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**OAK RIDGE  
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**COMPRESSIVE CREEP RESPONSE OF  
T1000G/RS-14 GRAPHITE/POLYCYANATE  
COMPOSITE MATERIALS**

LOCKHEED MARTIN 

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# TABLE OF CONTENTS

LIST OF TABLES .....	iii
LIST OF FIGURES .....	iv
LIST OF ACRONYMS .....	v
ABSTRACT .....	vi
INTRODUCTION .....	vii
1. RS-14 RESIN COMPRESSIVE RESPONSE .....	1
1.1 Resin Specimen Fabrication .....	1
1.2 Resin Compressive Strength .....	2
1.3 Resin Compressive Creep .....	3
2. T1000G/RS-14 COMPOSITE TRANSVERSE COMPRESSIVE RESPONSE .....	14
2.1 Composite Specimen Fabrication .....	14
2.2 Composite Transverse Compressive Strength .....	15
2.3 Composite Transverse Compressive Creep .....	16
3. DISCUSSION OF RESULTS .....	29
4. RECOMMENDATIONS .....	31
5. ACKNOWLEDGMENTS .....	32
6. REFERENCES .....	33

## LIST OF TABLES

Table 1.2-1. Resin compressive strength. ....	6
Table 1.3-1. Resin compressive creep test matrix. ....	9
Table 1.3-2. Results for RS-14 resin compressive creep tests. ....	10
Table 2.2-1. Results from composite transverse compressive strength tests. ....	19
Table 2.3-1. Test matrix for composite transverse compressive creep tests. ....	21
Table 2.3-2. Results for the T1000G/RS-14 composite transverse compressive creep tests ...	22

## LIST OF FIGURES

Fig. 1.2-1. Stress-strain responses for RS-14 resin tested at ambient temperatures. . . . .	7
Fig. 1.2-2. Stress-strain response for RS-14 resin tested at 275°F. . . . .	8
Fig. 1.3-1. Effect of process and test environments on RS-14 resin compressive creep. . . . .	11
Fig. 1.3-2. Effect of post-cure temperature on RS-14 resin compressive creep. . . . .	12
Fig. 1.3-3. Effect of test temperature on RS-14 resin compressive creep. . . . .	13
Fig. 2.1-1. Composite transverse compression test specimen geometries. . . . .	18
Fig. 2.2-1. Typical stress-strain curves for composite transverse compression. . . . .	20
Fig. 2.3-1. Total strains for composite transverse compressive creep tests. . . . .	23
Fig. 2.3-2. Effect of test temperature on composite transverse compressive creep. . . . .	24
Fig. 2.3-3. Effect of applied stress level on composite transverse compressive creep. . . . .	25
Fig. 2.3-4. Magnitude of creep strains for composite transverse compressive creep. . . . .	26
Fig. 2.3-5. Nonlinear least-squares regression fit for power law parameters. . . . .	27
Fig. 2.3-6. Ten-year prediction based on power law model. . . . .	28

## LIST OF ACRONYMS

ASTM	American Society of Testing and Materials
CV	Coefficient of Variation
ID	Inner Diameter
psi	pounds per Square Inch
ksi	$10^3$ pounds per Square Inch
Msi	$10^6$ pounds per Square Inch
OD	Outer Diameter

## ABSTRACT

The response of a T1000G/RS-14 graphite/polycyanate composite material system to transverse compressive loads is quantified via experimentation. The primary objective of the work was to quantify the effects of process environment and test environment on the T1000G/RS-14 compressive creep response. Tests were conducted on both the neat resin and the composite material system. In addition to the creep tests, static compressive strength tests were conducted to define the stress-strain response.

The creep behavior for the RS-14 resin was quantified by conducting a series of tests to study the effects of different process environments (air and nitrogen), different cure temperatures, and different test environments (air and vacuum). The combined effect on the RS-14 resin compressive creep of processing in nitrogen and testing under vacuum versus processing in air and testing in air was a 47% decrease in the creep strain after 2177 hr. The test environment appeared to have a greater effect on the resin creep than the process environment.

Following the conclusion of the resin creep tests, composite transverse compressive creep tests were conducted. The composite creep test cylinder was post-cured in a nitrogen environment prior to machining test specimens and all tests were conducted in a vacuum environment. The series of tests investigated the effects of initial stress level and test temperature on the creep behavior. At the end of the 2000-hr tests at 275°F on specimens stressed at 10,000 psi, the nitrogen-processed and vacuum-tested conditions reduced the composite transverse compressive creep strain by 19% compared to processing in air and testing in air. The effects of process and test environment on the creep behavior are not as great for the composite system as they were for the neat resin, primarily because of the low resin content in the composite material system.

At the 275°F test temperature there was a significant increase in the composite transverse compressive creep strain between the 6000-psi and 10,000-psi stress levels. Despite the reduction in creep strains due to processing in a nitrogen environment, dimensional stability of the T1000G/RS-14 composite at high transverse compressive stress levels and high temperatures may still be an issue when selecting this system for future applications.

## INTRODUCTION

The overall objective of this effort was to evaluate and characterize an advanced composite material system that could be used in higher temperature applications that exceed the capability of current epoxy resin systems. Two examples are future aerospace and energy storage applications. The primary structural component under consideration is a composite ring subjected to rotational body forces, where the fiber-direction strength is a more critical design parameter than stiffness. Also, the dimensional stability of the composite material system is a critical parameter. The results from these tests and corresponding data analyses were aimed at reducing the risk of selecting a composite material for possible use in future designs.

The quality of composite structural components required for this type of application is best achieved by using the wet filament winding process. The advantages of wet-filament-winding are that it permits instantaneous and in-situ ply-by-ply consolidation during the lay-up of the composite. Resin bleed-out is almost immediate with the application of each layer. These process characteristics lead to reduced fiber wrinkling and voids in thick constructions, particularly when compared to composites made via the pre-preg lay-up fabrication method in which resin bleed-out and ply consolidation occur after the total thickness has been laid up. Another advantage of wet-filament-winding is the capability to achieve very high fiber volume fractions which translates into high fiber-direction composite tensile strengths. The disadvantages of the wet-filament-winding process are typically low transverse shear strengths and a heavy dependence on the selected resin.

The material system that was selected for this investigation was an ultra-high-strength carbon fiber with a polycyanate resin. The highest strength carbon fiber currently on the market is produced by Toray Industries and is designated by T1000G. The reported strength for this fiber ranges from 900-950 ksi. The cyanate ester class of thermoset matrix resins, also called polycyanates, have inherent qualities that make them an excellent choice for a material system that will be exposed to a space or vacuum environment. In general, polycyanate resins have low moisture absorption, good fiber wet-out properties, low out-gassing, good radiation stability, good resistance to microcracking, and superior heat resistance. Also, the polycyanate resins are amenable to the wet-filament-winding

process with only slight modifications to existing processes for epoxy resins. The selected polycyanate resin was RS-14 produced by YLA, Inc.

The details of the fabrication and wet-filament winding process for the T1000G/RS-14 system were described by Frame and Starbuck<sup>1</sup>, and Frame<sup>2</sup>. Also, the experimental results for the static strengths and fiber-direction performance were discussed by Frame<sup>2</sup> and Starbuck<sup>3</sup>. A preliminary investigation of the dimensional stability of T1000G/RS-14, based on the results from transverse compressive creep tests, was conducted by Starbuck<sup>3</sup>. This work was expanded to study the effects of processing and testing environments on the transverse compressive creep response and the results are documented in this report.

## 1. RS-14 RESIN COMPRESSIVE RESPONSE

The primary objective of this study was to quantify the effects of process environment and test environment on the T1000G/RS-14 compressive creep response. Tests were conducted on both the neat resin and the composite material system. In addition to the creep tests, static compressive strength tests were conducted to define the stress-strain response. The results from the neat resin tests are discussed in this section.

The compressive strength and creep behavior for the RS-14 resin was quantified by conducting a series of tests to study the effects of different process environments, different cure temperatures, and different test environments. The different process environments were air and nitrogen. The different test environments were air and vacuum. The initial creep test matrix was redefined as a result of difficulty in the manufacturing of the test specimens. This led to investigating the effect of post-cure temperature by testing specimens that had been cured at 470°F, 520°F, and a third temperature that was in between these two temperatures.

### 1.1 Resin Specimen Fabrication

The test specimen geometry was a cast rod having a nominal 0.5 in. diameter and a length of 1.5 in. The slenderness ratio for the specimen, i.e., the ratio of the length to least radius of gyration, was equal to 12:1. For a uniform circular cross-section, the radius of gyration is 0.250 times the diameter. This was a preferred specimen size according to ASTM D695M-85, Standard Test Method for Compressive Properties of Rigid Plastics, which recommends a slenderness ratio in the range of 11:1 to 16:1.

The specimens were cast in a glass test tube treated with a mold release agent to facilitate removal after curing. Any entrapped air caused by pouring the resin into the test tube was removed by centrifuging. The specimens were pre-cured at 280°F at a ramp rate of 2°F/min and a hold time of 3 hr. The specimens were returned to ambient temperature, removed from the test tubes, and then post-cured. The ramp rate for the post-cure was 3°F/min with a hold time of 4 hr. For specimens cured in the nitrogen environment, there was a 3- to 4-hr purge time prior to initiating any cure cycle.

After the specimens were post-cured, they were machined to length with the end faces being flat and parallel.

The planned manufacturing process was to utilize an autoclave system without the pressure cycle for curing the specimens. The autoclave was a convenient method for controlling both the temperature and environment. The first batch of specimens were cured in the air environment at a post-cure temperature of 520°F. The second batch of specimens was to be identical to the first but with a nitrogen environment. However, during the post-cure cycle the autoclave heaters malfunctioned and the specimens were only cured at 470°F. It was determined that the autoclave heaters needed to be replaced and an alternative curing process was required to maintain schedule. Consequently, a vacuum furnace was used as a replacement for the autoclave. The cavity in the furnace was such that the specimens had to be separated into two batches. The two batches were then positioned at two different vertical locations in the furnace. After curing the specimens and removing them from the furnace it was observed that the two batches had different colors. It was postulated that this was caused by a thermal gradient in the furnace. The top batch, which was darker in color, was determined to have been cured at 520°F since this was where the control thermocouple was located. The bottom batch, which was lighter in color, was assumed to have been cured at a temperature less than 520°F but this was never verified via a separate temperature measurement.

