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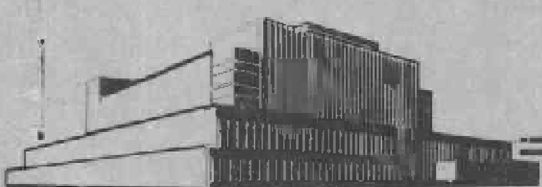

FOREST PRODUCTS LABORATORY
MADISON 5, WISCONSIN

UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE
In Cooperation with the University of Wisconsin

EFFECT OF INFRARED HEAT ON 45° TENSILE STRENGTH OF TWO REINFORCED PHENOLIC LAMINATES[1]

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Summary

A cooperative investigation was made to determine the effect of air-transmitted heat energy versus radiated heat energy on the strength of reinforced plastic laminates. The study was conducted on tensile coupons from phenolic laminates made with either glass fabric or asbestos fibers and machined at 45° to the natural axes. The strength in this direction is related largely to the shear strength of the material along the natural axes. Separate series of coupons were subjected to elevated temperatures in circulating air, to hot air and radiation, and to radiation alone. Circulating air was provided by ovens, and pure radiation was provided by tubular infrared lamps. Rates of heating on the surface were held constant in one series of tests but were varied in another.

The results of the first series, in which the temperature of the coupons was increased on the surface at a constant rate before loading, show the characteristic drop in strength with increases in temperature from air heating and a slightly greater drop in strength with infrared heating. The results of the second series in which the coupons were partially loaded and then heated with infrared on only one side at various rates from 1° to 29° F. per second until failure occurred, showed that the hot-side temperature increased with increasing rate of temperature rise; furthermore the center temperature (average of hot and cold side) was relatively unaffected by rate of temperature rise. It therefore appears that thermal shock and subsequent thermal gradient did not produce small internal fractures in these phenolic coupons.

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[1]This report covers work done in cooperation with the Bureau of Naval Weapons, Department of the Navy, under Order Nos. NAer 01967 and 19-61-8019-WEPS. Trade names are included in the report at the request of BuWeps, and with the permission of the materials suppliers.

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

Introduction

Strength evaluations of reinforced plastic laminates at elevated temperatures are made by heating with several methods. Strength evaluations are usually made on sample coupons so that their values may be projected to the strength of a structure. The method of heating the coupons may differ from that of heating the structure. The methods of heating differ in respect to method of heat transmission to the material and within the material. The methods of principal concern in this investigation are energy transfer by air and by radiation to the surface of the material.

In order to correlate strength studies of structures heated by radiation with studies of coupons heated by other means, such as hot air or ovens with circulating air, this study was initiated to investigate the mechanical strength of laminated plastic materials subjected to heat by different methods. Three heating methods were used: (1) heating by air only, (2) heating by radiation only, and (3) heating by a combination of these.

It was not practicable to investigate the effect of all of these heat sources on all of the mechanical properties such as tensile, compressive, flexural, shear, and bearing strength. Data from other reports[3,4,5] on parallel laminates show that increases in temperature result in decreases in strength properties. Temperature had the least effect on the tensile strength at 0° to the warp direction (or 0° to the machine direction of the asbestos felt) and greatest effect on interlaminar shear. An average effect was observed on flexure at 0°, compression at 0°, and tension at 45°. The tensile strength at 45° to the natural axes of the laminates was chosen to represent the effects of various heating methods because (1) it was likely to show an average effect of temperature and (2) it could readily be adapted to heating by various methods.

The 45° tensile strength is related largely to the shear strength of the laminates along their natural axes.[6] The natural axes are parallel and perpendicular to the warp direction for the glass-fabric laminates and to the machine direction of felt for the asbestos laminates.

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[3]Boller, K. H. and Kimball, K. E. Strength Properties of Reinforced Plastic Laminates at Elevated Temperatures (DC 2106 Resin and 181 Heat Cleaned Glass Fabric). Wright Air Development Center Technical Report 59-229. 1959.

[4]Boller, K. H. Strength Properties of Reinforced Plastic Laminates at Elevated Temperatures (CTL-91LD Phenolic Resin and 181-A1100 Glass Fabric). Wright Air Development Center Technical Report 59-569. 1959.

[5]Boller, K. H. Strength Properties of Reinforced Plastic Laminates at Elevated Temperatures (Phenolic-Asbestos, R/M Pyrotex Felt Style 41-RPD). Wright Air Development Division Technical Report 60-177, Part 1. 1960.

[6]Freas, Alan D. and Werren, Fred. Directional Properties of Glass-Fabric-Base Plastic Laminate Panels of Sizes That Do Not Buckle. Forest Products Laboratory Report No. 1803-B. November 1955.

Test Material

The tension specimens were 1/8 inch thick, 3/4 inch wide, and 9-3/8 inches long, necked down to a 1/4-inch-wide net section.

