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HIGH TEMPERATURE, SHORT TERM TENSILE STRENGTH OF C6000/PMR-15 COMPOSITES

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Tensile tests were conducted on 0° unidirectionally reinforced Celion 6000 graphite fibers in PMR 15 polyimide matrix. Tensile strengths for coupons subjected to short and long term uniform temperatures were obtained. Thick coupons, heated on one side to produce significant transient through thickness temperature gradients, were tested and compared to the strength of specimens with uniform temperature distributions.

All coupons were radiantly heated and reached maximum test temperatures within 15 sec. Tensile loads were applied to the coupons after 15 sec of elevated temperature exposure. Loading rates were selected so that specimen failures occurred within a maximum of 45 sec after reaching the test temperature. Results indicate that significant tensile strength remains beyond the material post cure temperature.

BACKGROUND

Medium range tactical missile airframe components have historically been designed with low cost metallic materials insulated by a variety of thermal protection coatings when flight regimes cause excessive aerodynamic heating of the structure. The major design considerations for missile body shells are strength, stiffness, weight, minimum thickness to permit maximum internal packaging volume, and minimum unit cost in production. Aluminum airframe structures are limited to moderate free flight Mach numbers due to the rapid reduction in strength at temperatures above 600°F. High strength metallic airframe structures are more difficult to manufacture and are subject to increasing material and fabrication costs. Ablation or subliming thermal protection systems are costly, increase the net thickness of components thereby resulting in constricted internal volume, and are susceptible to damage during handling operations and aircraft captive-carry.

The development of advanced continuous filament composite materials over the past two decades has been mainly restricted to applications in the aircraft and space technology areas and has used epoxy matrices which are limited to 250°F - 300°F for long term use. Polyimide matrix materials have recently been developed which can sustain temperatures up to 600°F for long durations. The maturation of graphite/polyimide composites, due to improved manufacturing technology and increasing filament production, has continuously reduced hardware cost, stimulating increasing applications.

The unique aspects of graphite/polyimide materials such as tailorable thermal expansions, stiffness and strength by appropriate selection of laminate orientation are several characteristics important to the missile airframe designer. The strength data at elevated temperatures of most advanced composite materials has been developed for aircraft applications which assume long term exposure at elevated temperature environments. Since many tactical missiles are characterized by flight times of less than two minutes duration at higher than cure temperature, advanced composite strength data obtained for long term high temperature exposure is potentially conservative for missile airframe applications. Therefore, a test program was conducted to determine if the strength of graphite/polyimide could be extended to temperatures up to 900°F for short durations.

Two issues are being addressed as part of the short time high temperature strength evaluation of advanced composites. First, the evaluation of tensile strengths of 0° unidirectional six ply thick graphite/polyimide coupons when subjected to uniform through thickness transient temperatures for exposure times of one minute or less. Second, the tensile strength evaluation of 20 ply thick 0° graphite/polyimide coupons wherein significant through thickness temperature gradients existed. For the latter case, high temperature exposure was limited to one minute and maximum temperatures reached were the same as for the thin (six ply) test cases. The purpose of tests conducted during the existence of thermal gradients across the thick test coupon was to determine whether higher tensile strengths could be achieved in those cases than for the uniform temperature case. When comparing strengths for both transient uniform and transient non-uniform coupon temperature histories, strengths for the non-uniform case were based on maximum face temperatures, not average through thickness temperatures.

Shown in figure 1, for tactical missiles, are typical ranges of maximum external surface structural component temperature ranges, maximum through thickness temperature differences, time to reach and time to maximum temperature, and total flight times.

Ranges shown in figure 1 represented the bases for the tensile coupon tests conducted during the presently reported experimental study.

TEST SPECIMEN PREPARATION, INSTRUMENTATION, AND TEST PROCEDURE

Tensile test, straight edged, 0° unidirectional tabbed coupons were cut from Celion 6000/PMR-15 panels fabricated by the Hamilton Standard Division of United Technologies Corp., Windsor Locks, Ct. Six ply and twenty ply panels, 9 in. x 10 3/4 in., were press-cured in heated ceramic platens. The six ply laminated panels were used to provide uniform through thickness transient temperature test coupons, while the thicker twenty ply laminated panels provided test coupons used for through thickness transient temperature gradient tensile tests. The unidirectional panels were fabricated from Fiberite prepreg HY-E 1666AE, Lot C1-467. The laminated panels were imidized and cured following a procedure developed by Hamilton Standard for fabrication of C6000/PMR-15 F100 engine nozzle flaps. The panels were postcured at 600°F for 12 hrs. in an air circulating oven.

Test coupon tab materials were fabricated using seven plies of 0°/90° 7781 fiberglass cloth/PMR-15 laminate for the six ply 0° coupons and twenty plies of 0° C6000/PMR-15 laminate for the thick twenty ply coupons. For the latter case, an additional twenty ply C6000/PMR-15 panel was specifically fabricated to provide tab material for the thick coupons. Tab lengths were 1 1/2 in. for both thin and thick coupons and were bonded to the panels using PMR-15 adhesive for subsequent cutting into straight edged tensile coupon shape. Tab angles for all test coupons were 90°. Coupon widths were 0.5 in. nominal for thin (six ply) and thick (20 ply) coupons. Panel cutting was performed with diamond blades to ensure high finishes on coupon edge faces.

