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THE EFFECT OF EXTREME TEMPERATURES ON THE  
ELASTIC PROPERTIES AND FRACTURE BEHAVIOR  
OF GRAPHITE/POLYIMIDE COMPOSITES

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**ABSTRACT**

The influence of elevated, room and cryogenic temperatures on the elastic moduli and fracture strengths of two graphite/polyimide composites was studied. The fracture behavior of notched specimens was modeled using an average stress failure criterion.

**INTRODUCTION**

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. 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 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 two graphite/polyimide composites 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 materials studied in this program, both notched and unnotched tension tests were conducted. The data reported in this paper are the results of the second phase of a two-phase program. The first phase covered tests on notched specimens that were 102 mm (4 in) wide or smaller, and has been previously reported

(Garber, Morris and Everett, 1982). The second phase of the program investigated the fracture behavior of test specimens that were up to 305 mm (12 in) wide. In addition, the second phase considered graphite/polyimide composites with two matrix materials.

### MATERIAL AND SPECIMENS

The material used for this study consisted of Celion 6000 graphite fibers in a matrix of PMR-15 or NR-150B2 polyimide. Specimens were cut from panels of  $[\pm 45]_{2S}$  and  $[0/45/90/-45]_S$  laminates. Ply thicknesses in the cured laminates ranged between 0.127 mm (0.005 in) and 0.152 mm (0.006 in). Fiber volume fractions varied from 54 to 69 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 51 mm (2 in) to 305 mm (12 in). Fracture specimens had center circular holes that ranged between 1.6 mm (1/16 in) and 76.2 mm (3 in) in diameter.

### TEST PROCEDURES

Tension and fracture tests were performed at 316, 24, and  $-157^{\circ}\text{C}$  (600, 75,  $-250^{\circ}\text{F}$ ). An environmental chamber, which was small enough to permit the specimens to be gripped outside the chamber, was used to control the test temperature. The maximum variation in temperature was  $\pm 5^{\circ}\text{C}$  ( $\pm 9^{\circ}\text{F}$ ) at the extreme temperatures. Heat was provided by electrical resistance elements, and cooling was provided by liquid nitrogen.

All tests were performed at a constant cross-head speed. For tension tests the cross-head speed was 0.008 mm/s (0.002 in/min), and for fracture tests it was 0.002 mm/s (0.005 in/min). For all experiments the test section within the environmental chamber was 203 mm (8 in). It took approximately 1 h

to reach 316°C (600°F), and 15 min to reach -157°C (-250°F). Specimens were allowed to soak at temperature for 10 to 15 min before being tested. It was assumed that mechanical properties were not affected by the total time in the environmental chamber (time to reach temperature plus soak time plus testing time). This assumption was based on previous results (Shyprykevich and Wolter, 1982) where it was found that the failure stress of a  $[0/\pm 45/90]_S$  laminate of AS/3501-5A graphite/epoxy was independent of soak time for the times encountered in the work reported herein. Similar results were reported (Rummler and Clark, 1979) for  $[0]_6$  and  $[0/\pm 45]_S$  laminates of HTS/710 graphite/polyimide.

Tension modulus values were determined from strain gage measurements. The accuracy and repeatability of the gages for determining elastic modulus has been established (Chapman, 1979). It was generally not possible to measure ultimate strains due to gage failures. Strains were not measured for the fracture tests.

## RESULTS AND DISCUSSION

### Unnotched Tension Tests

The results of the unnotched tension tests are shown in Figs. 1 and 2. Three replicate tests were performed at each temperature for each laminate. The results shown in the figures are average values of the replicate tests. It is seen from Figs. 1 and 2 that both axial tensile modulus and tensile strength are nearly independent of temperature for the fiber-dominated laminate, except for the strength of the laminate with the NR-150B2 matrix.

The situation is quite different for the matrix-dominated laminate. Figures 1 and 2 reveal that both modulus and strength decrease with increasing temperature, except for the strength of the laminate with the NR-150B2 matrix.

These figures also show that the modulus (initial slope of a stress-strain curve) is practically independent of the matrix material, but the strength appears to be more dependent upon the type of matrix.

