ELASTIC PROPERTIES AND FRACTURE BEHAVIOR OF GRAPHITE/POLYIMIDE COMPOSITES AT EXTREME TEMPERATURES

by

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TABLE OF CONTENTS

Acknowledgements	ii
List of Tables	iv
List of Figures	v
Introduction	1
Literature Review	4
Experimental Procedure	10
Results and Discussion	17
Summary and Conclusions	48
References	51
Vita	56
Abstract	

LIST OF TABLES

Table		Page
1	Lamina Material Properties	18
2	Laminate Material Properties	19
3	Notched Tension Test Results	
a	$[0/45/90/-45]_S$	21
b	$[45/0/-45/0]_S$	22
c	$[\pm 45]_{2S}$	23
d	$[0/45/90/-45]_{2S}$	24

LIST OF FIGURES

Figure		Page
1.	Axial Stiffness versus Temperature for Fiber-Dominated Laminates.	25
2.	Tensile Strength versus Temperature for Fiber-Dominated Laminates.	26
3.	Axial Stiffness versus Temperature for Matrix-Dominated Laminates.	27
4.	Tensile Strength versus Temperature for Matrix-Dominated Laminates.	28
5.	Poisson's Ratio versus Temperature.	30
6.	Notched Strength versus Temperature for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).	31
7.	Notched Strength versus Temperature for the $[45/0/-45/0]_S$ Laminates, Width = 64 mm (2.5 in.).	33
8.	Notched Strength versus Temperature for the $[\pm45]_{2S}$ Laminates, Width = 102 mm (4.0 in.).	35
9.	Notched Strength versus Width at Room Temperature for a Fixed Flaw Size of 4.8 mm (3/16 in.).	36
10.	Comparison of Experimental and Theoretical Failure Strengths for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).	38
11.	Comparison of Experimental and Theoretical Failure Strengths for the $[45/0-45/0]_S$ Laminates, Width = 64 mm (2.5 in.).	39
12.	Comparison of Experimental and Theoretical Failure Strengths for the $[\pm45]_{2S}$ Laminates, Width = 102 mm (4.0 in.).	42
13.	Characteristic Distance versus Temperature for the $[45/0/-45/0]_S$ and $[0/45/90/-45]_{2S}$ Laminates.	43

LIST OF FIGURES (CONCLUDED)

Figure		Page
14.	Characteristic Distance versus Temperature for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).	44
15.	Characteristic Distance versus Temperature for the Fiber-Dominated Laminates.	45

INTRODUCTION

In recent years the use of advanced fiber reinforced composite materials has expanded dramatically. The reasons are manifold, but the greatest advantage of composites over typical macroscopically homogeneous isotropic engineering materials is their "tailorability." The ability to select fiber and matrix materials nearly independently of each other and stack the resultant laminae in virtually any order has resulted in an almost unmanageable melange of new materials, each possessing unique properties. Because of the heterogeneity and anisotropy of the resultant materials, mechanical characterization techniques have not kept up with the rapid development pace.

The demand for advanced composite materials has grown most rapidly in areas in which the need for high specific strength and specific stiffness outweigh the high cost of composites relative to other structural materials: high performance aircraft, space structures, and sports equipment. Because of the extreme demands placed on the materials used and the high cost of structural failure, material characterization must cover an extensive list of properties: notched and unnotched strength, elastic and plastic properties, fatigue response, thermal, acoustical, and electrical conductivity, corrosion and wear resistance, impact resistance, and the effect of environmentally induced damage on these properties.

Graphite/polyimide is a composite material being developed to extend the useful temperature range of composite materials for such applications as jet engines, supersonic cruise aircraft, and space shuttle structures. Toward this end, the Composites for Advanced Space Transportation Systems (CASTS) program at the National Aeronautics and Space Administration (NASA) Langley Research Center was established to develop a graphite/polyimide composite that could be used for structural applications in the space shuttle environment. All structures, whether they are in an air or space environment, must be fastened to other structures. Since this fastening often results in holes being introduced into the structure, the so-called notch behavior of the basic material must be understood. The primary objective of this work was to develop an understanding of the notch behavior of a graphite/polyimide composite over the temperature range that would be experienced by part of the space shuttle structure.

In order to characterize the notch behavior of the graphite/polyimide material studied in this program, both unnotched and notched tension tests were conducted. The data reported in this thesis are the results of the first phase of a two-phase program. The first phase covers tests on unnotched specimens and notched specimens that are 102 mm (4 in.) wide or smaller. The second phase, to be reported at a later date, involves testing of notched specimens of eight and twelve inch widths. The notched fracture data are correlated through the use of two similar failure theories which take material orthotropy, flaw type, flaw size, and specimen width into account. Tests of unnotched laminates corresponding to the notched laminates were conducted in order to provide unnotched strength and elastic constants necessary for use in the failure models.

LITERATURE REVIEW

Because of the extensive research conducted on composites in a variety of areas, the review of available literature was focused on two areas that relate to the work performed for this project. The first area involves mechanical testing at extreme temperatures of polyimide matrix composites in general and PMR-15 in particular. The second area covers the models developed to predict failure of notched composite materials. While an exhaustive review of even these limited areas is hardly possible, the research surveyed here can be considered to be reasonably representative.

Mechanical Testing

Because of the relative newness of polyimide matrix systems, it is not surprising that the majority of research reported at the time this literature search was conducted involved material development, process development, and screening based on simple mechanical testing commonly involving flexural and short-beam-(interlaminar)-shear tests. The suitability of polyimides for high temperature use has been established by Jones [1] and Serafini, et.al. [2]. The chemical structure and the details of the chemical reaction by the in situ polymerization of monomer reactants (PMR) are elucidated by Serafini and Delvig [3] and the further development of PMR polyimides for high temperature use has been reported by Vannucci, et. al. [4] and Serafini, et.al [5]. The flexural strength of HTS/PMR versus formulated molecular weight at various temperatures has been determined by Winters and Serafini [6].

The results of studies on graphite/epoxy and graphite/polyimide process development in which the effects of different curing techniques on the mechanical properties of several material systems were evaluated have been reported by Scheck [7-9] and Maximovich [10]. These reports contain flexural properties as well as creep, fatigue and aging data and Scheck [9] reached the conclusion that graphite/polyimide composite structures can be as reliably designed and analyzed as are graphite/epoxy structures. Varlas [11] reported that graphite/polyimide is competitive with graphite/epoxy on the basis of specific tensile strength and interlaminar shear strength while Jones [12] concluded that processing void free polyimide laminates is comparable in simplicity and reliability to graphite/epoxy. Less encouraging is data presented by Vaughan, et. al [13] that appears to indicate that a polyimide matrix yields a lower fracture toughness than an epoxy matrix.

In addition to development and screening studies, there have been several papers [14-16] which present compilations of current research into epoxy and polyimide matrix composites. The consensus of opinion in these documents appears to be that polyimide matrix composites can be safely, easily, and reproducibly processed into void free composites which are comparable to epoxies at room temperature but superior at elevated temperatures. While the material cost of polyimides is similar to that of epoxies, the sophisticated bagging and high temperature tooling and autoclaving required for polyimide curing push the total cost up.

A number of researchers have addressed the problem of strength retention in polyimide matrix composites after various exposure times at elevated temperatures. Early work by Hanson and Serafini [17-18], Browning and Marshall [19], and Cavano, et. al. [20] indicates that polyimide based composites exhibit superior strength retention after long term exposure and virtually no short term strength loss. Other researchers [21-24] have confirmed that polyimides retain a significant fraction of elevated temperature strength even after long duration exposure at the high temperature.

Despite extensive research on polyimide matrix composite materials, little has been reported on the properties of PMR-15 polyimides in general or C-6000/PMR-15 in particular. Of the available data on PMR-15 based composites, the majority [6,14,16,25] takes the form of flexural strength, flexural modulus, and interlaminar shear strength values used to screen large numbers of candidate material systems. Tensile strength of HTS/PMR-15 is reported by Campbell and Burleigh [26] along with coefficients of thermal expansion and thermal conductivity but no information on notched fracture is given. The fracture behavior of notched C-6000/PMR-15 was investigated by Awerbuch, et. al. [27] but only crack-like slits were examined and only room and elevated temperature tests were conducted.

Failure Models

The first expression of a workable quantitative theory for predicting the fracture of cracked materials was presented by Griffith [28] in 1920. A brief history of the further development of what came to be known as the Griffith-Irwin theory of fracture mechanics was presented by Weiss and Yukawa [29] so the details will not be given here. The field of classical fracture mechanics, however, was developed to handle the fracture of homogeneous isotropic materials. It is not surprising that early failure models proposed for use with composite materials rely on isotropic failure concepts with appropriate changes made to accommodate anisotropic stress fields. Sih, Paris, and Irwin [30], Beaumont and Phillips [31], Phillips and Tetelman [32], Cruse [33], and Phillips [34] have presented work which extends the use of classical fracture mechanics to include anisotropic composite materials. Solutions by Ang and Williams [35], Savin [36], and Lekhnitskii [37] to the problem of the stress fields in anisotropic plates containing crack-like slits or holes were used by these researchers to modify classical theory. In a similar manner, Sih and Chen [38] proposed that a strain energy density criterion be used to predict composite failure. Although these theories account for material anisotropy, they do not account for material inhomogeneity and the propensity for composites to crack in a non-self-similar manner.

While so-called "shear lag" models have been proposed which separately account for fiber and matrix properties and their interaction [39-40], they are sufficiently complicated to daunt all but the most persistent. Another class of models which comprise an empirical fracture mechanics approach finds its simplest expression in the work of Waddoups, et. al. [41] in which the size of a region of intense energy is experimentally determined and used to predict failure using linear elastic fracture mechanics. Whitney and Nuismer [42-43] proposed a pair of fracture criteria which are empirical in nature but do not rely on classical fracture mechanics. The point stress and average stress criteria can both be used for composites with either a crack-like slit or a hole. The characteristic distances appearing in the models are assumed to be material properties. The popularity of these models is evidenced by the number of researchers using them [44-50]. Pipes et. al [51] proposed a modification involving an additional parameter, but the determination of the exponential parameter is dependent on an apparently subjective choice of the reference notch radius.

One apparent weakness of the point stress and average stress criteria of Whitney and Nuismer is related to the existence of out of plane stresses at the free edges of composites. Whitney and Kim [49] report experimental results which indicate that stacking sequence affects the unnotched strength but not the notched strength of composites. Since determination of the characteristic distance depends on normalization of the notched strength by the unnotched strength, different characteristic distances will be calculated for different laminates which possess the same elastic constants and differ only in stacking sequence. Poe [52-53] has proposed a fracture model which, while similar in some ways to the point stress criterion, does not contain the aforementioned ambiguity. This model has as yet only been developed to handle crack-like slits.

EXPERIMENTAL PROCEDURE

Materials and Specimens

The material used for this study consisted of Celion-6000 fibers embedded in a matrix of PMR-15 polyimide. Specimens were cut from panels of $[0]_8$, $[90]_8$, $[\pm 45]_{2S}$, $[\pm 45]_{4S}$, $[0/45/90-45]_{2S}$, $[0/45/90/-45]_S$, $[45/0/-45/0]_S$, and $[45/90/-45/90]_S$ laminates which were manufactured in 1978. Each panel was C-scanned to ensure that specimens were cut from void free sections of the panel. In some cases, because processing difficulties had not yet been completely eliminated, only one specimen could be obtained from an entire panel. The first four of these laminates were tested without notches in order to determine lamina elastic constants. The remaining four were tested without notches to get laminate elastic constants, and with circular holes of various sizes to determine their fracture behavior. Ply thicknesses in the cured laminates ranged between 0.127 mm (0.005 in.) and 0.152 mm (0.006 in.). While the fiber volume fraction records supplied by the manufacturer were incomplete, those values available varied from 54 to 67 percent.

Unnotched tension specimens were 25.4 mm (1 in.) wide by 406 mm (16 in.) long. Fracture specimens were 406 mm (16 in.) long with widths ranging from 19 mm (0.75 in.) to 102 mm (4 in.). Fracture specimens had center circular holes that ranged between 1.6 mm (1/16 in.) and 25.4 mm (1 in.) in diameter.