Thermocouples were bonded back-to-back at the center of each coupon, using M Bond-610 adhesive. The thermocouple adhesive was cured at 325° for 30 min. Thermocouple wires were perpendicular to the specimen axis to minimize shadowing of the radiant flux. Details of the thin and thick test coupons and thermocouple locations are shown in figure 2.

A modular radiant heating reflecting assembly, using a bank of quartz lamps on each side of the test coupon, was adapted to an MTS series 810 Material Testing System. Thermocouple output was fed back to a temperature controller which regulated power to the lamps in order to obtain desired transient temperature responses on each side of the Gr/Pi test coupons. Temperature rise times, temperature hold times, and temperature gradients as measured by the center located thermocouples could be controlled. A schematic of the controlled radiant heating apparatus and instrumented tensile test coupon affixed to the tensile tester is depicted in figure 3. The grips were insulated during all elevated temperature tests.

Temperature response tests were conducted on a six ply tension coupon to determine the ability to control transient through thickness temperature nonuniformity as well as to measure axial temperature variations which existed during transient heating.

To determine both these coupon temperature responses, one of the standard six ply coupons was instrumented with eight thermocouples distributed axially along the mid-center line on one side and four thermocouples on the opposite side. The thermocouples located at the axial center position were back to back. The remaining four opposite side thermocouples were axially offset from 0.10 in. to 0.45 in. Three thermocouples were embedded 0.40 in. in the tab. The location of the thermocouples is shown in figure 4. The thermocoupled coupon was placed in the tensile tester with grips locked on the tabs. A negligible mechanical load was applied sufficient to maintain the coupon in a fixed position during the application of heat. The most severe heating condition was one wherein the temperature of the center of the coupon, initially at 77°F, reached 800°F within 15 sec. Between 20 sec and 80 sec the measured temperature was controlled to monotonically increase from 845°F to 865°F. At 80 sec into the experiment the heat lamps were turned off. The maximum temperature difference between each side of center of the six ply coupon did not exceed 25°F and occurred when coupon center temperatures exceeded 850°F. Maximum temperature within the tab, adjacent to the bond line, reached 210°F after 88 sec. The results obtained from the previously described experiment were used to determine the effect of axial temperature variation on the test coupon stresses. A three dimensional finite element stress analysis was performed on a typical six ply tabbed Gr/Pi coupon, and the results will subsequently be discussed.

TEST RESULTS

Having established repeatability and ability to control transient prescribed temperature histories at the center of the coupon, as well as maintaining a uniform axial temperature distribution about the major axial portion of the test coupon gage length, tension tests during long term steady state heating and transient heating conditions were conducted. Steady state heating in the present context refers to 10 min. exposure to heating, at the end of which time coupon temperatures over the coupon gage length were uniform. Shown in figure 5 are the ultimate tensile strengths after 10 min. exposure to temperatures measured at the test coupon center. Because of the need to conserve test coupons for transient temperature tests, only one test at each steady state temperature was performed. However, while the initial set of six ply 0° coupons had observable tab offset (each side of different length) due to initial fabrication problems, four were tested at room temperature conditions. Three of these four tests resulted in an average ultimate stress 203 ksi with a spread of 4 ksi. The fourth coupon, with significant damage in the six ply region at the edge of the tab, resulted in failure at the tab at a stress of 172 ksi. An additional six ply panel was fabricated and the tab problem corrected. No data from coupons obtained from the problem tab panels is presented. While the test data from the C6000/PMR-15 coupons exceeds that in reference 1, it is important to note that two different Gr/Pi systems are compared in the results shown in figure 5. Also, the test coupon of (ref. 1) has a 4 in. gage section and 2.5 in. bevelled tabs. The increase in strength from 750°F and 850°F may be illusory because of the minimum specimens tested, and/or post curing effects. The former seems more likely since the 0° tensile strengths are fiber dominated.

In order to determine transient short term tensile strengths with uniform through thickness temperature distributions, six ply coupons were subjected to temperature histories as shown in figure 6. Loading was applied at a constant load rate at 15 sec following start up of heating, and all coupons failed within 40 sec. Assuming that the 0° tensile modulus is never reduced below $5x10^6$ psi for all short term temperatures to 850°F. max., this results in maximum strain rate during the tests of 0.05 in/in-min.

The corresponding ultimate tensile strengths for short term uniform through thickness elevated temperatures are shown in figure 7.

