FATIGUE TEST OF PHENOLIC LAMINATE
AT HIGH STRESS LEVELS AND
ELEVATED TEMPERATURES

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In Cooperation with the University of Wisconsin
FATIGUE TEST OF PHENOLIC LAMINATE AT HIGH STRESS LEVELS AND ELEVATED TEMPERATURES

By

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Summary

This report covers fatigue tests made on representative laminates of CTL-91LD resin reinforced with 181-A1100 glass fabric. Fatigue tests with axial loads applied parallel to the warp direction and at zero mean stress were made at the rate of about five cycles per minute. Specimens were tested at room temperature (73° F.), 400°, 500°, 600°, and 800° F. All the tests were made on a constant-load type machine and loading was to high stress levels. Starting with the average value found in the static control test, the loads were progressively lowered until a specimen would maintain the established load for about 200 cycles without failure. The specimens, with few exceptions, failed on that portion of the cycle, either in tension or compression, that had the lowest average value in the static control test.

Fatigue tests at room temperature showed that at 80 percent of the tension-control strength, specimens would sustain about 200 cycles of repeated loading. At the elevated temperatures, however, there were no clearly indicated trends between 1 and 200 cycles because of strength-time-temperature variations.

1 This report is one of a series (ANC-17, Item 61-1) prepared and distributed by the Forest Products Laboratory under U.S. Air Force Contract Nos. DO 33(616)58-1 and 33(616)61-06 and U.S. Bureau of Naval Weapons Order No. 19-61-8041-WEPS. Results reported here are preliminary and may be revised as additional data become available.

2 Maintained at Madison, Wis., in cooperation with the University of Wisconsin.
Introduction

Fatigue properties of a few plastic laminates under repeated loading have been reported in previous work. This previous work has been done at low to moderate stress levels and from about 1,000 to 10,000,000 repetitions of load, and fatigue tests were performed at both room temperature and elevated temperatures.

In modern flight vehicles that are intended for only limited use, long life is not necessarily critical in the design. The design requirements therefore are for structures that will maintain their integrity for a very limited time under extremes of environment. In describing environmental conditions, surface temperature, acceleration forces, and temperature differentials are a few of the conditions to be considered.

The work reported in this project covers fatigue tests of a typical heat-resistant phenolic-reinforced plastic where a specimen is subjected to from 1 to 200 cycles of reverse loading at a uniform temperature. Each cycle of loading consisted of equal tension and compression loads, and stresses were applied parallel to the warp direction. The evaluation of the CTL-91LD material was conducted at the Forest Products Laboratory from February to July 1961 at the request of and in cooperation with the ANC-17 Panel on Plastics for Flight Vehicles. Initial work relating to development of test procedures was done before this period and is described in Appendix A.

Material

Reinforced plastic laminates of heat-resistant phenolic resin and 181 glass fabric were purchased for this test program. A phenolic resin (CTL-91LD) was selected as being a typical heat-resistant resin in use at the present time. The glass fabric selected was 181 with A1100 finish, a typical reinforcing material.

Panels were fabricated at the Cincinnati Testing Laboratories from prepregs containing 36.1 percent resin and 5.2 percent volatile. The prepregs were laid up with their warp fibers parallel and with 30 plies per panel. The assembly was laid up between stainless-steel cauls, using 600 PT cellophane as the parting film, and placed in a hydraulic press having a platen temperature of about 260° F. Press pressure was raised to 200 pounds per square inch, and the temperature of the press was regulated between 260° and 270° F. for 1-1/2 hours. The hot panels were then removed from the press and were postcured for 48 hours at each of the following temperatures: 250°, 300°, and 350° F.
Test Procedure

Four panels, about 1/4-inch thick and 36- by 36-inches square, were purchased for use in this project. Upon receiving these panels, a 5-inch grid was laid out on each panel. Barcol hardness readings and thickness measurements were taken over the grid pattern, and the average values were calculated. The panels were also weighed and measured for length and width. Using the measured volume and weight, the specific gravity of each panel was calculated. Two of the panels that were well matched as to Barcol hardness, thickness, and specific gravity were then selected for the work described in this report. Physical properties of the two panels used are presented in table 1.

Tests to determine the basic tensile and compressive strength qualities and the resin content of the panels were made in accordance with the test methods of Federal Specification L-P-406b. Tension and compression tests were made on a mechanically operated, universal-type testing machine. Resin content was determined by measuring the loss in weight of the panel specimens after heating them in an electric oven. The mechanical properties of each panel are also shown in table 1.

