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# DEVELOP AND DEMONSTRATE MANUFACTURING PROCESSES FOR FABRICATING GRAPHITE FILAMENT REINFORCED POLYIMIDE (Gr/PI) COMPOSITE STRUCTURAL ELEMENTS

V.A. CHASE AND E.S. HARRISON

GENERAL DYNAMICS CONVAIR DIVISION
P.O. BOX 85357
SAN DIEGO, CA 92138

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V. A. Chase
and
E. S. Harrison

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By

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# 1.0 SUMMARY

This report documents work performed by General Dynamics Convair Division for the National Aeronautical and Space Adminstration, Langley Research Center, under NAS1-14784 Tasks A through F. The objective of this program was twofold: first, to characterize the NR150 polymide resin and fabrication processes; second, the demonstrations of these processes through the fabrication of major composite structural elements.

The program was divided into two phases: (1) Fabrication Process Development and (2) Demonstration Components. The Fabrication Process Development phase consisted of Tasks A, B, and C with the Demonstration Component phase Tasks D through F. The first phase of the program involved the selection of a graphite fiber, quality assurance of the NR-150B2 polyimide resin, and the quality assurance of the graphite/polyimide prepreg (Task A), fabrication process studies (Task B), and small specimen process qualification (Task C). This effort resulted in the limited definition of material and process controls and the generation of limited test data. A significant material variability problem was encountered and a much larger effort on Phase I was expended than planned.

In the second phase of the program, a limited number of components was fabricated before the NR-150B2 resin was removed from the market by the supplier, DuPont. At that point it was decided to terminate the technical effort on this program and summarize the work that was accomplished.

# 2.0 INTRODUCTION

This report presents the work accomplished by General Dynamics Convair for the National Aeronautics and Space Adminstration, Langley Research Center, under Contract NAS1-14784. This program consisted of experimental studies structured to develop material and processing parameters for the manufacturing of structural elements and to demonstrate the manufacturing feasibility of graphite/NR-150B2 polyimide prepreg materials. The program was divided into eight original tasks, six which were completed before the removal of the resin from the market, which consisted of:

| | |
|---|---|
| Task A | Quality Assurance Program |
| Task B | Fabrication Process Development |
| Task C | Specimen Fabrication and Test |
| Task D | Laminate Fabrication |
| Task E | Skin Stringer Panel Fabrication |
| Task F | Honeycomb Panel Fabrication |
| Task G | Chopped Fiber Moldings |
| Task H | Space Shuttle Component Fabrication |

Tasks G and H were not conducted because of the NR-150B2 resin not being available.

The Materials and Processes Department of General Dynamics Convair was responsible for the work performed on this program. Mr. V. A. Chase was the program manager, with Mr. E. S. Harrison conducting the material characterization testing, process development and fabrication of the structural elements was conducted by Mr. J. E. Hilzinger, Mr. C. Smith, and Mr. G. Brown, and adhesive bonding studies by Ms. V. Y. Steger.

## Quality Assurance Program

The objective of this task was to characterize the NR150B2 resin sytem, select a compatible graphite fiber, and establish parameters for graphite/NR-150B2 prepreg material. The resin solution was characterized for solids content, monomer purity, acid number, amine assay, viscosity, molecular weight, TGA, PSC, infrared spectra, gas chromatography, and gel time. Prepreg characterization included resin and volatile content, fiber volume, flow, tack, and gel time.

## Fabrication Process Development

The objectives of this task were to develop manufacturing processes for four different structural elements and obtain process control and inspection data. The four structural elements studied under this task were as follows:

(1) Flat laminates
(2) Hat stiffners
(3) "I" stiffners
(4) Honeycomb sandwich panels

## Task C Specimen Fabrication and Test

The objective of this task was to generate preliminary test data as a function of temperature and time at temperature for both flat laminate and honeycomb sandwich construction.

## Component Demonstration - Tasks D through F

The objective of these tasks was to fabricate designated structural elements using the established processes and deliver them to NASA Langley for their testing.

# 3.0

# TASK A QUALITY ASSURANCE PROGRAM

This task of the program was aimed at establishing controls on the graphite fiber, NR-150B2 resin binder, and the graphite/polyimide prepreg.

## 3.1 GRAPHITE FIBER EVALUATION AND SELECTION

Fiber weight loss data at 316C (600F) developed by Gibbs and others at DuPont raised doubts as to the suitability of the contract specified HTS graphite fiber for 316C (600F) service. Gibbs's work showed fiber weight loss for HTS after 500 hours at 316C (600F) ranging from >50% to <2%. Lot-to-lot variation was found, as well as variation within a fiber lot. Discussions with Hercules disclosed that an earlier change in fiber heat treat temperature was apparently responsible for the HTS fiber high weight loss. Subsequent fiber batches were prepared by Hercules with the heat treat temperature adjusted upward to that originally used. One of these batches (66-7) was evaluated by Gibbs who measured weight loss variation for specimens ranging from 0.2 to 16% after 500 hours at 316C (600F).

Because of the uncertainty of thermo-oxidation stability of graphite fibers, thermal aging studies were conducted at 316C (600F) for various graphite fibers and polyimide composites, directed toward obtaining adequate data to select a fiber for the this program. Results for exposure times up to 1000 hours are shown in Table 1 and Figures 1 and 2. The Modmor II and HTS-2 (Lot 80-2, low sodium) fibers exhibit excellent thermal stability. The HTS (Lot 76-8) made with higher heat treat conditions showed significant improvement over the low heat treat HTS (Lot 54-4), but still experienced significant weight loss. The Celion fiber was intermediate in terms of thermo-oxidative stability for the fibers evaluated. The HM-S fiber exhibited a much higher weight loss than expected with high scatter between specimens (17.3 and 10.1% for the two specimens after 1000 hours exposure). All other materials showed little scatter from specimen to specimen. Laminate specimens followed the same weight loss trend as the fibers except for the HM-S laminate, which was based on a different fiber lot (55-3).

A limited amount of fiber evaluation involving fabrication and testing of laminates was conducted.

A sample of Modmor II/NR 150B2 was obtained from Fiberite and a 12-ply unidirectional 12 cm X 12 cm (5 in. X 5 in.) laminate was prepared by autoclave molding. Teflon coated glass release fabric was used on both sides of the layup.

The bleeder consisted of two plies of 1534 and one ply 120 glass fabric. This was followed by a caul plate and two additional plies of 1534 glass fabric. The entire layup was bagged using Kapton film and given an initial cure as follows:

(1) With full vacuum heat to 185C (365F) at 1C (1.8F)/Min.
(2) Hold one hour at 185C (365F).
(3) Apply 200 psi while heating to 204C (400F) at 1C (1.8F) Min.
(4) Hold two hours at 204C (400F).
(5) Cool to 65C (150F) under pressure.

After the initial cure, the laminate was cut into two parts. One part was post-cured in an autoclave as follows:

(1) Apply full vacuum and heat to 316C (600F) at 1.1C (2F) minute.
(2) Apply 1379 $kN/m^2$ (200 psi) at 177C (350F).
(3) Hold one hour each at 316, 343, and 371C (600, 650, and 700F).

The second half of the laminate was postcured in a similar manner in a press at 6895 kN/m$^2$ (1000 psi).

Flexural and interlaminar shear properties at room temperature (RT) and 316C (600F) are shown in Table 2. Due to low resin flow, a fiber content of only 53.5% volume was obtained and mechanical properties were low. Surprisingly, the high pressure postcure offered little advantage over the normal autoclave cure.

Specimens from the Modmor II laminate were subjected to thermal aging at 316C (600F) and short beam shear strength was determined after 696 hours at RT and 316C (600F). A 93% strength retention at 316C (600F) was obtained after the 696 hours of thermal aging, demonstrating the excellent thermal stability of the Modmor II/NR-150B2 composite (Table 3).

Two samples of NR-150B2 polyimide prepreg based on Celion high tensile strength graphite fiber were obtained from Fiberite for evaluation. Celion fiber properties are shown in Table 4. The Celion 3000 filament yarn was processed with an epoxy sizing, while the Celion 6000 filament fiber received an NR-150B2 sizing prior to resin film impregnation. The amount of NR-150B2 size was less than optimum for prepregging according to Fiberite. Unidirectional 12 ply X 15.2 cm (6 in.) X 15.2 cm (6 in.) laminates were prepared by the autoclave molding process previously described. The same autoclave postcure was used except for an additional five hours at 399C (750F). Mechnical properties versus temperature for the laminates are shown in Table 5. The RT flexural strength of 1633 MN/m$^2$ (237 ksi) was the highest value ever measured at Convair for an autoclave molded graphite/NR-150B2 laminate at that time.

A basic ground rule in selecting a fiber for this program was that it be a high strength type. A major criterion used in the final selection of a graphite fiber for this program was long term thermal stability. While it appears as though the polyimide resin may act as an oxidation barrier for the fiber, the possibility of local areas having porosity must be anticipated for a large, complex structure. In this case, oxidation of the fiber could occur, resulting in a degradation of the structure. Therefore, the two high strength candidates based on thermal stability are Modmor II and HTS-2. The HTS-2 is a new, low-sodium fiber based on a different precursor than the original HTS. No polyimide experience was available with the fiber. Also, at the time of the evaluation, it was not yet readily available. Therefore, Modmor II was initially selected for use on the program. A final and significant factor in this selection was the willingness of Morganite Modmor Ltd. to certify a maximum weight loss of 2% for the fiber after 500 hours at 316C (600F).

However, shortly after this selection Morganite Ltd. discontinued the manufacture of Modmar II graphite fiber. A program decision was made to use the Celion graphite fiber because it appeared to be more readily available than the HTS-2 graphite fiber. Initial processing studies were, however, conducted with all 3 fibers.

## 3.2 NR-150B2 RESIN BINDER EVALUATION

The first step toward developing laminate fabrication processing methods involved a determination of resin viscosity versus temperature for the polyimide solution. This data is needed to provide guidance as to hold temperatures for the laminate to build up resin viscosity sufficiently to prevent excessive flow and resin starvation when autoclave pressure is applied. Figure 3 shows the test apparatus used for the viscosity measurements. Figure 4 shows viscosity versus temperature curves for both the NR-150B2/NMP solution (55% solids) and the NR-150B2/EtOH/NMP solution (54% solids). The first obvious difference in the solutions is the large difference in initial viscosity. The NMP-based materials viscosity was too high to obtain a measurement with the instrument until 60C (140F). A second obvious difference is the temperature at which the viscosity starts to increase due to polymerization and/or solvent loss. For the NMP solution, the viscosity change is due to polymerization since no significant amount of solvent is lost. For the EtOH/NMP solution the viscosity increase is a result of the combined effect of removal of the low boiling EtOH and polymerization reaction. For the NR-150B2/NMP the reaction rate appears to become significant at approximately 121C (250F).

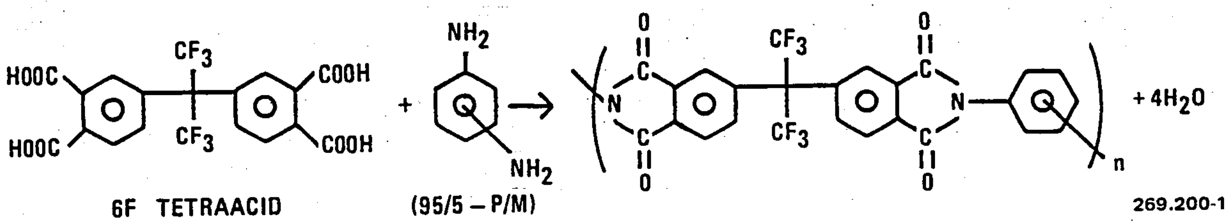
A five pound batch of prepreg (Lot 6C-55) based on Modmor II fiber and 60% solids NR-150B2-S5X was obtained from Fiberite. This prepreg was prepared from the same resin batch used to prepare the Celion 3000 prepreg (Lot 6D-26) received earlier. Both batches of prepreg were based on 60% solids NMP resin solution and ordred with a 28% cured resin content to allow "no-bleed" curing. Actual resin contents tended to be >30% as measured by Convair. A 10-pound quantity of the Modmor II prepreg from Lot 6C-55 was delivered to NASA/LARC. Physical appearance and handling characteristics of the prepreg were very good. Properties and characteristics of the prepreg and resin are given in Table 6. Properties for the initial batch of Modmor II prepreg based on 55% solids are included for comparison. Testing of the prepreg resin solution for viscosity versus temperature (Figure 5) gave a curve similar to that obtained for the 55% solution except that a higher viscosity was maintained across the temperature range. This can be attributed to the higher solids content. A minimum viscosity for the 60% solids solution was reached at 121C (250F) where the 55% solids solution reached a minimum at 88C (190F). For the higher solids material, higher temperature is required to achieve a solution (95C versus 75C) during the mixing operation. This apparently results in additional polymerization, which contributes to the higher viscosity. It appears reasonable that the difference in the viscosity curves can be attributed to the difference in solids content and degree of polymerization.

Acid and amine numbers were determined by chemical titration for the resin solution (combined Lots E14224-76 and E14224-78) with values of 1.55 and 0.789 meg./g. respectively being obtained. This compares to 1.68 and 0.864 meg./g. for the initial 55 solids solution resin batch (Lot E14224-34). The lower numbers for the high solids content resin batch support the assumption that polymerization reaction is occurring at the higher temperature (95C versus 75C) required for mixing the high solids solution. The acid/amine ratios for the initial and more recent resin batches are 1.94 and 1.97 respectively, showing reasonable agreement to the theorectical ratio of 2.0. Figures 6 and 7 are typical titration curves for the NR-150B2-S5X resin solutions.

High pressure liquid chromatograph (HPLC) studies were conducted on the 60% solids resin lot used for the recent batches of prepreg and compared to the earlier 55% solids resin lot (Figures 8 and 9). A scan was also run on the earlier NMP/EtOH based NR-150B2 as a matter of interest (Figure 10). Our efforts were centered on reverse phase/gradient elution techniques using a water/methanol mix (40-80%) as the eluting solvent and indicate minor differences between the 55% and 60% resin solids solutions over anticipated concentration dependency.

## Acid Content

NR-150B2-S5X resin binder as formulated has about 70% free acid groups. During the preparation of the prepreg due to different heating times or temperatures causes the acid content to be variable. As a check on results developed at Convair, samples were sent to Dr. Hugh Gibbs at DuPont. His results compare very well with the data generated at Convair and are shown in Table 7. A number of experiments were conducted in attempting to track the changes in NR-150B2 and NR-150B2-S5X resin during both preparation and subsequent processing. Our results are consistent with any simple age/advancement scheme which would be expected from the chemistry of the system.



We determined the total acid number of the material, supplied by Fiberite, in three forms: as the neat 60% dissolved solids resin, the film cast on release paper prior to fiber impregnation and, finally, the prepreg itself. The data obtained are presented in Table 8.

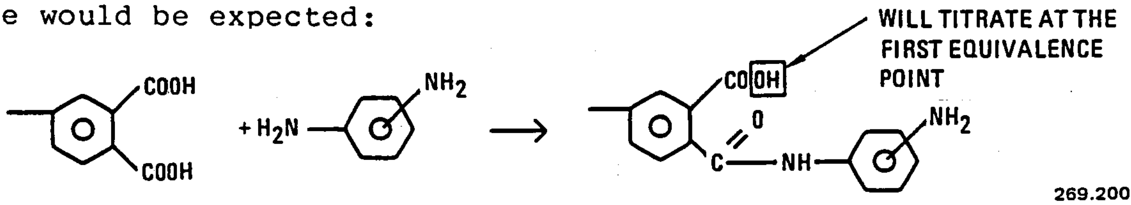
Some explanation of the following table is in order for a more complete understanding. Numerous corrections are required so that comparison of the results from various product forms may be made. The first row entry is for the sample of the 6F tetro acid, the second for 6F acid plus a stoichiometric amount of p-phenylene diamine (instead of the 95/5 p-phenylene diamine/m-phenylene diamine employed in the NR150B formulation). Since our titration yields results expressed in mg/g of sample, a correction for known acid content must be used to report a % of theory for both first and second equivalence points. In the case of acid plus diamine mixture, the correction factor is 0.816 (total acid as a percent of total sample weight). Similar considerations yield the results shown either measured (*) or calculated by the following scheme.

Resin plus volatile %*
-Volatile %*
Cured resin %
1.137 (1)
Dissolved solids %
.816 (2)
Dissolved acid %

Uncorrected equivalence point - % of theory
Dissolved Acid Content

(1) To correct for the loss of 4 moles of water to complete imidization
(2) As explained in text.

The results of 6F acid titrations show relatively poor accuracy, however, the results are within 10% of the expected values. We expected to see some scatter, but certainly not what was found; the % of theory results for the resin solution determinations shows fair consistency, but an unexpectedly low value range of 57 to 70% of theory for the first equivalence point and about 67% for the second. The single film specimen shows the expected decrease in free acid content brought about by the heat applied during the film casting procedures; while the prepreg results, which would be expected to show a continued loss in acid content, two out of three lots showed no change. The third lot of prepreg was solvent coated and, thus, would be expected to be near the % of the theoretical value for the first and second equivalence points found. If, indeed, some amic/acid formulation had occurred, a decrease in the second equivalence value would be expected:



269.200-2

Since the first and second equivalence points are virtually the same in all cases, the implication is that the imide ring closure is a fast step in the polymerization process. That is, the rate of imidization is fast as compared to amic/acid formation.

269.200-3

These results were obtained on material which had been stored for prolonged periods in the freezer. Titration of the dissolved resin, after filtering off the fibers, yielded virtually the same results. We similarily found no appreciable acid content on the unimpregnated sized fibers.

In conclusion, we believe careful attention to the thermal history during storage, filming, and prepregging the resin are required for the NR-150B-S5X polyimide resin.

## 3.3 GRAPHITE/NR-150B2 PREPREG EVALUATION

Work conducted by NASA/LaRC and DuPont has confirmed that the ethyl alcohol in the mixed solvent (EtOH/NMP) NR-150B2 system reacts with the 6F tetra acid to form various ester species. The degree of esterification is a function of time/temperature history for the material and directly affected the processing characteristics of the prepreg. To obtain a more stable material, a decision was made to use NR-150B2 polyimide prepreg based on only NMP solvent.

A five-pound quantity of Modmor II/NR-150B2 prepreg was obtained from Fiberite. Consistent with our plan to generate material property data that will allow development of a quality assurance specification, the prepreg material and resin solution were characterized for a variety of physical and chemical properties. The solution used for the prepreg preparation had a 55% present solids content, which proved to be too low for optimum prepreg preparation. As a result, the material had very high tack. Due to the low resin solids and resulting low viscosity, fiber collimation was not maintained and tow separation was evident. In general, the material had poor uniformity. It is evident that future prepreg should be based on a higher solids content resin solution.

The earlier NR-150B2 graphite prepreg based on the mixed solvent system typically had a volatile content of 12 ± 2%. With the all NMP solution, a volatile content of 20% was obtained in the prepreg. The first laminate from this prepreg was processed using the cure cycle desribed earlier, which had been developed for the mixed solvent prepreg. As anticipated, there was excessive resin flow resulting in resin starvation and high void content in the laminate. Properties for the laminate are shown in Table 9. It is obvious that processing conditions for the NMP-based prepreg are vastly different than for the mixed solvent material, necessitating the development of a completely differenct cure cycle.

At this time, an attempt was made to study volatile weight loss by TGA during postcure. Figure 11 is a TGA scan developed on laminate C-39 after the initial 204C (400F) cure. An approximate 0.8 CM by 0.8 CM (.3" x .3") square of composite was cut from the laminate and the TGA profile run at 0.5 C/MIN (33 F/MIN) scan rate. The purpose of the slow rate was an attempt to establish baesline data for postcure development studies.

The weight loss rate divides into five distinct segments:

$T_1$ - 65C (149F) - Absorbed moisture loss
$T_2$ - 215C (419F) - Retained solvent (NMP) loss below existing Tg
$T_3$ - 358C (676F) - Retained solvent (NMP) loss at or near advancing plasticized Tg
$T_4$ - 380C (676F) - Retained solvent loss above ultimate Tg
$T_5$ - 450C (842F) - Decomposition

These results were obtained on a laminate that showed essentially no voids prior to the postcure procedure.

The loss of solvent below the 204C (400F) cure temperature is not surprising since the small sample, with its very large surface area at the edges, should release residual solvent quite readily.

Because of the large edge (fiber ends) surface area, the diffusion rates from the TGA specimen is not representative of an actual laminate. However, the TGA curve represents a model of what is desirable to achieve in a laminate postcure. The heating rate is sufficiently low so as not to exceed the Tg of the plasticized laminate as the solvent is removed.

Lot C8-059 with Celion 3K fiber 8.66 kgs (19.1 lbs.) sized with NR-150B2 used the same (60.0%) resin solution solids level as Lot C8-166 with HTS-II fiber 2.27 kgs (5 lbs.). The prepreg properties (Table 10) did not indicate any unusual characteristics other than heavy resin concentrations on both tape surfaces and bare, dry fiber internally (Figure 12). An attempt was made to rework the material by wetting out single plies in a press. However, the material did not process satisfactorily.

## Material Quality Assurance

High pressure liquid chromatography (HPLC) has proven to be a very valuable analytical tool in the early studies. The presence of various esters of the 6F tetra-acid in the NMP/EtOH solvented material was first demonstrated by HPLC. Similar techniques have been employed for the all NMP solvent approach has been emphasized.

Our intent was to track the various resin batches through preliminary film casting and subsequent prepreg fabrication. Unfortunately, the traceability of more recent shipments from Fiberite were lost. There also have been some instances of a single prepreg batch being produced from two separate cast films of uncertain history. (Lot C8-059, 19.1 lbs. Celion).

Each operation in the resin to prepreg sequence is expected to result in some advancement of the material. HPLC is considered the best tool to describe the condition of the resin and provide a sensitive measure of degree of advancement for correlation with fabrication process changes.

The HPLC scans of two resin batches and two prepregged resin batches are shown in Figures 13 to 16. The most obvious difference is indicated by the arrow where the appearance (or disappearance) of a particular specie is easily seen. We are unable to correlate resin to prepreg as mentioned above. The scans are intended to only be illustrative. DuPont is currently attempting synthesis of some oliogomeric material, using stoichiometry unbalances in an attempt to identify the various later eluting peaks.

The following prepreg parameters were established:

1. NR150B2 resin with NMP solvent only.
2. NR150B2 sizing on Celion 6000 fiber.
3. Cured ply thickness: 0.017 ± 0.001 cm (0.005 ± 0.003 inch).
4. Areal fiber weight of prepreg: 145 ± 10 gm/$m^2$.
5. Resin content: 32 ± 2%.
6. Maximum volatiles: 14% at 204 (400F).
7. Resin flow: 10 to 20% at 177C (350F).
8. Drape must bend around a .800 cm (.3 in) diameter rod without fiber separation.

# 4.0

# TASK B FABRICATION PROCESS DEVELOPMENT

This task of the program was conducted to develop the required cure and postcure parameters required to fabricate graphite/NR-150B2 composite parts.

## 4.1 PROCESS EVALUATION

The processing of Modmor II/NR-150B2-S5X (100% NMP solvent) graphite/polyimide prepreg began with the autoclave curing of laminate C-4 in accordance with a standard cure schedule previously established for the HMS/NR-150B2 (EtOH/NMP solvent) material combination.

### Standard NR-150B2 Autoclave Cure Schedule

#### Initial Cure

(1) With full vacuum heat to 185C (365F) at 1C (1.8F)/minute.
(2) Hold one hour at 185C (365F).
(3) Apply 1400 kN/m$^2$ (200 psi) while heating to 204C (400F) at 1C (1.8F)/minute.
(4) Hold two hours at 204C (400F).
(5) Cool to 65C (150F) under pressure.

#### Postcure

(1) Apply full vacuum and heat to 316C (600F) at 1.1C (2F)/minute.
(2) Apply 1400 kN/m$^2$ (200 psi) at 177C (350F).
(3) Hold one hour each at 316, 343, and 371C (600, 650, and 700F).

Extremely high resin flow resulted during the initial cure producing a resin starved laminate having 74.8% fiber volume and approximately 15% void content. Subsequent curing cycles CR-1 through CR-4 added time and/or temperature in an attempt to increase the resin viscosity prior to pressure application and reduce resin flow. Cure schedules studies are summarized in Table 11.

An initial hold temperature of 121C (250F) without pressure was selected to increase resin viscosity sufficiently to obtain controlled resin flow. This hold temperature was based on a resin solution viscosity versus temperature curve which indicated the reaction rate to become significant at approximately 121C (250F). Also, this temperature would not give an excessively high reaction rate and should provide a cure process with wider tolerance on the temperature control, heating rate, etc. Holds at 121C (250F) showed improvement of mechanical properties with increased time, but the time required to obtain the necessary improvements proved excessive (>5 hr) for a practical cure schedule. As a result, laminate C-9 was prepared with a hold (2 hr.) at 149C (300F). This cure resulted in a substantial reduction in resin flow and gave a laminate with 1.8% void content after the 399C (750F) postcure. Photomicrographs of laminates C-4 through C-9 show progressive increase in quality of the laminates, (Figures 17-21). This is also reflected in Table 12 which gives the mechanical properties of the laminates. The CR4 cure schedule used for laminate C-9 is summarized below:

<u>Cure Schedule CR-4</u>

(1) Without vacuum heat at 1C (1.8F) min. to 149C (300F) and hold one hour.
(2) Apply full vacuum and hold an additional hour at 149C (300F).
(3) Heat to 185C (365F) at 1C (1.8F) min. and hold one hour.
(4) Apply 1400 kN/m$^2$ (200 psi) and heat to 204C (400F).
(5) Hold two hours at 204C (400F).
(6) Cool to 65C (150F) under pressure.

While this cure schedule gave a low void laminate, it went too far in advancing the resin and reducing flow as evidence of the low

(55%) fiber volume. This is reflected in the less than optimum flexural strength. A further contributing factor to low strength is the poor quality of the prepreg, which due to low resin solution viscosity (55% solids) had tow separation, variations in thickness, and resin content.

Laminate C-10 was prepared using the same CR-4 cure cycle and for laminate C-9 in order to evaluate the reproducibility of the process. Laminate C-10 had a void content of 1.7% indicating that the process is reproducible. Photomicrographs of laminate C-10 are shown in Figures 22 and 23.

While laminates C-9 and C-10 were very satisfactory in terms of void content, little resin flow was experienced, resulting in a higher than desirable resin content (55 and 52% fiber volume). Laminates C-11 and others prepared during this period involved attempts to obtain increased resin flow and higher fiber volume.

Laminates C-11, C-13, and C-15 (Figures 24, 25, and 26) involved successive modifications of the CR-4 cure schedule by reduction of hold time at 149C (300F) in one-half hour increments. For laminate C-11 the hold time at 149C (300F) without vacuum bag pressure was reduced to one-half hour (cure CR-4A) from the one hour used in the CR-4 cure. Mechanical properties were comparable to laminate C-10.

Laminate C-13 was prepared using cure cycle CR-4B which involved a one-half hour hold at 149C (300F) without vacuum pressure followed by an additional one-half hour at 149C (300F) with vacuum bag pressure. This cure cycle gave a void content of 2.7%, but gave no improvement as far as increasing the fiber volume. Mechanical properties were somewhat lower than what was obtained on the previous laminates.

Laminate C-15 was prepared using cure cycle CR-4C which consisted of a one-half hour hold at 149C (300F) without vacuum pressure followed by applying vacuum bag pressure and raising directly to 185C

(365F). On this laminate, a void content of 2.1% was measured with the fiber volume being substantially unchanged from the previous laminates. However, photomicrographs (Figure 26) of a specimen from this laminate did not confirm the calculated low void content indicating that the laminate was not uniform or a non-representative sample was used for the photomicrograph. Mechanical properties were similar to previous laminates except there appeared to be a tendency for a lesser retention of strength at 316C (600F).

Laminate C-12 was prepared using cure cycle CR-5 which consisted of a 1 hour hold at 149C (300F) without pressure followed by applying both vacuum and 1400 kN/m$^2$ (200 psi) autoclave pressure, holding for a second hour at 149C (300F) and then continuing the standard cure. This process resulted in high resin flow and a 66% fiber volume. However, a void content of 10.7% was calculated for the laminate. Examination of the photomicrograph of the laminate cross section (Figure 27) show the voids to be concentrated in two thirds of the laminate thickness with the remaining one third being very low in porosity. Surprisingly, this laminate gave the highest flexural strength measured at this time of 1316 MN/m$^2$ (188 ksi). Figure 28 illustrates the difference in rim flow for five of the cure cycles evaluation.

Laminate C-16 was prepared using cure cycle CR-6 which involved a half-hour hold at 149C (300F) with no pressure after which the laminate was increased to 185C (365F) for a one-hour hold at which time both vacuum and autoclave pressure were applied. High void content (13%) and high fiber volume (64%) were obtained on this laminate. Mechanical properties were practically non-existing with a flex strength at RT of 147 MN/m$^2$ (21 ksi). This laminate was inadvertently heated at a rate of 1.4C/min (2.6F/min) on the increase to 185C (365F) rather than the normal 1C/min (1.8F/min).

Laminates C-14, C-17, and C-20 were prepared at the same time to study the effect of flow for a cross ply panel and the possibility of controlling flow by different bleeder combinations on C-14 and C-17.

These laminates were cured using the CR-4 cure schedule, but with vacuum bag pressure being applied at the start of the curing operation. Examination of the bleeders for these laminates showed excessive resin flow cure schedule, but with vacuum bag pressure being applied at the start of the curing operation. Examinatin of the bleeders for these laminates showed excessive resin flow and specific gravity measurements (Table 12) are indicative of high void content.

Laminates C-18, C-19, and C-21 were cured at the same time in a manner similar to the previous three with vacuum pressure being reduced to 254 MM (10 in.) of mercury to give approximately 35 $kN/m^2$ (5 psi). Laminate C-19 used a neat bleeder sytem, that is, the bleeder was the same area as the laminate layup. Laminate C-21 had a cross ply orientation. Specific gravity measurements on these three laminates indicate some improvement in void content. Also, the neat bleeder approach appeared to offer promise in that the highest specific gravity for the three laminates was obtained for that laminate.

Laminates C-22 and C-23 were cured using cure cycles CR-9 and CR-10 and was an attempt to study the effect of slight vacuum bag pressure during the 149C (300F) hold. Laminate C-22 was held at 149C (300F) for two hours with 127 mm (5 inches) of mercury which produced 35 $kN/m^2$ (5 psi) pressure. Laminate C-22 was held at the same temperature pressure condition except for a time duration of only one hour. Unfortunately, excessive bleeder material was mistakenly laid up with these laminates causing excessive resin bleed which resulted in high porosity and high fiber content. Due to this error, the results were not meaningful to the fabrication process study.

An 11 Kg (5 lb) quantity of prepreg based on Celion 3000 fiber and 60% solids resin solution with a net resin content (no bleed approach) was evaluated. The Celion material was employed because of delays in obtaining Modmor II fiber and the necessity to maintain the fabrication process development effort. This material was a vast improvement in terms of uniformity and fiber collimation, with good tack and drape. The Celion prepreg was used to evaluate the possibility of performing the 149C (300F) advancement setup in an oven rather than the autoclave. Laminates C-28, C-29, and C-30 were vacuum bagged without bleeder and advanced in an oven at 149C (300F) for 1, 2, and 3 hours, respectively. After removal from the oven, they were rebagged with bleeder and subjected to the standard C-4 autoclave cure without the 149C (300F) hold. The laminates exhibited no bleed during cure and visually appear to be of good quality.

Earlier studies established the need for a temperature hold at 149C (300F) for the NR-150B2 prepreg material to increase the viscosity sufficiently to prevent excessive resin flow when vacuum bag/autoclave pressure is applied. An investigation was also conducted into the possibility of performing the 149C (300F) advancement step for the NR-150B2 initial cure in an oven rather than the autoclave. Laminates C-28, C-29, and C-30 (Celion 3000) were vacuum bagged without bleeder and advanced in an oven at 149C (300F) for 1, 2, and 3 hours respectively. After removal from the oven, they were rebagged with bleeder and subjected to the standard initial C-4 autoclave cure shown below without the 149C (300F) hold (Steps 1 and 2). The laminates exhibited no bleed during the initial cure and visually appear to be of good quality.

Initial Cure (CR-4)

(1) Without vacuum, heat at 1C (1.8F) min. to 149C (300F) and hold one hour.
(2) Apply full vacuum and hold one additional hour at 149C (300F).
(3) Heat to 185C (365F) at 1C (1.8F)/minute.
(4) Hold one hour at 185C (365F).
(5) Apply 1400 $kN/m^2$ (200 psi) while heating to 204C (400F) at 1C (1.8F)/minute.
(6) Hold two hours at 204C (400F).
(7) Cool to 65C (150F) under pressure.

Postcure

(1) Apply full vacuum and heat to 316C (600F) at 1.1C (2F)/minute.
(2) Apply 1400 kN/m$^2$ (200 psi) at 177C (350F).
(3) Hold one hour each at 316, 343, and 371C (600, 650, and 700F).
(4) Raise to 399C (750F) and hold for five hours.

Laminate C-31 (Celion 3000) involved an evaluation of the possibility of performing the 149C (300F) advancement step in an oven while unrestrained (no vacuum bag). However, excessive swelling and distortion of the layup was experienced. This distortion along with the extreme boardness of the layup after advancement made it impractical to cure laminate C-31 (Figure 29).

Postcure of Laminates C-28, C-29, and C-30 was conducted in accordance with the above postcure schedule. Upon removal from the autoclave, the laminates were found to have developed blisters obvious from visual examination. The laminates were inspected by ultrasonic C-scan at 5 MHz and different gain settings. At the lower gain settings the blisters tend to blend in and become obscured by what is assumed to be porosity; however, at higher gain setting, they are very distinct. Flexural, short beam shear and specific gravity specimens were cut from the laminates. Even though all three laminates had calculated void contents on the order of 6%, average RT flexural strength values ranged from 1631 to 1771 MN/m$^2$ (233 to 253 ksi) and SBS ranged from 90.3 to 97.3 MN/m$^2$ (12.9 to 13.9 ksi) (Table 13).

Laminates C-32 (Celion 3000) and C-33 (Modmor II) were prepared using the C-4 initial cure and postcured by the standard postcure schedule. Again, blistering was evident after postcure. Ultrasonic C-scans were conducted on these laminates at 5 MHz and at various gain settings. These are shown in Figures 30 and 31. Specimens were cut from the sound areas of laminates C-32 and C-33 and tested for flexural strength, SBS, fiber volume, specific gravity, and void content. RT flex values of 1890 and 1386 MN/m$^2$ (270 and 198 ksi), respectively, were measured for laminates C-32 (Celion 3000) and C-33

(Modmor II) and represent the highest values obtained for these materials to date. Also, void content for both laminates was <1% in areas that had not blistered.

Also postcured with laminates C-32 and C-33, was a large 15 by 15 by 1/8 inch (CD-1) unidirectional Modmor II laminate. This laminate was prepared to demonstrate scale-up from the typical 15x15 cm (6x6 in.) laminates used throughout the program for process development and for evaluation of thermophysical properties under a separate NASA/LaRC program. This laminate did not exhibit any visual signs of blistering; however, ultrasonic C-scan indicated delamination and porosity.

An additional set of Celion 3000 and Modmor II laminates (C-34 and C-35) were fabricated and postcured by the standard cycle with blistering occurring for both laminates. Figure 32 illustrates the type of blistering experienced with the blistered area being greatly increased in thickness over the sound portion of the laminate.

Both laminates show approximately 12% weight loss during initial cure and total weight losses of 18.7 to approximately 20.1% after final postcure. Neither layup exhibited flow during cure, with only minimum bleeder stain, and 6.6 to 8.5% volatiles remained to be removed during the postcure. By contrast, earlier dense laminates showed a weight loss of 10 to 12% (C9 and C10) during postcure and did not blister. Apparently the volatile removal was reasonably complete since glass transition temperatures (Tg) exceeding 350C were measured. No further testing was done on laminates C-34 and C-35.

At this point, a detailed analysis was conducted of the fabrication process in an effort to solve the laminate blistering problem. This analysis disclosed that the vacuum bag had presented problems of leakage during the 399C (750F) postcure on earlier laminates, which was alleviated by reducing the autoclave pressure. As a result the laboratory technicians had established a "standard procedure" of reducing the autoclave pressure from 1400 kN/m$^2$ (200 psi) to 350 kN/m$^2$ (50 psi) when the 399C (750F) postcure step was reached. When the autoclave pressure was released at 399C (750F) the laminate was above its Tg and contained residual NMP, which created sufficient internal pressure to cause blistering. It appeared logical that maintaining autoclave pressure throughout the postcure would alleviate the blister problem.

Laminates C-36 and C-37 were cured using the CR-4 initial cure schedule for evaluation of the effect of maintaining full autoclave pressure during postcure. Inadvertently, full vacuum was applied too early in the cure cycle caused very high resin flow and a resin starved laminate condition. These laminates were replaced by C-38 and C-39.

Laminate C-38 was used to evaluate the effect of 1400 kN/m$^2$ (200 psi) pressure throughout the postcure cycle. Upon removal from postcure, the laminate appeared sound. However, ultrasonic C-scan and physical and mechnical properties measurement showed the laminate to be delaminated and/or containing internal porosity (Tables 14, 15, and 16). The high-pressure postcure was sufficient to prevent visual blistering, but the rate of temperature increase was apparently high enough to create internal pressure from the residual solvent and resulted in delamination.

At this point, it became obvious that the postcure schedule, which had been successful with earlier laminates, was not adequate for the laminates presently being fabricated. This is attributed to their very low void content and the fact that the laminates have a higher fiber volume, which results in a longer path for volatiles to diffuse to the laminate surface.

Laminates C-40 (Celion 3000) and C-41 (Modmor II) were prepared using the CR-4 initial cure. Laminated C-40 was postcured by the following modified schedule (Standard B) under 1400 kN/m$^2$ (200 psi) autoclave pressure:

3 hrs at 288C (550F)
1 hr at 316C (600F)
2 hrs at 343C (650F)
1 hr at 371C (700F)
1 hr at 399C (750F)

This postcure differed from the standard postcure previously used in that the 3 hours 288C (550F) hold was added, an additional 1 hour hold at 343C (650F) was added, and the time at 399C (750F) was decreased from 5 to 1 hour. The rationale for this cycle involved an attempt to remove more of the NMP solvent at lower temperatures. After postcure the laminate was inspected by ultrasonic C-scan at different gain settings at 2.25 MHz. Typically, the C-scan is first conducted at a lower frequency where sound transmission is more readily obtained. If good scans are obtained, inspection is conducted at higher frequencies. In general, a good laminate is indicated when uniform dark scans are obtained at high frequency and low gain. The scans for laminate C-40 at 2.24 MHz and all gains indicated delamination and porosity over the majority of the laminate. Mechnical properties were low (Table 17) and, although a void content of approximately 1% was obtained, the specimen was obviously not representative of the laminate. Since Modmor II fiber was no longer available, laminate C-41 was not postcured.

Laminated C-44 (Celion 3000) was prepared by the CR-4 initial cure cycle with an additional two hours at 232C (450F) being added in an attempt to reduce the residual solvent level prior to postcure. The postcure cycle (Standard C) for laminate C-44 involved adding temperature holds at closer intervals for the lower temperature portion of the schedule as follows:

1 hr at 260C (500F)
1 hr at 288C (550F)
1 hr at 316C (600F)
1 hr at 343C (650F)
1 hr at 371C (700F)
5 hrs at 399C (750F)

After postcure, the laminate was inspected by ultrasonic C-scan at 2.25 and 5 MHz. Scans at 2.25 MHz at gain settings of 10 and 20 indicated uniformity and some porosity. However, the scan at 5 MHz indicated delamination, which was reflected in the low mechanical properties measured (Table 17). For laminate C-45 the CR-4 cure cycle was modified (CR-11) by applying vacuum bag pressure at the beginning of the cure cycle in an attempt to obtain some degree of resin flow and to lower the amount of residual solvent which must be removed during postcure. Excessive flow was experienced resulting in some porosity in the laminate.

At this point it became apparent that the problem of postcure removal of solvent from the dense thermoplastic NR-150B2 laminate was a very complex one. One approach for achieving a postcure would be to conduct an empirical study that would involve determining weight loss versus time at short temperature intervals over the postcure range. However, this approach would be very time consuming and would have to be worked out for a variety of laminate thicknesses. The ideal solution to the problem would be an indicator that would provide a signal when the temperature increase rate approaches a point where there is sufficient internal pressure to cause blistering or delamination. If this was possible the temperature rate could be changed or temperature holds could be initiated as the critical temperature is approaching. Molecular activity would be expected to increase as the laminate approaches blistering temperature and should be reflected in the dielectric properties of the material, which can be measured by a dielectric cure monitor. Certainly the temperature at which blistering occurs should be indicated (capacitance change) and provide information as to temperatures for initiating holds.

## Dielectric Monitoring During Initial Cure

Dielectric monitoring was initiated for the initial cure to 204C (400F) to further assure reproducible laminates. Through the use of the dielectric monitor resin viscosity change is indicated and allows applying pressure at the optimum temperature/time. Our experience has been to produce laminates with very low void content and optimum mechnical properties pressure must be applied during a narrow time-temperature band. For the epoxy systems that we have investigated this band encompasses a range from where maximum volatiles have been removed and the resin is at a viscosity where excessive resin flow does not occur to just before the gellation point, where the resin can no longer flow when pressure is applied. Developing similar parameters for the NR-150B2 system would provide a process control technique which will incure reproducibility.

While the monitor background for epoxy systems acted as a starting point, there are a number of very major differences that had to be considered for the NR-150B2. For example, a method of polymerization with high volatile content required that our normal monitoring procedures be modified. The electrode on the topside of the laminate (the steel tool acts as the bottom electrode) must be in contact with the resin to respond. The normal procedure has been to place the probe in the bleeder for epoxies. Because of the very low viscosity at temperatures below 149C (300F), vacuum cannot be applied prior to that point. This poses a problem in that with no vacuum the bleeder will not saturate, thus no voltage response. To compensate for this, we placed an insulated probe in the laminate. Because of the very small initial voltage change, the monitor recorder had to be set to a very high sensitivity scale.

Process gel tests were ran in an attempt to correlate bleeder saturation, visible volatile outgassing, and viscosity to voltage readings. This initial investigation indicated that the monitor voltage responses are truly indicative of viscosity change for the NR-150B2 system.

The following is an explanation of voltage/temperature profile for laminate C-27. (Figure 33).

1. As the temperature was increased from 24C to 149C (75 to 300F), very little voltage response was seen because there was no vacuum on the laminate thus no resin bled to provide contact with the electrodes. The electrodes for this laminate were placed on top of the bleeder material. This was changed to incorporate the electrode in the laminate.

2. When vacuum bag pressure was applied, an immediate voltage decrease was seen. This indicated resin flow and laminate compaction.

3. As the temperature was increased to 185C (365F) the voltage increased, indicating a viscosity increase.

4. When 1378 $kN/m^2$ (200 psi) was applied a voltage change was not seen in epoxy systems. This would indicate that the resin had advanced to a stage where 1378 $kN/m^2$ (200 psi) would not cause resin flow or compaction.

Laminate C-52 (based on Lot 6C-55, the original 60% resin content Modmor II prepreg) and Laminate C-53 (Lot C8-166, 2.27 Kg (5 lb.) HTS-II prepreg) were cured in accordance with the CR-4 cure schedule. Vacuum bag pressure was lost during the cure and visual quality of the laminates was poor. High porosity and low specific gravity was evident (Table 18). Laminate C-52 was retained for later postcure while C-53 was discarded due to poor quality.

Prepreg lots (HTS-II, Lot C8-166 and Celion, Lot C8-059) were reworked in an effort to wet out fiber in the middle of the tape by contact pressure in a press at 93C (200F) for 1 to 2 minutes.

The next cure involved the fabrication of three laminates, two being based on the reworked HTS-II and Celion prepreg with the third one being the as received Celion material (Laminate C-56 reworked HTS II, C-55 reworked Celion and C-54 as received Celion). The three laminates (C-55 and C-54) exhibited high resin flow whereas the HTS II (C-56) laminate showed no flow. Based upon the visual appearance of the three laminates only Laminate C-56 was retained for postcure.

The next cure attempt involved Laminate C-57 (HTS-II, Lot C8-166), Laminate C-58 (Celion, Lot C8-059), and Laminate C-59 (HTS-II, Lot C8-166). This cure (CR-12) was conducted using a dielectric monitor in an effort to determine the optimum point for applying pressure and to help assure reliability on future laminates fabricated. In addition, Laminate C-57 was cured without the standard caul plate. The cure consisted of heating to 149C (300F) at 1C (1.8F) per minute followed by a ten minute hold after which full vacuum pressure was applied. Following an additional ten minute hold, 1400 $kN/m^2$ (200 psi) autoclave pressure was applied. This cure was determined by the dielectric monitor response and represented the first successful use of the monitor for initial cure of the NR-150B2 system. This was accomplished by imbedding the electrode in the layup. The points of pressure application and dielectric response versus time and temperature are shown in Figure 34. These three laminates showed moderate resin flow during cure and visually appeared to be an improvement over the previously cured laminates.

Laminates C-60 (Celion batch C8-059) and C-61 (HTS-II, Lot C8-166) involved a continuation of the dielectric monitor cure study. This cure (CR-12A) involved a one hour hold at 149C (300F) at which time vacuum bag pressure was applied, followed by 15 minute hold at which time 1400 $kN/m^2$ (200 psi) autoclave pressure was applied. After an additional time of 45 minutes at 149C (300F) under pressure the laminate was raised to 204C (400F) for four hours. The dielectric monitor curves, temperature and pressure profiles are shown in Figure 35. The later application of vacuum and autoclave pressure was an attempt to obtain reduced flow which was considered excessive on the previous cure. Laminate C-60 (Celion) gave high flow and a very rough and irregular surface and high porosity. Laminate C-61 (HTS-II) showed no flow, had an irregular surface, and gave a poor ultrasonic C-scan even at a frequency of 2.25 KHz and a gain of 10 (Figure 36).

At this point, there was much concern that the quality of the recent prepreg was such that satisfactory laminates could not be readily achieved, and a decision was made to reject the prepreg material and use earlier prepreg which was on hand for fabrication of laminates to conduct postcure studies. Laminates C-62, based on the 1st lot of Celion prepreg made from 60% resin solids and laminate C-63, based on the 1st batch of Modmor II prepreg made from 60% resin solids, were cured in accordance with CR-4 cure cycle that was earlier proved successful. Unfortunately, a vacuum bag leak was experienced during the cure which resulted in poor quality of these laminates. As previously for the CR-4 cure cycle, no flow into the bleeder material was experienced. A dielectric monitor of this cure was conducted and is shown in Figure 37. At approximately 135C (275F) resin viscosity stabilized, as indicated by the dielectric curve, and remained stable for approximately 25 minutes at 149C (300F). When vacuum pressure was applied after one hour at 149C (300F), no indication of flow or compaction was evident. This was also the case when full autoclave pressure was applied at 135C (365F). C-Scans indicated porosity which was probably due to the loss of the vacuum bag pressure during the cure.

The next cure involved laminates C-64 and C-65 which was based on the earlier Modmor II fiber with 60% resin solids (Lot 6C-65). This cure was conducted by use of the dielectric monitor with vacuum bag pressure being applied after 15 minutes at 149C (300F). Dielectric monitor curves indicated flow occurring both at vacuum bag pressure and autoclave pressure application points (Figure 38). Both laminates experienced substantial flow confirming the response of the dielectric monitor. Ultrasonic C-scans were conducted for laminates C-64 and C-65 after the initial autoclave cure. Laminate C-64 gave excellent C-scans at both 2.25 MHz and 5 MHz (Figure 39). Laminate C-65 which experienced higher flow during cure gave poor C-scans at 5 MH even at a gain as high as 70.

The replacement order (Lot C8-206) for the rejected 8.66 Kg (19 lbs) of Celion prepreg was received and two preliminary laminates (C-66 and C-67) were cured using the dielectric monitor as an indicator for pressure application. Vacuum bag pressure was applied after 15 minutes at 149C (300F) and 1400 kN/m$^2$ (200 psi) autoclave pressure was applied 15 minutes after vacuum bag pressure. The dielectric monitor curve indicated flow and compaction at both pressure point applications (Figure 40). Laminate C-66 was C-scanned after initial cure and gave a good scan at 2.25 MHz at a gain of 3 with approximately 85% of the laminate being dark. At 5 MHz and higher gains, substantial portions of the laminates were revealed to be sound. Some of the earlier laminates from the recent prepreg showed evidence that pressure had been applied later than desirable. This cure was an attempt to investigate an earlier application of pressure and establih an initial data point for pressure application to the new batch of prepreg material.

At this point, the problem appeared to be excessive resin flow which results in starvation of the laminate and porosity. For laminate C-68 and C-69 the point of pressure application was extended to 45 minutes into the 149C (300F) hold with both vacuum bag pressure and autoclave pressure being applied simultaneously (Figure 41). In addition, only one ply of 120 glass bleeder was used for the laminates in order to prevent excessive resin flow and starvation. Laminates

C-68 and C-69 had good visual appearance. Laminate C-68 gave a good C-scan at 2.25 MHz at a gain of 2.1, at the same frequency and a gain of 1, a small area of porosity was indicated. Laminate C-69, which was from a roll of prepreg from the new lot having slightly higher resin content than used for C-68, gave much poorer C-scans than laminate C-68 under all conditions.

Laminates C-70, 71, 72, and 73 were cocured using a single 120 glass bleeder in an attempt to reduce resin flow when full vacuum and 1400 $kN/m^2$ (200 psi) pressure were applied early in the cure cycle after a 45 minute hold at 149C (300F). Relatively heavy flow was experienced on all four layups, as shown in Table 18 and high average cured ply thicknesses resulted. C-scans at 5 MHz indicated poor laminate densification with the thinner 6 ply layups looking best, although small cracks and warpage were evident on all. No postcure was conducted.

The next series of laminates, C-74 through C-79 combined the reduced bleeder system with a delayed vacuum and pressure application after a 70 minute hold at 149C (300F). Lower resin flow was obtained on all laminates and a slow 19 hour postcure ending after 4 hours at 371C (700F) produced an equivalent postcure weight loss except for the two six ply laminates which showed a higher cure weight loss and the highest overall total laminate weight loss. These laminates, however, produced the highest measured flexural and shear strengths of this series. Laminate C-78 C-scan was the best at all settings although the mechanical test results of C-76 to C-78 do not reflect as much difference.

Four additional unidirectional laminates, C-80 through C-83, were then processed using a cure cycle which further delayed vacuum and pressure application until 100 minutes. As before, both rolls of prepreg from Lot C8-206 were laid up as 6 and 12 ply laminates. Very low resin flow occurred during cure and the 12 ply laminates displayed substantial gaps on the top ply after the same slow postcure used on laminates C-70 through C-79. The six ply laminates, however, produced excellent C-scans before and after cure. Good specific gravities and superior ultimate flexural strengths of 160 $MN/m^2$ and 163 $MN/m^2$ (231 and 237 ksi) for laminates C-81 and C-83, respectively (see Table 19). Approximately 50% property retention was obtained at 316C (600F). This cure schedule with delayed vacuum and pressure application produced relatively low flow and resulted in fiber volumes below 60%.

The following series of laminations C-84 through C-94 were attempts to further refine the processing window for application of vacuum and pressure. An 80 minute hold at 149C (300F) was used for C-91 through C-94 after two earlier processing starts were terminated prematurely because of equipment malfunctions. The C-scans for these panels after cure, however, did not show the same high quality and densification obtained with laminates C-81 and C-83, which have calculated void contents of 2.0 and 0% respectively.

### Postcure Study

One laminate (C-39) previously cured by the CR-4 cure schedule and a second laminate (C-46) cured by a modificaton of CR-4 designated CR-11A which involved full vacuum bag pressure from the start of the initial cure, were autoclaved postcured together.

The standard postcure schedule was used except that 1400 $kN/m^2$ (200psi) autoclave pressure was applied from the start (Standard A) rather than at 185C (365F). The dielectric cure monitor was used in an attempt to obtain an indication of the temperature at which blistering was occurring. Electrodes 10.2 X 10.2 $cm^2$ (4x4 in.) of copper foil were placed on both sides of the 15.2 x 15.2 cm (6x6 in.) laminates with a layer of glass fabric between the electrodes and laminate to provide electrical insulation. This in effect, created a capacitor of the laminates. It was anticipated that an electrical response would be indicated at the temperature which blistering occurred. The dielectric response for the two laminates versus temperature is shown in Figure 42. To appreciate the significance of the curves, it is necessary to understand that the initially cured laminate is in a highly plasticized solvent, thereby achieving maximum glass transition temperature (Tg) for the polymer and maximum service temperature for the composite. A downward direction for the dielectric curve may be indicative of a number of phenomena including:

1. A thickness reduction of the laminate due to resin flow or shrinkage.
2. Increase in molecular freedom due to decrease in viscosity.
3. Increase in molecular freedom due to exceeding the polymer's Tg.
4. Normal increase in molecular activity due to elevated temperature.

In the case of the postcure study, the dielectric response curve indicates a number of temperatures at which increased molecular action occurs. For example, the first maximum activity is indicated at 316C at (600F). Upon holding the laminate at 316C at (600F), molecular activity decreases with time and actually levels off for laminate C-39. This is attributed to the plasticizing solvent being removed from the laminate with the leveling off of the curve indicating the diffusion rate at that temperature. When temperature is increased further to 343C (650F), the same phenomena is repeated. Upon increasing to 317C (700F), a very large increase in molecular activity occurs which would lead one to believe that blistering or delamination is most likely occurring at this temperature range. The larger change in the case of Laminate C-39 can probably be attributed to the fact that it was dense and high in resin content, therefore, having a higher solvent content and contained porosity which allowed the solvent to be more readily removed. Also disclosed by the curve is the fact that after 2 1/2 hours at 399C (750F) the molecular activity has stabilized indicating that the five hour postcure at 399C (750F) was not required.

Continuation of the postcure study by dielectric monitoring involved postcuring laminates under vacuum bag pressure at a constant heating rate in an air circulating oven. By this approach, it was possible to start with an initial high heating rate and determine the time at which this rate becomes excessive by a dielectric indication. Additional postcures were conducted at reduced heating rates to generate a family of curves. In this manner it was possible to establish that X heating rate was satisfactory up to Y temperature, that X1 heating rate was adequate up to Y1 temperature, etc. This data established a postcure schedule consisting of a continuous increase in temperature with periodic decreases in rate of increase, thereby yielding the minimum possible postcure time for the laminate.

The first dielectric monitored postcure at a constant heating rate was run at 1.2 C/min (2.2 F/min.). The dielectric response curve for laminates C-47 and C-41 which were postcured together under vacuum bag pressure in an oven are shown in Figure 43. Only one curve is plotted since the response for both laminates was identical. The curve indicates that this heating rate was adequate up to just below 260C (500F). The postcure was terminated at approximately 382C (720F).

The postcure study was continued using the dielectric monitor to indicate changes in the composite versus temperature with successive runs being made at reduced heating rates. The dielectric curve indicated this rate to be satisfactory up to approximately 254C (490F). Laminates C-52 and C-59 along with a standard laminate, which had been fully postcured, were postcured under vacuum bag pressure in an air circulating oven with a dielectric monitor. Since the laminates previously had been cured to 204C (400F) the rate up to 177C (350F) was accelerated. From 177 to 399C (350 to 750F) the average rate of temperature increase was 0.3 C/min. (0.7F/min.). The dielectric monitor curve indicated that this rate was adequate up until approxmiately 310C (590F), (Figure 44). Ultrasonic C-scans of laminates C-52 and C-59 indicated high porosity as would be predicted from the postcure dielectric monitor curve. No further testing was conducted on laminate C-52 and C-59.

Next, laminate C-27 (cured earlier) along with the standard postcured laminate (AA) was postcured under vacuum bag pressure in an air circulating oven using the dielectric monitor to measure laminate conditions or changes. The intent of this postcure was to reduce the heat-up rate above 274C (525F) to 0.2C/min (0.5F/min.). Difficulty was experienced in maintaining the heat-up at the desired rate and as a result an indication of laminate degradation occurred at approxmiately 357C (675F), (Figure 45). Ultrasonic C-scan was performed on the laminate before and after postcure indicating good quality initially but porosity after postcure. The C-scan combined with a 10% increase in thickness after postcure indicated delamination and porosity and no further testing was performed.

Laminates C-56 and C-57 were next postcured and monitored under vacuum bag in an air circulating oven along with laminate (AA) (standard postcured laminate). laminate c-56 had been cured by the cr-4 cure cycle and consequently was thicker and higher in resin content. laminate c-57 was cured by cure cycle cr-12, which involved earlier application of vacuum and autoclave pressure, and was low in resin content and high in porosity. this postcure again attempted to maintain a 0.2C/min. (0.5F/min.) rate from 274 to 399C (525 to 750F). Laminate C-56, which was dense with a higher resin content, showed a break in the dielectric curve at approxmiately 354C (670F). Laminate C-57 which was lower in resin content tended to follow the standard postcured laminate curve and showed an apparent break at approxmiately 371C (700F) (Figure 46). Ultrasonic C-scans for both laminates indicated poor quality and no further testing was conducted.

Laminates C-62 and C-63 were postcured under vacuum bag pressure in an air circulating oven following the same cycle as used on previous laminates C-56 and C-57. Ultrasonic C-scans were conducted for the laminates before and after postcure and in the case of laminate C-62 an improvement in quality was indicated. This would suggest that flow and compaction is occurring during the postcure cycle. However, porosity in these laminates was high and mechanical properties were disappointing (Tables 20 and 21).

The major difficulty in conducting the oven postcure appeared to be at temperatures above 343C (650F) and also problems in obtaining the desired heating rates. On laminates C-64 and C-66 it was decided to limit the oven cure to 343C (650F) and increase the time at this temperature to 16 hours. Figure 47 shows the dielectric monitor curve for the long term postcure at 343C (650F). Laminate C-64 showed very good ultrasonic C-scan pattern at 5 MHz down to a gain of 2 prior to postcure. However, after postcure the C-scan pattern was poor even at a gain of 60, indicating porosity and/or delamination had occurred during the postcure. Since no response was indicated by the dielectric cure monitor, it appears as though an autoclave pressure postcure will be required if dense laminates are to be achieved after postcure. Density measurements and mechanical properties for these laminates (Table 21) confirmed the C-scan results and further suggest that an autoclave postcure is desirable. Also, the Tg of 330C (626F) for laminate C-66 indicates that the time/temperature conditions were not adequate to achieve the ultimate Tg>350C (662F) for the polymer.

Laminates C-65 and C-67 were autoclaved postcured under 1378 $kN/m^2$ (200 psi) using the dielectric monitor. The laminates were postcured at a rate of 0.6C/min. (1.4F/min.) up to 343C (650F) and held at 343C (650F) for five hours. The temperature was planned to be increased from 343C to 371C (650 to 700F) at a rate of 0.1C/min. (.25F/min). Unfortunately, the autoclave temperature controller malfunctioned and manual override was used for the increase from 343C to 371C (650 to 700F) resulting in a higher rate of increase than planned. Actual rate of increase from 343C to 371C (650 to 700F) was 0.6C/min. (1.4F/min). Laminate C-65 gave an indication by the dielectric monitor slightly beyond 343C (650F) of the rate of increase being excessive. For laminate C-67 this indication was at approximately 371C (700F) under 1400 kN/m$^2$ (200 psi) (Figure 36). The panels were cured an additional five hours at 371C (700F) under 1400 kN/m$^2$ (200 psi). Laminate C-65 was ultrasonic C-scanned before and after postcure. Prior to postcure, acceptable C-scans were obtained at 2.25 MHz. At 5 MHz porosity was indicated in the center portion of the laminate. After postcure, laminate C-65 showed further degradation at both frequencies, however, a reasonable C-scan at 2.25 MHz and a gain of 3 was obtained for laminate C-65. Laminate C-67 was not subjected to ultrasonic C-scan prior to postcure. At 2.25 MHz and down to a gain of 1, good C-scans were obtained after postcure. However, at 5 MHz Laminate C-67 gave poor C-scans below a gain of 40. Laminate C-65 gave low mechanical properties both at ambient and 316C (600F) and had an indicated void content of 7.3% (Table 21). Laminate C-67 gave a RT flex strength of 1080 MN/m$^2$ (157 psi) and a 316C (600F) flex strength of 660 MN/m$^2$ (96 ksi). Measured porosity for laminate C-67 was 5.5%. The strength retention at 316C (600F) indicates that the 5 hr. @ 371C (700F) postcure is probably adequate, but the potential strength of the material was not achieved.

Laminates C-95, C-96, C-97, C-98, C-99 and C-100 were cured with the objective of continuing to narrow the pressure application band. The laminates fabricated included one each, six, twelve and twenty-two ply laminates from each of two rolls. Pressure and vacuum were applied after 57 minutes at 149C (300F). The six and twelve ply laminates from each roll had fair to good surface quality, but had an average cured ply thickness of .016 cm (6.4 mil), which is in excess of normal. The twenty-two ply laminates were also thick, in addition, they exhibited resin filled gaps that had been seen previously under a number of various conditions. These laminates were considered unacceptable and further processing was discontinued. The effort continued to narrow the pressure application range of NR-150 laminates, C-103 and C-104, by applying vacuum and pressure after 50 minutes at 149C (300F). Both of the laminats were unidirectional 15.24 x 15.24 cm, (6x6 in.) x 12 plies, one laminate was fabricated from each roll of batch C8-206. Encouraging looking laminates were produced using the cure cycle shown in Figure 48. Surface quality was good and ply thickness was .0148 down to .0147 cm (5.85 and 5.80 mils) per ply. C-scans with a gain setting of 2 x 10 at 5 MHz looked very good (Figure 49). To better understand the behavior of the resin during cure, resistivity measurements were taken on these laminates during cure. This process was developed by Dr. David Sears under Air Force Contract F-33615-77-C-5217, "Advanced Composite Process Control." Probes similar to those used for dielectric cure monitoring were placed on the top and bottom of the laminate in perfect registry. These probes had previously been gold plated to elminate the corrosion that could possibly effect the resistivity measurement. The bottom of the laminate was insulated from the layup plate with a ply of Teflon and two plies of 181 to prevent shorting of the probe (Figure 50). While this did not allow for additional bleed, there is a possibility that this fiberglass cushion did allow an avenue of escape for volatiles. To determine what effects this variable might of had, part of the laminates from the next two groups were bagged in the same manner. Laminate C-104 was postcured using the cycle shown in Figure 51. After postcure, a decrease in thickness was observed and no blistering occurred. C-scan results at a gain of 2 x 10 at 5 MHz showed very little change after cure (Figure 52). Flexural and short beam shear specimens were machined and tested. Test results are shown in Table 22. A portion of the C-104 laminate has also been designated as a standard for C-scan.

### Laminates C-116, C-117, C-118

The C-103/C-104 cure cycle was again attempted and accomplished with the exception of the temperature reaching 166C (330F) for five minutes prior to pressure application. It was felt that this slight deviation in the cure cycle would have very little effect on the laminates, however, the laminates were thicker than normal, .0157 to .0167 cm (6.2 to 6.6 mils) per ply. After postcure laminates C-117 and C-118 had cured ply thicknesses of .0149 to .0148 cm (5.89 and 5.84 mils) with a very good C-scan. Property data for C-117 is shown in Table 22.

A continuing effort was maintained on the C-103/C-104 type cure cycle. For this evaluation, two cross ply laminates, $(0\pm60)_{2S}$, C-119 and C-120 were fabricated. Both laminates had a good surface finish in addition to an acceptable ply thickness. Laminate C-119 was postcured using the standard cure cycle shown in Figure 4. After completion of postcure, laminate C-119 had a ply thickness of 144 cm (5.69 mils) per ply. However, after postcure, the C-scans showed a marginal laminate. We performed flex and shear tests on C-120. These test results are also shown in Table 22.

The purpose of the next group of laminates (C-121 through C-127) was to show cure cycle feasibility on .0317 cm (.125 in.) thick laminates, a six-ply 45.7 x 45.7 cm (18 x 18 in.) laminate and two 7.6 x 7.6 cm (3 x 3 in.) dielectric monitor laminates. The two thick laminates, C-121 and C-122 averaged .0157 cm (6.17 mils) per ply with good surface quality with very little crowning. The 45.7 x 45.7 cm (18 x 18 in.) six-ply laminate had an average resin/volatile loss of 12%, however, the ply thickness was .0165 cm (6.5 mils)/ply. C-scans at a gain of 2 x 10 at 5 MHz showed acceptable laminates.

### (300F) Cure

Because of special bagging materials required for cure temperatures of 149C (300F), 204C (400F), or higher, we felt that some evaluation should be pursued to lowering the initial cure temperatures and possibly even reducing cure pressure. Data generated over the past few months coupled with visual observations of small oven cured samples led to the conclusion that a starting point for a reduced temperature/pressure cure cycle should be around 149C (300F) and 700 $kN/m^2$ (100 psi). In addition, a pressure bump could be used to aid in the nesting and compaction of the fibers and pre-preg plies to eliminate large volume changes within the pre-preg layup. A single ply of 120 style bleeder cloth could be used to limit the resin flow and bleed. Laminates C-108 through C-113 were composed of six, twelve, and twenty-two ply laminates from rolls one and two of batch C8-206 and were cured according to Figure 53. Ply thickness on these laminates ranged from .0151 to .0164 cm (5.96 to 6.46 mils) per ply, which is considered to be too thick. However, surface quality was excellent and the resin at the edge of the laminates was a rich dark brown, rather than the dull brown witnessed in many previous laminates. Weight loss after cure (bleed plus volatiles) was around 7.5% rather than the normal 10 to 12%. C-scan results on the six and twelve ply laminates were excellent at a gain of 2 x 10 at 5 MHz and very good in most laminates at 2 x 1 MHz. The twenty-two ply laminate C-111, did not C-scan well before or after postcure.

Because of the encouraging results of laminate C-108 through C-113, the cycle was repeated with two new variables added. First, reduce the three 700 $kN/m^2$ (100 psi) pressure bumps to a single 700 $kN/m^2$ (100 psi) application and second, eliminate the top caul plate. There has been a problem up to this time with the fiber distribution lacking in uniform thickness within the pre-preg. Curing without the aid of a pressure equalizer such as a top caul plate accentuated this problem. Both laminates, each from a separate roll of pre-preg, were uneven in thickness. Average thickness measurements could not be taken because of the non-uniform condition. The surface finish of the laminates other than the hill and valley effect was excellent.

Areal fiber weights of pre-preg rolls were checked and found to be unacceptable. Roll number one of batch C8-206 had a variance of 44.52 to 53.8%; fiber weight (9.1%) over twelve specimens. Roll number two had a variance of 48.97 to 57.92%; fiber weight (17.4%) again over twelve specimens.

The last group of laminates, C-134 through C-137, were reruns of the 149C (300F), 700 $kNm^2$ (100 psi) bump cure cycle. While this is not a desirable cure cycle from a manufacturing standpoint, material processing characteristics may require this type of pressure application. This group of laminates consisted of twelve ply laminates, both cross ply and unidirectional orientations with and without caul plates. These laminats were cured and tested. Test results are shown in Table 22.

