMPRESSIVE DATA FOR NARMCO AND 3M RESINS SYSTEMS, 73°F[45]

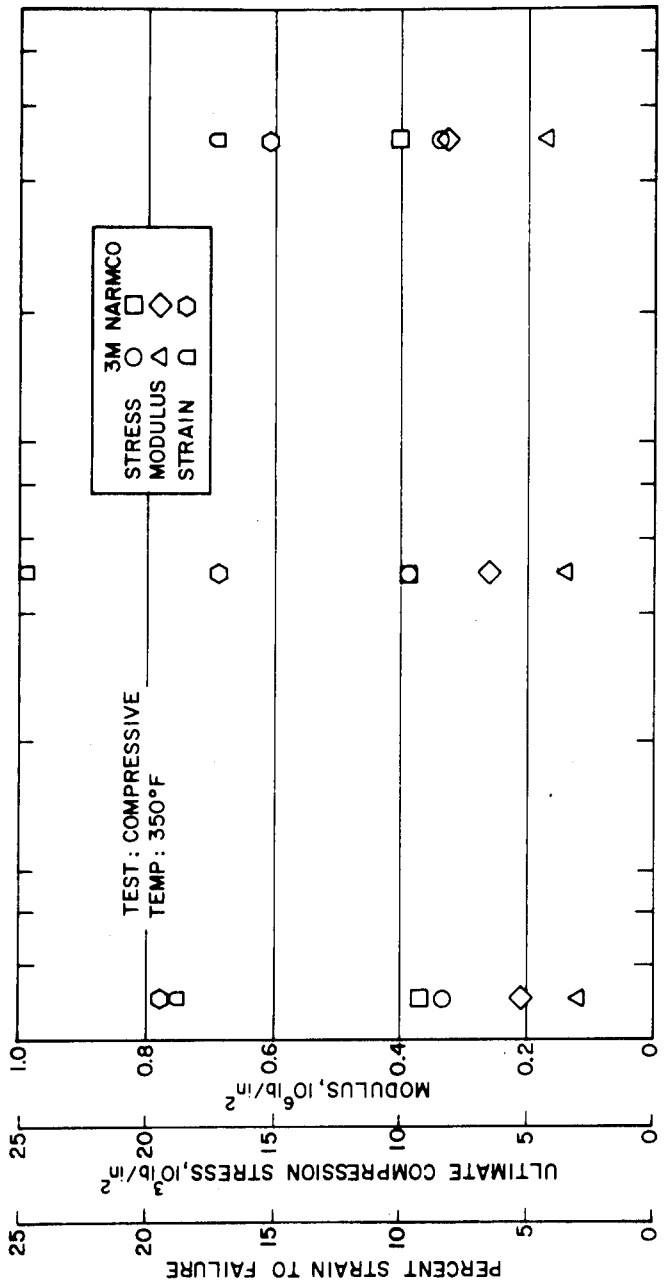


FIGURE 2-14. COMPRESSIVE DATA FOR NARMCO AND 3M RESIN SYSTEMS, 350°F[45]

matrices[46]. A typical test fixture is illustrated in Figure 2-15. The basic procedure is utilized as a flexure test method for both resin and metal matrix composites[46,47]. The recommended test specimen configuration and nominal dimensions are shown in Figure 2-16. The span to depth ratio is maintained at 32:1. As shown in Figure 2-16, the nominal specimen length is 3 inches; however, the length varies with specimen thickness to maintain the assigned span to depth ratio. The test can be conducted on any test machine capable of controlled crosshead motion. The recommended crosshead speed is 0.05 in/min.

Flexural strength is calculated according to the following equation:

$$F = \frac{3\,PL}{4\,Wt^2}$$

where
- F = Flexure strength
- P = Maximum load at failure
- L = Base span
- W = Specimen width
- t = Specimen thickness

Modulus of elasticity is calculated by obtaining the slope of the load-deflection curve by extending the initial straight line portion of the curve and graphically determining the ratio of load to deflection. Modulus is calculated according to the following equation:

$$E = \frac{11}{16}\,\frac{L^3}{Wt^3}\,\frac{\Delta P}{\Delta Y}$$

where
- E = Modulus
- L = Base Span
- W = Specimen width
- t = Specimen thickness
- $\frac{11}{16}$ = Constant
- ΔP = Increment of load, pounds
- ΔY = Increment of deflection, inch
- $\frac{\Delta P}{\Delta Y}$ = Slope of initial straight line portion of the loading deflection curve, pound per inch

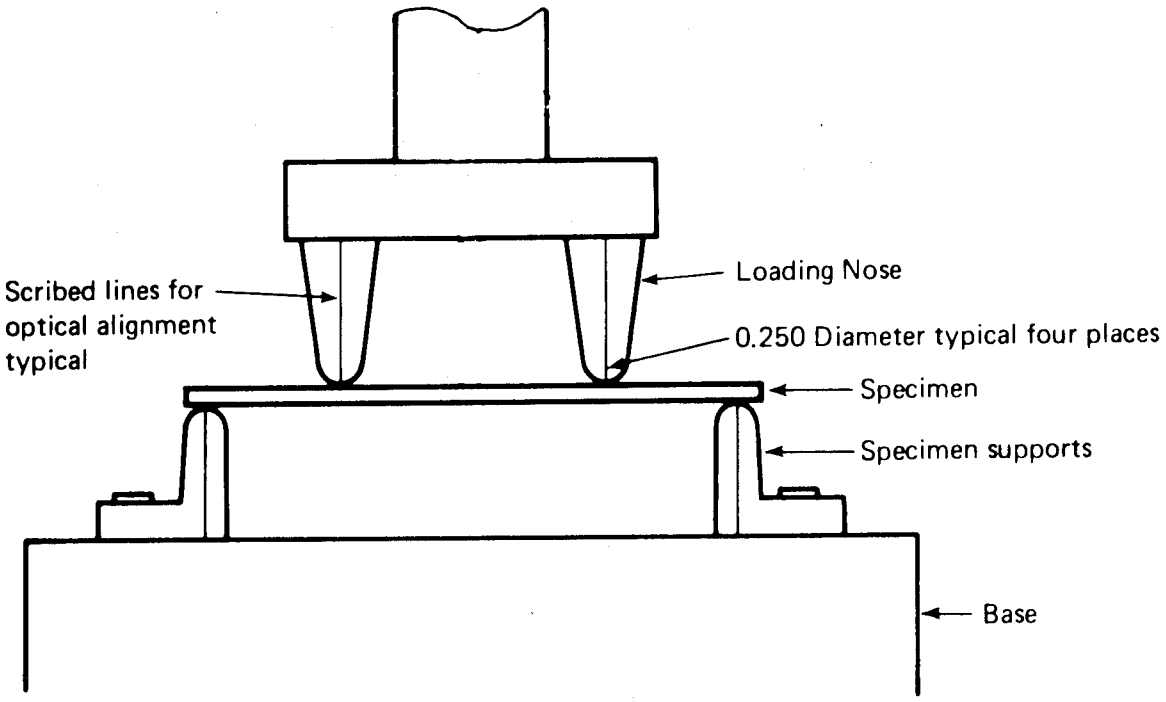


FIGURE 2-15. FLEXURE TEST FIXTURE[46]

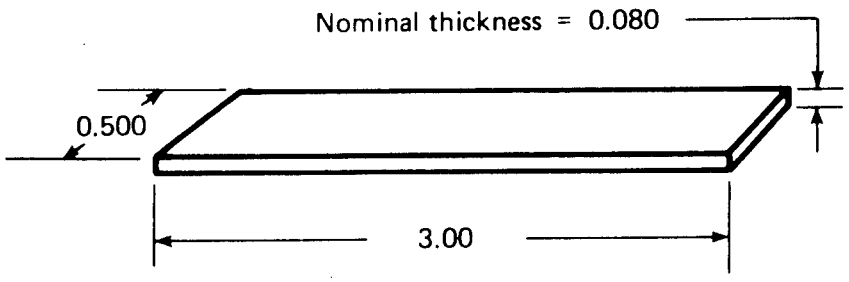


FIGURE 2-16. FLEXURE TEST SPECIMEN[46]

2.4 PREPREG

2.4.1 General

In view of the increased availability and utilization of both boron and graphite monolayer composite prepreg tape, it is considered advisable to include a summary of pertinent material acceptance and evaluational tests for these materials. A group of test procedures is presented which is utilized to determine if the prepreg materials meet the specifications required for fabrication of composites of reproducible physical and mechanical properties. The tests include tack, resin flow, resin gel time, volatiles, fiber and resin contents.

2.4.2 Tack

2.4.2.1 Graphite

Two 2 x 2 inch prepreg specimens are required for this test. A stainless steel plate of 1/4 inch thickness with a surface finish of RMS 100 or better is degreased with acetone or MEK, wiped with a halide free scouring powder, immersed in deionized water and allowed to dry at room temperature. The release paper is removed from one of the prepreg specimens and the specimen is applied to the plate surface with fibers oriented parallel to plate sides. The specimen is highly rolled to smooth out and remove wrinkles. The second prepreg square is then applied to the back of the above specimen with the fibers oriented in the same direction as the substrate specimen. An absence of motion of the specimen for a minimum period of 30 minutes constitutes good tack[49]. A minimum of two tests are conducted to determine tack quality.

Prepreg tack is also determined by a rolling ball technique. This method involves the free rolling of a chrome plated or stainless steel sphere - 0.750 inch diameter and weight of 27 grams, down an incline of 20° ± 0° on to a rectangular strip of prepreg. The distance that the sphere rolls on the specimen surface from start of incline to center of sphere is recorded. Five tests are conducted, each using a new prepreg strip[50]. The previously discussed plate method however is recommended as an acceptance test.

2.4.2.2 Boron

Tack tests for boron are the same as for graphite; however, test specimen dimensions are 3 inches wide by 4 inches long in the case of the recommended test procedure.

2.4.3 Resin Flow

2.4.3.1 Graphite

Prepare prepreg resin flow test layups (for use in a platen press) in the following manner: Remove the release paper from one side of two 2-inch-square specimens and press the exposed sides together with their fibers oriented at 90°. Remove and discard the release paper from one side of the specimen sandwich. Record the weight of the specimen as W_1. Fold a 6 by 12-inch sheet of aluminum foil in half, crease, then unfold the sheet and lay it on a flat surface. Stack two 3-inch-square pieces of fiber glass bleeder cloths together, then lay them on top of the aluminum foil, aligning the edges of the bleeder cloth with the center crease of the foil. Center a 3-inch-square porous tetrafluoroethylene (TFE) release cloth on top of the bleeder cloth. Center the exposed side of the specimen on top of the release cloth, remove the remaining piece of release paper from the specimen, weigh the piece of release paper, and record the weight of the release paper as W_2. Center a second 3-inch-square TFE release cloth on top of the specimen and place two 3-inch-square bleeder cloths on top of the relaese cloth. Fold the aluminum foil over the layup to form a 6 by 6-inch square, completing the layup.

The flow test is conducted as follows:

Set the temperature of platen press to 350 ± 5°F. Place the layup on the press and immediately apply 50 ± 3 psi to the specimen. When the layup has been in the press for 15 minutes ± 15 seconds, remove it from the press and allow it to cool to room temperature. Remove the specimen from layup and trim excess resin which has extruded around the edges. Weigh the specimen and record the weight as W_3. Calculate the percent of resin flow by:

$$\text{Flow w/o} = \frac{(W_1 - W_2) - W_3}{W_1 - W_2} \quad (100)$$

where W_1 = initial weight of the specimen with one piece of release paper still attached

W_2 = weight of the release paper in W_1 above

W_3 = final weight of the specimen after pressing

Average and record the data from two tests[49].

2.4.3.2 Boron

The preparation of the specimen requires four 3-inch-square pieces of prepreg. Remove the release paper from both sides of each of the four pieces and stack the plies evenly, alternating 0° and 90°. Weigh and record the weight as W_1 (all weights to the nearest 0.001 gram). Fold two pieces of 3 by 7-inch predried TX-1040 release cloth around the stack at 90° to each other.

Place the stack in a platen press preheated to 350 ± 10°F. Apply 50 psi within 5-10 seconds to insertion and cure for 15 minutes. Remove the laminate from the press and allow to cool. Remove the specimen from the release cloth and weigh; record as W_2. Calculate the percent of resin flow by:

$$\text{Flow w/o} = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 = initial weight of the specimen

W_2 = weight of the specimen after pressing

2.4.4 Gel Time

This procedure is applicable to both graphite and boron prepreg[49]. Specimens consisting of four 1-inch squares of prepreg are cut with edges parallel and perpendicular to the fiber direction and plied with fiber of alternate layers oriented at 90°. The specimen is placed between aluminum foil coated with a release agent and inserted in a platen press preheated to 300°F. The press is closed to a point sufficient to form beads of extruded resin. The operation should be timed with a stopwatch starting with press closing. The beads of extruding resin are probed with knife-edge rods until long strings or threads of resin cease to form. Record the time when the resin ceases to string. Conduct three tests (min) and record the data. Gel time is the average value.

2.4.5 Volatiles Content

The following procedure is applicable to both graphite and boron prepreg[49]. Filtering crucibles are conditioned in a beaker containing concentrated nitric acid (HNO_3) for a minimum of 1 hour at 100 ± 5°C (212°F). The crucible is washed, desiccated, and weighed (W_1). A 2-inch-square prepreg sample is placed in the crucible and weighed

(W_2). Prepregs with epoxy resin systems are conditioned in a nonrecirculating oven maintained at 93.5 ± 3°C (200°F) for a minimum of 90 minutes and a maximum of 120 minutes. The crucible is removed from the oven, desiccated, and reweighed (W_3). The weight percent of volatiles is obtained from

$$\text{Volatiles w/o} = \frac{W_2 - W_3}{W_2 - W_1} \quad (100)$$

Data are averaged from a minimum of three tests.

2.4.6 Resin Content

2.4.6.1 Graphite

One 2-inch-square specimen with volatiles removed is placed in a 150-250 ml beaker. The beaker is filled with 100 ml of nitric acid (HNO_3) and placed on a hot plate maintained at 100 ± 5°C (212°F). The sample is digested until fiber is completely separated as determined by visual examination. The acid and fibers are transferred into the original tared glass filtering crucible positioned in a filtering flask with vacuum trap and vacuum pump. Fibers are washed three times with 20 ml of HNO_3 and followed by water wash[51].

The crucible and fibers are dried at 93.5 ± 3°C (200°F) for a minimum of 60 minutes, cooled in a desiccator, and the weight (W_4) is obtained. The weight percent of resin is computed from:

$$\text{Resin content w/o} = \frac{W_3 - W_4}{W_2 - W_1} \quad (100)$$

where W_1 = is crucible weight

W_2 = is sample and crucible weight

W_3 = is specimen weight

W_4 = is weight after acid digest (fibers)

Compute weight percent of fiber from:

$$\text{Fiber content w/o} = \frac{W_4 - W_1}{W_2 - W_1} \quad (100)$$

2.4.6.2 Boron

One 2-inch-square specimen with volatiles removed is required. Prepare asbestos thimble by refluxing in a solution of dimethylformamide. Thimble is dried and weighed. Specimen is placed in thimble and weighed to nearest 0.001 gram (sample weight = W_1). The specimen is refluxed in chemically pure dimethylformamide at a rate which causes a 50 ml Soxhlet to dump every 30 minutes. Four complete washings are required, taking approximately 2 hours. Specimen is dried to a constant weight at 250°F[52,53].

Obtain filter weight and record as W_2. Resin content may be computed from:

$$\text{Resin content w/o} = \frac{W_1 - W_2}{W_1} \quad (100)$$

Data are averaged from a minimum of three tests. The Soxhlet method is also applicable to graphite.

2.4.7 Shelf Life

2.4.7.1 Graphite and Boron

Shelf life is the number of days that the prepreg material can be stored and continue to maintain specified properties. Shelf life is quoted at temperatures of 0°F, 40°F, and 80°F. The shelf life at 80°F is also referred to as the work life of the material.

In determining shelf life, the following four properties are measured by the test methods previously discussed:

Volatiles content
Resin content
Tack
Flow

Testing is conducted at intervals based on estimated shelf life for the resin system being tested. When one of the four properties cited above fails to meet specified requirements in a given test period, shelf life is based upon the previous successful test period. A typical example of a testing schedule is outlined below[49]:

80°F	40°F	0°F
1 day	3 days	9 days
3 days	9 days	27 days
9 days	27 days	81 days
21 days	81 days	243 days

2.5 COMPOSITE MATERIALS

2.5.1 Physical Properties

2.5.1.1 Density

The basic pycnometer method is recommended as the material acceptance test for composite density. The pycnometer is calibrated in this instance with mercury and the initial volume is computed from:

$$V = \frac{W_2 - W_1}{\rho\,Hg}$$

where W_1 = Weight of empty pycnometer

W_2 = Weight of pycnometer filled with mercury

$\rho\,Hg$ = Density of mercury at recorded temperature

The pycnometer is emptied, cleaned, and dried. Composite short beam shear or other suitable composite specimens weighing 4 to 6 grams are weighed (W_3), introduced into the pycnometer and again weighed (W_4). The vessel and its contents are evacuated to a pressure of 2 mm of mercury for 15 minutes, filled with mercury and the mercury temperature measured. If no bubbles are visible in the pycnometer, the vacuum is disconnected and the vessel and its contents are weighed (W_5). The average density of composite specimens is computed from the equation

$$\rho_c = \frac{W_3}{V - \frac{W_5 - W_4}{\rho Hg}}$$

where ρ_c = Composite density

W_3 = Weight of specimens

W_4 = Weight of specimens and pycnometer

W_5 = Weight of specimens and mercury filled pycnometer

2.5.1.2 Composition

In determining the composition of composites, the most important step involves selection of accurate techniques and test methods which indicate fiber, resin, and void contents. In view of current state-of-the-art fabrication processes for filamentary/resin composites, voidds, without regard to size, geometry, or distribution, can be considered a composite constituent. The significance of voids is demonstrated by their marked influence on certain composite mechanical properties; namely, shear, compressive, and longitudinal flexural strengths. Composite ignition or resin burnoff has been selected as the recommended procedure for determining the constituitive volume fractions of graphite and boron reinforced systems. The technique is simple and economic and is readily capable of defining, at last, the lower limits of void content.

2.5.1.2.1 Fiber Fraction

2.5.1.2.1.1 Graphite

Ignition (Gravimetric) - The weight fraction of fiber in the composite can be determined by simple ignition of the composite in air at 400 to 425°C for at least 4 hours. Corrections can be made, however, to allow for imcomplete burn off of the resin and oxidation of the fiber by determining the weight losses of both resin and fiber under identical conditions of temperature and time for the same ignition. This method is not applicable to graphite fiber types which exhibit excessive wright loss as a function of oxidation.

The weight percent (w/o) fiber is determined on the basis of initial composite sample weight and the weight of residue following ignition, assuming, however, complete combustion of the resin and an absence of fiber weight loss as a result of oxidation.

$$\text{Fiber content (w/o)} = \frac{W_2}{W_1} \quad (100)$$

where W_1 = Weight of composite

W_2 = Weight of residue remaining after ignition

With fiber content in weight percent, the fiber volume in the composite may be computed from:

$$V_f = \frac{\text{Fiber w/o } (\rho_C)}{\rho_F} \quad (100)$$

where V_f = Fiber Volume fraction

ρ_C = Composite density

ρ_f = Fiber density

Resin Digestion - This procedure is detailed in Section 2.4.6.1.

Point Count - The technique is microscopic and involves a point count analysis wherein a grid is superimposed on the specimen and the fibers contacting intersection grid lines are tabulated and summed to provide local fiber fraction determinations. This technique was suggested by Sands, et al, for determination of void fractions in filament wound structures[54]. This procedure is particularly adaptable to graphite fibers having irregular cross sections. The method can be conducted on any standard optical microscope or metallograph.

2.5.1.2.1.2 Boron

Resin Burnout (Ignition) - Determination of filament or resin content in boron epoxy composites by an ignition technique consists of burning weighed specimens of resin blanks and of the composite in an oxygen atmosphere to determine the amount of CO_2 evolved by each material[38]. CO_2 is collected in a selective gas adsorption train thereby

permitting determination of the percent carbon in the composite. Prior to conducting the procedure, a conditioning of the samples is suggested for removal of moisture and entrapped gases. The procedure recommended consists of desiccating the samples for 8 hours over a suitable granular desiccant. The conditioning may be omitted when resin content determination has been shown to be relatively unaffected by the treatment. The carbon percent as resin content of the blank (Specimen A) is recorded. Similarly, the carbon percent as resin content of the composite (Specimen B) is recorded. Total resin content is calculated as follows:

$$\text{Resin percent} = \frac{B}{A} \times \frac{W_1}{W_2} \times 100$$

where A = percent CO_2 in Specimen A (Resin Blank)

B = percent CO_2 in Specimen B (Composite)

W_1 = weight in grams of resin blank - Specimen A

W_2 = weight in grams of boron composite specimen - Specimen B

Resin Digestion - The basic procedure for this method is treated in Section 2.4 for composites with carrier material, for example, scrim cloth in the case of boron-epoxy. The weight of the carrier material may be computed from the product of the weight per square inch of carrier, and the number of plies of carrier in the specimen. Fiber content in weight percent may be computed from:

$$\text{Fiber content (w/o)} = \frac{W}{W_i} - \frac{mN}{\rho_c} \times 100$$

where W = measured weight of fiber in crucible after resin removal

W_i = initial composite specimen weight

m = weight per square inch of carrier material per inch of composite thickness

ρ_c = composite density from section

N = number of plies of carrier material per inch of composite thickness

Fiber content as volume percent is calculated as shown in Composite Ignition Method for graphite fiber content determination. The suggested mineral acid for use with boron-epoxy composite couple is reagent grade sulfuric acid.

Metallographic - This method utilizes an optical photographic technique for determination of fiber content. The technique is applicable only to reinforcements which are characterized by relatively uniform cross-sectional areas which show minimal variations as opposed to the point count technique which is independent of cross-sectional area measurements.

Selected sections of the composite or composite specimen are mounted and polished. Two photomicrographs, one each at magnifications of 200X and 1000X, are taken of selected areas of the specimens. Average filament diameter is obtained from the 1000X photomicrograph by taking the mean of 15 randomly selected filaments. Accuracy of the determination is dependent upon the resolution of the optical objectives used. Resolution is the minimum distance between two lines which can be resolved and is expressed by the equation:

$$\delta = \frac{1/2\lambda}{NA}$$

where λ = wavelength of light used in Angstroms
NA = numerical aperature of the objective

The number of filaments per given area can be determined by dividing the 200X photomicrograph into centimeter squares and counting the number of filaments per unit square. The total area occupied by the filaments is given by the product of average filament diameter and the number of filaments tabulated. Filament volume fraction is calculated from the equation:

$$V_f = \frac{\text{total cross-sectional area of filaments in area considered}}{\text{area considered}}$$

2.5.1.2.2 Void Content

2.5.1.2.2.1 General

In the compositional analysis of advanced fiber-reinforced resin matrix composites, void content is of major importance and should be considered. Void-free plastic matrix

composites cannot be fabricated using current state-of-the-art techniques. In several composite systems, it is known that total void content relates directly to fabrication process parameters of time, pressure, and temperature. The size and symmetry of the composite fabricated is also a contributing factor. The distribution of voids in a composite has been found to be influenced by specific properties of the constituents; for example, thermal characteristics, matrix viscosity, and volatility[55]. Further findings indicate that shapes, dimensions, and orientations of voids are functions of location, type of fabrication, fiber orientation, and processing parameters.

Voids in composite materials function only to degrade their mechanical properties. It is obvious, therefore, that the specific degree of such effects is of prime importance in the design of composite structures and substructures. The marked influence of void content on longitudinal flexural strength and shear strength is commonly observed in resin composites. Schultz observed degradation in unidirectional graphite/epoxy panels for compression, flexure, and shear strengths and for fatigue failure level at void contents ranging from essentially zero to 3 volume percent. A summary of the effects of voids on the behavior of unidirectionally reinforced laminate as detailed by Lenoe is shown in Table 2-3[56].

In view of the pronounced influence of low void volume fractions on the mechanical properties of advanced, low temperature filamentary composites, development and use of techniques for void characterization assumes major significance in quality control and acceptance of these materials. On this basis, therefore, void content has been included under the general heading of composite composition. Several test methods applicable to and commonly used for composite void content determinations are also employed for resin and fiber contents and have been previously defined. These methods include resin ignition (burnout), resin digestion (mineral acid and Soxhlet technique), and point count.

2.5.1.2.2.2 Density Method

The standard density method involves measurement of the density of the composite determination of the weight fractions and densities of the constituents.

TABLE 2-3 - SUMMARY OF POROSITY EFFECTS ON MECHANICAL PROPERTIES OF THORNEL 50/EPOXY MONOLAYER[56]

Property	Porosity Range	Fiber Volume	Associated Range in Mechanical Properties	Remarks
Longitudinal Flexure				
Modulus	1/2 to 11%	52	Modulus varied from 19 to 26 million psi	Linear
	2%	50 to 73	Modulus ranged from 24 to 34 million psi	Linear
Strength	1/2 to 11%	52	Ultimate flexural strengths varied from 155,000 to 69,000 psi	Linear
	2%	50 to 73	Ultimates varied 100,000 to 155,000	Linear
Transverse Flexure				
Modulus	1/2 to 11%	52	Modulus varied from 290,000 to 940,000 psi	Nonlinear
Strength	1/2 to 11%	52	Ultimate strengths ranged from 1000 to 6000 psi	Nonlinear
Longitudinal Compression				
Strength	1/2 to 11%	52	Ultimate strengths varied from 52,000 to 69,000 psi	Nonlinear
Short Beam Shear				
Strength	Low porosity, 2%	50 to 73	Strengths ranged from 5300 to 6800 psi	Nonlinear 33% decrease
Strength	1/2 to 11%	52	Strengths degraded from 6800 to 4200 psi	Nonlinear 27% decrease

$$\text{Void Percent} = 100\left[1 - \left(\frac{W_f}{\rho_f} + \frac{W_R}{\rho_R}\right)\rho_c\right]$$

where W_f = weight fraction of fiber

W_R = weight fraction of resin

ρ_f = density of fiber

ρ_R = density of resin

ρ_c = density of composite

A modification of the above method is based on the densities of the composite and its constituents. The accuracy of the method is dependent upon the precision of the required measurements[57]. Void volume fraction is calculated by the following equation:

$$\text{Void Volume Fraction} = V_F\left[\left(\frac{\rho_F - \rho_R}{\rho_R - \rho_A}\right) - \left(\frac{\rho_C - \rho_R}{\rho_R - \rho_A}\right)\right]$$

where V_F = fiber volume fraction

ρ_F = density of fiber

ρ_R = density of resin

ρ_A = density of air at STP

ρ_C = density of composite

As previously cited, the method demands greater precision in measurements of density than is normally required for quality control or material acceptance. Further, a knowledge of the statistical distribution of density of the constituent materials is necessary. As reported by Cuevas[57], variations of the density in E glass were reported to be of the order of 0.75%. An error of this magnitude introduced by using a manufacturer's quoted value of fiber density would lead to significant inaccuracy in void fraction determinations for high quality, low void-content laminates.

2.5.1.2.2.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis of resin matrix composites is a refinement of the resin ignition or resin burnout technique. The composite sample is heated in ambient air at a constant rate and the associated weight loss from oxidative degradation is recorded. The analysis curve generated from the reaction is nonlinear. In the case of many materials, an accurate resin content measurement (weight %) can be made by use of the relatively flat sector of the TGA curve prior to the final drop-off indicating rapid degradation. The identifiable flat region of the curve, however, is not classic for many materials. In case of carbon composites, this may occur if the fibers or their coatings contain a significant amount of volatile impurities, the degradation of which coincides with that of the resin matrix. Lenoe has noted the possibility that sample size has an influence on the observation as appeared to be the case for several Thornel-50 epoxy materials considered[56].

The procedure involves grinding the test sample and thoroughly mixing to yield a representative powder from which the samples are taken. Nominal sample weights of 100 mg are utilized. The sample is introduced into the instrument pan and the temperature is increased at a preselected constant rate - 5° to 10°C/min. The strip chart records sample weight loss versus temperature from ambient to a selected maximum temperature or until the sample is consumed. Resin content is determined directly from the TGA curve based on percent of total sample weight loss. The test procedure, however, involves a number of factors which can lead to variations. If the analysis is conducted employing ambient atmosphere, resultant variations in moisture content and gas composition should be considered. Variations in gas flow over the specimen as well as heating rates similarly should be considered. If the TGA method is employed to accurately characterize void content in a composite, particularly at low void volume fractions, the specific effects of sample size, heating rate, atmosphere and gas flow or velocity over the specimen must be defined.

2.5.1.2.2.4 Point Count

The basic "point-count" technique has been previously defined as a method of fiber content determination. This method is particularly applicable to determination of void content. In comparison with other techniques discussed the point-count technique characterizes the distribution and orientation of voids and provides a better statistical representation of the composite material examined. Figure 2-17 illustrates such volume fraction determinations versus flexural strength.

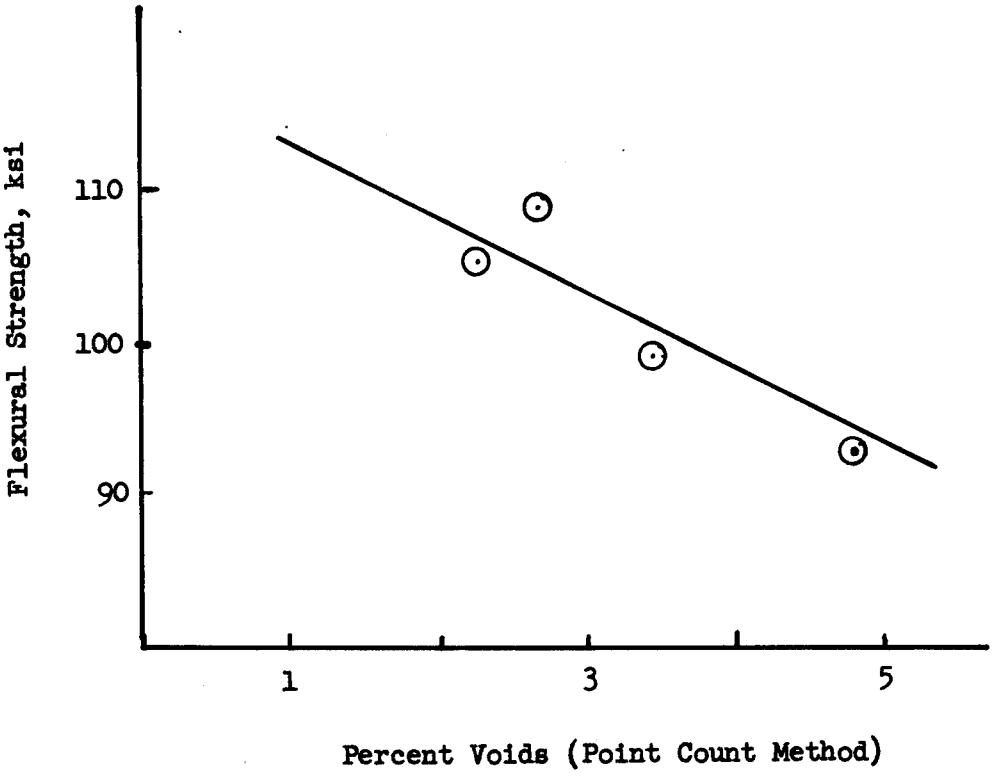


FIGURE 2-17. EFFECT OF VOID CONTENT ON COMPOSITE FLEXURAL STRENGTH[56]

2.5.1.2.2.5 NDT Methods

The application of NDT methods to the evaluation of advanced filamentary composite materials has been extensively investigated[56-60]. Test methods developed fall into two categories; namely, those utilized for characterization of flaws, for example, voids, cracks, delaminations, and filament discontinuities; and those employed in determination of mechanical properties.

Nondestructive test methods more applicable to void content and characterization are ultrasonic scanning, utilizing through transmission or pulse echo techniques, and radiography. In the case of ultrasonic scanning, energy passing directly through the test piece is recorded by the through transmission technique. Energy reflected from a discontinuity, or its related effect to the total response, is recorded by the pulse echo technique. Through-transmission has the advantage of being relatively unaffected by surface geometry, topography, and alignment of the test piece. Further only one signal is received for processing the total pass-through energy. The pulse echo method, however, is significantly affected by the above surface and alignment factors since the energy or sound passes through the test piece twice and thus results in higher attenuation and sound scatter degradation. Schultz[53] inspected graphite-epoxy panels using the pulse echo technique at a frequency of 5 Mhz. Hagemaier[58] conducted similar pulse echo inspections of boron, graphite, and glass resin composite face sheets (aluminum-core honeycomb specimens) at frequencies of 10, 10, and 2.5 Mhz, respectively. In view of current state-of-the-art detection employing ultrasonic techniques, results are largely dependent upon the experience of the operator. This is particularly true in the case of thin specimen panels in which delineation of back-echoes from internal or interior echoes is quite difficult. Although ultrasonic velocity measurements are known to be influenced by composite void characteristics such as distribution, volume, shape, and quantity, the sensitivity of such measurements are too low to permit correlation with any of these characteristics other than a crude estimate of component volume fraction.

The effective use of radiography in quantitative characterization of voids in filamentary composites is also limited. Use of x-ray radiography for void content analysis in graphite-resin systems is ineffective because of the close similarity in x-ray absorption coefficients of the constituents. Similarly, the effective use of neutron radiography for void content analysis of boron filament composites is limited. Thermal neutrons are highly attenuated by boron; thus a high density boron composite would be completely opaque to a thermal neutron beam.

Nondestructive test techniques, as applied to void content and porosity, require further development and must be optimized for particular composite systems to achieve high resolution for quantitatively predicting content as well as size, shape, and distribution and their specific influences on mechanical property degradation.

2.5.1.2.3 Recommendation

Composite ignition is recommended as the quality control or material acceptance test method on the basis of simplicity and obtainable accuracy. This method is applicable to composites in which the effects of ignition on its constituents are known. The recommended ignition interval is 8 hours at a temperature of 400-410°C, ±5°C, since limited oxidation of most available grades of fibrous graphite can occur in excess of this. Tests have shown in excess of 2% oxidative weight loss in carbon yarn after an ignition period of 24 hours at 415°C, as compared with less than 0.5 percent for an 8-hour interval at the same temperature. Specific corrections for both significant fiber weight loss in the case of carbon, and incomplete burnoff or combustion of resin, as well as the scrim cloth in the case of boron, must be applied to enhance and maintain accurate, reproducible results. In those instances in which oxidative weight losses are excessive for specific graphite fiber types, for example, Courtaulds Type A and Type HT*, chemical digestion (mineral acid) technique is recommended. A minimum of three tests should be conducted for each sample. Test methods delineated above for determination of fiber and resin volume fractions are also applicable to determining composite void content. The most accurate and comprehensive approach involves the metallographic "point-count" technique. This procedure provides information on the number, shape and distribution of voids. This method, however, is time consuming and is applicable to a more detailed study and analysis than is required for a material acceptance standard or test method. An acceptable composite may have one percent voids or less as opposed to a poorly fabricated composite which exhibits considerably higher void fractions. Higher void fractions usually imply lower fatigue resistance, increased susceptibility of fractions to water penetration, and increased scatter in strength properties of the composite. For purposes of quality control, the composite ignition or resin burnoff method is recommended. The accuracy of this method is capable of defining at least the lower limits of void content, namely, one volume percent or slightly less which exhibit minimal effects on composite mechanical properties. A minimal number of 3 tests

*Trade name. Courtaulds, Ltd., Coventry, England

is recommended for each sample. Ignition test parameters and required corrections, if any, as detailed for both resin and fiber volume fraction measurements apply directly to void content determinations.

2.5.1.3 Thermal Expansion

2.5.1.3.1 General

The significance of thermal expansion characteristics of filamentary composites is commonly overlooked as an important factor in their basic design and use. Thermal deformation characteristics of composite laminates can be grossly affected by fiber orientation, laminate stacking sequence, and the thermal expansion characteristics of the epoxy matrix material. In the case of resin matrices, expansion behavior is directly influenced by the following factors:

(1) Resin type

(2) Resin formulation, i.e., the specific catalyst or curing agent and the amount of such agents

(3) Curing cycle

The effects of the latter two factors were demonstrated by E. L. Harmon, et al.[63], who obtained thermal expansion curves on cast epoxy resin system and cured unidirectional laminates both parallel and transverse to the longitudinal fiber axis. The initial results obtained on the cast resin and composite specimen transverse to the fiber axis show type "S" curves as illustrated in Figures 2-18 and 2-19. A shift from expansion to contraction occurred between 150°F and 210°F (65°C and 99°C). Analysis of results indicated that the "S" curve was due to additional shrinkage of the specimens as a result of the "post cure" inadvertently carried out in the normal process of the thermal expansion measurement. As shown in Figures 2-20 and 2-21, the "S" or contraction segment of the curve was eliminated by post curing in the dilatometer with prolonged soak times in the 150° to 210°F (65°C to 99°C) range. An approximate shrinkage of 1.5 percent, however, occurred as a result of the post cure. Matrix shrinkage was completely eliminated by a 2-hour post cure at 350°F (177°C).

