NASA Contractor Report 165809

NASA-CR-165809
19820014441

Development and Demonstration of Manufacturing Processes for Fabricating Graphite/LARC 160 Polyimide Structural Elements

R.K. Frost, J.S. Jones
P.J. Dynes and D.H. Wykes

Rockwell International
Downey, CA 90241

Contract NAS 1-15371
DECEMBER 1981

National Aeronautics and
Space Administration

Langley Research Center
Hampton Virginia 23665

NASA Contractor Report 165809

Development and Demonstration of Manufacturing Processes for Fabricating Graphite/LARC 160 Polyimide Structural Elements

R.K. Frost, J.S. Jones
P.J. Dynes and D.H. Wykes

Rockwell International
Downey, CA 90241

Contract NAS 1-15371
DECEMBER 1981

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665

TABLE OF CONTENTS

TABLE OF CONTENTS (cont.)

TABLE OF CONTENTS (cont.)

TABLE OF CONTENTS (cont.)

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

LIST OF TABLES

LIST OF TABLES

LIST OF TABLES

1.0 SUMMARY

This final report describes the program effort performed by Rockwell International for the NASA/LaRC under Contract NAS1-15371. The objective of the program was to develop and demonstrate manufacturing technologies for the structural application of Celion graphite/LARC-160 polyimide composite material.

The program consisted of two parts: Process Development and Fabrication of Demonstration Components. Process development included establishing quality assurance of the basic composite material and processing, non-destructive inspection of fabricated components, developing processes for specific structural forms, and qualification of processes through mechanical testing. In the second part of the program, demonstration components were fabricated using the processes developed in part one. The demonstration components consisted of flat laminates, skin/stringer panels, honeycomb panels, chopped fiber compression moldings, and a Technology Demonstrator Segment (TDS) representative of the Space Shuttle aft body flap. The TDS, initially intended to be only a display article, was later directed in the program to a testable component. TDS test results will be reported in a separate final report.

2.0 INTRODUCTION

This program was conducted for the NASA Langley Research Center, Materials Division, Materials Application Branch under NASA contract NAS1-15371. Mr. Robert M. Baucom was the NASA Program Manager. Mr. Roger Frost of the Advanced Manufacturing Technology Department of Rockwell International, Downey, California was responsible for program management and technical direction. Acknowledgement is made for the technical assistance provided during the program by the following Rockwell personnel:

B. D. Bhombal	C. L. Hamermesh	J. D. Leahy
I. Bouton	P. J. Hodgetts	W. H. Morita
R. J. Demonet	K. C. Hong	M. A. Morrow
P. J. Dynes	D. W. Houston	F. Roje
H. E. Flanery	J. S. Jones	D. H. Wykes
S. R. Graves	C. C. Kammerer	

The initial objective of this program was to develop and demonstrate manufacturing technologies for structural application of Celion graphite/LARC-160 polyimide composite materials. Later this was expanded by contract modification to include mechanical ground testing of a Technology Demonstrator Segment (TDS), a three-bay test article representative of the Space Shuttle aft body flap.

This final report presents the accomplishments and results of the original contract requirements with regard to manufacturing technologies. Mechanical ground testing of the TDS will be documented in a subsequent report.

The manufacturing technologies phase of the program was divided into two parts, process development and the fabrication of demonstration components, each consisting of several tasks. The following briefly describes the objective.

Part 1. Process Development

Task (a) - Develop a quality assurance program including specification for Celion/LARC-160 polyimide materials, quality control of materials and processes, including studies of the effects of monomer and/or polymer variables and prepreg variables on the processibility of Celion/LARC-160 prepreg and on the mechanical properties of test specimens fabricated from the prepreg, and NDI of fabricated components.

Task (b) - Develop processes for fabricating laminates, hat and "I" stiffeners, honeycomb core panels, and chopped fiber moldings.

Task (c) - Fabricate specimens and conduct tests to qualify the processes for fabrication of demonstration components.

Part 2. Demonstration Components

Task (d) - Fabricate and NDI three (3) laminates 61x22-cm (24x48-in.) with 0^{o}, $\pm 45^{o}$ lay up symmetrical about the neutral axis. Laminate thickness to be 0.08-cm, 0.15-cm, and 0.32-cm (0.030 in., 0.060 in., and 0.125 in.).

Task (e) - Fabricate and NDI three (3) secondarily bonded hat-stiffened skin-stringer panels 23-cm (9 in.) wide x 122-cm (48 in.) long with three lengthwise stiffeners.

Task (f) - Fabricate six (6) honeycomb core panels 25.4x24.5-cm (10x10-in.) having 0.15-cm (0.060 in.) thick face sheets with 0^{o}, 90^{o} layup symmetrical about the neutral axis of the panel and 2.54-cm (1 in.) thick honeycomb core.

Task (g) - Fabricate six (6) chopped fiber moldings according to a specimen design mutually agreeable to Contractor and Contracting Officer's technical representative.

Task (h) - Fabricate a representative component of a Space Shuttle aft body flap that is mutually agreeable to the Contractor and Contracting Officer's technical representative.

Each of the above tasks is documented separately in the main body of this final report.

Use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.

3.0 PROCESS DEVELOPMENT

3.1 TASK (a) - QUALITY ASSURANCE

3.1.1 Selection of Prepreg Supply Source

On initiating the program, a survey of seven potential prepreg suppliers was conducted to determine their ability for providing Celion/LARC-160 prepreg materials to support the objectives of the contract. Each was evaluated by questionaire, Appendix A1. Following screening of returned questionaires, an on-site inspection of supplier prepreg facilities was made by a Rockwell survey team. The objectives of the survey were to determine the potential supplier's level of quality control procedures and record keeping, the type and adequacy of analytical test equipment, specific personnel capabilities, and their indicated cooperation and willingness to work with Rockwell to achieve reproducible prepreg material. Each was requested to provide samples of Celion/LARC-160 prepreg tape for evaluation. Four of the seven suppliers responded to this request.

The prepreg samples were subjected to physical tests to determine percent of volatiles, resin solids content, fiber areal weight, and calculated thickness per ply. Visual examinations were performed to evaluate fiber collimation, gaps/edge waviness, tack and drape. Three suppliers, one primary and two backup, were selected to provide Celion/LARC-160 prepreg for the program.

3.1.2 Material Specification

All prepreg material used for this program was ordered against the requirements shown in Appendix A2 included as a flysheet attachment with each purchase order. Test conditions and calculations imposed by item 5 of the flysheet were defined by document LTR 2433-4462 which is presented in Appendix A3. In addition, prepreg acceptability for program production use was based on chemical

analysis of the intermediate ester and neat resin used to ensure prepreg batch-to-batch repeatability. Each prepreg batch was subjected to physical and mechanical testing before being used for program requirements.

From this program activity and also that conducted against NASA/LaRC contract NAS1-15183, Graphite/Polyimide Design and Fabrication, Rockwell material Specification MB0130-152, Graphite/Polyimide Resin Prepreg - 600°F Applications was developed and is presented in Appendix B1. This specification includes both LARC-160 and PMR-15 resin impregnated graphite material systems and will be invoked when procuring these materials for production use.

3.1.3 Process Specification

Rockwell Process Specification MA0105-328, Fabrication of 600°F Polyimide Graphite made from Norborene Terminated Methylene Di or Larger Analine Benzophenonetetracarboxylic Base Resin, is presented in Appendix B2. This specification presents the improved processing technique developed for both LARC-160 (Contract NAS1-15371) and PMR-15 (Contract NAS1-15183). While this specification describes processing for flat laminates, using perforated metal caul plates, the staging and curing procedures are directly applicable to complex structural shapes and were used in fabricating detail elements of the Technology Demonstrator Segment required by Task (h) of the program.

3.1.4 Nondestructive Inspection Techniques

Ultrasonic C-scan was the primary nondestructive inspection (NDI) technique utilized throughout this program to detect porosity, voids and debonds in laminates and bonded structures.

C-scan sensitivity is one of the most important variables that must be considered in the quality assessment of laminates and bonded structures. To optimize calibration sensitivity, comparative reference standards having internal defects of known type and size were fabricated and used for sensitivity settings. At the beginning of the NASA Graphite/Polyimide Design and Fabrication Contract

(NAS1-15183), personnel at LaRC and Rockwell shared sample specimens of graphite/polyimide laminates having known defects. Ultrasonic C-scans and destructive correlative tests were made followed by the selection of an "A" sensitivity agreed to by both LaRC and Rockwell personnel. Therefore, "A" sensitivity has been used for ultrasonic C-scan inspection of products produced under this program with only minor exceptions as will be noted in appropriate sections of the test.

Solid laminate structures were C-scanned in the NDI laboratory by engineering personnel. Sandwich structures, however, were C-scanned in the production facility. The specification governing production C-scan procedure is presented in Appendix B3.

3.1.5 Chemical Characterization of LARC-160

The primary goal of this effort was to provide the methodology for specifying the chemical composition of LARC-160 polyimide resin materials. Once these analytical procedures were developed their sensitivity could then be assessed by analysis of a series of LARC-160 resin materials containing intentional composition and processing variations. This would thus provide the first step toward establishment of a chemical quality assurance specification for LARC-160.

One of the most successful techniques for characterizing composite matrix resins is high pressure liquid chromatography (HPLC). The objectives of the program were to describe the development of HPLC methods for characterizing LARC-160 resin and their application to the analysis of standard and Variables Study (discussed in 3.1.6) lots of commerical LARC-160 Resin materials.

3.1.5.1 LARC-160 Synthesis and Characterization Plan

A comprehensive characterization plan was devised for monitoring the chemical composition of LARC-160 at three stages during its manufacture. The monomeric LARC-160 ingredients and their idealized polymerization sequence are given in Figure 1. The three LARC-160 components are BTDE (diethyl ester of 3,3',

4,4' - benzophenonetetracarboxylic acid); the endcap NE (monoethyl ester of 5-norbornene-2,3-dicarboxylic acid); and Jeffamine AP-22, a liquid polymeric amine mixture containing approximately 85% methylene dianiline isomers with the remaining being mostly higher molecular weight tri-, tetra-and penta-functional amines. The commercial preparation of LARC-160 resin is outlined schematically in Figure 2. In the first step, BTDA and NA, the anhydride forms of BTDE and NE, respectively, are refluxed at 82-99°C (180-210°F) with alcohol for a total of approximately 60 minutes to give the BTDE/NE ester mixture. The alcohol used is an industrial mixture composed of 85.8% ethyl, 9.0% isopropyl, and 4.3% methyl alcohol with 0.9% methyl isobutyl ketone. The BTDE/NE ester mixture is a viscous liquid at room temperature containing several percent of unreacted alcohol. Neat resin is prepared in the next step by blending the liquid amine into the BTDE/NE ester mixture at 32°C (90°F). After mixing for five minutes, the material is stored in a freezer. Prepreg is produced in the final step by a proprietary hot-melt impregnation technique.

The chemical characterization plan called for HPLC analysis to be carried out subsequent to the BTDE/NE esters, neat resin, and prepreg stages of LARC-160 production. This approach was expected to help identify the introduction of chemical variations during manufacture and permit possible corrective measures to be taken during production.

3.1.5.2 Variables Study Materials

Many factors contribute to the consistent performance of an advanced composite matrix material. In the Variables Study, the most important LARC-160 formulation and processing variations were assessed by studying a series of modified resin materials prepared by the prepreg supplier. The formulation and process variations incorporated in the intermediate ester batches are described in Table 1. Several standard production batches from the same supplier which were analyzed are also identified. Only five of the intermediate ester Variables Study batches involved

modifications. Batches 7 through 10 contain ± 5% variations in the standard amounts of BTDA and NA normally used. Batch 13 was given a six hour reflux compared to the normal 1.75 hour processing time.

The formulation and process variations contained in the neat resin and prepreg batches is given in Table 2. Batches 1 through 10 contain percentage variations on the formulated weights of amine and anhydrides normally used to prepreg LARC-160. An extended resin cook time of two hours at 79°C (175°F) was used in Batch 11, compared to a normal operation involving about 10 minutes at 60°C (140°F).

Two polymeric amine substitutes for Jeffamine AP-22 were evaluated in Batches 14 and 15. They are Anchamine DL, a product of Pacific Anchor Chemical Corp., and Tonox-22 from UniRoyal. Final Batch 16 represents a control material for the Variable Study, prepared with the standard LARC-160 formulation and under the same processing conditions as used for the other standard processed materials for Variables Study.

Resin Batches 1 and 2 in the Variables Study were rejected and replaced by Batches 1A and 2A, respectively. Original Batches 1 and 2 were rejected due to improper hot melt impregnation procedures. Although original Batches 1 and 2 of the Variables Study were not processed into prepreg, their chemical composition is included for comparative purposes. Included as a second part of the Variables Study was a test of the repeatability of the supplier in producing three standard batches of LARC-160.

3.1.5.3 Liquid Chromatography

The development of a liquid chromatographic separation technique for LARC-160 involved the selection of a type of column absorption material, an eluting solvent or solvent combination, and a mode of detecting the eluting components. The most common type of column packing material was chosen which is the so-called "reverse-phase" material consisting of octadecyl silane bonded 10μm silica spheres. Reverse-phase separations are nearly always carried out with an

aqueous/organic solvent gradient. The presence of both acidic BTDE and NE together with basic amine ingredients results in the pH being a critical factor for good resolution in the separation of LARC-160. Adequate column retention and resolution in a reverse-phase separation requires that component species be maintained in their non-ionic forms by control of pH.

The ionization of carboxylic acid groups on BTDE and NE esters was suppressed by using a pH = 3.00 KH_2PO_4 buffer in the aqueous portion of the solvent gradient. This buffer serves not only to maintain BTDE and NE esters in their neutral forms but also enhances separation by a complex mechanism believed to be based on differences in the dissociation constants for various acidic groups present in BTDE and NE components. The basic amine components are present as protonated cations under the above acidic solvent conditions and are therefore poorly resolved.

A key feature of this technique was the adaption of low ultraviolet wavelength (200 NM) enabling the detection of the NE ester endcap. This in turn required the use of a solvent which was transparent at this wavelength. A water/acetonitrile solvent gradient was found to best fulfill all the requirements. Experimental details of the acidic reverse-phase techniques are given in Table 3.

The analysis of amine components in LARC-160 was made by an ion-pair HPLC method in which tetrabutyl ammonium phosphate is added to the aqueous solvent phase. The tetrabutyl ammonium cations are believed to ion-pair with anionic sample species, neutralizing their charge and improving retention and separation. This technique provides good separation of free amine ingredients in LARC-160, but BTDE and NE ester components are not as well resolved as with the buffered acidic solvent gradient. The experimental parameters used in the ion-pair method are given in Table 4.

3.1.5.4 Ester Analysis

BTDE/NE Ester Mixture

The initial step in LARC-160 synthesis is the co-esterification

of BTDA and NA. The products expected from this process are BTDE diester and NE monoester as shown in Figure 1. The HPLC separation of a BTDE/NE ester batch is shown in Figure 3. The complexity of the ester mixture is the result of several factors. First, the esterification is carried out using an alcohol mixture rather than pure ethyl alcohol. The major products detected are BTDE and NE ethyl esters; however, methyl esters are also present as BTDE monomethyl and BTDE methylethyl mixed esters. Isopropyl alcohol is apparently much less reactive in that none of its esters have been detected.

A second factor contributing to the complicated BTDE/NE ester composition is the manner in which the BTDE/NE ester mixture is prepared. In order to minimize the solvent content, only a small stoichmetric excess of alcohol is used for co-esterification. This results in a highly viscous reaction medium which tends to inhibit complete conversion of BTDA to BTDE diester and results in BTDE monoester and unreacted BTDA. When a large excess of alcohol is used in BTDA esterification, only diesters are produced. NA appears to be a more reactive anhydride than BTDA and is converted to monoester completely during esterification.

The identification of BTDE/NE ester components in Figure 3 was made by comparison with characteristic elution times of reference materials. A synthetic mixture of these BTDE and NE ester compounds was prepared by reacting the two anhydrides under conditions that favor the formation of a particular species. The HPLC separation of this mixture is shown in Figure 4. The isomeric makeup is that expected from the possible theoretical forms shown in Figures 5 and 6, except that three rather than four BTDE diester isomers are observed. The BTDE ortho diester isomer probably does not occur because of the much greater reactivity of the anhydride ring compared to the carboxylic acid groups toward esterification. Endo and exo isomer forms of NE esters were not separated in the present study. These reference ethyl ester compounds, together with others prepared from methyl alcohol and methyl/ethyl mixtures, made

possible the identification of the major BTDE/NE ester components.

BTDE/NE mixtures from the Variables Study, as well as several batches from larger scale standard production runs, were examined by HPLC. The relative concentration of each ester constituent was calculated as the peak area percentage of that component relative to the total area of all the peaks in the chromatogram. Although original Batches 1 and 2 of the Variables Study were not processed into prepreg, their chemical composition is included for comparative purposes.

Neat Resin and Prepreg

The characterization of BTDE and NE esters, ingredients in LARC-160 neat resin and prepreg, was made by the same HPLC method as that for analyzing BTDE/NE ester mixtures. The separation of a LARC-160 neat resin batch is shown in Figure 7. BTDE components and NE ester endcapper are separated, as are a number of resin intermediate compounds. The relative concentration of each BTDE and NE ester ingredient in the resin batches was expressed as its HPLC peak area percentage relative to the total ester peak area of the chromatogram.

The following is a discussion of the variation in the relative concentration of BTDE/NE ester components separated by HPLC as shown in Figure 3.

BTA

One form of the major anhydride ingredient which was detected in LARC-160 was BTA, the tetra-acid of BTDA. The identification of this component is complicated by the fact that if BTDA (anhydride) were present in LARC-160 resins, it would be hydrolyzed immediately to the tetra-acid form by the aqueous HPLC solvent gradient. It is therefore impossible to determine by HPLC whether the anhydride or tetra-acid were present in the resin. Infrared spectra of BTDE/NE ester mixtures, however, show neither of the strong anhydride absorption peaks at 1785 and 1860 cm^{-1} characteristic of BTDA (anhydride). This implies that primarily only the tetra-acid occurs in

the LARC-160 materials. The levels of BTA in the intermediate ester and resin batches examined is shown in Figures 8 and 9. Except for Batches 15 and 16 of the Variables Study only a small amount of BTA is detected in the LARC-160 materials. One significant feature of these data is that the trend in BTA concentration among the samples is the same for intermediate ester, neat resin, and prepreg. This indicates that variations in BTA present at the intermediate ester stage of production are carried through both resin formulation and hot-melt prepregging.

The presence of BTA can affect later prepreg processing in several ways. First, BTA is more acidic than BTDA diester and is thus more likely to form insoluble salts with amine components added in the neat resin manufacturing operation. Salt formation has been reported (Ref. 1) in a similar PMR resin in which the tetra acid, 2,2-bis (3',4'-dicarboxylphenl) hexafluoropropane, and an aromatic amine were utilized. The presence of organic salts in PMR resin could introduce compositional variations and prevent proper prepregging. Furthermore, BTA may also be more reactive than BTDE toward amines in forming polyamide acid which can result in faster build-up of viscosity during processing. The high concentration of BTA in Batches 15 and 16 is believed to be the result of hydrolysis of BTDA by moisture in the air. These two batches were prepared late in the program using the original lot of BTDA which had been opened frequently, allowing exposure to moisture. It is not known for certain whether the excessive levels of BTA are responsible for the poor physical properties of laminates prepared from Batches 15 and 16 in the Variables Study. Replacement materials for these batches prepared with fresh BTDA showed only a small trace of BTA present.

BTDE Monoethyl Ester

Another unexpected form of the basic BTDA ingredient present is BTDE monoethyl ester. The peak area percentage of the second of the two isomeric forms (see Figure 5) is plotted in Figures 10 and 11. The concentration trend in this component is also very similar

between the intermediate ester, neat resin, and prepregs. The concentration variations are also similar to those observed for BTA. This is not unexpected because both BTA and BTDE monoester are the result of incomplete esterification and reaction conditions that promote BTA formation also favor BTDE monoester. This compound, which is also believed to be hydrolyzed, contains the ortho dicarboxylic acid group and could affect resin processing similar to that of BTA described above.

BTDE Diethyl Ester

The major BTA ingredient in the ester mixture is the diethyl ester. The variation in the second of three BTDE diethyl ester isomers separated by HPLC is plotted in Figures 12 and 13. The relative variations in Figure 12 and 13 correspond to different levels of BTA and BTDE monoester described earlier. For example, Batches 15, 16 and 16A contain a lower relative concentration of BTDE diethyl ester due to the greater amounts of BTA and BTDE monoester in these batches of material. The use of relative peak area analysis does not provide very meaningful data for the major component of a system. It works best for detecting relative variations in the concentration of minor ingredients.

NE Monoethyl Ester

The relative levels of NE ester endcap in the LARC-160 intermediate ester mixtures is plotted in Figure 14. In contrast to the variety of BTDA products present in the ester mixtures, the NE endcap occurs predominantly as the monoethyl ester. The relationship between NE ester peak area percentage and initial formulation changes is somewhat ambigious due to the larger variations in BTDA related ingredients. It is found, however, among Variables Study

Batches 1 through 12, which are similar to BTDA related components, that correlations between NA formulation changes and peak area percentages can be made.

The relative levels of NE ester endcap in the LARC-160 resins are plotted in Figure 15. The irregularity in the data does not correlate with NA formulation changes and is believed to be related to resin cook variations. For example, the low level of NE ester in the extended resin cook time material (Batch 11) is a result of the preferential reaction of endcap with amine components to form resin intermediates. The greater reactivity of NE compared to BTDE esters have been discussed (Ref. 2 & 3). It is very difficult, therefore, to determine NE formulation errors by HPLC analysis.

BTDE Triethyl Ester

The extent of BTDE triethyl ester in the BTDE/NE ester batches examined is plotted in Figure 16. Excess ester reflux time in Variables Study batch 13 is indicated by an increased concentration of BTDE triester. The detrimental effects of the ortho ester group of this compound have been described for PMR-15 polyimide resin (Ref. 4). The amount of triester in the LARC-160 materials is, however, very small. BTDE triethyl esters could not be detected in LARC-160 neat resin or prepregs due to interference by ester/amine reaction products which elute at the same time.

3.1.5.5 Resin Intermediates

Polymerization of LARC-160 monomers begins during neat resin preparation and prepregging, as evidenced by the appearance of new resin intermediate peaks in the HPLC separation of neat resin in Figure 7. According to the simplified curing sequence in Figure 1, BTDE ester ingredients first react with amines to form polyamide acids. These species are then endcapped by the NE ester to give the desired molecular weight product. Imidization and cross-linking then occur in subsequent steps at higher temperatures. Contrary to this reaction scheme, the resin intermediates identified in LARC-160 are predominantly NE ester/amine compounds.

The identification of three resin intermediate peaks in the HPLC separation of LARC-160 was made by comparison with elution times of model compounds whose structures are given in Figure 17. The variation in MDA bis-nadimide is plotted in Figure 18 for the LARC-160 resin materials. The effect of extended resin cook time and temperature, 2 hours at 79°C (175°F) vs 10 minutes at 60°C (140°F), is clearly indicated for Batch 11 of the Variables Study. The HPLC separation of this material is shown in Figure 19. A large increase in both MDA mono- and bis-nadimide results from excess resin cook time.

The presence of MDA bis-nadamide and bis-nadimide can affect both the processing and cured physical properties of LARC-160. Cross-linking of MDA bis-nadimide will result in a low molecular weight cross-link with different physical properties compared to the idealized structure shown in Figure 1. The remaining resin, however, will be deficient in endcapper, thus increasing its molecular weight between cross-links. The overall result is to produce a less homogeneous network than is predicted by the idealized polymerization sequence for LARC-160.