### Notched Tension Tests

The effect of temperature on the notched strength of the  $[0/45/90/-45]_5$  quasi-isotropic laminate is shown in Fig. 3. For the laminate with the PMR-15 matrix, and for a given hole size, the effect of temperature is practically insignificant. A similar trend is noted for the unnotched laminate, Fig. 2.

On the other hand, the notched strength of the quasi-isotropic laminate with the NR-150B2 matrix is practically constant between room and elevated temperature, but the strength increases at cryogenic temperature. A similar trend is noted from Fig. 2 for the strength of the unnotched laminate. Regardless of the matrix material, Fig. 3 reveals that the effect of hole size is significant.

### Failure Model

A failure model was selected that would explain the effect of hole size and also predict the notched strength. The model selected was the average stress failure criterion (Whitney and Nuismer, 1974; Nuismer and Whitney, 1975). This model states that failure of a notched specimen will occur when the average stress over some distance ahead of the notch,  $a_0$ , equals the unnotched tensile strength. For a quasi-isotropic laminate this failure criterion may be written

$$\frac{\sigma_N^\infty}{\sigma_0} = \frac{2(1 - \xi)}{2 - \xi^2 - \xi^4} \quad (1)$$

where  $\xi = R/(R + a_0)$ ,  $\sigma_N^\infty$  is the failure stress for a notched infinite width plate,  $\sigma_0$  is the unnotched tensile strength, and  $R$  is hole radius. The infinite width plate notched strength is found by multiplying the experimentally determined notched strength for a finite width plate by a finite width correction factor (Nuismer and Whitney, 1975). The distance  $a_0$  is called a characteristic length.

Using Eqn. (1), the unnotched strength, the notched strength, and the finite width correction factor, a value of  $a_0 = 5.1$  mm (0.20 in) was found to model the fracture behavior of the C6000/PMR-15 quasi-isotropic laminate. As can be seen from Fig. 4, one value of  $a_0$  models the data at the three temperatures.

Using Eqn. (1) with  $a_0 = 5.1$  mm (0.20 in) failure stresses were predicted for plates that had widths ( $W$ ) of 102 mm (4 in), 203 mm (8 in) and 305 mm (12 in). The plates were tested at temperatures ( $T$ ) of 316°C (600°F) and 24°C (75°F). The results are shown in Table 1, where the predicted values are compared with experimental results. Good agreement was found.

A comparison between notched strengths for the C6000/PMR-15 and C6000/NR-150B2 quasi-isotropic laminates is shown in Fig. 5. Some of the specimens with the NR-150B2 matrix failed at the grips of the testing machine, while others failed between the hole and grips. Thus, the data for this laminate should be viewed with caution. More data are needed to establish a definite trend, and to determine if the average stress failure criterion will model the notched behavior.



TABLE 1 Comparison of Failure Stresses

T °C(°F)	W mm(in)	R mm(in)	$\sigma_N$ (Eqn.1) MPa(Ksi)	$\sigma_N$ (Exp.) MPa(Ksi)	Difference %
316(600)	102 (4)	12.7(0.50)	180(26.2)	199(28.8)	9.0
		4.8(0.19)	250(36.3)	281(40.7)	10.8
		2.4(0.09)	301(43.7)	297(43.1)	-1.4
24(75)	102 (4)	12.7(0.50)	159(23.1)	175(25.4)	9.1
		4.8(0.19)	220(32.0)	234(34.0)	5.9
		2.4(0.09)	265(38.5)	274(39.7)	3.0
	203 (8)	12.7(0.50)	244(35.4)	254(36.8)	3.8
		25.4(1.00)	201(29.2)	186(27.0)	-8.1
		38.1(1.50)	190(27.5)	186(27.0)	-1.9

#### SUMMARY AND CONCLUSIONS

It has been shown that elastic modulus and unnotched tensile strength for the fiber-dominated laminate are not highly temperature dependent. On the other hand, the elastic moduli and tensile strengths of the matrix-dominated laminates are quite sensitive to changes in temperature.

Notched tensile strength is dependent on hole size, but is practically independent of temperature for the C6000/PMR-15 laminate. The notched strength of the C6000/NR-150B2 laminate is also dependent on hole size, and appears to be temperature dependent. However, some of the specimens failed away from the hole, and the data should be viewed with caution.