Two phenolic laminates were used to evaluate the effects of various heat sources. One laminate was a glass-fabric-base material, furnished by Cincinnati Testing and Research Laboratories, Inc., and was fabricated by American Reinforced Sales. Data from the same lot of material are presented in another report.[4] The general fabrication and postcuring information was furnished by the supplier; average values of resin content, Barcol hardness, and specific gravity were obtained at the Forest Products Laboratory. Information on the material is as follows:

Resin--CTL-91LD
Catalyst--None
Fabric--181-A1100 glass fabric, parallel laminated back to back
Number of plies--14
Method of impregnation--"B" stage "prepreg" cloth used; material from standard production run which is impregnated on a Waldron-type treater, using dip pan, metered squeeze rolls, drying oven, cooling zone, and windup
Initial resin content--40 $\pm$ 2 percent including volatiles
Precure--None
Cauls and parting films--Steel cauls with a No. 8 mirror finish; 600 PT cellophane as releasing film
Curing cycle--200 pounds per square inch at 265° to 275° F. for 40 to 50 minutes
Removal from press--Cooled under pressure
Postcure--24 hours each at 250°, 300°, and 350° F.
Resin content--37.3 percent (from burnoff tests)
Barcol hardness--86
Specific gravity--1.79
Nominal size of panels--1/8 by 36 by 36 inches

This material, according to quality tests in compression and flexure, met the MIL-R-9299 requirements.

The other plastic material was a phenolic-asbestos laminate, fabricated and supplied by Raybestos-Manhattan, Inc. Results of other tests on the same lot of material have been reported.[5] Information furnished by the supplier regarding the laminates is:

Resin--R/M Style 984-RPD
Felt--R/M Pyrotex Felt Style 40-RPD (unimpregnated sheets of asbestos fibers having a slight orientation parallel to machine direction)
Prepreg felt--R/M Pyrotex Style 41-RPD
Method of impregnation--Prepreg made by passing Pyrotex Felt through resin bath and heating chamber

Initial resin impregnation--25 to 30 percent by weight
Number of plies in laminate--44
Orientation of plies in laminate--All parallel
Pressure in press--400 pounds per square inch
Temperature and time in press--300° F. for 30 minutes
Removal from press--Hot
Postcure--24 hours at 300° F., 24 hours at 350° F., 8 hours at 400° F.

Data obtained at the Forest Products Laboratory include:

Volatile content from burnoff tests--34 percent (some of this is possibly due to loss of water of crystallization of the asbestos.)
Barcol hardness--72
Specific gravity--1.88
Nominal size of panels--1/8 by 24 by 78 inches

Tension specimens were machined from these two types of laminates so that 12 replications could be tested at room temperature, at three elevated temperatures, and with three kinds of heating. The elevated temperatures were 300°, 500°, and 650° F.

Heating Methods

The three methods of heating used in this work are:

(1) Air Heating.--Air heating was accomplished by forcing hot air from an oven to the specimen through insulated tubing (figs. 1 and 2). The heat source was a 12- by 12- by 14-inch, 5-kilowatt electric oven. A fan in the oven forced air past the heating elements and discharged the hot air through a 3-inch-diameter hole in the top of the oven. The air was carried by a 3-inch-diameter flexible metal tube to a 4-inch-diameter chamber around the specimen (fig. 2). Inside this chamber a baffle caused the air to sweep around the flat sides of the specimen, thus equalizing the temperature around the specimen and along the 2-1/4-inch-long net section. The air passes through the chamber and returned to the base of the oven for reheating. Because of heat losses in the tube, temperatures in the oven were maintained considerably higher than those at the specimen. For example, an oven temperature of about 1,200° F. was needed to maintain a temperature of 650° F. at the specimen.

(2) Radiant Heat.--This heating method was accomplished by using four clear quartz-infrared lamps (9 inches long, 500 watts, 115 volts, type T3) on each side of the specimen. The lamps were placed 2-3/4 inches from the specimen and the rays were reflected by gold-plated reflectors (fig. 3). The center 5 inches of each lamp radiated energy at high intensity which peaked at about 10,000 angstroms according to General Electric Circular LS-128. The ends of the lamps were air cooled, causing a slight circulation of air in the reflector.

(3) Air-Disk Heating.--This heating method is a combination of air circulation and low-intensity radiant heat. The air circulation results from natural circulation in the chamber (fig. 4) and the radiant heat from electric heating elements shielded in the steel disks. The temperature of the radiant heat source was only a few degrees above the test temperature of the specimen. According to General Electric Circular LS-128, the wave length of the peak heat energy would be at about 50,000 angstroms. The heating chamber is about a 4-inch cube containing two 3-inch-diameter disks, each 400 watts. These 115-volt heaters were controlled with a variable voltage regulator and an a automatic controller to the test temperature.

The specimen temperature in each heating method was controlled by a 0.005-inch-diameter chrom-alumel thermocouple and automatic controller. The thermocouple was held on the surface of the specimen by a metal clip and a ceramic button 5/32 inch in diameter and 1/16 inch or less thick. This button, it was found, constituted a convenient method of pressing the thermocouple to the surface of the specimen and shielded it from radiation and convection.

Heating was controlled in the first series of tests so that the time to heat the specimens to a uniform temperature through their thickness was approximately constant for the three methods of heating. The heating time was about 4 minutes for each, and the rate of heating varied from about 1° F. per second at 300° F. to about 2-1/2° F. per second at 650° F.

In order to explore the effect of rate of heating, the time to heat specimens in another series was varied from about 10 seconds to 20 minutes. The heat was applied to only one side by means of infrared lamps. The rates of temperature rise on the hot side varied between about 0.9° F. per second and 29° F. per second.