## **1.2 Resin Compressive Strength**

Static compressive strength tests were conducted on specimens taken from each of the four groups of processing conditions. The specimens were strain gaged for determining the stress-strain response and modulus. The modulus was calculated using the initial linear portion of the stress-strain curve between 0.2-0.7% strain. Also, the yield stress and yield strain, calculated using the 0.2% offset method, were determined from the stress-strain curve. The results are shown in Table 1.2-1 for the ambient test temperature and for the elevated test temperature of 275°F. The stress-strain responses are provided in Fig. 1.2-1 and Fig. 1.2-2 for the ambient and 275°F test temperatures, respectively.

The results in Table 1.2-1 indicate there was a minimal affect of process environment on the ultimate compressive strength, yield strength, yield strain, and modulus of the RS-14 resin. The post-cure temperature had a greater effect in that the specimens cured at a lower temperature had slightly lower values for all these quantities. However, this effect was still less than 5%. This was not unexpected because the compressive strength of an unreinforced resin is not as sensitive to flaws as the tensile strength. Also, the compression test specimen geometry has a smaller surface to volume ratio than the tensile specimen geometry thereby making it less sensitive to the effects of carbamate formation and surface oxidation. The effects of test temperature were significant with a 38% reduction in strength and a 21% reduction in modulus for testing at 275°F compared to ambient.

### **1.3 Resin Compressive Creep**

The resin compressive creep tests were conducted in accordance with Table 1.3-1. All of the creep tests were conducted using 3000 psi as the initial applied stress level and 275°F as the test temperature. One additional test was conducted at 325°F to study the effect of test temperature on the measured creep response. The specimens were dead-weight loaded using 20:1 mechanical advantage lever-arm creep stands and special fixtures for applying the compressive loads. Each specimen was placed in an environmental chamber, pumped down to obtain the vacuum, heated to the prescribed test temperature, then pre-conditioned for 24 hr. Afterwards, a motorized weight pan was used to smoothly apply the load at a constant rate. As shown in Figs. 1.2-1 and 1.2-2, the 3000-psi applied stress level was within the initial linear portion of the stress-strain curve. A dummy specimen was placed in each environmental chamber to provide active thermal compensation for the strain gages. Thermocouples were used in each chamber to monitor the specimen temperature as well as the surrounding air temperature. A computerized data acquisition system was used to automatically collect the strain gage and thermocouple data. A sampling rate of once per second was used during loading and the first 2 hr. For the remainder of the test, data was taken every 30 min. The faster initial sampling rate was chosen to better quantify the initial creep response. The planned duration was 2000 hr with the actual test duration being equal to 2177 hr.

The measured total strain data was plotted as a function of time and then graphically reduced to determine the creep strains. The time (on the order of sec) required to apply the full load was recorded and the corresponding strain was defined as the initial strain to be subtracted from the total strain to obtain the creep strain. Generally, this under-predicts the creep strain as the material actually begins to creep during the load application. However, for studying the effects of process and test environments on the creep response this is an acceptable approach. In addition to the graphical interpretation of the data, a nonlinear least-squares regression algorithm was used to fit the data and determine the three parameters in the power law model. The algorithm was based on the Marquardt-Levenberg<sup>4</sup> method to fit the total strain versus time curve. For a material that behaves according to the power law, the log of the creep strains is a linear function of the log of time.

Prior to discussing the results, it should be noted that during the 2177-hr tests two specimens had anomalies in the data. The specimen cured at 470°F in nitrogen and tested under vacuum had a lead-wire short out that resulted in a loss of data. The wire was repaired and the specimen was returned to the prescribed test conditions. The recorded strain was inconsistent with the previous reading and this was explained by a change in lead-wire resistance. The difference in the gage readings was determined to be acceptable in terms of comparing the data with the other data sets. The specimen cured at 520°F in nitrogen and tested under vacuum experienced a loss of vacuum. A vacuum seal was replaced and the specimen was returned to the prescribed test conditions. However, when exposed to the air environment the specimen had a higher creep rate and did not return to the previous strain level. The creep rate prior to losing vacuum was used to correct the data and the adjusted strains were in excellent agreement with the earlier part of the data.

The results are summarized in Table 1.3-2 for both the graphical interpretation of the data and the power law parameters. General observations from the results follow. The test specimens run under vacuum had lower creep strains than the tests run in air. Also, as shown in Fig. 1.3-1, the vacuum-tested specimens exhibited lower creep rates than the air-tested specimens. Processing the specimens in a nitrogen environment produced lower creep strains than processing in air. The combined effect of processing in nitrogen and testing under vacuum versus processing in air and testing in air was a 47% decrease in the creep strain. The test environment appeared to have a greater effect on the resin creep than the process environment. The results also show lower creep

strains resulted from post-curing at a lower temperature (see Fig. 1.3-2). A possible explanation for this observation is a reduction in free volume produced by curing the polycyanate resin below the glass transition temperature. Finally, the effect of test temperature was very significant with the 325°F condition resulting in a 159% increase in creep strain over the 275°F condition (see Fig. 1.3-3).

The results for the nonlinear least-squares regression fit to the data indicated that the power law model was an excellent representation of the experimental data. In comparing the air versus vacuum test environment, the air-tested specimens had higher initial strains and time exponents but lower scale parameters. Conversely, for the air versus nitrogen process environment, the air cured specimens had higher scale parameters and lower time exponents. The effects of lowering the post-cure temperature were a decrease in the scale parameter and an increase in the time exponent. An increase in the test temperature resulted in an increase in all three of the power law parameters. One further observation from the power law parameters for the air-tested specimens was that the initial strains determined from the nonlinear data fits were larger than the strains determined graphically. Typically, this is not the case and the cause and/or explanation for it is unknown.

The results from the RS-14 resin compressive creep tests demonstrated that a reduction in creep strain is possible when the resin is processed in nitrogen and tested under vacuum. This will minimize or eliminate the formation of surface oxidation and minimize any other chemical aging effects that may be accelerated by exposing this resin system to an air environment at elevated temperatures.

Table 1.2-1. Resin compressive strength.

Fab *	Ambient temperature				275°F			
	Ultimate strength (psi)	Yield strength (psi)	Yield strain (%)	Modulus (psi)	Ultimate strength (psi)	Yield strength (psi)	Yield strain (%)	Modulus (psi)
Air/520	20,592	12,235	2.90	455,555	12,515	8915	2.62	367,044
N2/520T	20,925	12,130	2.92	451,655	13,027	8930	2.73	356,451
N2/520B	19,671	11,025	2.77	435,485	12,314	8325	2.74	336,364
N2/470	19,591	11,235	2.77	444,446	12,180	8255	2.49	354,641

\* Nomenclature:

Air/520 - post-cured in air at 520°F (autoclave)

N2/520T - post-cured in nitrogen at 520°F (top of vacuum furnace)

N2/520B - post-cured in nitrogen at &lt;520°F (bottom of vacuum furnace)

N2/470 - post-cured in nitrogen at 470°F (autoclave)

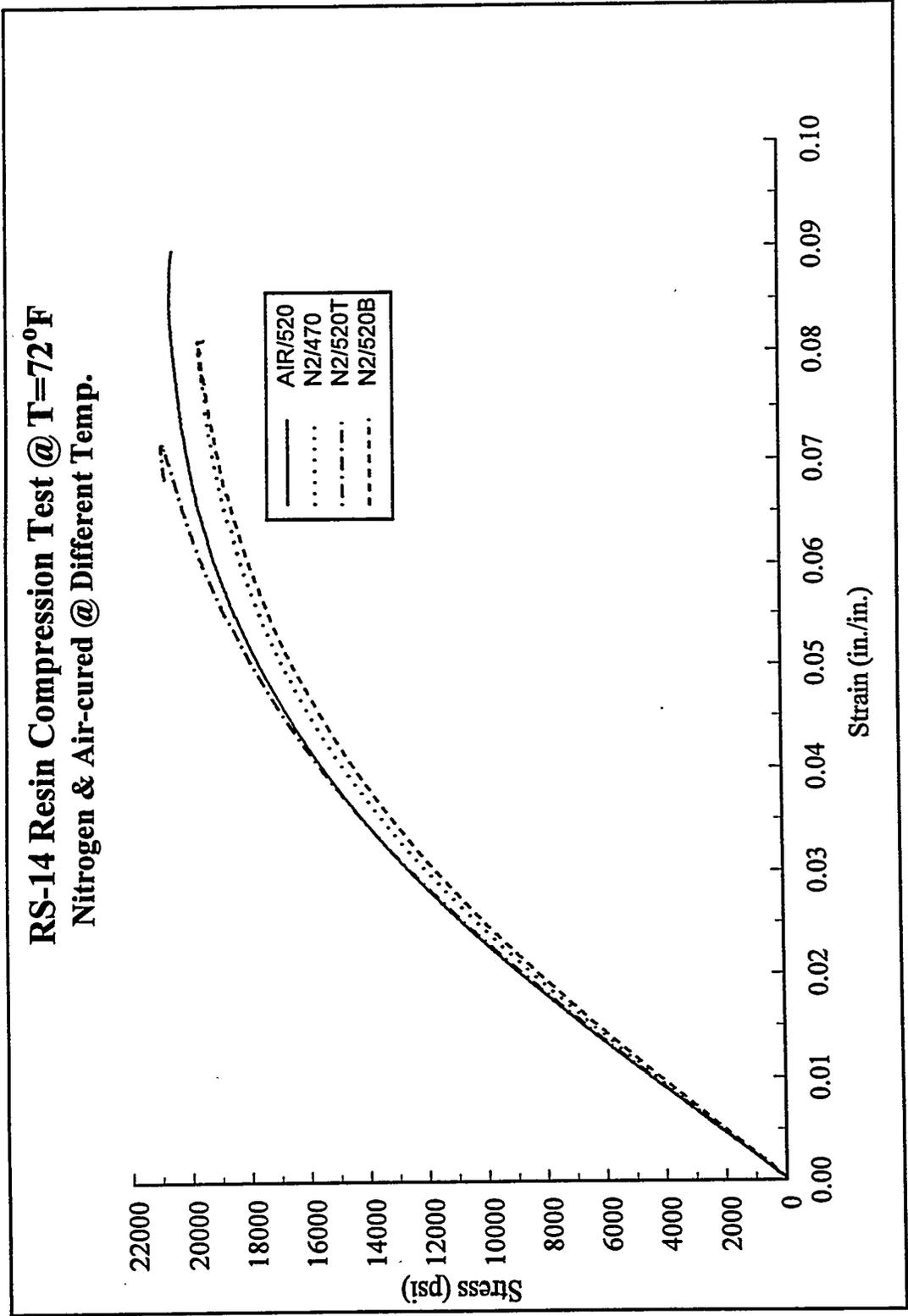


Fig. 1.2-1. Stress-strain responses for RS-14 resin tested at ambient temperatures.

