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TITANIUM REINFORCED BORON POLYIMIDE COMPOSITE

Final Report

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TITANIUM REINFORCED
BORON-POLYIMIDE COMPOSITE

Final Report

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North American Aviation
Columbus Division
North American Rockwell

FOREWORD

This final report covers the work performed on boron-polyimide during the period 27 June 1969 to 1 July 1971 on Contract NAS8-24511. This program was sponsored by the National Aeronautics and Space Administration, George C. Marshall Space Flight Center, Alabama with Mr. E. L. Brown the C.O.R. (Contracting Officer's Representative). The program was performed by the Structures R&D Group, Columbus Division, North American Aviation with Mr. J. E. Bartfield serving as Program Manager and Mr. G. A. Clark as Principal Investigator. Mr. K. I. Clayton was responsible for analysis and testing of the component and assisted in the preparation of the final report.

Publication of this report does not constitute NASA approval of the program results or conclusions. It is published only for the exchange and stimulation of ideas.

ABSTRACT

Processing techniques for boron polyimide prepreg were developed whereby satisfactory composites could be molded under vacuum bag pressure only. A post cure cycle was developed which resulted in no loss in room temperature mechanical properties of the composite at any time duration up to 16 hours at 650°F.

A design, utilizing laminated titanium foil, was developed to achieve a smooth transition of load from the titanium attach points into the boron reinforced body of the structure.

The box beam test article was subjected to combined bending and torsional loads while exposed at 650°F. Loads were applied incrementally until failure occurred at 83% design limit load.

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Section I

Introduction

1.0 Introduction

High modulus reinforced composites are seeing increased use because of their increased strength-to-weight and stiffness-to-weight ratios. The application of optimized, oriented, filamentary composite structures in aerospace applications has been limited by low stiffness, bearing characteristics, and in some instances temperature resistance. The low stiffness (composite modulus) has been overcome by the development of boron and high modulus graphitic filaments. The elevated temperature characteristics have been improved by such polymers as polyimides, polybenzimadazoles, polybenzothiazoles, etc. These resin systems have not, however, been completely characterized for their processing characteristics particularly as they apply to production type tooling. The bearing characteristics and concentrated load transfer through hard points still remain problem areas where minimum weight is required.

One good approach to reducing the weight of hard points is to incorporate lightweight metal shims to augment the strength of the composite. To minimize strains at the metal-composite interface, the component must have compatible linear coefficients of thermal expansion. A boron composite with any resin system is reasonably compatible with stainless steels but more compatible with titanium sheet materials. Graphite composites impose problems for incorporation of metal shims since they, in the longitudinal direction, have a slightly negative coefficient of expansion.

The intended use of any material imposes other considerations on how it will be processed. One application of interest which was to be considered in all the development effort,but not to be fabricated under this program,was a conical section thirty-two feet in diameter at the base, tapering to twenty-eight feet in approximately six feet. The size of such a section obviously precludes the press molding of the component in one piece. It also severely limits the facilities for autoclave or hydroclave molding. Thus the processing technique established for concentrated effort was conventional vacuum bag molding.

This program involved the development of the process technique for boron-polyimide prepreg, the lay-up and curing procedures for the prepregs when processed under vacuum bag pressure, the development and evaluation of titanium hard points for the smooth transition of loads from the titanium attach points into the boron reinforced body of the structure. This information, after data generation, was then applied to the modification of a box beam design which had previously been fabricated of boron-epoxy. After verification of the design, the component was fabricated and structurally tested at the maximum temperature capability of the system.

Section II

Summary

2.0 Summary

Polyimide resin prepregged onto scrim cloth may be tackified for application to boron by the use of wet steam. Also, for small quantities, low boiling solvents such as ketones are satisfactory for the purpose while the use of high boiling solvents such as n-methyl pyrrolidone (NMP) creates problems in subsequent processing.

N-methylpyrrolidone forms an azeotrope with water and excess solvent may be removed by controlled water immersion and subsequent staging at temperatures as low as 200F.

While the prepregged boron can be reprocessed, to remove the excess NMP it is not a procedure that is applicable to production operation. It appears that considerable development effort on the prepregging of boron-polyimide is required for the system to be considered for the fabrication of production parts using low pressure, vacuum bag techniques.

Composite specimens, using Pyralin 4707 resin, retain, at 650F,100 percent of their room temperature strength (within experimental error) if a post cure of 10 hours at 700°F is included in the cure cycle.

A titanium interleaved joint design has been developed which is readily fabricated, machined, and drilled. A laminated titanium foil, using polyimide adhesive, permits all drilling to be accomplished in the titanium without the complication of boron inclusions. Step lengths of 1/2 or 3/4 inch will transmit the load which can be carried by the individual boron-polyimide laminate plies.

When tested at 650°F, the box beam test article failed at 83% design limit load with failure initiating at one end of the spar to upper facing joint. Examination of the failed area showed an area where a questionable bond was obtained. Stress concentration of that point would cause a peeling action and the failure at less than the predicted strength.

Section III

Material and Process Development

3.1 Introduction

This phase of effort involved the selection of the two most heat resistant materials of those polyimides currently available, the development of the boron prepregging technique, the establishment of a reproducible lay-up and curing technique, and the fabrication of panels by different technicians to assure that the process is reproducible by more than one competent technician. An additional restraint imposed upon the fabrication process development was that it be a vacuum bag molding technique without augmented pressure.

3.2 Material Selection

Several polyimide resin systems were considered as potential binders for this laminating development. These were selected initially on the basis of their high temperature capabilities, general handling and curing characteristics.

Resins initially considered included Pyralin(a) 4707, RS(b) 6228, P13N(c) and Pyralin 35-520 prepreg. The first three systems are polyimide precursor solutions. The 4707 is supplied as a 44 per cent solids (polyimide cured 1 hr at 600°F) in N-methyl pyrrolidone/xylene solvent. Similarly, the RS 6228 is supplied in solution form. This is a newly developed, high-temperature resin, and technical literature was not available. The P13N is also a solution but its basic formulation is completely different from those previously discussed in that it polymerizes by an addition type reaction rather than a condensation type. The resulting product is one which cannot be bag molded and has considerably lower heat resistance than the condensation type polymers. On this basis, P13N was not given further consideration.

The Pyralin 35-520 polyimide prepreg has the following specifications:

Glass fabric	104 style E glass
Finish	A-1100 (soft)
Resin content (ignition)	78 ±4 percent
Volatiles (2-1/2 minutes at 750°F)	33 ±4 percent
Flow (5 minutes at 450°F; 15 psi)	33 ±4 percent

This fabric is the lightest weight glass cloth (0.6 oz/sq. yd.) commercially available as a resin carrier. It is a plain weave with a 60 x 52 construction and a nominal thickness of 1 mil. The tensile strength of this cloth is 40 x 15 (W&F) lb/in. This is not expected to contribute significantly to the properties of the finished laminate, but the nominal strength should contribute to the handling properties of the collimated boron prepreg.

(a) DuPont polyimide
(b) Monsanto
(c) TRW

3.3 Prepreg Technique and Process Development (Sub Contract)

A sub contract was issued to Battelle Memorial Institute to develop the boron-polyimide prepreg technique and the production process techniques, for that prepreg, using vacuum bag pressure only. These procedures were to be established using instrumental techniques. The information presented in this paragraph was developed by Battelle under sub-contract unless otherwise noted.

3.4 Thermal Analysis Studies

Several procedures were used in studying the handling behavior, chemistry, and processing characteristics of these polyimides. Considerable background on handling and processing was based on gravimetric data generated by exposures at controlled time and temperature. These data were supplemented by instrumental techniques including DTA, TGA, mass spectrometry and dissipation factor measurements.

DTA thermograms were obtained on these three remaining candidate materials and a TGA run made on the 35-520 prepreg. Single runs were made for each sample, and, hence, the results must be considered tentative.

The DTA thermogram (Figure 1) was made on Monsanto RS6228 resin at a heating rate of 5 C (9 F) per minute in a dynamic nitrogen environment. The sample exhibited an initial endotherm from room temperature to about 200 C (392 F), with many abrupt endospikes. This suggests skinning over during loss of solvent and volatiles. Bursts of trapped volatiles through the skin apparently cause the sudden spikes. Above 200 C only gradual exothermal activity was noted up to 450 C (896 F).

A similar heating rate and environment was used in the thermogram (Figure 2) for DuPont PI/4707. This thermogram indicates a considerable difference in drying behavior. The relatively smooth curve for the 4707 resin suggests a balance of solvents such that good diffusion control is obtained. This, of course, is inferred from the absence of "skinning" aberrations. Furthermore, the solvent system appears somewhat more volatile as evidenced by the endotherm peak occurring at a lower temperature. The Pyralin 35-520 prepreg was reported to use the Pyralin 4707 resin system. The similarity in DTA thermograms appears to confirm this. The thermogram on the prepreg, however, reflects the absence of solvents removed during drying and prepregging resulting in the smaller endotherm.

The DTA thermogram (Figure 3) obtained on DuPont 35-520 prepreg, under the same conditions, showed an initial endotherm from room temperature to about 140 C (284 F). At 140 C there was a sudden endotherm shift, possibly due to sample movement. From 140 C to 450 C (842 F) only gradual exothermal activity was noted.

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SAMPLE Monsanto RS 6228	WEIGHT 3.3mg.	REF. MATL.	WT.	ATMOSPHERE N_2	PRESSURE
PROGRAM RATE 5 °C/MIN	ΔT GAIN 80 μv 500 °C/F.S.	SAMPLE HOLDER		GAS FLOW .075 SCFH	

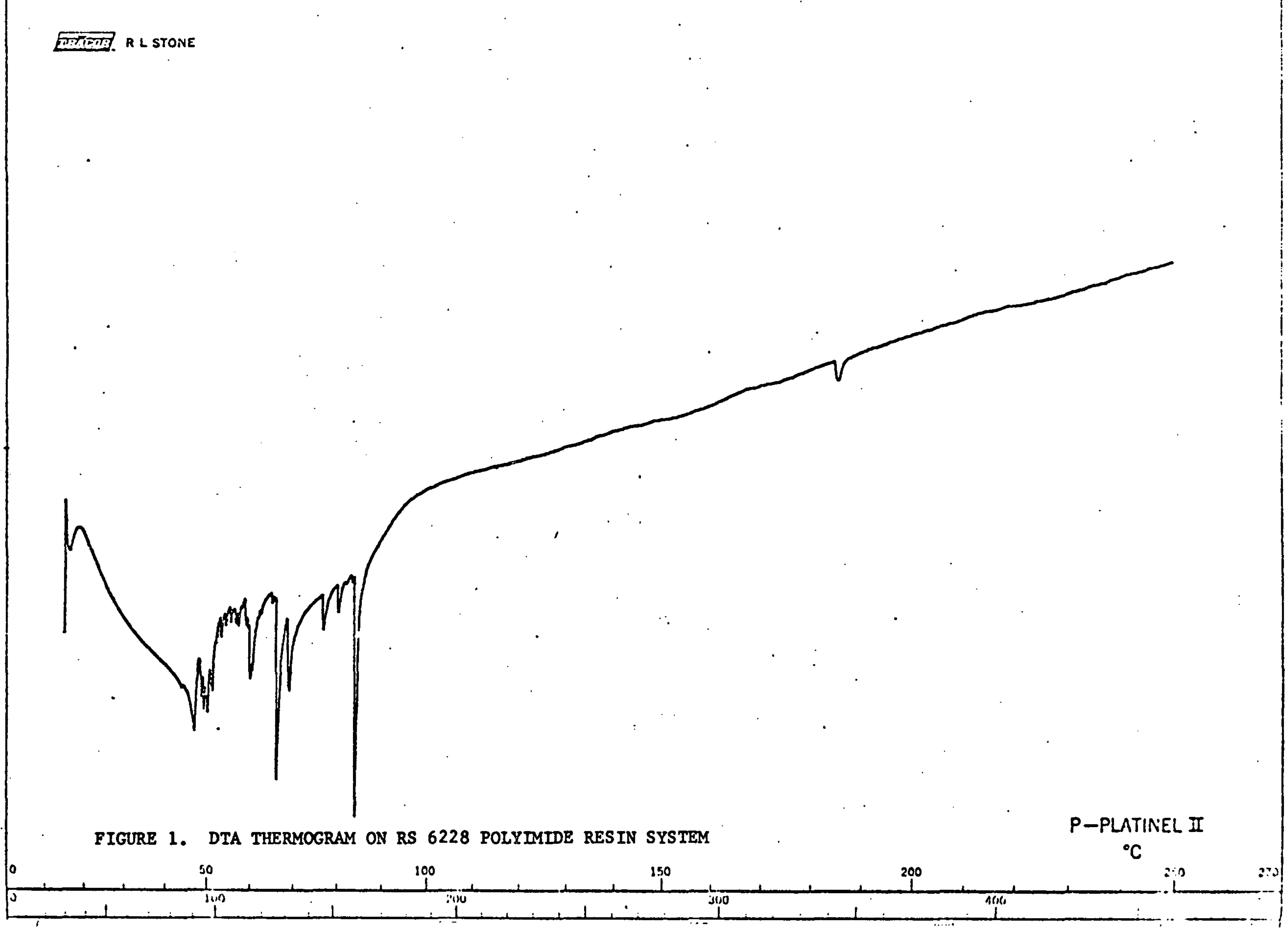


FIGURE 1. DTA THERMOGRAM ON RS 6228 POLYIMIDE RESIN SYSTEM

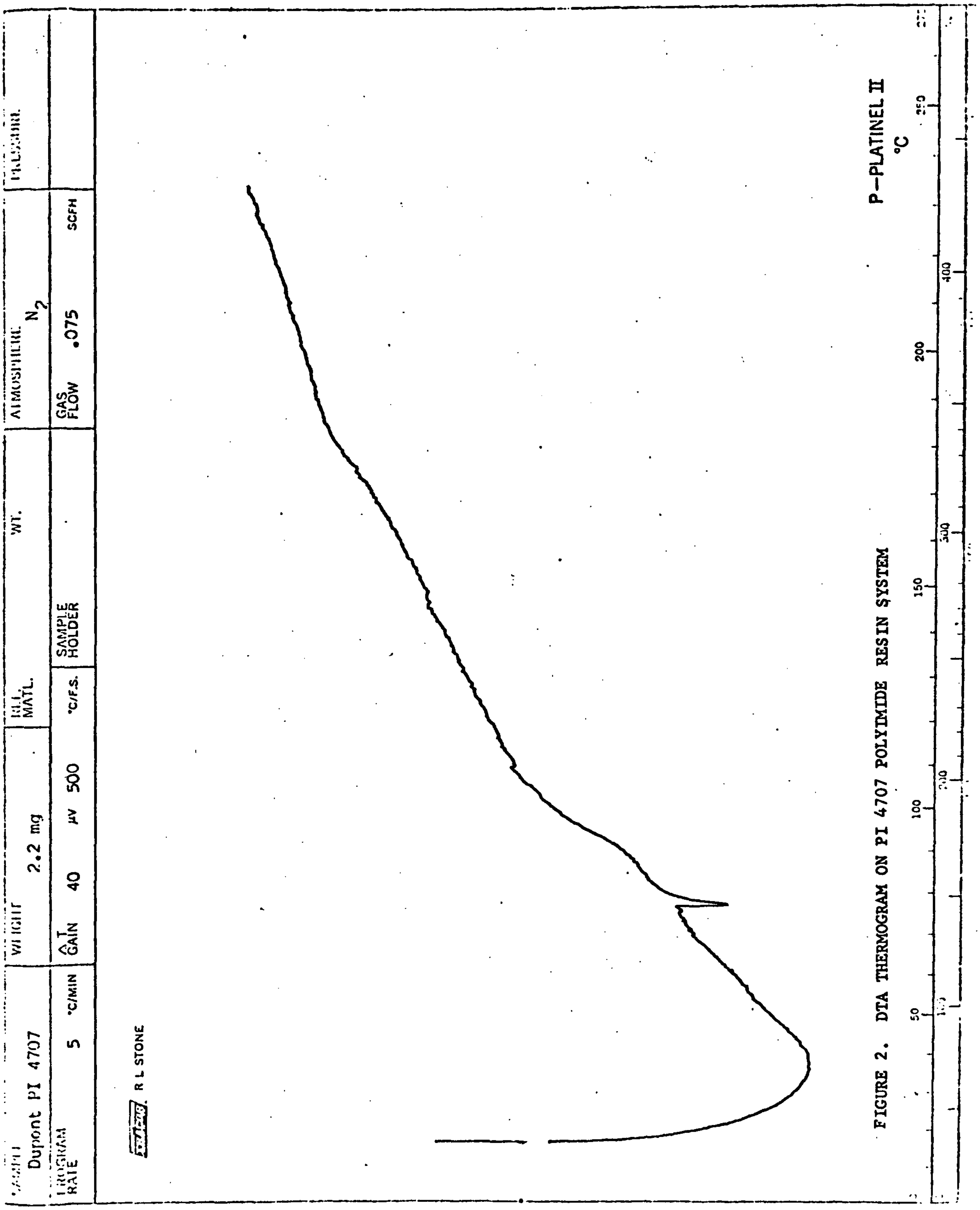


FIGURE 2. DTA THERMOGRAM ON PI 4707 POLYIMIDE RESIN SYSTEM

SAMPLE Dupont 35 - 520	WEIGHT 2.2	REF. MATL. WT.	ATMOSPHERE N_2	PRESSURE
PROGRAM RATE 5 °C/MIN	ΔT GAIN .40 μV 500 °C/F.S.	SAMPLE HOLDER	GAS FLOW .075 SCFH	

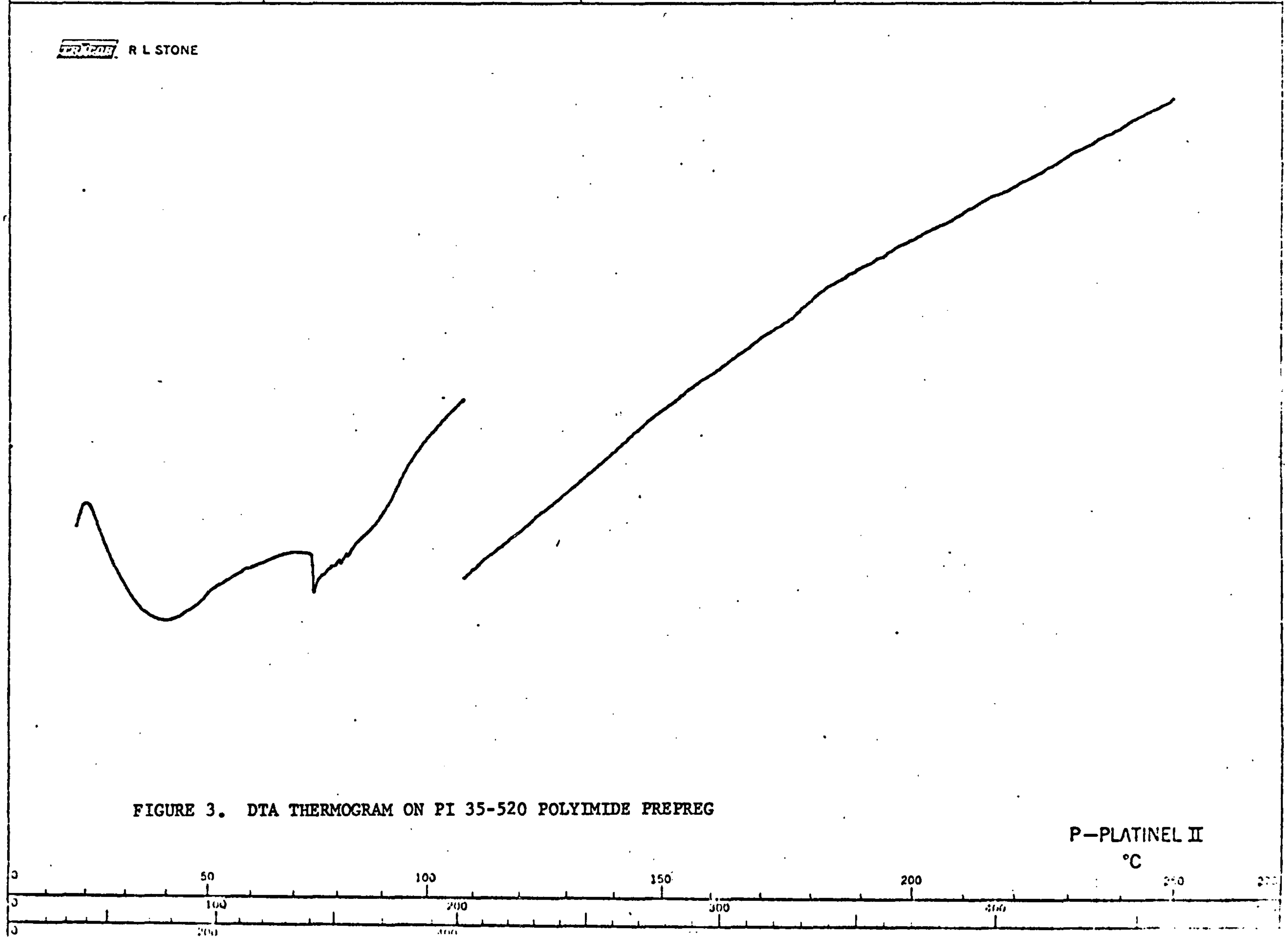


FIGURE 3. DTA THERMOGRAM ON PI 35-520 POLYIMIDE PREPREG

A TGA thermogram/mass spectrometer run was made on the same DuPont prepreg. The sample was exposed to hard vacuum and then heated at 4 C (7.2 F) per minute. Before beginning heating, the sample lost weight gradually, indicative of loss of solvent, water (probably not) or pyrrolidone. Upon heating, the rate of weight loss increased rather gradually to about 100 C (212 F) at which temperature the rate of weight loss increased significantly. This rate continued to about 350 C (662 F) at which temperature the weight loss rate appeared to diminish somewhat. During the major period of this TGA run, the low resolution mass spectrometer was set to monitor the presence of water. Results indicated that water evolution peaked at 110 C (230 F) and then fell to a relatively constant value at about 175 C (347 F) which probably represented the equipment background level. Accordingly, these results suggest that the reaction producing (condensation of) water is virtually complete at about 110 C (230 F). However, since considerable weight loss occurs above this, it seems likely that a different condensation product is being evolved. Confirmation of this was obtained in a later programmed TGA run.

An inflection was noted around 350 C in the DTA curves for the Pyralin PI 4707 and 35-520 prepreg and also in the TGA for the 35-520 prepreg. Although this may reflect the initiation of degradation, it does not appear to be significant based upon the TGA curve below about 480 C (896 F). The mass spectrometer was shifted from the water peak at 480 C (896 F) to scan the decomposition products being evolved. Evidence was noted during this scan of the presence of nitrogen, carbon dioxide, and some atomic oxygen.

A TGA-mass spectrometer run was made using the time-temperature program recommended by North American Rockwell for vacuum bag fabrication with the Pyralin 35-520 prepreg. The TGA (Figure 4) shows two regions of significant weight loss. The first of these occurs about 110 to 120 C (230 and 248 F) with the weight change approaching constancy after 6 hr. total.

The mass spectrometer was set to periodically scan through n-methyl pyrrolidone on the products from this TGA. Six scans were conducted at nominal temperatures of 57, 83, 115, 118, and two at 171 C. Evolution of water was observed to be increasing at 83 C (181 F) but was falling off at 115 C (239 F). This, of course, is consistent with the previous mass spectrometer run where the scan showed the peak evolution at 110 C (230 F). The present scan revealed the evolution of a material of around m/e 31. It is suspected that this is probably a volatile product (possibly methyl amine) resulting from the condensation of a modified polyimide precursor. This was observed during the first scan at 57 C (135 F). The evolution, however, was higher during the scan at 83 C (181 F) and appeared to peak at about 115 C (239 F) but the evolution remained high and relatively constant through the first scan at 171 C (340 F). A subsequent scan at 171 C revealed a relatively low evolution.

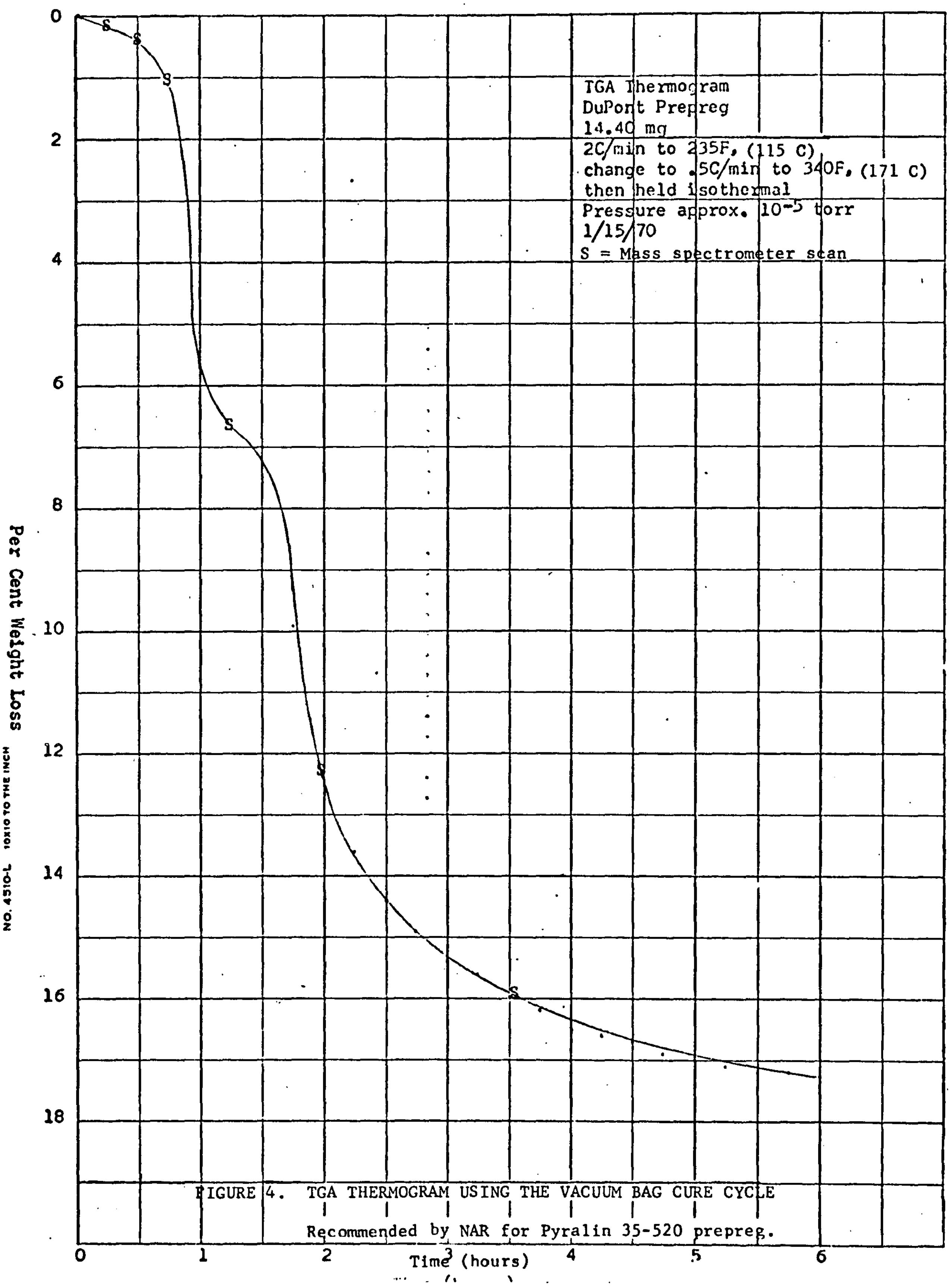


FIGURE 4. TGA THERMOGRAM USING THE VACUUM BAG CURE CYCLE

Recommended by NAR for Pyralin 35-520 prepreg.

Two other materials were observed in the mass spectra. These were about 92 and 99. It is believed that the first material probably is toluene, present as part of the solvent system. Evolution of this material appeared highest during the first scan and fell off slightly during subsequent scans. A significant amount still was noted during the scan at 118 C (244 F) but the level appeared around background value during the first and second scans at 171 C (340 F). Evolution of the m/e 99 material, probably n-methyl pyrrolidone, was fairly constant and rather low during the spectrometer scans until the first scan after reaching 171 C (340 F). Evolution during this scan was considerably higher, but the rate had fallen considerably in the second scan at 171 C (340 F).

It should be noted that equipment limitations exist in attempting to quantize mass spectrometer data obtained in the TGA-spectrometer train. Materials volatilized in the TGA heated zone are exposed to considerable cool adsorptive surfaces existing in the combined assembly. This, of course, militates against sharp quantitative cuts highly responsive to sample temperature. It is reasonable to project, however, that peak indications are meaningful since this represents the system flooding point for a given material. It is expected that the adsorptive system surfaces will affect the return to background values. Thus, it is believed that the data on materials is isolated and trends are realistic. Based upon these limitations, data to date indicate the projected cure schedule to be reasonable. Off-gases evolved during gelation generally appear fairly constant or falling. The exception to this appeared to be the n-methyl pyrrolidone which significantly increased when the laminate first reached 171 C. Accordingly, a possible modification of the cycle would be to hold the laminate temperature for a short time at about 140 to 150 C (285 to 300 F) to reduce the possible impact of n-methyl pyrrolidone volatilization on reaching 171 C(340 F).

3.5 Dissipation Factor Studies

Dissipation factor studies were made on DuPont PI 35-520 prepreg. The samples consisted of four plies of prepreg about 1-1/2 in. square. A thermocouple bead (36 gage) was placed between the two center plies to monitor temperature. Two plies of glass cloth were used on either face of the laminate to electrically insulate the dissipation factor monitors. These monitors consisted of strips of aluminum foil placed between the glass cloth (7628) and positioned so that ends of the strips were over the prepreg. The aluminum strips were long enough to extend just beyond the glass cloth insulation. The protruding ends of the foils were tapered to preclude foil to foil contact which would prevent dissipation measurements.

Direct contact of the foil electrodes to the small laminates also was shown to result in a shorted system. The use of glass cloth between the foils and the prepreg prevented the electrical short through the prepreg. Similarly, the use of glass cloth between the electrodes and sheet metal caul plates prevented an electrical short when the cauls were inserted in a heated press. During dissipation studies contact was made between the upper platen and caul to provide good heat transfer but pressure was not applied. A manually operated Heath Kit impedance bridge was utilized for measuring the dissipation factors.

The dissipation factor technique was used in various studies of the curing behavior of the DuPont PI 35-520 prepreg. A number of parameters were investigated to obtain insight on their criticality. These included measurements at various temperatures both in a press and vacuum bag assembly. The laminate assembly and monitoring procedures previously described were used throughout this work. For vacuum bag studies the assembly was inserted in a flat vacuum bag fabricated from Fairprene 5806 (glass-cloth reinforced silicone rubber). The edges were stitched for reinforcement and sealed with RTV 732. This system had sufficient resilience that a good vacuum seal was obtained by clamping the open side over the thermocouple and aluminum foil leads. Thin wooden strips were used on either side of the vacuum bag as back-ups for binder clips. The foil leads were folded over the top back-up strip and a clamp used over each lead. This provided mechanical protection for the rather delicate lead and permitted the binder clamp to be used as the electrical contact. The bag was heated in the same press used for the press studies. However, when the press was used as a vacuum bag heater, care was taken to prevent putting a load on the assembly.

Results of studies of conditions and procedures are summarized in Figure 5. Data from three curing procedures are summarized. These include the press assembly, vacuum bag, and a modified vacuum bag. In the vacuum bag procedure, evacuation was used throughout the run. In the modified procedure, the vacuum bag unit was heated before applying the vacuum.

Several interesting observations may be derived from Figure 5 and the procedures utilized in deriving the data. The first two observations were based upon press-cure studies. It was noted that the equilibrium dissipation factor tended to decrease rather quickly with time such that a given sample could not be programmed to a series of temperatures. Equilibrium dissipation factor in this figure accordingly relates to the value attained with a fresh sample at thermal equilibrium and no press load. This generally follows the lower dissipation factor curve. It was further observed that press-loading increased the dissipation factor value at constant temperature. This resulted in the higher dissipation factor line. Undoubtedly the increase in dissipation factor with pressure and temperature can be attributed both to the concentration of water and solvent vapor evolved and to molecular mobility.

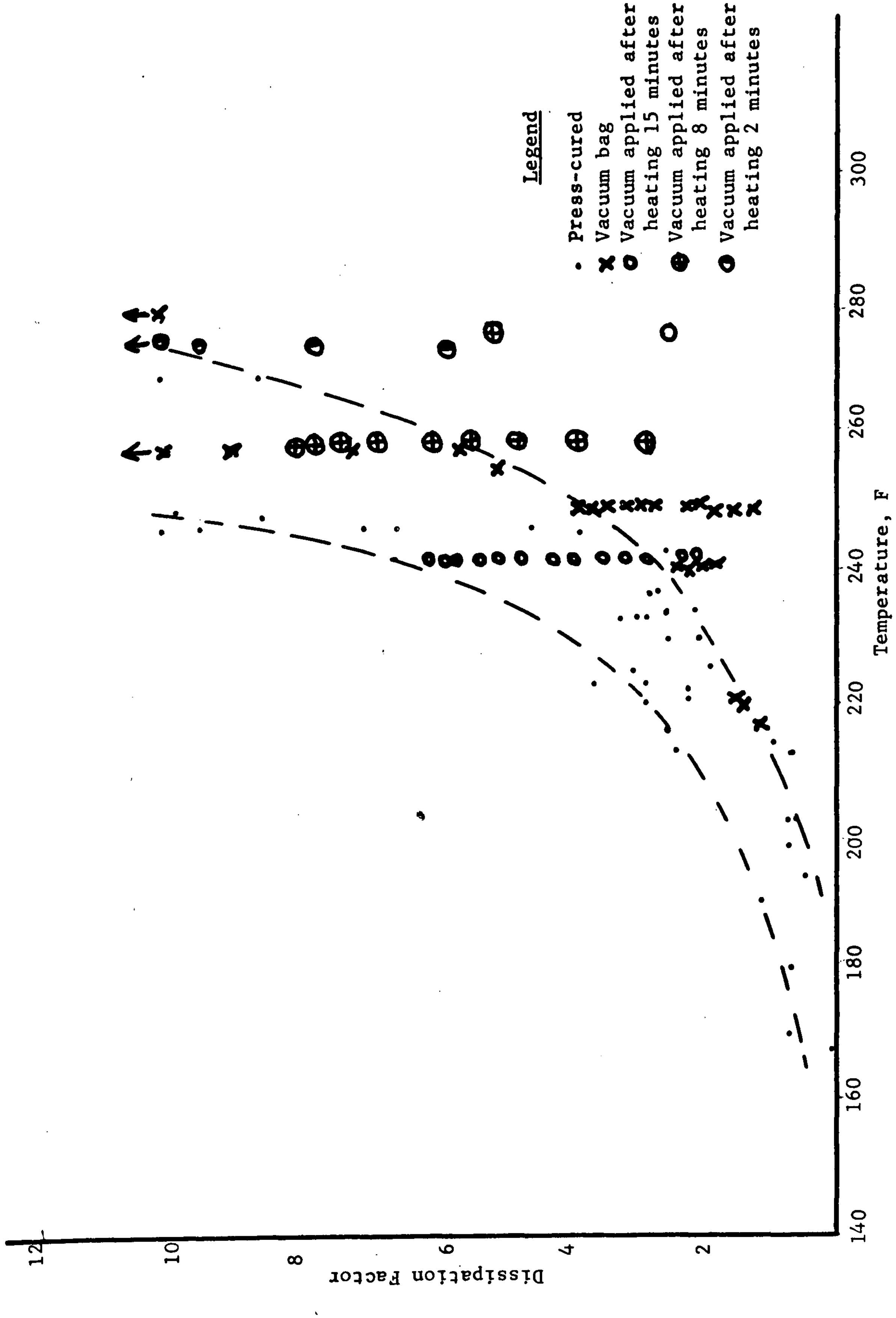


FIGURE 5. EFFECT OF LAMINATING CONDITIONS AND PROCEDURES ON DISSIPATION FACTOR MEASUREMENTS ON DU PONT 35-520 PREPREG

Correlation of the equilibrium dissipation factor vacuum bag data with the press data appears to fall reasonably well into the previously established pattern. The maximum dissipation factor values for runs at 240 and 250 F appear to approach the lower dissipation factor line. Vacuum runs at 260 and 280 F appear to quickly reach the lower dissipation factor value at temperatures such that the values fall within the boundaries established by heated press studies.

Results of modified vacuum runs wherein the evacuation was delayed during part of all of the heating period is reflected by deviations from the dissipation factor zone. This is quite pronounced in a run at 274 F which was not evacuated until 8 minutes after heating was initiated. During this period, imidization had proceeded to the extent that the maximum dissipation factor value was considerably below the correlation zone. A comparison run at 274 F was heated for 2 minutes (to 240 F) before applying vacuum. This did reach a maximum within the dissipation factor correlation as did a run at 260 F preheated for 8 minutes.

Results of these dissipation studies thus give background information on the effects of temperature, pressure, and evacuation factors on imidization rate and extent of the reaction.

The results of dissipation factor measurements of PI 35-520 prepreg with various treatments and at different temperatures also were studied. It is apparent that various treatments as well as temperature affect the magnitude of the dissipation factor.

The effect on dissipation factor (d.f.) of using water or n-methyl pyrrolidone (nmp) as softening agents for the prepreg is shown in Figure 6. It is believed that the difficulty of eliminating the nmp is evidenced both by the larger value and the prolonged observation of the dissipation factor. The high value for d.f. appears typical for runs at higher temperatures. The marked and prolonged observations of d.f. is likely due to rapid gelation of resin with consequent entrapment of water vapor. This is markedly different from the run at 220 F where the d.f. is noted only momentarily. Although previous work indicated that press loading during monitoring might be responsible for some of the observed differences in the various runs, care was taken to prevent pressurization in these runs. Furthermore, studies (Figure 7) utilizing a vacuum bag in which differences in loading should not be a factor showed the same effects of temperature. That is, an elevated temperature, 279 F, rapidly produced a highly dissipative and slowly decaying system. Runs at lower temperatures, 240 and 250 F, presumably did not achieve a rate of gelation sufficient to produce either a high dissipation factor or prolonged decay of dissipation factor. It seems apparent from this work that lower temperatures may result in a somewhat better fabrication cycle. Unfortunately, this factor must be balanced with the high viscosity and poor flow at low temperature. Accordingly, the cure tradeoffs needed are those which efficiently remove the resin volatiles with minimal entrapment and which have adequate flow to give a void-free laminate.

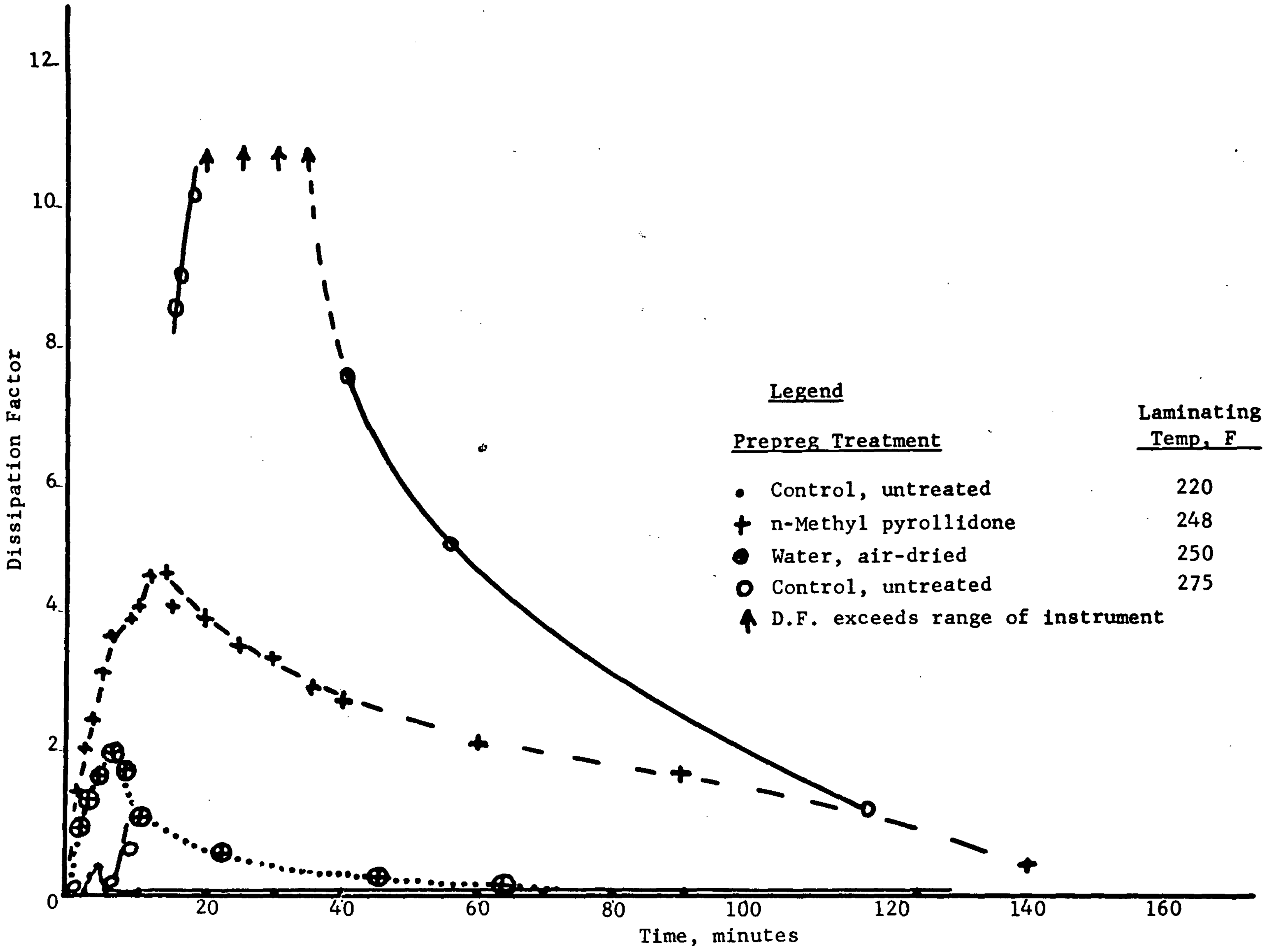


FIGURE 6. CORRELATION OF DISSIPATION FACTOR WITH PREPREG (DU PONT 35-520) TREATMENT AND PRESS-LAMINATING CONDITIONS

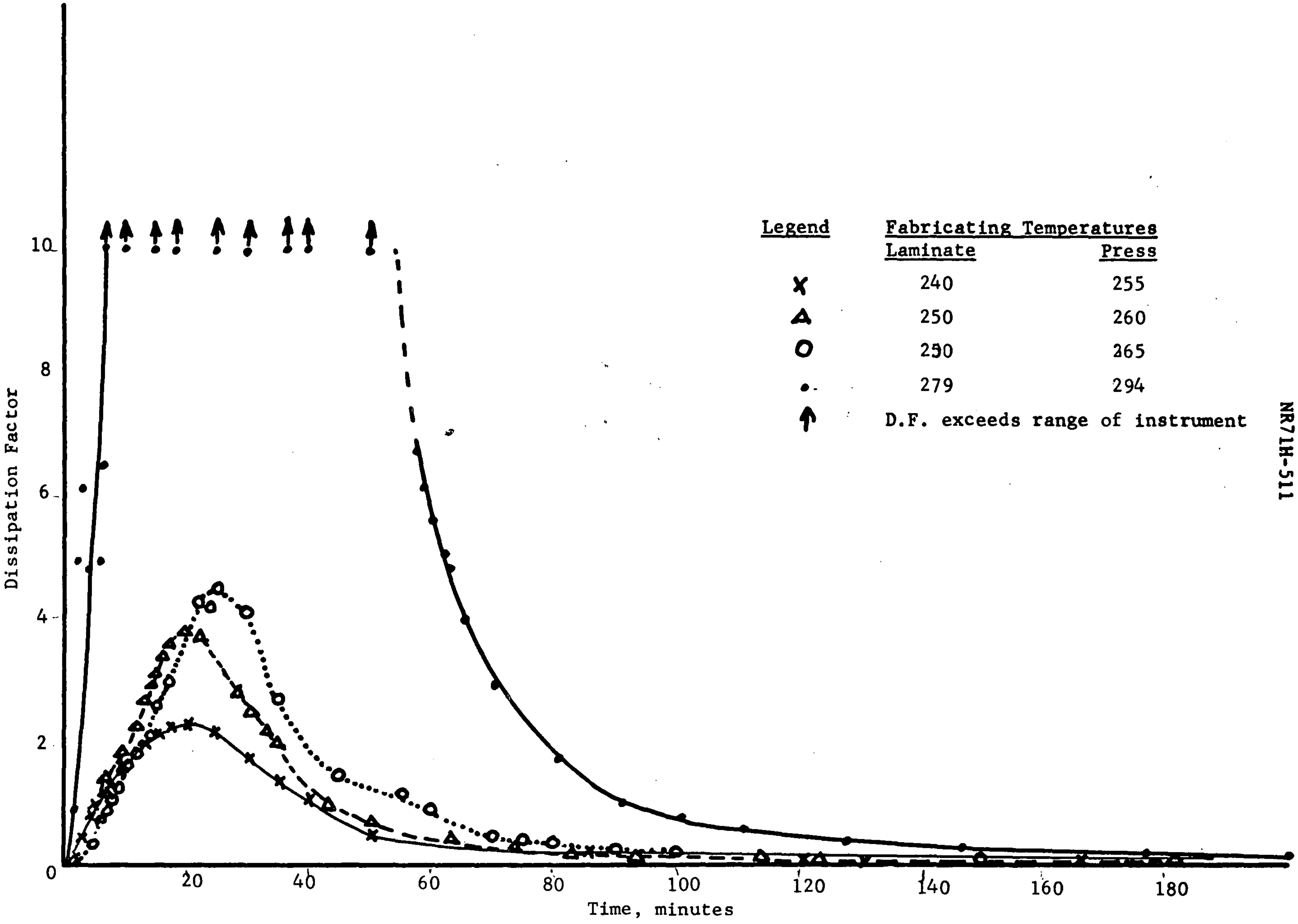


FIGURE 7. CORRELATION OF DISSIPATION FACTOR WITH TIME FOR VACUUM BAG LAMINATIONS USING DU PONT 35-520 PREPREG

Several modifications in vacuum cure cycles were studied in evaluating possible tradeoff procedures. Figure 8 correlates the laminate temperature and dissipation factor with time using a fabrication cycle suggested by DuPont for PI 35-520. This cycle does reduce the magnitude of the dissipation factor and the time required for decay. In general, the laminate appeared reasonably good. However, both foamed resin and unfilled voids were noted upon microscopic study of the specimen.

