

# Elastic-plastic and failure properties of a unidirectional carbon/PMR-15 composite at room and elevated temperatures

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## Abstract

A series of off-axis tensile tests at room and elevated temperatures have been conducted up to 316°C (600°F) to determine the elastic and plastic properties of a unidirectional carbon/PMR15 composite as a function of temperature. The transverse tensile and shear strengths of the composite as a function of temperature have also been determined. The effect of the specimen preparation process (type of machining) on the strength properties of the composite has also been evaluated. It has been shown that elastic (with the exception of Poisson ratios  $\nu_{12}$  and  $\nu_{21}$ ), plastic, and strength properties of the composite are significantly affected by elevated temperatures. It has also been demonstrated that the quality of machining can noticeably influence the normal and shear strength data at room and elevated temperatures. Even if the quality of machining is very high, failure of the specimens can occur either in the gage or grip sections. At room temperature, all specimens failed in the grip areas influencing the transverse tensile and shear strength measurements. However, the type of specimen failure does not noticeably affect the strength data at elevated temperatures. The transverse tensile and shear strength properties of the composite at room temperature could only be estimated by extrapolating the normal and shear strength vs temperature curves to room temperature. © 2000 Elsevier Science Ltd. All rights reserved.

*Keywords:* PMR-15; Polyimide composites; Shear strength; 10° off axis test

## 1. Introduction

PMR-15 is one of the most widely used thermosetting polyimide resins for high-temperature polymer–matrix composite applications. Of the high-temperature resins, PMR-15 displays the best overall balance of processing, behavior, thermo-oxidative stability, and retention of mechanical properties up to temperatures around 316°C (600°F). Developed in the 1970s at the NASA Lewis Research Center, the bulk of current applications of carbon/PMR-15 composites are in the aerospace industry. With a high stiffness to weight ratio, strength to weight ratio, and glass-transition temperature [Lo et al. [1] have measured the glass-transition temperature of PMR-15 to be 310°C (590°F)], carbon/PMR-15 composites are excellent candidates for structural components such as jet engine housings, wings, and nose cones.

For successful application of carbon/PMR-15 composites in advanced aerospace systems, it is essential for processing and design engineers to have a reliable database of their mechanical properties at room and elevated

temperatures. In particular, the effect of elevated temperatures on the mechanical behavior of unidirectional carbon/PMR-15 needs to be thoroughly understood for the design of more advanced carbon/PMR-15 composite systems such as cross-ply and woven fabric laminates. Extensive studies have been performed on the thermo-oxidative stability and the effects of aging at elevated temperatures on the mechanical behavior of carbon/PMR-15 composites [2,3]. However, a thorough investigation of the elastic-plastic (time independent) and failure properties of unidirectional carbon/PMR-15 systems as a function of temperature (without aging) has never been performed.

The elastic constants of a unidirectional composite are dependent on the elastic properties of the constituent fiber and matrix phases. As the temperature of the composite increases, the polymer matrix becomes more compliant, therefore the composite elastic stiffness constants decrease, especially the matrix-dominated constants (e.g. shear modulus, transverse Young's modulus). When the applied loads reach a certain level, most unidirectional polymer–matrix composite materials begin to exhibit non-linear mechanical behavior (especially when subjected to loading in shear and perpendicular to

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the direction of the fibers) owing to the non-linear behavior of the polymer matrix. This non-linearity becomes more significant as the temperature of the composite increases toward the glass-transition temperature of the polymer matrix.

Another important factor associated with the effect of temperature on the mechanical behavior of composite materials is the significant reduction of their strength properties, especially those dominated by the strength properties of the polymer matrix. Composites tend to exhibit lower shear and transverse tensions strength values as the temperature is increased. Even though higher temperatures may result in a more ductile, and thus tougher, polymer matrix, the loss in strength at elevated temperatures can be significant.

The off-axis tensile test has been a fundamental method of characterizing the mechanical response of unidirectional composite materials for many years. Fig. 1 shows the geometry of the off-axis tensile test. In 1977 Chamis and Sinclair [4] proposed the use of the  $10^\circ$  off-axis tensile test for the determination of the intralaminar shear strength of composite materials. The unidirectional fibers are oriented  $+10^\circ$  to the loading axis which creates a stress state that will cause the material to fail mostly due to shear. There is currently no ASTM standard for the  $10^\circ$  off-axis test specifically, however, ASTM D 3039-76 is a standard test method for off-axis tests in composite materials in general.

Pindera and Herakovich [5] examined the errors in the measured values of elastic properties in off-axis tensile tests due to the end-constraint effects. Traditionally, the specimen is gripped by straight-edged tabs, which produces parasitic shear stresses near the tabs. Sun and Chung [6] developed oblique tabs to reduce these errors by providing a uniform displacement field in the specimen.

The largest source of error in the off-axis tests performed on unidirectional composites may be premature failure due to poor machining. Virtually any machining operation on unidirectional composite materials will generate micro-cracks in the matrix or along the matrix-fiber interface along the edges of the specimen. Under an applied external load, the micro-cracks will propagate and significantly reduce the measured strength. This effect can occur for every loading angle except  $0^\circ$ . At lower temperatures the polymer matrix may become brittle, and this effect will become especially significant. At elevated temperatures, the polymer becomes more ductile, and the microcracks along the specimen edges caused by low quality machining should have a smaller effect on the measured composite strength properties.