Loading Method

Uniform Temperature Distribution

Cool specimens were placed in Templin-type grips. The specimens were then heated to a controlled temperature and the load was applied in a universal-type test machine with a movable head motion of 0.1 inch per minute. The temperature was held constant during loading and therefore the period of soak at constant temperature was a variable, depending on the magnitude of of the load. However, the average of the soak period for 12 replications at constant temperature for the phenolic-glass laminates varied from 1.03 to 1.79 minutes, and that for phenolic-asbestos laminates was from 1.77 to 2.79 minutes.

Nonuniform Temperature Distribution

In this series cool specimens were first loaded to a predetermined level and then, while the load was maintained, they were heated until failure occurred. The rate of heating was varied in an attempt to provide a fast rate of temperature rise so that the thermal shock without a subsequent soak period might cause the development of blisters and internal fractures.

Specimens were supported in Templin-type grips and load was applied in a universal-type testing machine. Loads of 20, 40, 60, and 80 percent of the room temperature strength were applied and maintained with an automatic load maintainer throughout the test period. Heating of the specimen on one side with infrared lamps was started when the load was reached and was continued until failure occurred. Target rates of temperature rise were 1°, 5°, and 100° F. per second; however, the 100° F. rise was impossible to obtain with the present equipment and a maximum of only about 25° F. per second was obtained with full power available.

Temperatures were observed on the surface of the specimen at the net section by means of thermocouples.

Discussion of Results

Uniform Temperature Distribution

The results of the tests to obtain the failing stress for the three conditions of uniform heating are presented in table 1 and figure 5.

Table 1 presents the maximum tensile stress obtained at 45° to the warp direction at the three elevated temperatures after three methods of heating. Each value represents the average of 12 replications and the variation in individual test values is indicated by the accompanying standard deviation value. Stress and standard deviation values are shown as pounds per square inch and the stress values are also shown as percentages of the room temperature tensile stress.

A comparison of the standard deviation values on table 1 show that the spread of individual test values is relatively small. The average standard deviation value for the phenolic-glass laminate is only 888 pounds per square inch and for the phenolic-asbestos laminate is only 1,625 pounds per square inch. These values are 3.5 and 4.4 percent of their respective room temperature strength values.

Figure 5 presents a comparison of the tensile strength values for the two distinct types of heating, air versus radiant heating, for the two types of laminates tested. The plotted points represent the average values and the shaded band reflects the area that is included within one standard deviation from each average value.

The data show that the difference in strength values between air heat and infrared heat at 300° and 500° F. is probably not significant. However, at 650° F. the strength after infrared heating is significantly less than that after air heat. At 650° F., the strength retention in terms of percentage of room temperature stress is about 13 percent less after infrared heating than after air heating.

All of the average strength values obtained by some degree of radiant heat, either air disk or infrared, are less than those obtained by air heat. Even though some differences are significant and some are not, the overall trend indicates that the penetrating rays of radiant heat cause some detrimental effect on strength of reinforced plastic laminates. The degree of that detrimental effect is small at temperatures up to 500° F., but at a temperature of 650° F. the detrimental effect has become greater.

While this work points out the effect of various heat sources on tensile strength at 45° to the principal axes, this effect is also expected to apply to the edgewise shear strength of the laminate.

Nonuniform Temperature Distribution

In an effort to isolate the effect of heat penetration due to infrared heat rays, it was theorized that the fast rate of heat rise due to penetration plus conduction would cause small internal fractures and blisters, and hence would reduce the failing stress at a given temperature or reduce the failing temperature at a constant stress. Within the framework of this experiment, specimens were tested with the latter method at four constant load levels and three heating rates.

Results are presented in tables 2 and 3. These tables show the maximum temperatures at failure obtained by heating with infrared on one side. Each value for failing temperature and time to failure is the average of five specimens. The time to failure is the heating time at constant load, and thus is also a stress-rupture time at a variable temperature.

If internal failures occur at the fastest rate of temperature rise, the failing temperature due to the fastest rate would be less than that due to the slowest rate of temperature rise. Results of tests do not appear to substantiate this assumption. The data on tables 2 and 3 show that:

(1) With low energy input to infrared lamps the time to failure (col. 5) at a constant load is longer than that with a high energy input. Hence the rate of temperature rise on the hot side (col. 6) varies with the input energy. The rate of temperature rise on the cold side was, of course, slower than that on the hot side.

(2) The temperature gradient (col. 7) at a constant stress level generally increases with increasing rate of temperature change (col. 6).

(3) Even though there is usually a substantial increase in thermal gradient with increases in rate of heating the hot side (col. 6), the average temperature (col. 3) at failure seems to be relatively unaffected by the rate of temperature change.

(4) Actually the hot side temperature (col. 2) tends to increase with increasing rates of temperature change (col. 6). This is contrary to the original assumption that the failing temperatures should decrease due to an acceleration of internal failures with increases in rate of temperature change.

