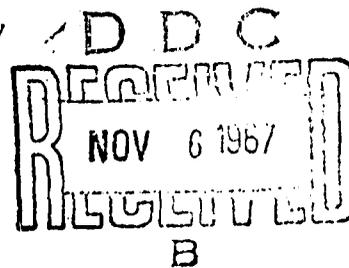


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Strength and Stress-Strain Properties of Rapidly Heated Laminated Ablative Materials

JULY 1967

Prepared by A. CHING and W. E. WELSH, Jr.
Aerodynamics and Propulsion Research Laboratory
Laboratory Operations
AEROSPACE CORPORATION



Prepared for SPACE AND MISSILE SYSTEMS ORGANIZATION
AIR FORCE SYSTEMS COMMAND
LOS ANGELES AIR FORCE STATION
Los Angeles, California

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STRENGTH AND STRESS-STRAIN PROPERTIES OF RAPIDLY
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FOREWORD

This report is published by the Aerospace Corporation, El Segundo, California, under Air Force Contract No. F 04695-67-C-0158 and documents research carried out from December 1965 through June 1967. On 18 September this report was submitted to Lt C. R. Lee for review and approval.

Approved


R. A. Hartunian, Director
Aerodynamics and Propulsion
Research Laboratory
Laboratory Operations

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.


Curtiss R. Lee, 2nd Lt, USAF
Project Officer

ABSTRACT

It is shown that the mechanical properties of rocket nozzle liners and reentry vehicle ablation materials can be determined by tests in which a high temperature plasma arc is used as a heat source. The arc flow is used to heat slender specimens, and a pneumatically actuated loading frame is used to apply stress. This method of testing provides heating and loading in time periods typical of the rapid heating during rocket firing or reentry, on the order of 10 sec. Earlier investigations of similar ablative materials have used specimens heated and charred (degraded) slowly prior to the application of load.

Strength and stress-strain properties in the warp direction were determined for carbon, graphite, and silica phenolic materials. The method appears promising for the determination of properties in various loading-laminate orientations and during thermal expansion, and for studies of heating and strain rate effects on strength at elevated temperatures.

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I. INTRODUCTION

A thorough knowledge of the mechanical properties (strength and physical constants) of laminated ablative materials at elevated temperatures is a necessity for sound design, analysis, and evaluation of reentry heat shields and rocket nozzle liners, as well as for comparison and evaluation of ablative materials. Unfortunately, information regarding these properties is not readily available at present. Previous mechanical testing of ablative materials above 2000°F has been performed on specimens which were slowly degraded (precharred) to a charred state in a high temperature oven, slowly cooled, inserted in a furnace, re-heated to a desired temperature, and then stressed for strength and elongation measurements.¹ Some question can be raised concerning the applicability of the results obtained in such tests. For example, the initial slow heating and subsequent thermal cycling are not representative of the rapid heating which occurs in reentry or rocket engine firing where the ablative materials may have been post-cured at a relatively low temperature but are not usually precharred. The chemical state of charred material could be sensitive to the charring rate. If this is the case, then it can be expected that the microstructural and strength characteristics are, to some extent, also sensitive to the thermal history.

For investigation of the influence of heating rate on the mechanical properties of ablative materials at elevated temperatures, a technique has been devised which permits much faster heating of material than in previous

work.¹ The apparatus and method are described, and results obtained for strength and stress-strain properties of rapidly heated, laminated ablative materials are reported. Initial strength results were previously reported in Aerospace Corp. report TR-1001(2240-10)-7, A New Technique for Mechanical Strength Testing of Rapidly Charred Ablation Materials, dated February 1967.

II. APPARATUS AND TEST PROCEDURE

A. HEATING AND THERMAL ENVIRONMENT

A conventional plasma arc provided hot gas to heat the test specimens of ablative material. This arrangement is shown in Fig. 1. A slender specimen was positioned such that its central region was heated by a flow of hot nitrogen gas from an electric plasma arc. The input power level was adjusted to yield the desired gas and specimen temperature. A similar test configuration with a somewhat cooler gas jet was used by Vassallo, et al.² in a study of the tensile strength of incompletely charred ablation materials. However, in those tests, failure strengths were obtained while the central region of interest was in a transient thermal environment.

The plasma arc used in the present study has a nominal rating of 200 kW and uses a thoriaated tungsten cathode and copper anode. Outlet temperature of the nitrogen is estimated to be 6500°K. The plasma arc nozzle provides a subsonic outlet flow of 0.500 in. diam. A test chamber encloses the strength-testing apparatus, test specimen, and arc-heated outlet flow. This chamber is pressurized slightly above atmospheric pressure with cool nitrogen so that atmospheric oxygen (which would oxidize the test specimen) can not diffuse into the test region. A 1-in.-diam. exhaust port is located at the downstream end of the test chamber.

Specimen temperatures (on the exterior observed surface) typically achieved a steady distribution after 4 to 10 sec of heating. A representative

steady specimen temperature field is shown in Fig. 2. The central external region of interest was viewed with an automatic recording pyrometer. The internal specimen temperature was not measured because of unfavorable geometry for thermocouple accuracy. Using a well-proven computer program for ablation response,* W. Herbig of Aerospace Corp. has made calculations which show that the central internal temperature rises to within 50°F of the external observed temperature in 5 sec after the start of heating. These calculations were made for a phenolic-carbon laminate rod, having a 0.12 by 0.15-in. cross section (used for tensile tests) and a maximum central external observed temperature of 4850°F. Further calculations are in progress for the other geometries and materials utilized in these tests. It is probable that some cases will require somewhat more than 5 sec for internal temperature equilibration.

B. TENSILE STRENGTH

The tensile test fixture was designed to fit the internal dimensions of the arc tunnel in order to avoid modification of the existing tunnel. Figure 1 shows a layout of the test fixture in the arc tunnel just downstream of the arc flow outlet nozzle. A compressed air cylinder used to provide the tensile load was located at the top of a "C"-shaped loading frame. To avoid imposition of bending moments on the specimen, universal joints were incorporated

*The program is described by C. Anderson, TOR-669(S6855-20)-3, Aerospace Corp., San Bernardino Operations, San Bernardino, Calif. (February 1966).

in the loading train above and below the specimen. Below the lower universal joint was a load cell. A cooling coil was installed around the upper end of the load cell to prevent its overheating. This permitted testing at higher temperatures and for longer durations. The output signal from the load cell was plotted on an x-y recorder to give a load-time curve.

A typical loading rate of 25 lb/sec was equivalent to about 0.20 in./min crosshead speed for the specimen geometry used. Nitrogen was used in the loading system to eliminate the possibility of oxygen entering the arc tunnel. A micrometer valve was used to control the gas flow to the compressed air cylinder to give the desired rate of loading.

Tensile test specimens were machined to an hourglass shape (stress concentration factor approximately 1.05) from thin panels of ablation material. The specimen thickness was constant at the as-received panel thickness. This configuration, shown in Fig. 3a, was adopted after exploratory tests of carbon and graphite laminates employing specimens with straight test sections failed in regions other than the central zone of maximum temperature. The failure away from the hot central zone was due to the increase in strength with temperature (above about 2500°F) that these two materials experienced. Straight test sections could be used for the silica laminate specimens, because this material shows a continuous drop in strength with increasing temperature. Sheet thickness was typically 0.12 in. (9-ply), and the cross sectional dimensions at the central minimum area location were typically 0.12 in. by 0.15 in.

The desired specimen temperature was attained by adjusting the input power level for the arc and the positioning of the loading frame with reference to the arc nozzle. Loading was commenced after the central temperature had remained constant for at least 3 sec, so that penetration of heat and charring within the central portion of the specimen would be ensured. Opening a gas valve to the loading cylinder caused linear increase of load with time over about 4 sec. Upon failure of the specimen, the arc flow was terminated. Typical temperature-time and load-time curves are shown in Fig. 4.

C. TENSILE STRESS-STRAIN

The tensile stress-strain tests used the same fixtures, instrumentation, and procedures that were used for the tensile strength tests described in Section B. However, the specimen geometry was modified to provide a central gauge section with reference holes, as shown in Fig. 3b, for observation of strain. In addition, motion pictures of the specimen test section were taken for the duration of each test. The framing rate was 48 frames/sec with an exposure time of 0.01 sec/frame. The amount of strain (elongation), which is the change in gauge length caused by stress (load) and defined by the upper and lower set of holes in the gauge section, was obtained from the movies by the use of a film reader. The frames just prior to loading were used to establish a reference gauge length. Subsequent readings from start of loading to failure gave a complete strain history of the specimen. A strain-time curve was derived from the data; and from that, in conjunction with the stress-time curve, the tensile stress-strain curve was constructed.

Time synchronization of stress and strain was done by manually marking the film at the instant of loading. Accuracy of this procedure was within about 5 frames.

D. INTERLAMINAR SHEAR STRENGTH

The same fixtures, instrumentation, and procedures used for the tensile strength tests described in Section B also were utilized for the interlaminar shear tests. The specimen geometry was changed, as shown in Fig. 5a, so that the interlaminar shear failure mode could be obtained. Two parallel cuts, one on each opposite face of the specimen and 0.375 in. apart, were sawed across the entire width of the specimen. The cuts were of sufficient depth to sever the center lamina located midway between the two faces of the laminate. Method 1042-B of the Federal Test Method Standard No. 406³ was used in the tests.

E. COMPRESSIVE STRENGTH

The compression test setup was similar to that used for tensile strength testing as described in Section B. Here, however, the specimen geometry was modified as shown in Fig. 5b to effect a compression failure mode, and the loading frame and fixtures were changed to provide a rigid load train (see Fig. 6). Otherwise, the same instrumentation and procedures were utilized.