The objective of this research was to determine the in-plane elastic, plastic, and failure properties of a unidirectional carbon/PMR-15 composite tested at room and elevated temperatures. This was accomplished by performing  $0^\circ$ ,  $10^\circ$ , and  $90^\circ$  off-axis tensile tests on a carbon/PMR-15 composite material. The macro-

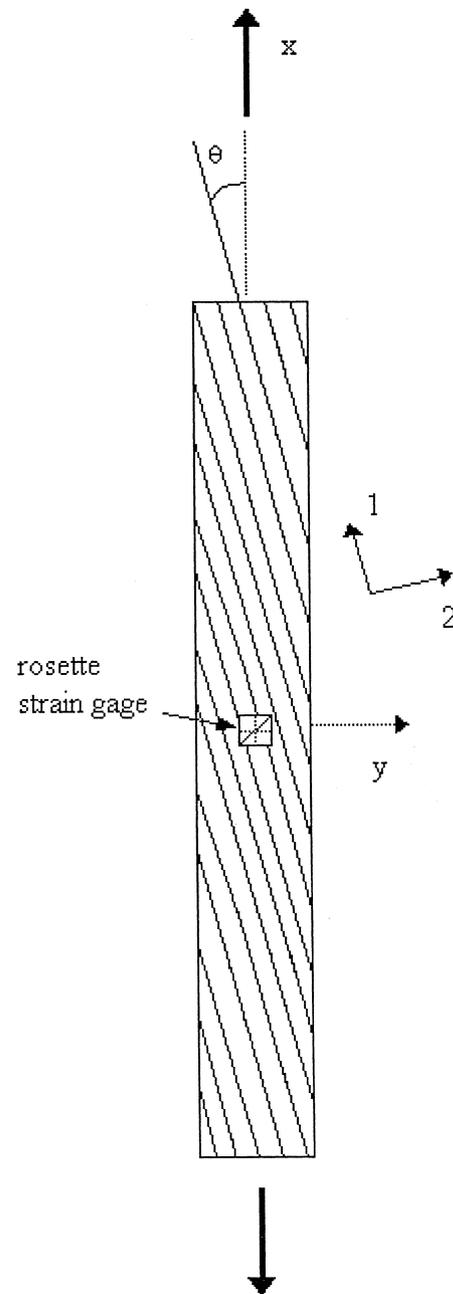


Fig. 1. Off-axis tensile test specimen.

mechanical response of the composite as a function of temperature was established.

## 2. Material and specimen preparation

The composite material used in this study was fabricated at the NASA Glenn Research Center in the form of two  $300\text{ mm} \times 300\text{ mm} \times 5\text{ mm}$  plates per the following specifications:

unidirectional graphite/polyimide  
Fabric: T650-35 unidirectional

Matrix: PMR-15  
 Ply Arrangements: 34-ply  
 Cure: simulated autoclave and postcure

Two different cutting techniques were employed in this research. In the first method, a 0.76 mm (0.03 in.) diameter stream of a highly pressurized mixture of water and abrasive was used to machine the specimens from the as supplied plates (waterjet cutting). In the second method, the samples were cut from the as supplied

The plates were C-scanned before machining and the results indicated excellent quality.

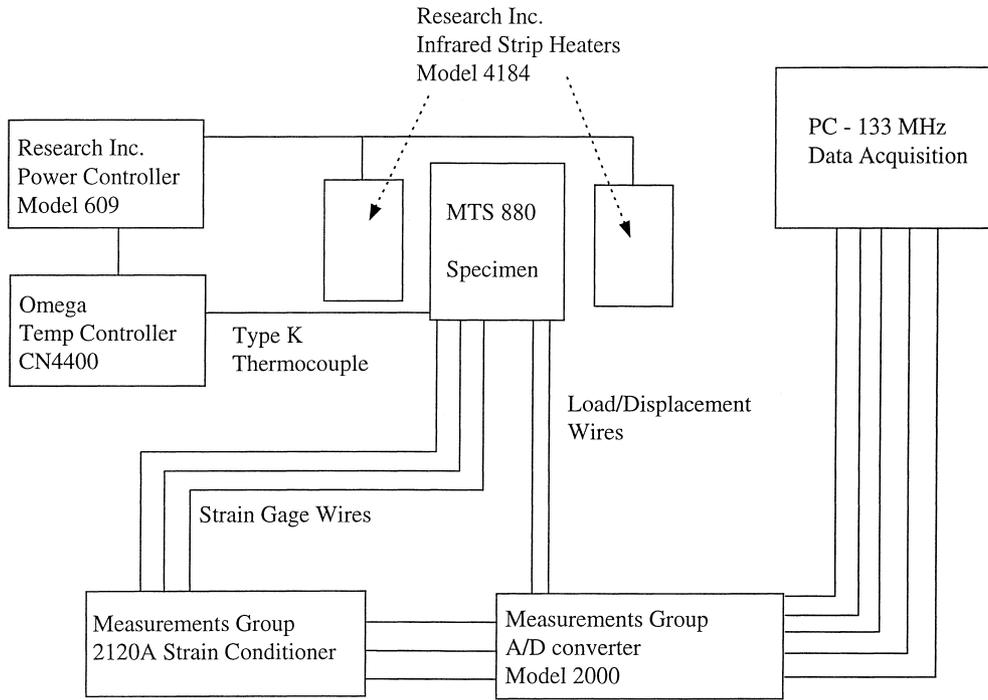


Fig. 2. Experimental setup.