Figure 9 shows the effect of delayed application of vacuum. It may be noted that this procedure is effective at high temperatures in reducing the magnitude and decay time of the dissipation factor. The general appearance of these test laminates and others produced during vacuum bag studies are summarized in Table 1. These results should be considered qualitative and are presented only for comparison of the respective merits of the various laminating procedures.

Several conclusions appear to be in order based upon this laminate evaluation and the dissipation factor studies of the PI 35-520 prepreg.

(1) Initial cure temperatures much above 275 F may produce gelation at a rate sufficient to preclude optimum flow as well as producing foam in resin-rich areas.

(2) Cure temperatures below 250 F do not provide adequate flow.

(3) Laminate fabrication under conditions which produce dissipation factors below 6.0 appear to result in poor flow and bonding.

(4) The best flow characteristics appear to be obtained around 260 to 275 F.

(5) Vacuum-bag lamination requires a critical control of laminating temperature for optimum composites.

(6) Vacuum bag laminating under conditions which produce dissipation factors above 8.0 appear to result in composites which contain both voids and foamed resin.

(7) Either preheating before evacuation or programmed heating may be used to reduce the level of prepreg volatiles to minimize foaming and voids.

(8) Resin flow undoubtedly is limited even under optimum vacuum laminating conditions. Accordingly,a high uniformity of resin distribution in laminate prepregs is imperative in production of high-quality composites.

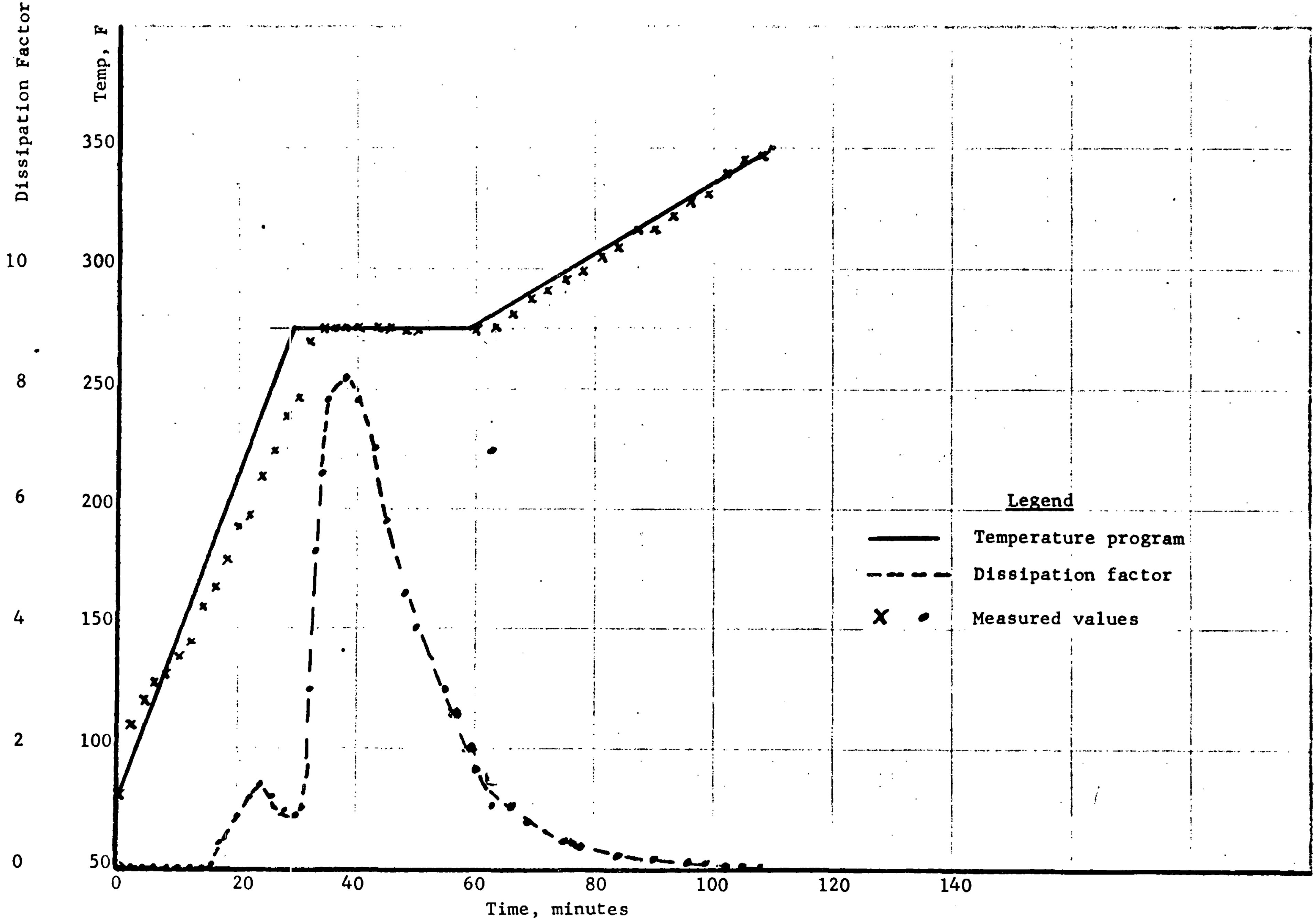


FIGURE 8. PROGRAMMED VACUUM BAG FABRICATION RECOMMENDED BY SUPPLIER FOR DU PONT 35-520 PREPREG

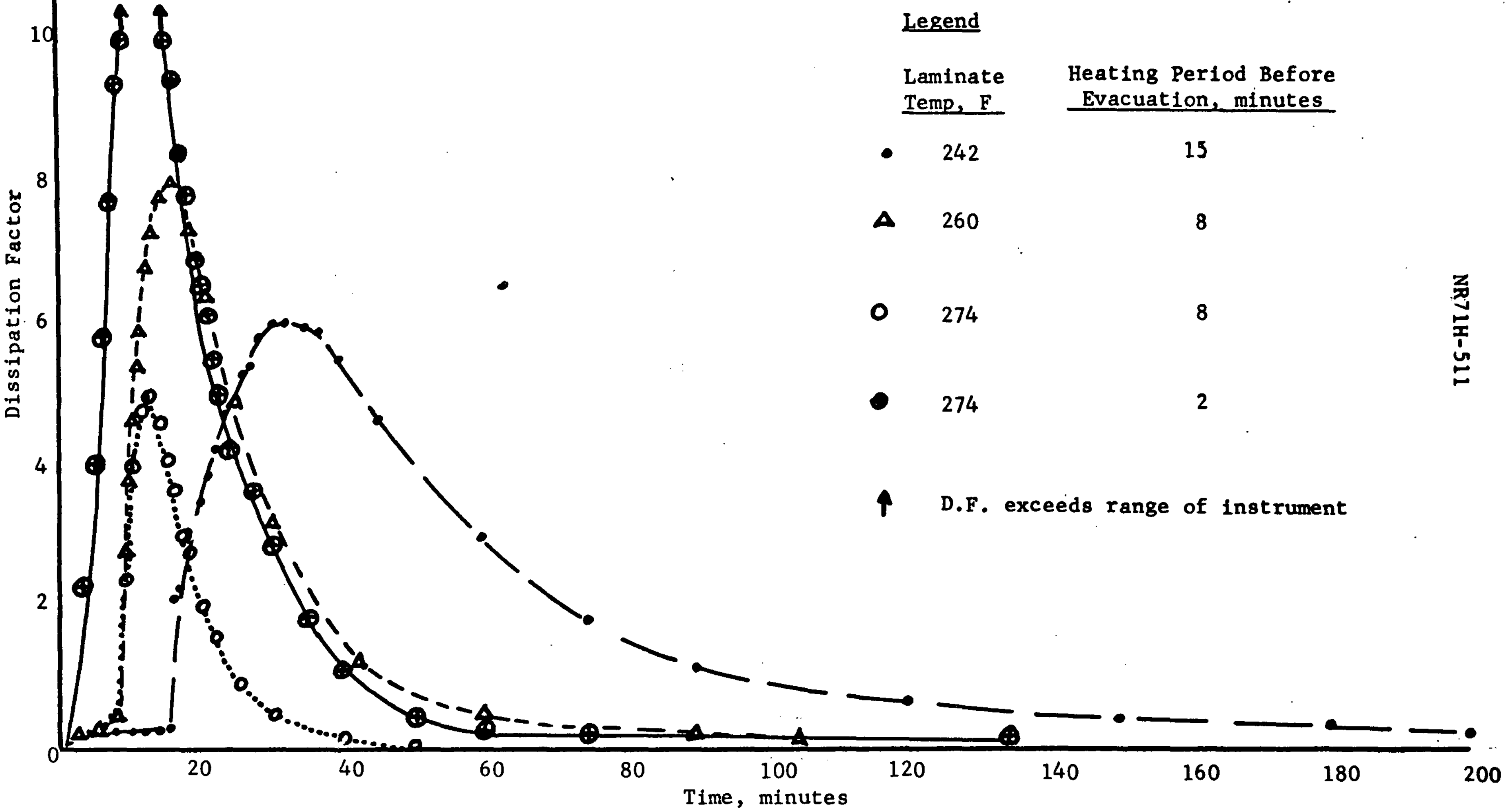


FIGURE 9. MODIFIED VACUUM BAG LAMINATION STUDIES WITH DU PONT 35-520 PREPREG

TABLE 1. SUMMARY OF VACUUM BAG LAMINATING STUDIES (DUPONT 35-520, 4 PLIES)

Laminating Temperature, F	Thickness, mils[(a)] Average	Thickness, mils[(a)] Range	Estimated Pre-Preg Volatiles, percent	Estimated Resin Loss, percent	Laminate Appearance
220	21.6	20.0-23.5	--	--	Fair, limited glass marking, unfilled voids.
241	20.3	18.8-22.0	--	--	Fair, limited glass marking, unfilled voids.
242[(b)]	17.2	15.0-19.0	12.7	6.7	Fair, nominal glass marking, unfilled voids.
250	19.1	17.0-21.5	--	--	Fair, good glass marking, unfilled voids.
259	17.7	15.5-20.5	--	--	Fair, nearly total glass marking, unfilled voids.
260[(c)]	17.7	15.5-20.0	12.3	11.3	Fair, nearly total glass marking, some unfilled voids.
263	15.9	14.5-16.5	20.6	8.0	Good, nearly total glass marking, some unfilled voids.
274[(d)]	17.4	14.8-19.5	23.0	None[(g)]	Fair, nominal glass marking, some voids, some foamed resin.
274[(e)]	18.4	17.0-19.8	15.0	2.2	Fair, nominal glass marking, unfilled voids.
274[(f)]	18.4	17.0-20.0	18.8	None[(g)]	Fair, nearly total glass marking, some unfilled voids, some resin foamed.
279	18.5	17.0-20.0	18.7	11.5	Fair, nearly total glass marking, considerable voids, considerable foam.

(a) Calculated laminate thickness, 12.5 mils assumes no resin loss nor surface marking from bleed glass. Such flow into fabric weave probably will result in high thickness values.
(b) Evaluation 15 minutes after heating started.
(c) Evacuation 8 minutes after heating started.
(d) Programmed heating approximately 7 F/minute to 275 F; 30 minutes hold; postcure program 1.5 F/minute to 350 F.
(e) Evacuation 8 minutes after heating started.
(f) Evacuation 2 minutes after heating started.
(g) Glass adhering to surface compensated for resin loss.

It was demonstrated that the dissipation factor technique was valuable in establishing a correlation for indicating resin fluidity useful in press fabrication. It becomes apparent that the vacuum bag fabrication technique required a much less rigorous heating program. The time-temperature program shown in Figure 10 was projected as a result of various fabrication and analytical studies. It will be noted that the sensitivity of the instrument was inadequate to monitor the dissipation factor of this laminate. The measurement shows, however, that the temperature schedule does not result at any time in a significant rate of volatile production.

Additional runs were made to establish the feasibility of using dissipation measurements for boron containing composites. Results are shown in Figure 11 for two vacuum bag laminations at 260 F. The unreinforced laminate showed a peak dissipation value of about 9. The boron reinforced system showed a corresponding peak value of about 0.5. Although the boron fiber tends to destroy the dissipation signal, a sufficiently sensitive instrument may be capable of monitoring changes in an inherently shorted system. Studies with an instrument having improved monitoring capabilities at low dissipation values appear highly desirable. It is believed that such studies would reveal a potential for d.f. control of vacuum bag fabrication similar to that used in press lamination procedures.

3.6 B-Staging Studies

A series of tests were performed to determine weight change of the 35-520 prepreg as a function of exposure to steam and drying time. The steam was at 212 F and was partially condensed (visible) as it contacted the specimen. Drying was in ambient air (75 F, 50 per cent RH). Weight changes were measured for up to 5 minutes steam exposure. They indicate that a net loss occurs after approximately 40 minutes (actually 35 minutes drying time) for a steam exposure of 5 minutes. The maximum weight gain was 35.1 percent, and the net weight loss was 29.3 percent after levelling off beyond 100 minutes. These figures are based on resin weight only.

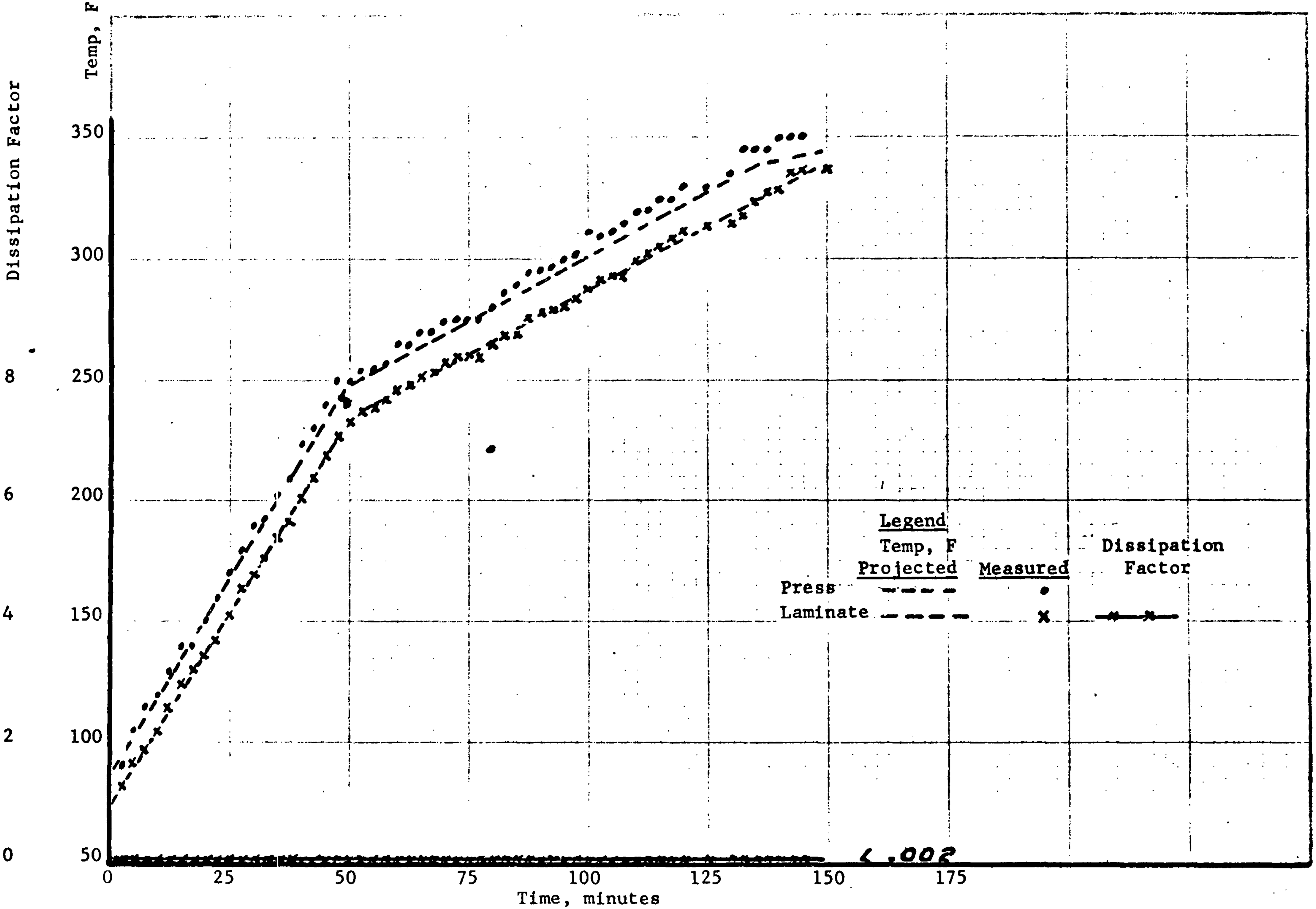


FIGURE 10. CORRELATION OF DISSIPATION FACTOR WITH BCL-RECOMMENDED FABRICATION CONDITIONS FOR DU PONT 35-520 PREPREG

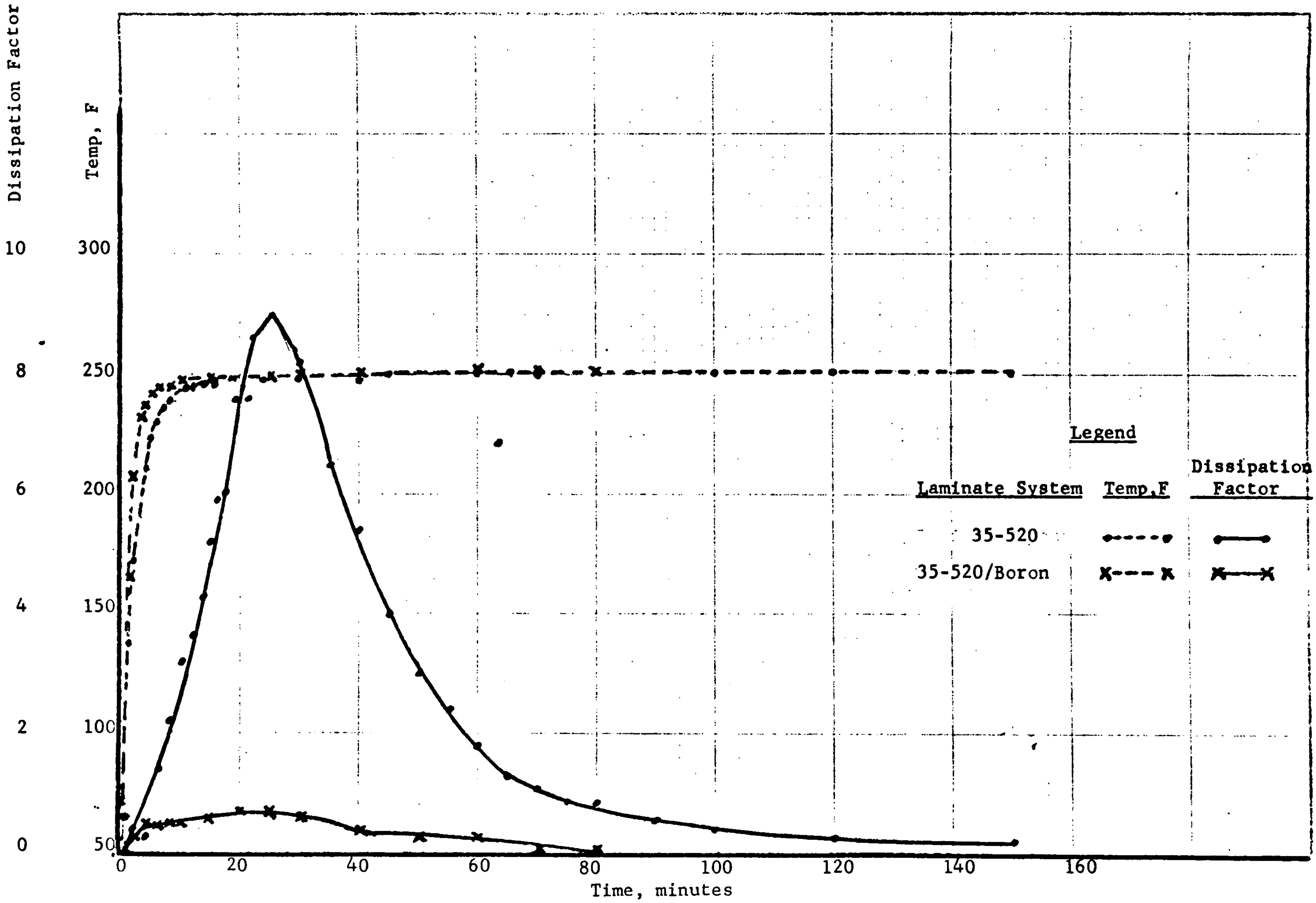


FIGURE 11. EFFECT OF BORON REINFORCEMENT ON DISSIPATION FACTOR DETERMINATION

The weight loss occurring after the initial water absorption is probably due to formation of an azeotrope between water and the solvent in the resin (n-methyl pyrrolidone). This is a reaction which might well be used to reduce the volatile content prior to imidization. To determine the efficacy of the azeotrope in reducing volatile content, a series of prepreg specimens were exposed to steam, air-drying, and subsequent B-staging cycles. Table 2 is a summary of weight loss data. The last four specimens listed in Table 2 were also cured at 350 F for 1 hr. and reweighed. The resulting weight changes consisted of any remaining volatiles plus water of condensation from the imidization reaction. Following the 350 F cure, the specimens were burned out at 1200 F in air to determine resin content. Table 3 shows the percentage weight loss during cure as a function of the B-stage cycle, based on resin weight only.

The above results indicate that imidization is occurring at lower temperatures than generally assumed. DuPont has stated that the 33 percent volatile content of the prepreg is approximately equally divided between the solvent (n-methyl pyrrolidone) and water of imidization. Considering that the specimen which was B-staged for 1 hr. at 240 F had a cumulative weight loss of 14.8 percent, it is obvious that a significant part of this weight loss must have been water of imidization as shown by the fact that only 5 percent additional weight loss occurred during the 350 F cure. The conclusion is that the final cured resin must have a significant amount of solvent retention. This conclusion is further supported by noting that the boiling point of n-methyl pyrrolidone is between 387 and 396 F. Thus, the imidization apparently starts around 240 F and proceeds rapidly enough (particularly at 350 F) that there is insufficient time and temperature for significant effusion of solvent. The major solvent effusion must, therefore, occur during the high temperature post cures generally specified for polyimides.

There is an apparent anomaly between the final weight loss obtained after 5 minutes steam exposure and air dry (29.3 percent) and weight losses occurring after 2 minutes and 10 seconds steam exposures followed by air drying (approximately 1/2 to 1 percent). The 29.3 per cent value appears abnormally high, since the total volatiles (solvent plus water of imidization) is nominally 33 percent. Possible explanations are that the long exposure actually caused some imidization to occur.

The above discussion and data apply to processing under ambient atmospheric pressure. If B-staging is done in vacuum, it may be possible to remove more of the solvent. However, significant solvent removal can take place only if the mean free path of the solvent molecule to an exposed surface is very short, i.e., the B-staging is done prior to laminating.

TABLE 2. WEIGHT CHANGE DATA FOR VARIOUS B-STAGES OF DUPONT 35-520 PRE-PREG

Steam Exposure	Percent Weight Change	Air Dry*	Cumulative Percent Weight Change	B-Stage Cycle**	Cumulative Percent Weight Change
None	--	2/78/46	- 0.18	1/220	- 8.33
2 min	+ 33.0	2/78/46	- 0.41	1/220	- 9.96
10 sec	+ 2.4	--	--	0.33/240	- 10.1
10 sec	+ 2.4	--	--	0.67/240	- 11.5
10 sec	+ 2.4	--	--	0.67/270	- 17.3
10 sec	+ 2.4	--	--	1/270	- 18.7
2 min	+ 33.7	2/76/39	- 0.59	1/240	- 14.8
2 min	+ 26.6	2/75/25	+ 0.97	6/240	- 25.0
2 min	+ 57.0	2/75/25	- 0.47	12/240	- 26.3
2 min	+ 29.4	2/75/25	- 1.1	24/240	- 28.4

Note - All weight percent changes are based on original resin weight only (assumed 78 percent of total pre-preg weight).

*Number of hours/temperature in F/relative humidity in percent.

**Number of hours/temperature in F.

TABLE 3. POLYMERIZATION WEIGHT LOSS VERSUS B-STAGE FOR DUPONT 35-520 PRE-PREG

B-Stage Cycle	Cumulative Percent Weight Change After B-Stage	Percent Weight Change During Cure*	Percent Resin Content
1/240	-14.8	-5.0	74.0
6/240	-25.0	-4.5	75.0
12/240	-26.3	-2.4	75.5
24/240	-28.4	-2.26	77.6

* Cure - 1 hour at 350 F.

To determine a lower boundary temperature for B-staging under vacuum, an absolute pressure of 0.01 atmosphere was assumed. Trouton's rule gives the molar heat of vaporization, ΔH_v, as:

$$\frac{\Delta H_v}{T_b} = 21$$

where T_b is the boiling point at 1 atmosphere. For n-methyl pyrrolidone, T_b = 473K. Thus:

$$\Delta H_v = 9930 \text{ cal./mole.}$$

This value can be used in the Clausius-Clapeyron equation:

$$\log_{10} \frac{P_2}{P_1} = \frac{\Delta H_2 (T_2 - T_1)}{2.303 R (T_2 T_1)}$$

where P = pressure, T = boiling point, and R is the gas constant. Subscript 2 refers to standard conditions (1 atmosphere), subscript 1 refers to the conditions desired, and the gas constant R is 1.987 for ΔH_v in cal/mole. Substituting these values in the equation gives a boiling point of approximately 71 C (160 F) for n-methyl pyrrolidone at 0.01 atmospheres.

To determine the effectiveness of a possible solvent extraction cycle prior to B-staging and curing, specimens of DuPont 35-520 were heated to 160 F in a vacuum oven at 0.01 atmosphere (7.6 mm Hg), and weight loss versus time was measured. The results are shown in Table 4. It is obvious that the largest fraction of the weight losses occurred within the first 2 hr. The average weight loss, based on resin weight only, was 11.1 percent.

It was also necessary to determine if this solvent extraction cycle advanced the B-stage (or reduced the solvent content) enough to preclude resin flow during the cure cycle. This was done by vacuum bag molding a laminate of six layers of the treated prepreg (without boron) at 350 F with no attempt to optimize the process. The results showed that flow existed after the vacuum heating cycle of 2 hr. at 160 F and 7.5 mm Hg absolute pressure.

TABLE 4. WEIGHT LOSS OF DUPONT 35-520 PRE-PREG
AT 160 F, 7.6 mm Hg

Specimen Number	Initial Weight, gm	Weights at Indicated Number of Hours From Start, gm					
		1	2	3	4	5	6
1	0.186	0.175	0.170	0.169	0.168	0.167	0.167
2	0.190	0.178	0.174	0.173	0.172	0.171	0.171
3	0.178	0.168	0.164	0.163	0.163	0.162	0.162

3.7 Cure Cycle Studies

Evaluation of the TGA and DTA data indicated two possible approaches for the cure cycle. One would be to accomplish the imidization in the 100-110 C range which would probably give a longer gelation period for the water of imidization to diffuse out of the resin mass. Counterbalancing this desirable factor would be a higher range of viscosity during gelation which would mitigate against the ease of diffusion as well as the ability to flow readily under low pressure (only 1 atmosphere is available in the vacuum bag process). The second approach would be to take the resin up to the 150 C range at a rate slow enough to avoid resin starvation and thus take advantage of the lower viscosity range during gelation to provide proper flow and compaction.

A series of small laminates were made using various cure cycles. The layup scheme for vacuum bagging is shown in Figure 12. The various cure cycles are given in Table 5. Specimens A-2 through A-6 were based on accomplishing the major part of the imidization at low temperatures (220 F), and varied mostly in the use of the solvent extraction cycle and the rate of temperature increase. Specimens A-7 through A-16 were based on the major imidization occurring at 280 F or higher, which is in the range where maximum total weight loss rate occurs.

One specimen (designated PI-12) was prepared without boron and was cured using a cycle suggested by DuPont. This cycle proved completely unacceptable and further attempts at using the higher heating rates and dwell periods recommended by DuPont were discontinued. In all fairness, the higher heating rates may be suitable for pressure molding cycles, but for "vacuum bag only" cycles with boron filaments, they generate volatiles too rapidly and force gelation during this rapid outgassing period.

Examination of specimen panels showed that mottling (actually caused by high void areas) always occurred on the surface adjacent to the caul plate. Thermocouple traces taken during the cure cycles did not indicate any significant temperature lag between the caul plate surface and the peel-ply surface of the laminate during cure. The only apparent explanation for this effect was that volatile removal was more effective on the peel ply surface of the laminate. To check this hypothesis, panels were molded with one or two layers of F-56 peel ply interposed between the caul plate and the laminate as well as on the top surface. Those molded with two interposed layers (A-16) looked particularly sound and had no noticeable mottled areas.

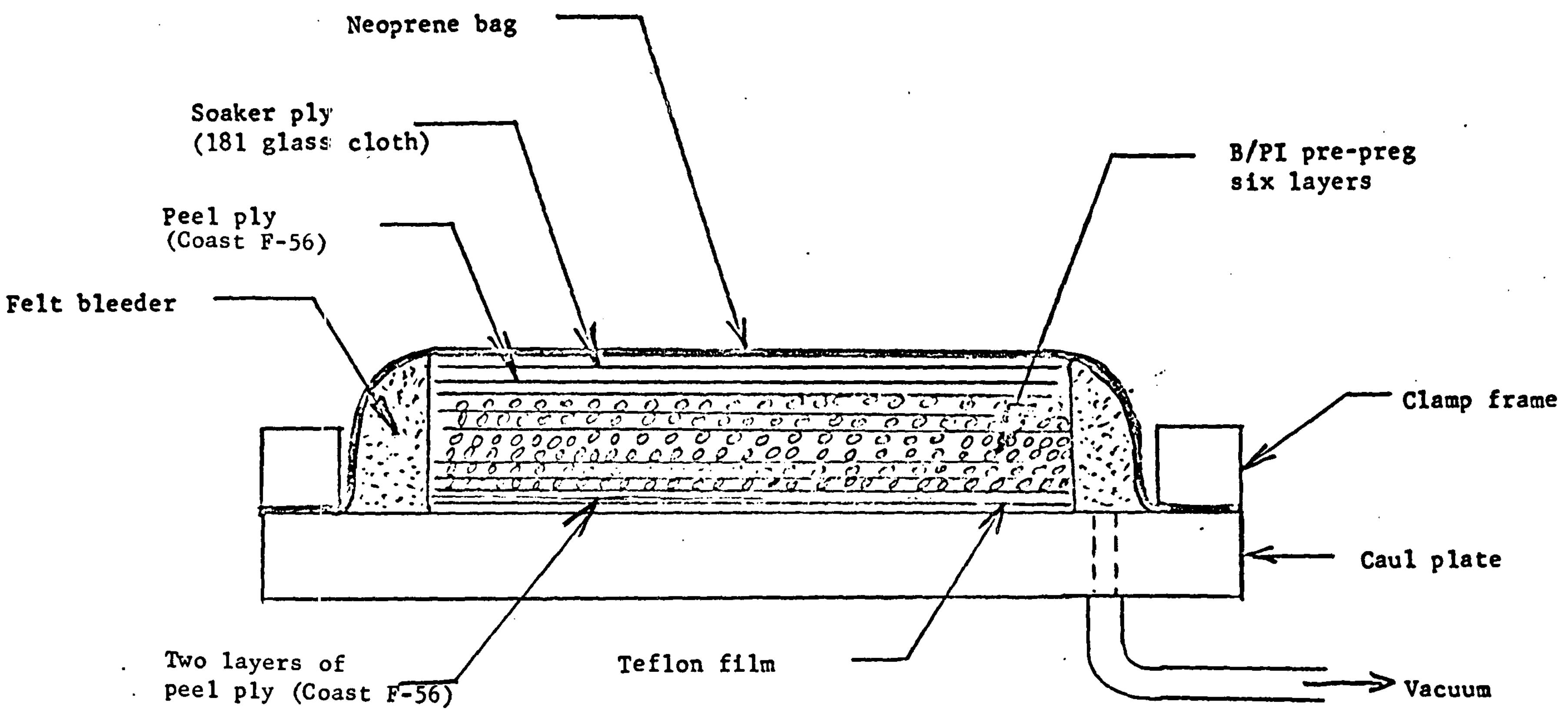


FIGURE 12. SCHEMATIC VACUUM BAG LAYUP

TABLE 5. EXPERIMENTAL CURE CYCLES FOR BORON/DUPONT 35-520

Specimen Number	Solvent Extraction Cycle	Cure Cycle (Oven Settings)	Average Ply Thickness, in.	Remarks
A-1	None	2 hr at 350 F	0.0075	Six plies, mottled
A-2	None	2 hr at 225 F 2 hr at 350 F	0.0062	Six plies, mottled
A-3	2 hr at 165 F 7.6 mm Hg	2 hr at 225 F 2 hr at 350 F	0.007	Three plies, mottled
A-4	1 hr at 160 F 7.6 mm Hg	1 hr at 220 F 2 hr at 350 F	0.0058	Six plies, mottled
A-5	1 hr at 160 F 7.6 mm Hg	30 min at 160 F 2 hr at 220 F 30 min at 280 F 30 min at 320 F 2 hr at 350 F	0.006	Six plies, mottled
A-6	None	Placed in RT oven 1 hr at 160 F 2 hr at 220 F 30 min at 280 F 2 hr at 350 F	0.0067	Six plies, mottled
A-7	None	20 min at 160 F 20 min at 200 F 60 min at 280 F 30 min at 320 F 120 min at 350 F	0.0052	Six plies, 1 percent mottled area
A-8	None	Same as A-7	0.0058	Six plies, 10 percent mottled area
A-9	None	20 min at 190 F 15 min at 240 F 60 min at 280 F 30 min at 320 F 120 min at 350 F	0.006	Six plies, 5 percent mottled area

TABLE 5 (Continued)

Specimen Number	Solvent Extraction Cycle	Cure Cycle (Oven Settings)	Average Ply Thickness, in.	Remarks
A-10	None	20 min at 140 F 20 min at 180 F 20 min at 220 F 60 min at 280 F 30 min at 320 F 120 min at 350 F	0.0058	Ten plies, 10 percent mottled area
A-11	None	20 min at 160 F 20 min at 200 F 20 min at 240 F 60 min at 310 F 30 min at 340 F 120 min at 370 F	0.0058	Ten plies, 10 percent mottled area
PI-12	None	105 min at 280 F 120 min at 350 F	--	Twelve plies of prepreg only (no boron), 100 percent mottled area
A-12	None	20 min at 170 F 20 min at 230 F 60 min at 290 F 30 min at 330 F 120 min at 360 F	0.0058	Ten plies, 1 layer of peel ply on caul plate side. No obvious mottling, lighter area may indicate subsurface porosity
A-13	1 hr at 160 F 0.1 atm pressure	20 min at 140 F 20 min at 180 F 20 min at 220 F 60 min at 280 F 30 min at 320 F 120 min at 350 F	0.0074	Ten plies, 1 peel ply on caul plate side. No mottling, but insufficent compaction and flow
A-14	None	Same as A-13	0.0057	Ten plies, 1 peel ply on caul plate side. Lighter area may indicate subsurface porosity
A-15	None	Same as A-12	0.0058	Ten plies, same as A-14
A-16	None	Same as A-12	0.0055	Twelve plies, 2 peel plies on caul plate side, no mottling, no lighter area

Table 5 lists the various cure cycles used in terms of oven settings. The cycle used for A-12 was found to yield the soundest laminates, especially when peel plies were used next to the caul plate to permit volatile extraction from both laminate surfaces. Since the actual resin temperature-time relationship is the critical cure parameter, curves for the range of best cure cycles have been derived from the thermocouple traces. The curves have been slightly simplified by approximating them with straight line segments, as shown in Figure 13. The A-12 and A-14 curves have been established as the recommended cycle limits. Basically, the recommended cycle consists of raising the laminate (resin) temperature to 235 F at a rate of 3.3 ± 0.1 F/minute, followed by an increase rate of 1.05 ± 0.05 F/minute to 340 F. At 3 0 F, the laminate is held for 90 minutes before allowing it to cool. The post-cure cycle is the typical one used for polyimides. Complete details of the recommended boron prepreg preparation, laminating procedure, cure cycle, and post-cure cycle are covered in Tables 6 and 7.

3.8 Test Data

A specimen cut from panel A-16 was mounted, polished, and examined metallographically. Attempts to use the Quantimet to determine void content failed due to the polishing "relief" which formed at the boron/polyimide interface. This prevented accurate discrimination by the Quantimet scanning beam due to reflective distortion. Consequently, a point count was made using a 64 intersection grid in the eyepiece of a metalloscope. This evaluation gave the following data for a 2560 intersection count:

Volumetric void fraction = 0.075

Volumetric boron fraction = 0.417

Volumetric resin fraction = 0.508
(including glass scrim)

Tensile tests were performed on 10 ply thick specimens, 5 in. long by 0.50 in. wide. Aluminum tabs 1-1/2 in. long by 1/2 in. wide by 1/16 in. thick were bonded to both sides of each end of the specimens to serve as gripping surfaces. Crosshead velocity was 0.02 in./minute.

Compression tests were performed on 0.1 in. by 0.1 in. by 0.5 in. long columns whose ends were ground perpendicular to the specimen axis. Due to the exceptionally good parallelism of the MTS test machine load surfaces, it was felt that special end preparation (such as bonded-on ball caps) would not be required on the specimens. Test results were inordinately low, however; this indicated the need of a better specimen design and loading system.