## High Pressure Cure

During a discussion with NASA Langley personnel it was suggested that a 2100 kN/m$^2$ (300 psi) cure cycle be explored. Laminates C-101 and C-102 were press cured at 2100 kN/m$^2$ (300 psi) after 80 minutes at 149C (300F). Surface quality was fair, however, resin filled gaps in the laminate were still present. Ply thicknesses were .0150 and .0152 cm (5.9 and 6.0 mil), respectively, which is still thicker than anticipated. C-scan evaluation showed that voids were present in both laminates.

Laminates C-114 and C-115 were fabricated as part of the 2100 kN/m$^2$ (300 psi) study. For this set of laminates the application of 2100 kN/m$^2$ (300 psi) was to have been applied after 50 minutes at 149C (300F). However, pressure was inadvertently applied upon reaching 149C (300F). This resulted in a .0117 cm (4.6 mils) per ply thickness and a 28% weight/bleed/volatile loss. No further processing of these laminates was attempted.

As part of the 149C (300F) 2100 kN/m$^2$ (300 psi) cure evaluation, laminates C-124 and C-125 were fabricated. Pressure and vacuum was applied after 50 minutes at 149C (300F). Ply thickness was down to .0119 to .0124 cm (4.71 and 4.90 mils) per ply with a 26.6% weight/bleed/volatile loss. C-scan results at a gain of 2 x 10 at 5 MHz were poor. While resin content was not run on these laminates, it would appear that pressure was applied prematurely causing excess resin loss. One ply of 120 style cloth was used as the bleeder, which was saturated in addition to excess edge bleed. No further procesing of these laminates was conducted.

## Cure Cycle Investigation of Single Solvent System

Three cures consisting of a total of ten laminates were processed with the purpose of obtaining additional dielectric monitor and resistivity data. These laminates consisted of unidirectional and cross-plied, six, twelve, and twenty-four ply laminates with and without caul plates. Refer to Table 22 for laminate data. It was suggested by Dr. Hugh Gibbs of DuPont Corp. that one possibliity for poor retention of 316C (600F) properties could be due to microcracking as a result of the lower initial cure temperature. To determine if this condition existed in laminates that had previously been tested, specimens from laminates C-103, C-108 and C-113 were inspected and found not to be microcracked (Figures 54, 55, and 56).

## Cure Laminate Characterization

Due to the poor retention of 316C (600F) properties Tg and TGA analysis were conducted on a number of laminates. It was determined that the Tg's ranged from 302-330C (576-626F), and .2 to .4% solvent remained after the 371C (700F) postcure. Preliminary investigations have shown that Tg's should be driven to 340-360C (644-680F), after five hours of postcure at 399C (750F). However, solvent content still fell in the range of .1 to .3% after a 399C (750F) postcure. Mechanical testing showed that driving the Tg to the 350C (662F) range still did not produce good 316C (600F) property retention. It was concluded that this very small amount of residual solvent, presumably NMP, was a definite factor. Laminates were fabricated using nine different lots of prepreg, five from Fiberite and four from U.S. Polymeric. All laminates with the exception of C-238 were precompacted, cured, and postcured at the same time. Laminate C-238 was not postcured because of limited postcure plate size and poor cure results. The precompaction, cure, and potcure cycles used for these laminates are shown in Figure 57 through 60.

During the postcure of these laminates, the autoclave control thermocouple shorted out causing an incorrect temperature reading.

This resulted in the final eight hour hold being at 343C (650F) rather than the prescribed 399C (750F). After postcure, glass transition temperatures were measured on three laminates. The Tg's for all three laminates were low, 130-190C (266-374F) rather than the normal 350C (662F). The laminates were rebagged and postcured again following the originally intended 399C (750F) cycle.

During the fabrication process a weight loss study was conducted to determine the approxmiate amount of volatiles remaining in the laminate at various processing steps. These numbers can then be compared to the prepreg volatile content, thus giving an indication of the amount of remaining volatiles at any particular point. Volatile content determinations are shown in Table 1 as determined when originally received and just prior to the fabrication of these laminates. Selected laminates were tested at room temperature and 316C (600F), as tabulated in Table 23.

Additional flexural test specimens were machined and subjected to heat aging for periods of 200, 500, and 1000 hours at 316C (600F). The specimens then were tested at 24C (75F) and 316C (600F). The test data are shown in Figure 61. The Celion 6000/NR-150B-S5X laminates showed no loss of strength out through 500 hours. After 1000 hours at 316C (600F) the strength retention has dropped to approximately 25% of the original strength at 316C (600F) and 50% of the original room temperature strength. Since there weren't any data points between 500 and 1000 hours, the point at which the strength really starts to decrease is not know. It is expected that good retention of strength may go out to as much as 700 to 800 hours followed by a significant drop in strength.

A laminate was fabricated, per the procedures outlined in the preceding section of this report, with the exception of the postcure. During postcure, the Data-trac on the autoclave malfunctioned and caused the part to heat from 316C (600F) to 371C (700F) in 10 minutes. After postcure, the laminate was found to be blistered. An additional set of two laminates were then fabricated. One laminate was processed through cure (C-503) and the second laminate (C-502) through postcure. No anomales were seen during the processing cycles. C-scan results of C-502 after postcure was excellent at a frequency of 5 Mega Hertz and a gain of 2. Flexure and short beam shear specimens were machined and tested. The following results were obtained:

| Test Temperature | 24C (75F) | 316C (600F) |
|---|---|---|
| Flexure Strength | | |
| MN/m | 2150 | 1474 |
| (ksi) | (312) | (214) |
| Short Beam Shear | | |
| MN/m | 84 | 55 |
| (ksi) | (12.2) | (8.04) |

Based on these studies, we selected the precompaction, cure, and postcure cycles shown in Figures 57 through 59 for the studies to be conducted in Task C-Specimen Fabrication and Test.

## 4.2 NON-DESTRUCTIVE EVALUATION (NDE) STUDY

An evaluation was conducted to determine the feasibility of using an infrared scanner for NDE of NR-150B2/graphite composites. The system used for this evaluation was based on an AGA 680-S infrared scanner, which employs a liquid nitrogen-cooled detector of indium autimonide (INSB). The system characteristics include a temperature resolution of 0.2C (0.3F) at 30C (86F), a faster frame rate of 16 frames per second, a line scan frequency of 1600 per second, with 210 lines (interlaced) per frame, and a resolving capability of 140 elements per line. The IR lens had a 15-degree field of view. The ultimate resolution at a lens-to-subject range of 46 cm (18 in.) is better than 0.812 mm (0.032 in.). The system operates on the principle that defect areas will have a different thermal conductivity which will be indicated on the IR scan pattern. The primary advantage of this approach over ultrasonic C-scan is the speed with which the test can be conducted. The test setup is shown in Figure 62. Figure 63 illustrates the display/control arrangement.

The test sequence used for this program involved heating the back surface of the panel while observing the changes in the infrared-detected heat-level and dispersion on the front surface. When an indication was detected on the cathode ray tube (CRT), a scope-photo was taken of the infrared display pattern. This method was sufficient for all but the thinnest section of the test panel. Here, the time was too brief (much less than two seconds). An auto-triggered scope camera would have been appropriate for the thin section, but was not available. For all thicker sections, the time was two seconds or greater, and manual triggering was adequate.

Heating to the panel was supplied by one or two 2700 Watt quartz lamps. Heating level was controlled by a variable transformer. The technique involved turning up the voltage to a predetermined level and time, then promptly turning it off.

The evaluation was conducted on test panel No. C-5 which was fabricated in a stepped fashion to give thicknesses of 40 plies, 24 plies, 12 plies, and 6 plies. The panel measured 61 x 30 cm (24 x 12 inches), with each section being six inches long. The properties for the laminate are given in Table 24. Due to the relatively high thermal conductivity of the solid laminate, each section of the panel had to be heated and observed individually.

Except for the 6 ply section, heat was applied for three seconds at a heater-to-panel spacing of 18-21 cm (7 - 8 1/2 inches). Heater power was varied by using 130 volts peak for the 40-ply and 24-ply sections, 100 volts peak for the 12-ply sections, and 80 volts peak for the 6-ply sections. Lower peak voltages were ineffective, as were greater spacings. The maximum peak voltage available was not fully adequate for the 40-ply section.

The time required for obtaining the IR patterns after the thermal pulse varied with laminate thickness. About 30 seconds was required for the 40-ply (too long, too low heat level), about seven seconds for the 24-ply, about three seconds for the 12-ply, and approximately 1± seconds for the 6-ply section. Noting the rough doubling of time required for the 6-12-24 ply sequence would lead one to project a nominal time around 12-13 seconds with 170 volts peak applied to the heaters for the 40-ply section. Other data indicate this to be a reasonable projection.

The length of time during which the IR pattern could be observed was very short; being on the order of one tenth of the time required for the indication to appear after first heat application. For the thinner sections, this necessitated a relatively long exposure (1 second) of the scope camera to assure capture of the IR pattern. Failure to secure a satisfactory exposure by this method determined the need for an auto-triggering scope camera mentioned previously. Review of the Polaroid scope photo of the C-5 test panel reveals an indication of an anomaly in the 12-ply section near the edge of the 24-ply section. This is shown most clearly where an isotherm technique was applied. Three sections of the panel are represented in Figure 64, a portion of the 40-ply section on the left, the 24-ply in the center, and a portion of the 12-ply on the right. In the 12-ply portion, below center, a dark squarish area is surrounded by white speckels. The white speckels represent the areas of high isotherm level, the dark area is revealed as one at a lower surface temperature, indicating a localized condition of reduced conductivity. Analysis of system and infrared camera data reveals the surface temperature difference between the dark area and the surrounding speckel area to be within 3C. For the large part the test panel was indicated to be uniform by the IR-scan.

The multi-thickness panel was next ultrasonic C-scanned to determine if there was a correlation to the IR thermogram. C-scans were conducted at 2.25 MHz and at two gain settings for each thickness of the panel (Figures 65-68). The standard procedure for the C-scan evaluation is to scan the panel at reduced gain levels until high contract is obtained between the dark (low porosity) and white (high porosity) areas. In terms of low porosity of the laminate (for a given laminate thickness) the lower the gain setting at which a dark scan can be obtained, the higher the quality.

The stripped laminate did not give good C-scans; however, this was not unexpected since the laminate was based on the mixed solvent resin system and also contained a fair degree of porosity. Interestingly, the anomaly indicated on the 12-ply laminate by IR scan (Figure 64) was not apparent on the ultrasonic C-scan (Figure 66). The conclusion resulting from the IR study is that the IR scan is not particularly suitable for identifying areas of typical porosity in a highly thermal conductive graphite laminate. The IR scan offers a rapid and effective means of NDT for locating delaminations on solid laminates and debonds in a honeycomb sandwich.

A C-scan is not normally performed on the laminate where physical properties such as low density or high porosity indicat a low quality laminate. However, three of the laminates fabricated during the processing study were ultrasonic C-scanned to obtain comparative data (Figure 69). Laminate C-10, based on cure cycle CR-4 which has resulted in void content <2%, was included in this series of laminates and gave excellent C-scans at low gain settings and were typical of those obtained for graphite/epoxy composites.

## 4.3 CHOPPED FIBER MOLDING

The problem of removing residual NMP from thick, high-pressure molding is considered a major difficulty with NR-150B2, which severely limits its application. The use of Diglyme solvent in lieu of NMP was considered a viable approach for alleviating this problem. Diglyme has been used successfully by DuPont for adhesive bonding with NR-150 polyimide. A purchase order for a 2.3 Kg (5 lb) quantity of NR-150B2/Diglyme prepreg had been issued to Fiberite with the intent of chopping the material to give a molding compound. However, as a result of concern by both DuPont and Fiberite in being successful in prepregging with the resin solution, the procurement was cancelled. This task was later dropped from the program.

# 5.0

# TASK C SPECIMEN FABRICATION AND TESTING

The objective of this task was to fabricate honeycomb sandwich test specimens, lap shear specimens, and solid laminates for preliminary composite test data.

## 5.1 ADHESIVE BONDING STUDY

### Preliminary Evaluation

NR-056X, the newest member of the NR-150 family of adhesives, was selected for the adhesive bonding study in this program.

NR-056X, developed by DuPont under a program sponsored by NASA/Langley Research Center (Contract NAS1-24620) consisted of a monomeric solution of 6F tetra acid, PPD and ODA (molar ratios of 1.0/0.75/0.25) in diglyme solvent. Upon curing, the NR-056X solution yields approximately 48 percent by weight of cured polyimide solids. It was the purpose of this program to evaluate and optimize NR-056X adhesive system for titanium-titanium, composites/composites, and composite/honeycomb core bonding.

Initially, efforts dwelt mainly on establishing a procedure to make good adhesive prepregs with NR-056X resin using a glass fabric as the carrier.

NR-056X resin (Lot No. E14224-101, procured from DuPont) was mixed with aluminum powder (Reynolds grade 1-131) to produce an adhesive solution with 72.5% solids; of which 65% by weight is the aluminum and 35% by weight is the cured resin solids. 112 Style E glass scrim was used in preparing the adhesive prepreg.

The roller blades set-up initially used for making the prepreg was not satisfactory. The resin was warmed up about 71C (160F) in a trough placed on a hot plate. A set of rods with a pre-set air gap was positioned directly above the trough. The scrim impregnated with resin would drip back into the trough. The prepregging had to be conducted in a well-ventilated hood in accordance with the industry health and safety regulations. The ventilation cooled the resin to the extent that the impregnated scrim became thick, viscous, and impossible to be pulled through the gap without severely distorting the fabric.

Because the resin mixture was nearly at room temperature during the impregnating process, the prepared prepregs were rather thick. Both 112 and 120 style prepregs had a thickness of 0.05/cm (20 mils) after the first coating and around 0.076 to 0.102 cm (30-40 mils) after the second coating process. The B-staged prepregs didn't have a uniform appearance (see Figures 70 and 71). Blisters and craters were present on both prepregs.

The fully cured prepregs have a more smooth surface compared to the B-staged prepreg (Figures 72 and 73). A weight loss of around 14% was observed for the cured films. The cured film thickness was around 0.043 cm (17 mils) for 120 scrim and 0.051 cm (20 mils) for 112 scrim. The films were cured at 316C (600F) for two hours under 1400 MN/$m^2$ (200 psi) platen pressure and postcured at 316C (600F) for 16 hours freestanding.

A second prepregging process investigated proved successful. Warmed to 71C (160F) the adhesive resin was applied with a squeegee onto the glass scrim, which was placed on silicone release paper. The impregnated scrim along with the release paper was pulled through the heated blades spaced for the desired prepreg thickness of 0.032 cm (13 mils). The prepreg was staged as follows:

40 min at 85C (185F)
20 min at 100C (212F)
20 min at 113C (235F)

The B-staged prepreg measured .020-.023 cm (8-9 mils) in thickness. Warmed adhesive resin was reapplied to the prepreg to attain a total thickness of 68 cm (27 mils). The prepreg was B-staged as follows:

40 min. at 85C (185F)
20 min. at 100C (212F)

The finished prepreg measured 0.050 - 0.055 cm (20-22 mils) in thickness, contained 10-11% volatiles, and had a shinny and smooth appearance.

Adhesive prepregs were prepared using 112 style E glass scrims with several fabric finishes: A1100, Volan A, heat-cleaned (Volan A +1 hr. at 426C (800F), and S-935 (a heat-resistant silane finish proprietary to J.P. Stevens Co.). No difference in resin wetting characteristic or adhesive prepreg appearance was observed as a function of fabric finish.

Adhesive films of filled NR-056X polyimide resin were successfully prepared using 112 style E glass scrim with A1100 and S935 finish. The prepared adhesive film was evaluated using lap shear tests (using 301 stainless steel adherends) at R.T. and 316C (600F) and flatwise tension test (using HRH 327 glass polyimide core and cured NR-150B2-S5X graphite polyimide laminates) at R.T.

The stainless steel test specimens were MEK wiped and alkaline cleaned with Ajax. Adhesive resin was brushed onto the metal surface and B-staged in an air circulating oven as follows:

5 min. at 85C (185F), 5 min. at 100C (212F), 5 min. at 120C (248F), and 5 min. at 145C (293F). The staged primer thickness was approxmiately .0051 cm (2 mils). The compostie adherend (a cured NR-150B2-S5X graphite laminate, 6 plies thick, was MEK wiped before and after a light abrasion using an 80 grit sandpaper. The adherend was primed identical to the steel surface. The test specimens were bonded and cured according to DuPont's instructions with the exception that only 700 kN/m$^2$ (100 psi) rather than 1400 kN/m$^2$ (200 psi) pressure was used for bonding the sandwich specimen. The ultimate compressive strength of HRH 327-2/16-6.0 glass polyimide core at 316C (600F) after 18 hours during postcuring of 316C (600F) is around 3858 kN/m$^2$ (560 psi). The bondline thickness of the cured specimens was .0203 to .0254 cm (8-10 mils).

The lap shear strength of the adhesive ranged between 6890 to 10,335 kN/m$^2$ (1000-1500 psi) at R.T. and between 4134 to 10,335 N/m$^2$ (600-1500 psi) at 316C (600F) (see Table 25). Test specimens bonded with A1100 scrim adhesive prepreg had slightly higher strength values than those with S935 scrim. However, the test results were inconclusive since the specimens failed adhesively. Figure 75 shows the test results. Discoloration of metal in the bonded area was observed, indicating the inadequacy of the prebond surface treatment. The adhesive was later evaluated using an alternate surface preparation method for bonding steel, as well as using titanium and composite adherends.

One flatwise tension test specimen bonded using the A1100 surface finish scrim-adhesive pre-preg yielded a flatwise tensile strength of 3,100 kN/m$^2$ (450 psi). The failed specimen (see Figure 76) showed that good filleting can be obtained with the adhesive when a good core/laminate contact is presented. At 10% volatile content the adhesive exhibited moderate to heavy flow (see Figure 77).

## Adhesive Selection

The selection of NR-150B2 adhesive for bonding graphite/NR-150B2-S5X polyimide composite structures was a result of extensive evaluations of elevated temperature resistant adhesive formulations. These included NR-150B2, NR056X, FM34, LARC13, and LARC160. The performance requirements for the adhesive include lap shear strength in excess of 14,000 kN/m$^2$ (2000 psi) at 316C (600F) and after 125 hours of aging at 316C (600F).

Briefly, the formulations evaluated here are catagorized as the condensation and the addition types of polyimides.

Polymerization of NR-150B2, NR056X, and FM34 is based on the condensation reaction of polyamic acid (formed by reacting aromatic carboxyllic acids or anhydrides with aromatic amines) under high temperature. Water is evolved as the condensation reaction by-product. Hence, these adhesives are termed "condensation" polyimides. A more detailed discussion on the chemistry of NR-150B2 and NR-056X can be found in References 2-4.

LARC13 and LARC160 were both developed by NASA Langley Research Center based on the polymerization of monomer reactant technology (5-8). Monomer reactants undergo condensation reactions to form nadic, end-capped imide prepolymer under high temperature. The prepolymer undergoes pryolytic polymerization, an addition reaction, under high temperature and pressure. Thus, LARC13 and LARC160 are termed "addition" polyimides. The monomer reactants for LARC13 consisted of nadic anhydride, 3.3'-methylene dianiline, and benzophenone tetracarboxylic acid anhydride. The monomer reactants for LARC160 were ethyl ester of norbornene dicarboxylic acid, Jeffamine AP-22, and diethyl ester of benzophenone tetracarboxylic acid.

The criteria for evaluating the adhesives consisted of lap shear strength at -55C (-67F), 24C (75F), and 316C (600F), shear strength retention at 316C (600F) after 160 hours of 316C (600F) exposure (thermal oxidative stability), and shear strength retention at R.T. after 20 days of exposure to 160F and 95-100 percent R.H. environment (durability in heat and humid environment).

These tests were also conducted to determine the effect of filler-to-resin ratio so that the optimal filler concentration could be used to prepare the adhesives.

## Adhesive Film Preparation

The FM34 adhesive, manufactured by American Cyanamide, was obtained as a supported film (112-style glass cloth with Volan A finish). The uncured film thickness was 0.036 cm (14-15 mils) and the volatile content was approximately 13 percent.

LARC 13 adhesive was prepared in the laboratory using resin solution obtained from the NASA Langley Research Center. The solution contained imide oligomer with 30 weight percent of aluminum powder in diglyme. This solution was brushed on 112-style glass cloth with A1100 finish and B-staged at 60C (140F) for 20 minutes and 149C (300F) for 10 minutes. These B-staging temperatures were based on prior work (2). However, the staging time was determined based on the appearance of the adhesvie film. Since the adhesvie solution was low in viscostiy, coating and B-staging were repeated three times to attain a nominal thickness of 0.030 cm (11-12 mils).

NR-150B2, NR-056X, and LARC160 were prepared by Young Lee Associates. In addition to aluminum powder (ALCOA 123, 325 mesh), 2 percent Cab-o-sil grade M-5 was added to the adhesive resins. Style 120 E-glass cloth with A1100 finish was used as the adhesive carrier.

The resin filler mixtures were stirred for several hours at 79-84C (175-185F) until the mixture was cast into a resin film. The glass cloth was then placed between the cast resin films and nip-rolled into an adhesive prepreg. The steel rollers used for this nip-rolling procedure were maintained between 82-99C (180-210F). The resultant adhesive prepregs ranged from 0.038 to 0.048 cm (15 to 19 mils) in thickness.

The volatile content of NR-150B2 and NR-056X was approximately 10-13 percent as determined by weight loss after two hours at 316C (600F) via TMA (thermal gravimetric analysis).

Volatile contents of LARC13 and LARC160 films were not measured since in the absence of cure pressure, the vorbornene moiety undergoes a reverse Diels-Alder reaction to form cyclopentadiene (Ref. 9).

## Adherend Surface Preparation

The procedures used for preparing adherends for adhesive bonding were as follows:

### Titanium

Titanium (6Al-4V-Ti) was first cleaned with MEK (methyl ethyl ketone), grit blasted with No. 320 grit glass beads, immersed in Alkaline Cleaner made of 1.13-1.36 Kg (2.5-3.0 lbs) of Turco Alkaline Rust Remover per 3.79 Ltr (1 gal.) of tap water maintained at 100C (200-212F) for 20-30 minutes, and finally, immersed in Pasa Jell 107 made of 10 percent by volume of Pasa Jell 107-C7 concentrate, 21 percent by volume 42$^{O}$Be'nitric acid, .11 Kg (0.25 lbs) of chromic acid per 3.79 Ltr (1 gal) solution at R.T. for 15-20 minutes. The adherends were primed within 4 hours after cleaning.

### Graphite/Polyimide Laminates

The Celion 6000/NR-150B2-S5X graphite composite adherends were lightly sanded with 280 grit sandpaper and MEK wiped prior to priming.

### Fiberglass/Polyimide Honeycomb Core

Fiberglass/polyimide honeycomb core (HRH327, 3/16" - 4.5 psf, 0.5 inch in thickness) were post-cured at 600F for 18 hours and vacuumed to remove dust and loose particles prior to priming.

### Bonding Procedure

BR34 primer and the FM34 adhesive were applied and cured in accordance with American Cyanamid recommended procedures. The primer brushed on the adherends was dried 30 minutes at R.T., 30 minutes at 93C (200F), and 45 minutes at 210C (410F). The cured primer thickness was approxmiately 0.005 cm (2 to 3 mils). The adhesive was cured at 316C (600F) and 280 kN/m$^2$ (40 psi) for 90 minutes. The heat-up time to 316C (600F) was 30 minutes.

LARC13 resin solution was used as the primer when bonding joints with LARC13 film adhesive. The primer brushed on the adherends was dried 20 minutes at 60C (140 F) and 10 minutes at 149C (300F) to a dried film thickness of 0.009 to 0.023 cms (0.2 to 0.5 mils). The adhesive was cured under a cure cycle of 60 minutes at 248C (480F), 30 minutes at 288C (550F), and 700 kN/m$^2$ (100 psi) bonding pressure, followed by a 16-hour free-standing postcure in a 316C (600F) oven.

NR-150B2 solution containing 30 weight percent of aluminum powder was used as the primer for the NR-150B2, NR-056X, and LARC160 adhesives. The applied primer was dried 30 minutes at R.T., 20 minutes at 121C (250F), and 20 minutes at 204C (400F).

The dried primer thickness was approximately 0.004 to 0.009 cm (1 to 2 mils).

A cure cycle developed by DuPont was used for NR-150B2 and NR-056X adhesives (4). This consisted of placing the specimens under full vacuum and 1400 $kN/m^2$ (200 psi), and heating to 316C (600F) at a rate of 1.7 to 2.8 C (3 to 5F)/minute, and then holding for 60 minutes at 316C (600F). The pressure was then released and the specimen held another 60 minutes under vacuum at 316C (600F). They were then cooled to R.T. under vacuum, and post-cured for 16 hours at 316C (600F) in an air-circulating oven. For bonding honeycomb core sandwich assemblies, the pressure was reduced to 700 $kN/m^2$ (100 psi).

The cure cycle used for LARC 160 consisted of placing the specimens under vacuum heating to 177C (350F) at 1.7 to 2.8C (3-5F)/minute, holding the specimen for 60 minutes at 177C (350F) under vacuum and 350 $kN/m^2$ (50 psi), then cooling to R.T. under vacuum and pressures, and then postcuring 10 hours at 316C (600F) in an air-circulating oven.

## Testing

Lap shear tests were conducted in an Instron test machine per ASTM D1002 at a crosshead speed of 0.13 cm (0.05 inch)/minutes. Specimens were conditioned at the test temperature for 10 minutes prior to testing.

Heat aging of test specimens at 316C (600F) was conducted in an air circulating oven maintained at 316C (600F).

The glass transition temperature, Tg, of the cured adhesives was measured using thermal mechanical analysis (TMA) at a heat-up rate of 20C (68F)/minute.

Flatwise tensile shear tests of honeycomb core sandwich assemblies were conducted in accordance with ASTM C297.

## Results and Discussion

The effect of filler-to-resin weight ratio, i.e. 63:35 and 35:65 on adhesive bond strength was investigated based on NR-056X and LARC160 resins. The test results presented in Table 26 showed that 65:35 filler-to-resin ratio by weight imparted slightly higher strength at 316C (600F) for both adhesives and slightly better strength retention after aging at 316C (600F) for NR-056X. The amount of filler had no effect on the durability of the adhesives.

Based on these results, 65:35 filler-to-resin ratio by weight was used in preparing NR-150B2, NR-056X, and LARC160 adhesive films.

Adhesive films prepared as described had moderately good drape and tack. Handling and laying up of the uncured adhesive films posed no difficulties.

The test results (see Table 27) showed the condensation polyimides were better in strength retention after 160 hours of exposure to 316C (600F) when compared to the addition polyimides. NR-150B2, NR-056X, and FM34 virtually retained 100 percent of their lap shear strength when tested at 316C (600F) after 160 hours of exposure to 316C (600F); whereas LARC13 and LARC160 showed only 60 percent strength retention. NR-150B2 passed the test among the condensation polyimides in terms of strength from -55C (-67F) to 316C (600F), while LARC160 exhibited higher strength than LARC13 at -55C (-67F) and at R.T. However, the shear strength at 316C (600F) and the effect of aging for 160 hours at 316C (600F) were comparable for both adhesives. This correlated well to the Tg measured. Tg of NR-150B2 and NR-056X were greater than 316C (600F), while LARC160 was approxmiately

293C (560F). Thus, it seemed that LARC13 and LARC160 were not amenable for bonding applications where the intended service temperature exceeded the Tg of the adhesives; certainly not for service at 316C (600F).

Since only NR-150B2 seemed to marginally meet the performance requirements previously mentioned, it was selected for adhesive bonding of graphite/polyimide laminates and glass/polyimide honeycomb core sandwich assemblies.

NR-150B2 used for bonding graphite/polyimide (Celion 6000/NR-150B2-S5X) laminates to HRH 327 glass/polyimide honeycomb core sandwich assemblies was evaluated using flatwise tensile and shear tests. Five out of six test specimens failed in the laminate face sheets at a failing stress level ranging from 770 to 1820 kN/m$^2$ (110 to 260 psi). These specimens did not measure the bond strength of the sandwich assemblies. One remaining specimen failed approximately 50 percent cohesive, 50 percent adhesive (actually lacking good laminate to core contact during cure as shown in Figure 78 and delamination in the composite face sheets was also observed (see Figure 79). The flatwise tensile strength of this specimen was 3150 kN/m$^2$ (450 psi).

The effect of long term heat aging of NR-150B2 adhesive at 316C (600F), was evaluated using titanium and composite single overlap shear test and titanium and composite double overlap shear test. The test results are summarized in Table 28.

The test results showed lap shear strength at 316C (600F), when bonding either titanium or graphite/polyimide. (Celion 6000/NR-150B2) adherends, to be approximately 11,200 kN/m$^2$ (1,600 psi) (Figure 80). With aging at 316C (600F) the titanium lap shear strength at R.T. dropped nearly 25 percent from 17,150 kN/m$^2$ (2,540 psi) to 13,720 kN/m$^2$ (1,960 psi) after 200 hours of exposure. The graphite/polyimide composite lap shear strength at R.T. dropped nearly 50 percent from 20,580 kN/m$^2$ (2,940 psi) to

10,920 kN/m$^2$ (1,560 psi) after 200 hours of exposure to 316C (600F). The lap shear strength at 316C (600F), bonded either with titanium or composites, showed almost no strength loss after 200 hours of aging at 316C (600F).

A similar trend was also observed with the double overlap shear test results. The lap shear strength of the adhesive at R.T. dropped approximately 35 percent after 200 hours of aging at 316C (600F) see Table 27. The lap shear strength of the adhesive at 316C (600F) was less affected by heat aging at 316C (600F).

Unless otherwise noted (i.e., the composite/composite double overlap shear specimens), almost all the specimens failed cohesively. The heat aged specimens showed failure occurred uniformly at the adhesive/glass cloth carrier interface. This seemed to indicate additional work should be conducted in the future in improving adhesive to cloth carrier compatibility and thereby improve the strength of the adhesive bond.

## 5.2 SOLID LAMINATE TEST DATA

Solid 8, 12, and 24 ply C-6000/NR-150B2-S5X laminates were fabricated from 9 different lots of prepreg from two different suppliers. The following precompaction, cure, and postcure cycles were used:

### Precompaction

Apply full vacuums at RT, heat to 132C (270F) at 2.2-2.8C/minute (4-5F/minute). Hold 90 minutes, cool under vacuum to below 52C (125F) at a rate not to exceed 2.8C/minute (5F/minute).

### Cure

Apply full vacuum at RT, heat to 149C (300F) at 2.2-2.8C/minute (4-5F/minute), apply 1400 kN/m$^2$ (200 psi), hold 30 minutes, heat to 204C (400F) at 2.2-2.8C/minute (4-5F/minute), hold for 120 minutes, cool under vacuum and 1400 kN/m$^2$ (200 psi) to below 65C (150F) at a rate not to exceed 2.8C/minute (5F/minute).

Postcure

Apply full vacuum and 1400 kN/m$^2$ (200 psig) at room temperature, heat to 177C (350F) at 2.2-2.8C/minute (4-5F/minute), heat to 288C (550F) at 0.6C/minute (1F/minute), heat to 399C (750F) at 0.3C/minute (0.5F/minute), hold eight hours, cool under vacuum and 1400 kN/m$^2$ (200 psi) to below 65C (150F) at a rate not to exceed 1.7C/minute (3F/minute).

Cutting plans (Figures 81-85) were developed for all unidirectional and multiaxis laminates for the development of the design data. This allowed for correlation of specimen location to the C-scan results if their was any erractic data. The data obtained was very uniform. It also ensured that room temperature and 316C (600F) tests of aged and nonaged specimens were taken uniformly throughout the laminate. All lamintes were C-scanned after postcure. Tables 29 and 30 identify the laminates for the test program. Typical C-scans are shown in Figures 86-88.

Photomicrographs were also taken prior to and after the 200 hour heat-aging at 316C (600F) (Figures 89 through 91). The photomicrographs show no change in laminate appearance after thermal aging. The measured glass transition temperature of this laminate was 374C (Figure 92). Thermal gravimetric analyses showed less than 0.2 percent volatiles remaining after postcure.

Unidirectional Mechanical Properties

Tensile tests were conducted on an eight ply laminate C-532; refer to Table 31 for individual test values. A reduction of less than 3 percent was seen when comparing room temperature properties of control specimens and specimens that had been aged for 200 hours at 316C (600F). A 12 percent reduction was seen when tested at 316C (600F): Table 32 shows the tensile properties for laminate C-532 after being aged at 316C (600F) for 200 hours. A comparison of nonaged and aged specimens is shown in Figure 93.

Flexural properties were conducted on a 12-ply laminate C-533 for quality assurance data. Refer to Tables 33 and 34 for test data. Short beam shear values were also obtained on this laminate. By comparing test data of control specimens and specimens aged at 316C (600F) for 200 hours, the maximum property degradation was established. However, as with most laminates tested during this program, a rather dramatic drop was seen in room temperature properties versus 316C (600F) properties. In this case, the resin dominated properties, had an approxmiately 40 percent loss in ultimate strength. The calculated void content for this laminate was 1.3 percent. Figure 94 shows the relationship of tensile strength, tensile modulus, and short beam shear strength for this laminate.

A 24-ply laminate was fabricated for compression specimens. This laminate did not scan well in all areas. Specimens were machined from areas that were considered acceptable. Again, very little loss of strength was seen when comparing nonaged and aged specimens at a given temperature but a loss was observed when comparing room temperature and 316C (600F) values. Refer to Tables 35 and 36 for individual specimen data and to Figure 95 for a comparison of nonaged and aged specimens.

## Crossplied Mechnical Properties

An eight-ply laminate, C-535, was fabricated to determine tensile specimens of a multiaxis laminate. As shown in Tables 37 and 38, the mean values follow the same trend of negligible property loss after aging when comparing test temperatures. Figure 96 shows a comparison of nonaged and aged specimens.

## Adhesive Bonding Laminate Property Verification

A 12-ply unidirectional laminate (C-536) and 16-ply multiaxis laminate (C-537) were fabricated for the adhesive bonding evaluation. From these, laminate flexural specimens were machined and tested to verify resin-dominated mechanical properties, as control specimens and after exposure at 316C. (600F). Both laminates followed the same basic trend that was seen on the previous laminates. Temperature exposures of 100 and 200 hours at 316C (600F) had little effect on flexural properties. Whereas control and aged specimen values of room temperature versus 316C (600F) tests had the typical 40 percent decrease. Refer to Tables 39 and 40 for individual specimen values and to Figure 97 for mean trends.

# 6.0

# TASKS D, E, AND F-FABRICATION OF DEMONSTRATION COMPONENTS

The objective of these tasks was to demonstrate that typcial flat laminates and structural shapes such as I's and hats can be fabricated from C-6000/NR-150B2-S5X graphite/polyimide composite materials.

## 6.1 STRUCTURAL ANALYSIS

Panels were fabricated and tested to support the design of the stringer panels. The skin/stringer compression panels (tee and hat) were sized by laminate crippling data to have an ultimate failure load of 3000 lbs/in., with a length of 48 in. and end fixity of 3.6. The wide column critical Euler load was well above the crippling load in both cases. Element buckling loads were also well above element crippling points.

As a starting point in the sizing of the required skin/stringer section, a known optimum isotropic section was used. This optimum isotropic section (aluminum) was then resized using a pseudoisotropic skin and orthotropic stringers. The resizing is an iterative process constrained by given skin material properties and thickness as well as stringer material properties. The resulting sections are shown in the following figures, with a wide column analysis of each.

Wide Column Analysis: Tee

$t_s = 0.06$ in., $b_s = 1.50$ in.

$t_w = t_f = 0.08$ in., $b_w = 1.50$ in., $b_f = 0.75$ in.

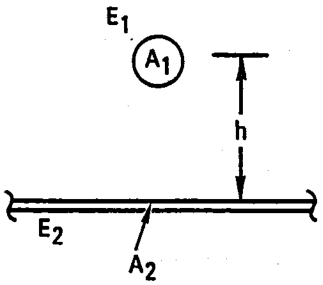
$$E_R = \frac{E_{stringer}}{E_{skin}} = 1.26$$

Area of Skin, $A_2 = t_s b_s = 0.09$ in.$^2$

Area of Stringer, $A_1 = 0.18$ in.$^2$

$$\frac{A_2}{A_1} = 0.5$$

$h = {}^{b_w}/_{1.5} = 1.0$ in.



$$\frac{I_{eff}}{in} = \frac{h^2 A_2}{b_2}\left[\frac{E_R}{E_R + \frac{A_2}{A_1}}\right] = 0.043 \text{ in/in}$$

$$N_{X_{cr}} = \frac{\pi^2 EIC}{L^2} = 5968 \text{ lbs/in}$$

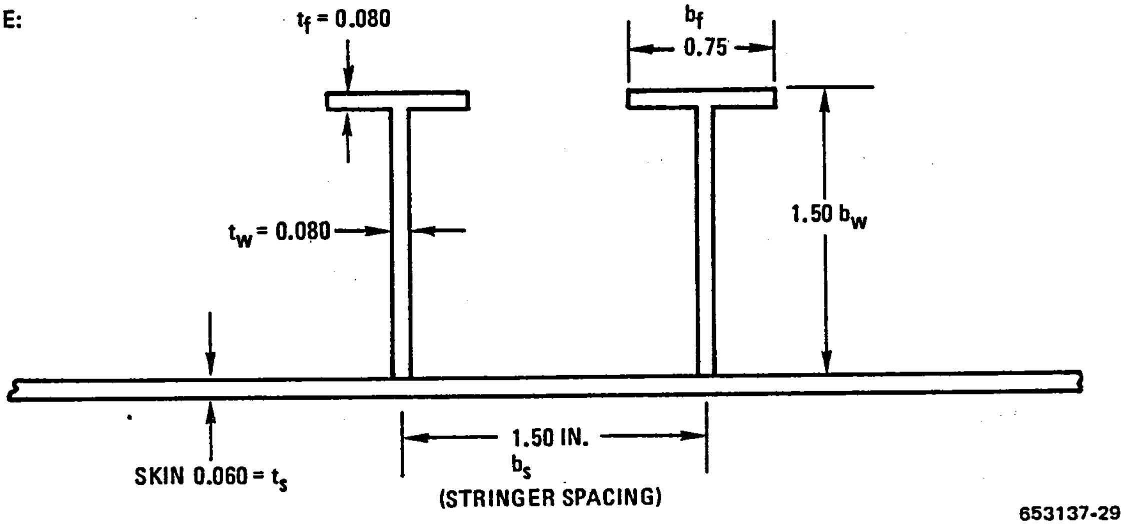
$$\left.\frac{\sigma_{eff}}{\sigma_{cc}}\right|_{skin} = \frac{14200}{16500} = 0.86,\ b_s/t_s = 25$$

$$\left.\frac{\sigma_{eff}}{\sigma_{cc}}\right|_{web} = \frac{17900}{32000} = 0.56, \frac{b_w}{t_w} = 18.75$$

$$F_u \Big|_{skin} = 38750 \text{ psi} >> 14,200 \text{ psi}$$

$$F_u \Big|_{skin} = 68200 \text{ psi} >> 17,900 \text{ psi}$$

TEE:



Layups:

Skin — $(45, 0, 90, 135, 0, 45)_s$, 12 ply

Tee — $(45, 0, -45, 0, 0, 45, 0, 135)_s$, 16 ply

Wide Column Analysis: Hot

$t_s = 0.06$

Stringer spacing, $b_{ss} = 4.95$

$t_w = 0.08$

$b_w = b_c = 2.20$

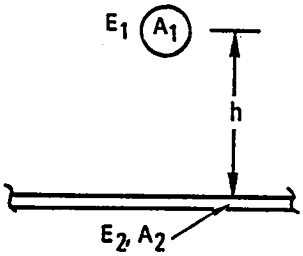
$b_f = 0.66$

$A_2 = t_s b_{ss} = 0.297$

$A_1 = 0.634 = t_w (2b_w + b_c + 2b_f)$

$$\frac{A_2}{A_1} = 0.47$$

$$h = \frac{t_w \frac{b_f}{b_w} + b_w + 2b_c}{4\frac{b_f}{b_w} + 4 + 2\frac{b_c}{b_w}} = 0.92 \text{ in.}$$



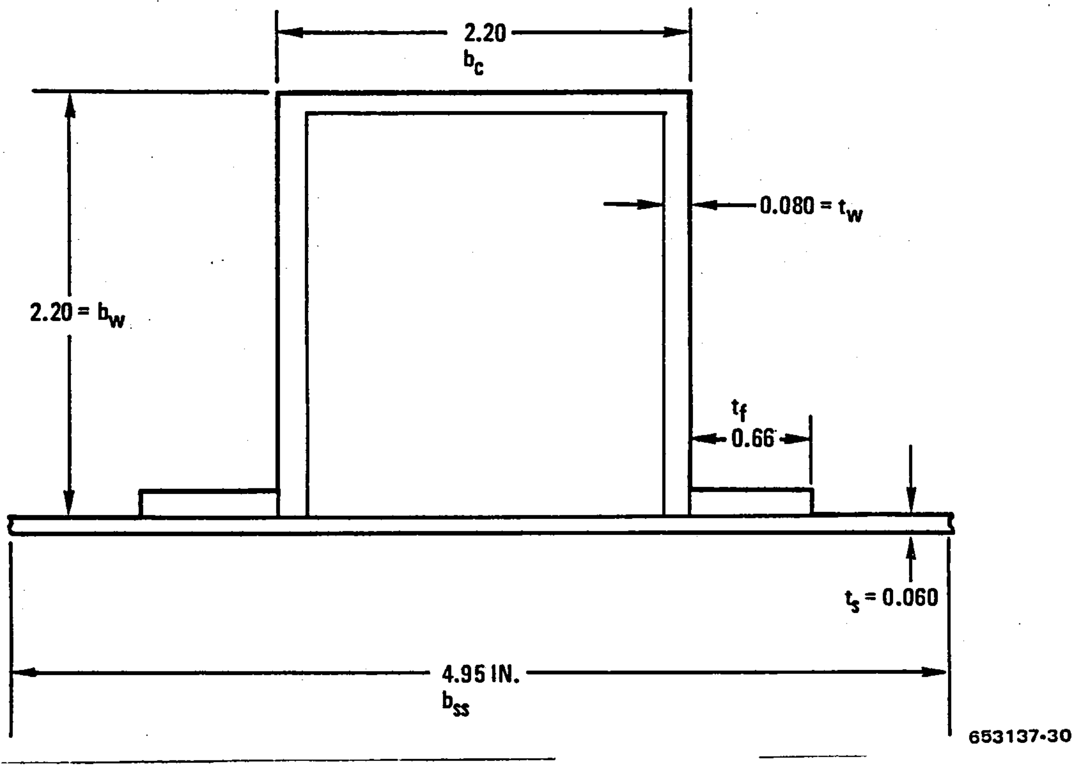
$$E_R = \frac{E_1}{E_2} = 0.037 \frac{\text{in}^4}{\text{in.}}$$

$$\frac{I_{eff}}{in.} = \frac{h^2 A_2}{b_{ss}} \left[ \frac{E_R}{E_R + \frac{A_2}{A_1}} \right] = 0.037 \frac{in^4}{in.}$$

$$N_{X_{cr}} = \frac{\pi^2 EIC}{L^2} = 5135 \text{ lbs/in.}$$

$$\left.\frac{\sigma_{eff}}{\sigma_{cc}}\right|_{skin} = \frac{13400}{14200} = 0.94, \frac{b_c}{t_s} = 36.7$$

$$\left.\frac{\sigma_{eff}}{\sigma_{cc}}\right|_{web} = \frac{17000}{22000} = 0.77, \frac{b_w}{t_w} = 27.5$$



Layups:

Skin, $(45, 0, 90, 135, 0, 45)_S$

Stringer, $(45, 0, -45, 0_2, 45, 0, 135)_S$

A plot of the crippling data used to size the sections, and assumed material properties used to calculate local buckling points is shown in Figure 98 and Table 41.

## 6.2 FABRICATION OF COMPONENTS

Bulk graphic tooling was used for the hat and "I" section. As shown in Figures 99 and 100, one of each tool configuration had a 1.29 cm (0.5 inch) cartridge heater running the length of the tool. This allowed the tool to be heated to approximately 77C (170F) to aid in the forming of the prepreg. The part was laid up on the heated tool and then allowed to cool and be transferred to the cure tool which was not heated. This freed the heated tool for additional layups. The rigidity of the formed prepreg allowed us to transfer the composite material from one tool to the other without damaging the layup.

Hat and "I" channels of the following ply configuration $(45, 0, -45, {}_2, 45, 0, 135)_S$, a total of sixteen plies thick are subjected to a precompaction operation. Between each eight plies, or one-half of the layup, one ply of porous armalon breather and one ply of 181 fiberglass bleeder are positioned. The 181 fiberglass is extended to the vacuum port and the entire layup is covered with a nylon vacuum bag. The vacuum bagged hat or "I" section was heated to 140C (285F) at 1-1.8C/minute (2-3F per minute) and held at full vacuum for 45 minutes. The layup was allowed to cool under vacuum to 43C (110F) and was then removed from the oven. Bleeder materials were removed, and the final eight plies were laid down on top of the precompacted first eight plies. The second eight plies were then subjected to the same precompaction operation.

For final cure, the bagging sequence was: one ply of porous armalon over the layup, two plies of 181 fiberglass bleeder, semi-porous barrier film (Celgard), one ply 181 fiberglass breather extended to the vacuum port, and vacuum bagged using vacalloy and a high temperature bag sealant.

The hats, and "I" channels were then cured accordingly:

1. Apply full vacuum to part at room temperature.
2. Heat to 162C (325F) at 1.-1.8C/minute (2-3F/minute).
3. Apply 175 kN/m$^2$ (25 psi) and dwell 30 min. at 162C (375F).
4. Heat to 216C (420F) and hold for 30 min.
5. Apply 1050 kN/m$^2$ (150 psi) pressure to the part and heat to 302C (575F) at 1 to 1.8C/minute (2 to 3F/minute), hold one hour.
6. Heat to 343C (650F) at 1 to 1.8C/minute (2 to 3F/minute), hold one hour.
7. Heat to 371C (700F) at 1 to 1.8C/minute (2 to 3F/minute), hold five hours.
8. Cool under pressure and vacuum to 65C (150F) and remove from the autoclave.

Most fabricated parts were then subjected to a post cure which required rebagging of the part in a vacalloy bag and post curing at 371C (700F) for 16 hours under vacuum and pressure of 700 to 1050 kN/m$^2$ (100-150 psi).

Adhesive bonding of the hat and "I" channels to the flat composite panels was accomplished according to the following procedure.

1. Abrade the graphite/polyimide surfaces to be mated with scotch brite, solvent wipe with MEK and air dry for 30 minutes.
2. Prime the adherends with NR-150B2 resin solution and allow to air dry 15 minutes, at 121C (250) for 20 minutes, and for 20 minutes at 205C (400F).
3. Bond surfaces together by placing under full vacuum and 1050 kN/m$^2$ (150 psi) pressure and heat to 316C (600F) at a rate of 1.8-2.8C/minute (3-5F/minute). Hold under these conditions for two hours, then cool to room

temperture before releasing vacuum and pressure. The bonded structure is then post cured free standing for 16 hours at 316C (600F).

Large flat laminates, honeycomb panels, "I" stiffened panels, and hat stiffened panels were fabricated and submitted to NASA Langley Research Center for evaluation. At this point the NR-150B2 resin binder was taken off the market and the large demonstration component could not be fabricated.

# 7.0

# CONCLUSIONS AND RECOMMENDATION

## 7.1 CONCLUSIONS

1. The NR-105B2 polyimide resin binder was found to varing in its advancement significantly depending on previous time and temperature history during the prepregging operation.

2. Strength retention at 316C (600F) was above 50% when compared to room temperature strength for the C-6000/NR-150B2-S5X graphite/polyimide composites.

3. Thermal stability at 316C (600F) for the C-6000/NR-150B2-S5X composites was demonstrated for at least 200 hours with no apprent loss in either room or elevated temperature strength after exposure.

4. A significant amount of basic resin chemistry studies are required for determining NR-150B2 resin binder quality assurance parameters before it could be considered to be reliable.

5. Graphite fibers are available which can withstand the high temperatures required for curing and postcuring the NR-150B2 resin system.

## 7.2 RECOMMENDATION

1. Studies should be continued for the development and evaluation of high temperature composites for use on future missile and aircraft structures.

2. Any resin system selected for evaluation should be available from more than one material supplier.

# 8.0

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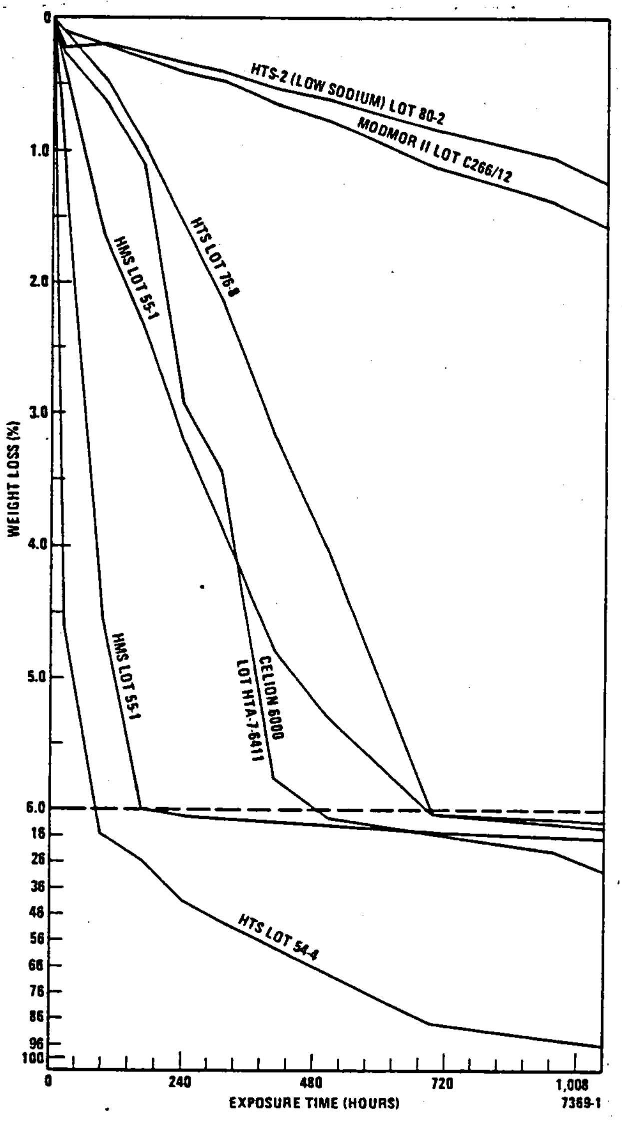


Figure 1. Weight Loss Versus Time At 316C (600F) For Graphite Fiber.

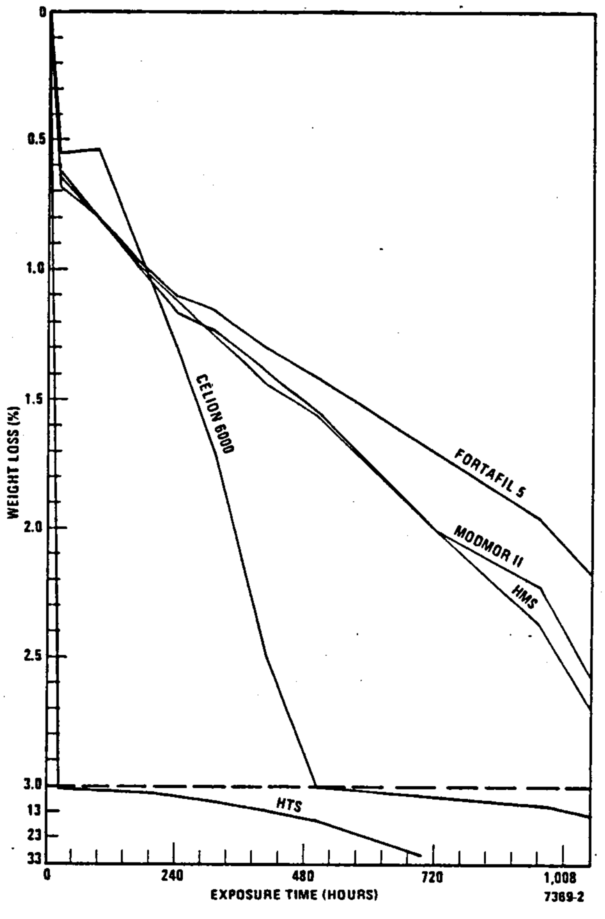


Figure 2. Weight Loss Versus Time At 316C (600F) For Graphite/ NR-150B2 Polyimide Laminates

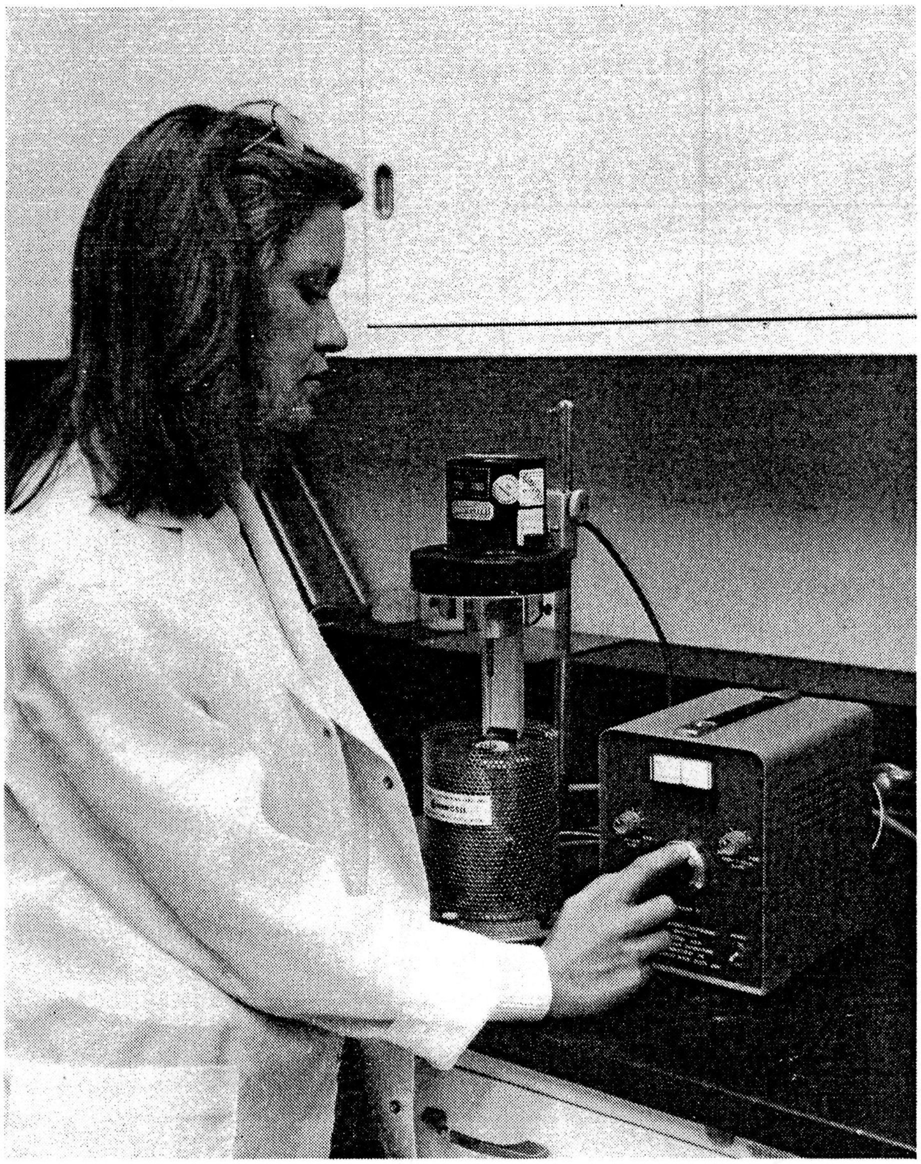


Figure 3. Determination of Viscosity Versus Temperature for NR-150B2 Polyimide Solutions

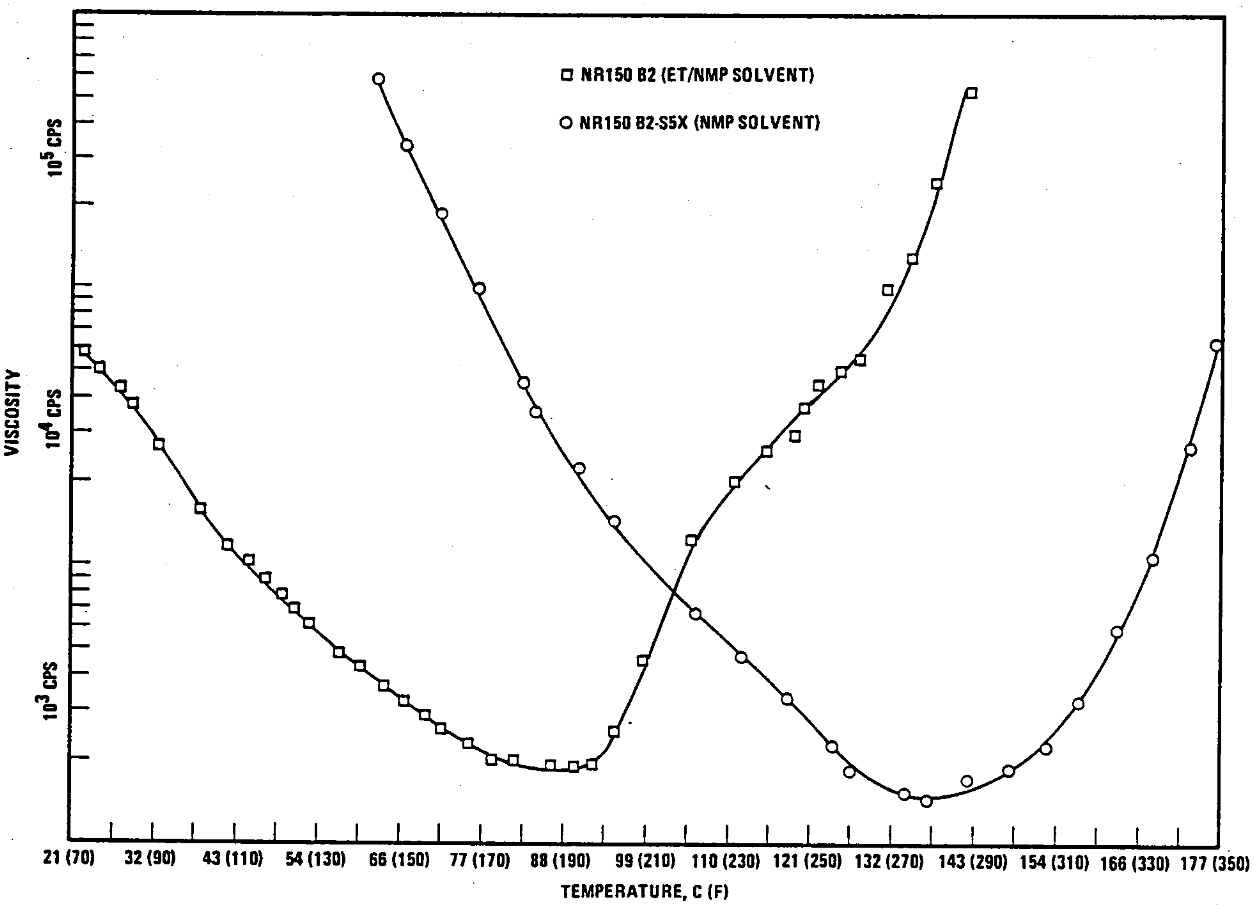


Figure 4. Viscosity Versus Temperature for NR-150B2 Solutions

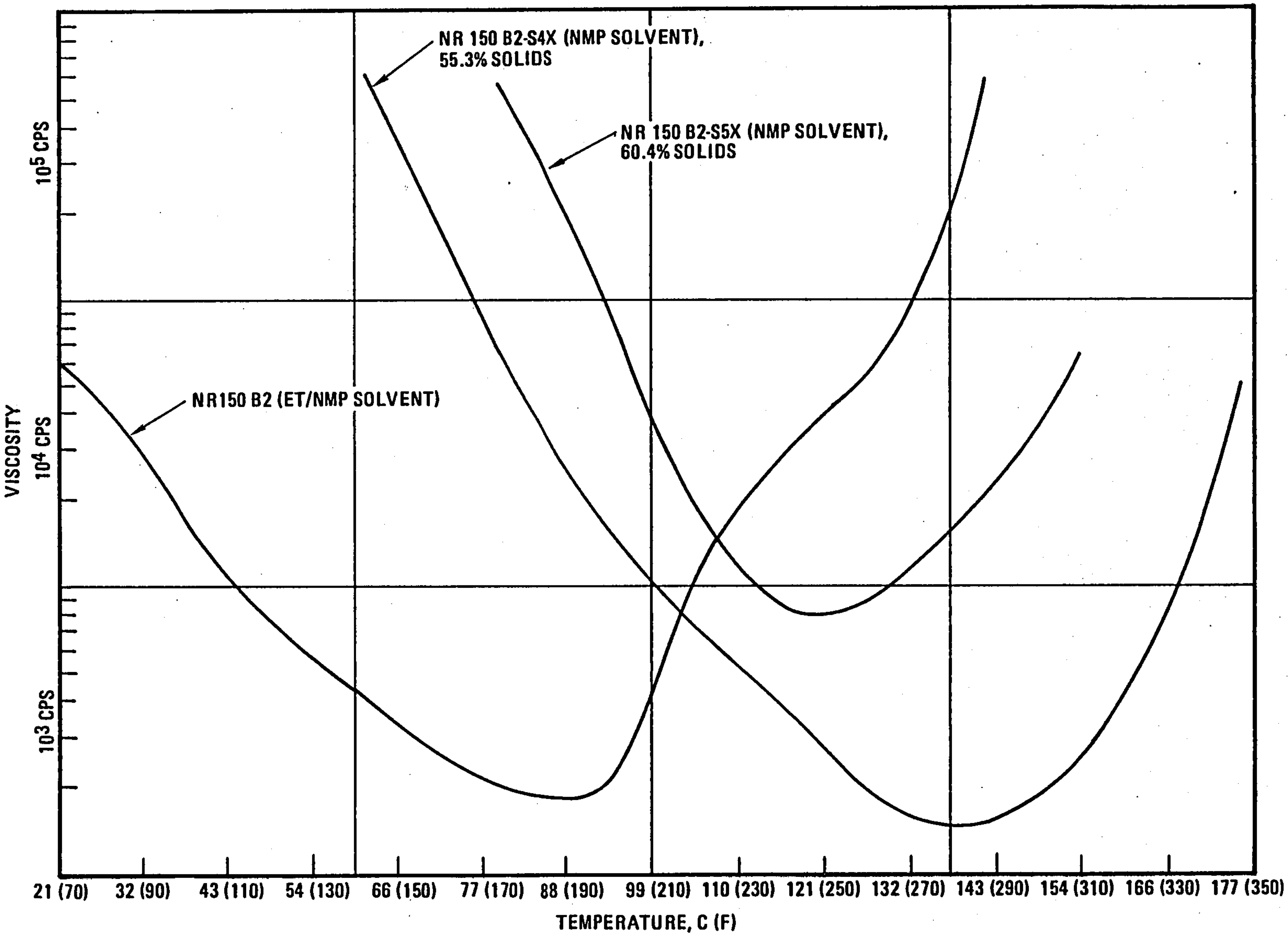


Figure 5. Viscosity Versus Temperature for NR-150B2 Solutions

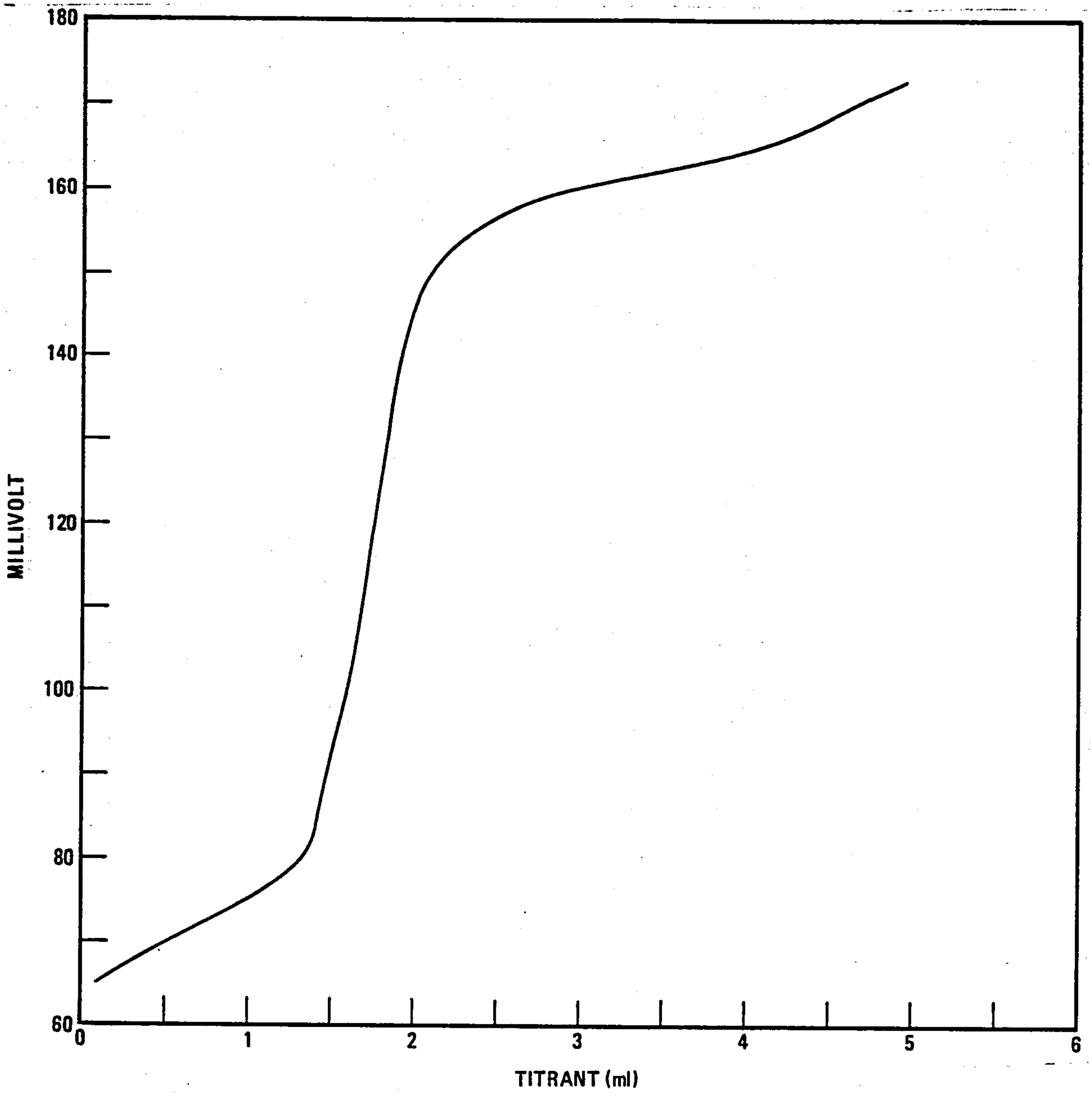


Figure 6. Acid Titration Curve for NR-150B2-S5X Resin Solution (Combined Batches E14224-76 and E14224-78)