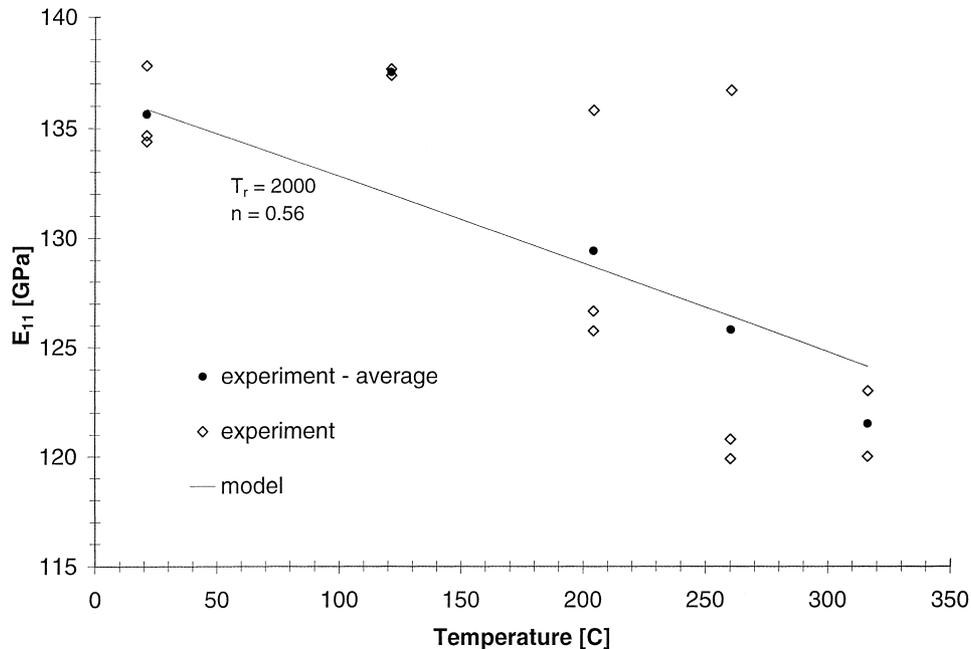


Fig. 3. Axial modulus (parallel to fibers) vs temperature.

plates using a cobalt band saw. The cutting surfaces were subsequently finished by grinding with a carbide end-mill.

### 3. Off-axis experiments

The 0°, 10°, and 90° off-axis test specimens were machined to the dimensions recommended by ASTM

D3039-76. High-temperature three-element rosette strain gages (Measurements Group WK-06-060WR-350) were mounted in the center of the specimens aligned as shown in Fig. 1.

The strain readings were conditioned (Measurements Group model 2120A) and acquired into the data acquisition software using a A/D converter (Measurements Group model 2000). A schematic of the experimental

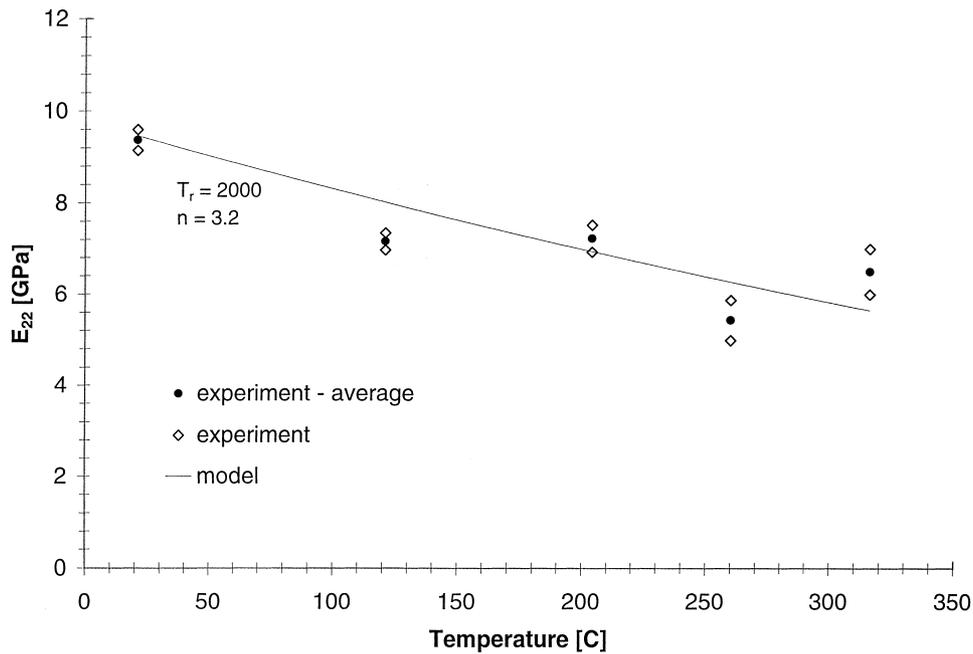


Fig. 4. Axial modulus (transverse to fibers) vs temperature.