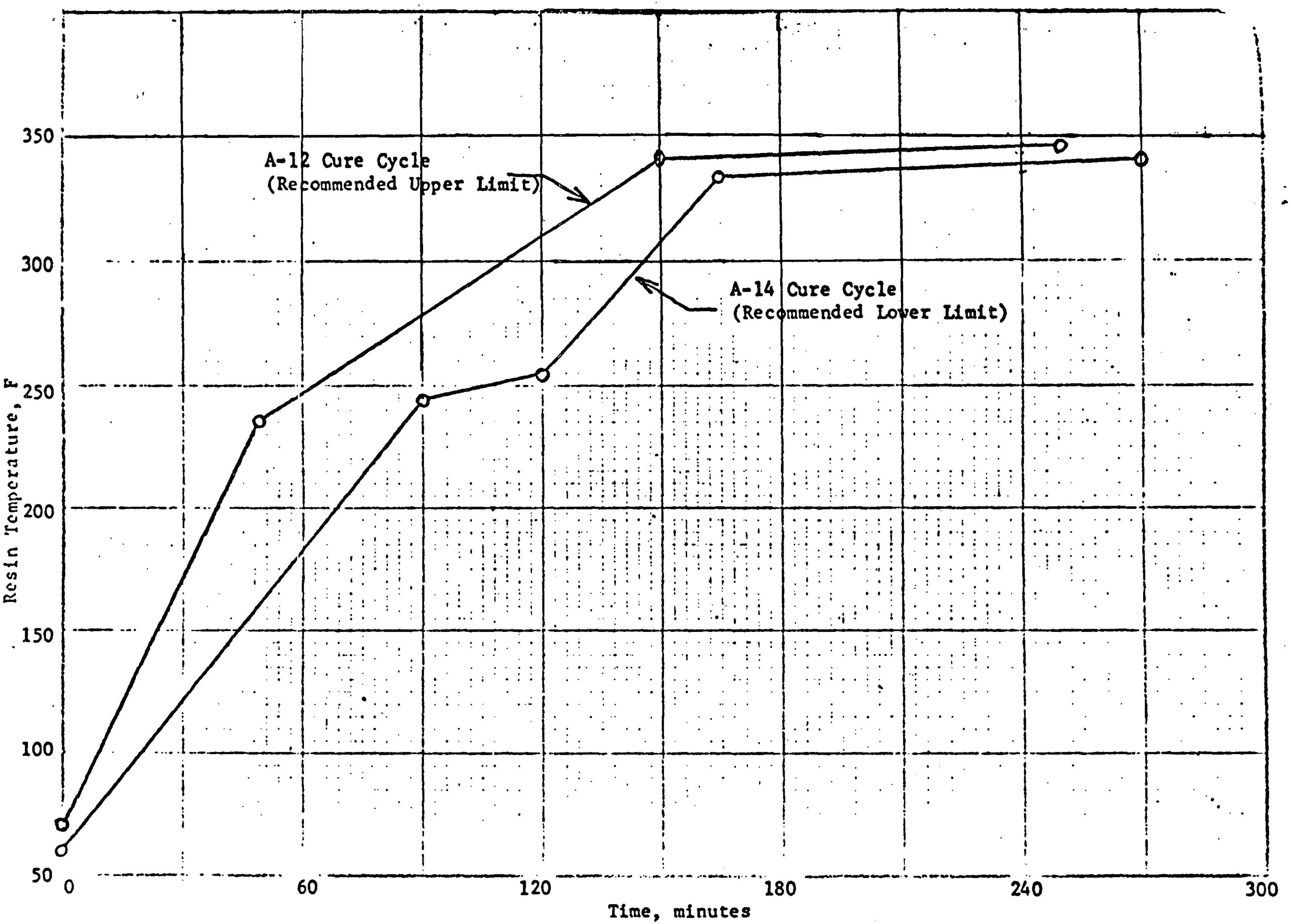


FIGURE 13. VACUUM BAG CURE CYCLES FOR B/35-520 LAMINATES

TABLE 6

PROPOSED PROCESS FOR PREPARATION OF B/PI PRE-PREG

I. Materials

A. Boron Filament - 0.004 in. diameter, W substrate, United Aircraft Corporation

B. Polyimide Prepreg - 35-520 Pyralin prepreg (DuPont)

Resin Content = 78 ± 4 percent (wt)
Volatiles = 33 ± 4 percent
Fabric - 104 Style, E-glass, A-1100 finish
Flow = 33 ± 4 percent (5 min at 450 F, 15 psi)

II. Filament Collimation

A. Clean surface of winding drum. Surface must be free of oil, grease, wax, dirt, resin, and any other foreign matter.

B. Diameter of any pulleys, guides, or tension reels for the boron filament should be 4 in. or more to avoid overstressing.

C. Set filament tension at 4 ounces maximum, measured at point of delivery to drum.

D. Set winding pitch at 0.005 in. (200 filaments per inch).

E. Maximum winding speed should be 100 ft/min linear velocity at drum surface.

III. Pre-Preg Application

A. Cut length of 35-520 pre-preg equal to circumference of winding drum.

B. Expose pre-preg to 100 percent relative humidity, 80 F to 90 F for 20 min or more. Pre-preg is ready for application to drum when resin is "gummy" and transfers to finger when touched. For best results, the humidity chamber should be somewhat foggy.

C. Tack end of pre-preg to surface of drum and wind pre-preg on drum, maintaining sufficient tension on free end to avoid wrinkles.

TABLE 6 (cont'd)

D. Using teflon doctor blades, press the pre-preg against the boron filaments firmly so that some resin (which still should be in the "gummy" stage) is extruded into the interstices between the filaments. Doctor blades pressure must not be high enough to drag the scrim cloth and create wrinkles.

E. Allow pre-preg to dry on drum at room ambient conditions (70 - 80 F, 30 - 80 percent RH) for 12 hours minimum before removal.

IV. Alternate Pre-Preg Preparation

A. Prior to winding boron filament on drum, wrap and fasten clean separator film (teflon or polyethylene) on drum surface. Separator film must fit drum snugly.

B. Wrap a single layer of 35-520 pre-preg over separator film and fasten. Pre-preg must be snugly fitted.

C. Using steam jets, soften resin in narrow band at end of drum where winding is to start. Wind boron filament on prepreg surface, advancing steam jets axially ahead of filament as winding progresses. Tension greater than 4 ounces may be necessary to imbed filament into softened resin.

D. Allow pre-preg to dry 12 hours before removal from drum.

TABLE 7

PROPOSED VACUUM-BAG LAMINATING PROCESS FOR B/PI

I. Lay-Up Procedure (10 to 12 ply laminate)

- A. Apply thin coat of release agent to caul plate.
- B. Place two layers of peel-ply (Coast Manufacturing F-56) on caul plate.

  Peel-ply must overlap vacuum ports.
- C. Build boron/polyimide laminate on top of peel-plies.
- D. Place bleeders (felt or rolled cloth) around edges of laminate, overlapping vacuum ports.
- E. Place two layers of peel-ply over top of laminate and extending over bleeders.
- F. Seal vacuum bag over caul plate.

  Note: For large laminates, it is desirable to have small vacuum holes in the caul plate or mold in the port area to assure adequate volatile removal from the bottom surface of the laminate. These holes may be spaced on a 6 in. grid pattern.

II. Cure Cycle

- A. Place bagged part in oven at room temperature.
- B. Apply vacuum (25 in. Hg minimum)
- C. Increase laminate temperature at a rate of 3.3 ± 0.1 F/min to 235 F.
- D. From 235 F to 340 F, increase laminate temperature at a rate of 1.05 ± 0.05 F/min.
- E. Hold at 340 F for 90 minutes.
- F. Reduce temperature to 150 F, maintaining vacuum.

III. Post-Cure Cycle

- A. Place in oven at room temperature.
- B. Raise oven temperature to 250 F and hold 1 hour.

TABLE 7 (cont'd)

C. Raise oven temperature to 400 F and hold 2 hours.

D. Raise oven temperature to 450 F and hold 2 hours.

E. Raise oven temperature to 500 F and hold 4 hours.

F. Raise oven temperature to 550 F and hold 4 hours.

G. Raise oven temperature to 600 F and hold 4 hours.

H. Raise oven temperature to 650 F and hold 2 hours.

I. Cool slowly in oven to 200 F before removal.

Note: It may be necessary to restrain part during post cure to avoid warpage.

Interlaminar (horizontal) shear strength was measured by short beam shear tests. The specimens were 12 plies thick (0.070 in.) by 0.6 in. long by 0.25 in. wide. The specimens were tested on a 0.4 in. span (5.7:1 span/depth ratio), with load and reaction supports having 1/8 in. radii.

All specimens were preconditioned for 48 hr. at 70 F, 30 percent RH. Elevated temperature tests were performed at 500 F, after the specimen was soaked for 1/2 hr. at 500 F. The results are shown in Table 8.

Results of the compression tests are not included in Table 8 due to premature specimen failures. The average ultimate compressive strengths at 70 F and 500 F were 59.3 and 50.1 ksi respectively. These values were about one-third the expected values. Examination of the specimens showed a tendency toward uneven crushing at the ends, often precipitating splitting of the specimen. Tests performed at North American Rockwell on the same material but using a different specimen design gave values in the 150 ksi range.

Two of the tensile specimens tested at 70 F showed marked primary and secondary moduli. The primary modulus of one specimen was 33.6 by 10^3 ksi to 0.12 percent strain (maximum strain was 0.62 percent). The other specimen had a primary modulus of 38.3 by 10^3 ksi to 0.13 percent strain (maximum strain was 0.52 percent). These specimens were cut from the same panel as two others which showed no primary-secondary moduli.

Short beam shear specimens tested at 70 F showed some flexural failures. The values reported for these specimens would be conservative. The remaining specimens had obvious horizontal shear failures. Judging by the very small coefficient of variation (4.6 percent), the specimens which failed in flexure must have also been close to shear failure. At 500 F, there were no actual failures of the specimens. In all tests at 500 F, the specimens deflected until compressive interference between the loading nose, supports, and the specimen occurred. This point was indicated by a sudden load increase occurring after a "plateau" indicating flexural creep. Strength values were calculated by using the highest load recorded prior to the sudden increase caused by compressive load transfer through the loading nose and supports.

TABLE 8. MECHANICAL PROPERTIES OF UNIDIRECTIONAL B/PI LAMINATE*

	70 F	500 F
Tension		
Ultimate (average), ksi	165.2	86.6
Standard deviation, ksi	21.4	6.47
Coefficient of variation	0.13	0.075
Modulus of elasticity, 10^3 psi	25.8	--
Strain at failure, percent	0.62	--
Interlaminar shear		
Ultimate (average), ksi	5.52	4.43
Standard deviation, ksi	0.25	0.59
Coefficient of variation	0.046	0.135

*Volumetric B fraction = 0.417
Volumetric void fraction = 0.075

3.9 Summary - Battelle Sub Contract

Vacuum bag processing of boron/PI using DuPont's Pyralin 35-520 yields satisfactory laminates when controlled rate temperature increases are used during cure. The temperature of the resin should be increased at a rate of 3.3 F/minute from room temperature to 235 F. At this point, the temperature increase rate should be reduced to 1.05 F/minute until a cure temperature of 340 F is reached. The laminate should be held at 340 F for 90 minutes to complete the cure. Complete details for pre-pregging, curing, and post-curing are given in Tables 6 and 7.

An important factor to remember is that sufficient effusion of volatiles from the laminate is a key factor in obtaining sound laminates, particularly where available pressure is low (as in the vacuum bag process). Volatile removal was enhanced by interposing bleeder plies between the caul plate and the laminate, as well as between the laminate and the bag. This technique worked well for laminates up to 20 plies thick.

Measurement of dissipation factor during cure is a feasible method for establishing in-process control. Difficulty was encountered when this technique was attempted on boron containing laminates, but it is believed that improved instrumentation can be devised to give the required sensitivity. Continuation of dissipation factor studies is strongly recommended.

3.10 Process Development Studies

3.11 Cure Cycle Development - Battelle Memorial Material

Approximately 22 sq. ft. of polyimide prepregged boron was supplied under the contract but Battelle indicated they were not interested in processing the material for the entire program. After a review of the recommended cure cycle, it was believed that one more applicable to production should be investigated. Two cure cycles based on previous experience were evaluated along with the Battelle Procedure as a control. The specific process for each laminate was as follows:

General: All panels were laid up flat on an aluminum caul plate which had the surface prepared with Frekote 33. Two bleeder plies (181 fabric) were placed on the plate and covered with an 0.001 inch porous teflon coated fabric. The three panels of boron polyimide prepreg (18 plies) were laid up unidirectionally and covered with the teflon coated fabric and two additional bleeder plies. A peripheral bleeder (181 fabric) was laid up to the thickness of the part and 2 inches wide around the laminate. This part was then covered with nylon, high temperature film, sealed and a vacuum drawn. The cure and post cure cycles for each panel are as follows

Panel No. 1 (Battelle Process)

Cure

a. place bagged part in oven at room temperature.
b. apply vacuum (25 in. Hg min.)
c. increase laminate temperature at a rate of three degrees/minute to 235°F.
d. increase laminate temperature to 340°F at one degree/min.
e. hold at 340°F for 90 minutes.
f. reduce temperature to 150°F while maintaining pressure.
g. remove from oven and de-bag.

Post Cure

a. place in oven at room temperature.
b. raise oven temperature to 250°F and hold 1 hour.
c. raise oven temperature to 400°F and hold 2 hours.
d. raise oven temperature to 450°F and hold 2 hours.
e. raise oven temperature to 500°F and hold 4 hours.
f. raise oven temperature to 550°F and hold 4 hours.
g. raise oven temperature to 600°F and hold 4 hours.
h. raise oven temperature to 650°F and hold 2 hours.
i. cool slowly in oven to 200°F before removal.

Panel No. 2

Cure

a. place bagged part in oven at room temperature.
b. apply vacuum (25 inches Hg min.)
c. increase temperature to 240°F in 45 minutes (approx. 3 1/3 degrees/min)
d. hold for 120 minutes
e. increase temperature to 320 in 180 minutes (0.4 to 0.5 degrees/min.)
f. continue increasing temperature to 340°F in 30 minutes.
g. hold 340°F for 90 minutes
h. reduce temperature slowly to 150°F while maintaining pressure.
i. remove from oven and de-bag.

Post Cure

a. place in oven at room temperature.
b. raise oven temperature to 350°F in 35 ±5 min. and hold for 40 minutes.
c. raise temperature to 500°F in 40 ± 10 min. and hold for 2½ ± ½ hours.
d. increase temperature to 600°F in 15 minutes and hold for 4 ± ½ hours.
e. reduce temperature to 150°F, under pressure, at a rate not to exceed 10 degrees/minute.

Panel No.3

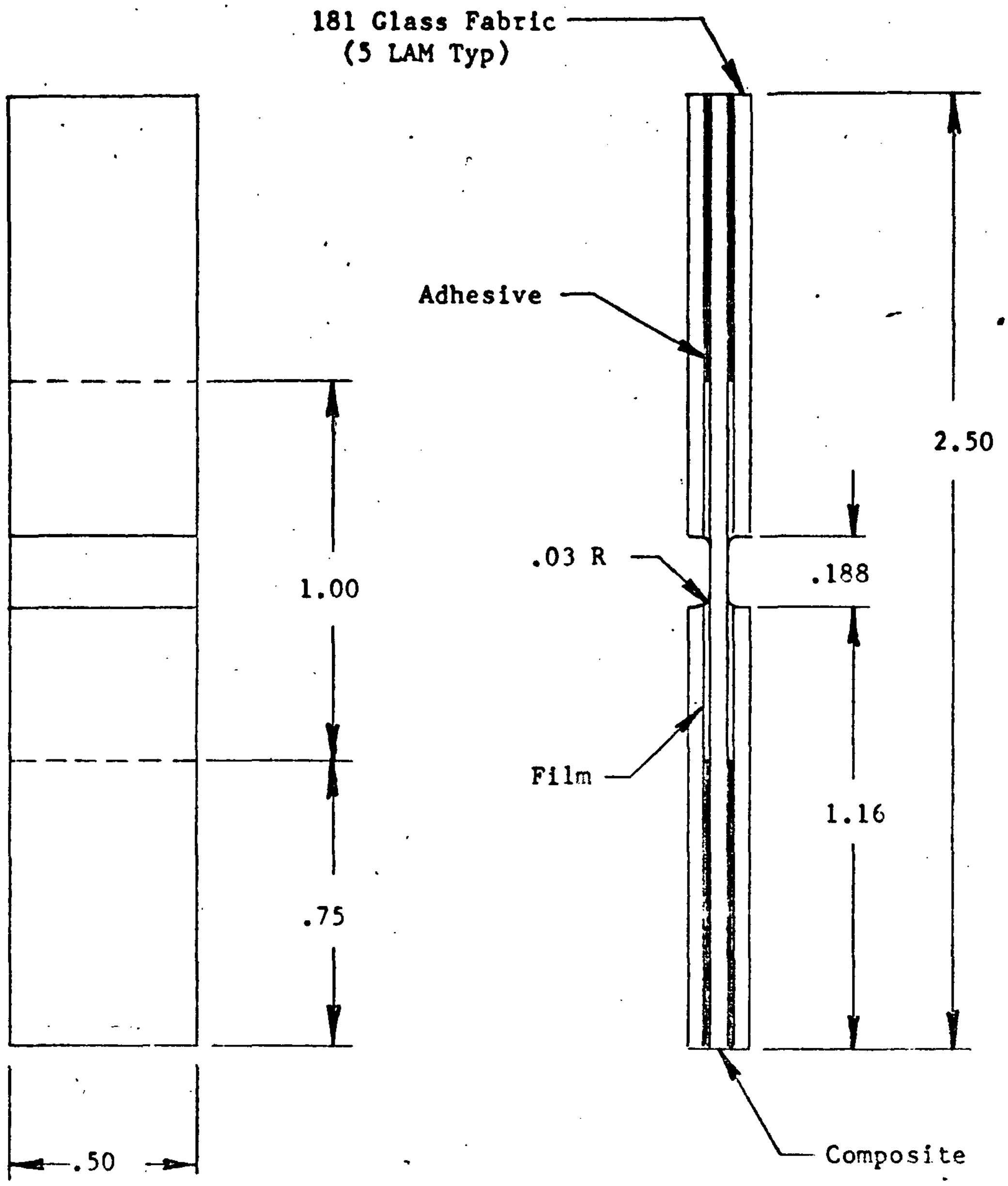
Cure

a. place bagged part in oven at room temperature.
b. apply vacuum (25 in. Hg min.)
c. heat to 260°F in 3 hours (approximately 1 degree/min) and hold for three hours.
d. increase the temperature to 310°F in approx. one hour and hold for one hour.
e. increase the temperature to 350°F and hold for 90 minutes.
f. reduce temperature to 150°F while maintaining pressure.
g. remove part from oven and de-bag.

Post Cure

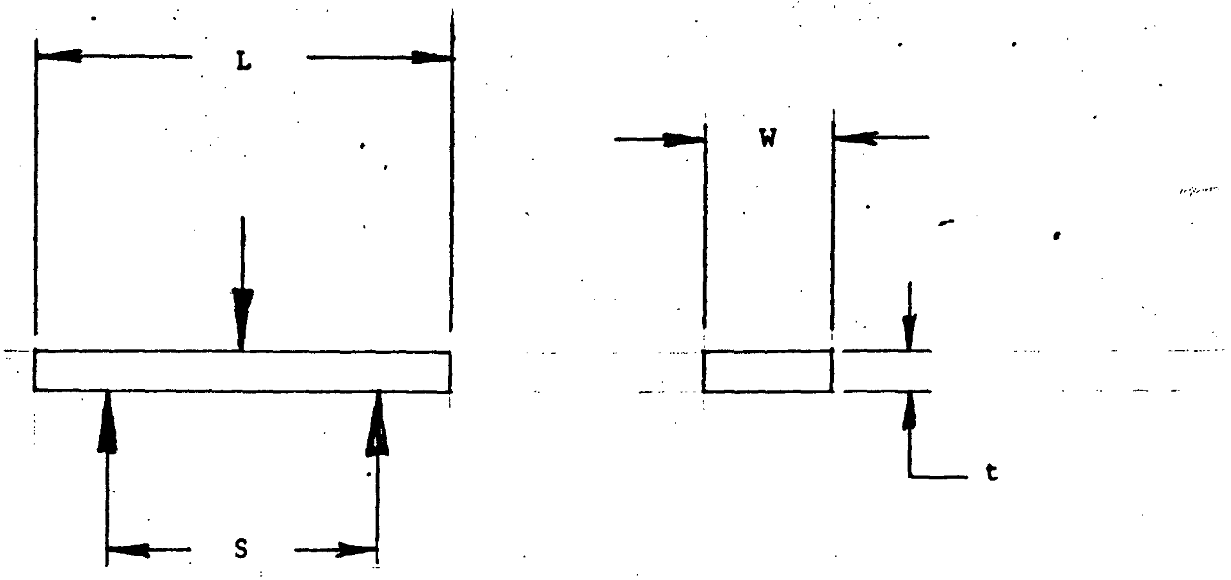
The post cure was identical to Panel No. 1.

The resulting panels were tested in compression and short (horizontal) beam shear at room temperature and at 500 F after 1/2 hour at 500 F on specimens fabricated in accordance with Figures 14 and 15. It should be noted that panels 1 and 2 were fabricated from boron prepared with the first batch of 35-520 polyimide impregnated fabric. Panel No. 3 used a second winding using the same batch of boron but another shipment of 35-520 material. The initial shipment was dry with virtually no tack while the second had considerably greater, but no excessive, tack. The results obtained on the three panels are shown in Table 9.



Composite thickness shall be 0.050 ± .005 inches for unidirectional material or for oriented material, multiple sequences to achieve 0.050 min.

Figure 14 The Standard Composite Compression Test Specimen



Specimen Dimensions

Length (L) 0.60 ± 0.01
Width (W) = 0.250 ± 0.003
Thickness (t) = Note (1)
Span (S) = Note (2)

Load and reaction supports shall be 1/8" radius steel rods filament direction shall be as specified.

Notes (1) Thickness$^{(t)}$ shall be 0.050 inches minimum or if oriented multiple sequences to achieve a thickness greater than 0.050 inches.

(2) Span$^{(S)}$ shall be calculated as follows:

The span (s) - depth (t) ratio shall be

8 to 1 for glass
5 to 1 for boron
4 to 1 for graphite

Figure 15 Horizontal Shear Test Specimen

TABLE 9. RESULTS-CURE CYCLE VARIATIONS

	Compression		Horizontal Shear	
	R.T.	At 500 F	R.T.	At 500 F
Panel #1 (Control)				
Max.	170,393	107,339	8,172	6,944
Min.	159,516	78,573	6,499	6,236
Ave.	164,073	94,857	7,586	6,509
Panel #2				
Max.	182,547	99,081	11,888	7,180
Min.	146,068	77,653	10,052	6,591
Ave.	166,181	88,553	10,821	6,870
Panel #3				
Max.	144,546	103,647	4,397	5,174
Min.	117,415	73,647	2,281	3,580
Ave.	136,574	89,030	3,605	4,353

Reviewing the data, presented in Table 9, on the effects of the three different cure cycles revealed that the panel #2 technique was better than the control in compression at room temperature and in horizontal shear at both room and elevated temperature. The same was true of the comparison between panels #2 and #3. Since panel #2 was considerably better than either of the other panels, it was agreed that the cure cycle of panel #2 would be used for future work. It was believed that the lower, elevated temperature compression results was a function of the post cure cycle and could be corrected.

3.12 Development of Source for Production Quantities of Prepreg

With Battelle not interested in supplying the prepregged boron to complete the program, other organizations were contacted concerning their interest in supplying material processed in accordance with the Battelle procedure. It was learned that only limited experience with polyimide was available from any source except with the TRW, P13N resin. Contacts were made to costs and representative sample quantities to assure quality material using the 35-520 material. Two organizations, R&D Division of Whittaker Corporation and Hercules, agreed to process one pound of filament for $200 when the boron was supplied to them. Hercules had second thoughts and increased the price by $1600 which they felt would be needed for a development phase. They did, however, suggest an alternative: they would give, free, a one pound sample of P13N impregnated material in order to be considered as a material source for the program. This offer was rejected until data were available to assure that the product had temperature resistance equal to or better than the Pyralin 4707. These data were never received. Whittaker requested that consideration be given to use of a low boiling solvent such as methyl ethyl Ketone (MEK) instead of the live steam of the Battelle process. One pound of boron filament was supplied to Whittaker for conversion to prepreg using the alternate solvent. The material was received, a panel fabricated and the following results obtained.

	Compression		Horizontal Shear	
	73 F	500 F(1)	73 F	500 F(1)
Max.	216,736	134,381	10,467	6,225
Min.	174,470	115,102	9,750	5,857
Ave.	186,828	124,808	10,100	6,059

(1) Tested at 500 after 1/2 hour at 500 F.

The results were considerably better than those obtained with the steam activated material and the visual examination of the broad goods showed greater uniformity. On this basis, the use of the low boiling solvent (MEK) was considered acceptable and the prepreg for the program was purchased from Whittaker with this deviation from the original requirements.

3.13 Lay-Up Technique Evaluation

It had been found under previous development work that a surface bleeder vacuum system produced composites with higher properties than when a peripheral system was used. A panel was made from part of the second shipment of Whittaker material using the 3M system, the schematic of which is shown in Figure 16. The properties obtained with this system are as follows.

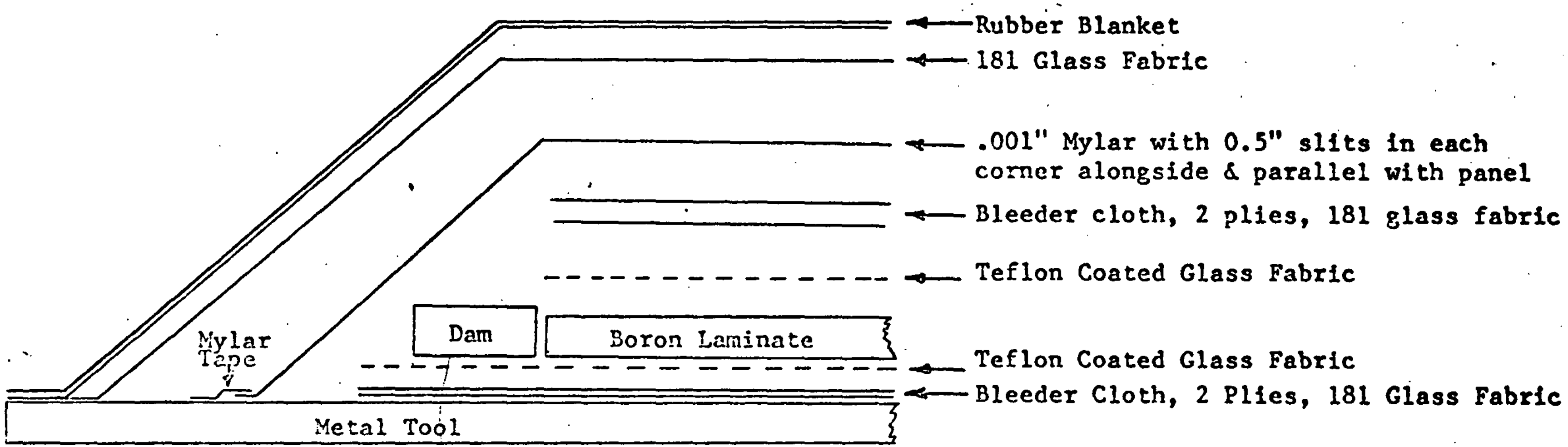
	Compression		Horizontal Shear	
	73 F	500 F	73 F	500 F
Max.	211,221	153,710	12,122	6,284
Min.	193,618	148,720	11,302	5,967
Ave.	200,560	151,215	11,591	6,128

Comparing these data with those previously obtained, the surface bleeder results in not only higher property values but also more uniform results.

3.14 Process Reproducibility

Based on the effort previously discussed, it was established that the reproducibility panels would be fabricated using the surface bleeder system and the cure cycle used for Panel #2, paragraph 4.3. The first reproducibility panel was fabricated from material from shipment #2 and had the good, glassy, dark brown appearance associated with satisfactory panels. The second panel was made by the same technician using some material of shipment #2 but mostly shipment #3. This panel had a light tan, powdery looking surface indicating an unsatisfactory panel. Attempts were made to modify the curing technique to one more forgiving of the prepreg variations. Five small panels (4" x 4") were made using Sheet 56, Shipment #5, Lot 0016. The fabrication variables and the test results were as follows:

FIGURE 16 VACUUM BAG LAYUP PROCESS



Panel	Description	Shear Results(1)
a	Standard cure (control)	2893 psi
b	Standard cure - 25 psi augmented pressure	(2)
c	Cure cycle same as (a) except 4 hr. dwell (instead of 2) at 250°F plus 16 hours at 350°F instead of 4 hours (Vacuum lost after 1 hour at 350°F)	3117 psi
d	Repeat of (c)	1959 psi
e	Same as (a) except long (17 hours) dwell at 250°F	2914 psi

Notes: (1) Average of 5 specimens
(2) Shear too low to permit machining

These results indicate that variations of dwell and cure times will not remove the solvent and result in the high strength, low void content composite which is desired. Another panel was made from the same sheet but using the original bag procedure developed by Battelle and the same cure cycle as the control. The results on this panel were comparable to those obtained on the previous panels (1926 psi average).

Another sheet of material (#55, Shipment #5, Lot 0016) which had higher tack was investigated to see if minor variations would have any affect. A control panel and a panel using a 265°F dwell for four hours and an overnight cure (17 hours) resulted in interlaminar shear strengths of 2251 and 4308 psi respectively. Again, these results were not adequate for component fabrication.

At this point it was concluded that the prepreg contained an excessive amount of high boiling solvent which could not be removed by the cure cycle only. Contact was made with the supplier of the production material (Whittaker R&D) to determine exactly what process they had used for the preparation of the production material vs. that used for the initial qualification material. Whittaker's detail manufacturing process specification "B-10123P" is as follows:

Scope

The purpose of this specification is to outline the critical parameters involved in the manufacture of a composite prepreg from 4 mil diameter boron filament and Du Pont polyimide prepreg 35-520.

Equipment Required

Use the 12" wide x 10' circumference or 18" wide x 15' circumference rotary drums equipped with multi-spool fiber feed.

Composite Prepreg Manufacture

1. Cover drum with 0.003" thick Mylar film, attaching securely in place with masking tape to prevent slippage.

2. Cut Du Pont 35-520 prepreg to required dimensions. Do not exceed width of drum.

3. Using masking tape, attach prepreg to Mylar-covered drum with the heavy resin surface of the prepreg on the outside.

4. Spray prepreg with a light mist of n-methylpyrrolidone (NMP) in the immediate area being wound with boron fiber. Avoid excessive use of NMP. Prepreg surface should be just wet enough that the fiber will be embedded securely in the resin.

5. Set winding equipment for correct fiber count. Attach fibers at the drum edge and begin winding at a constant rate of speed, maintaining the required tension on the fiber as specified on the drum chart.

6. Progressively moisten prepreg with NMP mist just ahead of the fiber winding. Avoid excessive wetting, using only enough NMP to wet the surface of the polyimide resin and allow the fiber to become firmly embedded in the resin.

7. When entire drum is wound, apply moderate pressure to fiber surface as the drum rotates to assure close contact between fibers and resin.

8. Cut composite prepreg sheet free from drum and cover with a film of 0.0005" Mylar. Place on 200°F heated table surface and seal vacuum cover sheet to surface of heated table.

9. Pull vacuum pressure to a minimum of 20" mercury.

10. During this compacting step make certain that the fibers are completely wet by the resin.

11. After 10 minutes treatment on the heated table under vacuum, remove composite prepreg and place on flat surface to cool.

12. After cooling, strip off 0.0005" Mylar film and cover with the specified WRD release paper.

13. Invert the prepreg and remove the 0.003" Mylar using dry ice treatment and re-cover with additional WRD release paper.

14. Bag in polyurethylene bags.

15. Store in refrigerator at -40°F. Ship in specified dry ice protected containers.

The specification indicates that the production material was processed using n-methylpyrrolidone (NMP). This has resulted in the excess high boiling solvent. In order to save the material, methods other than cure cycle or pressure were investigated. It was known that the NMP was soluble in water and, on reviewing the work done by Battelle on this program, that it formed an azeotrope with water having a boiling point lower than either component.

It was also known that Dr. L. Maus of the Tulsa Division, North American Rockwell, had submitted a patent declaration on how to remove NMP and other high boiling, water soluble, solvents from polyimide resins. Applying this information to the process development resulted in the following modifications being made from the original process to try to achieve the desired goals. Again using the material from sheet 55, Shipment 5 and Lot #0016, the following changes were made.

1) Each ply soaked in water for 3 minutes and oven dried 10 minutes at 150°F. A box coat of acetone was sprayed on the individual plies to tacify the material. The short beam shear strength after standard lay-up and cure was 5419 psi.

2) Each ply was soaked in water for three minutes and dried for 10 minutes. This cycle was repeated 3 times. When cured as before, no resin flow occurred. The laminate was extremely thick with insufficient integrity to machine.

3) Each ply soaked in water 15 minutes and allowed to air dry overnight at room temperature. Each ply was sprayed with MEK, laid-up and processed by the standard technique. The laminate was not worth testing.

Based on these data and all previous effort, it was concluded that the water was a contributor in reducing the volatiles or void content as well as increasing the flow. A second series were fabricated using Sheet 54, Shipment 5, Lot 0816. These included a new control and four other laminates where the only variation was the length of time for the water soak. The procedure was as follows:

Soak the material in water for the specified time.
Blot with a clean dry paper towel.
Immediately lay-up and cure the panel by the standard technique.

The results were as follows:

Soak Time	Laminate Thickness	Shear Strength (psi, ave.)
Control	.093	3717
2 min.	.090	5456
4 min.	.091	9690
6 min.	.096	16,448
8 min.	.113	12,683

Reviewing the thickness and strength in relation to previous work revealed that the resin content of the two and four minute cycles were too low for optimum boron composite. This would be due to the plasticizing effects of the azeotrope (NMP-H_2O mixture). As the length of time in the water goes up less azeotrope is formed because less NMP is available for its formation. This results from the leaching action of the water where the NMP is water soluble. Another panel was made duplicating the 8 minute laminate but leaving more water on the surface to see if flow would be increased. This panel showed a thickness increase (to .120 inches) and a 20 percent loss in strength to 10,373 psi. These results show that the six minute soak with a blot dry resulted in material which produced composites with the highest shear strengths obtained under this program. An attempt to reproduce these values under the same conditions resulted in a value of only 5773 psi compared to the 16,448 psi.

A thorough review was made of the handling of the material during the entire operation. It was established that all material, for the previous studies except the unblotted 8 minute soak and the last panel made, were soaked in a limited quantity of water which was not changed during the processing of sufficient material for four panels. This resulted in a reduction in the amount of NMP extracted due to its limited solubility in water. The second variation between the two panels was that the first panel was made of one batch of material where the last panel was made of five different batches. A review of the data obtained on the two panels reveals that the last panel had inadequate flow and probably poorer wetting than was obtained with the first panel. Based on this information, two courses were open for property improvement. One was to extract the NMP for the same length of time and increase the flow by the addition of a low boiling solvent or, two, to reduce the extraction (soak) time. Both approaches were tried. Five additional panels were fabricated using the six minute soak and one panel using a four minute soak but all with constantly changing water.

The fabrication variations of the six minute water soak material involved:

a) blot dry
b) oven dry-butyl alcohol spray
c) blot dry-butyl alcohol spray
d) blot dry-50 psi augmented pressure
e) blot dry-methyl isobutyl ketone spray

The blot dry process consisted of laying the soaked material on a white paper towel and blotting the exposed surface to remove the water droplets. The solvent spray (butyl alcohol or methyl isobutyl ketone) consisted of a light box coating of solvent which was allowed to stand for forty-five minutes prior to lay-up. Vacuum bag pressure was used for all panels except as noted above. The four minute soak was made identically to (a) above except the time in the water was reduced by two minutes.

The results of these tests along with some comparative data previously presented are shown in Table 10. These data show that the blot dry operation is somewhat better than the air dry but that the forty-five minute soak with butyl alcohol had little effect. The soak in the less volatile MIBK did result in shear strengths greater than the alcohol but somewhat less than the four minute water soak. The four minute soak, in constantly changing water, resulted in a laminate thickness (0.098 inches) more nearly that which would be considered optimum (0.095) for the number of laminations used.

All previous work on the evaluation of the immersion technique was accomplished immediately after immersion, it was, therefore, considered advisable to investigate the effects of immersion plus a so-called open assembly time to be more consistent with production operations. A series of three panels were fabricated to establish the production technique. The variables and the results are as follows:

Technique	Shear Strength (psi)
1) Four minute soak in cold running water, blot dry, set over night, lay-up bag and cure after 16 hours	11,137
2) Four minute soak in cold running water, blot dry, MIBK box coat spray, set over night. Complete as item 1.	8,923
3) Four minute soak in cold running water, blot dry, MIBK spray. Complete as before	9,638

Table 10

Shear Strength Results From Various Processing Techniques

Process Description	Thickness (Inches)	Shear Strength (psi) (2)
<u>Sheet 54</u>		
4 minute soak - water not changed - blot dry	.091	9,690
6 minute soak - water not changed - blot dry	.096	16,448
8 minute soak - water not changed - blot dry	.113	12,683
8 minute soak - water changed - material laid up wet	.120	10,373
<u>Sheets 49, 50, 51, 52, and 53</u>		
Water changed each laminate		
6 minute - blot dry	.117	5,773
6 minute - blot dry	.122	8,666
6 minute - oven dry - butyl alcohol spray	.126	7,332
6 minute - blot dry - butyl alcohol spray	.117	8,732
6 minute - blot dry - 50 psi augmented pressure	.091	(1)
6 minute - blot dry - methyl isobutyl alcohol spray	.103	11,816
4 minute - blot dry	.098	13,863

Notes (1) Panel contained large areas of light (voided) color and extremely low resin content.

(2) Typical Boron-Epoxy shear strength is 8 - 10,000 psi.

The value of technique #1 (11,137 psi) compares favorably with the four minute soak data (13,863 psi).

All subsequent panels will be fabricated using the four minute soak technique previously described.

3.15 Panel Fabrication and Data Generation

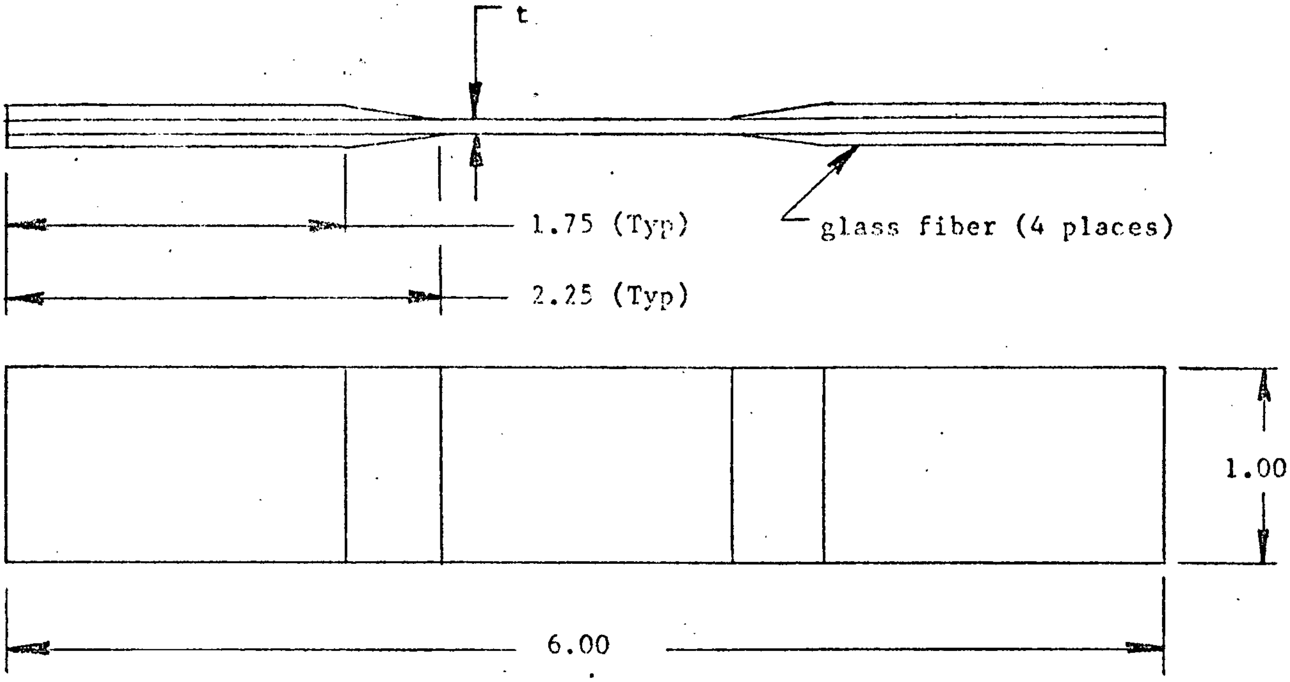
Using the techniques previously presented, five panels were fabricated to establish reproducibility. The five panels were fabricated by three different technicians with no more than two made by any one individual. These panels consisted of 20 plies of boron prepreg processed by the water immersion technique, oriented in accordance with the optimum orientation established for the specific loadings for the box beam, laid up in accordance with the processes described in paragraph 4.3 and cured per paragraph 4.4.

The optimum thickness for the 20 ply laminate is established as 0.106 inches. Four of the five panels varied from 0.107 to 0.111 inches while the fifth panel measured 0.111 ±003 inches. The latter panel was the second fabricated by one of the technicians who made two panels. The processed prepreg for this latter panel laid from Friday to Monday when the panel was laid up and cured. This indicated that more of the NMP-H_2O azeotrope evaporated in a 64 hour period then was experienced, under the test program, in a 24 hour period. Thus, if the processed material is to lay for more than one day, the time of water immersion must be shortened or the processed material must be sealed in a moisture proof bag.

Eight specimens of each configuration (tension, compression and short beam shear, Figures 14, 15 and 17 respectively) were machined for each panel. One specimen of each configuration, from each panel, was randomly selected for test at the specified conditions. These conditions were at room temperature for control and at the exposure temperature after 1/2, 8 or 16 hours exposure to that temperature. Temperatures were 550, 600, 650, 700, 750 and 800 degrees Fahrenheit.

The high, low and average results of the tensile tests are presented in Table 11 and are presented graphically in Figures 18, 19, 20 and 21. The compression results are similarly presented in Table 12 and plotted in Figures 22, 23, 24 and 25. The short beam shear data are presented in Table 13 and Figures 26, 27, 28 and 29.

These data indicated that the resin system was satisfactory for up to 16 hour exposure at 700 F since these values were at least 2/3 of those obtained at room temperature; the post cure used for these panels was inadequate in that the short time (1/2 hour) exposure data were considerably lower than the longer time exposures, where continuing curing occurred. This was true of all temperatures up to 750 F where deterioration of the resin due to temperature started to exceeded the improvements obtained by the increased cure time and temperature. Even at 750 F, the eight (8) hour data showed improvement over the 1/2 hour data. At 800 F, all results showed a decrease with increasing time at temperature.



t = 0.050 ± 0.005 for unidirectional material or, if oriented, multiple sequences to achieve a thickness greater than 0.050 inches.

Figure 17 Tensile Test Specimen

Table 11

Ultimate Tensile Strength - Reproducibility Panels

Condition	High	Low	Ave.
Room Temp.	61,900	47,357	54,200
* At 550 after			
1/2 hour	50,190	47,003	48,500
8 hours	52,719	44,984	49,300
16 hours	53,247	41,127	46,400
At 600 after			
1/2 hour	50,380	43,545	46,100
8 hours	46,799	41,236	44,300
16 hours	44,275	37,666	41,100
At 650 after			
1/2 hour	34,476	34,203	34,342
8 hours	44,705	38,030	40,400
16 hours	43,940	39,894	42,200
At 700 after			
1/2 hour	39,770	27,364	32,400
8 hours	39,926	34,664	37,100
16 hours	46,086	38,545	41,300
At 750 after			
1/2 hour	33,784	30,536	32,200
8 hours	38,276	36,640	37,400
16 hours	28,594	23,137	25,400
At 800 after			
1/2 hour	31,990	27,412	29,000
8 hours	25,575	23,133	24,200
16 hours	20,000	17,995	18,900

* Tested at stated temperature after the designated period of time at the stated temperature.