3.1.5.6 Unreacted Amines

The remaining ingredients to characterize in the LARC-160 resin system are the free or unreacted Jeffamine AP-22 amine compounds. An ion-pair HPLC technique was developed for this purpose. The separation of a standard production batch of LARC-160 resin by this method is shown in Figure 20. The identification of Jeffamine AP-22 components in the resin was made by comparison with the chromatogram of a pure Jeffamine AP-22 sample shown in Figure 21. Three major components are detected, including MDA, and two higher molecular weight MDA homologs. The relative MDA content was calculated as the peak area ratio of MDA to BTDE diester in the chromatograms. This ratio is plotted in Figure 22 for the LARC-160 batches. The results for Variables Study resin Batches 1-6 agree qualitatively with the Jeffamine AP-22 formulation changes incorporated in those materials. A low ratio is shown for Batch 11 because of its extended cooking time. The wide

variation in the data for the standard batches is most likely due to the effect of B-staging rather than formulation changes.

3.1.5.7 Chemical Characterization Conclusions

This chemical characterization activity of the program demonstrates the applicability of HPLC techniques for characterizing the chemical composition of LARC-160 polyimide resin. Monomeric ester and amine ingredients, as well as a number of resin advancement products, are detected and identified by HPLC analysis. These techniques not only provide the basis for improved quality control procedures but also reveal information regarding the mechanism of LARC-160 polymerization. Some specific conclusions resulting from this study are:

- Relative batch-to-batch chemical variations in BTDA in NA ingredients can be detected precisely, but absolute formulation deviations are difficult to determine because of the complexity of ester products and the effects of varying resin advancement composition.
- Resin B-staging or advancement caused by processing variations can be monitored from relative concentration of intermediate products formed.
- Complex conversion of BTDA to BTDE diester during esterification does not always occur, resulting in potentially detrimental BTDE monoesters and tetra acid (BTA) products.
- High levels of BTA can also result from hydrolysis of BTDA by exposure to air prior to formulation.
- NE ester endcap reacts preferentially with amine components, producing the bis-nadimide resin intermediate that modifies the ideal cured network structure.

- o Only a slight trace of BTDE triethyl ester is present in the resin materials examined.
- o Free amine components present in neat resin and prepreg can be characterized by an ion-pair HPLC method.
- o The chemical composition of neat resin and prepreg are quite similar, indicating that hot-melt impregnation has little affect on composition.

Based on the foregoing conclusions and observations made during the program, the following recommendations are given:

- o Preparation of BTDE/NE intermediate esters should be modified to permit complete conversion of BTDA to BTDE diester.
- o Pure methyl or ethyl alcohol should be considered for esterification. The use of a complex alcohol mixture introduces components whose behavior is unknown. The use of a pure alcohol would also greatly simplify the quality assurance of LARC-160.
- o Care must be taken to prevent exposure of BTDA to moisture in the air which results in hydrolysis to the tetra-acid.
- o The HPLC methodology needs to be made more reproducible. It is presently too difficult to obtain consistent data from one analysis to another.
- o Improved quantification of the HPLC methodology is required. The use of relative peak area ratios is not satisfactory for analyzing all ingredients in LARC-160.

3.1.6 LARC-160 Variables Study

This portion of the program was incorporated by contract modification with the objective (1) of establishing the limits within which LARC-160 polyimide resin could vary with regard to formulation and processing without detriment to prepreg quality and (2) to demonstrate prepreg batch-to-batch repeatability.

3.1.6.1 Formulation and Process Variables

Table 5 presents the variables in formulation and processing assessed. All resin formulation and processing and prepreg production was conducted under laboratory conditions primarily to better control the variable requirements and also because of the 1.4 Kg (3 lb) prepreg batch requirement which would have been cost prohibitive if accomplished on a production line. The prepreg tape produced was 15.2-cm (6 in.) wide.

A total of sixteen prepreg batches were produced for this study. Batches 1 through 10 varied resin stoichiometry, batches 11 through 13 varied processing, and batches 14 and 15 substituted Anchamine-DL and Tonox 22 respectively for the Jeffamine AP-22. Batch 16 was utilized for chemical standardization since all other standard batches of prepreg used for this program were produced on a production line because of the quantity required.

Chemical analyses were conducted on each of the 16 batches of prepreg material produced (see 3.1.5). Analysis of the starting materials, NA, BTDA, and AP-22 was done only once since these materials were procured in large quantities. The intermediate ester, neat resin, and resin extracted from the prepreg of each batch were chemically analyzed as noted in Table 5.

Laminate panels 15.2x15.2-cm (6x6 in.), 14 ply unidirectional, were fabricated from each batch of prepreg material produced for the variable study using the two stage imidizing and cure process (see 3.2.1.2). The laminate panels were postcured free-standing for four hours at 316°C (600°F) in an air circulating oven.

Laminate Physical Properties

NDI C-scan tests resulted in 100% transmission through all resin stoichiometry and processing variable panels except EX207, resin variable number 5. Panel EX207 had a 10% excess concentration of Jeffamine AP22 and showed 40% transmission. The two panels with amine components, Anchamine-DL and Tonox 22, showed 70% and 0% transmission respectively. NDI C-scan recordings are shown in

Figures 23 through 38.

Target fiber volume of 60 ± 2% was achieved in five of fifteen panels fabricated. Low and high fiber volumes in the remaining panels is attributed to prepreg resin content inconsistencies within each laboratory scale tape roll. Detailed composite physical properties are presented in Table 6.

Laminate Mechanical Properties

Flexural and short beam shear (SBS) properties were determined on each postcured panel at room temperature and 316°C (600°F). High flexural strength and modulus and short beam shear strength was achieved in all specimens tested with the exception of panel EX219 which employed the Tonox 22. Detailed properties are presented in Table 6.

Laminate TMA-Tg Properties

Figure 39 shows a plot of Tg temperature determined for laminates as a function of Jeffamine AP-22 concentration. The NE and BTDE quantities were held constant at the standard formulation concentrations. Although the number of data points is limited, and only one determination per laminate sample was made, the plot indicates that Tg increases with increasing AP-22 concentration up to +10%. Data were too limited with respect to variable NE/BTDE concentrations to determine a trend in Tg temperature variations.

Significantly, panel EX 219 (Tonox 22) yielded a higher Tg than all other laminates. Individual TMA-Tg curves are presented in figures 40 through 43.

3.1.6.2 Repeatability and Usability

Three 4.5Kg (10 lb) batches (23723, 23725, and 23727) of 30.4cm (12 in.) wide unidirectional tape were produced under production conditions with separate batches of resin formulated for each 4.5 Kg (10 lb) of prepreg to demonstrate formulation repeatability and material usability over a six month period at -18°C (0°F) storage and after ambient out-time exposure of seven days.

The resin batches were formulated within limits established from evaluating batches of prepreg 1 through 13 which varied in stoichiometry and processing. Formulation and processing limits for the three batches were as follows:

AP-22 $\pm$ 2.5% by weight
NA $\pm$ 2.5% by weight
BTDA $\pm$ 2.5% by weight

Reflux time - not to exceed 90 minutes at 82 $\pm$ 3°C (180 $\pm$ 5°F)

Cook time - not to exceed 115 minutes at 82 $\pm$ 3°C (180 $\pm$ 5°F)

Prepreg material physical properties specified were:

Resin solids: 37 $\pm$ 3%
Volatiles: 12 $\pm$ 3%
Fiber Areal Weight: 134 $\pm$ 3 grams/m^2

Samples of the neat resin and intermediate ester were provided with each batch of prepreg for HPLC analysis to determine repeatability and establish a standard for comparison of resin extracted from the prepreg during the storage period.

Although this phase of the variables study was to be completed in a six months period, equipment schedule priorities, equipment malfunctions, and unexplainable processing problems have not allowed a clear definition of usability after six months storage. Equipment priorities prevented laminates from being processed at the end of one month storage and laminates representing two months storage were not usable because of an autoclave malfunction. At the end of four month's storage, laminates were processed. However, these laminates were not deemed usable because of excessive resin bleed and fiber washing.

Chemical analysis of resin extracted from each of the three repeatability batches showed no marked difference to the samples of

neat resin supplied with each batch. A detailed examination of the prepreg supplier's records for the repeatability batches also showed no possible cause for the processing difficulties. Further chemical analysis by Rockwell did not disclose any extreme differences between the repeatability batches and other batches used successfully to date.

After eight months storage at -10.8°C (0°F), two 15.2x15.2-cm (6.0x6.0 in.), 14 ply unidirectional laminates, laid up from each of the three Celion/LARC-160 repeatability batches were autoclave cured.

Prior to curing, the laminates were imidized: one from each prepreg batch at 190°C (375°F) and the remaining laminates at 207°C (405°F). Imidization staging at the noted temperatures was conducted under $< 16.9 KN/m^2$ (5 in. Hg) vacuum bag pressure for one hour. The higher imidizing temperature was used, in an attempt to reduce resin flow during the cure process.

The laminates were autoclave cured for two hours at 316°C (600°F) under full vacuum and augmenting pressure of $1379 KN/m^2$ (200 psi). Total pressure was applied at the start of the cure and cure temperature was attained at the rate of 2.2°C (4°F)/minute. The cured laminates were forced cooled to $< 66°C$ (150°F) before release of pressure. The cured laminates exhibited surface and edge fiber washing. However, this condition was to a lesser degree for those laminates imidized at the higher temperature. NDI C-scan, "A" sensitivity, of the laminates showed five of the six having minor voids to a maximum of 3% of laminate area.

Flexural properties of the laminates from one batch (23723) were $1476 MN/m^2$ (214 ksi) at room temperature and $786 MN/m^2$ (114 ksi) and $765 MN/m^2$ (111 ksi) at 316°C (600°F) for laminates imidized at 190°C (375°F) and 207°C (405°F) compared to target values of $> 1572 MN/m^2$ (228 ksi) and $> 938 MN/m^2$ (136 ksi) respectively for the test temperature conditions. The remaining two batches (23725 and 23727) provided flexural strengths in the range of 1862 to $1924 MN/m^2$ (270 to 279 ksi) at room temperature and 931 to $1282 MN/m^2$

(135 to 186 ksi) at 316°C (600°F). Short beam shear values for the three batches were essentially equivalent: 110 to 122MN/m^2 (16 to 17.7 ksi) at room temperature and 42 to 61 MN/m^2 (6 to 8.8 ksi) at 316°C (600°F) compared to target values of $>$ 103 MN/m^2 (15 ksi) and $>$ 48 MN/m^2 (7 ksi) respectively for the test temperature conditions.

Two sets of 15.2x15.2-cm (6x6 in.) 14 ply unidirectional laminates were fabricated from each of the three repeatability batches after the material had been stored for eleven months. The laminates were imidized at 218C (425F) for 30 minutes at two pressures 16.9 KN/m^2 (5 in. Hg) and 6.7 KN/m^2 (2 in. Hg). This change is processing, over that used for the prior specimens, was in keeping with the improved processing developed for the program.

Fiber washing was evident after imidizing to approximately the same extent as seen on prior laminates made from these material batches. However, it is felt this would have been controlled by the use of edge dams.

The laminates were autoclave cured for three hours at 329°C (625°F) under full vacuum and augmenting pressure of 1379 KN/m^2 (200 psi). The laminates were not postcured. All laminates showed excellent C-scan quality as shown in Figures 44 through 49.

Mechanical properties of these laminates were not determined because of variable thickness caused by the fiber washing. However, specimens were taken from each laminate for determination of fiber volume and Tg. Results of these determinations are as follows:

Batch	Imidizing Pressure	Fiber Volume (%)	Tg
23723	16.9 KN/m^2	66	343°C
	6.7 KN/m^2	63	349°C
23725	16.9 KN/m^2	65	340°C
	6.7 KN/m^2	64	341°C

Batch	Imidizing Pressure	Fiber Volume (%)	Tg
23727	16.9 KN/m^2	70	343^oC
	6.7 KN/m^2	67	339^oC
	Target	60 ± 2	340^oC (after postcure)

Further effort on this particular study was impossible because of fabrication of the Technology Demonstrator Segment, Task (h), which was of higher priority. However, batch repeatability was demonstrated by the repetitive chemical analysis conducted in attempting to determine a possible cause for the "processing" problem.

With regard to usability after storage, experience with production materials used for this program has indicated no problem with materials that have been kept at -10.8^oC (0^oF) for over six months.

3.2 TASK (b) - DEVELOP PROCESSES

Processing of Celion/LARC-160 was developed for fabrication of laminates, hat and "I" stiffeners, honeycomb sandwich panels, and chopped fiber moldings. Primary effort was devoted to developing process procedures for laminate structures which provided the basis for process development of other structure variations.

3.2.1 Laminate Processing

Three laminate processing procedures were evaluated during this program: (1) in situ cycle, imidizing and cure, (2) two stage cycle, imidizing and cure, and (3) improved two stage cycle, imidizing and cure.

3.2.1.1 In Situ Cycle

The objective of the in situ cycle was to accomplish imidization and cure in one continuous operation. The in situ cycle is shown schematically in Figure 50 and is described as follows:

- o Apply 6.7 KN/m^2 (2 in. Hg) vacuum and maintain throughout cycle.
- o Raise temperature from RT to 163°C (325°F) at 1.7 to 2.8°C (3 to 5°F)/minute.
- o Hold for 1 hour at 163°C (325°F).
- o Raise part temperature from 163°C (325°F) to 329°C (625°F) at 1.7 to 2.8°C (3 to 5°F)/minute.
- o Apply 1378 KN/M^2 (200 psi) pressure at 274°C (525°F).
- o Cure at 329°C (625°F) for 2 hours.
- o Force cool to $<$ 149°C (300°F) prior to pressure release.
- o Postcure free-standing in an air-circulating oven. Raise temperature from RT to 316°C (600°F) at 5°C (9°F)/minute. Hold at 316°C (600°F) for 4 hours. Force cool to RT.

The tooling/layup configuration for the in situ cycle is shown in Figure 51. A limitation to this cycle was the inability to control resin flow resulting in higher than target fiber volume (60 $\pm$ 2% laminates.

Concurrent with the development of the in situ cycle, studies were conducted to evaluate minimum pressure/minimum temperature cure conditions.

Minimum Pressure Cure Study

Celion/ LARC-160 unidirectional, 32 ply laminates 12.7-cm (5 in.) wide by 11.5-cm (4.50 in.) long were prepared at cure pressure levels of 1.38 MN/m^2 (200 psi), 1.03 MN/m^2 (150 psi), .69 MN/m^2 (100 psi) and .34 MN/m^2 (50 psi). The in situ cycle time/temperature profile and pressure application point of 274°C (525°F) was employed in autoclave molding the laminates and final cure was accomplished at 329°C (625°F) for two hours using standard tooling. Panels were postcured at 316°C (600°F) for four hours. As indicated by NDI C-scan, "A" sensitivity, tests and actual void volume measurements, .69 MN/m^2 (100 psi) was the lowest molding pressure that produced $<$ 1% target void volume laminates. Panels

EX41 1.38 KN/m^2 (200 psi), EX47 1.03KN/m^2 (150 psi) and EX48 .69 KN/m^2 (100 psi) NDI C-scan, "A" sensitivity, recordings showed near 100% sound laminates with only two discrepant areas $\leq$3.2-mm (1/8 in.) diameter showing on EX48. NDI C-scan recordings of postcured laminates are shown in Figures 52 and 53. Therefore, the minimum pressure limit for processing Celion/LARC-160 prepreg appears to be somewhere between .69 MN/m^2 (100 psi) and .34 MN/m^2 (50 psi) since the 34 MN/m^2 (50 psi) pressure molded laminate EX49 showed total porosity in C-scan test and has a 6.4% void volume.

Significantly, panel EX 48 cured at .69 MN/m^2 (100 psi) had an optimum fiber volume of 60.2%, compared with panels cured at 1.38 MN/m^2 (200 psi) and 1.03 MN/m^2 (150 psi) which had fiber volumes of 69.9% and 67.7% respectively.

Composite physical, flexural and short beam shear properties of panels autoclave molded at 1.38 MN/m^2 (200 psi), 1.03 MN/m^2 (150 psi), .69 MN/m^2 (100 psi), and .34 MN/m^2 (50 psi) are compared in Table 7. Data indicated no definite trend related to flexural strength or elastic modulus properties in comparing cure pressure levels. Short beam shear strength, 316°C (600°F) properties, however, in all specimens cured at 1.03 MN/m^2 (150 psi), .69 MN/m^2 (100 psi), and .34 MN/m^2 (50 psi) fell slightly below the 48.2 MN/m^2 (7 psi) target level.

Minimum Cure Temperature Study

A study was conducted to determine the practical minimum temperature/time cycle for curing Celion/LARC-160 prepreg. This study was undertaken in an effort to take advantage of the possible use of reusable silicone rubber vacuum bags and/or premolded pressure caul sheets. These bags and/or caul sheets would be particularly advantageous in fabrication of hat and "I" beam stringers, sine wave ribs, and other complex shaped parts. Reusability is of prime importance for cost effectiveness. In addition, lower curing temperatures will reduce the chances of seal and bag

leakage.

Fabrication of unidirectional, 32 ply 11.5x12.7-cm (4.5x5.0 in.) laminates was accomplished using the two stage Celion/LARC-160 prepreg cure cycle and tooling with 1.38 MN/m^2 (200 psi) pressure applied at 274°C (525°F). Cure cycles employed to bracket temperature/time limits are described as follows: 316°C (600°F) 2 hours; 302°C (575°F) 3 hours; 288°C (550°F) 4 hours; and 274°C (525°F) 5 hours. Postcure of all laminates was accomplished at 316°C (600°F for four hours.

NDI C-scan, "A" sensitivity, tests on laminates EX74, EX69, EX70, EX71, EX72 cured at respective temperatures of 329°C (625°F), 316°C (600°F), and 302°C (557°F) and 274°C (525°F) had 99.5% sound laminate areas after final postcure at 316°C (600°F) for four hours. C-scan recordings of postcured laminates are presented in Figures 54 and 55.

As cured laminate TMA-Tg values gradually decreased with decreasing cure temperature. For example laminate EX74 cured at 329°C (625°F) had a Tg of 346°C (655°F) and EX72 cured at 274°C (525°F) had a Tg of 257°C (495°F). All laminates, however, after being postcured at 316°C (600°F) for four hours had essentially equivalent Tg values, with panel EX72 having the highest value of 360°C (680°F). TMA-Tg recordings are presented for comparison in Figures 56 through 60.

Composite physical and mechanical properties are presented in Table 7. Composite fiber volume decreased with decreasing cure temperature, from 68.4% for panel EX74, cured at 329°C (625°F) to 62.1%, for panel EX72, cured at 274°C (525°F). No definitive trend was noted in normalized room temperature flexural strengths and none of the panels achieved minimum target values. All panels except EX70, cured at 302°C (575°F) achieved target 316°C (600°F) strength. Room temperature and 316°C (600°F) short beam shear target strengths were achieved on all panels.

3.2.1.2 Two Stage Cycle

The two stage cycle was developed to improve resin flow control not obtainable by the in situ process. The following fully describes the two stage cycle including layup and debulking procedures.

Prepreg Tape Layup Procedure

Stack prepreg tape in the required ply orientation and number of plies, paper backing surface up, on a smooth tooling surface such as a glass plate. Supporting layup materials such as parting films, bleeders, porous teflon coated 104 fiber glass (TX1040 or 3TLL), caul plate, breathers and vacuum bag are shown in the layup sequence in Figure 61.

During layup, it is preferable to slice the edges of the tape using a straight edge in order to remove irregularities. If the prepreg tape edges are cut clean and uniform, this operation can be omitted. Normally, the Celion/LARC-160 tape will have adequate tack to adhere to itself, however, if tack is not adequate, a hot iron may be employed to aid layup. This is accomplished by ironing directly over the paper backing; either locally tacking or flat ironing to adhere the plies. The iron heat setting should be in the range of 176°C (350°F). Great care shall be exercised in insuring either absolute butt joints or a slight overlap of 0.76-mm (0.03 in) in the layup of tape elements. Tow splices should be flagged on the tape roll by the supplier, however, extreme care shall be taken to visually inspect each tape element. Any sections having tow splices must be removed.

Debulking Procedure

Debulking of stacked prepreg has been found to be advantageous in fabricating both flat and complex shaped laminates for the following reasons:

- Preconsolidation debulks the prepreg close to final laminate thickness; therefore, when augmented pressure is applied during final cure, less resin and fiber movement is required to achieve ultimate thickness.

- During the debulking operation, supporting materials such as TX1040 and bleeders are adhered to the laminate stack, forming a well consolidated unit that is easily handled during subsequent operations.
- Prestacked and debulked laminates are easily stored, under refrigeration, in sealed bags.
- The preconsolidated preforms, with bleeder materials in-place, are easily handled in vacuum forming operations during the fabrication of hat, "I", "Pi" and other complex shape elements.
- Where dry prepreg is used in the layup, the debulking operation not only consolidates and adheres the materials but also rejuvenates the resin tack, making the total stack pliable.

Two approaches to the flat laminate debulking operations have been developed and successfully qualified for use in Tasks (a), (b), (c), (d), (e), and (f).

Dry Preg Debulking

The following operations are to be performed after completing prepreg tape layup.

- Select a flat 4.6 to 6.34-mm (0.18 to 0.25 in.) thick aluminum caul plate the same size as the stacked layup and apply Frekote 33 mold release to the caul surface which will face the layup.
- Preheat the caul plate to 127 $\pm$ 6.6°C (260 $\pm$ 12°F).
- On removal from the oven (within 2 - 3 minutes), immediately assemble the caul plate to the stacked layup surface, seal in a vacuum bag and apply vacuum to 67 KN/m^2 (20 in. Hg).

  Note: The layup area shall have been previously prepared for rapid vacuum bag application so that minimal heat loss is incurred from the hot caul

plate. This operation softens the LARC-160 resin and causes it to flow into the TX1040 and bleeder materials, creating a consolidated prepreg/bleeder preform. The dry prepreg stack will become mildly tacky and pliable after this operation.

- o Allow the assembly to stabilize to room temperature before removing the vacuum bag.

Tacky Prepreg Debulking

The same procedures shall be employed as for dry prepreg except that heating of the caul plate is optional. If heat is used, the procedure described for dry prepreg shall be followed. The layup shall be debulked at room temperature under vacuum bag pressure, <67 KN/m^2 (20 in. Hg) for a minimum of four hours.

Imidizing Procedure

Principal concerns with the LARC-160 polyimide resin/graphite materials are ensuring (1) efficient, uniform removal of solvent and condensation reaction volatiles from large and complex surface areas, and (2) resin flow control in the composite prior to application of augmented pressure during the cure cycle.

Prepreg volatile removal techniques were developed using the tooling shown in Figure 62. The concept of uniform removal of volatiles is based on use of a perforated layup tool surface. Perforations in the surface act as individual, unrestrained vacuum ports serving local surface areas of about 6-cm^2 (1 in.^{2}). Vacuum channel separator strips are located in-line between the vacuum ports to support the caul layup surface and to provide an unrestrained venting system to a central manifold that, in turn, leads to the main vacuum source.

The laminate preform is imidized on porous tooling shown in Figure 62. The laminate is contained in a Celgard 4500 or 4510 polypropylene microporous membrane which allows removal of volatiles through the bottom perforated caul plate while preventing

resin loss into surrounding breathers. Volatiles are reduced to $< 3\%$ by this procedure. The imidizing cycle is shown in Figure 63.

A perforated top pressure caul without vacuum channels may be used rather than a perforated bottom plate, if desired, although this concept has not been proved for larger, thicker, laminates. If the top perforated caul is used, efficient methods for venting volatiles to the vacuum source must be employed.

Detailed Imidizing Procedure

- Apply one 7781 fiberglass breather ply and one layer of Celgard 4500 or 4510 to the surface of the perforated layup plate as shown in Figure 62. Celgard may be spliced with an electronic heat sealer or with a thin band of Kapton tape 6.35-mm (0.25 in.) wide.
- Transfer the debulked preform and the integral bleeders from the layup tool to the prepared perforated layup plate.
- Assemble all bagging components per Figure 62 and install thermocouples in the trim edge of the part.
- Install the assembly in an air-circulating oven and perform the imidizing cycle per Figure 63. Thermocouples placed within the part trim, not oven temperature, shall be used in controlling the imidizing cycle. Thermocouple data shall be autographically recorded.