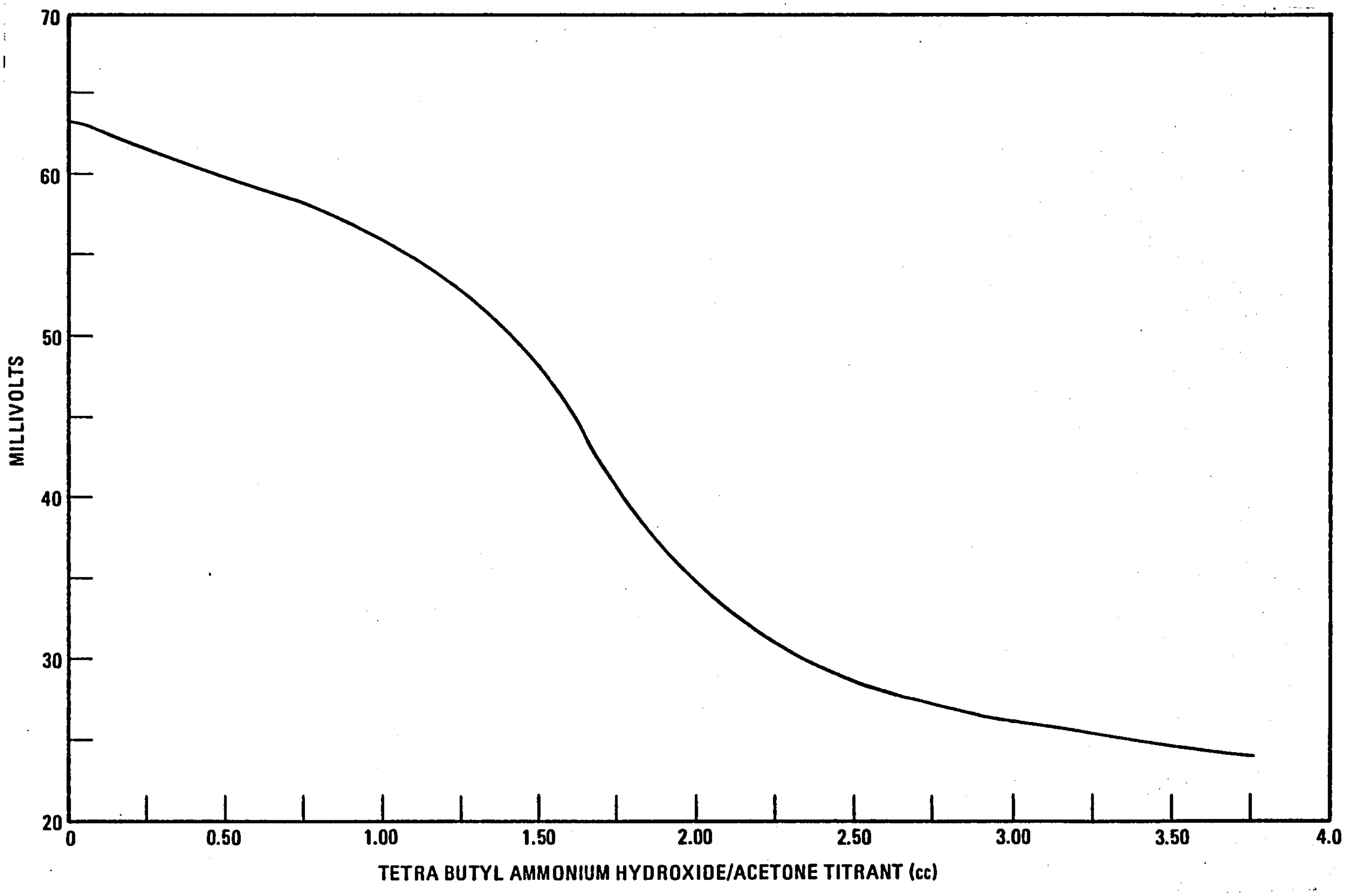


Figure 7. Amine Titration Curve for NR-150B2-S5X Resin Solution (Combined Batches E14224-76 and E14224-78)

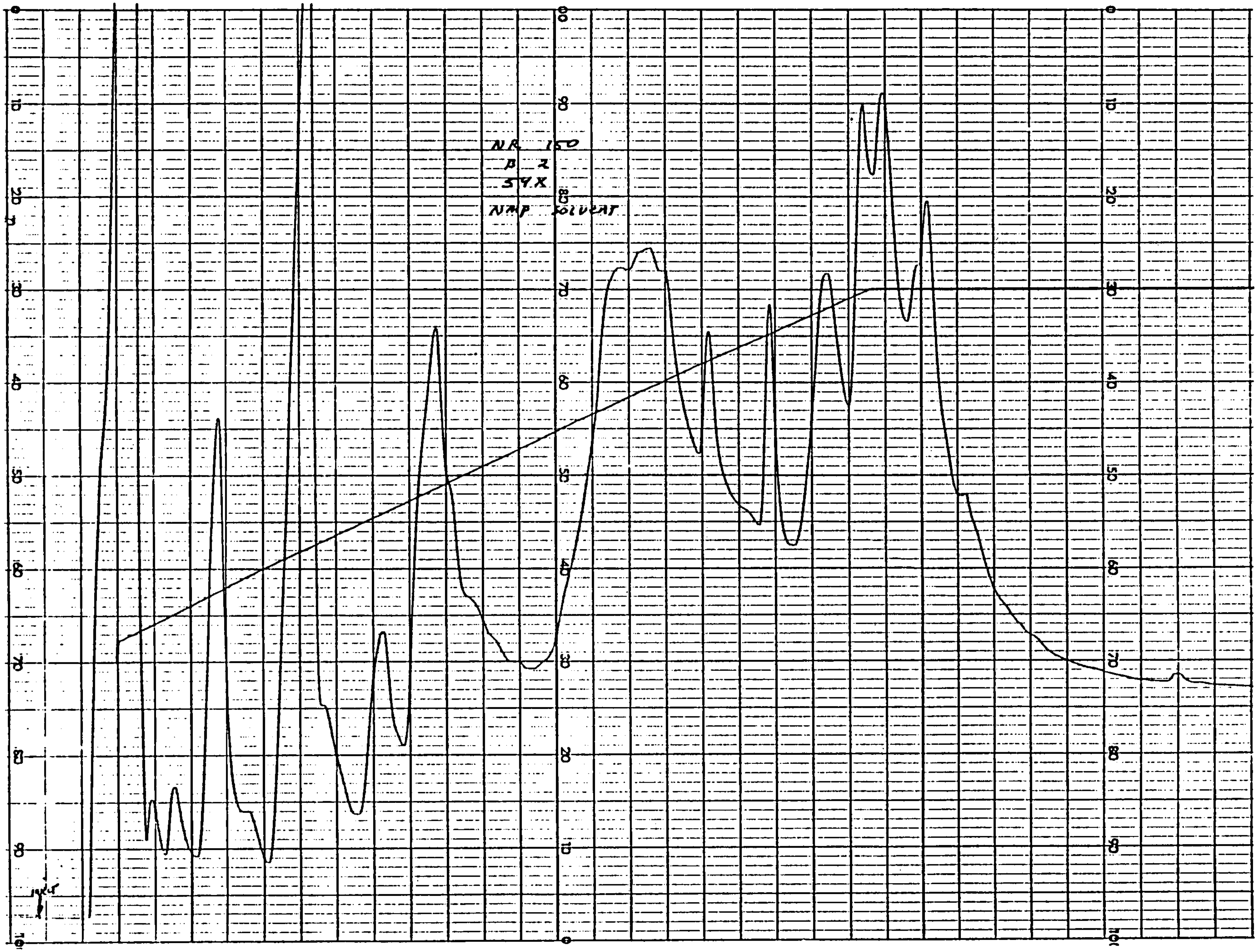


Figure 8. H.P.L.C. Scan for NR-150-B2-S5X Resin Solution (55% in NMP) Used for Initial Lot (6B-74) of Modmor II Prepreg

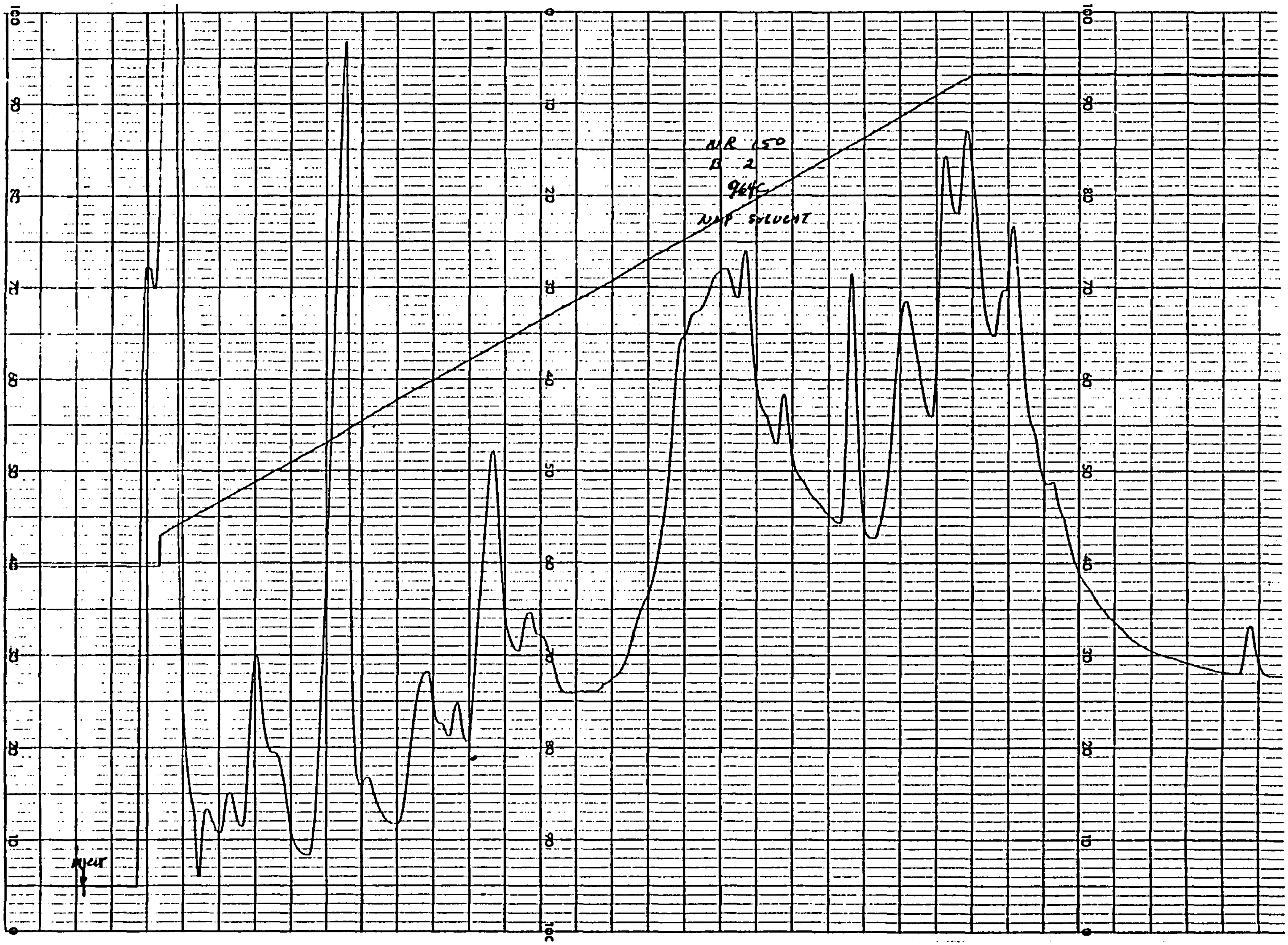


Figure 9. H.P.L.C. Scan for NR-150-B2-S5X Resin Solution (60% Solids in NMP) Used for Recent Lots of Celion 3000 (Lot 6D-26) and Modmor II (Lot 6C-55)

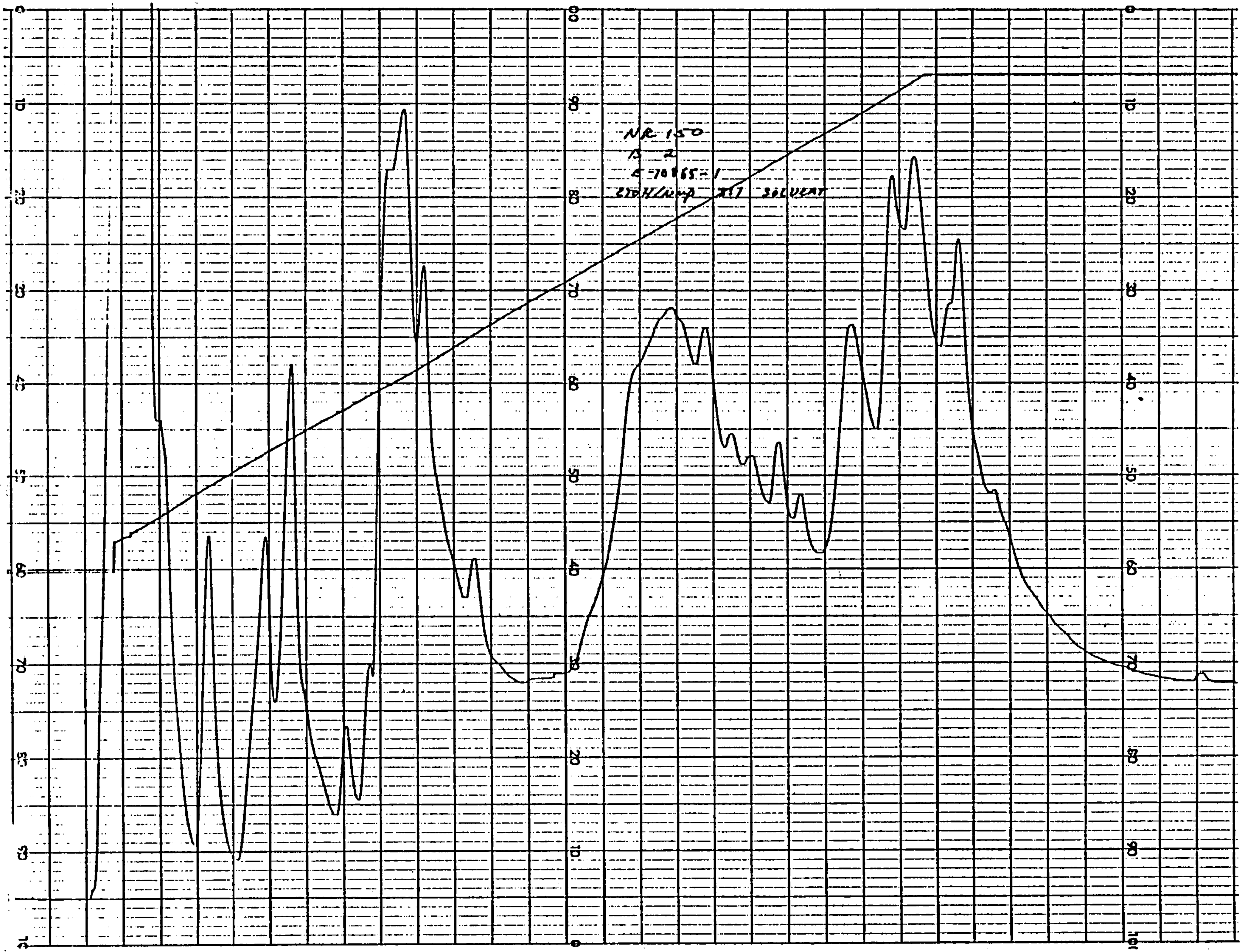
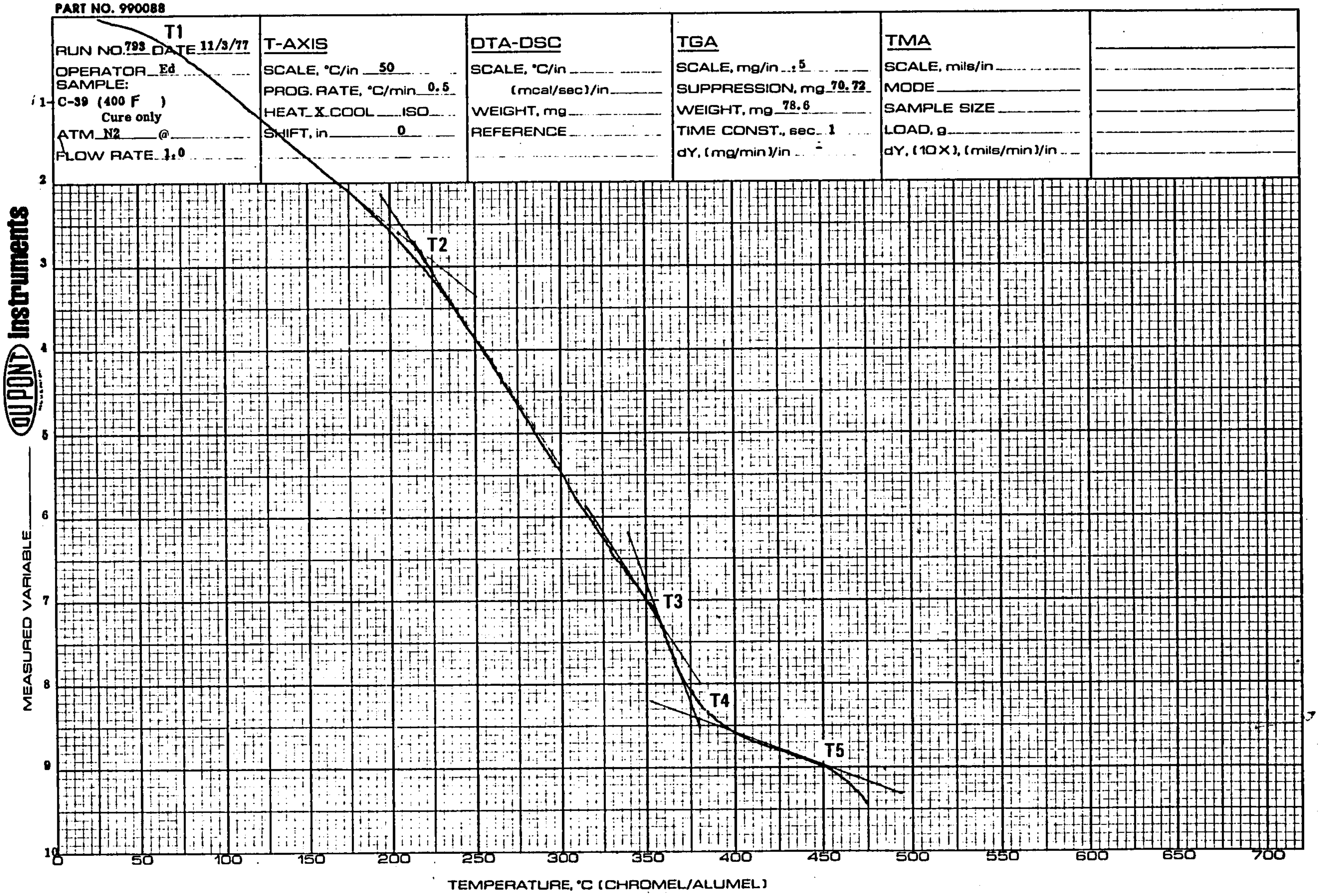


Figure 10. H. P. L. C Scan for NR-150B2 Based on ETOH/NMP Solvent



PART NO. 990088

RUN NO. 793 DATE 11/3/77

OPERATOR Ed

SAMPLE: C-39 (400 F ) Cure only

ATM N2 @

FLOW RATE 1.0

T-AXIS

SCALE, °C/in 50

PROG. RATE, °C/min 0.5

HEAT X COOL ISO

SHIFT, in 0

DTA-DSC

SCALE, °C/in

(mcal/sec)/in

WEIGHT, mg

REFERENCE

TGA

SCALE, mg/in .5

SUPPRESSION, mg 70.72

WEIGHT, mg 78.6

TIME CONST., sec 1

dY, (mg/min)/in -

TMA

SCALE, mils/in

MODE

SAMPLE SIZE

LOAD, g

dY, (10X), (mils/min)/in

Figure 11. Weight Loss During Postcure by TGA

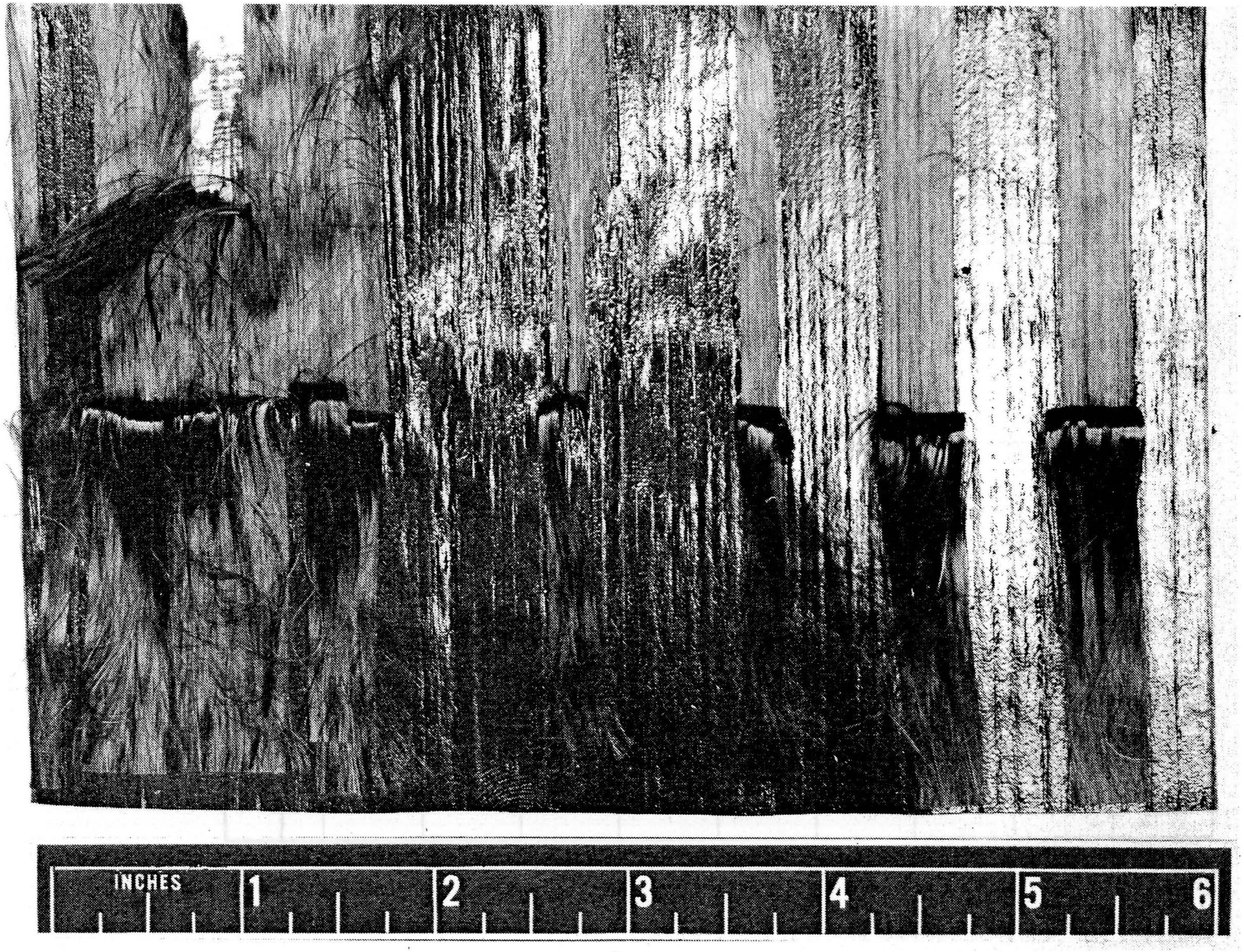


Figure 12. Dry Internal Fibers Due to Poor Impregnation of Prepregnation

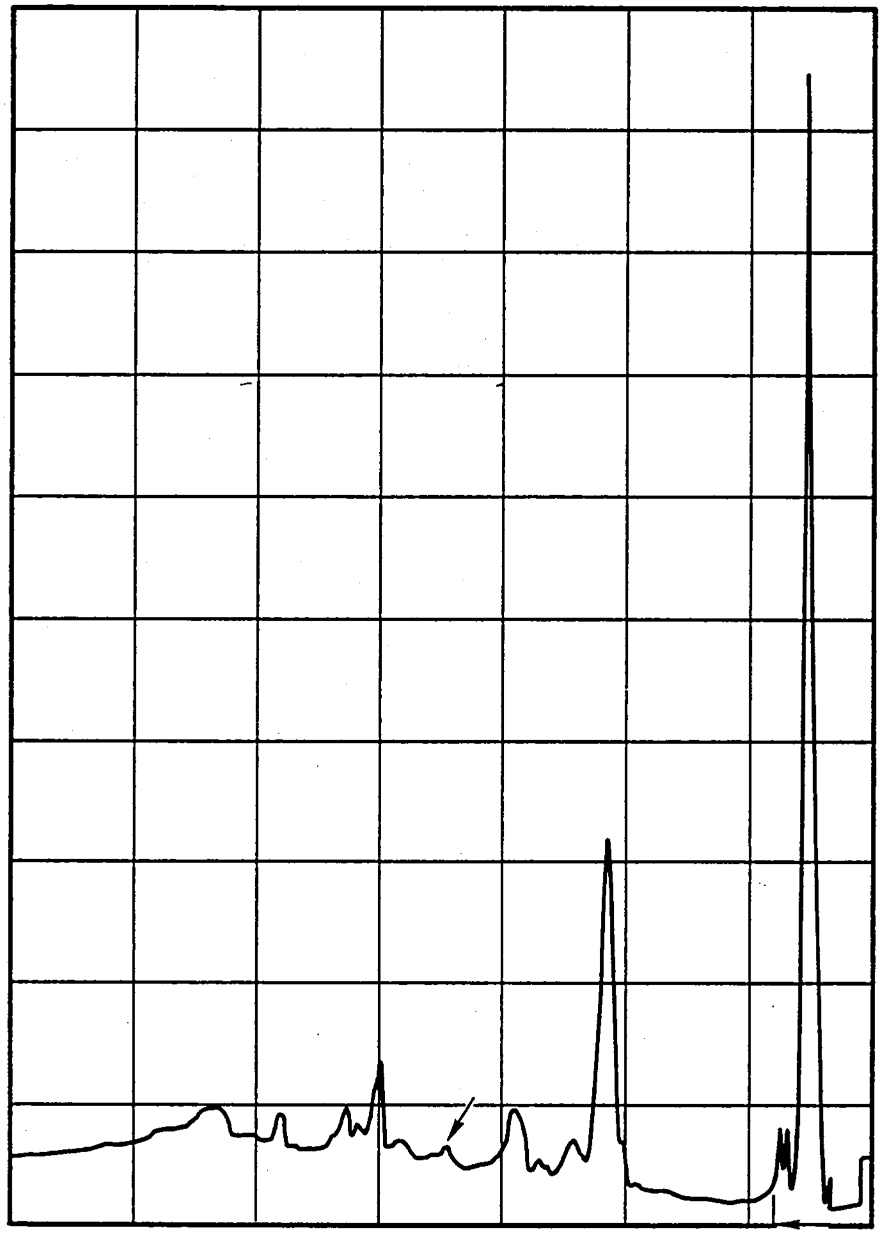


Figure 13. HPLC Scan for Resin E-14224-34

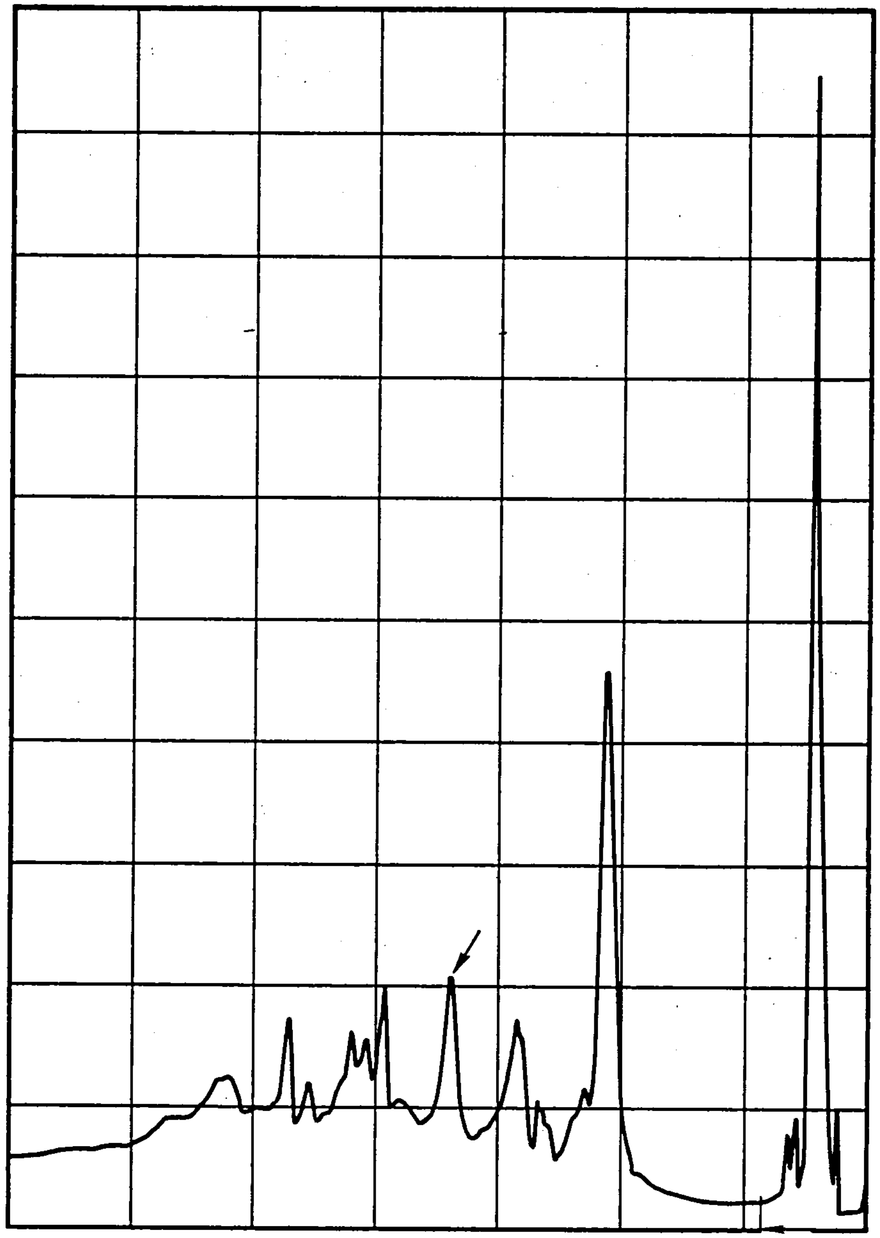


Figure 14. HPLC Scan for Resin 964-C

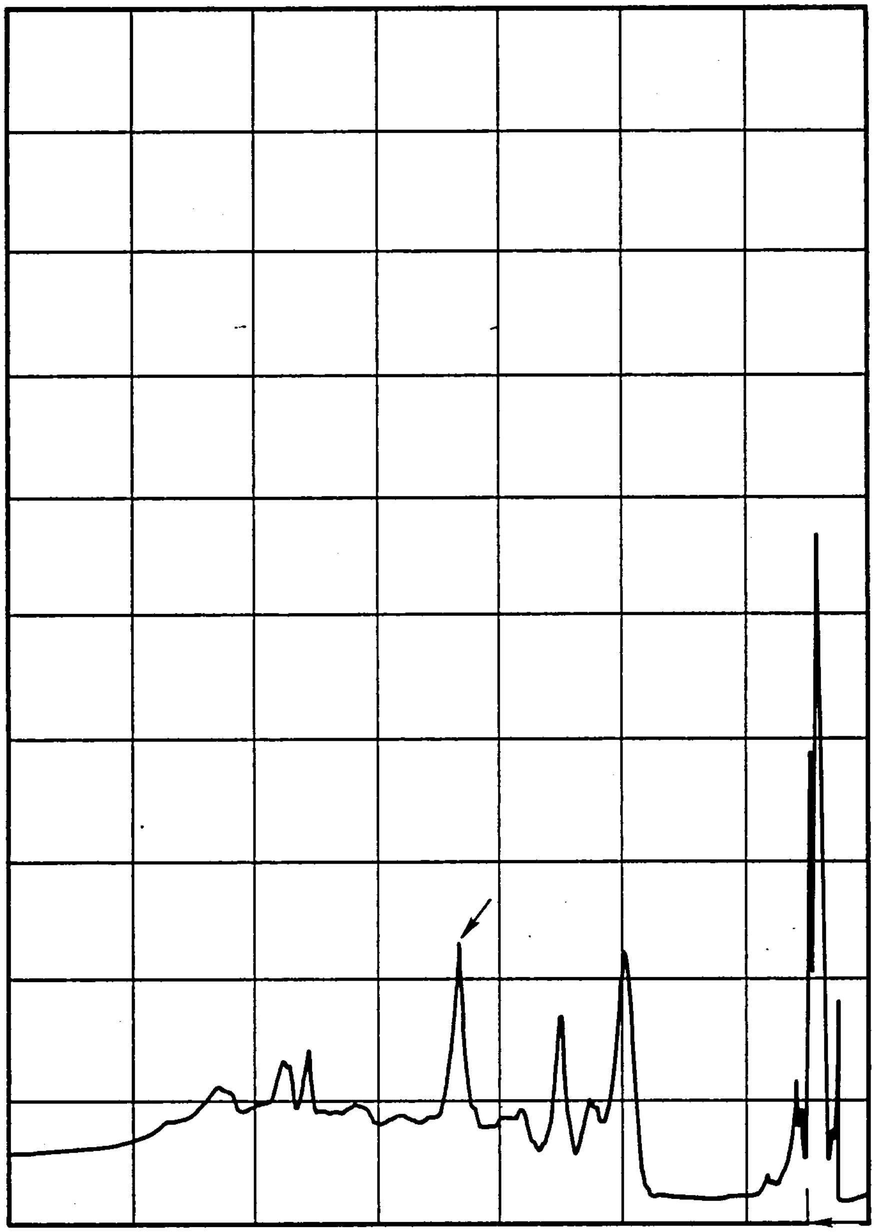


Figure 15. HPLC Scan for Pre-preg 6D-26

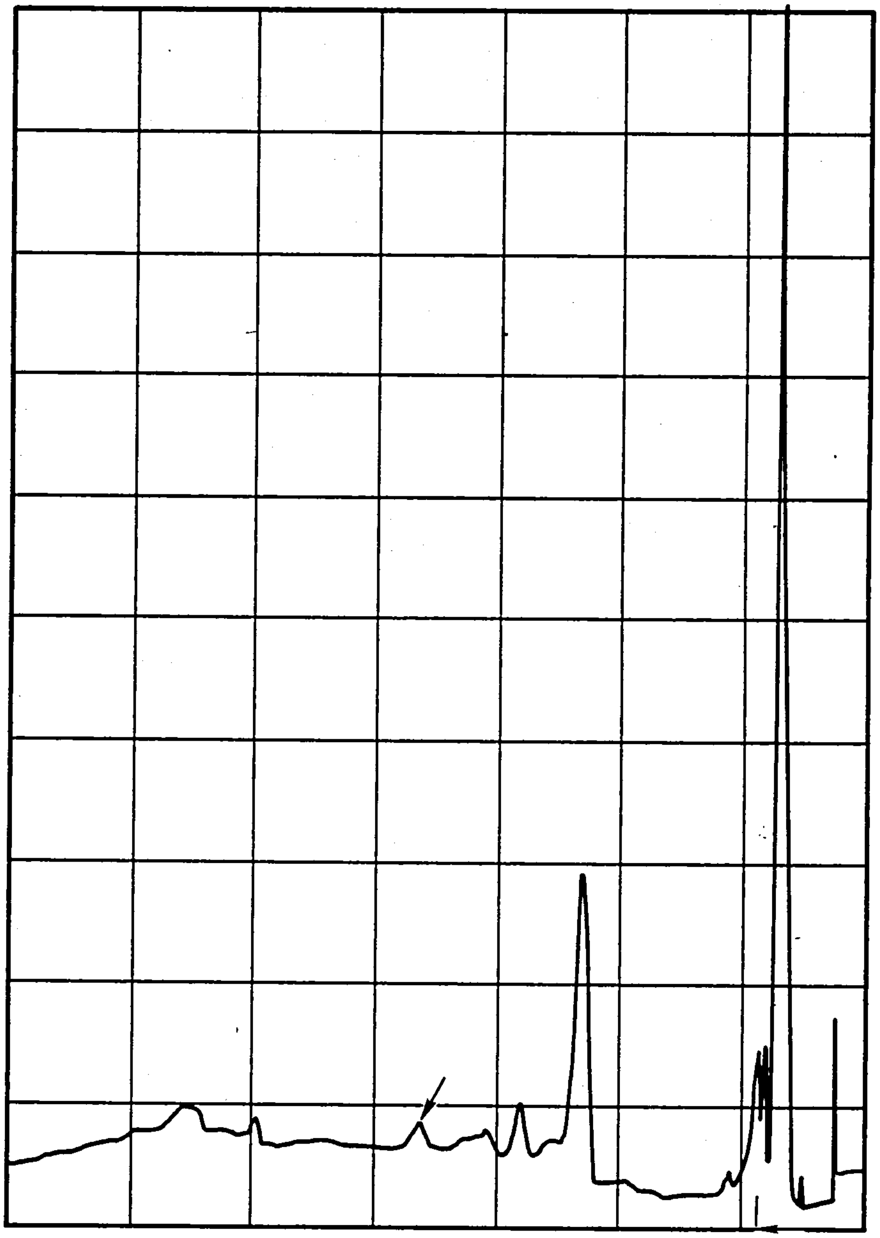
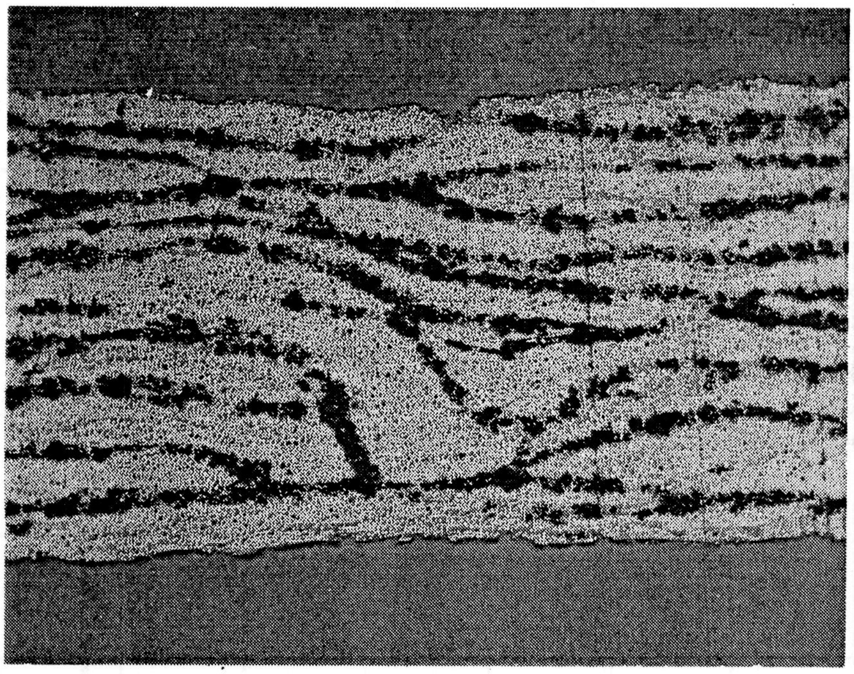
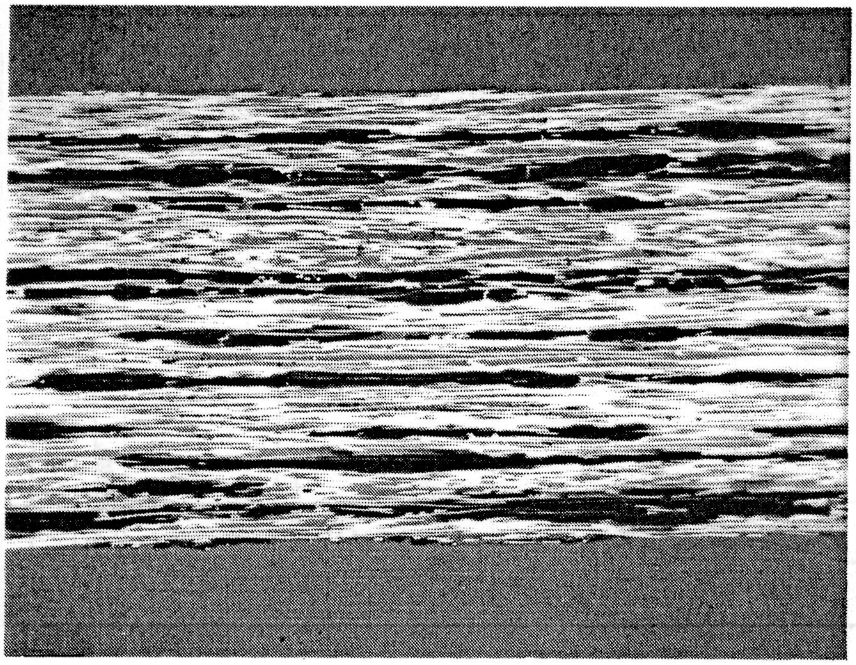


Figure 16. HPLC Scan for Pre-preg 6B-74



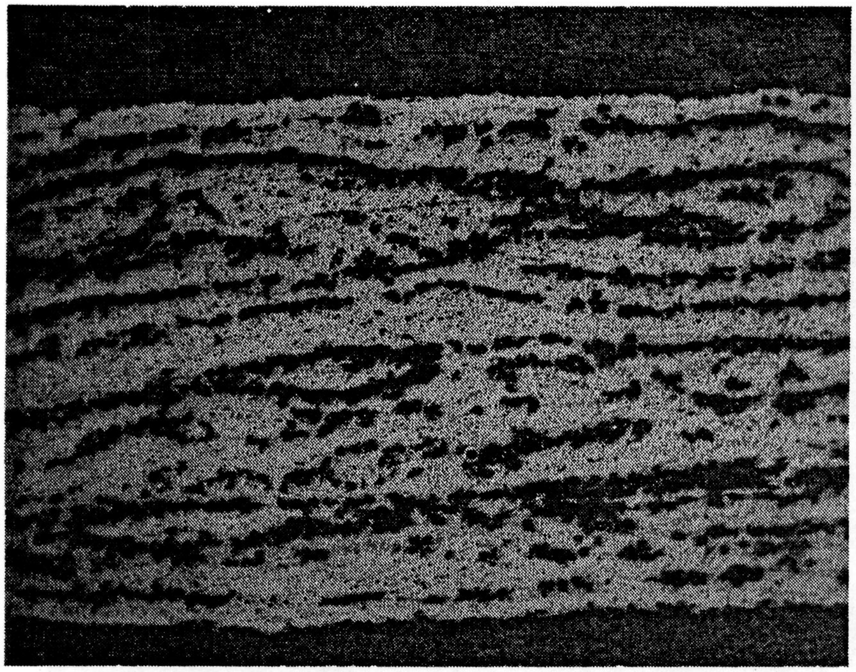
A. END VIEW (50X)



B. SIDE VIEW (50X)

NOTES: 1. STANDARD CURE
2. SP GR, 1.40
3. FIBER VOL %, 70.5
4. VOID %, 14.6

Figure 17. Photomicrograph of Laminate C-4



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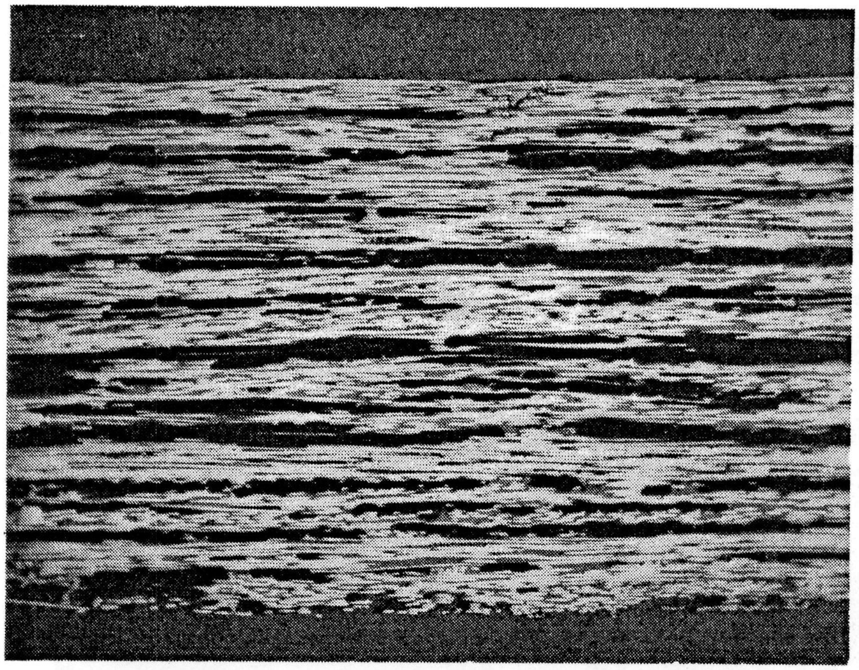
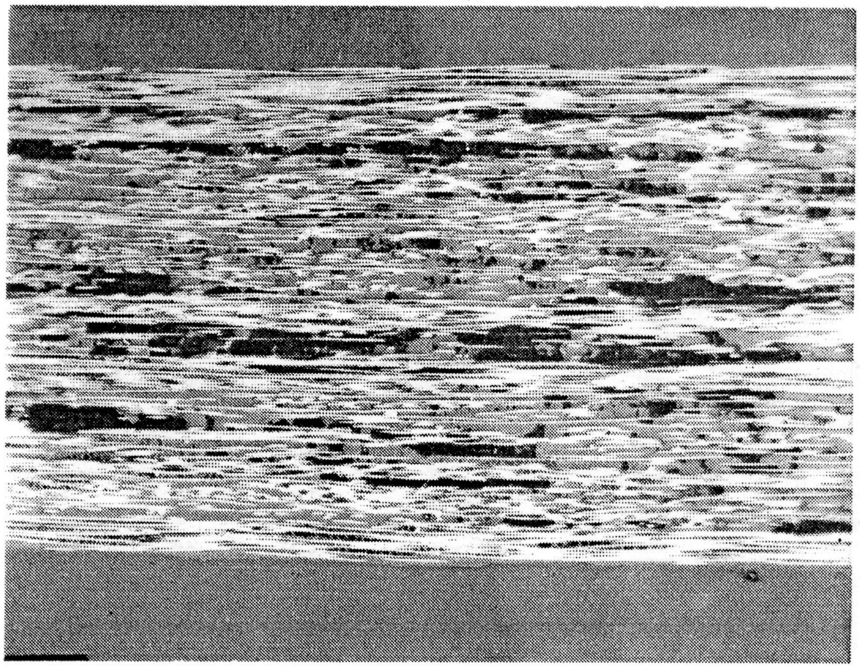


B. SIDE VIEW (50X)

NOTES:
1. STANDARD CURE + HOLD OF 1 HR AT 150F & 1 HR AT 250F
2. SP GR, 1.42
3. FIBER VOL %, 67.3
4. VOID %, 13.4

Figure 18. Photomicrograph of Laminate C-6

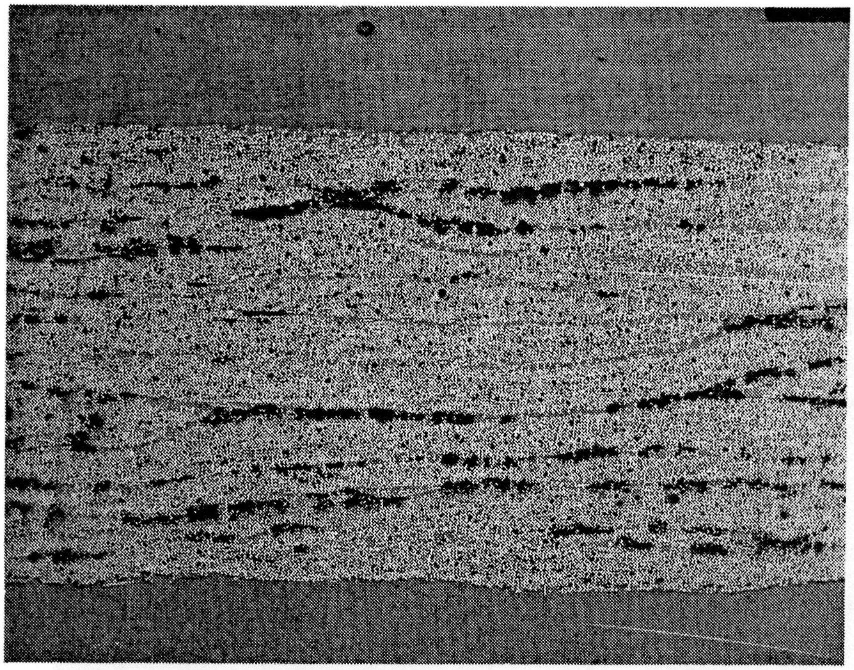
A. END VIEW (50X)



B. SIDE VIEW (50X)

NOTES:
1. STANDARD CURE + HOLD OF 3 HRS AT 250F
2. SP GR, 1.40
3. FIBER VOL %, 63.1
4. VOID %, 13.5

Figure 19.Photomicrograph of Laminate C-7



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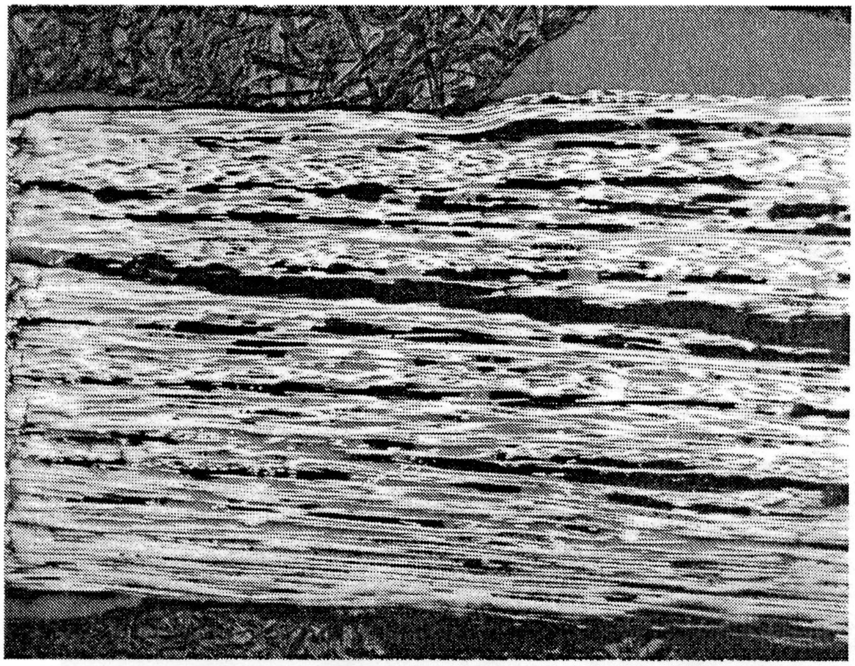
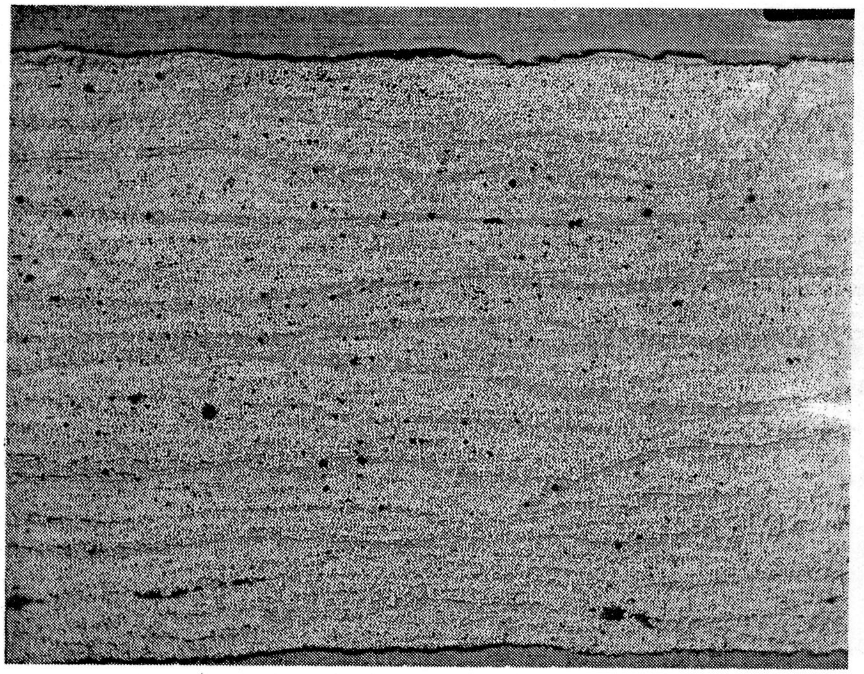


B. SIDE VIEW (50X)

NOTES:
1. STANDARD CURE + HOLD OF 5 HRS AT 250F
2. SP GR, 1.38
3. FIBER VOL %, 63.4
4. VOID %, 14.9

Figure 20. Photomicrograph of Laminate C-8



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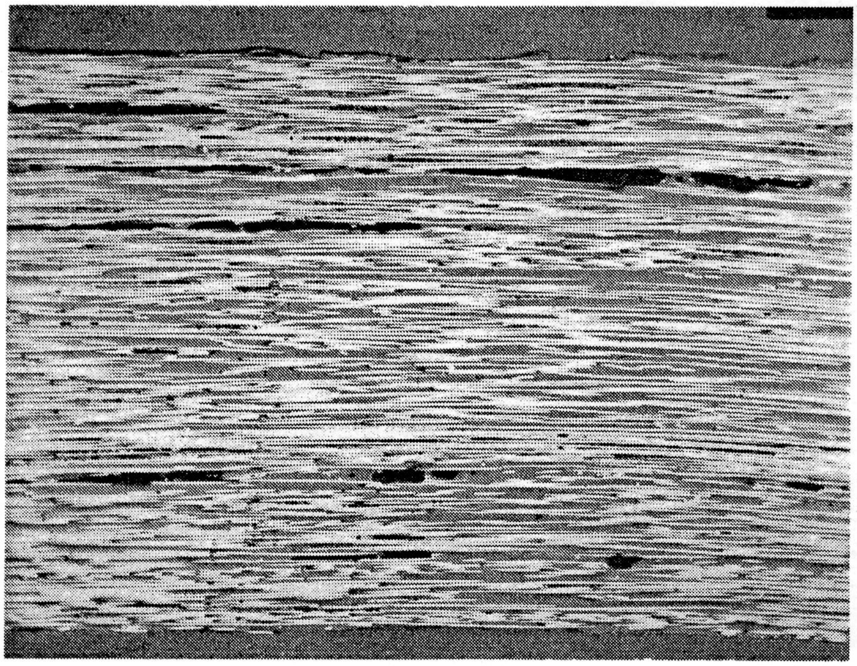
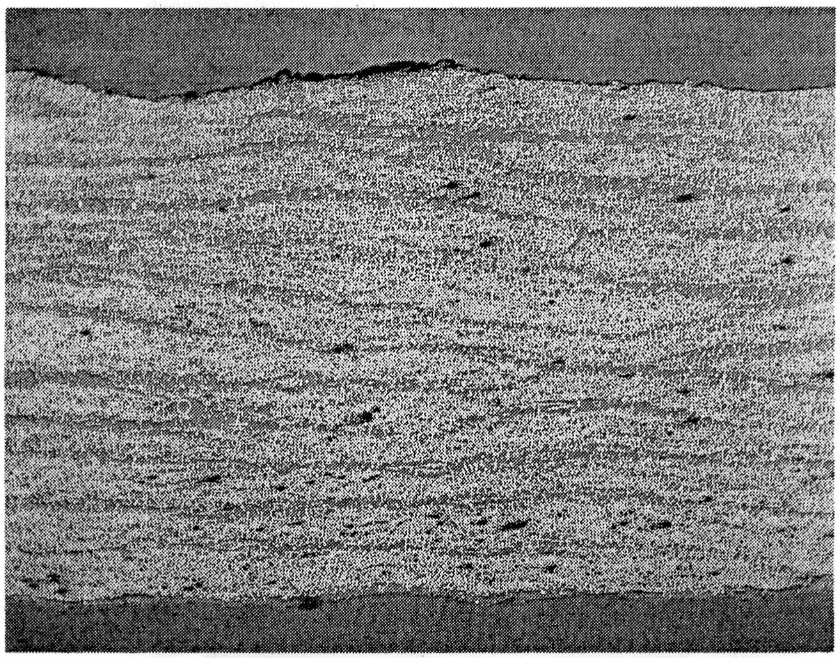


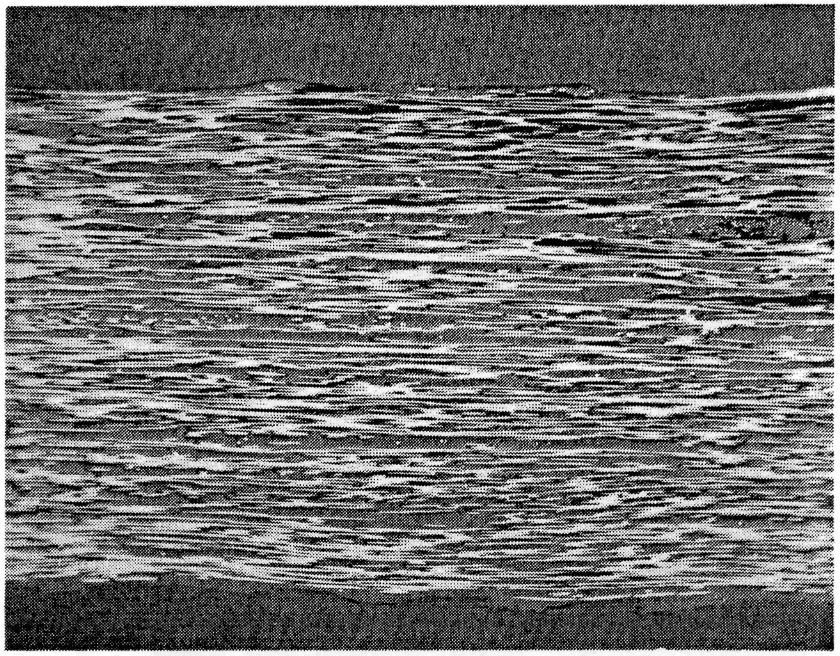
B. SIDE VIEW (50X)

NOTES: 1. STANDARD CURE + 2 HRS AT 300F (CR-4)
2. SP GR, 1.56
3. FIBER VOL %, 55.4
4. VOID %, 1.8

Figure 21. Photomicrograph of Laminate C-9



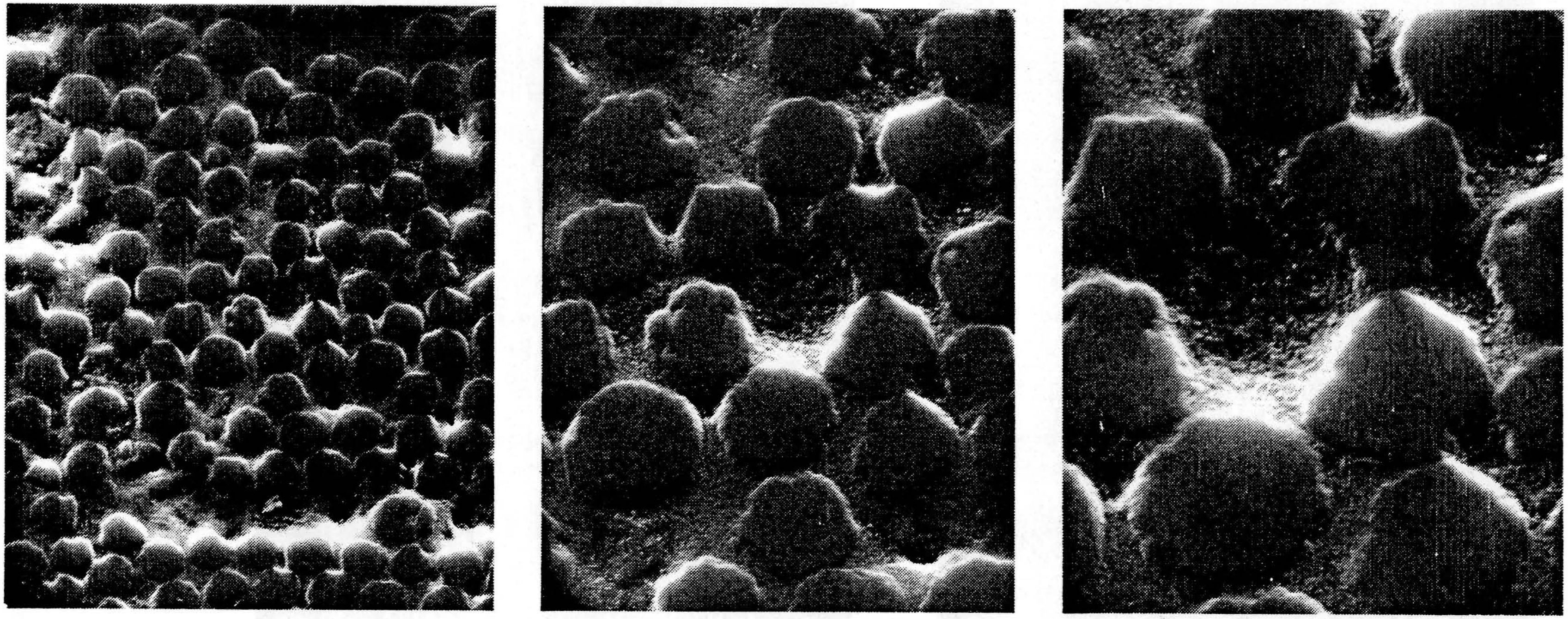
A. END VIEW



B. SIDE VIEW

NOTES: 1. CR-4 CURE (Same as Laminate C-9)
2. SP GR, 1.55
3. FIBER VOL %, 52.1
4. VOID %, 1.7