Additional information outside this experiment[7] supports the conclusion that rapid heating of 1/8-inch-thick flat laminates up to 1,000° F. in the range of rates used in these experiments does not induce sufficient thermal stresses to be the primary cause of strength deterioration. Temperature and duration of exposure to both temperature and stress are the predominating factors that affect strength deterioration. The effect of rate of heating on strength properties may, however, be quite different for full-size structural elements than for small coupons. In the coupons, internal failures and blisters might be less likely to occur because volatiles are free to escape along the edges of the coupons. Such escape of volatiles may not be possible in large structural parts.

Conclusions

The results of tension tests of two reinforced phenolic laminates machined at 45° to the natural axes indicate that infrared heating is slightly more detrimental than air heating. Even though rates of heating on the surface of these small tension specimens were the same for both methods, the effect of infrared rays in the interior apparently deteriorates the resin or its bond to the reinforcement to the extent that the overall tensile strength is diminished.

Strength tests of laminates with two different reinforcements, glass-fabric and asbestos felt, showed about the same degree of deterioration due to infrared heating so the transparency of the reinforcement apparently has little effect on the deterioration. The common denominator, the phenolic resin, would presumably be responsible for the change in strength.

Even though there are no direct data available to compare internal temperatures of these plastic laminates by various heating methods, it is believed that the interior temperature rises faster with infrared penetration than

[7] Boller, Kenneth H. Tensile and Compressive Strength of Reinforced Plastic Laminates After Rapid Heating. Wright Air Development Division Technical Report 60-804.

with conduction alone. Hence, the interior is at a higher temperature longer by infrared heating than by conductive heating. The resulting strength then depends on strength-time-temperature characteristics rather than on the type of heating.

When infrared heat was applied to only one side of a specimen under constant stress, results showed that rates of heating from 1° to 29° F. per second did not affect the average temperature when failure occurred. This indicates that the strength remains unaffected by rate of heating. This conclusion is limited to the rates, material, and size of specimens in this experiment. Other conditions, such as larger specimens or different shapes, could conceivably have radically different stress distributions that would cause rate of heating to differ from that in this experiment.

Table 1.--The 45° tensile strength of two phenolic laminates at elevated temperatures after heating by each of three methods.[1]

Temperature	Air heating			Air-disk heating			Infrared heating		
	Strength[2]	Percentage of room temperature strength	Standard deviation	Strength[2]	Percentage of room temperature strength	Standard deviation	Strength[2]	Percentage of room temperature strength	Standard deviation
°F.	1,000 p.s.i.	Percent	1,000 p.s.i.	1,000 p.s.i.	Percent	1,000 p.s.i.	1,000 p.s.i.	Percent	1,000 p.s.i.
Phenolic-glass (CTL-91LD and 181-A1100)									
75	25.44	100.0	0.95	....	....	....	....	....	....
300	21.25	83.5	.70	20.94	82.3	0.90	19.48	76.5	0.73
500	18.03	70.8	.72	17.62	69.2	.94	17.12	67.3	.96
650	15.31	60.1	1.14	14.36	56.5	.96	12.07	47.4	.88
Phenolic-asbestos (R/M Pyrotex Felt Style 41-RPD)									
75	37.27	100.0	2.22	....	....	....	....	....	....
300	31.76	85.2	1.37	29.78	79.9	1.52	29.92	80.4	1.58
500	30.13	80.9	1.45	28.94	77.7	1.53	29.13	78.2	1.07
650	29.25	78.5	1.64	26.07	70.0	2.17	24.03	64.5	1.70

[1]Rate of heating to each test temperature was approximately constant for the 3 methods of heating.

[2]Average of 12 replications.

Table 2.--Results of tests[1] of 45° tension specimens subjected to constant loads heated[2] at variable rates until failure occurred. Material was a phenolic-glass laminate (CTL-91LD and 181-A1100 glass fabric).

Constant average stress	Failing temperatures: Hot side	Failing temperatures: Average[3] at center	Failing temperatures: Cold side	Time to failure	Average rate of temperature change on hot side	Temperature gradient[4]
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1,000 p.s.i.	°F.	°F.	°F.	Sec.	°F. per sec.	°F. per in.
5.09	1,134	934	734	1,098	0.96	3,200
	1,162	930	699	226	4.80	3,704
	1,250	992	734	91	12.90	4,128
10.18	855	708	562	787	.99	2,344
	810	669	529	154	4.78	2,248
	929	718	508	54	16.03	3,368
15.27	562	466	371	472	1.03	1,528
	526	434	343	93	4.86	1,464
	592	435	279	23	23.47	2,504
20.36	318	278	238	224	1.08	640
	354	298	242	56	4.95	896
	373	281	189	12	24.25	1,472
25.44	75	75	75			

[1] Average of 5 specimens.

[2] Specimens were heated on one side with infrared lamps.

[3] Value is an average of the face temperatures.

[4] Based on nominal 1/8 inch thickness.