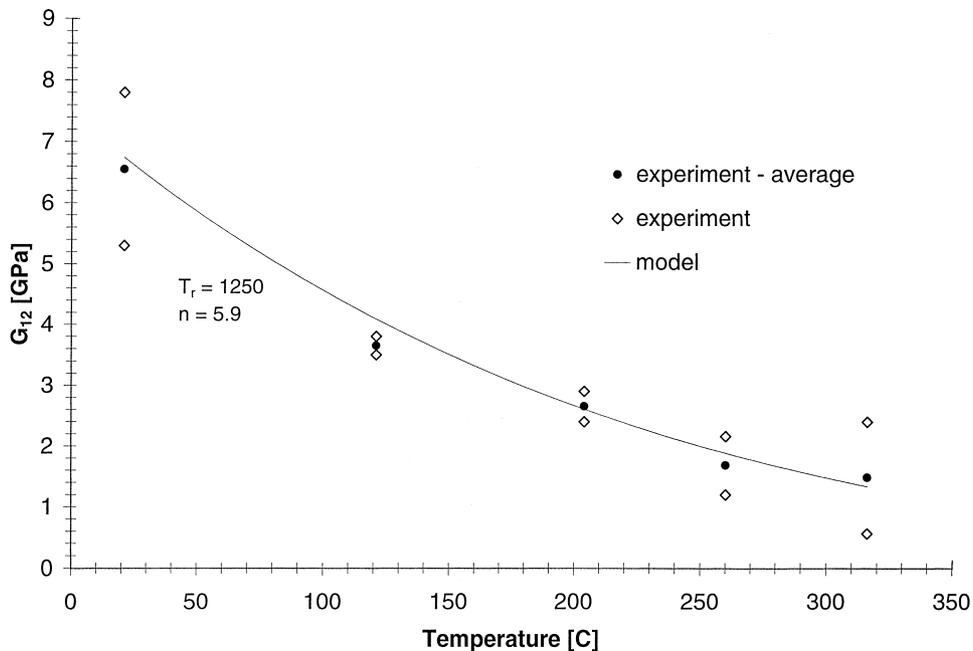


Fig. 5. Shear modulus vs temperature.

setup is shown in Fig. 2. Aluminum tabs were used at the gripped portions of the specimens to prevent crushing due to the serrated (diamond-faceted) grip faces. The grips for the  $0^\circ$  and  $90^\circ$  specimens were strait and those for the  $10^\circ$  specimen had an oblique angle of  $22^\circ$ . All of

the tabs had a beveled front edge in order to reduce the local stress concentrations from gripping. The tabs were adhered to the specimen using a high-temperature epoxy adhesive (CTD 943AD). The tests were performed on a servo-hydraulic MTS 880 with hydraulic

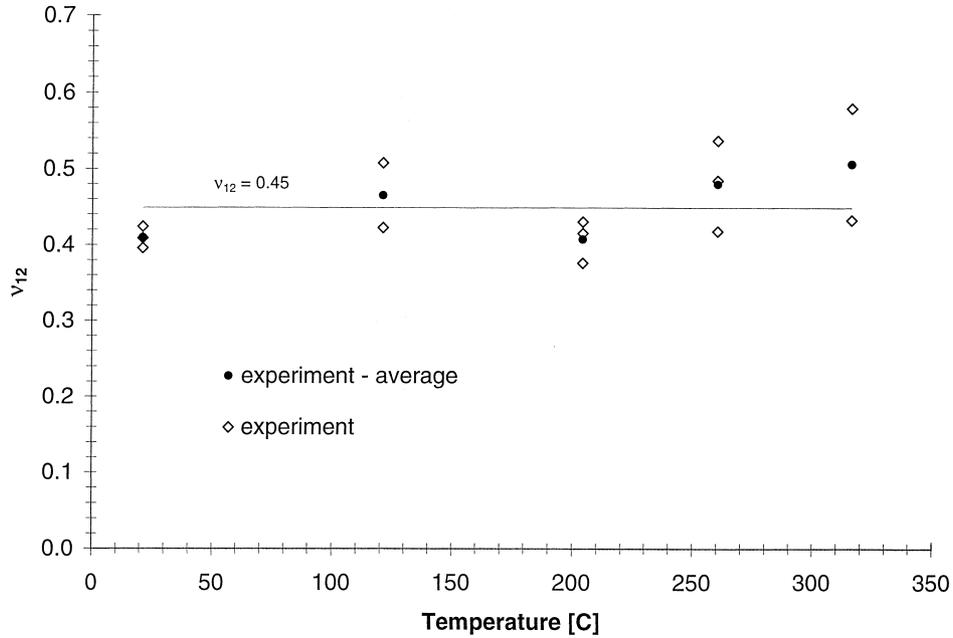


Fig. 6. Poisson's ratio vs temperature.

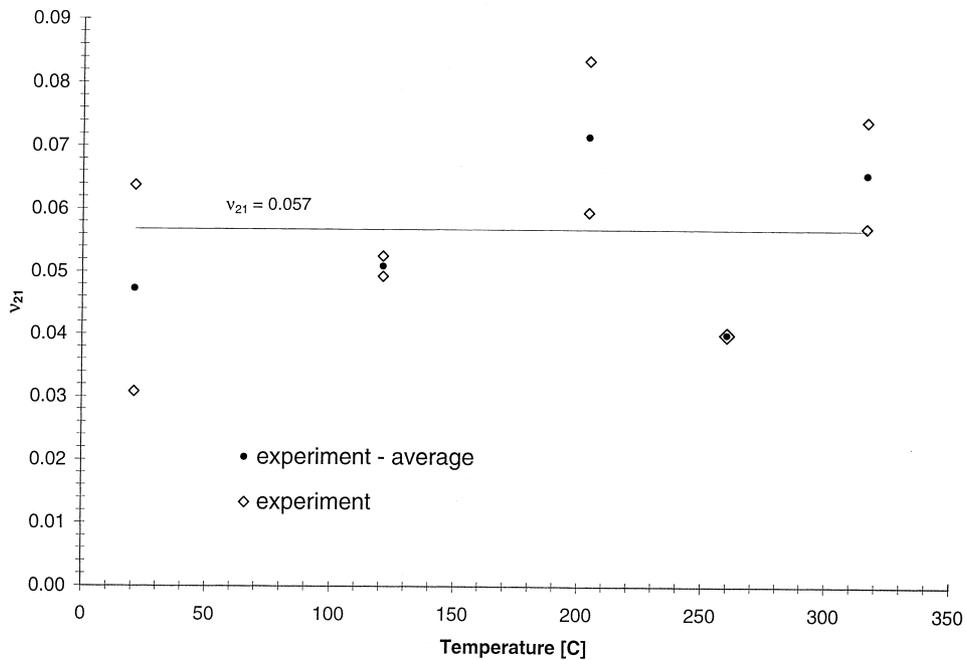


Fig. 7. Poisson's ratio vs temperature.