Test Procedures

Load Application

All specimens were tested in an Instron load frame in which one grip was attached through a universal joint to an immovable fixture containing a load cell while the other grip was rigidly attached to a movable cross-head. The universal joint permitted a slight degree of self-alignment and ensured that the load cell was only axially loaded. The load cell was calibrated using a system of shunt resistors contained in the test machine controller. The shunt calibration was checked against dead weights at the lower load ranges and found to be within 1 percent of actual load. All unnotched tension tests were conducted at a cross-head displacement rate of 0.0008 mm/s (0.002 in./min.). All notched fracture tests were conducted at 0.002 mm/s (0.005 in./min.).

Two different types of grips were used in this program: wedge-grips and U-grips. In order to isolate the specimen from the deeply serrated wedge faces while still providing the friction necessary to cause the wedges to tighten with increasing load, sandpaper was inserted between the wedges and the specimen. Silicon carbide, 320 grit sand-paper was placed on each side of the specimen with the grit side against the specimen. Silicon carbide, 180 grit sandpaper was inserted on each side between the 320 grit paper and the wedges with the paper side against the wedges. All specimens 25.4 mm (1.0 in.) wide or narrower were tested in 25.4 mm (1.0 in.) wedge grips. All specimens 50.8 mm (2.0 in.) wide were tested in 50.8 mm (2.0 in.) wedge grips. No specimens tested in the wedge-grips were observed to slip or fail in a manner which was obviously grip induced.

Because there were no large width wedge-grips available, all specimens wider than 50.8 mm (2.0 in.) were tested in U-grips originally designed at NASA-Langley. The U-grips were designed to be tightened by five bolts which passed through holes drilled in the grips. Each specimen had an identical pattern of holes machined to provide for passage of the bolts. Bakelite shims were inserted between the specimen and serrated grip faces because the specimen thickness was so much less than the opening provided by the grips. By carefully selecting shims of the proper thickness, the specimen was centered in the grip. Proper shim thickness also limited the amount of deformation of the grips caused by tightening the bolts so that the specimens were clamped uniformly over the entire grip area. The bolts were tightened to a torque of 16.6 kg-m (120 ft-lb); the maximum allowed for the strongest bolts available.

Environmental Control

Both unnotched tension tests and notched fracture tests were conducted at cryogenic, room, and elevated temperature. Room temperature tests were conducted in a constant temperature, constant humidity room. The temperature was 24±2°C (75±4°F) and the relative humidity was 50±5%. An environmental chamber was used to control the temperature for cryogenic and elevated temperature tests. The chamber was small enough to permit the specimen to be gripped outside the chamber thereby providing a benign environment for the grips and facilitating the isolation of the load cell from extreme temperatures. At no time did the load cell temperature measurably differ from ambient room temperature. A small triple-paned window in the door of the box permitted observation of the specimen.

Heat was provided by three electrical resistance elements located in a small chamber in the back of the box. Cooling was provided by liquid nitrogen contained in a self-pressurizing bottle with a constant pressure bleed-valve. A thermocouple located near the specimen in the test chamber provided the feedback signal to the temperature controller. The controller operated both the resistive heaters and a servo-valve placed in the nitrogen line between the bottle and the chamber containing the heaters. A recirculating fan forced air over the heating elements (or nitrogen) and through a baffle into the test chamber. The baffle ensured uniform temperature over the specimen.

Additional thermocouples were used before the start of the test program to check the accuracy of the controller. The maximum variation in temperature was ±5°C (±9°F) at both elevated and cryogenic temperatures. It took approximately one hour to reach elevated temperature:

316°C (600°F), and fifteen minutes to reach cryogenic temperature: -157°C (250°F). Specimens were allowed to soak at temperature for ten to fifteen minutes before being tested. This was felt to be sufficient time to reach uniform temperature but insufficient time to cause any aging problems (reference [19]).

Strain Measurement

Strains were measured for unnotched tension tests by bonded foil strain gages. The accuracy and repeatability of foil gages for determining elastic modulus has been established by Chapman [54], Weymouth [55], and Telinde [56]. One longitudinal and one transverse gage were mounted on each side of the specimen. The longitudinal gages were wired in series and connected so as to constitute one arm of a Wheatstone bridge circuit. The transverse gages were similarly connected to a separate bridge. All gages were connected with a three-wire temperature compensation method. Dummy gages of the same type were mounted on specimens of identical layup in the same configuration as on the test specimen. The two longitudinal dummy gages were connected in series and connected to the compensating arm of the longitudinal bridge. The transverse dummy gages were connected to the transverse bridge in the same manner. The appropriate dummy specimen was placed in the environmental chamber during cryogenic or elevated temperature tests or on the movable cross-head during room temperature tests. Care was taken to place the dummy specimen in such a way as to permit free expansion or contraction with changing temperature.

The four gages constituting the longitudinal half-bridge and the four gages constituting the transverse half-bridge were connected to different channels of a strain conditioner which contained provisions for circuit excitation, bridge balancing, and output signal amplification. Although the gages were not stacked, the polyimide is a sufficiently poor heat sink that low excitation voltage was necessary to prevent gage heating. The excitation voltage across the bridge was kept below 2 volts. Because of the extreme temperatures encountered during testing, the temperature dependent gage factor (supplied by the manufacturer) was used to determine the value of resistor to use for shunt calibration at each temperature. In order to eliminate drift problems, specimens were held unclamped until the proper test temperature was reached and then clamped into the grips after the strain gage circuit was calibrated. Strain and load signals were recorded on an X-Y-Y plotter during the test.

Although the half-bridge circuit used for these tests is not perfectly linear, it is accurate to within 2 percent for the strains attainable with this material. There was little electrical equipment in the testing room and the strain gage lead wires were short (less than one meter) so electrical noise problems were minimal. Careful inspection of the strain gages indicated no alignment problems. The transverse sensitivity factor of all gages was (according to the manufacturer) zero. Because of the care taken with the strain gages, strain measurements are considered to be extremely accurate at the low strain levels at which elastic constants were determined. There were, however, a large number of gage failures at high strain levels due either to the failure of the adhesive or to tearing of the gage backing.

RESULTS AND DISCUSSION

Results

The results of the unnotched tension tests are summarized in Tables 1 and 2. Three replicate tests were performed at each temperature for each laminate, except where noted. Results presented in this paper are average values of the replicate tests. In most cases, data values were within a few percent of the average values presented in this paper. The experimental elastic constants varied very little about the mean while the strength values varied by as much as ten percent about the mean. The principal exceptions occurred in the data for the $[90]_8$ and $[\pm45]_{2S}$ laminates. For the $[90]_8$ laminate, the tensile strength at elevated temperature varied almost 30 percent from the average while the tensile strength of the $[\pm45]_{2S}$ laminate varied about 20 percent at elevated temperature. As mentioned previously, it was not possible to determine ultimate tensile strains in many cases, due to failure of the gages from either adhesive failure or gage tearing at high strains.

Lamina constants E_{11}, E_{22}, and ν_{12} were determined from tests of the unidirectional laminates, and G_{12} was determined from tests of the [±45] laminates using Rosen's method [57]. The results appear in Table 1. Laminate constants for unnotched tension specimens were determined experimentally for the other laminates, and compared with laminate analysis [58-60] calculations based on experimental lamina constants (Table 2). As would be expected, good agreement was obtained.

TABLE 1.- LAMINA MATERIAL PROPERTIES*

Laminate	Temperature, deg C (deg F)	Specimen Thickness, mm (in.)	Unnotched Tensile Strength, MPa (ksi)	E_x, GPa (Msi)	ν_{xy}	G_{12}, GPA (Msi)
$[0]_8$	316 (600)	1.14 (.045)	1430 (208)	132[a] (19.1)	.333[b]	...
	24 (75)	1.12 (.044)	1450 (210)	128[a] (18.6)	.367[b]	...
	-157 (-250)	1.12 (.044)	1520 (221)	140[a] (20.3)	.364[b]	...
$[90]_8$	316 (600)	1.12 (.044)	17.7 (2.57)	5.93[c] (.860)	.024	...
	24 (75)	1.17 (.046)	27.7 (4.02)	8.55[c] (1.24)	.040	...
	-157 (-250)	1.14 (.045)	31.8 (4.61)	10.5[c] (1.52)	.049	...
$[\pm45]_{2s}$	316 (600)	1.30 (.051)	92.4 (13.4)	8.48 (1.23)	.813	2.10 (.304)
	24 (75)	1.17 (.046)	121 (17.6)	18.5 (2.68)	.776	4.67 (.678)
	-157 (-250)	1.17 (.046)	133 (19.3)	22.3 (3.23)	.636	6.61 (.959)
$[\pm45]_{4s}$	316 (600)	2.31 (.091)	95.1 (13.8)	d	.885	d
	24 (75)	2.31 (.091)	132 (19.1)	17.2 (2.49)	.771	4.76 (.690)
	-157 (-250)	2.24 (.088)	145 (21.0)	22.3 (3.23)	.737	6.41 (.930)

[a]E_{11}; [b]ν_{12}; [c]E_{22}; [d]Calibration error

*Material manufactured in 1978.

TABLE 2.- LAMINATE MATERIAL PROPERTIES*

Laminate	Temperature, deg C (deg F)	Specimen Thickness, mm (in.)	Unnotched Tensile Strength, MPa (ksi)	E_x, GPa (Msi)		ν_{xy}	
				Experimental	Theoretical	Experimental	Theoretical
$[0/45/90/-45]_s$	316 (600)	1.04 (.041)	419 (60.7)	46.1 (6.69)	47.5 (6.89)	.319	.332
	24 (75)	1.12 (.044)	369 (53.5)	47.9 (6.95)	49.4 (7.17)	.349	.315
	-157 (-250)	1.14 (.045)	325 (47.1)	50.5 (7.32)	55.6 (8.07)	.360	.303
$[0/45/90/-45]_{2s}$	316 (600)	2.36 (.093)	430 (62.4)	43.9 (6.37)	47.5 (6.89)	.316	.332
	24 (75)	2.39 (.094)	461 (66.8)	47.3 (6.86)	49.4 (7.17)	.345	.315
	-157 (-250)	2.29 (.090)	521 (75.5)	53.0 (7.68)	55.6 (8.07)	.339	.303
$[45/0/-45/0]_s$	316 (600)	1.04 (.041)	710 (103)	75.8 (11.0)	70.3 (10.2)	1.01	.813
	24 (75)	1.12 (.044)	686 (99.5)	73.8 (10.7)	73.1 (10.6)	.667	.700
	-157 (-250)	1.12 (.044)	689 (100)	77.9 (11.3)	82.0 (11.9)	.619	.651
$[45/90/-45/90]_s$	316 (600)	0.99 (.039)	143 (20.7)	19.1 (2.77)	18.1 (2.63)	.213	.209
	24 (75)	1.04 (.041)	166 (24.1)	19.5 (2.83)	21.2 (3.07)	.208	.203
	-157 (-250)	0.99 (.039)	185 (26.8)	23.9 (3.46)	24.9 (3.61)	.228	.198

*Material manufactured in 1978.

The results of the notched tension tests are summarized in Table 3. Three replicate tests were performed at each temperature for each laminate, except where noted. Results presented in this paper are average values of the replicate tests. In no case did notched strength vary more than eight percent from the mean values presented here. The test data were correlated using the Nuismer and Whitney point stress and average stress criteria [43]. Each criterion yields a characteristic distance, which appears in the table. More will be said about this distance in a later section.

Discussion

Unnotched Tension Tests

In order to see more clearly the effect of temperature on elastic properties, data also are presented in graphic form. The laminates can be divided roughly into matrix- and fiber-dominated laminates. It can be seen from Figs. 1 and 2 that both axial stiffness and tensile strength are nearly independent of temperature for the fiber-dominated laminates. An anomaly in the tensile strength of the 8-ply and 16-ply quasi-isotropic laminates is apparent in Fig. 2. The laminates were approximately equal in strength at elevated temperature. The 8-ply laminate, however, became weaker with decreasing temperature, whereas the 16-ply laminate became stronger. The situation is quite different for matrix-dominated laminates. As seen in Figs. 3 and 4, both axial stiffness and tensile strength increase with decreasing temperature.