The average stress failure criterion gives reasonable predictions of notched strength for the C6000/PMR-15 quasi-isotropic laminate. Due to uncertainty in some of the data for the C6000/NR-150B2 laminate, no attempt was made to model the data. However, the data tend to indicate that the characteristic length,  $a_0$ , may be temperature dependent.

### ACKNOWLEDGEMENT

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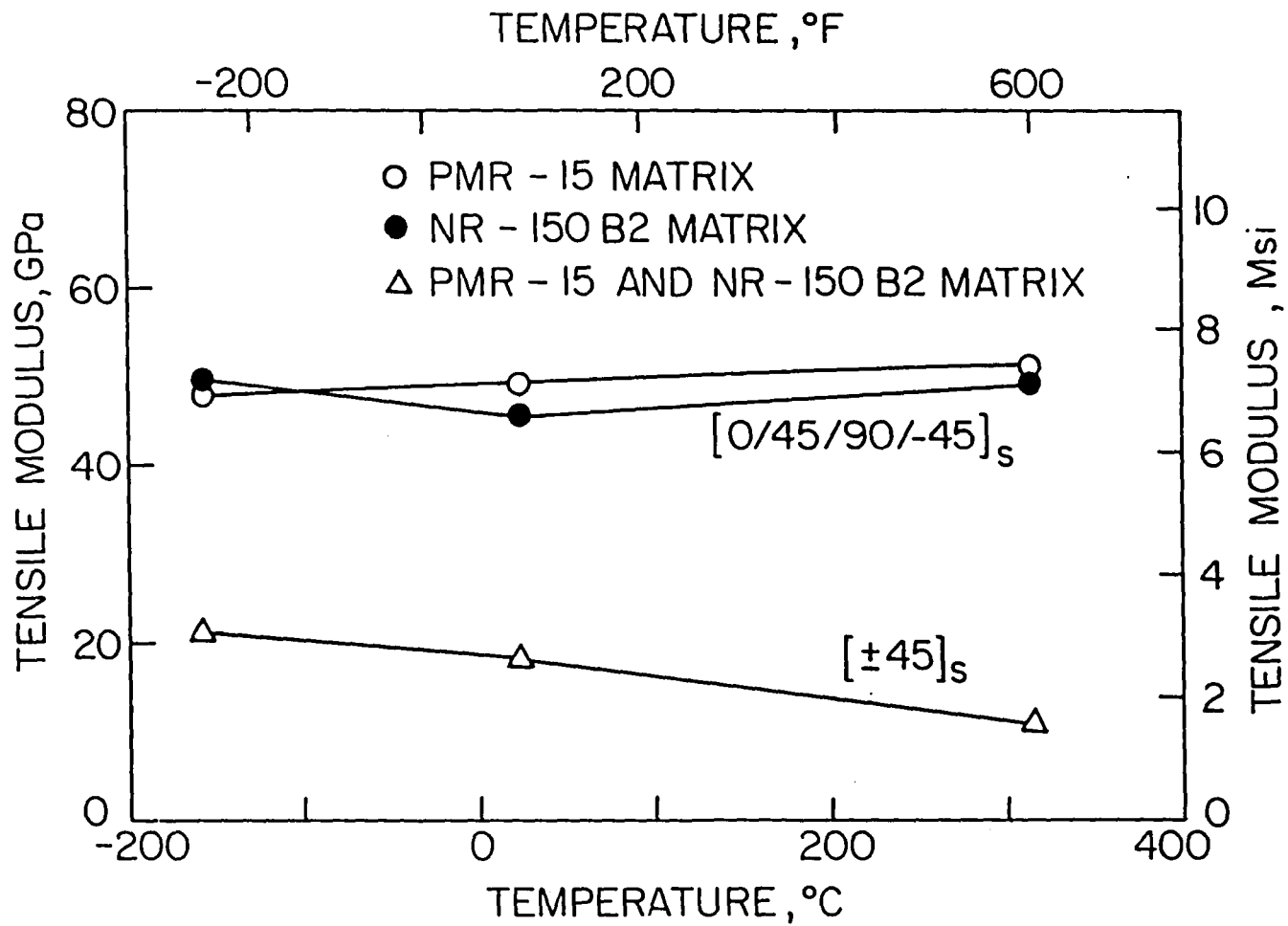