Control and fatigue specimens, 6 inches long and 1-1/2 inches wide with the center necked down to 1/2 inch, were prepared from the two opposite quarters of each panel used. Specimen blanks were cut on a bench saw using a carborundum wheel. The blanks were then rough shaped on a handsaw and the reduced section having a 4-inch radius was accurately machined using a carborundum wheel mounted in a vertical shaper. Each specimen from each quarter was numbered consecutively to identify the panel, the quarter, and the number. A cutting diagram is shown in figure 1.

Specimens used for control tests were selected at random alternately between each panel. The same method was used in selecting specimens for the fatigue test at each temperature.

Specimens were stored for at least 2 weeks prior to testing in a room where the temperature and humidity were regulated at 73° F. and 50 percent, respectively.

Control and fatigue tests at each test temperature were made on an Instron universal testing machine. Antibacklash springs were added to the movable head of this machine. It was then possible to mount the specimen between the load cell in the upper fixed head and the movable head and to apply both tensile and compressive loads. A 10,000-pound-range load cell that operates both in tension and compression was used. This machine is equipped with two load-limiting cams, which will reverse the direction of loading at any preset value of tension and compression load. In this study, equal loads in tension and compression (for a zero mean stress) were used. A variable speed content was
used to regulate the head speed at a rate that produced about five complete
reversals of stress each minute. The rate of head travel in inches per minute
was noted for each test and is presented in table 2. The grips used to hold the
test specimen are shown in figure 2. These grips were rigidly attached to the
load cell and to the movable head. They were mounted on threaded studs, and
it was possible to minimize eccentric loading by rotating the grips.

In the exploratory work at elevated temperatures, it was found that the load.
cell was sensitive to temperature when heat was applied directly to the load-
sensing element. A water jacket consisting of 1/4-inch-diameter copper tubing
was then made by coiling the tubing around the upper grip and circulating
cold water through it. The amount of cooling was regulated to within 5 degrees
of room temperature by throttling the water flow and periodically checking the
temperature of the load cell with a potentiometer and thermocouple taped to
the cell.

Specimens were heated in the area between the grips with two contact heaters
of nichrome wire connected in parallel, one on each side. Figure 3 shows the
heaters in place around a specimen. The heating elements were made with
nichrome ribbon wire wound around a mica sheet and were about 1-3/4 inches
by 2 inches in size. One side of the heater was backed with 1/4-inch asbestos
board while the side next to the specimen was covered with a thin copper sheet
insulated from the nichrome wire by a mica sheet. The heaters were attached
at one end to an asbestos spacer to form an open "U" shape that slipped onto
the specimen. The heat output could be varied with a powerstat and transformer
unit. The powerstat was connected to a 220-volt line and, in turn, to the
transformer, which was wired so that it delivered 16 volts and 22 amperes when
the powerstat was set at 100 percent of capacity. The nichrome ribbon coil
delivered uniform heat output over its length, which meant that the specimen
surface would be uniformly heated throughout the test area. The flow of
current to the heater was controlled by a thermocouple placed between the
surface of the specimen and the heater and connected to an electronic recording
controller and relay system.

Operation of the electric heater was checked with a thermocouple inserted into
a 1/16-inch-diameter hole drilled midway between the two heated surfaces of
a dummy specimen. In operation, it was found that the internal temperature
stabilized at the surface temperature of the specimen within 2 minutes after
the surface reached the test temperature. In making the control and fatigue
tests at elevated temperatures, the time the surface reached the test tempera-
ture was noted; the specimen was conditioned at this temperature for 4 minutes
before starting the test.
Static control tests of fatigue-shaped specimens were made in both compression and tension at each test temperature. These tests were made with the movable head traveling at 0.05 inch per minute, and maximum load only was recorded.

Fatigue tests were made at a zero mean stress at the rate of about 5 cycles per minute. These tests were timed with a stopwatch and the time checked against the chart recorder, which recorded each cycle against time. The chart was moving at the rate of 1/2 inch per minute. To regulate the rate of loading to about 5 cycles per minute, variable head speeds were used and are reported in table 2.