TABLE 3.- NOTCHED TENSILE TESTS RESULTS

(a) $[0/45/90/-45]_s$*

Temperature, deg C (deg F)	Specimen Width, mm (in.)	Specimen Thickness, mm (in.)	Hole Diameter, mm (in.)	Notched Tensile Strength, MPa (ksi)	Characteristic Distance	
					d_o mm (in.)	a_o mm (in.)
316	100	1.09	25.4	199	3.07	7.47
(600)	(3.94)	(.043)	(1.00)	(28.8)	(.121)	(.294)
	100	1.09	9.53	281	2.57	7.82
	(3.94)	(.043)	(.375)	(40.7)	(.101)	(.308)
	100	1.07	4.76	297	1.50	4.80
	(3.93)	(.042)	(.188)	(43.1)	(.059)	(.189)
	100	1.09	1.59	f	-	-
	(3.94)	(.043)	(.063)	-	-	-
24	100	1.09	25.4	175	3.07	7.47
(75)	(3.95)	(.043)	(1.00)	(25.4)	(.121)	(.294)
	100	1.12	9.53	234	2.21	6.38
	(3.95)	(.044)	(.375)	(34.0)	(.087)	(.251)
	100	1.07	4.76	274	1.70	5.82
	(3.95)	(.042)	(.188)	(39.7)	(.067)	(.229)
	100	1.07	1.59	306[a]	0.83	3.51
	(3.93)	(.042)	(.063)	(44.4)	(.033)	(.138)
	50.8	1.17	4.76	259	1.47	4.72
	(2.00)	(.046)	(.188)	(37.6)	(.058)	(.186)
	25.4	1.14	4.76	228	1.12	3.20
	(1.00)	(.045)	(.188)	(33.0)	(.044)	(.126)
	19.1	1.14	4.76	222	1.14	3.30
	(.752)	(.045)	(.188)	(32.2)	(.045)	(.130)
-157	100	1.07	25.4	217	8.10	26.4
(-250)	(3.94)	(.042)	(1.00)	(31.3)	(.319)	(1.04)
	100	1.09	9.53	278[c]	6.02	28.5
	(3.95)	(.043)	(.375)	(40.4)	(.237)	(1.12)
	100	1.12	4.76	290[a]	3.56	18.9
	(3.95)	(.044)	(.188)	(42.0)	(.140)	(.744)
	100	1.09	1.59	f	-	-
	(3.93)	(.043)	(.063)	-	-	-

[a]One specimen. [f]All specimens failed at grip. [c]Two specimens.

*Material manufactured in 1978.

TABLE 3.- CONTINUED

(b) $[45/0/-45/0]_s$*

Temperature, deg C (deg F)	Specimen Width, mm (in.)	Specimen Thickness, mm (in.)	Hole Diameter, mm (in.)	Notched Tensile Strength, MPa (ksi)	Characteristic Distance	
					d_o mm (in.)	a_o mm (in.)
316	63.2	1.17	25.4	298	3.02	8.08
(600)	(2.49)	(.046)	(1.00)	(43.2)	(.119)	(.318)
	63.2	1.17	9.53	476	2.49	8.31
	(2.49)	(.046)	(.375)	(69.0)	(.098)	(.327)
	63.2	1.09	4.76	503	1.37	4.80
	(2.49)	(.043)	(.188)	(72.9)	(.054)	(.189)
	63.2	1.09	1.59	684[c]	2.36	20.7[b]
	(2.49)	(.043)	(.063)	(99.2)	(.093)	(.815)
24	63.2	1.17	25.4	274	2.64	6.81
(75)	(2.49)	(.046)	(1.00)	(39.7)	(.104)	(.268)
	63.5	1.14	9.53	423	1.96	6.07
	(2.50)	(.045)	(.375)	(61.4)	(.077)	(.239)
	63.2	1.09	4.76	467	1.22	4.09
	(2.49)	(.043)	(.188)	(67.7)	(.048)	(.161)
	63.2	1.12	1.59	542	0.66	2.62
	(2.49)	(.044)	(.063)	(78.6)	(.026)	(.103)
	50.8	1.09	4.76	465	1.24	4.09
	(2.00)	(.043)	(.188)	(67.5)	(.049)	(.161)
	25.4	1.04	4.76	411	0.94	2.87
	(1.00)	(.041)	(.188)	(59.6)	(.037)	(.113)
	18.9	1.07	4.76	403[d]	0.99	3.05
	(.746)	(.042)	(.188)	(58.5)	(.039)	(.120)
-157	63.5	1.17	25.4	277[c]	2.69	6.93
(-250)	(2.50)	(.046)	(1.00)	(40.2)	(.106)	(.273)
	63.5	1.17	9.53	414[c]	1.83	5.49
	(2.50)	(.046)	(.375)	(60.0)	(.072)	(.216)
	63.5	1.12	4.76	441	1.04	3.23
	(2.50)	(.044)	(.188)	(63.9)	(.041)	(.127)
	63.5	1.07	1.59	546[c]	0.66	2.67
	(2.50)	(.042)	(.063)	(79.2)	(.026)	(.105)

[b]Not used in plot. [c]Two specimens. [d]Four specimens.

*Material manufactured in 1978.

TABLE 3.- CONTINUED

(c) $[\pm 45]_{2s}$*

Temperature, deg C (deg F)	Specimen Width, mm (in.)	Specimen Thickness, mm (in.)	Hole Diameter, mm (in.)	Notched Tensile Strength, MPa (ksi)	Characteristic Distance	
					d_o mm (in.)	a_o mm (in.)
316	100	1.17	25.4	86.2	e	e
(600)	(3.95)	(.046)	(1.00)	(12.5)	...	...
	100	1.27	9.53	104	...	...
	(3.95)	(.050)	(.375)	(15.0)	...	...
	100	1.22	4.76	114	...	...
	(3.95)	(.048)	(.188)	(16.5)	...	...
	100	1.22	1.59	115	...	...
	(3.95)	(.048)	(.063)	(16.7)	...	...
24	100	1.14	25.4	118	e	e
(75)	(3.95)	(.045)	(1.00)	(17.2)	...	...
	100	1.24	9.53	127	...	...
	(3.95)	(.049)	(.375)	(18.4)	...	...
	100	1.22	4.76	151	...	...
	(3.95)	(.048)	(.188)	(21.9)	...	...
	100	1.27	1.59	160	...	...
	(3.95)	(.050)	(.063)	(23.2)	...	...
	50.8	1.24	4.76	136	...	...
	(2.00)	(.049)	(.188)	(19.7)	...	...
	25.4	1.22	4.76	116	...	...
	(1.00)	(.048)	(.188)	(16.8)	...	...
	18.9	1.24	4.76	103	...	...
	(.746)	(.049)	(.188)	(14.9)	...	...
-157	100	1.22	25.4	125	e	e
(-250)	(3.96)	(.048)	(1.00)	(18.2)	...	...
	100	1.12	9.53	151	...	...
	(3.95)	(.044)	(.375)	(21.9)	...	...
	100	1.24	4.76	174	...	...
	(3.95)	(.049)	(.188)	(25.3)	...	...
	100	1.24	1.59	174	...	...
	(3.95)	(.049)	(.063)	(25.2)	...	...

[e]Nuismer-Whitney model not applicable.

*Material manufactured in 1978.

TABLE 3.- CONCLUDED

(d) $[0/45/90/-45]_{2s}$*

Temperature, deg C (deg F)	Specimen Width, mm (in.)	Specimen Thickness, mm (in.)	Hole Diameter, mm(in.)	Notched Tensile Strength, MPa (ksi)	Characteristic Distance	
					d_o mm (in.)	a_o mm (in.)
316	63.5	2.36	4.76	328	1.85	6.68
(600)	(2.50)	(.093)	(.188)	(47.5)	(.073)	(.263)
24	63.2	2.41	4.76	319	1.40	4.37
(75)	(2.49)	(.095)	(.188)	(46.2)	(.055)	(.172)
-157	63.5	2.34	4.76	345	1.22	3.58
(-250)	(2.50)	(.092)	(.188)	(50.0)	(.048)	(.141)

*Material manufactured in 1978.

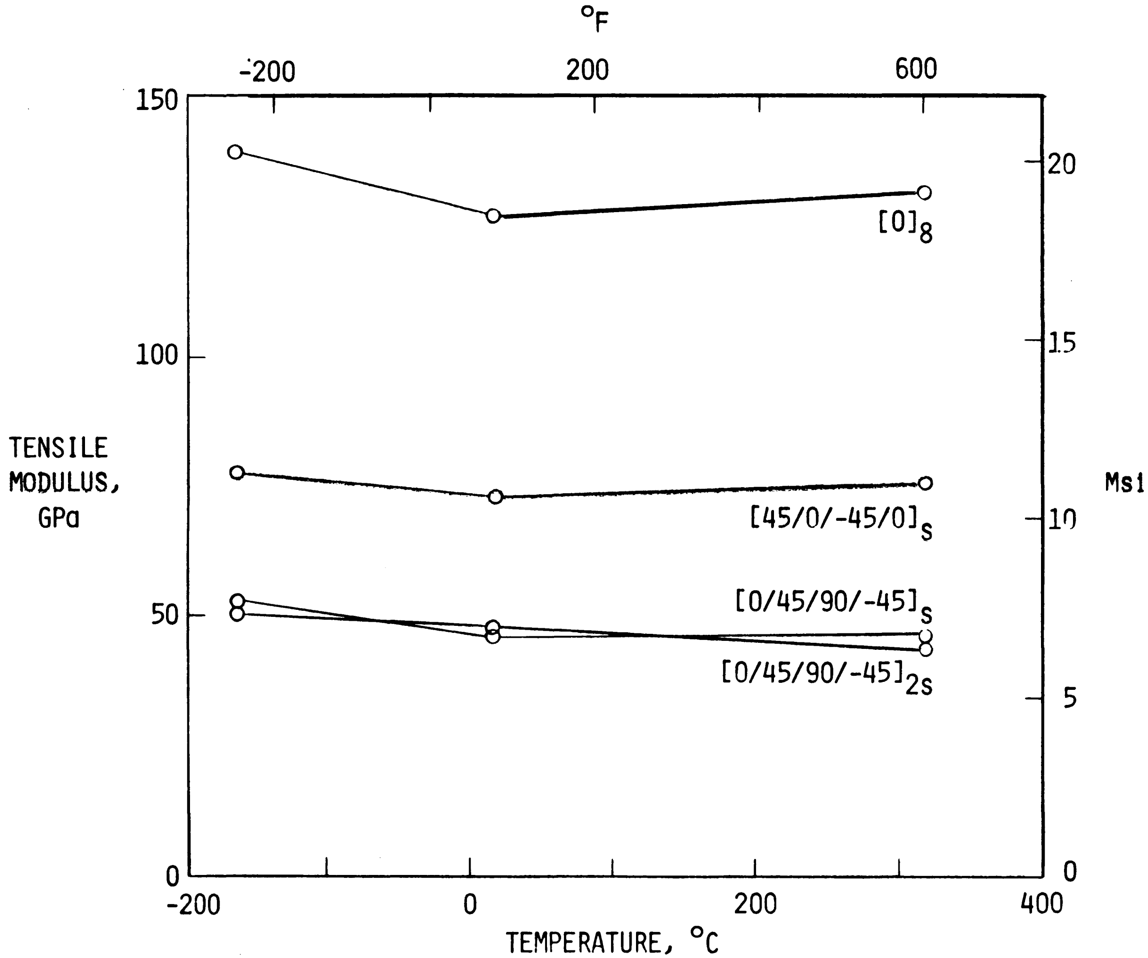

Figure 1. Axial Stiffness versus Temperature for Fiber-Dominated Laminates.