Upper and lower guide pins, designed with slip fits, ensured a near-axial loading. An alignment plate was used to set the upper and lower guide pins in position. The guide pins were restricted to axial motion with no

lateral movement or shake. Steel pins (0.312 in. diam.) were used to hold the specimen to the upper and lower clevises. Clamping plates held the specimen snug against the clevises.

The compressed-air cylinder used to provide the compressive load was located at the top of the loading frame. The load was transmitted from the air cylinder through the upper clevis into the specimen and thence to the lower clevis. The lower clevis rested on a stud protruding from a load cell at the bottom of the loading frame. From the lower clevis, the load was transmitted through the round-ended stud into the load cell.

The compressive specimen of Fig. 5b was proportioned so as to avoid failure by gross buckling. Photoelastic models with this geometry were used to check the load train alignment of the fixture at room temperature. Model thicknesses ranged from 0.125 in. to 0.250 in. The fringe or stress patterns showed near-axial loading with less than 5% bending involved for all thicknesses. This showed that the rigid load train and specimen geometry were adequate for compressive testing and that the specimen axis could be slightly off the load axis without the need of shimming to provide perfect alignment of the specimen with the load.

III. RESULTS

A summary of the strength and stress-strain test results of rapidly heated laminated ablative materials is shown in Table 1. Room temperature tensile and compressive strength data from Hitco, the manufacturer of the materials, are shown with the results of the present strength tests.

Ultimate tensile strength results for carbon, graphite, and silica phenolic materials are shown in Fig. 7. Photographs of typical fractured carbon and graphite tensile specimens are shown in Fig. 8. The tensile strength of silica phenolic was obtained from tensile stress-strain specimens, and a photograph of a typical fractured specimen is shown in Fig. 9. All materials exhibited a significant loss of strength between room temperature and 2500°F. The carbon and graphite showed a slight gain of strength between 2500°F and 4500°F, whereas the silica showed a continuing loss of strength with increasing temperature. The experimental scatter of data at any temperature level was less than $\pm 15\%$. No data are available for these materials with comparable fabric orientation from earlier tests conducted under slow heating conditions. However, the data of Schneider, et al.,¹ representing a composite of results for carbon laminates tested at various fabric orientations, are shown in Fig. 10 for qualified comparison. The lower strength levels indicated by these results may be due in large part to the effect of the low interlaminar shear strength of charred laminates (see Fig. 14) on the tensile strength of specimens that have a finite angle between

Table 1. Summary of Results

| Test | Hitco Carbon Phenolic CCA-1/EC201 Virgin, non- post-cured. | Hitco Graphite Phenolic G-1550/EC201 Post-cured at 300°F. | Hitco Silica Phenolic-Refrasil C-100-48/SC 1008 Post-cured at 325°F. |
|---|--|---|--|
| Tensile Strength (Warp Direction) | Fig. 7 | Fig. 7 | Fig. 7 |
| Tensile Stress- Strain (Warp Direction) | -- | Fig. 11 | Figs. 12 and 13 |
| Inter-Laminar Shear Strength | -- | Fig. 14 | -- |
| Compressive Strength (Warp Direction) | -- | Fig. 16 | Fig. 16 |

the fabric plies and the specimen axis. Thus, it cannot be concluded on the basis of available data that heating rate has a significant effect on the tensile strength of charred ablation materials. Further testing, in furnaces, on specimens with orientation similar to that of the specimens used in the present tests is required before a definitive comparison is possible.

Results of the tensile stress-strain tests are shown in Fig. 11 for graphite laminate and in Figs. 12 and 13 for silica laminates. Initial elastic modulus, E , and yield stresses are also shown in Figs. 11 and 12. With the use of a film reader, the strains were obtained from motion pictures taken during these tests.

For the graphite laminates, the failure strains* were not obtained, since failures occurred away from the gauge section with the failure stress lower than the ultimate value for the hot central area. Failures away from the central zone of maximum temperature for these specimens with straight test sections are explained in Section B. Only one specimen was tested at each temperature, and during these preliminary tests no temperature record was obtained. The temperatures were estimated to be about 3750°F and 4850°F, based on previous temperature calibration runs. The single-sample data for graphite laminates, shown in Fig. 11, are subject to verification in further tests. However, these curves give an indication of the nominal behavior that may be expected for this material.

The tensile stress-strain results for silica laminates at about 2850°F (Fig. 12) show good repeatability, whereas those at about 3100°F (Fig. 13) show some scatter. This scatter could be expected, since the temperature is near the melting point of about 3250°F for this material. All failures occurred within the gauge length for these tests. A photograph of a typical fractured specimen is shown in Fig. 9.

Interlaminar shear strength tests were performed on post-cured graphite phenolic specimens; the results are plotted in Fig. 14. Shear stresses of from 250 to 360 psi were obtained at temperatures from 2500 to 4500°F. Typical specimen fracture at all temperatures is shown in Fig. 15.

*If these strains are desired, a slight modification of the specimen geometry could assure failure within the gauge length.

The results of the compression tests at elevated temperatures for graphite and silica laminates are shown in Fig. 16. Both materials show a continuous drop in strength with increasing temperature for the ranges of temperature that were used. The tensile test results at these temperatures show increasing strength with increasing temperature for the graphite laminates and a decrease in strength for the silica laminates. The contrary trends of the compressive and tensile strengths of graphite laminates with increasing temperatures could be attributed to the resin strength, which is very low at these temperatures. The tensile strength is almost independent of the resin strength, whereas for compression, the cloth layers are dependent on the resin bond to provide support to preclude buckling of the individual layers.

The failure patterns of both graphite and silica laminates in compression appear to be the same. Although movies were taken of some of the tests, no well-defined failure mechanism was detected from them. Since these were the first tests done in compression, the movies were taken at a rate (150 frames/sec) to cover the full duration of each test from start of arc to specimen failure so that any flaw, such as delamination or erosion that could cause premature failure, or the failure triggering mechanism itself, could be detected. The films showed no well defined evidence of delamination or erosion; and failures in all cases were very abrupt, i. e., stable in one frame, complete failure in the following frame. It is evident that much higher framing rates would be necessary to study the compression

failure mechanism. No definite failure mode could be established from a study of the specimens after failure because the fractured area had been distorted by the loading mechanism. After failure, the piston rod continues to drive through to complete its travel of about 0.20 in. This causes the upper and lower sections of the specimen to wedge together. Examination of the specimens after each test show evidence of shear and delamination. A photograph of a typical fractured specimen is shown in Fig. 17.

Tensile strength and tensile stress-strain tests at elevated temperatures were run with ATJ graphite as a check on the performance of the tensile loading apparatus and on the method of determining strains with the use of movies. These tests were run at temperatures and time periods that were typical of those used for the laminated materials, and the same specimen configurations were used. The results showed good agreement with available data on tensile strength and initial elastic modulus from furnace type tests where the heating rates were considerably lower. Figure 18 shows the ATJ graphite strength results with the furnace test data of Martens, et al.,⁴ and the Air Force Materials Laboratory⁵ included for comparison.

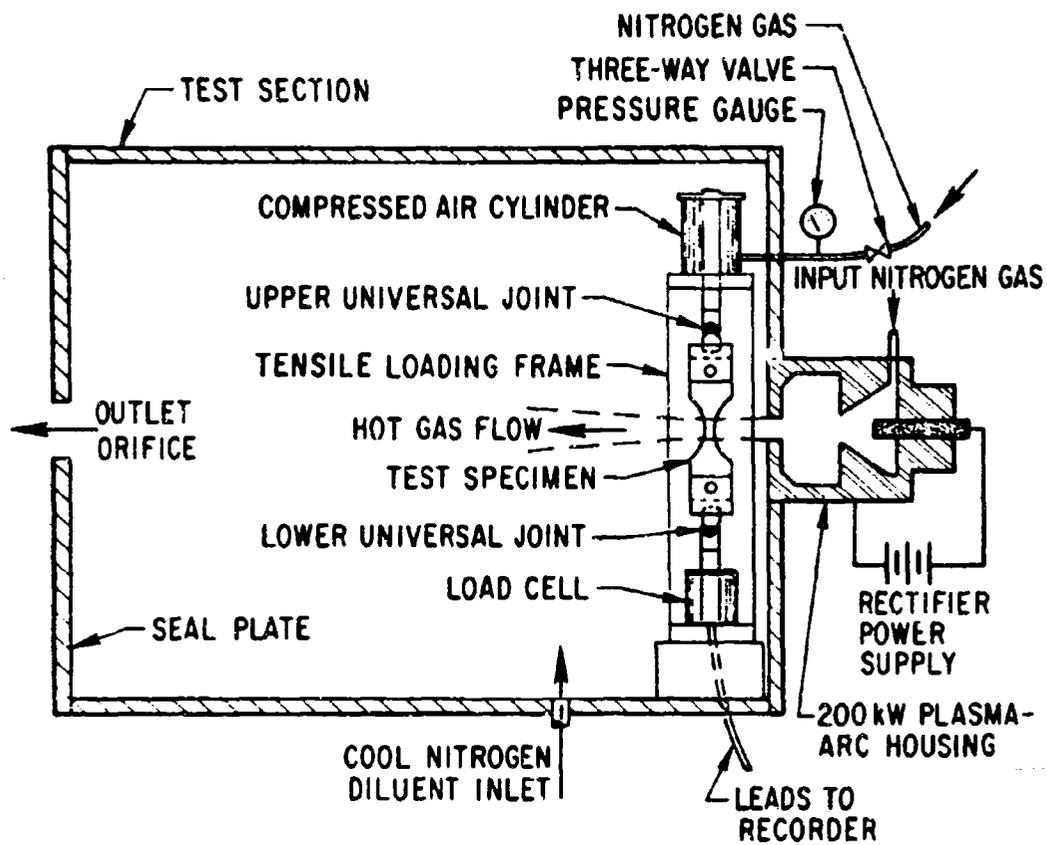
IV. FUTURE WORK

Efforts will be made to increase the maximum specimen temperature from 4850°F to a higher level by use of radiation shields. The effects of strain and heating rates, as well as time, at a given temperature on the ultimate strengths of ablative materials will be investigated. It has been suggested that laminated ablative materials are sensitive to strain rate at elevated temperatures. Reference 6 presents some high strain-rate results generated by General Motors Defense Research Laboratory on carbon and silica phenolic materials between room temperature and 600°F. Significant sensitivity was observed for both materials at the higher temperatures in compression tests.