Cure Procedure

Imidized flat laminates are autoclave cured on tooling shown in Figure 64. Since the laminate volatile content has been reduced to $< 3\%$ during the imidizing procedure it can now be treated analogous to an epoxy laminate in the cure process. Nonperforated cauls are employed with the bleeder arrangement shown in Figure 64. The autoclave cure cycle is shown in Figure 65.

Process development, performed for Task (b) and verified in Tasks (a), (b), (c), (d), (e), and (f), has shown that considerable latitude exists in cure levels. For example, ultimate cure temperature and pressure can be accomplished as low as 274°C (525°F) and 689 KN/m^2 (100 psi) respectively. Minimum cure temperature has been set at 287°C (550°F) and pressure 1378 KN/m^2 (200 psi).

Autoclave pressurization rates evaluated have been in the 5 to 7 minute range from 0 to 1378 KN/m^2 (0 to 200 psi). The pressure application window appears to be optimum in the temperature range of 274 to 287°C (525 to 550°F). Resin hot melt flow is in the range of 254 to 265°C (490 to 510°F), however, the apparent viscosity is very low. If pressure is initiated before 274°C (525°F) excessive resin losses have been realized, resulting in high fiber volume laminates.

Laminates are postcured at 316°C (600°F) for four hours in an air circulating oven in free-standing position. Postcuring studies during Task (b) proved that the composite Tg is significantly increased and mechanical properties at 316°C (600°F) are improved. Regardless of Tg or mechanical properties values attained after initial cure, postcured of all laminates has been specified to insure retention of laminate quality after exposure to the operating service temperatures.

Detailed Autoclave Curing Procedure

- Remove the Celgard membrane from the bottom surface of the laminate, being careful not to damage or disturb the imidized layup. Leave the 2.3 to 4.8-mm (0.09 to 0.18 in.) top pressure caul in place; do not remove bleeder.
- Transfer the laminate to the steel curing tool (nonperforated surface). The steel tool surface shall be prepared using Frekote 33 parting agent. A Kapton glide sheet shall be employed to separate the laminate from the tool surface. Tape all components in place with Kapton pres-

sure sensitive tape.

- Install type 162 fiberglass breathers (or equivalent) over the aluminum pressure caul and onto the tool surface. Adequate breather material shall be placed between the part and vacuum source to insure efficient removal of any residual volatiles.
- Install thermocouples into the edge of the part. These thermocouples shall be used in monitoring the recording time/temperature data during cure and shall be used to control the cure cycle.
- Install a 0.051-mm (0.002 in.) thick Kapton film bag over the breather and tool surface and seal around the periphery of the tool using a high-temperature sealant. Insure that no bridges exist or sharp protrusions bear against the vacuum bag. Make vacuum bag "ear" seals as required to insure adequate bag slack to prevent bridging.
- Install the steel clamping ring and secure with bolts around the periphery of the tool. The complete tooling arrangment is shown in Figure 64.
- Install the tooling in an autoclave and apply full vacuum to the tooling system. Apply 689KN/m^2 (100 psi) to the autoclave and inspect the system for leaks. If a vacuum leak greater than 33.6 KN/m^2 (10 in. Hg) occurs within five minutes, the source shall be located and repaired.
- Perform the cure cycle within the time temperature profile of Figure 65. The heat up rate may be less than the 3.9°C (7°F)/min. as shown depending on autoclave capability and/or tooling mass. No cool down rate is specified as this also depends on autoclave/tooling characteristics. Record all events such as application of various levels of vacuum, pressure and autographically record temperature from each thermocouple on parts and

autoclave. The part thermocouples shall be used in controlling the cure cycle. All part temperatures shall be in the range of 274 to 287°C (525 to 550°F) when 1378 KN/m^2 (200 psi) autoclave pressure is applied.

- o Remove bleeder materials and clean up parts.
- o Submit for NDI C-scan test.

Postcure Procedure

Postcure in an air-circulating oven by raising the oven and part temperature from room temperature to 316°C (600°F) at an average heat rise rate of 1.6 to 8.3°C (3 to 15°F)/minute and hold at 316°C (600°F) for four hours.

3.2.1.3 Improved Two Stage Cycle

Process studies, initiated under NASA/LaRC Contract NAS1-15183, were further developed under this program to simplify the imidizing and autoclave cure cycles. Prepreg tape layup and debulking procedures are as defined in 3.2.1.2. The objective of this effort were as follows:

- o Reduce the number of steps in the imidizing cycle.
- o Increase the prepreg preform imidizing temperature and/or time at temperature, thereby increasing the LARC-160 resin viscosity when hot melt occurs during the cure cycle. This would allow application of 1378 KN/m^2 (200 psi) pressure at the initiation of cure at room temperature and, in turn, eliminate the chance of error of pressure application in the temperature range of 274 to 287°C (525 to 550°F) with the existing cure cycle. Allowing the resin to seek its normal flow point while under constant pressure, also eliminates problems related to temperature non-uniformity due to varying part thickness or tooling mass.
- o Eliminate the intermediate 163°C (325°F) steps in the autoclave cure cycle by raising the temperature from

room temperature to final cure temperature within the established heat rise rate band.

Twenty-five panels, approximately 15.2x15-2-cm (6x6 in.) were fabricated and imidized as noted in Table 8. All panels were autoclave cured under the same bag with 1378 KN/m^2 (200 psi) pressure applied at the start of the cure. Actual heat rise rate for the cure cycle averaged 1.3°C (2.3°F)/minute. Panels were submitted for NDI C-scan testing and specimens removed for physical properties tests.

The following observations were made on panels cured using the improved cycle:

- High resin flow was noted on all panels imidized at 162 and 177°C (325 and 350°F) as indicated by saturation of surrounding fiberglass breather layers. Panels imidized at 191 and 199°C (375 and 390°C) showed excellent compaction characteristics and good resin beading at laminate edges. The panels imidized at 218°C (425°F) had minimal evidence of resin flow. The panels imidized for 30 and 60 minutes had overall excellent cosmetic appearance while those imidized for longer periods at 218°C (425°F) showed surface roughness discrepancies indicating inadequate resin flow.
- NDI C-scan testing results showed all panels imidized at 162°C (325°F) for time periods of 60, 90, 120, 150, and 180 minutes had void area discrepancies ranging between 40 and 80%. Void areas increased with lesser time at imidizing temperature. Panels imidized at 177°C (350°F) showed ultra sound penetration improvement starting at 150 minutes with approximately 7% void area showing. Panels imidized at 191°C (375°F) starting at 60 minute thru 150 minutes showed 100% ultra sound transmission except for some cellulose acetate fiber splice voids. All panels imidized at 199 and 218°C (390 and 425°F) had 100% ultra sound transmission.

As previously noted, panels imidizing at 218°F (425°F) for time periods exceeding 60 minutes showed surface roughness irregularities due to resin flow reduction.

NDI C-scan recordings of each panel evaluated are presented in Figures 66 through 90.

- Physical properties testing results verified imidizing time/temperature relation as observed in panel cosmetic appearance and NDI C-scan tests. All panels imidized at 163 and 177°C (325 and 350°F) had high fiber and void volumes. Panels imidized at 199 and 202°C (395 and 390°F) for time periods between 30 and 150 minutes achieved target fiber volume of 60 $\pm$ 2% and void volume $<$ 2%. Those panels imidized at 218°C (425°F) for 30 minutes also achieved target requirements. Panels imidized at 218°C (425°F) for longer time periods had low fiber volumes; void volumes were $<$ 2%. Detailed physical properties are presented in Table 9.

Results of this process improvement study indicated the best imidizing time/temperature bands to be in the range of 191°C (375°F) for 60 to 150 minutes, 199°C (390°F) for 30 to 150 minutes and 218°C (425°F) for 30 to 60 minutes.

The improved two stage cycle, employed in fabricating elements for the Technology Demonstrator Segment (TDS), Task (h), is presented schematically in Figure 91. The cure cycle permits temperature options based on tooling requirements e.g. formed silicone rubber blankets utilized on complex contoured details. Postcure time at temperature varies from four hours at the lower temperature to two hours when cured at 329°C (625°F).

3.2.2 "I" Stringer Processing

The following process description is specific for the "I" stringer design developed for this program. It is, however, applicable to the fabrication of any "I" stringer element with minor modifications.

Stock for the individual components of the "I" stringer ("C" channels, caps and radius fillets) are laid up and debulked as flat laminates per 3.2.1.2 and then vacuum formed and imidized. Vacuum forming of "C" channel and bottom surface top cap flat preforms enables easy, wrinkle-free shaping of components.

Preformed imidized radius fillets, that fill the interstices between "C" channels and caps to near net shape, and cap elements are easily handled and located in position during assembly for autoclave cure.

3.2.2.1 "I" Stringer Tooling

The following tools were required for the fabrication of the "I" stringer designed for this program:

- o "C" channel mandrels, 6061-T6 or equivalent aluminum alloy.
- o Cap forming silicone rubber pressure caul stabilized on an aluminum bar.
- o Imidizing tool consisting of a caul plate and perforated layup plate.
- o Preforming and imidizing tool for fillet stock.

Tools are shown in Figures 92 and 93.

3.2.2.2 Layup and Debulking

Layup flat stock for "C" channels per design requirements. Follow the layup and debulking procedures described in 3.2.1.2. During the layup procedure, the TX1040 and bleeder materials applied to "C" channels must be laid on a 45° bias to the rectangular flat preform. This is required to prevent wrinkles during preforming and imidizing operations.

3.2.2.3 Vacuum Forming "I" Stringer Elements

The "I" stringer elements; "C" channels, bottom surface top cap, and fillets are vacuum formed prior to imidizing.

Bottom Surface Top Cap

- Apply Frekote 33 parting agent to the aluminum "C" channel mandrels surfaces and oven dry for 15 minutes at 176°C (350°F).
- Assemble two "C" channels together with attachment bolts.
- Cut a 3.5-cm (1.48 in.) wide, 96.5-cm (38.0 in.) long strip of debulked $(0)_7$ ply prepreg stock. Remove TX1040 and bleeder ply.
- Assemble the $(0^\circ)_7$ layup to the "I" beam mold top cap surface, between two edge dams as shown in Figure 92.
- Place the silicone rubber surfaced aluminum pressure caul in place over the layup.
- Apply a breather and nylon film vacuum bag, seal and place in an autoclave. Apply $\geq$ 84 KN/m^2 ($\geq$ 25 in. Hg) vacuum and 689 KN/m^2 (100 psi) pressure. Raise the temperature to 65°C (150°F) and hold for 15 minutes. Force cool to room temperature.
- Remove the tooling from the bag.
- Cut the preformed laminate along the center line of the cap cleavage to separate the two mandrels. The cap $(0^\circ)_7$ preforms will adhere to each "C" mandrel after this operation. These remain in place for vacuum forming the "C" channels. Remove TX1040 from laminate.

"C" Channels

- Prepare a flat plate of suitable size for holding each "C" channel mandrel in a vacuum bag. Vacuum bag materials and seals shall be prepared previously so that a rapid seal can be made in the subsequent vacuum forming operation.
- Cut a 12.7-cm (5 in.) wide, 96.5-cm (38.0 in.) long strip of debulked ($\pm$ 45°), 4 ply prepreg stock. Remove

the TX1040 faying with the laminate surface facing the tool.

- Place the mandrel on the flat plate. Transfer the debulked laminate with integral 45° bias TX1040 and one ply 120 bleeder to the mandrel and secure in place on the ends with a small piece of tape.
- Drape the vacuum bag over the flat-debulked laminate on the mandrel, seal and draw vacuum. This operation will form the laminate with TX1040 and bleeder in place, over the "C" channel, wrinkle-free.
- Place the assembly in an air-circulating oven and raise the mandrel and part temperature to 65 to 93°C (150 to 200°F) and hold for 15 minutes.
- Allow the assembly to return to room temperature before releasing vacuum. The laminate preform, with integral 45° bias bleeders and porous TX1040 separators, will remain secure to the mandrel and is now ready for imidizing operations.

3.2.2.4 Imidizing "I" Stringer Elements

Imidizing of all "I" stringer elements was accomplished as described in 3.2.1.2. The following defines preparations prior to imidization of formed elements, "C" channels and fillets.

"C" Channels

- Drape a layer of Celgard 4500 or 4510 microporous membrane over the preform bleeder surface and secure in place, wrinkle-free, with pressure sensitive tape on the backside of the mandrel. Celgard is used to contain the resin and release volatiles during imidization.
- Place the two "C" channel mandrels on a flat plate suitable for applying a vacuum bag.
- Install thermocouples under the breather material over the part, outside trim lines. Data from the thermo-

couples shall be autographically recorded and used as the basis for controlling the imidizing cycle.

- Drape one ply of type 120 or 7781 fiberglass or Mochburg paper breather material over the Celgard film surface.
- Install a nylon film vacuum bag, drape in place over the preform bleeder surface and seal around the periphery of the flat aluminum plate. Insure that an efficient breather system such as multi-plies of type 162 fiberglass breather are connected between the parts and vacuum source. The "C" channel imidizing arrangement is shown in Figure 92.
- Place the bagged assembly in an air circulating oven and imidize per Figure 63. Monitor and record thermocouple and other cycle events such as vacuum data.

Fillets

The fillet imidizing tooling and molding concept is shown in Figure 93. The tooling is designed to augment vacuum bag pressure through a pressure augmenter plate to a maximum of 710 KN/m^2 (103 psi) when 101 KN/m^2 (30 in. Hg) vacuum is applied. This feature produces well defined and consolidated imidized fillet preforms.

- For each fillet preform, cut a 6.35-mm (0.25 in.) wide strip from the debulked fillet stock, remove TX1040 and bleeder, and place the strip in the fillet preform tool cavity.
- Install dams and pressure mandrels, thermocouples, pressure augmenter plate, breathers, vacuum bag, and seal per Figure 93.
- Place the assembly in an air circulating oven and imidize per Figure 63. Thermocouple data shall be used for controlling the imidizing cycle and shall be

recorded autographically.

3.2.2.5 Assembling of "I" Stringer Elements

- Remove Celgard, TX1040, and bleeder materials from outside surfaces of imidized "C" channel elements. Care shall be exercised to prevent damage to imidized preforms.
- Join the two "C" channel mandrels together with undersize diameter fasteners to allow for mandrel movement while under pressure during cure.
- Install two imidized 0° fillet elements in cleavage, top and bottom, between the "C" channels and secure in place at part ends, outside the part trim area, with a small piece of Kapton tape.
- Install tooling dams on top cap edges.
- Place the assembly on a flat steel tool for autoclave curing at $329^{\circ}C$ ($625^{\circ}F$), 1378 KN/m^2 (200 psi). The tool surface shall be prepared by coating with Frekote 33 parting agent. Cover tool surface with Kapton film glide sheet.
- Cut a 3.5-cm (1.48 in.) wide, 96.5-cm (38.0 in.) long strip of imidized 0°, 7 ply laminate stock and remove TX1040 and bleeder materials.
- Place the laminate in the top cap recess over the $(\pm 45^{\circ})_s$, 4 ply flanges of the "C" channels and 0° fillet elements.
- Install the top pressure caul. The pressure caul shall be prepared by coating with Frekote 33 parting agent.
- Install type 162 fiberglass breathers (or equivalent) over the "I" beam tooling and onto the tool surface. Adequate breather material shall be placed between the part and vacuum source to insure efficient removal of volatiles and protection of the bag.

- o Install thermocouples into the edge of the part. These thermocouples shall be used in monitoring and recording time/temperature data during cure and shall be used to control the cure cycle.
- o Install a 0.051-mm (0.002 in.) thick Kapton film bag over the breather and tool surface and seal around the periphery of the tool using a high-temperature sealant. Insure that no bridges exist or sharp protrusions bear against the vacuum bag. Make vacuum bag "ear" seals as required to insure adequate bag slack to prevent bridging.
- o Install the steel clamping ring and secure with bolts around the periphery of the tool.

3.2.2.6 Cure Procedure

- o Install the tooling in an autoclave and apply full vacuum to the tooling system. Apply 689 KN/m^2 (100 psi) to the autoclave and inspect the system for leaks. If a vacuum leak greater than 33.7 KN/m^2 (10 in. Hg) occurs within five minutes, the source shall be located and repaired.
- o Perform the cure cycle within the time temperature profile of Figure 65. Record all events such as application of various levels of vacuum, pressure and autographically record temperature from each thermocouple on parts and autoclave. The part thermocouples shall be used in controlling the cure cycle. All part temperatures shall be in the range of 274 to 287°C (525 to 550°F) when 1378 (KN/m^2) (200 psi) autoclave pressure is applied.
- o Remove bleeder materials, clean up parts, and submit to Quality Engineering for NDI C-scan test.

3.2.2.7 Postcure Procedure

- Postcure the "I" stringer in an air-circulating oven by raising the oven and part temperature from room temperature to 316°C (600°F) at an average heat rise rate of 1.6 to 8.3°C (3 to 25°F)/minute and hold at temperature for four hours. The "I" stringer shall be supported on a flat base, free standing, during postcure.

3.2.3 Hat-Section Stringer Processing

Processing procedures are defined for the specific hat-section stringer designed for this program but are applicable to any hat-section element with minor modifications.

Stock for the individual components of the hat-section stringer are laid up and debulked as flat laminates per 3.2.1.2 except for modifications noted herein. Only the $(0^{\circ})_{16}$ unidirectional cap reinforcement is imidized in accordance with 3.2.1.2. Imidization of the web/flange components is accomplished in situ during the autoclave cure cycle.

3.2.3.2 Layup and Debulking

Prepreg tape having a nominal 0.145-mm (5.7 mil) cured ply thickness and 152 $\pm$ 4 grams/m^2 areal fiber weight was used for this hat-section stringer design.

Flat laminate stock, $(0^{\circ})_{16}$ was laid up and debulked per 3.2.1.2 for the hat-section cap.

Two flat laminates, $\pm 45^{\circ}$ two ply, for inner and outer web/flange elements of the hat-section were laid up and debulked. However, the laminate stock for the inner element was debulked without bleeder material. In the layup of the laminate stock for both inner and outer elements, the TX1040 only and TX1040 with bleeder were applied to the laminate surface, as determined by the assembly, on a 45° bias to the rectangular laminate shape to prevent wrinkling during vacuum forming.

3.2.3.3 Imidizing $(0^{\circ})_{16}$ Cap Element

The $(0^{\circ})_{16}$ cap stock was imidized per 3.2.1.2 except that 172 KN/m^2 (25 psi) was applied after the 115°C (240°F) cycle to increase compaction and to reduce material movement during the cure process and thereby eliminated wrinkles in the cap area.

3.2.3.4 Shaping $(0^{\circ})_{16}$ Cap Element

Trim a 2.79-cm (1.1 in.) wide strip from the imidized $(0^{\circ})_{16}$ laminate stock parallel to the fibers using a sharp knife and straight edge. Remove TX1040 and bleeder.

Place the strip on the top of the mandrel and bevel the edges to match the angle of the tool using a sanding block.

3.2.3.5 Vacuum Forming Hat-Section Elements

- Apply Frekote 33 parting agent to the hat-section mandrel surfaces and oven dry for 15 minutes at 176°C (350°F).
- Cut a 15.2-cm (6.0 in.) wide strip from the debulked two ply +45° flat laminate (inner element) to desired length for the inner layer of the hat-section.
- Prepare a flat plate of suitable size for holding the mandrel in a vacuum bag. Vacuum bag materials and seals shall be prepared previously so that a rapid seal can be made in the subsequent vacuum forming operation.
- If the prepreg is dry and nontacky, heat the hat-section mandrel to 65 $\pm$ 6°C (150 $\pm$ 10°F) to promote improved drape for vacuum forming.
- Place the mandrel on the prepared flat plate. Transfer the debulked flat laminate to the mandrel and secure in place with tape at each end.
- Drape a layer of nylon film and Mochburg paper breather over the surface of the flat laminate.

- Drape the vacuum bag over the layup, seal and draw vacuum. Insure that the vacuum bag conforms to the radius areas by rubbing with a teflon paddle. This operation will form the flat debulked laminate with TX1040 in place over the mandrel, wrinkle-free. The laminate preform with integral 45^{o} bias TX1040 separators will remain secured to mandrel.

- Remove nylon bag, Mochburg breather, and bias ply of TX1040.

- Place the shaped $(0^{o})_{16}$ cap element prepared over the $\pm$ 45^{o} two ply layup on the tool cap. Tack in-place on each end with a small piece of tape.

- Cut a 15.2-cm (6.0 in.) wide strip from the debulked two ply $\pm$ 45^{o} flat laminate (outer element) to the desired length for the outer layer of the hat-section. Remove the single ply of TX1040. The bias oriented TX1040 and 120 fiberglass bleeder are to remain in place.

- Place the laminate on the $(0^{o})_{16}$ cap element to have the graphite surfaces in contact. Secure each end to mandrel with tape.

- Repeat the above vacuum forming operation. In order to prevent the outer plies from tacking to the flange of the inner plies, insert a strip of polyethelene, or F.E.P. film between the two preforms along each flange. During the vacuum forming operation rub and force the bag into the radius areas with a teflon paddle.

- Remove the vacuum bag and then carefully remove the two parting film strips from between each flange of the "hat".

- Tack the two flanges into final position.

- Drape a parting film such as mylon or F.E.P. over the vacuum formed hat assembly. Install the molded silicone

rubber caul over the parting film. Seal in a nylon film vacuum bag, place in an autoclave, apply vacuum and pressurize to 689 KN/m^2 (100 psi). Hold under pressure for approximately 15 minutes. This operation is performed to insure proper seating of prepreg preforms, bleeder materials and rubber caul.

- o Remove bag, rubber tooling and parting film and inspect for part conformance to the tooling.

3.2.3.6 Assembly for Cure

- o Apply Frekote 33 parting agent to the rubber caul. Air dry for 15 minutes minimum.
- o Install the rubber caul over the $\pm$ 45° bias 120 fiberglass bleeder surface of the preformed hat.
- o Place the mandrel on a flat steel tool suitable for curing parts at 1378 KN/m^2 (200 psi), 287°C (550°F). Install shims under the curved base of the tool to prevent bending the tool when autoclave pressure is applied.
- o Install 162 fiberglass breather material over the external surface of the rubber caul. Apply material as required to prevent bridging and any sharp protrusions from coming in contact with the bag. Adequate breather material shall be placed between the part and vacuum source to insure efficient removal of volatiles.
- o Install thermocouples into the edge of the part under the rubber caul. These thermocouples shall be used in monitoring and recording time/temperature data during cure and shall be used to control the cure cycle.
- o Install a 0.051-mm (0.002 in.) thick Kapton film bag over the breather and tool surfaces and seal around the periphery of the tool using a high-temperature sealant. Insure that no bridges exist or sharp protrusions bear

against the vacuum bag. Make vacuum bag "ear" seals as required to insure adequate bag slack to prevent bridging.

- o Install the steel clamping ring and secure with bolts around the periphery of the tool. The tooling arrangement is shown in Figure 94.

3.2.3.7 Cure Procedure

- o Install the tooling in an autoclave and apply full vacuum. Apply 689 KN/m^2 (100 psi) to the autoclave and inspect the system for leaks. If a vacuum leak greater than 33.7 KN/m^2 (10 in. Hg) occurs within five minutes, the source shall be located and repaired.
- o Perform the in situ imidizing the cure cycle within the time temperature profile of Figure 95. Record all events such as application of various levels of vacuum, pressure and autographically record temperature from each thermocouple on parts and autoclave. The part thermocouples shall be used in controlling the cure cycle. All part temperatures shall be in the range of 274 to 287°C (525 to 550°F) when 1378 KN/m^2 (200 psi) autoclave pressure is applied. The ultimate cure temperature shall not exceed 293°C (560°F).
- o Force cool the part to $< 65^{\circ}C$ ($< 150^{\circ}F$) prior to pressure release.
- o Remove the part from the tooling. Care shall be exercised to prevent tearing the rubber caul during removal from the surface of the bleeder material on the part.
- o Remove bleeder materials, clean up parts, and submit to Quality Engineering for NDI C-scan test.