Figure 1. Axial Tensile Modulus versus Temperature for Fiber and Matrix-Dominated Laminates

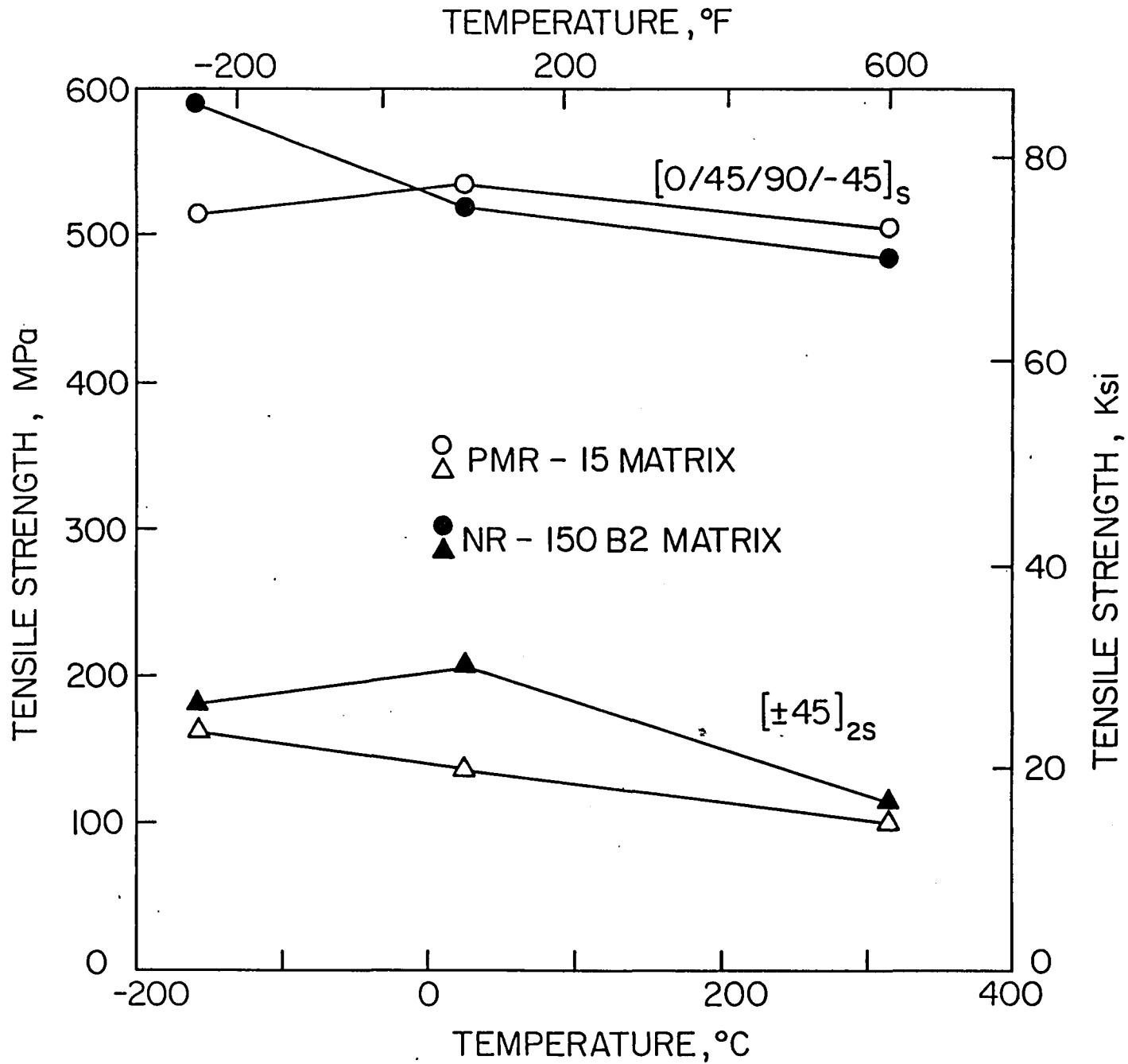


Figure 2. Tensile Strength versus Temperature for Fiber and Matrix-Dominated Laminates