Figure 22. Photomicrograph of Laminate C-10 at 50X

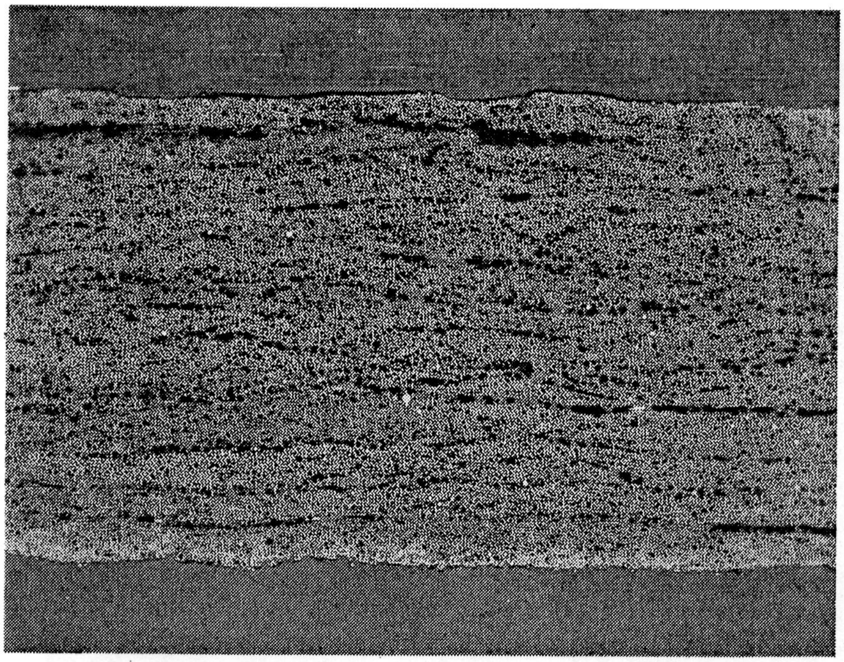


100X

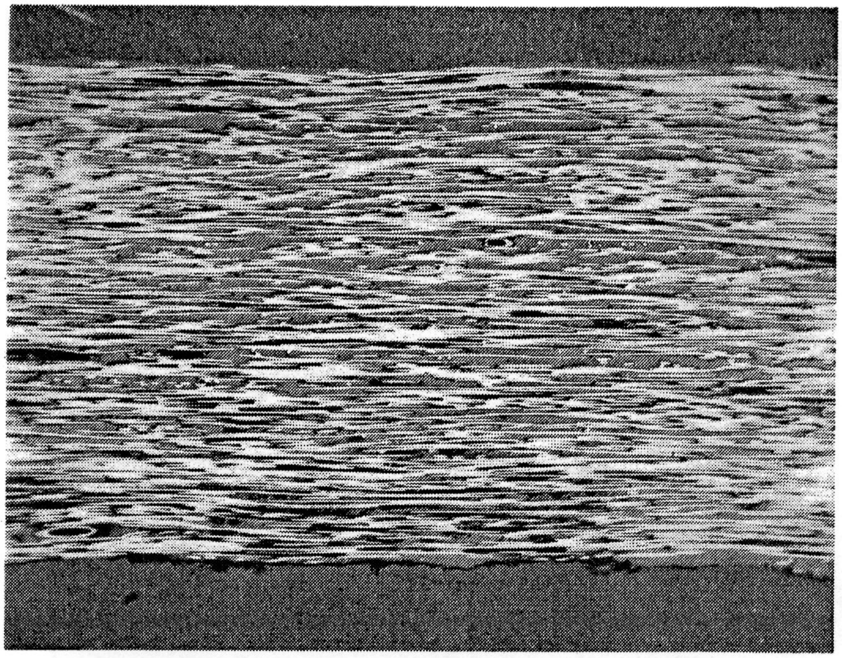
2400X

4000X

Figure 23. SEM of Laminate C-10



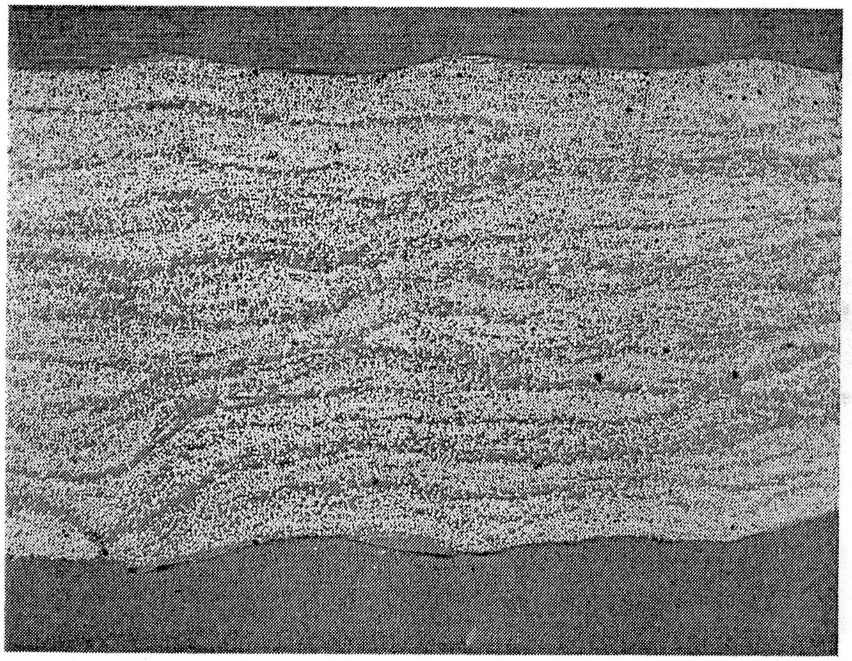
A. END VIEW



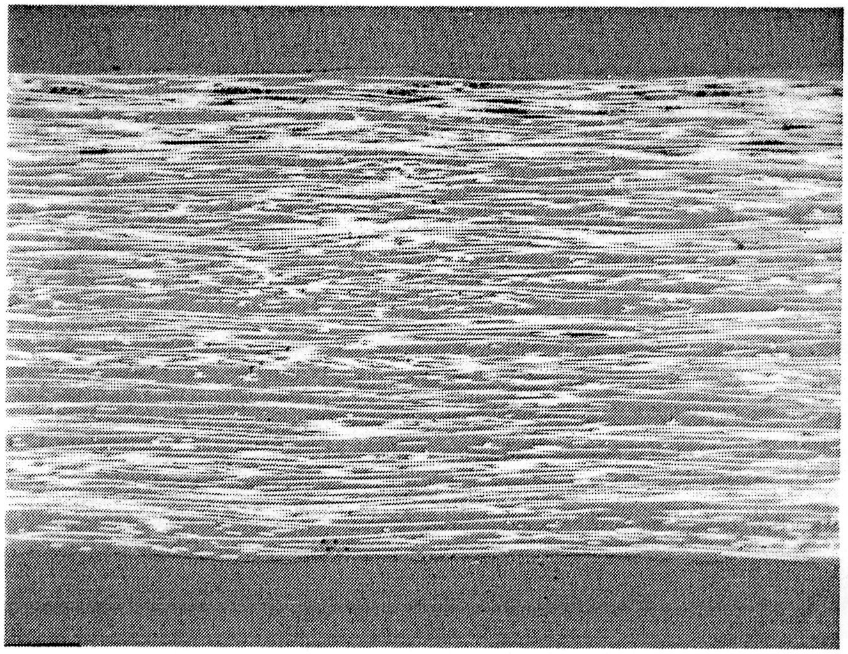
B. SIDE VIEW

NOTES: 1. CR-4A CURE
2. SP GR, 1.51
3. FIBER VOL %, 56.2
4. VOID %, 5.1

Figure 24. Photomicrograph of Laminate C-11 at 50X



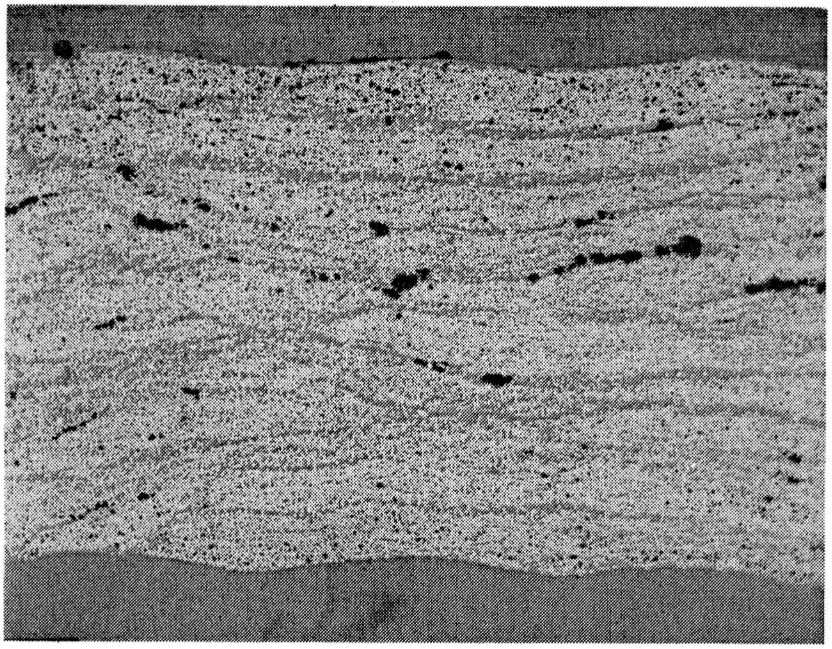
A. END VIEW



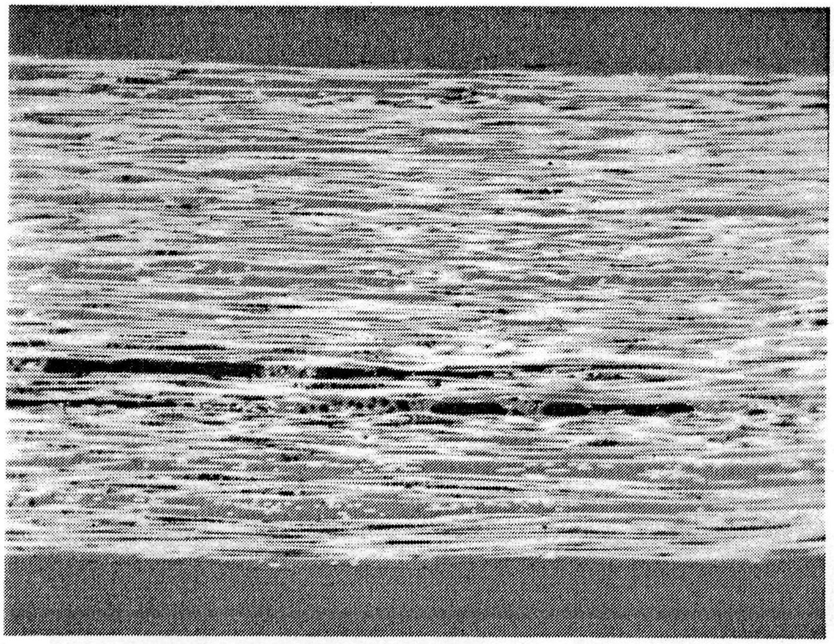
B. SIDE VIEW

NOTES:
1. CR-4B CURE
2. SP GR, 1.55
3. FIBER VOL %, 55.6
4. VOID %, 2.7

Figure 25. Photomicrograph of Laminate C-13 at 50X



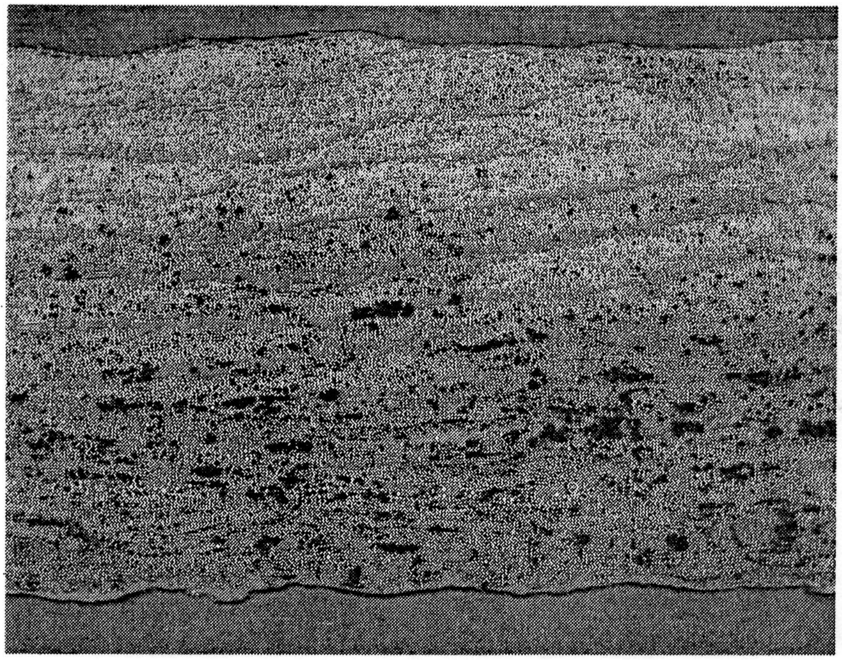
A. END VIEW



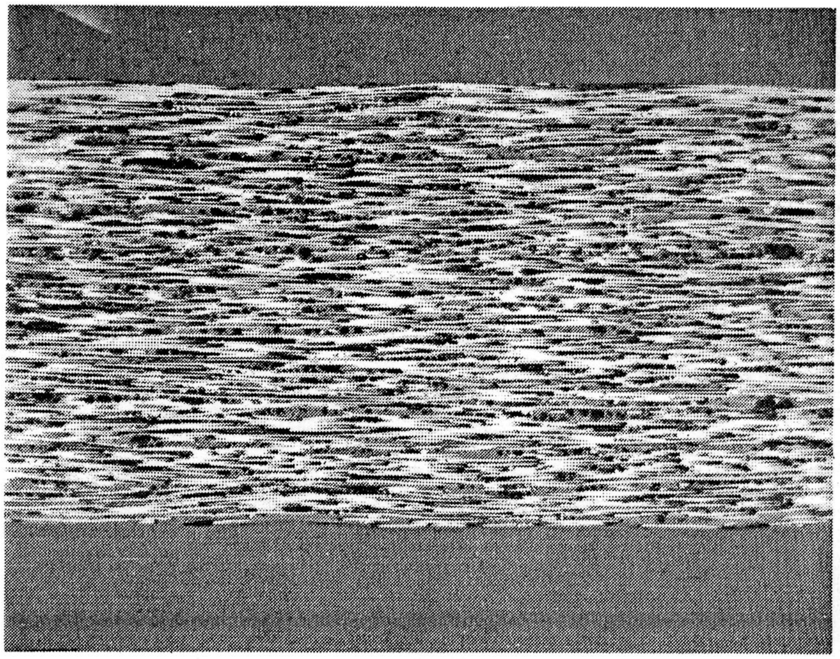
B. SIDE VIEW

NOTES:
1. CR-4C CURE
2. SP GR, 1.56
3. FIBER VOL % 56.1
4. VOID %, 2.1

Figure 26. Photomicrograph of Laminate C-15 at 50X



A. END VIEW



B. SIDE VIEW

NOTES: 1. CR-5 CURE
2. SP GR, 1.45
3. FIBER VOL, 66.0
4. VOID %, 10.7

Figure 27. Photomicrograph of Laminate C-12 at 50X

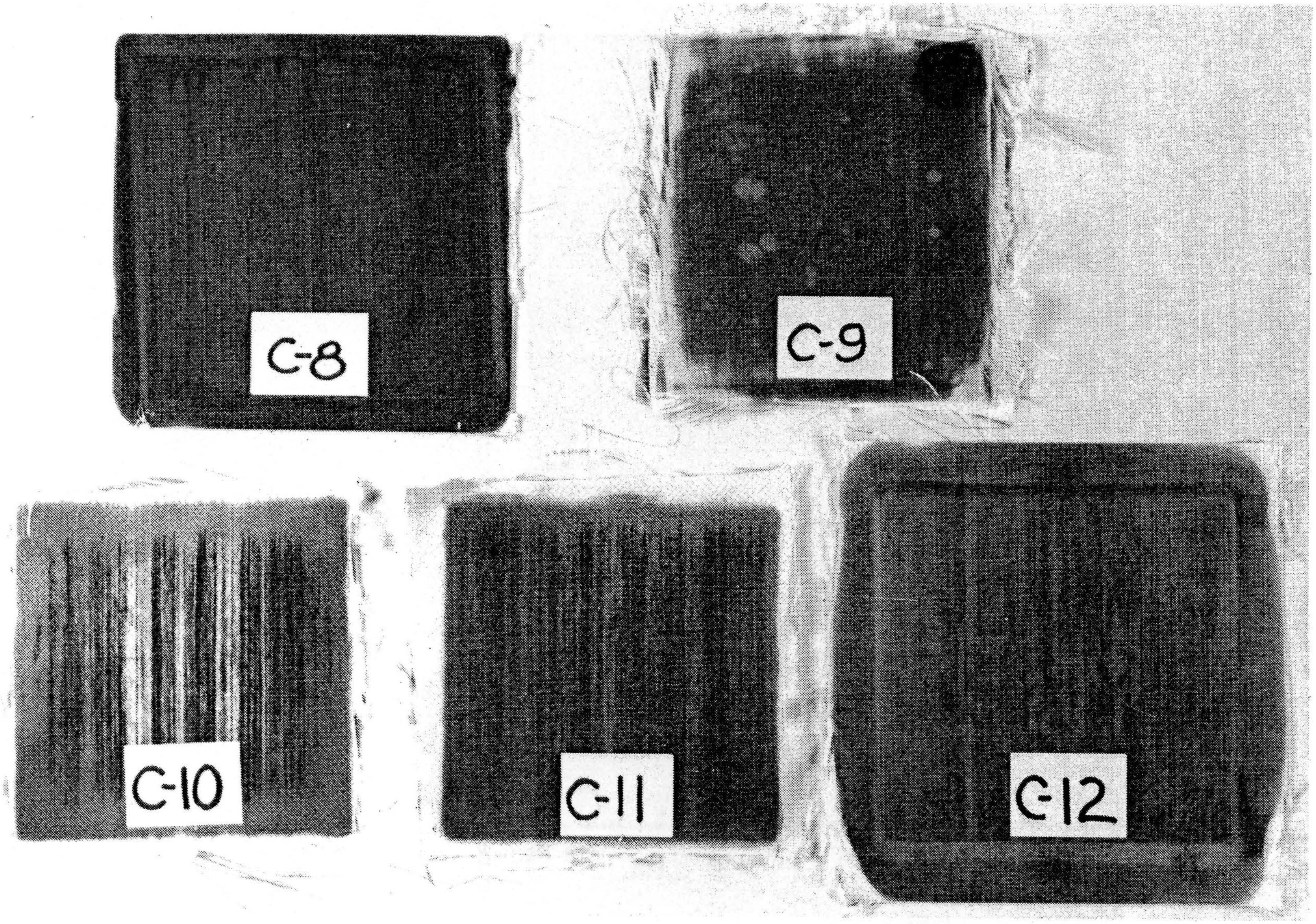


Figure 28. Glass Fabric Bleeder for Laminates C-8 through C-12
Shows Effect of Process Variations on Resin Flow

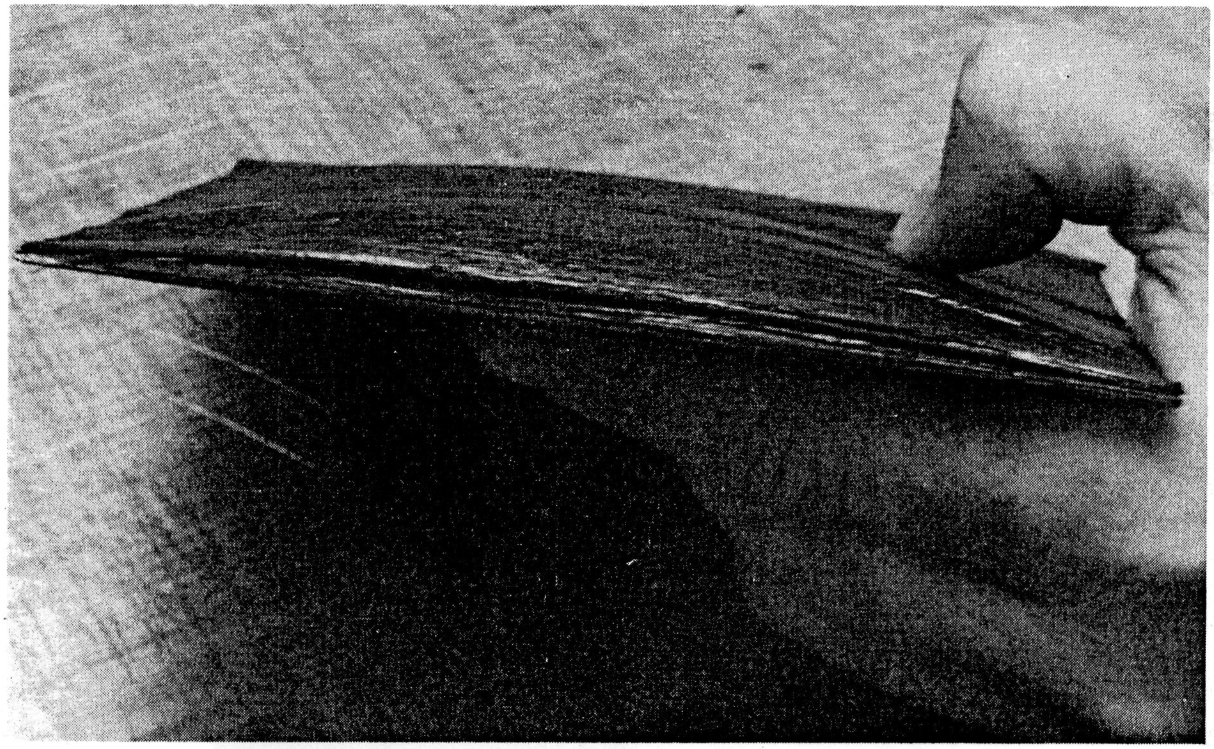
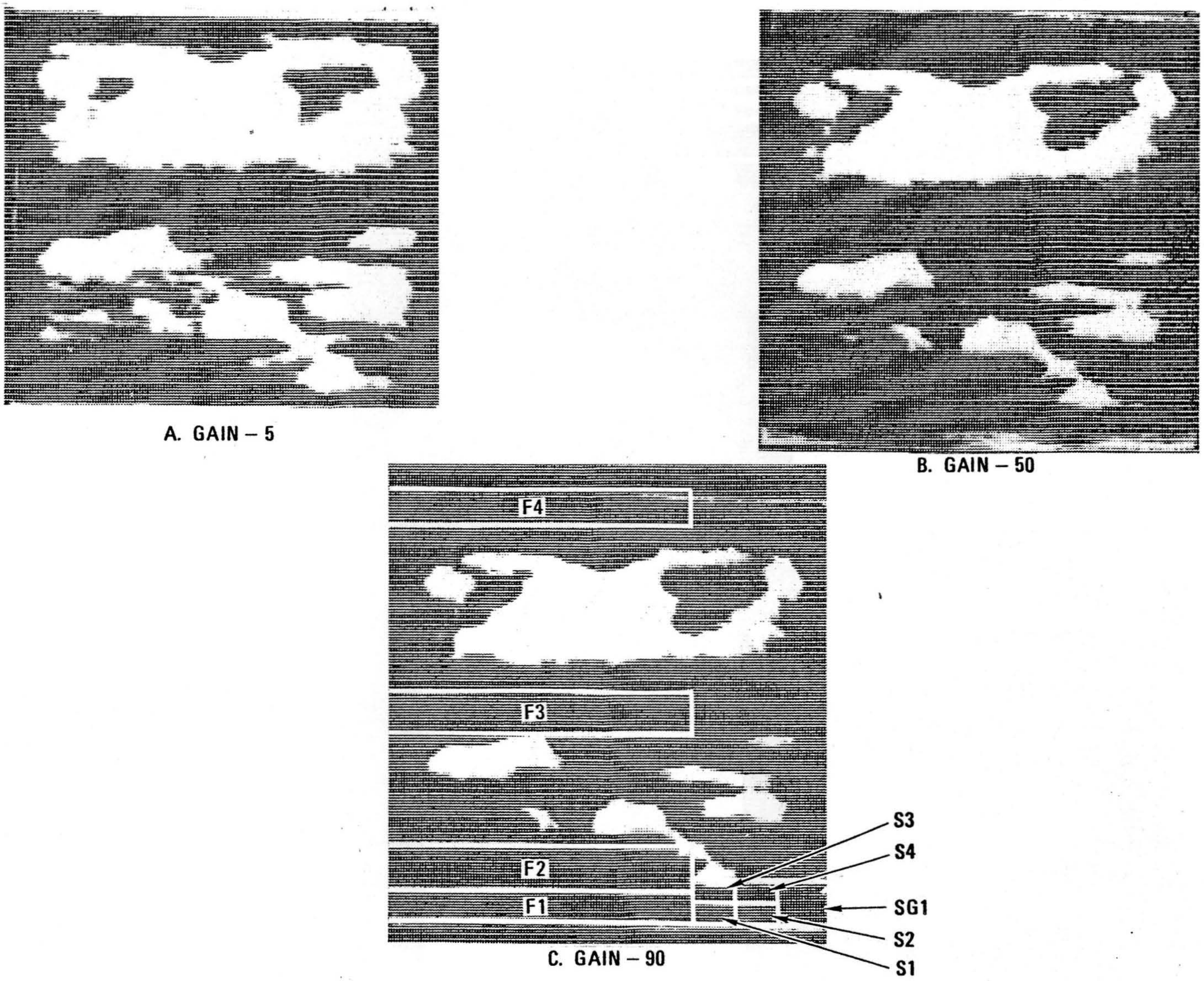


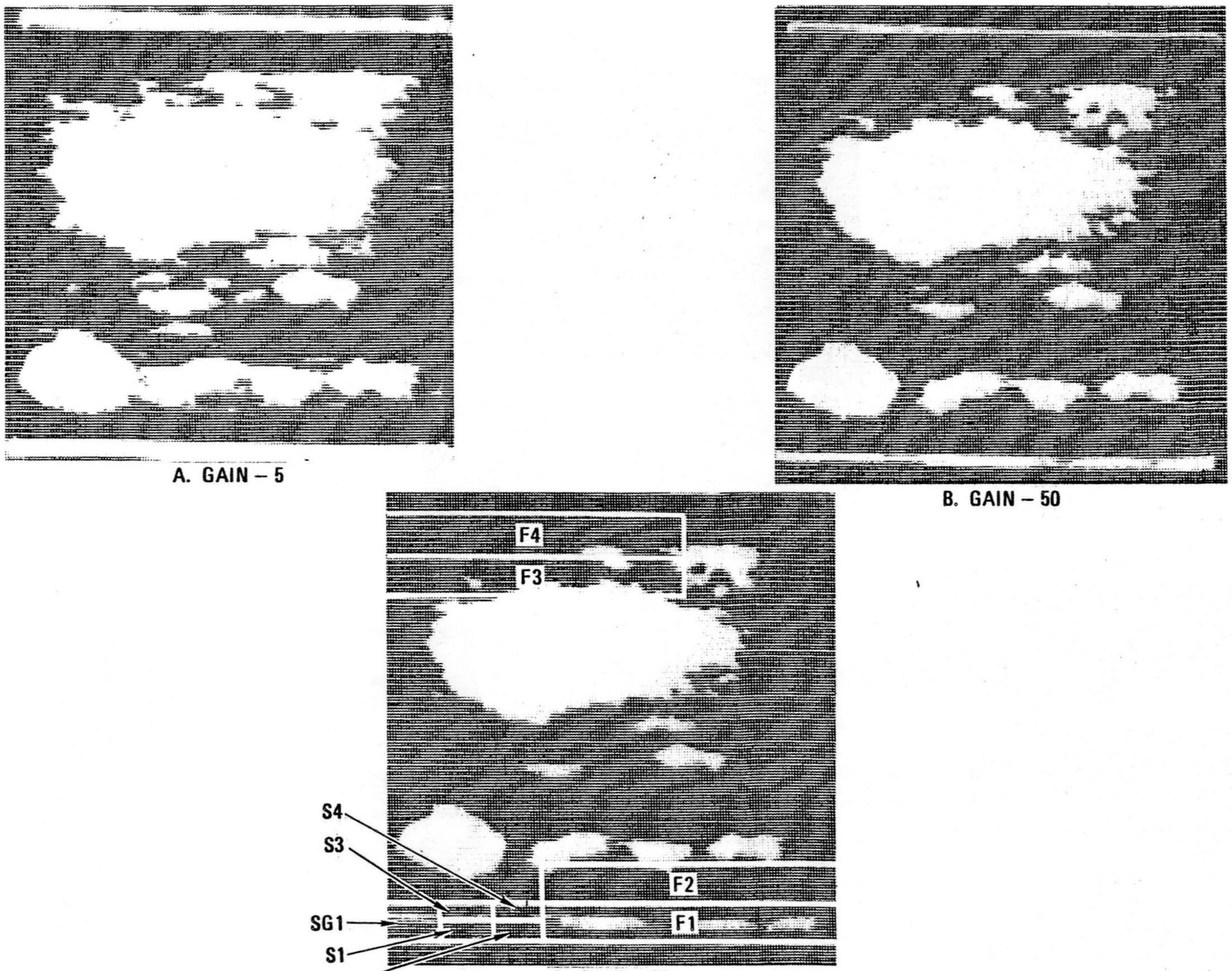
Figure 29, Laminate C-41 After Advancement in Oven at 149C (300F)


A. GAIN – 5

B. GAIN – 50

C. GAIN – 90

Figure 30, Ultrasonic C-Scan (5 MHz) and Specimen Cutting Pattern for Laminate C-32



A. GAIN – 5

B. GAIN – 50

C. GAIN – 90

Figure 31. Ultrasonic C-Scan (5 MHz) and Specimen Cutting Pattern for Laminate C-33

Figure 32. Typical Visual Blistering of NR-150B2 Lamination.

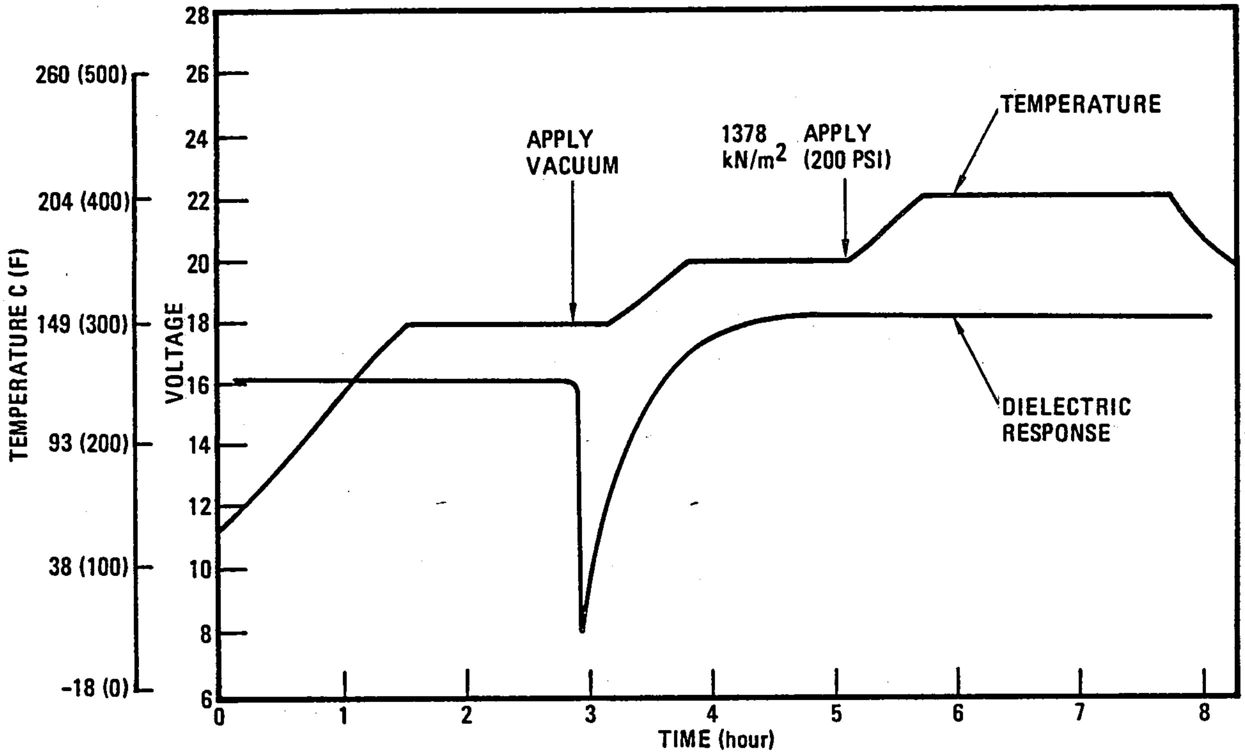


Figure 33.Voltage Temperature Profile for Laminate C-27 During Initial Cure

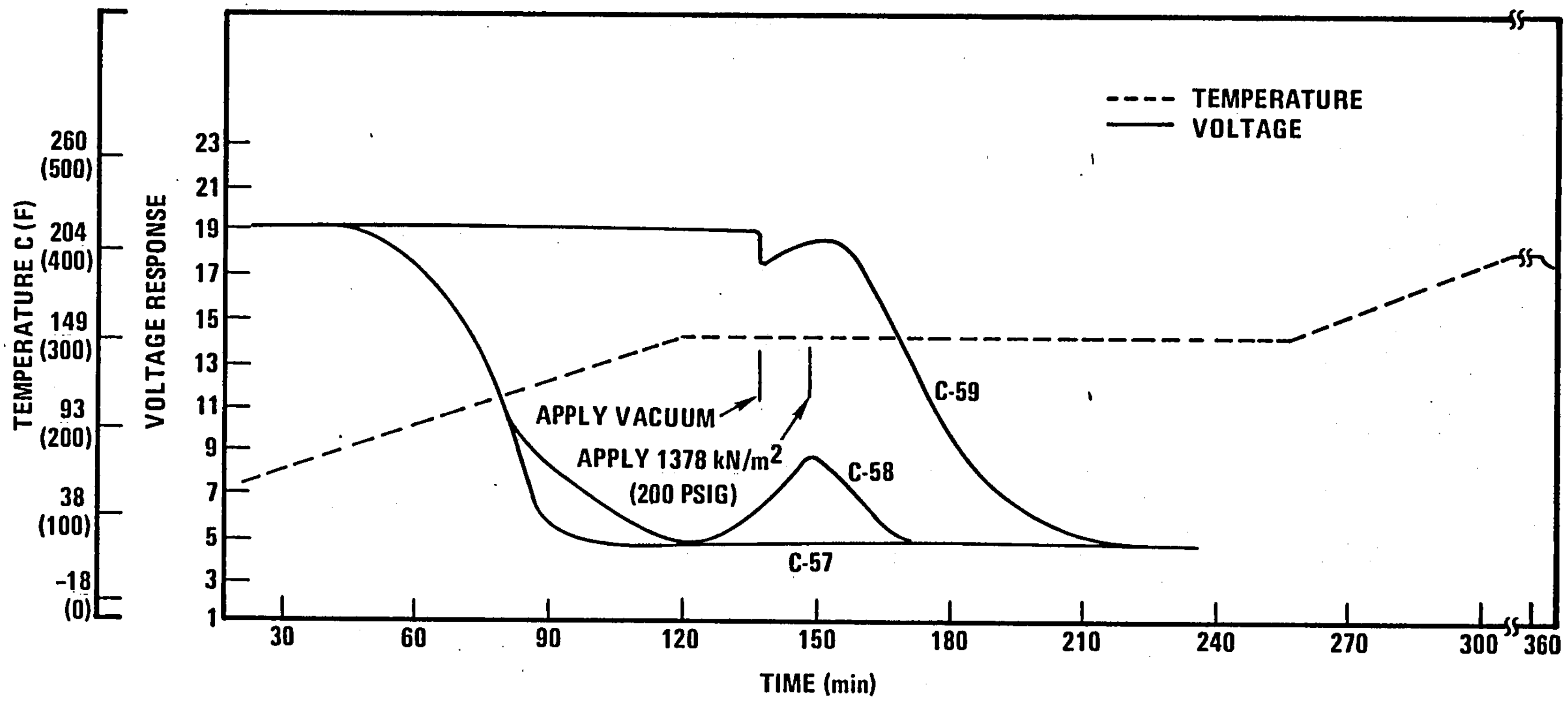


Figure 34. Dielectric Response vs Temperature for Cure of Laminates C-57, C-58 and C-59

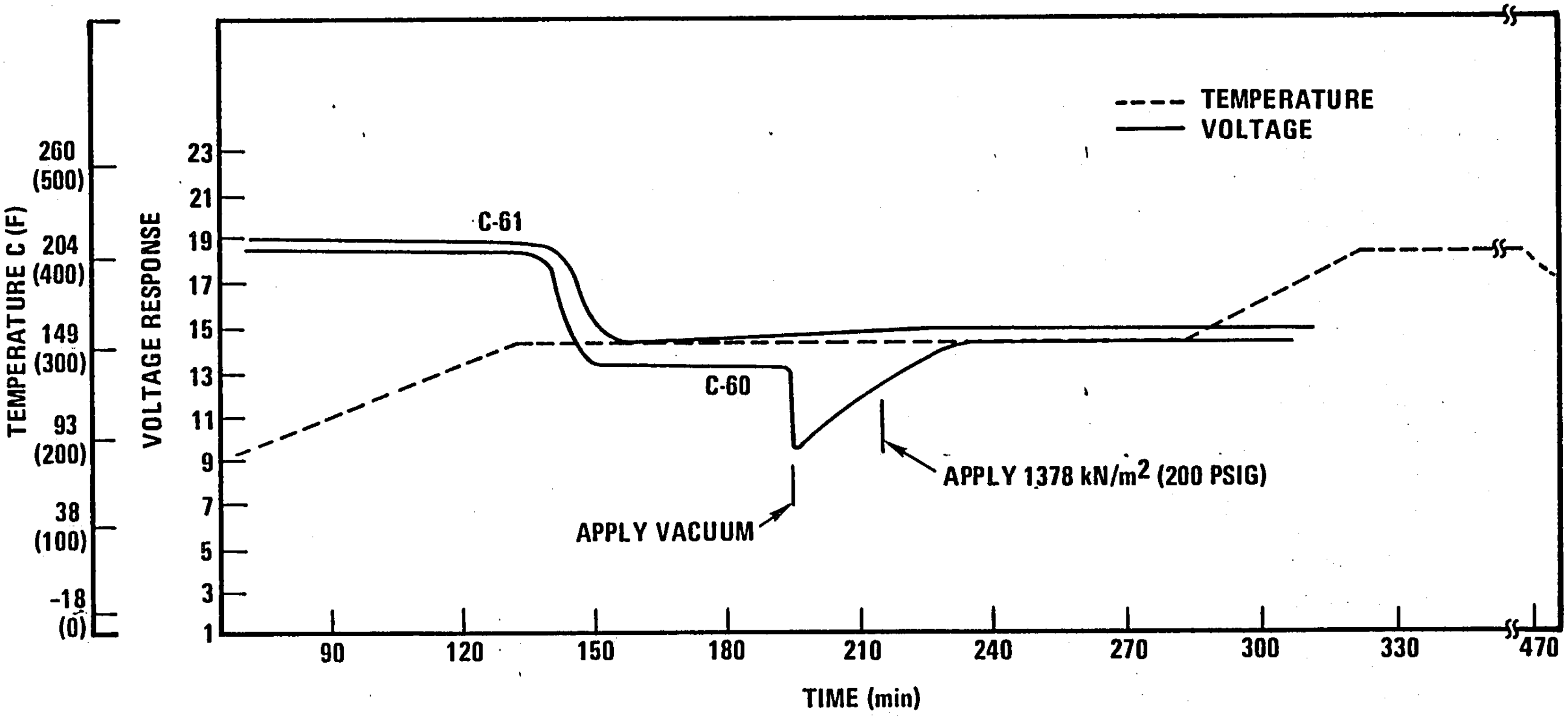
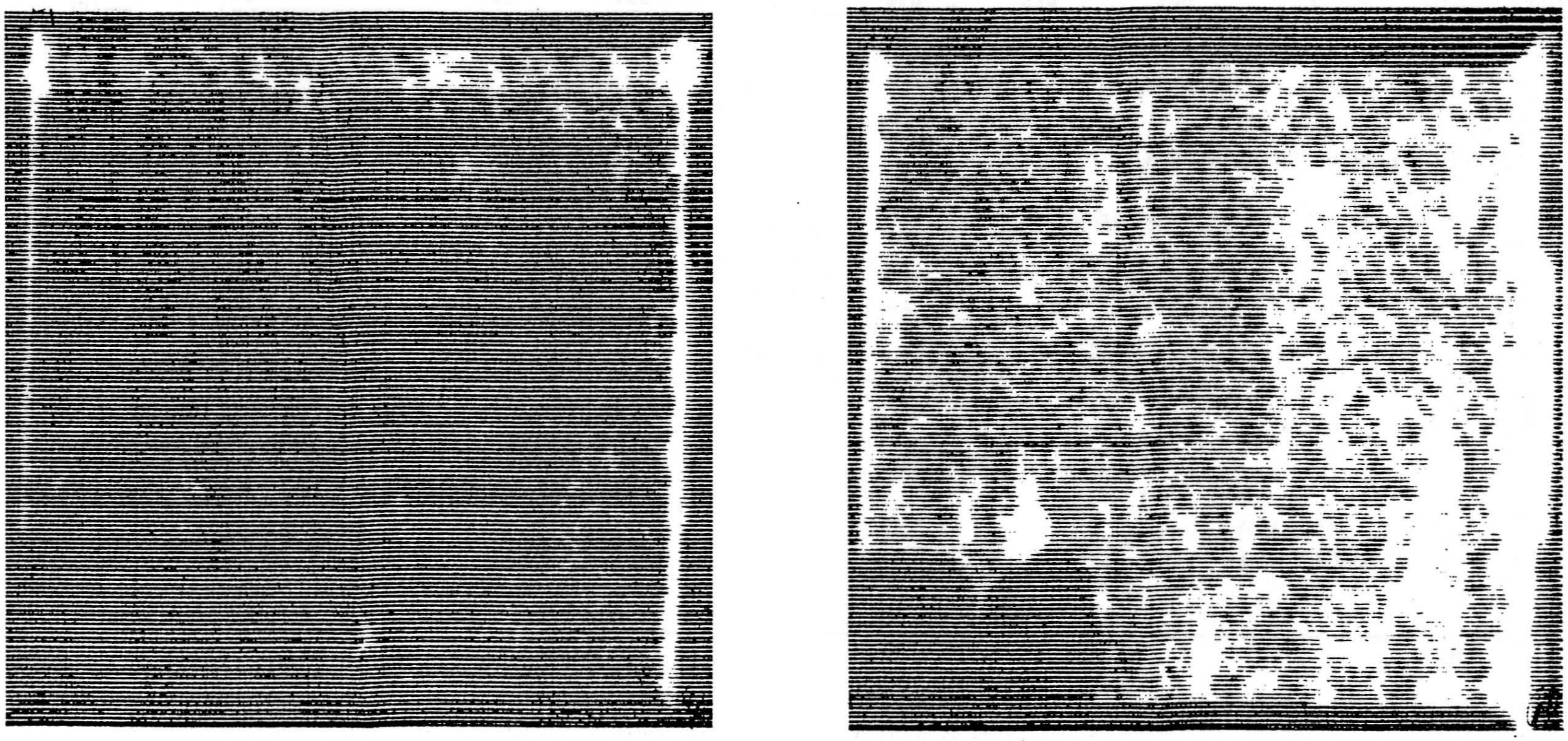


Figure 35. Dielectric Response vs Temperature for Cure of Laminates C-60 and C-61

LAMINATE C-61



2.25 MHZ GAIN = 10

2.25 MHZ GAIN = 5

Figure 36. Ultrasonic C-Scan for 12 Ply Unidirectional Graphite/NR-150 Laminate After Cure

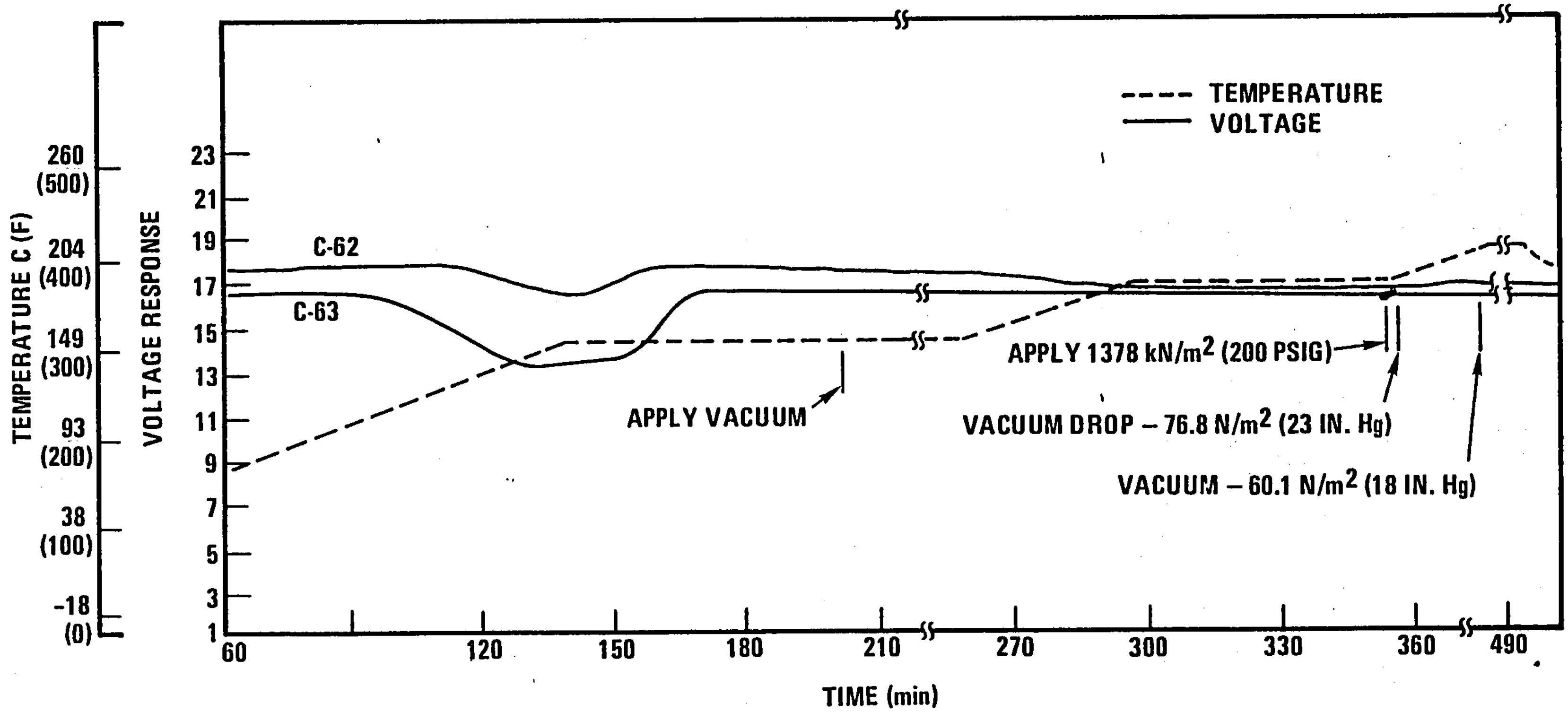


Figure 37. Dielectric Response vs Temperature for Cure of Laminates C-62 and C-63

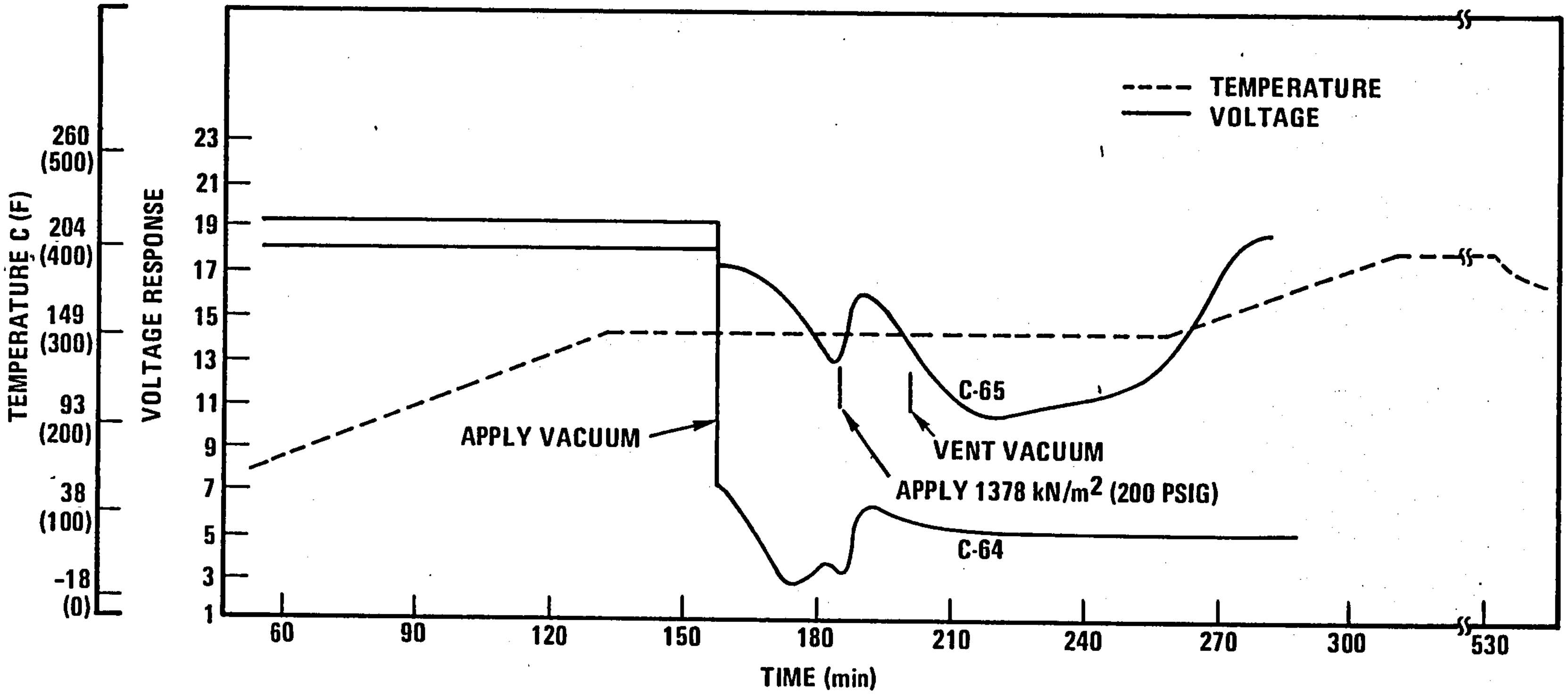
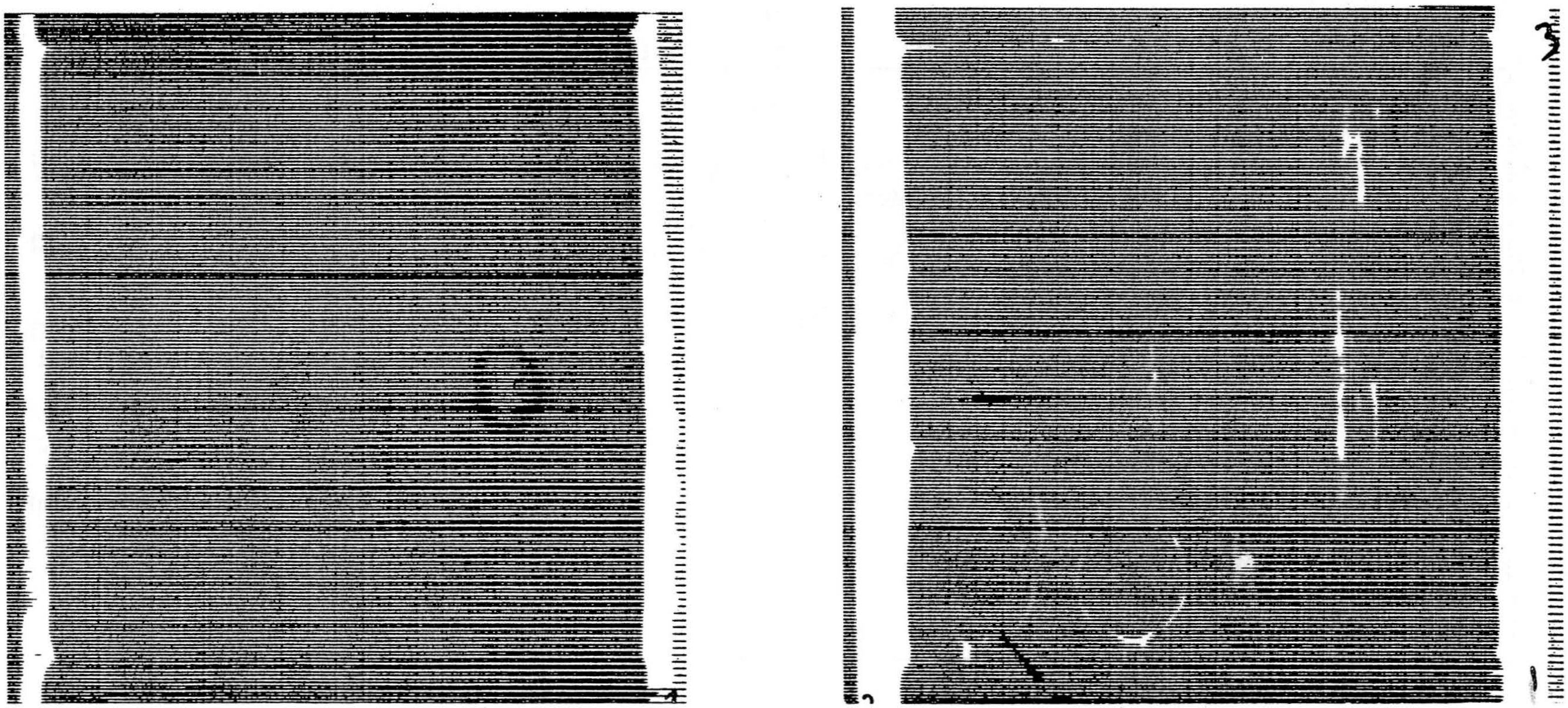


Figure 38. Dielectric Response vs Temperature for Cure of Laminates C-64 and C-65.



LAMINATE C-64

2.25 MHZ GAIN = 1

5 MHZ GAIN = 2

Figure 39. Ultrasonic C-Scan for 12 Ply Unidirectional Graphite/NR-150 Laminate After Cure

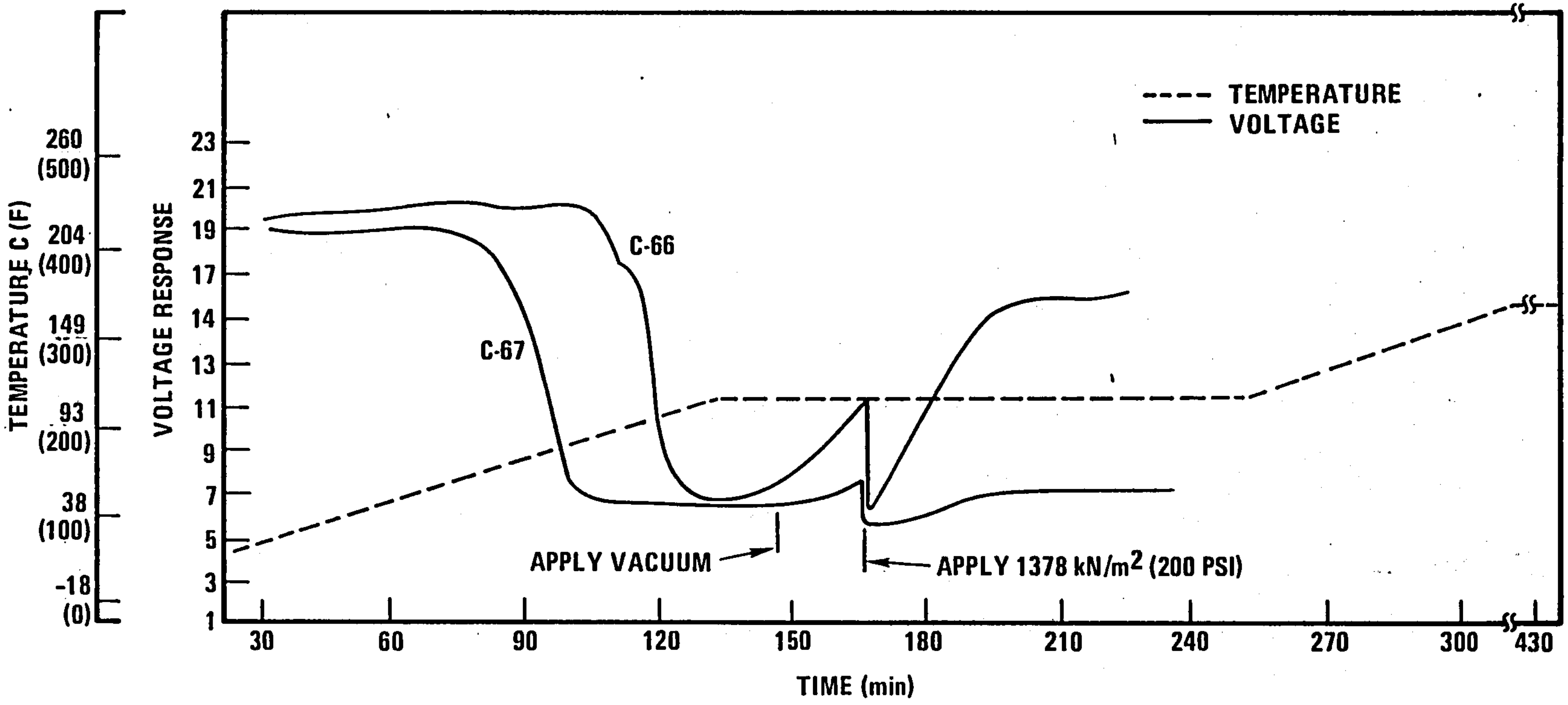


Figure 40. Dielectric Response vs Temperature for Cure of Laminates C-66 and C-67

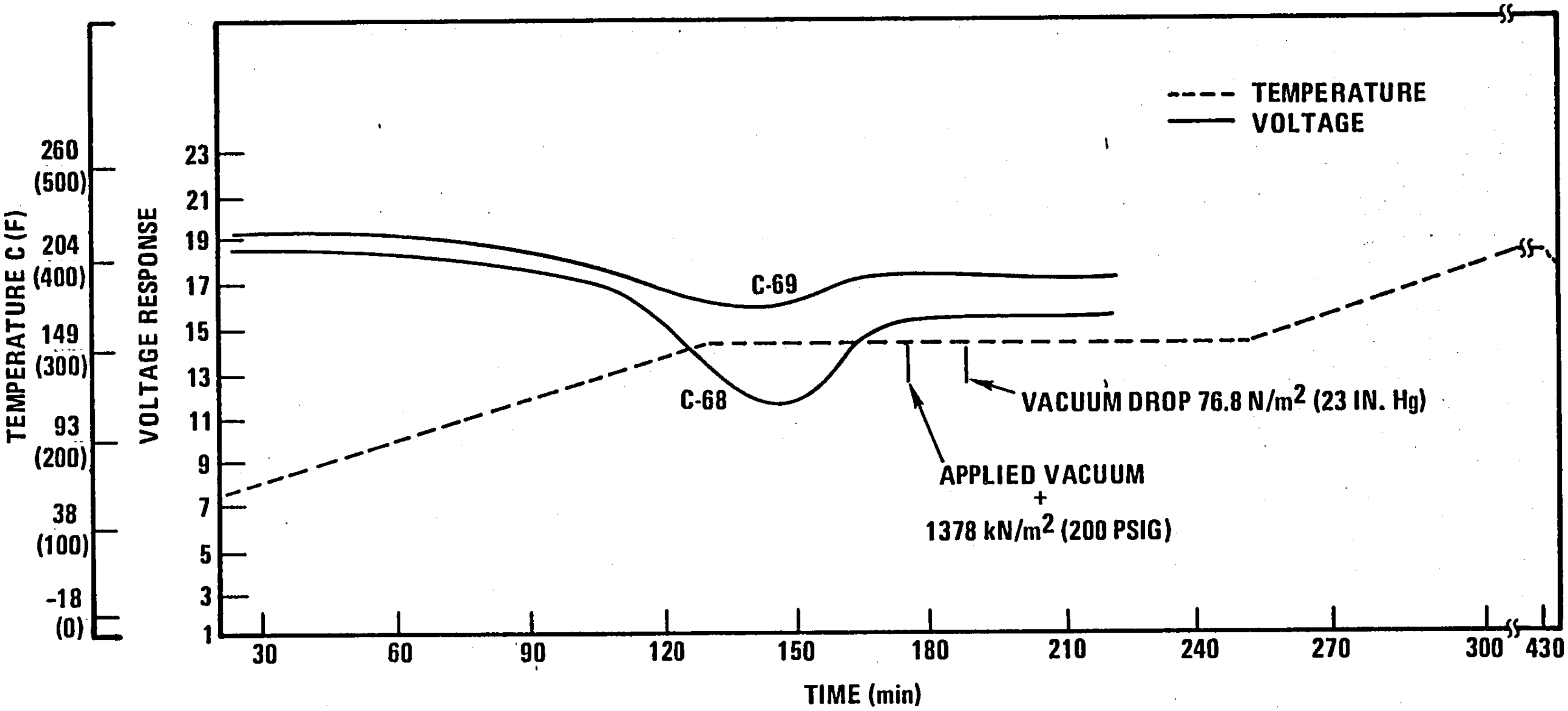


Figure 41. Dielectric Response vs Temperature for Cure of Laminates C-68 and C-69

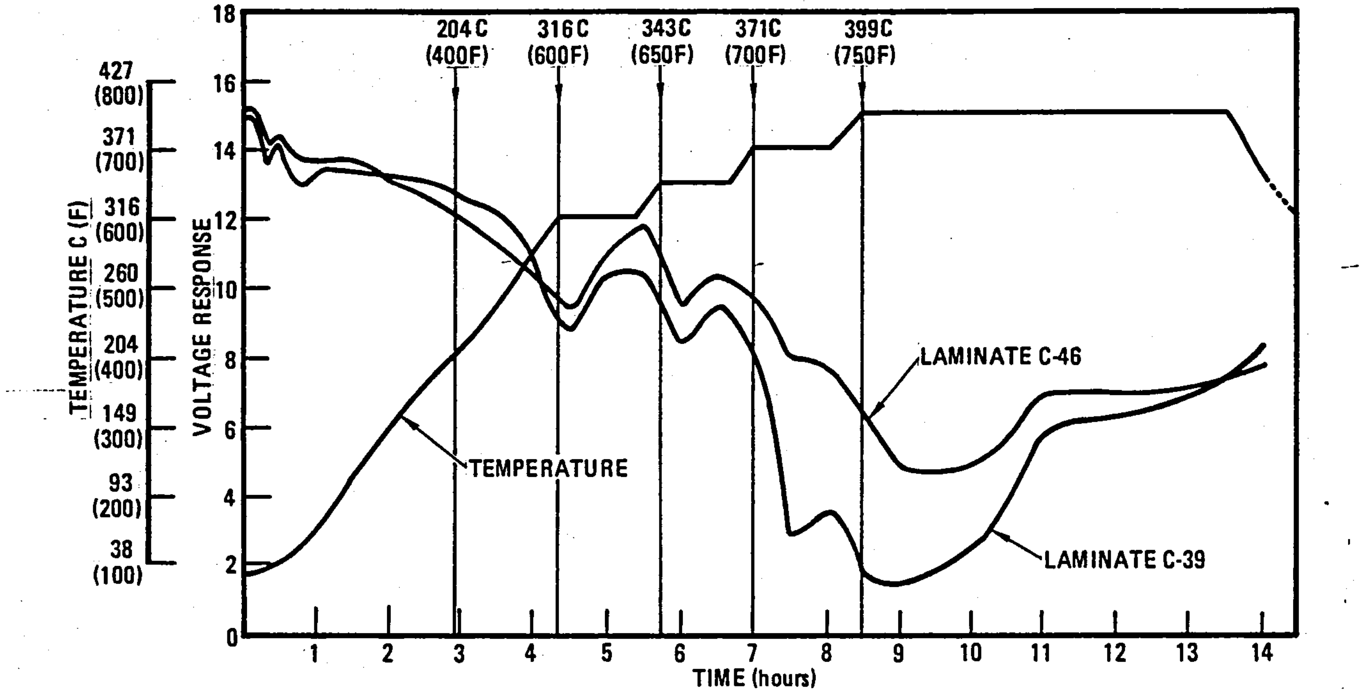


Figure 42. Dielectric Response vs Temperature for Graphite/NR-150B2 Laminates During Postcure

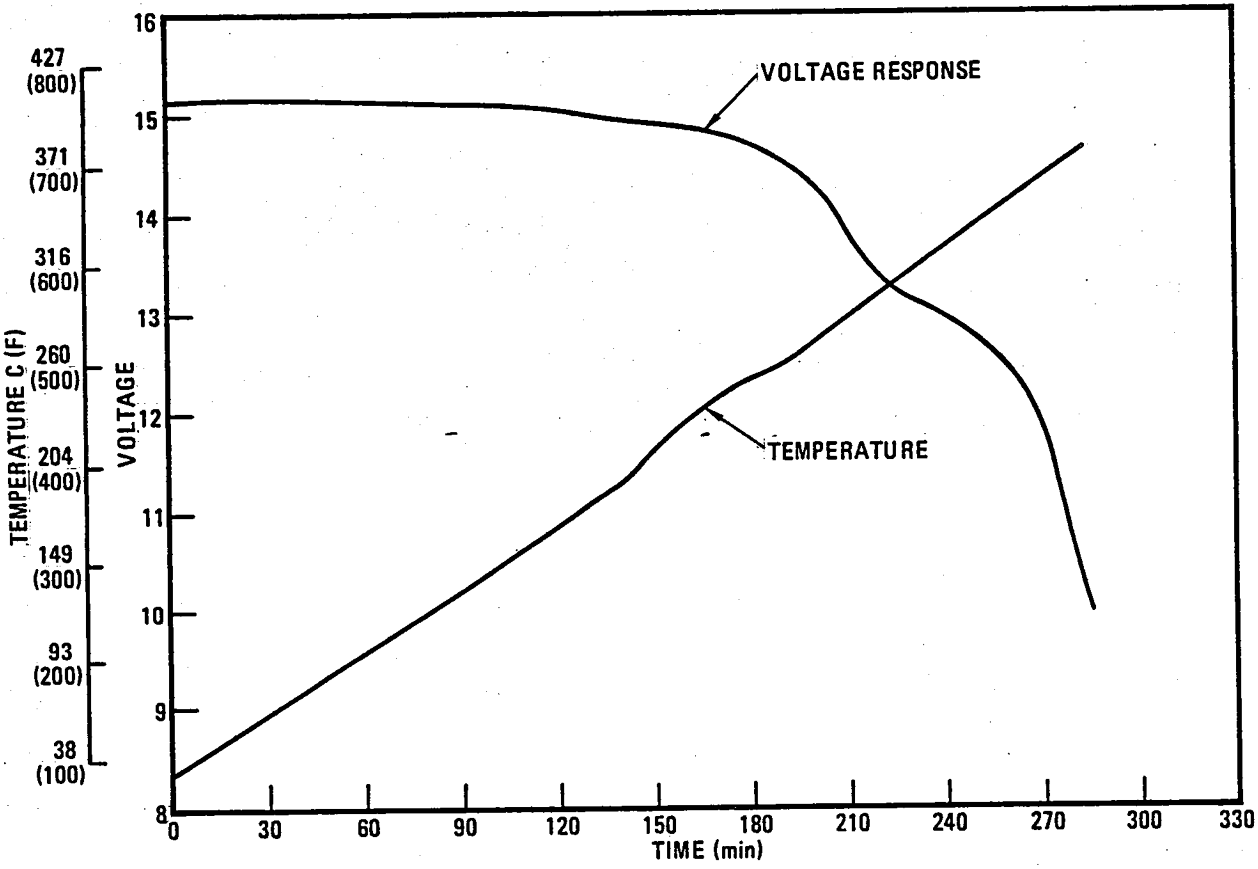


Figure 43. Dielective Response vs Temperature for Laminates C-47 and C-41 at 1.5C/Min (2.2F/Min.) Postcure Heating Rate.