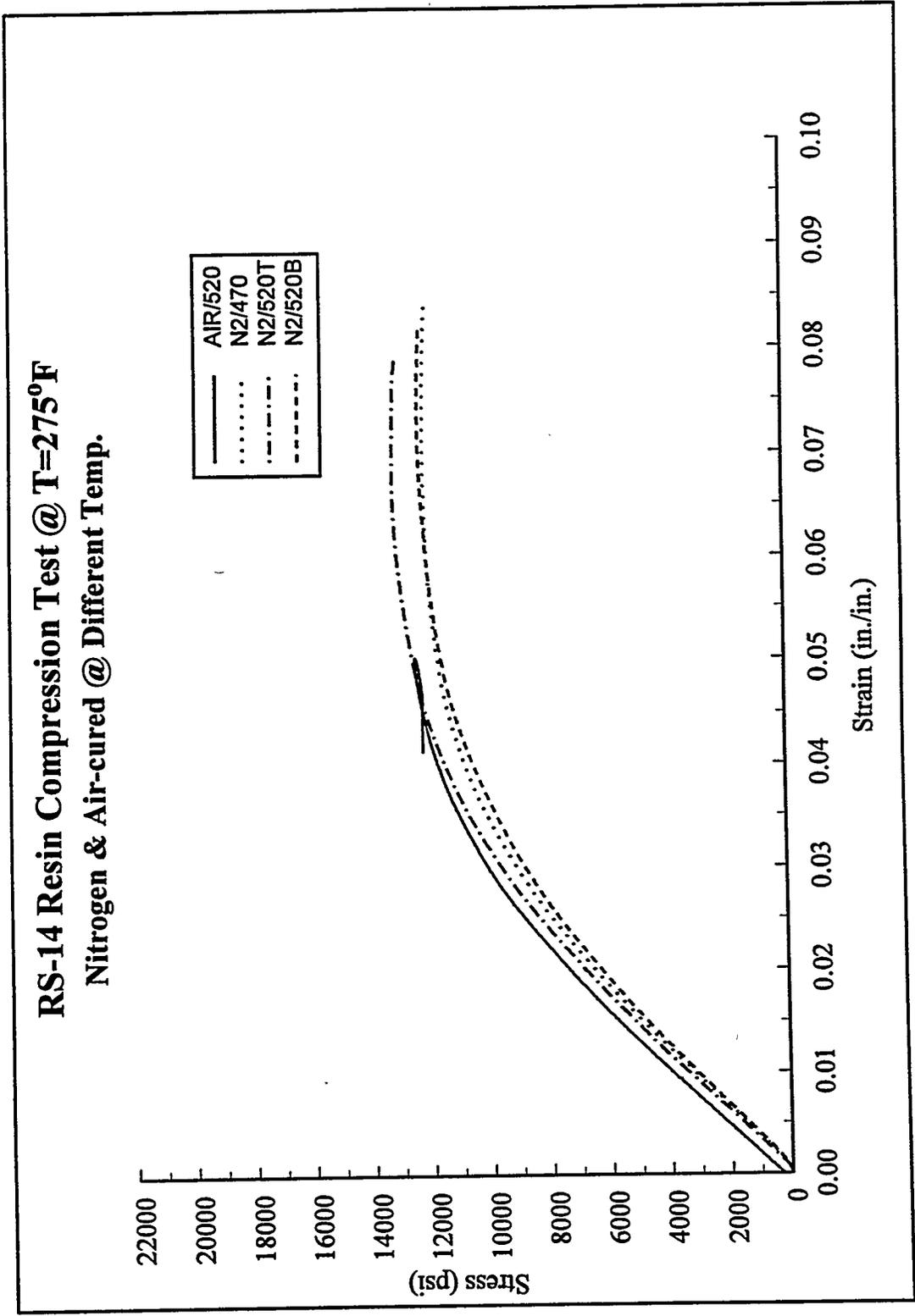


Fig. 1.2-2. Stress-strain response for RS-14 resin tested at 275 ° F.

**Table 1.3-1. Resin compressive creep test matrix.**

Test number	Process environment	Post-cure temperature (°F)	Test environment	Test temperature (°F)
5	Air	520	Air	275
B5	Nitrogen	520	Air	275
7	Air	520	Vacuum	275
B9	Nitrogen	520	Vacuum	275
A5	Nitrogen	470	Vacuum	275
C5	Nitrogen	< 520	Vacuum	275
B7	Nitrogen	520	Vacuum	325

Table 1.3-2. Results for RS-14 resin compressive creep tests

Spec. no.	Fab	Test temp. (°F)	Test environment	Graphical results			Power law results (nonlinear data fit) <sup>a</sup>			
				Initial strain (%)	Final strain (%)	Creep strain (%)	Initial strain (%)	Scale parameter	Time exponent	R
5	Air/520	275	Air	0.8548	1.8321	0.9773	0.8911	0.007270	0.3049	0.9995
7	Air/520	275	Vacuum	0.6861	1.3650	0.6789	0.4846	0.122578	0.1249	0.9990
B5	N2/520T	275	Air	0.8378	1.6133	0.7755	0.8770	0.004806	0.3154	0.9995
B9	N2/520T	275	Vacuum	0.7013	1.2168	0.5155	0.5637	0.081018	0.1327	0.9987
C5	N2/520B	275	Vacuum	0.7229	1.2211	0.4982	0.5981	0.074747	0.1341	0.9994
A5	N2/470	275	Vacuum	0.5992	1.0478	0.4486	0.5052	0.057570	0.1427	0.9987
B7	N2/520T	325	Vacuum	0.8680	2.2038	1.3358	0.6005	0.131797	0.1568	0.9991

<sup>a</sup>Power law equation:  $\epsilon(t) = \epsilon_0 + mt^n$

where:  $\epsilon(t)$  = total strain

$\epsilon_0$  = initial strain

m = scale parameter

n = time exponent

R = correlation coefficient

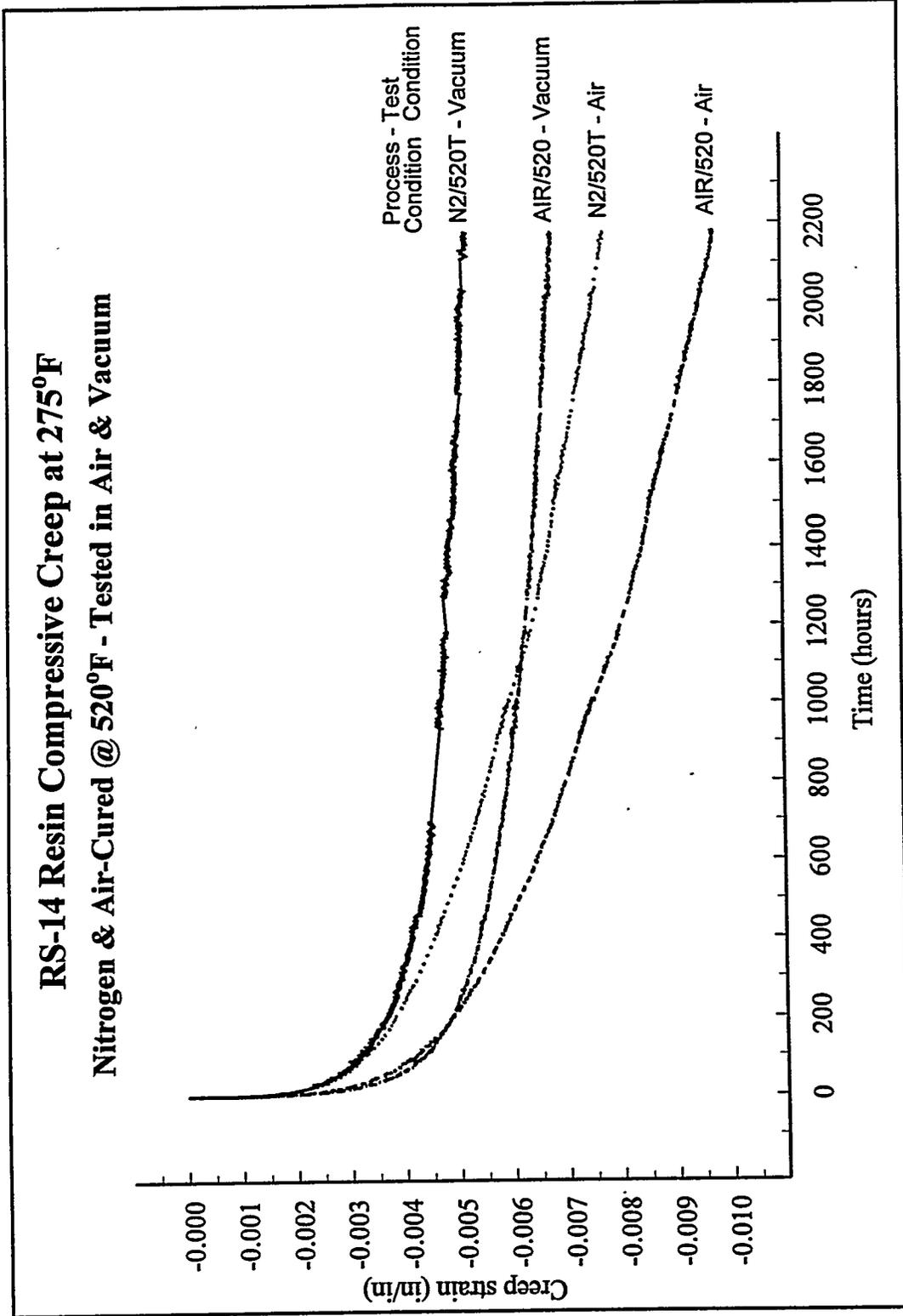


Fig. 1.3-1. Effect of process and test environments on RS-14 resin compressive creep.