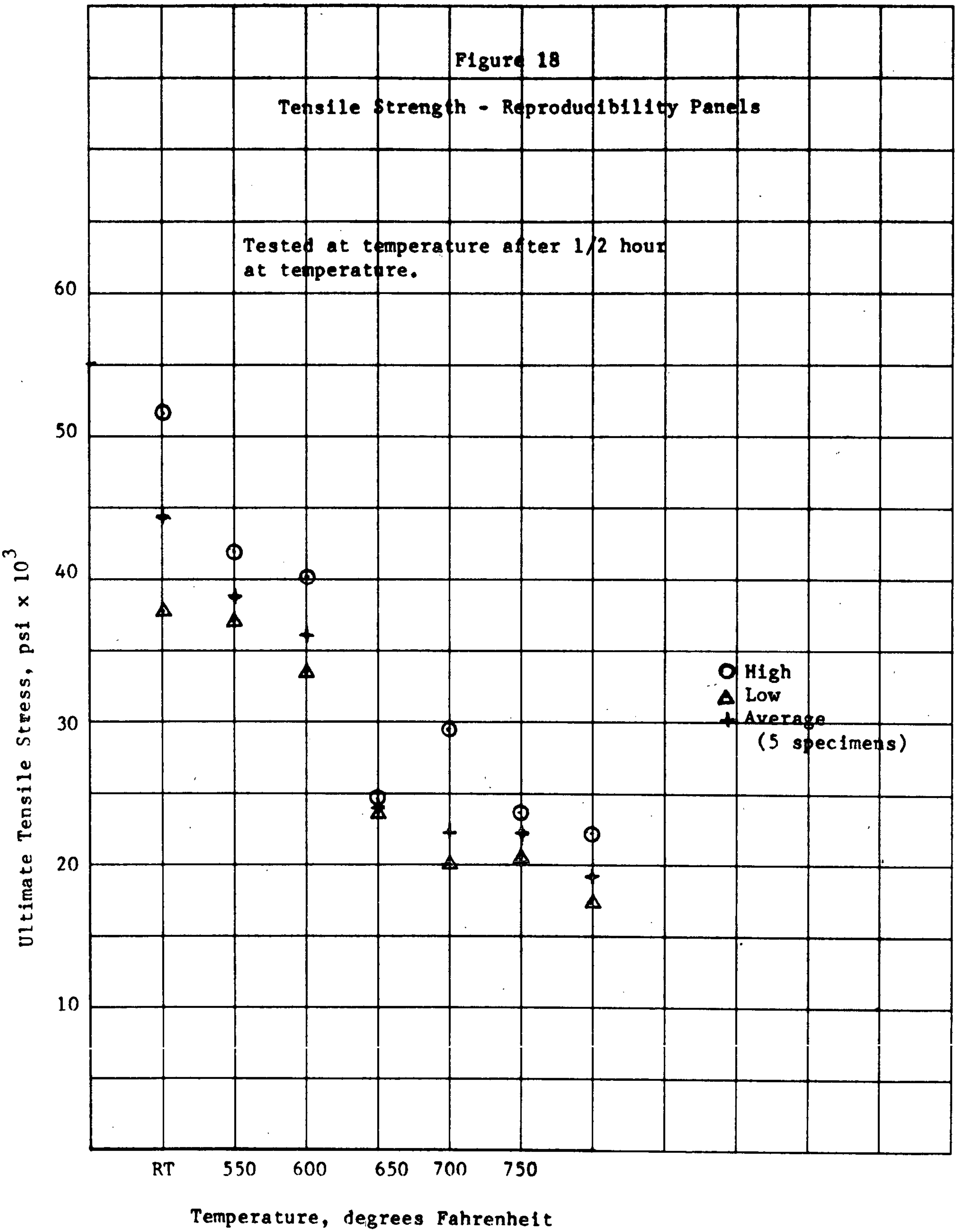


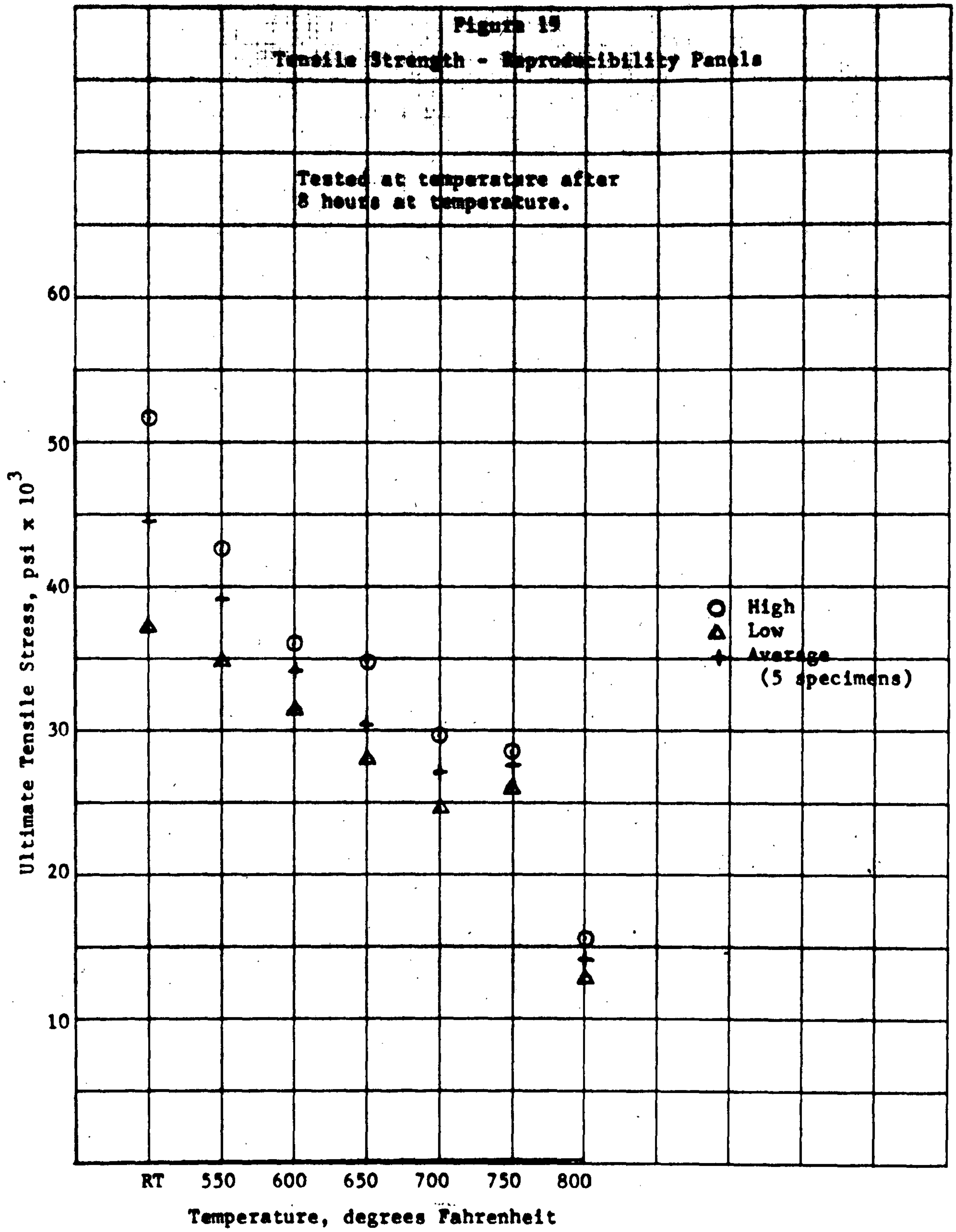
Figure 18

Tensile Strength - Reproducibility Panels

Tested at temperature after 1/2 hour at temperature.

Figure 15

Tensile Strength - Reproducibility Panels



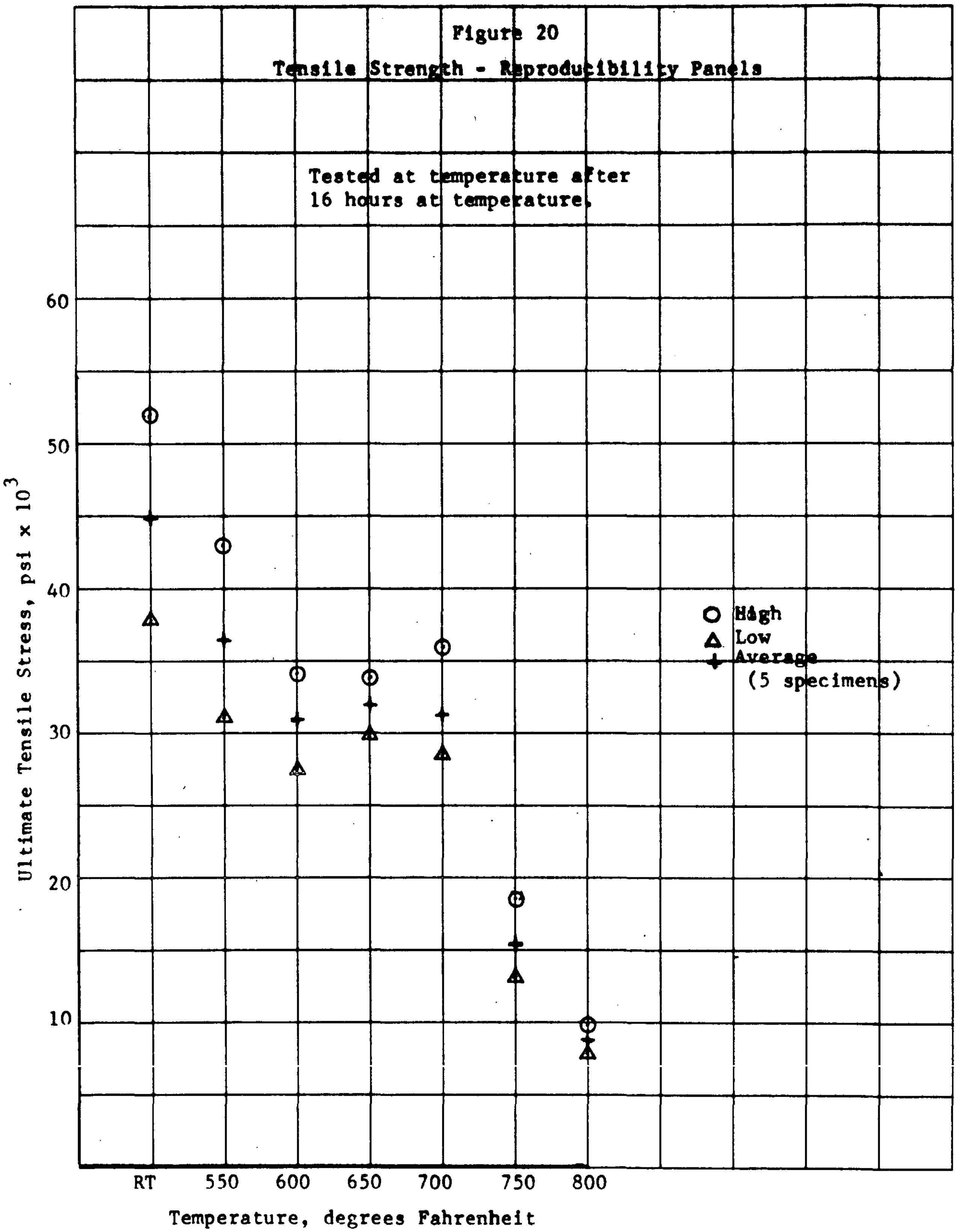


Figure 20

Tensile Strength - Reproducibility Panels

Figure 21
Tensile Strength (Average) - Reproducibility Panels

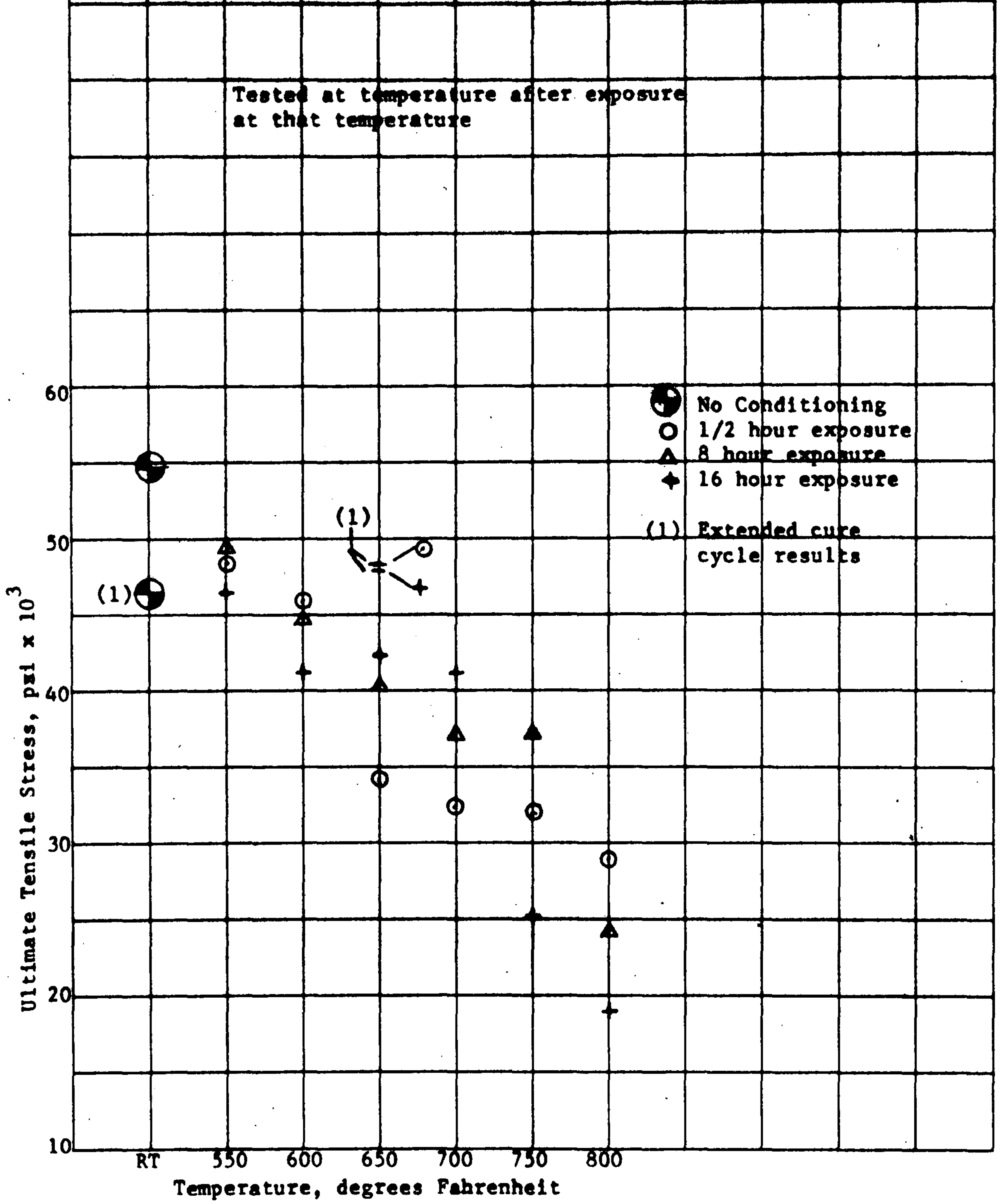


Table 12

Ultimate Compression Strength - Reproducibility Panels

Condition	High	Low	Ave.
Room Temp.	114,990	91,518	100,704
* At 550 F after			
1/2 hour	78,529	66,422	72,646
8 hours	112,847	89,184	97,885
16 hours	108,911	89,198	99,571
At 600 F after			
1/2 hour	50,679	44,785	46,950
8 hours	101,124	87,102	94,892
16 hours	106,437	87,403	97,924
At 650 F after			
1/2 hour	50,818	41,879	46,102
8 hours	82,025	68,921	74,562
16 hours	61,913	54,918	59,452
At 700 F after			
1/2 hour	34,141	25,296	29,779
8 hours	66,327	57,966	62,300
16 hours	74,411	62,214	67,455
At 750 F after			
1/2 hour	17,617	10,514	13,716
8 hours	55,585	52,322	53,671
16 hours	24,816	21,651	23,076
At 800 F after			
1/2 hour	17,507	13,693	15,893
8 hours	30,476	27,352	29,387
16 hours	1,695	1,075	1,283

* Tested at the stated temperature after the designated period of time at the stated temperature.

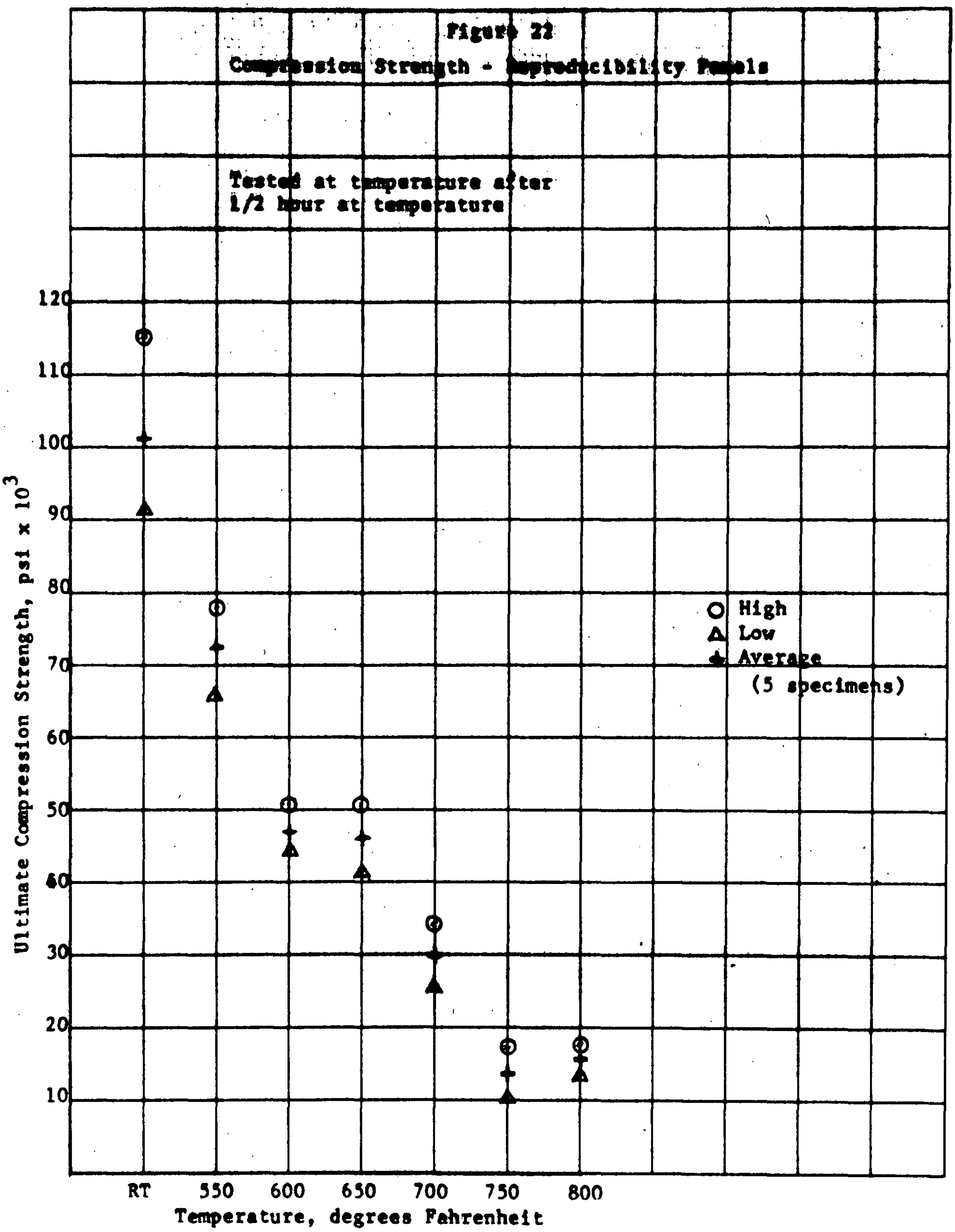


Figure 22

Compression Strength - Reproducibility Panels

Tested at temperature after
1/2 hour at temperature

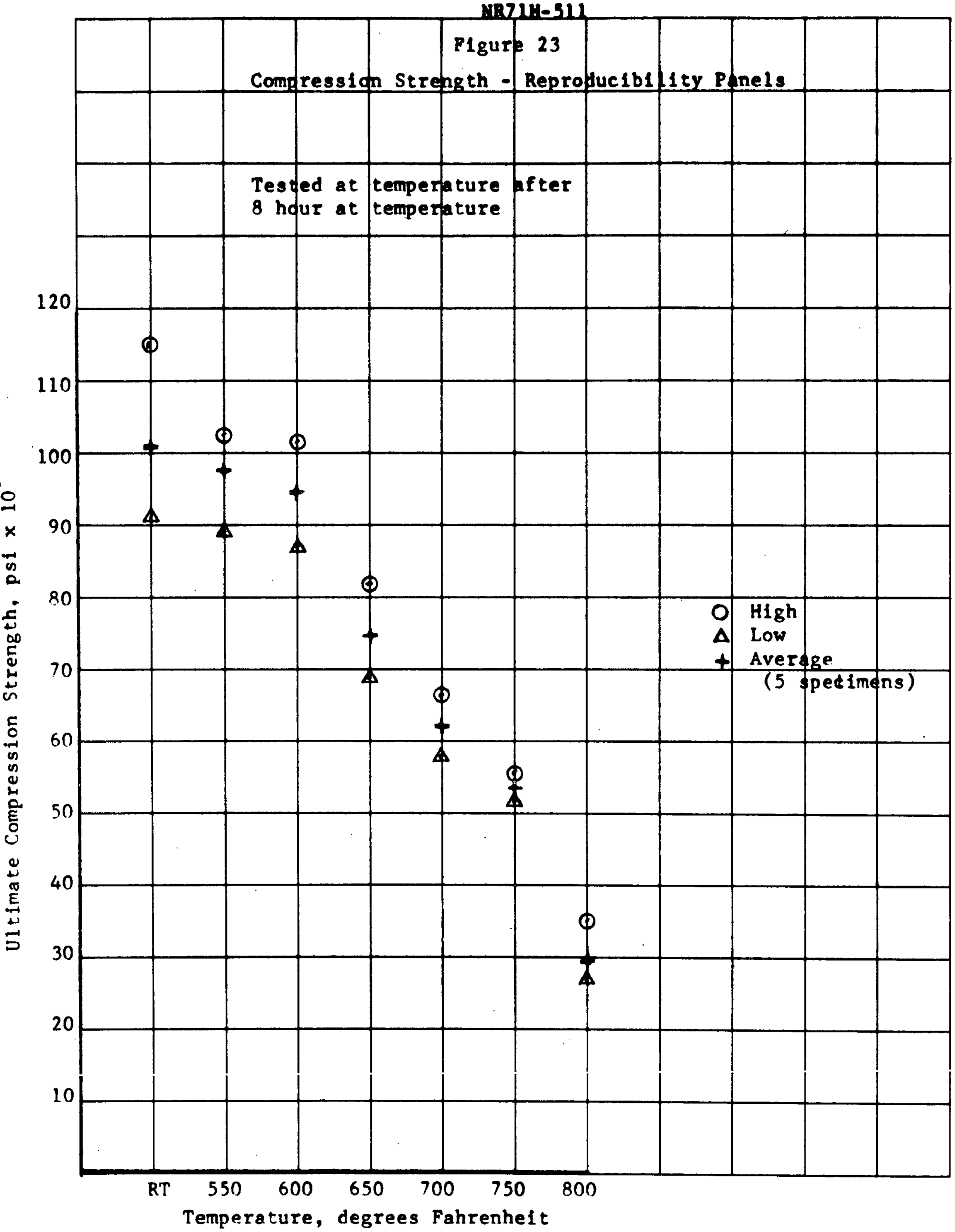


Figure 23

Compression Strength - Reproducibility Panels

Tested at temperature after 8 hour at temperature

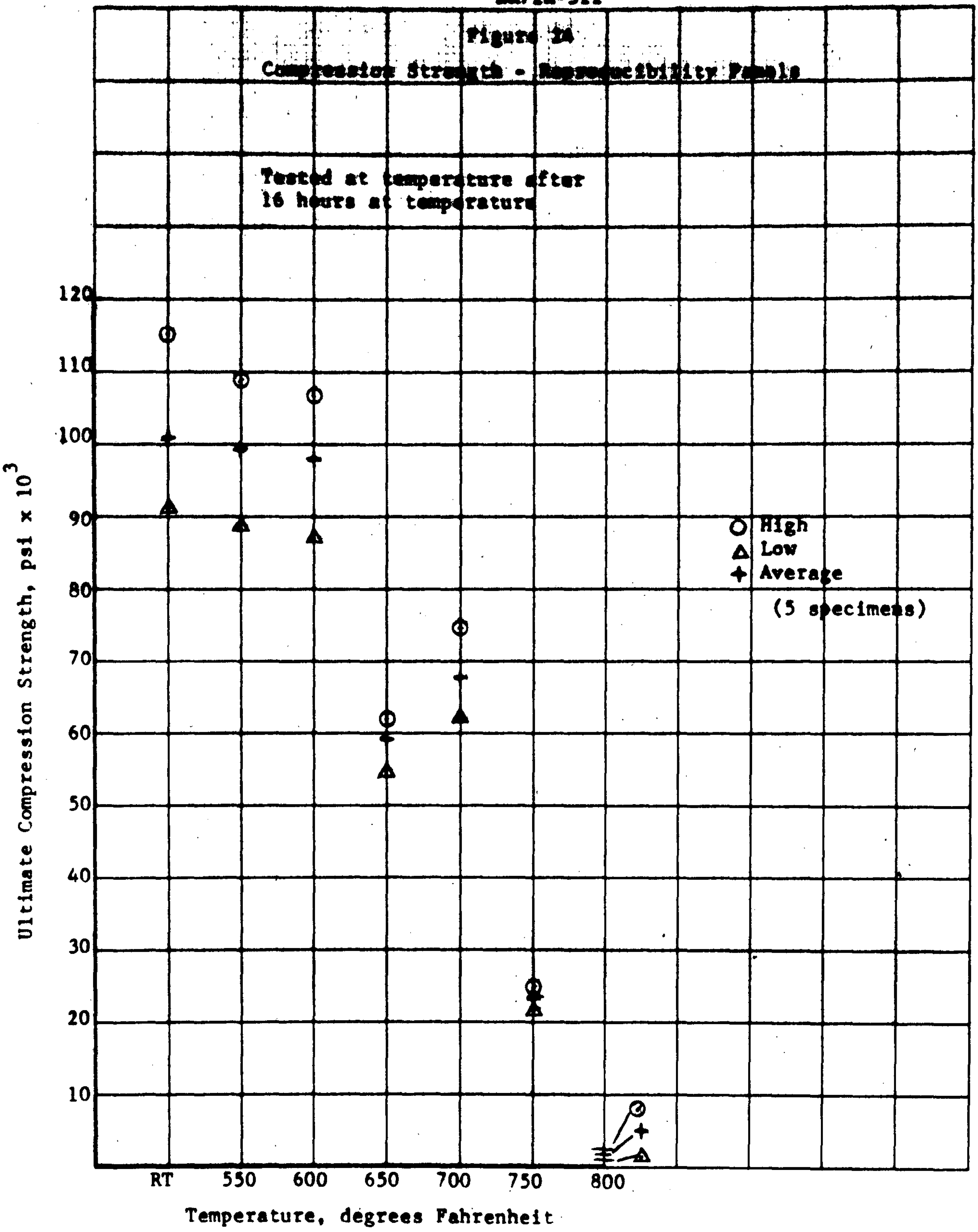


Figure 2A

Compression Strength - Reproducibility Panels

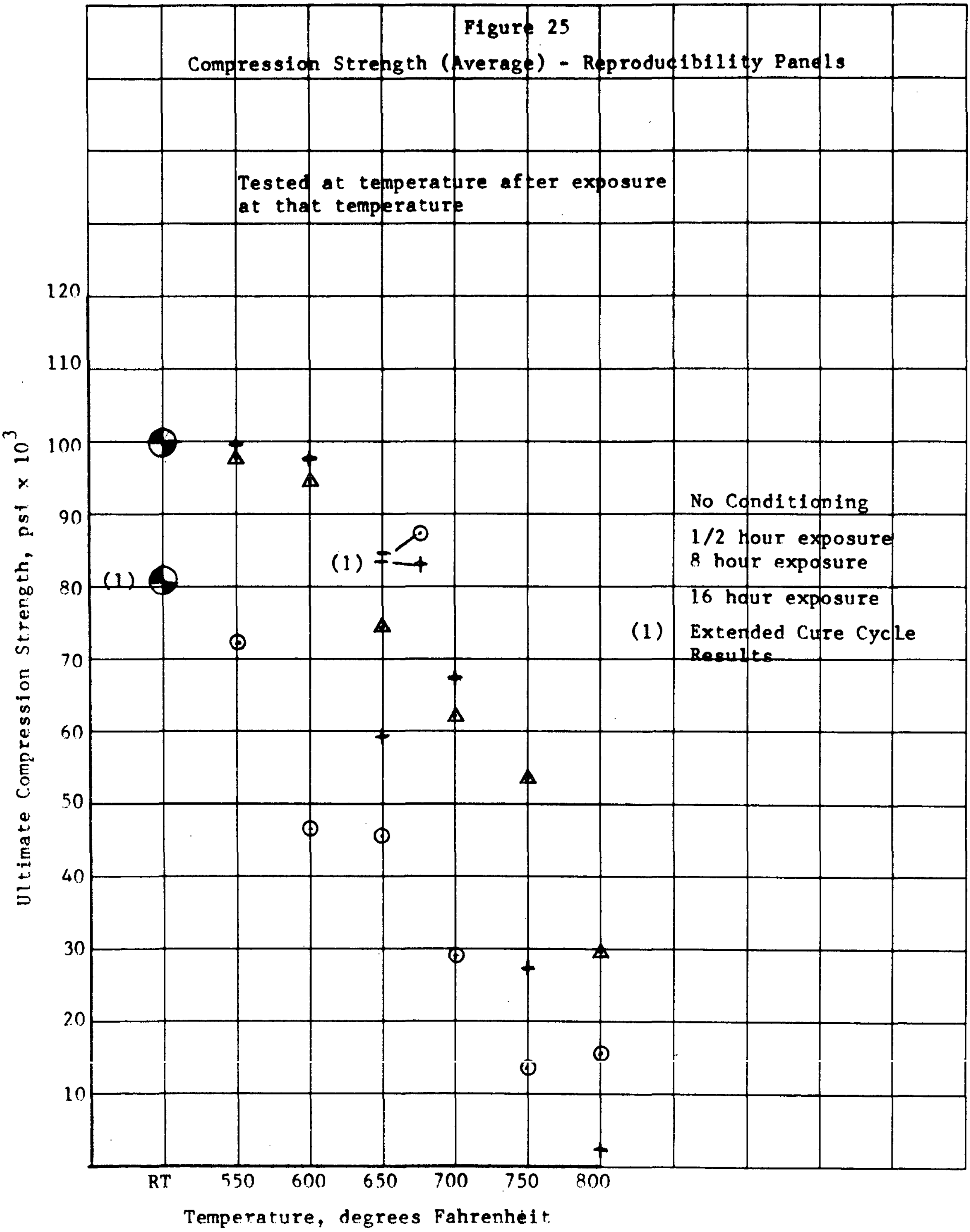


Figure 25

Compression Strength (Average) - Reproducibility Panels

Table 13

Ultimate Short Beam Strength - Reproducibility Panels

Condition	High	Low	Ave.
Room Temp.	6,067	4,800	5,318
* At 550 F after			
1/2 hour	4,226	3,857	4,027
8 hours	4,636	3,914	4,277
16 hours	4,909	4,018	4,523
At 600 F after			
1/2 hour	3,544	3,087	3,352
8 hours	3,816	3,631	3,745
16 hours	4,236	4,156	4,166
At 650 F after			
1/2 hour	3,024	2,205	2,507
8 hours	3,398	2,804	3,152
16 hours	4,287	3,690	4,019
At 700 F after			
1/2 hour	1,778	1,680	1,717
8 hours	3,545	3,052	3,323
16 hours	3,329	2,910	3,081
At 750 F after			
1/2 hour	1,840	1,410	1,570
8 hours	1,363	1,291	1,326
16 hours	1,563	1,005	1,307
At 800 F after			
1/2 hour	1,674	1,210	1,372
8 hours	1,613	1,260	1,378
16 hours	1,429	1,149	1,251

* Tested at the stated temperature after the designated period of time at the stated temperature.

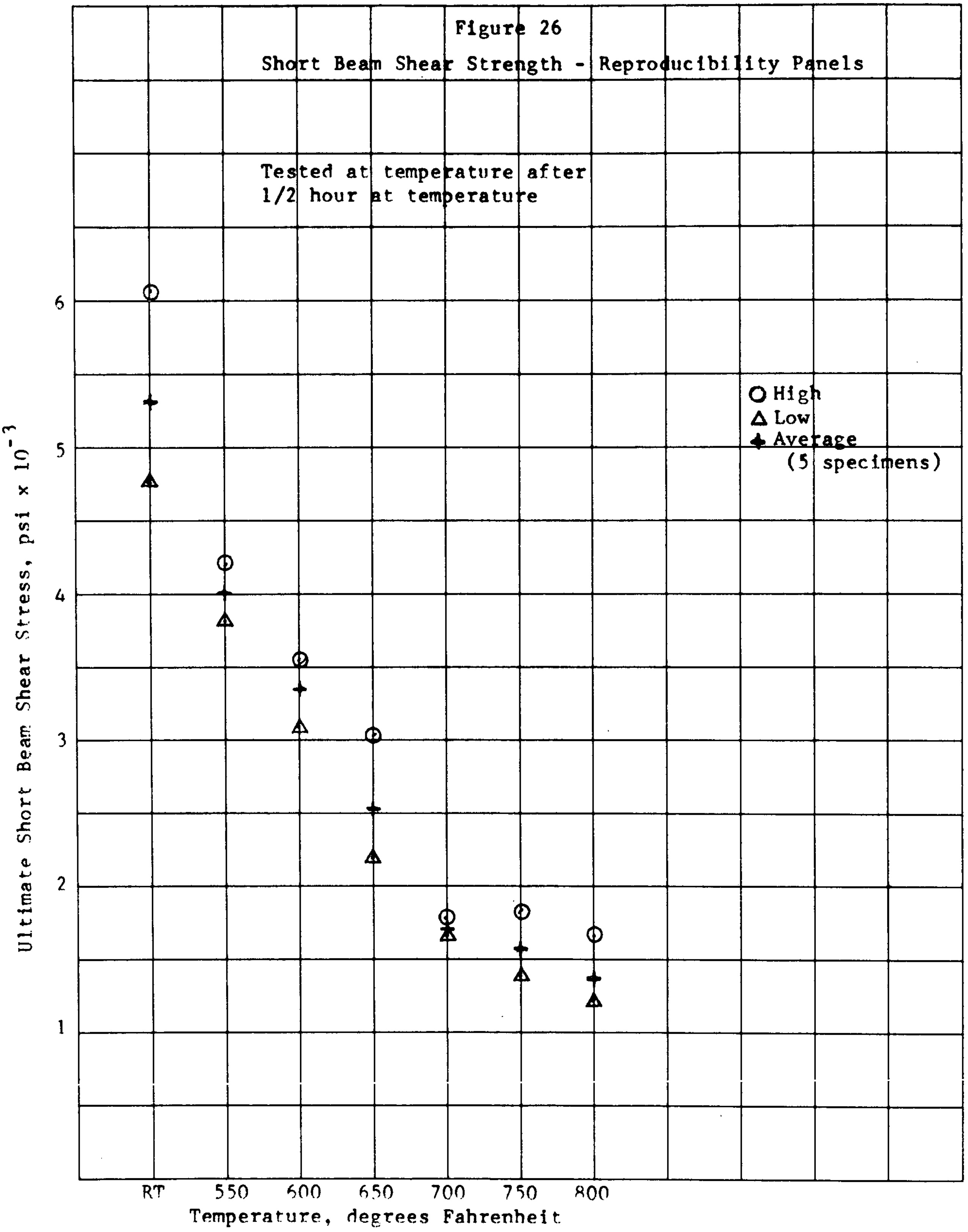


Figure 26

Short Beam Shear Strength - Reproducibility Panels

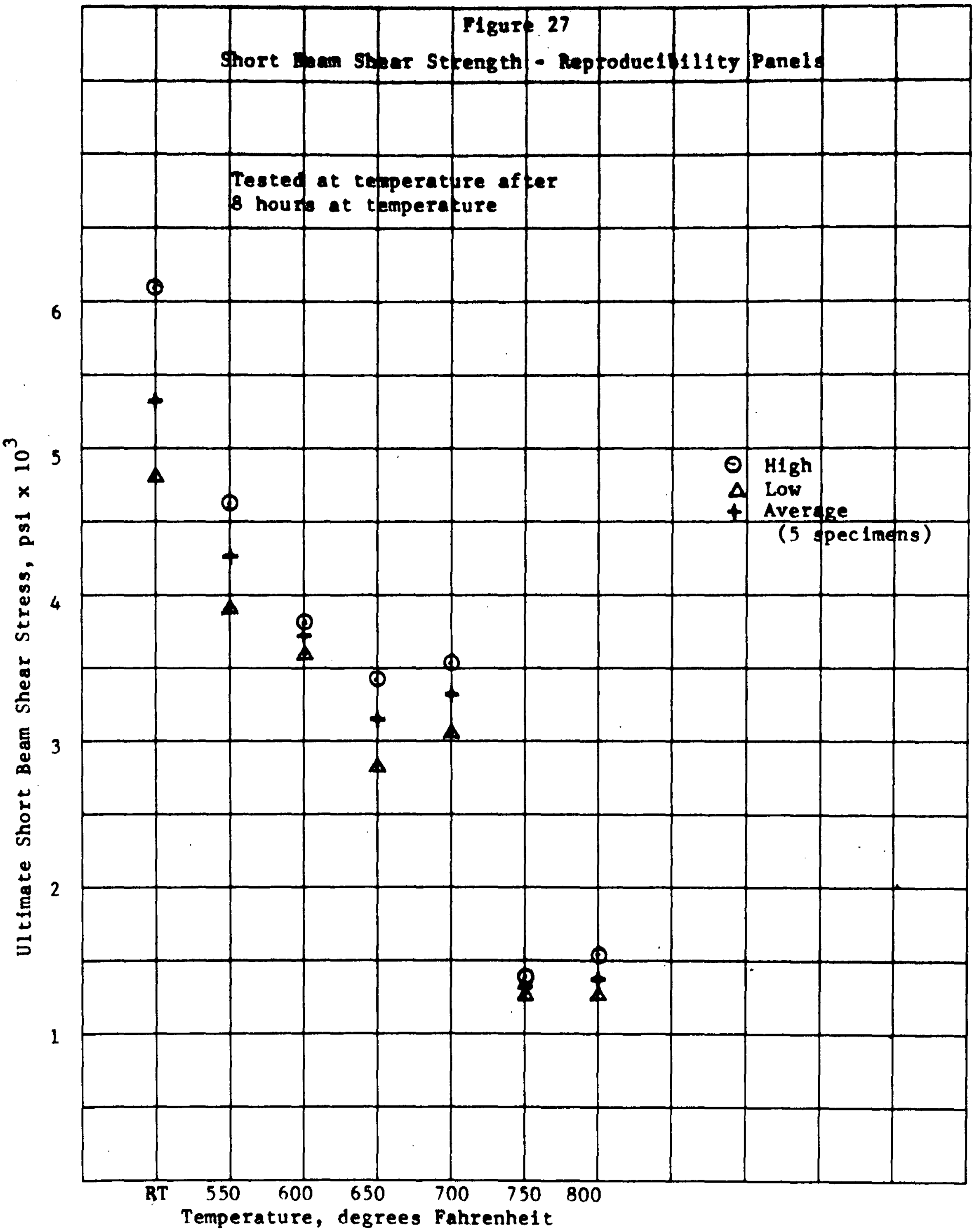


Figure 27

Short Beam Shear Strength - Reproducibility Panels

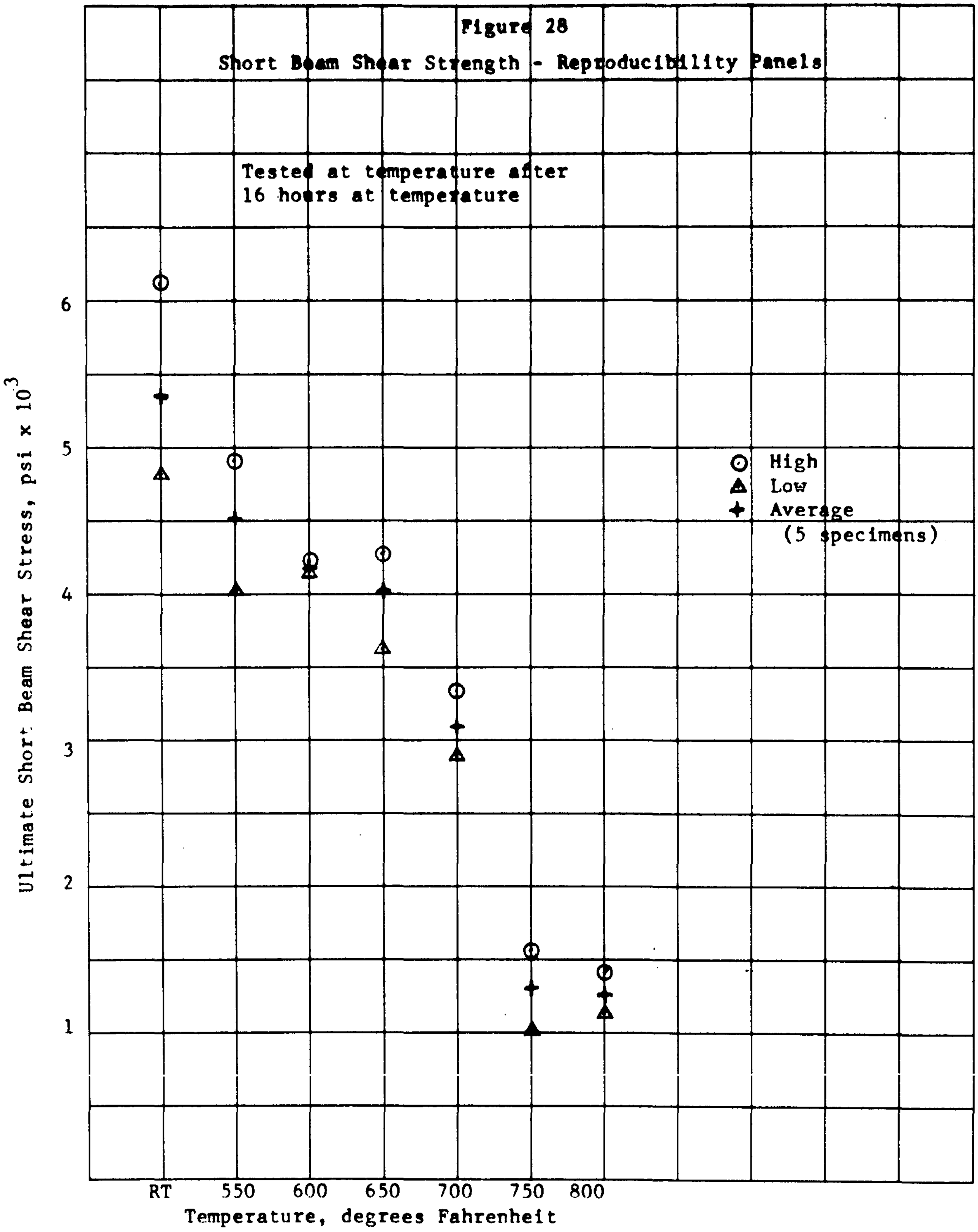
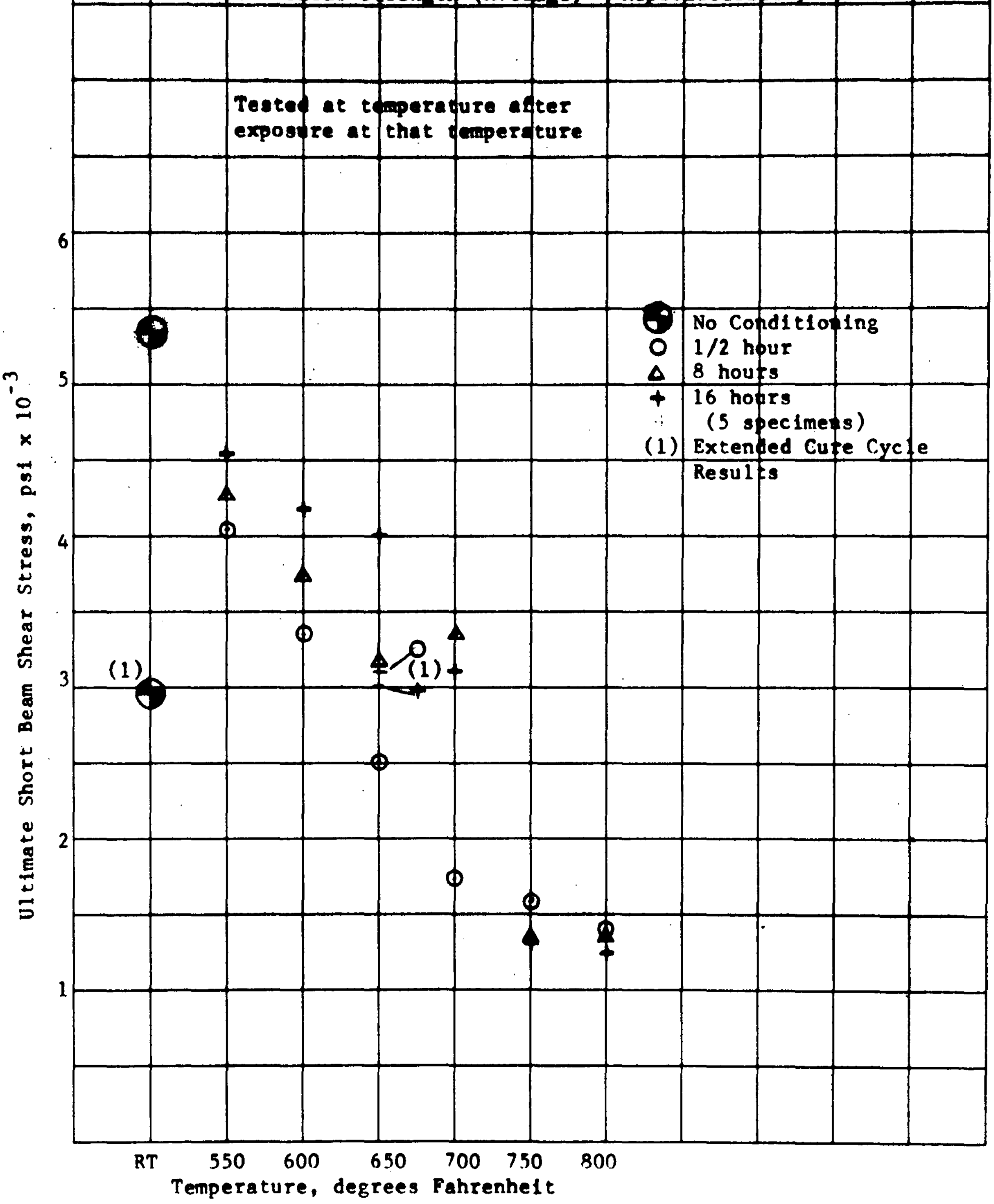


Figure 28

Short Beam Shear Strength - Reproducibility Panels

Figure 29

Short Beam Shear Strength (Average) - Reproducibility Panels



These data revealed that, for 700 F operation, a post cure at 700 F longer than 8 hours was required since the 16 hour data were higher than those obtained at 8 hours. A small program was established to determine if the post cure cycle could be improved to bring the 1/2 hour exposure data up to that obtained after 16 hours. All remaining specimens were exposed to 700 F for 10 hours and retested after 650 F exposures. These data are presented in Table 14, and plotted in Figures 21, 25 and 29. While the additional post cure reduces the room temperature strength of the composite in tension and compression, the reduced strength is still equivalent to the increased elevated temperature strength achieved by the long time at the higher temperature. In all exposures at 650 F up to 16 hours, the test results at temperature are within experimental error of the room temperature strength. Thus any component to operate at 650 F for short periods may use the room temperature results for design data.