Three tensile tests at each temperature were conducted as shown in figure 7. The primary results indicate that up to 850°F, approximately 70 percent of room temperature strength is maintained for up to 40 sec. Failure stresses for the six ply coupons were between 199-205 ksi at room temperature and between 125 and 165 ksi when subjected to short term 850°F transient temperatures. Relatively smooth strength decreases were obtained for coupons subjected to temperatures between R.T. and 850°F. Unlike the steady state ultimate strength behavior, significant strength decrease is apparent at 450°F, well below the post cure temperature of 600°F. Strength continues to decrease with temperature to 750°F and then remains constant but with increasing scatter to 850°F. This differs significantly from the steady state 0° tensile strength variation up to matrix post cure temperature experienced by graphite/epoxies (ref. 1) and graphite/polyimides (ref. 2).

In order to determine whether significant strength increases occur when high transient temperatures occur on only the external structural surfaces and large through thickness thermal gradients exist due to low transverse thermal conductivity, transient temperature histories were produced in thick, $[0°]_{20}$, coupons as shown in figure 8.

As in the case of $[0°]_6$ coupons, tensile loads were applied to the thick coupons following 15 sec of heating. After 20 sec of heating the maximum temperature on one side of the coupon was held constant while the temperature on the cooler side continued to increase. At 50 sec heating ceased and temperatures on both sides of the coupon instantaneously decreased. For the thermal gradient test, ultimate tensile stresses obtained for maximum coupon surface temperature of 650°, 750°, 850° and 950°F are shown in figure 9.

No significant increase in strength for coupons subjected to short term thermal gradients were observed when compared to the coupons subjected to short term uniform temperatures for the same maximum temperatures. Even when the short term thermal gradient strength data is compared to strengths obtained for long term temperature subjected coupons (figure 5), no significant increase in tensile strength is observed. (However, sufficient analysis has not been conducted to determine whether longer end tabs should be used for 20 ply straight coupons and if a uniform uniaxial state of stress exists in the gage section of 9 in. thick coupons.) The low value of room temperature ultimate stress, as shown in figure 9, for the $[0°]_{20}$ tensile specimen compared to the $[0°]_6$ specimen suggests at least a further investigation into the stress state in thick tensile coupons. Also, it should be noted that 5 percent less fiber volume was measured in the thick 20 ply panel than in the thin six ply panel from which the test coupons providing the data in question were obtained (both fiber volume measurements were obtained from one random sample of each panel).

THREE DIMENSIONAL FINITE ELEMENT IDEALIZATION OF THERMAL STRESS EFFECTS

To better understand the effect of axial thermal gradients in the $[0°]_6$ Gr/Pi test coupon, the test coupon including end tabs and adhesive were analyzed using the finite element idealization.

Symmetry of the test coupon enables the stress analysis to be performed using one-eighth of the coupon. The analysis is based on a 20 node isoparametric orthotropic brick element using the SAPV Structural Analysis Program (ref. 3). The finite element model represents a 6 laminae 0° unidirectional Gr/Pi end tab, a 0.002 in. PMR-15 adhesive layer, and a 6 laminae 0° unidirectional Gr/Pi specimen.

The end tab was modeled using three elements (two laminae) in the thickness direction, the adhesive layer by one element in the thickness direction, and the test specimen by 6 elements in the thickness direction, one for each lamina. The width of the elements was reduced in the tab end region in the axial direction of the center of the test coupon. It is assumed that the test coupon is stress free at the post cure temperature of 600°F. The detailed finite element geometrical model is shown in figure 10. The tab and adhesive element widths are 0.05 in. in the tab/specimen region and increase to 0.25 in. for the remainder of the tab. The specimen element width is 0.1 in. and increases to 0.5 in. for the remainder of the specimen length.

The measured temperature distribution for the $[0°]_6$ ply coupon at 25 sec after heating is shown in figure 11. Through thickness variations were small and neglected in the finite element analysis.

Material properties used in the finite element analysis are given below:

Graphite/Polyimide Specimen (refs. 4 and 5)

$$E_{11} = 20\times10^6 \text{ psi} \quad E_{22} = E_{33} = 1.2\times10^6 \text{ psi}$$

$$G_{12} = G_{13} = G_{23} = 1\times10^6 \text{ psi}$$

$$\nu_{12} = \nu_{13} = \nu_{23} = 0.33$$

$\alpha_{11} = 1\times10^{-7}$ in/in-°F

$\alpha_{22} = \alpha_{33} = 1.44 \times 10^{-5}$ in/in-°F at 70°F

$\alpha_{22} = \alpha_{33} = 1.58 \times 10^{-5}$ in/in-°F at 300°F

$\alpha_{22} = \alpha_{33} = 1.15 \times 10^{-5}$ in/in-°F at 900°F

PRM-15 Adhesive (ref. 6)

$E = 0.47 \times 10^6$ psi

$G = 0.17 \times 10^6$ psi

$\nu = 0.36$

$\alpha = 28 \times 10^{-6}$ in/in-°F

The important results for calculated stresses are shown in figure 12. Recall that the stresses calculated are due solely to steady temperature distribution measured experimentally. The stress free state was assumed to exist at the post cure temperature of 600°F.