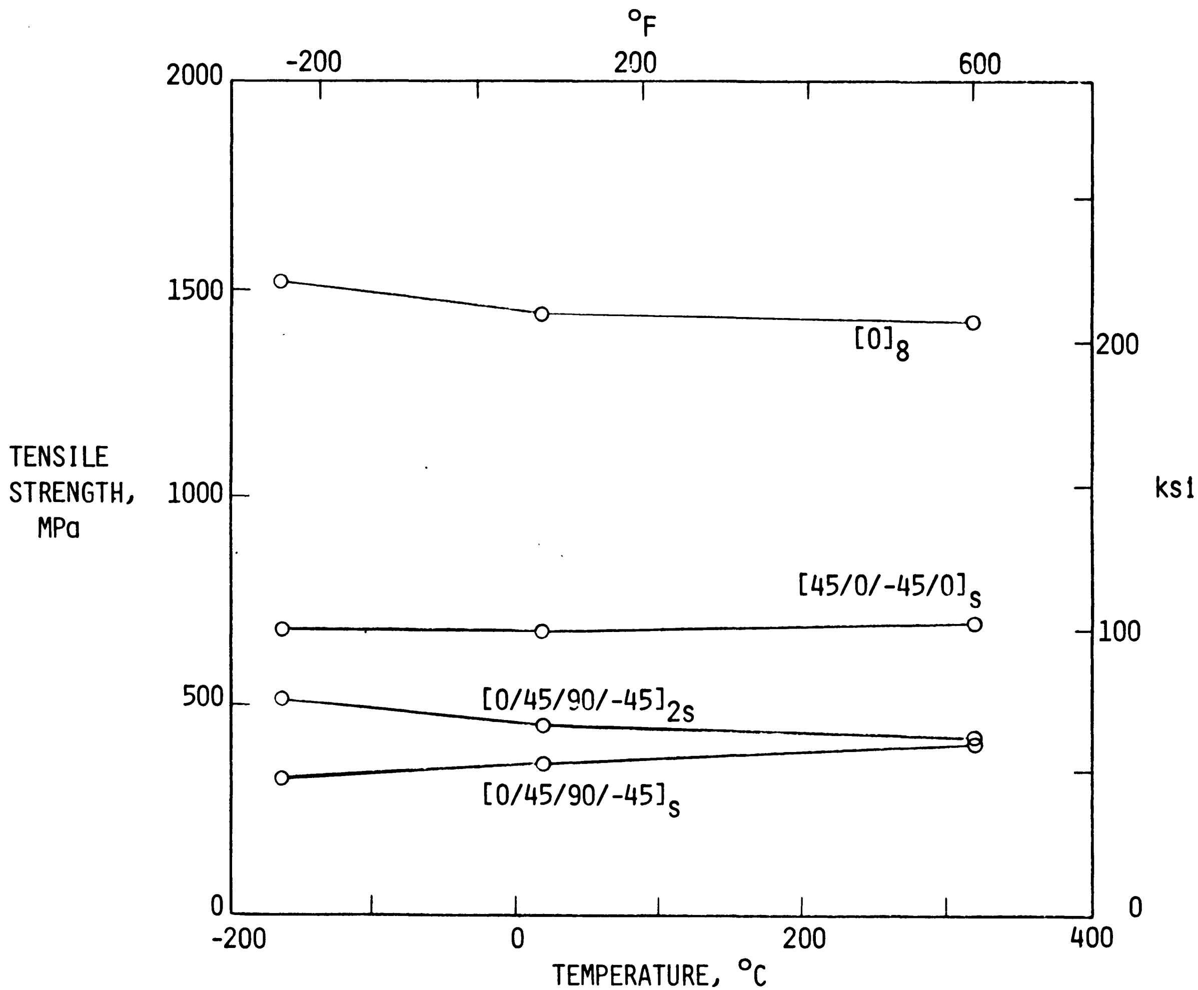

Figure 2. Tensile Strength versus Temperature for Fiber-Dominated Laminates.

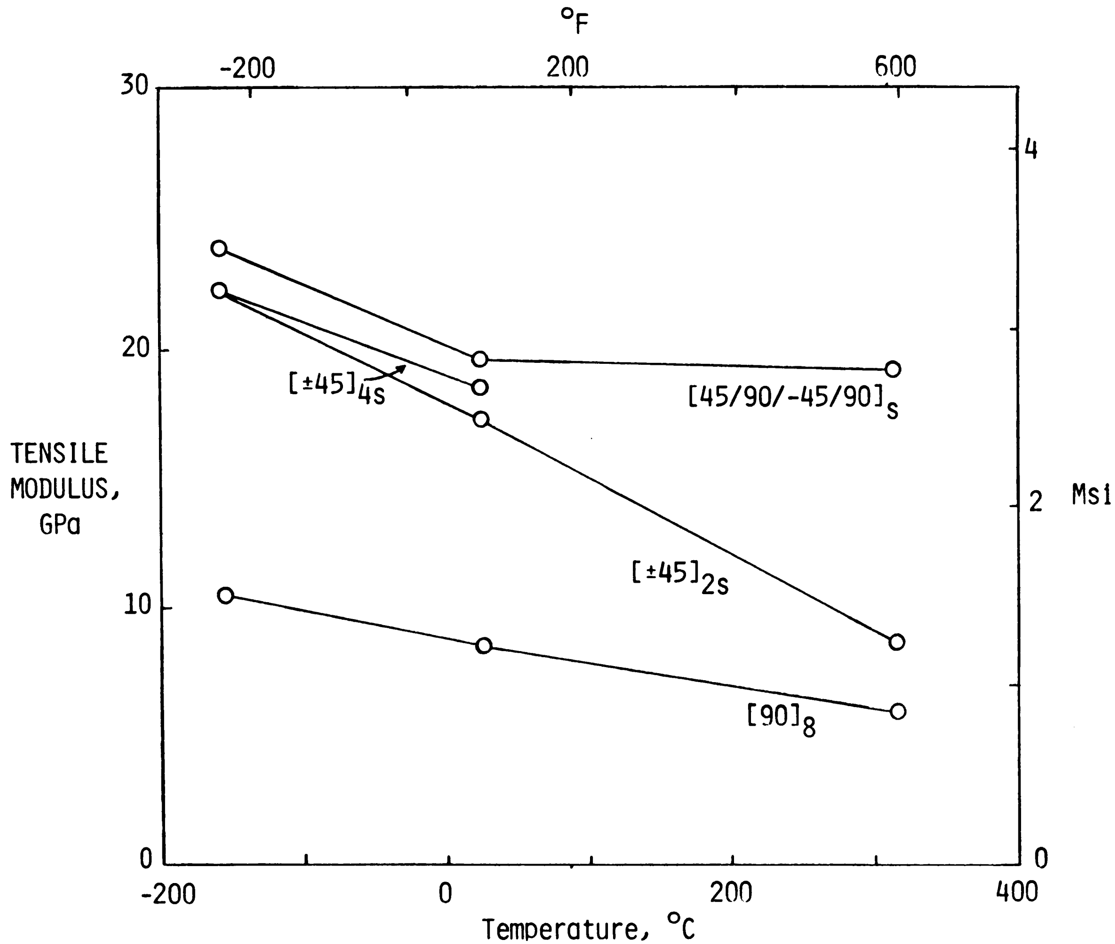

Figure 3. Axial Stiffness versus Temperature for Matrix-Dominated Laminates.

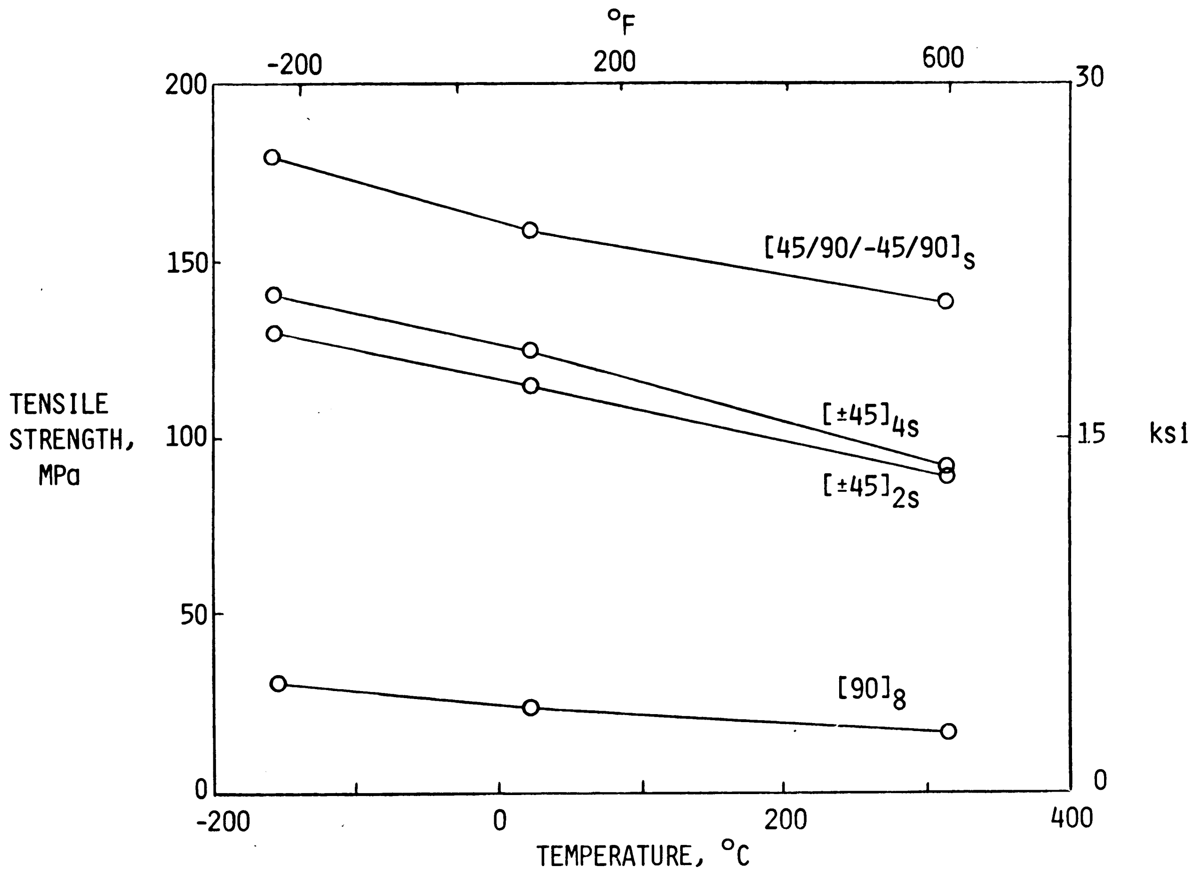

Figure 4. Tensile Strength versus Temperature for Matrix-Dominated Laminates.

The effect of temperature on Poisson's ratio is somewhat different, as shown in Fig. 5. For comparison purposes, the laminates may be divided into three groups. The laminates containing 45-deg plies but no 90-deg plies exhibit high values of Poisson's ratio, with the ratio increasing with increasing temperature. Laminates containing 90-deg plies but no 0-deg plies exhibit low values of Poisson's ratio, with no significant effect of temperature. The remaining laminates have intermediate values of Poisson's ratio which closely resemble the values for some isotropic metallic materials. For these laminates there is no significant effect of temperature.

While there is relatively little data available on C-6000/PMR-15 it is possible to make a few comparisons with other researchers. Elastic constants presented in [27] agree very well with data shown here. The only substantial difference occurs for the unnotched strength of the $[90]_8$ laminate at room temperature. Although all $[90]_8$ specimens failed in the test section, the unnotched strength determination for this laminate at any temperature is very sensitive to differences in gripping techniques and specimen alignment so it is not surprising that a disagreement between values should occur here.