grips at a displacement rate of 0.5 mm/min. The specimens were aligned in the grips by lining up the centerlines of the specimens with the centerlines indicated on the grip wedges. The alignment was then checked with a square tool to make sure the specimen side surface and the top grip surface were perpendicular.

Infrared heat lamps were used (Research Inc. model 4184) to heat the specimens up to the elevated temperatures. A thermocouple (type K) was mounted onto the heated surface of the specimen. The thermocouple temperature was read by a temperature controller (Omega

model CN4400) which regulated the intensity of the infrared heat lamps (Research Inc. model 609). The specimens soaked at the elevated temperature for 35 min so that the entire specimen was in thermal equilibrium. It was assumed that equilibrium was achieved when the specimen thermal expansion ceased. The load was continuously monitored and kept at 0 N by adjusting the applied specimen displacement. When displacement adjustments were no longer necessary, it was assumed that the specimen had reached thermal equilibrium (35 min).

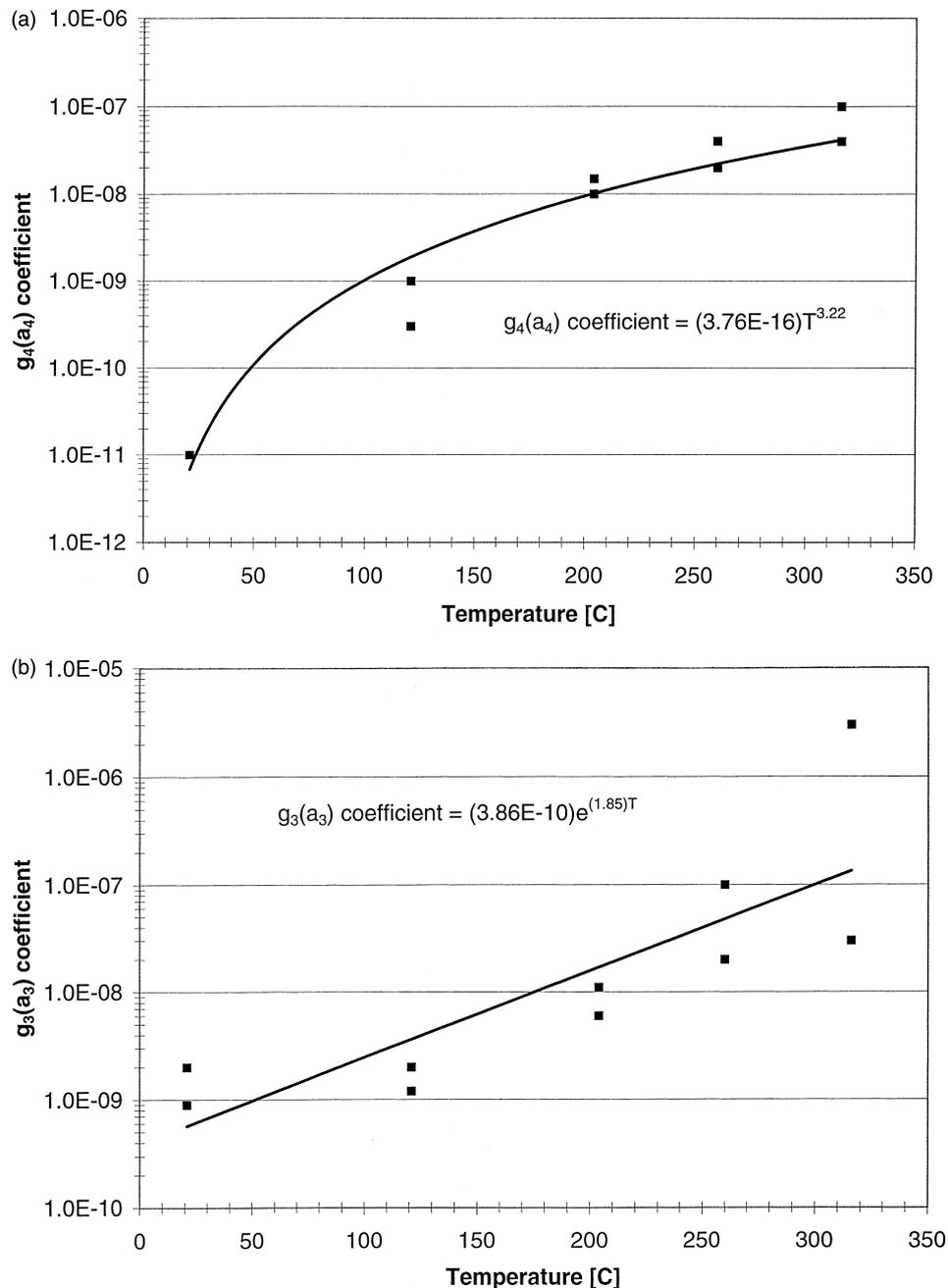


Fig. 8. Plasticity parameters vs temperature for (a)  $g_4(a_4)$  coefficient and (b)  $g_3(a_3)$  coefficient.