Table 3.--Results of tests[1] of 45° tension specimens subjected to constant loads heated[2] at variable rates until failure occurred. Material was a phenolic-asbestos laminate (R/M Pyrotex Style 41-RPD)

Constant average stress	Failing temperatures: Hot side	Failing temperatures: Average[3] at center	Failing temperatures: Cold side	Time to failure	Average rate of temperature change on hot side	Temperature gradient[4]
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1,000 p.s.i.	°F.	°F.	°F.	Sec.	°F. per sec.	°F. per in.
7.45	1,246	1,035	825	1,254	0.94	3,368
	1,212	993	775	244	4.66	3,496
	1,238	1,015	793	104	11.25	3,560
14.90	1,001	835	668	940	.99	2,664
	1,001	825	650	194	4.79	2,808
	1,105	870	636	71	14.48	3,752
22.35	756	640	524	672	1.01	1,856
	654	540	425	119	4.85	1,832
	741	550	358	36	18.81	3,064
29.80	195	172	150	105	1.17	360
	267	213	159	39	4.95	864
	349	244	139	10	28.99	1,680
37.27	75	75	75			

[1] Average of 5 specimens.

[2] Specimens were heated on one side with infrared lamps.

[3] Value is an average of the face temperatures.

[4] Based on nominal 1/8 inch thickness.

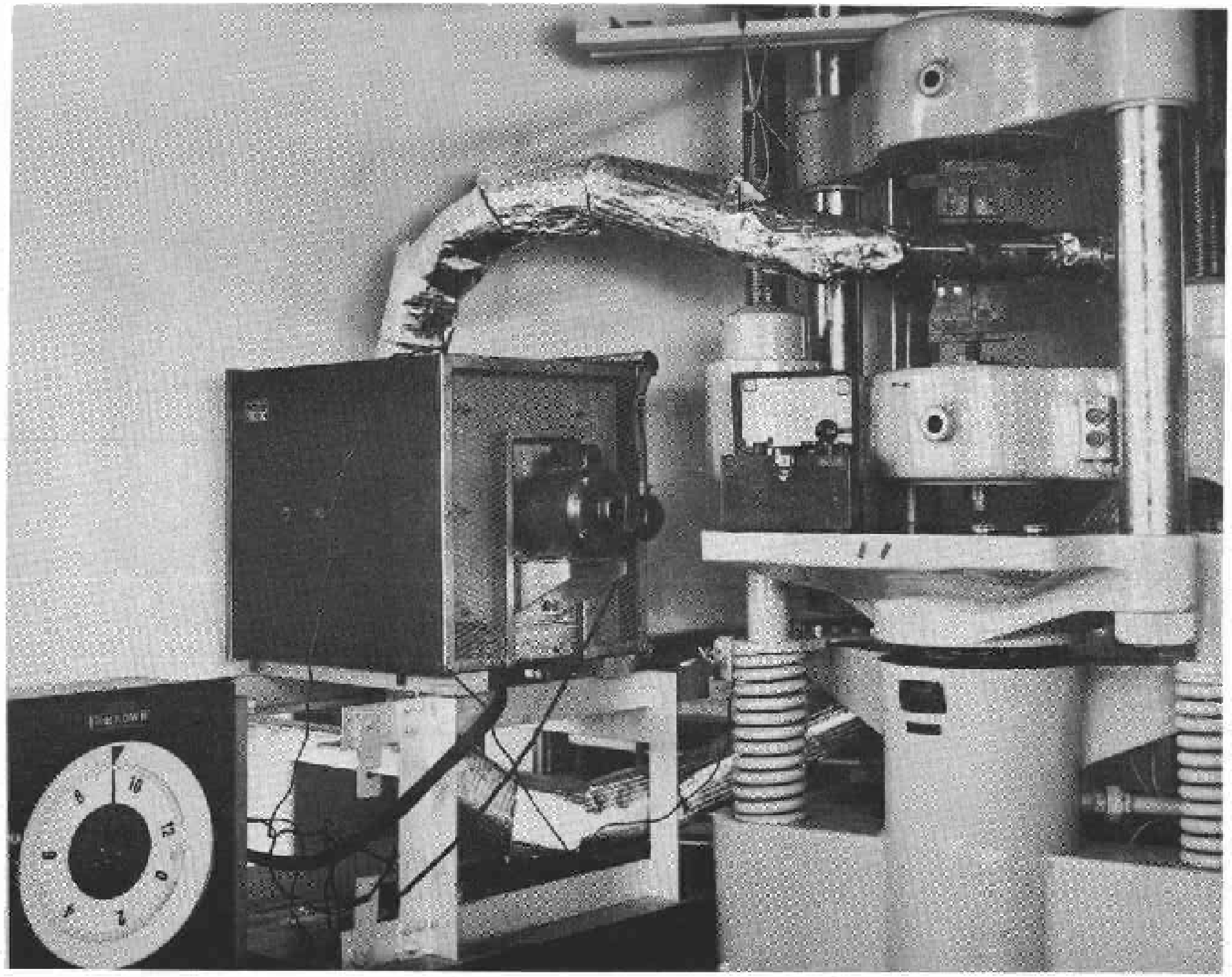

Figure 1.--Apparatus for hot air heating and testing reinforced plastic laminates. Air heated in the electric oven (left center) is conducted up through the flexible tube and around the specimen, held in the test grips.