Figure 2-22 illustrates coefficient of expansion of a unidirectional graphite-epoxy composite specimen. The measurements were obtained parallel to the longitudinal axis of the fibers. In a unidirectional composite, the thermal expansion coefficient can be

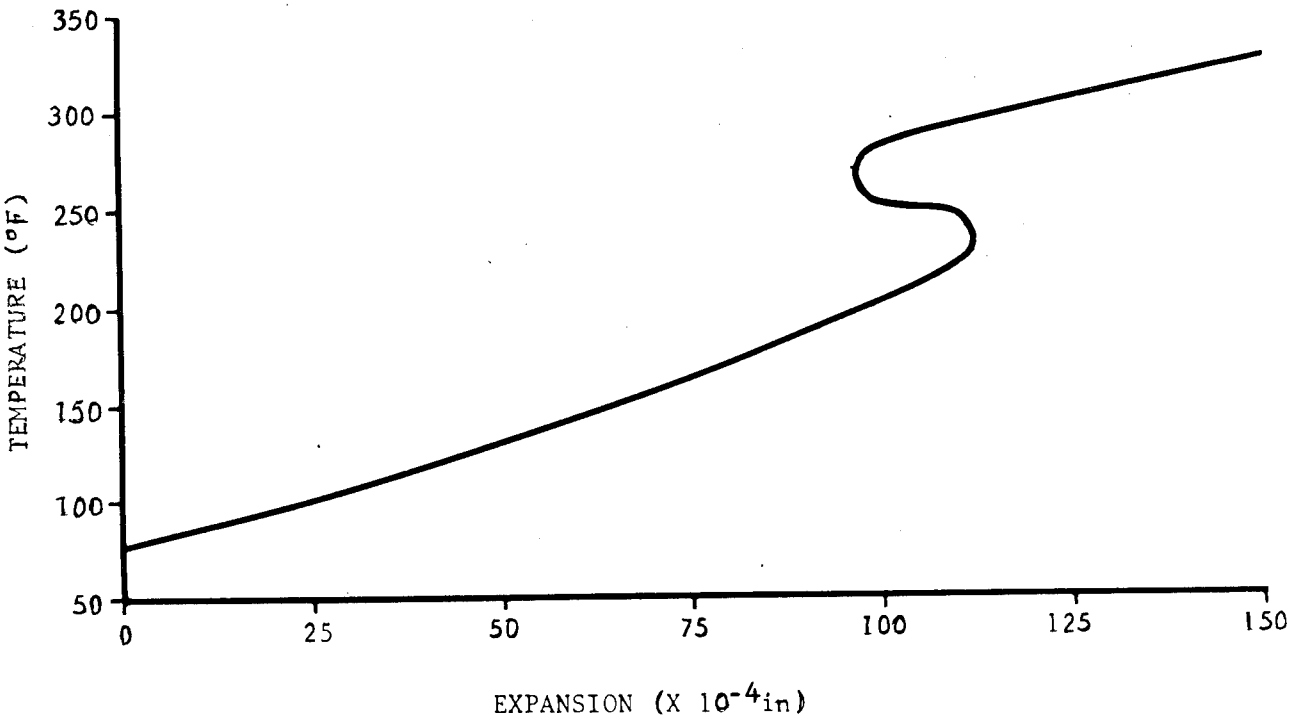


FIGURE 2-18. TYPICAL EXPANSION CURVE OF E-798 CAST RESIN PRIOR TO POSTCURE[63]

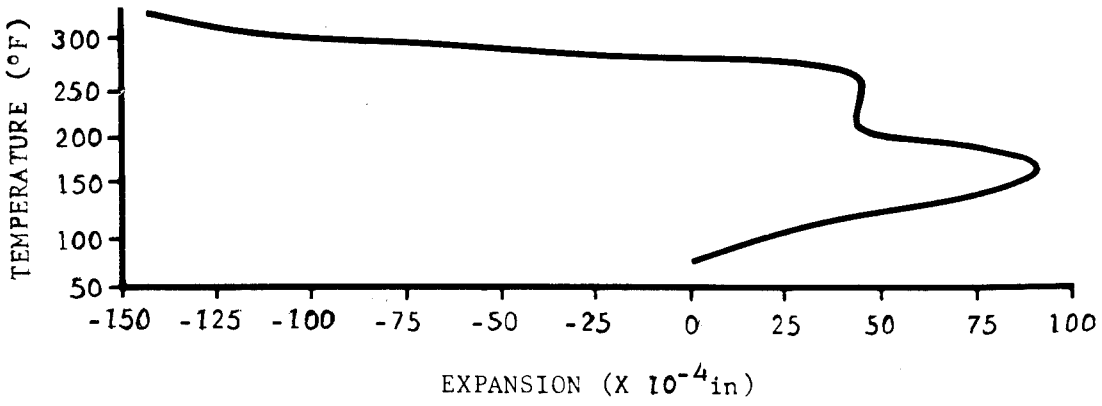


FIGURE 2-19. TYPICAL EXPANSION CURVE OF TRANSVERSE SPECIMEN OF THORNEL 50/E-798 EPOXY LAMINATE PRIOR TO POSTCURE[63]

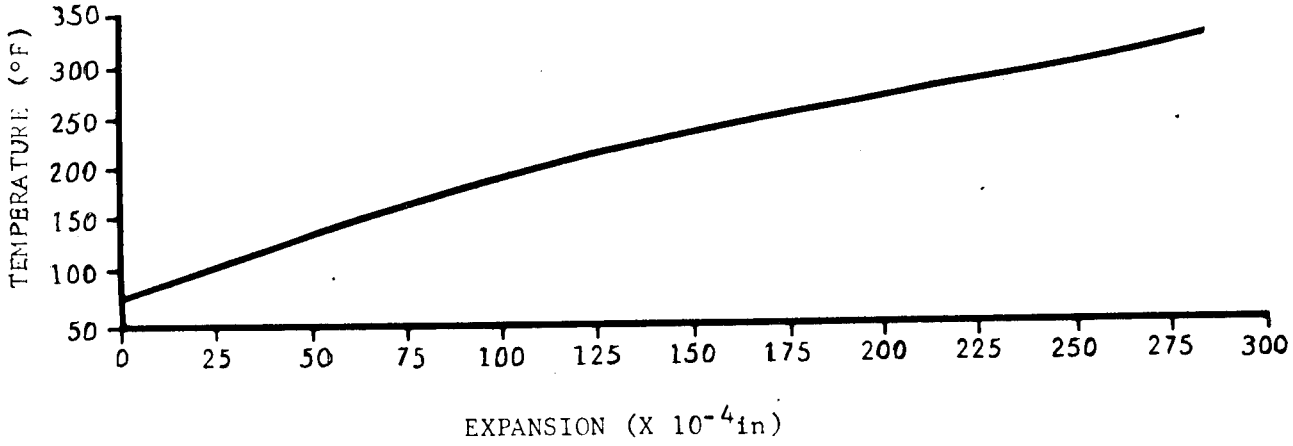


FIGURE 2-20. TYPICAL EXPANSION CURVE OF E-798 CAST RESIN AFTER POSTCURE[63]

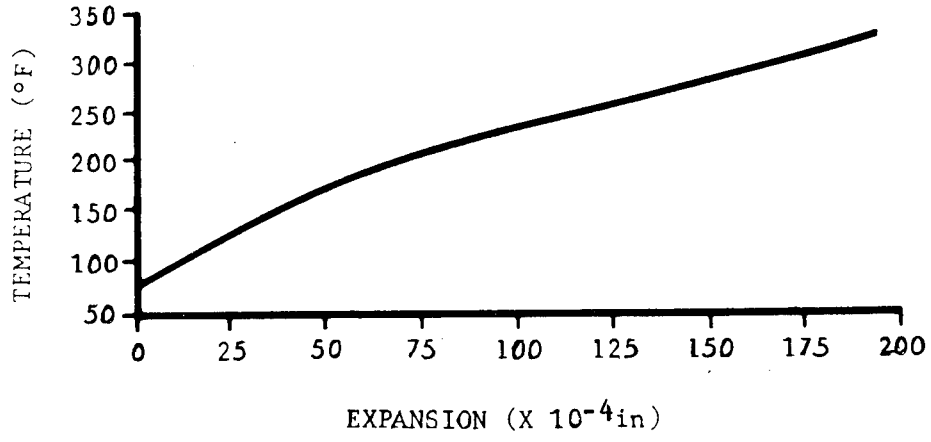


FIGURE 2-21. TYPICAL EXPANSION CURVE OF TRANSVERSE SPECIMEN OF THORNEL 50/E-798 EPOXY LAMINATE AFTER POSTCURE[63]

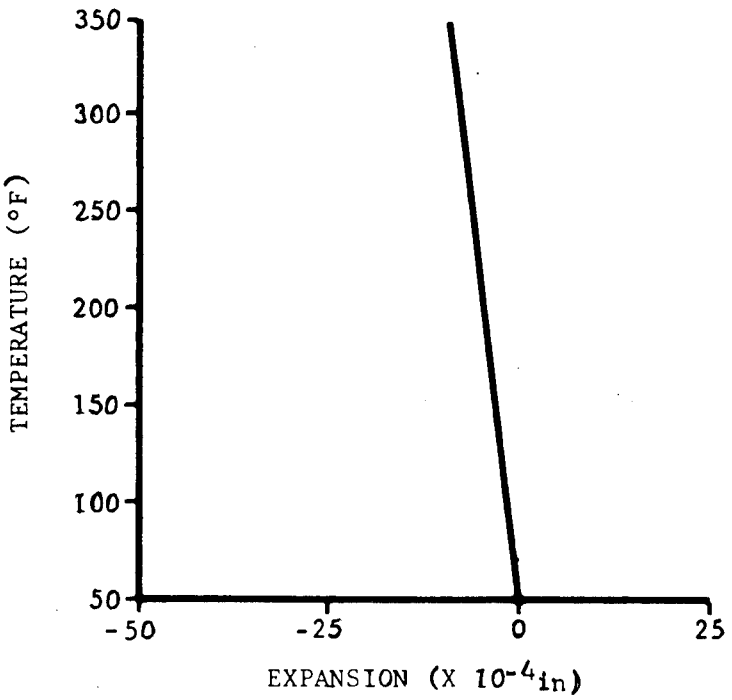


FIGURE 2-22. EXPANSION CURVE OF LONGITUDINAL SPECIMEN OF THORNEL 50/E-798 EPOXY LAMINATE

predicted in the longitudinal direction from the following equation which is based on equilibrium and compatibility considerations[1,3]

$$\alpha_L = \frac{\alpha_m V_m E_m + \alpha_f V_f E_f}{V_m E_m + V_f E_f}$$

where α_L = thermal expansion coefficient (longitudinal direction-unidirectional composite)

α_m = thermal expansion coefficient of matrix

V_m = matrix volume f

E_m = matrix modulus

α_f = thermal expansion coefficient of fiber

V_f = fiber volume fraction

E_f = fiber modulus

In graphite epoxy composites, the tensile modulus of the fiber is two or three orders of magnitude greater than that of the matrix. This, therefore, makes the longitudinal coefficient of the unidirectional composite nearly identical to that of the fiber as indicated by the above equation. Thermal expansion coefficients of a unidirectional composite thus vary with the angle between the direction of measurement and that of the fiber as illustrated in Figure 2-23[1]. The measurements were conducted at 100°C. The transverse expansion coefficient of the unidirectional composite indicates that the constituents exert a more proportionate effect. The coefficient also shows a sensitivity to the configuration of the fiber in the matrix.

The relation of thermal expansion coefficient to various balanced 6-ply graphite epoxy composites is illustrated in Figure 2-24[1]. It is noted that the coefficient, which is negative in the longitudinal direction, remained negative up to a ply angle of 30°. Figure 2-25 illustrates a similar phenomenon in the boron epoxy system both at room temperature and 340°F (171°C). The value of the coefficient, in both instances, falls slightly below the longitudinal value, reaching a minimum at approximately 18° in the case of the graphite epoxy and 30° in the case of the boron epoxy system. This behavior was observed experimentally by Halpin and Pagano[4] in the swelling of composites. Data for the theoretical curve shown in Figure 2-24 was derived using Halpin and Pagano's approach.

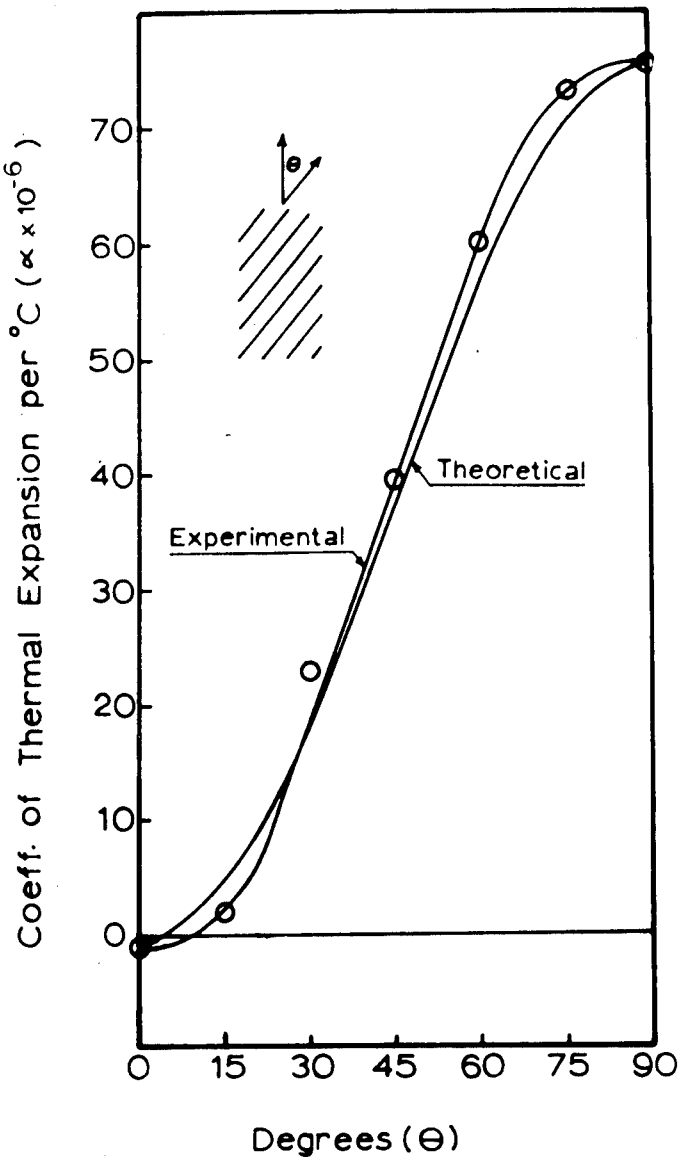


FIGURE 2-23. ANGULAR VARIATION OF THERMAL EXPANSION COEFFICIENTS FOR UNIDIRECTIONAL GRAPHITE-EPOXY COMPOSITES[1]

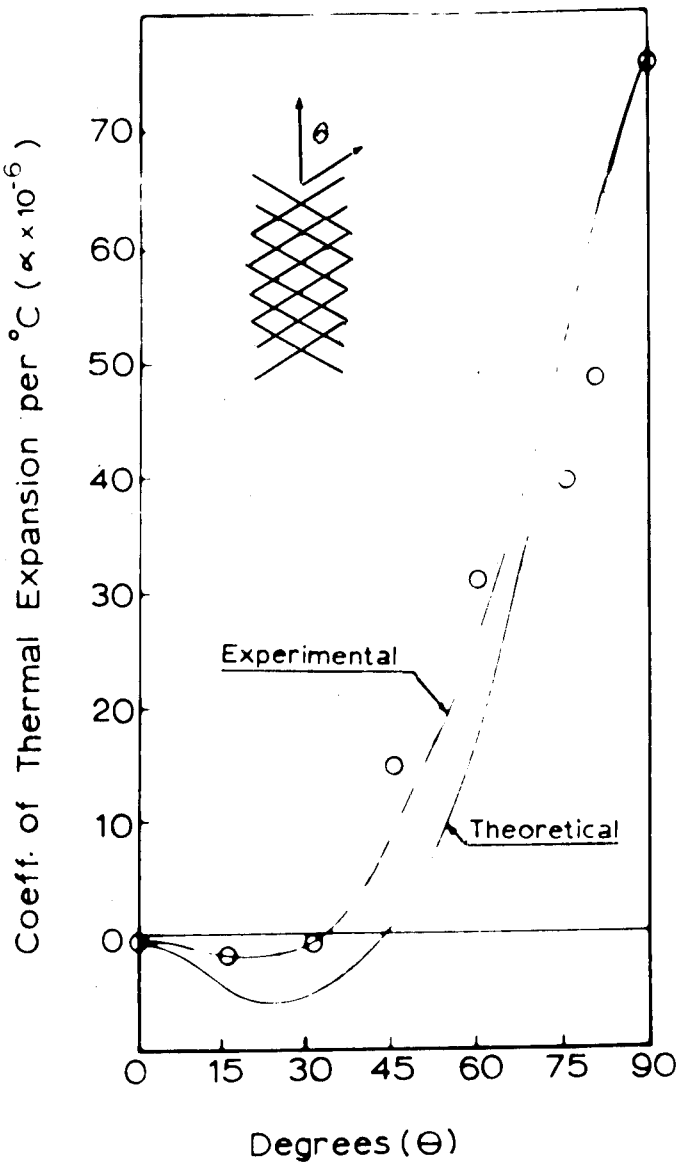


FIGURE 2-24. THERMAL EXPANSION COEFFICIENTS OF ANGLE-PLY GRAPHITE-EPOXY COMPOSITES AS A FUNCTION OF PLY ANGLE[1]

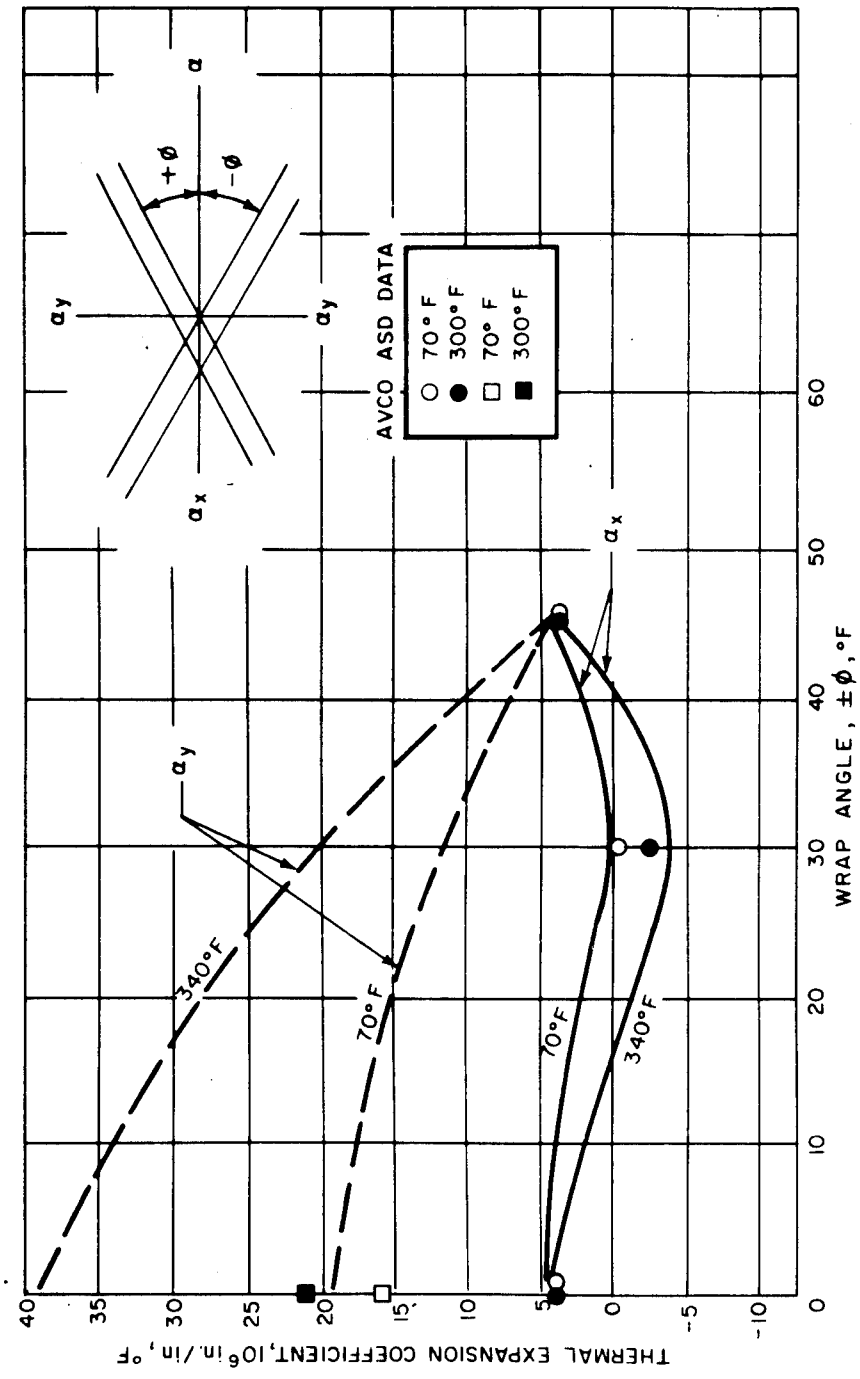


FIGURE 2-25. THEORETICAL AND EXPERIMENTAL THERMAL EXPANSION COEFFICIENT 50-VOLUME PERCENT BORON EPOXY[38]

The recommended procedure for thermal expansion measurement of composites, inclusive of specimen preparation, is detailed in ASTM Designation D-696-70[36]. Major emphasis has been directed toward the thermal expansion behavior of epoxy resins and advanced filamentary-epoxy composites because of the significant influence of such behavior on their strength properties, dimensional stability and mechanical compatibility in structural assemblies.

2.5.2 Mechanical Properties

2.5.2.1 Tension (Uniaxial)

A large variety of coupon specimen types have been used in determining the tensile properties of advanced filamentary composites. In the attempted adaptation of the conventional "dog-bone" configuration to composite testing, a buildup of stress concentrations at the intersection of the tab fillet and the test section was observed. This resulted in premature tensile failures within this sector of the specimen and yielded low strength values. These results stimulated design and use of flat, rectangular laminate coupons with end tabs. Load introduction is made through the bonded tabs which provide for lateral and longitudinal compliance as required to inhibit end failures. Figure 2-26 illustrates a variety of tab coupon configurations and materials.

The straight-sided, tab-ended tensile coupon is the most widely used specimen configuration and, with care in testing, can provide acceptable test results. Use of this basic IITRI type coupon[64] and the test procedure involved is recommended as an acceptable quality control test method for unidirectional and angle-ply boron-epoxy and graphite-epoxy composites. Figure 2-27 illustrates a modified IITRI type longitudinal tensile coupon applicable to both boron-epoxy and graphite-epoxy systems and recommended for use with unidirectional laminates, or laminates with the predominant reinforcement aligned with the load axis. A nominal thickness of 0.040 inch is recommended. End tabs should be bonded with a high temperature adhesive which will allow elevated temperature tests within the 350°F to 400°F (177°C - 204°C) regime. Tests should be conducted using a tensile machine with controlled constant crosshead speed at a rate of 0.02 to .05 in/min. Strain measurements may be monitored by a strain extensometer or single element axial strain gages.

Use of strain gages to monitor longitudinal deformation should be bonded back-to-back with the center of each gage accurately aligned within the center of the coupon gage

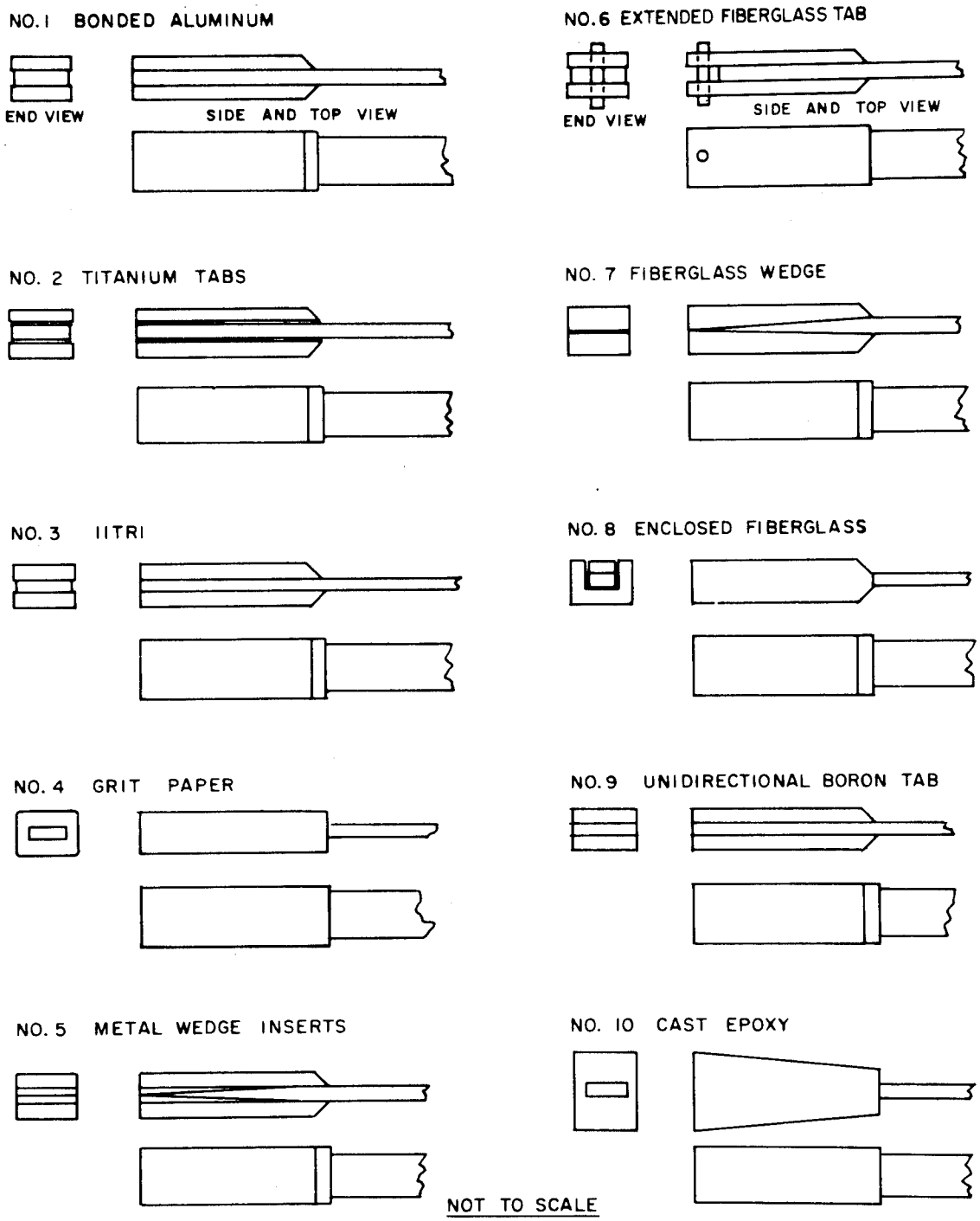


FIGURE 2-26. TAB CONFIGURATIONS FOR STRAIGHT-SIDED TENSILE COUPONS[38]

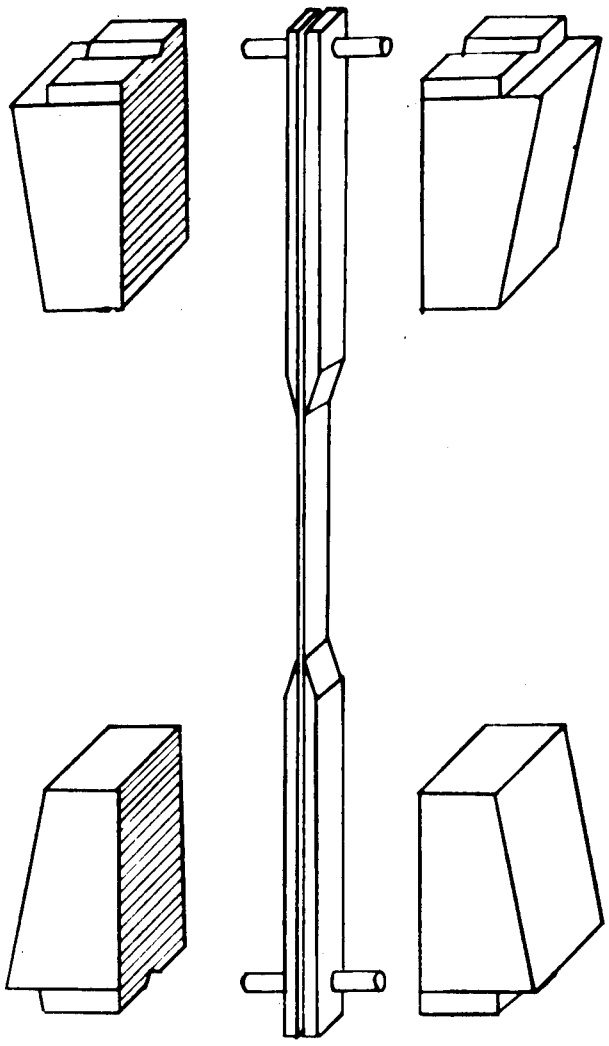


FIGURE 2-27. TEST CONFIGURATION INCLUDING SELF-ALIGNING FIXTURE[104]

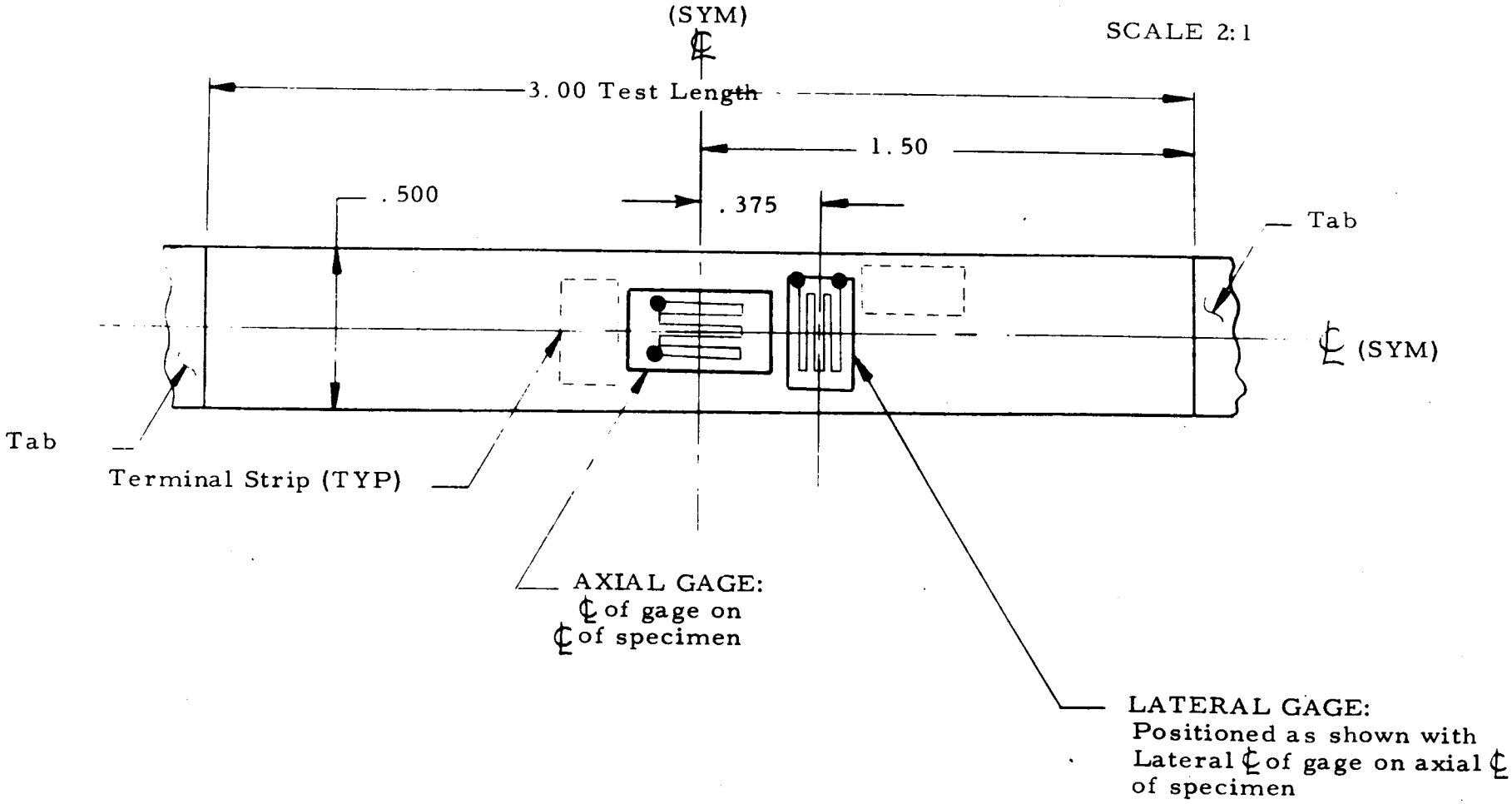
section. For simultaneous measurement of longitudinal and lateral deformation associated with axial tensile tests determining Poisson's ratio, back-up strain gages, as illustrated in Figure 2-28, should be used. Use of the averaging-type strain gage extensometer is recommended for routine tensile tests as required for materials screening and acceptance. For elevated temperature tests, micro-measurement strain gages, Type SA-06-187BB-120 or equivalent, can be used.

Tensile coupons should be mounted in the test machine employing standard file-faced, self-aligning grips. To minimize introduction of bending and/or torsional forces within the coupon as a result of poor axial alignment in the grips, the wedge-type grips should be modified as illustrated in Figure 2-27, to permit use of centering pins. The modification markedly improves axial alignment and further functions to diminish the tendency of specimen slippage within the grips. A test is considered valid only when failure is confined within the test region or gage length; that is, at least one specimen thickness away from the bonded tab ends.

The transverse laminate tensile specimen, as shown in Figure 2-29, does not require end tabs. No. 240 emery paper is sufficient for use between the specimen and the test grips to protect the specimen. The nominal specimen thickness recommended for both boron-epoxy and graphite-epoxy laminates is 0.080 inch.

Significant factors which can influence the mechanical behavior and responses of the straight-sided boron-epoxy tensile coupon have been treated by Lenoe, et al[65]. These include specimen preparation, tab material and the adhesive employed, strain rate, and specimen dimensions. These variables and their specific effects are also applicable to graphite-epoxy composite coupons.

Specimen preparation includes the basic laminating process as well as the cure and post-cure cycles employed. The process should be governed to yield maximum but reproducible composite properties. No special surface treatment is required for application of tabs to the coupon other than that stipulated for the specific tab adhesive utilized. Of the variety of tab materials available the fiberglass tab and design is recommended. The fiberglass tab is adequate for elevated temperature test as governed by the capability of the adhesive employed. Adhesives requiring a maximum cure temperature-time cycle of 350°F (177°C) and 1 hour at 25 psi pressure exhibit useful temperature ranges from -324°F (-198°C) to a maximum of 600°F (315°C). In the application and use of a selected



NOTE: Gages are to be backed-up

FIGURE 2-28. STRAIN GAGE POSITIONS[104]

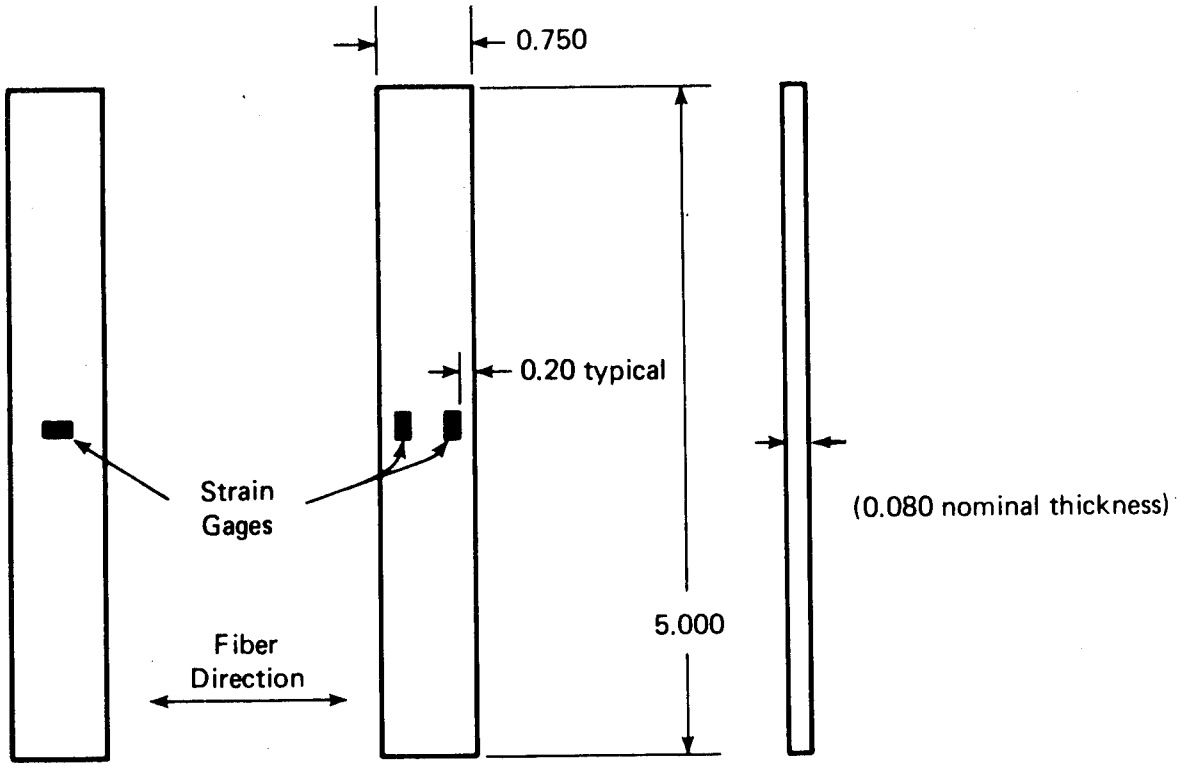


FIGURE 2-29. TRANSVERSE TENSILE TEST SPECIMEN

adhesive for tab bonding, uniformity of the bond line is an important factor in obtaining reproducible test data.

Strain rate dependence of filamentary reinforced resins is strongly influenced by the resin materials. Strain rate sensitivity tests were conducted at room temperature on unidirectional boron-epoxy coupons over the range of 0.008 to 0.60 in/in/min[38]. Similar responses were observed for each strain rate. There was no observable difference in mechanical properties and behavior. Strain rate sensitivity evaluation of resin (including tensile, compression, and shear properties at 73°F (22.7°C) and 350°F (177°C) for strains of 0.005, 0.05, and 0.5 in/in/min, as well as thermal expansion, tension creep and relaxation), indicated approximately 20 percent cumulative change in composite modulus, strength, and strain to failure over the range of two decades of strain[65]. This viscoelastic behavior would be most evident in crossply and angle-ply composites; negligible influence would be observed in unidirectional composites.

For material acceptance or quality control test methods and procedures, it is advisable to set a uniform strain rate. Under special testing conditions and certain specific applications; however, time rate effects must be considered.

The effects of specimen dimension, i.e., length, width, and thickness, appear to exert little influence on unidirectional composite tensile coupons. Data generated from an investigation of boron-epoxy coupon lengths of 4-1/2 and 9 inches, width .25 to 1.0 inch, and thickness of 6, 12, and 18 plies, is illustrated in Figures 2-30 and 2-31. The scatter shown was attributed to tab bond alignment and edge constraints. The data does suggest, however, some decrease in tensile strength for specimens with a thickness in excess of 12 plies, and this indicates a maximum limit on specimen thickness.

The influence of specimen width on cross-ply and angle-ply composites is illustrated in Figure 2-32[65]. Coupon specifications are indicated in the figure. For the 0°/90° and ±22°, a 10% strength decrease with increasing width is shown. In contrast, strength increases in excess of 20 percent for ±60° orientations is observed with increasing width.

2.5.2.2 Compression

Compression test procedures can be characterized by the method of applying load to the test area and the specific method of stabilization employed. The principal loading procedures are: direct end loading of the specimen, and beam bending. The major methods

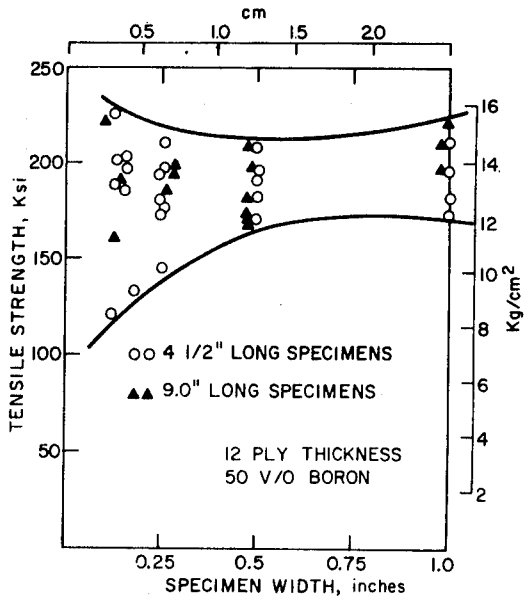


FIGURE 2-30. INFLUENCE OF SPECIMEN WIDTH AND LENGTH ON TENSILE STRENGTH OF UNIDIRECTIONAL BORON EPOXY[65]

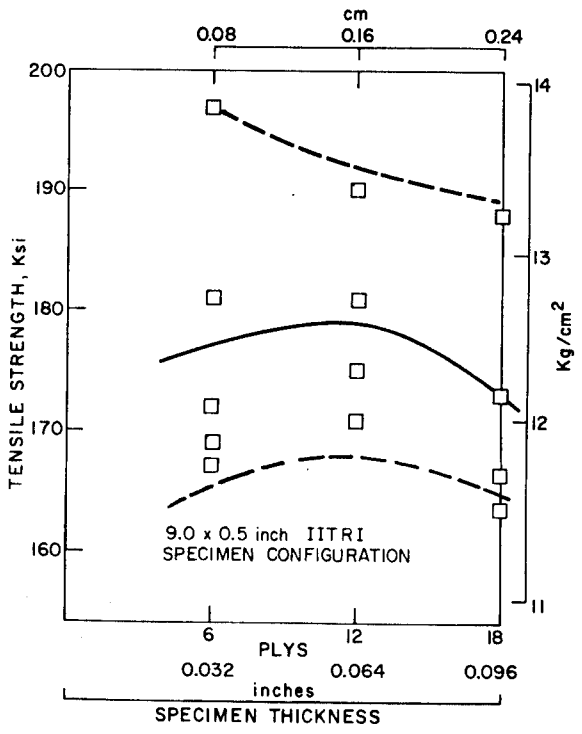


FIGURE 2-31. INFLUENCE OF PLY THICKNESS ON TENSILE STRENGTH OF UNIDIRECTIONAL BORON EPOXY[65]

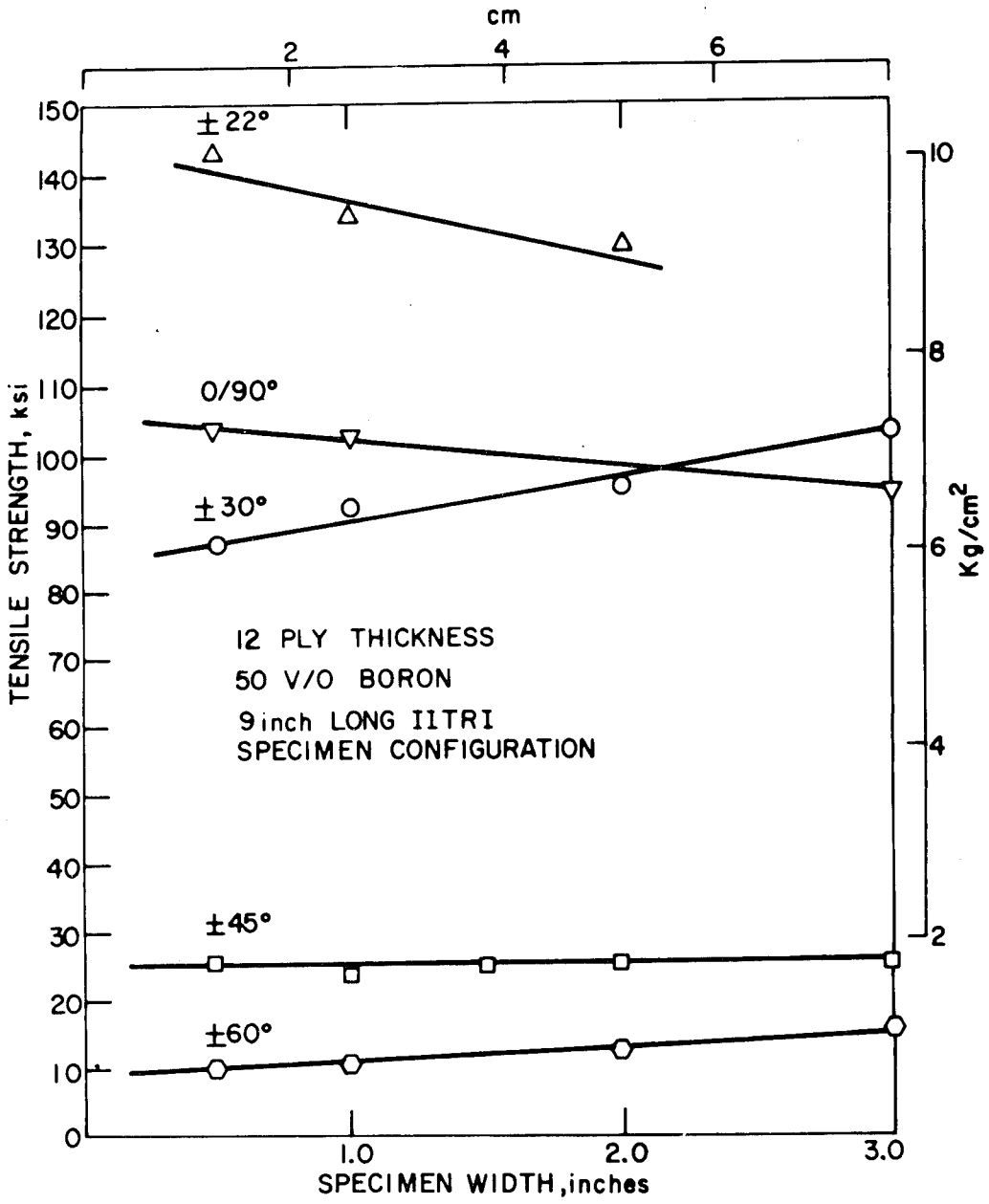


FIGURE 2-32. INFLUENCE OF SPECIMEN WIDTH ON TENSILE STRENGTH OF CROSS-PLY AND ANGLE-PLY BORON EPOXY[65]

of stabilization include plate, short-column, honeycomb, and finger. The method more readily adaptable to quality control and material acceptance procedures is the rectangular plate type coupon specimen as detailed in ASTM Specification D695-69. The basic sample specifications and test procedure is treated in Section 2.3.3.