#### 4. Results and discussion

The elastic properties of the unidirectional carbon/PMR-15 composite were experimentally determined as a function of temperature using the off-axis tensile tests and subsequently modeled using the relationship proposed by Ha and Springer [7]:

$$C = C_0 \left[ \frac{T_f - T}{T_f - T_0} \right]^n \quad (1)$$

where  $C$  is an elastic constant (e.g. axial modulus or shear modulus) at temperature  $T$ ,  $C_0$  is the reference modulus at  $T_0$  (room temperature), and  $T_f$  is the final temperature where  $C$  is zero.  $T_f$  and  $n$  are material parameters that serve as curve-fitting parameters when applied to experimental data. A general form of Eq. (1) has been thoroughly used by researchers as NASA Glenn Research Center to model material degradation behavior of materials due to environmental effects [8]. A non-linear least squares analysis was used to determine  $T_f$  and  $n$ .

The material non-linearity of the unidirectional carbon/PMR-15 composite was modeled using the same method as used by Odegard and Kumosa [9] for a unidirectional graphite/epoxy composite. In this method a series of tensile tests (with the fibers oriented at  $0^\circ$ ,  $10^\circ$ , and  $90^\circ$  away from the loading axis) was performed to characterize the non-linear behavior of the unidirectional material for use in the flow rule developed by Hansen et al. [10]. A scalar hardening parameter, which is a coefficient in the associated flow rule, is defined as a function of the following linear functions (see Ref. [9] for details):

$$g_n(a_n) = (\text{coefficient})a_n \quad (2)$$

where  $n=3$  and  $4$ , and  $a_n$  are stress invariants with respect to the material symmetry. The coefficients of the linear functions were determined for each temperature using the experimental data from the off-axis tests.

The applied stresses and strains (measured from the load cell and strain gages, respectively) were transformed into the material coordinate system for the off-axis tensile tests in order to obtain stresses and strains with respect to the fiber orientation (Fig. 1). This information was used to calculate  $E_{11}$  and  $\nu_{12}$  from the  $0^\circ$  test;  $E_{22}$ ,  $\nu_{21}$ , and  $g_4(a_4)$  from the  $90^\circ$  test; and  $G_{12}$  and  $g_3(a_3)$  from the  $10^\circ$  test (see Ref. [9] for details on obtaining the elastic constants and plasticity parameters).

The effect of elevated temperatures on the elastic properties  $E_{11}$ ,  $E_{22}$ ,  $G_{12}$ ,  $\nu_{12}$ , and  $\nu_{21}$  is clearly demonstrated in Figs. 3–7, and the effect of temperature on the plastic parameters  $g_3(a_3)$  and  $g_4(a_4)$  can be seen in Fig. 8(a) and (b). The results presented in Figs. 3–8 are also listed in Tables 1 and 2.

The transverse tensile and shear strength data vs temperature obtained from the specimens machined by waterjet cutting are presented in Figs. 9 and 10. The strength data are shown together in Table 3 with the strengths obtained from the specimens cut by the band saw and surface grinding technique. It is indicated in Figs. 9 and 10 and Table 3 how the  $10^\circ$  and  $90^\circ$  specimens failed during testing. The failure of the specimens either in the grip or gage sections is distinguished. For the  $90^\circ$  tests, the distinction between the failure either within the gage section or grips was obvious. For the  $10^\circ$  off-axis tests, failure in the grips was assumed when the failure surface intersected the front edge of the aluminum tabs. Essentially, the entire failure surface had to be completely located in the gage section to be considered as failure in the gage section.

It can be seen that the effect of temperature on the longitudinal modulus  $E_{11}$  (Fig. 3) is rather weak, however noticeable. Despite considerable scatter in the data,

Table 1  
Elastic properties of the unidirectional carbon/PMR-15 composite material

Temperature [°C (°F)]	$E_{11}$ (GPa)	$E_{22}$ (GPa)	$G_{12}$ (GPa)	$\nu_{12}$	$\nu_{21}$
21 (70)	137.8	9.6	5.3	0.40	0.06
	134.7	9.2	7.8	0.41	0.03
	134.4			0.42	
121 (250)	137.4	7.0	3.8	0.51	0.05
	137.7	7.4	3.5	0.42	0.05
204 (400)	135.8	6.9	2.4	0.38	0.06
	126.7	7.5	2.9	0.42	0.08
	125.8			0.43	
260 (500)	136.7	5.9	1.2	0.42	0.04
	120.8	5.0	2.2	0.48	
	119.9			0.54	
316 (600)	120.0	7.0	0.6	0.43	0.06
	123.0	6.0	2.4	0.58	0.07

Table 2  
Coefficients of the plasticity parameters of the unidirectional carbon/PMR-15 composite material

Temperature [°C (°F)]	$g_4(a_4)$	$g_3(a_3)$
21 (70)	$(1.0e-11)a_4$	$(2.0e-9)a_3$
	$(1.0e-11)a_4$	$(9.0e-10)a_3$
121 (250)	$(1.0e-9)a_4$	$(1.2e-9)a_3$
	$(3.0e-10)a_4$	$(2.0e-9)a_3$
204 (400)	$(1.0e-8)a_4$	$(1.1e-8)a_3$
	$(1.5e-8)a_4$	$(6.0e-9)a_3$
260 (500)	$(2.0e-8)a_4$	$(1.0e-7)a_3$
	$(4.0e-8)a_4$	$(2.0e-8)a_3$
316 (600)	$(1.0e-7)a_4$	$(3.0e-6)a_3$
	$(4.0e-8)a_4$	$(3.0e-8)a_3$

the general trend is clear with  $E_{11}$  decreasing almost linearly with temperature if the average data points are considered. The fitting parameters from Eq. (1) are presented in Fig. 3. The average tensile modulus  $E_{11}$  decreased from 135.6 GPa at room temperature to 121.5 GPa at 316°C (600°F) (approx. 10% reduction with respect to room temperature).