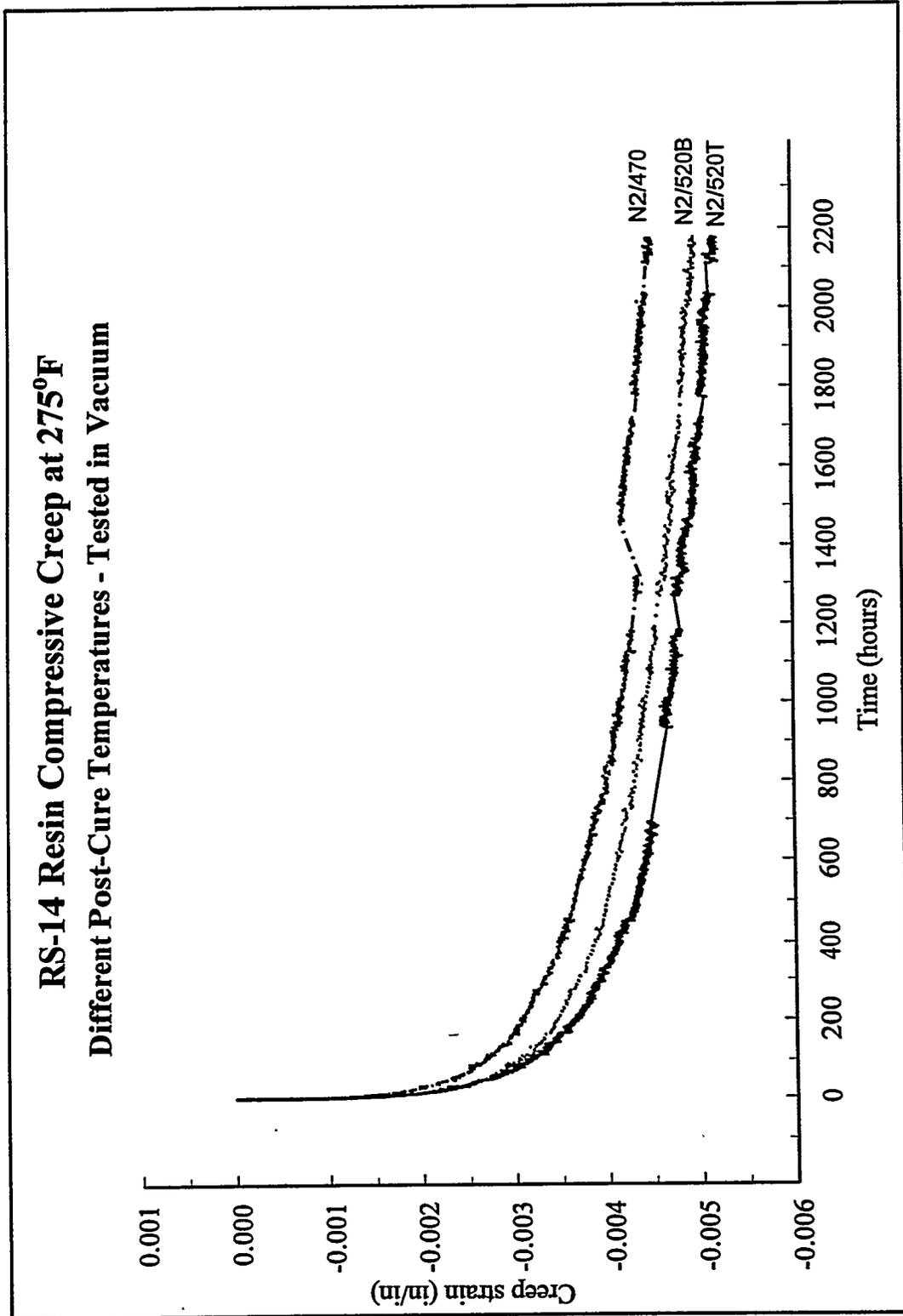


Fig. 1.3-2. Effect of post-cure temperature on RS-14 resin compressive creep.

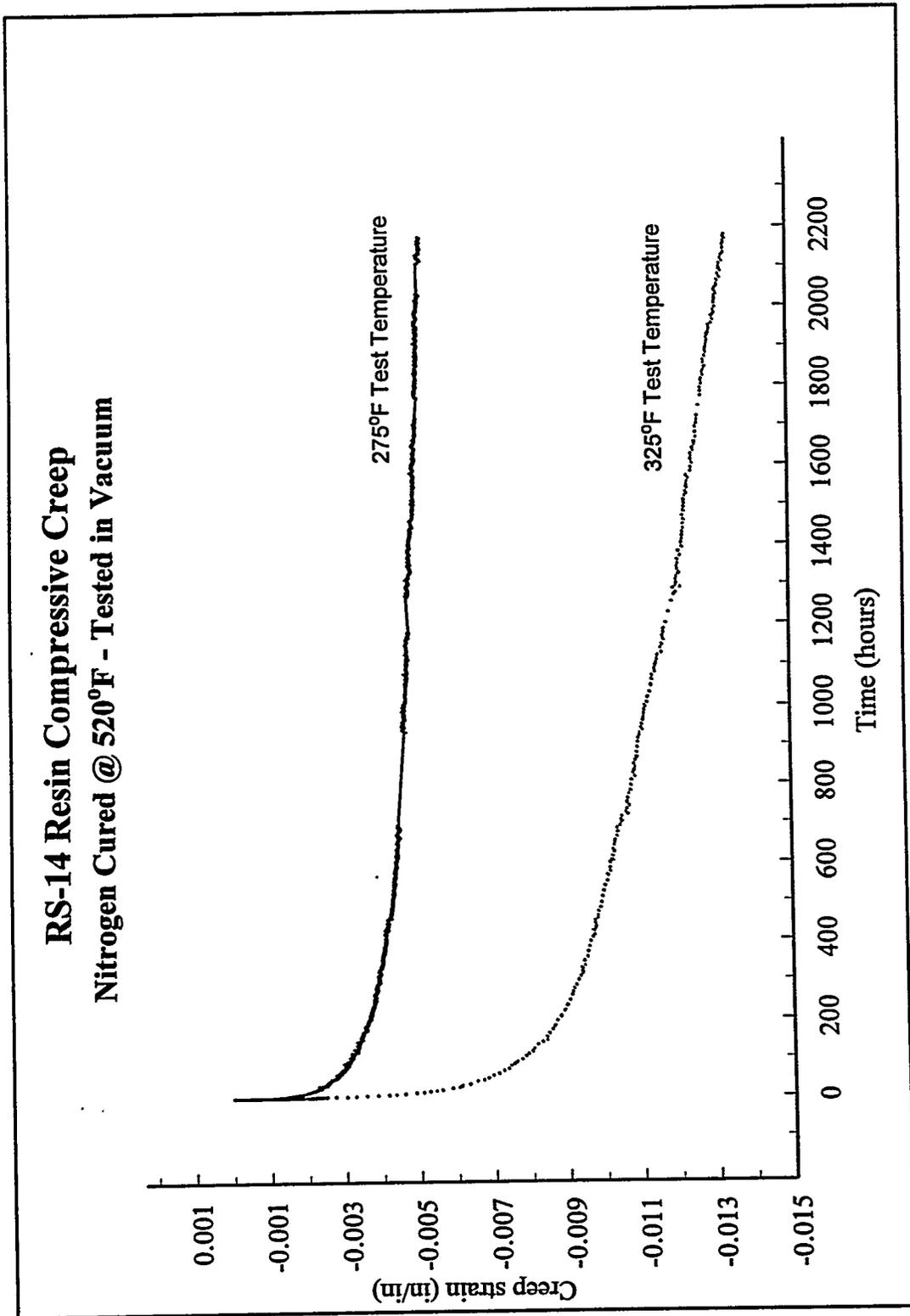


Fig. 1.3-3. Effect of test temperature on RS-14 resin compressive creep.

## 2. T1000G/RS-14 COMPOSITE TRANSVERSE COMPRESSIVE RESPONSE

Following the conclusion of the resin creep tests, the effects of the process and test environment on the composite creep response were quantified by conducting another set of tests. This series of tests also investigated the effects of initial stress level and test temperature on the creep behavior. All of the specimens were post-cured in a nitrogen environment and all tests were conducted in a vacuum environment. In addition to the creep testing efforts, transverse compressive strength tests were conducted to calculate a B-Basis allowable and to determine the transverse compressive stress-strain response of the composite material system.

### 2.1 Composite Specimen Fabrication

The specimens were machined from a 1 in. radially thick composite cylinder. The cylinder was wet-filament wound on a 14 in. diameter internally heated steel mandrel using T1000G fiber and RS-14 resin. The 1 in.-thick cylinder was wound in nominal 1/4in.-thick stages per day over a 4-day period. Each stage took approximately 4 hr to complete before the mandrel could be moved from the winding machine to the oven. To minimize the potential for carbamate formation from exposure of the resin to ambient humidity in the vicinity of the winding machine (Shimp and Ising<sup>5</sup>), winding was accomplished on days when the relative humidity in the laboratory was 40% or less. The pot temperature was maintained between 175°-180°F and the fiber tension was 12 lb. Every layer was compacted and wiped during winding. An additional two compaction passes with wiping were also applied after every five-layer winding increment.

The internal heaters of the mandrel were used to maintain the mandrel OD surface temperature between 170°-180°F. The composite surface temperature was also maintained between 170°-180°F. As the winding progressed, the thickening composite acted as an insulation layer and an external strip heater was required to maintain the composite surface temperature.

The last carbon fiber layers wound on the first, second, and third stages were wiped dry prior to moving the mandrel to the oven for pre-cure. The purpose was to remove excess resin so that there would not be an excessively thick resin layer between the composite stages after cure. Excess resin that bled out during the pre-cure was sanded to roughen the surface and degreased with acetone

prior to winding the next stage. The last layer of the fourth and final stage was left slightly resin rich to protect the carbon fiber at the OD of the composite.

After completion of each 1/4 in.-thick stage, the mandrel was moved from the winding machine to a rotisserie cart and sealed within a metal canopy. The canopy was purged with nitrogen gas and the mandrel temperature was raised to 380°F at approximately 0.5-1 °F/min and held at that temperature for 3 hr to complete the pre-cure.