The maximum transverse coupon normal stress distribution indicates failure of the matrix between fibers in the tab region. Examination of the temperature distribution (figure 11) shows that coupon temperatures are below 200°F. Since the tab is fixed on the outer face, the large transverse tensile stress is in response to constraining the coupon contraction. At the stress level predicted severe microcracking would have been initiated leading to a redistribution of transverse stress. Since the tensile tests are fiber dominated, no significant effect of the transverse cracking on the 0° tensile would be expected if damage between the matrix and fiber surface also does not result. The early strength decrease with temperature as shown in figure 7 may be an indication of early fiber/matrix interface failure. Further experimentation and photomicrographic examination of sectioned tab region is indicated to better understand the failure mechanism under elevated temperature highly transient environments.

CONCLUDING REMARKS

Tensile tests at transient elevated temperatures on $[0°]_6$ Celion 6000/PMR-15 graphite polyimide material for maximum temperatures up to 850°F were conducted. It was shown that controlled quartz heat lamps can provide near uniform through thickness temperature distributions for 60-70 sec for six ply coupons. Thick, 20 ply 0° coupons were used to provide controlled maximum surface temperature and prescribed transient temperature gradients.

During uniform through thickness transient temperature tests, 0° strength decreased moderately at 450°F, while 70 percent of the room temperature strength existed for peak transient temperature up to 850°F. Tensile strengths up to 115 ksi were obtained for 20 ply 0° coupons when the outer surface maximum temperature reached 950°F in less than one minute. A three dimensional finite element analysis of a $[0°]_6$ heated coupon, exhibiting a significant axial thermal gradient, indicated high transverse tensile normal stress in the tab region of the tensile coupon. Finally, in order to better understand failure of thick unidirectional coupons subjected to short term through thickness and axial temperature gradients, a detailed stress and thermal analysis should be further studied to determine edge and thickness effects and to establish an optimum tensile coupon configuration.

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● MAX SURFACE TEMP	– 750°F	– 1000°F
● ΔT AT MAX TEMP	– 100°F	– 175°F
● TIME TO MAX TEMP	– 15 sec	– 30 sec
● TIME AT MAX TEMP	– 0 sec	– 10 sec
● TOTAL FLIGHT TIME	– 24 sec	– 70 sec

Figure 1 Typical medium range tactical missile temperature response.

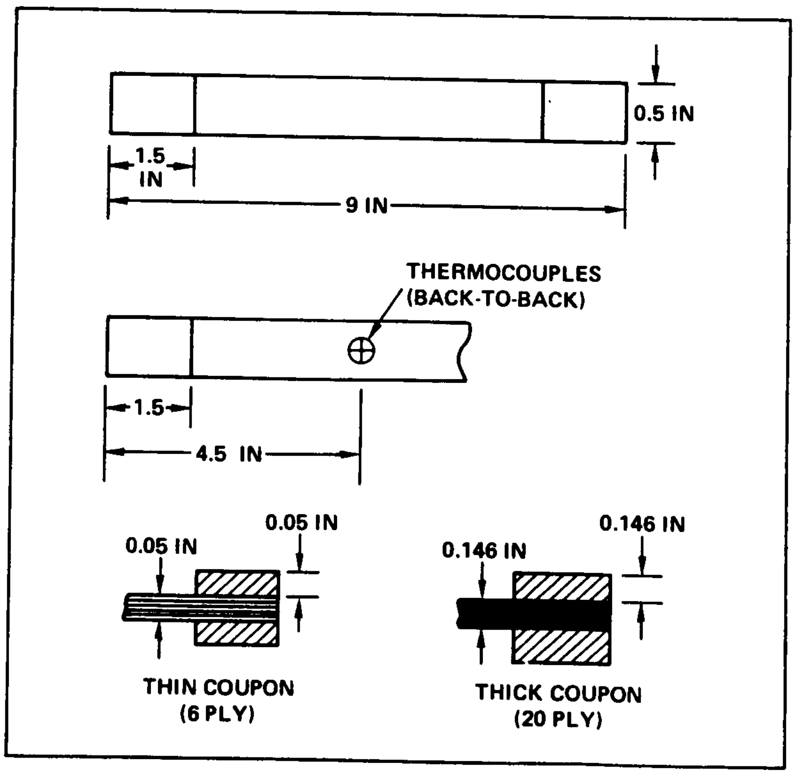

Figure 2 Test coupon and instrumentation details.