In the determination of compression properties, however, it would appear that one design cannot satisfy the accuracy required for both strength and modulus measurements. The straight-sided specimen provides accurate modulus measurements, however, end crushing, even in the presence of restraining clamps, inhibits ultimate strength measurements. A necked specimen with an abbreviated gage length should afford added stability as required for ultimate strength determinations.

Hogatt[66] employed a straight-sided graphite epoxy specimen (3-inch length by .502-inch width) for modulus determinations using an ASTM Class B-2 extensometer. The specimen was loaded to 50 percent of ultimate at a crosshead speed of 0.05 in/min. Compressive strengths were determined using the necked specimen. All failures with the necked specimen were confined to the center of the gage length which proved satisfactory for unidirectional laminates. For comparison, the compressive properties of unidirectional and multi-directional composite materials was determined employing sandwich beam and tubular specimens, respectively. The sandwich beam gave slightly higher properties, while tubular specimens gave markedly higher strength and modulus than the corresponding flat or plate specimen. The plate specimen data, however, exhibited significant scatter at fiber loadings ranging from approximately 0.47 to 0.60 volume fraction. As expected, compressive strength increased with fiber volume fractions. The values, however, were considerably lower than theoretical compressive values. This was attributed to the twisted graphite yarn. Dastin, et al.,[67] employed a modification of the test fixture recommended in the above specification and obtained satisfactory failure modes and ultimate strengths for several conditions of fiber loadings and test temperatures. The fixture effectively inhibited edge crushing, end brooming, and buckling of the plate-type specimen. The ultimate stresses were reproducible and exhibited minimal scatter. Measurement of modulus using a LVDT (compressionmeter) attached to the edge of the specimen yielded erratic results at various strain levels and induced stiffness reversal in some specimens. Thus, only the initial strain measurements are accurate. Compressive properties are shown in Figure 2-33. The more immediate advantages derived from the improved compressive specimen and test fixture are cited as follows:

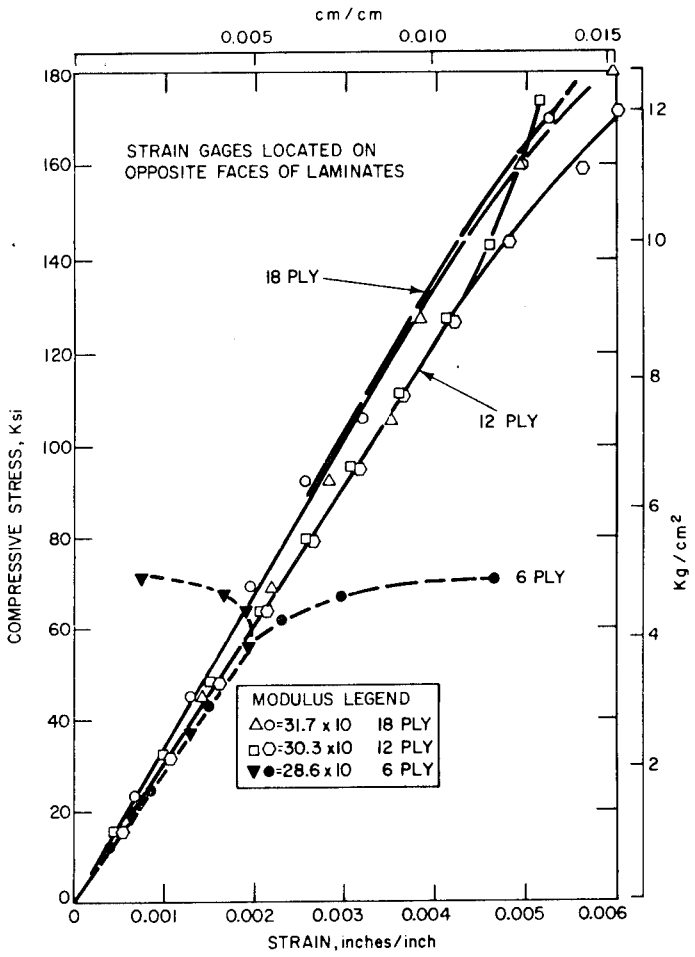


FIGURE 2-33. TYPICAL COMPRESSIVE STRESS-STRAIN RESPONSE OF BORON EPOXY[65]

1. A resonable size specimen is utilized; that is, 3/4 inch wide by 3 inches long, applicable over a thickness range of 0.06 to 0.125 inch. These dimensions closely parallel those specified in ASTM Specification D-695-69.
2. Test method is usable over a temperature range of -65°F (-54°C) to 450°F (232°C) and is both simple and in expensive. Further, both strength properties and failure modes are consistent and satisfactory for a compression coupon.
3. The method offers a potential approach for direct strain measurements utilizing a simple, clip-on compressionmeter (LVDT).

Boron-epoxy compression coupons of tubular and flat configuration (as specified in ASTM Specification D-695-69) were evaluated by Lenoe, et al[65]. Tolerance and alignment were found to be extremely crucial in minimizing scatter and obtaining high strength values. In rectangular coupons of laminates of unidirectional and 90° fiber orientations, the maximum obtainable nominal strengths were a strong function of specimen geometry. The parameters investigated were ply level, length, and width. Strength variations >100 percent as a function of gage lengths were noted. Influence of these parameters are illustrated in Figure 2-34[65].

Tubular coupons yielded relatively small strength variations. Ultimate strength values were considerably higher than for rectangular coupons, as previously noted in the case of graphite epoxy laminates[66].

The compression test method recommended for screening and acceptance of boron-epoxy and graphite epoxy composites is the basic procedure detailed in ASTM Specification D-695-69, employing the rectangular coupon configuration. The method is both simple and economic and is responsive to both specimen and test fixture modifications for enhanced accuracy.

2.5.2.3 Flexure

Flexural test methods are treated in Section 2.3.2. The four-point flexural test method and specimen geometry detailed therein is recommended as an acceptance quality control test procedure for boron-epoxy and graphite-epoxy composites.

The flexural test is employed principally as a method for determining laminate quality since it simultaneously applies tension, compression and horizontal shear. The method

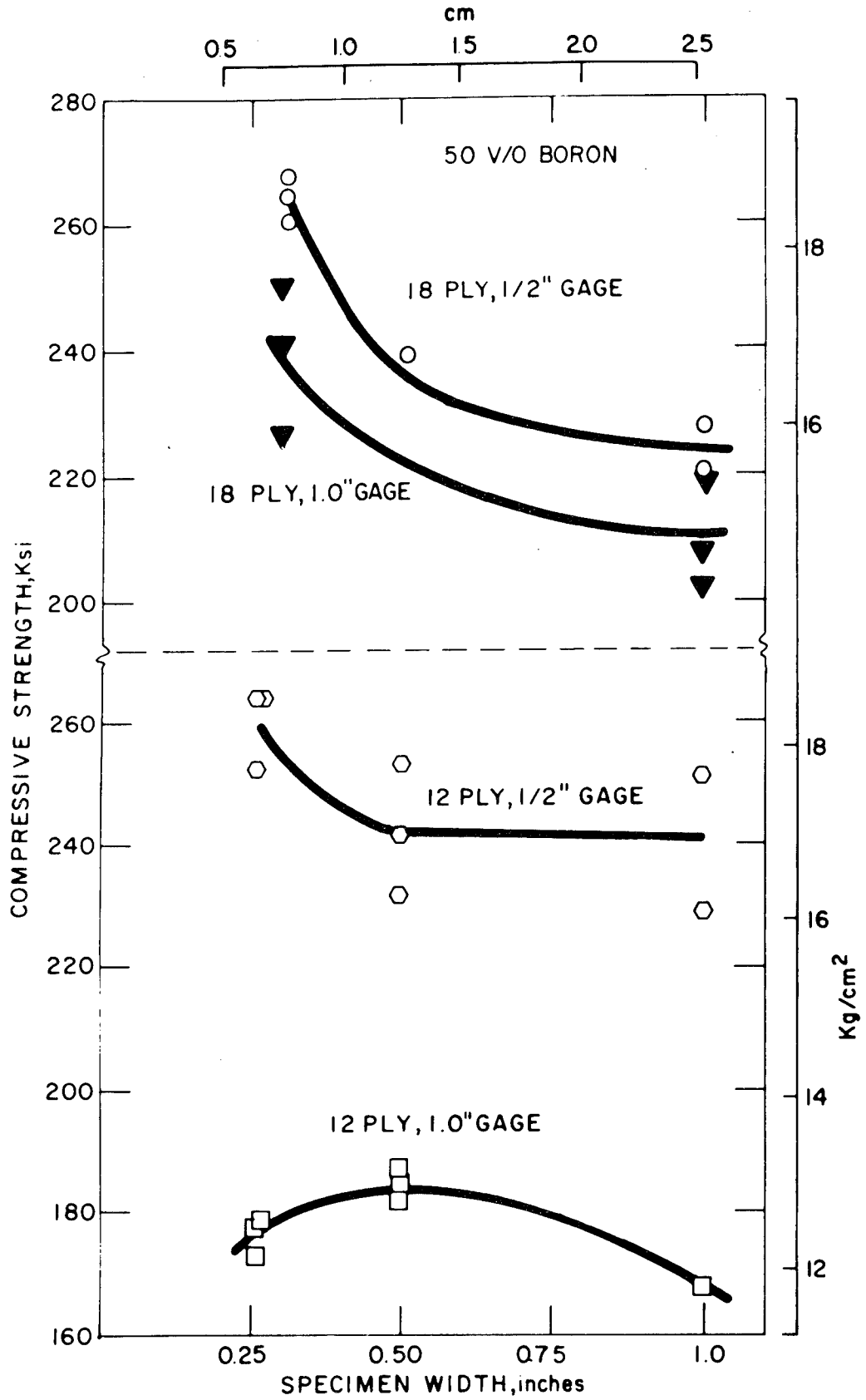


FIGURE 2-34. INFLUENCE OF SPECIMEN WIDTH, LENGTH, AND NUMBER OF PLIES ON COMPRESSIVE STRENGTH OF UNIDIRECTIONAL BORON EPOXY[65]

is not designed for establishing fundamental mechanical properties. Maximum failing stress, as calculated by the linear elastic (Mc/I) formula does not apply after the proportional limit is exceeded. Only an apparent modulus-of-rupture stress can be calculated. Stress calculations are further complicated in cross-ply laminates, since the longitudinal modulus value varies in each ply; only strain distribution remains linear. For quality control, however, interlaminar shear is the most effective measure of laminate quality.

Flexural strength and modulus are markedly influenced by several variables including filament orientation, filament loading, specimen configuration, span-to-depth ratio, and load point contact; that is, three- versus four-point loads, and the diameter of load contact points. Several test method standards recommended three-point specimen loading, and the specimens are generally similar in size[68,69,70]. A General Dynamics specification recommends 3-point loading for longitudinal tests as opposed to 4-point for transverse tests of unidirectional composites[71]. The rate of loading for several test procedures is based on the elasticity of the material treated.

Figures 2-35 and 2-36 illustrate influence of beam width, number of plies, and test temperature on the flexural strength of unidirectional boron-epoxy composites of span lengths of 2.0 and 2.5 inches, respectively. Figure 2-37 shows the influence of beam width, load contact diameter, span, and loading mode on the transverse flexural strength of unidirectional boron-epoxy composite samples.

2.5.2.4 Shear

Interlaminar shear as applied to material acceptance or quality control represents the most effective method for measurement of laminate quality. Currently, there are three basic specimen types for determining interlaminar shear strength; namely, the opposed double notch, torsion bar, and the short beam. The latter specimen type and test method is the recommended acceptance test. The Federal Test Method Standard 406, Method 1042, ARTC-11 Method VI, ASTM Specification D-2345-65, and Grumman specimens employ the opposed double notch both with or without stabilizing plates[68,70,72,73]. Notches must be deep enough to sever the middle ply for the full specimen width. Figure 2-38 illustrates clamped plates as applied to the specimen to inhibit or, at least, offset, peel action at the notch.

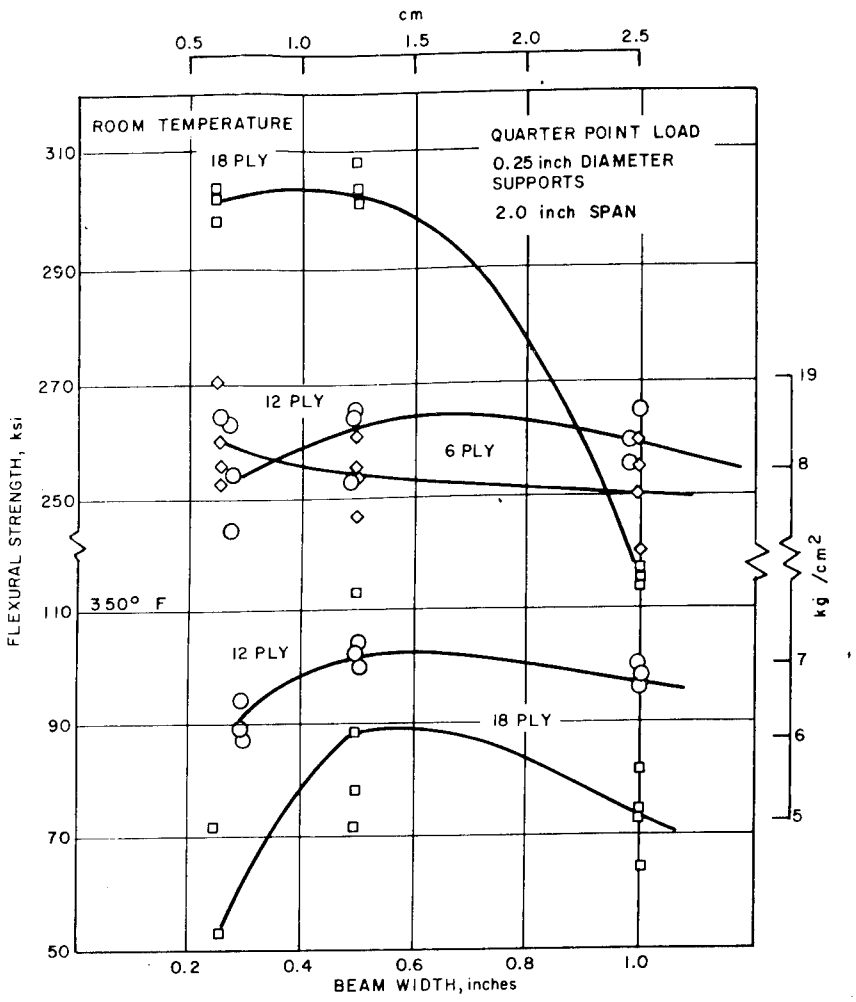


FIGURE 2-35. INFLUENCE OF BEAM WIDTH, NUMBER OF PLIES, AND TEST TEMPERATURE ON FLEXURAL STRENGTH (2.0-IN. SPAN) OF UNIDIRECTIONAL BORON EPOXY[65]

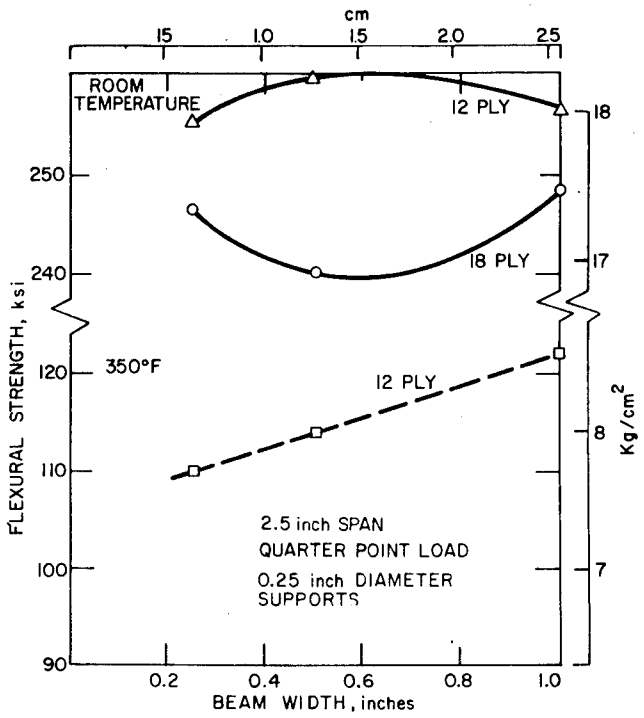


FIGURE 2-36. INFLUENCE OF BEAM WIDTH, NUMBER OF PLIES, AND TEST TEMPERATURE ON FLEXURAL STRENGTH (2.5-IN. SPAN) OF UNIDIRECTIONAL BORON EPOXY(65)

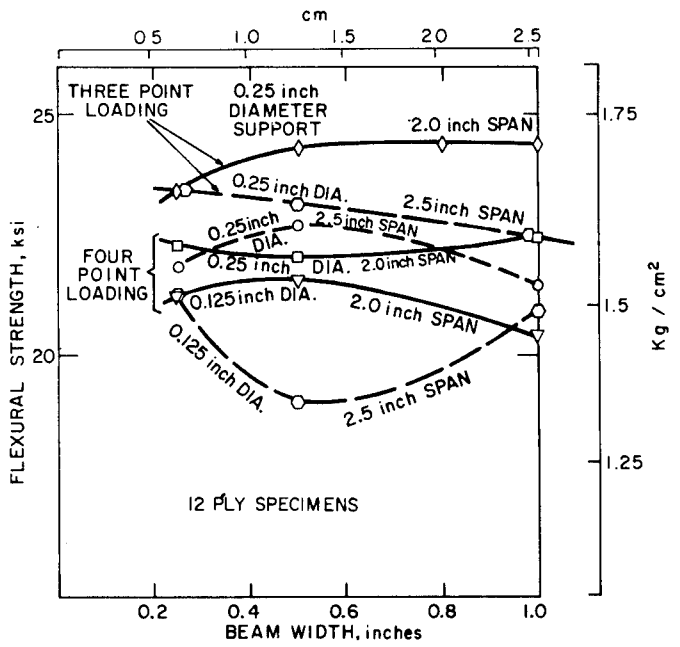


FIGURE 2-37. INFLUENCE OF BEAM WIDTH, LOAD SUPPORT DIAMETER, SPAN, AND LOADING MODE ON TRANSVERSE FLEXURAL STRENGTH OF UNIDIRECTIONAL BORON EPOXY(65)

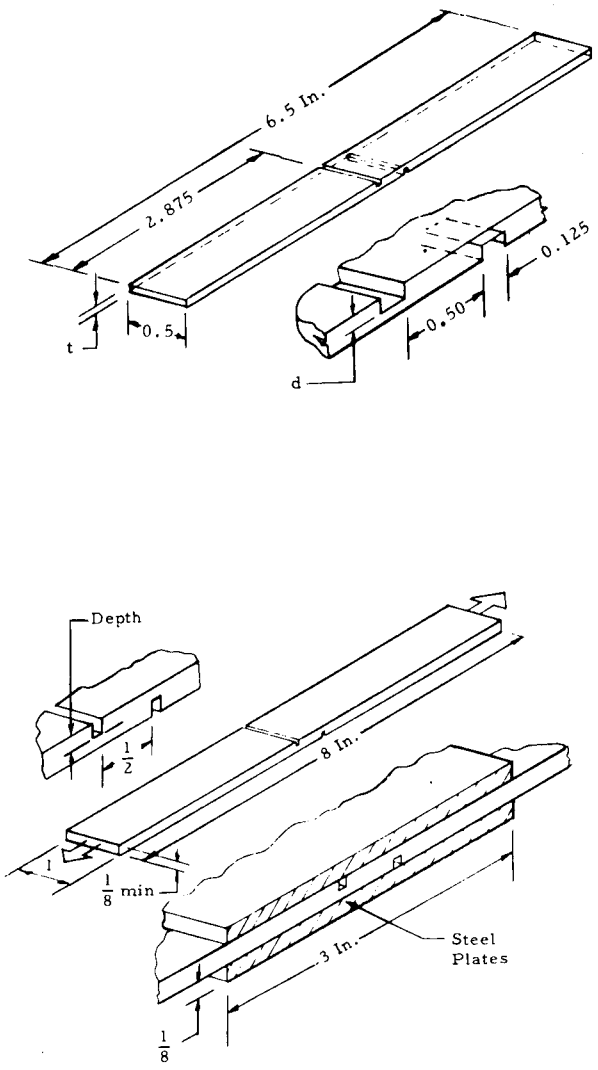


FIGURE 2-38. THREE TYPES OF COMPOSITE NOTCH-SHEAR SPECIMENS[68,70,72,73]

The major objection to the notched specimen, excluding the undesirable tendency of peeling, is the lack of uniform shear within the test section. Actually, maximum shear exists immediately adjacent to the notches with a correspondingly lower value occurring at the midpoint between the notches. This phenomenon resembles closely the shear loading distribution in a bonded lap joint. The stress yielded from this method is highly dependent upon the following factors:

1. Thickness of the laminate.
2. Distance between the notches.
3. Longitudinal modulus of the material tested.

The torsion bar or rod test method has been employed in many instances in the testing of graphite-epoxy composites for interlaminar shear strength. The test procedure simply entails loading a short unidirectional composite rod torsionally to failure. Maximum stress in the rod is calculated using the classical equation:

$$\sigma = 2T/\pi R^3$$

The equation, however, assumes a straight-line stress distribution will be existent. On the basis of this assumption, therefore, a considerably higher interlaminar shear stress will be indicated than can normally be achieved.

Of the three methods cited, bending of a short beam is the most suitable for use as an interlaminar shear test method. The method is defined in ASTM Standard D-732. The test method is significantly influenced by the following factors:

1. Specimen geometry, i.e., width, thickness, and length.
2. Fiber orientation.
3. Beam span.
4. Composite void content.
5. Load support diameter.

This test is considered satisfactory only when pure shear failure occurs. Nonshear or mixed mode failures constitute invalid data. Such failures are commonly generated when the interlaminar shear strength is too high with respect to the ultimate flexure strength, and when span-to-depth or width-to-depth ratio has been improperly selected. Figure 2-39 illustrates the effect of span-to-depth ratio on the apparent interlaminar shear strength and its relation to specific failure modes in graphite resin composites[66].

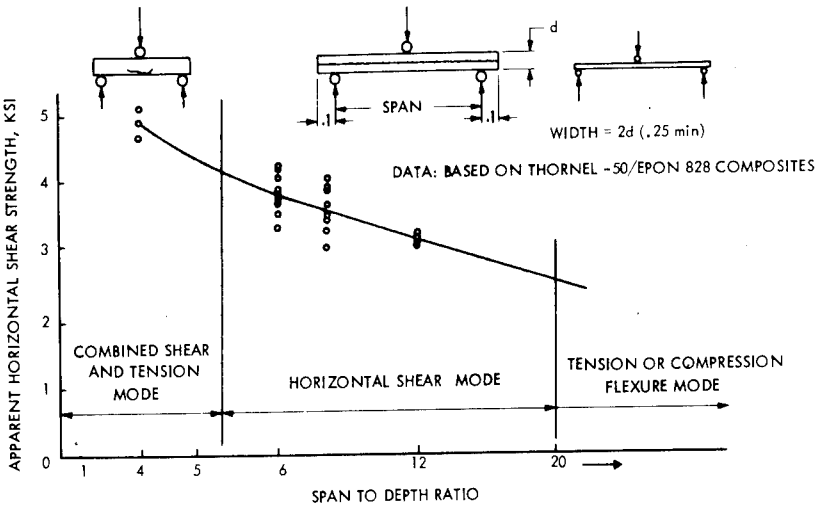


FIGURE 2-39. GENERAL CHARACTERISTICS OF INTERLAMINAR TEST FOR GRAPHITE/EPOXY COMPOSITES[66]

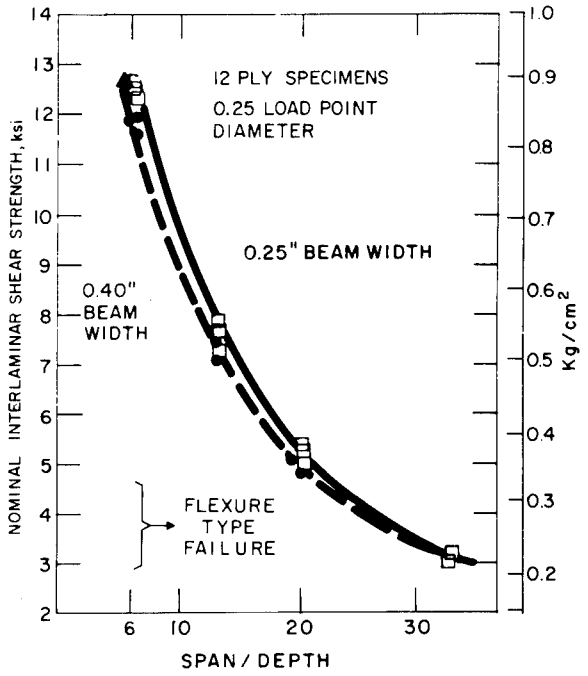


FIGURE 2-40. INFLUENCE OF SPAN TO DEPTH AND BEAM WIDTH ON NOMINAL INTERLAMINAR SHEAR STRENGTH OF UNI-DIRECTIONAL BORON EPOXY[65]

Figure 2-40 similarly demonstrates the influence of span-to-depth and beam width on the ILSS of unidirectional boron-epoxy composites. For the span-to-depth ratios used, the probability of flexural failure will also increase as the ILSS of boron-epoxy is enhanced without corresponding increases in flexural strength.

Figure 2-41 represents a plot of the minimal flexural-to-shear strength ratio (S_z/S_{xz}) required to ensure shear failure at a given span-to-depth ratio[74]. The graph, which is applicable to both composite systems treated, illustrates the valid-invalid span/depth ratios for short beam shear testing if the flexural-to-shear strength ratios are known approximately. The graph also includes a margin restricting the outer fiber flexural stress to 70 percent of the flexural strength and thus gives enhanced assurance that pure shear failure will occur. Employing this criterion, a span-to-depth ratio of 5 would not be used unless the flexural-to-shear strength ratio was, at least, 14.5. This curve can be most useful in determining future span-to-depth ratios as composites ILSS values increase without corresponding increases in flexural strengths, or where interlaminar shear values are desired for lay-ups where flexural strengths are reduced. In either case, a reduced span-to-depth ratio can be selected to yield a valid short beam shear test.

Test methods discussed and recommended in this section, exclusive of axial tension, transverse tension, and composite thermal expansion tests, are applicable to material screening and thus should be considered only for use as quality control test methods and procedures.

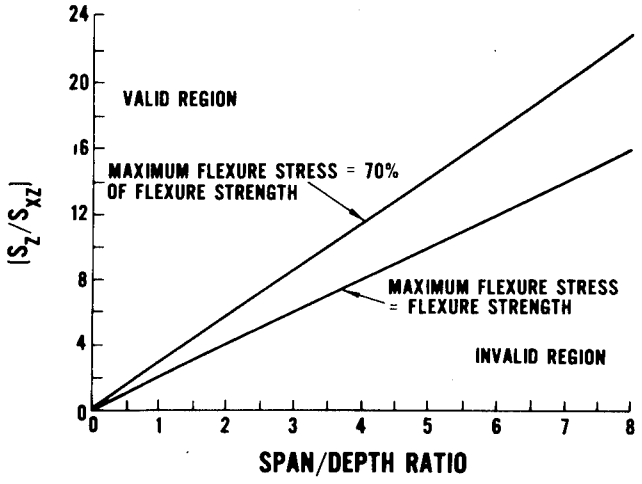


FIGURE 2-41. RATIO OF FLEXURAL STRENGTH-TO-SHEAR STRENGTH VERSUS SPAN-TO-DEPTH RATIO[74]

3.0 DESIGN TEST DATA

3.1 MECHANICAL PROPERTY TESTS

3.1.1 General

Failure characteristics and behavior of composite materials are considerably more difficult to quantitize for specific design purposes than are most homogeneous materials. Mechanical property data for composite materials generally show significant scatter, particularly when samples from different sources are compared. This phenomenon relates directly to the number of variables which influence the response of the material. Specific definition of these influences, both from a statistical and empirical point of view is quite complex. As an anisotropic material, different failure and response mechanisms in composites operate in each major material direction. Even when statistical distribution functions defining strength in each major material direction are the same, the function parameters normally differ. The problem of generating reliable design data is further compounded by the frequent required use of small test samples which are employed to simulate the full-scale end-item structural demands and performance.

Influence of the effects of processing (fabrication and specimen preparation) and testing variables on composite mechanical and physical properties should be carefully determined. Processing effects to be considered are: (1) the number, geometry, and distribution of voids, (2) variation of interfilament spacing and size and distribution of resin-rich areas, (3) variation of interfacial bonding of composite constituents. Testing effects previously discussed in Section 2.5, include the following:

1. Influence of test specimen geometry.
2. Dimensional effects (length, width, thickness).
3. Strain-rate effects.

The influence of voids has been shown to be particularly significant on shear, compressive, and transverse tensile strengths. Greszczuk[75] reports a 40 percent decrease in interlaminar shear strength with a void content of 4 volume percent. Foye[76] has cited a 30 percent reduction in composite compressive strength as a function of a void content of 5 volume percent. The effects of voids on the elastic properties, using a number of theoretical approaches, has been defined by Jones[77], as shown in Figure 3-1.

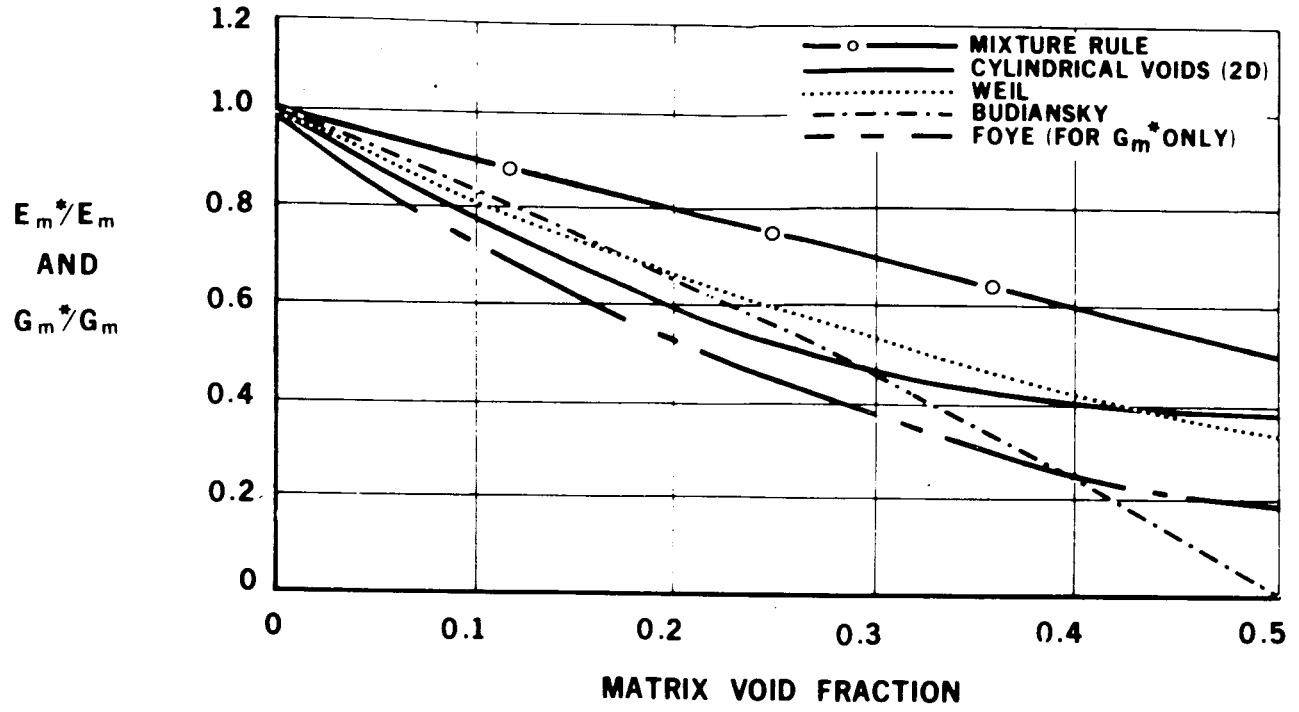


FIGURE 3-1. EFFECT OF VOIDS ON ELASTIC PROPERTIES OF MATRIX[77]

The effects of testing variables on the mechanical properties of composites have been discussed in the previous section. The test specimen is of prime importance in the establishment of reliable design data, particularly in terms of specimen dimensions and configuration for flat, balanced cross-ply, and angle-ply composites. Since composite allowables are a function of application, the test method selected represents the specific application and should provide the most realistic data for the given application. These requirements, however, are often difficult to achieve in advanced filamentary composites because of strong edge effects and a significantly wide range of Poisson's ratios.

3.1.2 Tension

3.1.2.1 Uniaxial

Uniaxial tensile specimen configurations and corresponding test methods considered include the straight ITTRI-type coupon, sandwich beam, and the filament wound tube. The ITTRI-type coupon is recommended as a standard test configuration for establishing design allowable tensile data for boron-epoxy and graphite-epoxy composite materials[78].

3.1.2.1.1 Straight-Sided Coupon

The straight-sided tensile coupon and tensile test method has been treated in detail under Section 2.5.2.1 of this report. In order to enhance both reliability and reproducibility of test data, the classic edge effects characteristic of the straight-sided coupon can be minimized by increasing specimen width in excess of the suggested 1/2-inch. Lehman[79] increases specimen width to 3/4-inch for longitudinal tensile (0°) tests, and 2 inches for transverse tensile (90°) and all patterned laminate tests.

3.1.2.1.2 Sandwich Beam

Uniaxial tensile tests are also conducted using the sandwich beam flexural configuration. The beam dimension, core, and opposing face sheet materials are designed so that the composite is a critical test material. Design considerations include the opposite face-sheet strength, core shear strength, weight and cell size, adhesive strength and curing temperature, beam span, test section length, and the allowable amount of deflection at midspan. The beam dimensions, core, and face sheets must be selected so that the composite face sheet is stressed to failure. Interpretation of the test data is based on assumed membrane stress in the face sheets. Table 3-1 summarizes sandwich beam dimensions and materials as used by AVCO for boron-epoxy composites[38].

TABLE 3-1 - AVCO AEROSTRUCTURES DIVISION SANDWICH BEAM DESIGN[38]

Composite Face Sheet Construction (degrees)	Opposite Face Sheet		Core		Adhesive	Beam Dimensions					Load Pad
	Material	Thickness t	Material	Thickness c		Width	Test Span s	Moment Arm m	Total Length L	Overhang h	Width w
		(in.)		(in.)		(in.)	(in.)	(in.)	(in.)	(in.)	(in.)
0 0/90	7075-T6 Aluminum	0.125	1/8-5052-0.006-NP (23.4)	1.5	FM1000	1	1.5	9.75	22	0.5	1
90	FRP/Epoxy	0.080	1/8-5052-0.001-NP (4.5)	1.5	FM1000	1	1.5	9.75	22	0.5	1
±a 0/±a	Titanium* 7075-T6 Aluminum	0.040 0.060	1/8-5052-0.002-NP (8.1)	1.0	FM1000	1	2.0	6.00	16	1.0	1

*6Al-4-V

Sandwich beam tests tend to have less stress concentrations than coupons. However, in thicker laminates there is evidence of shear lag, which overloads the inner ply and thus reduces the failing stress. With this specimen type, a relatively heavy core is required to limit the specimen to practical lengths. The core stiffening effect may be objectionable, particularly with thin laminates or laminates with low transverse stiffness. The sandwich beam tests generally yield higher tensile strengths for unidirectionally oriented laminates and large-angle-plied laminates than comparable coupon-type tests. The test also tends to modify the measured stress-strain response of boron-epoxy laminates as compared to typical coupon-type tests. This phenomenon is strongly demonstrated in the case of transverse strain measurement, as shown in Figures 3-2 and 3-3.

3.1.2.1.3 Tubular

The tubular specimen type yields the most reliable data on the mechanical properties of filament-wound structures[80]. One of the major advantages of tubular or cylindrical specimens, from a design standpoint, is the relatively large size required which gives an ideal representation of the response of an actual finalized structure. Other advantages associated with this specimen type are as follows:

1. For filament-wound tubular applications, this test provides the most efficient method for testing angle-ply materials. Generally, flat specimen tests of such materials are subject to significant edge effects.

2. For unidirectional laminates, the edge effect, characteristic of other type specimens, is eliminated by virtue of filament continuity from one end of the tubular specimen to the other.

Although this test specimen type exhibits several apparent advantages over other configurations, it is considerably more expensive than the others treated in terms of quantity of required material, specimen fabrication, and labor. Tubular specimens are illustrated in Figure 3-4[81,82]. The two specimens shown represent two alternative designs for axial tensile testing and differ essentially in the basic scheme for reinforcing the loaded extremes of the tube. The design illustrated in Figure 3-4B yields improved results by virtue of elimination of the inherent bending stresses which occur in the design in Figure 3-4A. Such stresses are directly related to a lack of symmetry of the loaded region with respect to the middle surface of the specimen. Further, the graded increase in wall thickness of the latter specimen design as provided near the loading doubler eliminates stress concentration.