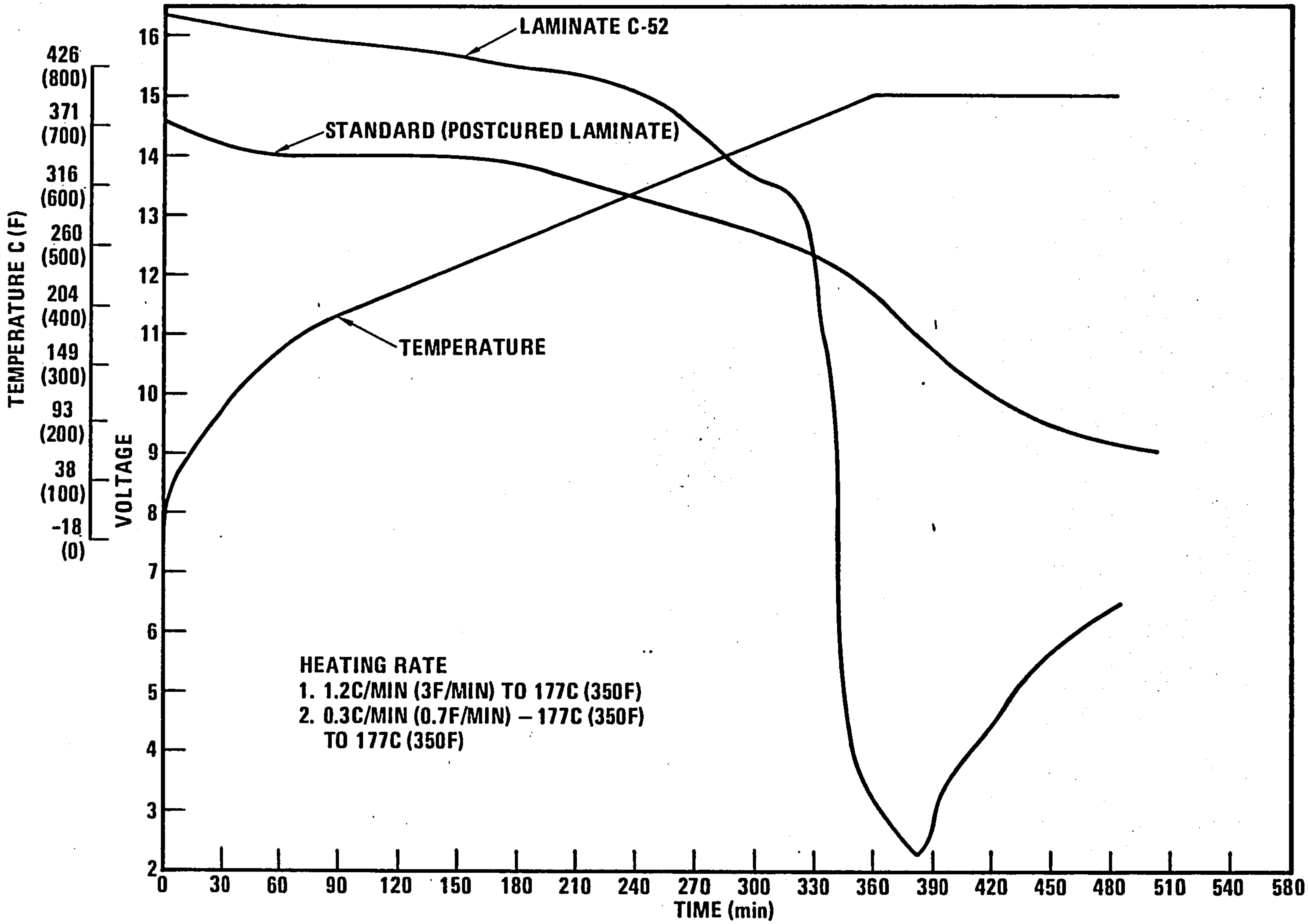


Figure 44. Dielectric Response of NR-150 B2 Laminate During Postcure

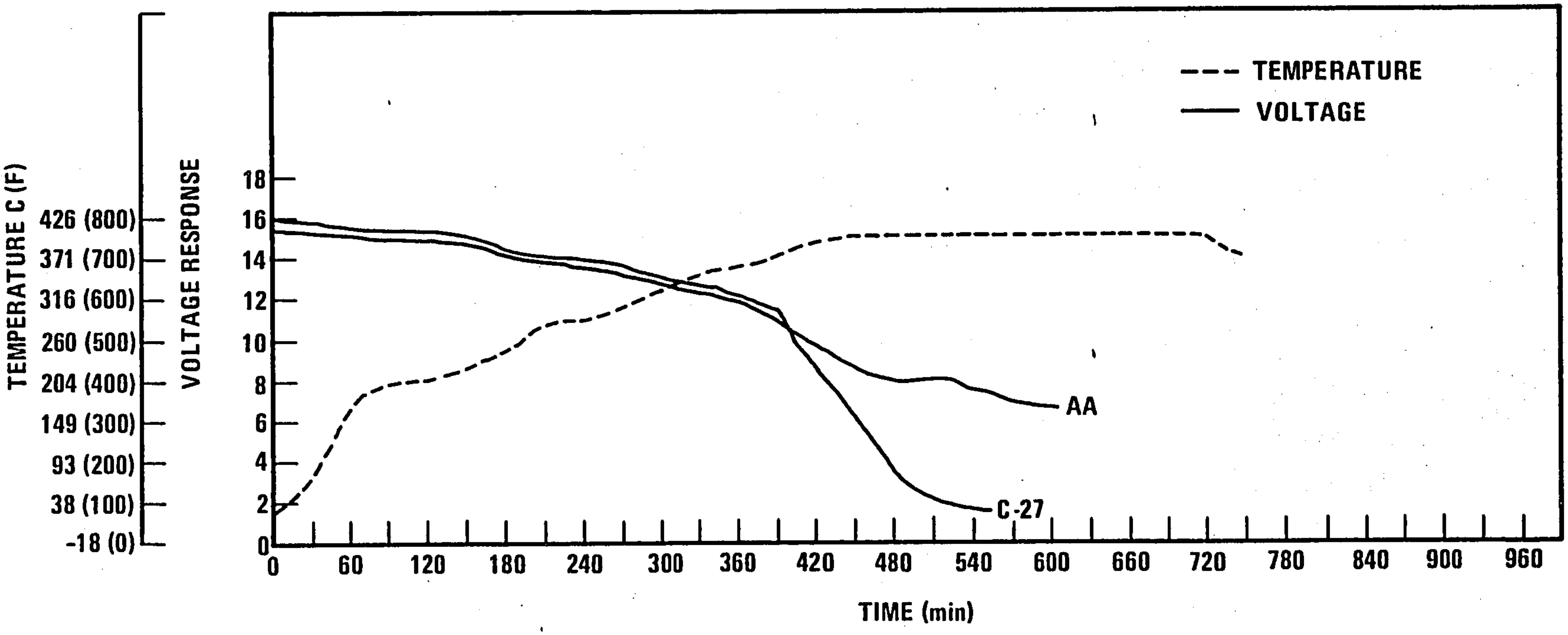


Figure 45. Dielectric Response vs Temperature for Laminates AA and C-27 Postcure

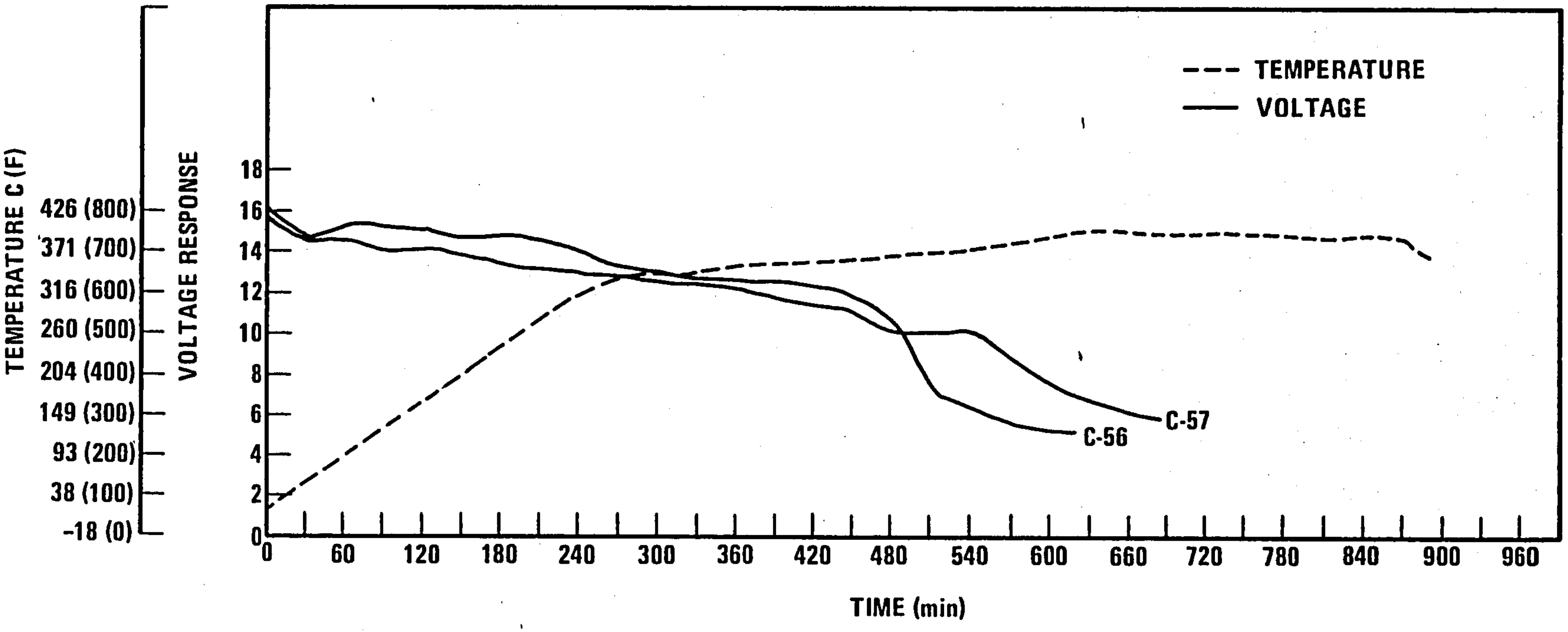


Figure 46. Dielectric Response vs Temperature for Laminates C-56 and C-57 Postcure

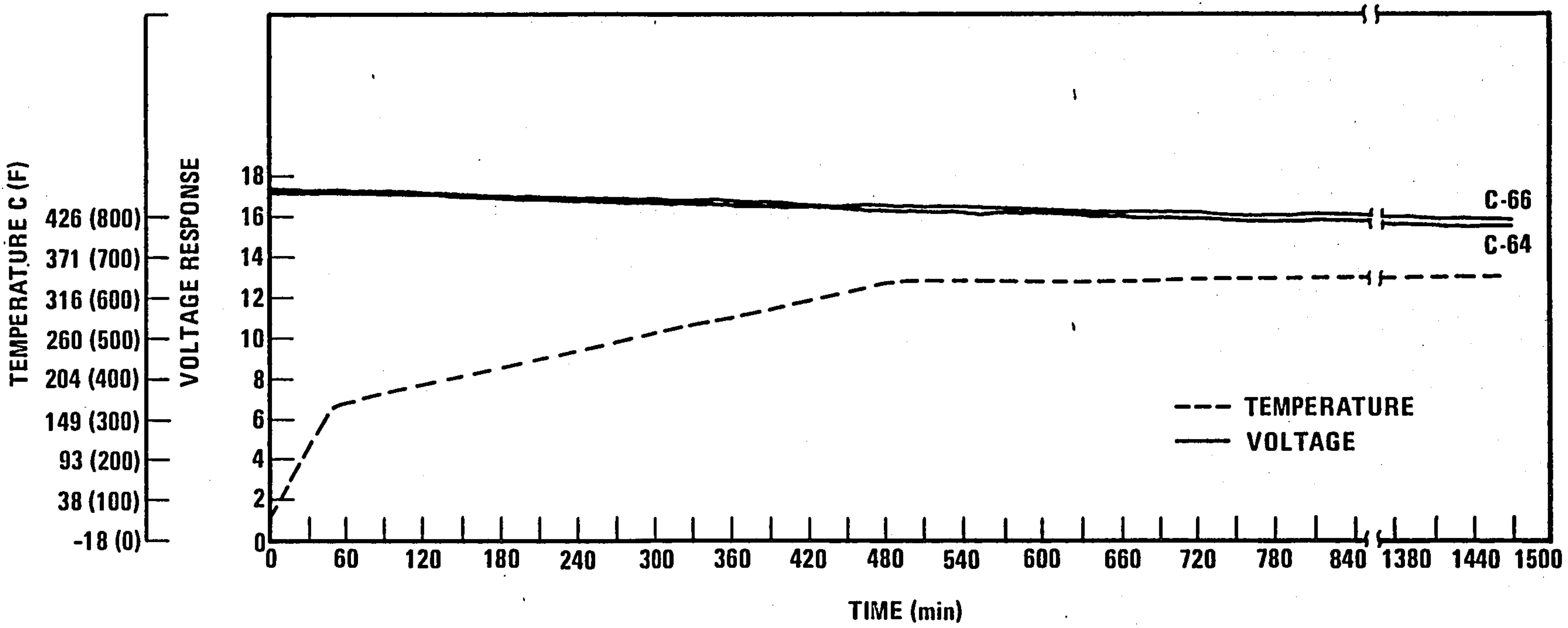


Figure 47. Dielectric Response vs Temperature for Laminates C-64 and C-66 Postcure

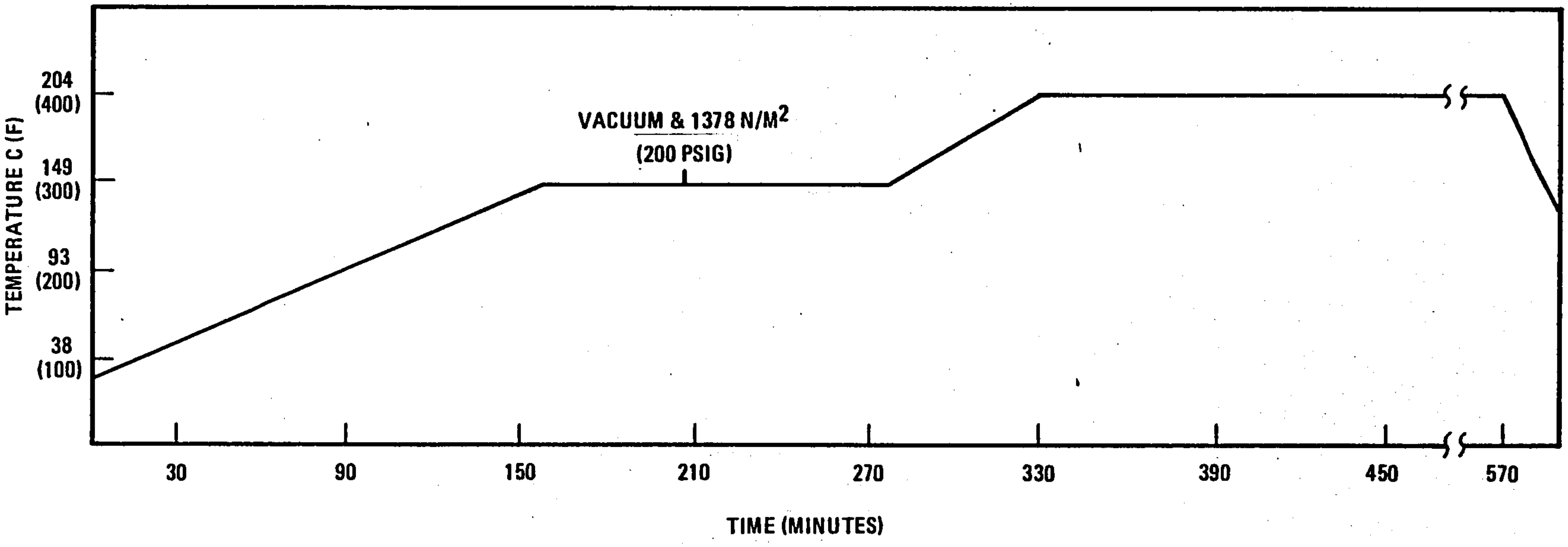
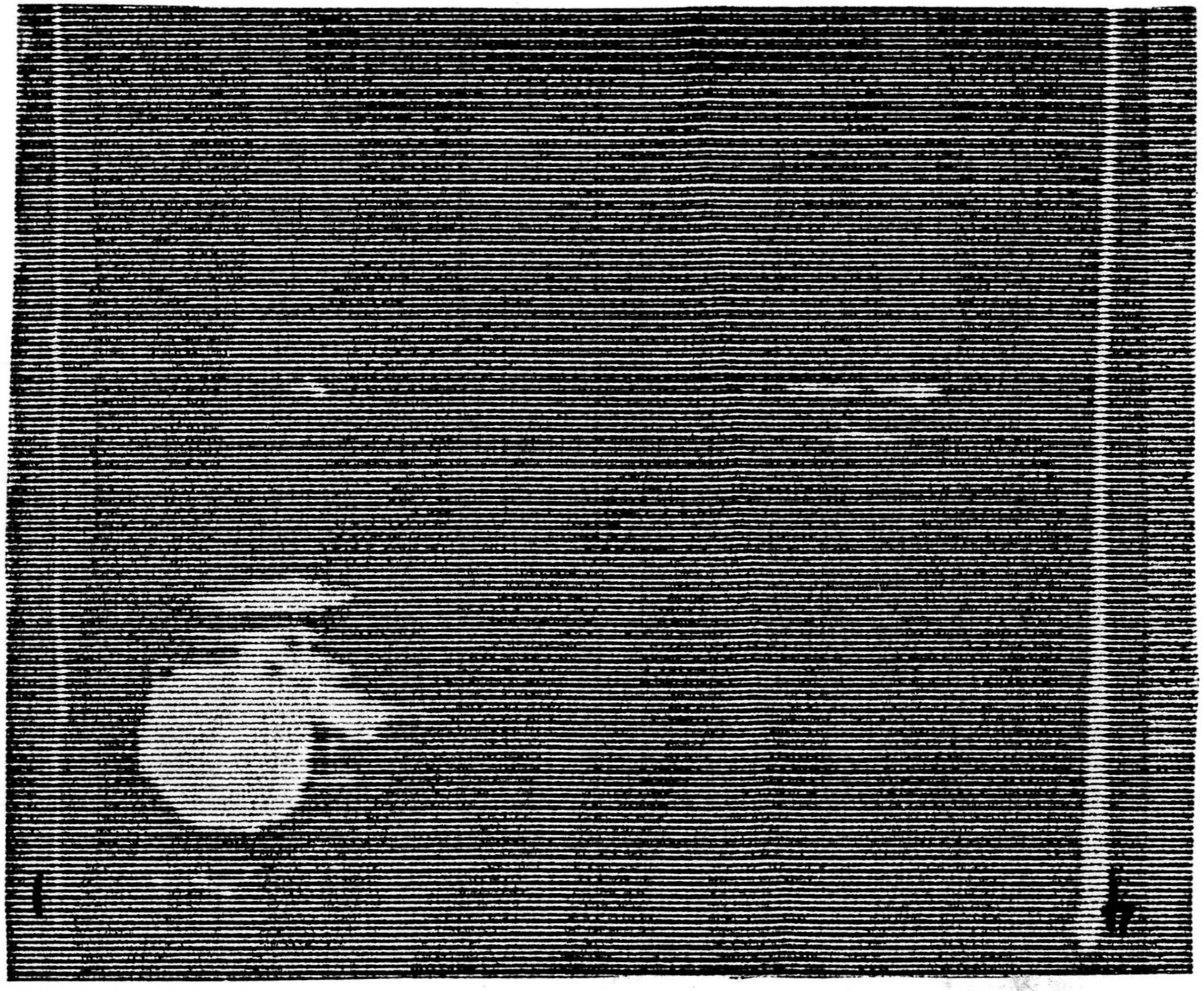


Figure 48. C-103/104 Cure Cycle



5 MHZ GAIN = 20

Figure 49. Ultrasonic C-Scan of Laminate C-103 after Cure.

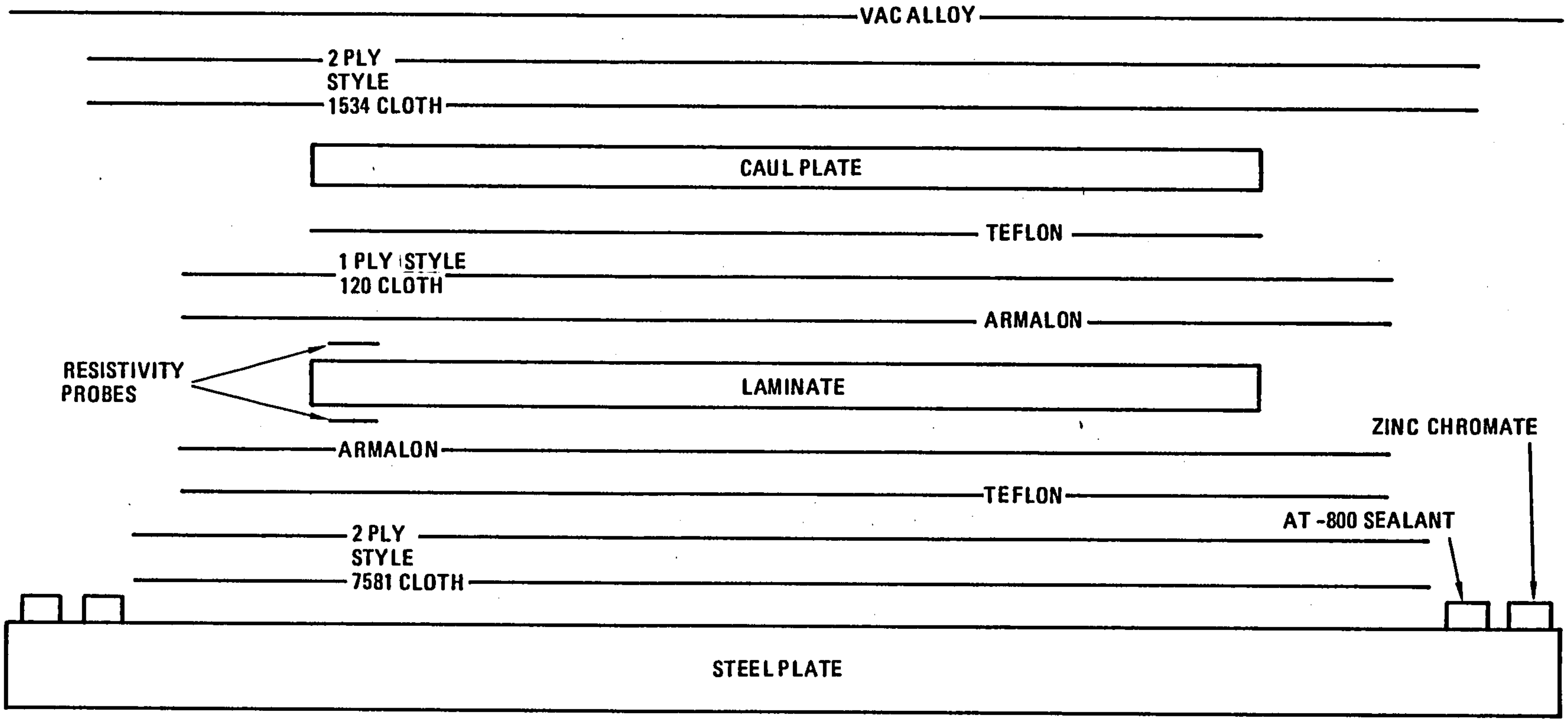


Figure 50. Laminates C-103, 104 Bagging Sequence.

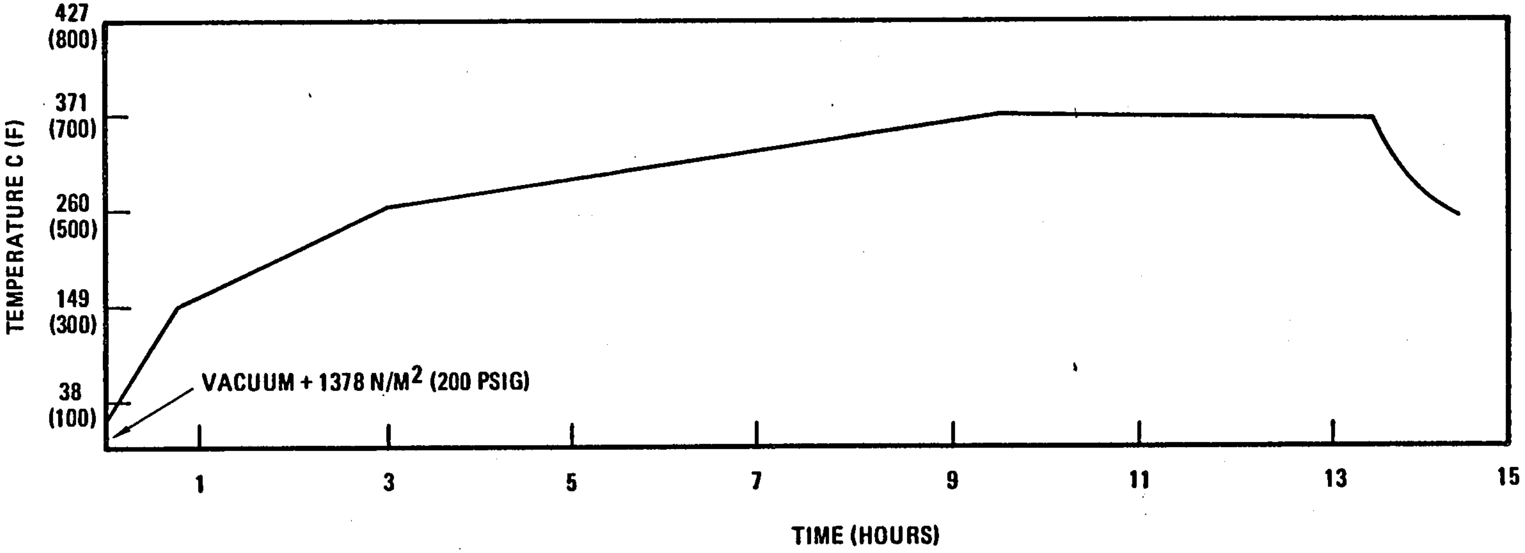
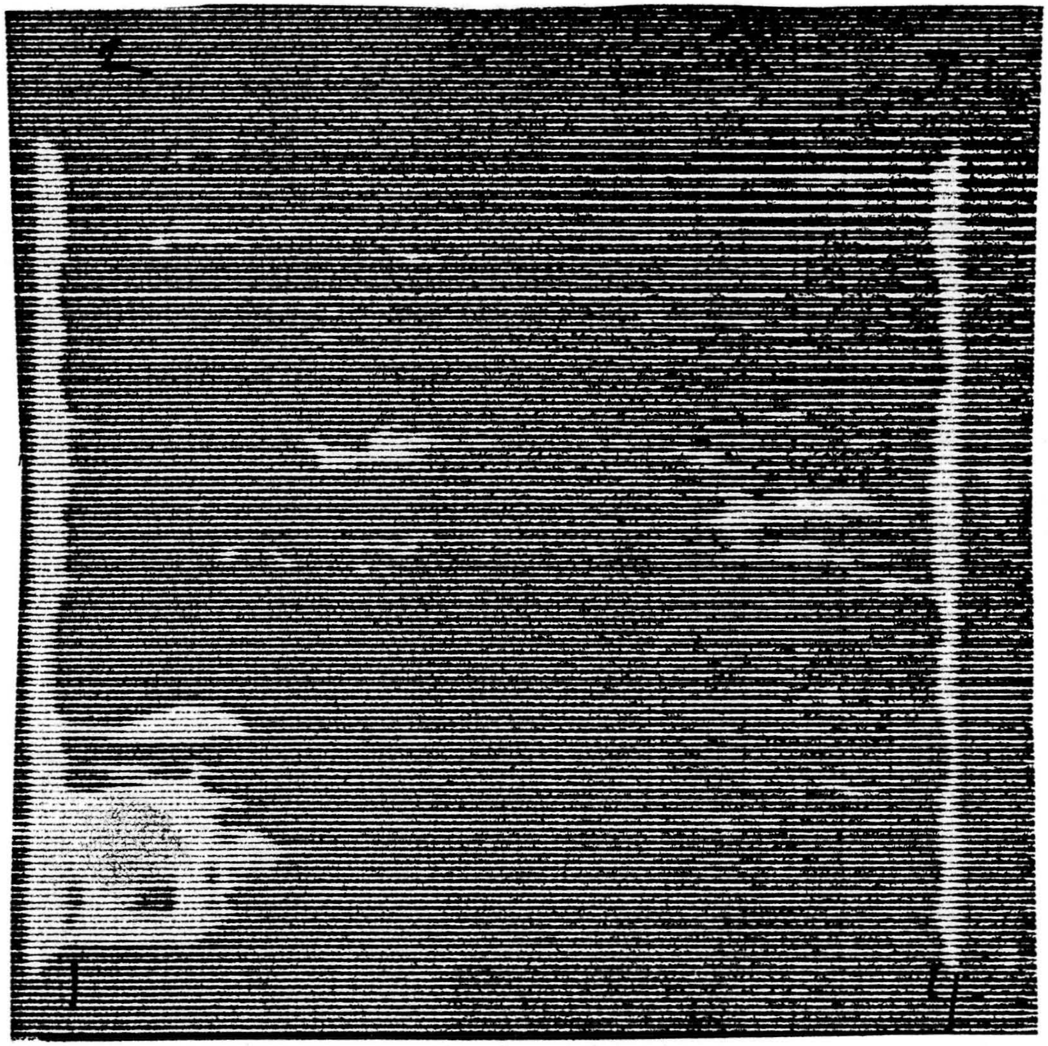


Figure 51. C-103/104 Postcure Cycle



5 MHZ GAIN = 20

Figure 52. Ultrasonic C-Scan of Laminate C-103 after Postcure.

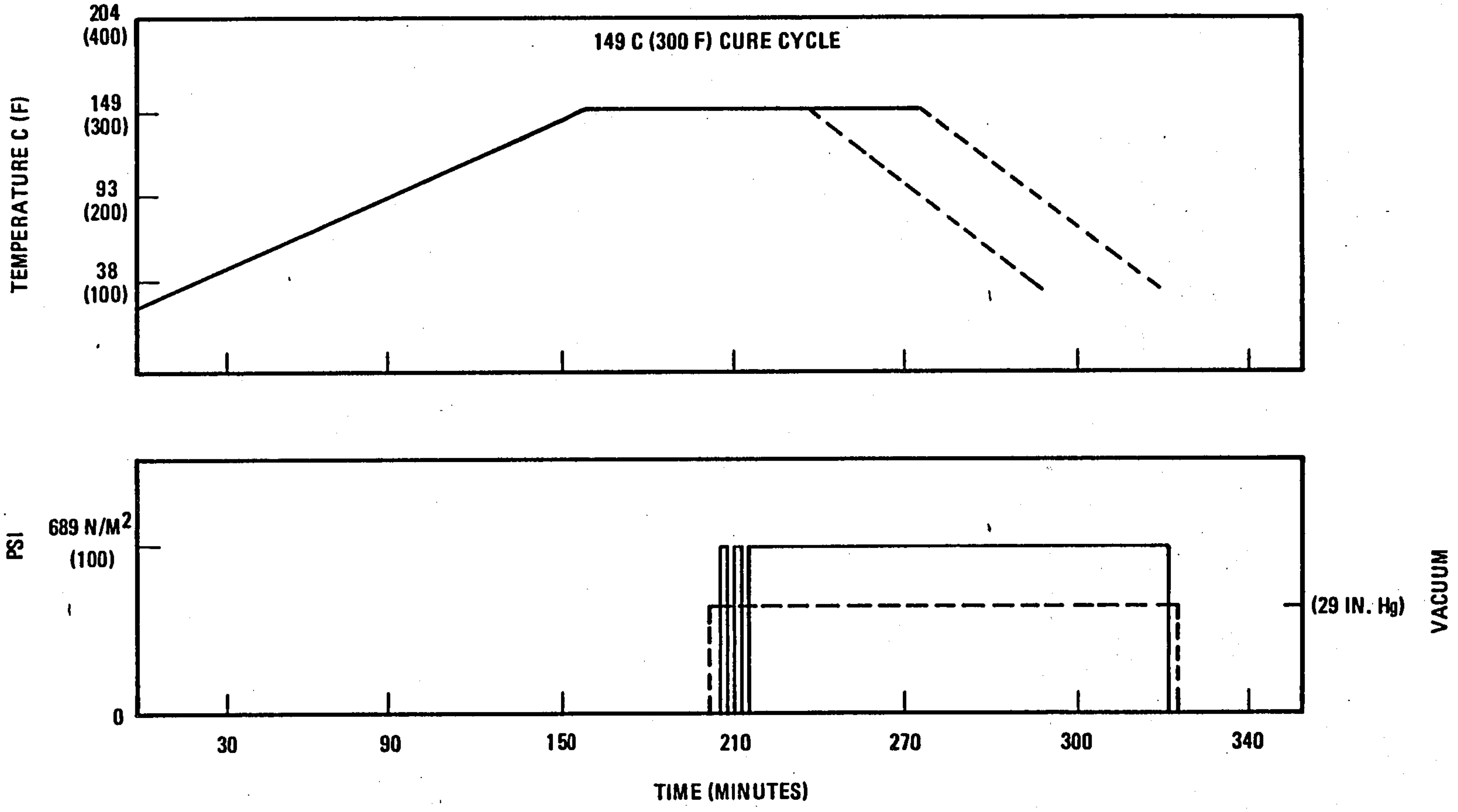


Figure 53. C-108 - 113 Cure Cycle

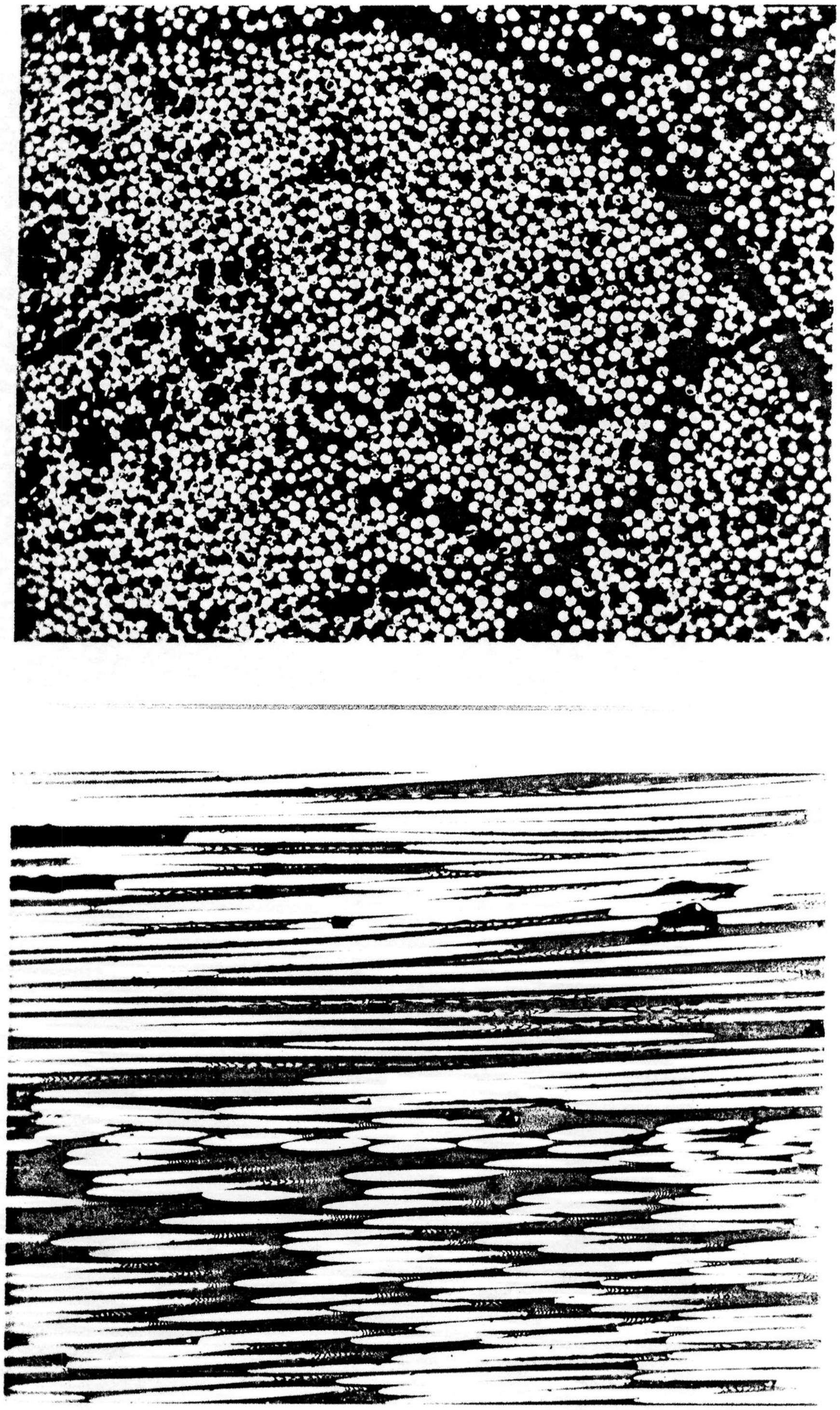


Figure 54. Photomicrograph of Laminate C-103

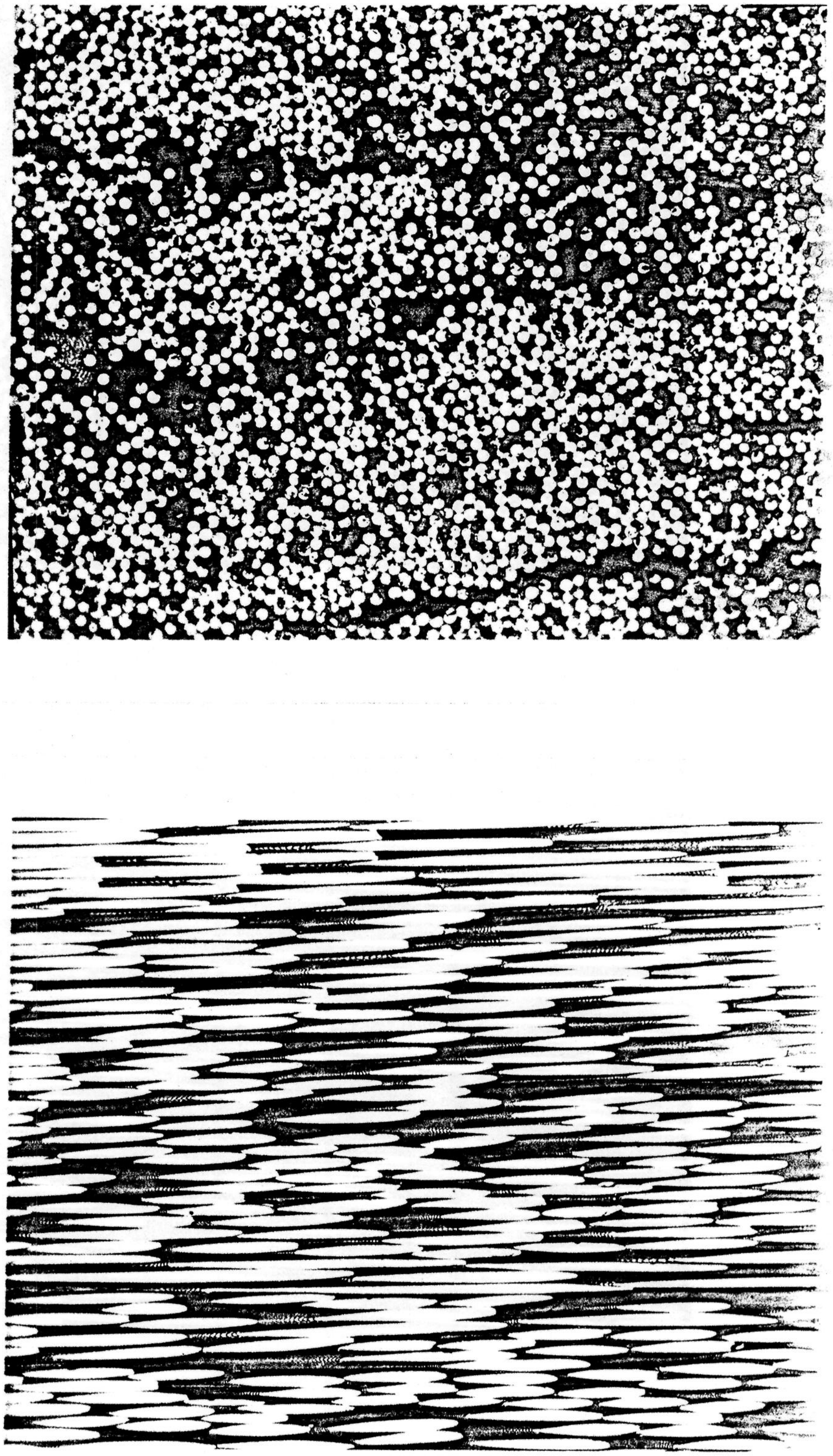


Figure 55/ Photomicrograph of Laminate C-108

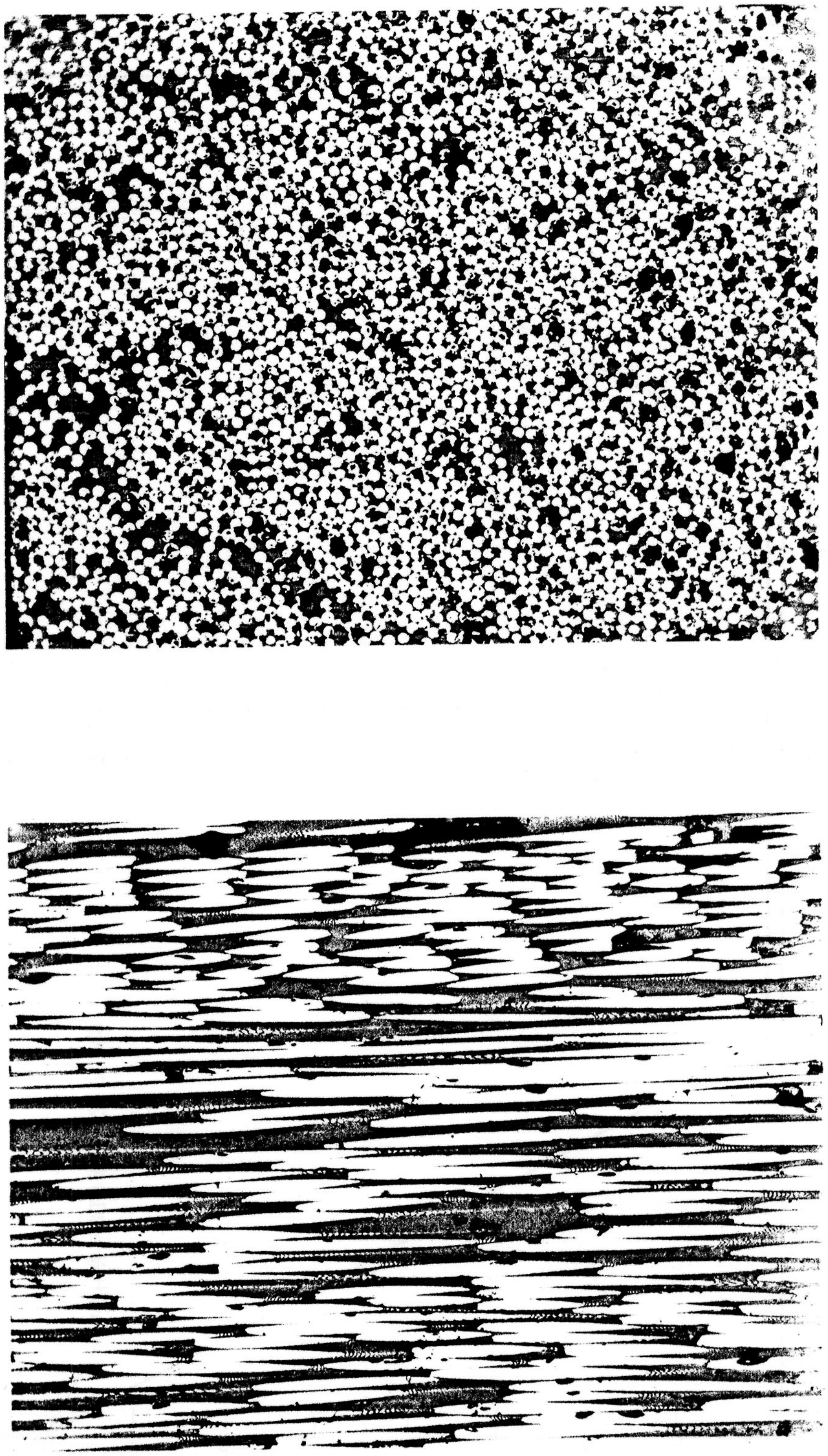


Figure 56. Photomicrograph of Laminate C-137

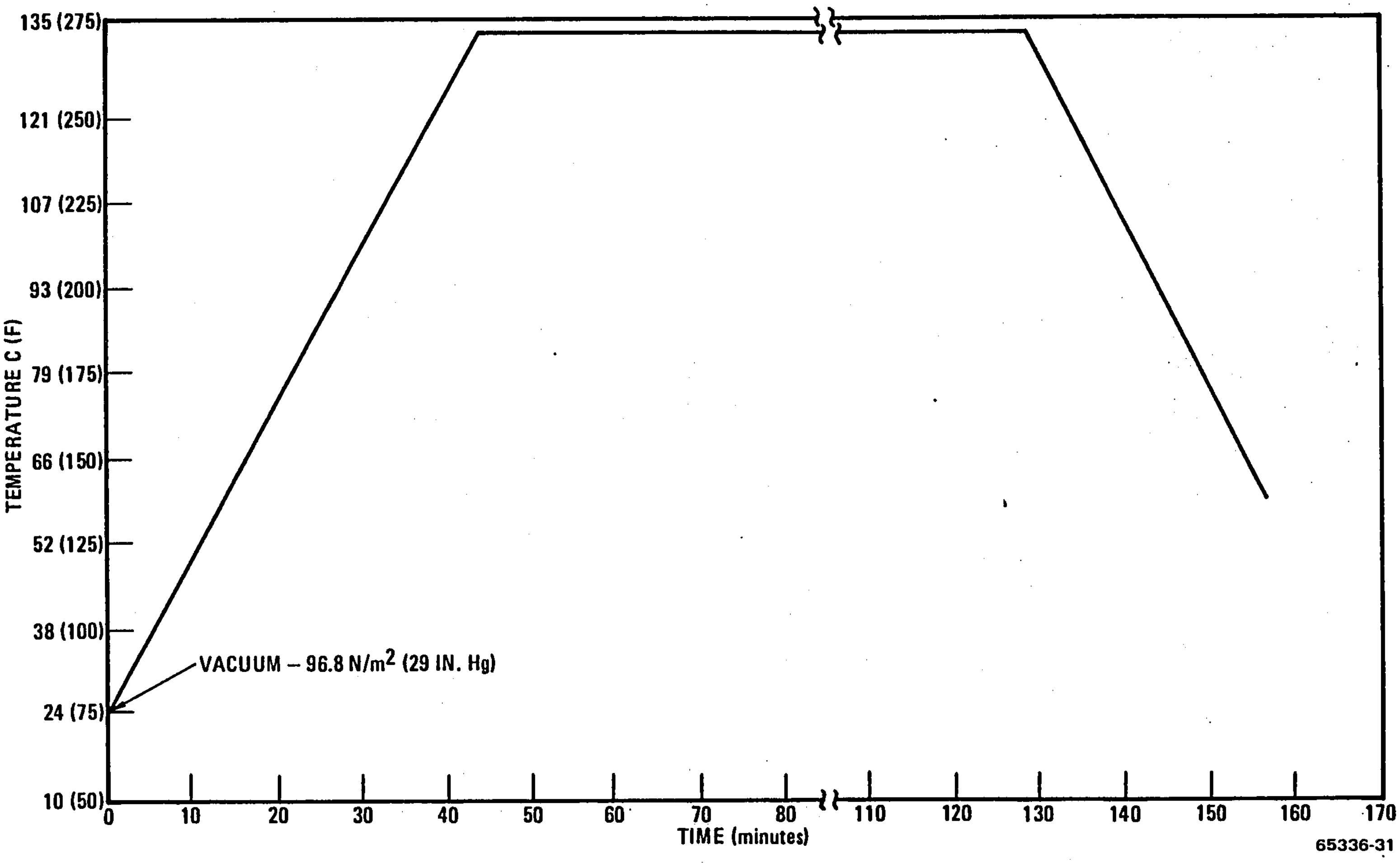


Figure 57. Precompaction Cycle for NR-150B2 and NR-150B2-S5X

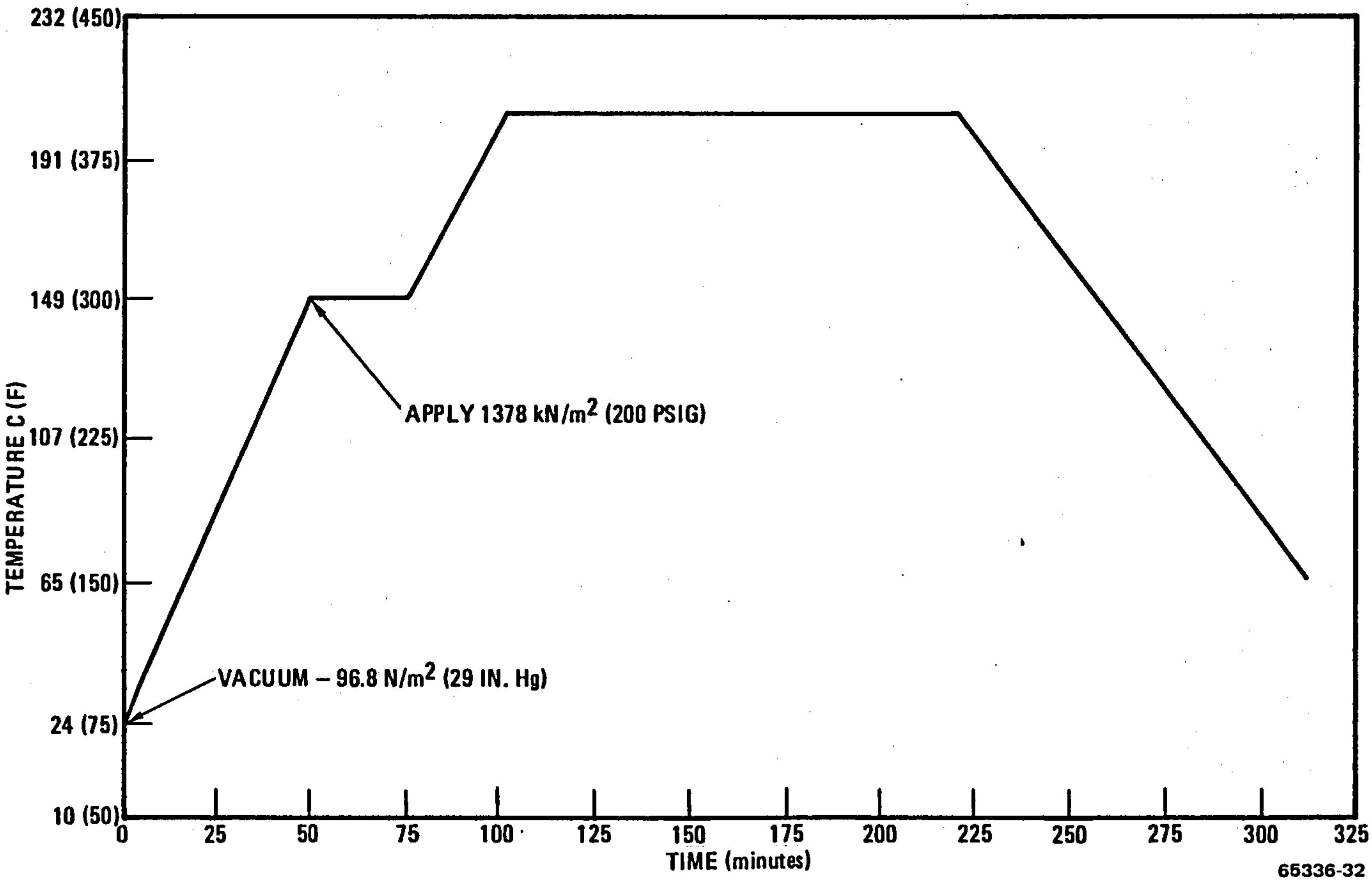


Figure 58. Cure Cycle for NR-150B2 and NR-150B2-S5X

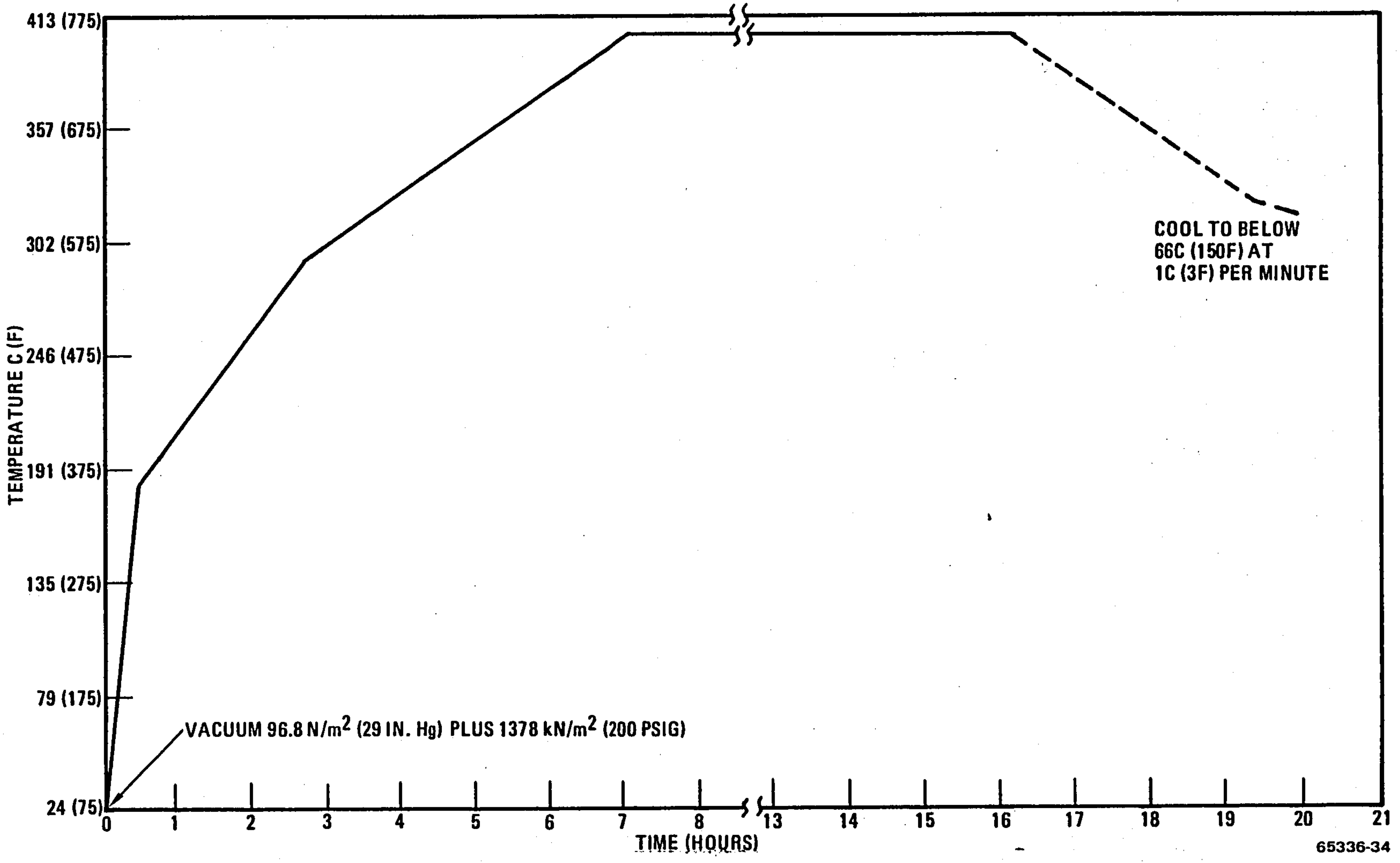


Figure 59. Postcure Cycle for NR-150BZ-S5X

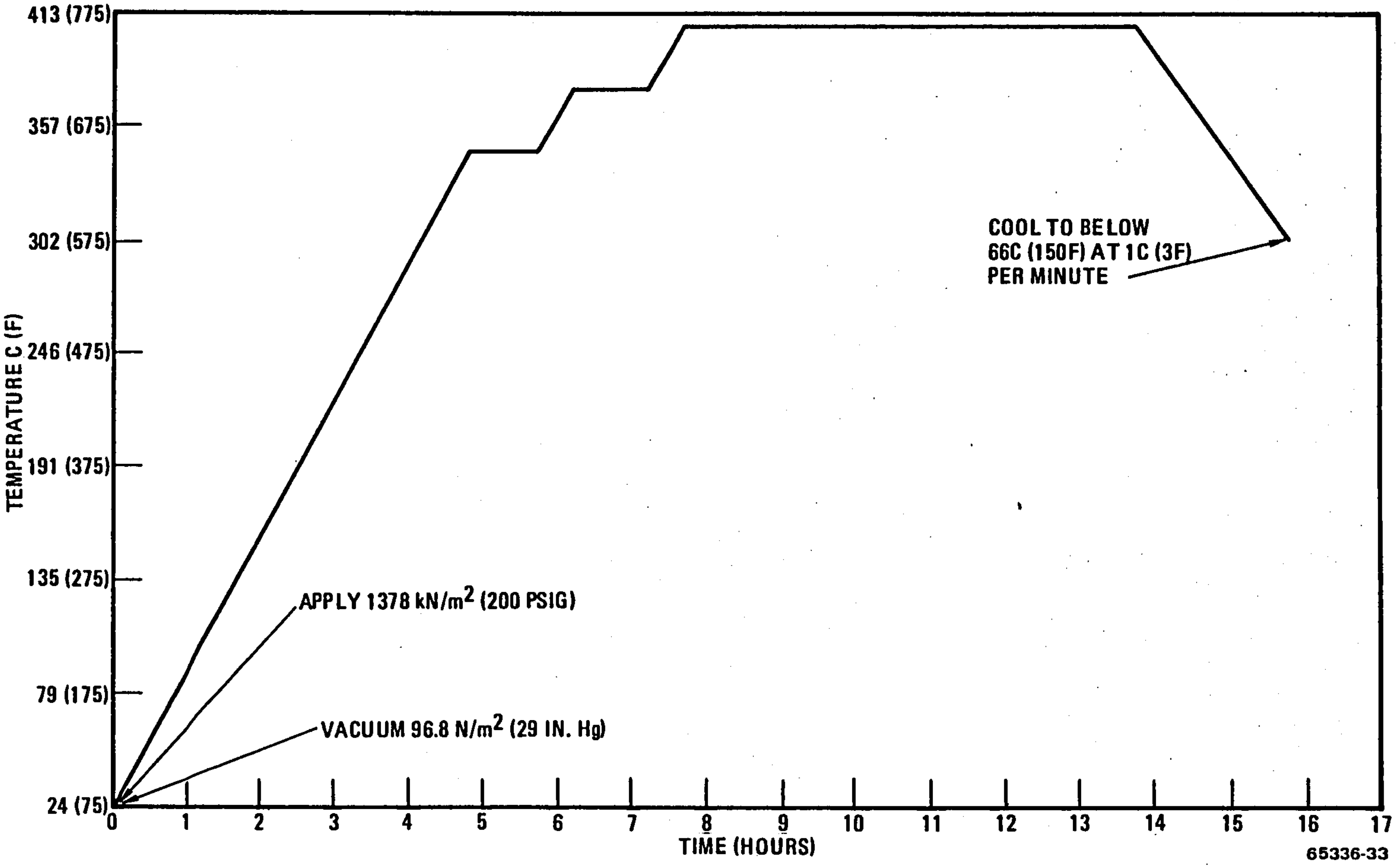


Figure 60. Postcure Cycle for NR-150B2

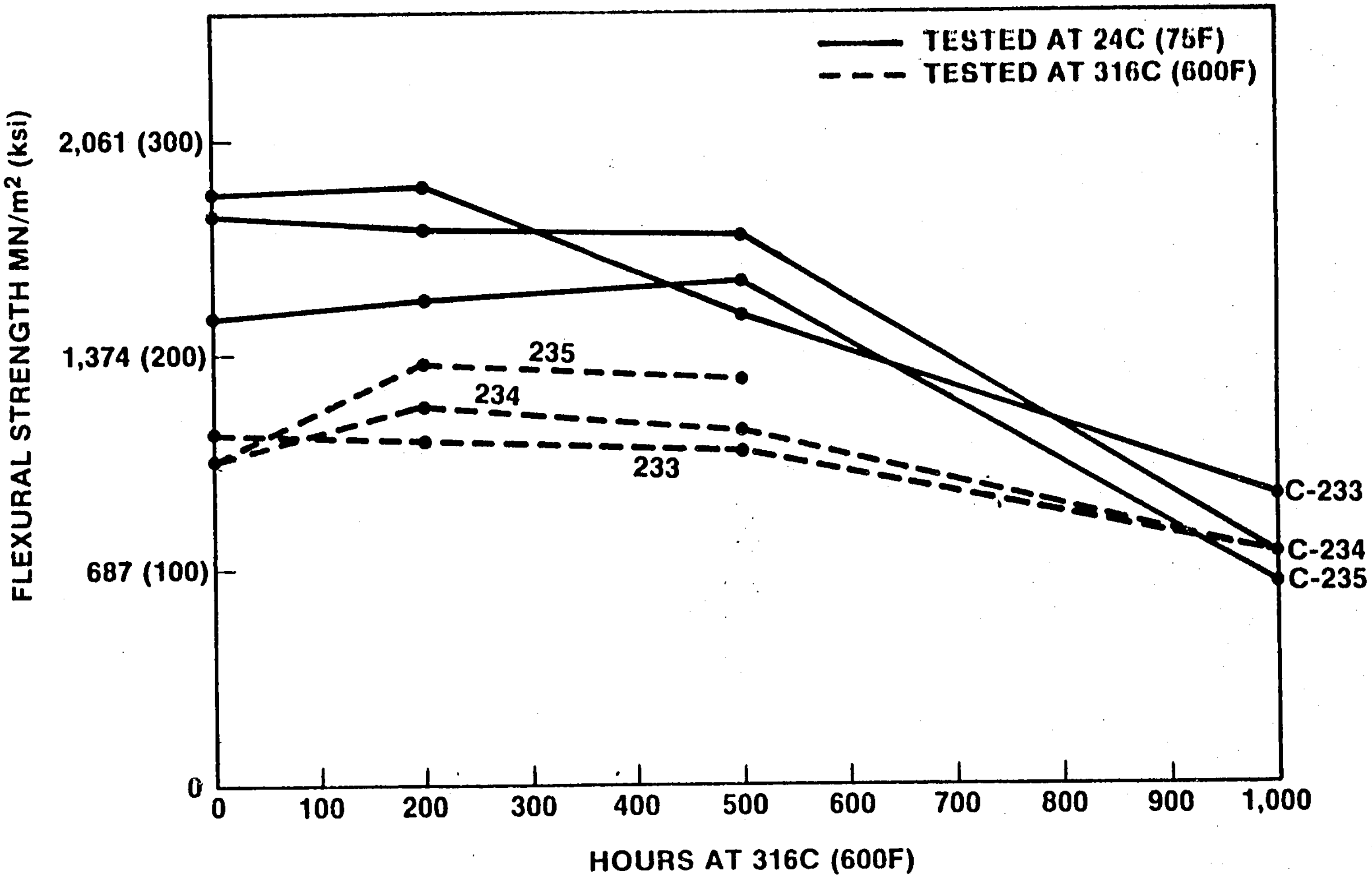
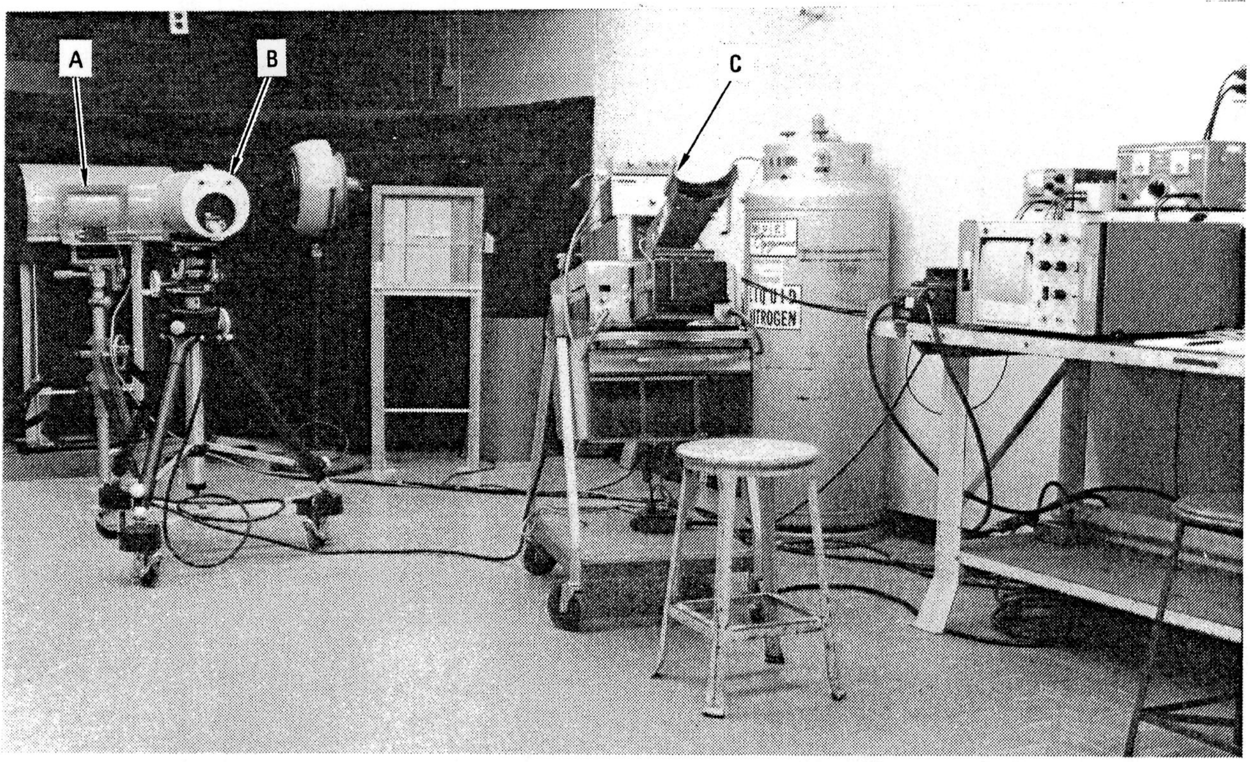


Figure 61. Heat Aging Test Results for C-6000/NR-150B-S5X Unidirectional Laminates



A. CURVED PANEL TEST SPECIMEN

B. IR CAMERA

C. DISPLAY/CONTROL

Figure 62. I.R. Scanner Arrangement

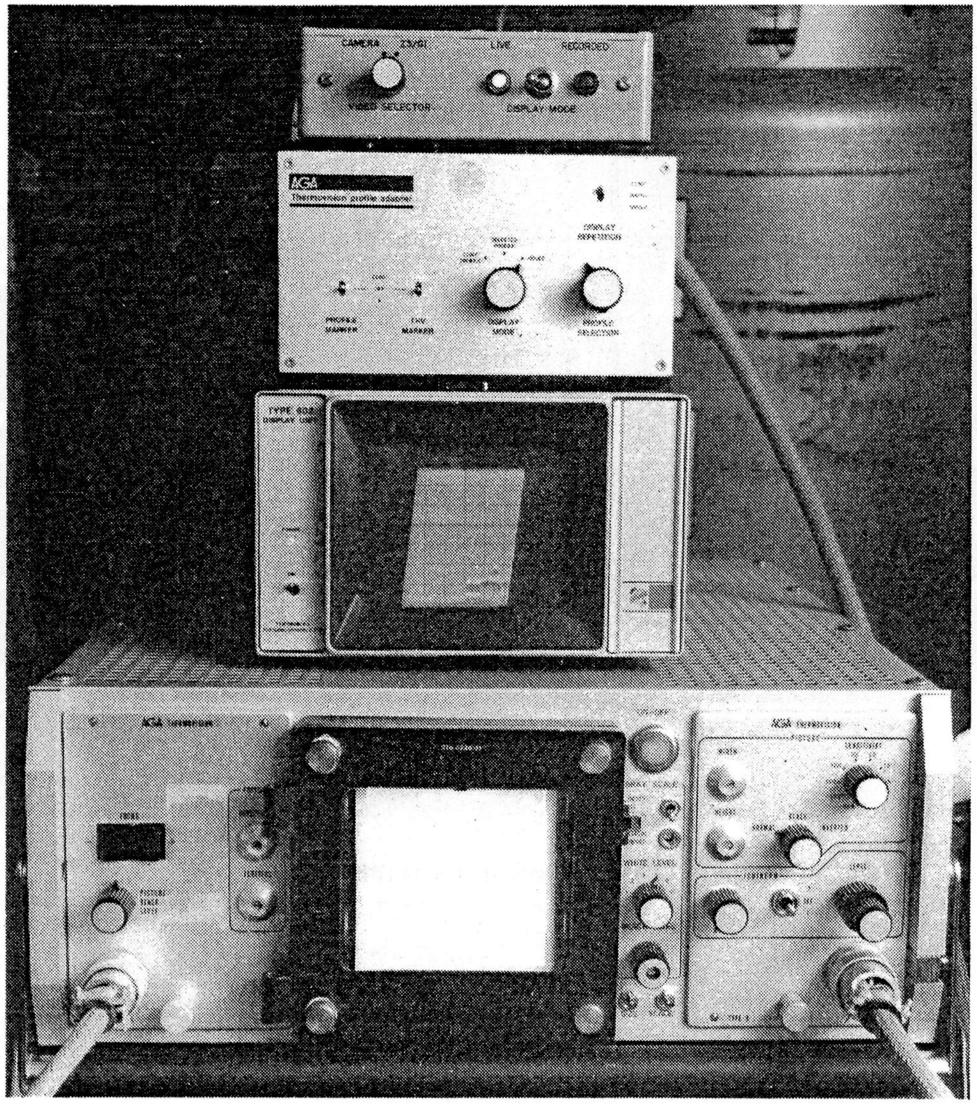


Figure 63. I.R. Scan Display/Control

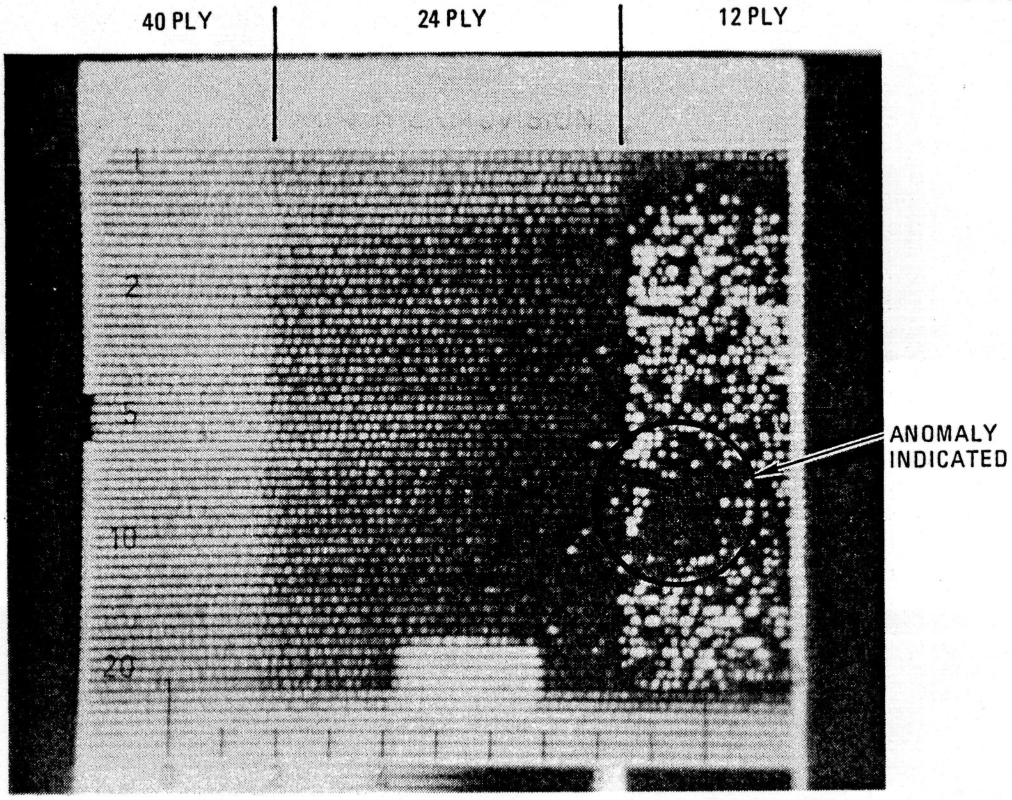
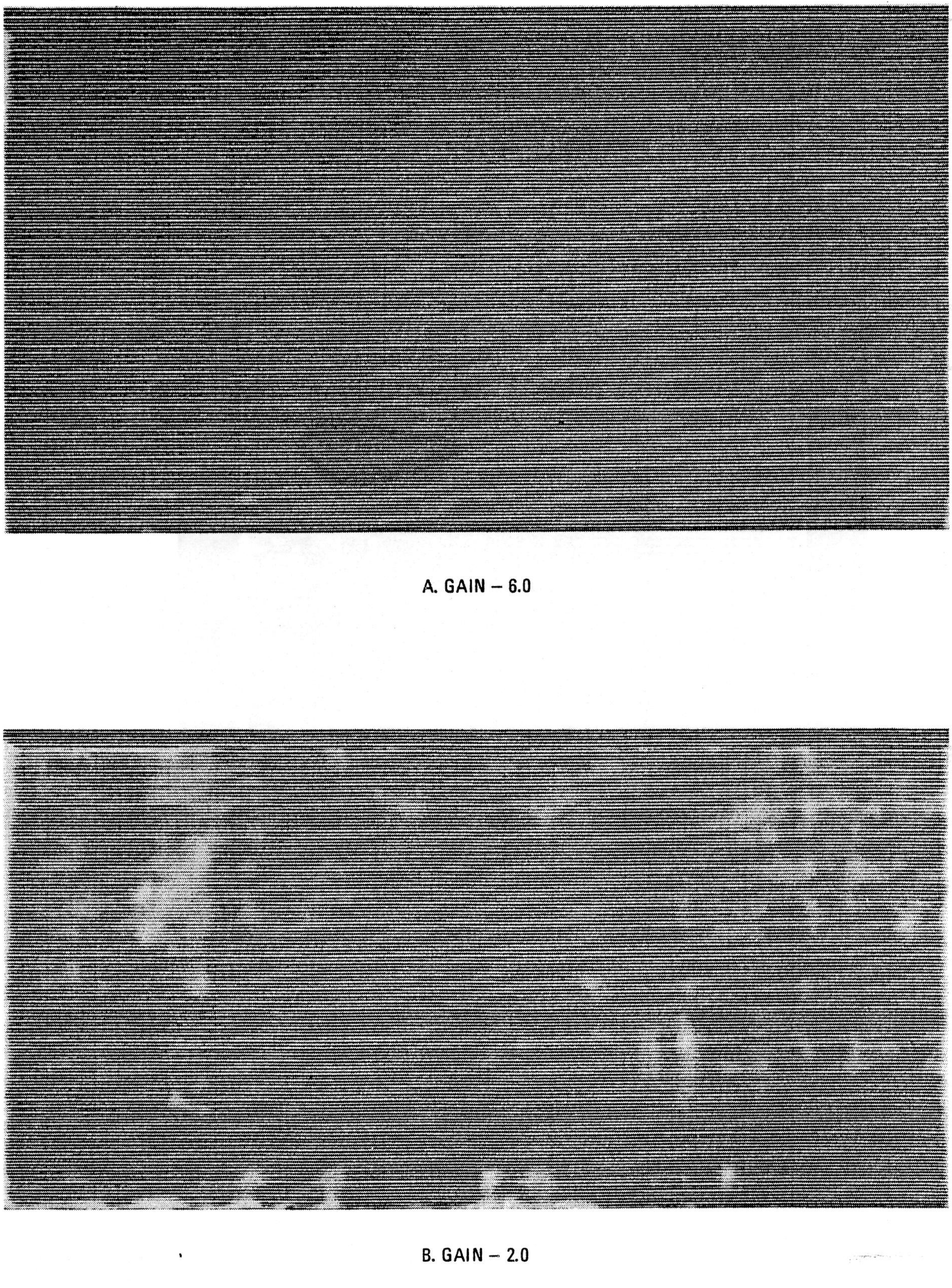


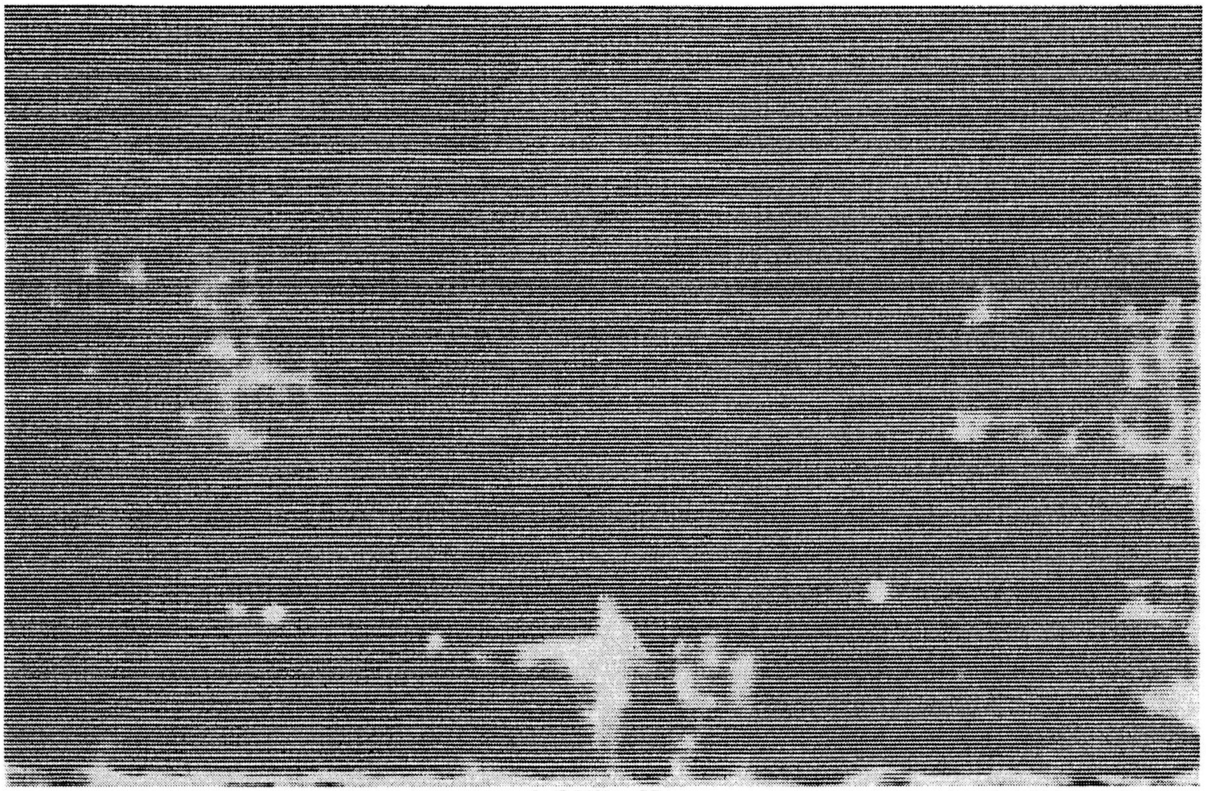
Figure 64. Thermogram for Variable Thickness GR/PI Panel.