It is planned to use the method that was used for tensile stress-strain to obtain the following: compressive stress-strain, thermal expansion, Poisson's ratios, very short time creep, and permanent set or the strain remaining after the removal of stress.

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5. "Research and Development on Advanced Graphite Materials," WADD TR 61-72, Vol. XIV (April 1964).
6. Breshears, S. R., "Comparative Properties of Heatshield Materials," Aerospace Corp. Report No. ATR-67(S2815-33)-1 (December 1966).



SIDE VIEW (CENTRAL CROSS SECTION)

Fig. 1. Experimental Arrangement.

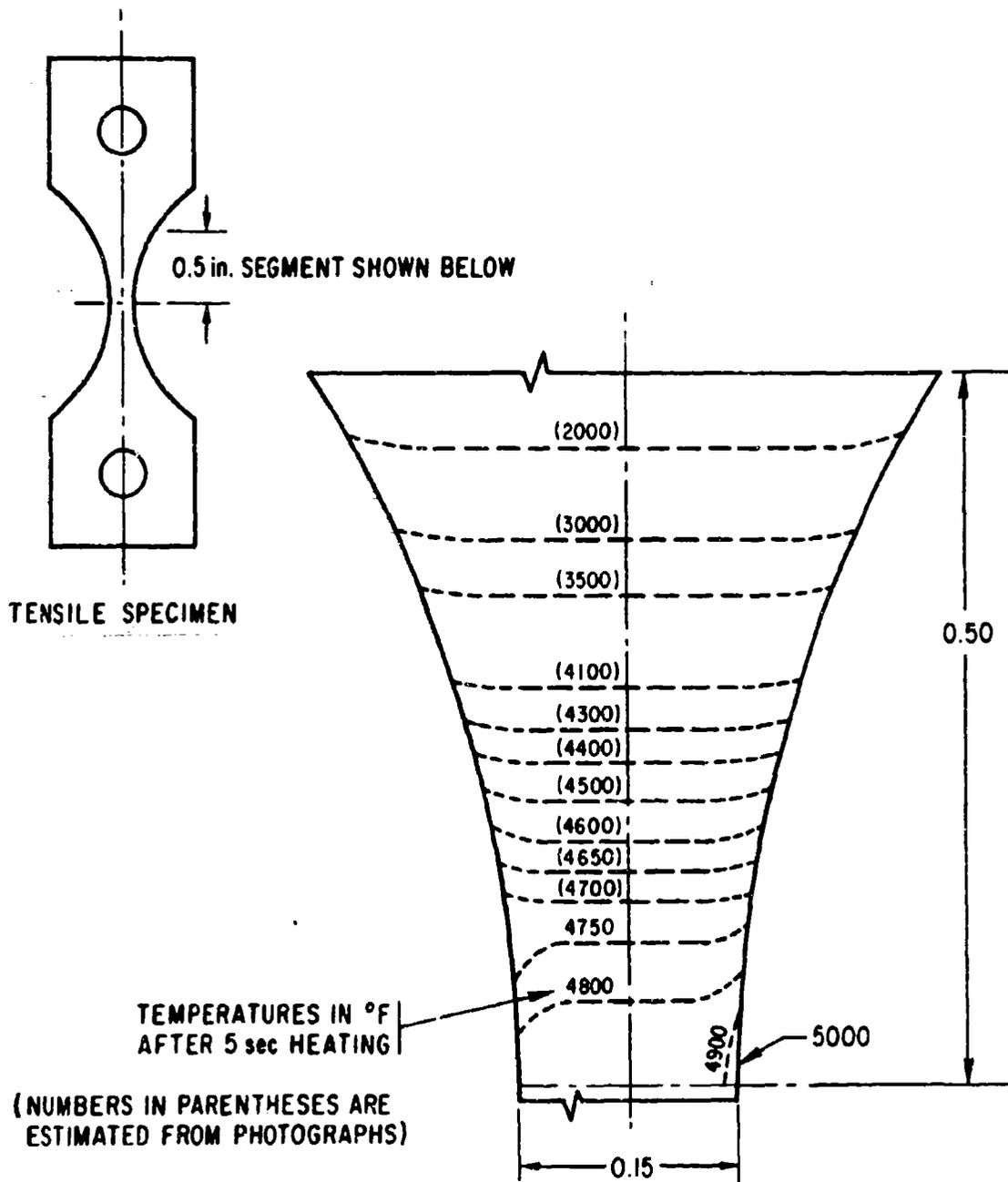
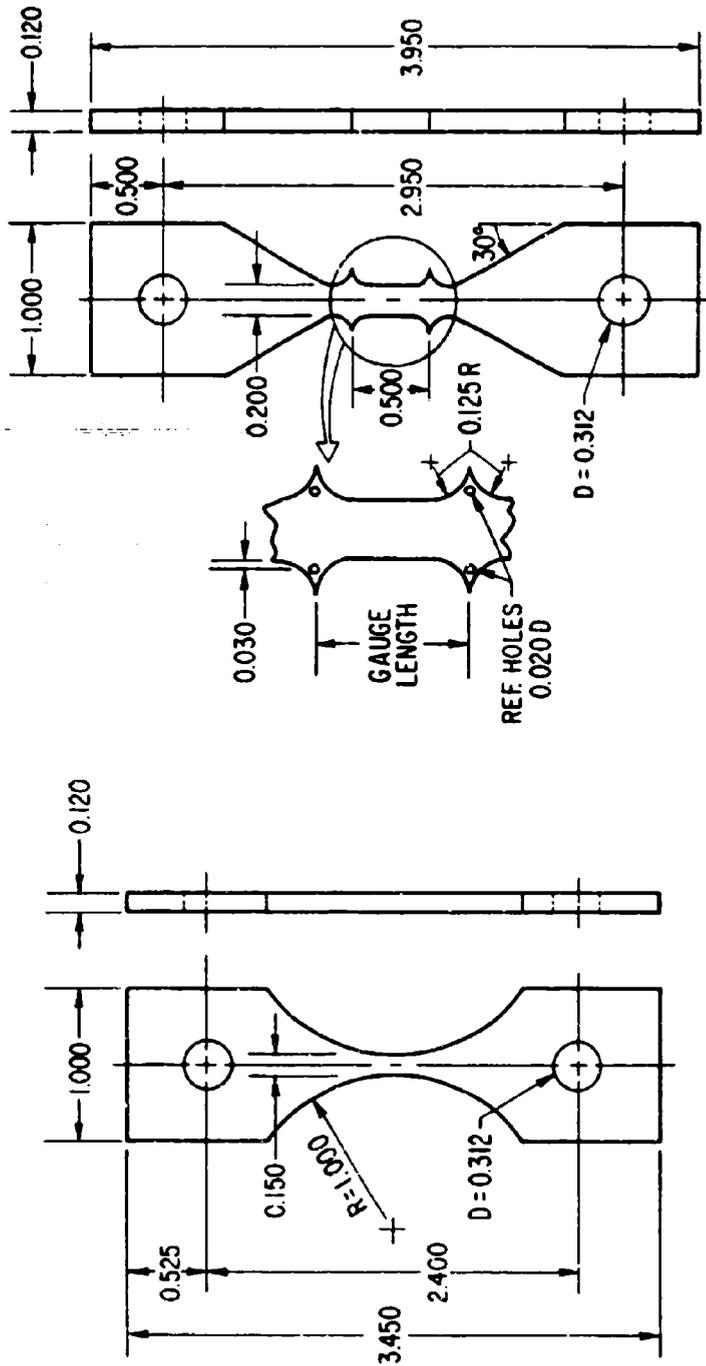


Fig. 2. Temperature Field on Exterior Side Surface of Tensile Specimen.



(DIMENSIONS IN INCHES)

Fig. 3. Typical Tensile and Tensile Stress-Strain Specimen Geometries.