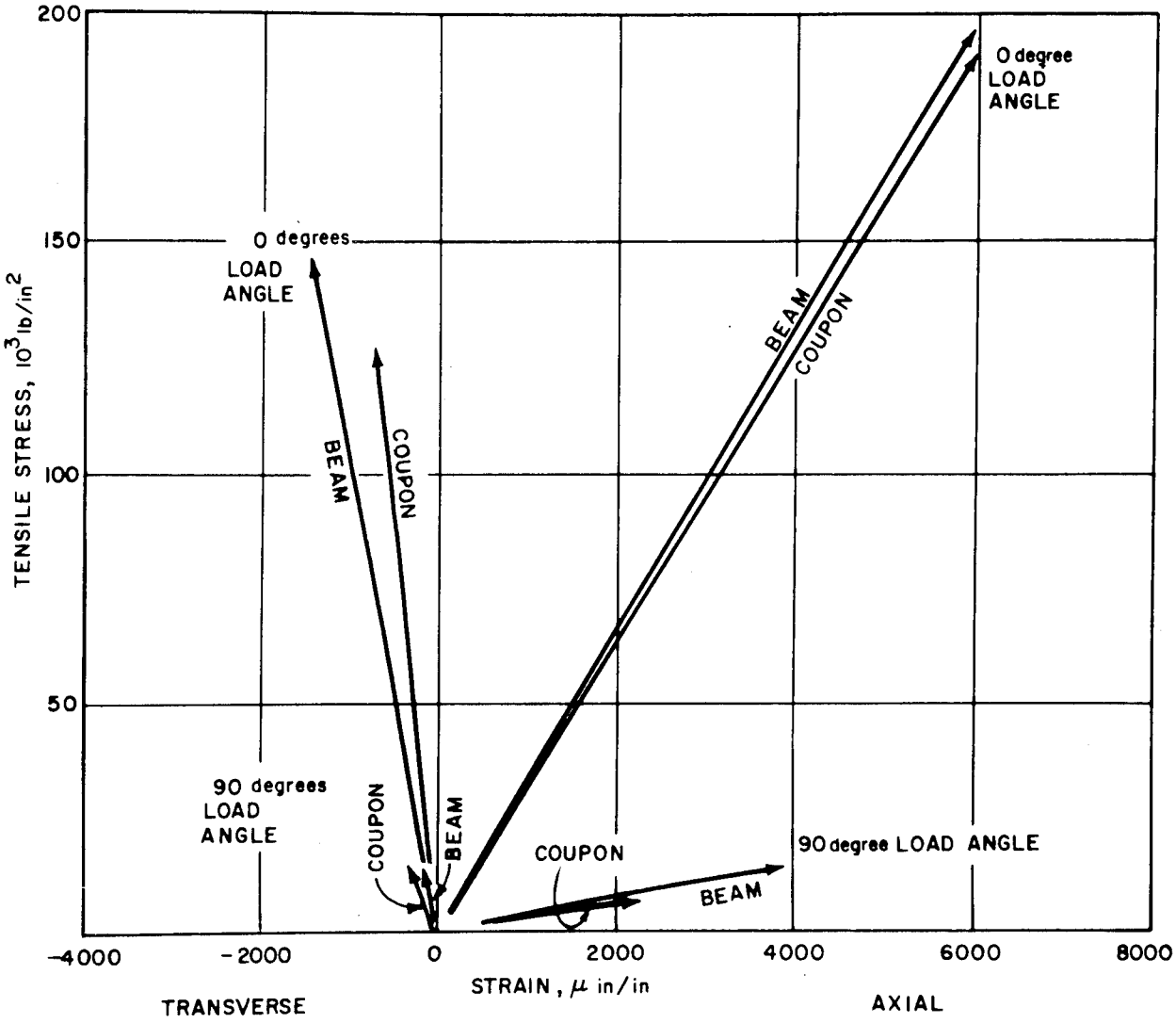


FIGURE 3-2. TYPICAL STRESS-STRAIN RESPONSE 0-DEGREE LAMINATES BORON/SP272 6-PLY COMPARISONS OF BEAM AND COUPON TESTS

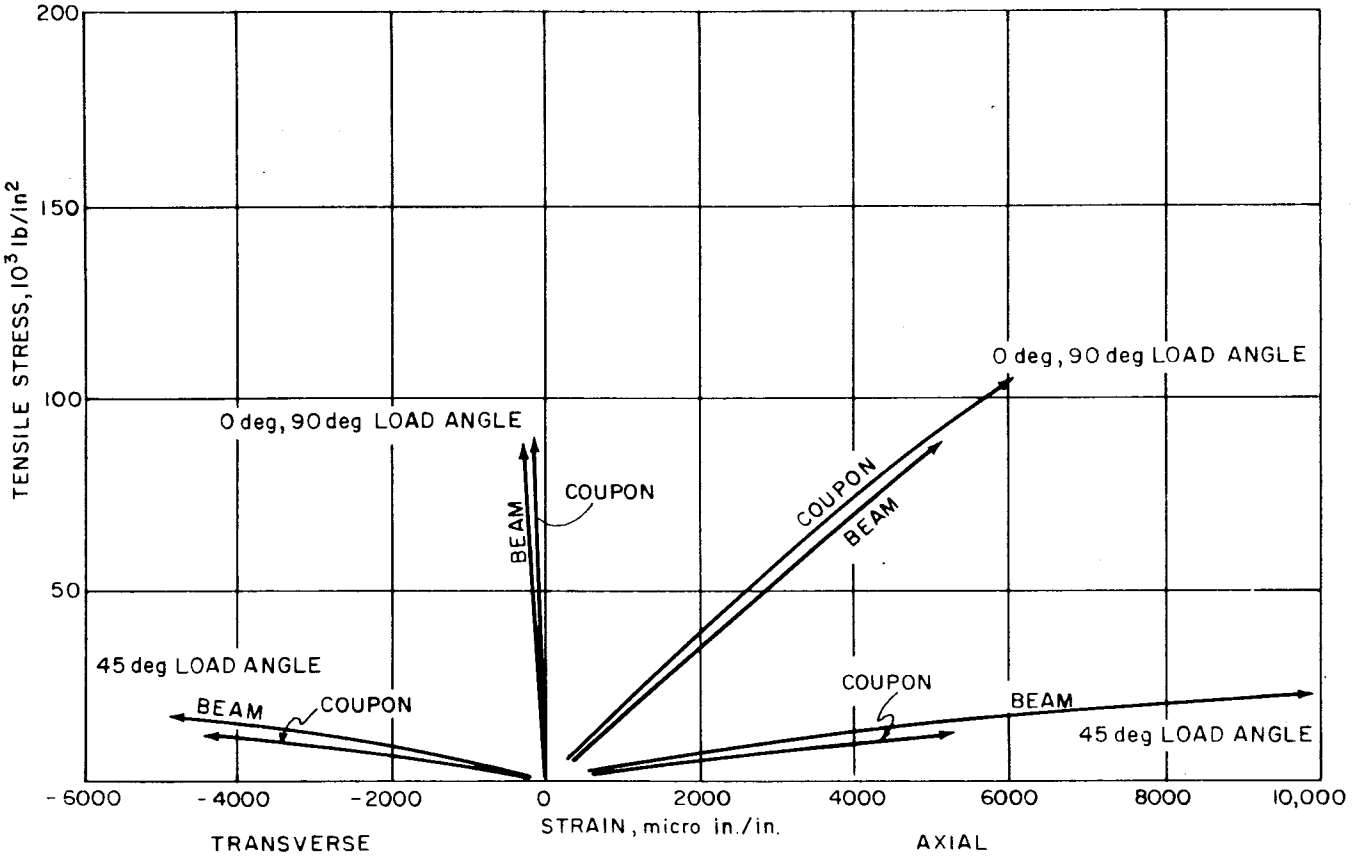
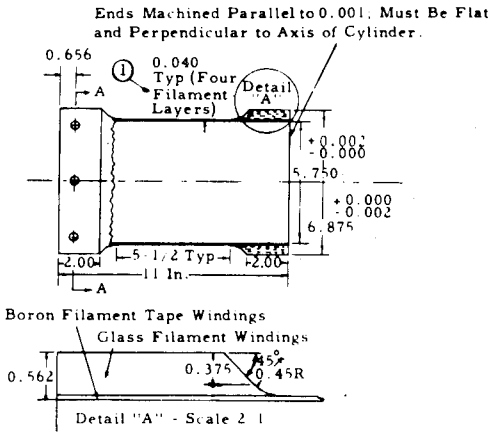


FIGURE 3-3. TYPICAL TENSION STRESS-STRAIN RESPONSE 0/90-DEGREE LAMINATES BORON/SP272 8-PLY COMPARISONS OF BEAM AND COUPON TESTS



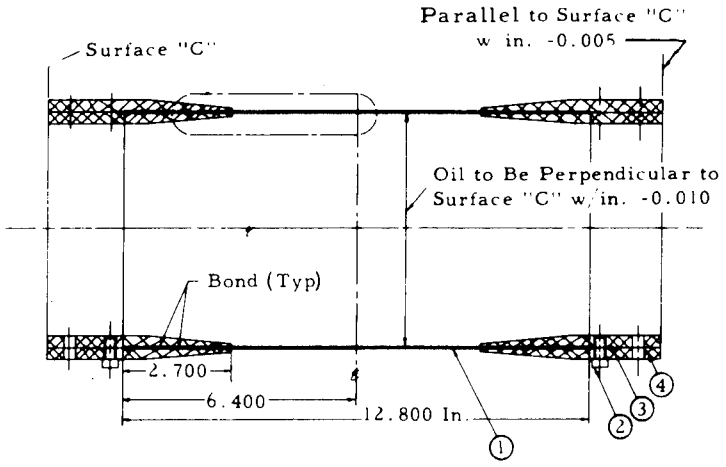


FIGURE 3-4. TUBULAR TYPE AXIAL TENSION SPECIMENS[81,82]

3.1.2.1.4 Recommendations

The ITTRI type coupon is recommended as the standard test configuration for development of design allowable tensile data for boron-epoxy and graphite-epoxy composite materials. In those instances, however, where the scatter of test data generated is excessive and deemed unacceptable, the sandwich beam test method is recommended.

3.1.2.2 Biaxial

One of the major requirements in development of composites into the range of primary structures is reliable biaxial data. The generation of a uniform, biaxially stressed composite test specimen, however, presents even greater difficulty than is encountered in metal testing because of the anisotropic properties of filamentary composites and the large variation of Poisson's ratio which may exist in the principal reference directions. Currently, the most commonly used technique to simulate a controlled biaxially stressed region is the application of loads to the arms of a cruciform test specimen. The fundamental approach uses the beam-type specimen[83, 84]. A typical sandwich crossbeam specimen, with minimum instrumentation high-elongation rosette strain gages, is shown in Figure 3-5 for biaxial tension-tension[64, 79]. In the recommended General Dynamics specimen shown, specimen facings are single-piece crosses of required thickness with area dimensions of 2 by 22 inches. Reversing the direction of loading will produce biaxial tension-compression loading. Figure 3-6 illustrates a hydraulic cushioned biaxial crossbeam stress fixture.

An additional method of biaxial testing commonly used with metals is the application of internal pressure and axial loading to thin-wall cylindrical specimens. The test configuration, as applied to hollow, thin-walled anisotropic composite cylinders, is shown in Figure 3-7. The specimen is a helical wound composite tube[85]. Processing of the material differs from that employed for flat laminates and the curved filaments tend naturally to induce a residual bending stress. In view of the fabrication complexities and other problems associated with load introduction into the specimen, the current practical utility of this specimen type for obtaining flat laminate properties has yet to be defined.

3.1.2.3 Transverse

Transverse tension specimen configurations and test procedures have been effectively treated in the previous sections under uniaxial tension as applied to both material acceptance and design data.

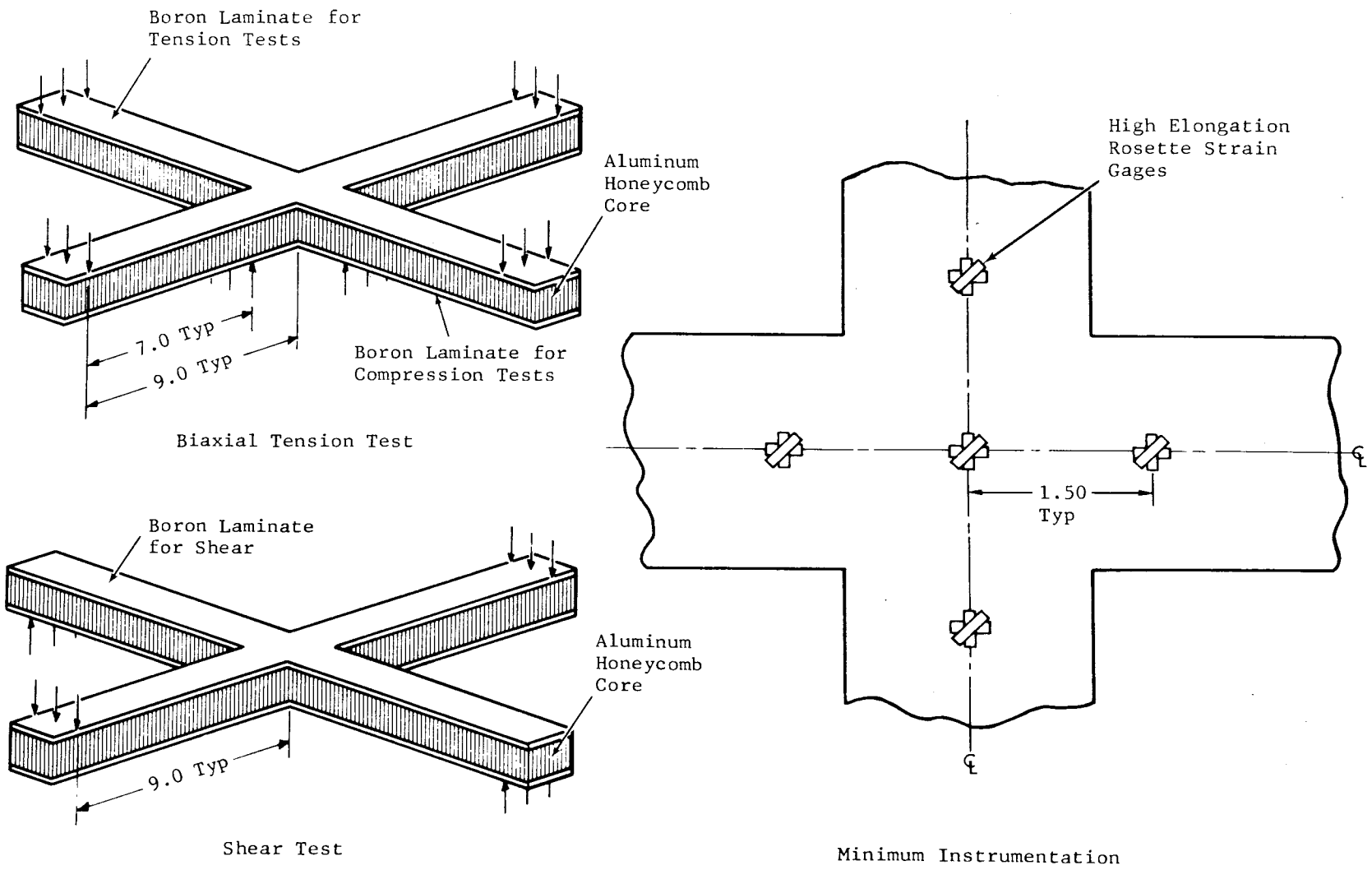


FIGURE 3-5. SANDWICH CROSS-BEAM TEST CONFIGURATIONS

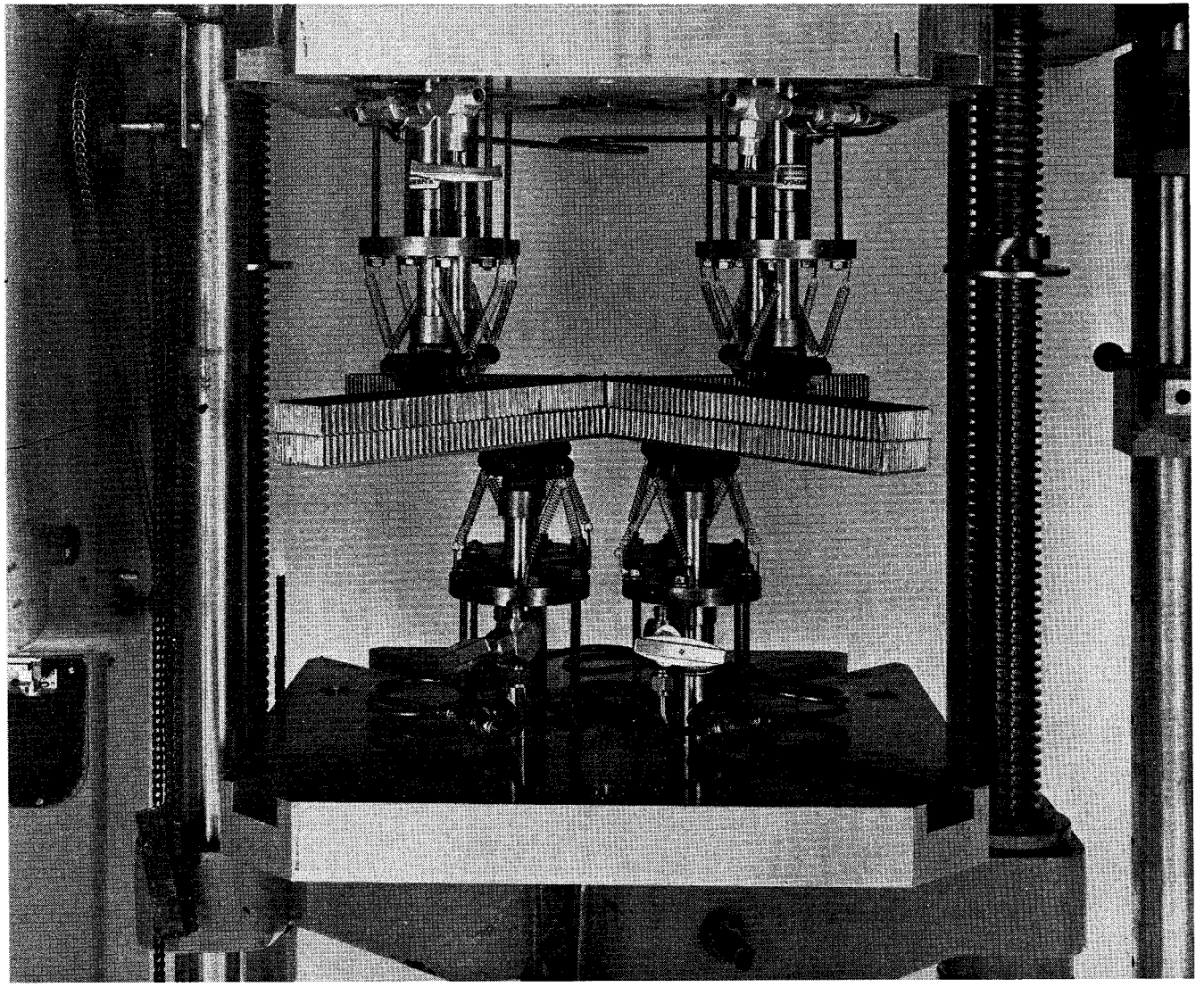


FIGURE 3-6. HYDRAULIC CUSHIONED BIAXIAL CROSSBEAM STRESS FIXTURE[64]

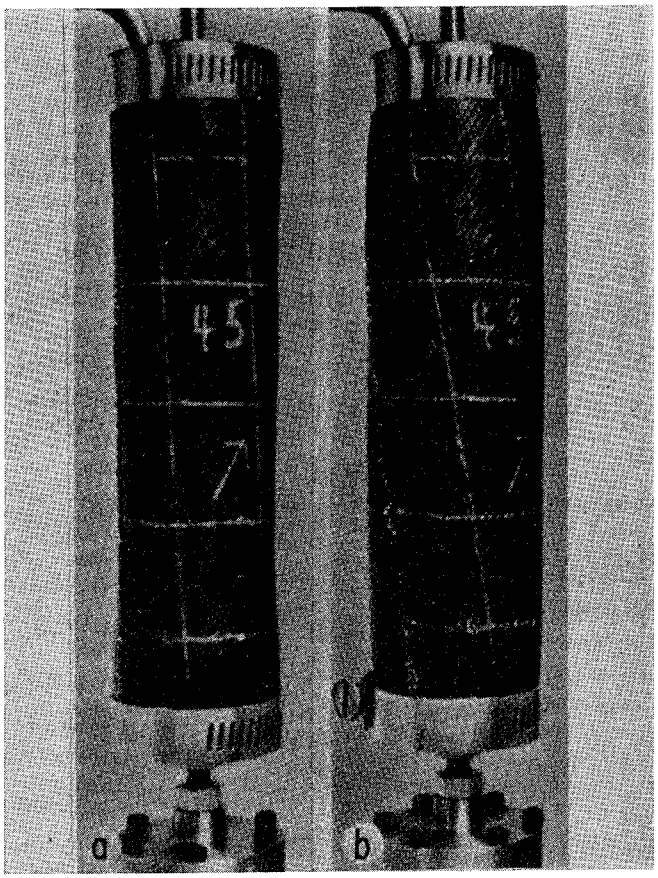


FIGURE 3-7. HELICAL WOUND THIN-WALLED TUBE UNDER (A) UNIFORM TENSION AND (B) INTERNAL PRESSURE[85]

3.1.3 Compression

3.1.3.1 Uniaxial

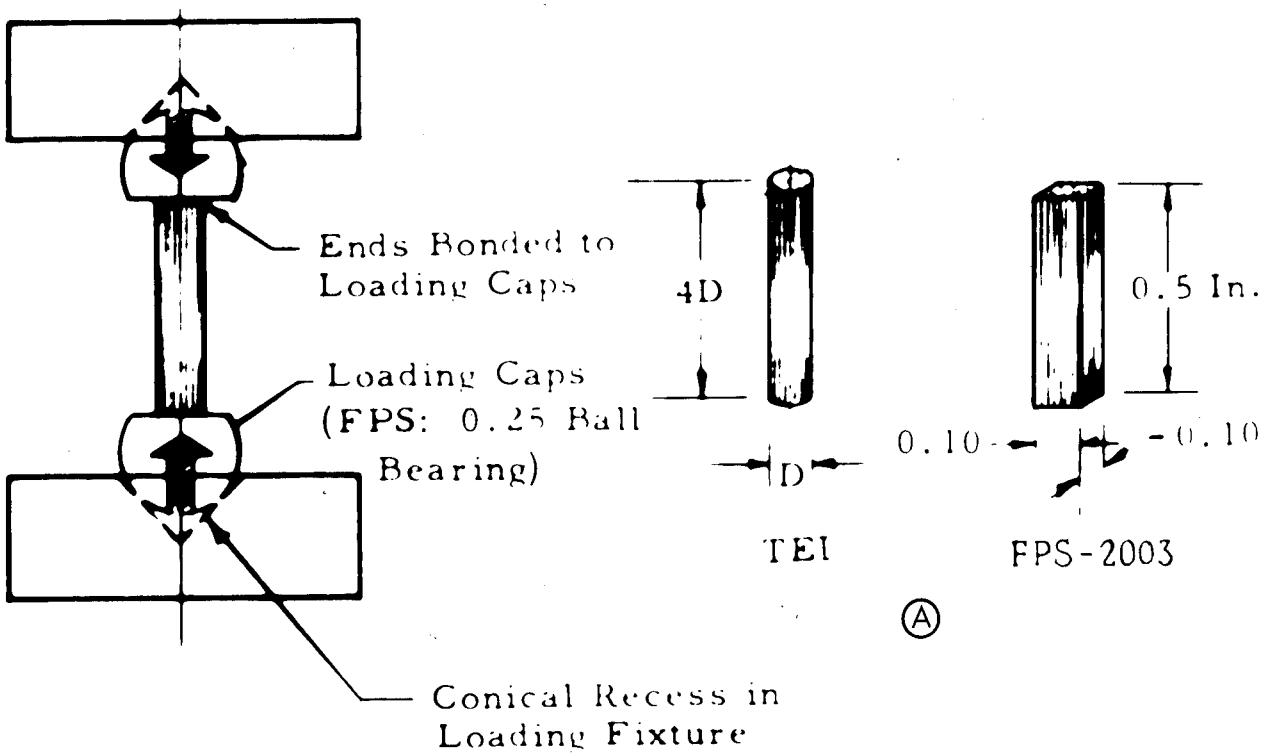
Uniaxial compression test methods, employing both flat coupon and tubular specimen types, have been treated in Section 2.5 of this report. Both specimen types and test procedures discussed were directed toward material acceptance and quality control test methods for composite materials.

Short-column type specimens, as shown in Figure 3-8, can be considered for determinations of compressive allowables for both boron-epoxy and graphite-epoxy systems. The TEI unidirectional composite rod[64], illustrated in Figure 3-8A, provides realistic upper-limit compressive strengths for fully stabilized sections. The small partially stabilized plate specimen, as shown in Figure 3-8B, is reported to yield relatively conservative results; however, it is not satisfactory for laminates less than six plies[86]. The short column compressive test developed by the Celanese Corporation for determining compressive strengths of high modulus graphite fibers yield reproducible results exhibiting low coefficients of variation for 0.125-inch-thick specimens[87].

Specimen configurations and test methods applicable to determination of compressive design data for honeycomb-stabilized structures include those given in the following recommended specifications; namely, ASTM C364-61, MIL-STD-410A, General Dynamics (Fort Worth Division) Sandwich Beam, Grumman Sandwich Beam, and FPS-2003 (B-007)[88,89,90,91,71]. Specimen configurations are shown in Figure 3-9. Specimen preparation procedures and material specifications are detailed in the references cited. Stress calculations for beam specimen configurations assume ineffective core and bending couple at midplane of faces.

The General Dynamics Sandwich beam test specimen, which is most commonly used, and is recommended here, is shown in Figure 3-10, which includes recommended instrumentation[64]. The basic test procedure, including both compression and tension and the computation of these properties is treated below. The procedures and calculations are valid for both boron-epoxy and graphite-epoxy composite systems.

Compression testing is conducted using four-point loading of a sandwich beam composed of two face sheets separated by a deep honeycomb core. One face sheet is the composite test specimen with the fiber orientation to be tested. The other is a face sheet of a material and size carefully selected to preclude its influence on the test results.



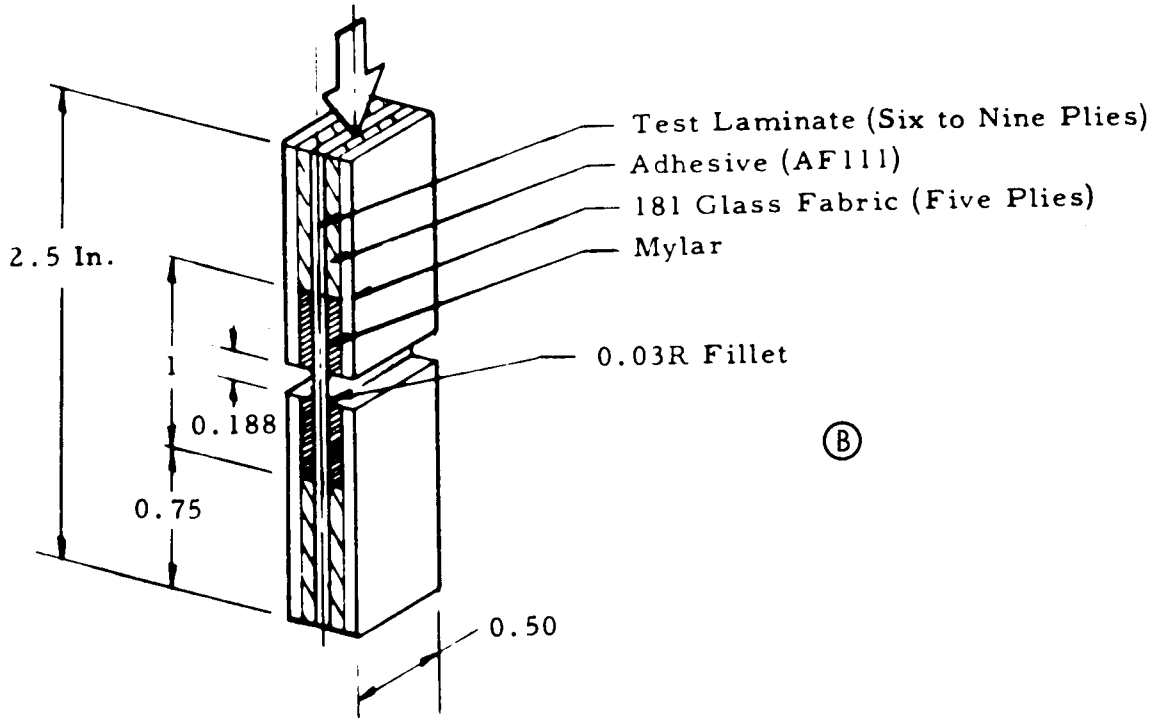


FIGURE 3-8. COMPRESSION TEST SPECIMEN TYPES[71,86]
A - COMPOSITE ROD
B - PARTIALLY STABILIZED PLATE

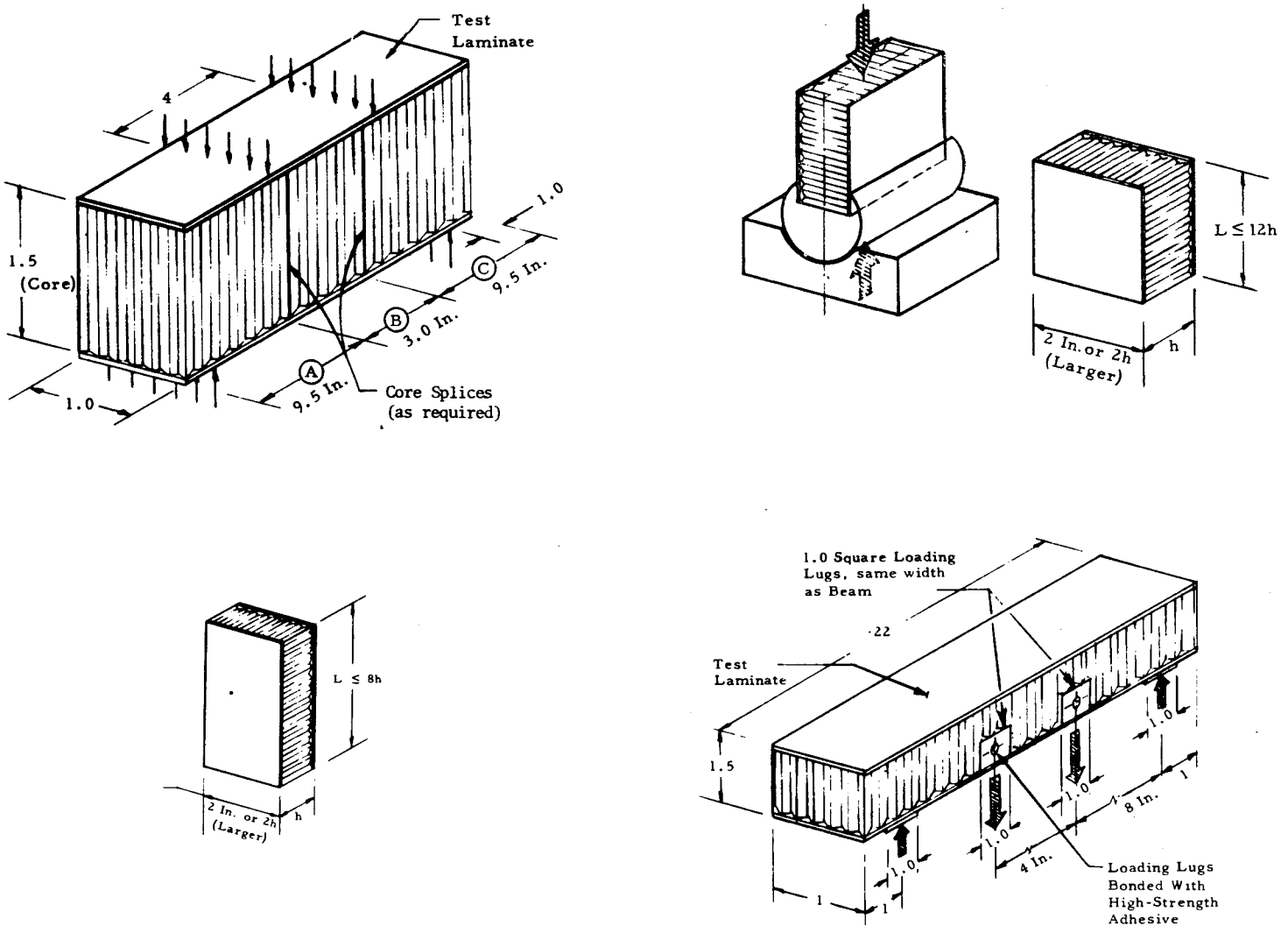
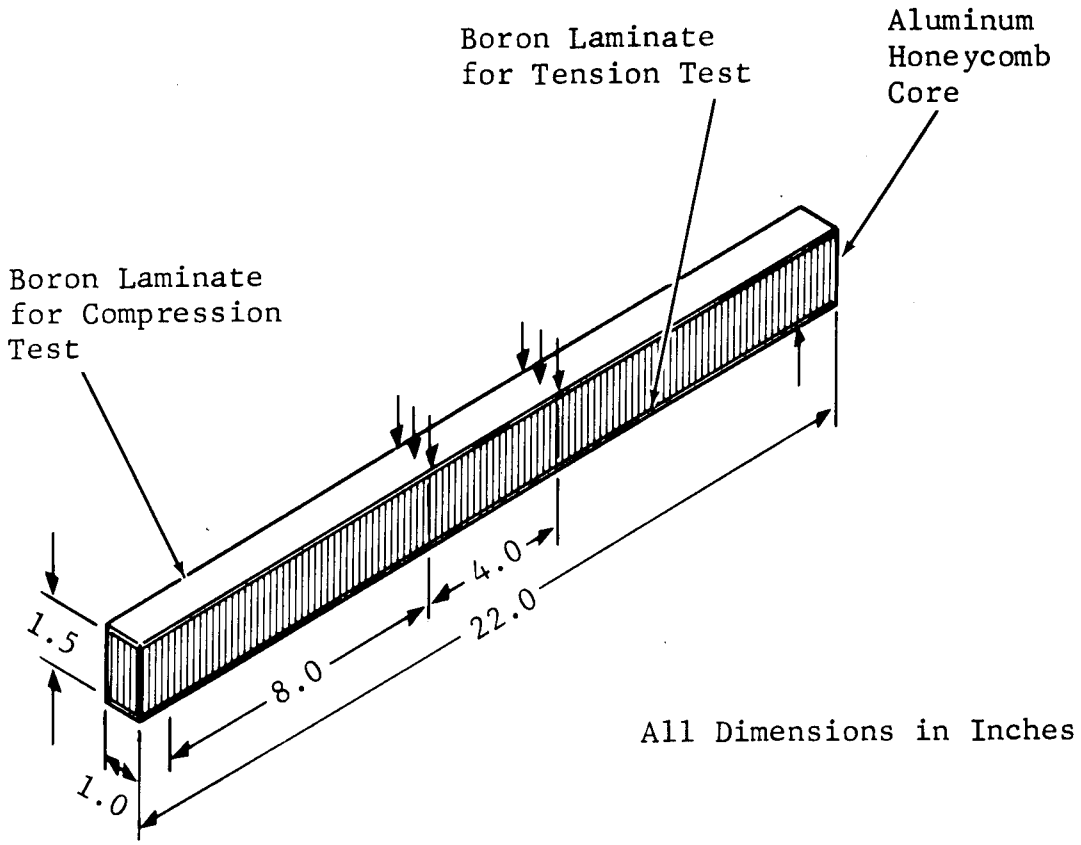
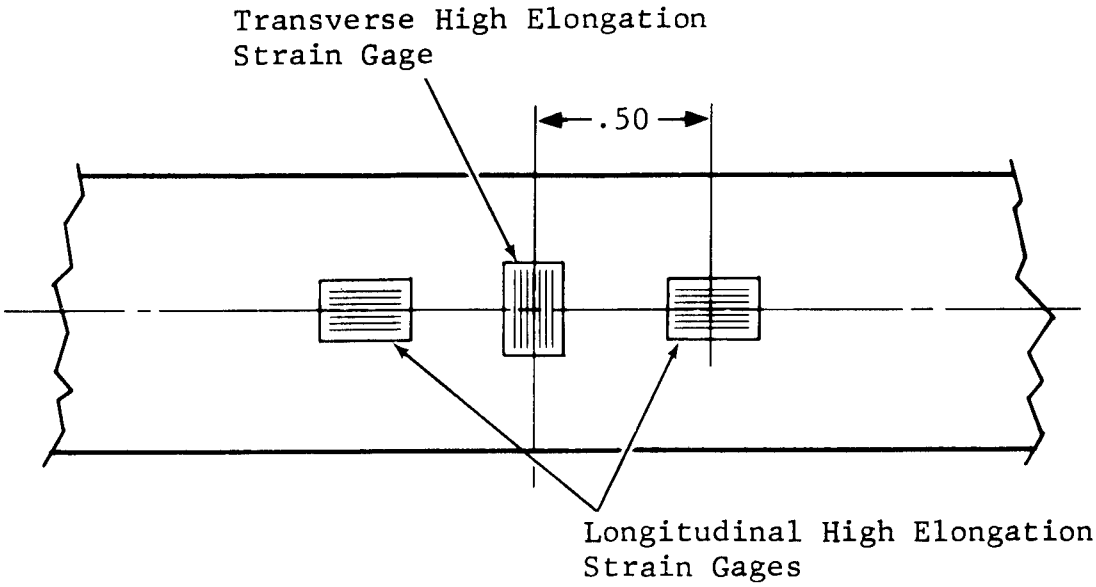


FIGURE 3-9. VARIOUS CONFIGURATIONS OF HONEYCOMB-STABILIZED COMPRESSION SPECIMENS[88,89,90,91]



Test Specimen and Loading



Instrumentation

FIGURE 3-10. UNIAXIAL SANDWICH BEAM TEST CONFIGURATION[64]

The beam dimensions, core, and opposite face-sheet materials are designed so that the composite is the critical test material. Design considerations include the opposite face-sheet strength, core shear strength, weight and cell size, adhesive strength and curing temperature, beam span, test section length, and amount of deflection allowed at midspan.

In the first approach to beam design, it might appear that modulus times thickness of the composite face and the opposite face must be the same. However, for thick beams, different values may be employed with negligible effect on the neutral axis location of the beams. This allows some flexibility in the choice of opposite face-sheet materials and thicknesses. Figure 3-10 illustrates the beam configurations for compression testing of boron, high-modulus graphite, and high-tensile-strength graphite.

Ultimate compressive strength is calculated from the following equation:

$$F_{cu} = \frac{M\,P}{2t_1\left[t_c + \frac{(t_1 + t_2)}{2}\right]}$$

where:
- F_{cu} = ultimate compressive strength
- M = moment arm
- P = applied load
- t_1 = composite thickness
- t_c = core thickness
- t_2 = opposite face thickness

For completeness, the beam design procedure is described below:

1. Obtain core shear strength for the weight of core being considered.
2. Calculate the maximum load the beam may be subjected to without core shear failure, using the following relation and making reasonable assumptions of thickness dimensions.

$$r_c = \frac{P}{t_1 + t_2 + 2t_c}$$

where: τ_c = allowable core shear stress

and $E_1 t_1 \cong E_2 t_2$

where: E_1 = modulus of composite in compression

E_2 = modulus of opposite face sheet in tension

3. Calculate the moment arm that is required to produce the estimated failure strength of the composite being tested from the relation:

$$M = \frac{F_{cu}\, 2t_1 \left[t_c + \frac{t_1 + t_2}{2}\right]}{P}$$

where: F_{cu} = estimated ultimate compressive strength

4. Overall beam length is calculated as follows:

L = desired gage length + 2 (moment arm) + overhang

If L exceeds available testing facility, t_c should be increased, t_1 decreased, or a heavier core selected.

5. Check the stresses in both faces by using the following general equations for bending of beams with face-sheet materials of equal width but dissimilar materials:

$$F_{cu_1} = \frac{M(P)\left[t_c + t_1 + t_{2E} - \left(\frac{t_1 t_c + \frac{t_1^2}{2} + t_1 t_{2E} + \frac{t_{2E}^2}{2}}{t_1 + t_{2E}}\right)\right]}{I_{NA}}$$

where: F_{cu_1} = ultimate compressive stress in composite face

I_{NA} = moment of inertia about neutral axis

$$I_{NA} = t_1 (t_c + t_1/2 + t_{2E})^2 - (t_1 + t_{2E})\left[\frac{t_1 t_c + \frac{t_1^2}{2} + t_1 t_{2E} + \frac{t_{2E}^2}{2}}{t_1 + t_{2E}}\right]^2 +$$

$$\frac{t_{2E}^3}{3} + \frac{t_1^3}{12}$$

where: $t_{2E} = \frac{E_2}{E_1} t_2$

and $$F_{tu_2} = \frac{\frac{E_2}{E_1} (M) (P) \left[\frac{t_1 t_c + \frac{t_1^2}{2} + t_1 t_{2E} + \frac{t_{2E}^2}{2}}{t_1 + t_{2E}} \right]}{I_{NA}}$$

where: F_{tu_2} = ultimate tensile stress in opposite face sheet

Other symbols previously defined.

If F_{cu_1} does not agree with the estimated value, the neutral axis has been shifted significantly by improper selection of opposite face sheet material. Pick another E_2 such that $E_2 t_2$ is more nearly equal to $E_1 t_1$.

If failure of the opposite face sheet in tension is indicated, changes in material or thickness must be made until failure in the composite face is indicated.

Adhesive employed to bond face sheets to the core is based upon lap tensile data at the required temperature.

3.1.3.2 Biaxial Compression

(Refer to Section 3.1.2.2, which treats biaxial tension)

3.1.4 In-Plane Shear

Test methods for determining interlaminar shear properties of composites have been treated in the Materials Acceptance Section. Test methods discussed included short beam, torsion, and notched shear. Shear properties in the plane of the composite laminate, however, are the most difficult and least standardized of the major mechanical properties of composite materials. The basic problem is rooted in the inherent difficulty of the application of pure and uniform shear stresses while excluding secondary and normal stresses. The most direct means of achieving pure shear in a filamentary composite is by application of torque load on a thin-walled cylinder. The required preparation of special test specimens, however, does not readily lend itself to testing of flat laminates or composite parts.