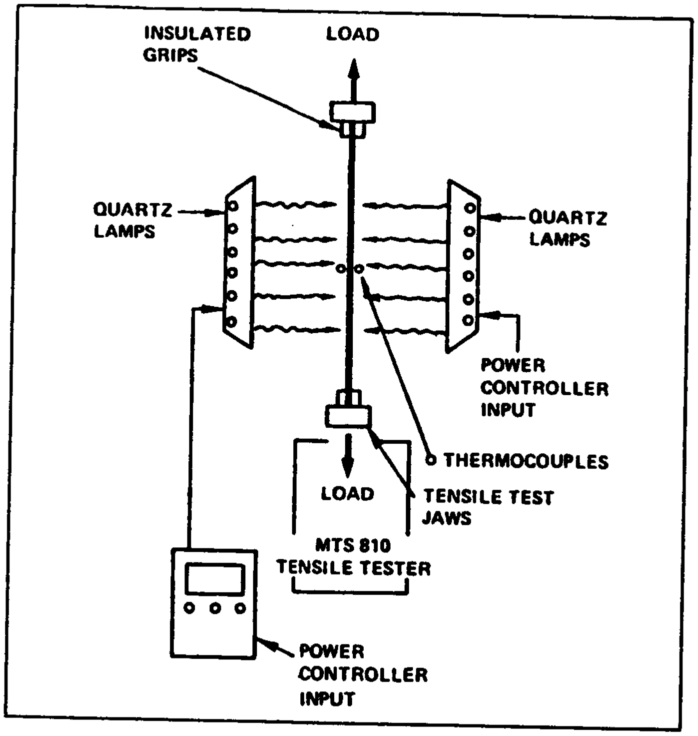

Figure 3 Transient heating/tensile loading schematic.

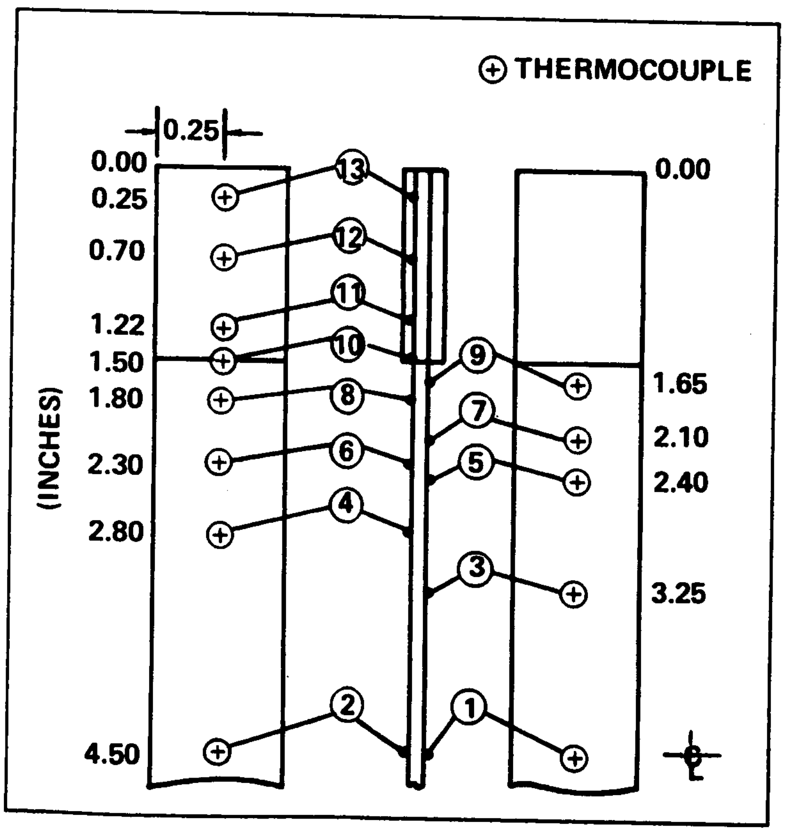

Figure 4 Thermocoupled test coupon (6 ply - 0^{o}).

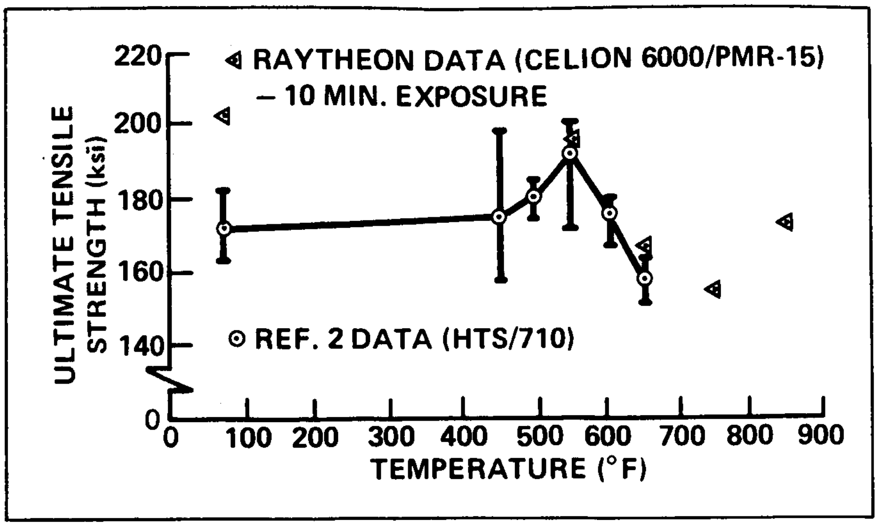

Figure 5 Long term uniform temperature tensile strength of 0^{o} Gr/Pi.