3.2.3.8 Postcure Procedure

Postcure the hat-section in an air-circulating oven by raising the oven and part temperature from room temperature to 316°C (600°F) at an average heat rise rate of 1.6 to 8.3°C (3 to 15°F)/

minute and hold at temperature for four hours. The hat-section shall be supported on a flat base, free standing, during postcure.

3.2.4 Honeycomb Sandwich Processing

Laminate face sheets are processed in accordance with procedures defined in 3.2.1.3 except for postcuring which is accomplished after bonding the sandwich structure.

3.2.4.1 Prime Laminate Face Sheets

- Secure face sheets to a flat surface or in a holding frame.
- Abrade faying surfaces with Scotch Brite, Type A, pads and water. A water break free test will be performed and each skin force dried at 121°C (250°F) for 30 minutes in an air circulating oven.
- Prime skin faying surfaces by spraying two box coats of 35% solids BR34 aluminum powder filled polyimide resin. (A box coat consists of two spray coats, with the second spray coat applied 90° to the first to ensure even coverage). The primer will be allowed to air dry for 45 minutes minimum, staged in an air circulating oven by raising the temperature from room temperature to 51.7°C (125°F) and then to 204°C (400°F) in 13.9°C (25°F) increments every 15 minutes. This procedure is required to prevent blistering the primer.
- Skins will be stored in clean kraft paper until ready for use.

3.2.4.2 Prime Honeycomb Core

- Vapor and spray clean in trichlorethylene and oven dry at 121°C (250°F) for 30 minutes.
- Apply 35% solids BR34 primer to the core cell faying edges in four box coats. Air dry 45 minutes minimum and stage primer in an air circulating oven per 3.2.4.1.

- Primed core elements will be stored in clean kraft paper until ready for use.

3.2.4.3 Assemble Sandwich Panel and Bond

- Apply adhesive film, FM34B-18, .44 Kg/m^2 (0.09 psf) to the face sheets, assemble with honeycomb, and vacuum bag for bonding.
- Apply 84.4 to 94.5 KN/m^2 (25 to 27 in. Hg) vacuum and 2.76 KN/m^2 (40 psi) autoclave pressure.
- Raise temperature from room temperature to 177°C (350°F) at 2.8°C (5°F)/minute.
- Cure two hours at 188°C (370°F).
- Postcure sandwich panels free standingin an air-circulating oven by raising the temperature at 5-7°C (9-12°F)/minute to 316°C (600°F) and holding at 316°C (600°F) for four hours. Force cool panels to room temperature.

3.2.5 Chopped Fiber Molding Processing

Unidirectional prepreg tape 15.2-cm (6 in.) wide was procured for this process development to the following physical properties requirements:

- Resin Solids: 38 ± 3%
- Volatiles: 12 ± 3%
- Fiber Areal Weight: 67 ± 3 grams/m^2
- Calculated thickness, 60% fiber volume: 0.0064-cm (2.5 mils)/ply

The tape material was chopped to produce random size pieces 1.25 to 2.54-cm (0.5 to 1.0 in.) long, in the filament direction, by 0.25 to 3.0-cm (0.1 to 1.2 in.) wide.

Development of chopped fiber molding processing was accomplished using an ASTM D790 flexure specimen molded in an ASTM D647 compression mold. The flexure mold and typical molded coupons are shown in Figure 96.

The following two stage process was developed for chopped fiber molding:

- Spread chopped unidirectional prepreg uniformly over a teflon sheet positioned in a shallow pan.
- Imidize material in an air-circulating oven by raising the temperature from room temperature to 190°C (375°F) and staging at temperature for one hour.
- Load mold with net weight imidized material to obtain a target fiber volume of 60%.
- Place mold in a press preheated to 316°C (600°F) and close to contact position.
- Apply 13790 KN/m^2 (2000 psi) pressure when part temperature reaches 204°C (400°F).
- Cure one hour at 316°C (600°F).

Postcure chopped fiber molding free standing for four hours at 316°C (600°F).

3.3 TASK (c) - FABRICATION AND TEST

3.3.1 Fabrication - Mechanical Properties Specimens

Initial laminate panels to be used for mechanical properties testing were laid up and autoclave cured using the single stage in situ imidizing and cure process described in 3.2.1.1. These panels were used to obtain all postcured condition mechanical properties specified in the test matrix, Table 10. Resin flow control proved to be a problem using this cure cycle, sometimes resulting in high composite fiber volumes in the range of 64 to 68%. All laminates for specimen fabrication had essentially zero void content as determined analytically, and by NDI C-scan test.

Process optimization studies performed in Task (b) led to a two stage processing requiring an imidizing cycle where volatiles are removed to $< 2\%$ from the stacked prepreg prior to the autoclave cure. Resin flow control was maintained during imidization by low vacuum levels and a Celgard 4500 or 4510 microporous polypropylene film which allows volatile matter to escape through a perforated tooling plate while the membrane contains the low viscosity resin. Excess resin was absorbed into bleeder materials calculated to yield a laminate with a target $60 \pm 2\%$ fiber volume.

The autoclave cure was accomplished between two flat tooling plates. Since the major portion of volatile matter was removed in the imidizing cycle, the laminates were treated similarly to epoxy materials. Final laminate cure was accomplished at 287°C (500°F) for three hours or 326°C (625°F) for two hours.

This two stage process was employed in the fabrication of all laminates for mechanical properties specimens that were aged for 125 hours at 316°C (600°F). All panels had essentially zero void with fiber volumes in the 61 to 63% range. NDI C-scan recordings confirmed high quality and are shown in Figure 97 through 102. The detailed description of this two stage processing is presented in 3.2.1.2. Flexural and tensile specimens were molded from chopped fiber using the compression molds shown in Figure 96 and 103. The

molding process used is described in 3.2.5.

3.3.2 Testing - Mechanical Properties

Testing was performed in accordance with the matrix, Table 10. Three specimens for each test mode and temperature were tested in the postcured and aged, 125 hours 316°C (600°F), conditions.

3.3.2.1 Beam Test Description

Tension and compression critical beams were employed to determine $(0)_t$ tension (F_{tu}, E_t, ε_{ult}, $\mu\%$) and $(0^\circ)_t$ and $(0^\circ, \pm 45^\circ, 90^\circ)_s$ compression (F_{cn}, E_c, $\varepsilon_{ult}\mu\%$) properties. The beam designs are presented in Figure 104.

Analytical studies (Ref. 5) performed by Mr. Mark Shuart, NASA-LaRC, proved that the 352 Kg/m^3 (22 pcf) honeycomb core significantly affects the measured strength and elastic modulus properties of composite specimens. A computer program was developed by NASA-LaRC to assess the actual effect this core has on the laminate properties and to establish property adjustment factors. Bulk core properties for both aluminum, 352 Kg/m^3 (22 pcf), and 301 CRES, 639 Kg/m^3 (40 pcf), core materials were developed by Rockwell International and transmitted to NASA-LaRC for use in the computer program in developing the composite property adjustment factors. These data are shown in Table 11. Specific adjustment factors are given in the individual mechanical property data Tables 12 through 17.

During test, individual specimens were stabilized at each test temperature for 10^{+10}_{-0} minutes prior to application of stress at a head travel of 1.27-mm (0.05 in.)/minute. Data were obtained by autographic recording of axial strain gages installed on the composite specimens at the beam midpoint.

3.3.2.2 Tensile Test Description

Tensile coupons were employed to determine $(0^\circ)t$, $(90^\circ)t$, $(+45^\circ)s$ and $(0^\circ, \pm 45^\circ)s$ and $(0^\circ, \pm 45^\circ, 90^\circ)s$ tension properties. Specific properties determined were F_{tu}, E_t, ε_{ult}, $\mu(\%)$ and ν.

The $(0^{o})_{t}$, $(90^{o})_{t}$ and $(+45^{o})_{s}$ coupons employed a straight sided design and the $(0^{o}, \pm 45^{o}, 90^{o})$s coupons were necked down in the test section. Specimen design is shown in Figure 105.

During test, specimens were loaded at a head travel of 1.27-mm (0.05 in.)/minute after stabilizing for 10^{+10}_{-0} minutes at temperature. Data were obtained using biaxial strain gages mounted back-to-back on two of three specimens in each test group.

Load/strain data were obtained incrementally in testing post-cured condition specimens by digital readout using a data logger. Stress/strain data plots were made using a Hewlett Packard 9820 computer system. The remaining single specimen in each group was instrumented with clip-on hang down extensometers.

In testing the 125 hour 316°C aged coupon specimens, load/strain data was obtained autographically from biaxial strain gages at a constant head travel of 1.27-mm (0.05 in.)/minute.

3.3.2.3 Compression Test Description

Compression coupons were employed to determine $(90)_{t}$ and $(+45)_{s}$ compression properties, F_{cu}, E_{c} and ε_{ult} μ (%). The compression specimen is a 7.62-cm long x 2.54-cm wide (3.000X1.000 in.) coupon. Specific tolerances and test fixture design are presented in Figure 106. During test, specimens were loaded, after stabilizing at each test temperature for 10^{+10}_{-0} minutes, at a constant head travel of 1.27-mm (0.05 in.)/minute. Load/strain data were obtained autographically using a hang down deflectometer.

3.3.2.4 Flexural and Short Beam Shear Test Description

Specimens were machined from 0°, 26 ply, nominal 0.063-mm (2.5 mils)/ply, 1.65-mm (0.0565 in.) thick test panels. Specimen configurations were in accordance with ASTM D790 (flexural) and ASTM D2344 (short beam shear). Respective span to thickness ratios for each test are 32:1 and 4:1. Strain measurements were made autographically during eact test using an isolated deflectometer positioned at the specimen midpoint. Elastic modulus properties were derived from load/strain curves obtained in the flexural tests

and the load/strain curves obtained in the SBS tests were used to give a positive indication of when actual specimen failure occurred. Specimens were loaded at a head travel of 1.27-mm (0.05 in.)/minute after being stabilized at the test temperature for 10^{+10}_{-0} minutes.

3.3.2.5 Tensile and Flexural Chopped Fiber Test Description

Specimen configuration as molded were in accordance with ASTM D651 (tensile) and ASTM D790 (flexural). The tensile and compression specimens were tested at room temperature and 316°C (600°F). For the 316°C testing, the specimens were stabilized at temperatures for 10^{+5}_{-0} minutes.

3.3.3 Test Results - Mechanical Properties

3.3.3.1 Tension

Data obtained during testing are summarized in Table 18. The effects of test temperature and postcuring versus aging (125 hour, 316°C (600°F) conditioning) on composite mechanical properties are presented graphically in Figure 107.

Testing problems were experienced in some cases with beam specimens at 204°C and 316°C (400°F and 600°F) when either composite facing-to-core or steel facing-to-core bond failures occurred. Test data were tabulated at the composite stress level reached when bond failure occurred and are therefore not averaged.

Tension test results of $(0^\circ)_t$ beam specimens adjusted per paragraph 3.3.2.1 show that the postcured specimens have higher strength than the aged under all conditions except room temperature. All tensile strengths were quite high starting at 2068 MN/m^2 (300 ksi) at -168°C (-270°F) and steadily decreasing to 1648 MN/m^2 (239 ksi) at 316°C (600°F). The $(0^\circ, \pm 45^\circ, 90^\circ)_s$ and $(\pm 45^\circ)_s$ tensile coupons in the postcured condition also maintained higher strength than the aged counterparts although the spread was very close. There was virtually no decrease in the postcured $(0^\circ, \pm 45^\circ, 90^\circ)_s$ tensile strength between -168°C (-270°F) and 316°C (600°F). The resin critical $(90^\circ)_t$ aged tensile coupon specimens

showed good strength retention in comparison with the postcured units, having slightly higher -168°C (-270°F) and room temperature strengths and slightly lower 204°C (400°F) and 316°C (600°F) strengths.

Elastic modulus properties of the fiber critical $(0^\circ)_t$ specimens were not significantly affected regardless of test temperature while the $(0^\circ, \pm 45^\circ, 90^\circ)_s$, 316°C (600°F) test specimens showed some modulus loss. For the resin critical $(\pm 45^\circ)_s$ and $(90^\circ)_t$ coupons, a gradual decrease in elastic modulus properties was noted between -168°C (-270°F) and 316°C (600°F).

Detailed tensile properties and failure modes from beam tests are presented in Tables 12 and 13 and properties from coupon testing are presented in Tables 19 through 26. Stress/strain curves from beam and coupon testing are presented in Appendix C.

3.3.3.2 Compression

Data obtained during test are summarized in Table 27. Effects of test temperature and postcuring versus aging (125 hours, 316°C (600°F) conditioning) on composite mechanical properties are presented graphically in Figure 108.

Testing problems occurred in compression test of 204°C (400°F), $(0^\circ, \pm 45^\circ, 90^\circ)_s$ beams as discussed in the tensile results 3.3.3.1.

Analysis of compression test results indicated somewhat different trends in strength properties than found in tension, with lower ultimate strengths in the fiber critical orientations, $(0^\circ)_t$ and $(0^\circ, \pm 45^\circ, 90^\circ)_s$, and higher strengths in the resin critical orientations, $(90^\circ)_t$ and $(+45^\circ)_s$. For the $(0^\circ)_t$ beam tests the aged, room temperature strength was higher than the postcured specimens as found in tension tests. A greater loss from room temperature compression strength was noted at 316°C (600°F) than in the tension tests, a 37% reduction for postcured and 50% reduction for aged condition. Beam specimens with $(0^\circ, \pm 45^\circ, 90^\circ)_s$ fiber orientation, postcured condition, showed only 33% strength loss from room temperature to 316°C (600°F) while the aged spec-

imens had a 20% loss indicating a postcure effect. The resin dependent $(90^{o})_{t}$ and $(\pm 45^{o})_{s}$ coupon specimen strengths were almost identical in both postcured and aged conditions at each test temperature except for the postcured 316°C (600°F) tested $(\pm 45^{o})_{s}$ specimens, indicating the influence of the resin. Strength losses from room temperature to 316°C (600°F) ranged from 54% in the postcured $(\pm 45^{o})_{s}$ specimens, while the aged specimens lost only 20%, again indicating a postcure effect on the resin.

Elastic modulus properties of the $(\pm 45^{o})_{s}$ specimens at each test temperature were increased after aging at 316°C (600°F), while the $(90^{o})_{t}$ specimens showed no significant difference. $(0^{o})_{t}$ and $(0^{o}, \pm 45^{o}, 90^{o})_{s}$ specimens showed no significant change in elastic modulus properties, regardless of test temperature or aged condition. Detailed compression data and failure modes from beam tests are presented in Tables 14 through 17. Table 28 presents the data for coupon testing. Stress/strain curves are presented in Appendix C.

3.3.3.3 Flexural

Results of flexural strength tests on postcured and aged specimens show a drop in strength from room temperature to 316°C (600°F) of 51% and 34% respectively. Specimens tested at -168°C (-270°F) yielded respective strength increases from room temperature of 18% and 6.1%. The aged specimens demonstrated higher strengths at all test temperatures except -168°C (-270°F). Elastic modulus properties were not significantly affected regardless of aged conditions or test temperature. Failure modes of -168°C (-270°F) and room temperature tested specimens were by outer fiber tension and by compression in specimens tested at 204°C (400°F) and 316°C (600°F). Detailed data are presented in Table 29 and the relative performance of postcured and aged specimens is shown graphically in Figure 109.

3.3.3.4 Short Beam Shear

Results of short beam shear tests on postcured and aged

specimens show exceptionally good strength retention at all test temperatures. The strengths of postcured specimens was slightly higher at -168°C (-270°F) and room temperature than the aged specimens, equivalent at 204°C (400°F) and slightly lower at 316°C (600°F). All failure modes were by interlaminar shear. The relative performance of postcured and aged specimens is presented graphically in Figure 110 and tabulated data in Table 30.

3.3.3.5 Tension and Flexural-Chopped Fiber Molding

Four batches of chopped fiber molding material were evaluated. These varied in areal weight of the starting prepreg tape and chopped fiber length. The material batches and variations are noted below in the order of evaluation.

Batch A - Areal weight: 66.8 gm/m^2
Fiber length: 1.27 to 2.54-cm (0.5 to 1 in.)

Batch B - Areal weight: 153.7 gm/m^2
Fiber length: 1.27-cm (0.5 in.)

Batch C - Areal weight: 60.4 gm/m^2
Fiber length: 1.27-cm (0.5 in.)

Batch D - Areal weight: 67 gm/m^2
Fiber length: 2.54-cm (1 in.)

Flexural specimens were molded from all batches. Tension specimens were molded from batches A and B. Average properties are presented in Table 31.

3.3.4 Fabrication - Structural Elements

Hat-section and "I" stiffened panel design requirements, design assumptions for optimization of panel configuration, analysis, and supportive calculations for the designs are presented in Appendix D.

3.3.4.1 Hat-Stringer Stiffened Skin Elements

The detailed process description for fabricating hat-section elements is presented in 3.2.3. Difficulties were encountered during fabrication of some later 193-cm (76 in.) long hat elements in the form of ($\pm 45^{\circ}$) layers locally wrinkling along the upper cap corners. Wrinkling was caused by insufficient compaction of the 30.5X193-cm (12.0X76.0 in.) 16 plies thick unidirectional cap preform during the imidizing cycle. However, NDI C-scan test results showed that essentially void free parts were attained.

The wrinkling problem was resolved by modifying the unidirectional cap imidizing procedure by applying 84KN/m^2 (25 in. Hg) vacuum plus 172KN/m^2 (25 psi) autoclave pressure at the end of the 114°C (240°F) cycle. Resultant preforms were reduced in bulk thickness from 3.30 to 3.56-mm (0.13 to 0.14 in.) to 2.79 to 1.41-mm (0.11 to 0.095 in.) which decreased material movement during final compaction in the cure process. The Celgard contained the resin during pressurization and no excessive losses were noted. Additional debulking of the lay up was accomplished after final lay up using a molded silicone rubber caul at room temperature under 689 KN/m^2 (100 psi). The resultant preform on the aluminum mandrel closely matched the shape of the rubber caul, producing smooth, wrinkle-free surfaces. Hat elements were autoclave molded using the in situ cure process described in 3.2.1.1. Excellent NDI C-scan test results were obtained on all hats in the cured and postcured conditions, as typically shown in Figure 111.

Concave warpage of the hat stringers occurs along the element flange and inside cap length with a maximum flatness deviation of approximately 5.08-mm (0.20 in.) at the midpoint as shown in the photograph, Figure 112. This condition was partially removed after bonding the three hat elements (EX191, EX193, EX195) to skin (EX197) and was almost completely removed when the 193-cm (76 in.) long skin/stringer assembly was cut into five 30.48-cm (12 in.) long test sections, Specimen No's EX195-1, -2, -3, -4, -5. This lengthwise concave warped conditions is caused by imbalance in the hat

design, where the major quantity of fiber contained in the cap section places the part's neutral axis off center. This condition would have posed a problem in the test of the stringer stiffened skin elements that were to be delivered to NASA-LaRC in Task (e) of the program, since nonuniform cap-skin loading would result from the concave condition.

To resolve this problem, the 127-cm (50 in.) long hat mandrel tool was modified by reverse rolling it concave to the cap surface, 6.35-mm (0.25 in.) at the midpoint. This modification, 1.27-mm (0.05 in.) more than actually observed in the elements as they are removed from the tool, was made on the assumption that upon removal from the reverse cured tool after curing, the hat element would approach a flat condition. Any minor longitudinal warping, concave or convex, would be eliminated when the hat is bonded to the skin.

This approach was verified in molding hat elements EX249 and EX250 to be used in fabricating hat-stiffened skin/stringer elements in Task (e). These parts were fabricated, per specific procedures defined in 3.2.3 on the reverse formed tooling. Resultant hat elements were flat and linear with no warpage. The photograph in Figure 112 shows the flatness of the hat element.

Due to the hat tooling mass, heat rise rates during the in situ imidizing cure cycle are extremely low. For example in the final critical temperature range between 257 to 271°C (500 to 525°F) the average heat rise rate was only 0.51 to 0.55°C (0.92 to 1.0°F)/minute. Figure 113 gives actual heat rise rate ranges observed during two hat element autoclave curing cycles. This indicates that the LARC-160 system is apparently not affected by long dwell periods close to the hot melt resin flow point, demonstrating that a large processing window exists. Heating rate comparisons are plotted in Figure 113 for typical flat panels, which show 340 minutes total cure time versus 590 minutes for hat elements, not counting cool-down.

The vacuum bag oven cure employing the pressure augmentation process was used to bond the three hat elements to skin using FM34B-18, 439 grams/m^2 (0.09 psf), adhesive. Tooling was improved for fabrication of the subsequent 193-cm (76 in.) long article, EX195, by employing inverted "T"-bars to distribute bonding pressure to hat flange/bond areas. This innovation improved handling and assembly of tooling elements. The tooling concept is shown in photographs, Figures 114 through 118. A section of the NDI C-scan recording of the hat-to-skin bond is shown in Figure 119.

3.3.4.2 "I" Stringer Stiffened Skin Elements

The detailed process description for fabricating "I" section elements is presented in 3.2.2. NDI C-scan tests showed considerable void areas in the caps. To determine the void characteristics, 120X photomicrographs were taken of discreet cap areas where both 100% and 0% sound penetrations were recorded. From these it was determined that the void shapes were irregular micron sized pits distributed throughout the cap thickness. The cap side showing 100% sound penetration showed no voids in the photomicrographs. Actual respective void volumes detemined analytically were 8.31% and 1.13% and fiber volumes 58-62%. NDI C-scans and photomicrographs of the "I"-stringer cap are shown in Figure 120.

"I"-stringers were bonded to the skin assembly with FM34B-18 adhesive film on a 104 glass cloth carrier using the vacuum bag pressure augmentation process. Since the maximum pressure augmentation area-to-bond area available in this part design is only 1.5:1, an autoclave was required in the bonding operation. A minimum 3:1 pressure augmentation area to bond is required for one atmosphere oven bonding operations. The bonding sequence is shown in Figures 121 through 125.

3.3.4.3 Honeycomb Sandwich Panel Elements

Fabrication of sandwich panel EX150, 63.5x71.1x4.85-cm (25.0x28.0x1.91 in.) was accomplished in accordance with processes described in 3.2.4. The skins were comprised of unbalanced (0°_2,

$\pm$ 45°, 0°), 5 ply, nominal 0.144-mm (5.7 mil)/ply unidirectional tape. NDI C-scan recordings indicated a good skin to core bond was attained.

Celion 1K, 34x35, 5 harness satin weave graphite fabric/ LARC-160, 20 ply doubler stock was fabricated using the two stage processing, imidization and autoclave curing. Panel size was 30.5x60.9x0.33-cm (12x24x0.130 in.). NDI C-scan showed 100% transmission; a recording is shown in Figure 126. Doubler stock was machined to a tapered configuration and bonded to the ends of each sandwich element with FM34B-18 adhesive film.

3.3.5 Preparation and Testing - Structural Elements

All structural elements were tested on a 1957KN (440,000 lb.) capacity Tinius Olsen universal testing machine. Test preparation and testing was accomplished as follows:

- Specimen ends were ground flat and parallel to within $\pm$ 0.127-mm ($\pm$ 0.005 in.).
- One half of all structural elements were aged 125 hours at 316°C (600°F). Initial weights, and percent weight loss after aging are shown in Table 32.
- Hat-and "I"-stiffened skin/stringer panels were stabilized at each end by potting in place to approximately 1.27-mm (0.50 in.) thick to match equivalent thickness precision ground tool steel load plates. Potting materials were selected based on test temperature and processed as shown in Table 33. Sandwich panel ends were stabilized by the tapered doublers described in paragraph 3.3.4.3. Doublers were clamped between parallel bars resting on the test bed during test to prevent specimen ends from spreading.
- Bi-axial gages were positioned and bonded at the midpoint of the center stringer cap and skin, in a back-to-back pattern and in rosette, 0°, 45°, 90°, on one web of the center stringer. Sandwich panels were instrumented

with back-to-back strain gages in rosette, 0^{o}, 45^{o}, 90^{o}, at the midpoint.