The problem is further compounded by induced filament bending stresses generated from curvature in all plies with the exception of those which are longitudinally oriented[92].

The "picture-frame" test, adapted from metal testing, employs a four-bar linkage fixture to translate shear to the periphery of a square specimen by application of a tensile force to one pair of corners along a diagonal of the frame[93,94]. Figure 3-11 illustrates the picture-frame fixture with specimen[95]. Dastin reported that the in-plane shear strength of bidirectional reinforced laminates can be accurately determined utilizing the "picture frame" test[67]. The ultimate stresses are repeatable and consistent with acceptable theory; however, the strains beyond the initial load level were considered questionable. As a result, the test was recommended for material characterization rather than design[67].

The rail test also employed for metals has been adapted for in-plane shear determinations in composite materials. Boller[96] developed a modified picture frame test procedure called the "rail shear" test. The test requires a laminate, 1/16 to 1/8 inch thick, 3 inches wide by 6 inches long, supported along the 6-inch length by two pairs of rails, having a 1/2 inch wide unsupported section. The length of the test section is approximately 5 inches, as a result of shortening the 6-inch side with a 1/2-inch radius. Shear distortion is measured by a detrusion gage located midway between the rails and midway along the 5-inch dimension. The detrusion gage accurately measures the angular distortion for a total of 0.10 radian to ± 0.000025 radian[96]. The rail shear apparatus and test specimen are illustrated in Figures 3-12 and 3-13.

Lehman[79] modified the standard configuration of the rail shear specimen by the addition of heavy steel doublers bonded to the grip areas to promote uniform load distribution across the gage section as shown in Figure 3-14. The results achieved using the modified specimen exhibited minimal scatter in ultimate strength.

Several other shear test methods suitable solely for shear modulus determinations are based on plate, coupon, and beam specimens. The most widely used of these is the plate-twist test which has generally proven reliable for orthotropic laminates when deflections are small.

Plate specimens are required to be perfectly flat. Deformation or warpage of the specimen as a function of curing can significantly influence plate deflection. Modulus

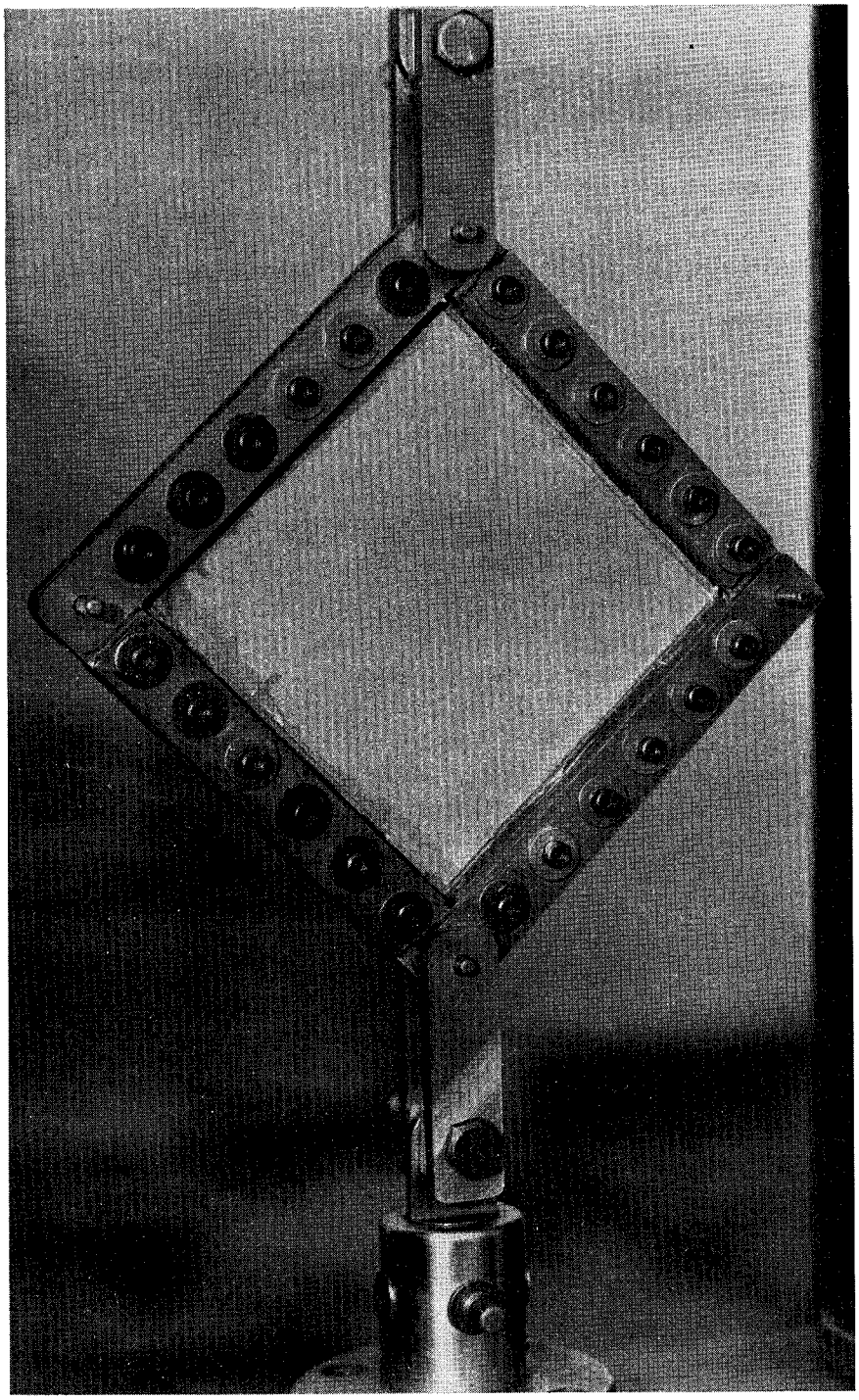


FIGURE 3-11. PICTURE FRAME FIXTURE WITH TEST ASSEMBLY

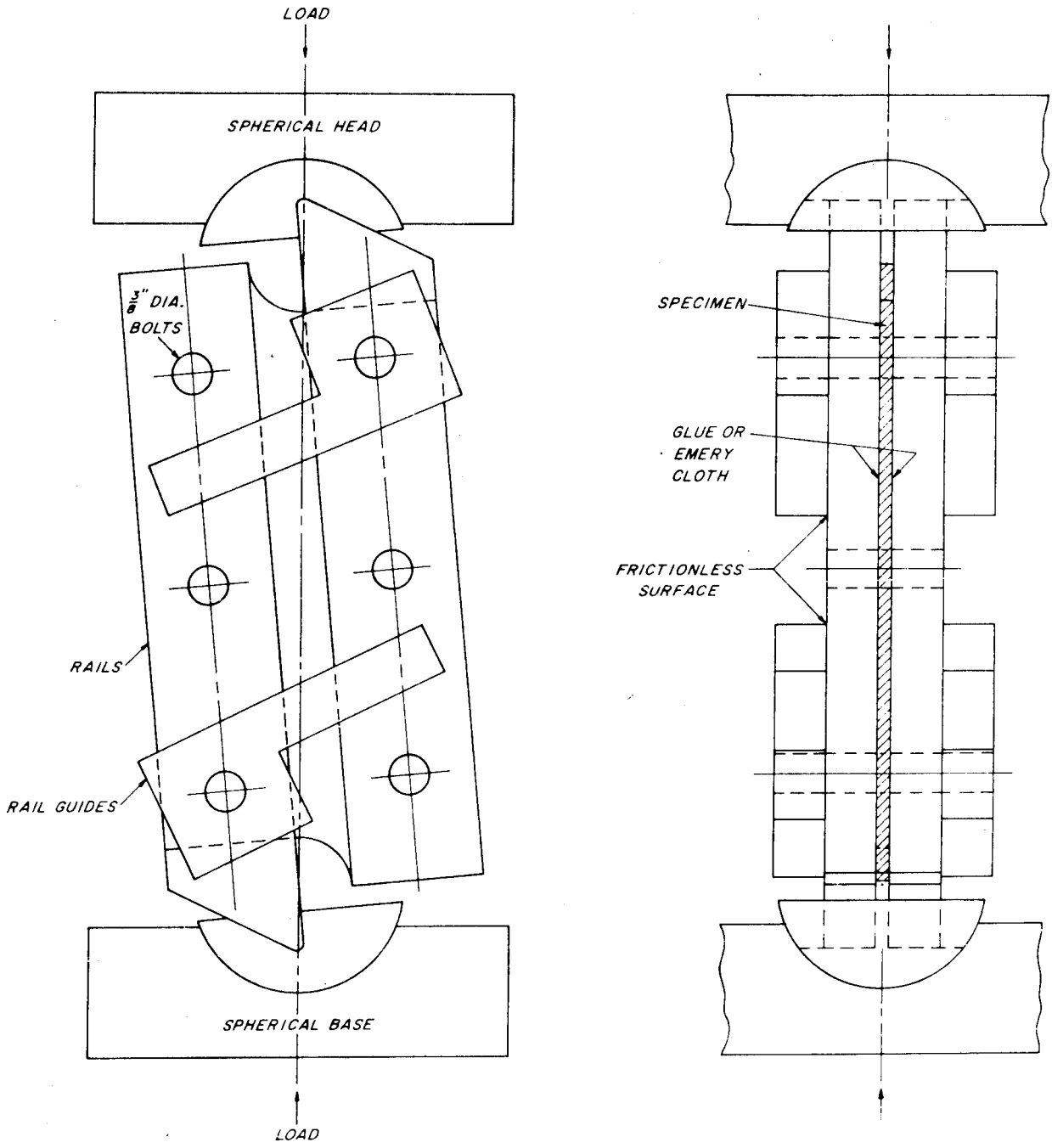


FIGURE 3-12. RAIL SHEAR TEST APPARATUS[96]

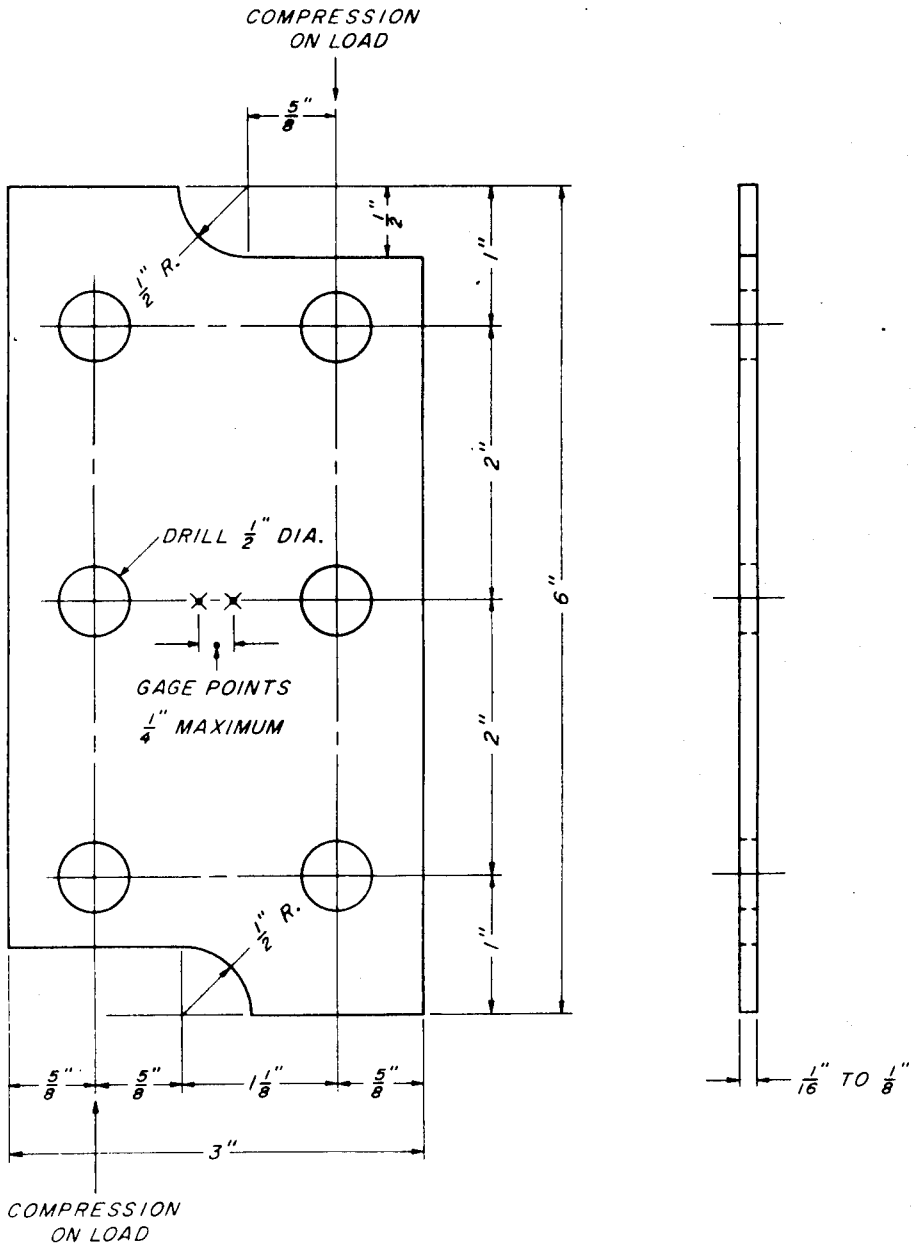


FIGURE 3-13. RAIL SHEAR SPECIMEN[96]

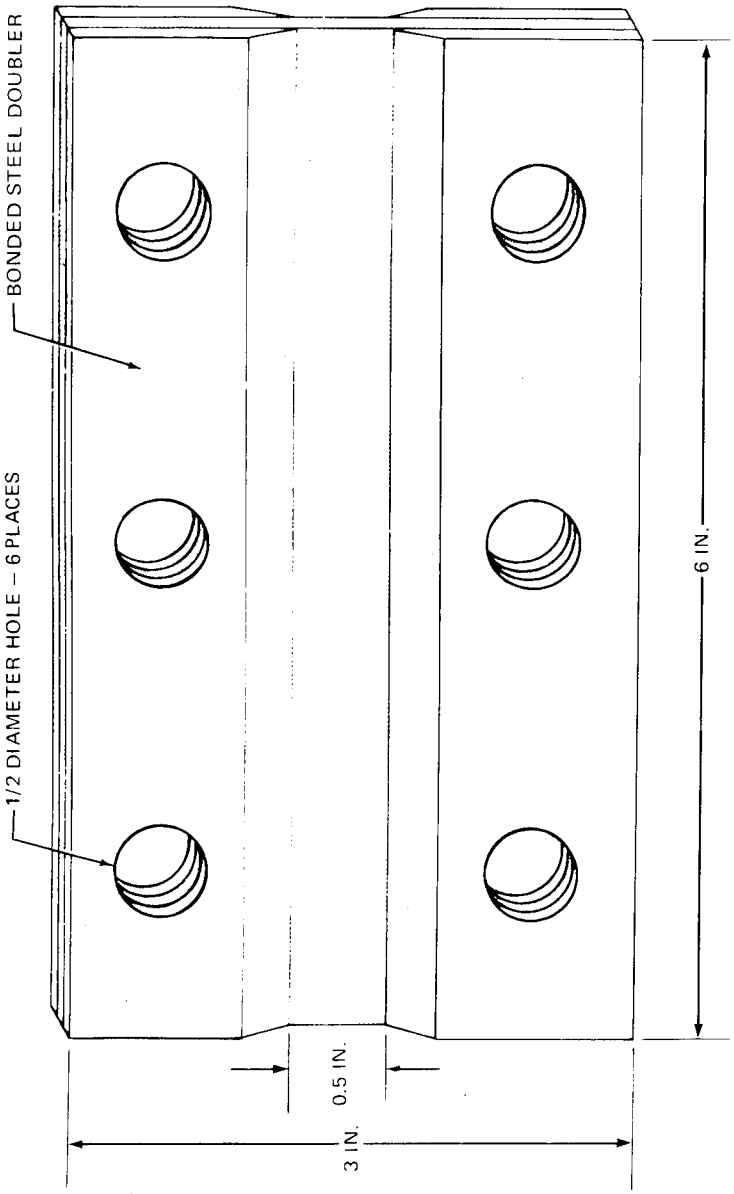


FIGURE 3-14. MODIFIED RAIL SHEAR SPECIMEN WITH BONDED DOUBLERS[79]

values obtained from this test method are generally higher than those derived from other test methods. Detailed treatment of basic theories involved in determination of modulus using the plate-twist technique are defined by Witt[97] and Beckett[98].

Grezczuk[99,100] developed a test method for determining shear modulus and other major elastic constants of filamentary composites which utilizes strain gage rosette-instrumented tensile coupons which contain oriented filaments as illustrated in Figure 3-15. The shear modulus is determined by the following equation[99].

$$G_{LT} = \frac{1}{2(m_A - m_C) + (2m_B - m_A - m_C)(\cot\alpha - \tan\alpha)}$$

where the m's are the strain-stress ratios

$$m_A = \frac{\epsilon A}{\sigma 1} \qquad m_B = \frac{\epsilon B}{\sigma 1} \qquad m_C = \frac{\epsilon C}{\sigma 1}$$

where: α = fiber orientation angle

ϵ = measured strain

σ = applied tensile or compressive stress

Principal shear modulus is obtained by conducting a tension test of the instrumented coupon. For the case where the fiber angle (α) is 45°, the original equation cited above simplifies to:

$$G_{LT} = \frac{1}{2(m_A - m_C)}$$

Several ring shear test methods are used to determine in-plane shear modulus. Two such methods to be considered are the General Electric ring[82] and the Douglas split ring tests[100].

The ring shear test simply involves use of a test fixture in which one half of the ring is restrained, and the unrestrained half is deflected as a cantilever beam. The test may be employed nondestructively to determine modulus values prior to testing to failure to determine hoop tensile or compressive properties of the composites.

The split-ring test involves the application of two loads of equivalent magnitude but in opposing directions at the point where the ring is split. The out-of-plane deflection is

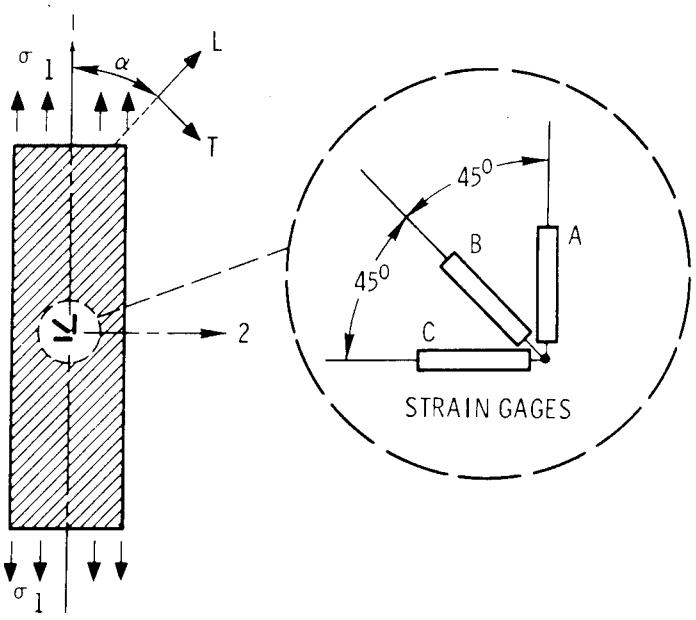


FIGURE 3-15. SHEAR MODULUS SHEAR SPECIMEN TENSILE COUPON WITH ORIENTED FIBERS[100]

predominantly due to shear deformation and thus makes the test ideal for shear-modulus determinations. Equations which relate the ring deformation to the physical and mechanical properties are treated in detail in the reference cited. The method is relatively simple and yields accurate data.

Finally, shear loading can be introduced into a cross beam specimen as illustrated in Figure 3-16. Opposed bending provides orthogonally oriented tensile and compressive stresses of equal magnitude, thus resulting in shear on a 45° axis in the test area or section. Because of the corner notch effects and variable effective width of the test section, the loading lacks true uniformity and the results can be significantly affected.

Currently, none of the in-plane shear test methods treated can be considered a superior method; however, the modified rail-shear specimen and the method utilized to survey shear-strain measurement within the test area, as developed by Lehman[79] appears to generate the most reliable design data. This procedure can be considered for recommendation as the design test method for determination of in-plane shear in advanced composite materials.

3.1.5 Impact

Impact testing of advanced filamentary composites should be conducted according to standard metal testing procedures; namely, the Izod and Charpy tests[101].

For impact properties of resin matrix composites, the cantilever beam or Izod type test is recommended. The test specimen, which requires a notched specimen at all times, is held as a cantilever beam and is fractured by a blow delivered at a fixed distance from the edge of the specimen clamp. The notch in the specimen functions to concentrate the stress, minimize plastic deformation, and direct the fracture to the area of the specimen immediately behind the notch. This tends to reduce data scatter. In view of the elastic and viscoelastic properties of resins, however, the response to a given notch may vary. Composite tests should be conducted at various critical loading angles with respect to the principal fiber orientation. Observation and close examination of the fracture surface and crack characteristics is important in this test.

A typical notched specimen with dimensions as specified by ASTM D256-56 is shown in Figure 3-17. The recommended notch depth is 0.10 inch. In the area of advanced filamentary composites, impact data is currently lacking. Until considerably more data

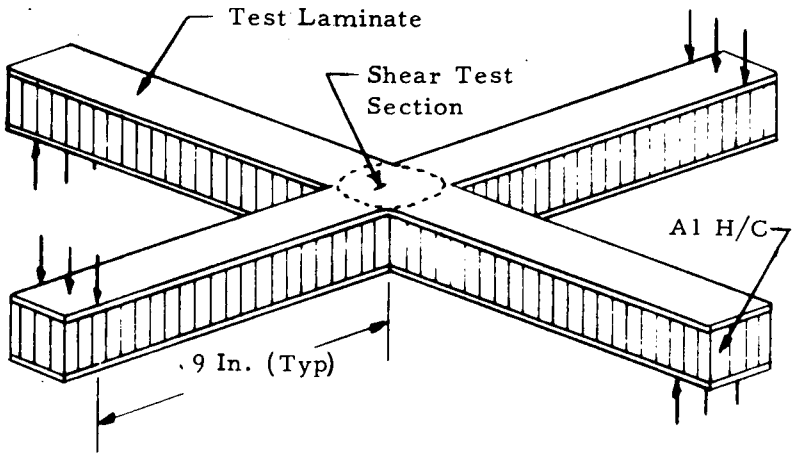


FIGURE 3-16. SHEAR LOADING OF CROSSBEAM SPECIMEN[83,84]

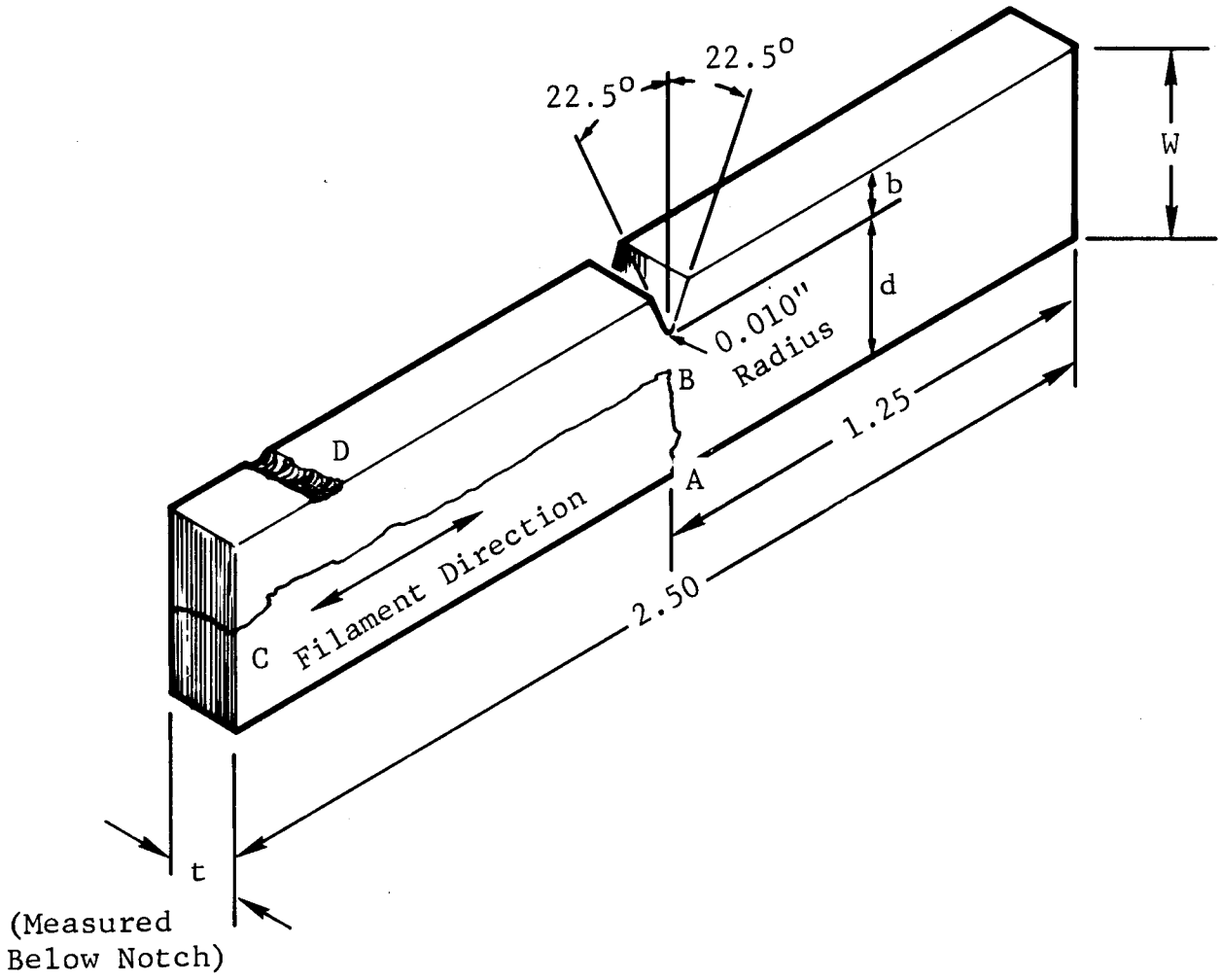


FIGURE 3-17. IZOD IMPACT SPECIMEN CONFIGURATION[64]

becomes available on several filament-resin combinations, it is difficult to determine if impact testing of composites actually yields any significant or meaningful information.

3.1.6 Bearing Tests

Bearing tests of filamentary reinforced composites designed to determine basic material properties, as opposed to specific joint strengths, are very similar to bearing test methods employed for metals, with the exception, however, of edge distance and bearing strength as defined by Federal Test Methods Standard and ASTM Specifications[44,102]. In this case, bearing strength is related to the slope of the tangent of the load deflection curve. The maximum bearing stress is the maximum load sustained by the bearing area. Test specimens are illustrated in Figure 3-18.

As illustrated, the method uses a double shear joint employing a hardened steel pin with the test specimen interposed between the steel loading plates. It is recommended that the hole should be drilled undersize and reamed to the specific diameter required to accommodate the bearing pin. The ARTC[70] type plates have a specially designed pin hole. A practical application of the test procedure is illustrated in the extensive series of bolt bearing tests in boron epoxy reported by Rogers[103] in examining the effects of edge distance, thickness, and laminate composition on the bearing strength under loading through standard and countersunk bolts. The range of variables treated was limited to the region of applicability in the specific fuselage component. The test specimen configurations and defining terminology are shown in Figure 3-19. The range of variables treated are given in Table 3-2. Testing was accomplished by placing the specimen in the test fixture and loading slowly to failure. Characteristic behavior of the boron-epoxy specimens during the loading to failure is illustrated in Figure 3-20 and represent the three types of load/deflection curves of the observed behavior[103]. Fracture characteristics of the boron-epoxy specimens are categorized into the four types illustrated in Figure 3-21. Laminates used by Rogers in this phase of the investigation were of the 0°/±45°/90° family.

Included in the investigation was an exploratory study of graphite-epoxy bolt bearing strengths employing the same specimen design and test apparatus utilized in the boron-epoxy system. The graphite-epoxy specimens exhibited a well-defined yield point and a significant amount of deformation prior to fracture. The latter is illustrated in Figure 3-22.

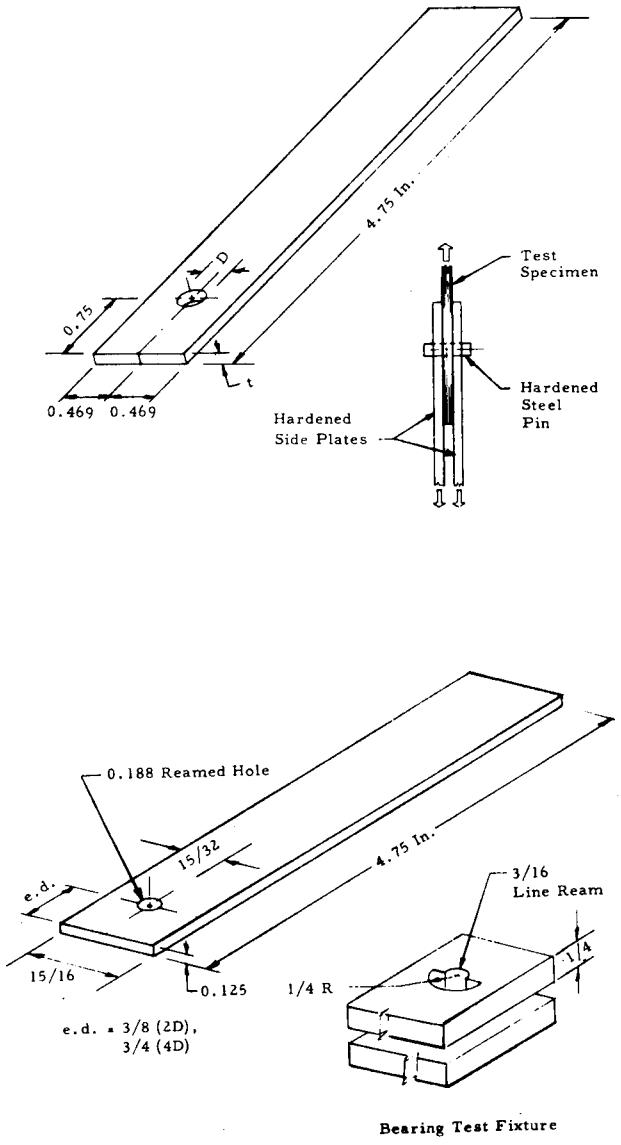
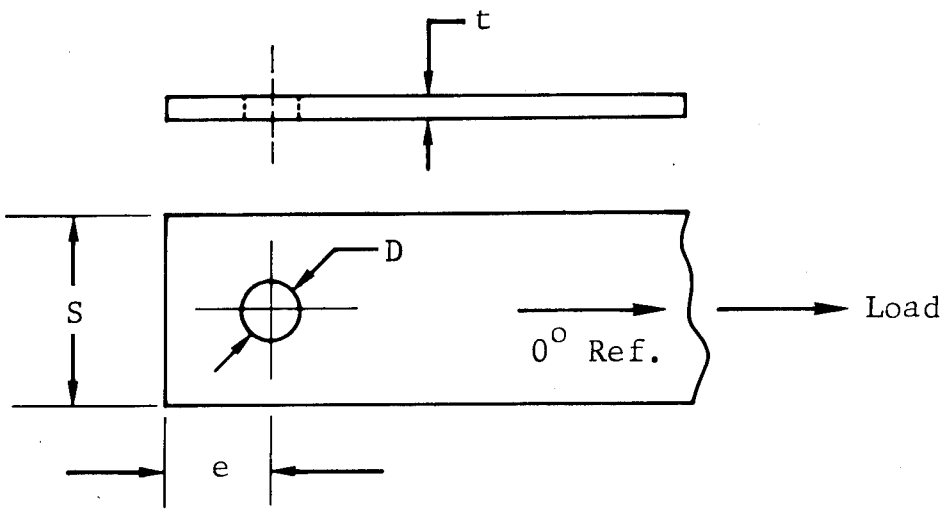
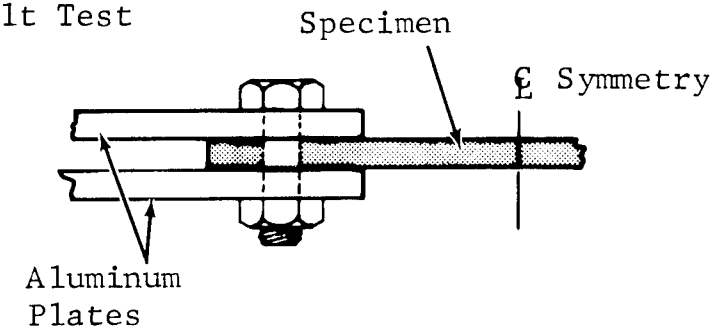


FIGURE 3-18. BEARING TEST SPECIMENS[44,71,102]



Standard Bolt Test



Countersunk Bolt Test

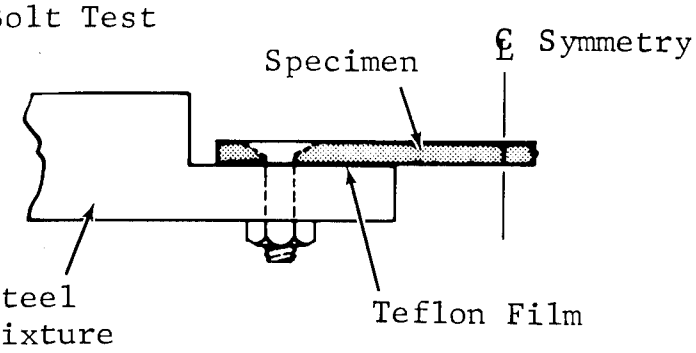


FIGURE 3-19. BOLT BEARING SPECIMENS AND TEST APPARATUS[103]

TABLE 3-2 - RANGE OF VARIABLES TREATED IN BOLT BEARING TESTS[103]

			Laminates		
e/D	S/D	D/t	No. 0°	No. ±45°	No. 90°
2.0	2.80	1.8	6	10	0
2.5	4.00	2.2	0	10	6
3.0	5.26	4.2	6	14	0
3.5	6.58		8	12	0
4.0	7.53		6	12	2
5.5	7.89				

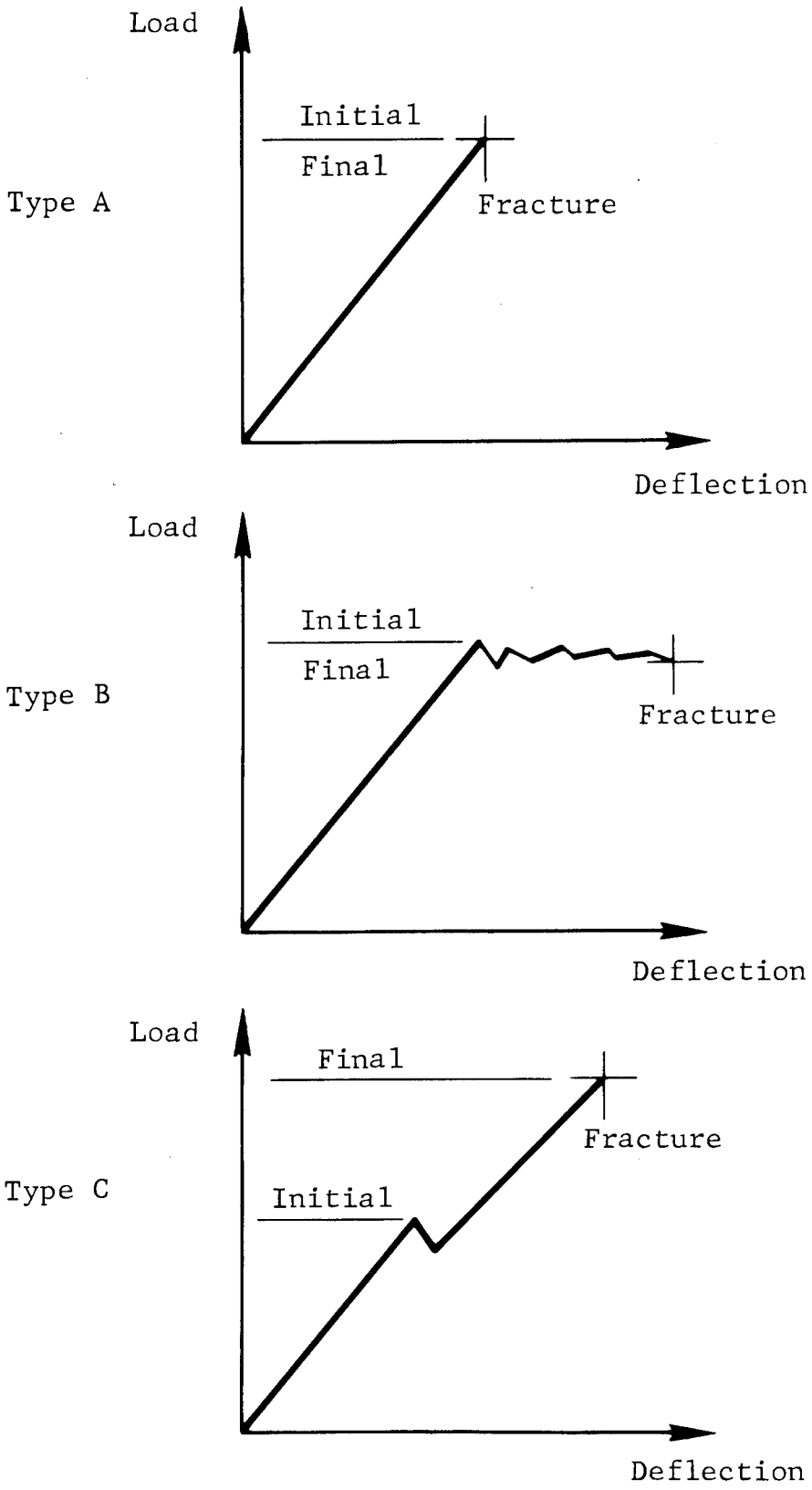


FIGURE 3-20. LOAD/DEFLECTION BEHAVIOR OF BORON/EPOXY BEARING SPECIMENS[103]

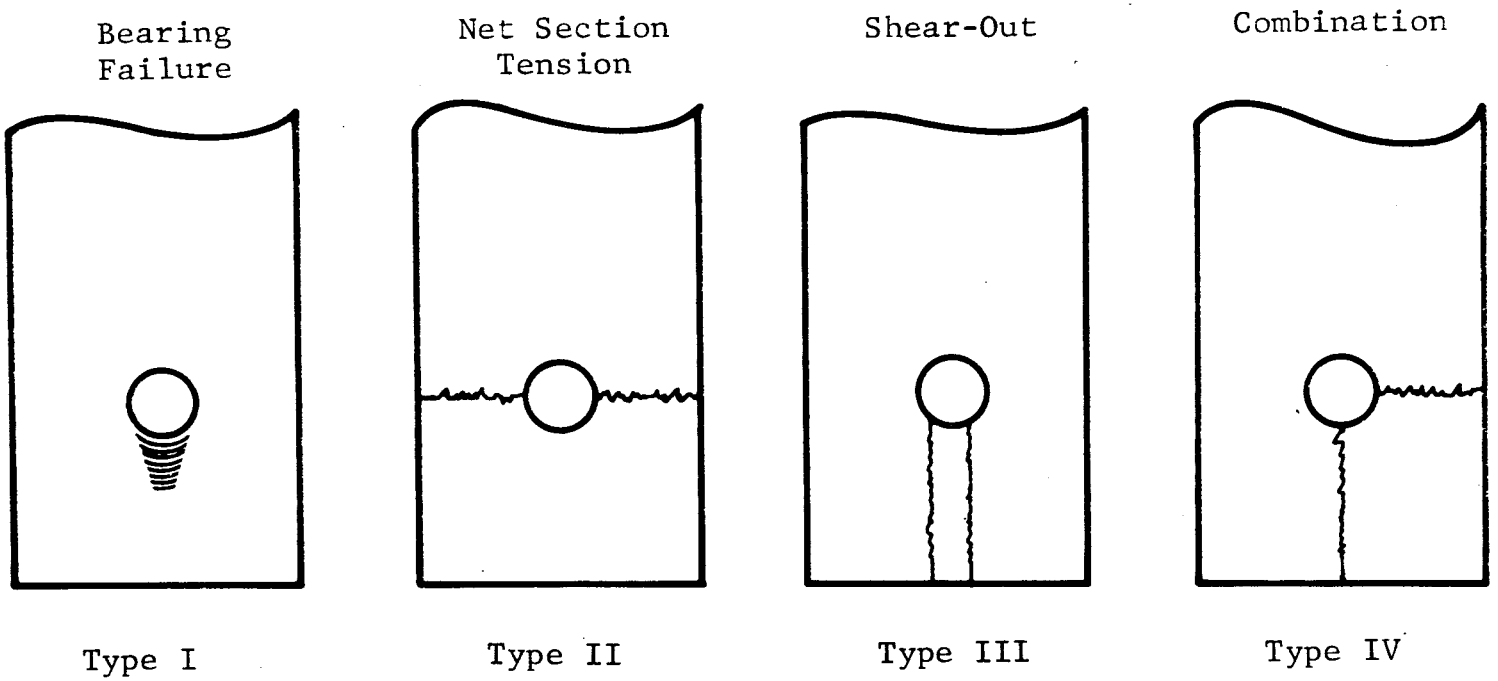


FIGURE 3-21. FRACTURE CHARACTERISTICS OF BORON/EPOXY BEARING SPECIMENS[103]

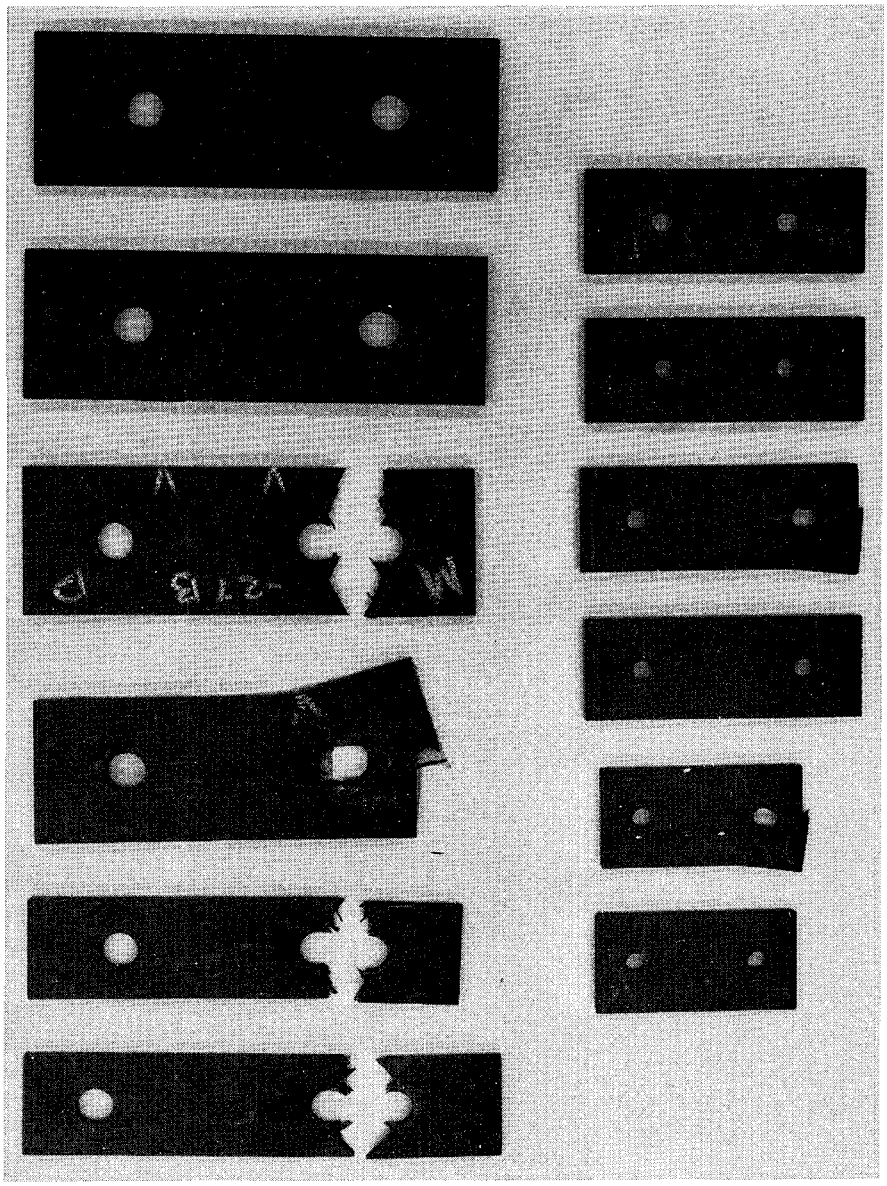


FIGURE 3-22. GRAPHITE/EPOXY BOLT BEARING SPECIMENS[103]

This abbreviated review of the actual utilization of an engineering or design-type test in an end-item component design and development is illustrative of the absolute requirement for the development of reliable, reproducible design test methods for establishment of industry-wide accepted standard procedures.