Table 14

Average Ultimate Strength Reproducibility Panels Extended Post Cure

Test	Original Post Cure Cycle	Extended Post Cure Cycle(1)
Tension		
Room Temperature	54,200	47,005
(2) At 650 F after		
1/2 hour	34,342	47,950
16 hours	42,200	47,962
Comp.		
Room Temperature	100,704	81,187
(2) At 650 F after		
1/2 hour	46,102	83,700
16 hours	59,452	82,896
Short Beam Shear		
Room Temperature	2,507	2,981
(2) At 650 F after		
1/2 hour	3,152	3,260
16 hours	4,019	3,051

(1) Same as original cycle with an additional 10 hours at 700 F incorporated.

(2) Tested at the stated temperature after the designated period of time at the stated temperature.

SECTION IV

Interleaving Studies

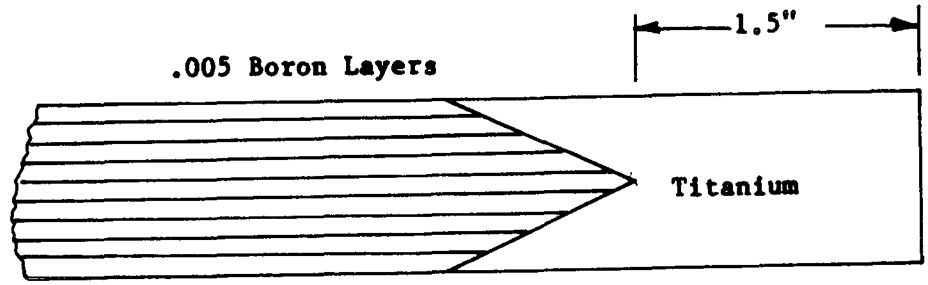
4.1 Introduction

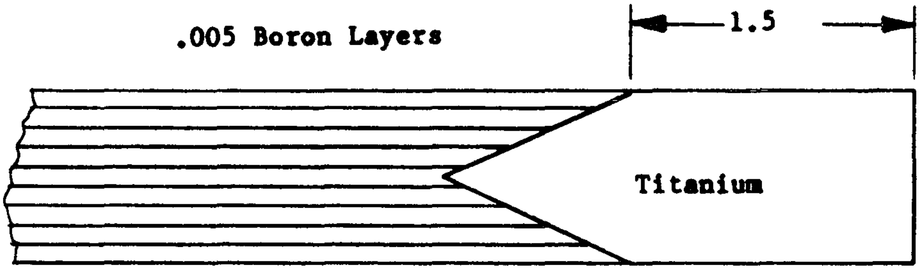
Previous programs sponsored by Department of Defense organizations have shown that the bolt bearing strength of a mechanically attached boron composite is of such low level that severe weight penalties resulted. It has also been established that adhesive bonding could not be utilized for transfer of the maximum loads which the composite was capable of carrying. Previous efforts to incorporate titanium metal into the joint have improved the situation to a degree but have also resulted in increased manufacturing costs due to the problems of drilling through the boron composite-titanium combination. This program was initiated to investigate designs which would result in a smooth transition of the stress loads from the titanium attach point into the fiber reinforced portion of the structure.

Several types of joints were to be considered in the design analysis including 1) bonded double scarf, 2) bonded single scarf, 3) bonded single lap and 4) titanium interleaving with bolt attachments. One of the considerations for the design was that it should be applicable to a large ring structure such as a missile transition stage.

4.2 Analysis of Joint Configurations

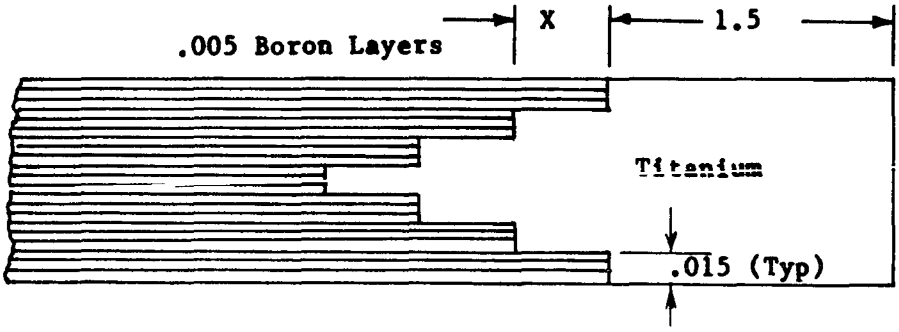
Figure 30 shows two variations of a double scarf design where infinite other variations would be possible by changing the angle θ. Figure 31 shows a modified double scarf where the titanium is stepped rather than a straight scarf. None of these configurations was really applicable to production processing. The straight double scarf joint, where the boron composite would be considered the tongue, would be virtually impossible to lay-up unless the titanium were made in two pieces. This would require very delicate machining for the two pieces to fit when one considers the possibility of the ring being 30-35 feet in diameter. The designs where the titanium is the tongue and the modified or step configuration would require that part of the composite be laid up, the titanium ring positioned and the balance of the composite applied. Pressure would have to be applied to reduce the thickness of the initial plies or the machined rings would not fit the lay-up. A two step cure, where only one-half of the required composite was laid-up, could be used to eliminate this problem but other problems of making the composite match the scarf or steps on the blind side appear insurmountable. The single scarf joint, Figure 32, or a stepped single scarf hold the most promise for a solid titanium doubler but again complex machining and lay up problems exist. The strength obtainable by these joints also could be a problem. Figure 33, shows a single lap joint, and, while this was more feasible from a production standpoint the load transfer is not adequate. Figure 34 shows two interleaving techniques either of which can be made satisfactory by changing the value of "X" in either configuration. Analytically, but not confirmed experimentally, the "A" configuration would give the greater shear strength since a greater area is available for bonding between the composite and the titanium.





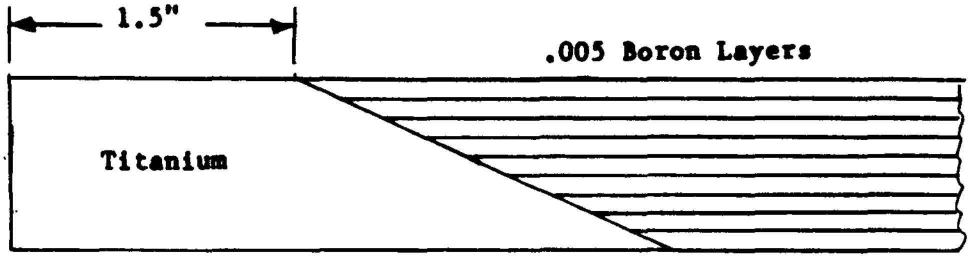
Double Scarf Joints

Figure 30

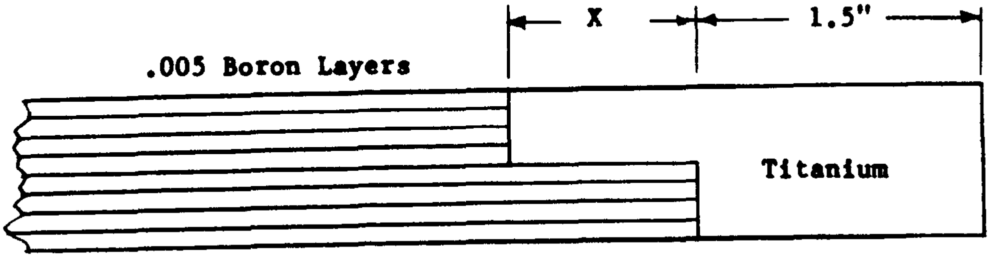


Modified Double Scarf Joint

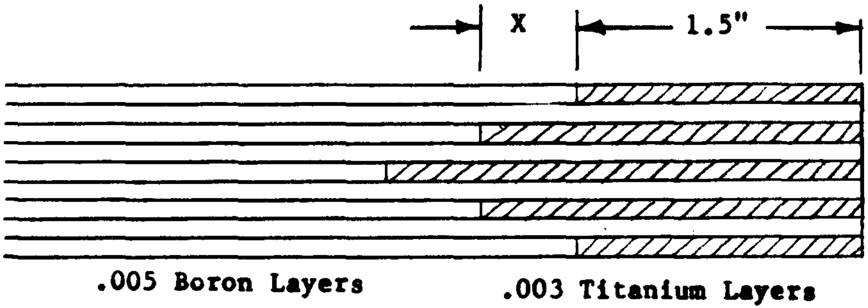
Figure 31



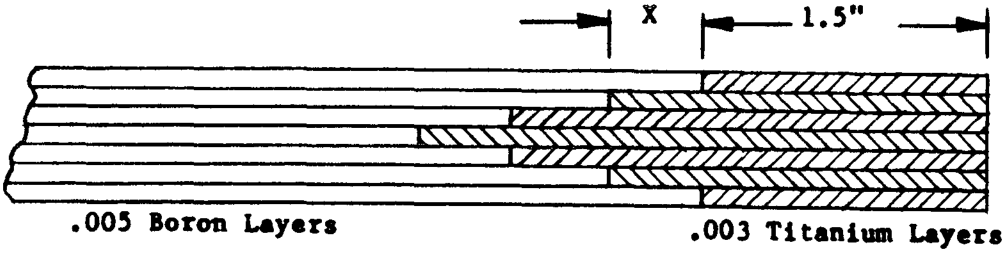
Single Scarf Joint
Figure 32



Single Lap Joint
Figure 33



"A"



"B"

Interleaved Configurations
Figure 34

This configuration creates problems, however, with hole drilling since the boron composite and titanium machine completely differently. The "B" configuration can be made, by varying "X", to satisfactorily carry the load and to have in the laminate titanium adequate bolt bearing properties. Edge trim machining or drilling was, with this configuration, no problem in that there is no boron where these operations must be accomplished. No further effort was to be given to the "A" configuration if the "B" design met all requirements during the experimental fabrication.

4.3 Laboratory Development Program

To effectively incorporate titanium into any composite structure several items had to be considered. These included the titanium surface preparation for bonding, selection of adhesive prime, selection of adhesive material, coefficient of expansion (in both directions) of the components and the establishment of load carrying capability of various overlap lengths.

4.4 Thermal Expansion Data

The coefficient of thermal expansion of the composite, oriented as required for the beam facing sheet, was determined using a Harrup Automatic Recording Dilatometer. The coefficient was determined parallel and perpendicular to the longitudinal axis and are presented in Table (15). The average coefficients of the composite in the 0 degree and 90 degree directions and of the 3 Al-2 1/2 V titanium alloy are 3.15, 3.65 and 5.3 x 10^{-6} respectively. Thus the titanium has greater expansion than the composite in either direction. Other materials such as zirconium, tantalum and molybdenum have coefficients of expansion more closely matching that of the specific composite orientation but it was believed that no major problem would be encountered with the use of titanium.

It should be noted, Table (16), that a permanent dimensional change occurred in the composite after exposure to 800 F. In the 90 degree direction the specimen shrank approximately 1% while in the 0 degree direction it grew by approximately the same amount.

4.5 Titanium Surface Preparation for Bonding

A review of various programs where bonding of titanium was accomplished revealed that three different procedures for surface preparation were used. It should be noted that none of the programs involved the same alloy as used (3 Al-2 1/2V) in this program. Three different methods, presented below, were selected for evaluation with the 3 Al-2 1/2 V alloy.

4.6 Adhesive Evaluation

The design of the beam requires two different types of adhesive. For conventional applications such as secondary bonding of facings to core and assembly of components, the only candidate material was FM 34, a polyimide adhesive. This material would not be satisfactory for bonding the titanium interleaves into the boron composite since it results in a glueline thickness of 0.007 to 0.010 inches. The BR 34 primer was then investigated. Two sets of specimens were prepared for bonding by spraying a 60% solids solution of BR 34 Primer on the faying surfaces. One set of details had a 0.003 to 0.004 inch thick coating and the second set 0.005 to 0.006 inch thick coating. The sprayed surfaces were air dried for 30 minutes followed by a 30 minute oven dry at 220°F. Lap shear specimens were made with a 1/2 inch overlap using and cured by the following schedule.

45 minutes to 240 ± 10 F
Hold temperature for 4 hours
180 minutes to 320 ± 10 F
30 minutes to 350 ± 10 F
Hold six hours minimum (17 hours used)

This cycle was followed by an unrestrained post cure of:

30 minutes to 350 F and hold 30 minutes
45 minutes to 500 F and hold 2 hours
15 minutes to 600 F and hold 4 hours
Allow to cool to less than 200 F prior to removal from oven - do not force to cool.

The shear strength with either thickness of primer is satisfactory with strengths in excess of 2500 psi at room temperature and approximately 1500 psi at 500 F after 1/2 hour at 500. The glue line thickness may be somewhat thin, however, for the .0024 inch foil to build up to the .0053 thickness of the boron ply. Also, since the optimum cure cycle for the adhesive could not be used for the interleaving, additional specimens were fabricated using the cure cycle of the prepreg. A second set of lap shears were fabricated using the 35-520 prepreg since that material was used for the boron prepreg and would obviously have a compatible cure cycle. The results of these two sets of specimens were:

Table 15

Thermal Expansion Data

Temperature Range (26°-426°C) (78°-800°F)
Approach rate - 50°C/hr.

Specimen No.	% Expansion	Coefficient (x 10^{-6}) °C	°F
1 - 90	.33	8.25	4.58
2 - 90	.23	5.75	3.18
3 - 90	.23	5.75	3.18
1 - 0	.27	6.75	3.74
2 - 0	.25	6.25	3.46
3 - 0	.17	4.25	2.36
Ave. coefficient in 90 direction		6.58	3.65
Ave. coefficient in 0 direction		5.75	3.15

Table 16

Dimensional Change in Material (Inches)

Specimen No.	Original Length	Length After Exposure	Change in Length
1 - 90	1.5095	1.5080	-0.0015
2 - 90	1.5095	1.5080	-0.0015
3 - 90	1.5080	1.5060	-0.0020
	Ave. dimensional change in 90° direction		-0.00166
1 - 0	1.5020	1.5040	+0.0020
2 - 0	1.5045	1.5050	+0.0005
3 - 0	1.5030	1.5040	+0.0010
	Ave. dimensional change in 0° direction		

Method #1

Solvent wipe metal with soft cloth (MEK).
Sprex clean 15 minutes at 180°F. (Alkaline Clean)
Rinse with hot (150°F) tap water then cold tap water
Pickle in the following water solution at room temperature for 5 minutes:

Nitric acid 15% by weight
Hydrofluoric acid 3% by weight

Rinse in tap water at room temperature.
Apply Pasa Jell 107 liquid to the titanium surface by dip or with an acid resistant brush. Cover the entire surface by cross brushing.
Allow to dry for 20 minutes.
Rinse thoroughly with water.
Air dry not over 150°F.
Bond within 16 hours.

Method #2

Sand the bonding surfaces
Swab the surface with a solution of trisodium phosphate, sodium fluoride and hydrofluoric acid.
Rinse until the ph of the surface is between 5.5 and 8.
Test for "water break free" surface
Wipe dry and air dry.

Method #3

Alkaline clean
Acid clean using Nitric-Hydroflouric solution
Rinse in cold water to wet metal surfaces. Then immerse for 2 to 2 1/2 minutes at room temperature in the following solution:

Trisodium Phosphate	6.7 oz./gal.
Sodium Fluoride	1.2 oz./gal.
Hydrofluoric Acid (50%)	3.4 oz./gal.
Water	Remainder

Rinse in cold water. Soak in hot water at 140° - 150°F for 15 minutes minimum. Rinse in cold water and dry.

Single lap shear specimens were fabricated using material whose surface was prepared by each of the three methods. All surfaces were primed with BR 34 reduced to 60% solids. The primer was cured per the manufacturer's instructions and the lap shear specimens bonded with FM 34 (polyimide) adhesive. Methods 1 and 2 were completely unsatisfactory with no single result exceeding 1000 psi. Method #3 resulted in specimens with an average shear strength of 2280 psi.

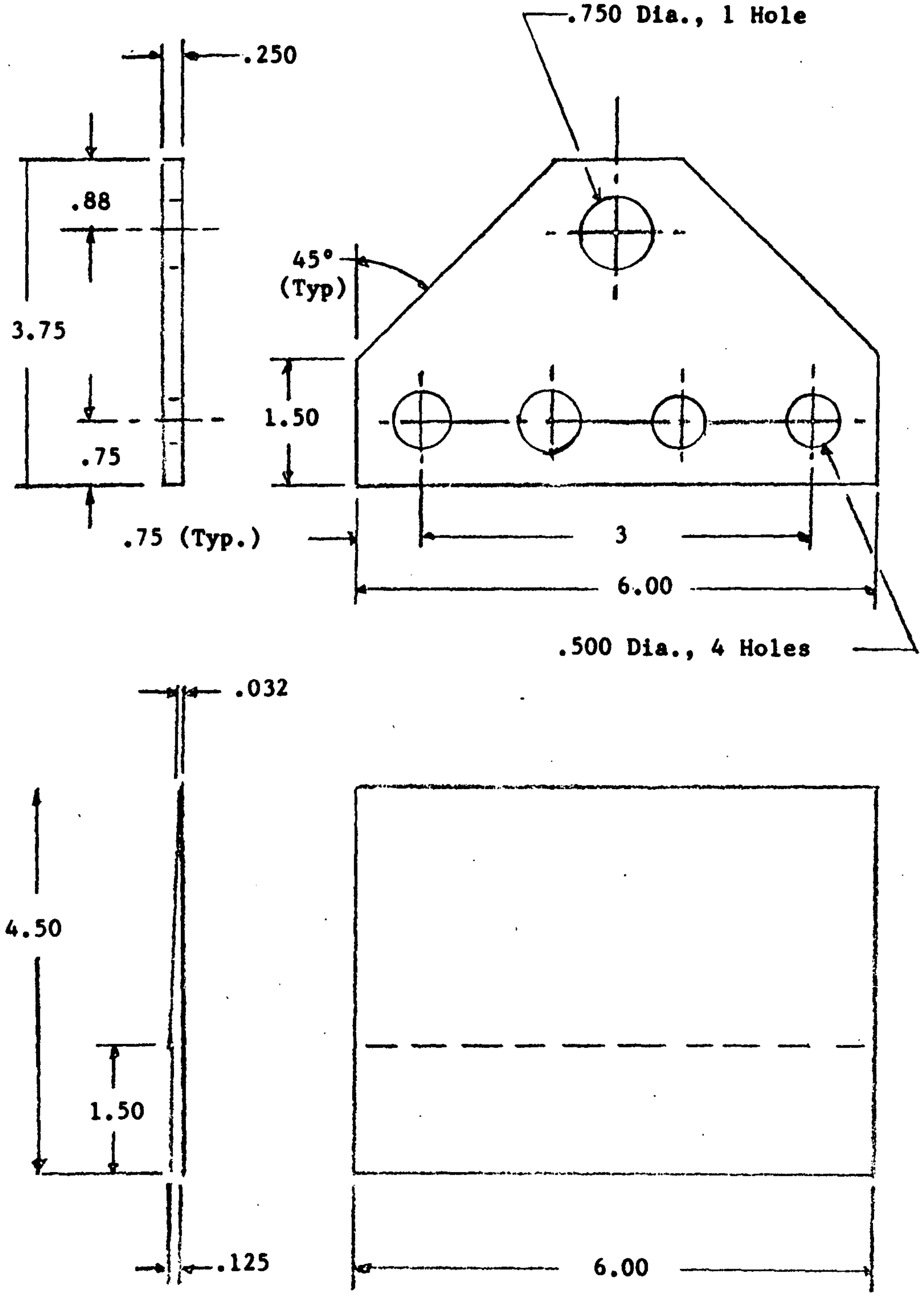


FIGURE 35 Details of Test Fixture - Interleaving

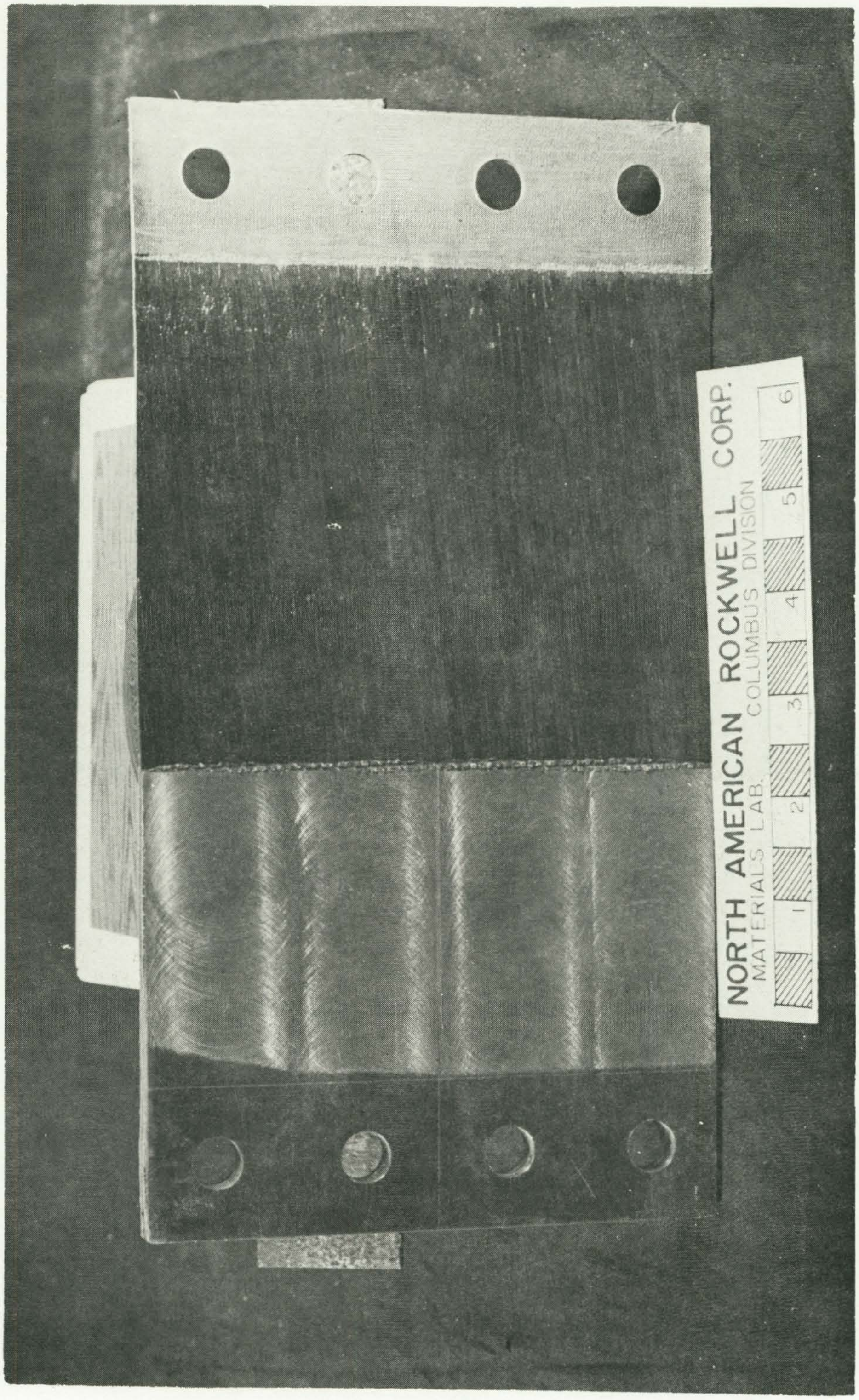


Figure 36 Interleaved Test Specimen

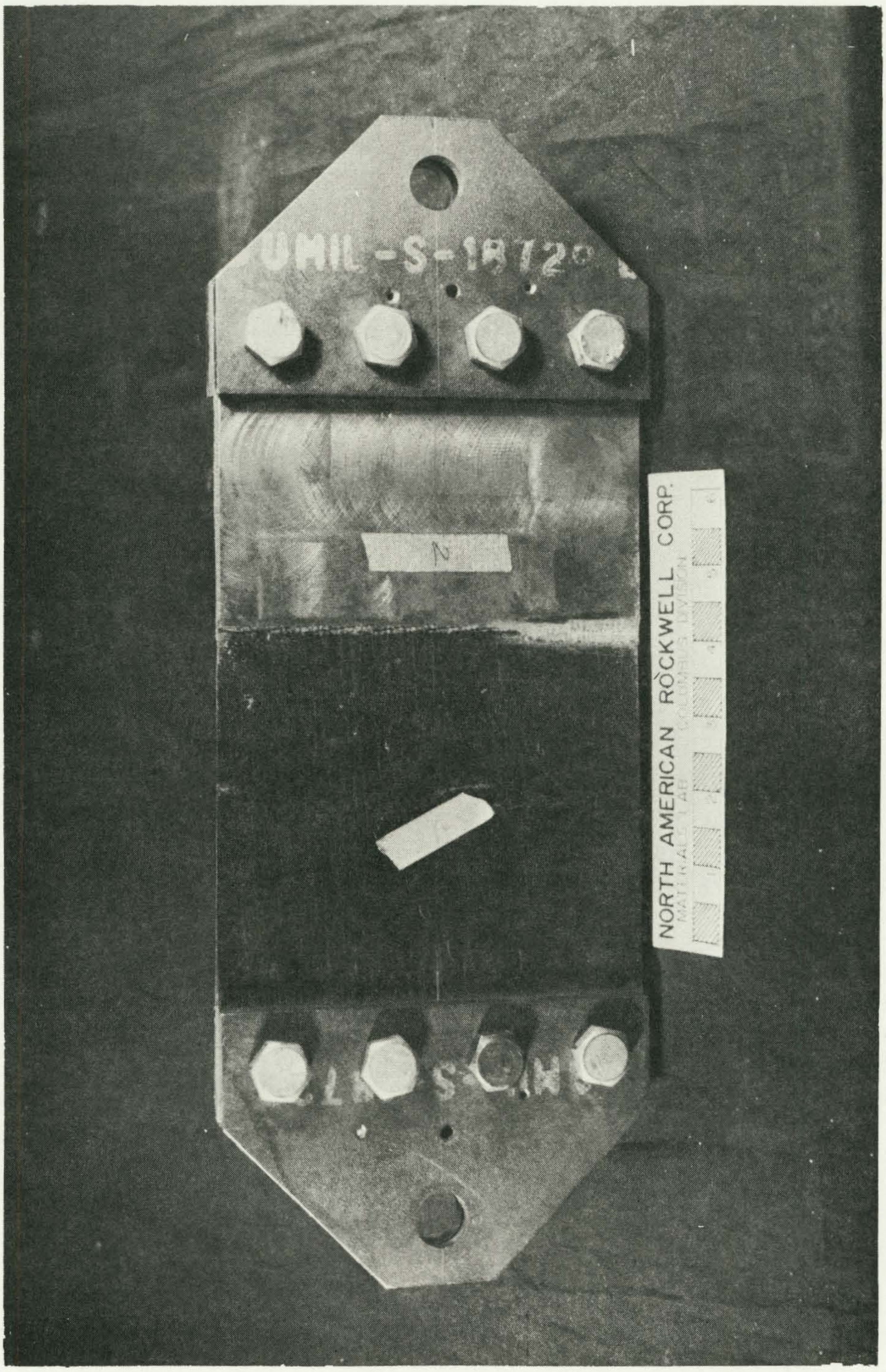


Figure 37 Completed Test Specimen

		Lap Shear Strength (psi)
35-520 bond	High	1350
	Low	1280
	Ave.	1310
FM-34	High	1520
	Low	1060
	Ave.	1290

The FM 34 resulted in the highest and lowest individual values. The 35-520 bond was extremely uniform with only -30 and +40 psi deviation from the average. Based on these data the 35-520 material shall be used for all future work where titanium foil is bonded to the wet boron prepreg.

4.7 Interleaving Joint Properties

Since this portion of the program was started before completion of the adhesive evaluation, the first panel for the interleaving studies was fabricated using BR 34 primer and a 3/4 inch overlap of each titanium foil to boron composite joint. The surface of the titanium was prepared for bonding as noted in para. 3.2. The BR #34 primer was applied to a thickness of 0.005 ± .001 inches immediately after cleaning. The primer was air dried for 30 minutes at room temperature followed by 30 minutes in an air circulating oven at 220°F. The prepreg was processed using the four minute soak-blot dry technique. The assembly was cured using the cure cycle discussed in Section III. The panel itself was made sufficiently wide to result in four, six inch wide straight sided specimens. In order to test these specimens it was necessary to bond a tapered shim on one end of the specimen and to attach a fixture at the other end. A drawing of the tapered shim and the fixture are shown in Figure 35. Figure 36 shows the specimen with the shim bonded on one end and the 1 1/2 inch titanium interleaving foil at the other end. Figure 37 shows the specimen completely bolted in the test fixture ready for test. The first specimen however, was pinned rather than bolted as shown.

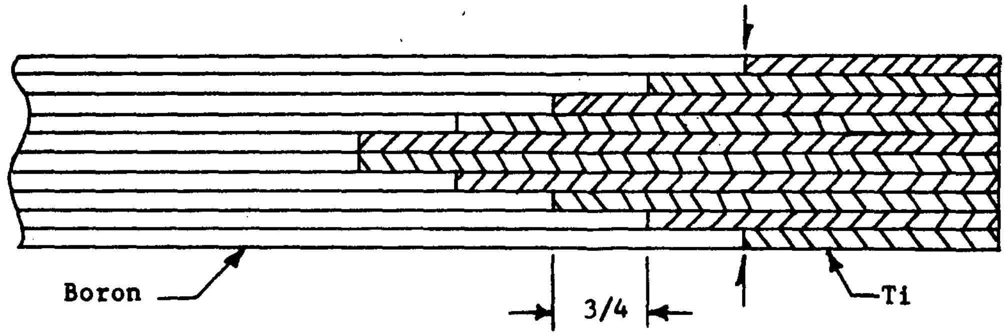
The requirement for the joint is that it must satisfactorily meet the load requirements of the skins for the boron-epoxy beam which was previously tested. These loads were 3925 lbs/linear inch of facing width.

The first specimen failed as a bearing failure of the laminated titanium at a load of 3200 lbs. for a six inch width or 640 lbs./inch of width. The second specimen was made with the steel plates bonded to the titanium still with one row of bolt holes. This specimen failed at 9000 lbs. or 1500 lbs/linear inch of width as a combined bolt bearing and titanium to titanium bond shear failure.

A third specimen duplicated the second except close tolerance holes were drilled into the titanium through plates which held the laminated titanium in compression to prevent delamination during drilling. The plates were bolted to the specimen and the bolts torqued down. A bearing failure again occurred of 9600 lbs. or 1600 lbs/linear inch. The specimens and fixtures were then redesigned to incorporate additional bolts to increase the bolt bearing of the titanium above the required 3925. This redesign also increased the bolt size from 1/2 inch to 5/8 inch to further increase the bearing strength.

The first specimen tested with the new configuration utilized a 5 inch titanium length rather than the 3 inch previously used, thus, permitting the second row of bolts. The specimens failed as a titanium tensile failure through the first row of bolt holes at 11,500 pounds or 1917 pounds per inch of width. This results in a stress of 136,400 psi in the titanium at failure.

In order to force the failure into the bonded joint, one side of the loading fixture was bonded to the panel and the other half of the fixture attached with bolts. The bond area added enough load carrying capability that the failure occurred across that section of the specimen where the effective thickness of the titanium in the laminate was only 8 plies or 0.0192 inches thick as shown below.



Failure occurred at 16,750 pounds (2790 pounds/inch of width) or at 71% of the required load. This was equivalent of 145,400 psi tensile stress in the effective titanium thickness.

From these data it became apparent that a one for one interleaf of 0.0024 inch titanium would not carry the required stress. Using a tensile allowable of 140,000 psi and a load requirement of 3925 pounds per linear inch, it was readily found that, assuming the adhesive has no tensile strength, an effective titanium thickness in the laminate of 0.027 or 0.0034 inches per ply was required to carry the design load.

A search was conducted for a source of titanium foil with a thickness greater than 0.0035 inches but less than 0.004 since it was believed that the 0.0013 inch thickness (boron molded thickness per ply 0.0053 - titanium thickness 0.004) was the minimum permissible thickness for the glue line. Fansteel Corporation indicated that they could supply the same alloy in 10 inch widths in a thickness of 0.0037 ± 0.0003 inches. The Fansteel foil when received was 0.00355 inches, well within the tolerance established for the material.

Six inch wide specimens were fabricated using the same procedures as previously except that the new adhesive system discussed in paragraph 3.3 was utilized. Specimens were made with overlap lengths of 1/2, 3/4 and one inch. One specimen of each lap length was tested at room temperature after the steel tabs were bonded on the boron laminate with AF 126-2 (low temperature) adhesive. The results of these tests were: 1/2 inch overlap - 21,250 pound load, 3/4 inch overlap - 20,000 pound load and 1 inch overlap - 16,000 pound load. The 650 F tests were not successful since the bonded steel tabs to carry the bearing loads to test the interleaving sheared from the test specimen at low load levels under 650 F exposure. New split, titanium end plates were machined for **rebonding** to the 650 F specimens but their condition was not satisfactory for further testing.

Two additional 3/4 inch overlap specimens were made identical to the previous six inch wide specimens except that the boron composite was of the same orientation as those for the box beam skins. The split titanium doublers were bonded to the laminate. The room temperature results were 17,000 lbs. for the six inch specimen with the failure occurring as a tensile failure of the laminated titanium. The second specimen was tested at 650°F after a one half hour exposure at that temperature and the result was a 13,000 pound total load with the initial failure, a sheared bond between the boron composite and the doubler.

4.8 Conclusions

The impregnating resin, Pyralin 35-520, is more satisfactory for laminating the titanium foil for the edge attachment since the cure cycle of the conventional adhesive, FM 34, is not compatible with the cure cycle required for the boron-polyimide.

Overlap lengths of either 1/2 or 3/4 inches are satisfactory for load transfer from the boron composite to the laminated titanium thus an overlap length of 5/8 inch ± 1/8 inch would be satisfactory for production. This conclusion is for the orientation tested rather than a generalized statement.

SECTION V

Design and Ultimate Load Prediction Analysis

5.1 Design

A simulated OV-10A center wing box beam was chosen as the major component to be fabricated using boron/polyimide facings with interleaved titanium joints. The detail design is similar to the boron/epoxy box beam generated by the Columbus Division for the Air Force Materials Laboratory under United States Air Force Contract No. F33615-67-C-1802, entitled, "Fabrication Techniques for Advanced Composite Attachments and Joints". Physical dimensions of the box beam were maintained, but the scarf joint (skin splice) and drag fitting attach points in the lower skin panel were eliminated while interleaved titanium joints were incorporated into the spars and skin panels. Material substitutions were made as required to develop structural capability at elevated temperature. The resultant detail design of the demonstration test box is shown in the following:

Figure 38 - Drawing TT-17543 Insert, Wing Non-metallic Center Box

Figure 39 - Drawing TT-17544 Door, Wing Non-metallic Center Box Assy. Of

Figure 40 - Drawing TT-17545 Rib, Wing Non-metallic Center Box Assy. Of

Figure 41 - Drawing TT-17546 Panel, Wing Non-metallic Center Box Upper Skin, Assy. Of

Figure 42 - Drawing TT-17547 Panel, Wing Non-metallic Center Box, Lower Skin, Assy. Of

Figure 43 - Drawing TT-17548 Spar, Wing Non-metallic Center Box, Assy. Of

Figure 44 - Drawing TT-17549 (Sheet 1 & 2) Beam, Wing Non-metallic Center Box, Assy. Of

FOLDOUT FRAME I

FOLDOUT FRAME II

TT-17543

REVISIONS				
ZONE	LTR	DESCRIPTION	DATE	APPROVED
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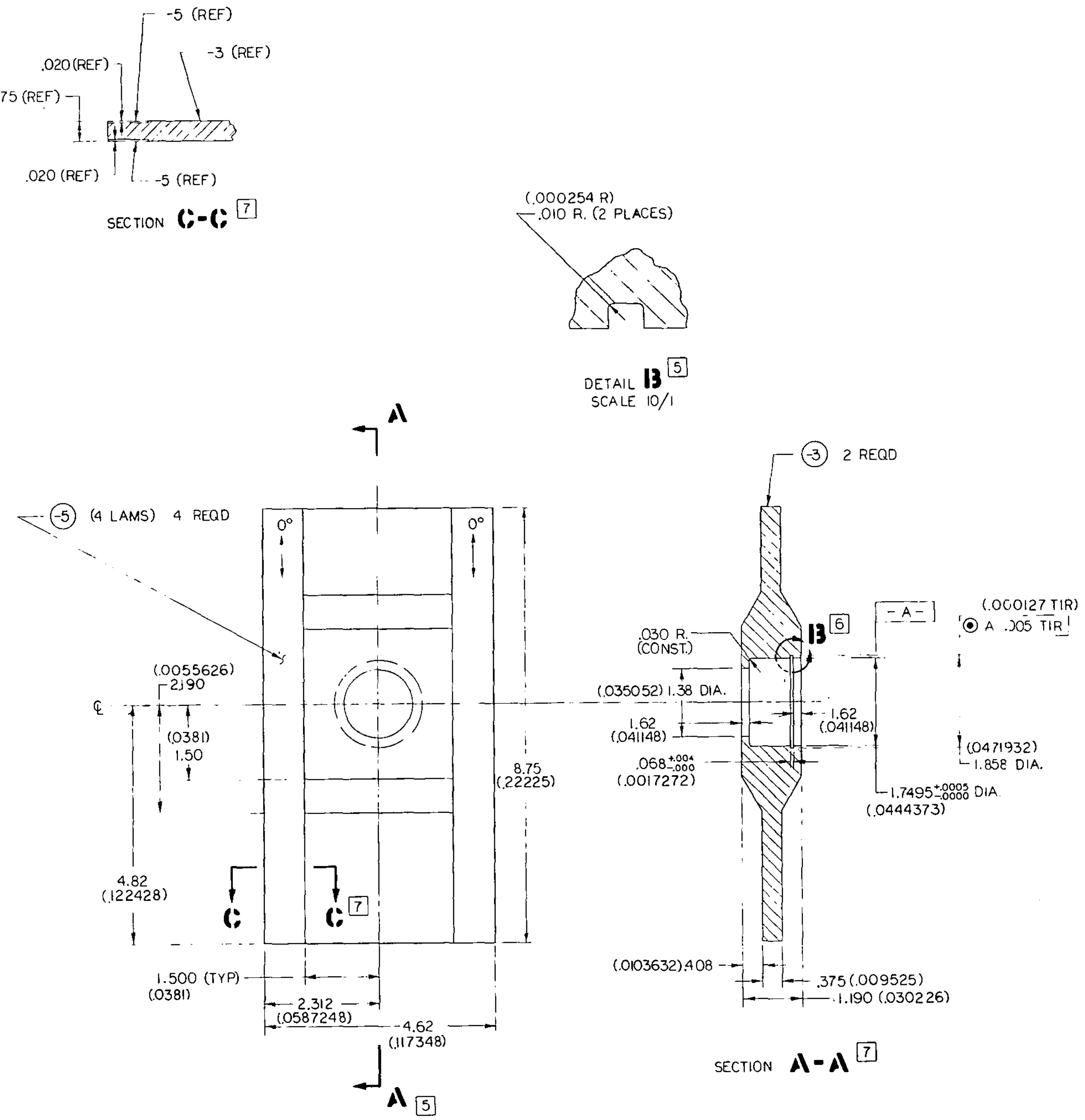
DISPN



② 5- MAY BE PURCHASED FROM WHITTAKER R&D SAN DIEGO, CALIF.