- Hat and "I"-stiffened skin/stringer panel edges were clamped to provide fixity; sandwich panel edges were not clamped.
- Specimens were positioned in the test machine on a special spherical seat fixture designed to ensure optimum axial alignment. A pre-load of 2244 to 8896 N (500 to 2000 lb.) was applied and the specimen was aligned by adjusting the spherical seat to match back-to-back skin and hat cap axial strain gage deflections to a tolerance of 50μ. Typical specimens are shown in position on the test machine in Figures 127 through 130.
- After stabilizing at test temperature, a compressive load was applied incrementally up to the individual specimen calculated design ultimate. Strain measurements were taken at each loading increment. Specific target ultimate loads are shown in Table 34.

3.3.6 Test Results - Structural Elements

Data are summarized in Table 35 and load/strain curves are presented in Figures 131 through 150. Figures 151 through 165 show failures modes of elements that failed during test.

All of the room temperature elements met the design ultimate load requirement of 525KN/m (3000 lb./in). Specimen EX109/EX110A hat stringer failed while being held at the predicted ultimate load, showing good correlation between theory and design practice. A small degree of strength degradation was noted in the hat stiffened skin/stringer element EX109/EX110B during $-168^{o}C$ ($-270^{o}F$) testing where failure occurred at 117 KN (26,250 lb.), 3.4% below room temperature design ultimate. This specimen had previously been tested to design ultimate of 120.8 KN (27,150 lb.) at room temperature and then fatigue tested to 265,000 cycles, 5% to 67% of design ultimate (compression/compression). Premature failure may have been caused

by the combination of previous static and fatigue testing and resin embrittlement at $-168^{o}C$ ($-270^{o}F$).

The strain gage data showed, for the most part, linear compression properties for the section designs tested. This was most apparent in the tests on the two honeycomb panels, which represent a balanced section and the six "I"-stringer panels which are unbalanced. The unbalanced hat-stiffened skin/stringer elements showed fairly linear strain increase until just before failure where it was found that probable local instabilities caused fairly large excursions in the gage readings in some test cases. The nonlinearity would be aggravated if the section designs were more unbalanced.

In terms of structural efficiency, the hat-stiffened panel yielded the lightest weight design. It should also be noted that the $(0^{o}, \pm 45^{o}, 90^{o})_{s}$ skin configuration of the hat-and "I"-stiffened panels was dictated as a design requirement, and thus these sections did not represent optimal designs. A measure of the structural efficiency of these configurations may be obtained by plotting the design parameters as shown in Figure 166. The relative weights per unit area of the three configurations are as follows:

Hat-Stiffened panel:	$4.5 Kgm/m^2$ (0.939 lb/ft^2)
"I"-stiffended panel:	$5.1 Kgm/m^2$ (1.04 lb/ft^2)
Honeycomb panel:	$5.6 Kgm/m^2$ (1.14 lb/ft^2)

4.0 DEMONSTRATION COMPONENTS

4.1 TASK (d) - LAMINATE FABRICATION

Three laminates, 64x127-cm (25x50 in.), were fabricated using 0.127-mm (5 mil), 30.5-cm (12 inch) wide prepreg tape. Laminate ply orientation was (0^{o}, $\pm 45^{o}$) symmetrical about the neutral axis. The laminates, indentified as CL6C-11, CL12C-6, and CL24C-7, were 0.75, 1.5, and 3.0-mm (0.030, 0.060, and 0.120 in.) thick having 6, 12, and 24 plies of prepreg tape respectively.

Two stage processing, imidization and cure, of the laminates was accomplished as described in 2.3.1.2. After post curing for four hours at $316^{o}C$ ($600^{o}F$), the laminate panels were C-scanned and trimmed to finished dimensions, 60x120-cm (24x48 in.). C-scans are shown in Figures 167, 168, and 169. Laminate physical properties are presented in Table 36.

4.2 TASK (e) - SKIN/STRINGER PANEL FABRICATION

The secondarily bonded hat-section stringer configuration, rather than the "I" stringer was selected on the basis of the test data (presented in 3.3.6) which showed that both met load requirements, with the hat configuration having a weight savings advantage.

Prepreg tape, 0.145-mm (5.7 mil) thick and 30.5-cm (12 in.) wide, was used for fabricating the laminate skins and hat-section elements for the three demonstration articles required by this task. Laminate and hat-section processing is described in 3.2.1.2 and 3.2.3. Assembly bonding of the three hat-section to the laminate skin was accomplished as described in 3.3.4.

The completed skin/stringer panels, measuring 26x122-cm (10.2 x48 in.) are shown in Figure 170. An end view of one panel is presented in Figure 171. NDI C-scan recordings of the FM34B-18 adhesive stringer to skin bond areas are shown in Figures 172 and 173. C-scans typical of the laminate skins and hat-sections are shown in Figures 174 and 111.

4.3 TASK (f) - HONEYCOMB PANEL FABRICATION

Six honeycomb sandwich panels 25.4x24.4-cm (10x10 in.) were required by this task. These panels consisted of 0.15-cm (0.060 in.) thick, $(0^o, 90^o)_t$ face sheets bonded to 2.54-cm (1 in.) thick glass/polyimide (HRH 327) honeycomb cure having a .48-cm (3/16 in.) cell size and 64.04 Kg/m^3 (4.0 lb/ft^3) density. The face sheets were bonded to BR34 primed core with FM34 adhesive, 0.44 Kg/m^2 (0.09 psf). Face sheet to core bonding was accomplished as defined in 3.2.4.

Two 12-ply laminates, designated as CL12C-8 and CL12C-9, 61x 91-cm (24x36 in.) were laid up using 0.127-mm (5 mil) prepreg tape. The layup was such that the $(0^o, 90^o)_t$ fiber orientation was symmetrical about the neutral axis of the honeycomb core. Laminate cure was as defined in 3.2.2. Postcure of the laminates was accomplished in two stages: two hours at 316°C (600°F) free standing and two hours at 316°C (600°F) in the bonded condition which also postcured the adhesive bond line. Physical properties of the postcured laminates are shown in Table 35. Specimens for physical properties determination were taken from laminate trim areas and received the full four hour exposure.

C-scans of laminates CL12C-8 and CL12C-9, showing the location of the three face sheets 28x56-cm (11x22 in.) cut from each are presented in Figures 175 and 176. Face sheet pairs 8C9C, 8A8B, and 9A9B were bonded to honeycomb core. C-scans of the three sandwich panels, identified as 8A, 9A, and 9C, and showing the location of each 25.4x25.4-cm (10x10 in.) panel, designated #1 through #6, are shown in Figures 177, 178, and 179.

4.4 TASK (g) - CHOPPED FIBER MOLDING FABRICATION

Six chopped fiber molding were fabricated using the longer fiber length/lower areal fiber weight molding material (Batch D) noted in 3.3.3.5. The moldings were made using the NASA/BAC matched metal molds utilized in performing a similar task for Contract NAS1-15009 (Graphite/PMR-15 Composite Materials). The molding procedure is

is defined in 3.2.5. Part configuration is shown in Figure 180. Figures 181 and 182 show the charged matched metal mold and a finished part.

4.5 TASK (h) - TECHNOLOGY DEMONSTRATOR SEGMENT

The initial intent of this task was to fabricate a representative demonstration component of the Space Shuttle aft body flap. It was intended that the component design would incorporate all processes and structural configurations developed in task (a) through (g) to demonstrate manufacturing feasibility of graphite/LARC-160 to full-scale structures. An example of such a demonstration component, presented in the program proposal, is shown in Figure 183.

The requirement of this task "to fabricate a representative demonstration component" was later changed "to fabricate a representative structural test component". This structural test component has been given the designation of Technology Demonstrator Segment (TDS). The completed TSD, ready for installation of instrumentation for ground testing, is shown in Figure 184.

In changing from a demonstration to a structural test component, the complexity of the task changed correspondingly. All aspects of design, tooling, NDI, fabrication, and assembly became more critical. To implement this change, the scope of Contract NAS1-15843 (Develop, Demonstrate, and Verify Large Area Composite Structural Bond with Polyimide Adhesive) was amended to fabricate the cover panels, ribs, and leading edge covers of the TDS. Fabrication of the remaining TDS components and final assembly operation was accomplished under this program, Contract NAS1-15371.

All fabricated elements of the TDS, i.e., solid laminate structures, laminate skins, and bonded honeycomb panels, were nondestructively inspected and the results recorded and filed. NDI records for the TDS are not included in this report because of

volume considerations and also because many of the recordings, being very large, would lose definition when reduced for report inclusion.

4.5.1 TDS Selection Rationale

Increase in orbiter inert weight during maturation of the design adversely affects the deliverable and recoverable payload weight capability. The decreased deliverable payload could be restored with additional Shuttle system propulsion, but the decreased recoverable weight cannot be restored in this manner. However, reduction of the basic orbiter weight could result in the restoration of both the deliverable and recoverable payload weight. Significant weight savings are predicted for application of advanced composites in orbiter structural components.

Early Shuttle orbiter studies showed that the use of advanced composites on the vertical tail, elevon, and aft body flap would achieve significant weight savings, particularly if high-temperature graphite/polyimide (Gr/PI) were employed. In 1976, the NASA selected the orbiter body flap as a demonstration component for the Composites for Advanced Space Transportation Systems (CASTS) program. Since that time, orbiter composite-structures IR&D studies have emphasized the body flap. In FY 1976, a preliminary design concept for a body flap was identified. Adhesive bonding of joints was used throughout, thus eliminating stress-concentration and fatigue problems associated with mechanical fasteners. In FY 1977 through 1980, the design data base for Gr/PI structure was expanded through an extensive test program of body flap related subelements.

The body flap was chosen because it is a large, relatively simple, and easily retrofittable structure. It is subjected to extreme acoustic (165dB OASPL), aerodynamic (13.8 KN/m^2), and thermal (1482°C) environment. The flight environment would thus thoroughly test the advanced structural concepts and demonstrate feasibility of application to other Orbiter structures.

Structural weight reduction and increased performance can be realized by taking advantage of the large strength-to-weight and stiffness-to-weight ratios of advanced composites. Savings of up to 145Kg (320 lb) of the total body flap structure/TPS weight can be realized by application of 316°C (600°F) structural allowable Gr/PI. In comparison to the baseline aluminum structure (177°C structural allowable), Gr/PI has reduced TPS requirements; and the TPS tiles can be directly bonded to the Gr/PI substructure because of the thermal compatability and stiffness of the components.

The design of the TDS simulates a section of the orbiter aft body flap incorporating three ribs and extending from the forward cover panels to the rear spar as shown in Figure 185. This section is 137x152-cm (54x60 in), 43-cm (17 in) at the front spar, and 18-cm (7 in) at the rear spar.

Specific objectives of the TDS were as follows:

- o Verify advanced composite design/analysis techniques.
- o Develop and verify manufacturing techniques for large, complex Gr/PI structure.
- o Demonstrate the integrity of Gr/PI all bonded structure to sustain orbiter aerodynamic, thermodynamic, and acoustic environments.

4.5.2 TDS Front Spar Fabrication

The TDS front spar panel (Appendix E, SS79-00253) is a bonded structure consisting of a honeycomb sandwich panel having a 13.33-cm (5.25 in) diameter access opening edged with a "U" shaped ring laminate, solid laminate "h" shapes framing the honeycomb panel, and laminate doublers backing the "h" section.

4.5.2.1 Honeycomb Sandwich Panel Fabrication

Laminate fabrication and sandwich adhesive bonding processes were performed as described in 3.2.1.2 and 3.2.4 except for minor modifications in the laminate bagging assembly procedures. Bagging assembly for imidizing and curing of the 4-ply (0^{o}, $\pm 45^{o}$, 90^{o}) laminates for the honeycomb sandwich are shown in Figures 186 and 187. Assembly bagging of laminate skins to honeycomb core for adhesive bonding is presented in Figure 188. A C-scan recording of one honeycomb sandwich panel prior to machining is shown in Figure 189.

4.5.2.2 "h" Frame Fabrication

A three section tool, shown in Figure 190, was used to fabricate the "h" frame members for the front spar panel. The "h" frame was comprised of the three laminate layup shapes: flat, "Z" and "U". Each laminate layup was imidized on the respective tool section. After imidization "Z" and "U" laminate layups and tool sections were assembled, an imidized unidirectional fillet section was placed in the space between "Z" and "U" laminates, and the flat laminate and flat tool plate were positioned to complete the layup/tooling assembly. This was bagged and autoclave cured. Laminate processing was in accordance with 3.2.1.2. A completed section, before machining, is shown in Figure 191.

4.5.2.3 "U" Closeout Ring Fabrication

The "U" closeout ring was fabricated in four 100^{o} arc segments from woven Thornell 300/LARC-160. The segments were fabricated using the tool shown in Figure 192. Laminate processing was in accordance with 3.2.1.3.

4.5.2.4 Front Spar Panel Assembly

A two piece aluminum picture frame tool was developed for assembly bonding of the front spar panel. Figure 193 presents a detail of the tool showning its application for bonding the "h" frames and doublers. FM34B-18 adhesive was used for bonding. Bond processing was in accordance with 3.2.4. Elements of the front spar assembly and the completed assembly are shown in Figure 199 and 195.

4.5.2.5 Front Spar Tee Fabrication

The three configurations of the front spar Tee members, SS79-00253-003, -004 and -005 are presented in Appdendix E. A typical tool for Tee fabrication is shown in Figure 196. The Tee members consists of three sections (two opposed "L" laminates and a flat laminate) and a unidirectional fillet to fill the space at the laminate junction.

The Gr/PI laminates and fillet section were imidized on the appropriate tooling. Typical imidization setup for an "L" laminate is shown in Figure 197. Following imidization, the tooling was assembled with the Gr/PI fillets in place. The assembly was bagged as shown in Figure 198 and autoclave cured. Proccessing was in accordance with 3.2.1.3. A typical Tee is shown in Figure 199.

4.5.3 TDS Rear Spar Fabrication

The TDS rear spar, SS79-00253-006 is presented in Appendix E. It was fabricated on steel tooling, shown in Figure 200, dimensionally corrected for differences in thermal coefficient of expansion between the Gr/PI laminate and the tool. Laminate layup, imidization and autoclave cure was conducted in accordance with 3.2.1.3. After C-scan inspection, load introduction Pi sections were bonded in three places using FM34B-18 adhesive on the spar OML in line with the TDS ribs. The completed aft spar is shown in Figure 201.

4.5.4 Rib Modification for Load Introduction Plates

Installation of the load introduction plates on the TDS for subsequent mechanical testing required flat and parallel rib interface surfaces. To meet this requirement, it was necessary to shim the interface surfaces of the rib Pi caps (SS79-00251-002) and the front spar Tee (SS79-00253-003) to achieve co-planar and parallel surfaces. Shims were fabricated from graphite fabric/LARC-160 and bonded to the TDS rib assembly in the areas shown in Figure 202 using FM34B-18 polyimide adhesive. Only minimum machining was required to establish the co-planar and parallel condition after bonding.

After shim bonding, the location of holes for attaching the load introduction plates to the rib was established using a trim/drill template. These locations in the rib were potted to provide a solid area for bolt clamp-up pressure in the rib honeycomb core. The potted rib and load introduction plates were match drilled to the trim/drill template using diamond core drills.

4.5.5 Alignment of Ribs to Covers

TDS cover panels (SS79-00250-008) were net trimmed and the location of the three rib assemblies was carefully laid out on the IML skin of the one panel. The three ribs were rigged into position and securely spring-clamped to the cover forward and aft closeout channels (SS79-00250-005 and -006) after establishing the optimum inplane condition for the aft spar attach caps (SS79-00251-003) and the open rib Pi caps (SS79-0025-002) aft of the Tee (SS79-00253-003). At optimum rigging, the open rib Pi caps were inplane while the center rib aft spar attach was out of plane by approximately 0.25-mm (0.010 in.).

In the clamped position, tooling holes were drilled in the rib Pi cap base and the cover IML skin/inner aft closeout channel leg, two places each rib, and slightly aft of the Tee in the rib Pi cap base and the cover IML skin, also two places each rib. Mechanical fasteners were installed in the aft tooling holes and the spring clamps replaced with C-clamps.

The clamped partial assembly was placed on the second cover panel. The two cover panels were squared using large 90° tooling knees and perpendicularity of the three ribs to the cover panels confirmed. The ribs were clamped to the cover panel, tooling holes drilled and mechanical fasteners installed as previously described. Figures 203 and 204 show the TDS in the completed rigged condition and the mechanical fasteners at the aft end holding ribs to covers.

Tees (SS79-00249-004 and -005) were positioned with respect to the rib Tees (SS79-00253-003), clamped in place and tooling holes drilled for precise location.

4.5.6 Assembly of the TDS

Assembly of the TDS was accomplished in three stages as follows:

- o Stage 1 - Bonding of the ribs and front spar Tees to upper and lower cover panels.
- o Stage 2 - Bonding of the lower leading edge cover panel and aft spar to the above.
- o Stage 3 - Mechanically attaching the upper leading edge cover and front spar panels to complete the assembly.

4.5.6.1 TDS Assembly - Stage 1

All bond faying surfaces of the upper and lower cover panels, ribs, and front spar Tees were cleaned to obtain a water break surface and primed as defined in 3.2.4. FM34B-18 adhesive film, .44 Kg/m^2 (0.09 psf), was applied to the prepared surfaces. Covers, rib, and Tees were assembled to the rigged positon established in 4.5.5.

This assembly was prepared for autoclave bonding as shown in Figure 205. The open channel closeouts, at the forward and aft end of the cover panels, were filled with honeycomb core, as shown in Figure 206, to prevent buckling under bonding pressure. All glass breather plies were taped in place to prevent movement and facilitate the bagging operation. The assembly was enclosed in an envelope bag which was appropriately tailored for the two cavities formed between ribs and covers. Thermocouples were placed to monitor the cure. Figure 207 shows the assembly with breather in place ready for bag installation. A view of the envelope bag through one of the two cavities is shown in Figure 208. Five vacuum valve stems were installed in the envelope bag: four active and one static. The bagged assembly was autoclave cured under full vacuum and 172 KN/m^2 (25 psi) augmenting pressure at 191°C (375°F) for two hours.

After cure all bagging materials were removed and the bond areas were visually and nondestructively inspected. NDI utilized harmonic bond testing, Figure 209, and ultrasonic pulse echo contact testing with a delay, Figure 210. Manual recordings were made of any discrepant areas.

The bonded assembly was postcured for two hours at (316C) 600°F.

4.5.6.2 TDS Assembly - Stage 2

The bond faying surfaces of the ribs, cover panels, lower leading edge cover and aft spar assembly were prepared for bonding. FM34B-18 adhesive film was applied to the prepared surfaces and the lower leading edge cover and aft spar assembly positioned. The open channel closeouts of the lower leading edge cover and mating, forward, open channel of the lower cover were filled with honeycomb core. A teflon tube was inserted in the aft open channel closeouts of the upper and lower covers as shown in Figure 211. These tubes extended through the bay and provided equalized pressure during cure.

Bagging, cure, and NDI were as noted in 4.5.6.1. Figures 212 through 216 show the assembly with breather plies in place and after bagging. After cure and NDI, the bonded assembly was postcured.

4.5.6.3 TDS Assembly - Stage 3

Attachment holes in the front spar panels were diamond core drilled using an applied trim/drill tool. The drilled panels were positioned to the front spar Tees for match drilling of holes in these attaching members. All drilling operations were performed with proper backup to prevent fiber breakout on drill exit.

The upper leading edge cover shown in Figure 217 was trimmed to net dimensions on the ends and aft edge. This was positioned on the assembly and forward end trimmed to match the rib trim as shown in Figures 218 and 219 respectively.

Attachment locations were laid out on the upper leading edge cover at the aft edge and ribs were diamond core drilled. Potted inserts were installed for the rib attachments. The cover was clamped in the assembled condition and holes match drilled through the ribs and upper Tee elements of the front spar.

The completed TDS has been delivered for installation of strain gages, thermocouples, and deflection transducer mounting pads in preparation for the ground test phase of this program.

5.0 CONCLUSIONS

The objectives of this program, to develop processes and fabricate demonstration components, have been accomplished. Principal accomplishments and their importance to potential application of graphite/LARC-160 material to Aerospace structures are presented in the areas of process development and component fabrication.

5.1 PROCESS DEVELOPMENT

Quality Assurance of the material system has been furthered by implementation of specifications for material procurement and fabrication processing. Also, nondestructive inspection techniques have been advanced by the cooperative efforts of Langley Research Center and Rockwell International in the establishing of "A" standards for C-scan inspection.

The chemical characterization activity demonstrated the applicability of high pressure liquid chromatography (HPLC) techniques for characterizing the chemical composition of LARC-160 polyimide resin and its mechanism of polymerization. However, this investigation also determined that the HPLC methodology needs further refinement to obtain better reproducibility and improved quantification to satisfactorily analyze all components of the LARC-160 system. At present, it can be said that HPLC provides an indication of material acceptability which must be fully substantiated by appropriate mechanical testing.

The Resin Variables Study indicated the basic range within which changes in both resin formulation and processing could occur without degradation. However, it must be noted that the variation matrix presented in the text could be greatly expanded to include more subtle variations which could not be evaluated within the scope of this program.

Specific processing was developed for fabrication of flat laminates, stiffened panels, honeycomb sandwich panels, and chopped fiber moldings. These processes were demonstrated by fabricating demonstration components which were delivered to LaRC.

Developed processes were qualified by fabrication and mechanical testing. The test data presented in this report provides an adequate starting point for further effort directed to the establishment of design standards necessary for effectual application of the LARC-160 system.

5.2 DEMONSTRATION COMPONENTS

The fabrication of demonstration components, laminates, stringer stiffened panels, honeycomb sandwich panels, and chopped fiber moldings, Task (d) through (g), demonstrated the applicability of developed processes to scaled-up structure. Fabrication of the Technology Demonstrator Segment (TDS), a full-size segment of the Space Shuttle aft body flap, demonstrates the applicability of the processes to a manufacturing environment. The TDS is one of the largest all bonded Gr/PI structures fabricated to date.

The results of this program demonstrates that Gr/LARC-160 is a viable material system for structural application. To achieve its potential, however, further development effort must be expended.

6.0 REFERENCE

1. Gibbs, H. H., and J. R. Ness, "Development of Quality Control Techniques for NR150 Polyimide Adhesive and Binder Materials", SAMPE Journal, January/February (1979).

2. Lauver, R. W., W. B. Alston, and R. D. Vannucci, "Stability of PMR-Polyimide Monomer Solutions," 34th Annual Conference of SPI Reinforced Plastics/Composites Institute, New Orleans, Louisiana, January 29-February 2, 1979.

3. Young P. R., and G. F. Sykes, Polymer Preprints, "Characterization and Aging Effects of LARC-160," ACS/CSJ Chemical Congress, Honolulu, Hawaii, April 1-6, 1979.

4. Lauver, R. W., and R. D. Vannucci "Characterization of PMR Polyimides-Correlation of Ester Impurities with Composite Properties," Proceedings 24th National SAMPE Symposium and Exhibition, San Francisco, California, May 8-10, 1979, p. 522.

5. Shuart, M. J. and Herakovich, C. A. "An Evaluation of the Sandwich Beam in Four point Bending a Compressive Test Method for Composites" NASA Technical Memorandum 78783, 1978.