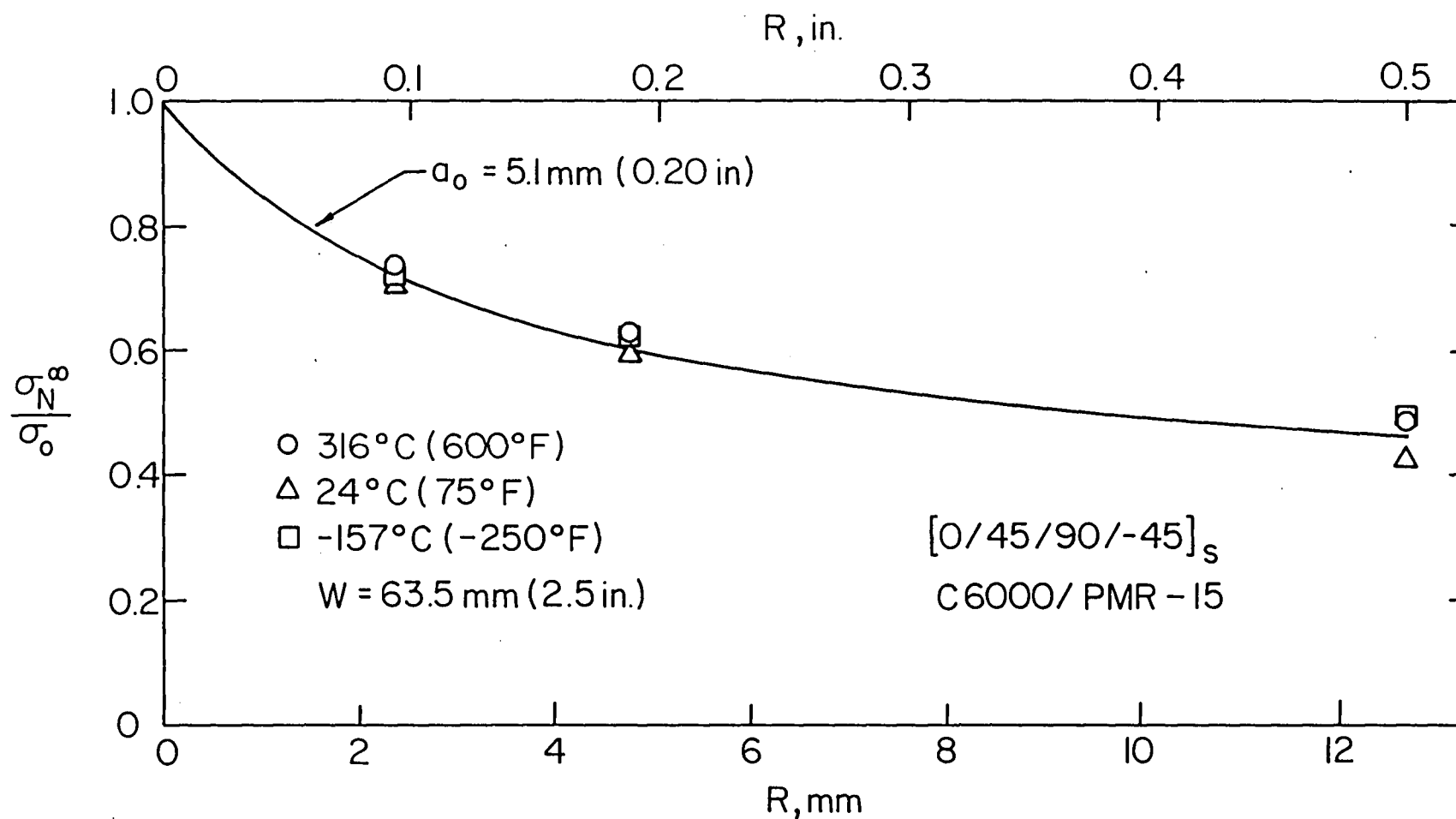


Figure 4. Comparison of Experimental and Predicted Strengths for the C6000/PMR-15 Laminate