The effect of elevated temperatures on the transverse tensile ( $E_{22}$ ) and shear ( $G_{12}$ ) moduli is considerably stronger (see Figs. 4 and 5). In these two cases, the amount

of scatter is less than in the case of  $E_{11}$  vs temperature. Considering the average data points, the relation between  $E_{22}$  and temperature is almost linear whereas the dependence of  $G_{12}$  upon temperature is significantly non-linear. The fitting parameters for the  $E_{22}(T)$  and  $G_{12}(T)$  curves from Eq. (1) are presented in Figs. 4 and 5, respectively.

Regarding the Poisson ratios ( $\nu_{12}$  and  $\nu_{21}$ ), no clear evidence of an effect of temperature on these two properties is observed (see Figs. 6 and 7). Therefore, it is assumed

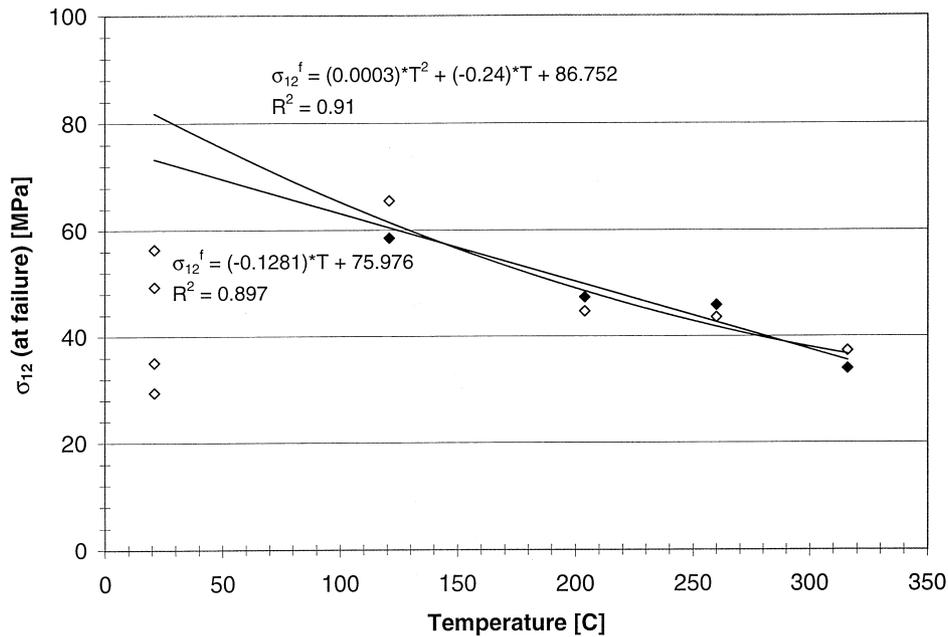


Fig. 9.  $\sigma_{12}$  (at failure) vs temperature. Open and solid diamonds indicate failure in grip and gage sections, respectively.

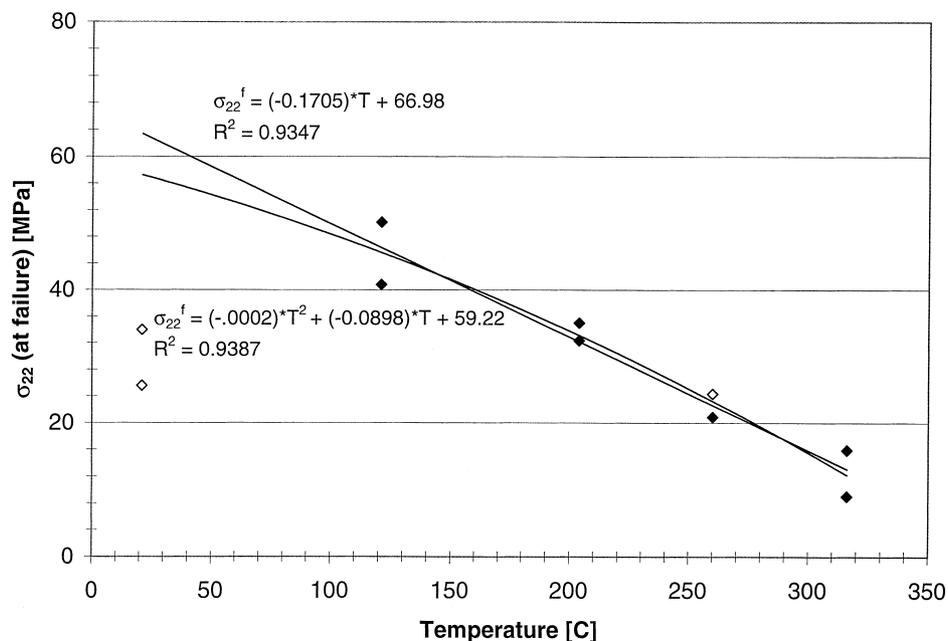


Fig. 10.  $\sigma_{22}$  (at failure) vs temperature. Open and solid diamonds indicate failure in grip and gage sections, respectively.

that they are independent of temperature. The average value of  $\nu_{12}$  and  $\nu_{21}$  (for all temperatures) is 0.45 and 0.057, respectively.