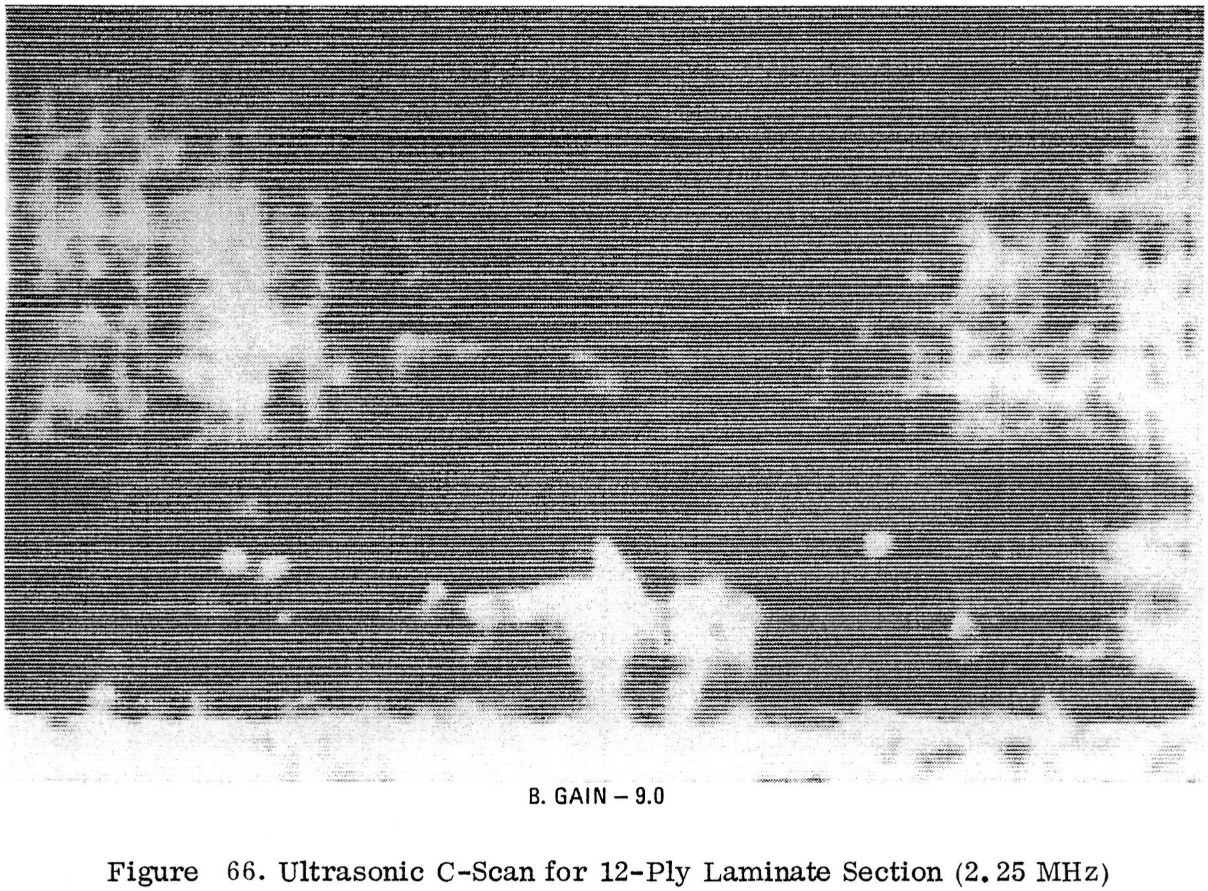
A. GAIN – 6.0

B. GAIN – 2.0

Figure 65. Ultrasonic C-Scan for 6-Ply Laminate Section (2.25 MHz)

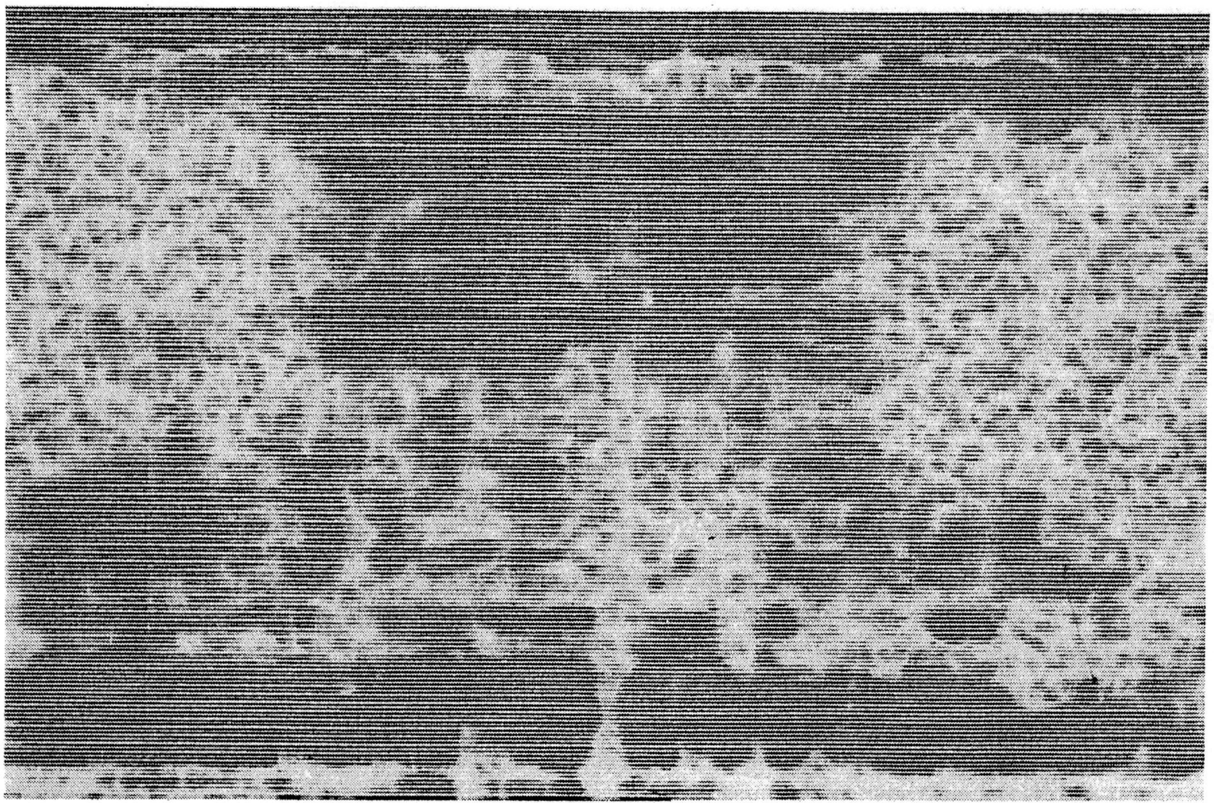


A. GAIN – 20

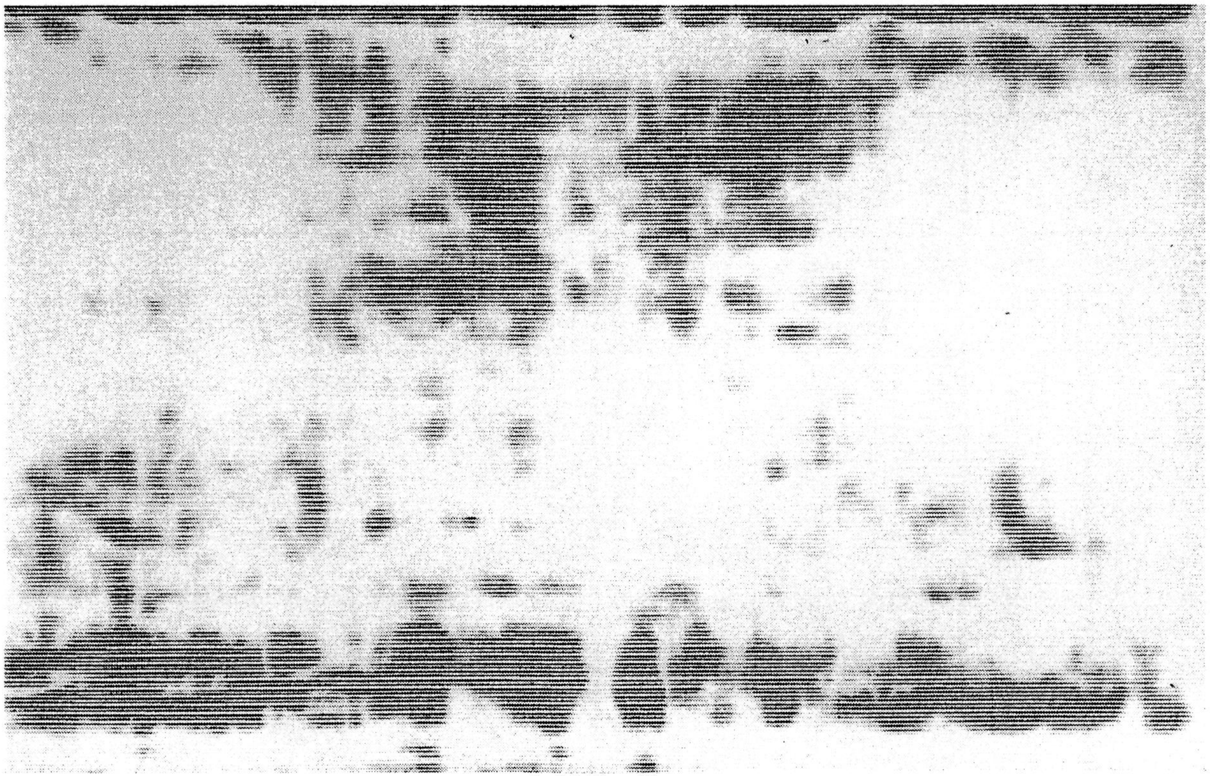


B. GAIN – 9.0

Figure 66. Ultrasonic C-Scan for 12-Ply Laminate Section (2.25 MHz)

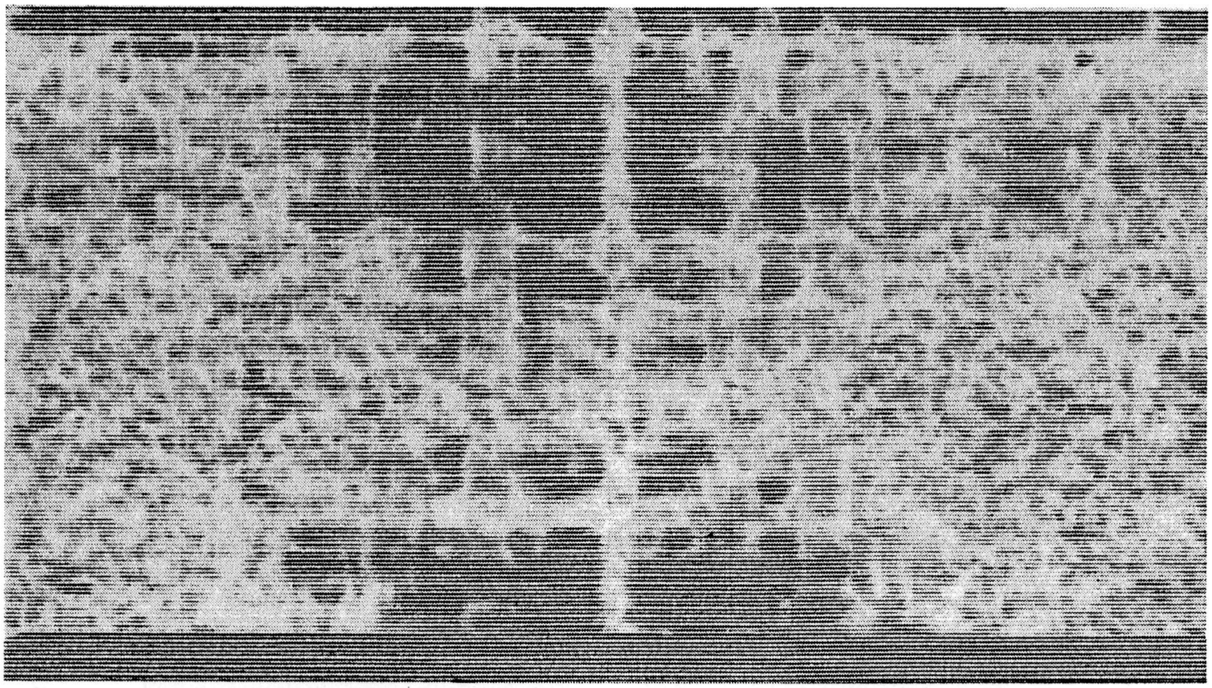


A. GAIN – 70

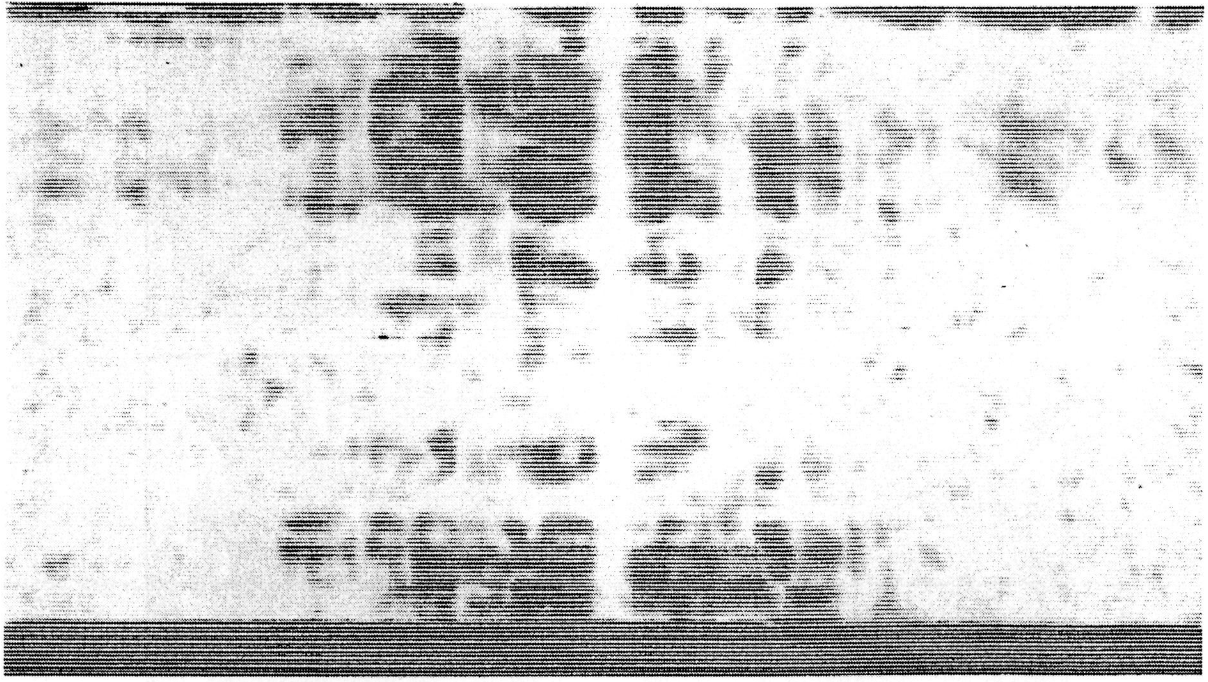


B. GAIN – 20

Figure 67. Ultrasonic C-Scan for 24-Ply Laminate Section (2.25 MHz)



A. GAIN – 70



B. GAIN – 20

Figure 68. Ultrasonic C-Scan for 40-Ply Laminate Section (2.25 MHz)

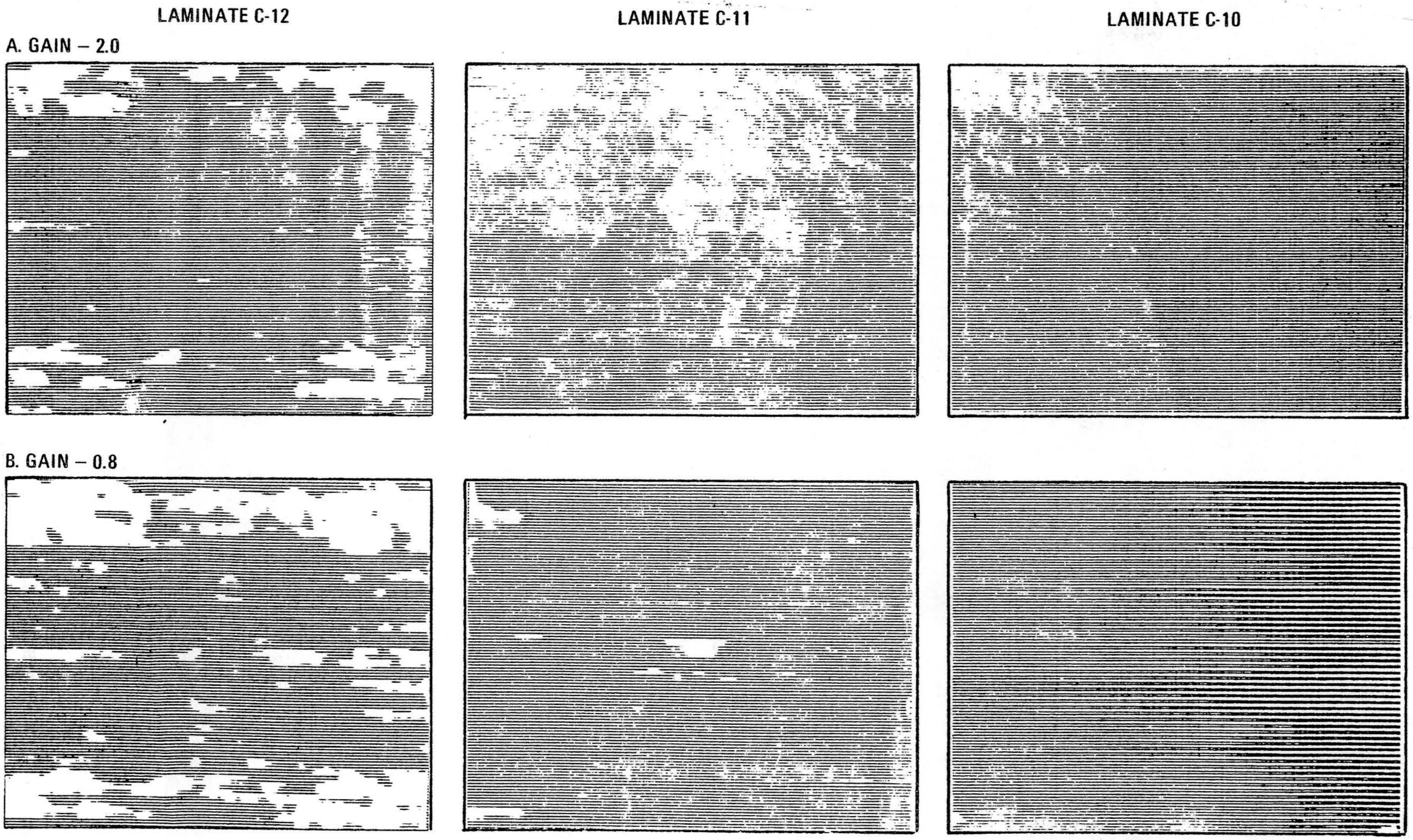


Figure 69. C-Scan for 12-Ply Modmor II/NR-150B2 Graphite/Polyimide Laminates (5 MHz) (Sheet 1)

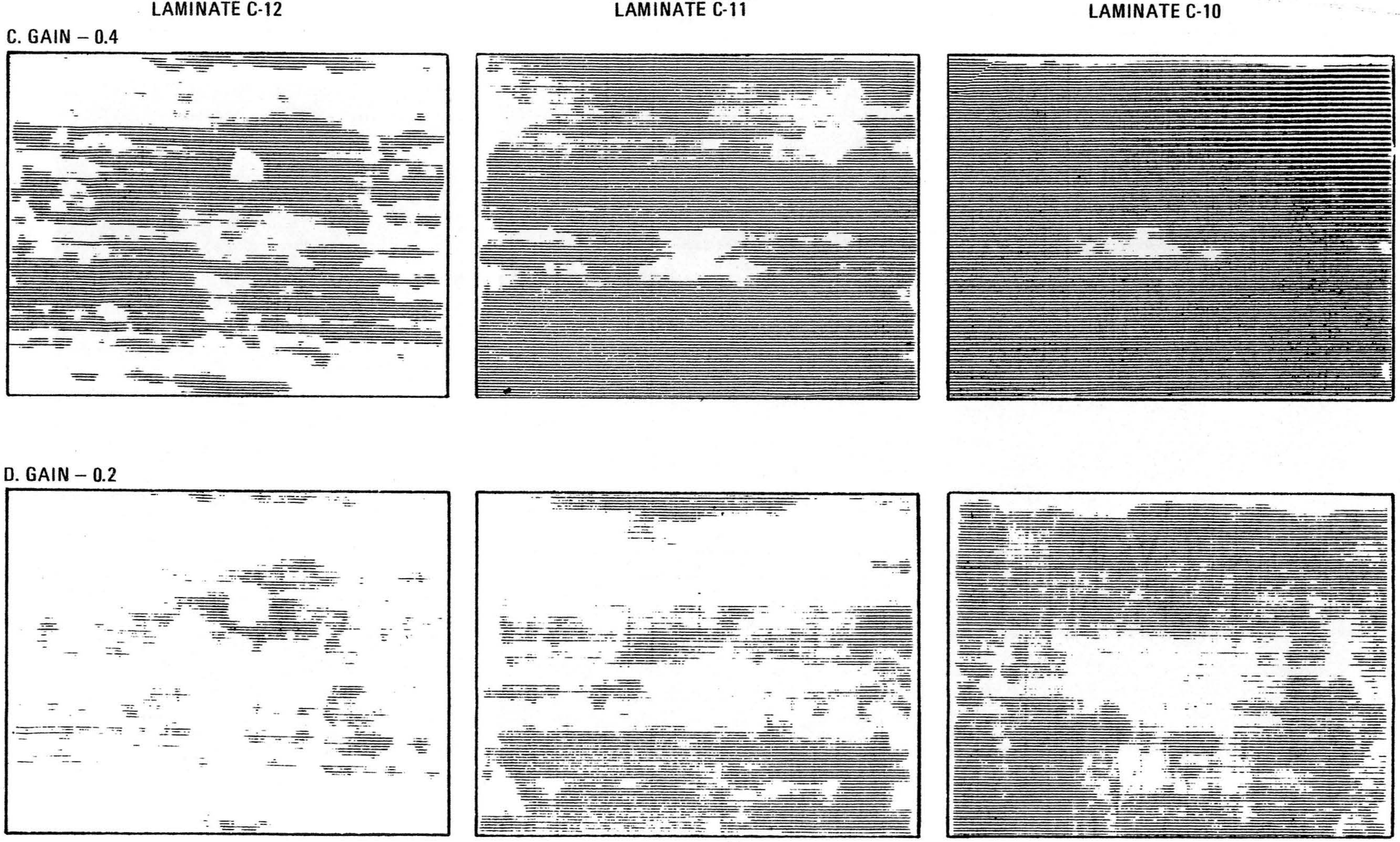


Figure 69. C-Scan for 12-Ply Modmor II/NR-150B2 Graphite/Polyimide Laminates (5 MHz) (Sheet 2)

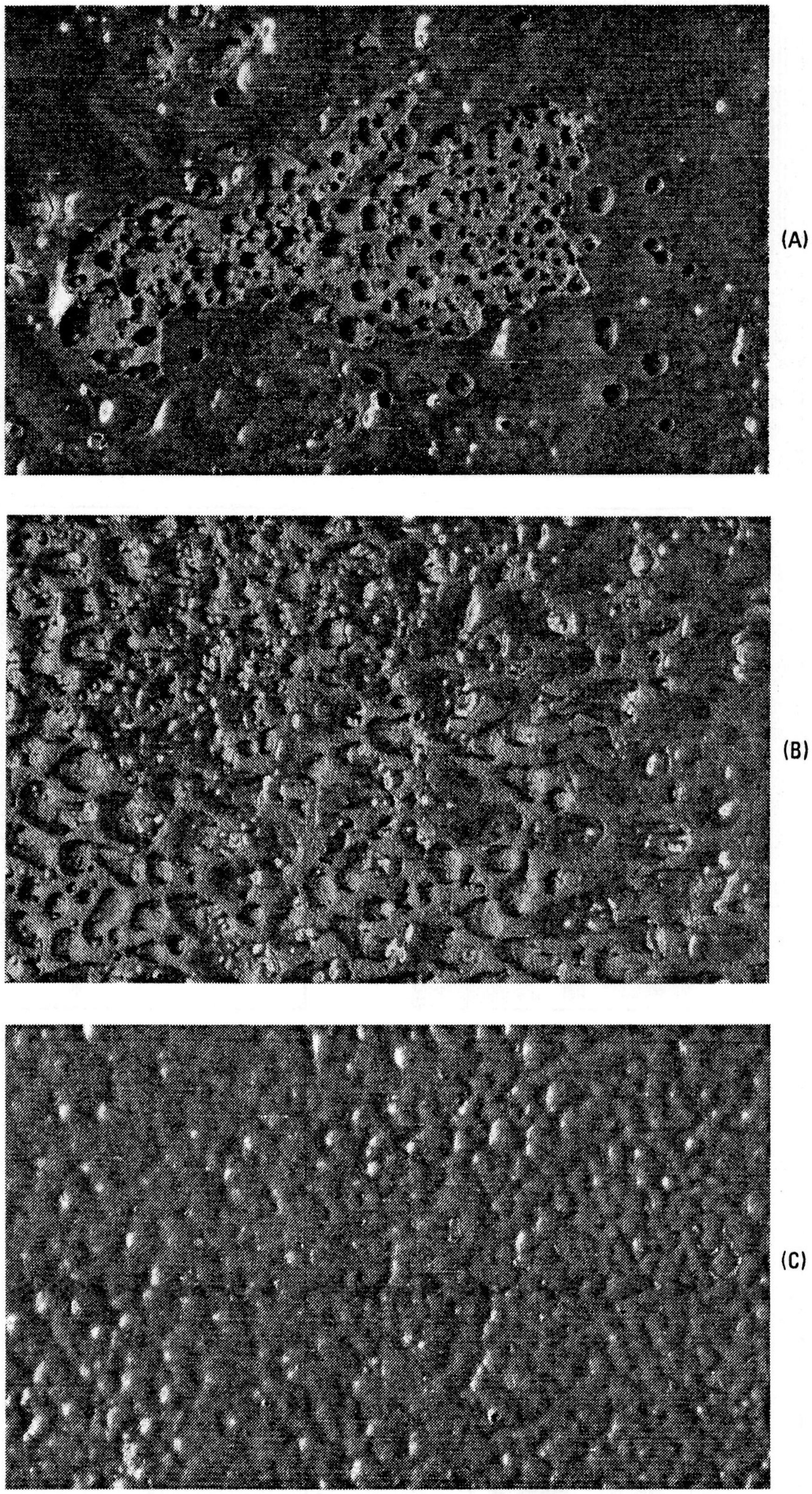


Figure 70. B-Staged 120 Impregnated Adhesive Film
(7X Magnification)

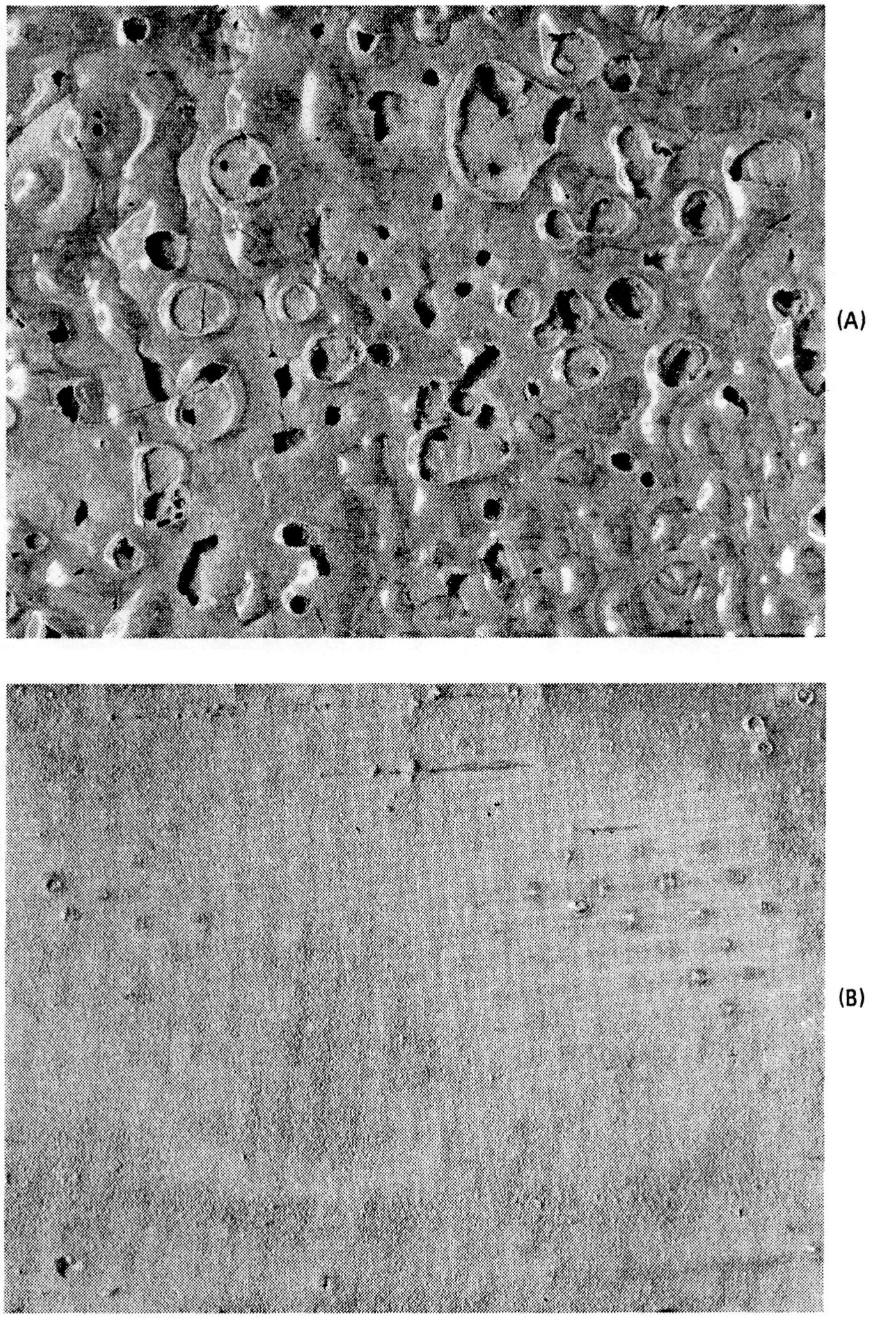


Figure 71. B-Staged 112 Impregnated Adhesive (7X Magnification)

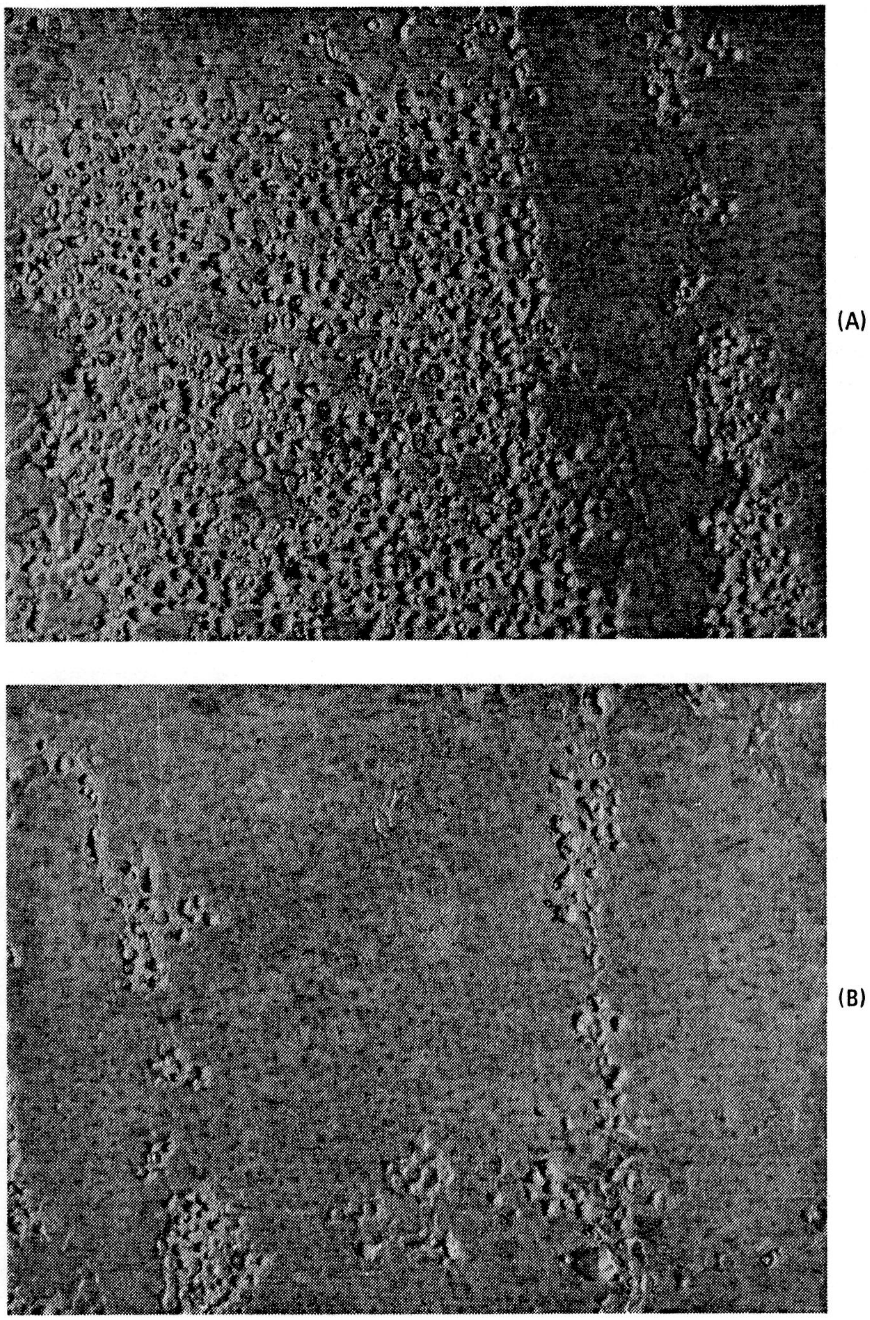


Figure 72. Cured 120 Impregnated Adhesive (7X Magnification)

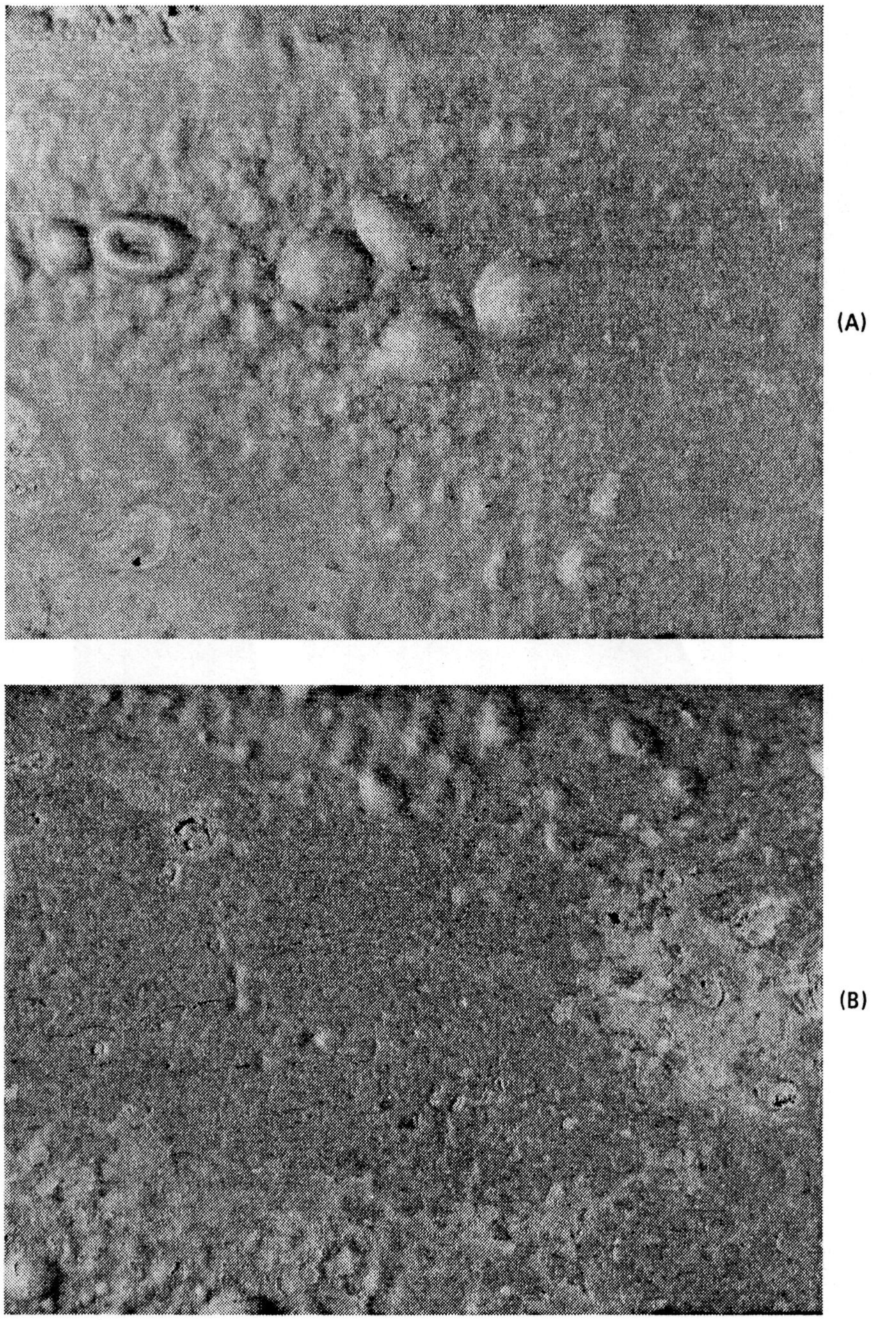


Figure 73. Cured 112 Impregnated Adhesive (7X Magnification)

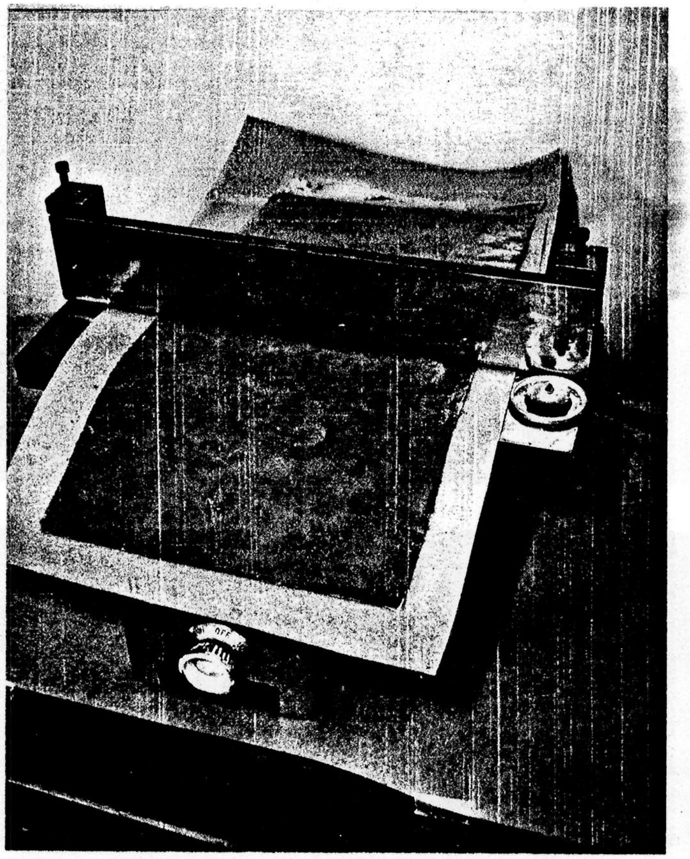


Figure 74. Set-Up for Preparing Adhesive Prepreg

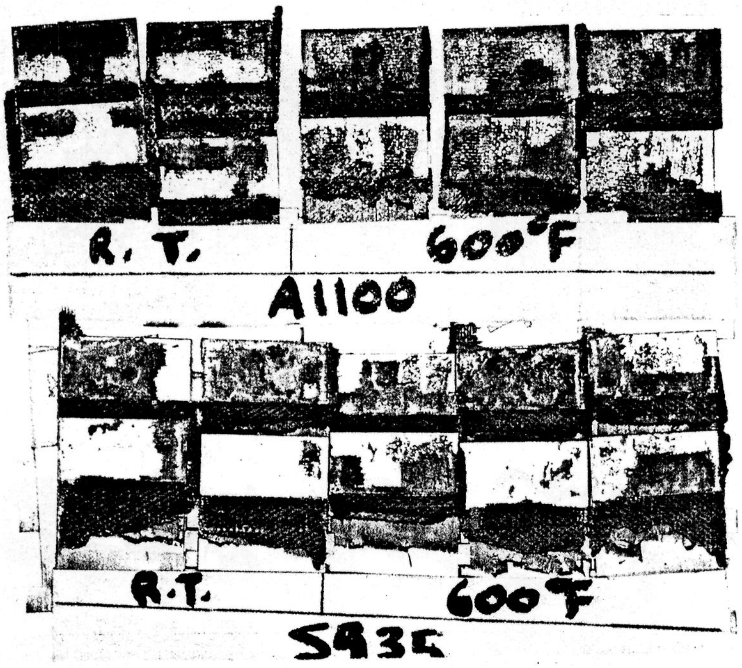


Figure 75. Failed Lap Shear Test Specimens

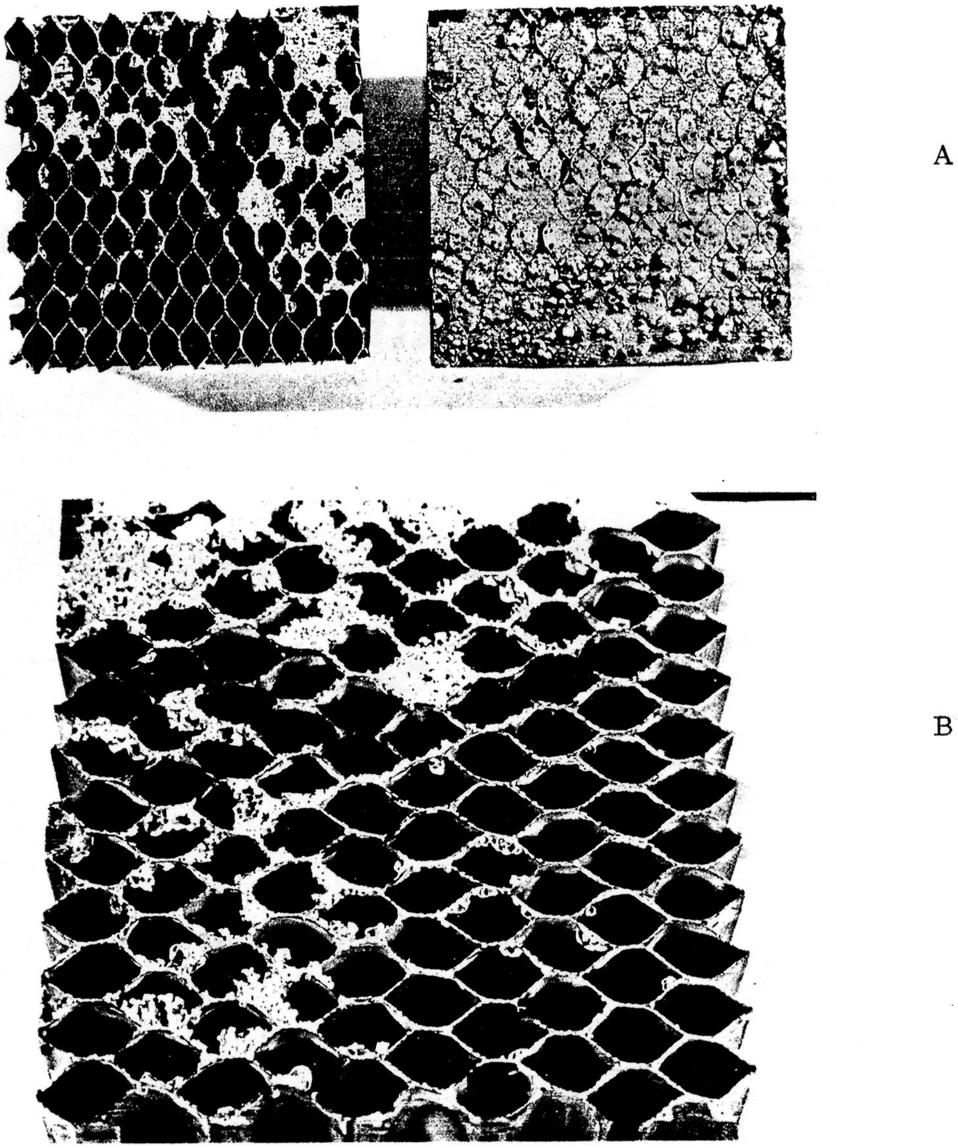


Figure 76. Failed Flatwise Tension Test Specimen
(NR 150 Graphite Laminates/HRH Polyimide
Honeycomb Core)

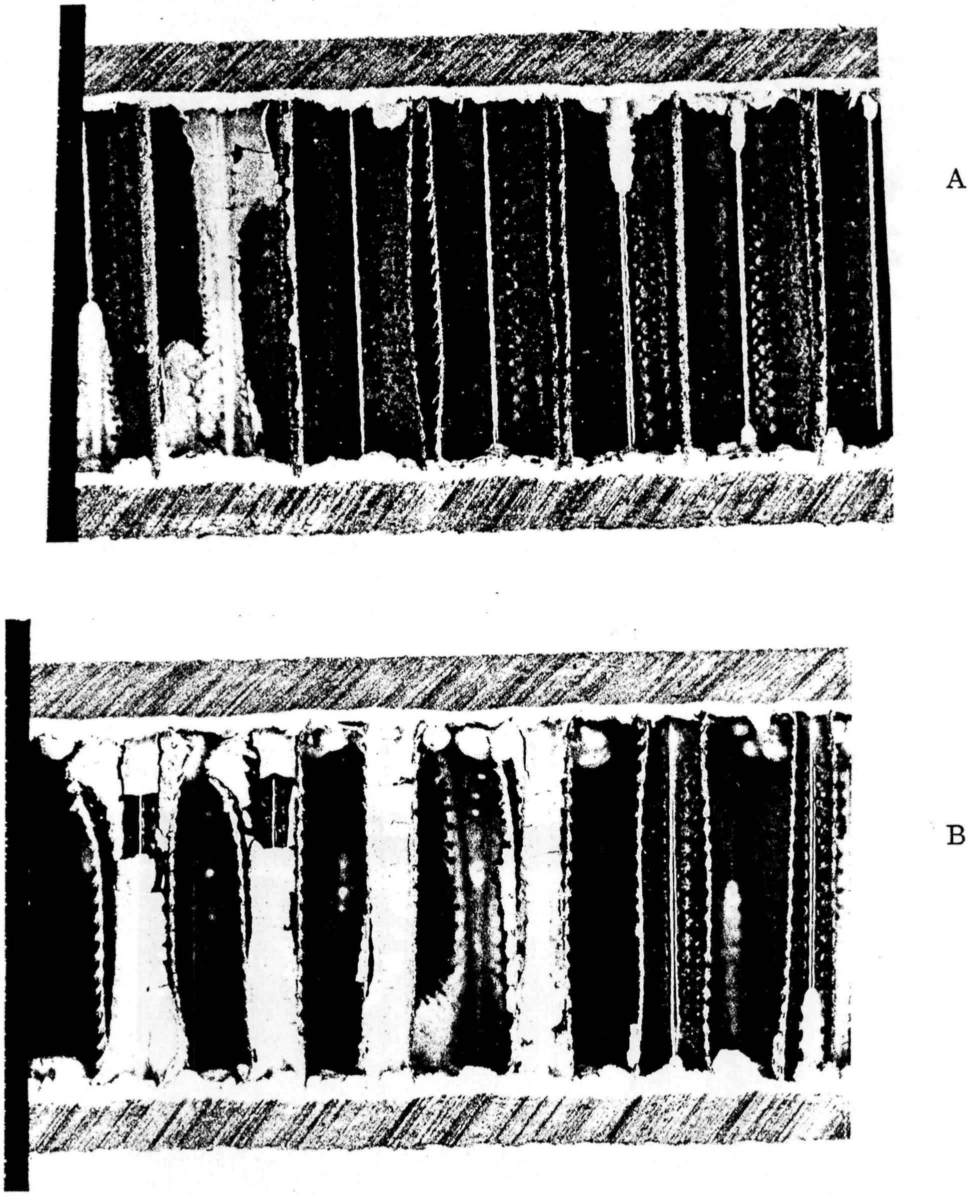
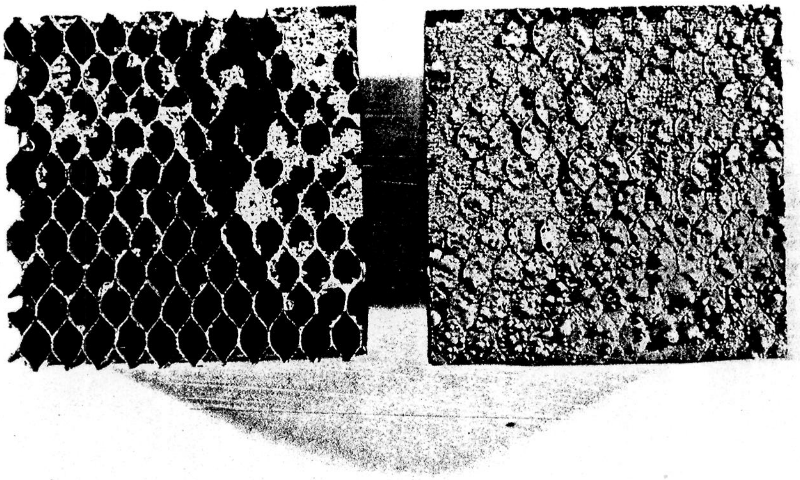


Figure 77. Adhesive Flow Observed in a Bonded Sandwich Specimen



✓ Figure 78. Flatwise tensile specimen of Celion 6000/NR150B2 composite laminate bonded to HRH327 glass/polyimide core using NR150B2 adhesive after testing.

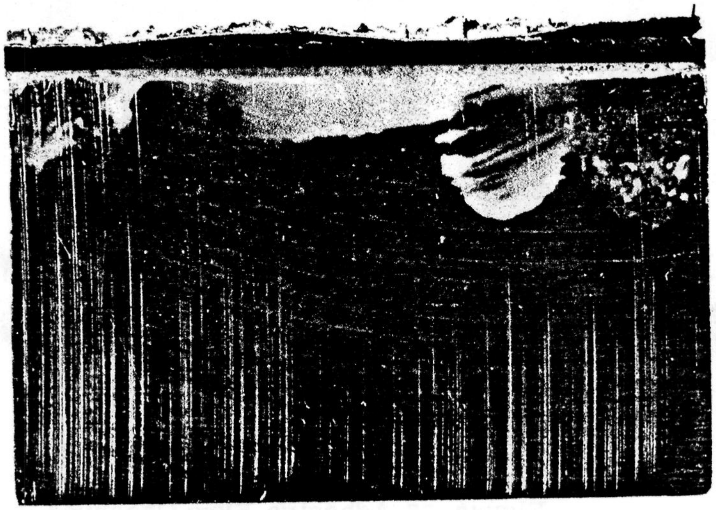


Figure 79. Delamination of Celion 6000/NR-150B2 laminate observed after flatwise tensile testing.

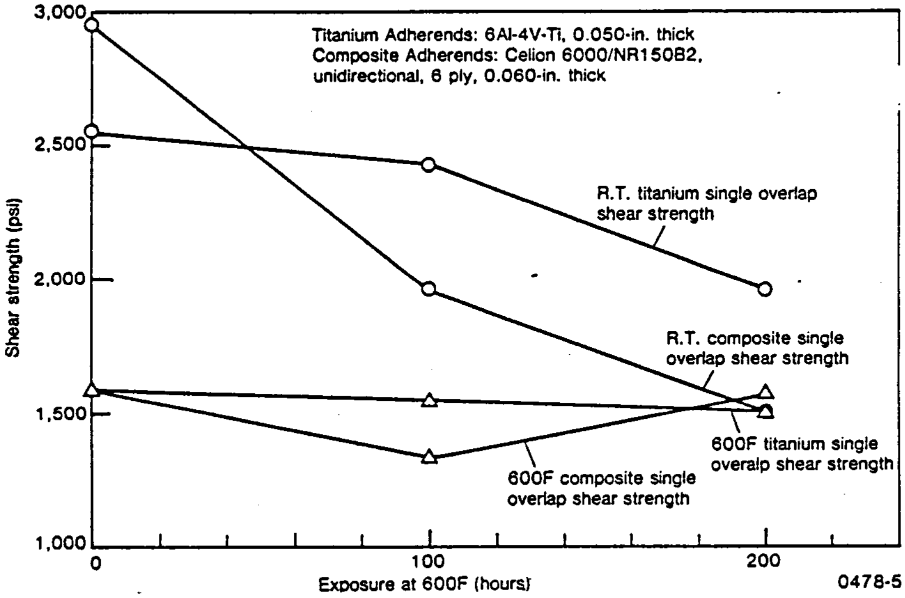


Figure 80. Effect of 316C(600F) Exposure on the Shear Strength of NR-150B2 Evaluated Using the Single Overlap Shear Test.