① 4- MAY BE PURCHASED FROM FIBERITE CORP. WINONA, WIS.

3- FABRICATE PER NR SPEC ST0605HA0015

2- IDENTIFY PER NR SPEC LA0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP

1- PARENTHETICAL DIMENSIONS ARE IN SI UNITS

NOTES: UNLESS OTHERWISE NOTED

ITEM OR FIND NO.	QUANTITY REQUIRED -1	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE	MATERIAL	THKNS GAUGE (MATERIAL)	WIDTH OD/ID (MATERIAL)	LG (SIZE)	DRAWING OR SPECIFICATION NUMBER	ZONE
037										
036										
035										
034										
033										
032										
031										
030										
029										
028										
027										
026										
025										
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013										
012										
011										
010										
009										
008										
007										
006										
005										
004										
003	4		-5	STIFFENER	BORON POLYIMIDE PREPREG	.0052		.197 SQ FT	②	8
002	1		-3	INSERT	HI-E-9020 POLYIMIDE MOLD COMPOUND				①	5
001			TT-17543	INSERT ASSY OF						

PARTS LIST

ITEM	QTY	NEXT ASSY	USED ON	END ITEM NO.	THRU
-1	4	TT-17548	TEST		

REQD PER END ITEM — APPLICATION — EFFECTIVITY

HEAT TREAT

FINISH

INSPECT PER MIL-I-6870 (ST0501LT0007 CLASS I)

UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE IN INCHES.
MFG TOLERANCES ON:
UNDIMENSIONED ± .03
.XX (DECIMALS) ± .03
.XXX (DECIMALS) ± .010
ANGLES ± 30'
HOLES NOTED "DRILL"
.013 THRU .040: + .001 − .001
.041 THRU .130: + .002 − .001
.131 THRU .229: + .003 − .001
.230 THRU .500: + .004 − .001
.501 THRU .750: + .005 − .001
.751 THRU 1.000: + .007 − .001
1.001 THRU 2.000: + .010 − .001
DO NOT SCALE PRINT

CONTR NO.
DWN BY JUNGEBERG HOWLAND 10-27-70
CHK BY
APVD

North American Aviation/Columbus
North American Rockwell
COLUMBUS, OHIO 43216

INSERT - WING NON-METALLIC
CENTER BOX
(TEST SPECIMEN)

SIZE E CODE IDENT NO. 89372 TT-17543

SCALE 1/1 & NOTED SHEET

FORM 311-G-2 REV. 9-69

FIGURE 38

FOLDOUT FRAME I

FOLDOUT FRAME II

ZONE	LTR	REVISIONS DESCRIPTION		DATE	APPROVED
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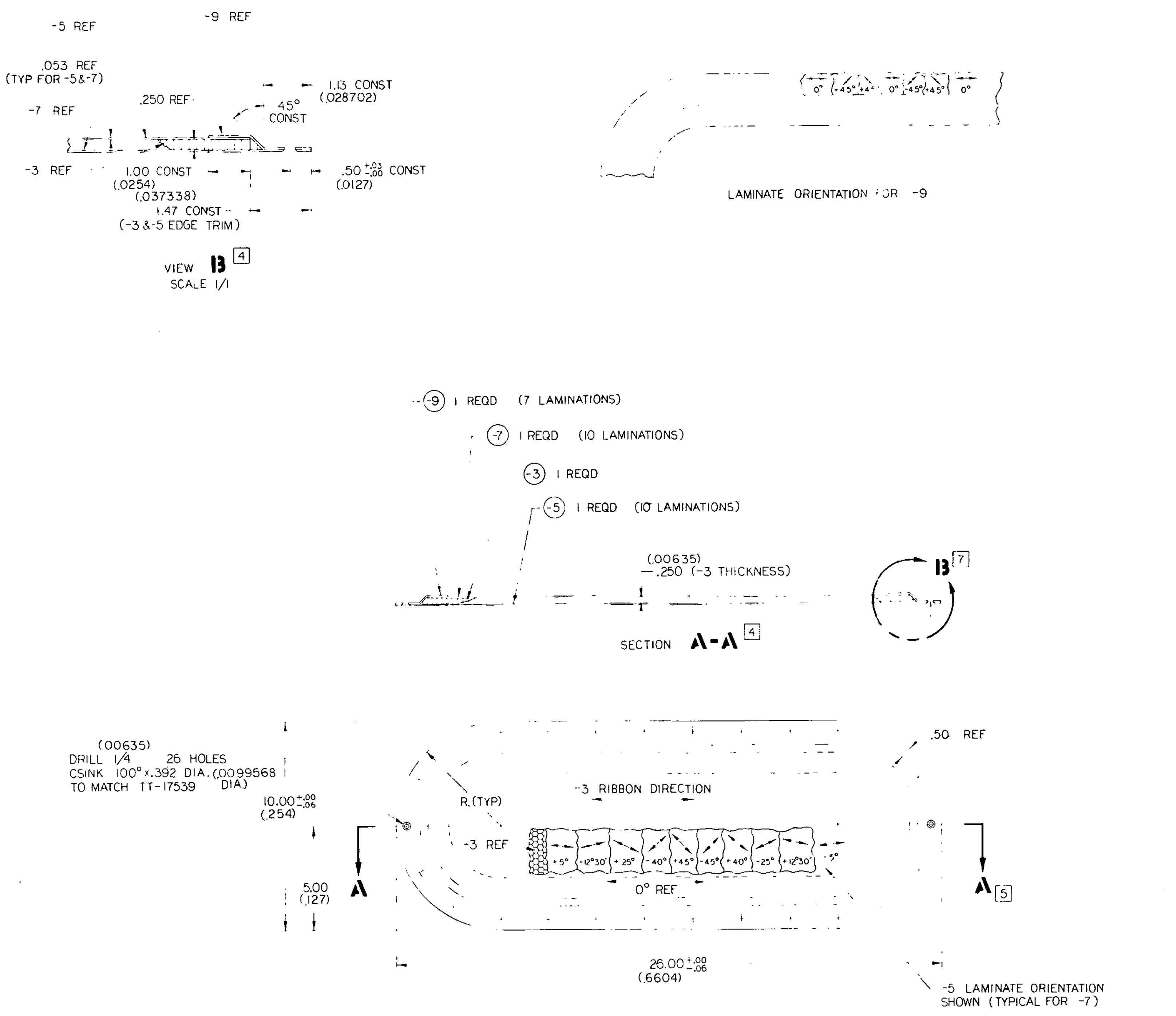

(3) 11- MAY BE PURCHASED FROM EI DUPONT, WILMINGTON, DEL.
(2) 10- MAY BE PURCHASED FROM WHITTAKER R&D SAN DIEGO, CALIF.
(1) 9- MAY BE PURCHASED FROM HEXCEL PRODUCTS INC. DUBLIN, CALIF.
8- SKIN GAP BETWEEN -1 AND TT-17546 TO BE 0 TO .06
7- BEND RADII .06
6- HOLES EQUALLY SPACED BETWEEN DIMENSIONED POINTS
5- 125/ ALL MACHINED SURFACES
4- IDENTIFY PER NR SPEC LA0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP
3- MACHINE PER NR SPEC LA0103-004
2- PARENTHETICAL DIMENSIONS ARE IN SI UNITS
1- FABRICATE PER NR SPEC ST0605HA0015
NOTES: UNLESS OTHERWISE NOTED

ITEM OR FIND NO	QUANTITY REQUIRED -1	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE	MATERIAL	THKNS GAUGE	WIDTH OD/ID	LG	DRAWING OR SPECIFICATION NUMBER	ZONE
037										
036										
035										
034										
033										
032										
031										
030										
029										
028										
027										
026										
025										
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016										
015										
014										
013										
012										
011										
010										
009										
008										
007										
006										
005	1		-9	CLOSEOUT	2501 GLASS FABRIC PREPREG	.010		8.10 SQ FT	(3)	
004	1		-7	FACE	BORON POLYIMIDE PREPREG	.0052		11.20 SQ FT	(2)	
003	1		-5	FACE	BORON POLYIMIDE PREPREG	.0052		11.20 SQ FT	(2)	
002	1		-3	CORE	HRH 327 - 1/4 - 6.0 HONEYCOMB CORE	.250	7.75	23.75	(1)	
001			TT-17544	DOOR ASSY OF						

PARTS LIST

ITEM	QTY	NEXT ASSY	USED ON	END ITEM NO.	THRU
-1	1	TT-17549	TEST		

REQD PER END ITEM — APPLICATION — EFFECTIVITY

HEAT TREAT

FINISH

INSPECT PER MIL-I-6870
(ST0501LT0007 CLASS II)

UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE IN INCHES.
MFG TOLERANCES ON:
UNDIMENSIONED ± .03
.XX (DECIMALS) ± .03
.XXX (DECIMALS) ± .010
ANGLES ± 30'
HOLES NOTED "DRILL"
.013 THRU .040: + .001 − .001
.041 THRU .130: + .002 − .001
.131 THRU .229: + .003 − .001
.230 THRU .500: + .004 − .001
.501 THRU .750: + .005 − .001
.751 THRU 1.000: + .007 − .001
1.001 THRU 2.000: + .010 − .001
DO NOT SCALE PRINT

CONTR NO.
DWN BY JUNGEBERG HOWLAND 10-27-70
CHK BY
APVD

North American Aviation/Columbus
North American Rockwell
COLUMBUS, OHIO 43216

DOOR - WING NON-METALLIC
CENTER BOX ASSY OF
(TEST SPECIMEN)

SIZE E CODE IDENT NO. 89372 TT-17544
SCALE 1/2 & NOTED SHEET

FORM 311-G-2 REV. 9-69

FOLDOUT FRAME I

FOLDOUT FRAME II

ZONE	LTR	DESCRIPTION	DISPN	DATE	APPROVED
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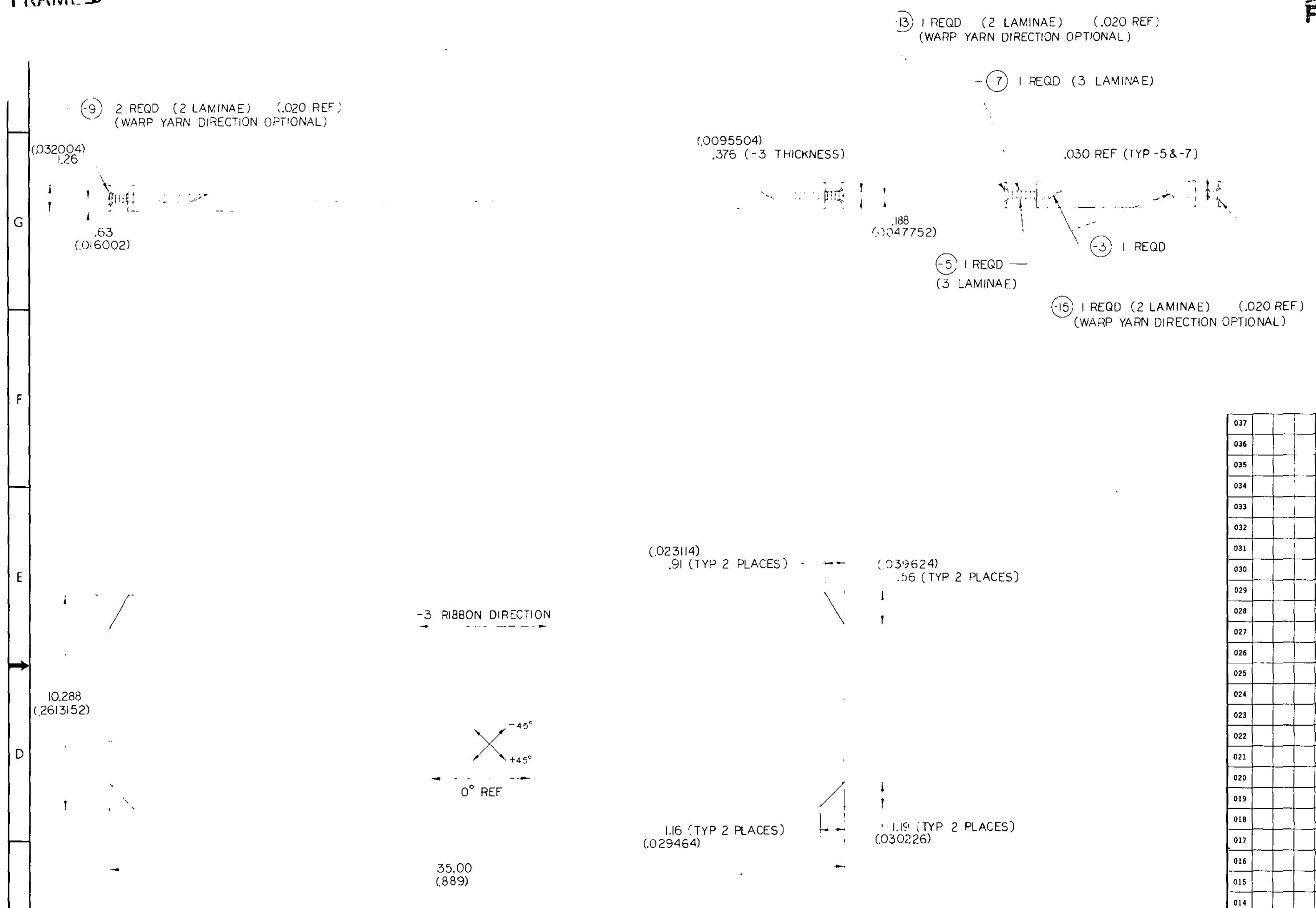

9- PARENTHETICAL DIMENSIONS ARE IN SI UNITS
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⟨1⟩7- MAY BE PURCHASED FROM HEXCEL PRODUCTS INC. DUBLIN, CALIF.
6- WARP YARN DIRECTION FOR -5 & -7 TO BE ±45° ALTERNATED
5- BEND RADII .06
4- $\overset{125}{\surd}$ ALL MACHINED SURFACES
3- MACHINE PER NR SPEC LA0103-004
2- IDENTIFY PER NR SPEC LA0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP
1- FABRICATE PER NR SPEC ST0605HA0015
NOTES: UNLESS OTHERWISE NOTED

ITEM OR FIND NO.	QUANTITY REQUIRED -1	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE	MATERIAL	MATERIAL THKNS GAUGE	MATERIAL WIDTH OD/ID	SIZE LG	DRAWING OR SPECIFICATION NUMBER	ZONE
037										
036										
035										
034										
033										
032										
031										
030										
029										
028										
027										
026										
025										
024										
023										
022										
021										
020										
019										
018										
017										
016										
015										
014										
013										
012										
011										
010										
009										
008										
007	1		-15	CAP	2501 GLASS FABRIC PREPREG	.010		.60 SQ FT	⟨2⟩	4
006	1		-13	CAP	2501 GLASS FABRIC PREPREG	.010		.15 SQ FT	⟨2⟩	5
005	1		-9	CAP	2501 GLASS FABRIC PREPREG	.010		.15 SQ FT	⟨2⟩	8
004	1		-7	CHANNEL	2501 GLASS FABRIC PREPREG	.010		8.25 SQ FT	⟨2⟩	4
003	1		-5	CHANNEL	2501 GLASS FABRIC PREPREG	.010		8.25 SQ FT	⟨2⟩	4
002	1		-3	CORE	HRH 327-1/4-6.0 HONEYCOMB CORE	.381	10.31	35.00	⟨1⟩	4
001			TT-17545	RIB ASSY OF						

PARTS LIST

ITEM	QTY	NEXT ASSY	USED ON	END ITEM NO.	THRU
-1	2	TT-17549	TEST		
REQD PER END ITEM		APPLICATION		EFFECTIVITY	

HEAT TREAT

FINISH

INSPECT PER MIL-I-6870 (ST0501LT0007 CLASS II)

UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE IN INCHES.
MFG TOLERANCES ON:
UNDIMENSIONED ±.03
.XX (DECIMALS) ±.03
.XXX (DECIMALS) ±.010
ANGLES ±30'
HOLES NOTED "DRILL"
.013 THRU .040: +.001 −.001
.041 THRU .130: +.002 −.001
.131 THRU .229: +.003 −.001
.230 THRU .500: +.004 −.001
.501 THRU .750: +.005 −.001
.751 THRU 1.000: +.007 −.001
1.001 THRU 2.000: +.010 −.001
DO NOT SCALE PRINT

CONTR NO.
DWN BY JUNGEBERG HOWLAND 10-27-70
CHK BY
APVD

North American Aviation/Columbus
North American Rockwell
COLUMBUS, OHIO 43216

RIB – WING NON-METALLIC CENTER BOX ASSY OF (TEST SPECIMEN)

SIZE E
CODE IDENT NO. 89372
TT-17545
SCALE 1/2
SHEET

FORM 311-G-2 REV. 9-69

FIGURE 40

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5.2 Ultimate Load Prediction Analysis

The critical design condition results in an upbending moment of 3,150,000 inch pounds ultimate, a nose up torque of 660,000 inch pounds ultimate, and a vertical shear of 21,250 pounds ultimate. A design optimization was conducted under the previous Air Force contract to satisfy the multiple load and stiffness requirements with the least number of boron/epoxy composite laminae. The lower (tension) skin panel facing was determined critical for the boron/epoxy box beam which was designed for combined edgewise loading consisting of 3925 pounds-per-inch in the axial (spanwise) direction and an edgewise shear flow of 400 pounds-per-inch.

A six inch wide tension specimen was fabricated to the design configuration of the boron/polyimide skin panel facings, incorporating a 3/4 inch overlap per ply interleaved titanium joint with ten oriented boron/polyimide layers, and tested at room temperature in an attempt to verify the design strength of the critical lower cover skin. Failure occurred at 17000 pounds through the test jig attach holes in the titanium. The net tension area of titanium foil at the failed area, each outer layer of titanium foil being ineffective for load transfer, is 0.0994 square inches resulting in a stress level of 171,000 psi for the laminated titanium. The stress level in the boron/polyimide laminate at the time of failure was 53,460 psi ultimate. The axial load carrying capability of the wide tension specimen was 2833 pounds per inch, indicating no severe stress concentration factors have been introduced by the interleaving joint concept.

Based on previously presented data in Section III, an F_{tu} = 47,000 psi for the oriented boron/polyimide at both R.T. and 650°F was used to evaluate the strength of the cover panel facings. Based on previously presented data in Section IV, an F_{tu} = 136,400 psi for the laminated titanium foil at R.T. was used to evaluate the strength of the interleaved joints of the cover panel facings. An F_{tu} = 100,000 psi was assumed for the titanium foil at 650°F.

The net cross-sectional area of an .053 inch thick x 38 inch wide lower skin panel facing is 2.014 square inches. At 47,000 psi, this results in a predicted failure axial tension load of 94,658 pounds (2491 pounds per inch). The interleaved titanium joints at the panel ends have demonstrated a greater load carrying capability. In addition, the maximum load buildup in the skin panel is at the wing-to-fuselage attach station rather than the test load introduction points at the ends of the box beam. Since a combined state of stress exists, an ultimate allowable axial tension load of 2350 pounds per inch is predicted at failure. Since 3925 pounds per inch represents the original design ultimate load, the projected 2350 pounds per inch at failure represents 60% design ultimate load.

In comparison, structural test failure of the boron/epoxy center wing box specimen occurred while holding 66 2/3% design ultimate load at R.T. Recorded stress across the tension skin indicated 45,480 psi at failure compared to a design allowable of 75,000 psi and a test coupon verification of 86,210 psi. The failure of the boron/epoxy tension cover was apparently due to the biaxial stress state induced by the high Poisson's ratio effect between the oriented skin and the scarf joint insert along the centerline of the tension cover panel. The strength of the boron/polyimide beam is predicted to be the same for either room temperature or elevated temperature (650°F) environments, whereas the boron/epoxy beam would have zero load carrying capability at 650°F.

Section VI

Component Fabrication

6.1 Introduction

The processing techniques developed in Sections III and IV were incorporated into a process specification which is presented in this report as Appendix A. Appendix A details the processes sufficiently that a production fabrication department can, with a minimum of initial assistance, proceed with component fabrication. Paragraph references in this section refer to those paragraphs of Appendix A. Surfaces of all tools were processed in accordance with paragraph 3.1.3 to assure satisfactory part release.

6.2 Fabrication of Details

6.3 Glass Inserts and Doublers

All glass fabric details, such as the end closeouts for the spar and surface panel sandwiches, the rib facings and caps, clips for spar to surface panel attachment, groove doubler of the spar and the tongue of the tongue and groove joint and the doublers around the door in the upper surface panel, were laid up of polyimide prepreg and bagged in accordance with paragraph 3.16. Cure was accomplished in accordance with the same paragraph. The cured laminates were trimmed as required for its intended use, wrapped in clean kraft paper and stored until required for installation.

6.4 Random Fiber Bearing Inserts

The spar bearing inserts were molded in matched tooling using conventional debulking and curing techniques. Prior to curing, layers of boron prepreg were incorporated on the flat surface along each edge of the part. These methods are shown in paragraph 3.1.4.

6.5 Surface Panel and Spar Facings

The titanium foil was sheared to the several widths as shown on the drawing. Slits were cut into the foil width on two inch spacing such that a five inch width of metal remained uncut to result in solid adhesive bonded titanium laminate at the edge. The titanium foil was cleaned in accordance with paragraph 3.1.8.1 and primed per paragraph 3.1.8.1.2 immediately prior to initiation of the component lay-up. The boron prepreg was water soaked in accordance with paragraph 3.1.5.1 to remove the excess n-methyl pyrrolidone. The components were laid-up in accordance with drawing requirements. In those areas within the composite where the titanium was not contacted by boron-polyimide prepreg, a layer of 35-520 prepregging film was incorporated to laminate the titanium. Upon completion of the lay-up, the assembly was bagged and cured in accordance with paragraph 3.1.5.2.

6.6 Fabrication of Sub-Assemblies

6.7 Ribs

The surfaces of the rib facings and rib caps to be bonded were hand sanded to remove the glossy surface per paragraph 3.1.8.2. One rib facing was placed on one-half of the tool and adhesive applied to the flat surface. The other facing was treated in a like manner on the other half of the tool. The core was positioned on the one facing and the two halves of the tool mated. The rib caps with adhesive applied were located on the rib flanges and held in position by heavy steel straps. The assembly was then prefit, pressurized, cured and post cured in accordance with paragraph 3.1.9.

6.8 Spars

The surfaces to be bonded were cleaned per 3.1.8. The details were placed on the tool in the following sequence.

The attachment clip was positioned on the tool and adhesive applied to the horizontal surface. One spar facing was then positioned. Adhesive was applied over the entire exposed facing area. The two bearing inserts were then placed in the assembly. Honeycomb core was inserted as required. The groove doubler, repositioned on its detail fabrication tool, was incorporated into the lay-up. Adhesive was applied to the second spar facing and this was positioned over the core and doubler. The attaching clip, with adhesive applied to the faying surface, was then placed on the assembly. The upper half of the tool was positioned, pressure was applied per paragraph 3.1.9.4 with the cure and post cure accomplished per paragraph 3.1.9.6. Access openings were cut into each spar approximately 14 inches from each end. Titanium ring doublers, cleaned and processed per paragraph 3.1.8 were bonded on the spar at the access openings.

6.9 Surface Panels

The design of the upper and lower surface panels required identical techniques for processing except that a structural door was incorporated into the design of the upper cover. The fabrication of the upper cover, only, will be discussed. Prior to the lay-up of the upper cover, material was removed from the area where the structural door was to be installed. This material was saved to make the door facings. All faying surfaces were hand sanded to remove the surface gloss and solvent wiped to remove dust and contamination per paragraph 3.1.8. The upper facing was placed on the tool and adhesive applied over the entire surface. The end close outs and core were then positioned. The lower facing, to which adhesive had been applied, was positioned on the assembly. The assembly was bagged and cured in accordance with paragraph 3.1.9 using vacuum bag pressure. Post cure was not accomplished until after the Z member around the door was installed. Adhesive was applied to the precured glass doublers and positioned around the access opening. Pressure was applied and cure was accomplished in the same manner as before. Post cure of the entire assembly was accomplished in accordance with paragraph 3.1.7.

6.10 Structural Door

The boron polyimide laminate which had been removed for installation of the door was used for the fabrication of the door. The facing sections were trimmed to dimension and the surface prepared per para. 3.1.8. The outer skin was placed on a flat tool, adhesive applied over the entire area and the core, adhesive and inner skin positioned. Adhesive was then applied to the Z doubler and it was also placed in the assembly. Bagging, cure and post cure were accomplished in accordance with para. 3.1.9.

6.11 Final Assembly

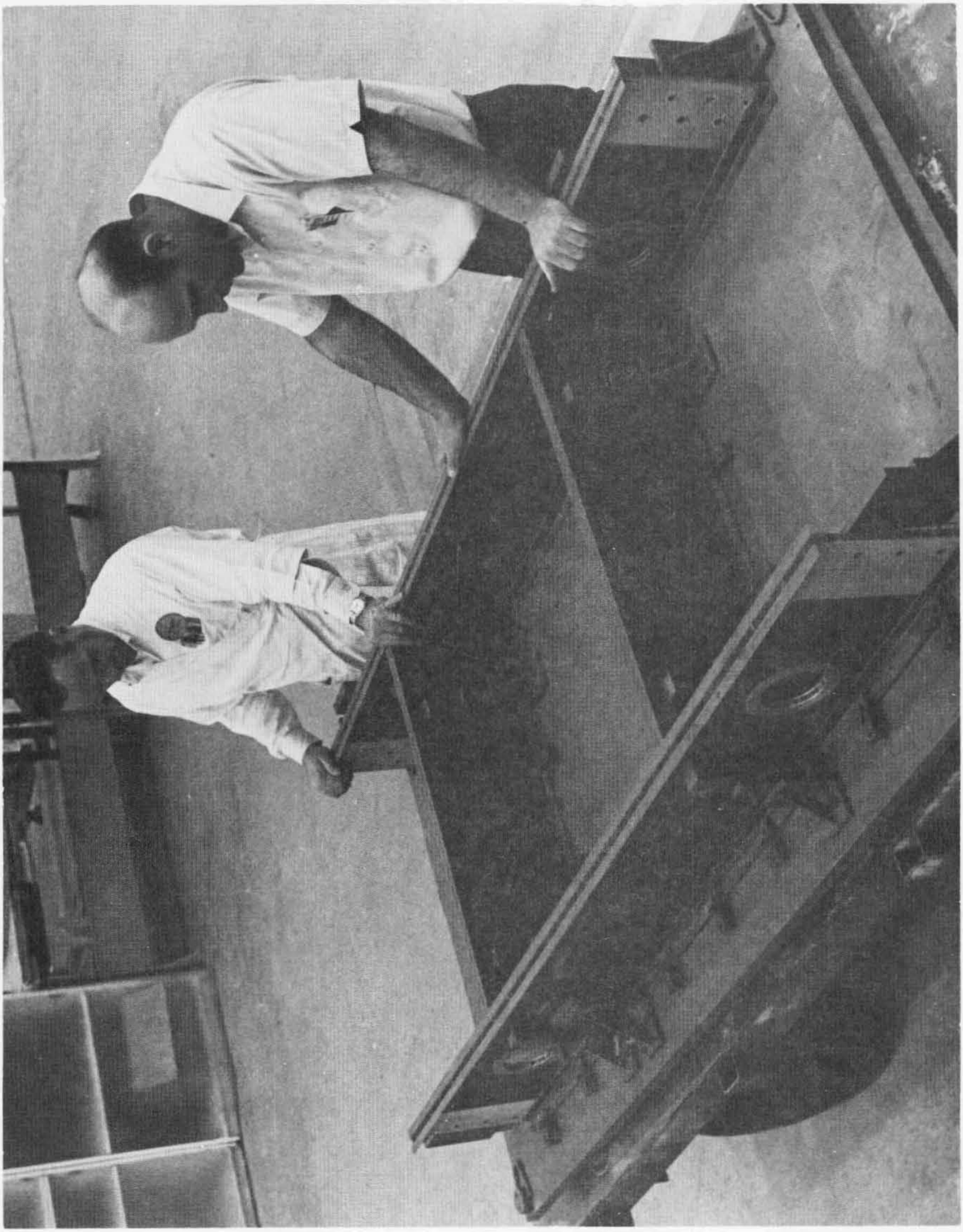
Where drilling was required for structural test mounting, it was accomplished in sub assembly. Since this only occurred in laminated titanium at the ends of the spars and surface panels, conventional metal drilling tools were satisfactory. The drilling of the holes around the structural door which involved both boron and glass composites was accomplished using high speed drills followed by clean-up with a reamer.

All bonds were accomplished using FM 34 film adhesive where the prefit, application and cure was accomplished in accordance with paragraph 3.1.9. Cleaning of all details was in accordance with paragraph 3.1.8.

The final assembly sequence was as follows. The spars and ribs were assembled as shown in Figure 45. Pressure was applied by controlled movement of the angles attached to the tool itself. The tongue of the tongue and groove joint was bonded to the assembly to establish the exact spar depth dimension over the entire beam. This is also shown in Figure 45.

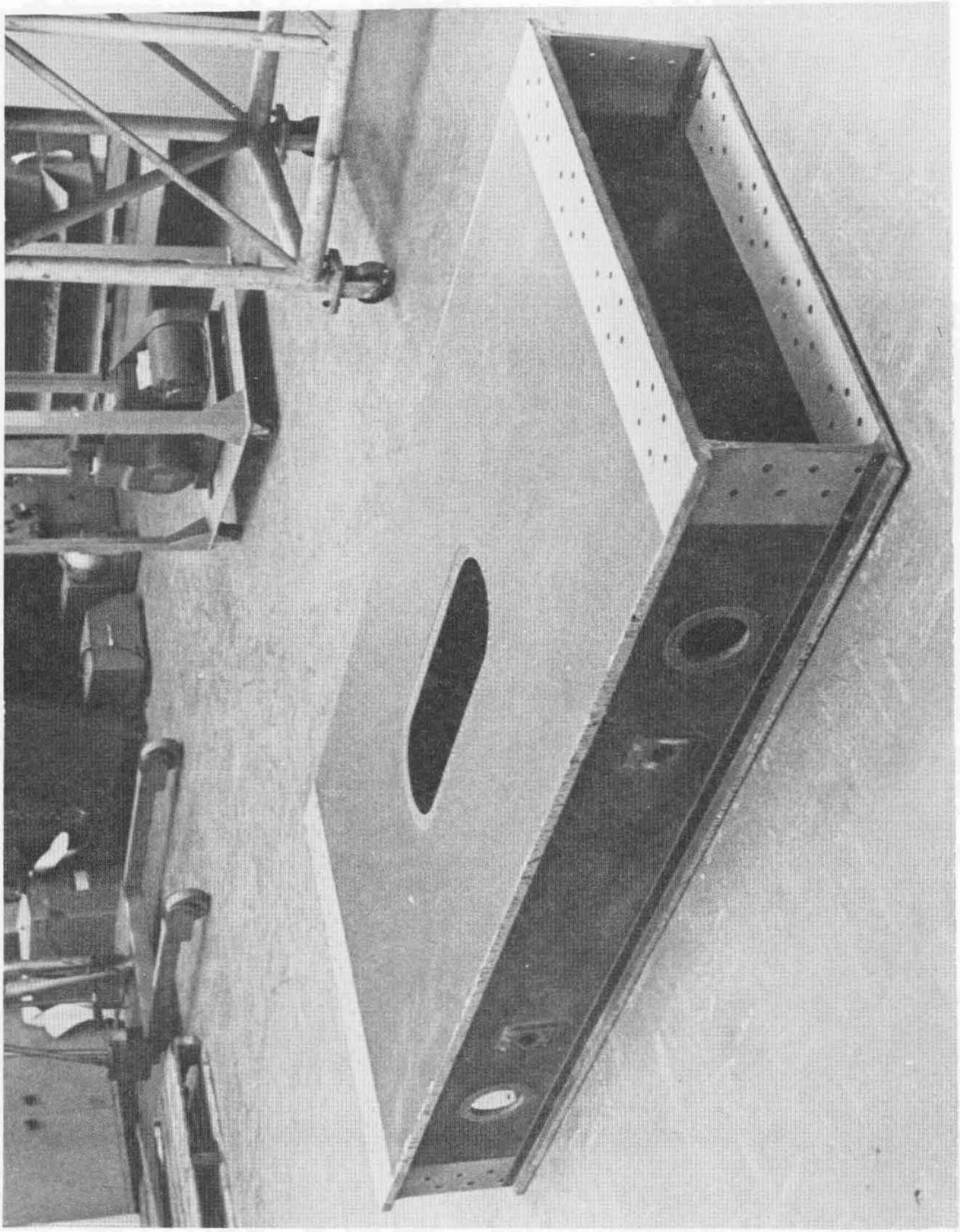
The upper surface panel, containing the opening for the structural door, was then bonded to the internal structure followed by the bonding of the lower surface. Figures 46, 47 and 48 show, respectively, the completed beam without the door, the door in relation to its opening and the completed beam.

The final operation to complete the beam for testing was to ream the holes in the spar bearing inserts to final dimensions for insertion of the bearing. With the accomplishment of this effort the box was ready for instrumentation and test.



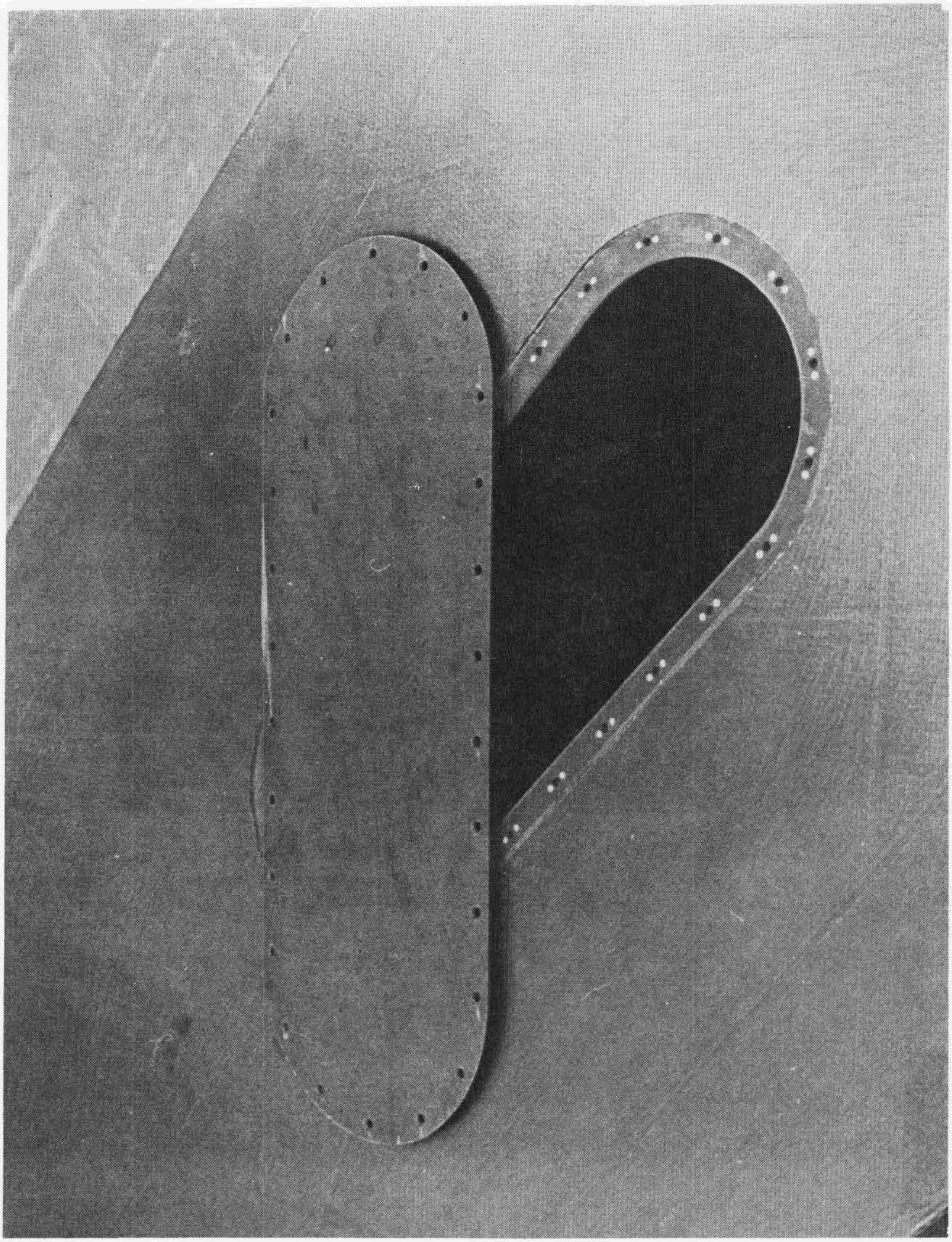
Assembly - Internal Structure

Figure 45



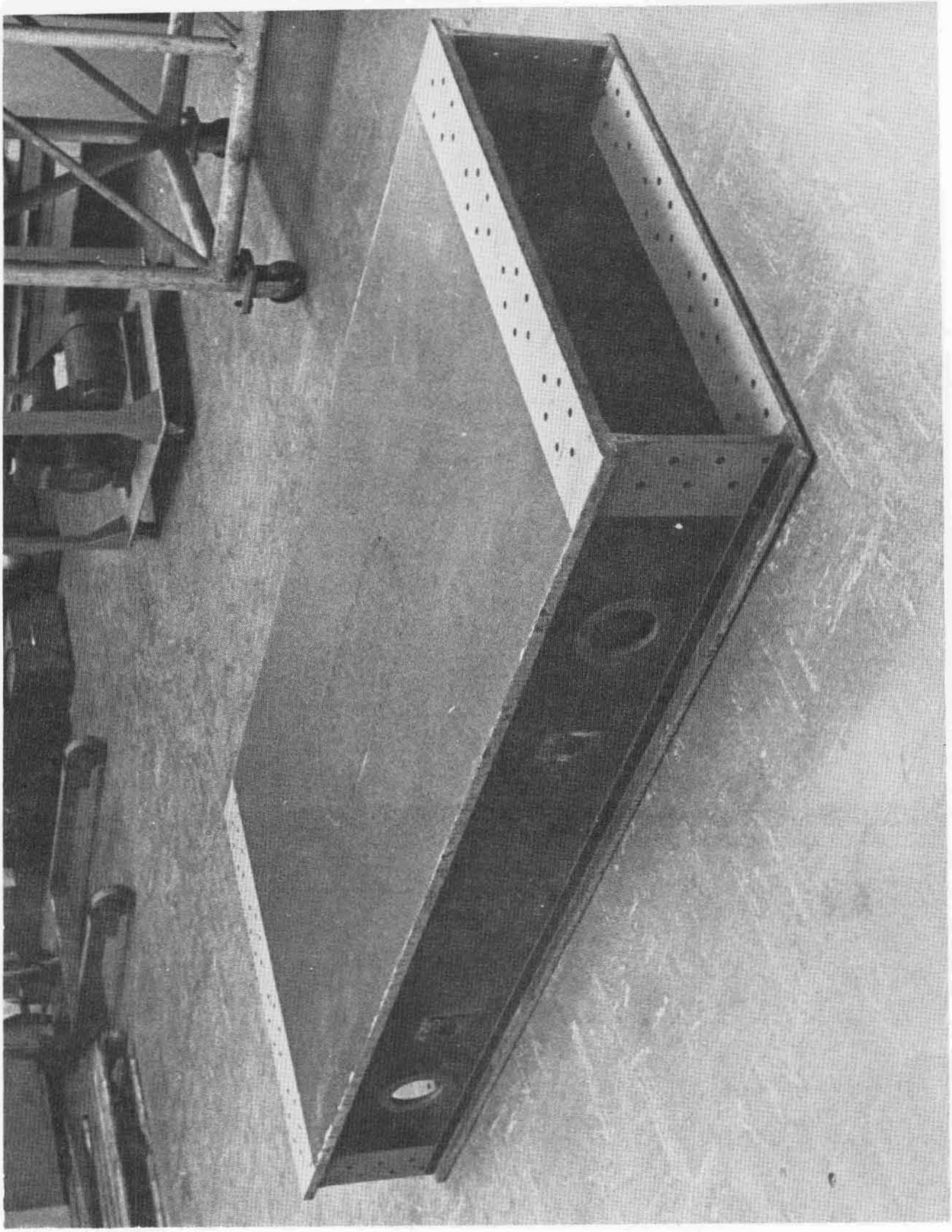
Completed Beam Without Structural Door

Figure 46



Upper Surface Panel Opening and Structural Door

Figure 47



Completed Box Beam

Figure 48

Section VII

Boron Polyimide Box Beam Test

7.1 Introduction

The test on the OV-10A center wing section boron polyimide composite box was conducted to demonstrate the structural integrity of the assembly when subjected to external loads in a thermal environment.

7.2 Test Conditions

The test loads and thermal environment as derived from test data were as follows:

Ultimate External Loads: (Bending) M_x = +1,890,000"# Up
(Torque) M_y = + 396,000"# Nose Up
(Shear) V = + 12,750 # Up

Thermal Environment: 650°F on upper and lower skins

7.3 Summary

The boron polyimide box beam test was conducted in three phases. The first test run was conducted by applying up bending (M_x) only to 75% of design limit load at room temperature. The second run consisted of full external loads (Mx, M_y, V) to 75% of design limit load under room temperature. The final test run was conducted by applying heat of 650°F to the skin surfaces of the test section and then applying full external loads until failure occurred at 83% of design limit load. The failure originated in the bond between the front spar cap and the upper skin on the L/H side. Figures 49 thru 54 show the failure of the test specimen.

During the first two runs at room temperature, strains and deflections were recorded in 10% increments of design limit load (Ref. Figures 55 and 56 for location of strain and deflection gages respectively and Tables 17 and 18 for tabulated data). Run No. three was conducted by obtaining a zero at room temperature; applying heat to 650°F; stabilize for five minutes; read out on strain gages, deflection gages, and monitor thermocouples before applying combined external loads to failure with another readout on all instrumentation at 50% of design limit load. (Ref. Table 19) for tabulated strains, Figure 57 for deflection data, and Figure 58 for thermocouple locations and tabulation of final recorded temperatures). A plot of the monitored strain, axial gage "A1" for compression and axial gage "A4" for tension, is shown as Figure 59

7.4 Test Setup

The test setup for the boron polyimide box beam was basically the same as for the previously tested boron epoxy box beam except that temperature equipment was added. Six banks of lamps, each consisting of six ALT8-612 reflector units with a proportionate number of 1000 T3CL quartz lamps, were positioned at 6 inches from each skin surface to obtain uniform heating of the skins. (Ref. Figure 60 for the heating arrangement)

The external load test setup consisted of installation of the assembly in steel fixtures with reactions at 16.875 from centerline both left and right on front and rear spars (total of four spherical bearing reaction points). The shear, moment, and torque were applied by hydraulic struts thru universal clevises and whiffletree linkage as shown on Figures 61 thru 66 . Pressure was supplied to the hydraulic struts by a hydro-pump and the loads were proportioned by an Edison machine. A dump switch is always used on the Edison machine for hydraulic pressure. For this test a master deadman switch was incorporated which instantaneously dumped both the applied mechanical loads (bending and torsion) plus the thermal input. The dead weight balance was independent.