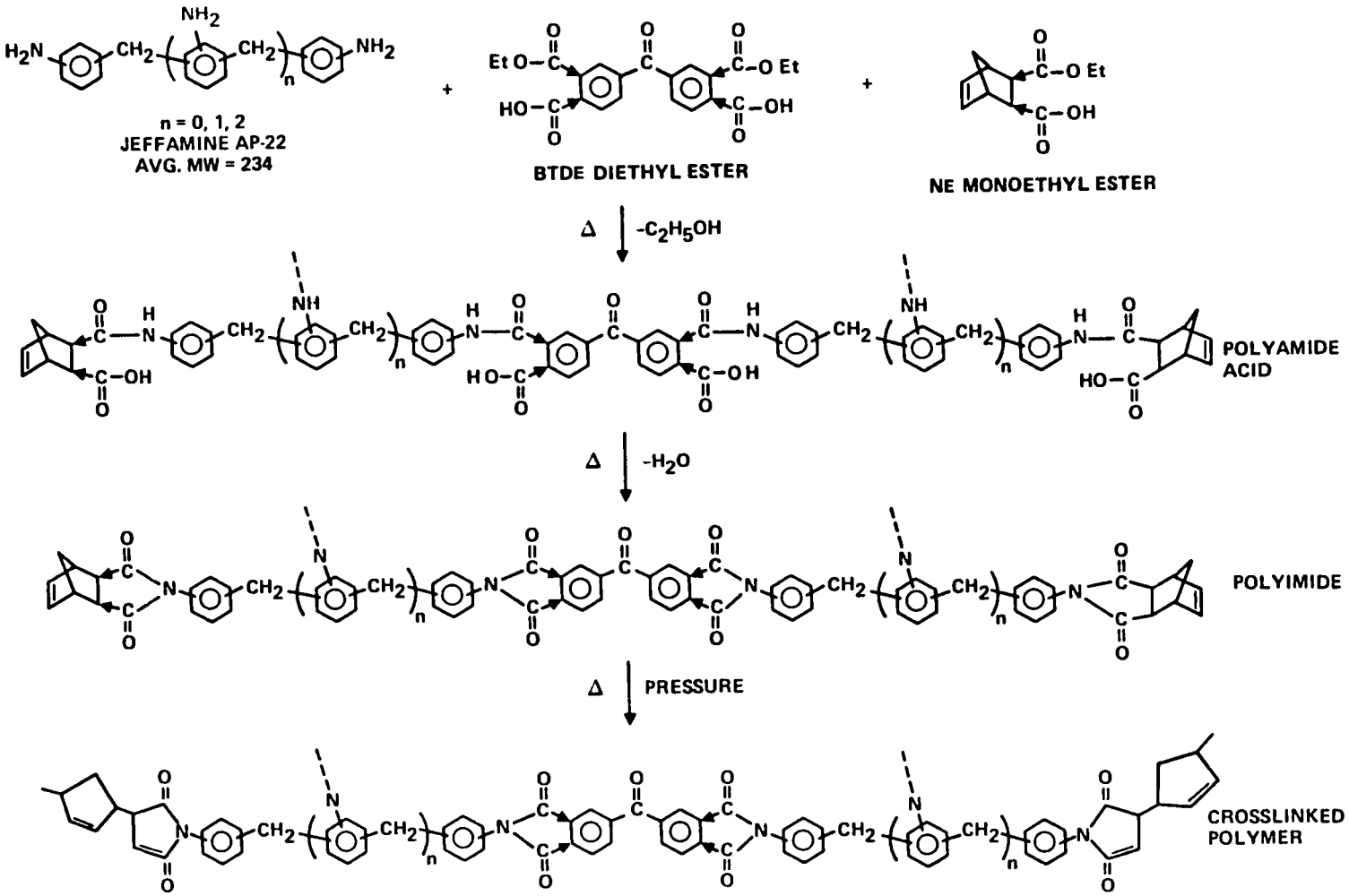


Figure 1. Idealized Polymerization Sequence for LARC-160 Polyimide Resin

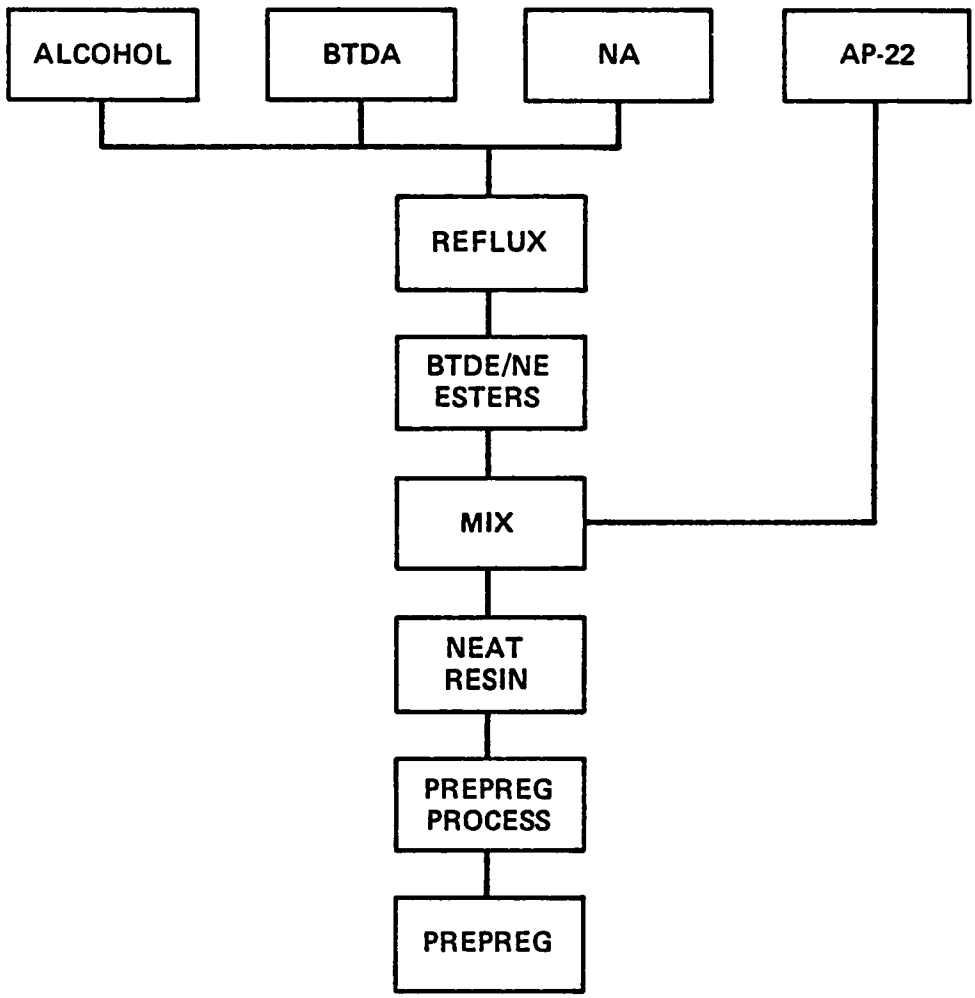


Figure 2. Schematic Outline for the Synthesis of LARC-160 Polyimide Resin

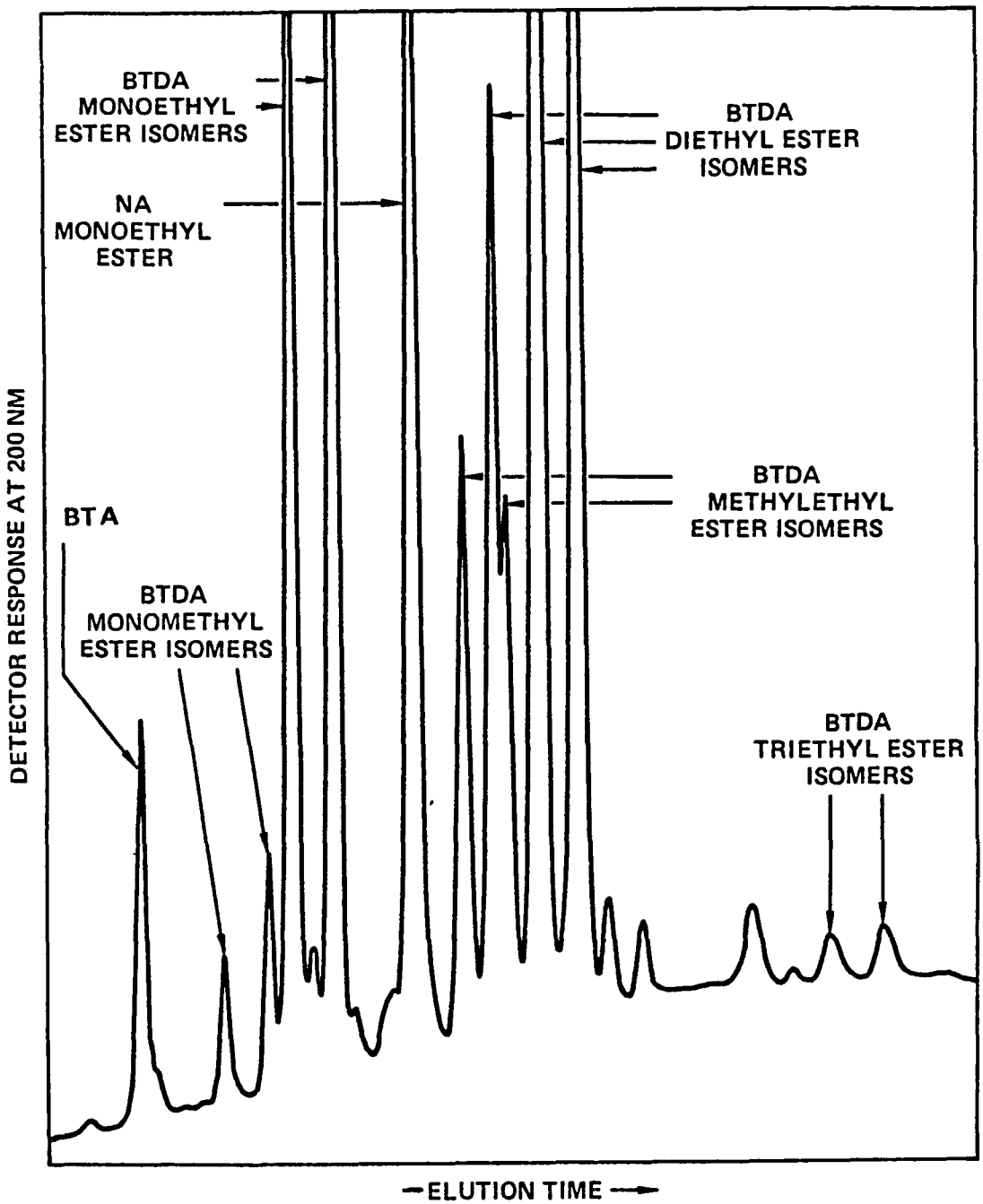


Figure 3. Liquid Chromatographic Separation of LARC-160 Intermediate Ester Mixture

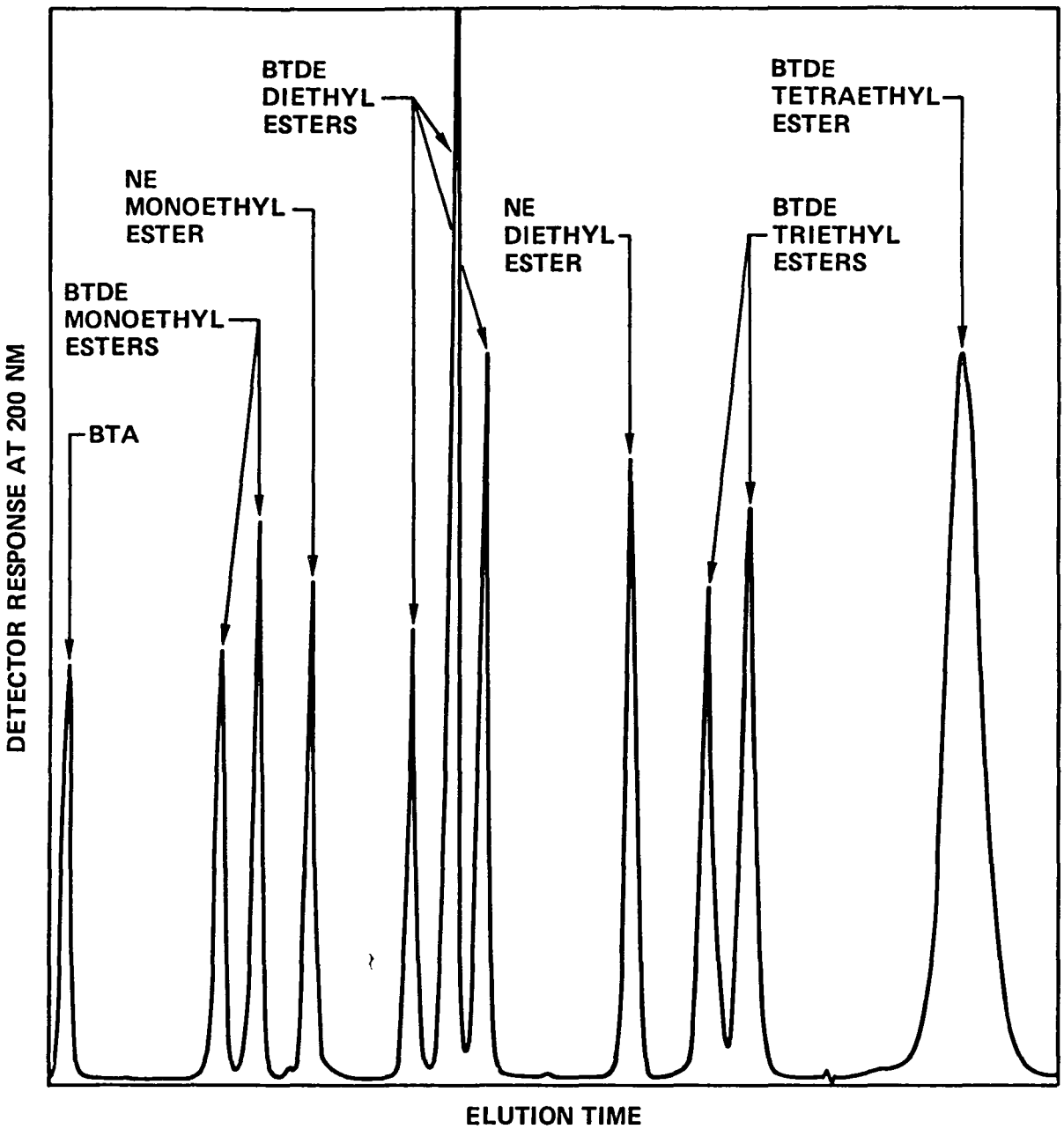


Figure 4. Liquid Chromatographic Separation of Synthetic Mixture of BTDA and NA Ester Compounds

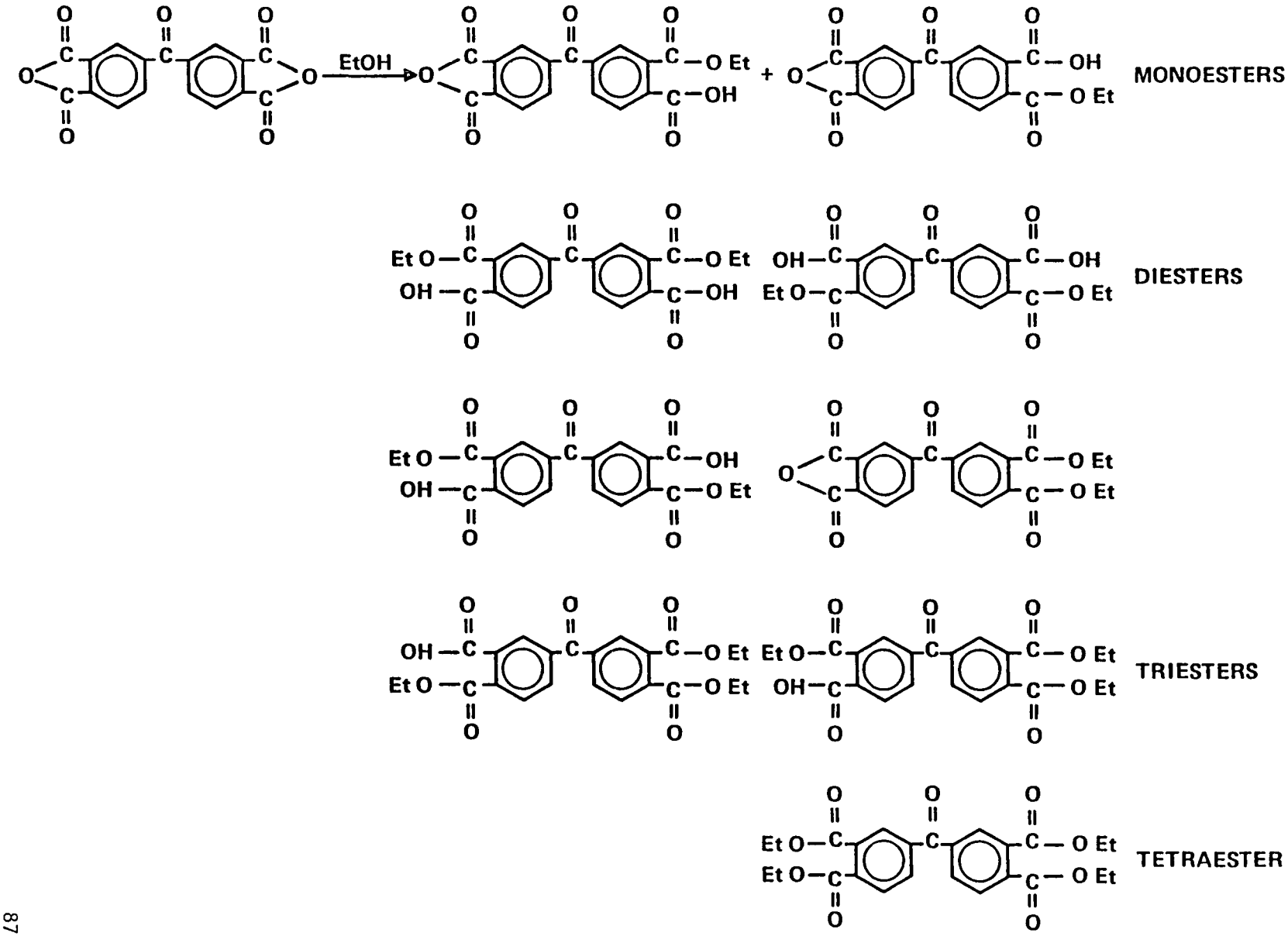


Figure 5. Chemical Structures of Theoretical Isomer Products of BTDA Esterification