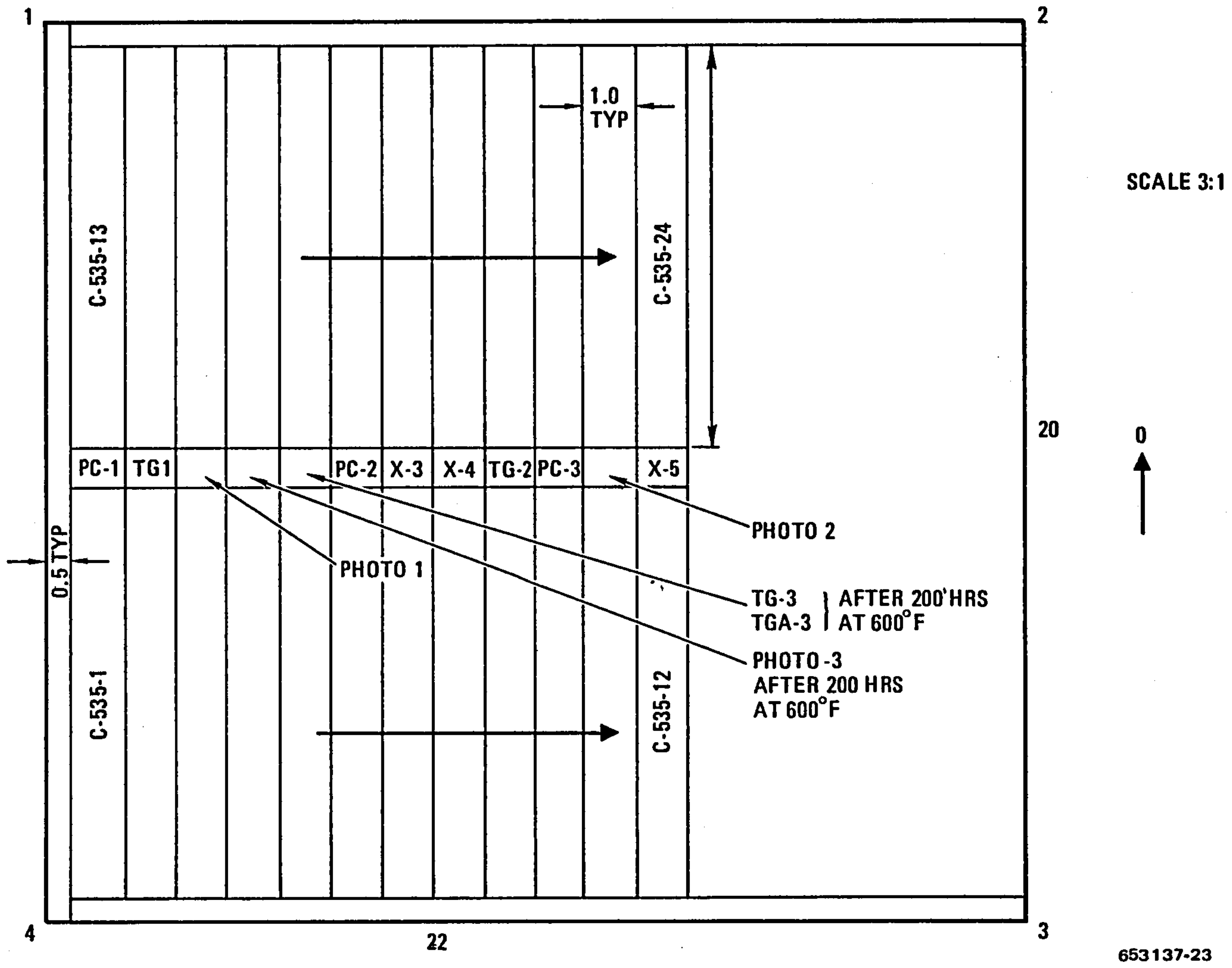


Figure 81. Cutting Plan for Test Laminate #1

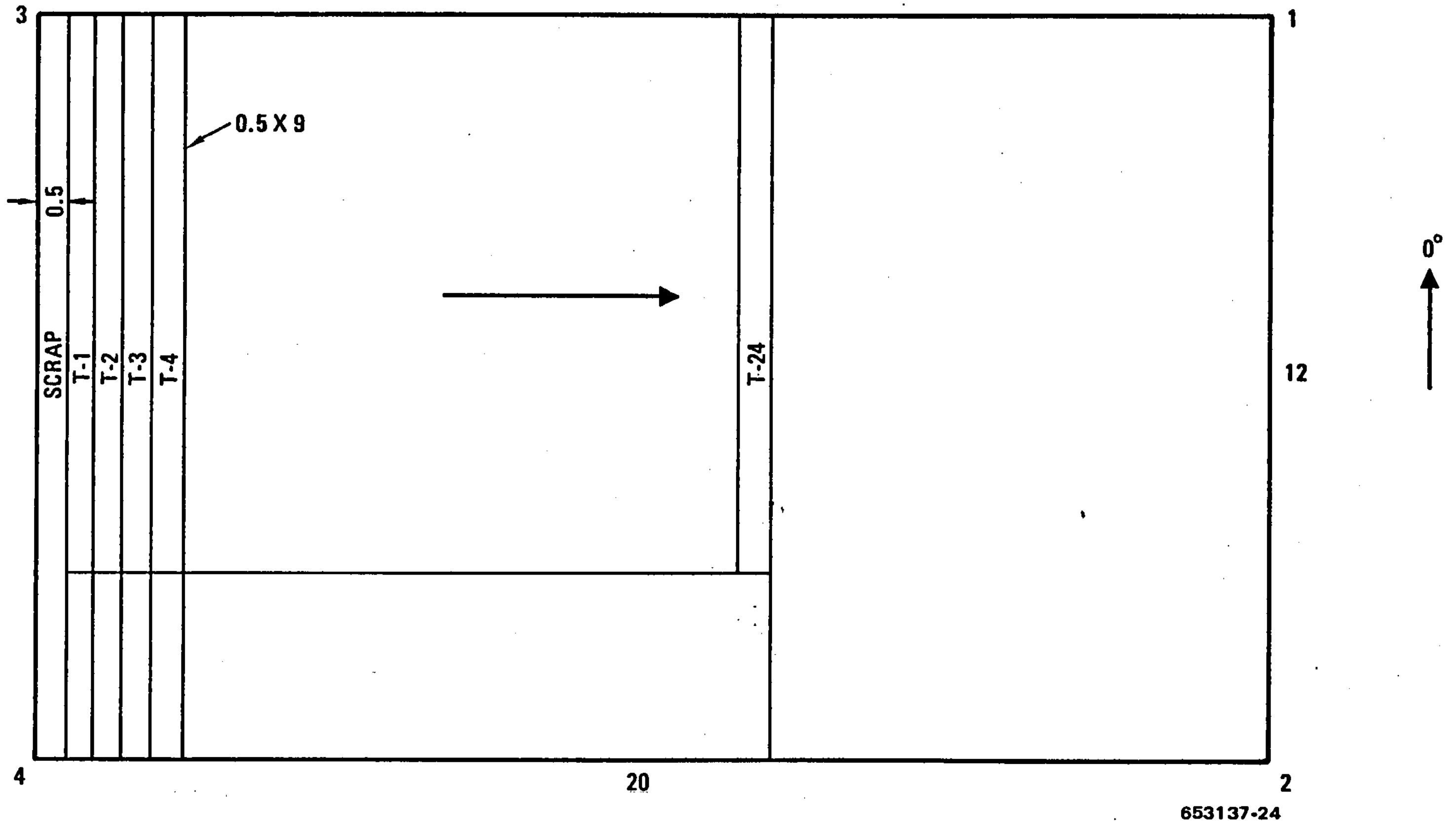


Figure 82. Cutting Plan for Test Laminate #2

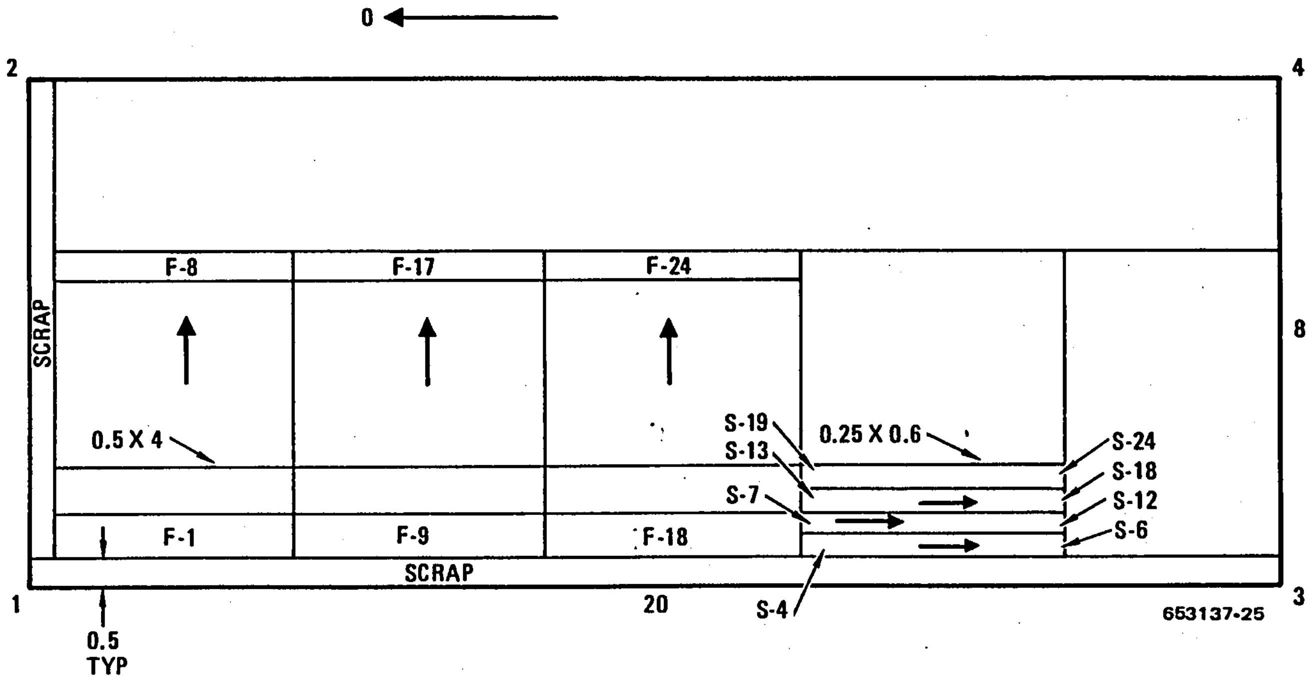


Figure 83. Cutting Plan for Test Laminate #3

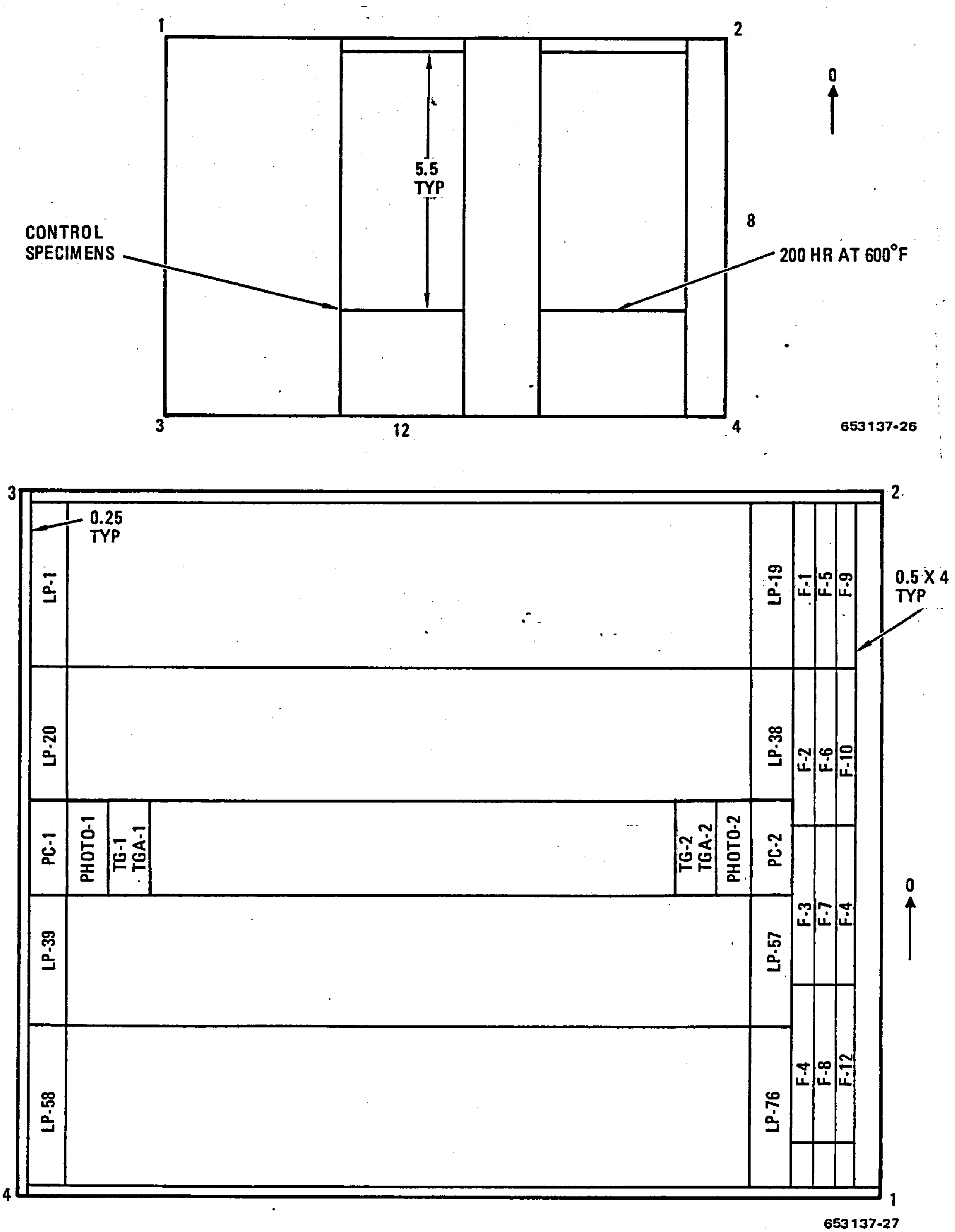


Figure 84. Cutting Plan for Test Laminate #4

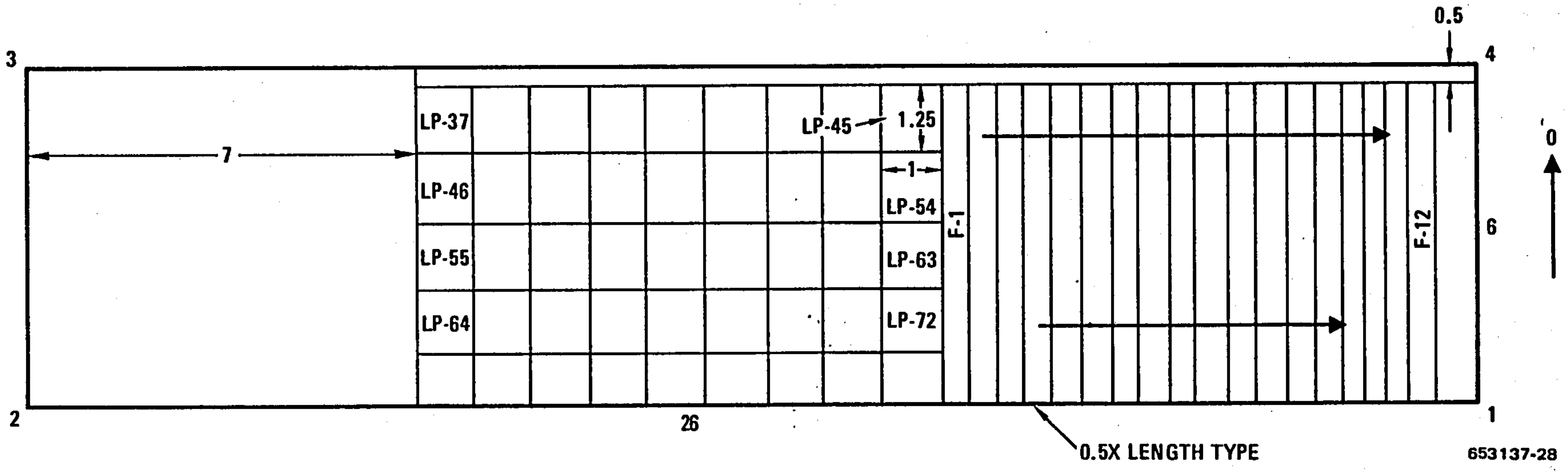
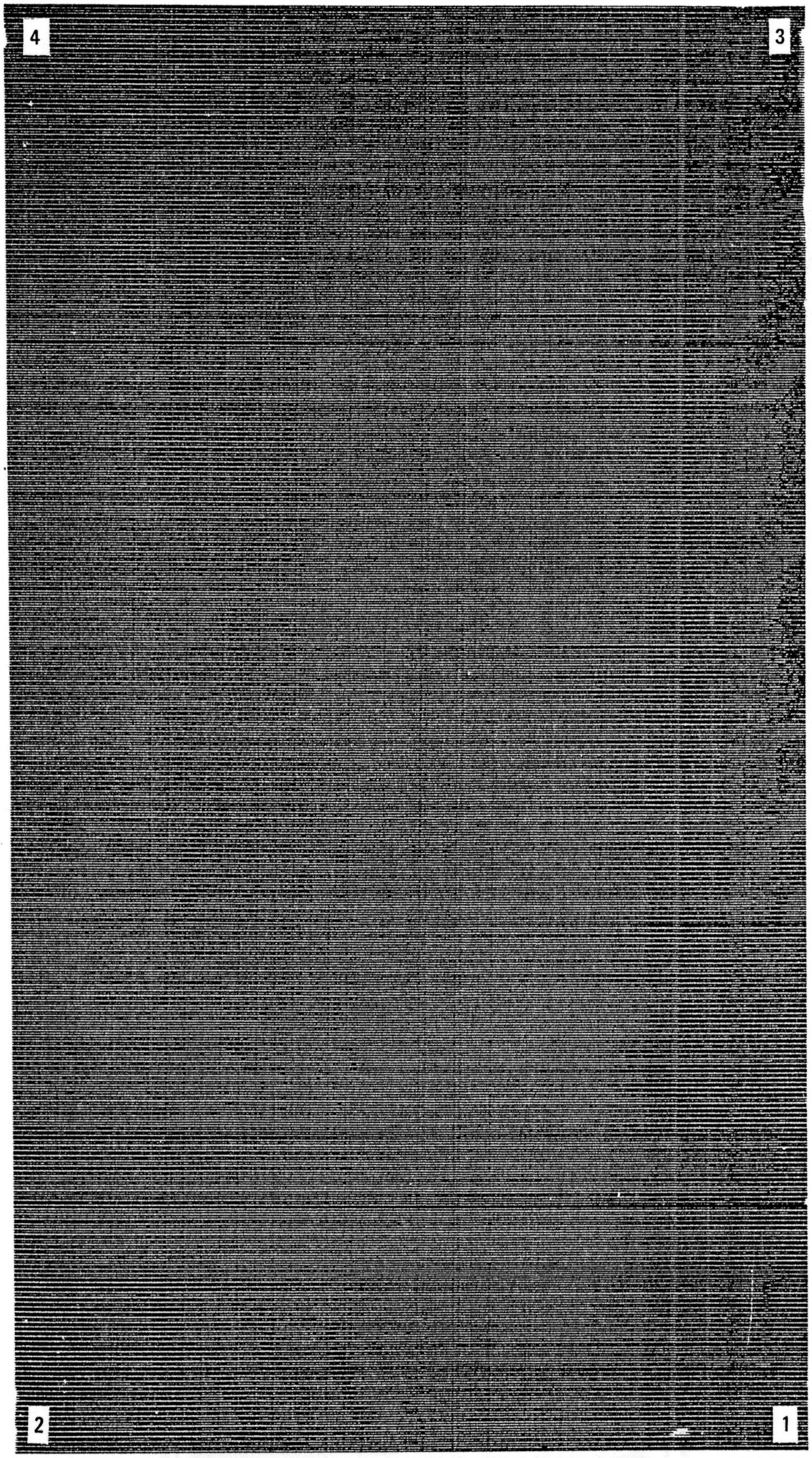


Figure 85. Cutting Plan for Test Laminate #5



653137-1

Figure . 86. C-Scan of Laminate C-532 $(0)_8$, 5 MHz at a Gain of 2

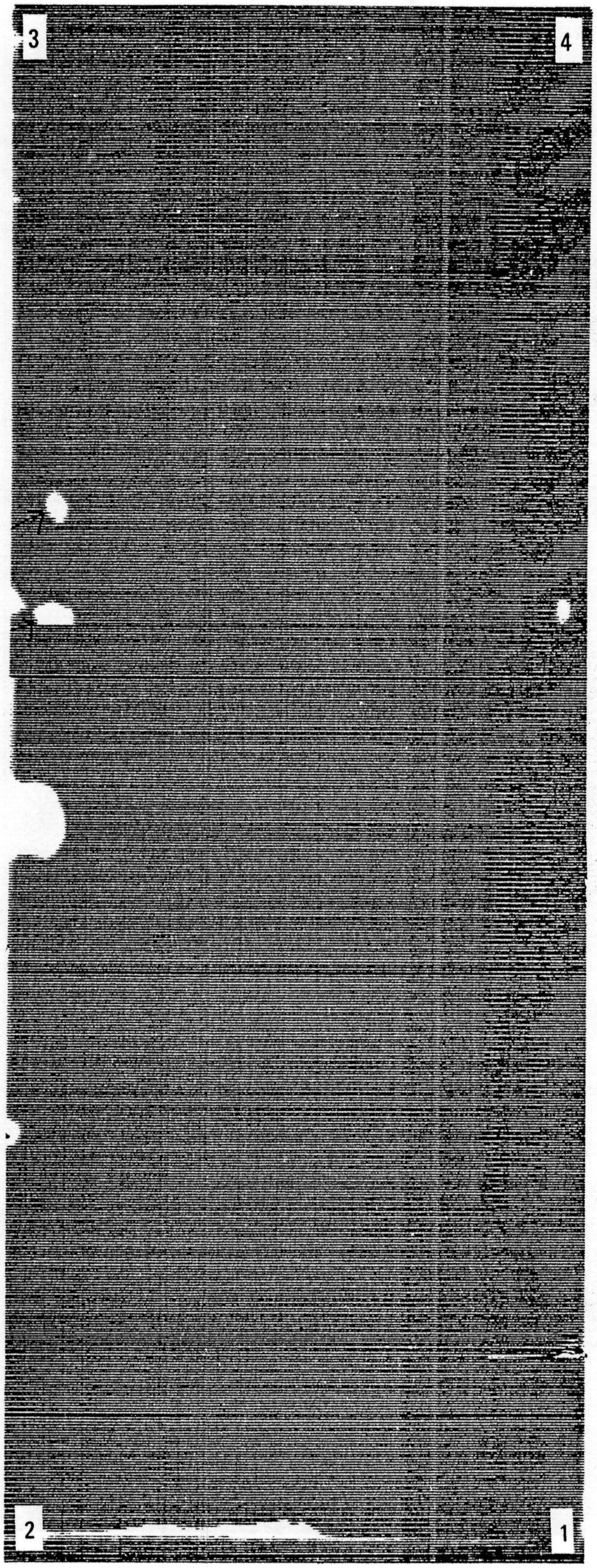


Figure 87. C-Scan of Laminate C-533 $(0)_{12}$, 5 MHz at a Gain of 2

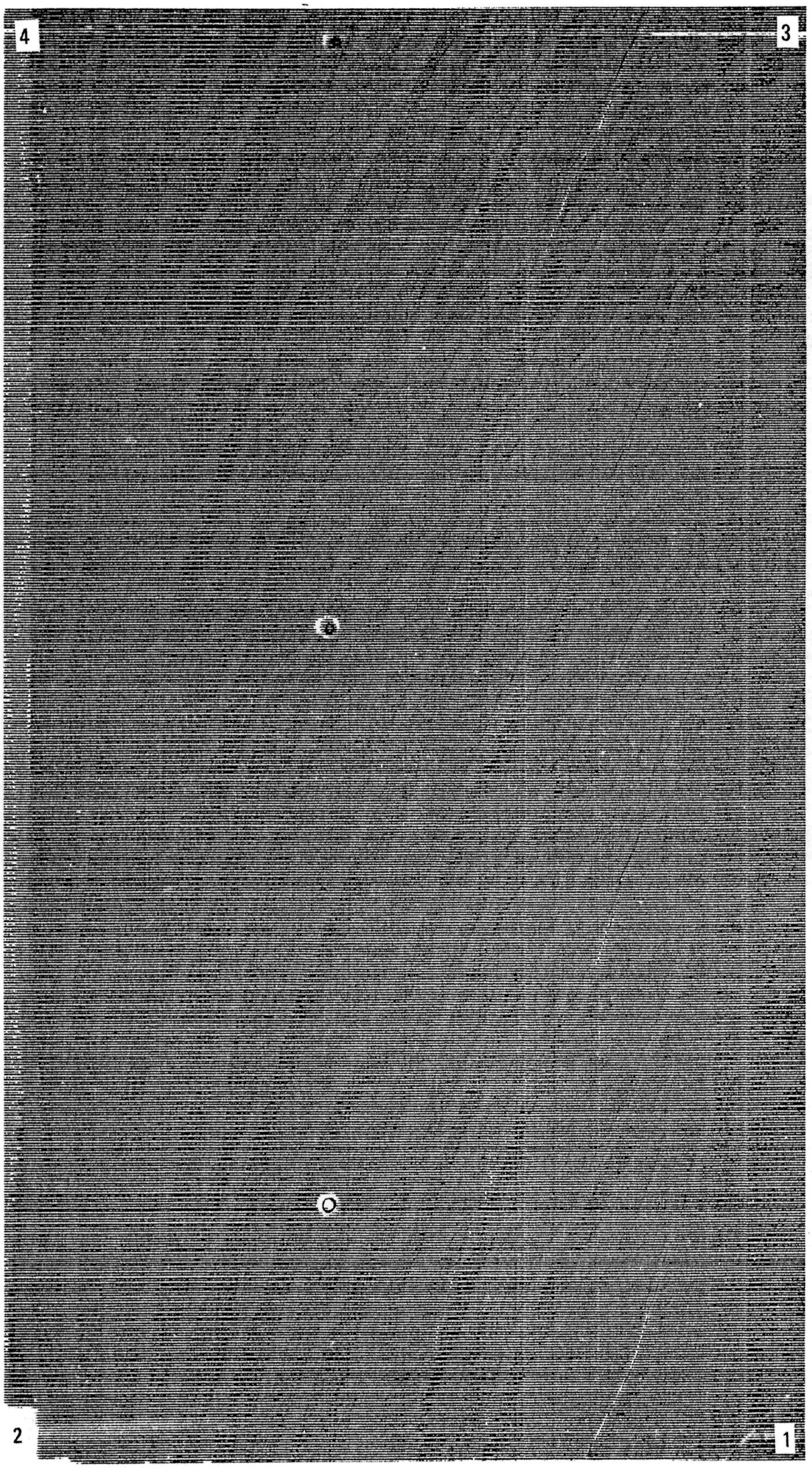


Figure 88. C-Scan Laminate C-535 (0, 45, 135, 90) 5 MHz at a Gain of 1

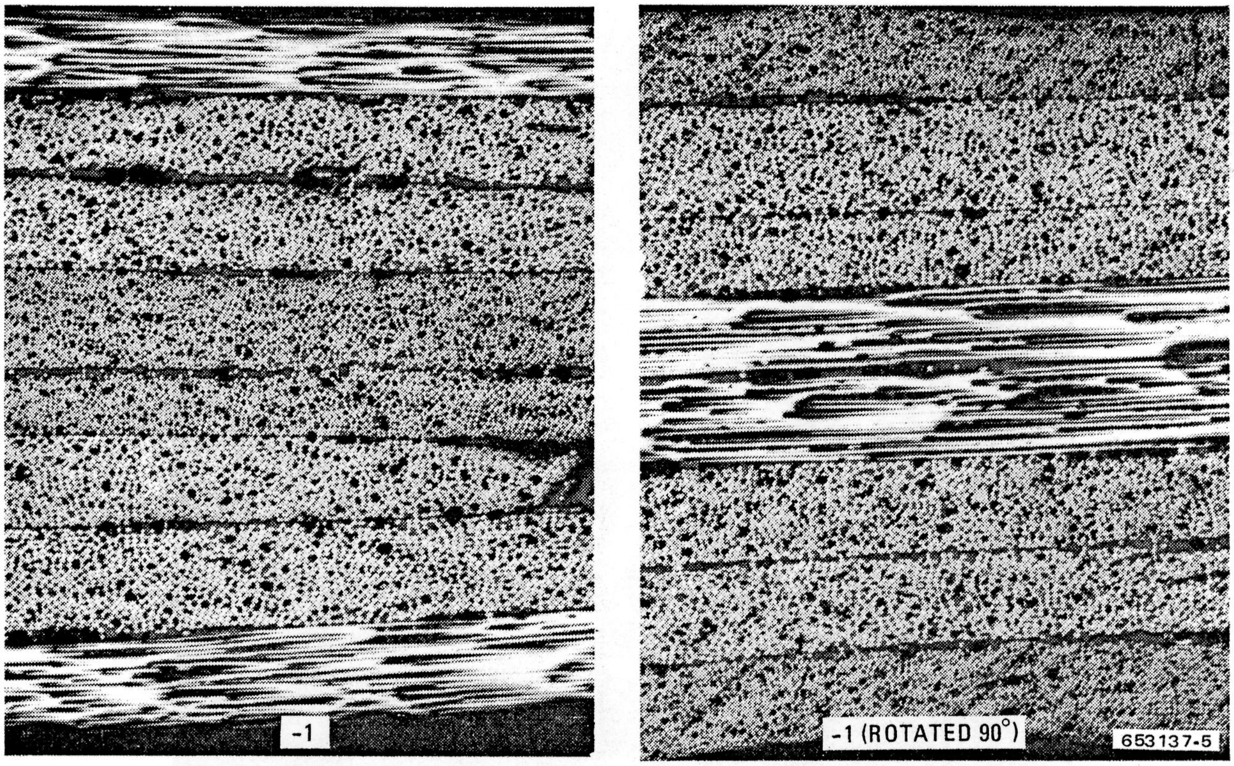


Figure 89. Photomicrograph of Laminate C-536 $(0,45,135,90)_S$, Mounts 273 and 276 (100×)

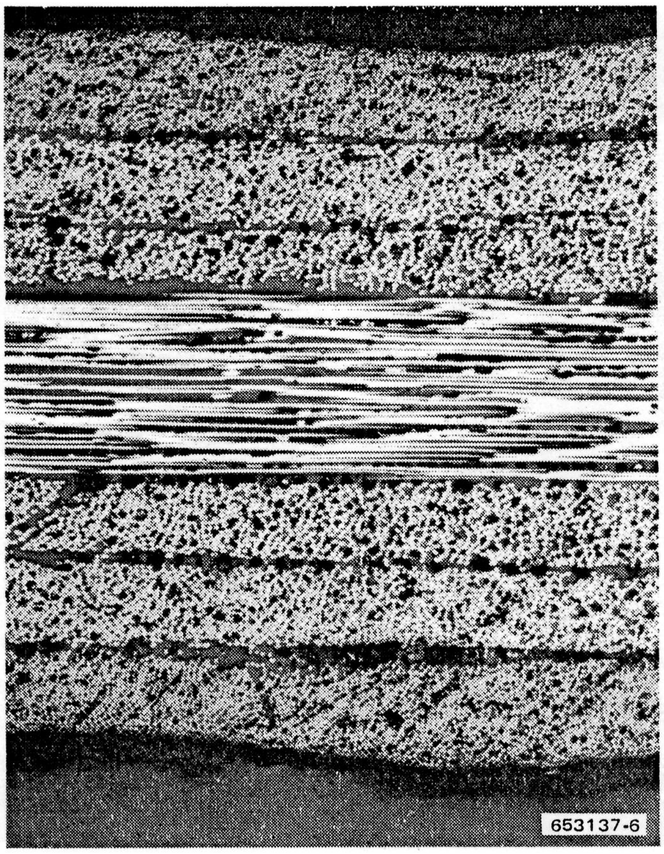


Figure 90. Photomicrograph of Laminate (C-535 $(0,45,135,90)_S$, Mount 273 (100×)

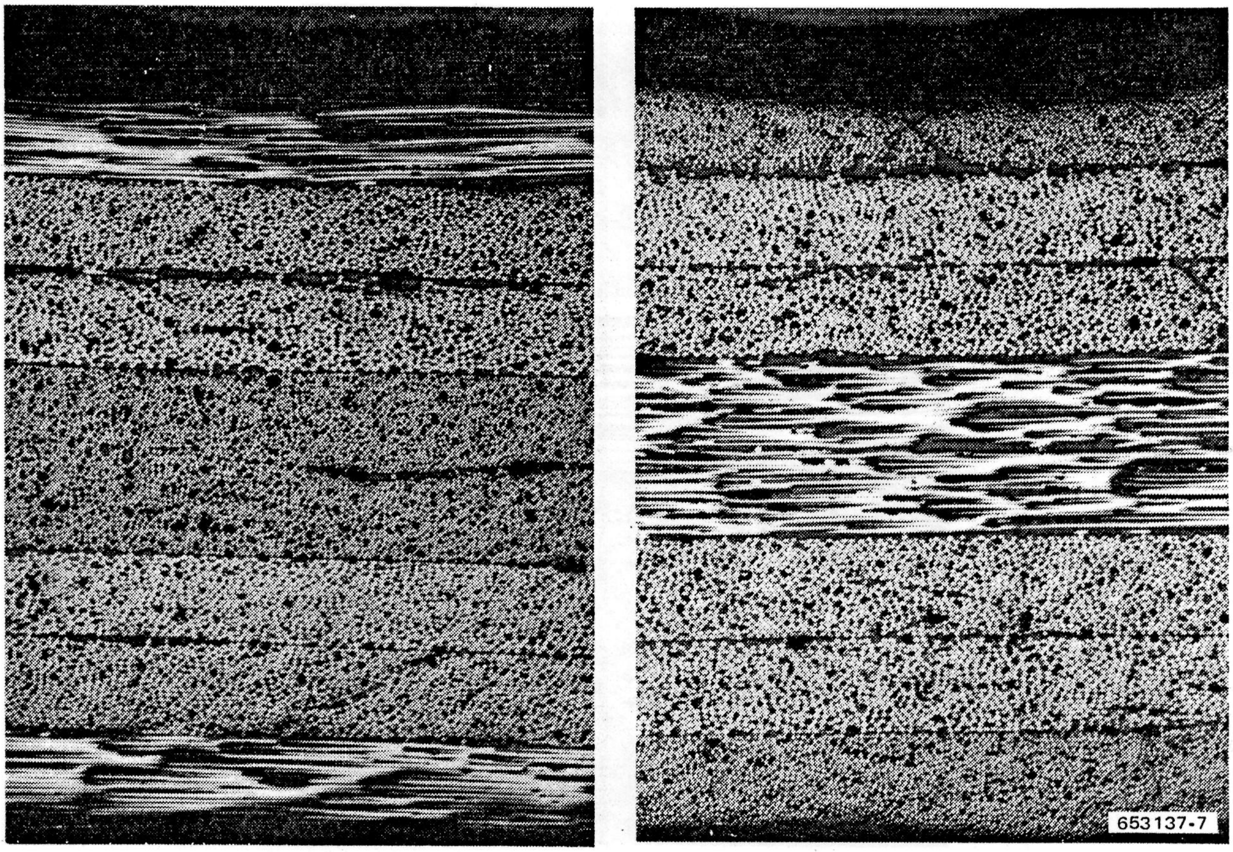


Figure 91. Photomicrograph of Laminate C-535 $(0, 45, 90, 135)_S$, Photo-3 After 200 Hours at 316C (600F) Mount 291 (100X)

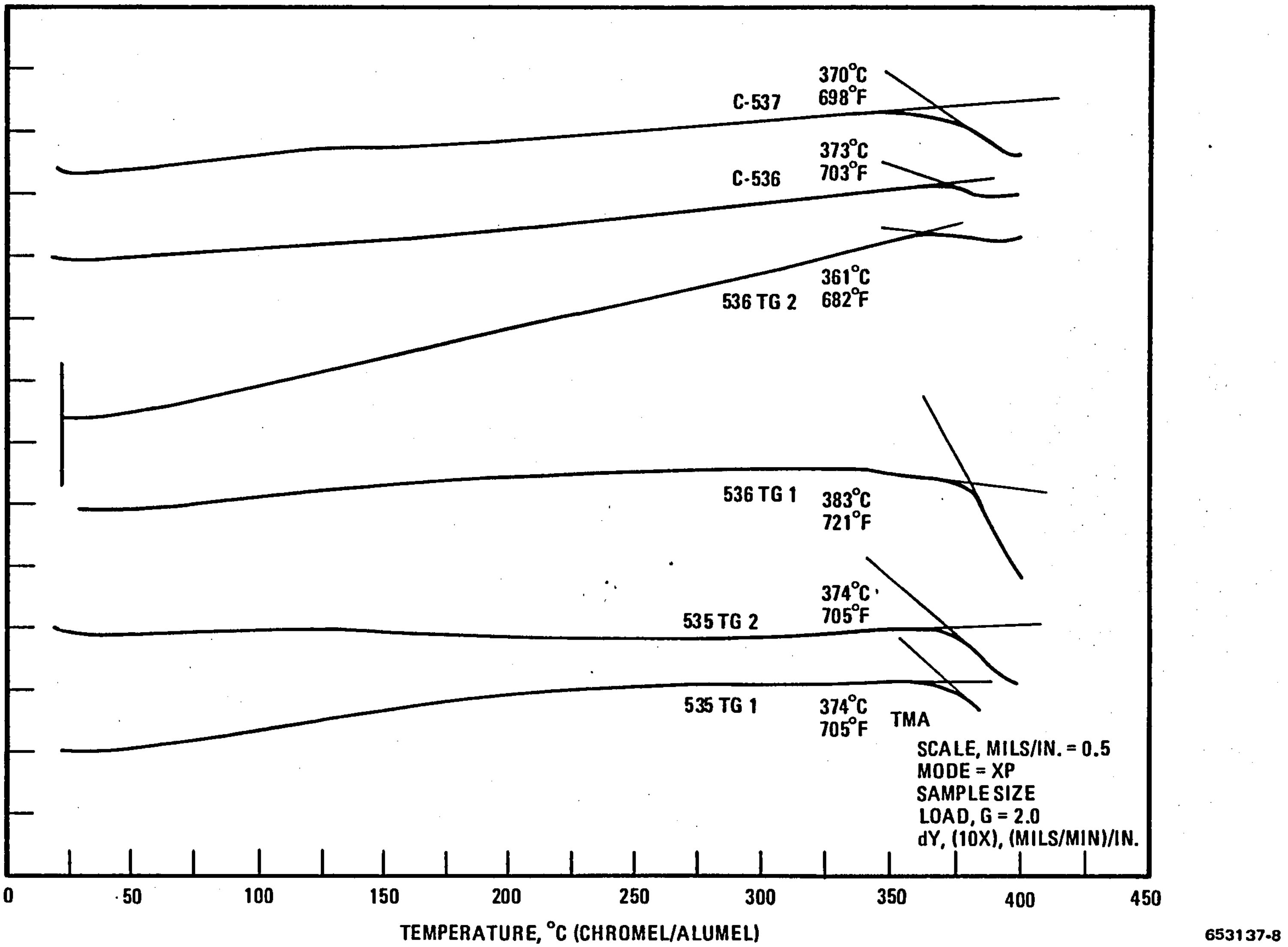


Figure 92. NR-150B2-S5X Postcured Laminate Tg

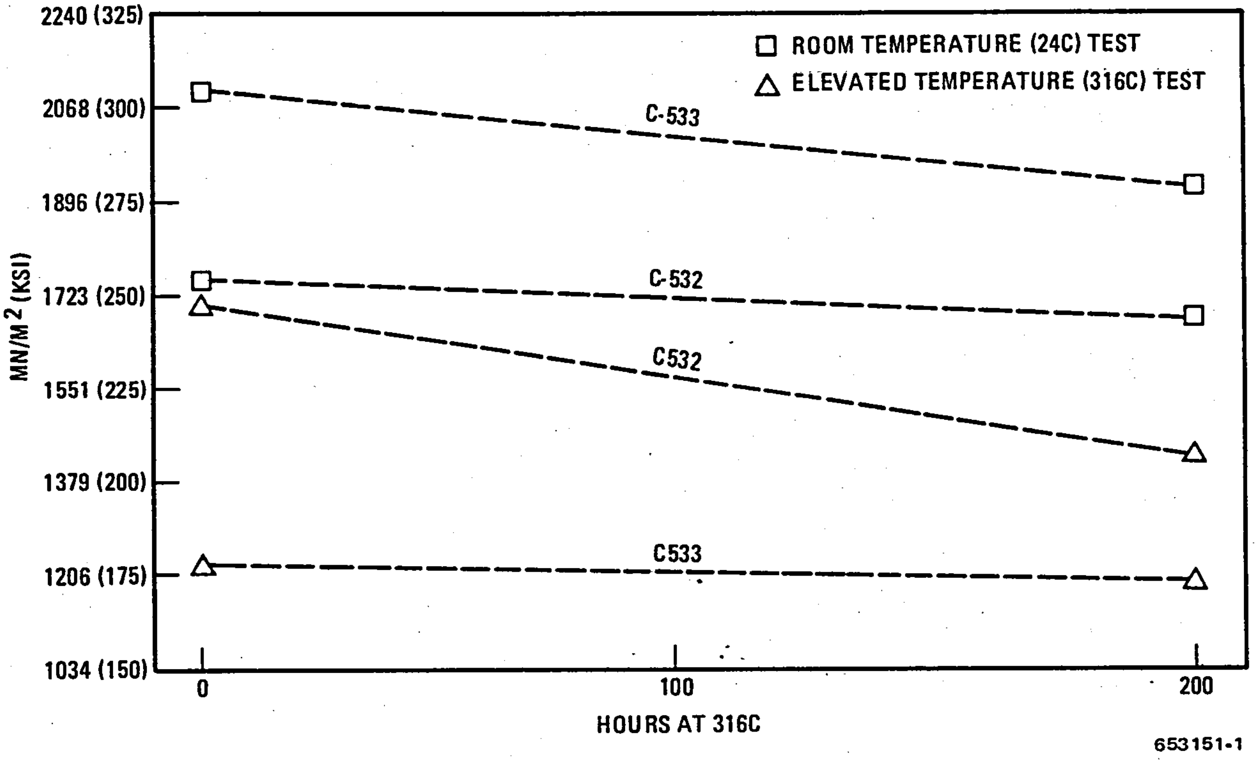
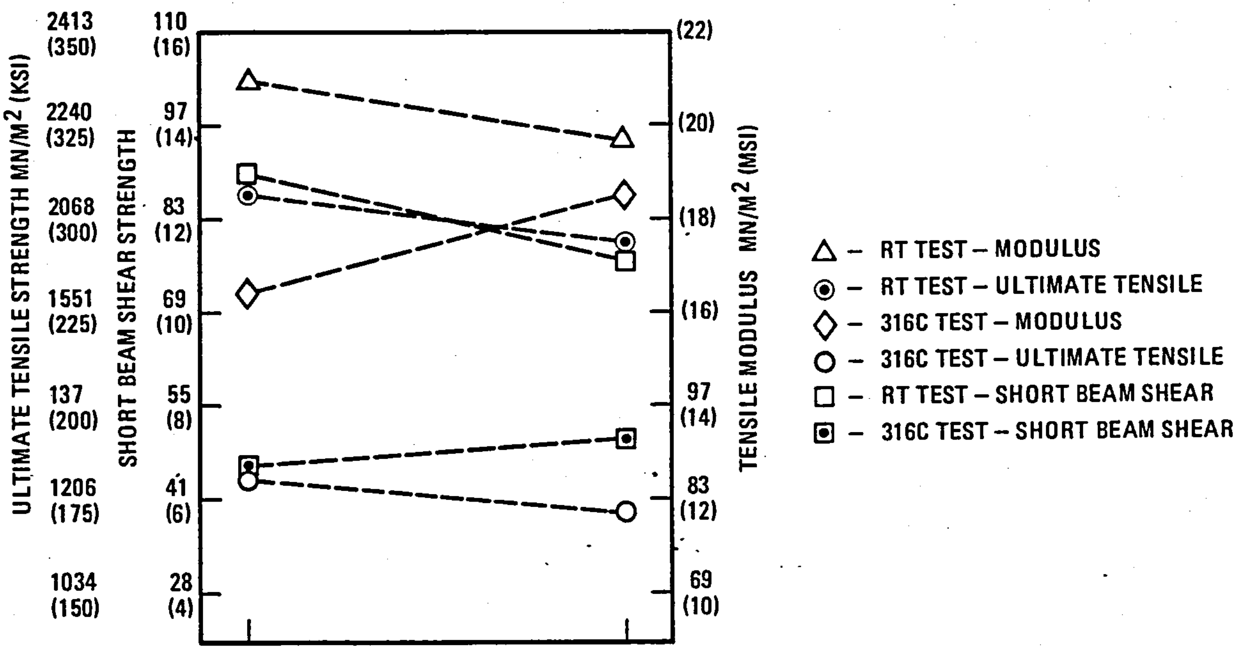


Figure 93. Tensile Properties of Laminates C-532 $(O)_8$, C-533 $(O)_{12}$ and C-535 $(O, 45, 135, 90)_S$



653151-2

Figure 94. Mechanical Properties of Laminate C-533 $(O)_{12}$ as a Function of Time at 316C (600F)

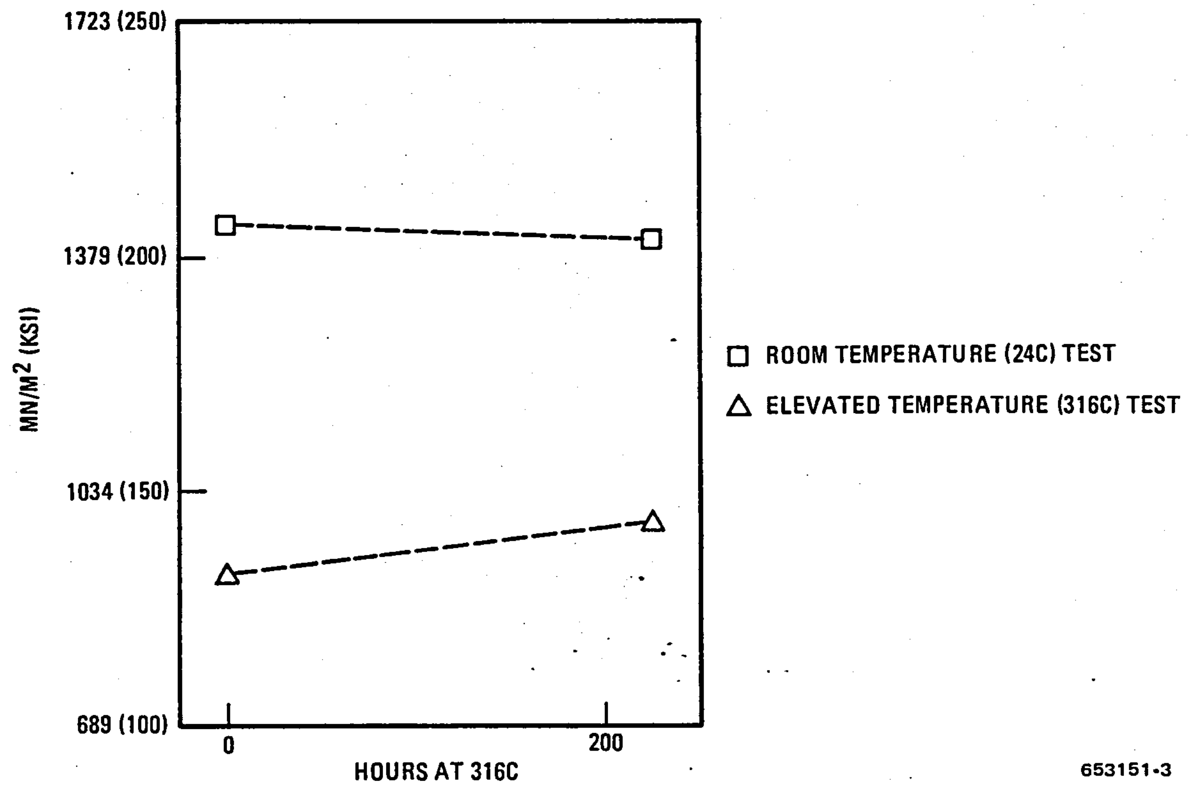


Figure 95. Compression Properties of Laminate C-538 $(O)_{24}$ as a Function of Time at 316C (600°F)

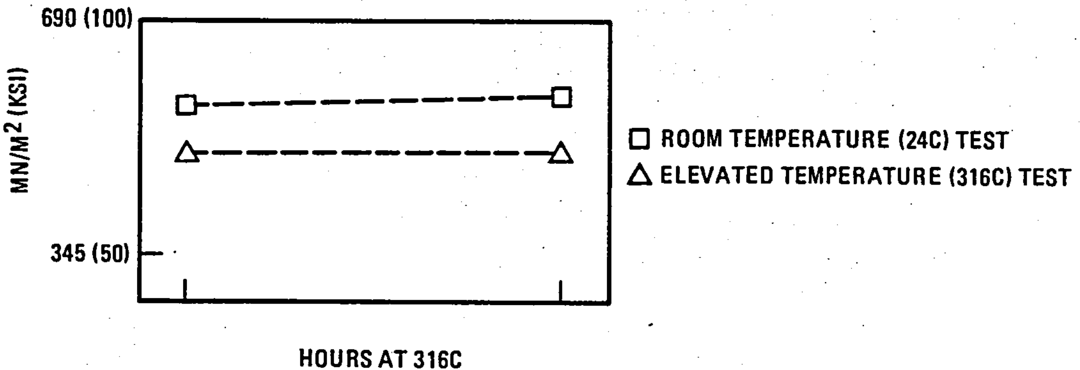


Figure 96. Tensile Properties of Laminate C-535 as a Function of Time at 316C (600°F)

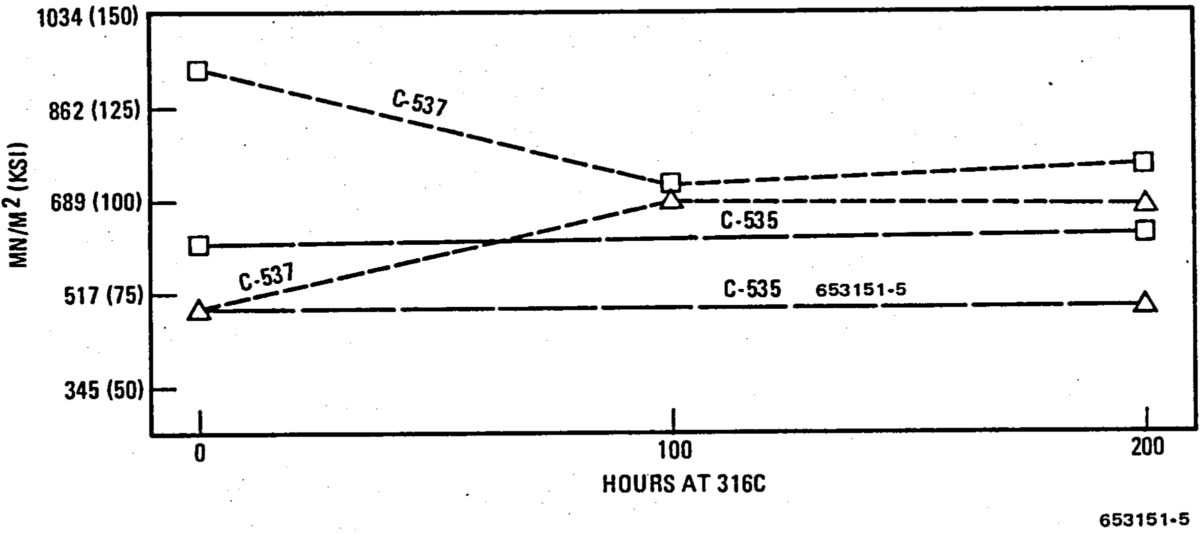
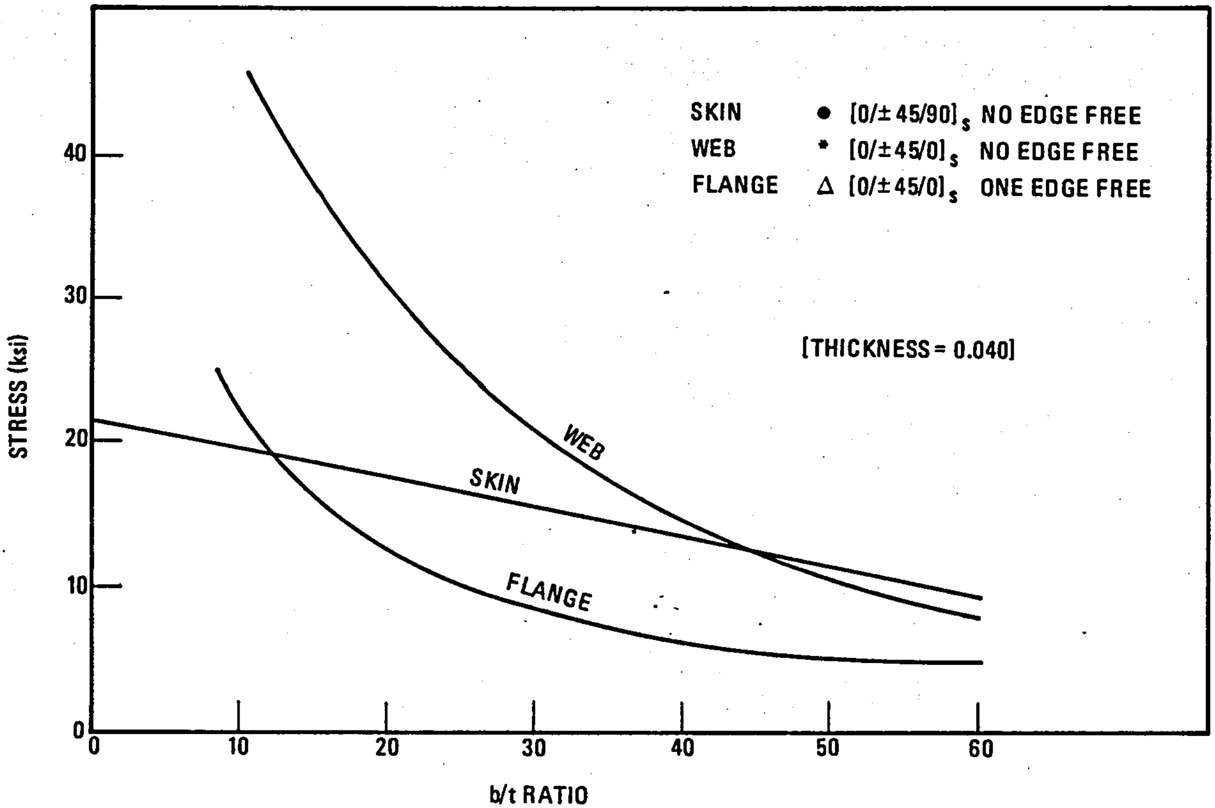


Figure 97.Flexural Properties of C-536 $(O)_{12}$ and C-537 $(0, 45, 135, 90)_{2S}$ as a Function of Time at 316C (600°F)



653137-19

Figure 98. Crippling Test Results

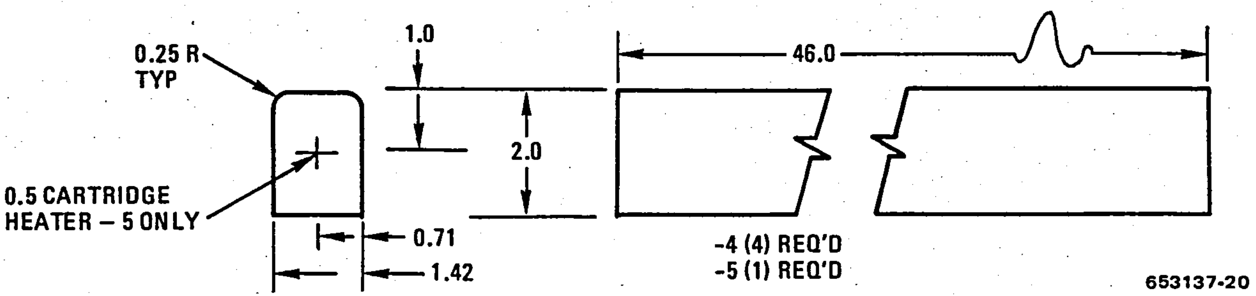


Figure 99. "I" Configuration

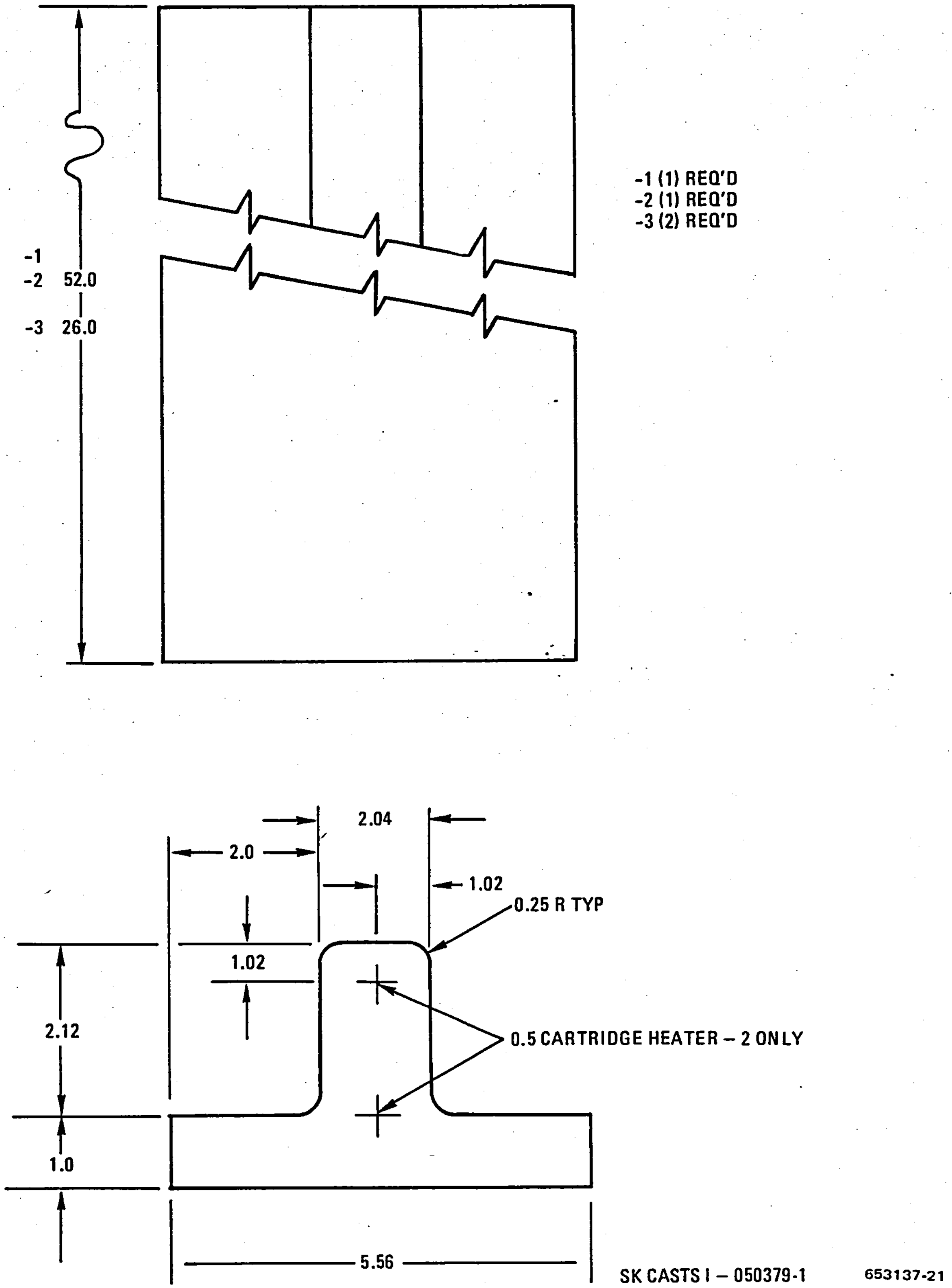


Figure 10a Hat Configuration

## TABLE 1.

## WEIGHT LOSS (%) FOR GRAPHITE FIBERS & LAMINATES (NR-150B2) AT 316C (600F)

| Specimen | Time, (hr) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 24 | 96 | 168 | 240 | 312 | 408 | 504 | 696 | 912 | 1008 |
| 1. Modmor II Fiber (Lot C266/12) | 0.11 | 0.20 | 0.30 | 0.41 | 0.47 | 0.64 | 0.76 | 1.11 | 1.37 | 1.57 |
| 2. HTS Fiber (Lot 76-8 high heat treat) | 0.10 | 0.45 | 0.95 | 1.55 | 2.13 | 3.17 | 4.02 | 6.92 | 10.37 | 12.52 |
| 3. HTS-2 Fiber (Lot 80-2, Low Sodium) | 0.23 | -- | 0.26 | 0.34 | 0.40 | 0.53 | 0.60 | 0.83 | 1.04 | 1.22 |
| 4. HTS Fiber (Lot 54-4, Low heat treat) | 4.57 | 15.51 | 25.18 | 37.99 | 47.95 | 59.47 | 68.48 | 87.25 | 93.23 | 95.93 |
| 5. Celion 6000 Fiber (Lot HTA-7-6411) | 0.27 | 0.61 | 1.59 | 2.96 | 3.45 | 5.75 | 8.42 | 14.86 | 21.09 | 29.21 |
| 6. HMS Fiber (Lot 55-1) | 0.26 | 3.07 | 4.26 | 5.49 | 6.36 | 7.52 | 8.16 | 10.52 | 12.41 | 13.67 |
| 7. Modmor II/NR-150B2 Laminate | 0.64 | 80 | 0.99 | 1.16 | 1.23 | 1.39 | 1.55 | 2.00 | 2.52 | 3.00 |
| 8. Fortifil 5/NR-150B2 Laminate | 0.63 | 80 | 0.97 | 1.11 | 1.16 | 1.30 | 1.41 | 1.70 | 1.95 | 2.18 |
| 9. Celion 6000/NR-150B2 Laminate | 0.55 | 0.54 | 0.93 | 1.30 | 1.71 | 2.49 | 3.57 | 7.16 | 10.33 | 14.71 |
| 10. HMS/NR-150B2 Laminate Fiber Lot 55-3) | 0.61 | 0.76 | 0.94 | 1.11 | 1.23 | 1.41 | 1.52 | 1.94 | 2.30 | 2.58 |
| 11. HTS/NR-150B2 Laminate Fiber Lot 54-4) | 3.43 | 4.14 | 5.04 | 6.92 | 9.08 | 12.52 | 16.12 | 29.84 | -- | -- |

Note: (1) Fiber data based on average for 2~1.5gm specimen.

(2) Laminate data based on average for 3 flexural specimens.

## TABLE 2

## MODMOR II/NR-150B2 COMPOSITE PROPERTIES (FIBERITE LOT 5D-76)

| | | 1379 kN/m$^2$ (200 psi) 371C (700F) POSTCURE (1 hr) | 6895 kN/m$^2$ (1000 psi) 371C (700F) POSTCURE (1 hr) |
|---|---|---|---|
| Flexural Strength, MN/m$^2$ (ksi) | | | |
| | RT | 1020 (148) | 1048 (152) |
| | 316C (600F) | 538 (78) | 621 (90) |
| Short Beam Shear St., MN/m$^2$ (ksi) | | | |
| | RT | 55.8 (8.1) | 55.8 (8.1) |
| | 316C (600F) | — | 27.6 (4.0) |
| Specific Gravity | | 1.43 | 1.48 |
| % Fiber, Wt. | | 58.6 | 63.6 |
| % Fiber, Vol. | | 49.3 | 53.5 |
| Thickness, (12 Ply-0$^\circ$) cm (in.) | | 0.188 (0.074) | — |
| Void Content, % | | 7.0 | 6.1 |

Prepreg Characteristics: Resin content, Wt. % - 43:1; Volatile content, Wt. % - 11.8; Fiber content, Wt. % 42.3

Comments: Low flow, low fiber content resulted in high resin content.

## TABLE 3.

## SHORT BEAM SHEAR STRENGTH FOR MODMOR II/NR150B2 COMPOSITE AFTER 696 HRS. @ 316C (600F)

| Conditioning | Test Temperature, C(F) | Short Beam Shear Strength, $MN/m^2$ (psi) |
|---|---|---|
| None | RT | 55.85 (8100) |
| | 316C (600F) | 27.6 (4000) |
| 696 Hrs. @ 316C (600F) | RT | 48.3 (7000) |
| | 316C (600F) | 25.5 (3700) |

Notes:

(1) Low initial properties due to 53.5% fiber volume and 6.1% void.
(2) 86% and 93% strength retention for RT and 316C (600F) respectively after thermal aging.
(3) 2% weight loss during thermal aging.

## TABLE 4

## PROPERTIES FOR CELION 3000 AND 6000 GRAPHITE FIBER(1)

| | Celion 3000 | Celion 6000 |
|---|---|---|
| Tensile Strength, $MN/m^2$ (ksi) | 2861 (415) | 2916 (423) |
| Tensile Modulus, $GN/m^2$ (msi) | 235. (34.1) | 228. (33.1) |
| Ultimate Elongation, % | 1.2 | 1.3 |
| Filament diameter, microns | 7 | 7 |
| Filament Count | 3000 | 6000 |
| Specific Gravity | 1.75 | 1.75 |

(1) Celanese data

## TABLE 5.

## PROPERTIES FOR NR-150B2 POLYIMIDE RESIN COMPOSITES BASED ON CELION 3000 AND CELION 6000 GRAPHITE FIBERS

| | | Celion 3000 | Celion 6000 |
|---|---|---|---|
| Flexural Strength, MN/$m^2$ (ksi) | | | |
| | RT | 1634 (237) | 1393 (202) |
| | 232C (450F) | 883 (128) | 820 (119) |
| | 316C (600F) | 738 (107) | 655 (95) |
| | 371C (700F) | 352 (51) | 414 (60) |
| Short Beam Shear Strength, MN/$m^2$ (ksi) | | | |
| | RT | 82 (11.9) | 67 (10.3) |
| | 232C (450F) | 50 (7.3) | 43 (6.2) |
| | 326C (600F) | 37 (5.3) | 30 (4.3) |
| | 371C (700F) | 24 (3.5) | 17 (2.4) |
| Laminate Specific Gravity | | 1.58 | 1.54 |
| Fiber Content, % wt. | | 68.3 | 67.2 |
| Fiber Content, % vol. | | 63.3 | 62.1 |
| Cured Ply Thickness, mm (in.) | | 0.117 (0.0046) | 0.114 (0.0045) |
| Glass Transition Temp., C(F) | | 354 (669) | 352 (666) |
| Calculated Void, % Vol. - | | 2.5 | 4.1 |
| Fiber Density, gm/cc - | | 1.75 | 1.75 |

## TABLE 6.

## PREPREG & RESIN DATA

| Fiber Type | Modmor II | Celion 3000 | Celion 3000 | Modmor II |
|---|---|---|---|---|
| Prepreg Lot No. | 6B-74 | 6D-26 | 6D-26 | 6C-55 |
| Resin Lot No. | E14224-34 | E14224-76 +<br>E14224-78 | → | |
| Solvent(s) | NMP | NMP | NMP | NMP |
| DuPont Resin Solids (%) | 55.3 | 60.4 | 60.4 | 60.4 |
| Roll No. | 2 | 1 | 2 | 1 |
| Mfg. Date | 5/20/77 | 8/23/77 | 8/23/77 | 9/1/77 |
| Delivery Date | 5/26/77 | 9/1/77 | 9/1/77 | 9/9/77 |
| Resin Content, % | | | | |
| Fiberite | 44.6 | 28.1 | 27.4 | 33.2 |
| Convair | 36.9 | 32.5 | 34.0 | 32.9 |
| Flow, % | | | | |
| Fiberite | ND | 25.2 | 25.8 | 24.1 |
| Convair | 22.0 | 22.1 | 22.4 | 21.6 |
| Volatiles, % | | | | |
| Fiberite | 20.2 | 16.1 | 16.7 | 14.1 |
| Convair | 16.6 | 12.2 | 12.1 | 14.0 |
| Roll Weight, kg (lb) | 2.13(4.7) | 2.49(5.5) | 2.49(5.5) | 2.58(5.7) |
| Width, cm (in.) | 7.62(3.0) | 7.62(3.0) | 7.62(3.0) | 7.62(3.0) |
| Drape | OK | OK | OK | OK |
| Tack | OK | OK | OK | OK |
| Gel Time (min.) | | | | |
| Fiberite at 204C (400F) | - | 1.5 | 1.3 | - |
| Convair at 204C (400F) | 0.6 | 1.0 | - | 0.9 |
| Fiberite at 177C (350F) | 5.1 | 4.4 | 3.5 | 3.8 |
| Convair at 177C (350F) | 2.5 | 3.0 | - | 2.9 |
| Fiberite at 149C (300F) | - | 11.4 | 11.0 | - |
| Convair at 149C (300F) | 7.5 | 4.2 | - | 4.5 |
| Process Gel Temp., C (F) | 140.6(285) | 171(340) | 171(340) | 171(340) |
| Areal Weight, gm/ft$^2$ | 21.44 | 22.58 | 25.83 | 25.0 |

Table 7. Analysis of Prepreg Based on "Celion" and NR-150 from General Dynamics

| Du Pont Notebook Reference | NR-150 Binder | Prepregger | Lot No. | % Vol. Rem. | % Cured Resin Solids | Composition, % of Theoretical |  | % $H_2O$ |
|---|---|---|---|---|---|---|---|---|
|  |  |  |  |  |  | Acid | Ester |  |
| E17307-136-A | B2 S5X | Fiberite | C8-206 | 16 | 36 | 51 | - | 0.95, 1.02 |
| B | B2 S5X | Fiberite | C8-406 | 13 | 39 | 70 | - | 0.85 |
| C | B2 | Fiberite | C8-469 | 12 | 38 | 63 | 17 | 1.15 |
| D | B2 S5X | Fiberite | C8-533 | 15 | 36 | 59 | - | 1.05 |
| E | B2 S5X | U.S. Poly. | 2W-4354 | 12 | 30 | 42 | - | 0.56, 0.50 |
| F | B2 S5X | U.S. Poly. | 2W-4366 | 12 | 36 | 81 | - | 0.78 |
| G | B2 S5X | U.S. Poly. | 2W-4367 | 13 | 35 | 81 | - | 0.59 |
| H | B2 S5X | U.S. Poly. | USP-1 | 17 | 35 | 44 | - | 0.32 |

Table 8. NR150B2/NR150B2-S5X Analysis

| Sample No. | Type | Resin + Vols. % | Vol. % | Cured Resin % | Dissolved Solids % | Dissolved Acid % | 1st Eq. Pt. |  | % Theory (4.16 mg/g) | 2nd Eq. Pt. |  | % Theory (8.33 mg/g) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  |  |  |  | Uncorr. | Corr. |  | Uncorr. | Corr |  |
| 6 FTA | Solid | - | - | - | 100 | 100 | 3.95 | 3.95 | 95.0 | - | - | - |
| 6 FTA & ppd | Solid | - | - | - | 100 | 81.6 | 3.12 | 3.82 | 91.8 | 5.98 | 7.33 | 88.0 |
| 64B28658 | Resin | 100 | 47.4* | 52.6 | 59.9 | 8.9 | 1.42 | 2.90 | 69.7 | - | - | - |
| E15588-62 | Resin | 100 | 47.3* | 52.7 | 60.0 | 49.0 | 1.35 | 2.76 | 66.3 | 2.80 | 5.71 | 68.5 |
| C8-206 | Resin | 100 | 47.2* | 52.8 | 60.1 | 49.1 | 1.16 | 2.37 | 57.0 | 2.68 | 5.47 | 65.7 |
| C8-206 | Film | 100 | 33.0* | 67.0 | 76.1 | 62.1 | 1.26 | 2.03 | 48.8 | 2.81 | 4.52 | 54.3 |
| 2W436 | Prepreg | 41.9* | 12.0* | 29.9 | 34.0 | 27.7 | .830 | 3.00 | 72.1 | 1.85 | 6.70 | 80.0 |
| C8-533 | Prepreg | 51.0* | 16.0* | 35.0 | 39,8 | 32.7 | .648 | 1.98 | 47.7 | 1.32 | 4.04 | 48.0 |
| C8-406 | Prepreg | 50.1* | 13.5* | 36.6 | 41.6 | 33.9 | .746 | 2.20 | 52.9 | - | - | - |

*Measured quantities

## TABLE 9.

## PROPERTIES FOR INITIAL NR-150B2/MODMOR II LAMINATE BASED ON ALL NMP RESIN SOLUTION

| Flex. Strength $MN/m^2$ (ksi) | |
|---|---|
| RT | 506 (73.4) |
| 316C (600F) | 239 (34.6) |
| **Short Beam Shear Strength $MN/m^2$ (ksi)** | |
| RT | 23 (3.4) |
| 316C (600F) | — |

NOTES:

(1) First laminate (12 ply, 0°, 15.2 cm (6 in.) × 15.2 cm (6 in.) from first batch of prepreg based on all NMP.