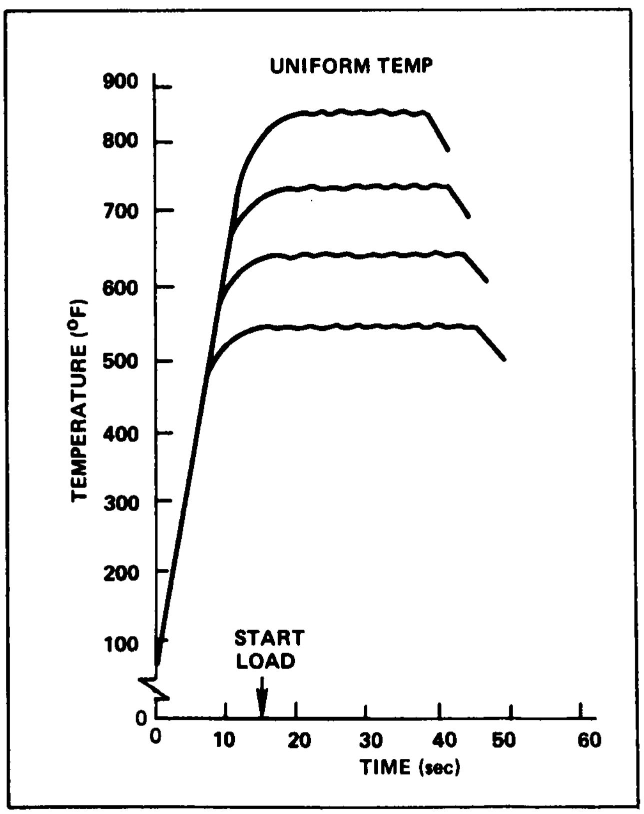

Figure 6 Transient temperature histories for 0^{o} 6 ply tensile coupons.

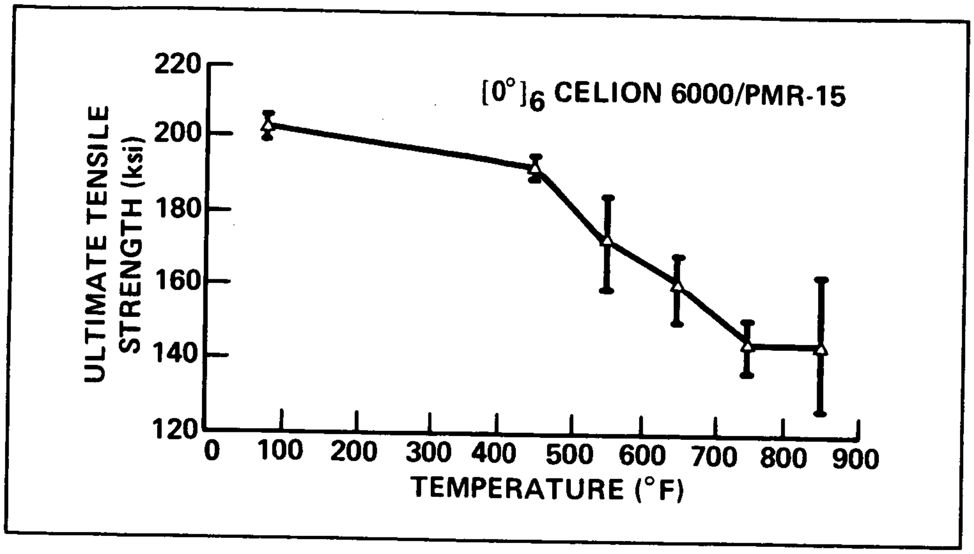

Figure 7 Short term uniform temperature tensile strength.