Discussion of Results

The physical properties and results of the quality tests on the two panels used in this study are shown in table 1 along with the strength requirements in tension and compression of specification MIL-R-9299. Both panels exceeded the strength requirements of MIL-R-9299. These panels were also well matched in resin content, specific gravity, average thickness, and Barcol hardness.

The testing machine used for fatigue test had a maximum range of 5,000 pounds in tension and compression. Because of the high strength of the material, this capacity was not large enough to test all groups of specimens. Hence, the net section of the control and fatigue specimens to be tested at room temperature (73° F.) and 400° F. was reduced in width from 1/2 to 3/8 inch. The additional reduction was made using the same 4-inch radius that had been used with the regular specimens.

Six specimens were loaded to failure in tension and 6 in compression at each test temperature. The results of these tests were used as control values for the fatigue tests. At room temperature, the tensile strength was lower than the compressive strength; at elevated temperatures, the opposite was true. Since fatigue tests were to be made at zero mean stress, the lowest value in tensile or compressive strength determined the starting stress level for the fatigue test. The results of the control tests and fatigue tests are presented in table 2.

The failure of the specimen during the fatigue test was observed, and a chart record for each specimen was recorded. It was therefore possible to tell whether failure occurred during the tension or compression side of the fatigue cycle. In both these tests and the exploratory tests (Appendix A), the specimens, with few exceptions, failed on that portion of the load cycle that had the lowest strength in the control tests.
The results of the fatigue tests are plotted in figure 4, and S-N curves were drawn through the empirical data. Stress is expressed as the percentage of the room temperature tensile control strength. The S-N curve for fatigue tests at room temperature (73° F.) shows the usual increase in fatigue cycles with decrease in applied stress, and the fatigue strength at 200 cycles is reduced about 20 percent below the control strength.

At elevated temperatures, appreciable changes in fatigue strength were not observed, and the S-N curves are practically horizontal lines. The change from 2 to 200 cycles, expressed as percentage of room temperature tensile strength, varied from 0 to 3 percent. The percentage change at any elevated temperature would, of course, be larger if fatigue strength was expressed in terms of the compressive control strength at that temperature. Calculations made on this basis show that the fatigue strength at 200 cycles was no more than 10 percent below the control value.

The fact that fatigue strength drops rather rapidly at room temperature and is not greatly changed at elevated temperature is reasonable for this phenolic material. Data in figure 6 of WADD Technical Report 60-804, show that compressive strength decreases rapidly with the initial application of heat and for some temperatures is a minimum value after about 2 or 3 minutes' exposure. The strength then increases with increased time and temperature until the strength again decreases. This second decrease in strength is associated with permanent degradation of the laminate, and the time of exposure at which it occurs varies with the test temperature. In this fatigue evaluation, each specimen was held at a uniform test temperature at least 2 minutes before loading. If the specimen withstood 200 cycles of repeated loading, about 40 additional minutes of heating were involved. It can be seen from figure 6 of the referenced report that the compressive strength at 400°, 500°, and 600° F. increased markedly after 2 to 6 minutes at temperature and continued to increase beyond 40 minutes of heating; hence, the increase in strength due to time at elevated temperature generally offsets the effects of repeated loading, and a relatively flat S-N curve is to be expected.

Time temperature data at 800° F. are limited, and the effects noted in the preceding paragraph are not as pronounced. It appears, however, that the trend of the fatigue data is about the same up to about 150 cycles of loading, and thereafter the fatigue strength is reduced. This reduction in fatigue strength after 100 to 150 cycles is expected in view of the degradation of laminates noted in the earlier work after continued exposure to temperature.

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Previously fatigue data had been obtained at the Laboratory on reinforced plastics with a fatigue machine operating at 900 cycles per minute. With this machine, load was applied at constant cycles but at variable loading rates because the load-time relationships are essentially a sine curve. In the current study, the load was applied with a universal testing machine at a constant rate of head travel for each specimen, and the load-time relationships are a series of linear relationships between the maximum applied stresses. Rate of head travel was controlled in an attempt to provide 5 cycles of loading per minute, and this was achieved reasonably well as noted in table 2. It is evident, however, that both the rate of applying loads and the load-time relationships were different in the previous and current studies. There is no positive way of judging the effect of these two variables on fatigue strength, but limited data from room temperature tests, as discussed in Appendix A, indicate that the effects may not be of major significance within the limits evaluated.