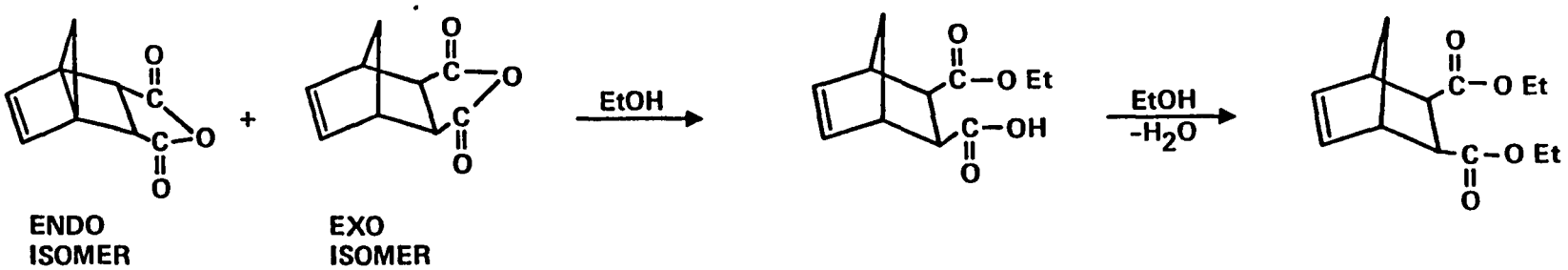


Figure 6. Chemical Structures of Theoretical Esterfication Products of NA

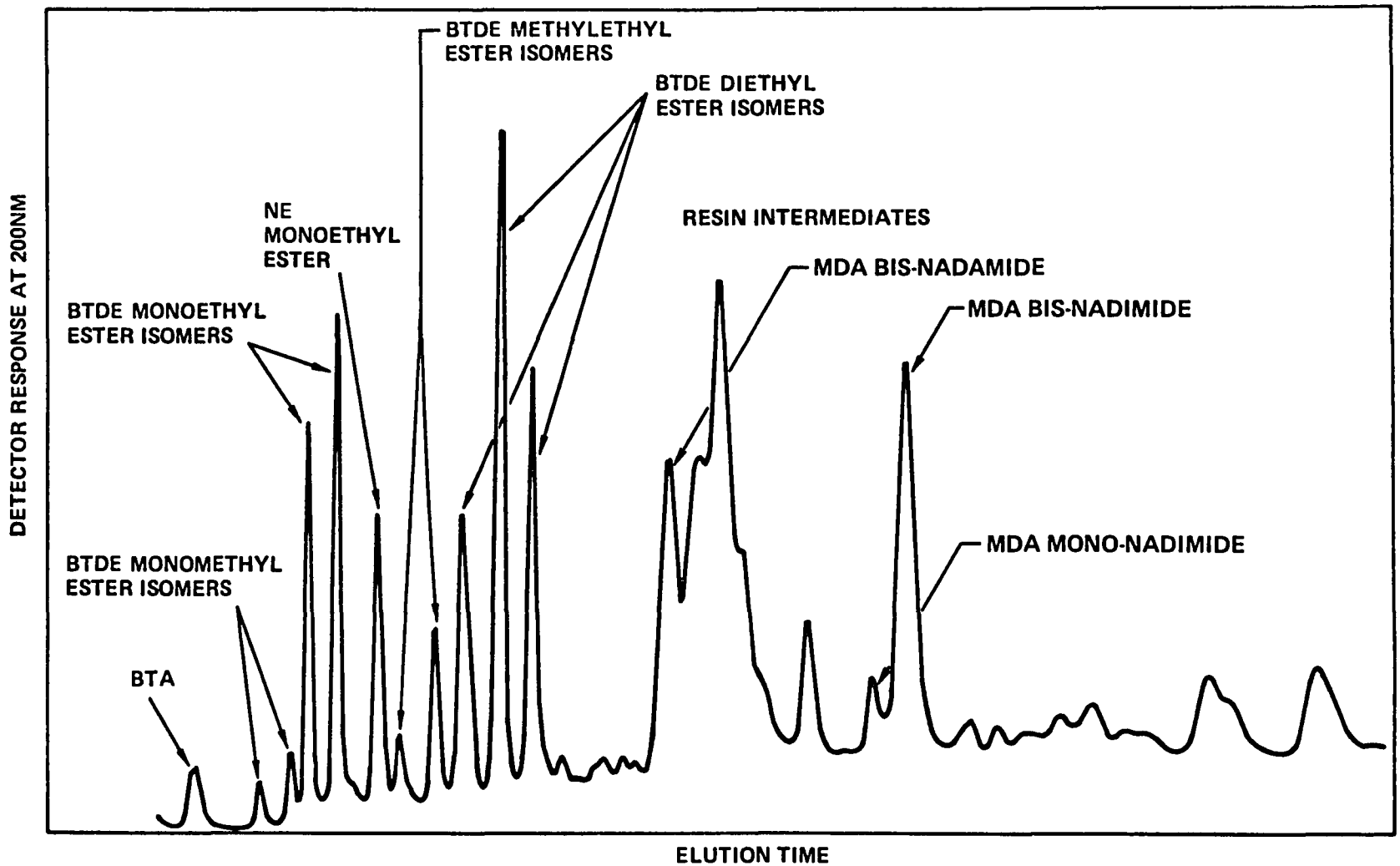


Figure 7. Liquid Chromatographic Separation of LARC-160 Polyimide Resin

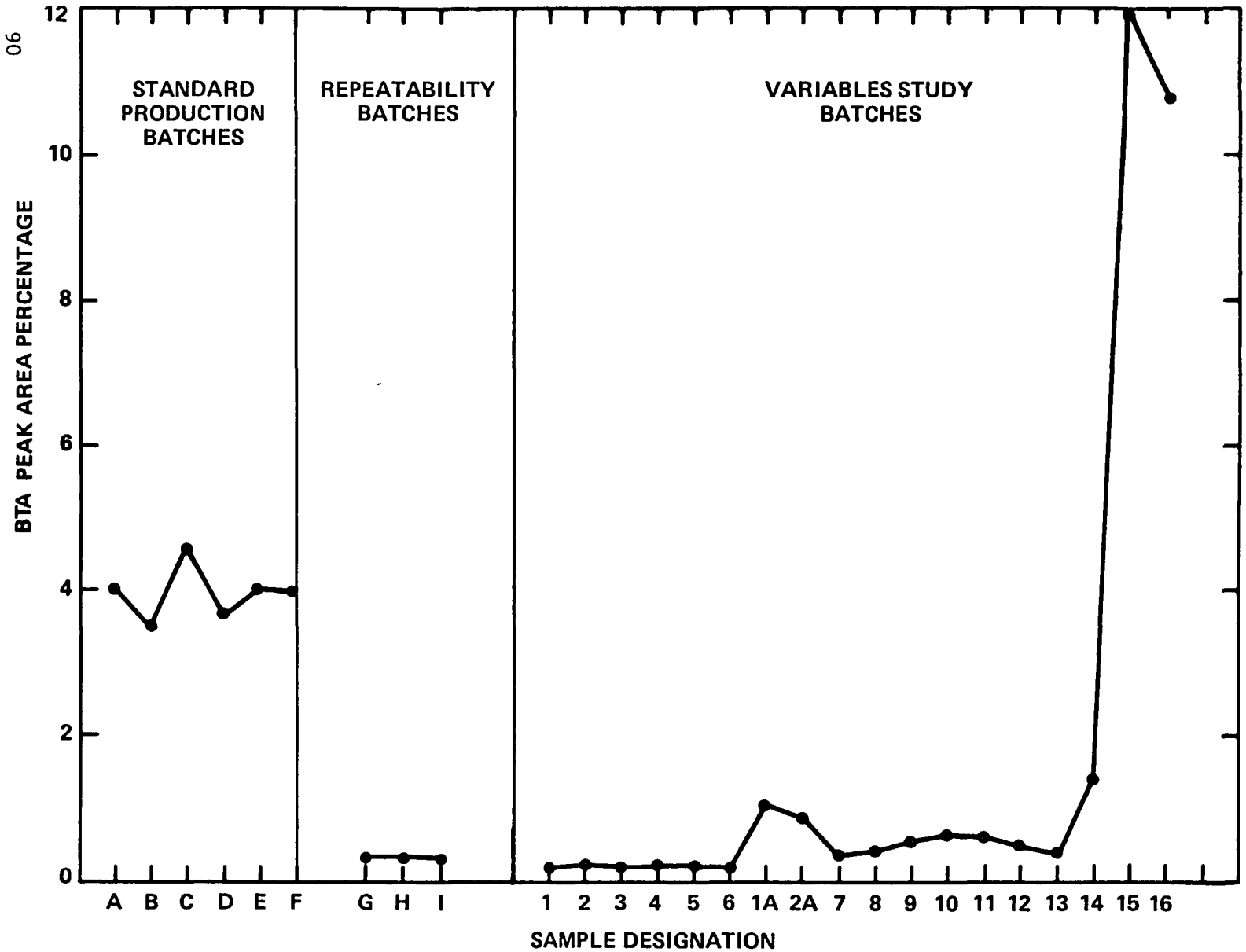


Figure 8. Relative BTA Concentration in LARC-160 Intermediate Ester Mixtures by HPLC Analysis

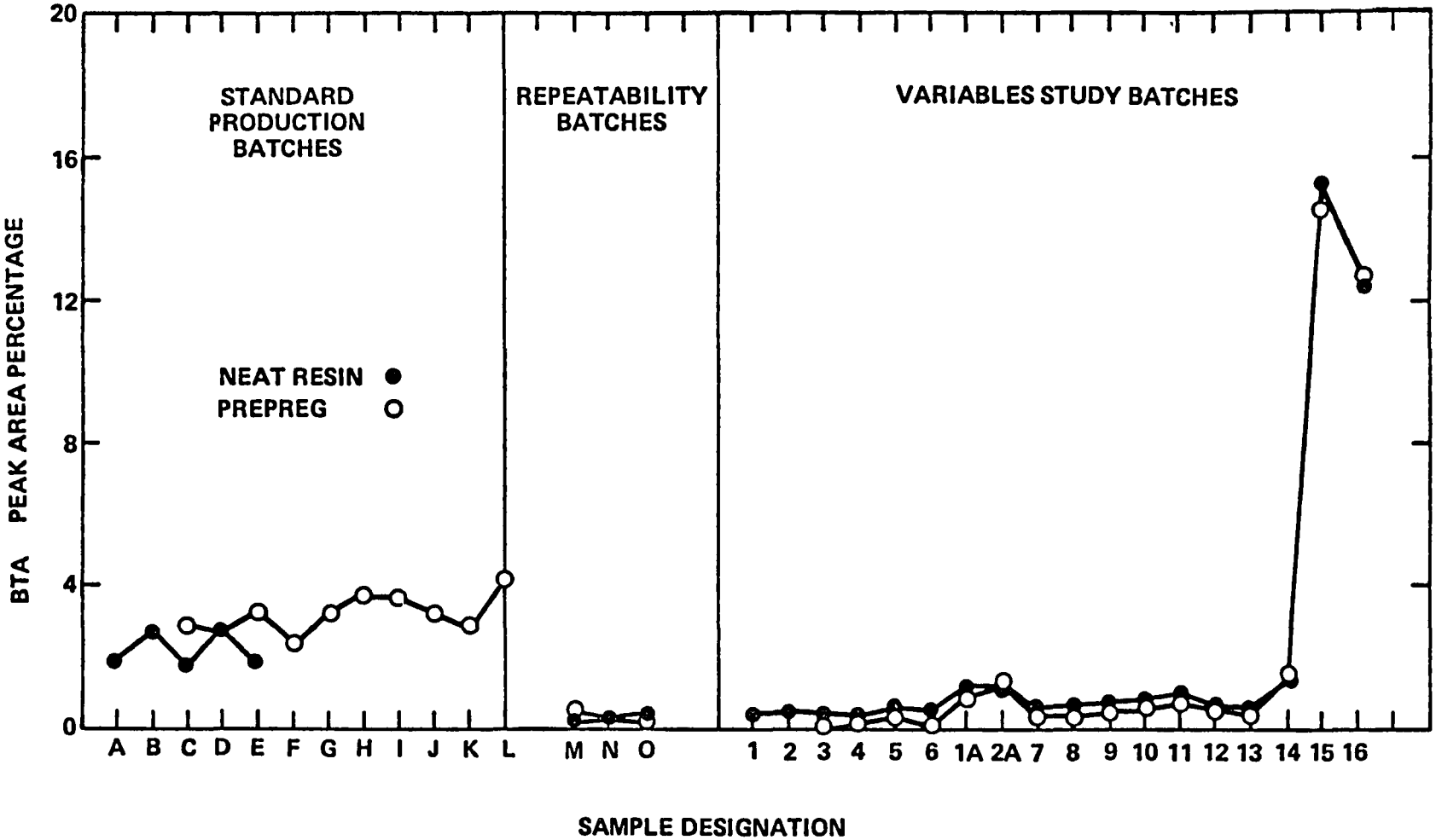


Figure 9. Relative BTA Concentration in LARC-160 Neat Resins and Prepregs by HPLC Analysis

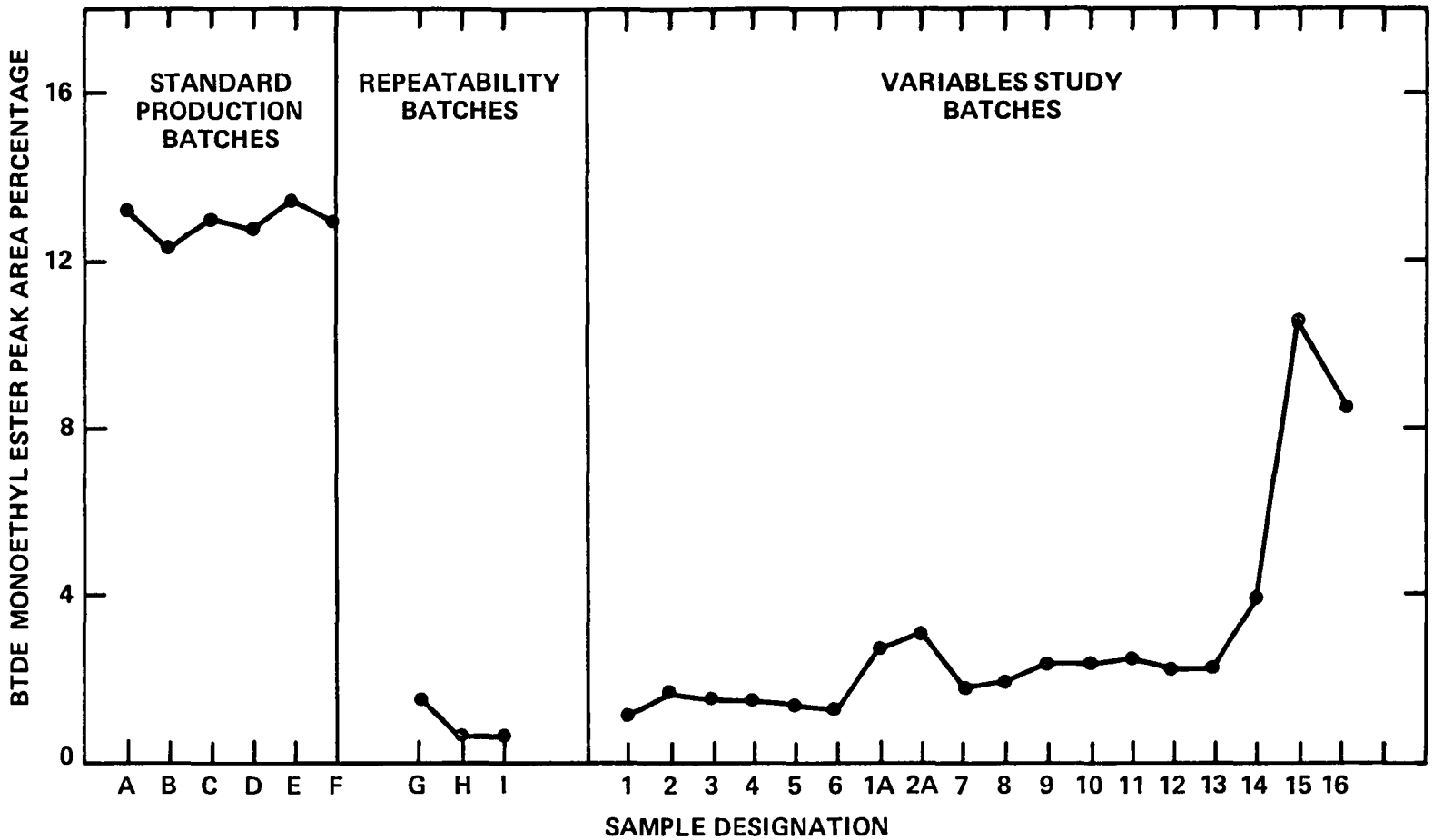


Figure 10. Relative BTDE Monoethyl Ester Concentration in LARC-160 Intermediate Ester Mixtures by HPLC Analysis

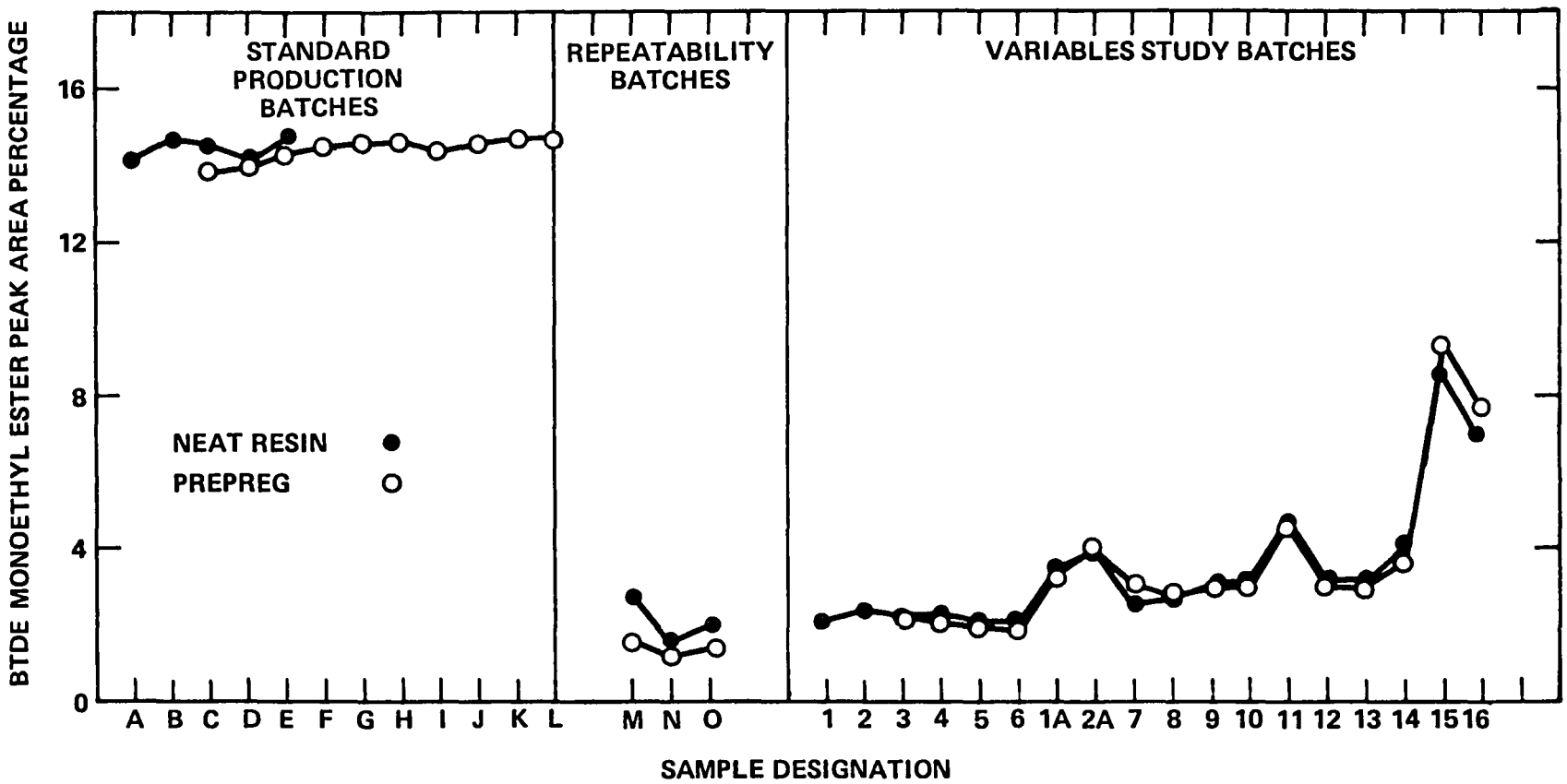


Figure 11. Relative BTDE Monoethyl Ester Concentration in LARC-160 Neat Resins and Prepregs by HPLC Analysis

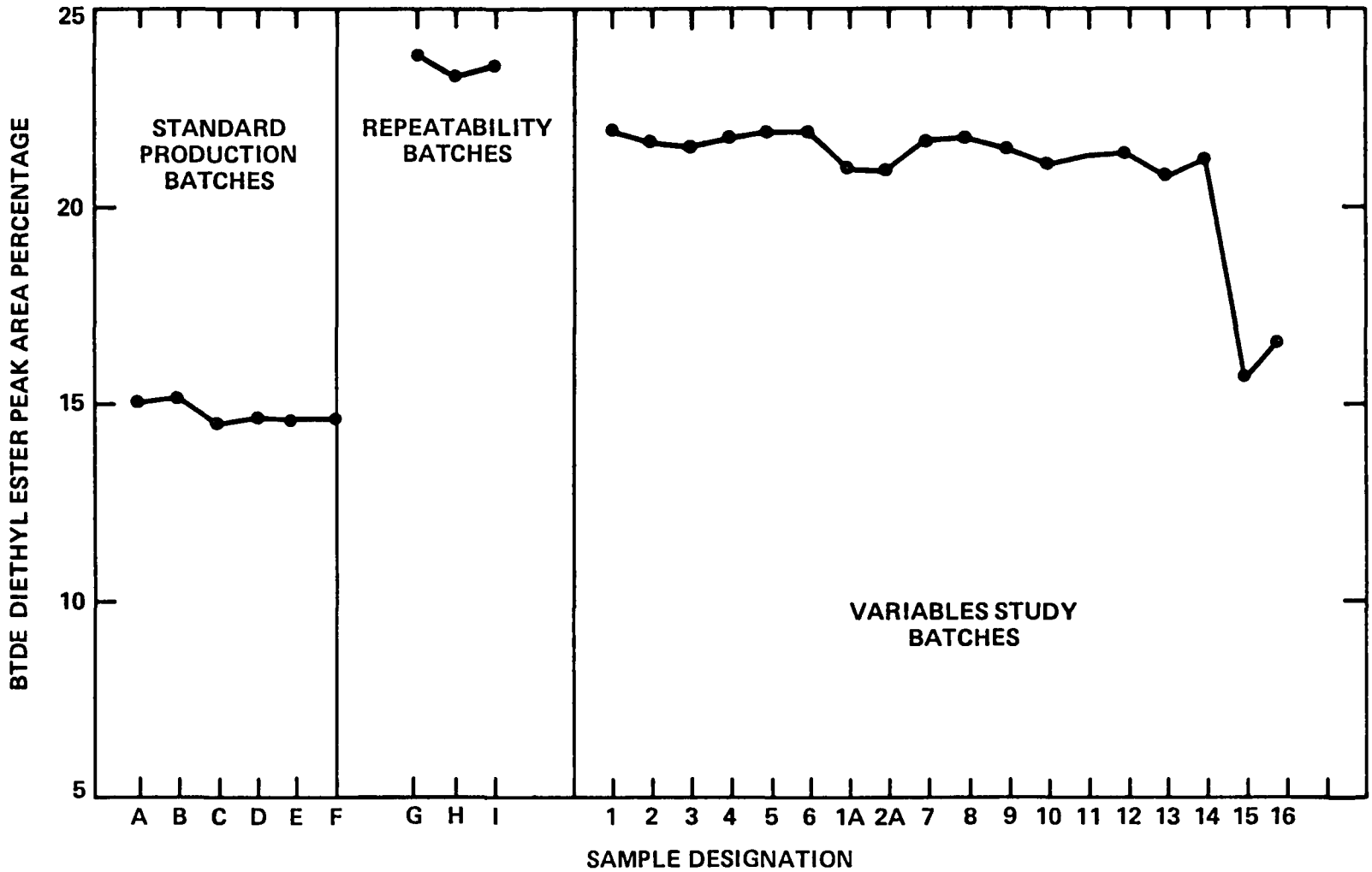


Figure 12. Relative BTDE Diethyl Ester Concentration in LARC-160 Intermediate Ester Mixtures by HPLC Analysis

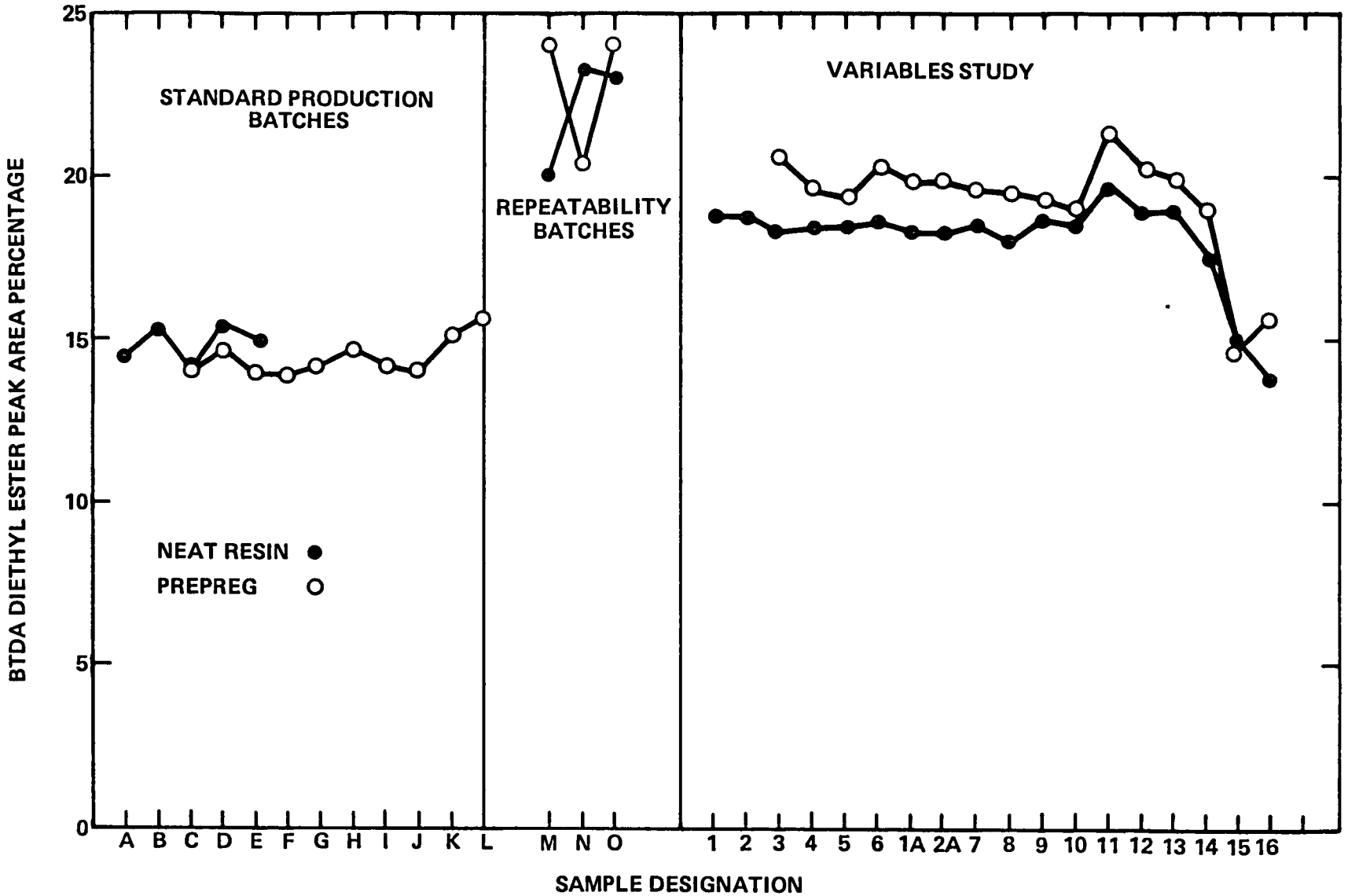


Figure 13. Relative BTDE Diethyl Ester Concentration in LARC-160 Neat Resin and Prepregs by HPLC Analysis