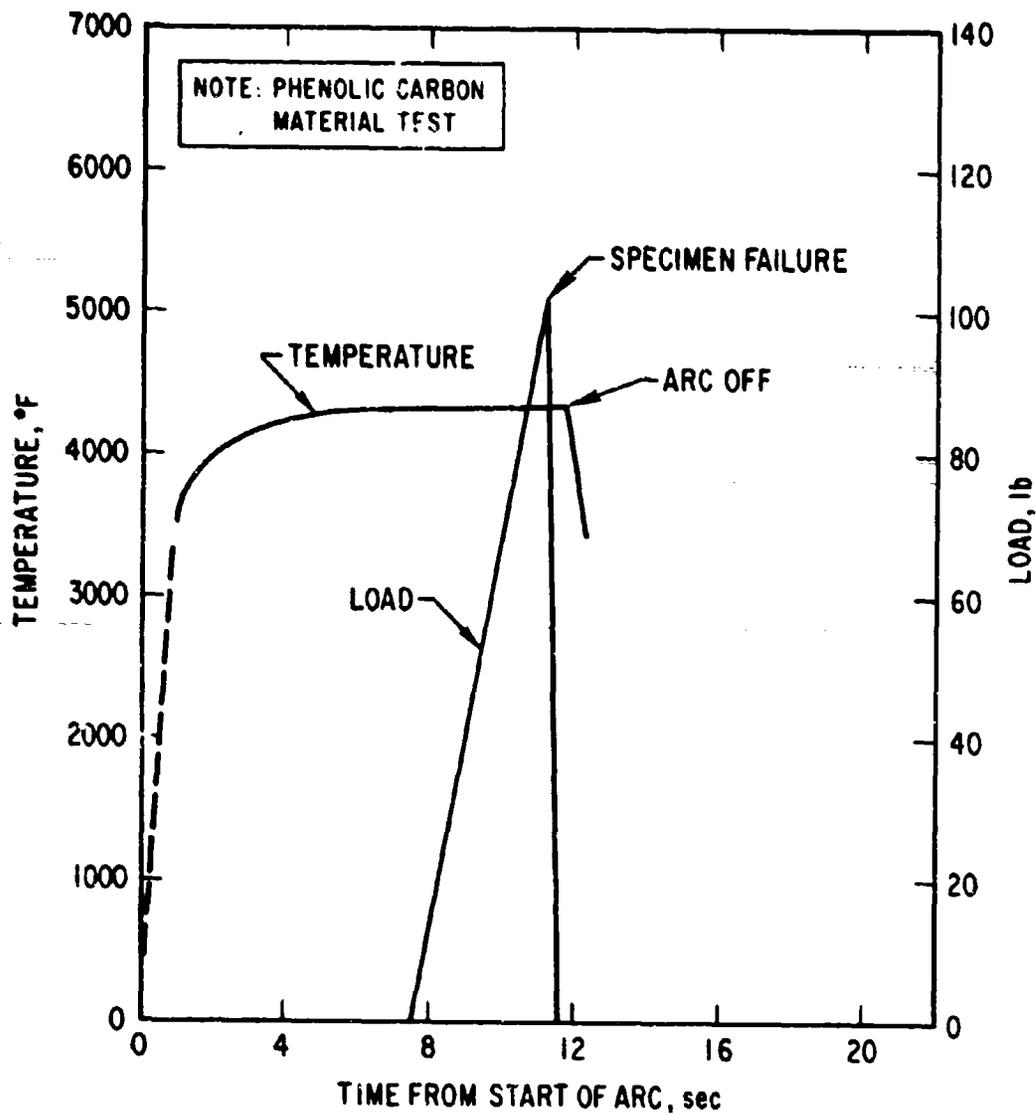
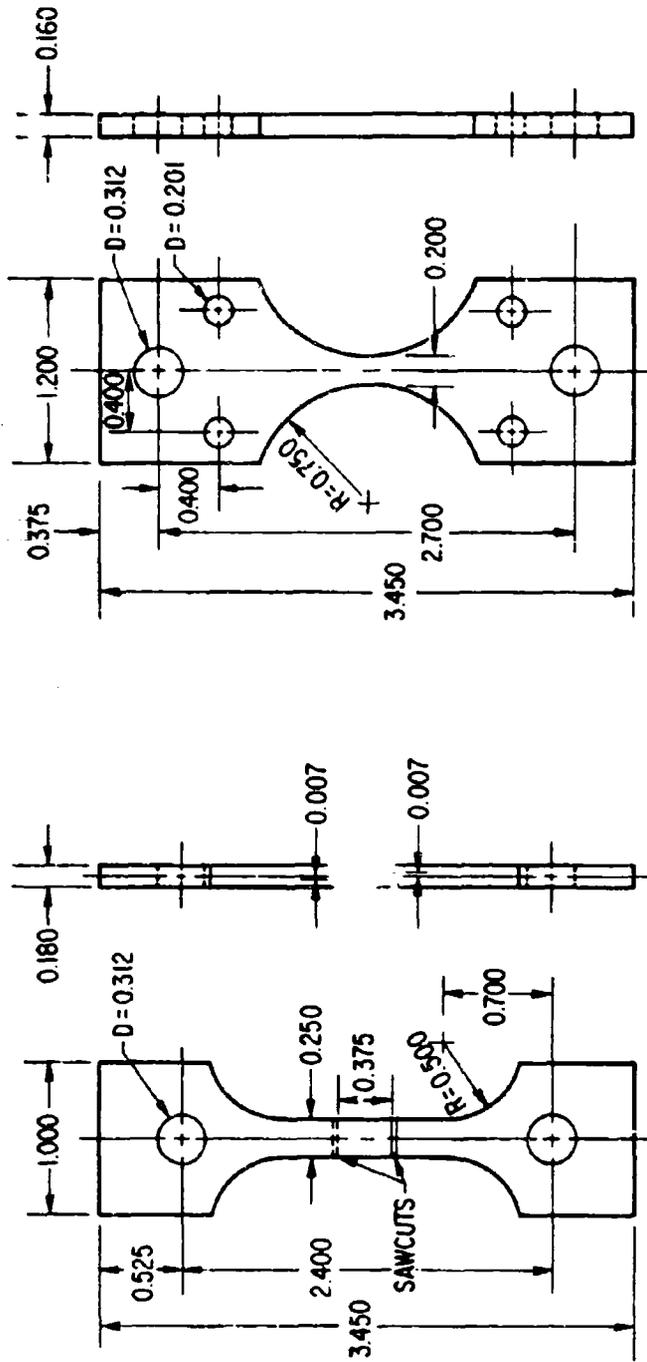


Fig. 4. Typical Temperature and Loading Histories.



a. INTERLAMINAR SHEAR SPECIMEN

b. COMPRESSIVE SPECIMEN

(DIMENSIONS IN INCHES)

Fig. 5. Typical Interlaminar Shear and Compressive Specimen Geometries.

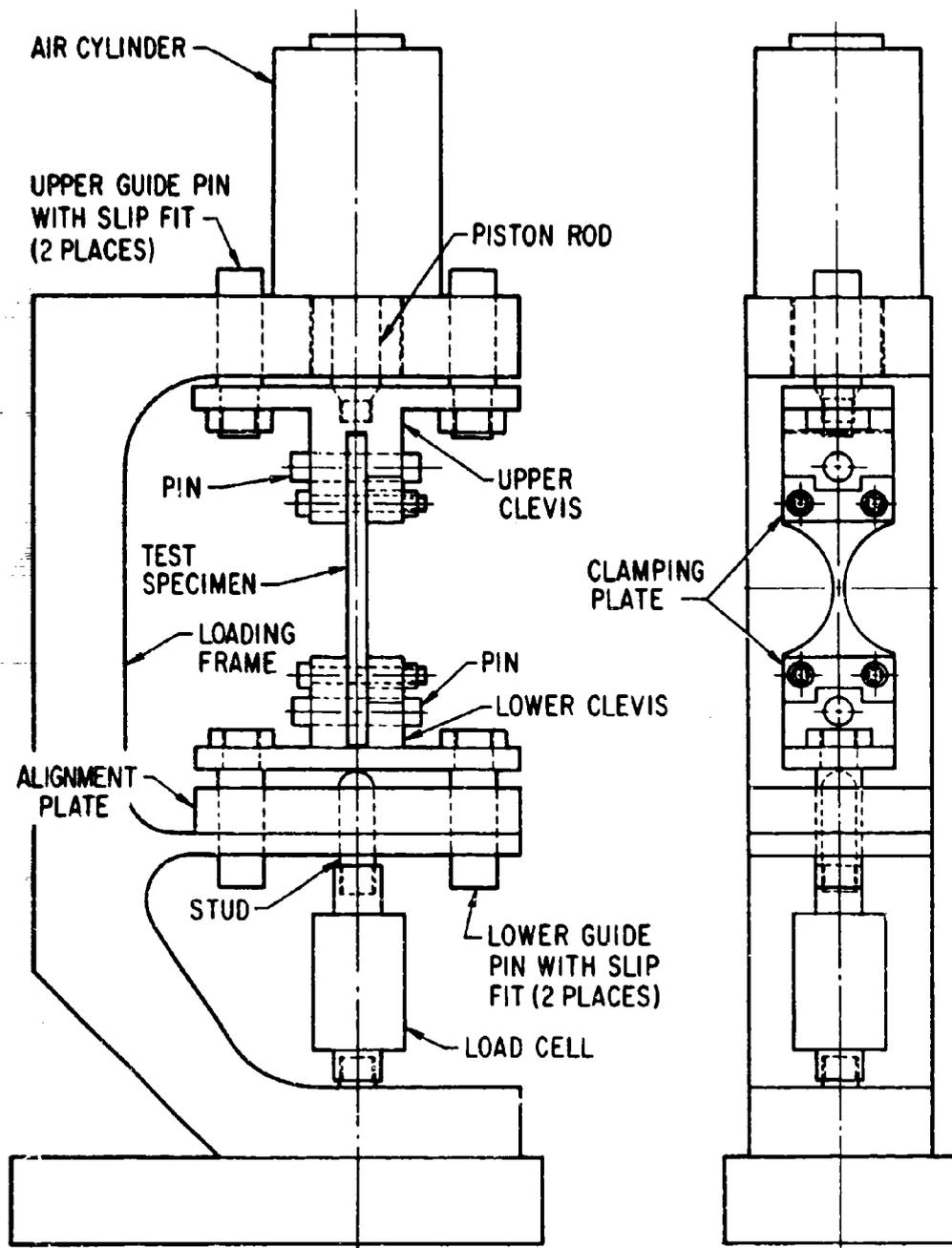


Fig. 6. Compressive Strength Test Fixture.