The results of these tests show the strength-time-temperature variations complicate any analysis of fatigue properties. As a result, fatigue data should be obtained after conditions representative of those that will occur in service. In the current evaluation, load was applied about 2 minutes after the test temperature had been reached, and therefore the loading was done approximately near the estimated minimum initial strength at that temperature. Perhaps lower fatigue strengths would have been obtained if fatigue loading was started at the same time as heating, or as soon as the surface reached the test temperature. Under these conditions, a thermal gradient would exist during the first part of the test. Another factor that may affect the fatigue strength is the rate of heating.

In this study, the rates of loading in cycles per minute were kept as near constant as possible. In future tests, however, it may be advantageous to maintain a constant head speed and let the cycles per minute vary.

This study did not investigate the effect of stress risers, such as a small hole at the net section of the specimen. Previous fatigue work at higher rates of loading has shown only a small reduction in the fatigue properties with a stress riser. The effect at high stress levels, however, may be greater.

Conclusions

Fatigue tests were conducted on a heat-resistant phenolic laminate made of CTL-91LD resin and 181-A1100 glass fabric. Load was applied to high stress levels, parallel to the warp direction and at zero mean stress. Specimens were unnotched and were tested at various temperatures. Based on this evaluation, the following observations were made.
(1) Failure under repeated loading at high stress levels occurred on that portion of the load cycle, tension or compression that had the least strength in the static control test.

(2) At 73° F., the fatigue strength at 200 cycles was about 80 percent of the control strength.

(3) At elevated temperatures, the fatigue strength up to about 150 cycles was essentially constant. This effect reflects the complex strength-time-temperature relationships. Permanent degradation effects due to time of heating were noted at 800° F. after about 150 cycles of loading.

Exploratory tests at about 5 cycles per minute and at high stress levels were also made on another phenolic laminate. This material had previously been evaluated between about 1,000 to 10,000,000 cycles at 900 repetitions of load per minute. The results of the exploratory tests indicate that the data at 73° F. would be reasonably well represented by an extension of the S-N curve from the previous work.

APPENDIX A

Exploratory Tests

A series of exploratory tests were made prior to the evaluation described in the report for the purpose of setting up suitable test techniques. The Instron machine was modified to permit the cyclic loading, special grips were made to hold the specimens, a heating unit was made that would enclose the net section of the specimen in the Instron machine during loading, a cooling device was improvised to cool the head near the load cell, and ventilation ducts were provided to remove volatiles during high-temperature tests.

Included in the exploratory work were a few fatigue tests of a phenolic laminate made with BV 17085 phenolic resin and 181 Volan A glass fabric. Specimens were cut from remnants of panels that had been evaluated in fatigue several years ago. A description of the materials and the test data are presented in WADC Technical Report 55-389. Some difficulties were experienced in the initial exploratory work, but techniques were refined so as to be considered acceptable. One of the problems, for example, was to provide for uniform loading during test so as to avoid eccentricities of stress.

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The results of these exploratory tests are of interest and are plotted in figures 5 and 6. Data from an initial series made at room temperature are plotted as open circles in figure 5. The scatter of points at the high stress levels is partly due to lack of uniformity of loading. It is evident, however, that the data would be reasonably well represented by an extension of the S-N curve of the earlier report. A single straight line would represent reasonably well the fatigue characteristics at 300° F. At 500° F., however, the exploratory data fall in essentially a horizontal pattern and well below the projection of the curve from earlier data. Although the characteristics of strength versus time at temperature had not been established for the BV 17085 laminate, it appears that the reaction is similar to that described for CTL-91LD laminates. In the earlier work, BV 17085 specimens were heated in an oven for about an hour before testing, and it is likely that this resulted in increased strength. In the exploratory tests, BV 17085 specimens were tested about 2 minutes after reaching temperature and therefore were near the minimum strength after initial heating.

The exploratory work emphasizes that in addition to temperature, the rate of heating and the time at temperature are important factors in the fatigue resistance of these laminates.
Table 1.--Properties of reinforced plastic panels of CTL-91LD resin and 181-Al100 glass fabric

<table>
<thead>
<tr>
<th>Panel No.</th>
<th>Average thickness 1</th>
<th>Specific gravity</th>
<th>Resin content</th>
<th>Barcol hardness</th>
<th>Tension properties 2</th>
<th>Compressive strength 2</th>
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<td>26.8</td>
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<td>2.90</td>
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</table>

ROOM TEMPERATURE VALUES

MIL-R-9299 REQUIREMENTS

|---|---|---|---|---|---|---|---|---|---|---|---|

1Thicknesses varied between 0.230 and 0.247 inch for Panel 712 and between 0.230 and 0.246 inch for Panel 715.