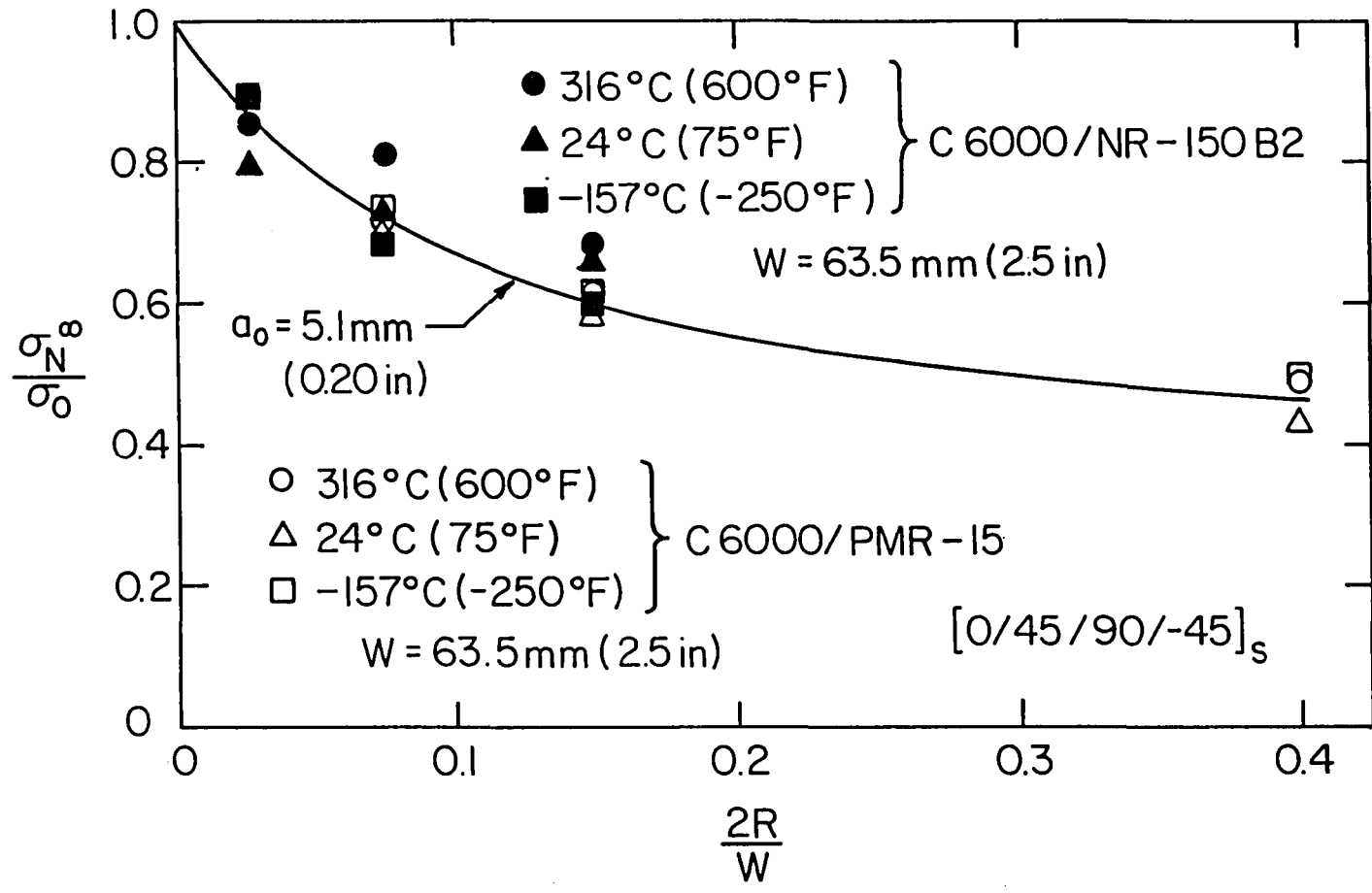


Figure 5. Comparison of Experimental and Predicted Strengths for the C6000/PMR-15 and C6000/NR-160B2 Laminate

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