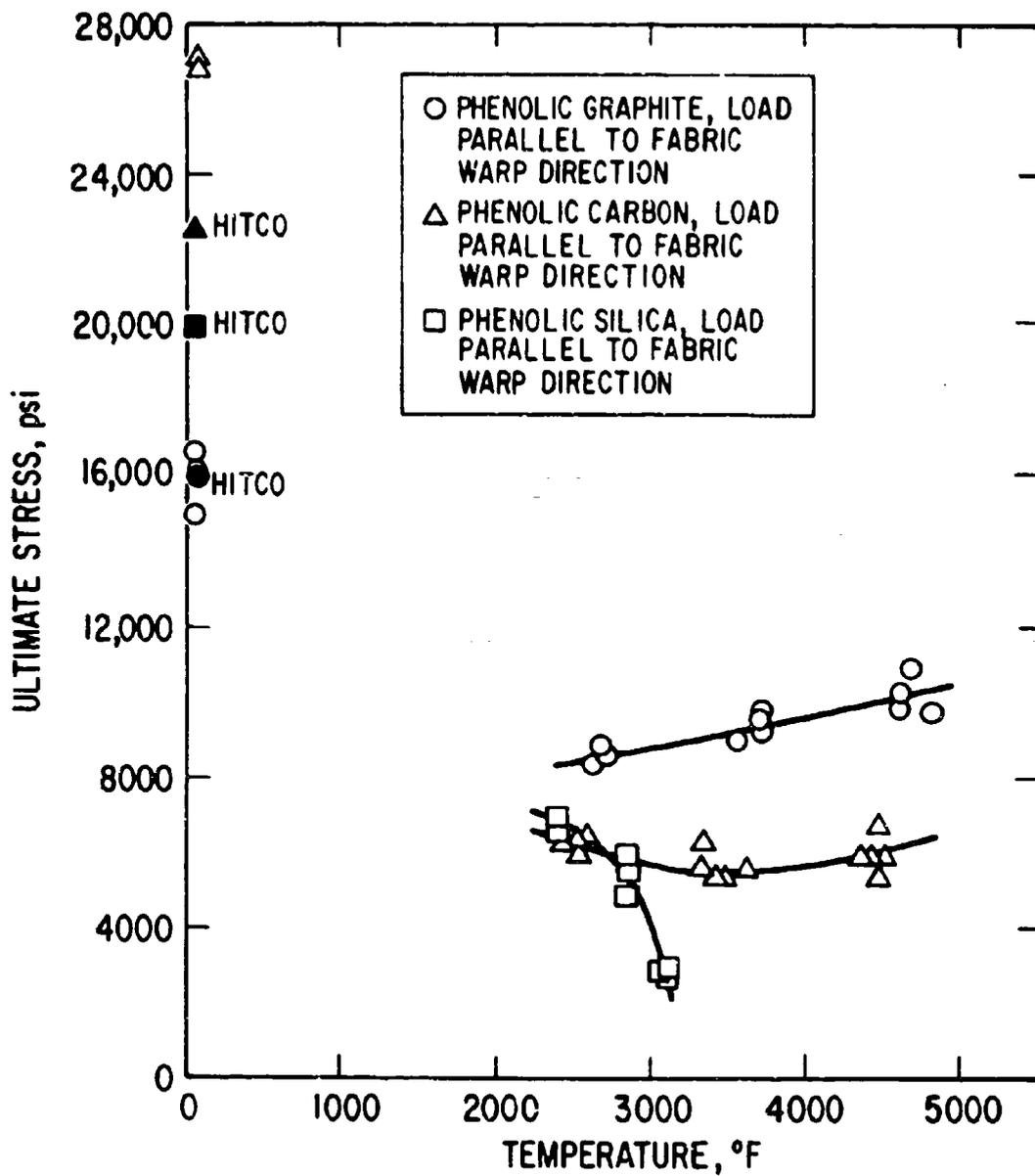
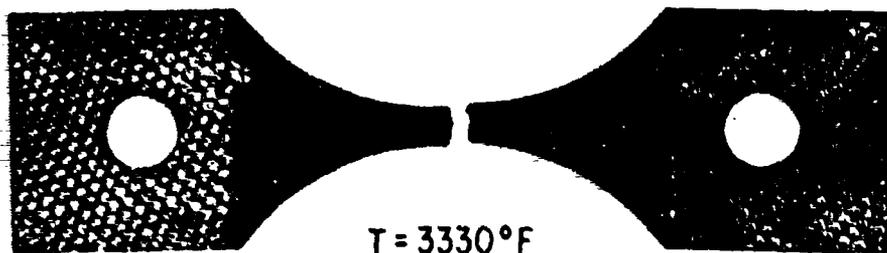
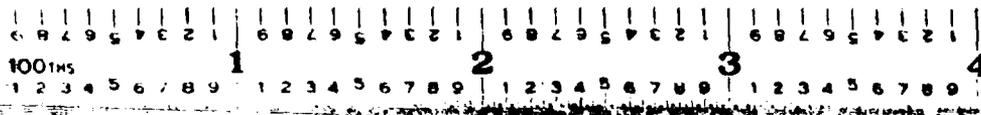


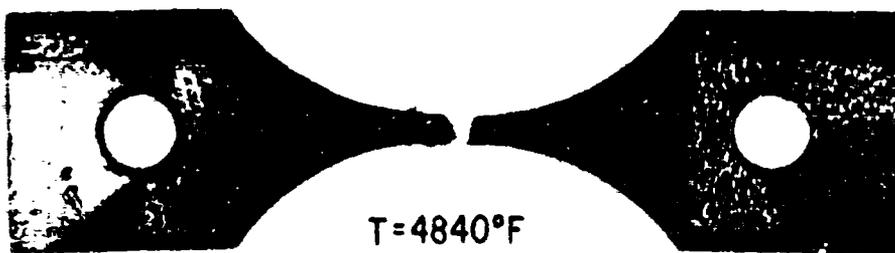
Fig. 7. Ultimate Tensile Stress in Warp Direction vs Temperature.



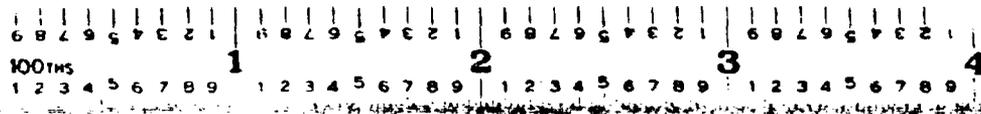
T=3330°F



a. CARBON PHENOLIC SPECIMEN
(CENTRAL CROSS SECTION = 0.12 in. x 0.150 in.)

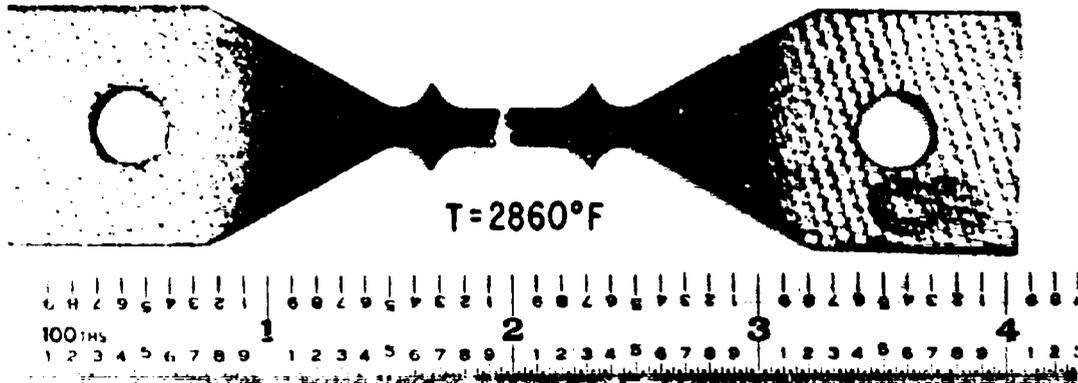


T=4840°F

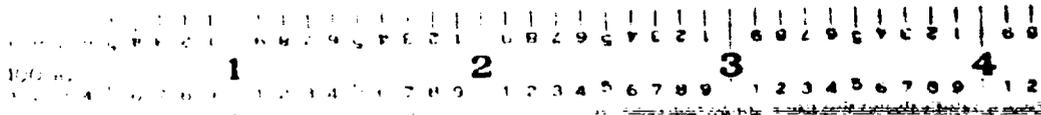


b. GRAPHITE PHENOLIC SPECIMEN
(CENTRAL CROSS SECTION = 0.12 in. x 0.125 in.)

Fig. 8. Typical Tensile Fracture of Carbon and Graphite Phenolic Specimens at All Temperatures.



FRONT VIEW



SIDE VIEW

Fig. 9. Typical Tensile Fracture of Silica Phenolic Stress-Strain Specimen at All Temperatures.