3.1.7 Fatigue

Fatigue properties and characteristics of composite material is of major significance in the effective design of composite structures. The basic concept of fatigue of composites is no different than that for metals; however, it is considerably more complex. This is due principally to the anisotropy of materials.

Recommended fatigue test methods for resin matrix composite laminates are adapted from the test methods for boron epoxy as specified by Grumman[104]. Test methods will be treated under the specific type of specimen loading practices; namely, axial, internal pressure, and spectrum.

3.1.7.1 Axial Loading

Fatigue characteristics of both notched and unnotched composites are determined by coupon-type specimens for unidirectional and multidirectional fiber orientations at room temperature and 375°F (190°C). The IITRI type coupon is employed for axial tension loading, both notched and unnotched and at both room and elevated temperatures. The recommended specimen thicknesses for varying fiber orientations are summarized in Table 3-3.

Coupon specimen configuration recommended for axial loading is illustrated in Figure 3-23 showing recommended notch diameter and location. Tests are conducted in a repeating stress testing machine capable of applying constant force for axial, bending, or torsional stresses or any combination of these at a controlled constant rate. Machine requirements include: (1) automatic control of loads and preloads within 2 percent of desired load; (2) a counter capable of recording number of applied cycles in specified increments; (3) disengagement of both counter and testing upon specimen failure. Specimen failure should occur within the coupon gage length or, at least, one specimen thickness from the tab ends. Prior to testing, the maximum stress level to which the specimen is to be stressed must be determined and expressed as percent of average ultimate strength. If unknown, ultimate strength, as determined by previous axial tension

TABLE 3-3 - PLY DESCRIPTION[104]

Designation	No. of Plies	Thickness Range, In.	Ply (Fiber) Orientation ± 1/2°
A	6	.0294-.0330	Unidirectional 0°
B	16	.0798-.0880	Unidirectional 90°
C	8	.0392-.0440	0, 90, 0, 90, 0, 90, 0, 90° Asymmetrical
E	9	.0441-.0495	0, 45, 135, 0, 90, 0, 135, 45, 0

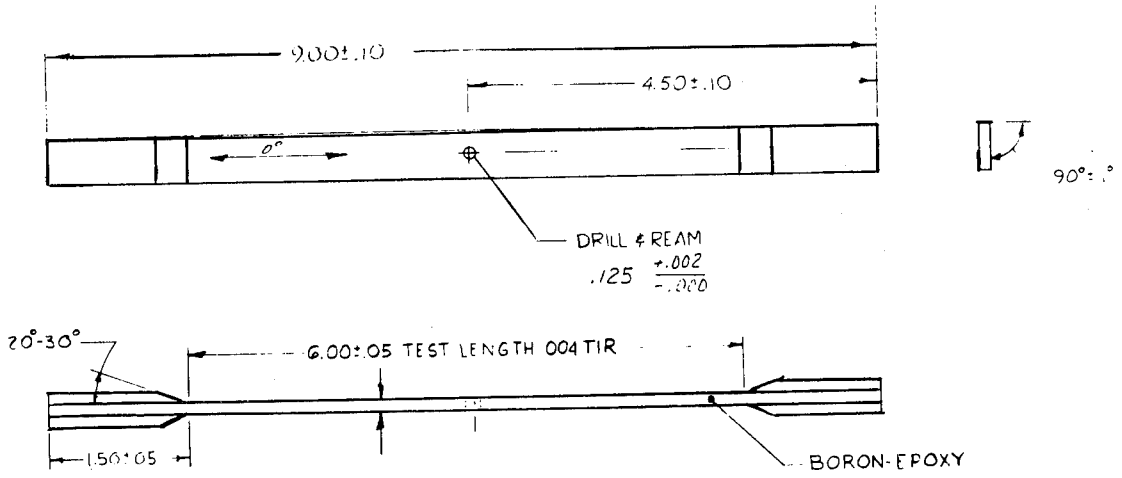


FIGURE 3-23. NOTCHED FATIGUE SPECIMEN[104]

or compression static tests, may be used. The stress ratio is expressed in terms of minimum applied stress versus maximum applied stress. Applied loads and stress ratios can be established using the formulas:

Maximum load: $P_{max} = F_{max} A' = F_{max} (b-d)t'$

Minimum load: $P_{min} = F_{min} A' = F_{min} (b-d)t'$

Stress Ratio: $\frac{F_{min}}{F_{max}}$

where:
- P = load in pounds
- $F_{(max)}$ = stress having highest algebraic value in loading cycle in psi
- $F_{(min)}$ = stress having lowest value
- A' = theoretical or normalized net area in square inches
- b = measured specimen width
- d = notch length or diameter
- t = measured specimen thickness

The test is conducted to specimen failure or until desired number of cycles have been applied.

For elevated temperature test, thermocouples are applied to the surface of the test section for temperature measurement and control. The instrumented specimen, inclusive of end attachment assemblies, can be wrapped with resistance heating tape. Specimen should be allowed to soak at prescribed test temperature for 30 to 40 minutes prior to test initiation.

For subnormal temperatures, the instrumented specimen, inclusive of end attachments, can be installed in an insulated test chamber through which air or another suitable gas is circulated at the prescribed temperature. A 30- to 40-minute soak at the prescribed temperature is suggested prior to test initiation.

3.1.7.2 Internal Pressure Loading

This method is designed for study of the fatigue characteristics of cured fiber-resin laminates acted upon by cyclic internal pressure. The test specimen is a thin-walled cylindrical laminate representative of a high-quality flat laminate. The test specimen with recommended dimensions is illustrated in Figure 3-24. Influence of the end caps in development of bending stresses within the cylinder should be determined. The specified number of cycles applied for any one test should be limited to 10,000. Residual properties of test specimens achieving the designated fatigue limit should be determined.

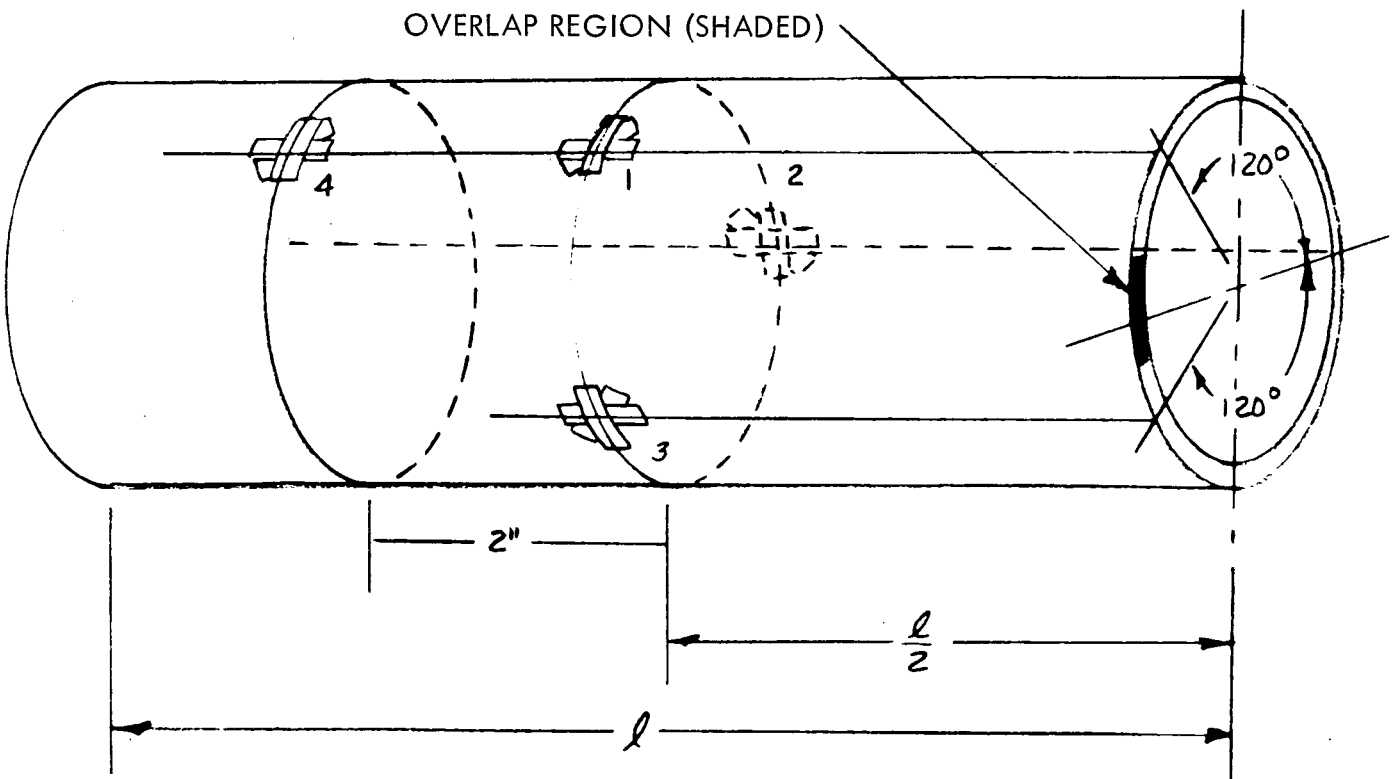
A detailed treatment of test cylinder end tabs and attachments and the basic test apparatus is given in the Reference 104. The pressurization apparatus uses water plus 10 percent soluble oil as the pressurization medium.

For each group of tests conducted, it is recommended that one specimen should be instrumented with strain rosettes positioned at the four locations depicted in Figure 3-24. For each additional specimen in the group, a strain rosette should be installed at location No. 2 only as shown in the above figure. The test should be conducted at a constant pressurization rate of 1000 psi/min. Concurrent with load application, a continuous deflection curve should be recorded for each of the strain circuits as a function of applied pressure. Prior to testing, the stress ratio should be determined, and wall thickness and both inside and outside specimen diameters should be measured to the nearest 0.001 inch. Minimum dimensions, based on the determinations in the central region of the specimen, should be recorded. Testing is continued to failure, or until prescribed number of fatigue cycles have been applied. Residual hoop stress should be determined upon completion of test.

Normalized loading conditions are calculated to three significant figures using the following formulas:

$$\text{Maximum applied pressure} \quad P_{max} = \frac{F_\theta^{max} t'}{R_m}$$

$$\text{Minimum applied pressure} \quad P_{min} = \frac{F_\theta^{min_{t'}}}{R_m}$$



Note: Rosettes 1, 2, and 3 are equally spaced (120° intervals) around center of test section; rosette 2 is approximately 180° from overlap region. Rosette 4 is spaced on a section 2 inches (or other specified distance) from the center section.

ℓ = Length of Test Specimen

FIGURE 3-24. THIN WALLED CYLINDER INSTRUMENTATED WITH STRAIN GAGE ROSETTES[104]

$$\text{Stress Ratio} \qquad R = \frac{F_\theta^{\min}}{F_\theta^{\max}}$$

where P = pressure in psi

$F_\theta^{(max)}$ = hoop stress having highest algebraic value in the cycle in psi

$F_\theta^{(min)}$ = hoop stress having lowest algebraic value in the cycle in psi

t' = theoretical or normalized thickness (ply thickness x number of plies)

R_m = mean radius of cylinder in inches

Reported results should include:

1. Specimen identification
2. Minimum and maximum applied stress
3. Number of cycles to failure or termination
4. Residual hoop stress
5. Failure mode

3.1.7.3 Spectrum Loading

This method defines the fatigue characteristics of resin matrix composites under spectrum loading employing a beam-type specimen. The significant test area is limited to the composite facing of a honeycomb sandwich beam that is tested as a simply supported beam under symmetrical two-point loading. Specimen configuration and dimensions are shown in Figure 3-25. Since the specimen is tested as a supported beam under symmetrical two-point loading, it has been designed to incorporate a spliced three-piece honeycomb core.

The center portion of the core where vertical shear is zero during the test, is of lightweight honeycomb, while the ends where maximum vertical shear is active are of heavyweight honeycomb. The design minimizes the possibility of transverse failure occurring in the test facing due to high Poisson's effect of the heavyweight core.

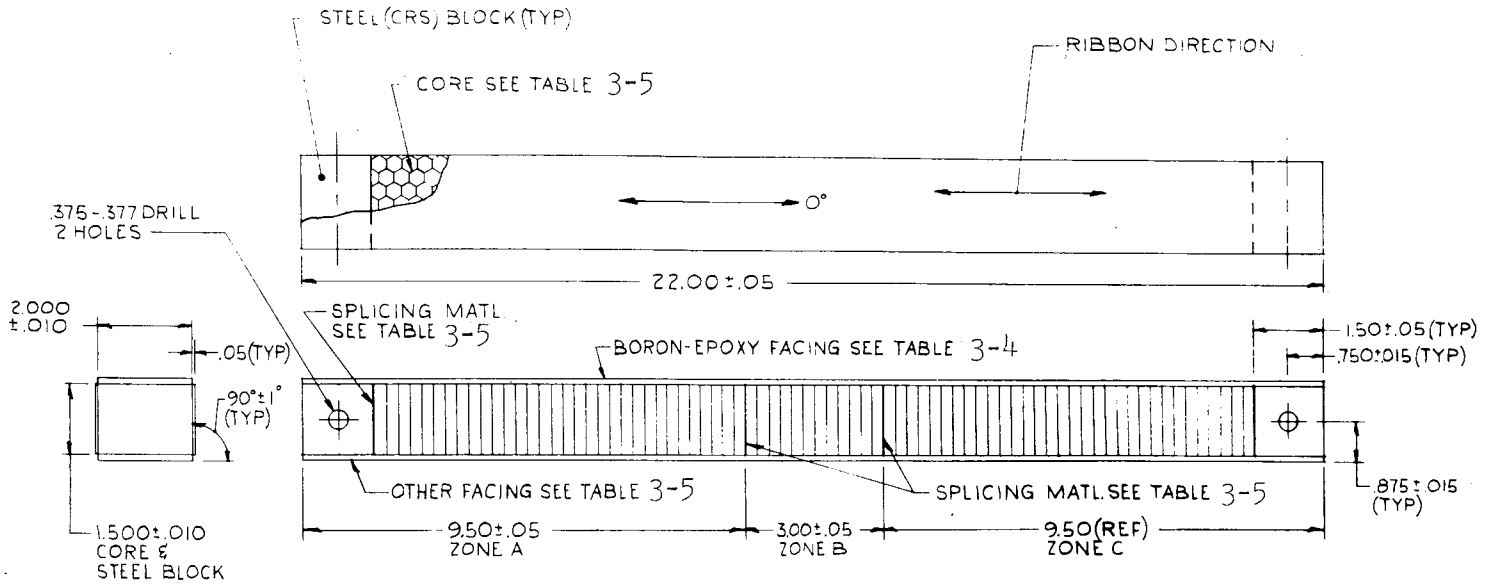


FIGURE 3-25. SPECIMEN CONFIGURATION OF HONEYCOMB SANDWICH BEAM FOR SPECTRUM LOADING[104]

Table 3-4 summarizes the recommended composite facing thicknesses and ply orientations. Table 3-5 defines the specimen relative to core designation, splicing material, etc. Specimen loading and support method is illustrated in Figure 3-26. The test can be conducted in any device capable of applying and measuring the prescribed loads and the number of applied events. The accuracy should be within ±2% for all loading conditions. A counter for recording the occurrences and a disengaging device capable of stopping the test upon specimen failure is required. For repeated stress loading, the constant force type universal fatigue machine as defined for axial loading should be used. Recommended cycling rate is 1800 cpm. When applicable, specimen deflection should be measured at the center of the primary span using a dial indicator calibrated in increments of .001 inch.

Prior to testing, the stress ratio desired should be defined. If the ultimate strength of the composite is unknown it may be established by static tests. The stress cycling rate must be determined. Applied loads can be calculated using the formulas:

Maximum applied Load:

$$P_{max} = \frac{F_{max}\, 2bt' \left[t_c + \frac{t' \times t}{2}\right]}{a}$$

Minimum applied load:

$$P_{min} = \frac{F_{min}\, 2bt' \left[t_c + \frac{t' - t}{2}\right]}{a}$$

where P = load in pounds

$F_{(max)}$ = stress having highest algebraic value in fatigue cycle in psi

$F_{(min)}$ = minimum stress

b = specimen width

t' = theoretical or normalized specimen thickness

t_c = core thickness

t = thickness of sacrificial skin

a = moment arm length

TABLE 3-4 - BORON FACING DESCRIPTION [104]

Designation	No. of Plies	Thickness Range Inches	Ply (Fiber) Orientation ±1/2°
F	8	.0392-.0440	0, 45, 135, 90, 90, 135, 45, 0°
S	11	.0539-.0605	45, 135, 0, 0, 0, 90, 0, 0, 0, 135, 45°

TABLE 3-5 - SPECIMEN DESCRIPTION (BORON FACING PER TABLE 3-4) [104]

Temp. Range, °F	Aluminum Honeycomb Core Designation		Other Facing	Splice Material		Adhesive Material	
	Zones A & C	Zone B		Type	Cure Cycle	Type	Cure Cycle
RT to +375	1/8 - 2024(T81)-23.1N	1/8-2024(T81)-6.7N	.125" Thick			Metlbond 329 Type lb	1 Hr/350 °F/ 25-45 psi
-67 to RT	1/8 - 5052(H39)-23.1N		4340 Steel (160-180 ksi)	AF 3002	1 Hr/ 350°F		

NOTES: (1) Specimens shall be fabricated with the boron surface of the boron facing at the adhesive faying surface. This surface shall be prepared for bonding in accordance with GMPS 3005.

(2) The steel facing and blocks are to be prepared for bonding by vapor degreasing, dry honing per GSS 7012 and then primed with EC 2333 within 1 hour.

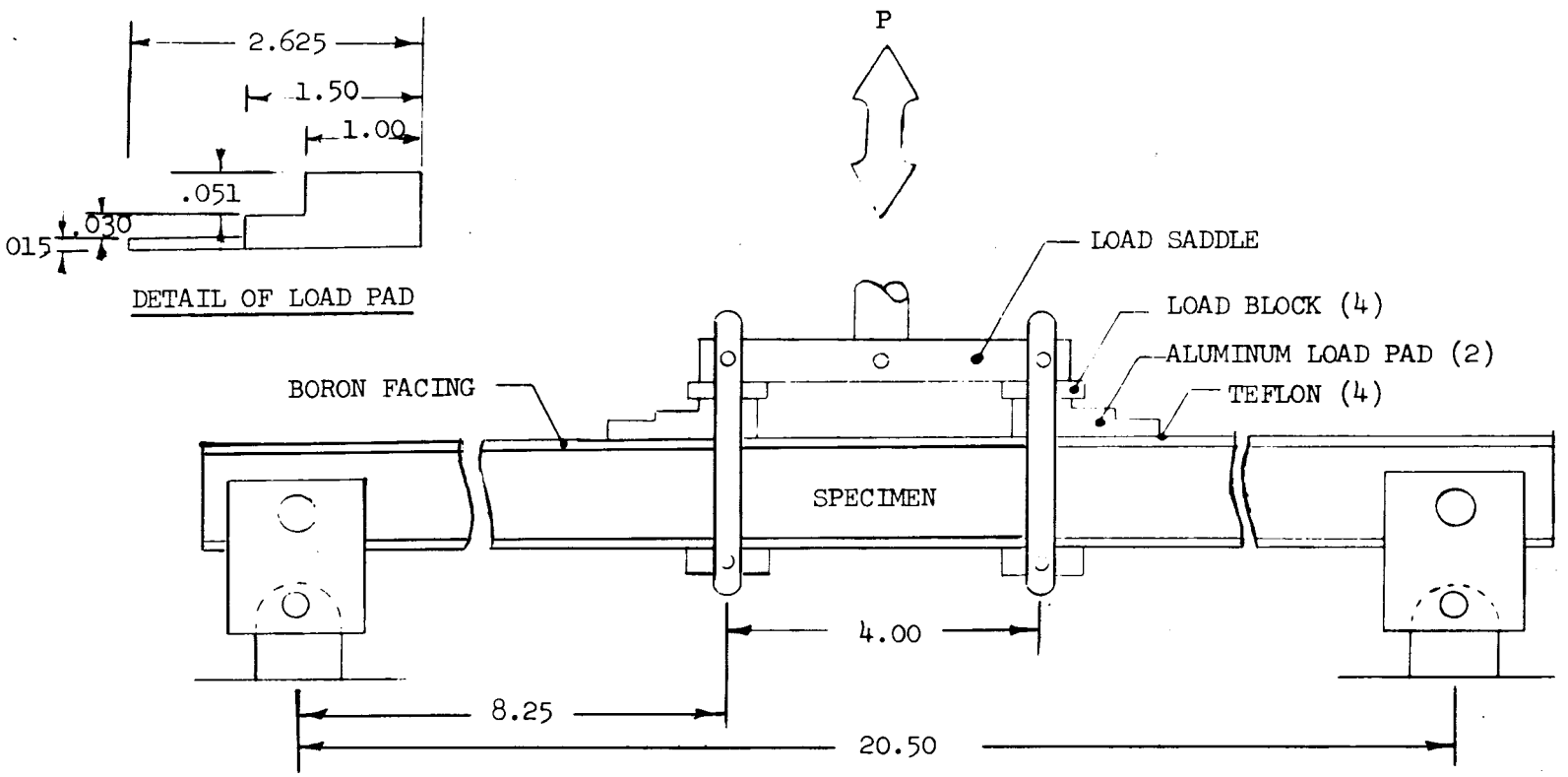


FIGURE 3-26. LOADING AND SUPPORT FOR BEAM SPECIMEN[104]

Upon completion of application of the fatigue conditions desired, either the residual tensile or residual compressive strength, depending upon the mode of stress applied during fatigue loading should be determined by employing static test procedures.

3.1.8 Creep

Data generated from short-term mechanical property tests have provided the basis for predicting the behavior and use potential of low-termperature advanced composite materials. Relatively little data, however, are currently available on the behavior and dimensional stability of resin matrix composites under service environments for extended time periods. Creep represents one of the long-term characteristics of structural materials which determine their reliability for use in certain applications. Creep phenomena in conventional structural materials have been extensively studied. In contrast, only limited studies of creep in composites have been conducted. In view of current and projected use of resin matrix composites, a detailed characterization study of the creep and stress-rupture characteristics is required to predict their reliability in structural applications.

A standard specimen for creep testing of filamentary composites has not been established. The IITRI-type specimen, however, is presently considered to be the most effective, and provides data comparable with static results. In general, therefore, any specimen configuration suitable for tensile tests can be considered for creep tests. An Instron Universal test machine or equivalent can be employed to obtain creep (strain time) with specimens held at constant load. The basic recommended practices guiding creep testing are specified in ASTM D674-56 (1969)[105].

Some of the problems experienced include nonuniform thermal expansion of test specimens during heating to assigned test temperatures and bowing of specimens of nonsymmetrical laminates. Determination of specimen temperature during the test is one of the most important single measurements. Minute variations in temperature may produce large variations in creep rate. The gage length of the specimen should be adequately instrumented with thermocouples to ensure accurate temperature measurements during the tests. A controlled temperature furnace or box should be maintained at a constant temperature preferably by an automatic device. Reliable temperature measurements should be made and recorded at frequent intervals to ensure accurate determinations of mean temperature. The maximum variation in temperature throughout the test procedure should be minimized. Similarly, temperature distribution over the specimen gage length

should be held to the least possible minimum, and the extent of such variation should be recorded.

Accuracy and sensitivity of the strain (specimen extension) measuring equipment should be suitable for the material. A sensitivity of not less than 0.001 in/in should be maintained for an anticipated creep of 50 percent; however, sensitivities on the order of 1×10^{-6} inch are generally required.

Creep tests conducted on 0°, 90° and 0°/45° fiber orientations in boron-epoxy laminates at room temperature and 270°F (130°C) employing an Instron testing machine are shown in Figures 3-27 to 3-32. Test objectives and parameters are shown in each figure. For most cases illustrated, immediate strain recovery upon release of load was nearly equal to original strain during load application. Total creep recovery (immediate + secondary) was seldom complete. Recovery was greater for room temperature tests than for corresponding tests at 270°F (130°C). As illustrated also, maximum strain recovery was indicated for 0° oriented laminates. Creep, as summarized in Table 3-6, was most predominant at ±45° oriented laminates and least predominant for 0° oriented laminates as anticipated.

Figures 3-33 and 3-34 illustrate effect of temperature on creep properties of 30°/60° oriented graphite-epoxy laminates at 90 and 80 percent of ultimate strengths, respectively[106]. Figures 3-35 and 3-36 illustrate the effect of stress level on creep-rupture time for 30°/60° graphite-epoxy laminates at various temperatures.

3.1.9 Crack Propagation

Fracture in both homogeneous materials and anisotropic composite materials most generally involves initiation and propagation of cracks. In view of the significance of crack phenomena, particularly in the case of resin matrix composites, a brief review of test methods designed for study of crack propagation is treated.

3.1.9.1 Single Sweep Method

In their study of crack propagation in resins, Carey and Boyle[107] utilized a notched epoxy specimen of rectangular symmetry (1 x 9 inches) provided with three conductive strips. Time required for a crack to propagate between two strips was measured by an oscilloscope set up in a single-sweep, externally triggered mode. The specimen is

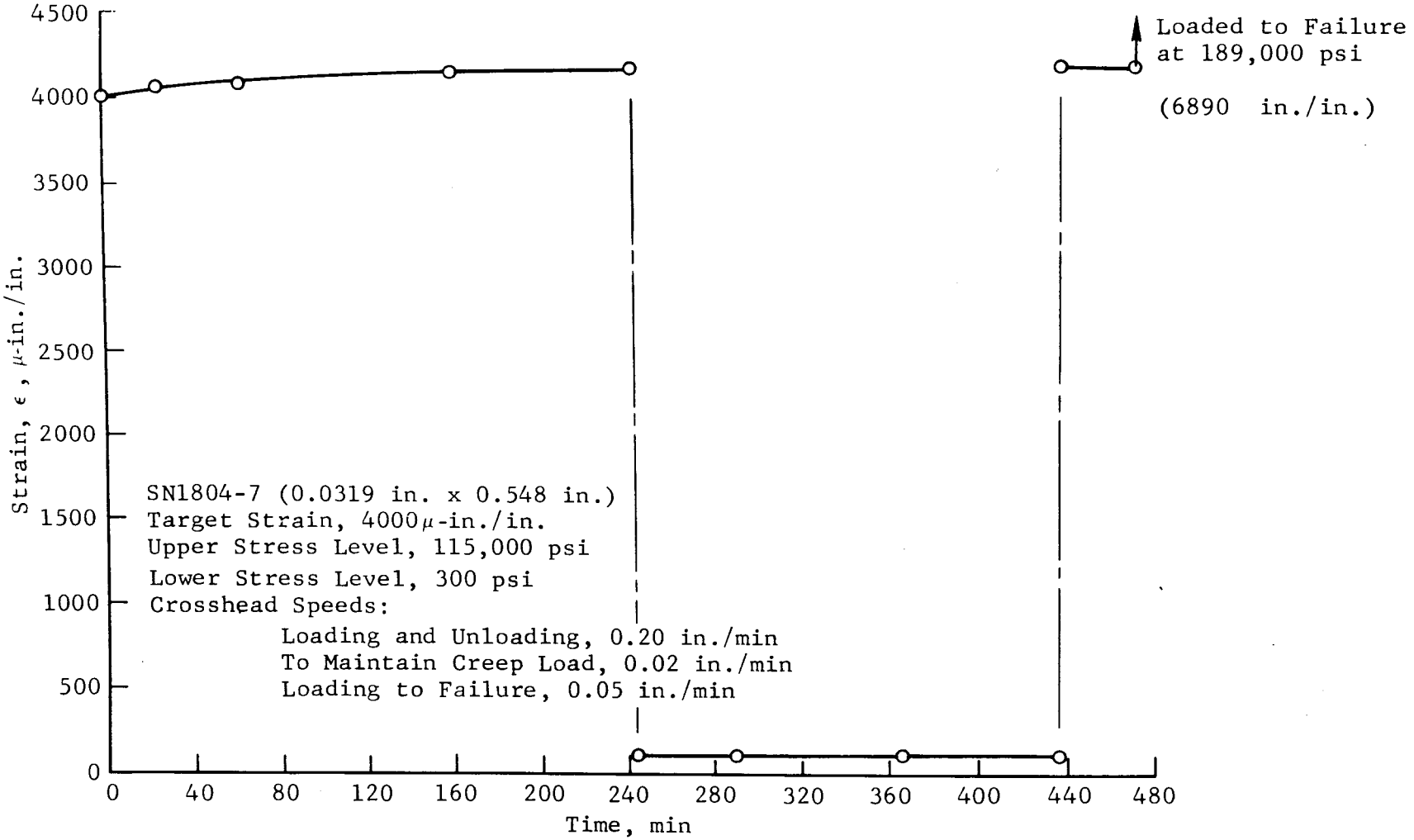


FIGURE 3-27. CREEP TEST, 0° ORIENTATION AT R.T.[64]

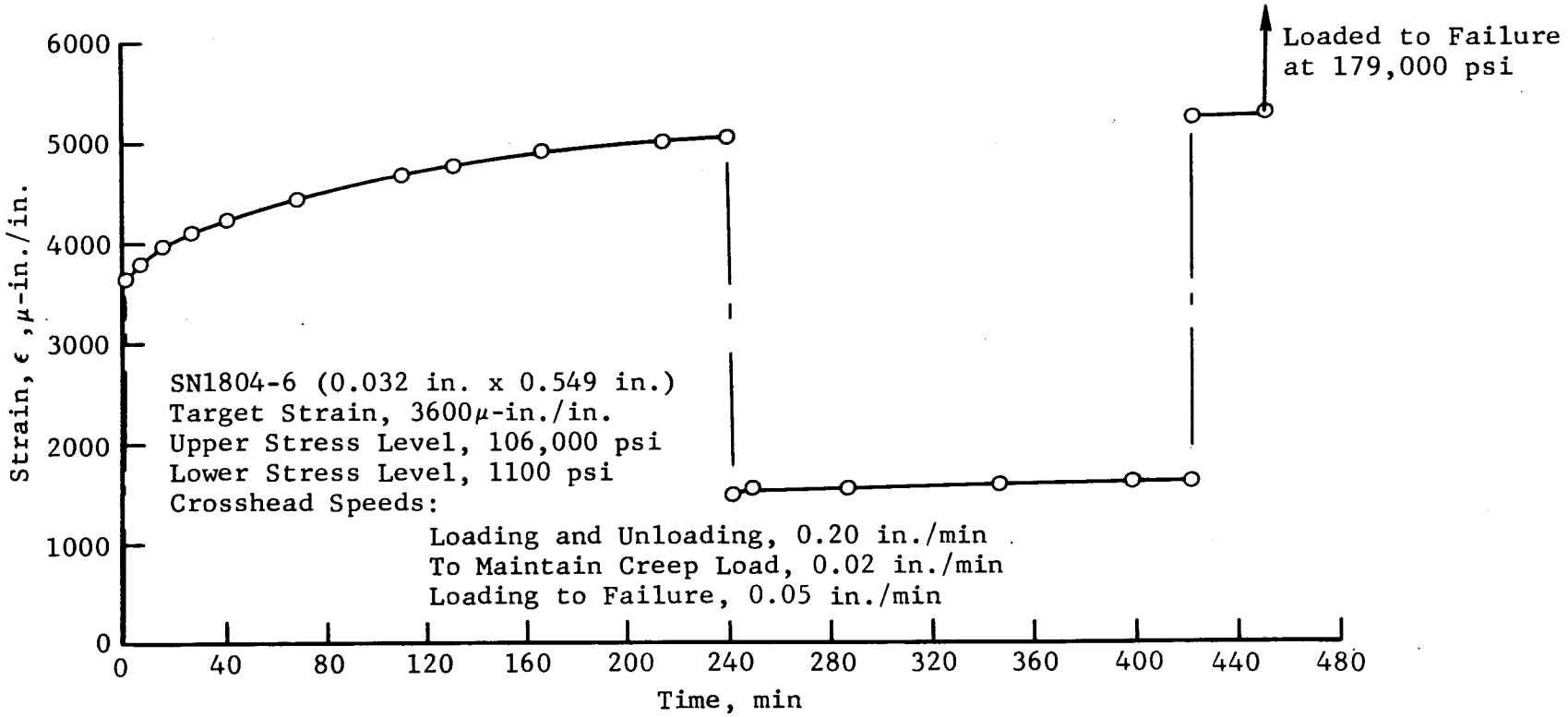


FIGURE 3-28. CREEP TEST, 0° ORIENTATION AT 270°F[64]

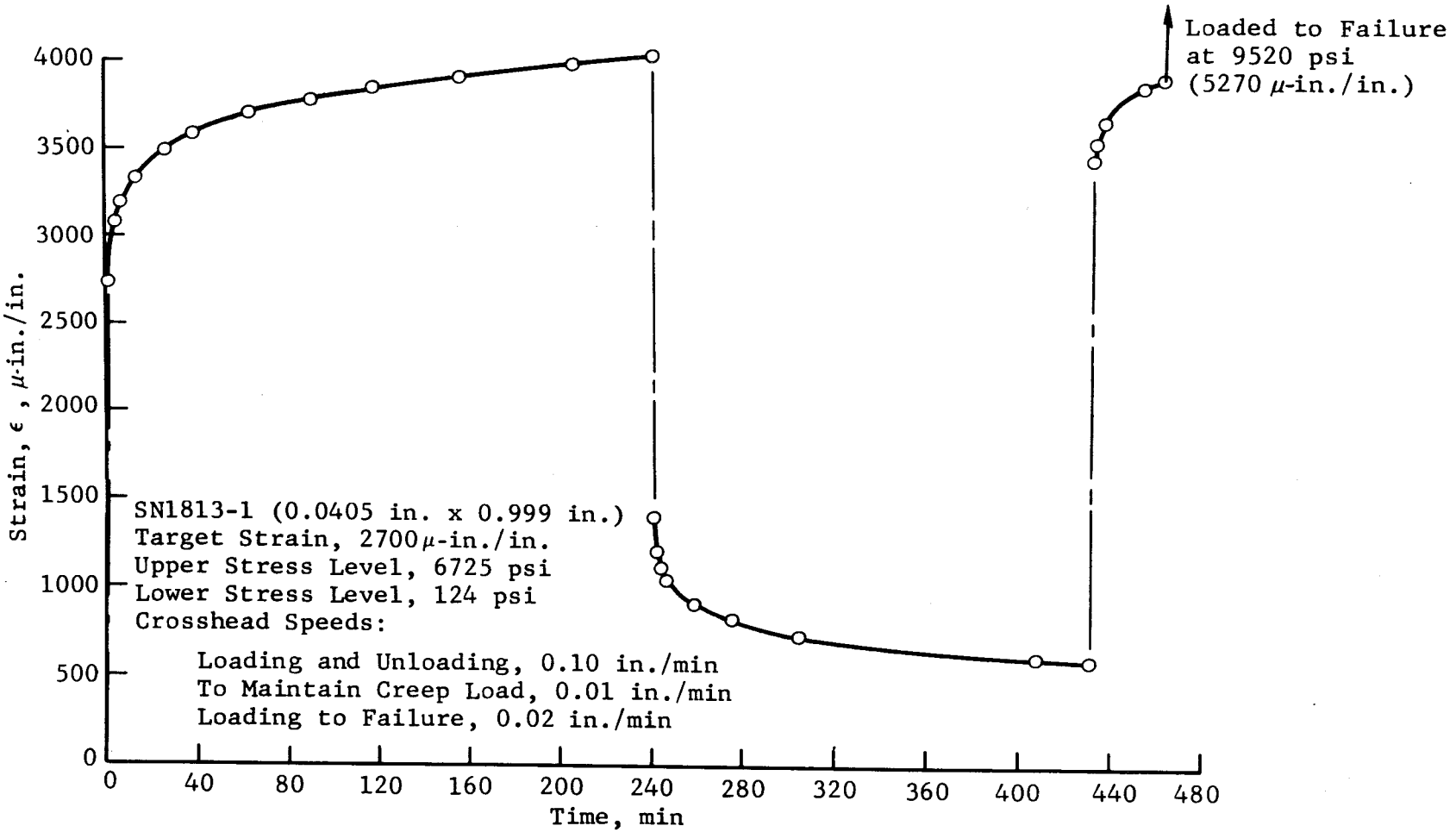


FIGURE 3-29. CREEP TEST, 90° ORIENTATION AT R.T.[64]