Instrumentation consisted of micro-measurement WK-05-250BP-120 high temperature axial strain gages and WK-05-250WR-120 high temperature rosette strain gages installed with M-bond 610 adhesive as shown on Figure 55. Sixteen deflection transducers were used with low thermal expansion nichrome wire as shown on Figure 56 Chromel alumel thermocouple wires were installed to control the temperature through Research, Inc. Ignitron units and several were installed for monitor read-out as shown on Figure 58. . All instrumentation was permanently recorded on Gilmore data plotters, allowing instant, visible, and continuous checks on linearity.

BORON POLYIMIDE BOX BEAM TEST
75% DLL DRY RUN
NO TEMPERATURE

Gage No.	% D.L.L.	Bending Only		Bending & Torsion	
		50	75	50	75
A-1		-960	-1440	-1280	-1930
A-2		+790	+1190	+1040	+1530
A-3	(Door)	-320	- 490	- 300	- 520
A-4		+860	+1300	+ 930	+1420
A-5		-970	-1440	-1060	-1570
A-6		+770	+1180	+ 810	+1210
R-1	(Outb'd)	-850	-1290	-1020	-1560
R-1	(45°)	- 20	- 20	+ 150	+ 250
R-1	(Aft)	+850	-----	+ 920	-----
R-2	(Outb'd)	+760	+1160	+ 900	+1370
R-2	(45°)	- 30	- 50	- 220	- 340
R-2	(Aft)	-690	-1030	- 810	-1230
R-3	(Outb'd)	-720	-1120	- 780	-1200
R-3	(45°)	-110	- 180	- 10	- 20
R-3	(Aft)	+430	+ 690	+ 400	+ 650
R-4	(Outb'd)	+830	+1250	+ 920	+1380
R-4	(45°)	+120	+ 160	0	0
R-4	(Aft)	-660	-1000	- 690	-1050
R-5	(Outb'd)	-790	-1200	- 660	-1170
R-5	(45°)	+130	+ 330	+ 310	+ 400
R-5	(Aft)	+880	+1380	+ 760	+1330
R-6	(Outb'd)	+660	+1150	+ 810	+1240
R-6	(45°)	+ 70	+ 140	+ 90	+ 100
R-6	(Aft)	-700	- 990	- 640	-1000
R-7	(Inb'd)	- 10	- 20	+ 60	+ 90
R-7	(45°)	- 20	- 30	+ 10	+ 20
R-7	(Up)	- 10	- 10	+ 80	+ 110
R-8	(Outb'd)	- 20	- 40	+ 200	+ 260
R-8	(45°)	- 50	- 70	+ 500	+ 820
R-8	(Up)	- 10	- 10	0	+ 40
R-9	(Inb'd)	- 20	- 30	- 50	- 70
R-9	(45°)	- 50	- 80	- 780	-1200
R-9	(Up)	+ 20	+ 30	+ 40	+ 70
R-10	(45° Dwn)	- 70	- 100	- 30	- 50
R-10	(Inb'd)	-340	- 490	- 70	- 30
R-10	(45° Up)	+ 30	+ 50	+ 530	+ 830

BORON POLYIMIDE BOX BEAM TEST
75% DLL DRY RUN
NO TEMPERATURE

(Continued)

Gage No.	% D.L.L.	Bending Only		Bending & Torsion	
		50	75	50	75
R-11	(Outb'd)	-200	---	+ 50	---
R-11	(45°)	+ 30	+ 80	+ 800	+1170
R-11	(Up)	+180	+ 260	- 110	- 130
R-12	(Outb'd)	+ 20	+ 30	- 70	- 100
R-12	(45°)	+ 70	+ 100	+ 760	+1150
R-12	(Up)	- 50	- 70	+ 70	+ 100
R-13	(Outb'd)	+190	+ 260	+ 180	+ 240
R-13	(45°)	- 10	- 20	- 710	-1120
R-13	(Dwn)	-170	- 250	- 160	- 200

NOTE:

1. Refer to Figure 55 for gage locations.

BORON POLYIMIDE BOX BEAM TEST
DEFLECTION DATA

*Extrapolated from Previous Readings

Gage No. \ % DLL	Room Temperature Run				Load - Temp Run		
	Bending Only		Bending & Torsion		Temperature = 650°F		
	50% (In)	75% (In)	50% (In)	75% (In)	0 (In)	50% (In)	83% (In)*
1*	0	0	0	0	+.002	+.002	+.002
2*	0	0	+.002	+.004	0	+.002	+.003
3	+.080	+.122	+.146	+.230	+.002	+.176	+.288
4	+.066	+.112	+.064	+.106	-.002	+.058	+.098
5	0	0	+.008	+.014	0	+.010	+.017
6	0	0	-.006	-.012	0	-.004	-.007
7	-.002	-.002	0	0	+.002	+.004	+.005
8	0	0	-.004	-.006	0	-.002	-.003
9	-.002	-.010	-.002	0	-.010	-.016	-.020
10	-.016	-.026	-.020	-.030	0	-.026	-.043
11	-.008	-.012	-.002	0	0	0	0
12	0	-.002	-.006	-.016	-.002	-.010	-.015
13	-.006	-.006	+.006	+.014	0	+.004	+.007
14	-.010	-.014	0	0	+.002	-.004	-.008
15	+.080	+.124	+.156	+.240	+.002	+.168	+.278
16	+.066	+.114	+.074	+.120	+.010	+.098	+.156

NOTE:

* Gages 1 and 2 were located on the reaction jig at 1.5" from front spar centerline.

1, + Deflection is up

2, Refer to Figure 56 for gage locations.

BORON POLYIMIDE BOX BEAM TEST
FINAL RUN
COMBINED LOADS AND TEMPERATURE

*Extrapolated From Previous Readings

Gage No.	% DLL	Strain (μ ″/″)			Actual Temp	Corrected Strain (μ ″/″)		
		650°F 0 Load	650°F 50% Load	650°F 83% Load*		650°F 0 Load	650°F 50% Load	650°F 83% Load*
A-1		-1880	-3110	-3920	601	-1600	-2830	-3630
A-2		-1880	- 670	+ 130	627	-1540	- 330	+ 470
A-3	(Door)	-1470	-1850	-2100	610	-1160	-1540	-1790
A-4		-1910	- 800	- 70	666	-1510	- 400	+ 330
A-5		-1910	-2890	-3540	601	-1630	-2610	-3260
A-6		-1760	- 810	- 350	627	-1440	- 490	- 30
R-1	(Outb'd)	-1340	-2240	-2840	588	-1060	-1960	-2560
R-1	(45°)	-----	-----	-----	588	-----	-----	-----
R-1	(Aft)	- 470	+1030	+2030	588	- 190	+1310	+2310
R-2	(Outb'd)	-1760	- 600	+ 160	623	-1420	- 260	+ 500
R-2	(45°)	- 930	-1290	-1530	623	- 590	- 950	-1190
R-2	(Aft)	- 630	-1600	-2240	623	- 290	-1260	-1900
R-3	(Outb'd)	-1210	-2100	-2690	610	- 910	-1800	-2390
R-3	(45°)	-1260	-1100	- 990	610	- 960	- 800	- 690
R-3	(Aft)	-1230	- 410	+ 160	610	- 930	- 110	+ 460
R-4	(Outb'd)	- 570	+ 410	+1060	653	- 280	+ 790	+1440
R-4	(45°)	-----	-----	-----	653	-----	-----	-----
R-4	(Aft)	-3050	-4980	-6250	653	-2670	-4600	-5870
R-5	(Outb'd)	-1580	-2580	-3240	588	-1300	-2300	-2960
R-5	(45°)	-1080	- 780	- 580	588	- 800	- 500	- 300
R-5	(Aft)	- 600	+ 500	+1230	588	- 320	+ 780	+1510
R-6	(Outb'd)	-1620	- 480	+ 270	623	-1280	- 140	+ 710
R-6	(45°)	-1240	-1530	-1720	623	- 900	-1190	-1380
R-6	(Aft)	- 750	-1480	-1960	623	- 410	-1140	-1620
R-7	(Inb'd)	- 240	- 190	- 160	250	- 340	- 290	- 260
R-7	(45°)	- 260	- 290	- 310	250	- 360	- 390	- 410
R-7	(Up)	- 280	- 190	- 130	250	- 380	- 290	- 230
R-8	(Outb'd)	+2000	+2410	+2680	273	+1900	+2310	+2580
R-8	(45°)	+ 180	+ 780	+1180	273	+ 80	+ 680	+1080
R-8	(Up)	- 280	- 190	- 120	273	- 380	- 290	- 220
R-9	(Inb'd)	+ 610	+ 610	+ 610	273	+ 510	+ 510	+ 510
R-9	(45°)	+ 110	-1180	-2030	273	+ 10	-1280	-2130
R-9	(Up)	- 360	- 310	- 280	273	- 460	- 410	- 380

BORON POLYIMIDE BOX BEAM TEST
FINAL RUN
COMBINED LOADS AND TEMPERATURE (Cont'd)

*Extrapolated From Previous Readings

Gage No.	% DLL	Strain (μ"/")				Corrected Strain (μ"/")		
		650°F 0 Load	650°F 50% Load	650°F 83% Load*	Actual Temp	650°F 0 Load	650°F 50% Load	650°F 83% Load*
R-10	(45°Dwn)	+ 70	+ 20	- 10	273	- 30	- 80	- 110
R-10	(Inb'd)	+1810	+1820	+1830	273	+1710	+1720	+1730
R-10	(45° Up)	- 130	+ 490	+ 900	273	- 230	+ 390	+ 800
R-11	(Outb'd)	+ 280	+ 310	+ 330	250	+ 180	+ 210	+ 230
R-11	(45°)	- 100	+ 710	+1250	250	- 200	+ 610	+1150
R-11	(Up)	-----	-----	-----	250	-----	-----	-----
R-12	(Outb'd)	+ 200	+ 80	0	250	+ 100	- 20	- 100
R-12	(45°)	+ 90	+ 830	+1320	250	- 10	+ 730	+1220
R-12	(Up)	+ 90	+ 200	+ 270	250	- 10	+ 100	+ 170
R-13	(Outb'd)	+ 200	+ 330	+ 420	250	+ 100	+ 230	+ 320
R-13	(45°)	+ 10	- 790	-1290	250	- 90	- 890	-1390
R-13	(Dwn)	- 390	- 570	- 690	250	- 490	- 670	- 790

NOTE:

1. Refer to Figure 55 for gage locations.

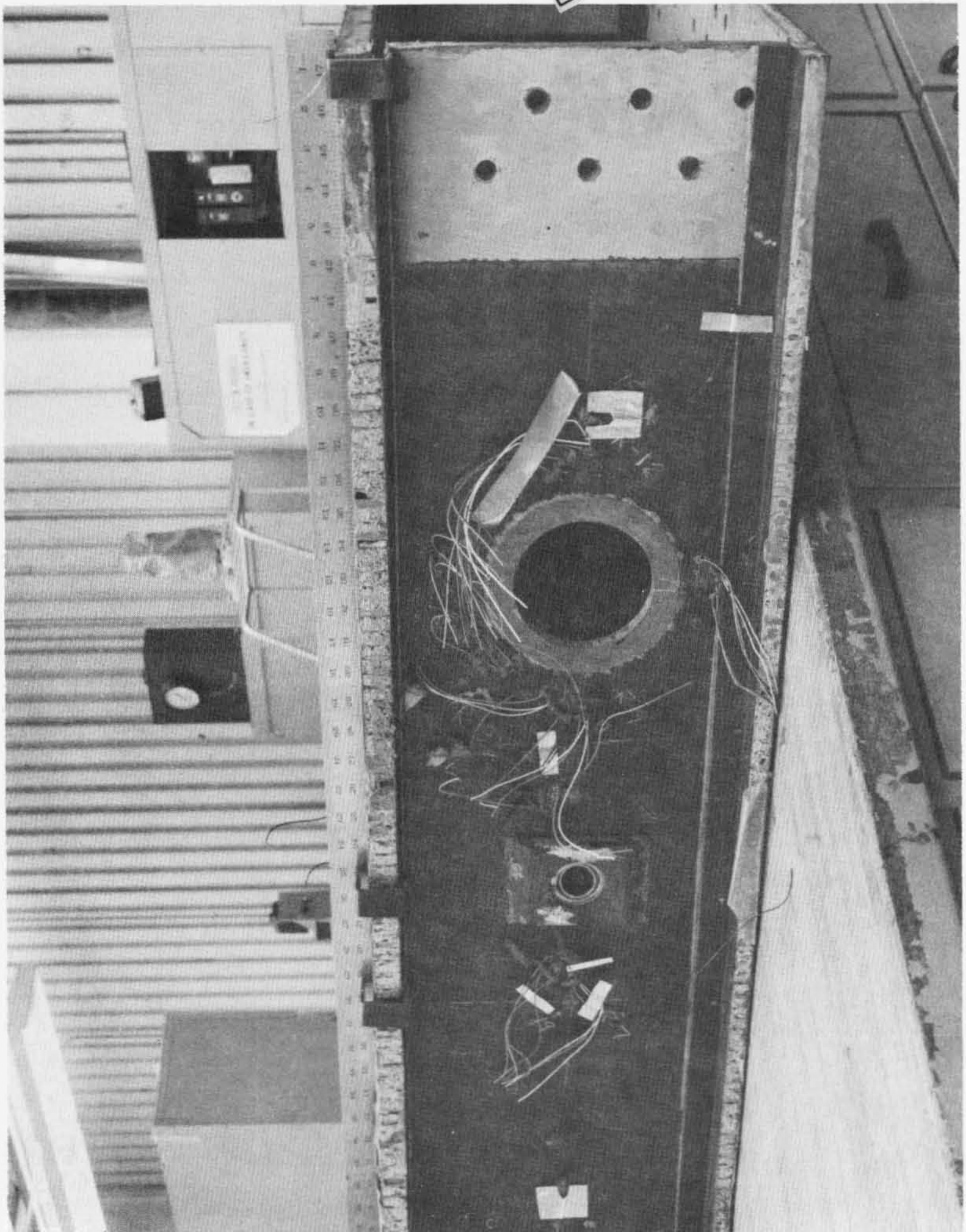


Figure 49 Failed Specimen

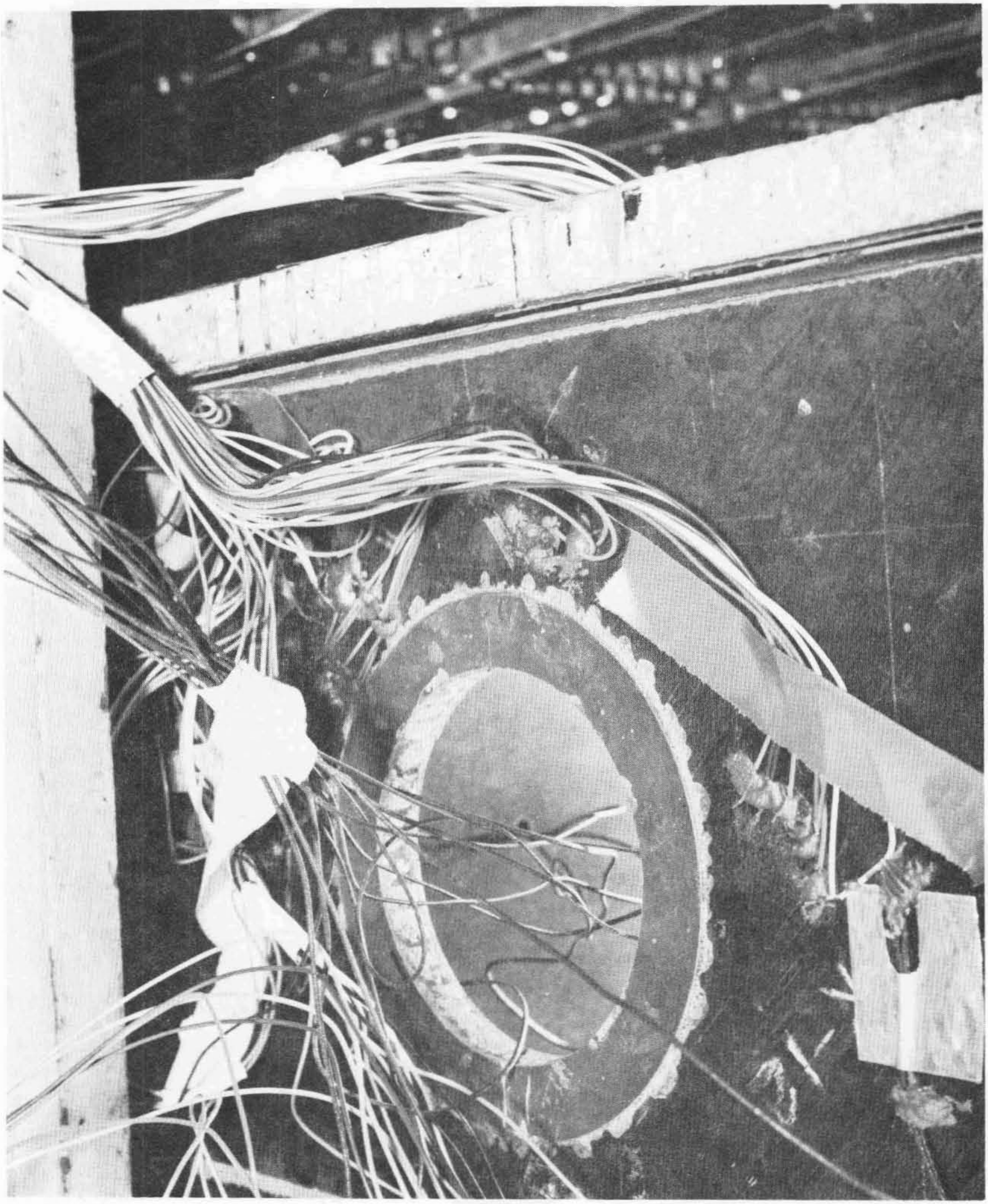


Figure 50 Close-Up Failed Specimen in Test Fixture

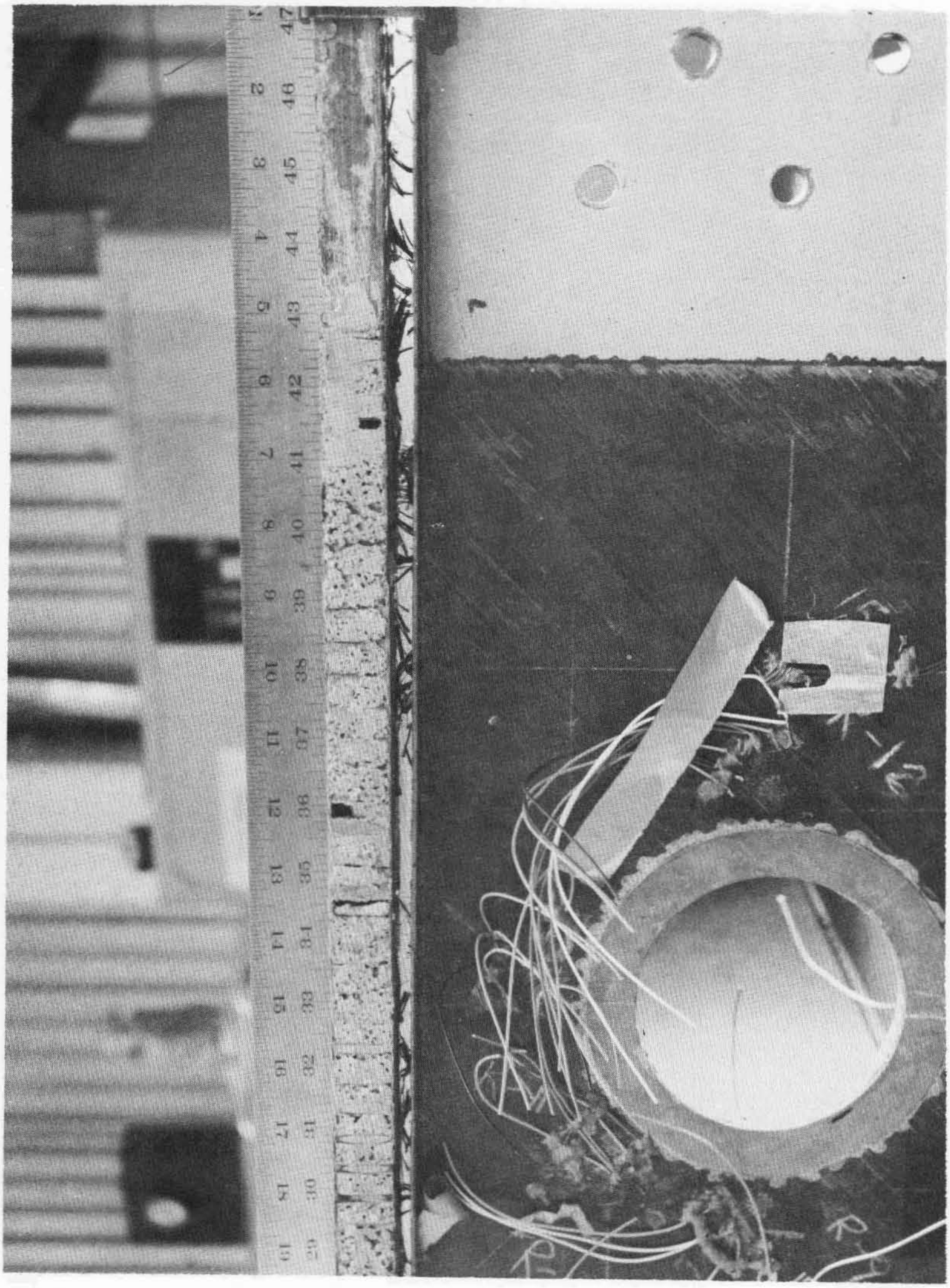


Figure 51 Close-Up of Spar to Facing Failure

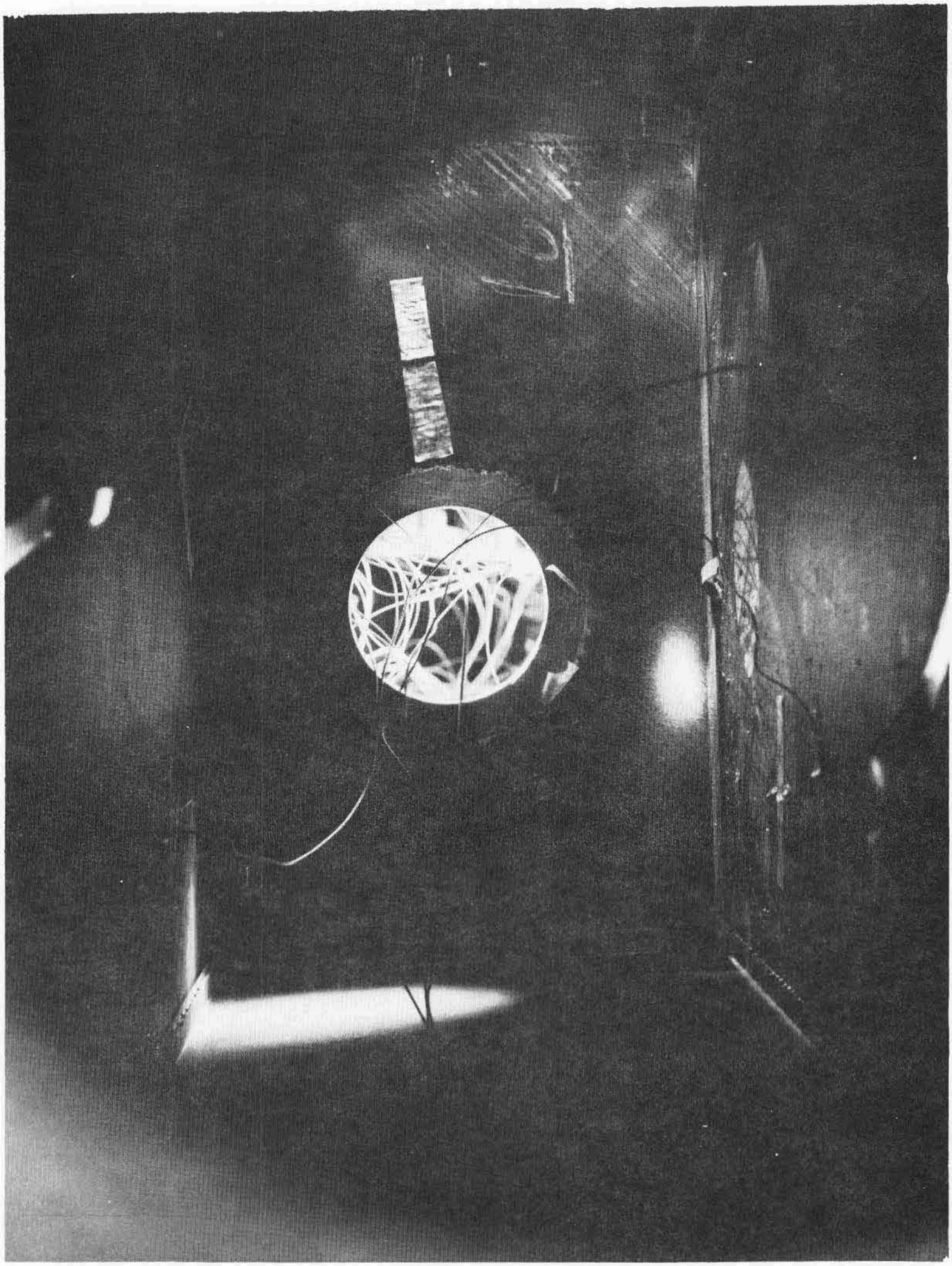


Figure 52 Component Spar, Rib, Facing, Joint Failure

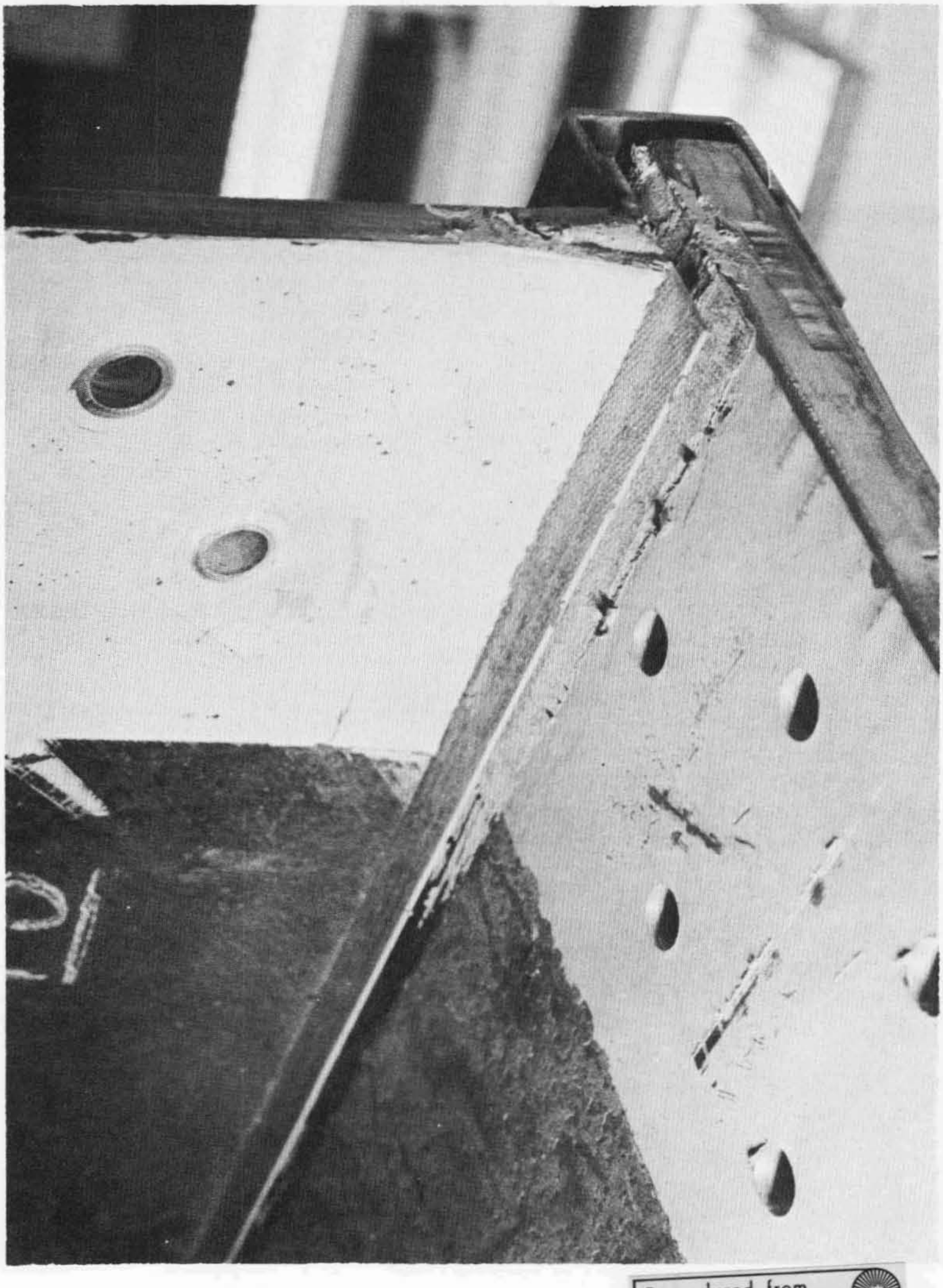


Figure 53 Close-Up of Spar to Facing Failure

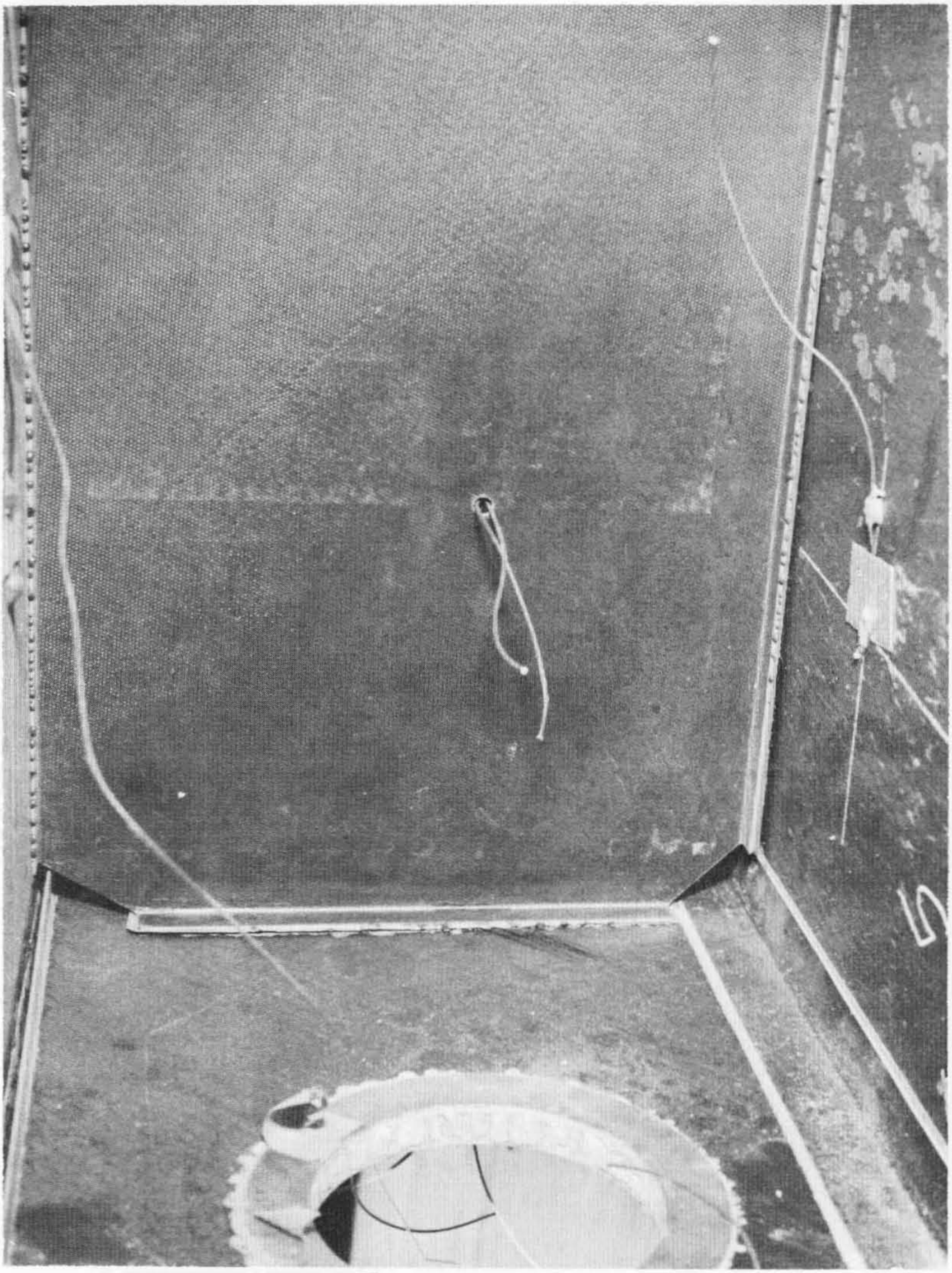
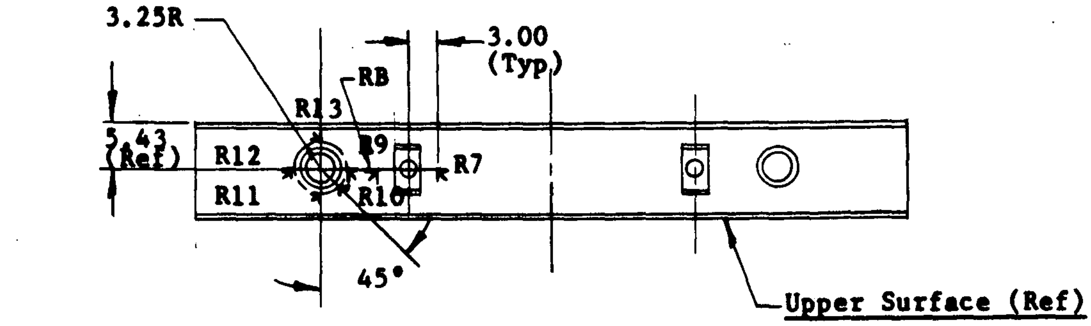
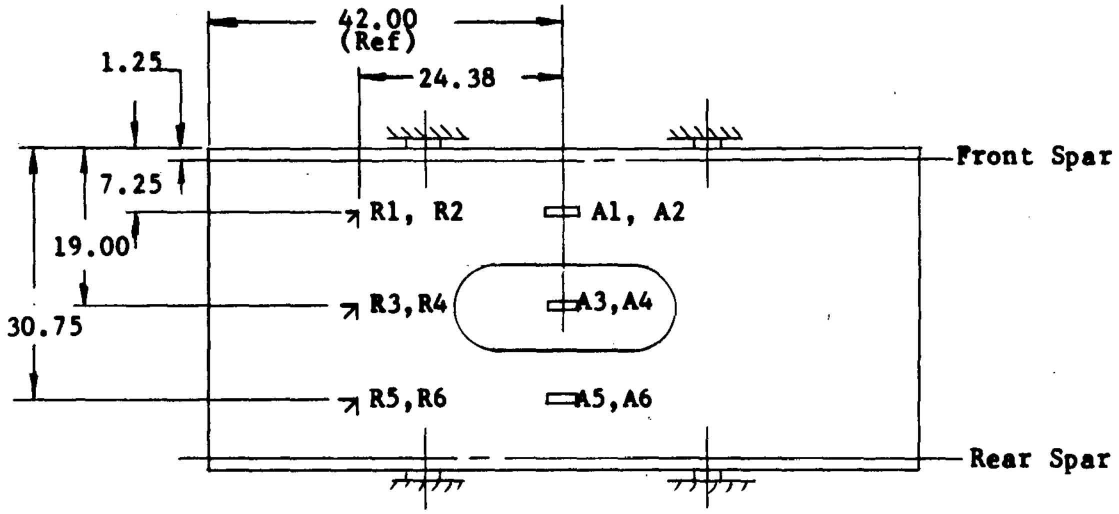


Figure 54 Close-Up of Spar, Rib, Facing Joint Failure



View Front Spar

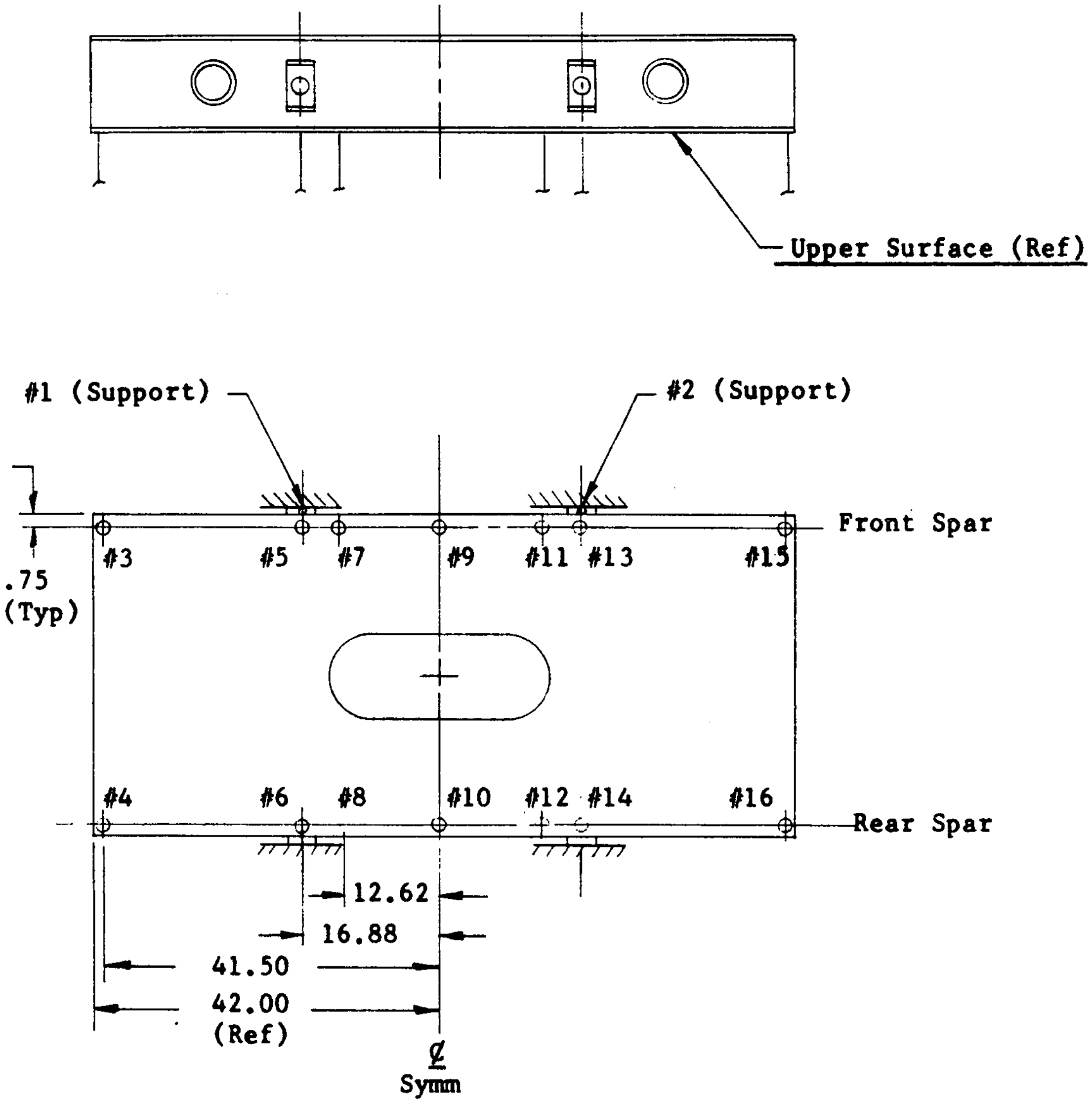


Plan View (Upper Surface)

NOTE:

1. Rosette Gages are designated by "R" and axial gages by "A".
2. Odd numbered gages are on upper surface and even numbers on the lower surface.