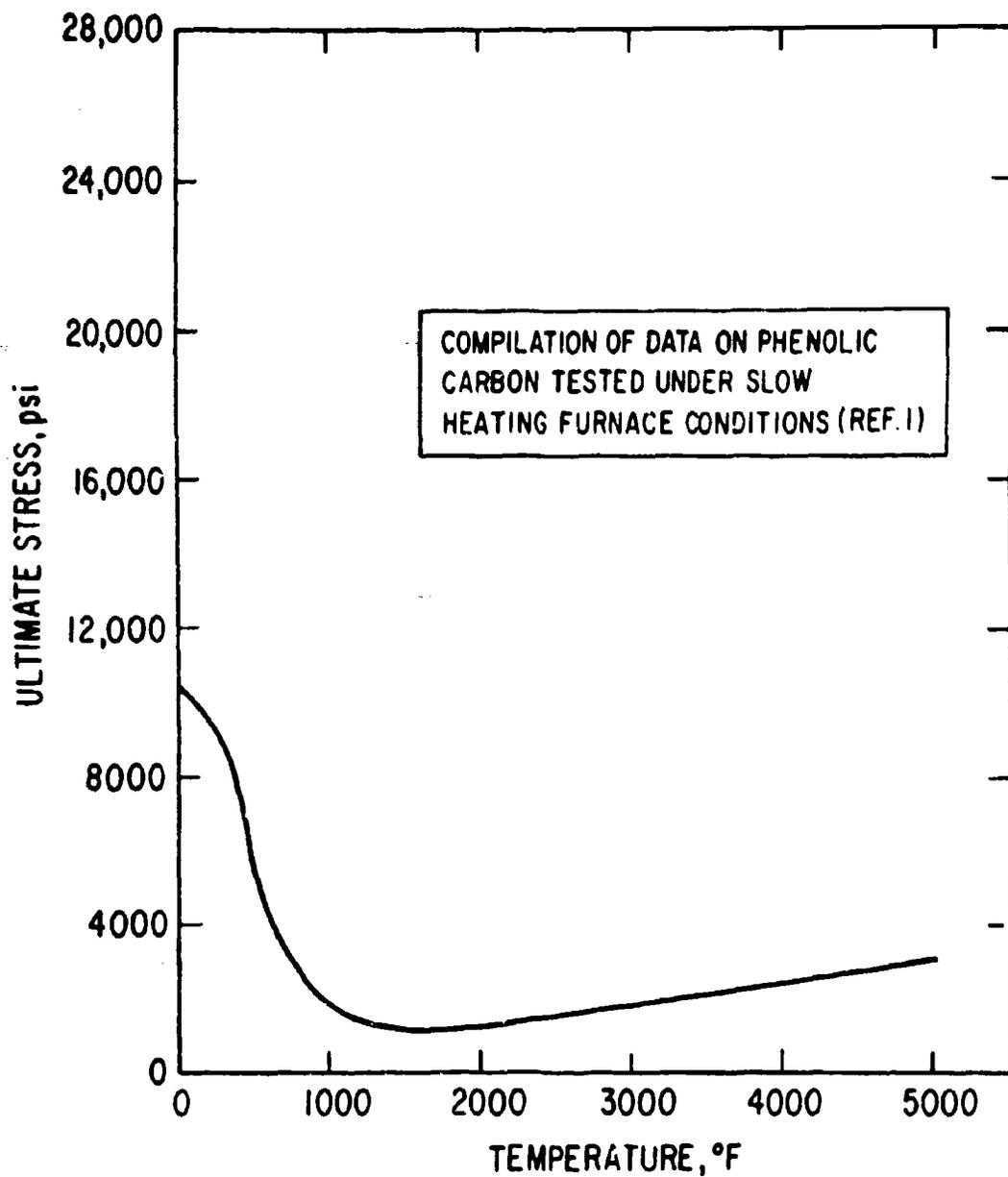


Fig. 10. Ultimate Tensile Stress in Tangential Direction vs Temperature.

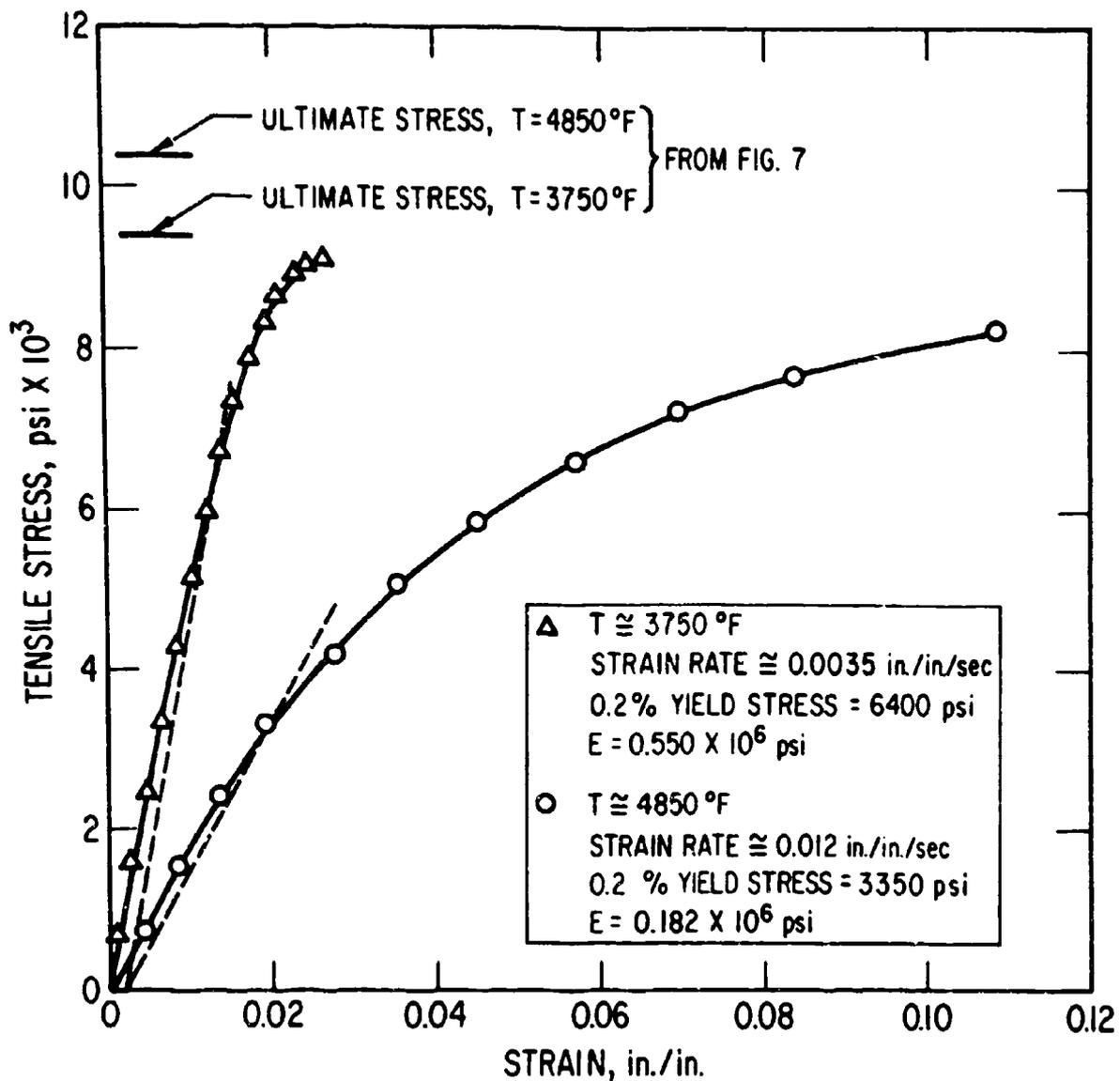


Fig. 11. Tensile Stress-Strain Curves for Graphite Laminate in Warp Direction. (Failures occurred away from hot central gauge area. Failure stresses and strains were not obtained for the given temperatures.)

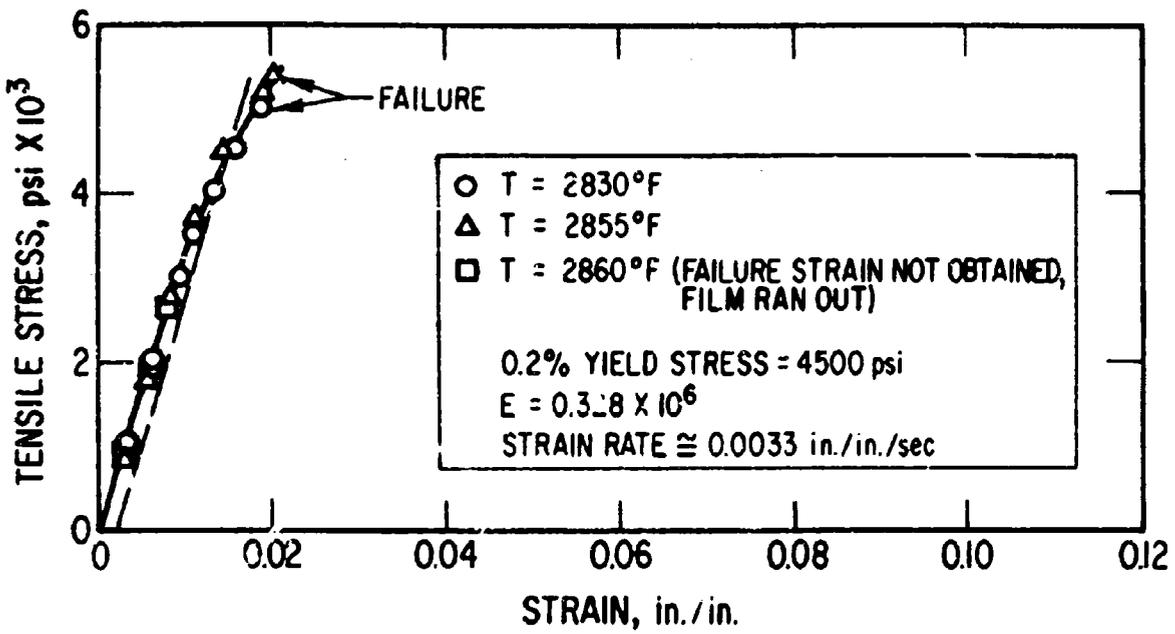


Fig. 12. Tensile Stress-Strain Curves for Silica Phenolic in Warp Direction at about 2850° F.