The temperature dependence of the plasticity parameters  $g_3(a_3)$  and  $g_4(a_4)$ , illustrated in Fig. 8(a) and (b), clearly indicate that the non-linear behavior of the composite significantly increases as a function of temperature. The best curve fits for these two sets of data are indicated in the figures. The logarithmic scales in Fig. 8(a) and (b) emphasize the large increase in the non-linear behavior as a function of temperature.

Both the transverse tensile and shear strengths decrease significantly with temperature (see Figs. 9 and 10). Since a significant number of specimens failed in the grip section, the shear strength results from these tests are indicated in Figs. 9 and 10 with white diamonds, whereas the results obtained from the specimens which failed in the gage section are marked by black diamonds. Not a single specimen failed in the gage section at room temperature. Therefore, the shear and transverse tensile strength values at room temperature from specimens without grip effects are not available. It can be clearly observed in Figs. 9 and 10 that the difference between the strength data obtained from the specimens which failed in the gage and grip sections diminishes with an increase in temperature. This statement applies to both  $\sigma_{22}$  and  $\sigma_{12}$ . Therefore, the strength values at elevated temperatures are independent of failure location. This effect can be clearly seen especially in the case of  $\sigma_{12}$ . Consequently, it is stipulated that the effect of stress concentration due to gripping vanishes at elevated temperatures when testing unidirectional graphite/polyimide composites.

Since the correct shear and transverse tensile strength data at room temperature (without grip effects) were not available, an attempt was made to determine these values by extrapolating from the elevated temperature results. Since a general trend is not clear, both linear and quadratic curves were fitted to the elevated temperature strength data (121–316°C). The curve fits are shown in Figs. 9 and 10 with the fitting parameters and the  $R^2$  value. If linear curve fits are considered, the tensile and shear strengths at room temperature should be 63.4 and 73.3 MPa, respectively. From quadratic fits, these values are 57.2 and 81.8 MPa. Most likely, the actual values could be somewhere between these two estimates.

The quality of machining should not affect the elastic and plastic property measurements. However, it is clear that the specimen preparation process does have a dramatic effect on the shear and transverse tensile strength determination from the off-axis tests performed on the unidirectional carbon/PMR-15 composite. As can be noticed in the data presented in Table 3 for tests at all temperatures, the 10° and 90° off-axis tensile tests performed on the mechanically cut specimens (band saw

Table 3  
Strength properties of the unidirectional carbon/PMR-15 composite material

Temperature [°C (°F)]	$\sigma_{22}$ (MPa)	$\sigma_{12}$ (MPa)
21(70)	12.1 <sup>a</sup> — grip	33.7 <sup>a</sup> — grip
	4.9 <sup>a</sup> — grip	38.4 <sup>a</sup> — grip
	25.6 — grip	49.3 — grip
	34.0 — grip	29.5 — grip
121(250)	40.8 — gage	65.6 — grip
	50.2 — gage	58.6 — gage
204(400)	25.8 <sup>a</sup> — grip	37.9 <sup>a</sup> — gage
	24.1 <sup>a</sup> — gage	40.0 <sup>a</sup> — grip
	35.0 — gage	47.4 — gage
	32.4 — gage	44.75 — grip
260(500)	4.0 <sup>a</sup> — gage	31.3 <sup>a</sup> — gage
	22.3 <sup>a</sup> — gage	28.5 <sup>a</sup> — grip
	20.9 — gage	43.6 — grip
	24.4 — grip	45.9 — gage
316(600)	9.04 — gage	37.3 — grip
	15.9 — gage	34.0 — gage

<sup>a</sup> Machined with band saw followed by grinding.

and subsequent surface grinding) consistently yielded significantly lower shear and transverse tensile strength results compared to the data from the specimens cut by the waterjet technique. Therefore, caution should be observed regarding the quality of the specimen preparation process when performing 10° and 90° off-axis tension experiments for the shear and transverse tensile strength determination of unidirectional brittle polymer matrix composites at room and elevated temperatures.

## 5. Conclusions

1. The elastic and plastic properties of a unidirectional graphite/polyimide composite are significantly affected by elevated temperatures. Major reductions in  $E_{22}$  and  $G_{12}$  were observed with temperature in this research. The effect of temperature on  $E_{11}$  was less pronounced, however noticeable. The plastic parameters  $g_3(a_3)$  and  $g_4(a_4)$  increased dramatically with an increase in temperature.
2. The shear and transverse tensile strength properties were also significantly reduced with temperature. At elevated temperatures, the specimens failed either in the grip or gage sections for both 10° and 90° off-axis tests even if the best type of specimen preparation process was employed. However, the type of failure did not have any noticeable effect on the shear and tensile strength values at elevated temperatures. At room temperature, all 10° and 90° off-axis specimens failed in the grip areas. In

order to estimate the shear and transverse tensile strengths of the composite at room temperature, these values were extrapolated from the elevated temperature data.

3. The effect of the specimen preparation process on the shear and transverse tensile strength determination for unidirectional graphite/polyimide composites was found to be very strong. The strengths obtained from the specimens cut by waterjet were significantly higher at room and elevated temperatures in comparison with the strength values from the specimens machined mechanically.

### Acknowledgements

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