Notched Tension Tests

The effect of temperature on notched strength will be examined first. Figure 6 indicates a slight temperature effect for the quasi-isotropic laminate. Higher strengths were obtained at elevated and

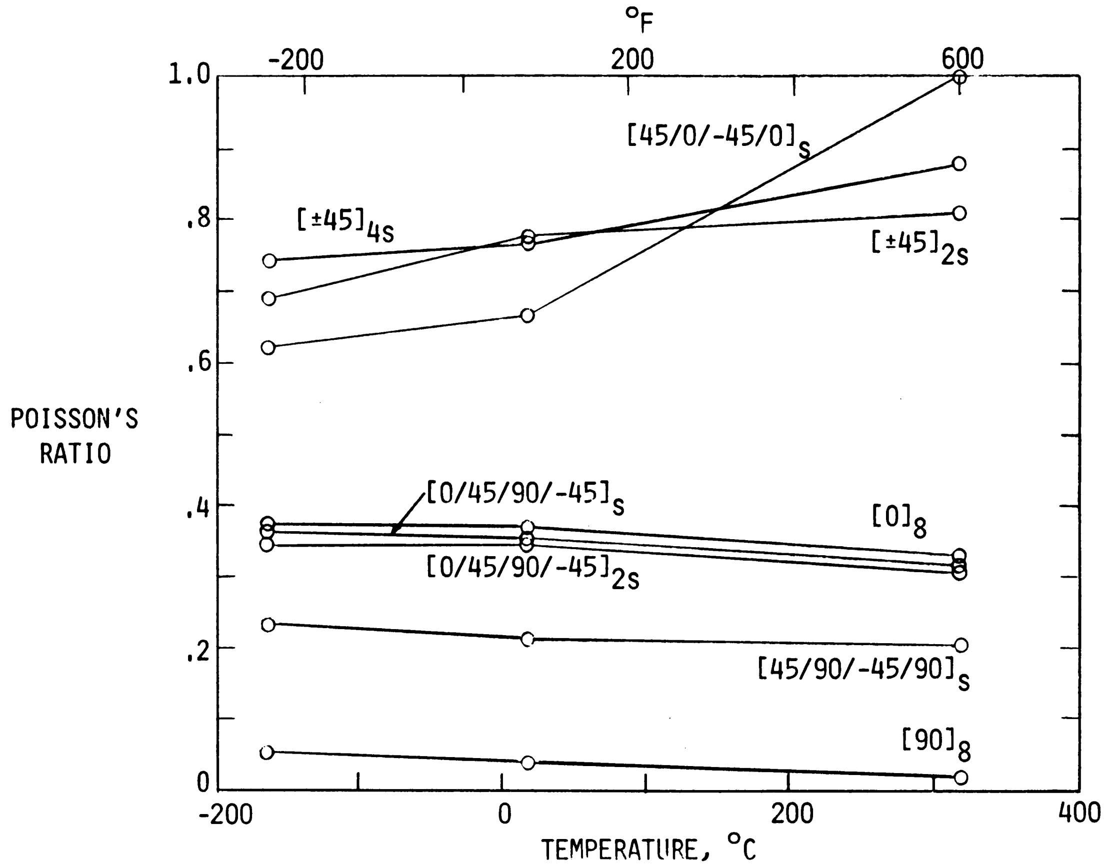

Figure 5. Poisson's Ratio versus Temperature.

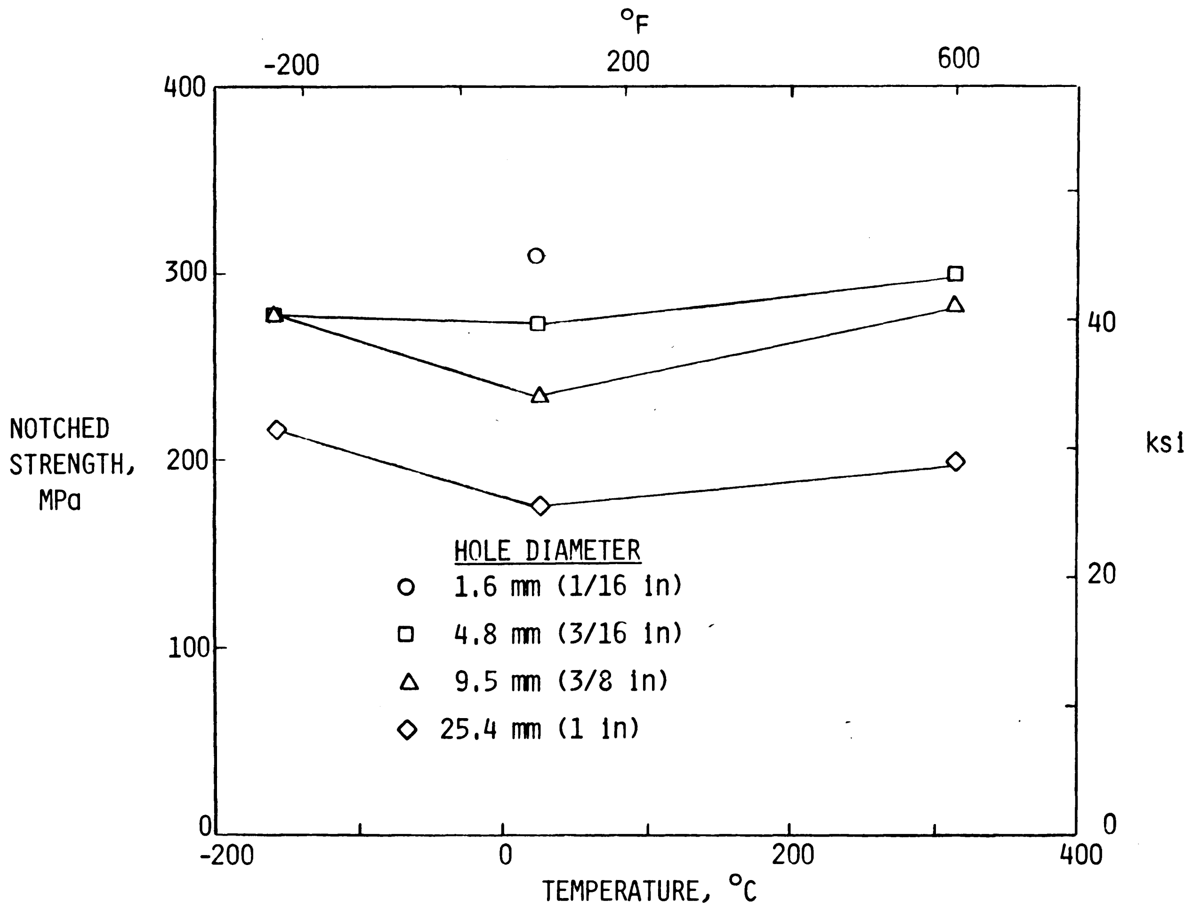

Figure 6. Notched Strength versus Temperature for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).

cryogenic temperatures than at room temperature. On the other hand, the strength of unnotched quasi-isotropic laminates decreased with decreasing temperature (Fig. 2). The notched strength of this laminate at cryogenic temperature should be viewed with caution, as approximately half of the specimens failed at the grips.

There are two possible explanations which are immediately apparent for why notched fracture specimens should fail at the grip rather than at the notch. The first involves the gripping method. The grip design required that five through-holes be drilled in the specimen ends to permit passage of the bolts. The decrease in specimen thickness due to the Poisson effect as the load increased may have caused the load to be introduced in bearing on the bolt hole rather than by friction at the grip edge. Many of the specimens which failed at the grip show a fracture surface terminating at a bolt hole. The second explanation involves the quality of the material tested. The specimens were manufactured while processing techniques were being developed. It is possible that flaws or voids sufficiently large to cause premature failure escaped detection during screening.

Due to the large number of grip failures at -157°C (-250°F) for the quasi-isotropic laminate, all tests of the $[45/0/-45/0]_S$ laminate were conducted on specimens 64 mm (2.5 in.) wide. Figure 7 shows only a slight temperature effect for this laminate, except for the smallest hole (one of three specimens did not fail at the hole nor at the grip).

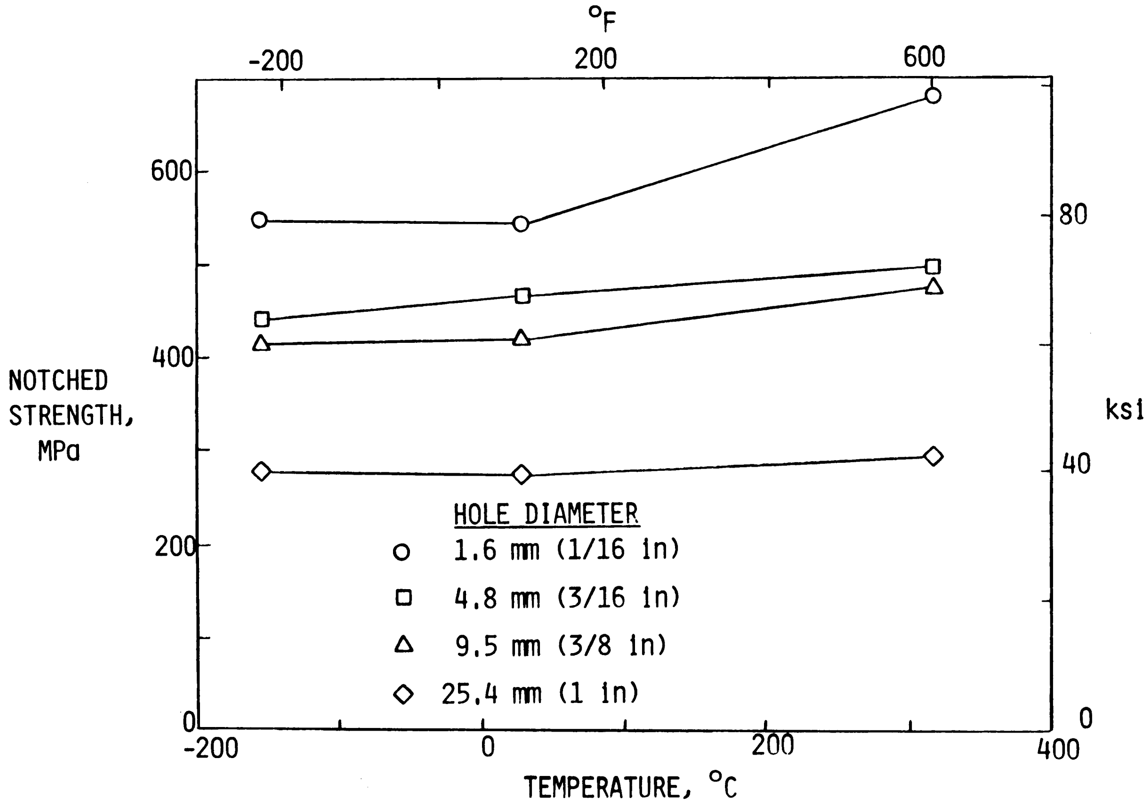

Figure 7. Notched Strength versus Temperature for the $[45/0/-45/0]_S$ Laminates, Width = 64 mm (2.5 in.).

Cryogenic and room temperature strengths were nearly identical with a slightly higher strength at elevated temperature. Similar trends were noted for the unnotched laminates (Fig. 2). As with the quasi-isotropic laminate, the effect of hole size on strength is very noticeable.

The data for the $[\pm45]_{2S}$ laminate, as shown in Fig. 8, should be viewed with caution. Many of the specimens showed extensive ply pullout after failure, making it impossible to determine with any degree of certainty whether failure initiated at the hold, the grip, or the region of high thermal gradient where the specimens passed through the oven wall. This suggests that [±45] specimens should be tested in a different manner.

Despite the uncertainty as to the location of failure initiation, the data shown in Fig. 8 clearly indicate the existence of a notch effect which is temperature dependent. As would be expected for a matrix-dominated laminate, both strength and the apparent notch effect decrease with increasing temperature.