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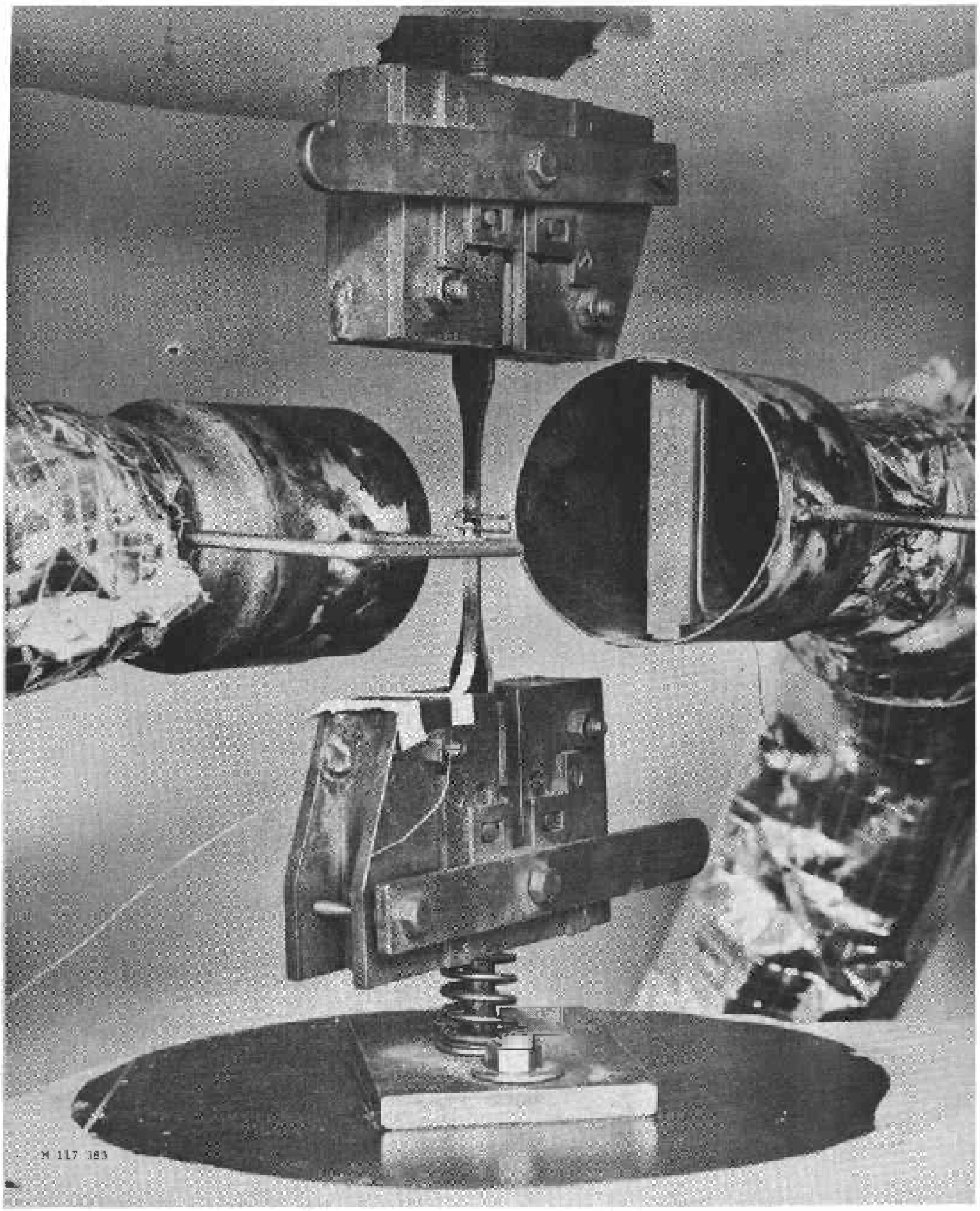

Figure 2. --Closeup showing hot air chamber and specimen in the grips for tension test. The vertical baffle in the chamber interrupts air flow and helps to equalize temperatures around the specimen.