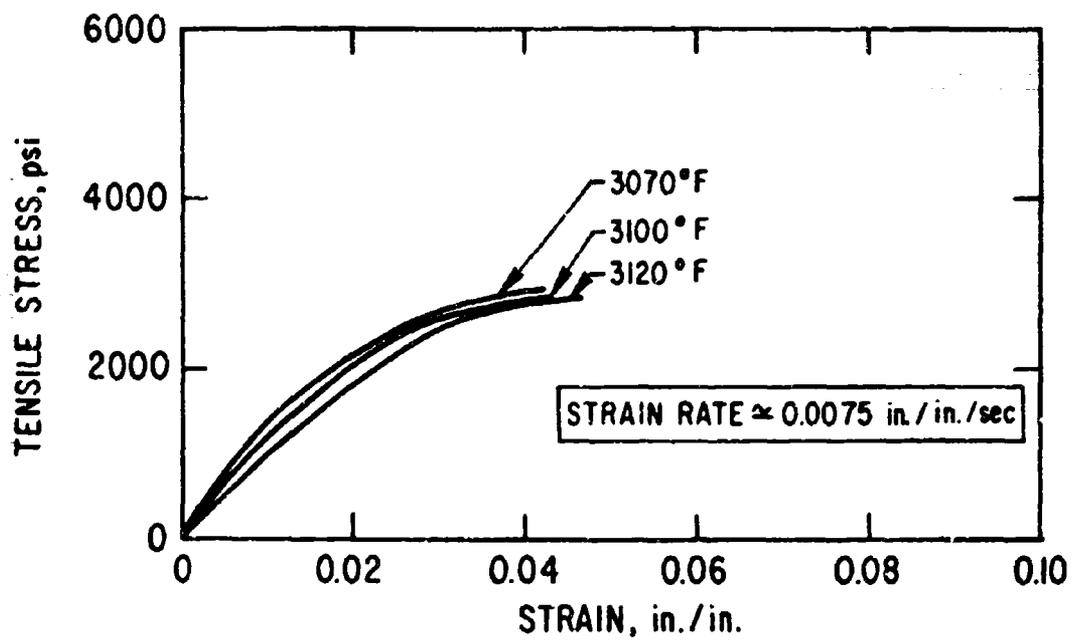


Fig. 13. Tensile Stress-Strain Curves for Silica Phenolic in Warp Direction at about 3100° F.

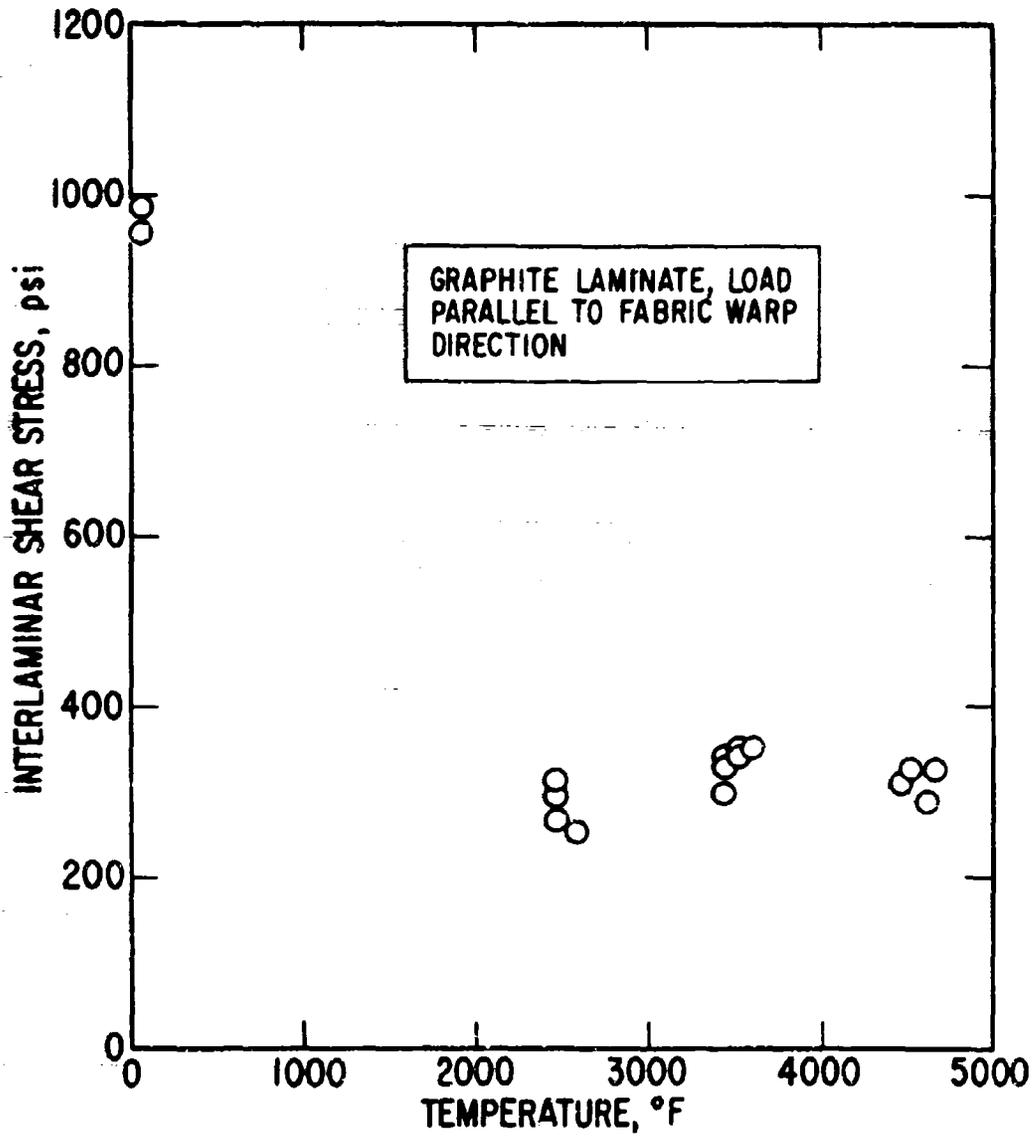
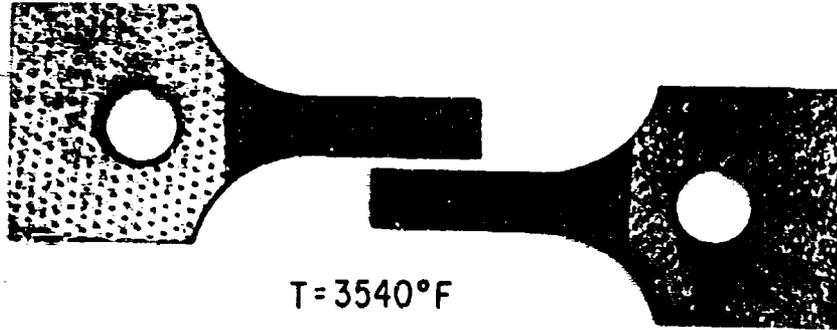
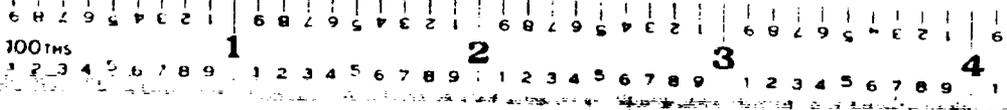


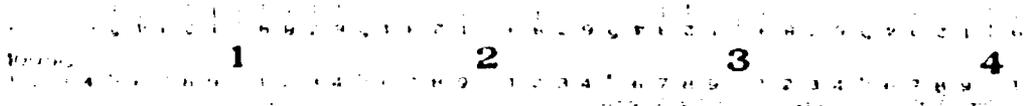
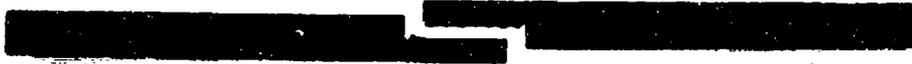
Fig. 14. Interlaminar Shear Stress vs Temperature.



T=3540°F



FRONT VIEW



SIDE VIEW

Fig. 15. Typical Interlaminar Shear Fracture of Graphite Phenolic Specimen at All Temperatures.