Figure 55 Boron Polyimide Box Beam Test Strain Gage Locations



NOTE: Refer to Table 18 for tabulated data

Figure 56 Boron Polyimide Box Beam Test Deflection Gage Locations

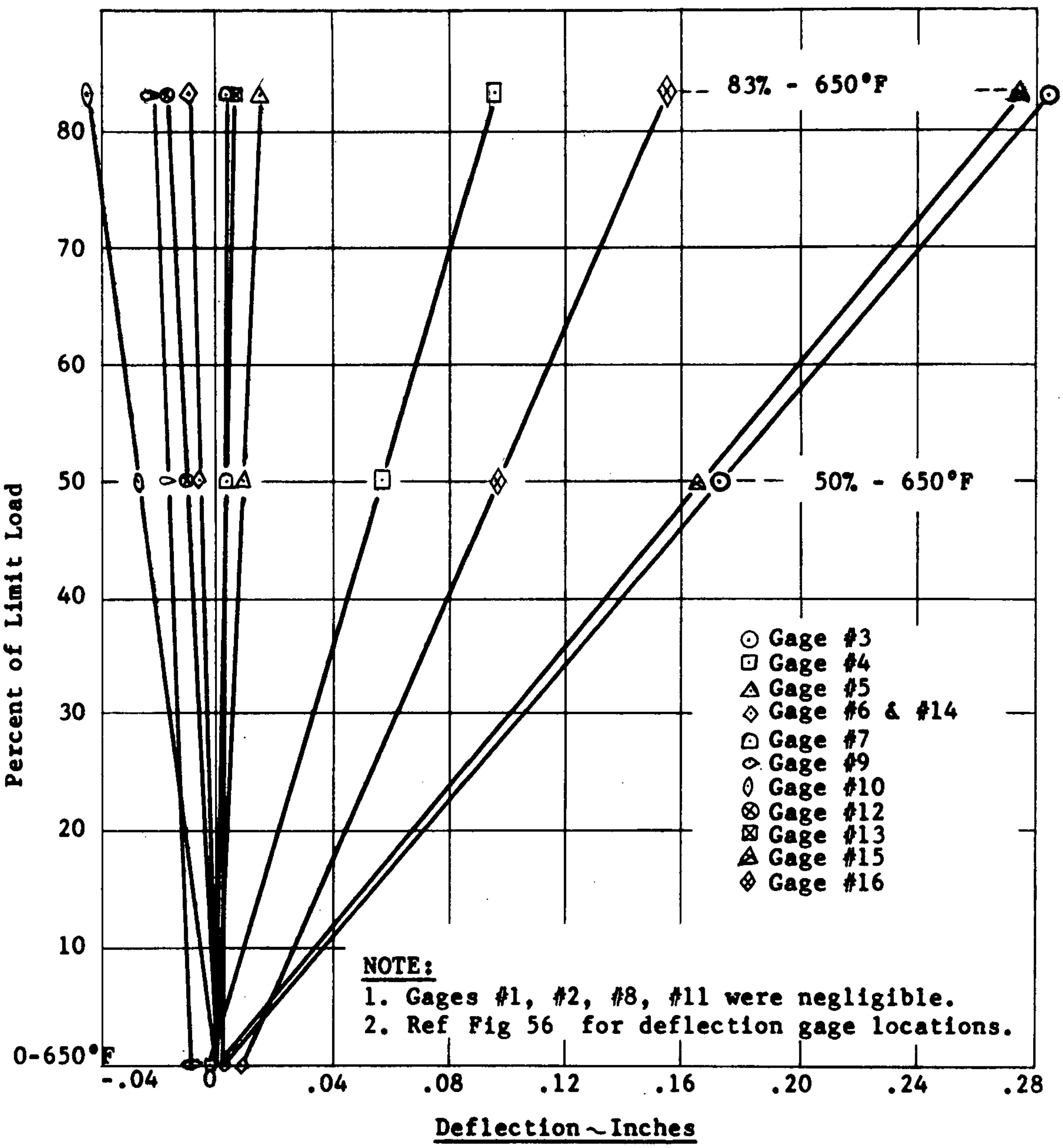
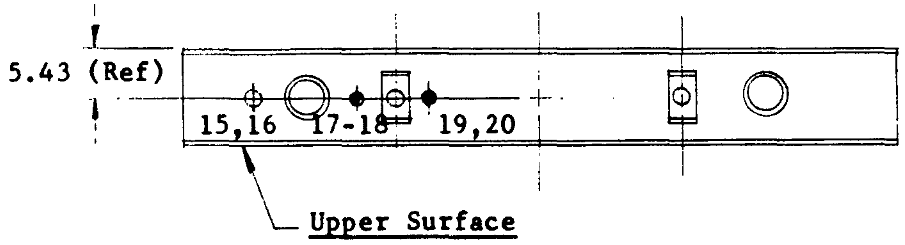
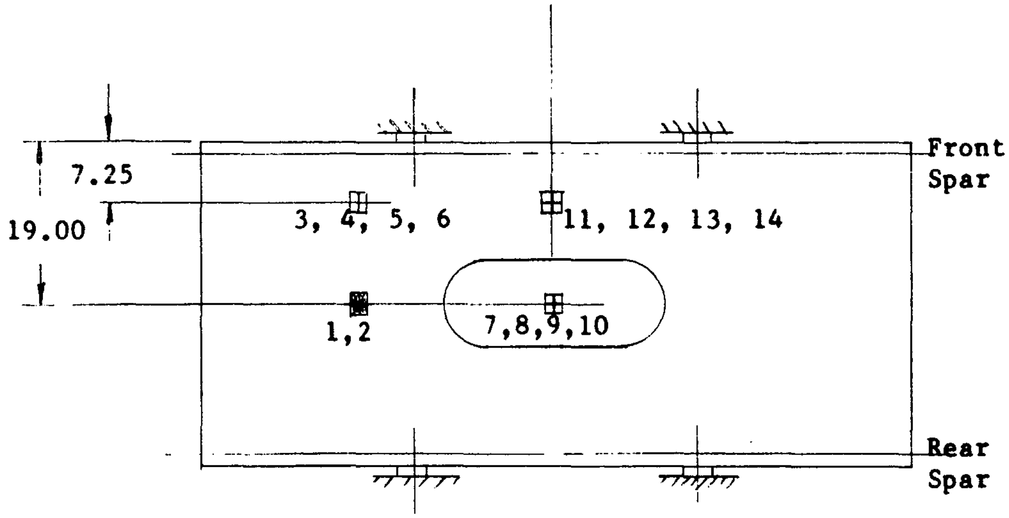


Figure 57 Boron Polyimide Box Beam Deflection Vs. Per Cent Limit Load and Temperature Run



View Front Spar



Plan View Upper Surface

Thermocouple No.	Temp. (Final) °F
1	610
2	653
3	588
4	310
5	421
6	623
7	614
8	456
9	441
10	666
11	601
12	367
13	435
14	627
15	250
16	250
17	273
18	250
19	250
20	250

NOTE:

Located inside and outside of both skins

Outer surface only of upper and lower skins

Inside and outside surface of front spar only

Outside surface only of front and rear spars

Thermocouple number sequence - upper to lower skins and front to rear spar

Figure 58 Boron Polyimide Box Beam Test Temperature Data

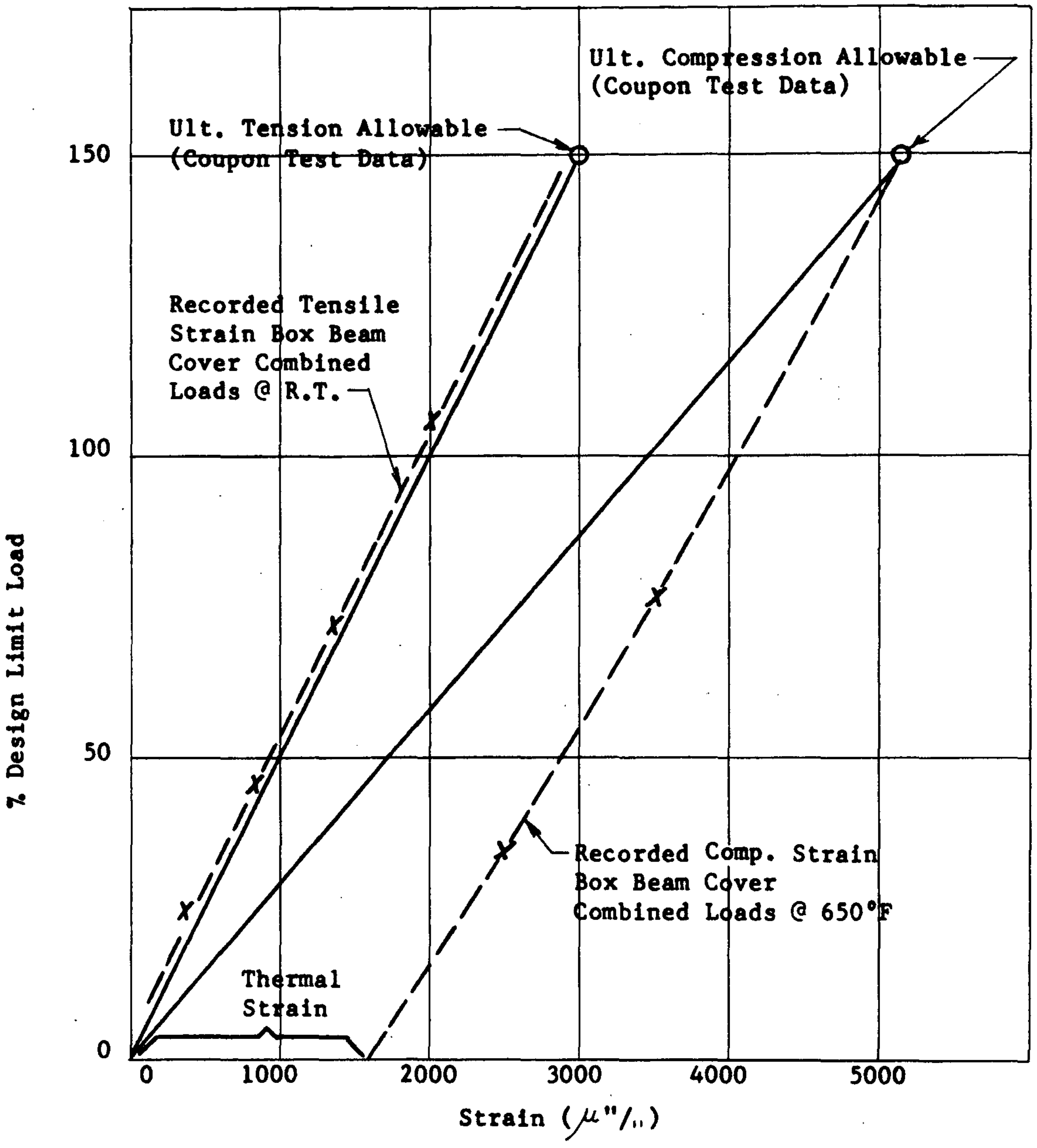
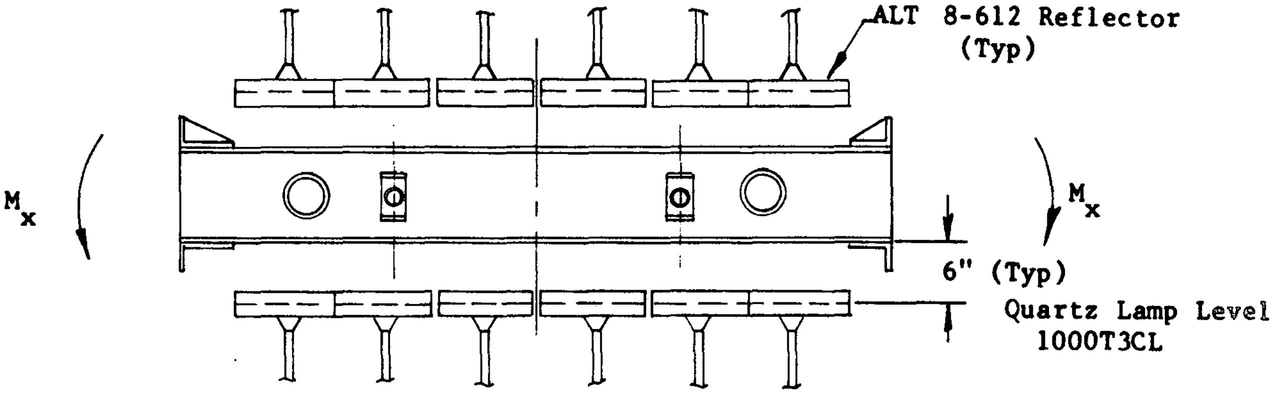
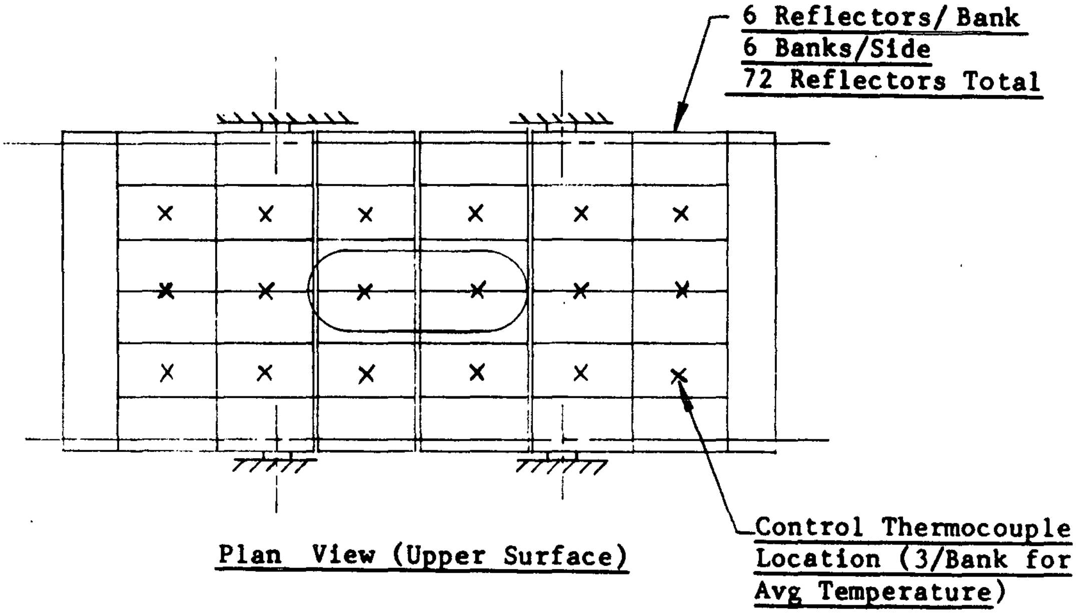


Figure 59 Monitored Strain Gage Data



View Front Spar



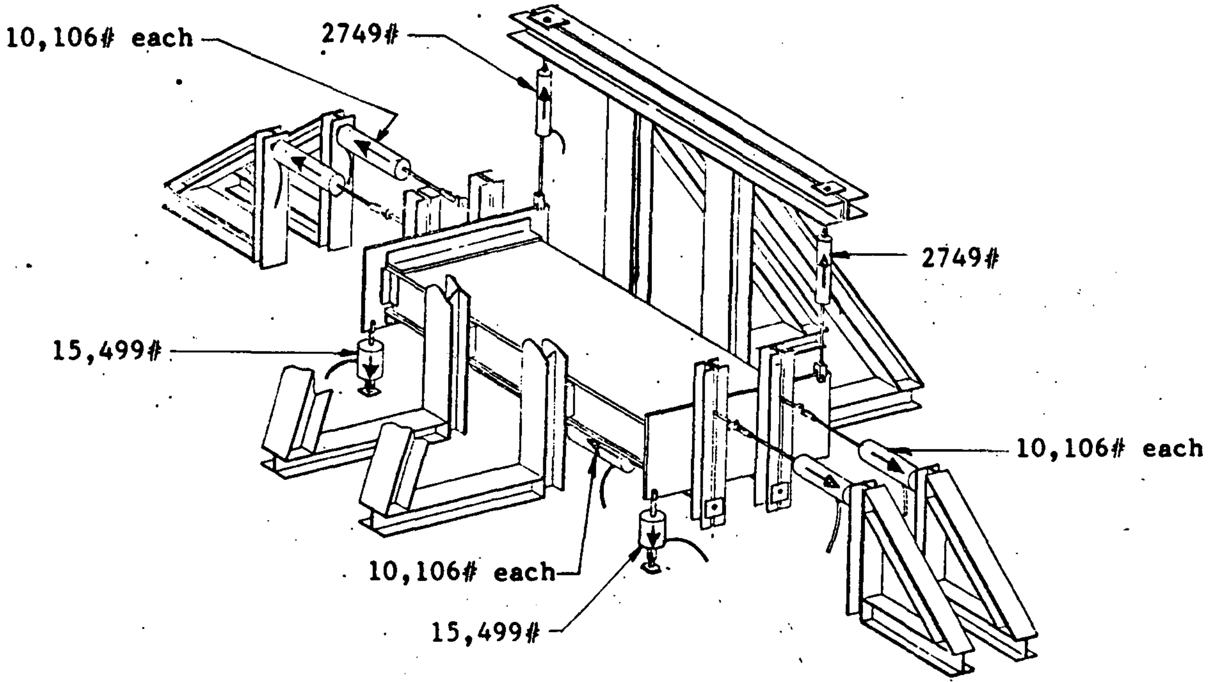
Plan View (Upper Surface)

NOTE:

X Indicates Location of Control Thermocouples Total of 192 1000 T3CL Quartz Lamps used with 72 ALT 8-612 Reflector Units

Figure 60 Boron Polyimide Box Beam Test Heating Arrangement

Figure 61 Structural Test Setup
Boron-Polyimide Box Beam
Ultimate Loads Shown = 150% Design Limit Load



M_x = +1,890,000"# Up Bending

M_y = + 396,000"# Nose Up Torque

V = + 12,750# Up

<u>NOTE:</u>

<u>BOX BEAM WAS INVERTED FOR SIMPLICITY OF TESTING (UPPER SURFACE FACING DOWN)</u>

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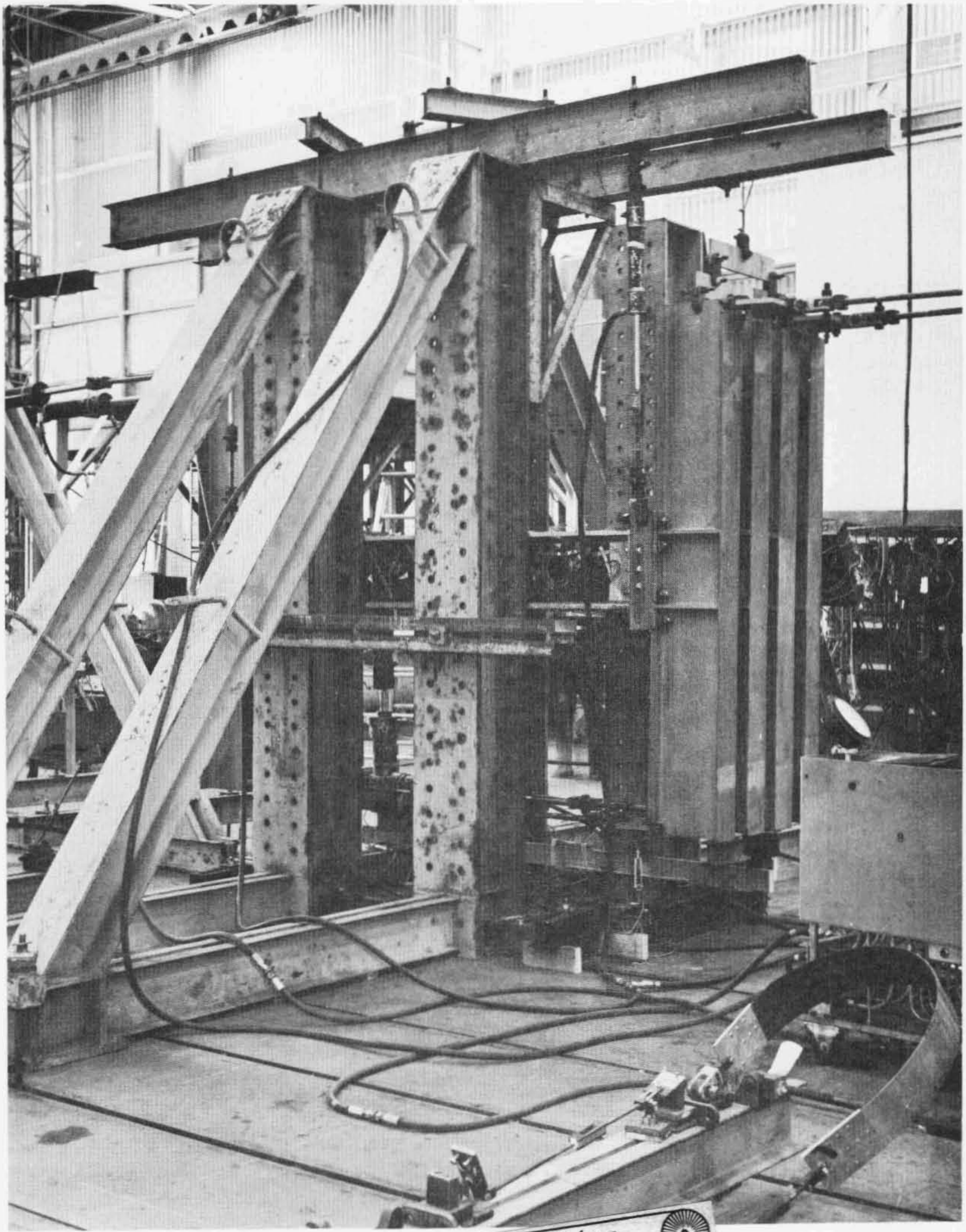


Figure 64 Test Set Up Looking Forward - Room Temperature

Figure 65 Test Set Up - Tension Cover

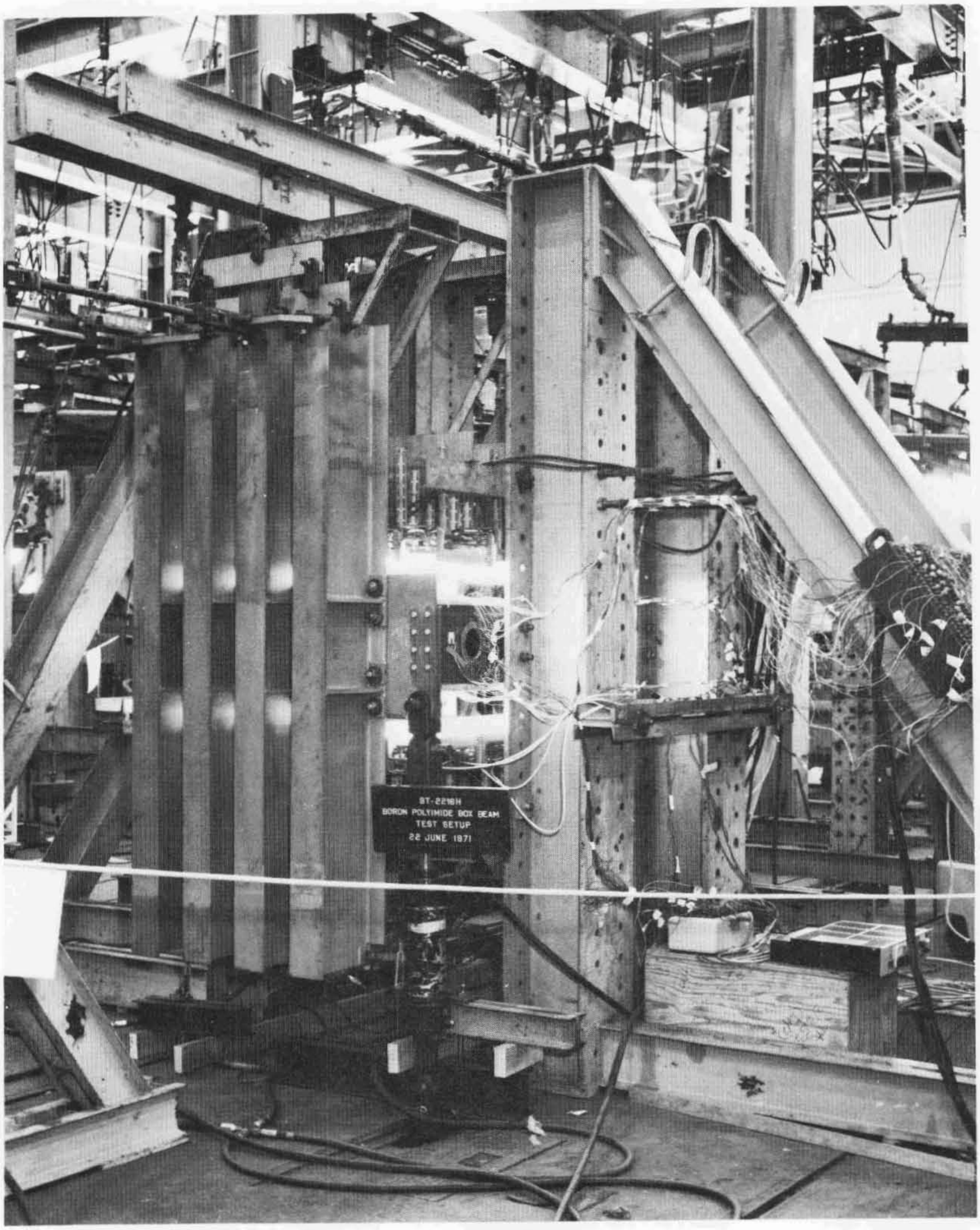


Figure 66 Test Set Up - Looking Aft 650°F

Section VIII

Conclusions and Recommendations

8.1 Conclusions

Techniques have been developed by which boron-polyimide prepregs can be reprocessed to permit fabrication by conventional vacuum bag techniques using a special bleeder system.

The processing technique cannot be considered a production process since it must be modified as the amount of high boiling solvent varies.

Post cure cycles have been developed which eliminate the low physical properties normally associated with polyimide resins when tested at an elevated temperature after short time (1/2 hour) exposure at that temperature. The maximum operating temperature of the Pyralin 4707 system is 650°F for relative, short time operation (up to 16 hours).

The smooth transition of load from the composite to adjacent structure has been successfully accomplished using a titanium foil interleaving technique. Machining and drilling is greatly simplified by this method since drilling is accomplished only through laminated titanium foil.

Design optimization methods are currently available for designing large complex primary structure utilizing oriented boron-polyimide.

The ultimate allowable tension and compression strains of the box beam test specimen cover skins were in agreement with the actual recorded strains when tested under combined loading at 650°F until premature failure at a secondary bond failure precluded completion of the structural test.

8.2 Recommendations

Studies should be conducted jointly by the resin suppliers and prepreggers to establish those controls required to obtain material of sufficient reproducibility and processability so that the fabricator need not additionally process the incoming material in order to achieve quality composites.

Controls should be developed whereby materials could be supplied for the various methods of fabrication such as vacuum bag, autoclave or positive pressure molding.

Effort should be expended to develop more ductile, (less brittle) high temperature adhesive formulations since the single high temperature resistant adhesive formulation is extremely brittle and process critical.

Appendix A

Process Specification

Boron-Polyimide Simulated OV-10A Center Wing Section

BORON-POLYIMIDE SIMULATED OV-10A CENTER WING SECTION

LIST OF CONTENTS

PARA. NO.	
1.	SCOPE
2.	APPLICABLE DOCUMENTS, AND MATERIALS
3.1	REQUIREMENTS
3.2	QUALITY

1. SCOPE

This specification outlines the materials and procedures to be employed in the fabrication of the boron-polyimide simulated OV-10A Center Wing Section.

2. APPLICABLE DOCUMENTS AND MATERIALS

Materials	Source
Boron-Polyimide Tape (35-520 prepreg)	Whittaker R&D
Polyimide Glass Prepreg (35-520)	DuPont
Polyimide Glass Prepreg (35-504)	DuPont
BR 34 Primer	American Cyanamid
Glass-Polyimide Molding Compound #HI-E-9020	Fiberite Corp.
181 Style Glass Fabric	Mil-C-9084
Mold Release-Freekote 33	Commercial
Adhesive - FM 34	American Cyanamid
Release Fabric (0.001 inch-permeable)	Taconic, Inc.
Cork Dam Material-Corprene DK 153	Armstrong Products
Polyvinyl Alcohol Film (0.008 inch)	Reynolds Metals Co.
Nylon Bag Film	Richmond Corp.
Carbon Paper (NKI 621-8 lbs.)	Interchemical Corp.

3. REQUIREMENTS

3.1 General Requirements

3.1.1 Material Certification

The materials shall be procured with certification for meeting the applicable specifications. Receiving Inspection tests shall be conducted upon material receipt to assure conformance to the specifications. In addition, recerticication of various materials shall be accomplished as listed herein.

3.1.1.1 Adhesives

Lap shear tests shall be conducted at room temperature within one week of use if storage in accordance with paragraph 3.1.2 is maintained or one day prior if storage history is questionable.

3.1.1.2 Boron Prepreg

Material shipments which are not consumed within 60 days but stored in accordance with paragraph 3.1.2 shall be retested in short beam shear to determine satisfactory conformance to the specification. This recertification shall be accomplished within 14 days prior to material use.

3.1.1.3 Polyimide-Glass Prepreg & Molding Compound

Material stored at 37 ±3°F or below need only be recertified after 6 months storage. Material stored at room temperature shall be recertified after 3 months.

3.1.2 Material Storage

3.1.2.1 Polyimide Prepregs

These materials stored at 37 ±3°F shall be retained in the original protective covering and shall be permitted to warm up to a temperature at least 5 degrees above the dew point in the working area prior to removal from the protective wrapping. Any moisture collected on the wrappings shall be removed prior to opening the package of the stored material. White gloves shall be used when handling the unprotected material to prevent contamination.

3.1.2.2 Glass Fabric

All glass fabric shall be stored in such a manner that it will not become contaminated with grease, oil, dust or other foreign substance.

3.1.2.3 Core Material

Honeycomb core must be stored in such a manner to prevent contamination and damage from stacking and handling.

3.1.2.4 Cured and Metallic Materials

Precured laminates, metallic materials and other details shall be stored in the "as molded" or as received condition. After preparation of the surfaces for bonding they shall be interleaved with Kraft paper and stored in such a manner as to prevent contamination from dirt, dust, oil and grease. Handling shall only be with clean white gloves.

3.1.3 Tool Preparation

The surface of the tool shall be examined and any foreign substance, on the molding surface, removed by lightly sanding. The surface shall then be cleaned with a clean rag moistened with methel ethyl ketone or acetone.

Freekote 33 Mold Release is sprayed (aerosol can) on the tool as a thin, box coat and allowed to air dry for 15 minutes prior to use. Excess material shall be wiped off. When processed without an elevated temperature bake, the release material must be reapplied for each part.

3.1.4 Lay-Up and Cure of Glass Molding Compound

The molding compounds have a bulk factor of approximately 12 (uncompacted) to 1 (molded). If the required volume has not been provided in the tool, a compaction procedure is as follows:

With press platens at 550°F, preheat tool to 400°F Place as much of the pre weighed molding compound as possible in the tool cavity. Close tool to stops. Open and add remainder of required compound. Close tool to stops and allow temperature to increase to 550 for 1 hour.

Allow to cool to 150°F before removal of pressure. Post cure, unrestrained, as follows

Room temperature to 575°F in 5 hours
Hold at 575 for 16 hours
Raise temperature to 650°F in 3 hours
Hold at 650 F for 1 hour
Allow to cool slowly in the post cure oven

3.1.5 Processing of Preimpregnated Boron

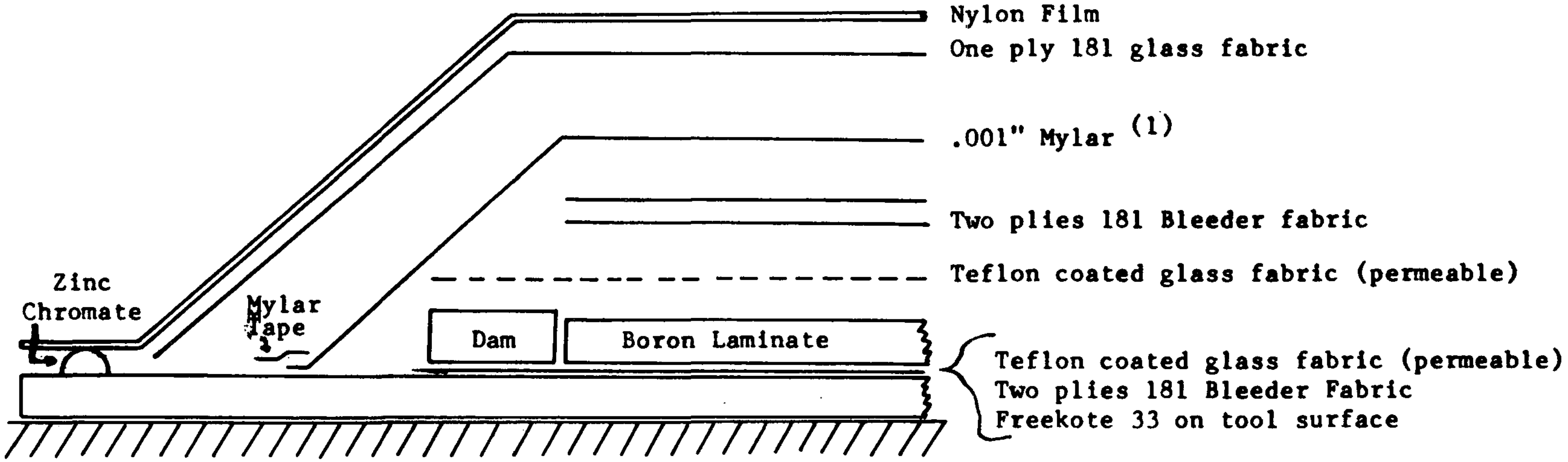
3.1.5.1 Processing of Boron Prepreg for NMP Extraction

Excess N-methylpyrrolidone (NMP) shall be removed from the prepreg by soaking in constantly over flowing bath of distilled water. The material shall be placed in the tank, allowed to soak four (4) minutes and removed from the water. Excess water shall be removed by placing the processed prepreg on clean, dry, cheesecloth which contains no grease, wax or other contaminating materials. The upper surface of the prepreg shall be blotted to remove water droplets using the same material.

3.1.5.2 Lay-Up and Cure of Prepreg

The boron tape shall be laid up to the blueprint orientation and thickness and bagged in accordance with the schematic shown in Figure 1. All boron tape shall be laid-up with the glass carrier side down and on completion of the lay-up shall be closed out with one ply of dry 104 style fabric. Full vacuum (25 inches Hg, min.) shall be drawn and the following cure cycle, based on thermocouple readings in the laminate, shall be followed:

Heat to 240 ± 10°F in 45 minutes
Hold for 2 hours
Raise the temperature to 310°F over a 3 hour period
Then to 350°F in 30 minutes
Hold for 1 1/2 hours at 350
Cool below 150°F under pressure



NOTES: (1) Slit 0.5" deep at 1" increments around periphery

Figure 1 Vacuum Bag Technique

3.1.6 Molding of Polyimide-Glass Fabric Details

The glass fabric prepreg shall be laid-up in accordance with the drawing requirements. It shall be bagged in the conventional manner using no dams, teflon fabric or other items shown for the boron prepreg but using one ply of 181 bleeder fabric for each ply of prepreg used. A full vacuum, 25 inches Hg, min.) shall be applied and cure shall be accomplished in accordance with the following:

Raise the temperature to 260°F in 3 hours
Hold at 260 for 16 hours
Raise temperature to 310°F and hold one (1) hour
Raise to 350°F and hold for 90 minutes
Cool to 150 prior to release of vacuum

Post cure shall be accomplished as follows

4 hours at 400°F followed by 4 hours at 500°F

3.1.7 Post Cure of Polyimide Sub-Assemblies

To obtain the maximum temperature resistance a step post cure is required reaching a maximum temperature of 700°F. The exact post cure cycle is shown in Figure 2. This cure was accomplished on major sub-assemblies such as upper and lower surface panels and the front and rear spars.

3.1.8 Preparation of Details for Bonding

3.1.8.1 Preparation of Titanium for Bonding

3.1.8.1.1 Cleaning

The titanium shall be processed, for bonding, as follows:

1) Alkaline clean by immersion at 170-200F.

2) Acid clean by immersion in Nitric-Hydroflouric solution not to exceed three minutes

3) Rinse in cold water

4) Immerse in the following solution for 2 - 2 1/2 minutes

Trisodium phosphate	6.7 oz./gal.
Sodium Fluoride	1.2 oz./gal.
Hydrofluoric Acid	3.4 oz./gal.
Water	Remainder

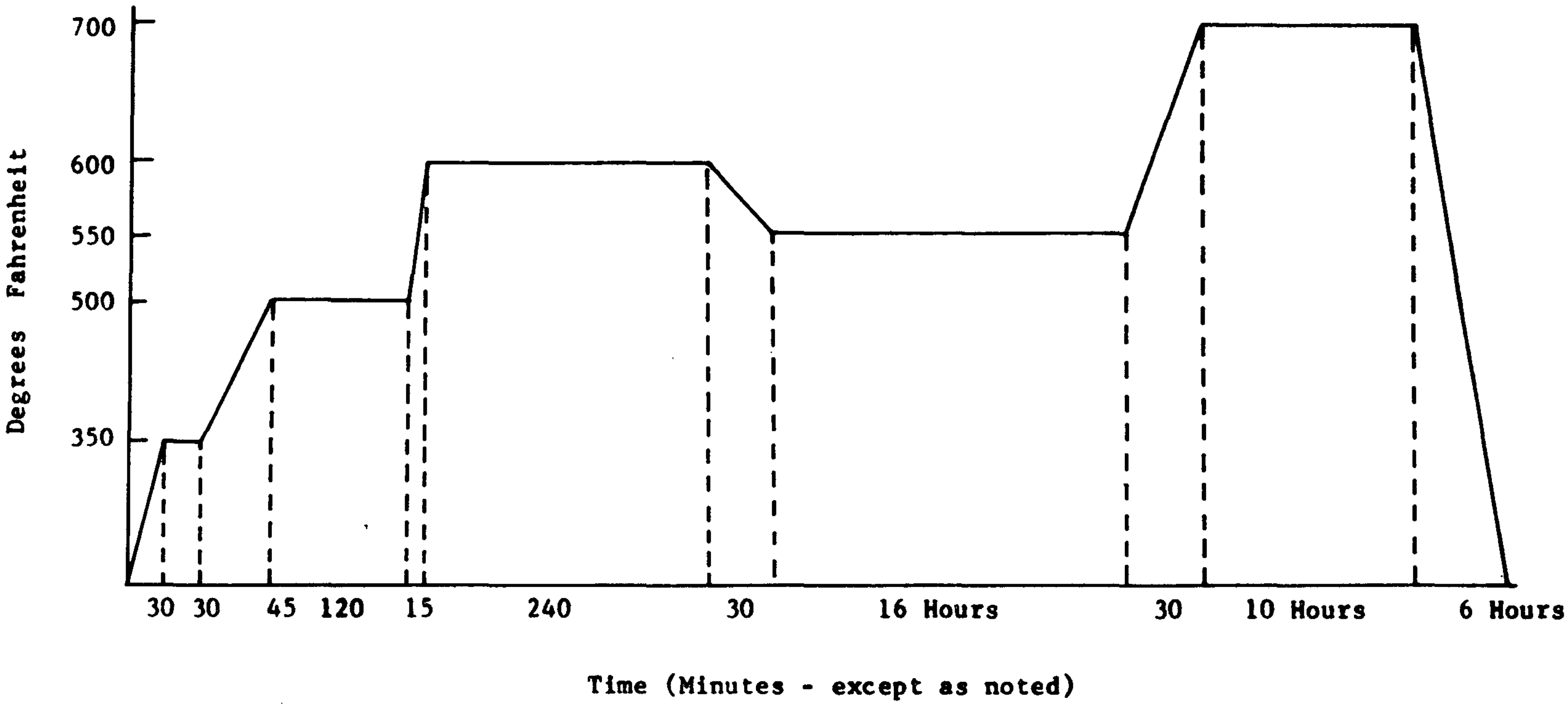


Figure 2 Post Cure Cycle: Polyimide-Boron Laminate

5) Rinse in cold water

6) Soak in hot water (145 ±5°F) for 15 minutes

7) Rinse in cold, dionized water and dry

3.1.8.1.2 Application of Primer

BR 34 Primer shall be cut to 60% solids using BR 34 thinner. A box coat, resulting in approximately 0.0005 inches of thickness shall be applied and air dried for 30 minutes followed by 30 minutes at 220F and 60 minutes at 350F. Adhesive shall be applied within one hour or the surfaces shall be processed in accordance with the procedure shown under "Solvent Cleaning".

3.1.8.2 Preparation of Premolded Glass and Boron Details

These surfaces shall be thoroughly scuff sanded preferably with a jitterbug using 200 grit sandpaper, followed by solvent cleaning per paragraph 3.1.8.2.1. Surfaces shall be considered satisfactorily prepared when all resin gloss has been removed from the laminate surface. In no case may the sanding extend into the reinforcing material. The adhesive shall be applied within 30 minutes of surface preparation.

3.1.8.2.1 Solvent Cleaning

Within one hour prior to adhesive application, all sanded bonding surfaces shall be hand cleaned with clean cheesecloth and methyl ethyl ketone. The solvent used shall have been previously certified as free from contaminants and shall be stored in a separate container which has been identified for solvent cleaning use only. This solvent shall be applied by pouring onto the clean white cheesecloth; this will eliminate contact of the solvent supply with the cleaning tissue and reduce the possibility of solvent contamination.

Bonding surfaces shall be vigorously scrubbed with solvent-saturated cloths and immediately wiped dry with additional clean tissues before the solvent has evaporated. Surfaces upon which the solvent has been allowed to air dry are not suitable for adhesive bonding. Clean rubber gloves shall be worn throughout the solvent cleaning operation. A minimum of three separate solvent application-wipe dry operations shall be performed on all bonding surfaces; additional cleaning cycles shall be used, if required, until fresh white drying tissues show no trace of discoloration.

3.1.8.2.2 Handling of Cleaned Details

After final cleaning, details shall be handled with clean white cotton gloves only. If transportation is required between the cleaning and bonding areas, details shall be protected by wrapping in fresh, clean Kraft paper.

3.1.9 Bonding of Components

3.1.9.1 Prefit

Each faying surface where bonding is required shall be prefit prior to surface preparation of the details. The operation is accomplished by making the complete assembly but substituting the prefit materials for the adhesive. The prefit materials and their use are as follows: place one ply of tissue paper against a faying surface, a piece of carbon paper (face up) on top of the tissue paper and an 0.008 polyvinyl alcohol sheet next to the other faying surface. The black surface of the carbon paper shall be against the polyvinyl alcohol sheet. Pressure and temperature shall be applied by the same techniques and equipment to be used for the bonding operation. The prefit temperature shall be 250 ± 10°F and shall be held 5 minutes. The assembly shall be allowed to cool (below 150°F), the vinyl sheets removed and examined for mismatch. This is a "go" - "no go" procedure and should not be considered quantitative. A continuous, uniform impression indicates satisfactory mating. Lack of impression indicates unsatisfactory match and will require rework and a repeat of the prefit operation.

A quantitative method for determining the mismatch involves the use of a thermoplastic foam sheet. Insertion of this material in place of the adhesive and proceeding with the pressure and cure cycle will transmit the mismatch to the foam sheet where it can be measured to ±0.002 inch. Corrections can then be incorporated with quantitative knowledge of the mismatch. After correction, the polyvinyl alcohol system will be used for final prefit and record purposes.

Where high spots occur, these may be sanded or strip plies removed at the discretion of Inspection if the resulting material is within drawing tolerance. Where low spots (outside of tolerance) occur, the material shall be rejected and submitted to Material Review for action.

3.1.9.2 Handling of Adhesive

Upon removal from refrigeration, allow the outer layers of adhesive to warm to room temperature before unrolling to avoid cracking of the film or moisture condensation on the adhesive surface. Return adhesive to refrigerated storage immediately after the amount of adhesive required for use is removed to avoid overaging of the material.

3.1.9.3 Application of Film Adhesive

The film adhesive is a tacky film and care must be exercised in application to bonding surfaces. Wherever possible it is suggested that the detail to be bonded be used as a template in cutting adhesive patterns. Clean bonding surfaces per 3.1.8 immediately prior to adhesive application. Allow the outer separator sheet to remain in position until immediately prior to assembly of details.

3.1.9.4 Pressure Application

After assembly of details, sufficient pressure to assure intimate contact of details shall be applied by vacuum bag, autoclave or clamps. In all applications, minimum pressure utilized shall be 10 psi but shall not exceed 50 psi.

3.1.9.5 Bonding of Titanium to Wet Boron Polyimide Prepreg

This bond shall be accomplished using the Pyralin 35-520 laminating resin since the cure cycle of the FM 34 is not compatible with that of 25-520. This cure and post cure cycle is as shown in paragraphs 3.1.5.2 and 3.1.7.

3.1.9.6 Bonding of Precured and Solid Materials

Precured and metallic details and assemblies shall be bonded using the FM 34 adhesive. After application of the adhesive and pressure application, cure shall be accomplished by heating the bond line to 350°F in 75 ± 15 minutes and holding for one hour. A post cure, to achieve 650°F temperature resistance, is required. The completed beam assembly shall be post cured for 16 hours at 350°F followed by three (3) hours at 700°F where the 700° is achieved in approximately 90 minutes after completion of the 16 hour exposure.

3.2 Quality Assurance

Specific quality provisions have not been developed except to assure conformance to the processes presented in paragraph 3.1.