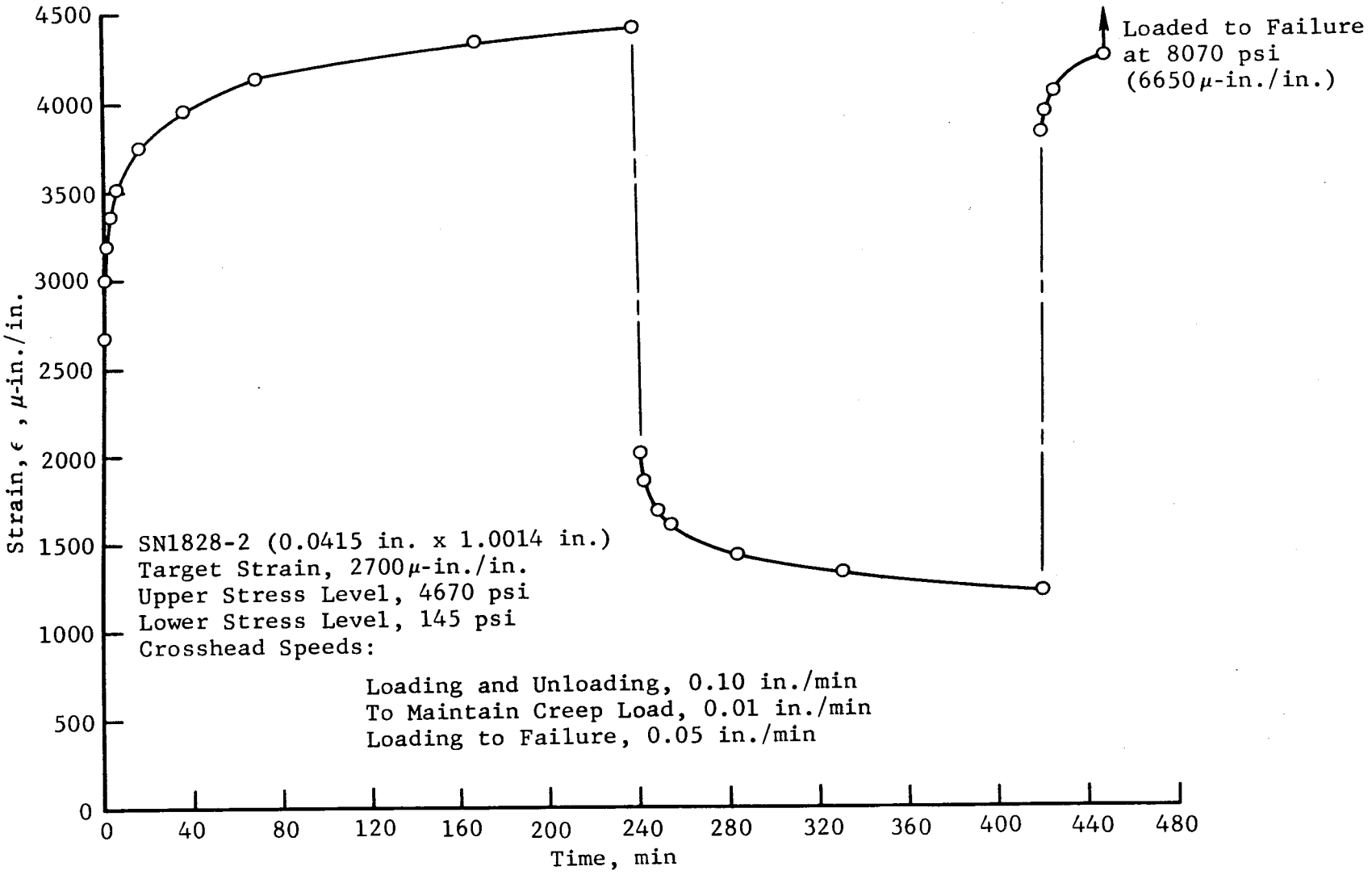


FIGURE 3-30. CREEP TEST, 90° ORIENTATION AT 270°F[64]

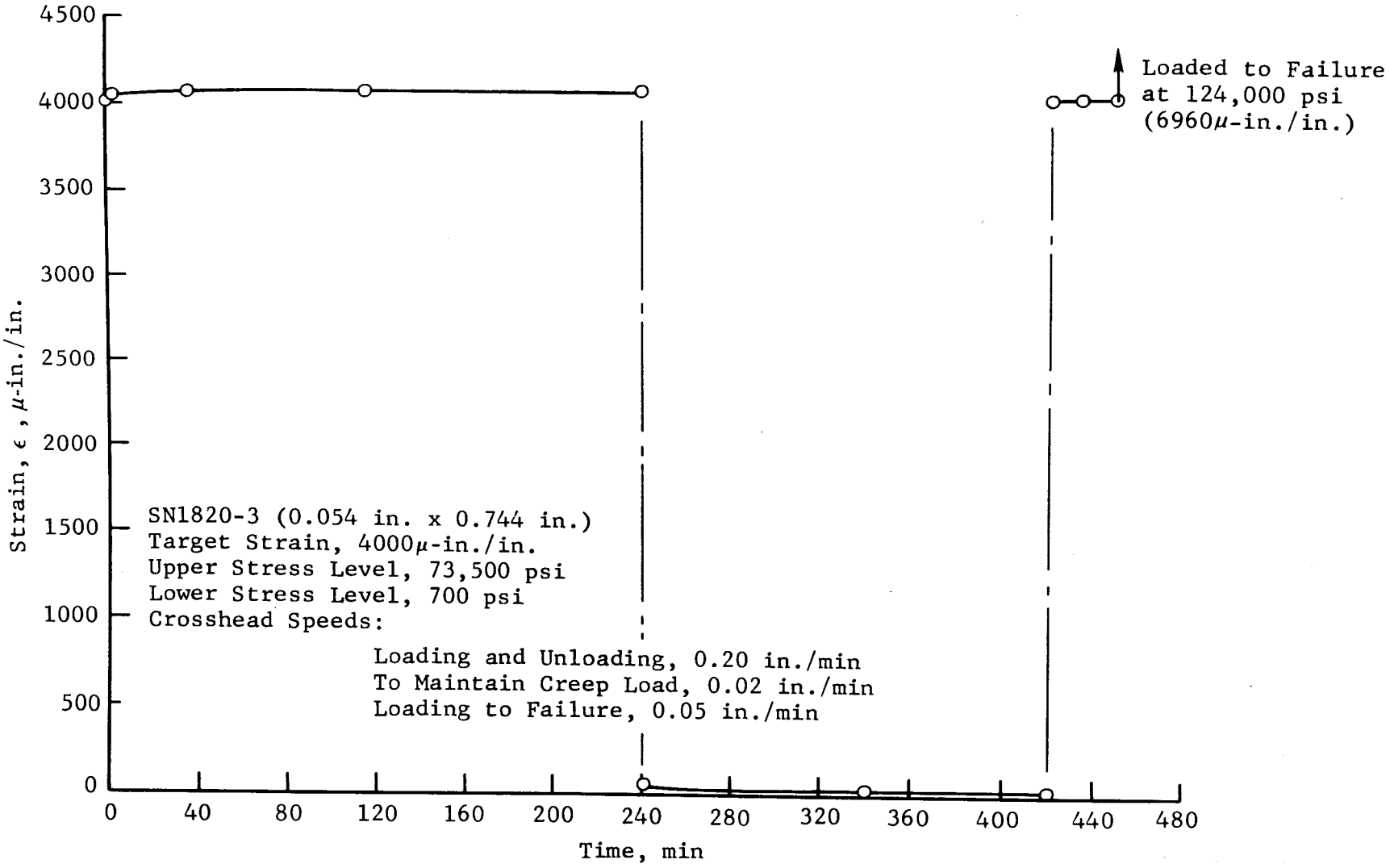


FIGURE 3-31. CREEP TEST, 0°/±45° ORIENTATION AT R.T.[64]

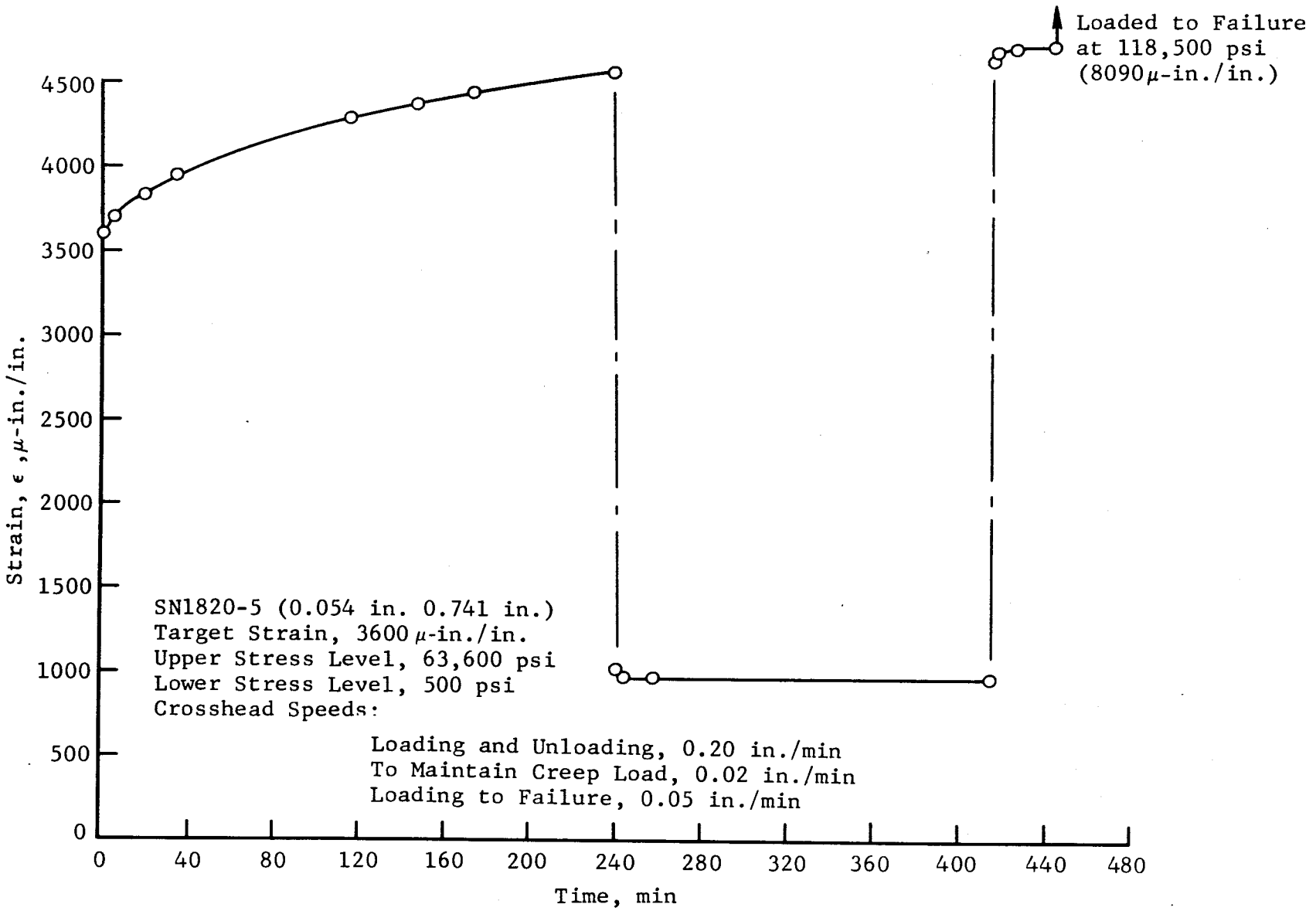
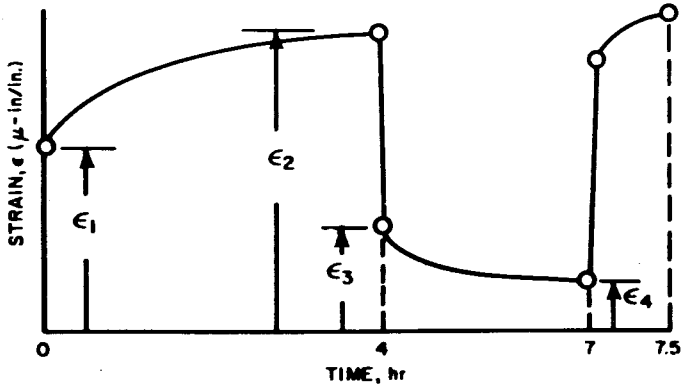


FIGURE 3-32. CREEP TEST, 0°/±45° ORIENTATION AT 270°F

TABLE 3-6 - CREEP TEST DATA FROM BORON/NARMCO 5505 COMPOSITES

Specimen Orientation (degrees)	Test Temp. (°F)	Specimen Number	Thickness (in.)	Width (in.)	Initial Strain (μ-in./in.)	Creep Stress (psi)	Recovery Stress (psi)	Percent Creep[a] During 4-Hours	Immediate Recovery,[b] Percent of Original Strain	Percent Recovery[c] During 3-Hours
0	R.T.	1804-7	.0319	.548	4,010	115,000	300	4.04	101.5	97.6
0	270	1804-6	.0320	.549	3,650	106,000	1,100	38.2	97.8	68.3
90	R.T.	1813-1	.0405	.999	2,730	6,725	124	47.5	96.0	85.1
90	270	1828-2	.0415	1.001	2,670	4,670	145	65.3	90.3	72.7
0/±45	R.T.	1820-3	.0540	.744	4,020	73,500	700	1.49	100.4	99.4
0/±45	270	1820-5	.0540	.741	3,600	63,600	500	26.4	98.5	79.0
90/±45	R.T.	1827-2	.0526	1.001	2,710	11,600	330	15.6	98.8	91.2
90/±45	270	1827-1	.0527	.999	2,730	10,130	350	31.2	96.5	82.1
±45	R.T.	1811-2	.0415	.949	8,940	15,400	300	90.4	134.8	84.0
±45	270	1811-3	.0417	1.004	5,750	7,330	95	136.8	109.3	65.7
±45	R.T.	1811-6	.0413	1.002	9,130	14,900	110	-	110.5[d]	-
±45	270	1811-4	.0414	1.003	6,090	6,600	90	-	110.5[d]	-
±45	R.T.	1811-5	.0415	1.005	3,000	7,440	130	-	110.8[d]	-
±45	270	1811-1	.0412	1.018	2,160	2,575	100	-	68.2[d]	-



a. $\frac{(\epsilon_2 - \epsilon_1)}{\epsilon_1} \times 100$

b. $\frac{(\epsilon_2 - \epsilon_3)}{\epsilon_1} \times 100$

c. $\frac{(\epsilon_2 - \epsilon_4)}{\epsilon_2} \times 100$

d. Recovery during first cycle only.

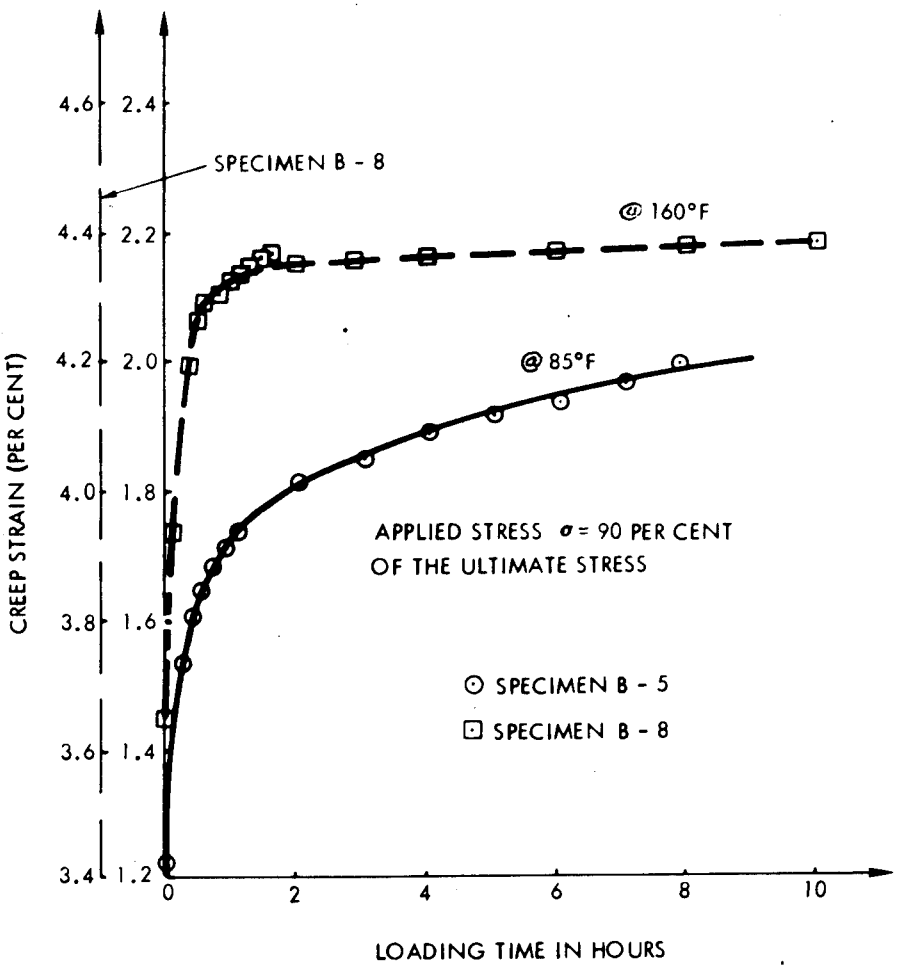


FIGURE 3-33. THE EFFECT OF TEMPERATURE ON THE CREEP STRAIN OF (30 DEG -60 DEG) LAMINATES STRESSED AT 90 PERCENT OF ITS ULTIMATE[106]

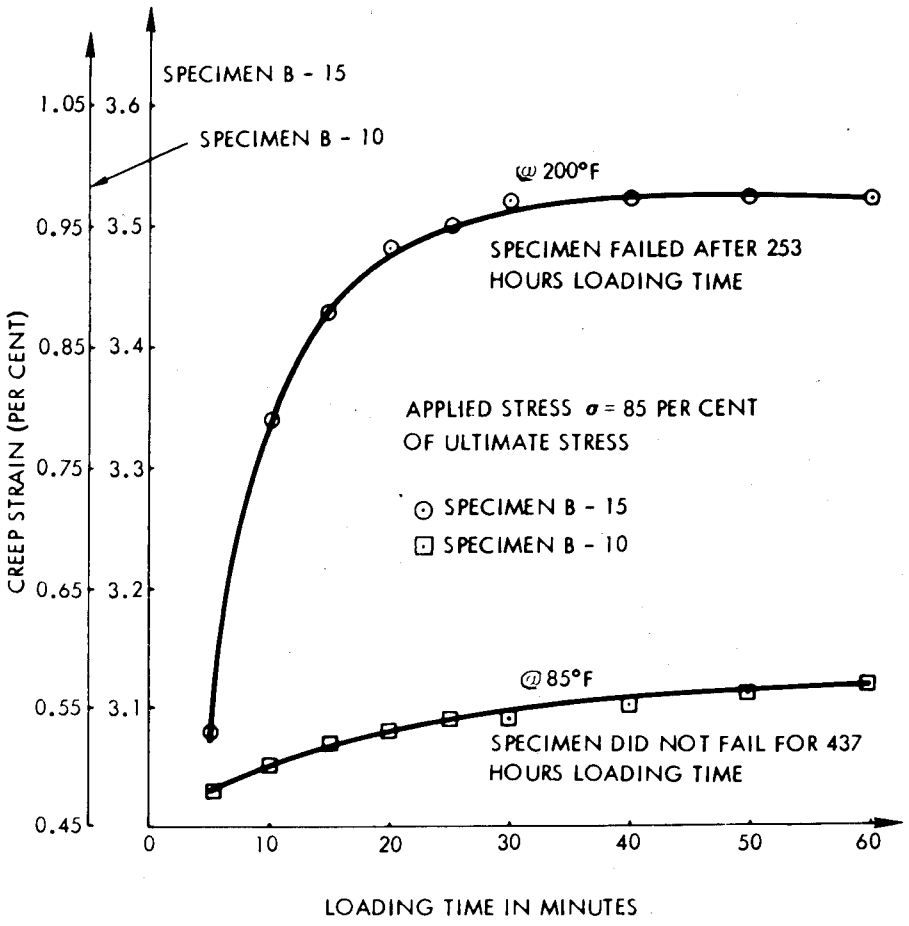


FIGURE 3-34. THE EFFECT OF TEMPERATURE ON THE CREEP STRAIN OF (30 DEG -60 DEG) LAMINATES STRESSED AT 85 PERCENT OF ITS ULTIMATE[106]

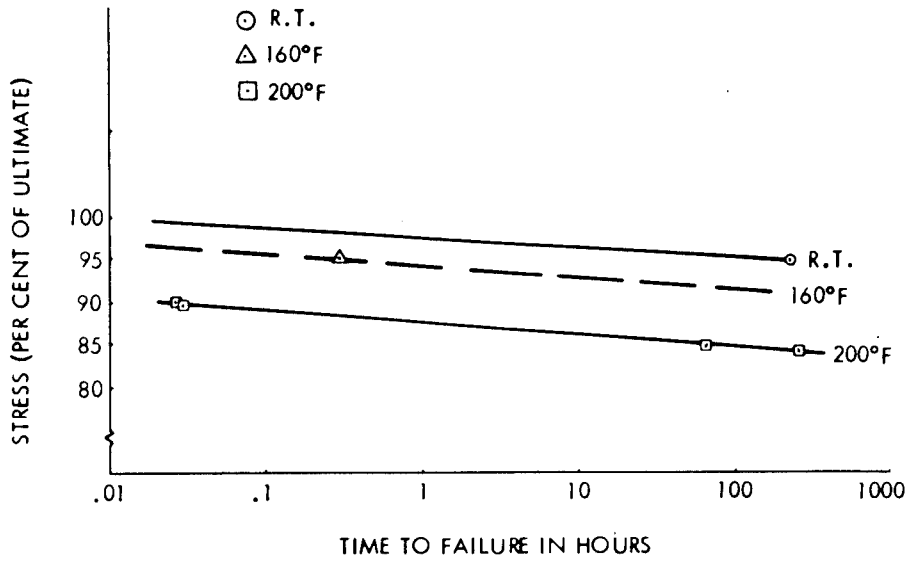


FIGURE 3-35. THE EFFECT OF STRESS LEVEL ON THE CREEP RUPTURE TIME FOR (30 DEG, -60 DEG) LAMINATES TESTED AT DIFFERENT TEMPERATURES

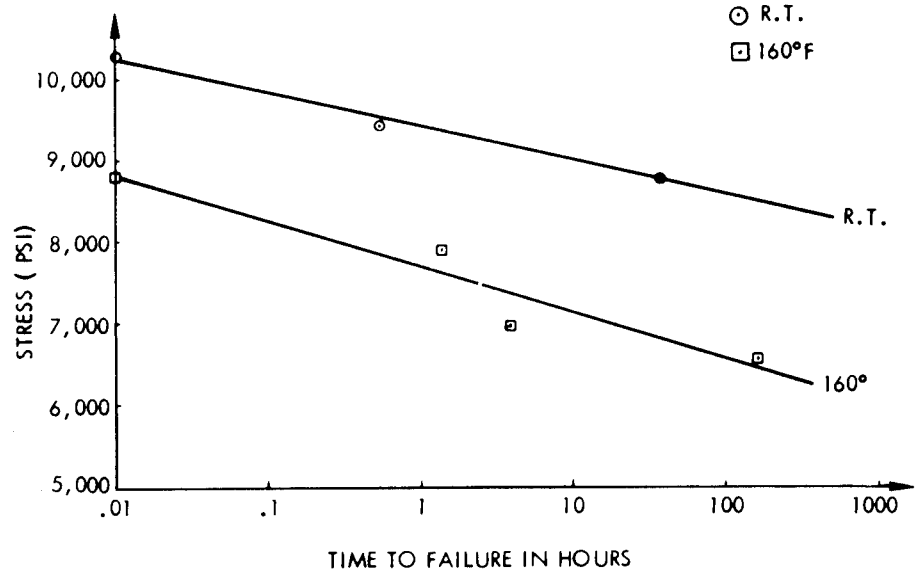


FIGURE 3-36. THE EFFECT OF STRESS LEVEL ON THE CREEP RUPTURE TIME FOR (45 DEG, -45 DEG) LAMINATES TESTED AT DIFFERENT TEMPERATURES

shown in Figure 3-37. The conductive strip immediately adjacent to the apex of the notch was successfully employed to trigger the scope sweep prior to the start of the time measurement trace.

The effect of tensile load rate, 0.02, 0.05, 1.0, and 10.00 inch/min., on crack propagation in EPON 828/1031 epoxy was determined. Since the results showed no significant trend, a load rate of 0.05 in/min. was used in all succeeding studies. The types of failure observed by Carey and Boyle fell between a basic straight line fracture associated with high crack propagation rate and a disordered sinusoidal type break associated with a low propagation rate.

3.1.9.2 Cantilever Beam Shear

Lenoe[54] applied the techniques developed by Corten[108] and Wu[109] to investigate crack growth in cantilever beam specimens. The technique involves introduction of high shear stresses around a machined notch and observation of crack growth under various load levels. The apparatus is shown in Figure 3-38.

The available graphite epoxy material (Thornel 50-epoxy 0° and 90° fiber orientations) dimensions did not permit observations of crack growth. Stress levels were too high and the fixture alignment was too crude. It was indicated that successful use of the techniques require thicker composite specimens and modification of the apparatus.

3.1.9.3 Slotted Tube Test

Lenoe[56] also investigated the use of circumferentially wrapped thin walled tubes slotted with an elliptical crack parallel to the fibers. Torsion tests were conducted and attempts were made to initiate crack growth; however, inter-laminar failure was generated away from the stress riser. Failure stress indicated defective material.

3.1.9.4 Single Fiber Technique (Photoelastic)

This technique employed a single, axially aligned fiber specimen in a plastic matrix as shown in Figure 3-39, and involves attempted introduction of radial tensile failure by means of Poisson's ratio expansion of the plastic matrix under compressive load[56]. Calculations employed were based on an extremely simplified elastic analysis given by the equation[56]:

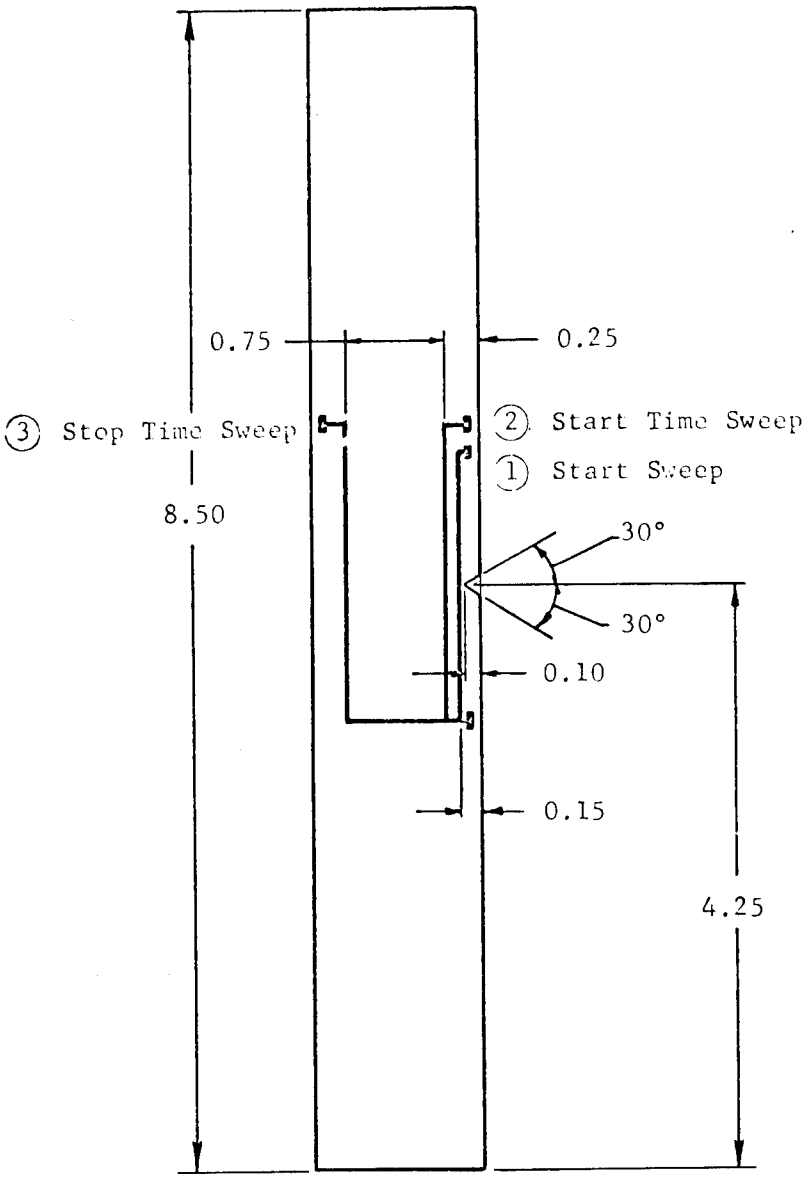


FIGURE 3-37. CRACK PROPAGATION SPECIMEN[107]

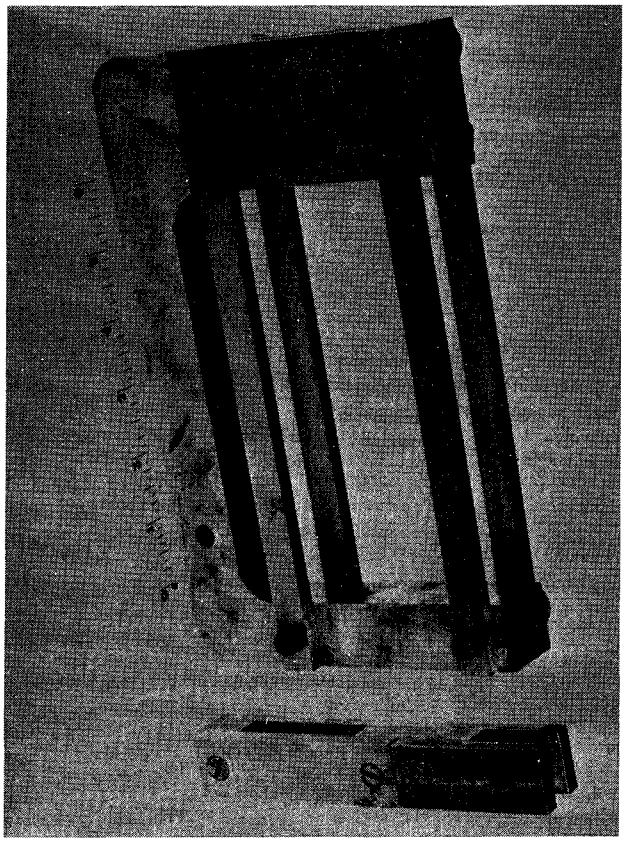


FIGURE 3-38. CRACK PROPAGATION TEST FIXTURE[56]

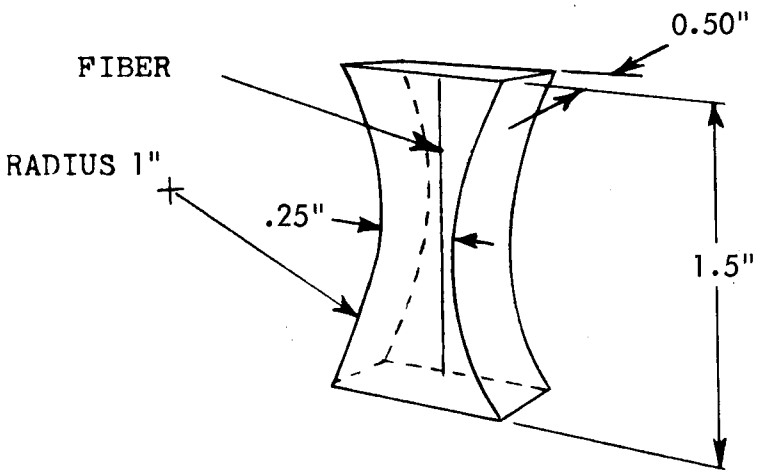


FIGURE 3-39. SINGLE FIBER TEST SPECIMEN, RADIAL TENSILE STRENGTH DETERMINATION[56]

$$\sigma r = \sigma z \frac{(V_R - V_F)\ E_F}{(1 + V_R)\ E_F + (1 - V_F - 2V_F^2)\ E_R}$$

where σ_r = Radial Tensile Stress at the Interface

σ_z = Maximum Axial Compressive Stress in Resin

V_R, V_F = Poisson's Ratio, Resin and Fiber

E_R, E_F = Yount's Modulus of Resin and Fiber

Centrally located single boron filament and Thornel 50 yarn specimens were fabricated to determine the effects of various stress risers at the fiber resin interface on failure. Each specimen was observed under polarized light while incremental increases in compressive load were applied, and the specimen observed for interface failure. Interface failures were observed; however, failure did not occur at projected stress riser sites, for example, at an air void. The apparent failure of this approach was related to the following possible factors:

1. Specimen geometry may introduce stress states significantly different from the assumed simple elastic mode.
2. The modulus and Poisson's ratio of the yarn fiber (Thornel 50) were variable and that failure would therefore occur at some other critical site rather than at the assumed stress concentration.

3.1.9.5 Pulsating Tensile Stress[104]

This method defines two procedures for study of crack propagation characteristics in boron-epoxy composites under axial loading of IITRI-type tensile coupon. Each procedure requires application of a pulsating stress on notched specimens. The stress is a prescribed percentage of the ultimate static strength. The stress ratio (R) in both test procedures is equal to zero, and if a crack fails to initiate at the prescribed stress level within a given number of cycles, the maximum stress is raised incrementally, and testing repeated until a crack is observed or failure occurs.

Specimen configuration is detailed in Figure 3-40. A precision, sharp cornered notch is machined at the geometric center of the rectangular cross-sectioned specimen. Table 3-7 summarizes suggested tab materials for use at various temperature ranges.

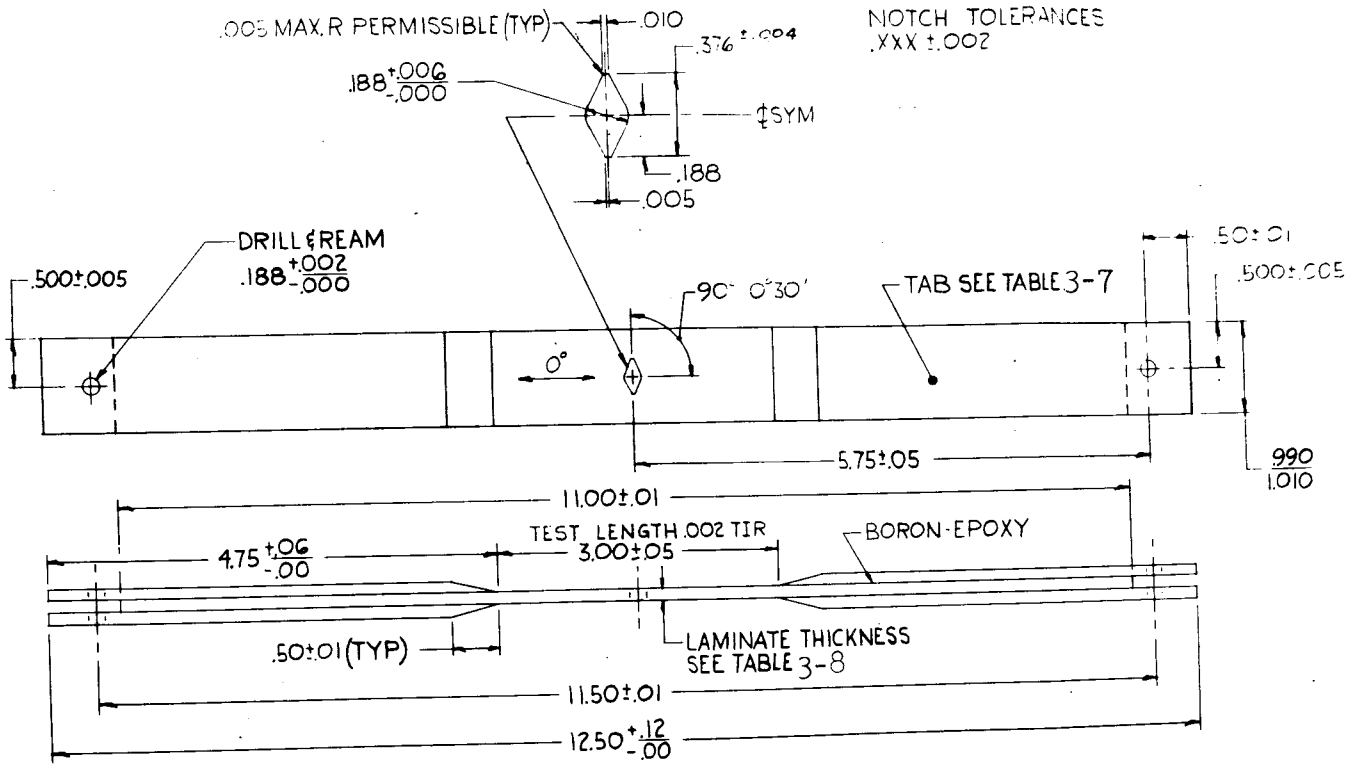


FIGURE 3-40. CRACK PROPAGATION SPECIMEN WITH SHARP CORNERED NOTCH[104]

TABLE 3-7 - TAB DESCRIPTION[104]

Test Temp. Range, °F	Tab: No. of Plies	Tab: Ply Orientation	Tab: Material	Tab: Cure Cycle	Adhesive: Material	Adhesive: Cure Cycle
RT to 375	8(.080 in.)	0,90,0,90,0, 90,0,90	Conolon 506/ Style 181	1 hr/350°F/15 psi P.C. 4 hr/375°F	Metlbond 329, Type Ia	1 hr/350°F/25-45 psi
-67 to RT	8(.080 in.)	0,90,0,90,0, 90,0,90	Scotchply 1002	GSS 11102	FM 1000	1 hr/350°F/25-45 psi Predry 150°F/1 hr.

TABLE 3-8 - PLY DESCRIPTION[104]

No. of Plies	Thickness Range Inches	Ply (Fiber) Orientation ±1/2°
6	.0294-.0330	Unidirectional 0°
9	.0441-.0495	0, 45, 135, 0, 90, 0, 135, 45, 0°
8	.0392-.0440	0, 45, 135, 90, 90, 135, 45, 0°

Table 3-8 summarizes suggested specimen thicknesses for various ply orientations. The test configuration with self-aligning fixture is shown in Figure 3-41. Tests can be conducted on an Instron Universal testing machine or equivalent testing machine of constant rate-of-crosshead-movement type, consisting of the following: a fixed member, movable member, drive mechanism, and load indicator. A low power microscope is used to observe crack initiation and growth.

Specimen width and thickness and the length of the notch is measured to the nearest 0.001 inch. The minimal cross-sectional dimensions (based upon three determinations along the length of the test gage) and length of the notch shall be recorded. The specimen is installed and centered in the test machine. A low powered microscope is focused on the notched region of the specimen to observe crack initiation and growth during the test.

A stress equal to 80% of the ultimate strength, unless otherwise specified, shall be applied and immediately released at a constant crosshead rate of 0.05 in/min. This procedure shall be repeated until failure occurs or completion of the prescribed number of cycles is achieved. If failure does not occur, the maximum stress level shall be increased by some prescribed increment and loading continued.

The second procedure is identical to the above with the exception that stress shall be held on the specimen for a prescribed period of time before being released.

The applied loads are calculated to three significant figures using the following formula:

$$P = F(b-d)t' = FA'$$

where F = Stress applied in psi
b = Specimen width in inches
d = Measured width of notch in inches
t' = Normalized or theoretical thickness
A' = Theoretical or normalized area in square inches

The problem of fracture in composites is quite complex. Although some success has been achieved in predicting strength of laboratory specimens employing crack propagation criteria[110], it is not known whether such findings can be applied to full-scale composite structures. Approaches to determination of crack propagation in resin and

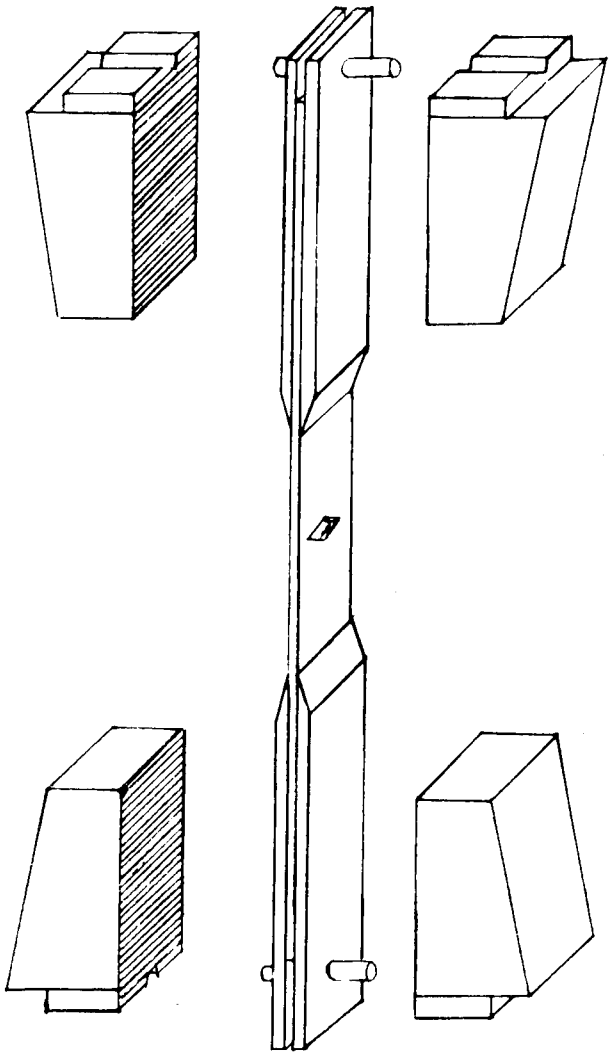


FIGURE 3-41. TEST CONFIGURATION INCLUDING SELF-ALIGNING FIXTURE[104]

composite materials treated above are representative of current efforts employed in the attempted identification of sources and mechanisms of crack initiation directed toward ultimately increasing the strengths of these materials.