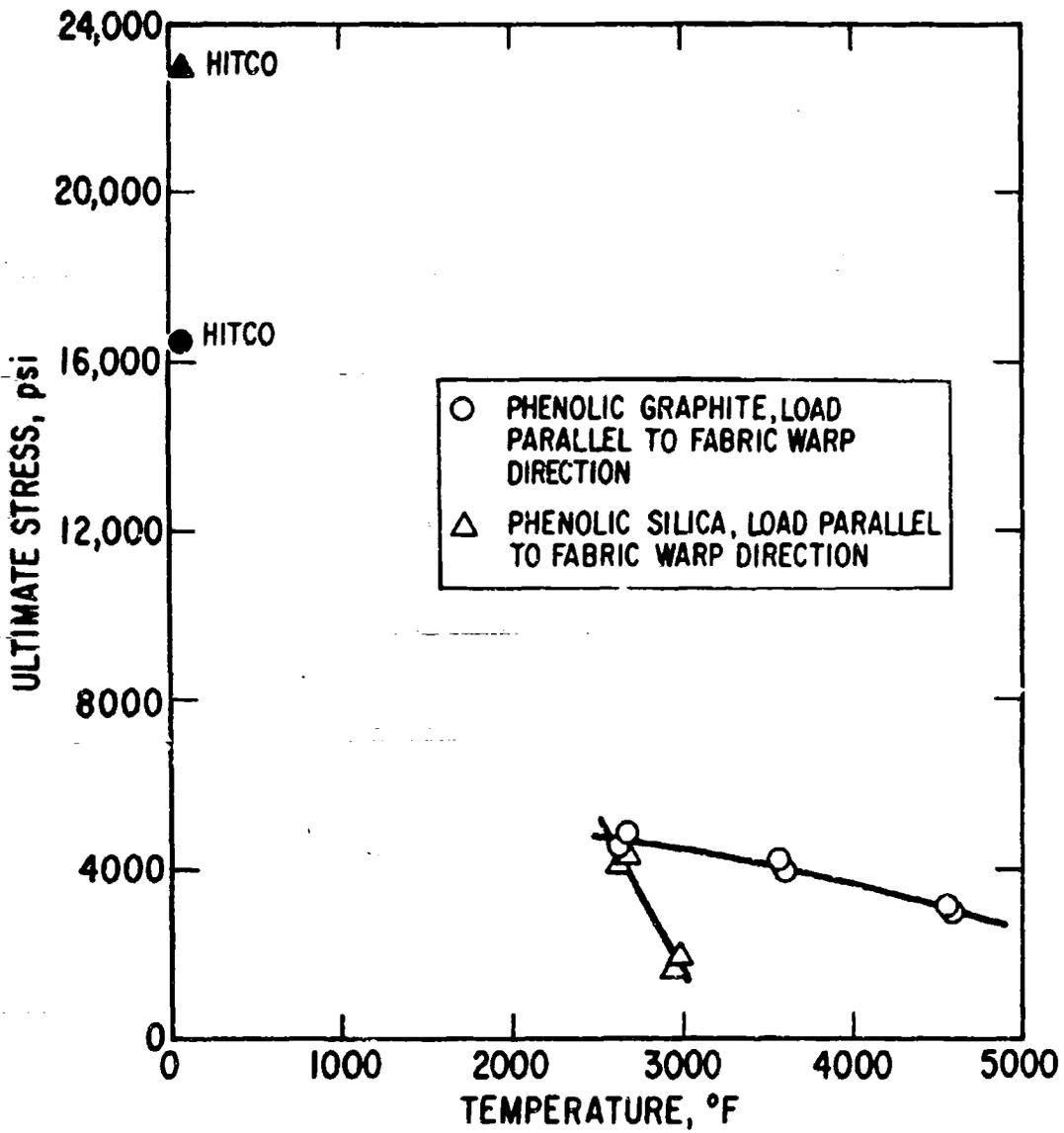
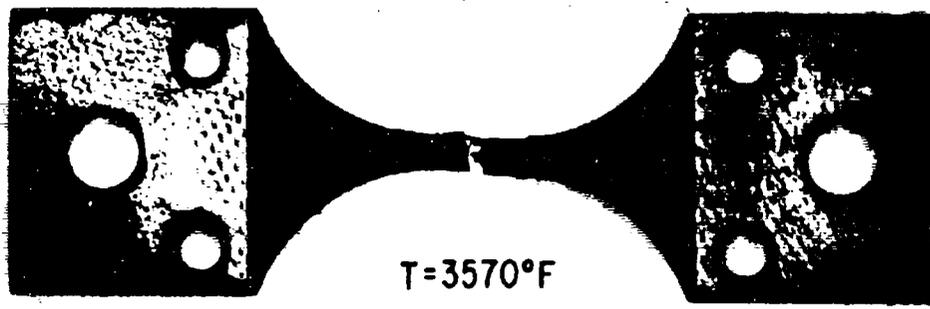
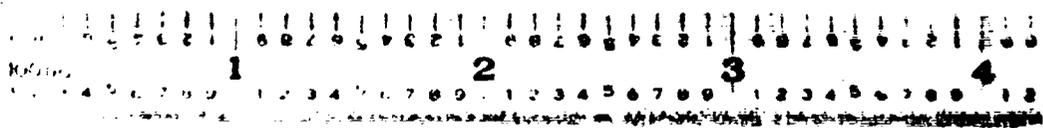


Fig. 16. Ultimate Compressive Stress in Warp Direction vs Temperature.



FRONT VIEW



SIDE VIEW

Fig. 17. Typical Compressive Fracture of Graphite and Silica Phenolic Specimens at All Temperatures. (Graphite Specimen Shown)

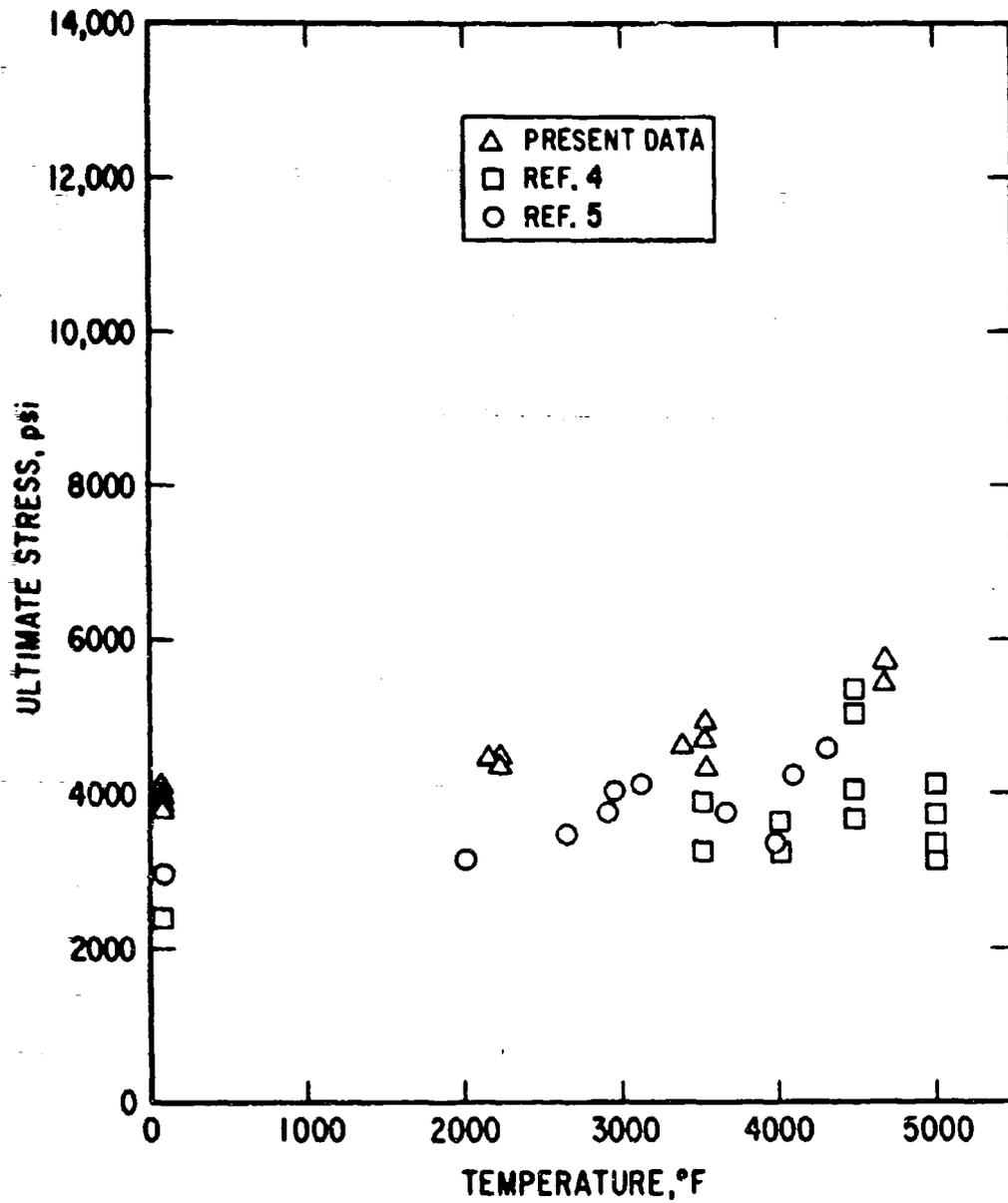


Fig. 18. Comparison of ATJ Graphite Tensile Stress Parallel to Grain.

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| 13 ABSTRACT <p>It is shown that the mechanical properties of rocket nozzle liners and reentry vehicle ablation materials can be determined by tests in which a high temperature plasma arc is used as a heat source. The arc flow is used to heat slender specimens, and a pneumatically actuated loading frame is used to apply stress. This method of testing provides heating and loading in time periods typical of the rapid heating during rocket firing or reentry, on the order of 10 sec. Earlier investigations of similar ablative materials have used specimens heated and charred (degraded) slowly prior to the application of load.</p> <p>Strength and stress-strain properties in the warp direction were determined for carbon, graphite, and silica phenolic materials. The method appears promising for the determination of properties in various loading-laminate orientations and during thermal expansion, and for studies of heating and strain rate effects on strength at elevated temperatures.</p> | | |

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