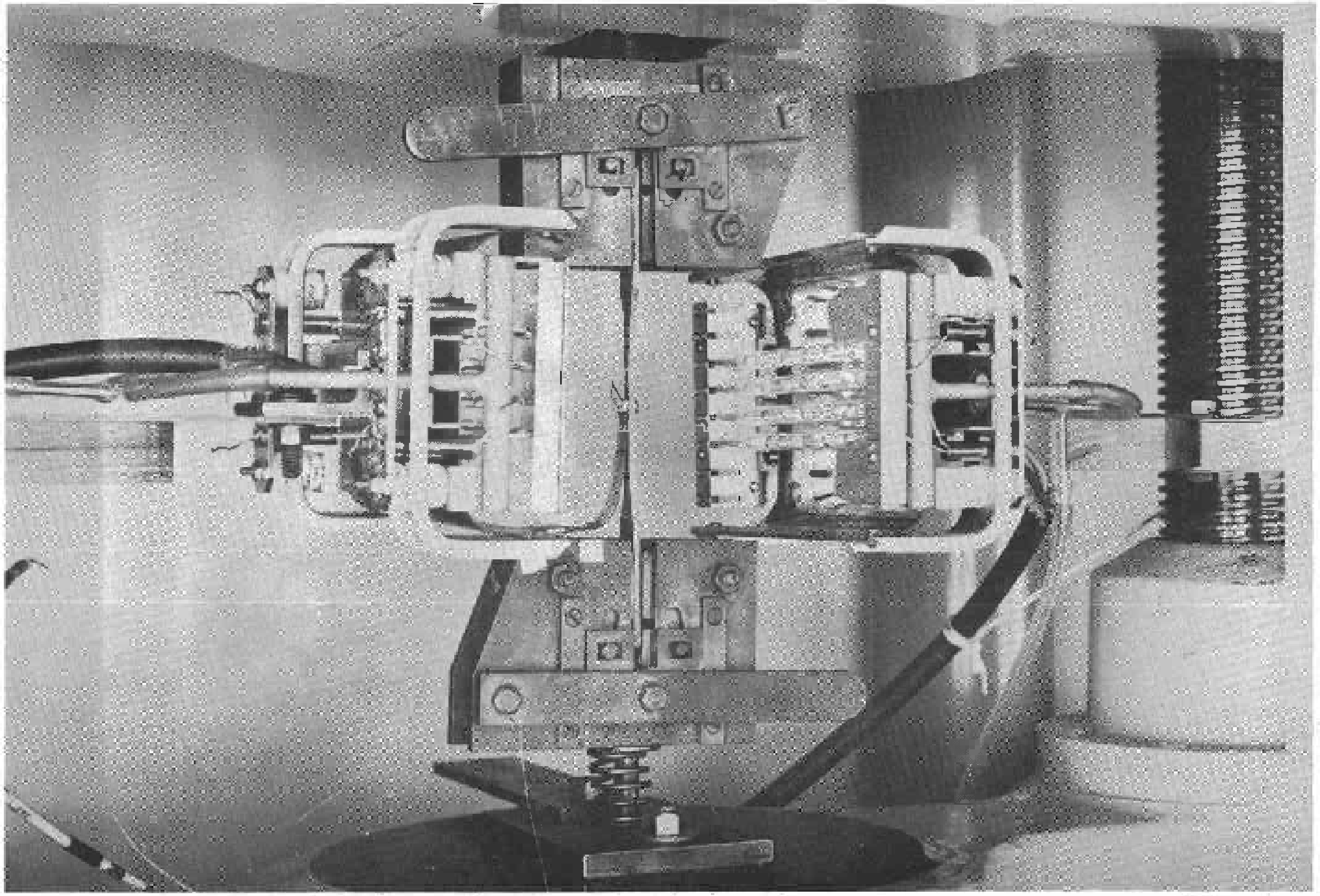

Figure 3. --Infrared heating units concentrated radiant energy on the tension specimen held in the test grips.

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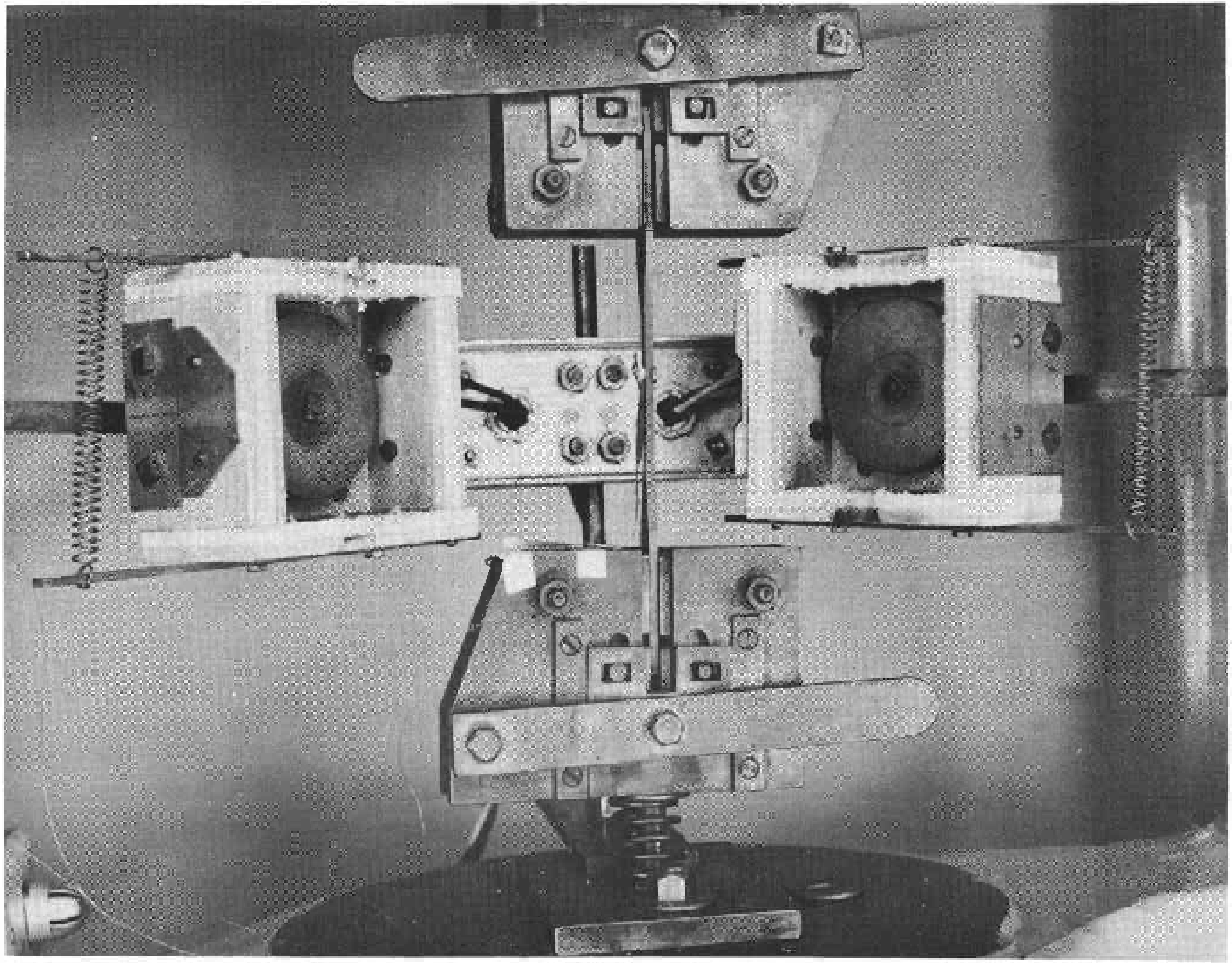