3.1.10 Environmental Tests

A primary consideration in selection, design, and utilization of advanced composites for structural applications is the variety of environmental conditions to which the structure will be exposed and the influence of such conditions on its mechanical properties and structural stability. Specific environmental conditions considered detrimental to epoxy-based composites should be evaluated with particular emphasis directed toward the critical effects on the design criteria for structures as well as composite joints and attachments. The effects of certain environmental conditions, for example, temperature and humidity, are of major importance in the fabrication of high-strength composites.

Environmental tests include a broad spectrum of conditions and should be treated under separate cover. This section does not include the natural environmental tests, such as temperature, humidity, ultraviolet and salt spray, commonly conducted in a weatherometer. Test methods treated herein are confined to abrasive erosion, sonic fatigue and ballistic impact.

3.1.10.1 Abrasive Erosion[47]

This test procedure utilizes a rectangular composite test specimen 1.5 inches in length by 0.075 inch wide. The test equipment is shown in Figure 3-42. Test parameters and conditions established are listed as follows:

Abrasive Medium - Coarse Road Dust (0-200 microns).

Abrasive Concentration - 0.0185 - 0.020 Grams/ft^3 Air.

Abrasive Velocity - 0.5 Mach (Calibrated by hot wire anemometer).

Angle of Impingement - 45° to Panel Surface
90° to 0° Filament Axis
90° to Panel Surface
90° to 0° Filament Axis

The selected composite specimen is weighed on an analytical balance (0.1 mg) prior to and following each incremental time period of exposure. Specimens are cleaned before

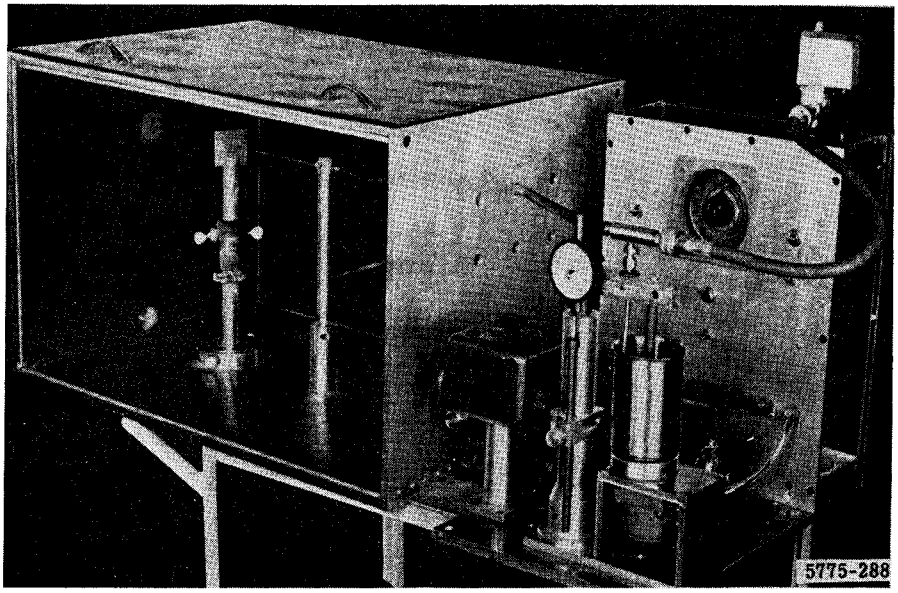


FIGURE 3-42. ABRASIVE EROSION TEST FACILITY[47]

and after each exposure by a water wash followed by methanol rinse and dried by dry air blasting.

Weight losses are converted volume losses by weight/density calculations. Boron-epoxy erosion test results are shown in Table 3-9[47]. Since the composites eroded primarily by resin loss with limited effect on the boron, volume losses cited in the table were calculated using both resin density (0.04 lb/in^3) and composite density (0.076 lb/in^3). Figure 3-43 illustrates erosion rates for boron-resin as compared with 7178 aluminum and Ti-6Al-4V.

3.1.10.2 Sonic Fatigue

Current use of advanced fiber-resin composites in aircraft structures, where high noise environment may become a significant factor in their design, has necessitated the development of reliable test methods which will determine the specific effects of exceedingly high sound pressure levels on their structural and mechanical properties. Basic sonic fatigue tolerance and design criteria must therefore be established for this category of composites.

3.1.10.2.1 Siren Test Facility

Advanced filamentary composite panels can be readily exposed to simulated high intensity sound environments to determine their sonic fatigue resistance. The most effective apparatus employed has been the AFFDL-type Small Acoustic Test Facility[111]. The test chamber illustrated in Figure 3-44 is equipped with two sirens which produce a maximum power output of 50KW over a frequency range of 50 HZ to 10KHZ in two overlapping increments. The sirens are coupled to an 800-cubic-foot termination chamber through a 16-foot catenoidal horn. Immediately down stream of the sirens is a one-foot-square test section in which sound pressure levels (SPL) up to 174 dB are attainable and in which panel specimens are installed singly for exposure to grazing incidence sound energy. Specimen panels are installed in the test fixture with the 0° fiber reference axis oriented vertically. A typical test panel specimen is shown in Figure 3-45. Sound pressure level is monitored by a microphone positioned to the rear of the panel specimen. A pulsed frequency tracking signal from the siren is provided for use as a frequency reference during response amplitude analysis. Thermocouples are used to monitor siren air temperature and panel surface temperature.

TABLE 3-9 - EROSION TESTING - RECORD OF VOLUME LOSS[47]

Specimen Identification	Cumulative Volume Loss (10^{-6} $in.^3$)									
	Weight Loss/Composite Density (Minutes)					Weight Loss/Matrix Density (Minutes)				
	15	30	45	60	120	15	30	45	60	120
Al 7178 T6	34.588	77.823	131.86	181.59	421.55	—	—	—	—	—
Al 7178 T6	—	—	—	194.59	391.27	—	—	—	—	—
Ti(6Al-4V) Annealed	22.05	57.881	95.087	132.30	283.90	—	—	—	—	—
Ti(6Al-4V) Annealed	—	—	—	117.13	—	—	—	—	—	—
B-resin III X	60.921	87.039	101.55	121.86	168.28	115.75	165.38	192.95	231.55	319.72
B-resin III X	—	—	—	60.921	133.46	—	—	—	115.75	253.58
B-resin XVII X	29.013	46.421	52.223	60.921	92.842	55.125	88.200	99.225	115.75	176.40
B-resin XVII X	—	—	—	69.631	118.95	—	—	—	132.30	226.00
B-resin XX X	31.921	52.223	60.921	63.829	133.46	60.650	99.225	115.75	121.28	253.58
B-resin XX X	—	—	—	81.236	136.37	—	—	—	154.35	259.10

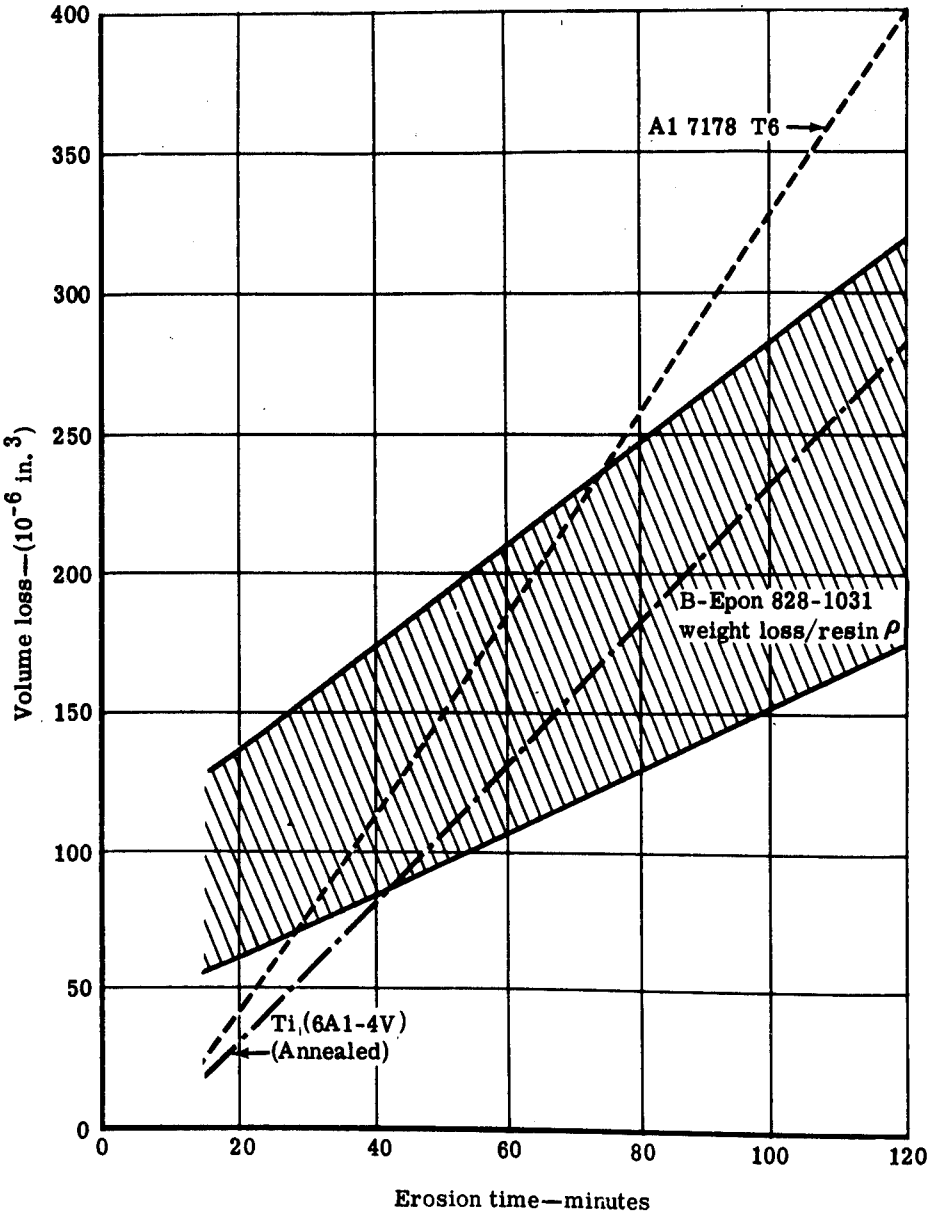


FIGURE 3-43. EROSION RATES FOR Al 7178 T6, Ti 6Al-4V AND B-RESIN COMPOSITE MATERIALS[47]

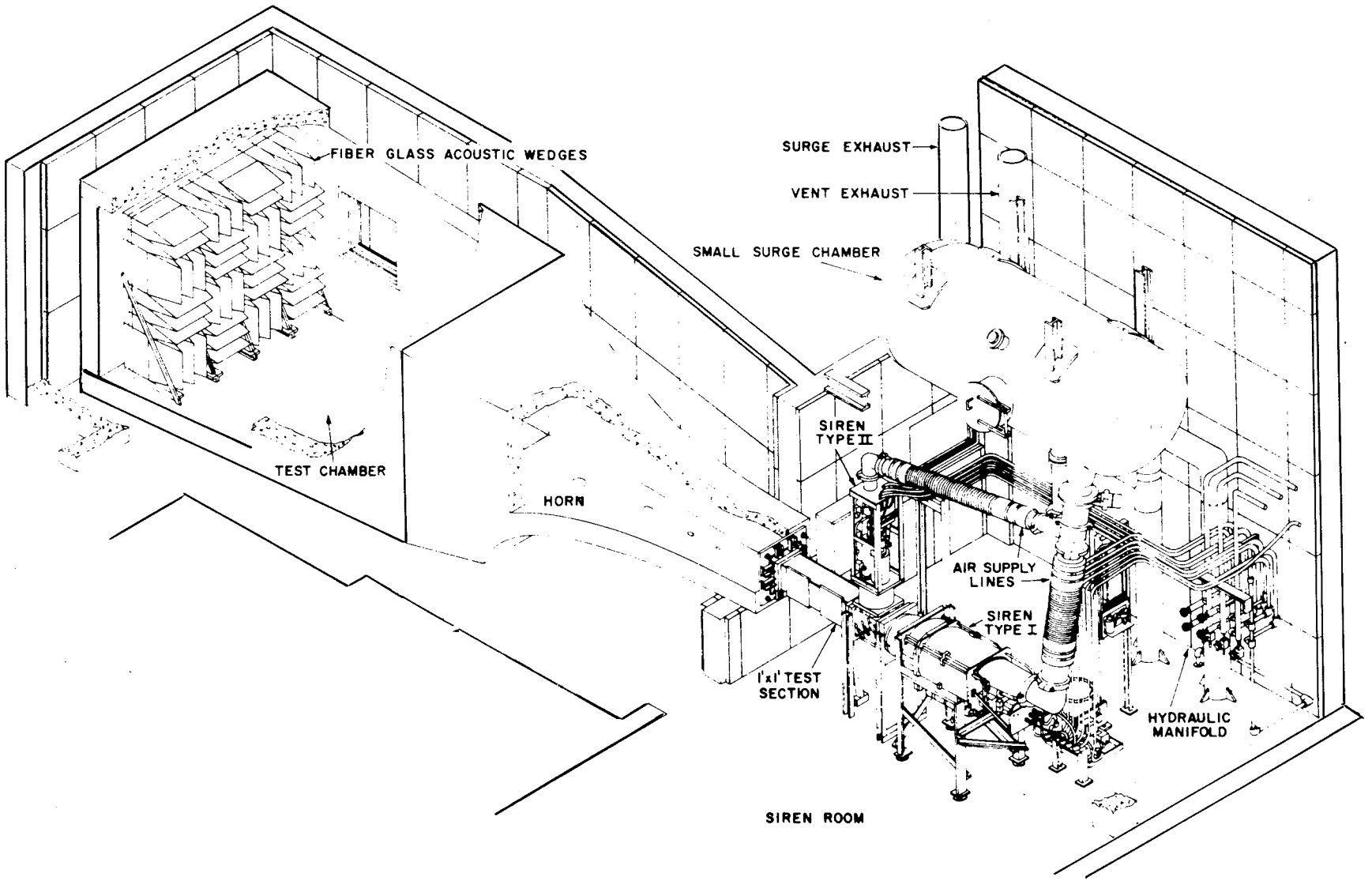


FIGURE 3-44. SMALL SCALE TEST CHAMBER SONIC FATIGUE FACILITY[111]

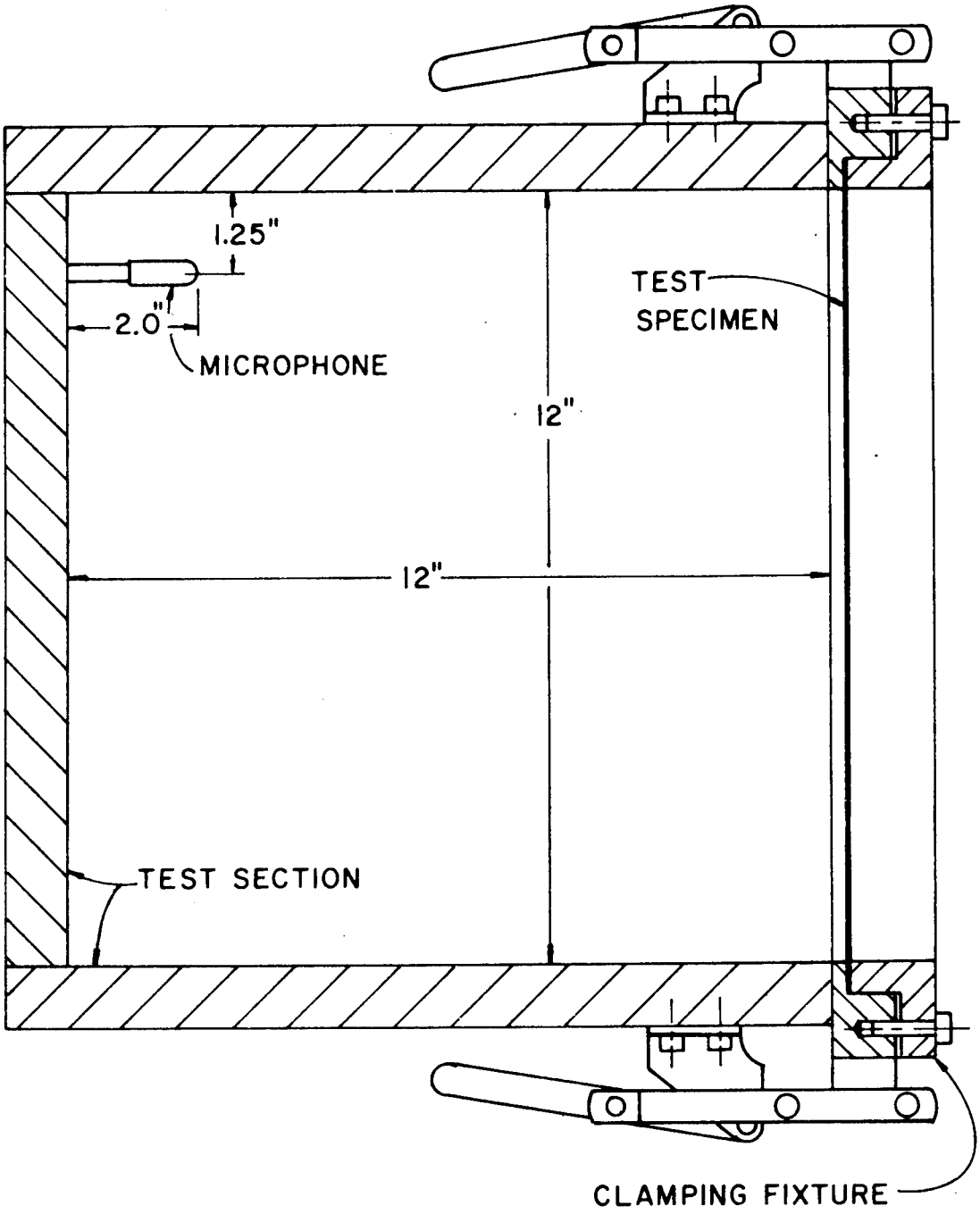


FIGURE 3-45. COMPOSITE PANEL TEST SPECIMEN INSTALLED IN ONE FOOT SQUARE TEST SECTION FOR GRAZING INCIDENCE IMPINGEMENT[111]

3.1.10.2.2 Sonic Test Procedure

3.1.10.2.2.1 Acoustic Noise Response

Response testing involves determination of frequency data required to ascertain the linearity of strain for increases in loading and to determine resonant frequencies for endurance tests. Response data is obtained at sound pressure levels, for each panel configuration listed in Table 3-10, at 140 dB through 158 dB in 6 dB increments by sweeping the siren frequency from 70 hz to 550 hz while holding the SPL constant. Each sweep run is monitored by a recording oscillograph in addition to storing the strain gage and microphone outputs and the tracking signal on magnetic tape for later analysis. Strain gage instrumentation of panel specimens is illustrated in Figure 3-46 for each panel configuration treated.

On the basis of strain levels observed during response testing of boron-epoxy panels, Rupert[111] estimated that a loading of 160 dB would be sufficient to induce failure without accumulating a large number of test hours. Upon establishing the sound pressure level at 160 dB, the region of maximum amplitude of the response curve was examined to avoid tuning-in the specimen too fine. If the resonant frequency decreased by 10%, the specimen was examined for possible failure. Visible failures varied from severe damage to nearly nondetectable ply separation. Detection of broken fibers by microscopic examination of an exposed panel was technically classified as a failure. Time-to-failure is not an exact determination. Although less meaningful, time-to-failure detection was proposed for use. The response of various panel configurations to acoustic excitement is shown in Figure 3-47.

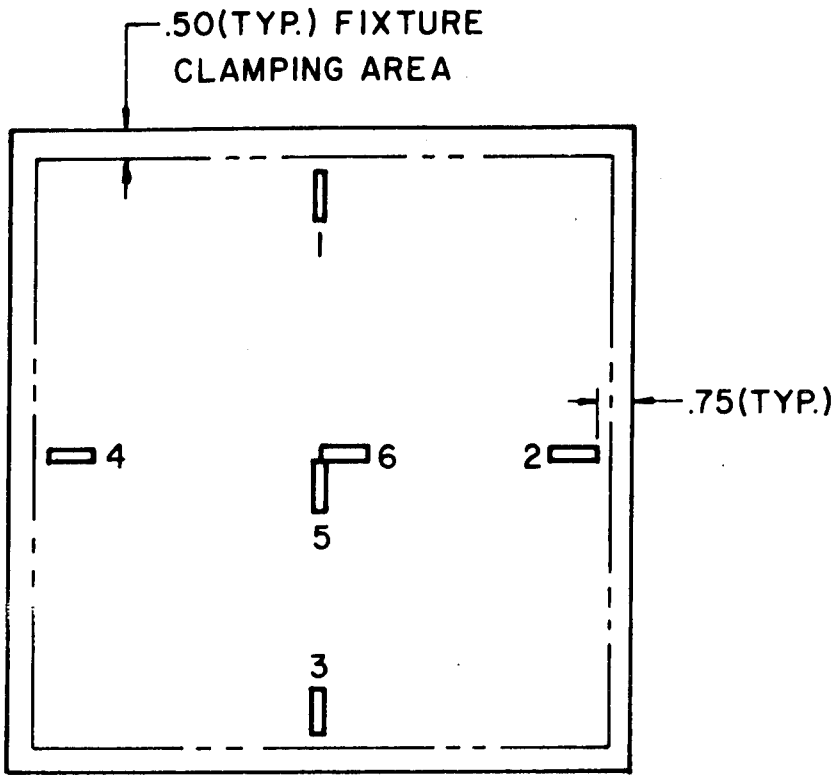
3.1.10.2.2.2 Sonic Fatigue Life

Composite panel specimens are exposed to sound pressure levels of greatest response. An accumulation of this specified test parameter is continued until failure is detected in the panel. An additional procedure involves exposure of the test panel to a specified sound pressure level at its frequency of highest response in increments of time until failure occurs.

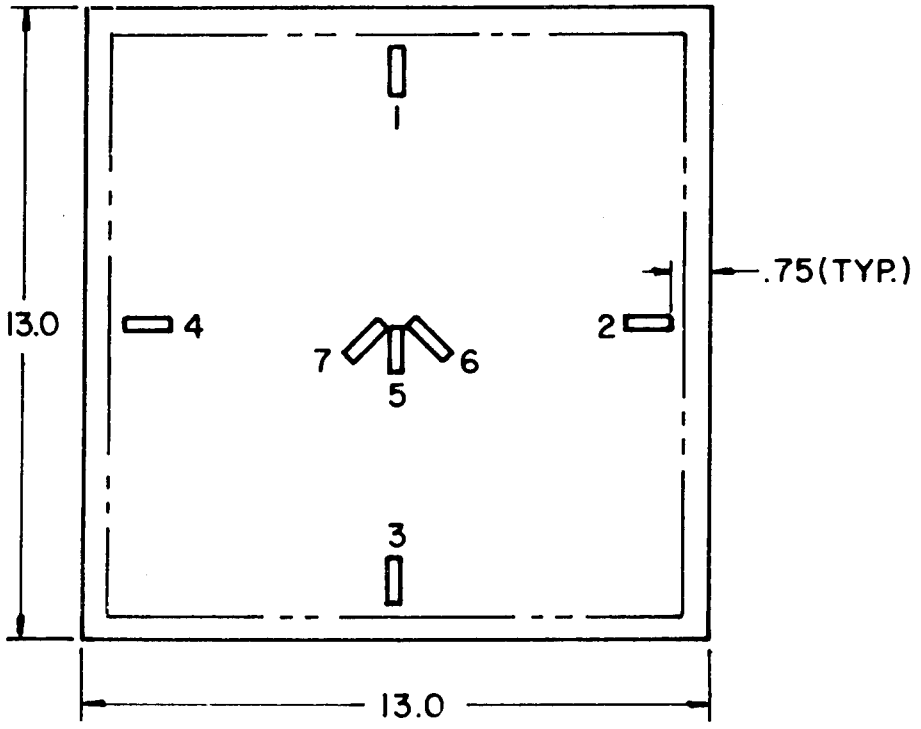
Shockey, et al., submitted boron-epoxy panels for testing in the described sonic test facility[112]. The following conclusions were described from their test results.

TABLE 3-10 - SPECIMEN PANEL CONFIGURATION[112]

Panel	Fiber Orientation
A	0°/ 90°/ 0°/ 90°/ 90°/ 0°/ 90°/ 0°
B	ALL 0°
C	45°/ -45°/ 45°/ -45°/ -45°/ 45°/ -45°/ 45°
D	0°/ -45°/ +45°/ 90°/ 90°/ +45°/ -45°/ 0°
E	+22.5°/ -22.5°/ +67.5°/ -67.5°/ -67.5°/ +67.5°/ -22.5°/ +22.5°
F	ALL 45°



(a) PANEL CONFIGURATIONS A AND B



(b) PANEL CONFIGURATIONS C,D,E AND F

FIGURE 3-46. STRAIN GAGE INSTRUMENTATION OF PANEL SPECIMENS[111]

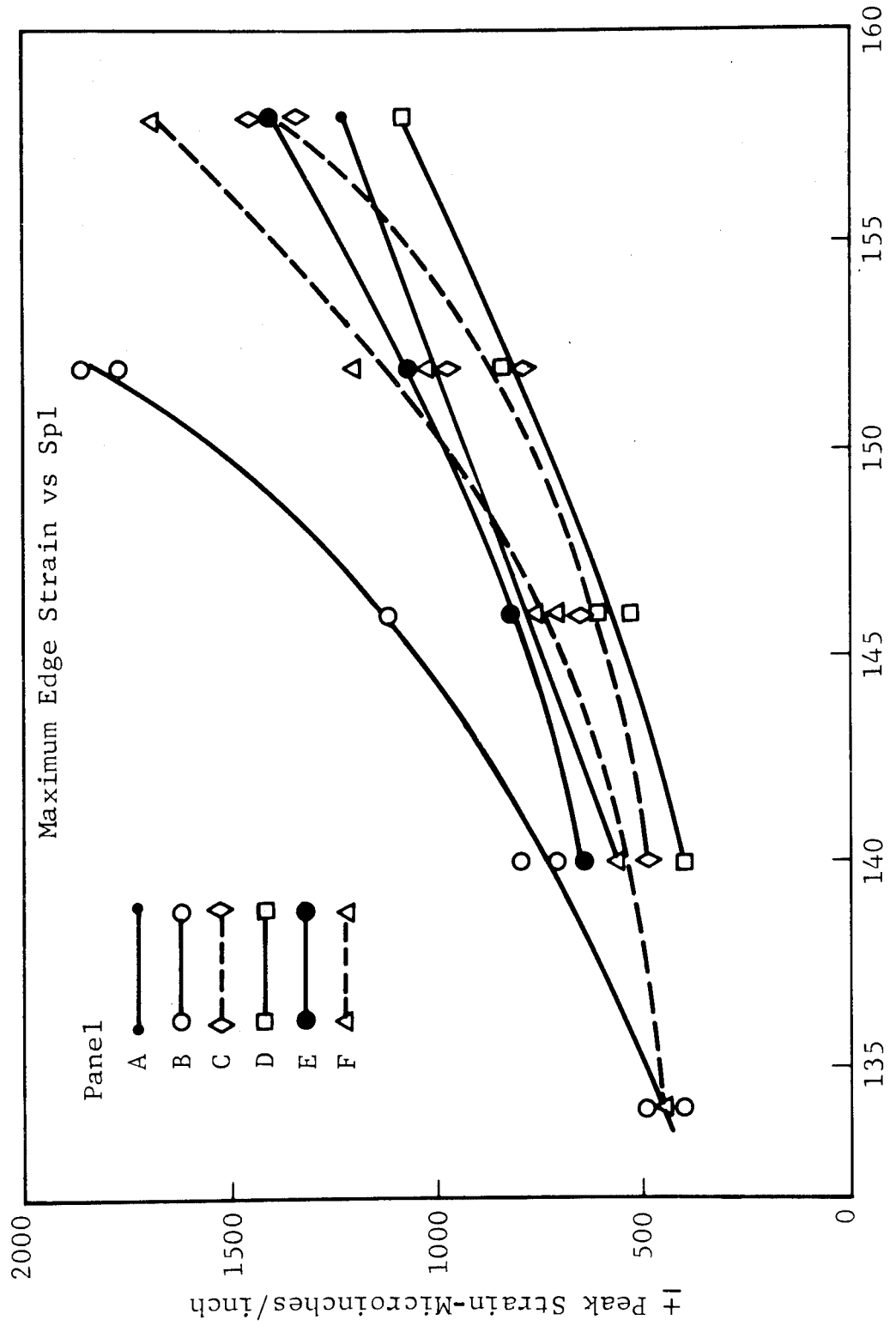


FIGURE 3-47. RESPONSE COMPARISON OF VARIOUS PANEL CONFIGURATIONS TO ACOUSTIC EXCITATION[112]

1. Panels with lamina in only one direction demonstrated, as expected, no significant resistance to sonic fatigue.
2. Edge delamination which occurs before cracks appear effectively unloads the highest stresses and prolongs the time to failure.
3. The greatest resistance to sonic fatigue was exhibited by the D panels which had lamina in four directions with the outer and central lamina in the direction of the x and y axes of the panel.
4. Orienting the lamina so that no fibers in the direction of the x and y axes of the panel shortened the panel fatigue life. (Compare E panels with D panels.)

A large variety of sonic test chambers and corresponding composite test specimen dimensions and configurations are currently in use. The AFFDL Small Acoustic Test Facility was selected for defining the above treated sonic test procedures because of its high power output (50 kw), broad frequency range - 50 hz to 10 khz, and high SPL (sound pressure levels) up to 174 dB for a one-square-foot test section.

3.1.10.3 Ballistic Impact

In the ballistic impact test employing the Crossman 0.177 cal. rifle[47], the suggested dimensions for the rectangular composite specimens are as follows:

Resin matrix - 4 in. x 1.0 in. x t
Metal matrix - 2.6 in. x 1.25 in. x t

where: t = 0.05 in. for coreless panels
t = 0.10 in. for cored panels

The test procedure involves use of a Crossman 0.177 cal. gas-operated rifle modified to permit controlled pressurization. The rifle is used to impact a 0.36-gram copper coated steel pellet against the specimen which is rigidly held at each end. Impact is made at 90° to the panel surface. The test apparatus is shown in Figure 3-48. Figure 3-49 shows the calibration. Projectile velocity is measured with an Avtron Model T-333 Counter Chronograph which records time, in milliseconds, required for a projectile to pass between two screens positioned at a known distance apart along the flight path.

Upon calibration of the test projectile velocity and corresponding energy the type and extent of damage are recorded. Description of specimen damage should be supplemented by photographs. The measure of resistance of materials to ballistic impact can be made either by relative comparisons of damage on different materials or the effect of impact on some specific mechanical property, for example, dynamic modulus, of that particular material.

In the ballistic impact test, a specific specimen configuration is not required. The composite specimen can conform to any configuration which may become useful in attaining more detailed evaluation of the effect of ballistic impact on the material.

August and Lubin[113] conducted ballistic impacts on boron-epoxy laminates to determine extent of entry and exit damage using 30 cal. and 50 cal. AP projectiles at 0° obliquity and at muzzle velocities ranging from 1249 to 1616 ft/sec. Panel fiber orientations were 0°/90°/±45° and 0°/90°. An additional direct approach to testing and assessment of ballistic impact damage and the effect of holes involved impacting of stressed and unstressed boron-epoxy panels with 30 and 50 caliber projectiles[114]. Panels of a size sufficient to yield reasonable adsorption energy characteristics were designed for tension loading. Both plain and laminate honeycomb sandwich constructions are used.

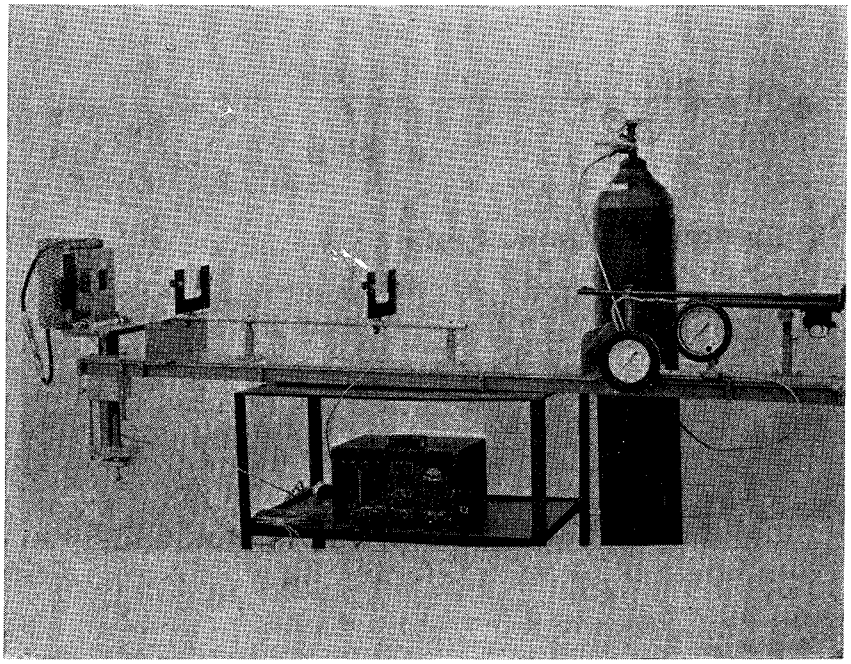


FIGURE 3-48. BALLISTIC TESTING APPARATUS[47]

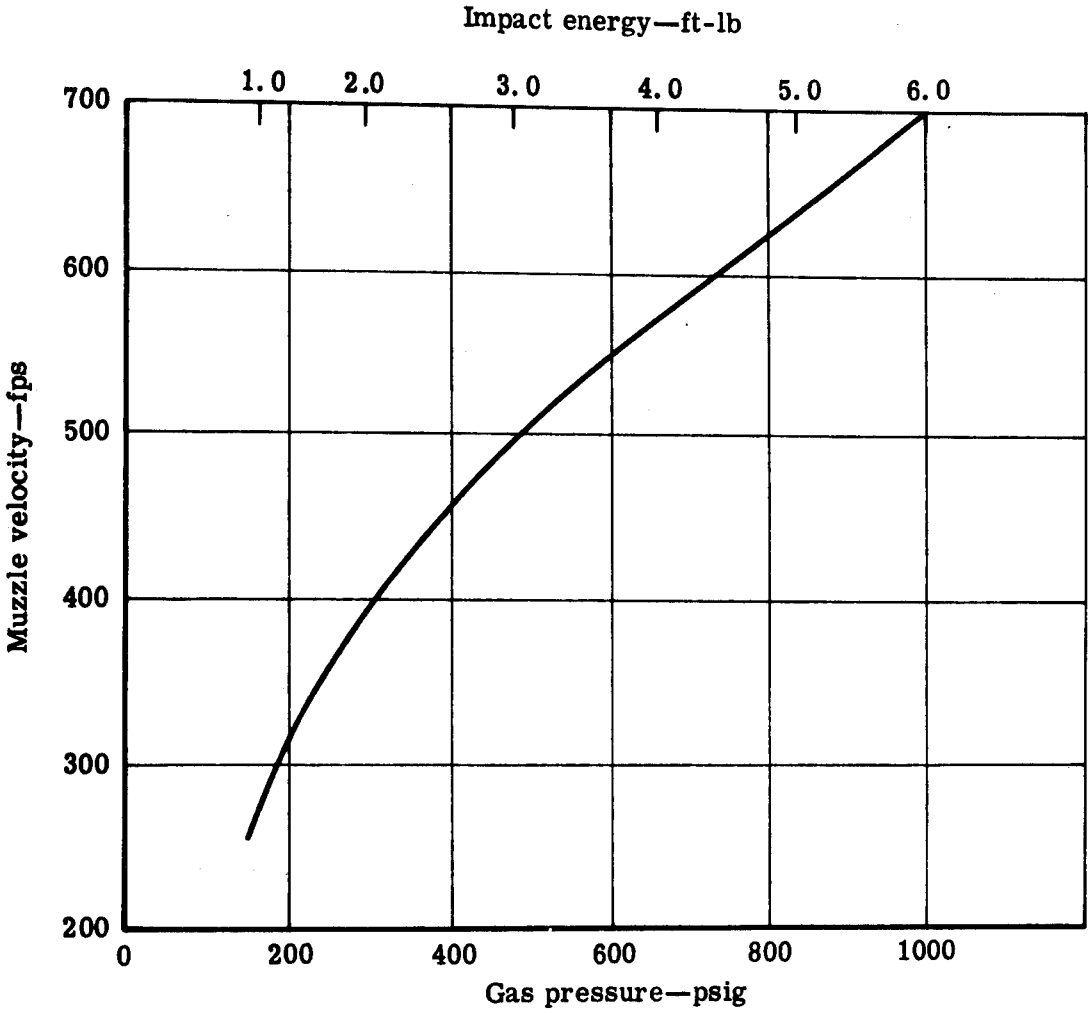


FIGURE 3-49. CALIBRATION CURVE FOR BALLISTIC IMPACT RIG. CROSSMAN 160 PELLGUN, 0.36 GRAM SPHERICAL STEEL BALL[46]

4.0 STRUCTURAL ELEMENT TESTS

4.1 GENERAL

The principal objective of structural element tests is the development of basic engineering data and the establishment of reliable analytical and practical design techniques for composite structures. Such a test program is quite complex and is devisable into several significant phases. It is not within the scope of this report to treat in detail structural element testing programs for any specific composite configuration or structure. A general treatment of such tests is appropriately confined to structural design guides or manuals. This section will review, however, tests for basic composite elements from which primary aircraft structures, for example, wing, fuselage, and tail assemblies, are designed and fabricated.

4.2 BASIC ELEMENTS

Basic structural elements and an established testing program have been detailed by Lackman[115]. Composite element and panel configurations included flat and curved laminates, crippling element (channel zee and hat types), and panel cutouts. A testing program for flat sheet or laminates was designed to evaluate fully uniaxial and biaxial compression, in-plane shear, and combined loadings. Additional tests; namely, pressure panel and thermal gradient, both singly and combined, as well as creep buckling for two edge conditions were included.

Suggested curved panel tests included uniaxial compression, shear, combined shear/compression, pressure and compression/pressure, and pressure and shear. Crippling element testing is confined to evaluation of structural behavior in the critical postbuckling range. Tests in the basic element program should be conducted at both room temperature and an assigned elevated temperature commensurate with the anticipated structural environment. Creep buckling tests are conducted at elevated temperature only, while fatigue tests are conducted at ambient temperatures. All specimens should be instrumented, as required, with three 6G strain gage rosettes suitable for use on composite materials at the assigned test temperature.

In the basic structural elements treated above, unstiffened panel tests and the skins of skin-stringer panels should consist of approximately 16-ply material or approach a thickness of 0.080 inch. All other tests should be conducted using approximately 8-ply

(0.040 inch) thick composite. Variation of panel aspect ratio (width/thickness) and radius of curvature provides various width/thickness and radius/thickness ratios for use in checking required analytical prediction methods.

Tests for cutouts in panels should include tension, compression, and shear. A detailed analysis of each such test should be conducted to provide an experimental basis for design of required holes for service lines and access in aircraft structures. Additional tests should include cyclic testing of pressure panels, both with and without in-plane compressive loading to generate data to check analytical fatigue life prediction techniques. One of the two panels for each data point should further be cycled under a constant amplitude loading, while the remaining panel is subjected to a spectrum loading which will simulate service conditions.

During the course of the basic element tests, Lackman suggests that tension coupon tests be conducted on source composite laminate materials to monitor the properties and relate these to corresponding panel test results. This procedure should provide a more reliable comparison of theory to experimental results.

Each of the basic shape unstiffened and honeycomb sandwich type panels should be subjected to nondestructive forced vibration tests to determine their natural frequency under simply supported end conditions.

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