2Average value for four specimens.
Table 2.—Results of control end fatigue tests on laminates of CTL-91 LE phenolic resin reinforced with 181-A1100 glass fabric.

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Tension:</th>
<th>Compression:</th>
<th>Cycles:</th>
<th>Percent:</th>
<th>Head:</th>
<th>Failure:</th>
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<td>Tension:</td>
<td>Compression:</td>
<td>Cycles:</td>
<td>Percent:</td>
<td>Head:</td>
<td>Failure:</td>
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<td>56,700</td>
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<td></td>
<td></td>
<td></td>
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<td>29</td>
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1Plus sign indicates specimen did not fail.
2If final failure was in tension, the percentage was calculated using the applied tension stress; if in compression, on the applied compression stress.
3T designates initial failure occurred while load cycle was in tension, C designates failure while load cycle was in compression, and NF indicates specimen did not fail.
4Room temperature conditions are 73° F. and 50 percent relative humidity.
5Specimens were at uniform temperature for at least 2 minutes before starting test.
6Failed during compression loading of first cycle. Data not plotted.

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Figure 1. -- Cutting diagram for 36-inch square laminated panels used in fatigue evaluation.

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Figure 2. --Position of specimen in grips for room temperature tests. Electric heaters are shown to the left of specimen.
Figure 3. --Electric heaters in place around specimen. Water jacket is shown in position on upper grip and ventilation duct is at the right.
Figure 4. -- Fatigue data from tests of unnotched specimens of CTL-91LD phenolic resin reinforced with 181-A1100 glass fabric. Tests made parallel to the warp direction, at zero mean stress, and at about five cycles per minute.
Figure 5.--Fatigue data from tests of unnotched specimens of BV 17085 heat-resistant phenolic resin reinforced with 181-Volan A glass fabric. Tests made parallel to the warp direction at zero mean stress and 73°F. Exploratory work performed at five or six cycles of load per minute; others at 900 per minute.
Figure 6.—Fatigue data from tests of unnotched specimens of BV 17035 heat-resistant phenolic resin reinforced with 181-Vylon A glass fabric. Tests made parallel to the warp direction at zero mean stress at 300°, 500°, and 600° F. Exploratory work performed at five or six cycles of load per minute; others at 900 per minute.
The following are obtainable free on request from the Director, Forest Products Laboratory, Madison 5, Wisconsin:

<table>
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<tr>
<th>List of publications on Box and Crate Construction and Packaging Data</th>
<th>List of publications on Fire Protection</th>
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<td>List of publications on Chemistry of Wood and Derived Products</td>
<td>List of publications on Logging, Milling, and Utilization of Timber Products</td>
</tr>
<tr>
<td>List of publications on Fungus Defects in Forest Products and Decay in Trees</td>
<td>List of publications on Pulp and Paper</td>
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<tr>
<td>List of publications on Glue, Glued Products and Veneer</td>
<td>List of publications on Seasoning of Wood</td>
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<tr>
<td>List of publications on Growth, Structure, and Identification of Wood</td>
<td>List of publications on Structural Sandwich, Plastic Laminates, and Wood-Base Aircraft Components</td>
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<tr>
<td>List of publications on Mechanical Properties and Structural Uses of Wood and Wood Products</td>
<td>List of publications on Wood Finishing</td>
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<tr>
<td>Partial list of publications for Architects, Builders, Engineers, and Retail Lumbermen</td>
<td>List of publications on Wood Preservation</td>
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<tr>
<td>Partial list of publications for Furniture Manufacturers, Woodworkers and Teachers of Woodshop Practice</td>
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Note: Since Forest Products Laboratory publications are so varied in subject no single list is issued. Instead a list is made up for each Laboratory division. Twice a year, December 31 and June 30, a list is made up showing new reports for the previous six months. This is the only item sent regularly to the Laboratory's mailing list. Anyone who has asked for and received the proper subject lists and who has had his name placed on the mailing list can keep up to date on Forest Products Laboratory publications. Each subject list carries descriptions of all other subject lists.