After the pre-cure of the fourth and final stage, the mandrel was allowed to return to ambient temperature and the seal of the metal can was inspected to ensure that there would be no air leakage into the canopy during the elevated temperature post-cure. Following inspection, the canopy was purged with nitrogen gas and the mandrel temperature was raised to 500°F at approximately 0.5-1 °F/min and held at that temperature for 4 hr to complete the post-cure of the composite.

Oxygen levels were measured with an oxygen analyzer during the 500°F portion of the cure cycle and ranged between 1 to 2%. The outer resin layer of the composite cylinder was examined visually and had a brown tint instead of the dark black color typical of cylinders that have been post-cured in air. This information suggests that the process used to fabricate the T1000G/RS-14 compressive creep test cylinders was successful at preventing resin surface oxidation during the cure cycle.

A 1-3/4 in. wide ring was cut from this cylinder for machining both the creep and static strength specimens. The machined circumferential width for the creep specimens was 0.5 in., whereas, the strength specimens were 1.0 in. wide (see Fig. 2.1-1). This was to minimize the effects of machining and local surface damage on the static compressive strength measurement. Similar to the resin creep specimens, this geometry produced a radius of gyration within the specified limits of the ASTM D695M-85 standard test method.

## **2.2 Composite Transverse Compressive Strength**

The composite transverse compressive strength was determined by loading the specimens transverse to the fibers along the direction parallel to the axis of the cylinder. The specimens were strain gaged to determine the ultimate strain and to calculate an initial modulus. A strain range of 0.1 to 0.6% was used for the modulus calculations. A total of 20 specimens were tested at both

ambient and 275°F test temperatures. This provided 10 tests at each temperature, which was a sufficient number of data points to reliably calculate a B-Basis allowable. The results are shown in Table 2.2-1 along with the data from previous tests conducted on specimens that had been cured in an air environment. The results indicate that the process conditions did not significantly affect the B-Basis allowable. The average strength for the nitrogen-cured specimens actually decreased, but with a corresponding reduction in the scatter the B-Basis was slightly greater than the air-cured results. Similar to the neat resin strength results, the effect of test temperature was significant with a 22% reduction in strength at 275°F compared to the ambient temperature strength. However, there was a less than 10% reduction in modulus and ultimate strain. A typical stress-strain curve for both test temperatures is provided in Fig. 2.2-1.

### 2.3 Composite Transverse Compressive Creep

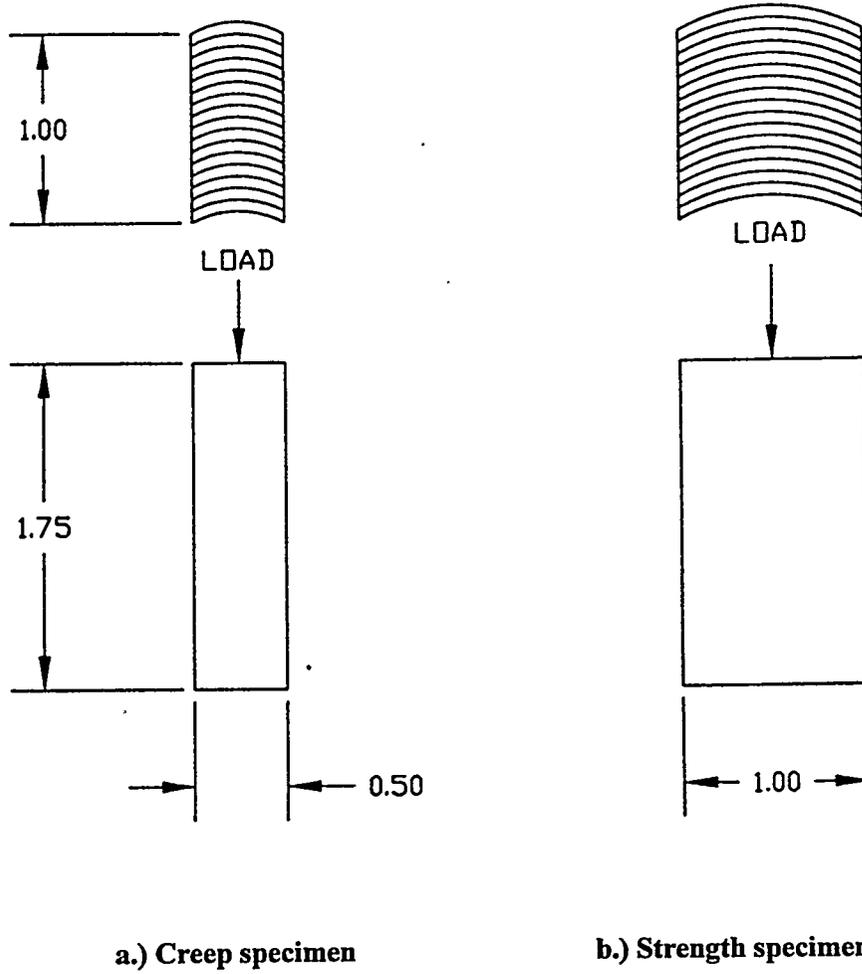
The test matrix for the composite transverse compressive creep tests is shown in Table 2.3-1. The specimens were loaded transverse to the fiber direction using the same creep stands and fixtures as described for the resin creep tests. Also, the same type of instrumentation and data acquisition system was utilized. The data was collected and reduced for a total test duration of 2000 hr. The results for the 10,000-psi stress level at 275°F were compared to the results from a baseline test conducted on an air-cured specimen tested in an air environment. According to the stress-strain curves plotted in Fig. 2.2-1 for the nitrogen-cured specimens, the 6000-psi stress level was well within the initial linear portion of the curve. However, the 10,000-psi applied stress was very close to the point where the stress-strain response begins to deviate from the initial linear response.

The measured total strains are plotted in Fig. 2.3-1 and the data was reduced as described in Sect. 1.3. The graphical interpretation of the results along with the power law parameters are summarized in Table 2.3-2. The results for the graphical interpretation of the data show a significant increase in creep strain between the 200°F test temperature and the 275°F temperature (Fig. 2.3-2) and between the 6000-psi applied stress and the 10,000-psi stress (Fig. 2.3-3). This is also shown graphically in Fig. 2.3-4, where the creep strains are plotted for the various test conditions. The specimen that was nitrogen-cured and vacuum tested at 275°F and subjected to the 10,000-psi stress, i.e., specimen 29, had creep strains that were 19% less than the equivalent air-cured specimen, C6.

The effects of process and test environment on the creep behavior are not as great for the composite system as they were for the neat resin. This is primarily because of the low resin content in the composite material system.

The power law model provided an excellent representation of the experimental data as shown in Fig. 2.3-5. The creep strains plotted in this figure were calculated by subtracting from the total strain the initial strain determined from the nonlinear least-squares regression fit of the data. This figure also demonstrates the reduced creep of the nitrogen-cured/vacuum-tested specimen compared to the air-cured/air-tested specimen. In using the initial strain from the power law fit to compute creep strains, the differences between the two data sets are not as great as the graphical results previously discussed. It should be pointed out, that not all of the reduced creep is due to the processing and testing environments because the C6 specimen had a slightly higher applied stress, 10,658 psi, versus 10,020 psi for specimen 29. In terms of the power law parameters, the air-cured/air-tested specimen had a larger initial strain, a smaller scale parameter, and a larger time exponent than the nitrogen-cured/vacuum-tested specimen. This is consistent with the trends observed in the neat resin compressive creep tests. The effects of an increase in stress on the power law parameters were larger initial strains, larger scale parameters, and smaller time exponents. The effects of an increase in temperature were shown to be larger initial strains and larger time exponents.

The power law model was used to make a 10-year prediction for the transverse compressive strains. The total strain predicted after ten years is in excess of 1.0% only for the highest stress level of 10,000 psi and the largest test temperature of 275°F. The static stress-strain curves indicate that a transverse compressive strain of 1.0% corresponds to 70% of the ultimate strain when the composite system is exposed to 275°F. Consequently, under these conditions there is still a small safety factor on the transverse compressive strain. The 10-year predicted creep strains are compared in Fig. 2.3-6 for the air-cured/air-tested specimen (C6) and the nitrogen-cured/vacuum-tested specimen (29). For the 10-year exposure, the reduction in creep strains for the nitrogen-cured/vacuum-tested specimen compared to the air-cured/air-tested sample is 18.8%.



**Fig. 2.1-1. Composite transverse compression test specimen geometries.**

**Table 2.2-1. Results from composite transverse compressive strength tests.**

Process condition	Test temperature (°F)	Average strength (psi)	C.V. (%)	B-basis (psi)	Ultimate strain (%)	Modulus (psi)
Nitrogen	72	22,769	5.28	19,390	1.57	1.57 x 10 <sup>6</sup>
Nitrogen	275	17,813	2.47	16,480	1.43	1.46 x 10 <sup>6</sup>
Air	275	20,119	7.66	16,292	1.58	1.50 x 10 <sup>6</sup>