(2) Cured in accordance with schedule discussed earlier in this section.

| | |
|---|---|
| (3) SP. GR. | 1.40 |
| (4) Fiber Vol., % | 74.8 |
| (5) Void Content, % | 14.6 |

# TABLE 10.

## PREPREG & RESIN DATA

| Fiber Type | Celion 3000 | HTS-2 |
|---|---|---|
| Prepreg Lot No. | C8-059 | C8-166 |
| Resin Lot No. 14224 | -109/-113 | -113/-61 |
| Solvent(s) | NMP | NMP |
| DuPont Resin Solids (%) | 60.0 | 60.0 |
| Roll No. | 1A, 1B | |
| Mfg. Date | 11/22/77 | 12/29/77 |
| Delivery Date | 12/1/77 | 1/5/78 |
| % Resin | | |
| Fiberite | 32.8 | 34.5 |
| Convair | 34.4 | 35.4 |
| % Flow | | |
| Fiberite | 26.0 | 25.4 |
| Convair | 25.5/26.6 | 22.2 |
| % Volatiles | | |
| Fiberite | 15.4 | 16.1 |
| Convair | 16.0/17.0 | 15.6 |
| % Fiber | | |
| Fiberite | 51.8 | 49.4 |
| Convair | 49.1 | 49.0 |
| Roll Weight, kgs (lbs) | 8.66 (19.1) | 2.27 (5) |
| Width, Cm (In.) | 15.2 (6) | 7.62 (3) |
| Drape | ok | ok |
| Tack | ok | ok |
| Gel Time (Min) | | |
| Fiberite @ 204C (400F) | 1.0 | 1.5 |
| Convair @ 204C (400F) | 0.8 | .9 |
| Fiberite @ 177C (350F) | 6.7 | 4.8 |
| Convair @ 177C (350F) | 2.0 | 2.9 |
| Fiberite @ 149C (300F) | 8.5 | 7.8 |
| Convair @ 149C (300F) | 3.2 | 4.5 |
| Areal Weight, gm/FT$^2$ | 25.1 | 22.7 |
| Comments | Surfaces Resin Rich Center of Fiber Dry | Surfaces Resin Rich Center of Fiber Dry |

## TABLE 11.

## CURE SCHEDULE SUMMARY

| Cure | Used for Laminate No. | Description |
|---|---|---|
| Standard | C-4 | See Section 2.2 |
| CR-1 | C-6 | Standard except hold of 1 hr at 66C (150F) +1 hr at 21C (250F). |
| CR-2 | C-7 | Standard + hold of 3 hr at 121C (250F) |
| CR-3 | C-8 | Standard + hold of 5 hr at 121C (250F) |
| CR-4 | C-9,<br>C-10 | No vacuum until 1 hr at 149C (300F), hold 2nd hr w/vac, raise to 185C (365F) and hold 1 hr, apply 1400 $kN/m^2$ (200 psi) heat to 204C (400F) and hold 2 hr |
| CR-4A | C-11 | CR-4, except vacuum bag pressure time at 149C (300F) of 1/2 hr |
| CR-4B | C-13 | CR-4, except 1/2 hr w/o vacuum + 1/2 hr with vacuum at 149C (300F) |
| CR-4C | C-15 | CR-4, except total time at 149C (300F) is 1/2 hr w/o vacuum; vacuum applied after 149C (300F) hold during heatup to 185C (365F). |
| CR-5 | C-12 | CR-4, except full vacuum and 1400 $kN/m^2$ (200 psi) applied after 1 hr at 149C (300F), held 2nd hr at 149C (300F) |
| CR-6 | C-16 | CR-4, except 1/2 hr w/o vacuum at 149C (300F), vacuum applied after 1 hr at 185C (365F) |
| CR-7 | C-14,<br>C-17 &<br>C-20 | CR-4, but with full vacuum from start |
| CR-8 | C-18,<br>C-19 &<br>C-21 | CR-4, but with 10-in. vacuum from start |
| CR-9 | C-22(1)<br>C-24 | CR-4, but with 5-in. vacuum from start |
| CR-10 | C-23 | CR-4, but with 5-in. vacuum from start and only 1 hr at 149C (300F) |

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(1) Faulty layup

TABLE 12.

PROPERTIES FOR MODMOR II/NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITES (12 PLY)

| Laminate No. | C-4 | C-6 | C-7 | C-8 | C-9 | CR-10 | CR-11 | CR-12 | CR-13 | CR-14 | C-15 | C-16 | C-17 | C-18 | C-19 | C-20 | C-21 | C-22 | C-23 | C-24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cure | STD | CR-1 | CR-2 | CR-3 | CR-4 | CR-4 | CR-4A | CR-5 | CR-4B | CR-7 | CR-4C | CR-6 | CR-7 | CR-8 | CR-8 | CR-7 | CR-8 | CR-9 | CR-10 | CR-9 |
| Postcure | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD | STD |
| Orientation | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0° | 0/90° | 0/90° | 0° | 0° | 0° |
| Flexural Strength $MN/m^2$ (ksi) | | | | | | | | | | | | | | | | | | | | |
| RT | 511(73) | 469(67) | 700(100) | 833(119) | 1190(170) | 1141(163) | 1085(155) | 1316(188) | 994(142) | (50) | 1057(151) | 147(21) | 350(50) | 1162(166) | 1113(159) | 238(34) | 427(61) | 602(86) | 490(70) | 714(102) |
| 232C (450F) | 357(51) | 413(59) | 462(66) | 490(70) | 966(138) | 966(138) | 917(131) | 896(128) | 763(109) | — | 665(95) | 147(21) | — | — | — | — | — | — | — | — |
| 316C (600F) | 245(35) | 301(43) | 336(48) | 406(58) | 833(119) | 945(135) | 875(125) | 784(112) | 602(86) | (38) | 595(85) | 119(17) | 259(37) | 588(84) | 665(95) | 154(22) | 252(36) | 350(50) | 378(54) | 392(56) |
| Short Beam Shear Strength $MN/m^2$ (ksi) | | | | | | | | | | | | | | | | | | | | |
| RT | 24.5(3.5) | 39.2(5.6) | 49(7.0) | 40.6(5.8) | 69.3(9.9) | 82.6(11.8) | 62.3(8.9) | 63(9.0) | 49.7(7.1) | (3.3) | 59.5(8.5) | 16.8(2.4) | 25(3.6) | 50(7.1) | 51(7.3) | 13(1.9) | 31(4.5) | 38(5.5) | 30(5.4) | 41(5.9) |
| 232C (450F) | — | 35(5.0) | 33.6(4.8) | 39.2(5.6) | 58.1(8.3) | 45.5(6.5) | 57.4(8.2) | 46.2(6.6) | 41.3(5.9) | — | 40.6(5.8) | 15.4(2.2) | — | — | — | — | — | — | — | — |
| 316C (600F) | — | 22.4(3.2) | 27.3(3.9) | 23.8(3.4) | 45.5(6.5) | 41.3(5.9) | 43.4(6.2) | 32.9(4.7) | 31.5(4.5) | (2.9) | 31.5(4.5) | 16.8(2.4) | 21(3.0) | 16(4.5) | 17(4.8) | 5(1.3) | 11(3.1) | 28(4.0) | 25(3.6) | 27(3.9) |
| Specific Gravity | 1.40 | 1.42 | 1.40 | 1.38 | 1.56 | 1.55 | 1.51 | 1.45 | 1.55 | 1.35 | 1.56 | 1.41 | 1.39 | 1.43 | 1.48 | 1.44 | 1.40 | 1.32 | 1.38 | 1.34 |
| Fiber Content, % Wgt. | 74.8 | 71.9 | 68.0 | 68.4 | 60.7 | 57.4 | 61.5 | 70.7 | 60.9 | 75.4 | 61.3 | 69.0 | 73.9 | 69.3 | 69.8 | 73.6 | 68.6 | 68.3 | 69.1 | 65.0 |
| Fiber Volume, % | 70.5 | 67.3 | 63.1 | 63.4 | 55.4 | 52.0 | 56.2 | 66.0 | 55.6 | 71.1 | 56.1 | 64.0 | 69.5 | 64.5 | 65.1 | 69.2 | 63.8 | 63.4 | 64.3 | 59.9 |
| Cured Ply Thickness, mm (mil) | 0.112(4.4) | 0.114(4.5) | 0.137(5.4) | 0.112(4.4) | 0.132(5.2) | 0.114(4.5) | 0.114(4.5) | 0.104(4.1) | 0.117(4.6) | 0.122(4.8) | 0.117(4.6) | 0.124(4.9) | 0.124(4.9) | 0.114(4.5) | 0.124(4.9) | 0.124(4.9) | 0.124(4.9) | 0.135(5.3) | 0.132(5.2) | 0.132(5.2) |
| Glass Transition Temp., C | 353 | 359 | 357 | — | 360 | 351 | 351 | 359 | 352 | 355 | 356 | 356 | 337 | 355 | 360 | — | 354 | 360 | 360 | 360 |
| Void, % | 14.6 | 13.4 | 13.5 | 14.5 | 1.8 | 1.7 | 5.1 | 10.7 | 2.7 | 17.5 | 2.1 | 13.0 | 15.1 | 14.3 | 8.8 | 11.7 | 13.1 | 18.3 | 14.4 | 16.3 |

## TABLE 13.

## PROPERTIES FOR NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITE C-28 TO C-33 (12 PLY, 0°)

| Laminate No. | C-28 | C-29 | C-30 | C-32 | C-33 |
|---|---|---|---|---|---|
| Fiber | Celion 3000 | Celion 3000 | Celion 3000 | Celion 3000 | Modmor II |
| Cure | C-4(1) | C-4(1) | C-4(1) | C-4 | C-4 |
| Postcure | STD | STD | STD | STD | STD |
| Flexural Strength MN/$m^2$ (ksi) | | | | | |
| RT | 1631 (233) | 1645 (235) | 1771 (253) | 1890 (270) | 1386 (198) |
| 316C (600F) | 763 (109) | 980 (140) | 756 (108) | 238 (134) | 714 (102) |
| Short Beam Shear Strength MN/$m^2$ (ksi) | | | | | |
| RT | 97.3 (13.9) | 90.3 (12.9) | 89.6 (12.8) | 105.7 (15.1) | 49.7 (7.1) |
| 316C (600F) | 38.5 (5.5) | 38.5 (5.5) | 39.9 (5.7) | 38.5 (5.5) | (ND) |
| Specific Gravity | 1.54 | 1.55 | 1.55 | 1.60 | 1.55 |
| Fiber Content, % Wgt. | 71.9 | 71.5 | 77.4 | 64.6 | 60.5 |
| % Volume | 67.0 | 66.6 | 73.1 | 59.2 | 56.4 |
| Cured Ply Thickness, mm (mil) | .160 (6.3) | .150 (5.9) | .145 (5.7) | .130 (5.1) | .152 (6.0) |
| Glass Transition Temp., C | 356 | 359 | 356 | 357 | 358 |
| Void, % | 6.2 | 5.5 | 6.8 | .8 | 0 |

(1) After Vacuum Bag/Oven B-Staging

## TABLE 14.

## PROPERTIES FOR NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITE C-34 TO C-39 (12 PLY, 0°)

| Laminate No. | C-34 | C-35 | C-36 | C-37 | C-38 | C-39 |
|---|---|---|---|---|---|---|
| Fiber | Celion 3000 | Modmor II | Celion 3000 | Modmor II | Celion 3000 | Modmor II |
| Cure | CR-4 | CR-4 | CR-4 | CR-4 | CR-4 | CR-4 |
| Postcure | STD | STD | None | None | STD-A | None |
| Flexural Strength MN/m$^2$ (ksi) | | | | | | |
| RT | | | | | 364-1144<br>(52-163) | |
| 316C (600F) | | | | | — | |
| Short Beam Shear Strength MN/m$^2$ (ksi) | | | | | | |
| RT | | | | | 77 (11.0) | |
| 316C (600F) | | | | | — | |
| Specific Gravity | | | | | 1.47-1.60 | |
| Fiber Content, | | | | | | |
| Wgt, % | | | | | 63.7 | |
| Volume, % | | | | | 56-59 | |
| Cured Ply Thickness, mm (in) | | | | | | |
| Initial Cure | .170 (.0067) | .173 (.0068) | .114 (.0045) | .137 (.0054) | .155 (.0061) | .121 (.0069) |
| Postcure | .149 (.0059) | — | | | .151 (.0059) | |
| Glass Transition Temp., C | 355 | 353 | | | 362 | |
| Void, % | | | | | .8-8.7 | |
| Wgt. Loss, % | | | | | | |
| Cure | 12.1 | 11.6 | 24.3 | 26.9 | 11.5 | 10.6 |
| Postcure | 6.6 | 8.5 | | | 5.2 | |
| Total | 18.7 | 20.1 | | | 16.7 | |
| Comments | No flow, Blister | No flow, Blister | Very High Flow | Very High Flow | Slight Flow, Delam | No Flow |

## TABLE 15.

## PHYSICAL PROPERTIES OF LAMINATE C-38

| Specimen No. | Fiber Volume, % | Specific Gravity | Porosity, % |
|---|---|---|---|
| 1 | 59.2 | 1.60 | 0.8 |
| 2 | 56.5 | 1.58 | 1.5 |
| 3 | 59.3 | 1.50 | 7.0 |
| 4 | 58.2 | 1.47 | 8.7 |

## TABLE 16.

## FLEXURAL STRENGTH OF LAMINATE C-38 SPECIMENS VS ULTRASONIC C-SCAN INDICATED DEFECTS

| C-Scan Evaluation | Flexural Strength, $MN/m^2$ (ksi) |
|---|---|
| Minor Porosity | 1141 (163) |
| Major Porosity | 945 (135) |
| Severe Porosity | 798 (114) |
| Possible Delamination | 364 (52) |

## TABLE 17.

## PROPERTIES FOR NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITE C-40 TO C-45 (12 PLY, 0°)

| Laminate No. | C-40 | C-41 | C-44 | C-45 |
|---|---|---|---|---|
| Fiber | Celion 3000 | Modmor II | Celion 3000 | Celion 3000 |
| Cure | CR-4 | CR-4 | CR-4D | CR-11 |
| Postcure | STD-B | — | STD-C | STD-C |
| Flexural Strength MN/m$^2$ (ksi) | | | | |
| RT | 1064 (152) | — | 714 (102) | 1379 (197) |
| 316C (600F) | 448 (64) | — | — | 721 (103) |
| Short Beam Shear Strength MN/m$^2$ (ksi) | | | | |
| RT | 73.5 (10.5) | — | 60.9 (8.7) | 58.8 (8.4) |
| 316C (600F) | 17.5 (2.5) | | — | 35 (5.0) |
| Specific Gravity | 1.55 | — | 1.53 | 1.54 |
| Fiber Content, | | | | |
| Wgt, % | 64.6 | — | 65.3 | 78.6 |
| Volume, % | 57.5 | — | 60.1 | 74.6 |
| Weight Loss, % | | | | |
| Initial cure | 5.6 | 6.9 | 6.8 | 5.2 |
| Postcure | 5.9 | — | 6.8 | 5.2 |
| Glass Transition Temp., C | 322 | — | 360 | 350 |
| Void, % | 0.6 | — | 2.1 | 5.5 |
| Wgt. Loss, % | | | | |
| Initial cure | 12.6 | 10.2 | 11.6 | 26.2 |
| Postcure | 8.0 | | 6.7 | 2.6 |
| Total | 20.6 | | 18.3 | 28.8 |
| Comments | No Flow | No Flow | No Flow | High Flow |

## TABLE 18. PROPERTIES FOR NR-150B2-S5X GRAPHITE/ POLYIMIDE COMPOSITE (LOT C8-206)

| Laminate No. | C-70 | C-71 | C-72 | C-73 | C-74 | C-75 | C-76 | C-77 | C-78 | C-79 |
|---|---|---|---|---|---|---|---|---|---|---|
| Fiber | Celion → | | | | | | | | | |
| Cure | ← 45(1) min. → | | | | ← → | | 70(1) min. | ← → | | |
| Orientation | 0 | X | 0 | X | 0 | 0 | 0 | X | 0 | X |
| No. Ply | 12 | 12 | 6 | 6 | 12 | 6 | 12 | 12 | 6 | 12 |
| Flexural Strength MN/m$^2$ (ksi) | | | | | | | | | | |
| RT | | | | | 970 (141) | 1330 (193) | 545 (79) | 890(120) | 1450 (211) | 495 (72) |
| 316C (600F) | | | | | 557 ( 81) | 915 (133) | 344 (50) | 530( 77) | 647 ( 94) | 331 (48) |
| Short Beam Shear Strength MN/m$^2$ (ksi) | | | | | | | | | | |
| 24C (75F) | | | | | 67 (9.8) | 73 (12.0) | 36 (5.2) | 65 (9.5) | 77 (11.2) | 38 (5.5) |
| 316C (600F) | | | | | 34 (4.9) | 29 ( 4.2) | 20 (2.9) | 32 (4.6) | 28 (4.0) | 18 (2.6) |
| Specific Gravity | 1.58 | 1.59 | 1.61 | 1.61 | 144/1.55 | 1.55/1.61 | 1.57/1.48 | 1.42 | 1.56 | 1.48 |
| Fiber Content, | | | | | | | | | | |
| % Wt. | | | | | 64.3 | 67.9 | 65.9 | 63.3 | 65.1 | 64.9 |
| % Volume | | | | | 59.0 | 62.9 | | | 59.9 | |
| Cure Ply Thickness, Mil/Ply | | | | | | | | | | |
| Cured | 6.72 | 6.72 | 6.19 | 6.22 | 6.58 | 6.52 | 6.49 | 6.15 | 6.69 | 6.00 |
| Postcured | | | | | 6.02 | 5.45 | 6.15 | 6.11 | 5.73 | 5.88 |
| Weight Loss, % | | | | | | | | | | |
| Cured | 17.1 | 15.0 | 22.4 | 20.9 | 11.4 | 16.3 | 11.7 | 12.2 | 16.0 | 11.8 |
| Postcured | | | | | 11.1 | 9.6 | 11.8 | 12.7 | 10.1 | 11.5 |
| TOTAL | | | | | 22.5 | 25.9 | 23.5 | 24.9 | 26.1 | 23.3 |
| REMARKS | Warp Cracks | Poor C-scan | C-scan Poor at 5MHz 1K at 2.25 | Warp Cracks | 3.5% Void | 0.2% Void | | | 3.1% Void | |

(1) Time to vacuum and pressure application

## TABLE 19. PROPERTIES FOR NR-150B2-S5X GRAPHITE/ POLYIMIDE COMPOSITE (LOT C8-206)

| Laminate No. | C-80 | C-81 | C-82 | C-83 | C84-90 | C-91 | C-92 | C-93 | C-94 |
|---|---|---|---|---|---|---|---|---|---|
| Fiber | Celion → | | | | | | | | |
| Cure | ← 97 min(1) → | | | | | ← 80 min. → | | | |
| Orientation | 0 | 0 | 0 | 0 | various | 0 | 0 | 0 | 0 |
| No. Ply | 12 | 6 | 12 | 6 | | 6 | 12 | 6 | 12 |
| Flexural Strength $MN/m^2$ (ksi) | | | | | | | | | |
| RT | | 1590 (231) | | 1630 (237) | | | | | |
| 316C (600F) | | 861 (125) | | 846 (123) | | | | | |
| Short Beam Shear Strength $MN/m^2$ (ksi) | | | | | | | | | |
| RT | | 67 (9.7) | | 72 (10.5) | | | | | |
| 316C (600F) | | 71 (4.9) | | 58 ( 4.7) | | | | | |
| Specific Gravity | 1.43 | 1.56 | 1.46 | 1.61 | | | | | |
| Fiber Content, | | | | | | | | | |
| % Wt. | 60.4 | 61.7 | 62.3 | 62.4 | | | | | |
| % Volume | | 56.3 | | 57.0 | | | | | |
| Cured Ply Thickness, Mil, Ply | | | | | | | | | |
| Cured | 6.86 | 6.52 | 6.64 | 6.56 | 6.33 | 6.47 | 6.38 | 6.42 | |
| Postcured | 6.44 | 5.94 | 6.27 | 5.94 | | | | | |
| Weight Loss, % | | | | | | | | | |
| Cured | 12.3 | 12.0 | 12.5 | 14.8 | 13.2 | 7.1 | 13.5 | 8.3 | |
| Postcured | 5.6 | 8.7 | 8.1 | 8.1 | | | | | |
| TOTAL | 17.9 | 20.7 | 20.6 | 22.9 | | | | | |
| REMARKS | | 20% Void | | Zero Void | Equip. Malfunction | | | | |

(1) Time to vacuum and pressure application

## TABLE 20. PROPERTIES FOR NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITE (12 PLY, 0°)

| Laminate No. | C-27 | C-52 | C-53 | C-54 | C-55 | C-56 | C-57 | C-58 | C-59 | C-60 | C-61 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Fiber | MODII | MODII | HTS-2 | CEL | CEL | HTS-II | HTS-II | CEL | HTS-II | CEL | HTS-II |
| Cure | CR4 | CR4 | CR-4 | CR-4 | CR-4 | CR-4 | CR-12 | CR-12 | CR-12 | CR-12A | CR-12A |
| Postcure | Oven | Oven | - | - | - | Oven | Oven | | Oven | - | - |
| Orientation/Plies | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 |
| Prepreg Lot No. | 6B-74 | 6C-55 | C8-166 | C8-059 | C8-059 | C8-166 | C8-166 | C8-059 | C8-059 | C8-059 | C8-166 |
| Specific Gravity | 1.57 | 1.38 | 1.25 | | | 1.29 | | 1.31 | 1.43 | | |
| Cured Ply Thickness, mils | 5.4 | 6.7 | 9.0 | 7.4 | 7.5 | 8.2 | 6.7 | 5.5 | 6.3 | | |
| Postcure; Thickness, mil/ply | 6.0 | 7.6 | | | | | 6.4 | | 5.9 | | |
| Wgt Loss; Cure, % | 17.9 | 6.7 | 18.7 | 19.7 | 16.7 | 16.5 | 10.3 | 9.4 | 12.4 | | |
| Postcure, % | 10.5 | 7.6 | | | | | 9.8 | | 3.7 | | |
| Total, % | 28.4 | | | | | | 20.1 | | 16.1 | | |
| Remarks | (1) | (2) | (2) | (3) | (3) | (4) | (5) | (5) | (5) | (6) | (7) |

Remarks:
(1) High oven heating rate
(2) Lost vacuum and reduced pressure
(3) High resin flow
(4) No resin flow
(5) Maximum cure temperature 400 F for 40 minutes
(6) One full 1-120 bleeder
(7) No resin flow

## TABLE 21. PROPERTIES FOR NR-150B2-S5X GRAPHITE/POLYIMIDE COMPOSITE (12 PLY, 0°)

| Laminate No. | C-62 | C-63 | C-64 | C-65 | C-66 | C-67 | C-68 | C-69 |
|---|---|---|---|---|---|---|---|---|
| Fiber | Mod. II | Cel. JK | Mod. II | Mod. II | Cel. 6K | Cel. 6K | Cel. 6K | Cel. 6K |
| Cure | CR4 | CR4 | CR1 | 2B | CR1 | 2C | CR1 | 2D |
| Postcure | Oven (4) | Oven (4) | Oven (5) | A/C | Oven (5) | | | |
| Orientation/Plies | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 | 0/12 |
| Prepreg Lot No. | C8-26 | C6-55 | 6C-55 | 6C-55 | C8-206 | C8-206 | C8-206 | C8-206 |
| Flexural Strength (ksi) | | | | | | | | |
| RT | 128 | 88 | 110 | 137 | 164 | 157 | | |
| 316C (600F) | 54 | 51 | 33 | 83 | 66 | 96 | | |
| Short Beam Shear Strength (ksi) | | | | | | | | |
| RT | 10.2 | 6.7 | 6.6 | 5.1 | 7.9 | 11.6 | | |
| 316C (600F) | 3.2 | 2.6 | 3.2 | 3.5 | 4.3 | 4.8 | | |
| Specific Gravity | 1.45 | 1.40 | 1.44 | 1.45 | 1.46 | 1.53 | 1.52 | 1.47 |
| Fiber Content, | | | | | | | | |
| % Wgt. | 66.7 | 62.2 | | 66.7 | | 66.1 | | |
| % Volume | 63.0 | 56.7 | | 62.9 | | 60.8 | | |
| Cured Ply Thickness, mil | 6.8 | 6.9 | 6.6 | 6.6 | 7.7 | 6.2 | 6.0 | 6.1 |
| Postcure, mil/ply | 6.9 | 7.0 | 6.5 | 6.3 | 6.2 | 5.5 | | |
| Glass Transition Temp., C | -- | --- | --- | --- | 330 | --- | | |
| Void, | 7.2 | 13.5 | Blew | 7.3 | ---- | 5.5 | | |
| Wgt. Loss; Cure | 9.3 | 9.3 | 10.2 | 10.8 | 10.8 | 19.0 | 9.3 | 15.5 |
| Postcure | 7.9 | 10.6 | 9.7 | 11.5 | 10.8 | 9.3 | | |
| Total | 17.2 | 19.9 | 19.9 | 22.3 | 21.6 | 28.3 | | |

Table 22. Properties of NR-150B2-S5X Laminates.

| LAMINATE | | C-103 | C-104 | C-108 | C-110 | C-113 | C-117 | C-120 | C-134 | C-135 | C-137 | C-140 | C-147 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FIBER | | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K | CEL 6K |
| RESIN | | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X | 150B2S5X |
| SIZE | | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY | EPOXY |
| ORIENTATION | | $(0)_{12}$ | $(0)_{12}$ | $(0)_6$ | $(0)_{12}$ | $(0)_{12}$ | $(0)_{12}$ | $(0\pm60)_{s2}$ | $(0\pm60)_{s2}$ | $(0\pm60)_{s2}$ | $(0)_{12}$ | $(0)_{12}$ | $(0)_6$ |
| LOT NO. | | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 | C8-206 |
| ROLL NO. | | 1 | 2 | 2 | 1 | 2 | 2 | 2 | 1 | 2 | 2 | 2 | 2 |
| CURE (DATE) | | 3-15 | 3-15 | 3-20 | 3-20 | 3-20 | 3-25 | 3-22 | 3-24 | 3-24 | 3-24 | 3-24 | 3-24 |
| POSTCURE (DATE) 371/399C 700/750[1] [2] | | 3-25/5-4[1] | 3-25/- | 3-25/- | 3-25/5-4[1] | 3-25/5-4[1] | 3-25/- | 4-27/- | -/5-18[2] | - /5-18[2] | -/5-4[1] | 4-27/5-18[2] | - /5-18[2] |
| SPECIFIC GRAVITY AFTER CURE | | 1.507 | 1.537 | 1.616 | 1.587 | 1.583 | 1.475 | -- | 1.673 | 1.628 | -- | 1.60 | 1.587 |
| SPECIFIC GRAVITY AFTER POSTCURE | | 1.518 | -- | 1.628 | 1.602 | 1.608 | 1.585 | -- | 1.593 | 1.593 | -- | 1.560 | 1.712 |
| CURED PLY THICKNESS MM (MILS) | | .148 (5.85) | .147 (5.80) | .16 (6.32) | .156 (6.17) | .164 .46 | .158 (6.22) | .170 (6.70) | .167 (6.59) | .166 (6.54) | .167 (6.57) | .150 (5.9) | .150 (5.9) |
| POST CURED PLY THICKNESS MM (MILS) | | .142 (5.6) | .142 (5.58) | .139 (5.5) | .148/.142 | .144/.142<br>(5.85/5.6) | .148 (5.82)<br>(5.65/5.6) | .150 (5.9) | .147 (5.80) | .134/.140<br>(5.29 /5.52) | .132 (5.22) | .131/.142<br>(5.15/5.6) | .148 (5.83) |
| Tg | | 310/- | -- | 330/- | 305/- | 302/- | -- | -- | -- | -- | -- | -- | -- |
| TGA | | .36%/- | -- | .44%/- | .30%/- | .36%/- | -- | -- | -- | -- | -- | -- | -- |
| WGT. LOSS CURE | | 12.3 | 12.4 | -- | 5.2 | 6.0 | 6.5 | 12.2 | 6.9 | 5.8 | 5.9 | 5.9 | 12.36 |
| WGT. LOSS POSTCURE | | -- | -- | -- | -- | -- | -- | 7.5 | +.1 | +.2 | -- | /+.1 | 1.6 |
| WGT. LOSS TOTAL | | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 13.96 |
| FLEXURAL STRENGTH | MN/m² | 1343/1330 | 1109 | 1585 | 1502/1509 | 1660/1302 | 1054 | 523 | 677 | 871 | 1612 | 772 | - |
| RT | (ksi) | (195/193) | (161) | | (218/219) | (.41/189) | (153) | (75.9) | (98.3) | (126.4) | (234) | (112.2) | -- |
| 316 | MN/m² | 587/587 | 579 | | 531/813 | 440/847 | 593 | 336 | 617 | 623 | 641 | 663 | |
| (600F) | (ksi) | (85.2/85) | 84 | 157 | 77/118 | (63.9/123) | 86 | 48.8 | 89.6 | 90.4 | 93 | 86.2 | -- |
| SBS | | | | | | | | | | | | | |
| RT | MN/m² | 71/75 | 77.2 | 75.1 | 87/785 | 121/59 | 57.2 | 36 | 61 | 68 | 77 | 63 | |
| | (ksi) | (10.3/10.9) | (11.2) | (10.9) | (12.6/11.4) | (17.6/8.6) | (8.3) | (5.2) | (8.9) | (9.9) | (11.2) | (9.2) | -- |
| 316<br>600 | MN/m² | 32/43 | 47.5 | 29 | | 41/38 | 54.4 | 21 | 35 | 33 | | 33 | |
| | (ksi) | (4.6/6.2) | (6.9) | (4.2) | -- | (5.9/5.5) | (7.9) | (3.0) | (5.1) | (4.8) | -- | (4.8) | -- |

[1] 4 hours at 399C (750F) under vacuum and 1378 MN/m² (200 psi).

[2] 16 hours at 399C (750F) under vacuum and 1378 MN/m² (200 psi).

Table 23. Test Data for NR-150B2 and NR-150B2-S5X Laminates.

| Laminate | Lot | Orientation | Laminate Size (inch) | % Volatiles |  | Prepreg % Resin Solids | Precompaction Bleeder/Side | % Volatile Loss |  |  |  | Specific Gravity (gm/cc)[4] |  |  | Mils /Ply |  | LAMINATE |  |  | Tg (°C) After Post Cure |  | Flexural Strength $MN/M^2$ (KSI) |  | SBS $MN/M^2$ (KSI) |  | C-Scan After 1st Post Cure |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | As Received | As of 9-28 |  |  | Prec. | Cure | Post Cure 1st. | Post Cure 2nd. | After Cure | After post Cure 1st. | After post Cure 2nd. | Cure | Post Cure | % Resin Content | % Fiber Volume | % Void Content | 1st. | 2nd. | 24C | 316C | 24C | 316C |  |
| C-229 | C8-404 (B2)<br>Epoxy Size | $(0)_{12}$ | 18 x 18 | 13.5 | 11.9<br>(11.8-12) | 38.6 | 1-7581 | 5.1 | 2 | 4 | .5 | 1.60 | 1.61 | 1.59 | 5.75 | 5.3 | 21.9 | 71 | <4 | 130 | 358 | 1481<br>(215) | 778<br>(113) | 92.3<br>(13.4) | 43.4<br>(6.3) | 5/20<br>90% Acceptable |
| C-230 | C8-406 (B2)<br>B2 Size | $(0)_{12}$ | 18 x 18 | 13.6 | 12.9<br>(11.3 -13.9) | 36.9 | 1-7581 | 6 | 2.9 | 2.4 | .9 | 1.62 | 1.63 | 1.60 | 5.5 | 4.9 | 31.9 | 62.2 | <1 | -- | -- | -- | -- | -- | -- | 5/20<br>95% Acceptable |
| C-231 | C8-469 (B2)<br>B2 Size | $(0)_{12}$ | 6 x 6 | 12.5 | 10.1<br>(9.9 - 10.5) | -- | 1-7581 | 8.5 | 1.2 | 4.6 |  | 1.50 | 1.51 | 1.49 | 6.4 | 5.5 | 30.2 | 59.4 | <3 | -- | -- | -- | -- | -- | -- | 5/20<br>10% Acceptable |
| C-232 | USP-1 (S5X)<br>B2 Size | $(0)_{12}$ | 6 x 6 | 10.8 | 13.3<br>(12.1-14.1) | 41.5 | 1-7581 | 8.5 | 4 | 7 |  | 1.63 | 1.63 | 1.61 | 5.3 | 4.9 |  |  |  | -- | -- | -- | -- | -- | -- | 5/2<br>90% Acceptable |
| C-233 | 2W4354 (S5X)<br>B2 Size | $(0)_{12}$ | 18 x 18 | -- | 11.4<br>(11.3-11.6) | 28.9 | 1-120 | 1 | 6 | 4.8 |  | 1.64 | 1.59 | 1.54 | 5.1 | 4.8 | 25.3 | 66 | <6 | 190 | 370 | 1874<br>(272) | 1137<br>(165) | 888<br>(12.9) | 36.5<br>(5.3) | 5/2<br>100% Acceptable |
| C-234 | [2]2W4354/USP-1<br>B2 Size | $(0)_{12}$ | 12 x 12 | 16.6 | -- | -- | 1-120 | 1.7 | 6.3 | 5.9 |  | 1.64 | 1.61 | 1.60 | 5.4 | 4.7 | 31 | 64 | <1 | -- | -- | 1846<br>(268) | 1034<br>(150) | 94.4<br>(13.7) | 40.6<br>(5.9) | 5/2<br>100% Acceptable |
| C-235 | C8-533 (S5X)<br>B2 Size | $(0)_{12}$ | 6 x 6 | -- | 14.7<br>(13.5-15.5) | 37.5 | 1-7581 | -- | -- | -- |  | 1.64 | 1.65 | 1.63 | 5.7 | 5.1 | -- | -- | -- | -- | -- | 1495<br>(217) | 1137<br>(165) | -- | -- | 5/2<br>100% Acceptable |
| C-236 | 2W4366 (B2)<br>B2 Size | $(0)_{12}$ | 18 x 18 | -- | 12.7<br>(11.2-13.7) | 38.1 | 1-7581 | 7.7 | 2.6 | 1.9 |  | 1.56 | 1.47 | 1.51 | 5.2 | 5 | -- | -- | -- | -- | -- | -- | -- | -- | -- | 5/20<br>10% Acceptable |
| C-237 | 2W4366 (B2)<br>B2 Size | $(0)_{12}$ | 6 x 6 | -- | 12.7<br>(11.2-13.7) | 38.1 | 1-7581 | 9.4 | 2.7 | 4.5 |  | 1.65 | 1.48 | 1.45 | 5.5 | 5.3 | -- | -- | -- | -- | -- | -- | -- | -- | -- | 5/20<br>10% Acceptable |
| C-238 | 2W4367 (B2)<br>Epoxy Size | $(0)_{12}$ | 18 x 18 | -- | 12.4<br>(11.9-12.7) | -- | 1-7581 | 10.3 | 2.1 | - |  | -- | -- | -- | 5.3 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| C-239 | C8-206 (S5X) | $(0)_{12}$ | 6 x 6 |  | 15.1<br>(14.8-15.4) | 32 | 1-7581 | 6.8 | 2.7 | 4.1 | .9 | 1.61 | 1.61 | 1.61 | 4.7 | 4.3 | 21.9 | 71.8 | <2 | 185 | 368 | 2005<br>(291) | 1247<br>(181) | 82.6<br>(12.0) | 41.3<br>(6.0) | 5/2<br>100% Acceptable |
| C-240 | C8-404 (B2)<br>B2 Size | $(0,45,135,90)_s$ | 6 x 14[1] | 13.5 | 11.9<br>(11.8-12-0) | 38.6 | 1-120 | -- | -- | -- |  | -- | -- | 1.52 | 5.6 | 5.1 | -- | -- | -- | -- | -- | 985<br>(143) | (461)<br>(67) | -- | -- | 5/20<br>90% Acceptable |
| C-241 | C8-404 (B2)<br>B2 Size | $(0,45,135,0)_s$ | 12 x 14[1] | 13.5 | 11.9<br>(11.8-12.0) | 38.6 | 1-120 | 7.7 | 4.1 | 2.0 | -- | -- | -- | 1.49 | 5.7 | 5.3 | -- | -- | -- | -- | -- | -- | -- | -- | -- | 5/20<br>70 % Acceptable |

1. All Fiber Celion 6000
2. Leaf Stack 2 different lots
3. Bleed occured after cure - this number includes resin
4. Example 5/20 = Freq. of 5 Mg $H_2$, Gain of 20

## TABLE 24

## PROPERTIES FOR MULTIPLY STEPPED, NDI LAMINATE

| No. Plies | Specific Gravity | Fiber Weight % | $T_g$, °C | Void % |
|---|---|---|---|---|
| 6 | 1.54 | 65.2 | 359 | 5.9 |
| 12 | 1.52 | 63.9 | 356 | 7.3 |
| 24 | 1.54 | 63.7 | 360 | 5.9 |
| 40 | 1.57 | 61.6 | 360 | 3.7 |

NOTES:

(1) Cured by standard cure cycle.

(2) 30 cm (12 in.) × 61 cm (24 in.) × 15 cm (6 in.) steps.

(3) HMS/NR150B2 (EtOH/NMP solvent).

Table 25. Stainless Steel Lap Shear Test Results

| Adhesive Scrim | | Lap Shear Strength, psi | | | |
|---|---|---|---|---|---|
| | $N/m^2$ | R.T. (ksi) | Failure Mode | $316^o$ (600F) $N/m^2$ (kis) | Failure Mode |
| A1100 | 9,024 | (1320 | ∼ 90% A | 9,163 (1330) | ∼50-60% A |
| | 10,610 | (1540) | ∼ 85% A | 10,610 (1540) | ∼40-50% A |
| | | | | 10,128 (1470) | ∼50-60% A |
| S935 | 7,786 | (1130) | ∼ 95% A | 5,512 (800) | ∼80% A |
| | 7,166 | (1040) | ∼95% A | 4,630 (672) | ∼90% A |

Table 26. Effect of Variation in Filler Content on the Strength of the Adhesive.

| Base Polyimide Resin | NR056X | | LARC 160 | |
|---|---|---|---|---|
| Filler-to-Resin Ratio by Weight | 65:35 | 35:65 | 65:35 | 35:65 |
| Titanium lap shear strength (psi) | | | | |
| 10 min. at -55C(-67F) ±10F | 2,480 | 2,310 | 2,870 | 2,020 |
| R.T. | 2,070 | 2,060 | 2,190 | 2,260 |
| 10 min. at 60±10F | 1,510 | 1,450 | 1,850 | 1,440 |
| 10 min. at 316C(600F)after aged 160 hr at 316C(600F) | 1,540 | 1,250 | 1,100 | 1,110 |
| R.T. after 20 days of exposure to 160F, 95-100%R.H. | 1,260 | 1,270 | 2,250 | 2,340 |

Table 27. Properties of Polyimide Adhesives

| Resin Classification | Condensation Polyimides | | | Addition Polyimides | |
|---|---|---|---|---|---|
| Adhesives | FM34 | NR150B2 | NR056X | LARC13 | LARC160 |
| Filler-to-Resin Ratio by Weight | - | 65:35 | 65:35 | 30:70 | 65:35 |
| Uncured Bondine Thickness (mils) | 14-15 | 15-18 | 15-18 | 11-12 | 17-19 |
| Cured Bondine Thickness (mils) | 13-16 | 12-13 | 14-15 | 4-5 | 8-9 |
| Titanium Lap Shear Strength(psi) | | | | | |
| 10 min. at 316C(600F)±10F | 2,070 | 2,700 | 2,480 | 1,840 | 2,870 |
| R.T. | 2,810 | 2,340 | 2,070 | 1,580 | 2,190 |
| 10 min. at 316C(600F)±10F | 1,340 | 2,030 | 1,510 | 1,850 | 1,850 |
| 10 min. at 316C(600F)after aged 160 hrs at 316C(600F) | 1,590 | 1,910 | 1,540 | 1,080 | 1,100 |
| R.T. after 20 days of exposure to 100F, 95-100% R.H. | 1,770 | 1,790 | 1,260 | 1,630 | 2,250 |
| Tg, $^{o}$F (measured using TMA at 20C/min. heat-up rate) | - | 660 | 640 | - | 560 |

Table 28. Effect of 316C (600F) Exposure on the
Shear Strength of NR-150B2

| | Lap Shear Strength, psi | | | | | |
|---|---|---|---|---|---|---|
| | Tested @ R.T. After | | | Tested @ 600F After | | |
| Test Specimen Configuration | 0 hr Aging @ 600F | 100 hr Aging @ 600F | 200 hr Aging @ 600F | 0 hr Aging @ 600F | 100 hr Aging @ 600F | 200 hr Aging @ 600F |
| Titanium Single Overlap Shear<br>0.050" 6AR-4V-Ti | 2540 | 2420 | 1960 | 1580 | 1560 | 1530 |
| Composite Single Overlap Shear | 2840 | 2000 | 1660 | 1600 | 1320 | 1610 |
| Titanium Double Overlap Shear | 2810 | 2640 | 1810 | 1960 | 1320 | 1600 |
| Composite Double Overlap Shear | 1100* | 1020* | 730* | 470* | 830* | 910* |

* Failure occurred as a result of adherend delamination.

Table 29. Unidirectional Laminate Design Data Test Plan

| Test | Laminate Number | Laminate Size | Laminate Orientation | Test Direction | Test Temperature | Specimen Number |
|---|---|---|---|---|---|---|
| $F_{TU}$, $E_T$[1] | C-532 | 12 × 20 | $(0)_8$ | 0° | RT | T-1, 5, 9, 13, 17, 21 |
| | | | | | 600 | T-2, 6, 10, 14, 18, 22 |
| | | | | | RT[2] | T-3, 7, 11, 15, 19, 23 |
| | | | | | 600[2] | T-4, 8, 12, 16, 20, 24 |
| $F_{FU}$, $E_T$ | C-533 | 8 × 20 | $(0)_{12}$ | 0° | RT | F-1, 5, 9, 13, 17, 21 |
| | | | | | 600 | F-2, 16, 10, 14 |
| | | | | | RT[2] | F-3, 7, 11, 15, 19, 23 |
| | | | | | 600[2] | F-4, 8, 12, 16, 20, 24 |
| $F_{SU}$ | C-533 | 8 × 20 | $(0)_{12}$ | 0° | RT | S-1, 2, 3, 4, 5, 6 |
| | | | | | 600 | S-7, 8, 9, 10, 11, 12 |
| | | | | | RT[2] | S-13, 14, 15, 16, 17, 18 |
| | | | | | RT[2] | S-19, 20, 21, 22, 23 |
| $F_{CU}$ | C-538 | 8 × 12 | $(0)_{24}$ | 0° | RT | C-1, 5, 9, 13 |
| | | | | | 600 | C-2, 6, 10, 14 |
| | | | | | RT[2] | C-3, 7, 11, 15, 19, 23 |
| | | | | | 600[2] | C-4, 8, 12, 16, 20, 24 |

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[1] Modulus determine on selected specimens only

[2] After 200 hours at 600F

Table 30. Multiaxis Laminate Design Data Test Plan

| Test | Laminate Number | Laminate Size | Laminate Orientation | Test Direction | Test Temperature | Specimen Number |
|---|---|---|---|---|---|---|
| $F_{TU}$, $E_T$[1] | C-535 | 20 × 22 | $(0,45,135,90)_s$ | 0° | RT | T-1,3,5,7,9,11 |
|  |  |  |  |  | 600 | T-13,15,17,19,21,23 |
|  |  |  |  |  | RT[2] | T-2,4,6,8,10,12 |
|  |  |  |  |  | 600[2] | T-14,16,18,20,22,24 |

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[1] Modulus determine on selected specimens only

[2] After 200 hours at 600F

Table 31. Mechanical Properties of Laminate C-532 $(O)_8$ Nonaged Specimens

| Specimen Number | Test Temperature | | $F_{TU}$ MN/m$^2$/ksi | $E_T$ MN/m$^2$/msi |
|---|---|---|---|---|
| T-1 | 24C (75F) | | 1849/268 | - |
| T-5 | | | 1814/263 | 161/23.4 |
| T-9 | | | 1628/236 | - |
| T-13 | | | 1718/249 | 157/22.7 |
| T-17 | | | 1738/252 | - |
| T-21 | | | 1752/254 | 157/22.7 |
| | | $\bar{X}$ | 1750/253.6 | 158/22.9 |
| | | N | -/ 6 | -/ 3 |
| | | S | 77/ 11.2 | 3/ 0.4 |
| | | $C_y$ | -/ 4.4% | -/ 1.7% |
| | | $N^1$ | 1464/212.2 | 132/19.2 |
| T-2 | 316C (600F) | | 1725/250 | |
| T-6 | | | 1677/243 | |
| T-10 | | | 1856/269 | |
| T-14 | | | 1497/217 | |
| T-18 | | | 2007/291 | |
| T-22 | | | 1449/210 | |
| | | $\bar{X}$ | 1702/246.7 | |
| | | N | -/ 6 | |
| | | S | 212/ 30.7 | |
| | | $C_y$ | -/ 12.45% | |
| | | $N^1$ | 1424/206.4 | |

Percent Resin Content: 25.9
Percent Fiber Content: 74.1
Percent Void Content: 4.2
Specific Gravity: 1.557
Percent Fiber Volume: 65.93

$N^1$ - Normalized to 62 percent fiber volume

Table 32. Mechanical Properties of Laminate C-532 $(O)_8$ After 200 Hours at 316C (600F)

| Specimen Number | Test Temperature | | $F_{TU}$ MN/m$^2$/ksi |
|---|---|---|---|
| T-3 | 24C (75F) | | 1625/235.5 |
| T-7 | | | 1759/255.0 |
| T-11 | | | 954/137.8 |
| T-15 | | | 1839/266.5 |
| T-19 | | | 1464/212.2 |
| T-23 | | | 1838/266.4 |
| | | $\overline{X}$ | 1705/247.1 |
| | | N | -/ 5 |
| | | S | 161/23.3 |
| | | $C_y$ | -/ 9.49% |
| | | $N^1$ | 1426/206.8 |
| T-4 | 316C (600F) | | 1586/230 |
| T-8 | | | 1669/242 |
| T-12 | | | 1448/210 |
| T-16 | | | 1414/205 |
| T-20 | | | 1469/2213 |
| T-24 | | | 1442/209 |
| | | $\overline{X}$ | 1504/218.17 |
| | | N | -/ 6 |
| | | S | 100/ 14.55 |
| | | $C_y$ | -/ 9.0% |
| | | $N^1$ | 1414/205.2 |

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$N^1$ - Normalized to 62 percent fiber volume

Table 33. Mechanical Properties of Laminate C-533 $(O)_{12}$ Nonaged Specimens

| Specimen Number | Test Temperature | | $F_{FU}$ MN/m$^2$/ksi | $E_{FU}$ MN/m$^2$/msi | | $F_{SU}$ MN/m$^2$/ksi |
|---|---|---|---|---|---|---|
| F-1 | 24C (75F) | | 2152/312 | - | | |
| F-5 | | | 2221/322 | 149/21.6 | | |
| F-9 | | | 2069/300 | - | | |
| F-13 | | | 2042/296 | 148/21.5 | | |
| F-17 | | | 2111/306 | - | | |
| F-21 | | | 2049/297 | 138/20 | | |
| | | $\overline{X}$ | 2107/305 | 141/21 | | |
| | | N | -/ 6 | -/ 3 | | |
| | | S | 69.38/10.1 | 5.77/0.89 | | |
| | | $C_y$ | -/ 3.3% | -/ 4.2% | | |
| | | $N^1$ | 1892/273 | 126/18.8 | | |
| S-1 | | | | | | 78/10.6 |
| S-2 | | | | | | 93/13.5 |
| S-3 | | | | | | 64/ 9.3 |
| S-4 | | | | | | 97/14.1 |
| S-5 | | | | | | 98/14.2 |
| S-6 | | | | | | 91/13.2 |
| | | | | | $\overline{X}$ | 87.1/12.5 |
| | | | | | N | -/ .6 |
| | | | | | S | 14/ 2.03 |
| | | | | | $C_y$ | -/ 16% |
| F-2 | 316C (600F) | | 1152/167 | 103/15 | | |
| F-6 | | | 1124/163 | - | | |
| F-10 | | | 1235/179 | - | | |
| F-14 | | | 1338/194 | 120/17.4 | | |
| F-18 | | | 1255/182 | - | | |
| F-20 | | | 1331/193 | 118/17.1 | | |
| | | $\overline{X}$ | 1239/179.7 | 114/16.5 | | |
| | | N | -/ 6 | 3/ 3 | | |
| | | S | 89/ 12.9 | 9/ 1.3 | | |
| | | $C_y$ | -/ 7.2% | -/ 7.9% | | |
| | | $N^1$ | 1112/161.4 | 102/14.8 | | |

Table 33. Mechanical Properties of Laminate C-533 $(O)_{12}$ Nonaged Specimens, Contd

| Specimen Number | Test Temperature | $F_{FU}$ MN/m$^2$/ksi | $E_{FU}$ MN/m$^2$/msi | $F_{SU}$ MN/m$^2$/ksi |
|---|---|---|---|---|
| S-7 | 316C (600F) | | | 41/5.94 |
| S-8 | | | | 46/6.74 |
| S-9 | | | | 39/5.70 |
| S-10 | | | | 48/6.92 |
| S-11 | | | | 46/6.65 |
| S-12 | | | | |
| | | | | X 44/6.39 |
| | | | | N -/ .5 |
| | | | | S 4/ .54 |
| | | | | $C_y$ -/8.4% |

| | |
|---|---|
| Percent resin content: | 24.9 |
| Percent fiber content: | 75.1 |
| Percent void content: | 1.27 |
| Specified gravity: | 1.609 |
| Percent fiber volume: | 69.05 |

$N^1$ - Normalized to 62 percent fiber volume

Table 34. Mechanical Properties of Laminate C-533 $(O)_{12}$ Aged at 316C (600F) for 200 Hours

| Specimen Number | Test Temperature |  | $F_{FU}$ MN/m$^2$/ksi | $E_F$ MN/m$^2$/msi |  | $F_{SB}$ MN/m$^2$/ksi |
|---|---|---|---|---|---|---|
| F-3 | 24C (75F) |  | 1924/279 | - |  |  |
| F-7 |  |  | 1835/266 | 137/19.8 |  |  |
| F-11 |  |  | 1959/284 | - |  |  |
| F-15 |  |  | 1980/287 | 134/19.4 |  |  |
| F-19 |  |  | 1917/278 | - |  |  |
| F-23 |  |  | 1904/276 | 137/19.8 |  |  |
|  |  | X | 1917/278 | 136/19.7 |  |  |
|  |  | N | -/ 6 | -/ 3 |  |  |
|  |  | S | 50/ 7.3 | 1.6/ 0.23 |  |  |
|  |  | $C_y$ | -/ 2.6% | -/ 1.19 |  |  |
|  |  | $N^1$ | 1721/249.6 | 122/17.7 |  |  |
| S-13 |  |  |  |  |  | 77/11.2 |
| S-14 |  |  |  |  |  | 83/12.1 |
| S-15 |  |  |  |  |  | 87/12.6 |
| S-16 |  |  |  |  |  | 80/11.6 |
| S-17 |  |  |  |  |  | 79/11.4 |
| S-18 |  |  |  |  |  | 77/11.1 |
|  |  |  |  |  | X | 80/11.6 |
|  |  |  |  |  | N | -/ 6 |
|  |  |  |  |  | S | 4/ 0.58 |
|  |  |  |  |  | $C_y$ | -/5% |
| F-4 | 316C (600F) |  | 1124/163 | - |  |  |
| F-8 |  |  | 1221/177 | 126/18.2 |  |  |
| F-12 |  |  | 1186/172 | - |  |  |
| F-16 |  |  | 1228/178 | 123/17.9 |  |  |
| F-20 |  |  | 1193/173 | - |  |  |
| F-24 |  |  | 1200/174 | 132/19.2 |  |  |
|  |  | X | 1191/172.8 | 127/18.4 |  |  |
|  |  | N | -/ 6 | -/ 3 |  |  |
|  |  | S | 37/ 5.3 | 5/ .68 |  |  |
|  |  | $C_y$ | -/ 3.1% | -/ 3.6% |  |  |
|  |  | $N^1$ | 1069/155.1 | 114/16.5 |  |  |

Table 34. Mechanical Properties of Laminate C-533 $(O)_{12}$ Aged at 316C (600F) for 200 Hours, Continued

| Specimen Number | Test Temperature | $F_{FU}$ MN/m$^2$/ksi | $E_F$ MN/m$^2$/msi | $F_{SB}$ MN/m$^2$/ksi |
|---|---|---|---|---|
| S-19 | 316C (600F) | | | 47/6.9 |
| S-20 | | | | 50/7.2 |
| S-21 | | | | 50/7.2 |
| S-22 | | | | 50/7.2 |
| S-23 | | | | 50/7.3 |
| S-24 | | | | 46/6.7 |
| | | | | X 49/7.1 |
| | | | | N -/6 |
| | | | | S 2/ .23 |
| | | | | $C_y$ -/3.3% |

$N^1$ - Normalized to 62 percent fiber volume

## Table 35. Mechanical Properties of Laminate C-538 $(O)_{24}$ Nonaged Specimens

| Test/Specimen Number | Test Temperature | | $F_{CU}$ MN/$m^2$/ksi | $E_{CU}$ MN/$m^2$/ksi |
|---|---|---|---|---|
| 1890/C-1 | 24C (75F) | | 1196/173.4 | |
| 1891/C-5 | | | 1690/244.9 | 142/20.64 |
| 1892/C-9 | | | 1493/216.4 | |
| 1893/C-13 | | | 1365/197.9 | 147/21.25 |
| | | X | 1435/208.1 | 144/20.9 |
| | | N | -/ 4 | - |
| | | S | 208/ 30.2 | - |
| | | $C_v$ | -/ 14.5% | - |
| | | $N^1$ | 1260/182.8 | 127/18.4 |
| /C-2 | 316C (600F) | | 1034/150 | |
| /C-6 | | | 890/129 | |
| /C-10 | | | 924/134 | |
| /C-14 | | | 807/117 | |
| | | X | 914/132.5 | |
| | | N | -/ 4 | |
| | | S | 94/ 13.7 | |
| | | $C_v$ | -/ 10.3% | |
| | | $N^1$ | 803/116.3 | |

| | |
|---|---|
| Percent resin content: | 22.26 |
| Percent fiber content: | 77.74 |
| Percent void content: | 3.2 |
| Specific gravity: | 1.589 |
| Percent fiber volume: | 70.59 |

$N^1$ - Normalized to 62 percent fiber volume

Table 36. Mechanical Properties of Laminate C-538 $(0)_{24}$ Aged 200 Hours at 316C (600F)

| Test/Specimen Number | Test Temperature | $F_{CU}$ MN/m$^2$/ksi |
|---|---|---|
| /C-3 | 24C (75F) | 1338/194 |
| /C-7 | | 1393/202 |
| /C-11 | | 1490/216 |
| /C-15 | | 1503/218 |
| /C-19 | | 1420/206 |
| | | X 1429/207.2 |
| | | N -/ 5 |
| | | S 68/ 9.96 |
| | | $C_v$ -/ 4.8% |
| | | $N^1$ 1255/182 |
| /C-4 | 316C (600F) | 986/143 |
| /C-8 | | 1200/174 |
| /C-12 | | 1034/150 |
| /C-16 | | 944/137 |
| /C-20 | | 800/166 |
| | | X 993/144 |
| | | N -/ 5 |
| | | S 145/ 21.0 |
| | | $C_v$ -/ 14.0% |
| | | $N^1$ 872/126.4 |

Table 37. Mechanical Properties of Laminate C-535 $(0, 45, 135, 90)_S$ Nonaged Specimens

| Specimen Number | Test Temperature | $F_{TU}$ MN/m$^2$/ksi | $E_T$ MN/m$^2$/ksi |
|---|---|---|---|
| T-1 | 24C (75F) | 630/91.3 | - |
| T-3 | | 557/80.8 | 57/8.3 |
| T-5 | | 631/91.5 | - |
| T-7 | | 475/68.8 | - |
| T-9 | | 519/75.2 | 58/8.4 |
| T-11 | | 548/79.5 | - |
| X | | 556/81.2 | 57/8.3 |
| N | | -/ 6 | - |
| S | | 62/ 9 | - |
| $C_v$ | | -/11% | |
| $N^1$ | | 502/72.8 | 51/7.4 |
| T-13 | 316C (600F) | 494/71.6 | |
| T-15 | | 457/66.2 | |
| T-17 | | 517/74.9 | |
| T-19 | | 528/76.6 | |
| T-21 | | 470/68.1 | |
| T-23 | | 509/73.8 | |
| X | | 496/71.9 | |
| N | | -/ 6 | |
| S | | 28/ 4.04 | |
| $C_v$ | | -/ 5.6% | |
| $N^1$ | | 445/64.5 | |

Percent resin content: 22.26
Percent fiber content: 77.74
Percent void content: 5.2
Specific gravity: 1.556
Percent fiber volume: 69.72

$N^1$ - Normalized to 62 percent fiber volume

Table 38. Mechanical Properties of Laminate C-535 $(0, 45, 135, 90)_S$ After 200 Hours at 316C

| Specimen Number | Test Temperature | | $F_{TU}$ MN/m$^2$/ksi |
|---|---|---|---|
| T-2 | 24C (75F) | | 611/88.6 |
| T-4 | | | 543/78.7 |
| T-6 | | | 578/83.8 |
| T-8 | | | 627/90.9 |
| T-10 | | | 505/73.3 |
| T-12 | | | 602/87.3 |
| | | X | 577/83.7 |
| | | N | -/ 6 |
| | | S | 46/ 6.7 |
| | | $C_v$ | -/ 8% |
| | | $N^1$ | 518/75.1 |
| T-15 | 316C (600F) | | 457/66.2 |
| T-18 | | | 498/72.2 |
| T-20 | | | 592/85.9 |
| T-22 | | | 451/65.4 |
| T-24 | | | 483/70.1 |
| | | X | 496/71.96 |
| | | N | -/ 5 |
| | | S | 57/ 8.27 |
| | | $C_v$ | -/11.5% |
| | | $N^1$ | 445/64.5 |

$N^1$ - Normalized to 62 percent fiber volume

Table 39. Mechanical Properties of Laminate C-536 $(O)_{12}$
(Adhesive Study Laminate Verifications)

| Specimen Number | Thermal Conditioning | Test Temperature | | $F_{FU}$ MN/m$^2$/ksi |
|---|---|---|---|---|
| F-1 | None | 24C (75F) | | 1890/274 |
| F-5 | | | | 2138/310 |
| F-9 | | | | 2145/311 |
| | | | X | 2062/299 |
| | | | N | -/ 3 |
| | | | S | 145/ 21 |
| | | | $C_v$ | -/ 7% |
| | | | $N^1$ | 1856/269 |
| F-2 | None | 316C (600F) | | 1042/151 |
| F-6 | | | | 1117/162 |
| F-10 | | | | 1186/172 |
| | | | X | 1115/161.7 |
| | | | N | -/ 3 |
| | | | S | 72/ 10.5 |
| | | | $C_v$ | -/ 6.5% |
| | | | $N^1$ | 1005/145.7 |
| F-3 | 100 hours at 316C (600F) | 24C (75F) | | 1842/267 |
| F-7 | | | | 1980/287 |
| F-11 | | | | 1890/274 |
| | | | X | 1904/276 |
| | | | N | -/ 6 |
| | | | S | 69/ 10 |
| | | | $C_v$ | -/ 3.9% |
| | | | $N^1$ | 1715/248.7 |

Table 39. Mechanical Properties of Laminate C-536 $(O)_{12}$ (Adhesive Study Laminate Verifications), Contd

| Specimen Number | Thermal Conditioning | Test Temperature | $F_{FU}$ MN/m$^2$/ksi |
|---|---|---|---|
| F-4 | 100 hours at 316C (600F) | 316C (600F) | 1242/180 |
| F-8 | | | 1290/187 |
| F-16 | | | 1193/173 |
| | | X | 1241/180 |
| | | N | -/ .3 |
| | | S | 48/ 7 |
| | | $C_v$ | -/ 3.9% |
| | | $N^1$ | 1117/162 |

| | |
|---|---|
| Percent resin content: | 24.8 |
| Percent fiber content: | 75.2 |
| Percent void content: | 1.8 |
| Specific gravity: | 1.601 |
| Percent fiber volume: | 68.8 |

N - Normalized to 62 percent fiber volume

Table 40. Mechanical Properties of Laminate C-537 $(0, 45, 135, 90)_{2S}$ (Adhesive Study Laminate Verification)

| Specimen Number | Thermal Conditioning | Test Temperature | | $F_{FU}$ $MN/m^2$/ksi |
|---|---|---|---|---|
| F-1 | None | 24C (75F) | | 883/128 |
| F-7 | | | | 807/117 |
| F-13 | | | | 883/128 |
| | | | X | 857/124.3 |
| | | | N | -/ 3 |
| | | | S | 44/ 6.35 |
| | | | $C_V$ | -/ 5.1% |
| | | | $N^1$ | 773/112 |
| F-2 | None | 316C (600F) | | 490/ 71 |
| F-8 | | | | 508/ 73.7 |
| F-14 | | | | 538/ 78 |
| | | | X | 512/ 74.2 |
| | | | N | -/ 3 |
| | | | S | 24/ 3.53 |
| | | | $C_V$ | -/ 4.8% |
| | | | $N^1$ | 462/ 66.9 |
| F-3 | 100 hours at 316C (600F) | 24C (75F) | | 684/ 99.1 |
| F-9 | | | | 835/121 |
| F-15 | | | | 606/ 87.8 |
| | | | X | 708/102.6 |
| | | | N | -/ 3 |
| | | | S | 117/ 16.9 |
| | | | $C_V$ | -/ 16.5% |
| | | | $N^1$ | 638/ 92.5 |
| F-4 | 100 hours at 316C (600F) | 316C (600F) | | 621/ 90 |
| F-10 | | | | 862/125 |
| F-16 | | | | 586/ 85 |
| | | | X | 690/100 |
| | | | N | -/ 3 |
| | | | S | 150/ 21.8 |
| | | | $C_V$ | -/ 21.8% |
| | | | $N^1$ | 622/ 90.18 |

Table 40. Mechanical Properties of Laminate C-537 (0, 45, 135, 90)$_{2S}$ (Adhesive Study Laminate Verification) (Continued)

| Specimen Number | Thermal Conditioning | Test Temperature | | $F_{FU}$ MN/m$^2$/ksi |
|---|---|---|---|---|
| F-5 | 200 hours at 316C (600F) | 24C (75F) | | 710/103 |
| F-11 | | | | 766/111 |
| | | | X | 747/107 |
| | | | N | -/ 2 |
| | | | S | 39/ 5.6 |
| | | | C | -/ 5.3% |
| | | | N | 674/ 96.5 |
| F-6 | 200 hours at 316C (600F) | 316C (600F) | | 690/100 |
| F-12 | | | | 655/ 95 |
| | | | X | 672/ 97.5 |
| | | | N | -/ 2 |
| | | | S | 24/ 3.5 |
| | | | $C_V$ | -/ 3.6% |
| | | | $N^1$ | 606/ 87.9 |

| | |
|---|---|
| Percent resin content: | 23.9 |
| Percent fiber content: | 76.1 |
| Percent void content: | 3.3 |
| Specific gravity: | 1.581 |
| Percent fiber volume: | 68.75 |

N - Normalized to 62 percent fiber volume

Table 41. Assumed Material Properties for Local Buckling

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Skin: $(0, 45, 90, 135, 0, 45)_s$, $t = 0.060$

$E_X = 8.4+6$ psi

$D_{11} = 200.6$ $D_{22} = 110.8$ $D_{66} = 39.1$ $D_{12} = 37.7$

Stringers: $(0, \pm 45, 0_2, \pm 45, 0)_s$, $5 = 0.080$

$E_X = 10.6+6$ psi $E_y = 3.1+6$

$D_{11} = 547.1$ $D_{22} = 152.6$ $D_{66} = 111.9$ $D_{12} = 108.5$

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16. Abstract

A study was conducted to assess the merits of using graphite/polyimide, NR-150B2 resin, for structural applications on advanced space launch vehicles. The program was divided into two phases: (1) Fabrication Process Development and (2) Demonstration Components. The first phase of the program involved the selection of a graphite fiber, quality assurance of the NR-150B2 polyimide resin, and the quality assurance of the graphite/polyimide prepreg. In the second phase of the program, a limited number of components were fabricated before the NR-150B2 resin system was removed from the market by the supplier, Du Pont.

The advancement of the NR-150B2 polyimide resin binder was found to vary significantly based on previous time and temperature history during the prepregging operation. Strength retention at 316C (600F) was found to be 50% that of room temperature strength. However, the composite would retain its initial strength after 200 hours exposure at 316C (600F). Basic chemistry studies are required for determining NR-150B2 resin binder quality assurance parameters. Graphite fibers are available that can withstand high temperature cure and postcure cycles.

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