Figure 4.--Interior of air-radiant heat chamber with specimen in the grips, for tension tests of reinforced plastic laminates.

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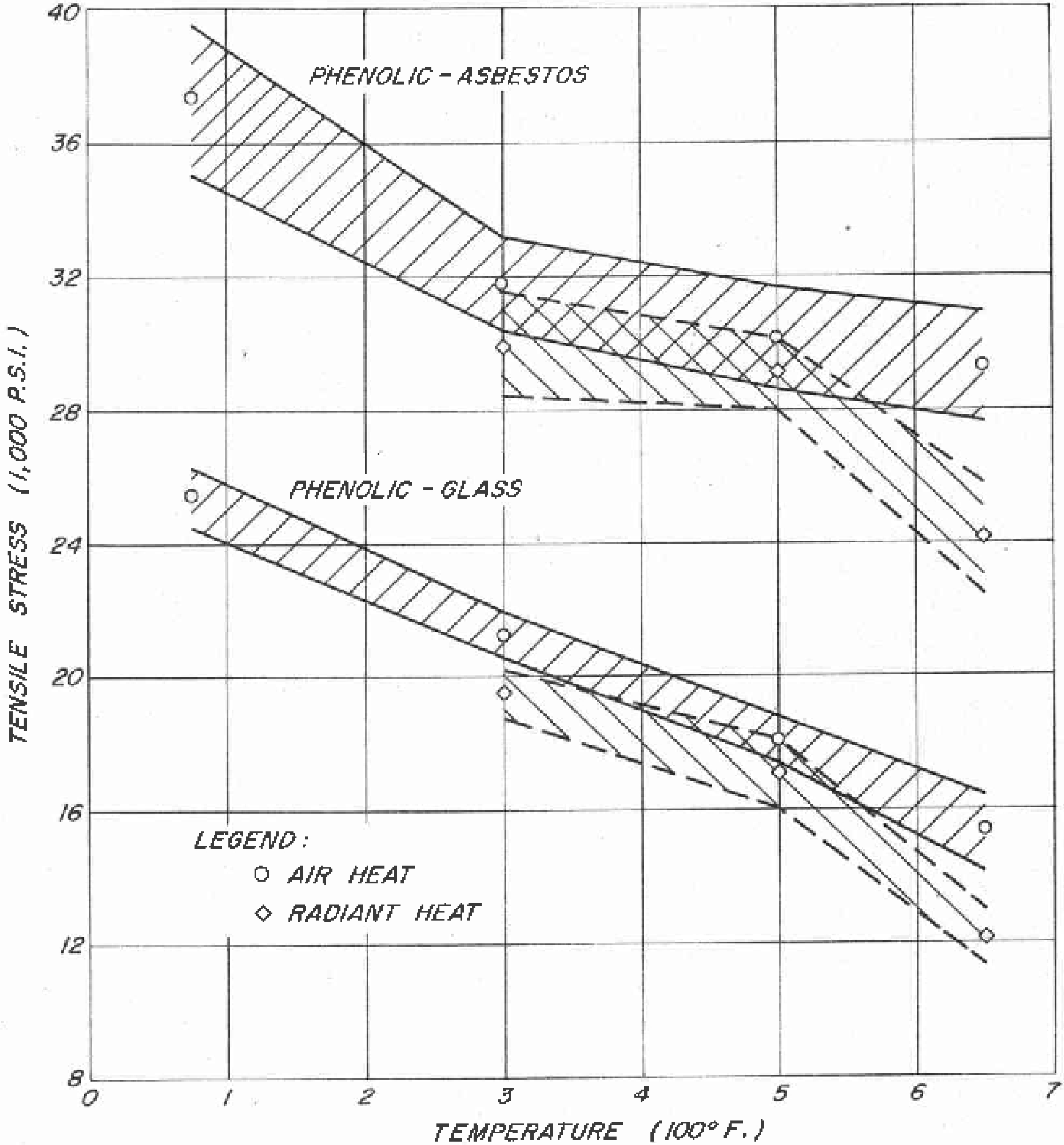

Figure 5.--Effect of various heat sources on the 45° tensile strength of two reinforced phenolic laminates. Plotted points are average tensile values, while the envelopes include the area that is within one standard deviation of each average value.

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