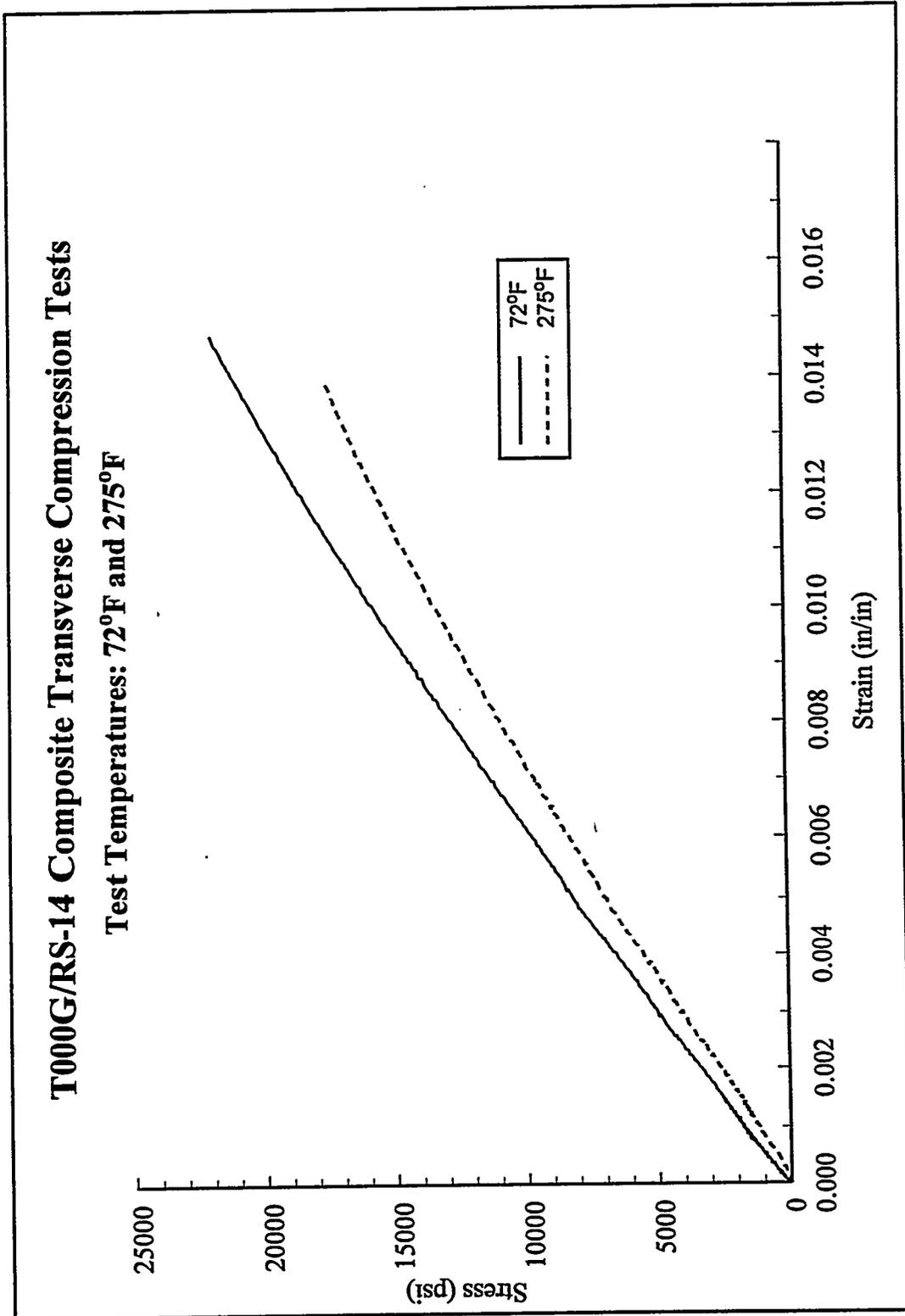


Fig. 2.2-1. Typical stress-strain curves for composite transverse compression.

**Table 2.3-1. Test matrix for composite transverse compressive creep tests.**

Test number	Stress level (psi)	Test temperature (°F)	Test environment
1	10,000	72	Vacuum
2	6,000	140	Vacuum
3	10,000	140	Vacuum
4	6,000	200	Vacuum
5	10,000	200	Vacuum
6	6,000	275	Vacuum
7	10,000	275	Vacuum

Table 2.3.2. Results for the T1000G/RS-14 composite transverse compressive creep tests

Spec. no. <sup>a</sup>	Temp. (°F)	Stress (psi)	Graphical results			Power law parameter (nonlinear data fit) <sup>b</sup>				10-year prediction	
			Initial strain (%)	Final strain (%)	Creep strain (%)	Initial strain (%)	Scale parameter (%)	Time exponent	R	Final strain (%)	Creep strain (%)
21	140	5999	0.4334	0.4727	0.0393	0.4094	0.0203	0.0719	0.9951	0.4924	0.0830
23	200	6002	0.4465	0.5008	0.0543	0.4219	0.0191	0.0903	0.9974	0.5336	0.1117
27	275	5998	0.4753	0.5832	0.1079	0.4470	0.0184	0.1272	0.9986	0.6693	0.2223
17	72	10,164	0.5498	0.5917	0.0419	0.5229	0.0215	0.0742	0.9953	0.6150	0.0921
19	140	9996	0.5923	0.6681	0.0758	0.5419	0.0538	0.0538	0.9980	0.6964	0.1544
30	200	10,012	0.6711	0.7597	0.0886	0.6097	0.0535	0.0652	0.9997	0.8016	0.1919
29	275	10,020	0.6918	0.9504	0.2586	0.5809	0.0849	0.0939	0.9994	1.1143	0.5334
C6	275	10,658	0.7497	1.0701	0.3204	0.6662	0.0550	0.1268	0.9995	1.3236	0.6574

<sup>a</sup> Specimens 17, 19, 30, 29, 21, 23, 27 were processed in nitrogen and tested under vacuum, and specimen C6 was processed in air and tested in air.

<sup>b</sup> Power law equation:  $\epsilon(t) = \epsilon_0 + mt^n$ , where:

$\epsilon(t)$  = total strain

$\epsilon_0$  = initial strain

$m$  = scale parameter

$n$  = time exponent

$R$  = correlation coefficient

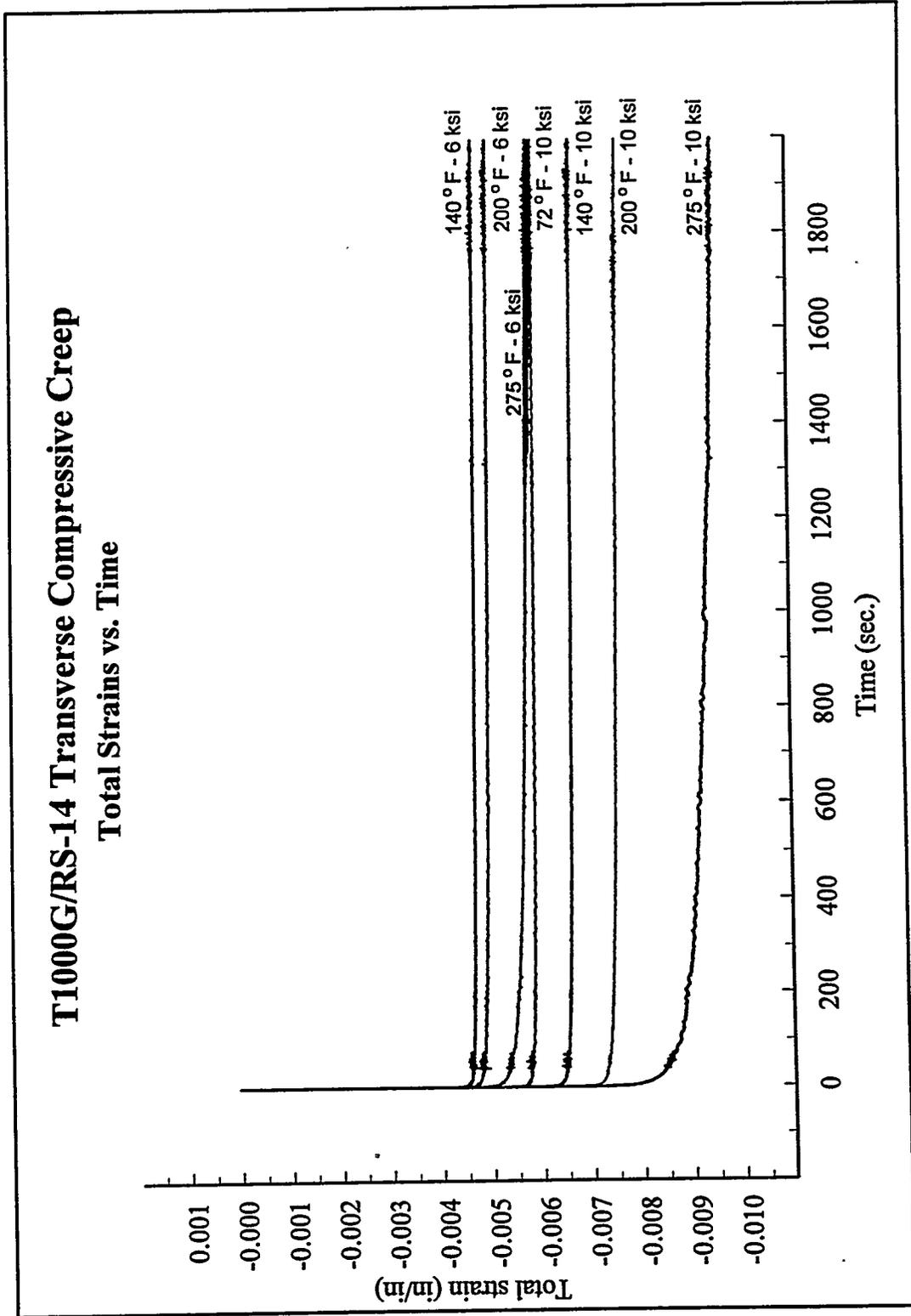
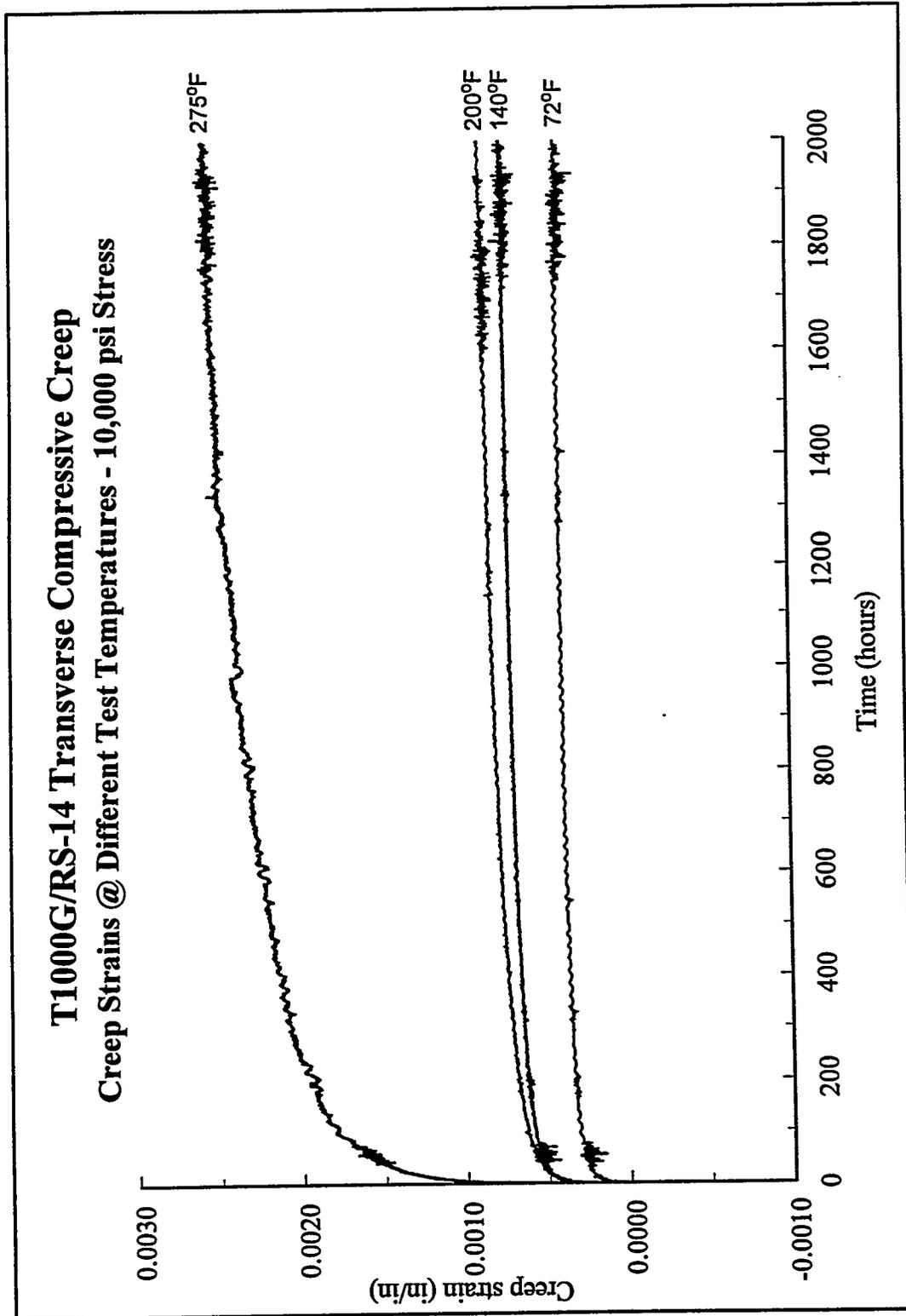
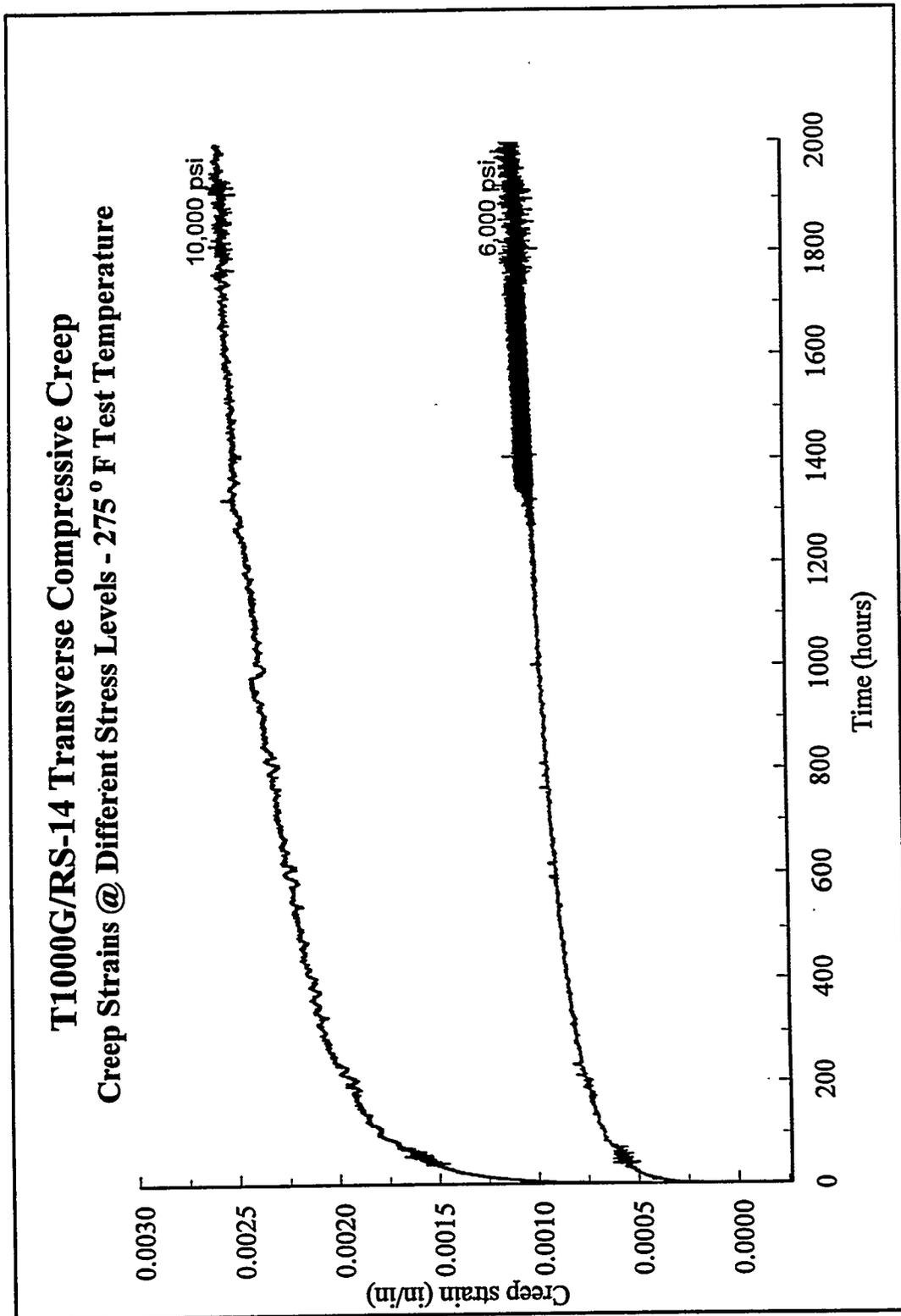


Fig. 2.3-1. Total strains for composite transverse compressive creep tests.



**Fig. 2.3-2. Effect of test temperature on composite transverse compressive creep.**



**Fig. 2.3-3. Effect of applied stress level on composite transverse compressive creep.**

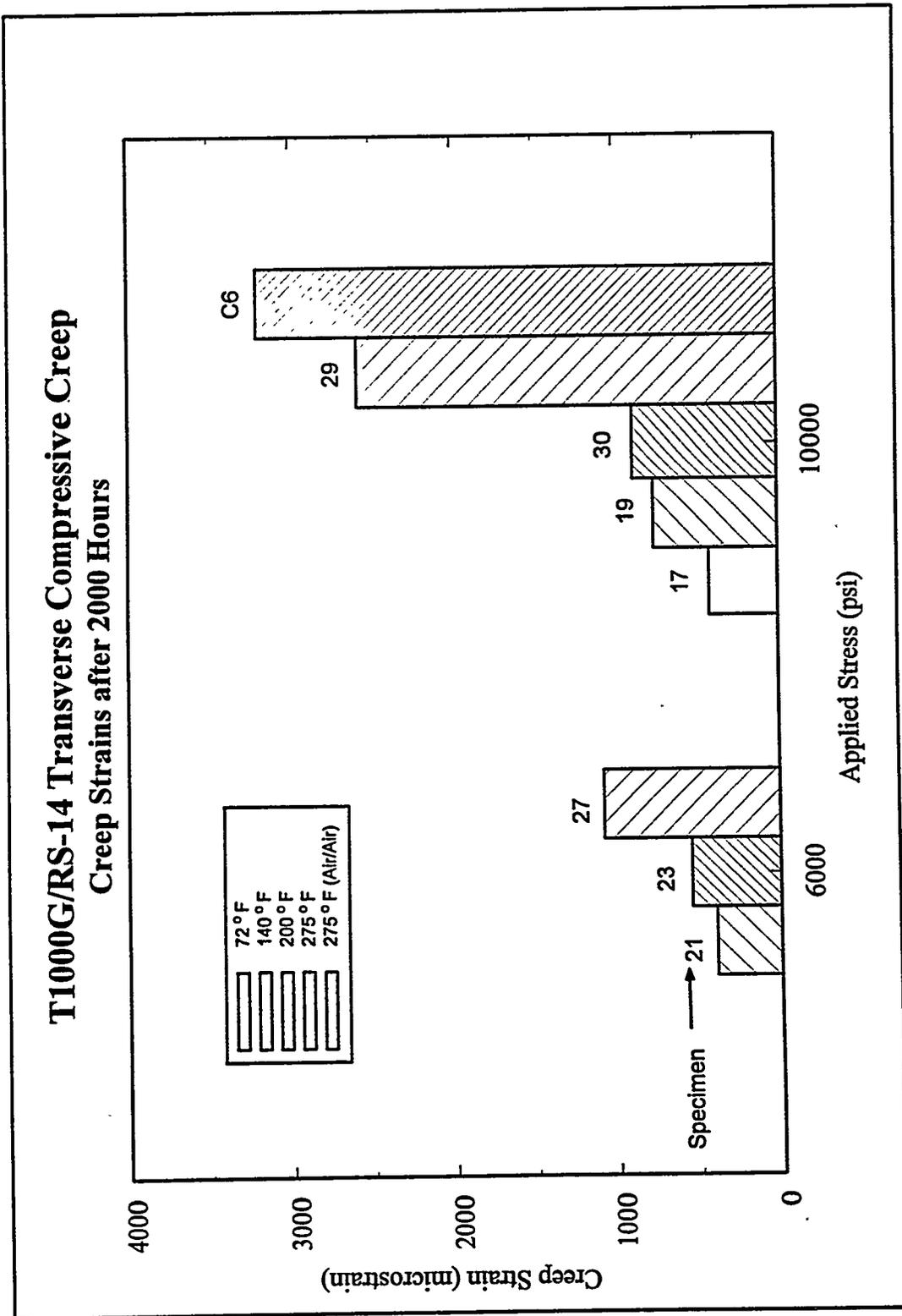


Fig. 2.3-4. Magnitude of creep strains for composite transverse compressive creep.