Figure 9 shows the effect of width on notched strength for a fixed flaw size and temperature. For all three laminates there is little width effect for plates wider than 51 mm (2 in.); that is, plates with a hole diameter to width ratio less than 0.094 may be treated as infinite plates. As mentioned previously, no tests were conducted on 102 mm (4 in.) plates for the $[45/0/-45/0]_S$ laminate. It should be noted that all data points in Fig. 9 are the average of three identical tests. As

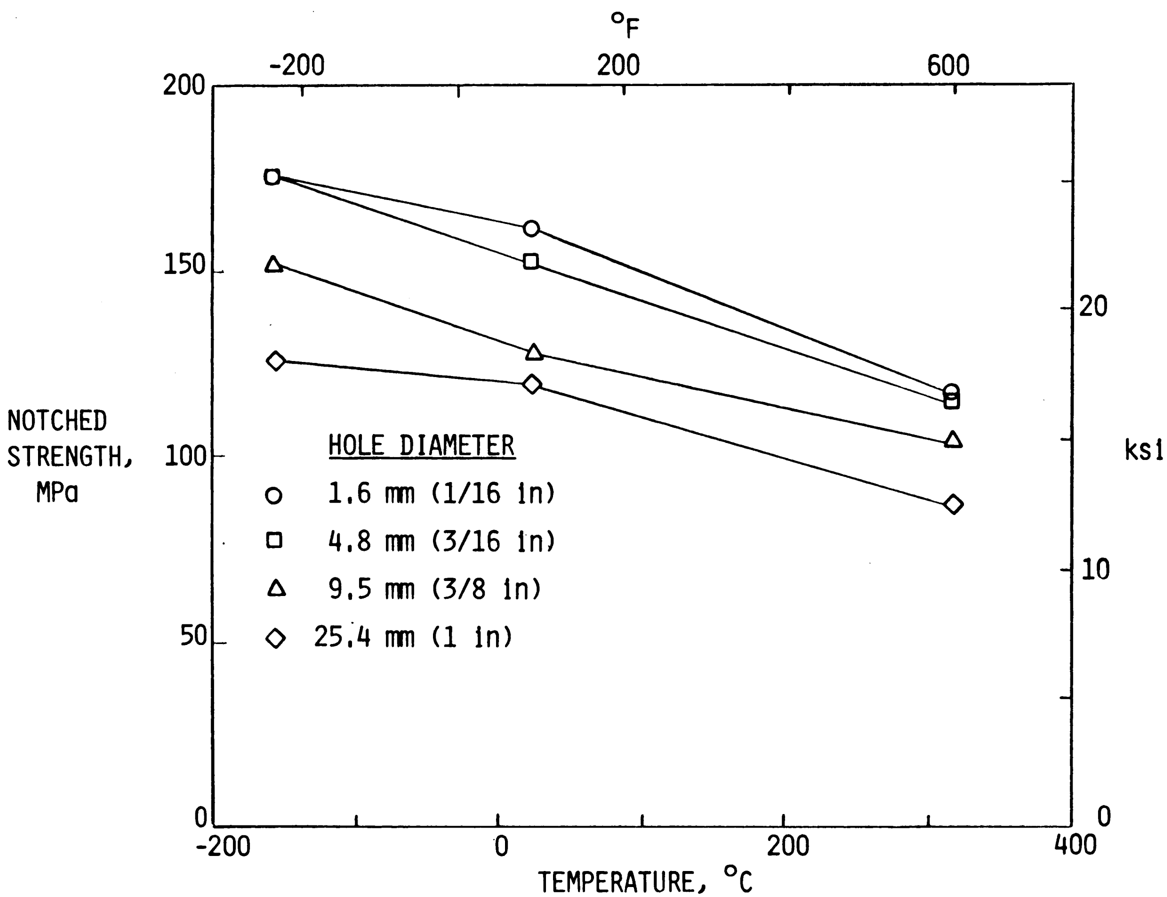

Figure 8. Notched Strength versus Temperature for the $[\pm45]_{2S}$ Laminates, Width = 102 mm (4.0 in.).

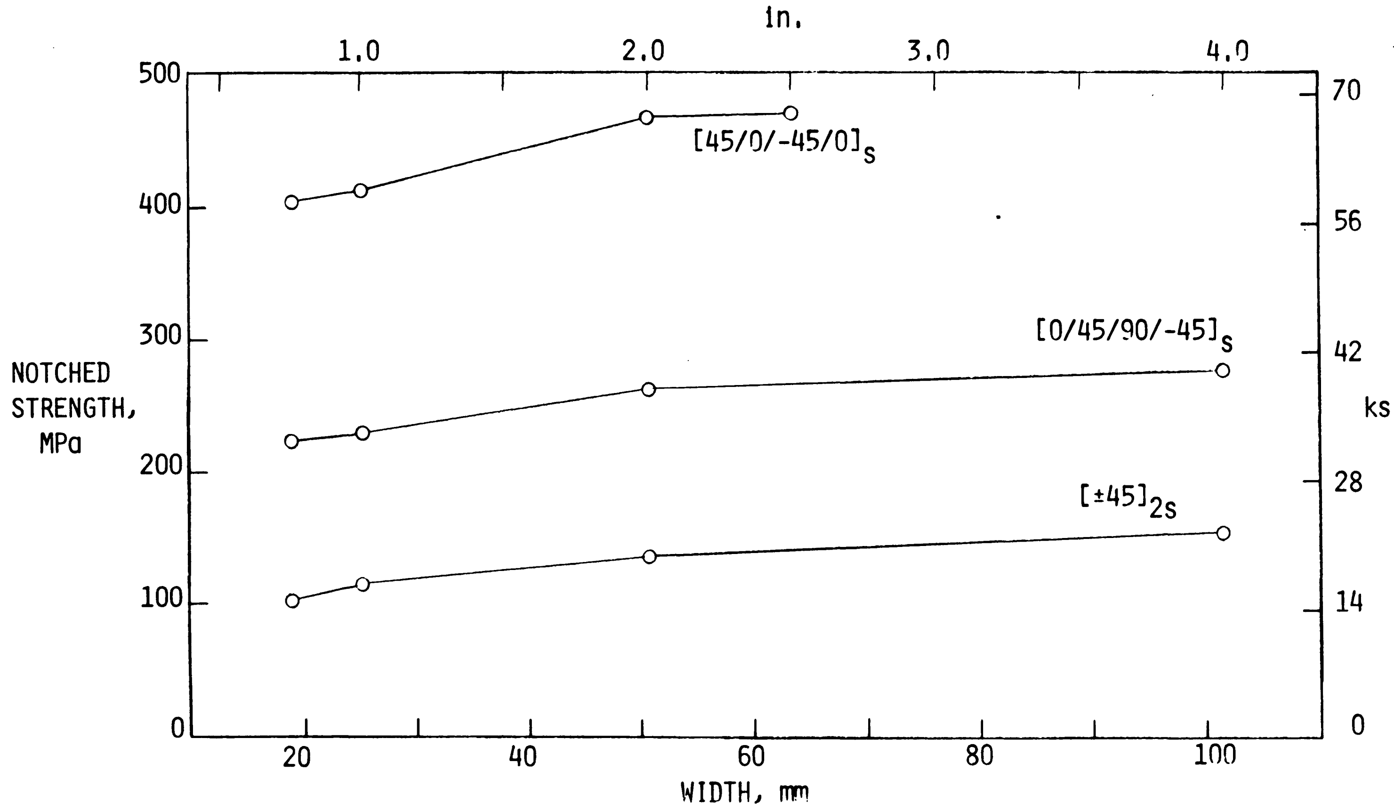

Figure 9. Notched Strength versus Width at Room Temperature for a Fixed Flaw Size of 4.8 mm (3/16 in.).

mentioned previously, notched strength varied no more than eight percent about the mean values presented here.

Failure Model

Figures 10 and 11 compare the experimental notched strengths with strengths predicted by the Nuismer and Whitney point stress and average stress models [42]. The point stress model states that failure of a notched tension specimen will occur when the stress at some distance, d_0, ahead of the notch equals the unnotched tensile strength. In equation form, this failure criterion is

$$\frac{\sigma_N^\infty}{\sigma_0} = 2[2 + \zeta^2 + 3\zeta^4 - (K_T^\infty - 3)(5\zeta^6 - 7\zeta^8)]^{-1}$$

where $\zeta = R/(R + d_0)$, σ_N^∞ is the failure stress for a notched infinite plate, σ_0 is the unnotched tensile strength, K_T^∞ is the orthotropic stress concentration factor for an infinite width plate, and R is hole radius. The infinite plate notched strength is found by multiplying the experimentally determined notched strength for a finite width plate by a finite width correction factor [43].

The average stress model states that failure of a notched specimen will occur when the average stress over some distance ahead of the

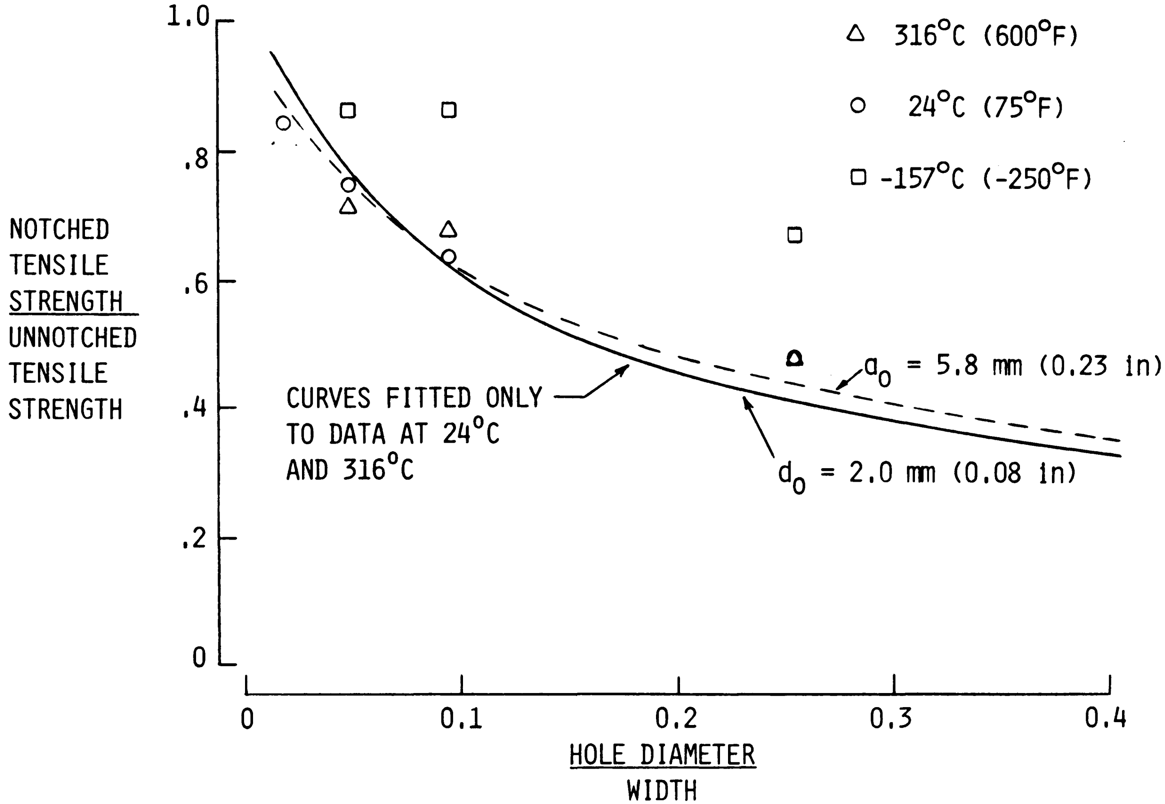

Figure 10. Comparison of Experimental and Theoretical Failure Strengths for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).

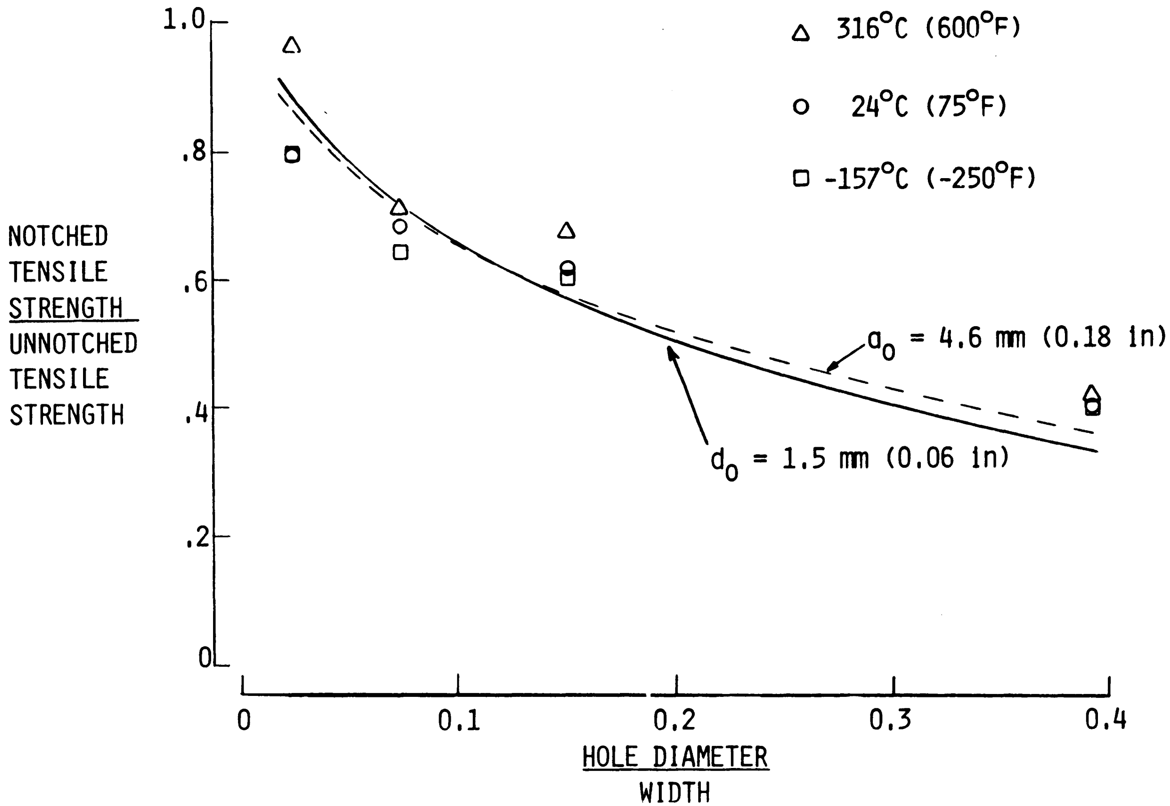

Figure 11. Comparison of Experimental and Theoretical Failure Strengths for the $[45/0\text{-}45/0]_S$ Laminates, Width = 64 mm (2.5 in.).

notch, a_0, equals the unnotched tensile strength. This failure criterion may be written

$$\frac{\sigma_N^{\infty}}{\sigma_0} = 2(1 - \xi)[2 - \xi^2 - \xi^4 + (K_T^{\infty} - 3)(\xi^6 - \xi^8)]^{-1}$$

where $\xi = R/(R + a_0)$. The distances d_0 and a_0 are called characteristic distances.