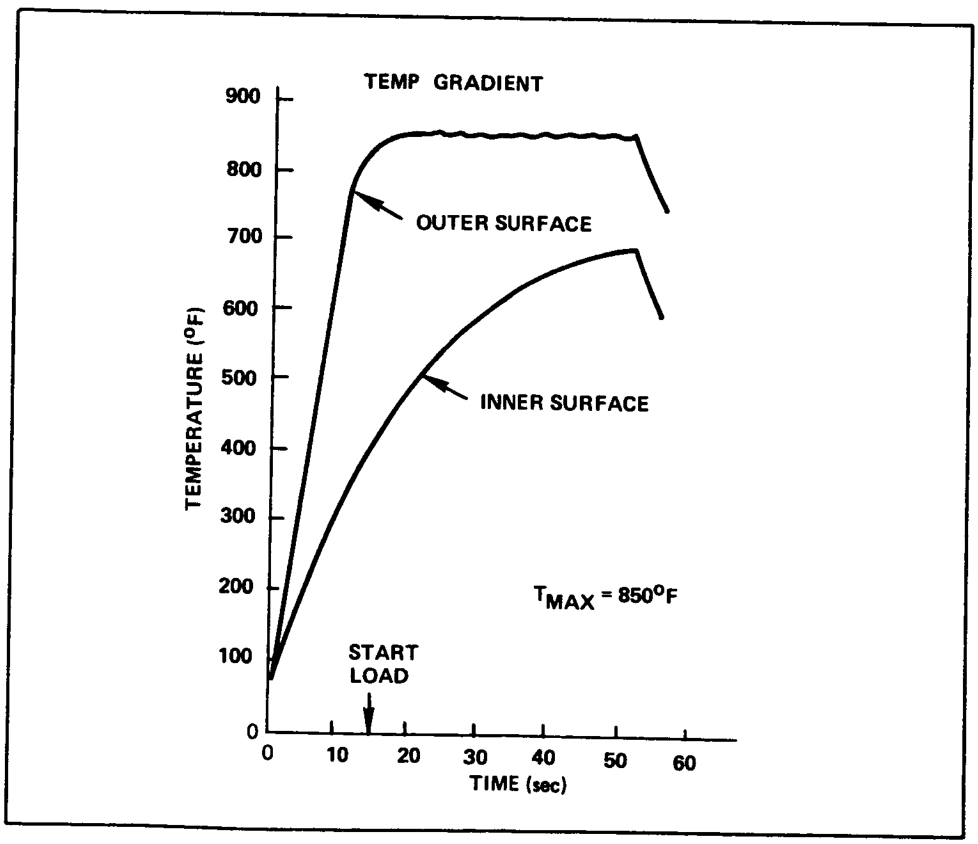

Figure 8 Transient temperature history for $[0^{\circ}]_{20}$ coupons.

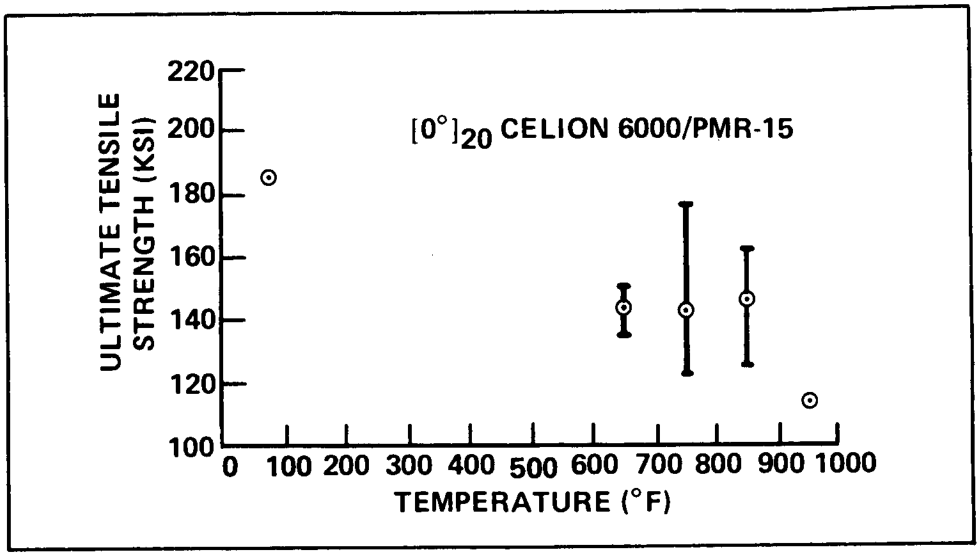

Figure 9 Short term thermal gradient tensile strength.

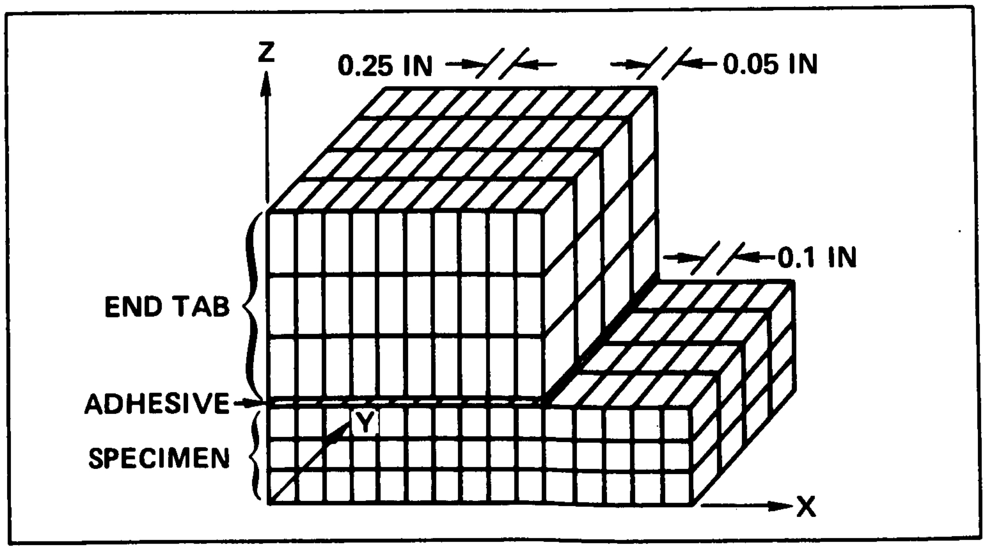

Figure 10 F.E. idealization of test coupon.