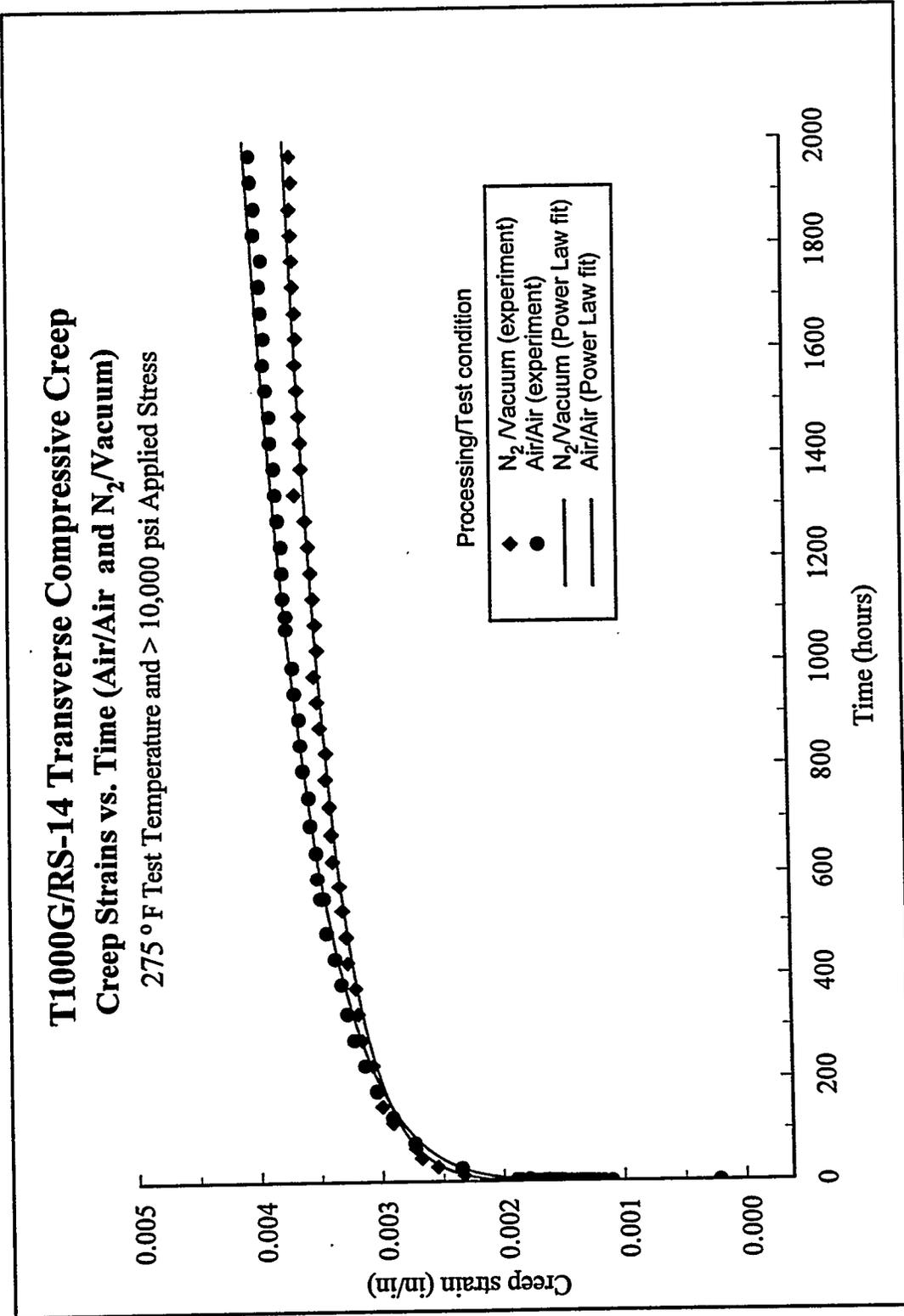


Fig. 2.3-5. Nonlinear least-squares regression fit for power law parameters.

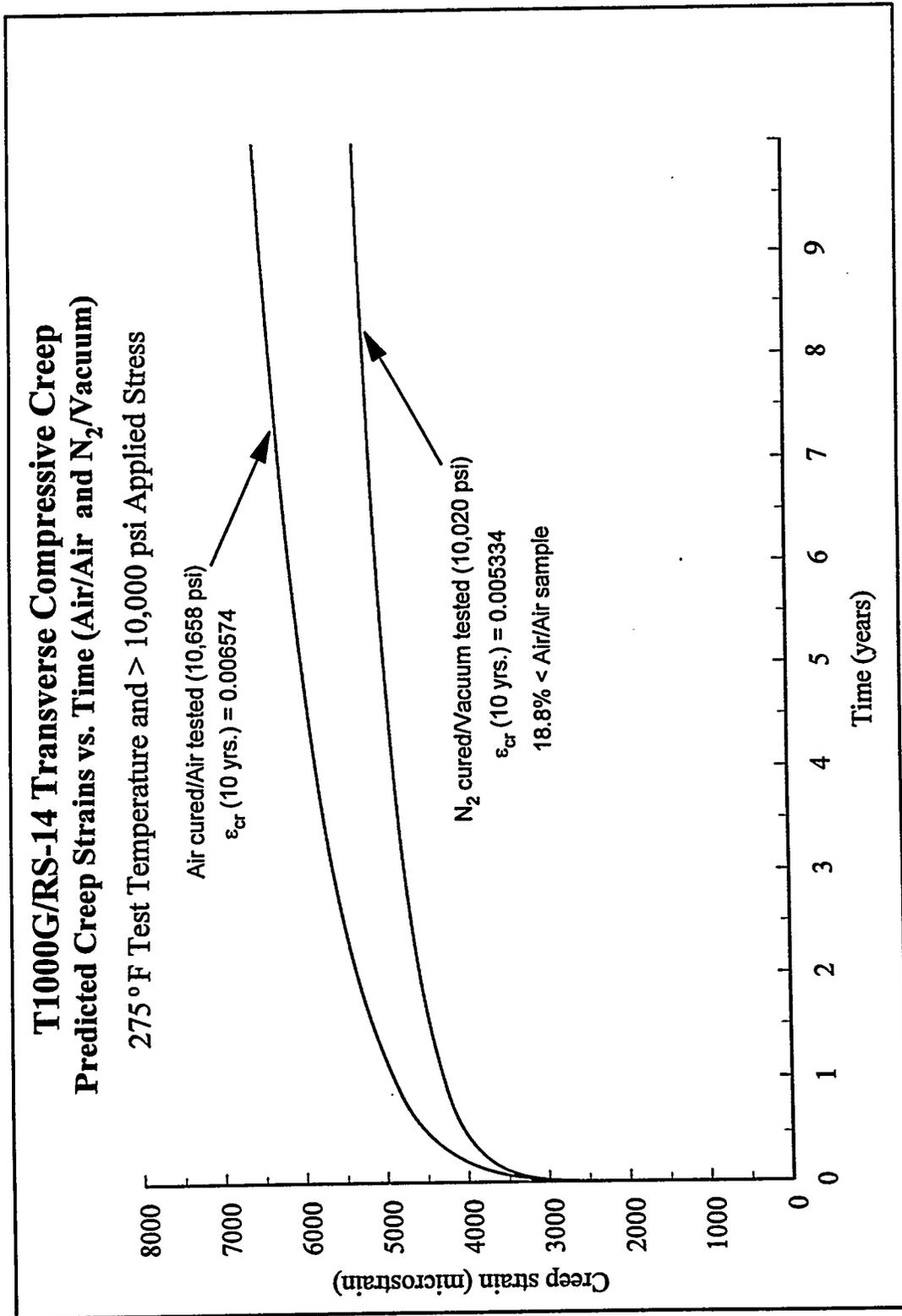


Fig. 2.3-6. Ten-year prediction based on power law model.

### 3. DISCUSSION OF RESULTS

Composite material characterization tests were completed to characterize the compressive behavior of the RS-14 resin and the T1000G/RS-14 composite system. Mechanical property tests were conducted for both static strength and creep, and the significant findings are as follows.

1. The process environment and post-cure temperature had a minimal effect on the RS-14 resin compressive strength and modulus.
2. There was a 38% reduction in strength and a 21% reduction in modulus for testing the RS-14 resin in compression at 275°F versus ambient temperature.
3. Processing the T1000G/RS-14 composite in a nitrogen environment did not increase the B-Basis allowable for transverse compressive strength.
4. There was a 22% reduction in the composite transverse compressive strength at 275°F compared to the ambient test temperature.
5. The combined effect of processing in nitrogen and testing in vacuum versus processing in air and testing in air on the RS-14 resin compressive creep was a 47% decrease in creep strain after 2177 hr, where the testing environment had the largest effect on the measured response.
6. A lower post-cure temperature resulted in smaller resin compressive creep strains.
7. There was significant increase in composite transverse compressive creep strains between the 200 and 275°F test temperatures, and between the 6,000 and 10,000 psi applied stress levels.

8. At the end of the 2000-hr tests, the nitrogen-processed and vacuum-tested conditions reduced the composite transverse compressive creep strain by 19% compared to processing in air and testing in air. The 10-year prediction for the reduction in creep strain was 18.8%.

#### 4. RECOMMENDATIONS

The power law exponent for creep of epoxy resin systems is typically around 0.2 - 0.3. The results from the resin compressive creep tests indicate that the RS-14 polycyanate resin will have lower creep strains than an epoxy system provided that precautions are taken during processing and testing to minimize the effects of moisture exposures.

The results from the composite transverse compressive creep tests under vacuum at 10,000 psi initial stress and 275°F temperature conditions led to a 1.1% total strain predicted after 10 years. The static ultimate strain for the T1000G/RS-14 material was measured to be 1.4% under elevated temperature test conditions. The results also showed that the nitrogen process and vacuum test environments significantly reduced the creep strains. However, under the conditions of high transverse compressive stress and high temperature the safety margin may not be sufficient for dimensional stability.

The results for the 10-year predictions were used, along with an approximate linear extrapolation, to obtain estimates for strains that correspond to relaxed conditions of an applied stress of 3000 psi and a temperature of 140°F. The results are a total strain of 0.23% with only 0.03% creep strain, where 85% of the total strain is comprised of the initial elastic strain. This is significantly less than the 1.1% total strain predicted for the 10,000 psi and 275°F conditions. This is an encouraging result, however it must be verified by conducting future tests at 3000 psi and 140°F.

The dimensional stability of the T1000G/RS-14 system, in terms of its transverse compressive creep response, may be acceptable at higher temperatures provided that the compressive stresses are kept to a minimum. This may be accomplished by using a laminate architecture that increases the transverse stiffness.

## 5. ACKNOWLEDGMENTS

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