It is apparent from Fig. 10 that one value of a_0 or d_0 gives fair predictions of the notched strength of the $[0/45/90/-45]_S$ laminate for both room and elevated temperature. These characteristic distances were found using the appropriate failure criterion, the unnotched strength, and the experimentally determined notched strengths. The values of a_0 and d_0 are average for all data at the two temperatures and various hole sizes. The average stress criterion gives slightly better results than the point stress criterion. Average values of a_0 and d_0 also were determined from the cryogenic temperature data for this laminate. Due to uncertainty in data from both notched and unnotched tests, however, curves for these values are not shown in the figure.

As shown in Fig. 11, the notched strengths of $[45/0/-45/0]_S$ laminates at all three temperatures can be predicted fairly well using one value of a_0 or d_0. Again, the average stress criterion gives a slightly better fit.

However, as shown in Fig. 12, the fracture data for the matrix-dominated [±45] laminate cannot be correlated using the Nuismer-Whitney model [43]. For this laminate, notched strengths greater than unnotched strength were measured. Similar results have been found for a graphite/epoxy laminate at room temperature [61].

Figures 13 to 15 depict characteristic distance as a function of temperature. Figure 13 shows the behavior of a_0 and d_0 for a 16-ply quasi-isotropic laminate for three temperatures. Also shown in the variation of a_0 and d_0 with temperature for the $[45/0/-45/0]_S$ laminate. The characteristic distances show nearly linear response with temperature for both laminates.

As shown in Fig. 14, the characteristic distance-temperature behavior for the 8-ply quasi-isotropic laminate is different from the 16-ply behavior shown in Fig. 13. As illustrated in Fig. 14, the elevated and room temperature value of a_0 and d_0 are practically identical, but characteristic distances at cryogenic temperature are much higher. This difference in behavior may be due to two factors.

First, due to a large number of grip failures, the average values of a_0 and d_0 at cryogenic temperature, as shown in Fig. 14, may not be as accurate as the values at room and elevated temperature. However, it is felt that the number of tests at a given temperature does not

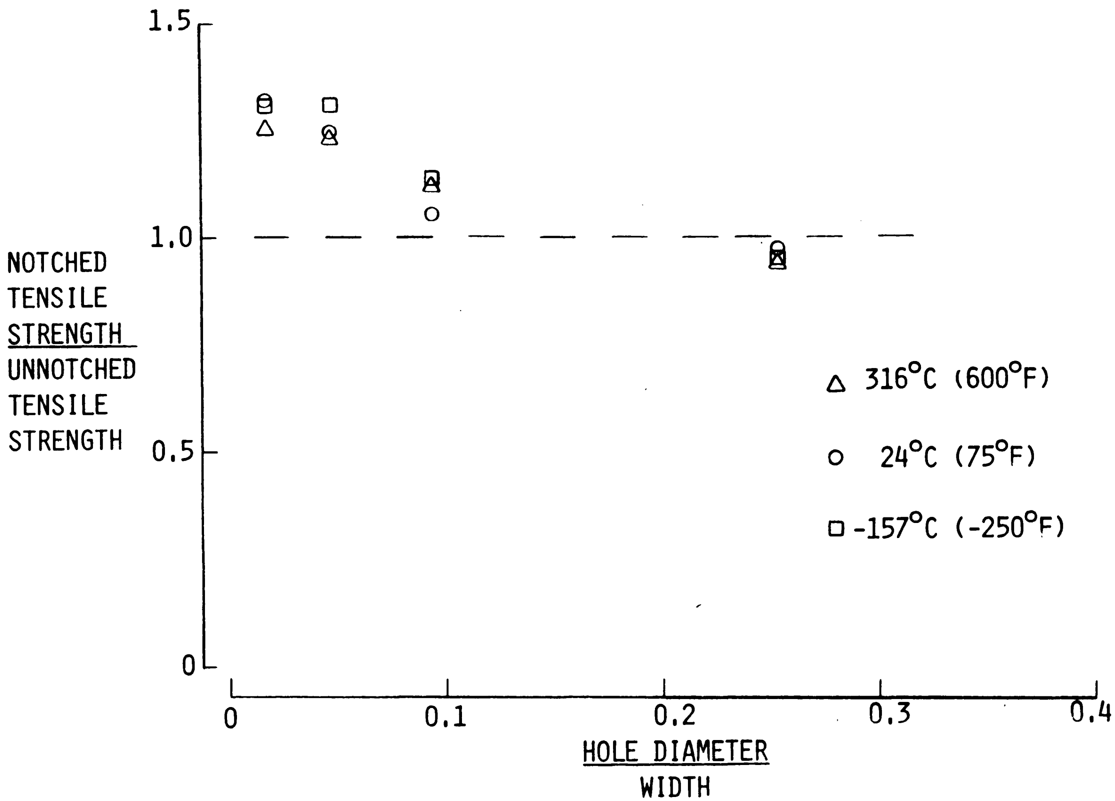

Figure 12. Comparison of Experimental and Theoretical Failure Strengths for the $[\pm 45]_{2S}$ Laminates, Width = 102 mm (4.0 in.).

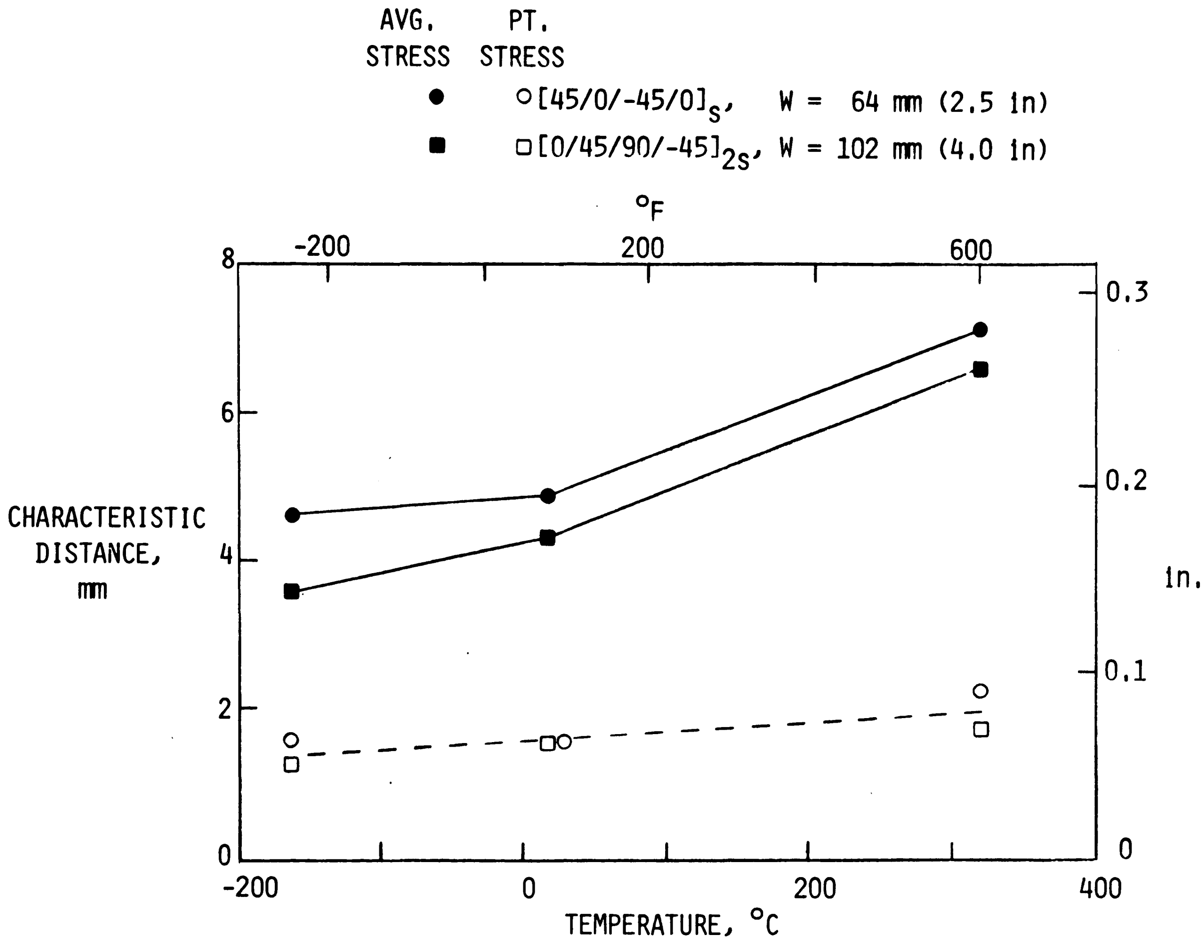

Figure 13. Characteristic Distance versus Temperature for the $[45/0/-45/0]_S$ and $[0/45/90/-45]_{2S}$ Laminates.

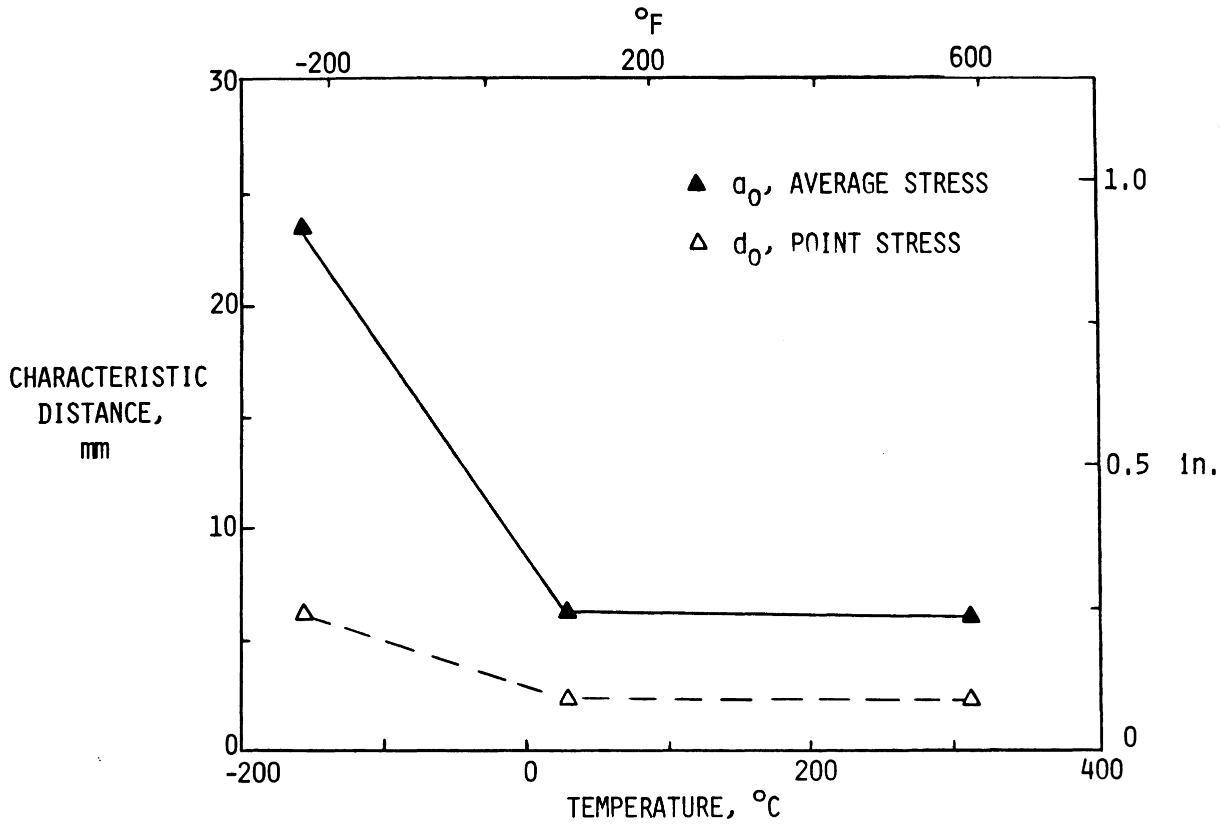

Figure 14. Characteristic Distance versus Temperature for the $[0/45/90/-45]_S$ Laminates, Width = 102 mm (4.0 in.).