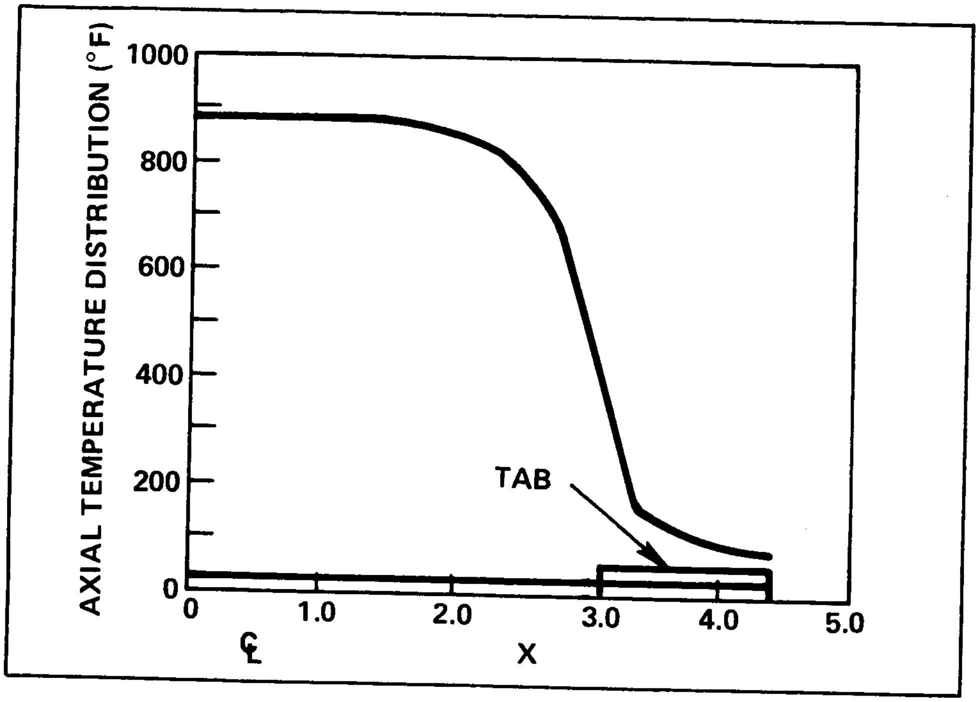

Figure 11 Temperature versus axial position along $\left[0^{\circ}\right]_6$ for maximum axial temperature gradient.

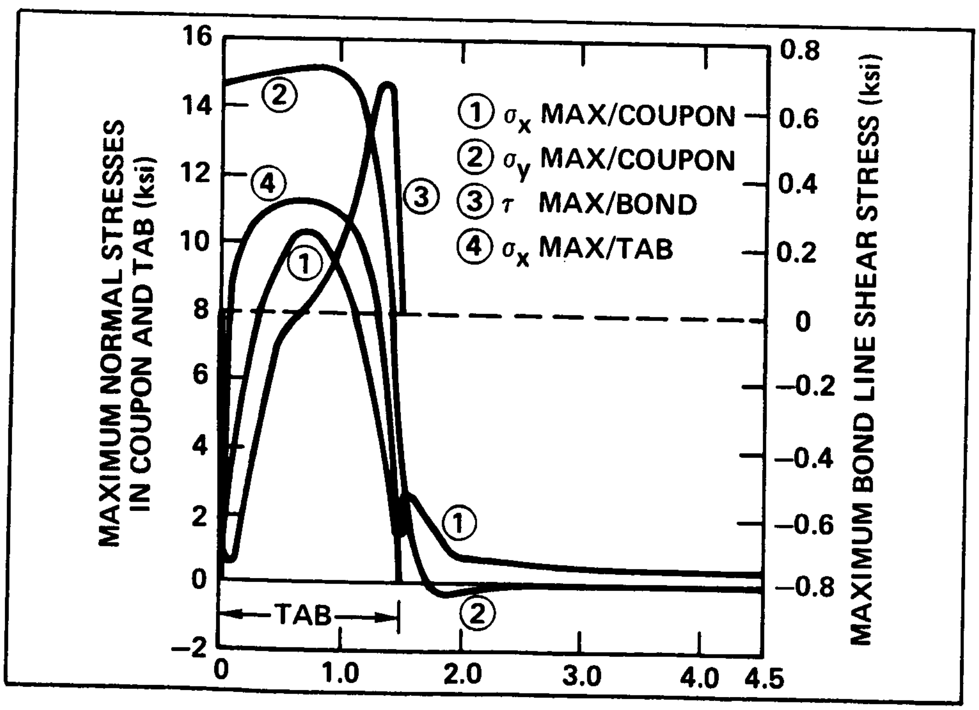

Figure 12 Maximum stress distributions in coupon, bond line, and end tab due to heating (stress free temperature = 600°F).