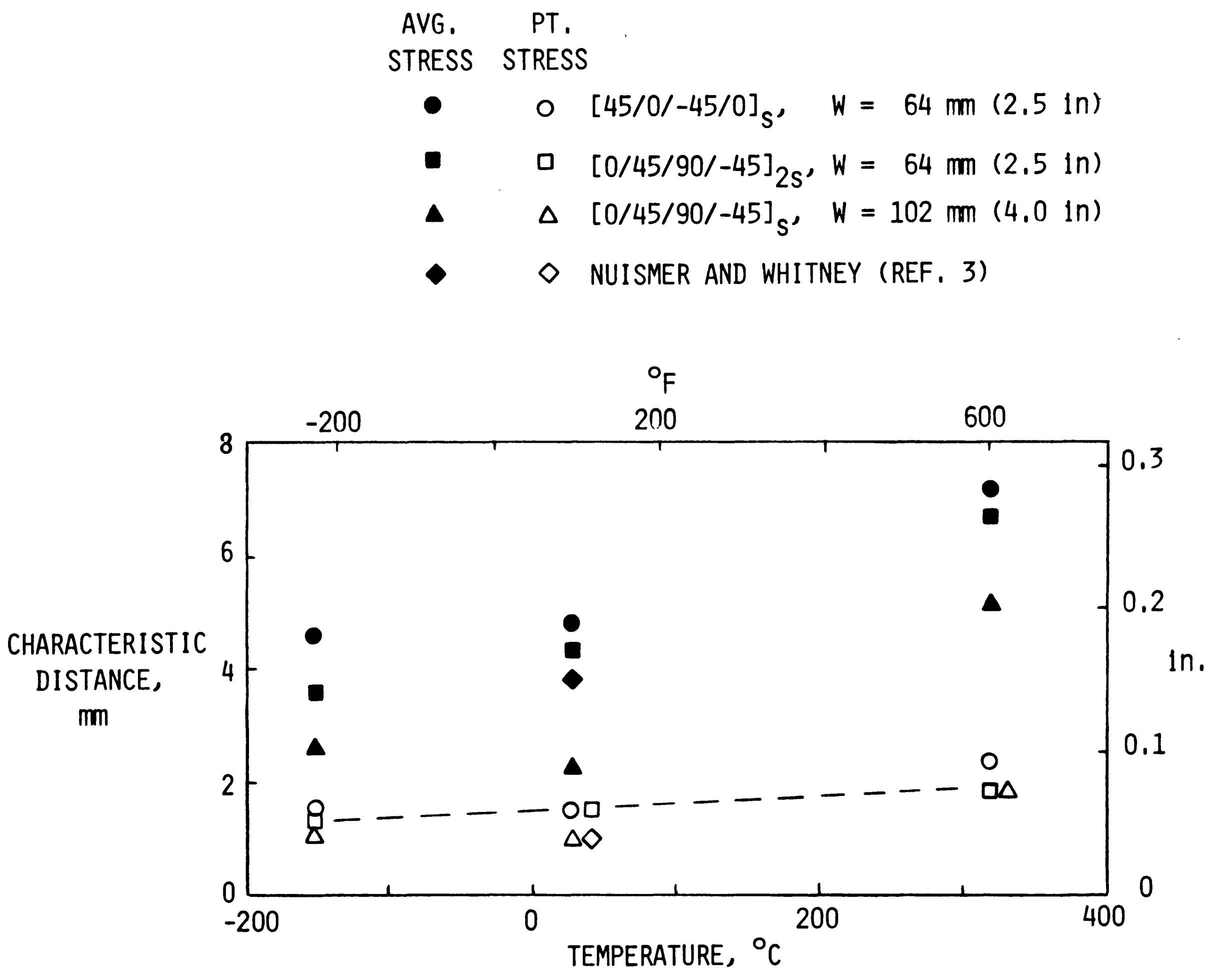

Figure 15. Characteristic Distance versus Temperature for the Fiber-Dominated Laminates.

explain adequately the large difference in a_0 and d_0 for low temperature as compared to the other two temperatures.

Second, the differences in a_0 and d_0 shown in Fig. 14 may be due to the extreme sensitivity of the characteristic distance calculation of the ratio of notched to unnotched strength. Slight changes in the ratio $\sigma_N^{\infty}/\sigma_0$ cause a_0 and d_0 to change dramatically. The noticeable decrease in the unnotched strength of 8-ply quasi-isotropic laminate with decreasing temperature (Fig. 3) appears to be responsible for the unusually high values which were calculated for the characteristic distance at cryogenic temperature, as shown in Fig. 14.

In order to reduce the effect of an apparent unnotched strength anomaly on the characteristic distance calculations, the a_0 and d_0 values were recalculated for the 8-ply quasi-isotropic laminate using the unnotched strength of the 16-ply laminate for normalization. This procedure appeared to be justified in view of the exceptional trend in the unnotched tensile strength data for the 8-ply quasi-isotropic laminate. The decrease in strength with decreasing temperature apparent in Fig. 3 is significantly different from the behavior of the other fiber-dominated laminates and from the behavior of the matrix-dominated laminates shown in Fig. 4. The modified results are shown in Fig. 15, along with all other point and average stress criteria data. All the data in this figure show an approximately linear relationship with temperature. The data are in fair agreement with the room temperature data for similar laminates of graphite/epoxy as reported by Nuismer and Whitney [43]. Although not shown in Fig. 15, the room temperature values of a_0 are similar to those found by Morris and Hahn [46,47] for similar laminates of graphite/epoxy.

Since the Celion-6000/PMR-15 composite was in the early stages of development, no attempt was made to obtain a better fit to the data shown in Figs. 10 and 11. However, the mathematically simple models of Nuismer and Whitney appear to predict the notched strengths of the fiber-dominated laminates studied. A more accurate prediction for various temperatures awaits further development of the Celion 6000/PMR-15 material such that more consistent values of strength are obtainable.

SUMMARY AND CONCLUSIONS

The results presented here indicate that elastic modulus and unnotched tensile strength for the fiber-dominated laminates are not highly temperature dependent. However, the corresponding moduli and strengths for the matrix-dominated laminates are quite sensitive to changes in temperature.

Notched tensile strength is dependent on both hole size and temperature, regardless of the type of laminate. Notched plates with hole diameter to plate width ratios less than 0.094 may be treated as infinitely wide plates, at least at room temperature.

The Nuismer-Whitney point stress and average stress models [43] give fair predictions of notched strength for the $[0/45/90/-45]_S$ laminate. Due to uncertainty in the cryogenic data resulting from the large number of grip failures, little confidence can be placed in the values for a_0 and d_0 determined for this temperature. The available data, however, tend to indicate that a_0 and d_0 are temperature dependent. Due to the relative insensitivity of the strength calculations to small changes in characteristic distance, it is felt that one value of a_0 or d_0 may be sufficient for room and elevated temperatures, as evidenced by Fig. 10.

The temperature dependence of a_0 or d_0 for the $[45/0/-45/0]_S$ laminate is much less than for the quasi-isotropic laminate. In fact, one value of a_0 or d_0, along with the corresponding Nuismer-Whitney model, results in fair predictions of the notched strength of the $[45/0/-45/0]_S$ laminates for all three temperatures. Since this study was conducted during the early stages of development of the Celion-6000/PMR-15 material, no attempt was made to determine the functional form of the temperature dependence of the characteristic lengths.

The Nuismer-Whitney models are of no value when predicting the notched strength of the $[\pm45]_{2S}$ laminate, regardless of temperature.

The testing techniques followed in this program appear, for the most part, to be adequate for the task of producing reliable, repeatable data. Two aspects of the testing procedure, however, should be modified to produce better and more complete results. First, strain gages and gage adhesives should be more carefully matched to the strain levels and environmental conditions to be experienced by the specimen. While the gages and adhesives used in this testing program were sufficient to reliably yield strain measurements for the determination of elastic constants at low strain levels, they failed at strains below the ultimate strains of the composites being tested. Second, gripping techniques for four inch wide specimens, and gripping techniques for the $[\pm45]_{2S}$ and $[\pm45]_{4S}$ laminates should be changed. Wedge grips or hydraulic or pneumatic grips should be used rather than grips which require holes in the specimens or grips which do not compensate for the decrease in specimen thickness due to the Poisson effect. For tests of

$[\pm 45]_{nS}$ specimens, the grips should be placed inside the environmental chamber because the properties of this matrix-dominated laminate vary so much with temperature.

Because Celion-6000/PMR-15 was in the early stages of development when the specimens for this study were manufactured, there is some question about the reproducibility of the data presented in this thesis in comparison with data derived from material prepared at a later date. Although C-scan and fiber volume fraction records were incomplete, the limited information available indicated that there were panels from which few specimens could be obtained because of large flawed areas in the panels and that fiber volume fraction varied ten percent from the mean. Despite the range of fiber content, the elastic constants appear to be reasonably accurate. The strength values presented in this thesis, particularly the unnotched strengths of the eight ply quasi-isotropic laminate, may be significantly low because of the processing problems experienced by the manufacturer. Whether the characteristic distances presented here are significantly affected by those processing problems or not, can only be determined by additional testing on materials prepared at a later date.

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ELASTIC PROPERTIES AND FRACTURE BEHAVIOR OF

GRAPHITE/POLYIMIDE COMPOSITES AT EXTREME TEMPERATURES

by

Donald P. Garber

(ABSTRACT)

The influence of elevated and cryogenic temperatures on the elastic moduli and fracture strengths of several Celion-6000/PMR-15 laminates was measured. Tests were run at -157,24 (room temperature), and 316°C (-250, 75, and 600°F). Both unnotched and notched laminates were tested. Several failure criteria, developed to predict the uniaxial fracture strength of epoxy laminates, were used to predict the fracture strength of polyimide laminates.

Lamina elastic moduli were measured at each temperature by testing unnotched $[0]_8$, $[90]_8$, and $[\pm 45]_{2S}$ laminates. The measured values were used with classical laminate theory to predict the elastic constants in $[0/45/90/-45]_S$, $[0/45/90/-45]_{2S}$, $[45/0/-45/0]_S$, and $[45/90/-45/90]_S$ laminates. With few exceptions, the predictions agreed with the moduli measured experimentally. As for the ultimate tensile strength, although the 8-ply and 16-ply quasi-isotropic laminates were about equally strong at elevated temperature, their respective strengths diverged at the lower temperatures. The 8-ply laminates lost strength as the temperature decreased, whereas the 16-ply laminates became stronger.

The notched laminates had layups of $[\pm45]_{2S}$, $[0/45/90/-45]_S$, and $[45/0/-45/0]_S$. The measured moduli, the ultimate strengths, and the point stress or average stress criterion of Nuismer and Whitney were combined to calculate the characteristic lengths associated with each criterion. Characteristic lengths were compared to determine the effect of temperature.