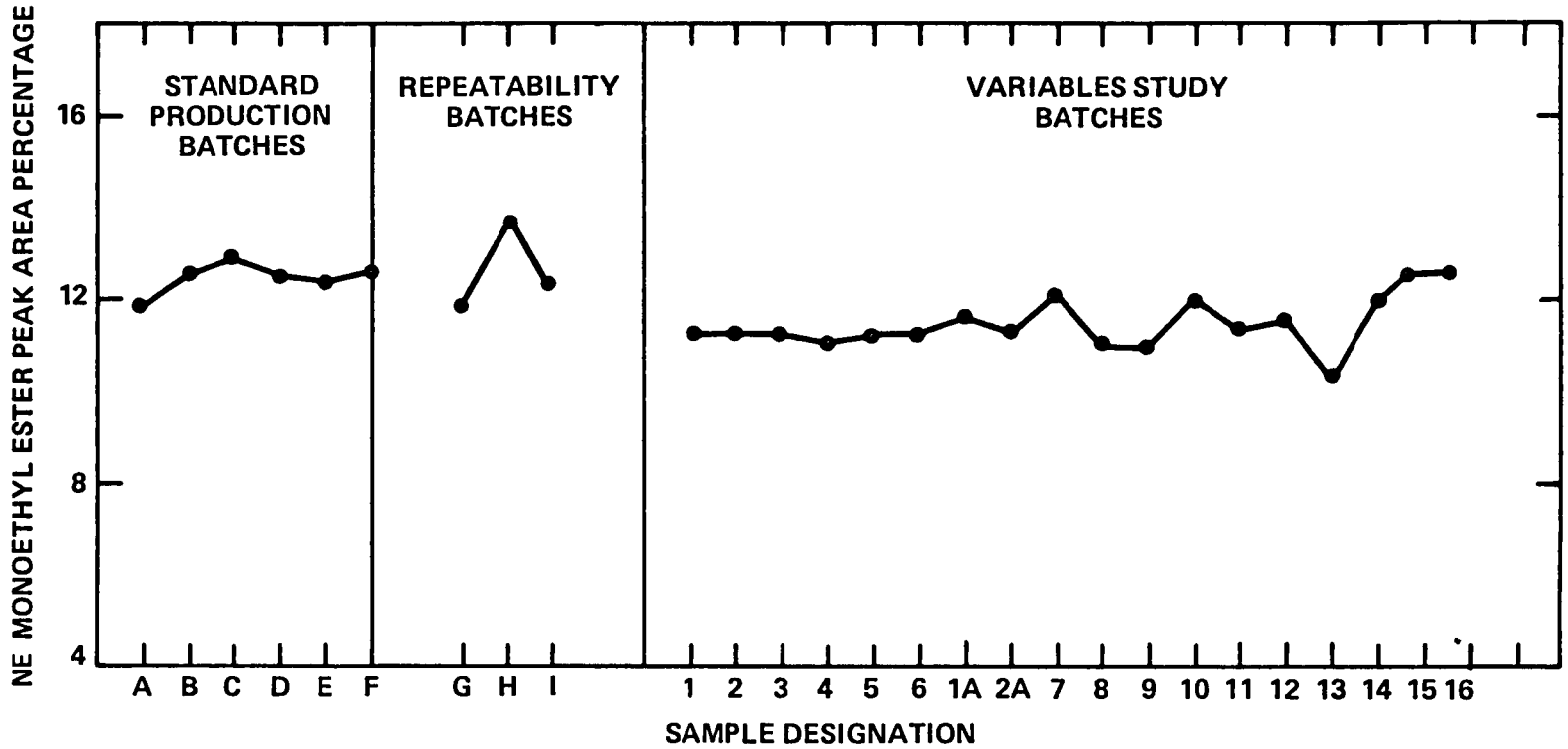


Figure 14. Relative NE Monoethyl Ester in LARC-160 Intermediate Ester Mixtures by HPLC Analysis

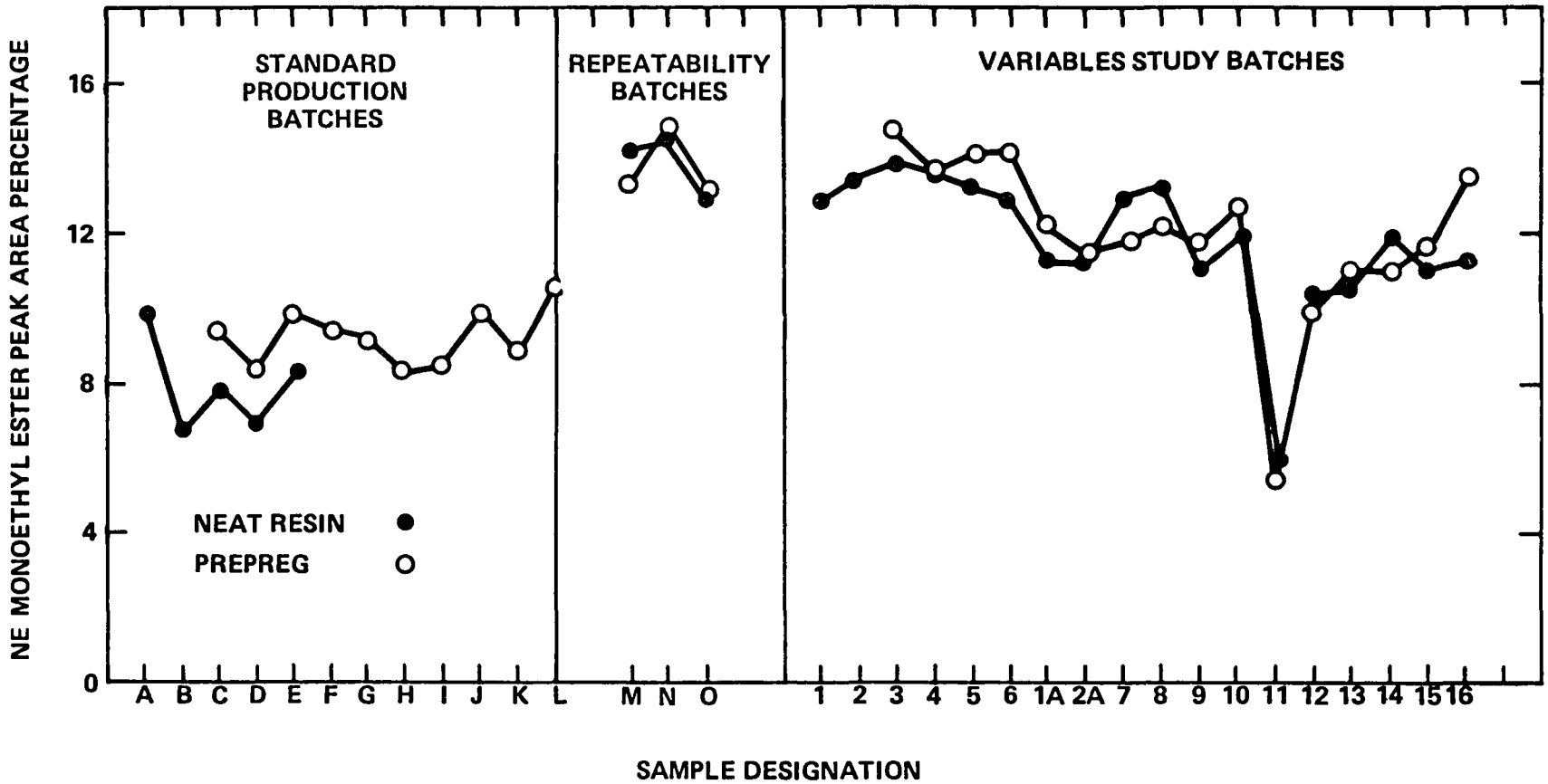


Figure 15. Relative NE Monoethyl Ester in LARC-160 Neat Resin and Prepregs by HPLC Analysis

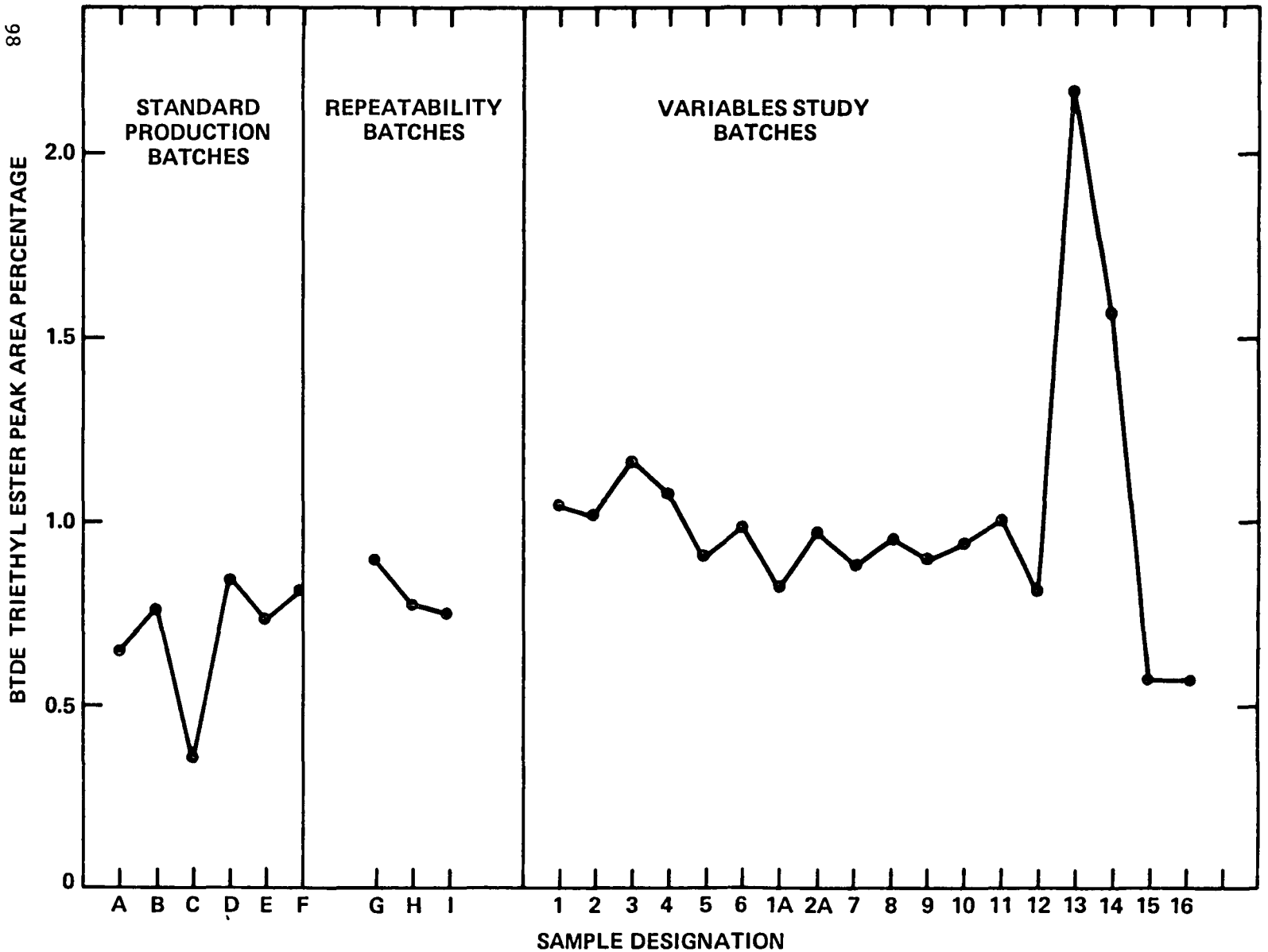
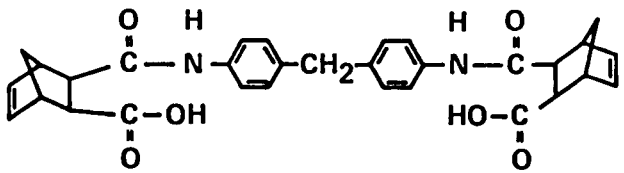
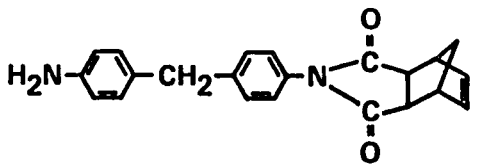


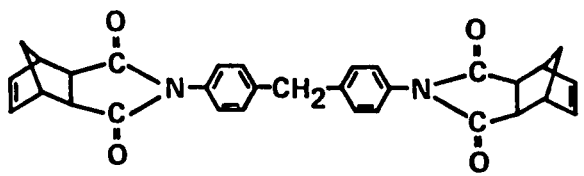
Figure 16. Relative BTDE Triethyl Ester Concentration in LARC-160 Intermediate Ester Mixtures by HPLC Analysis



p,p METHYLENE DIANILINE
BIS-NADAMIDE



p,p METHYLENE DIANILINE
MONONADIMIDE



p,p METHYLENE DIANILINE
BIS-NADIMIDE

Figure 17. Chemical Structures of LARC-160 Resin Intermediates

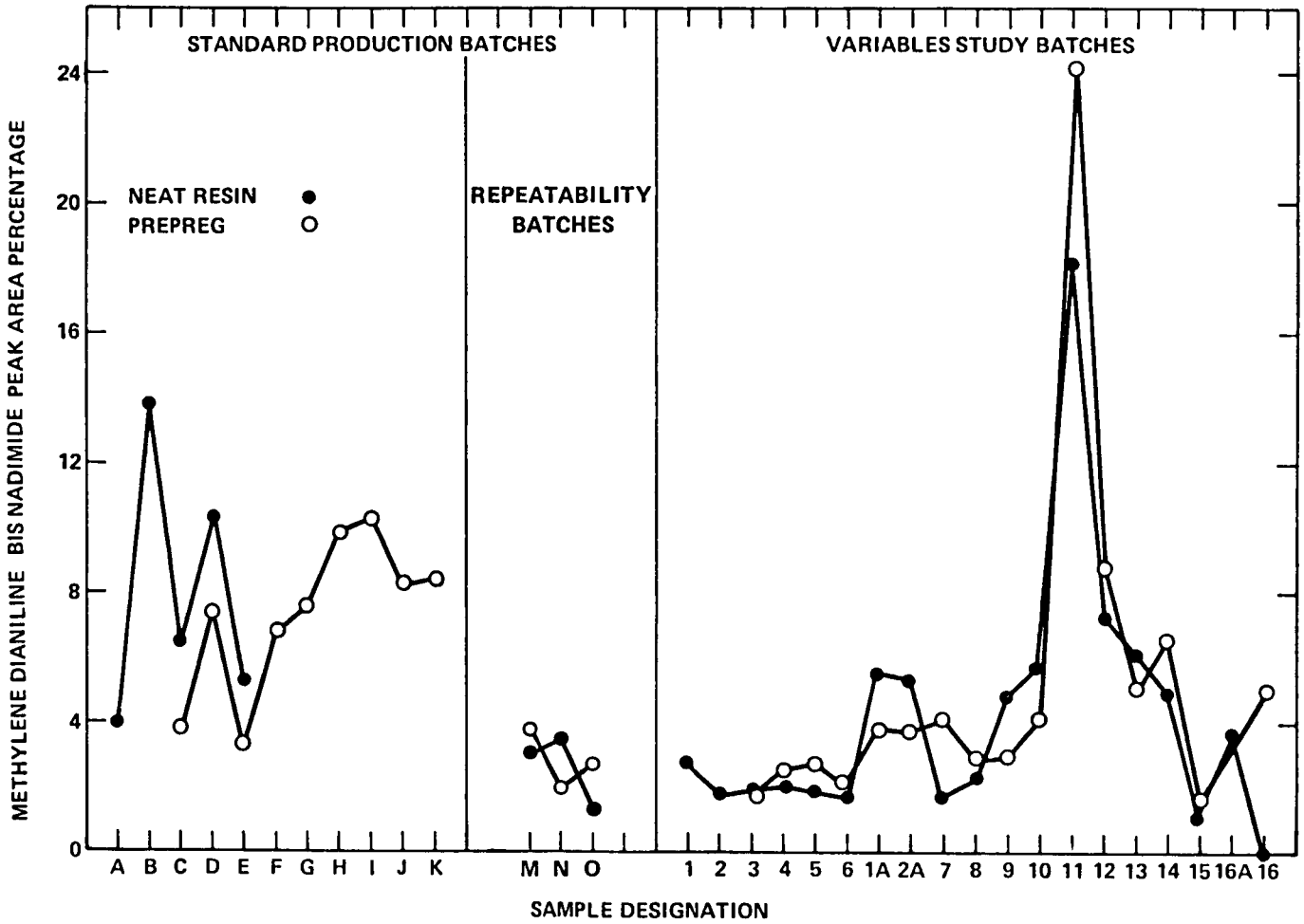


Figure 18. Relative Methylene Dianiline Bis-Nadimide Resin Intermediate Concentration in LARC-160 Neat Resins and Prepregs

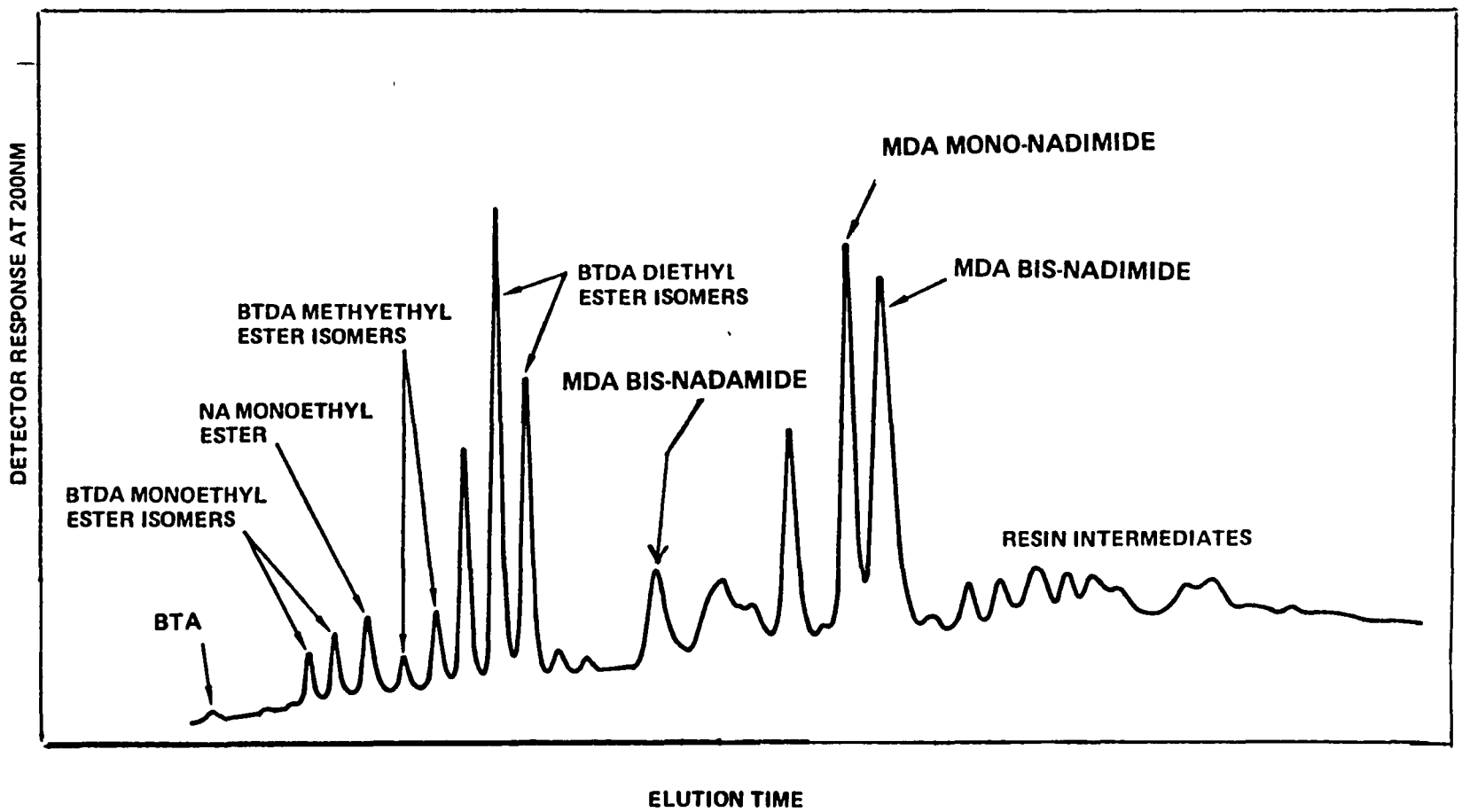


Figure 19. Liquid Chromatographic Separation of LARC-160 Polyimide "Variables Study," Batch 11, Extended Resin Cook Time Variation

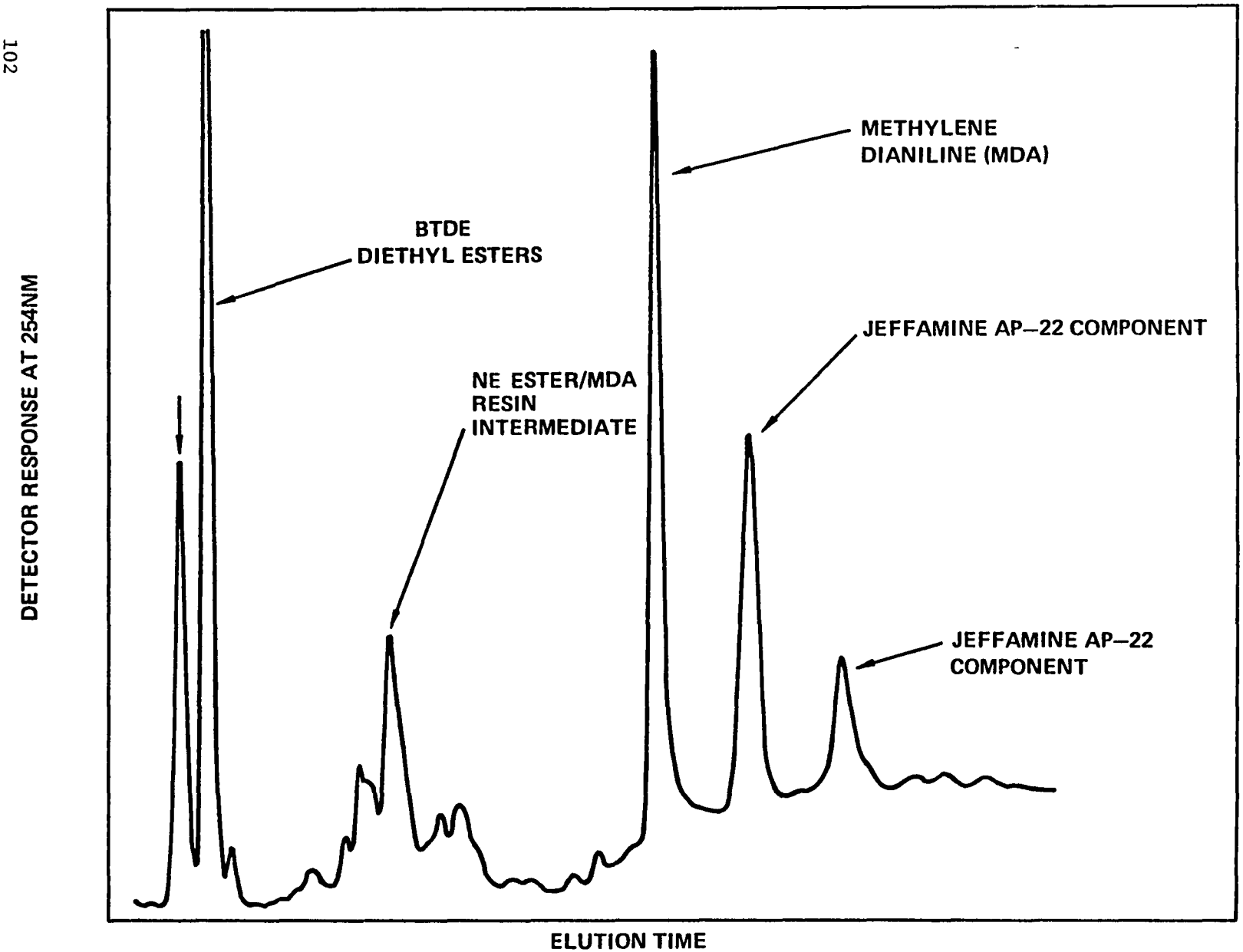


Figure 20. Ion-Pair Liquid Chromatographic Separation of LARC-160 Polyimide Resin

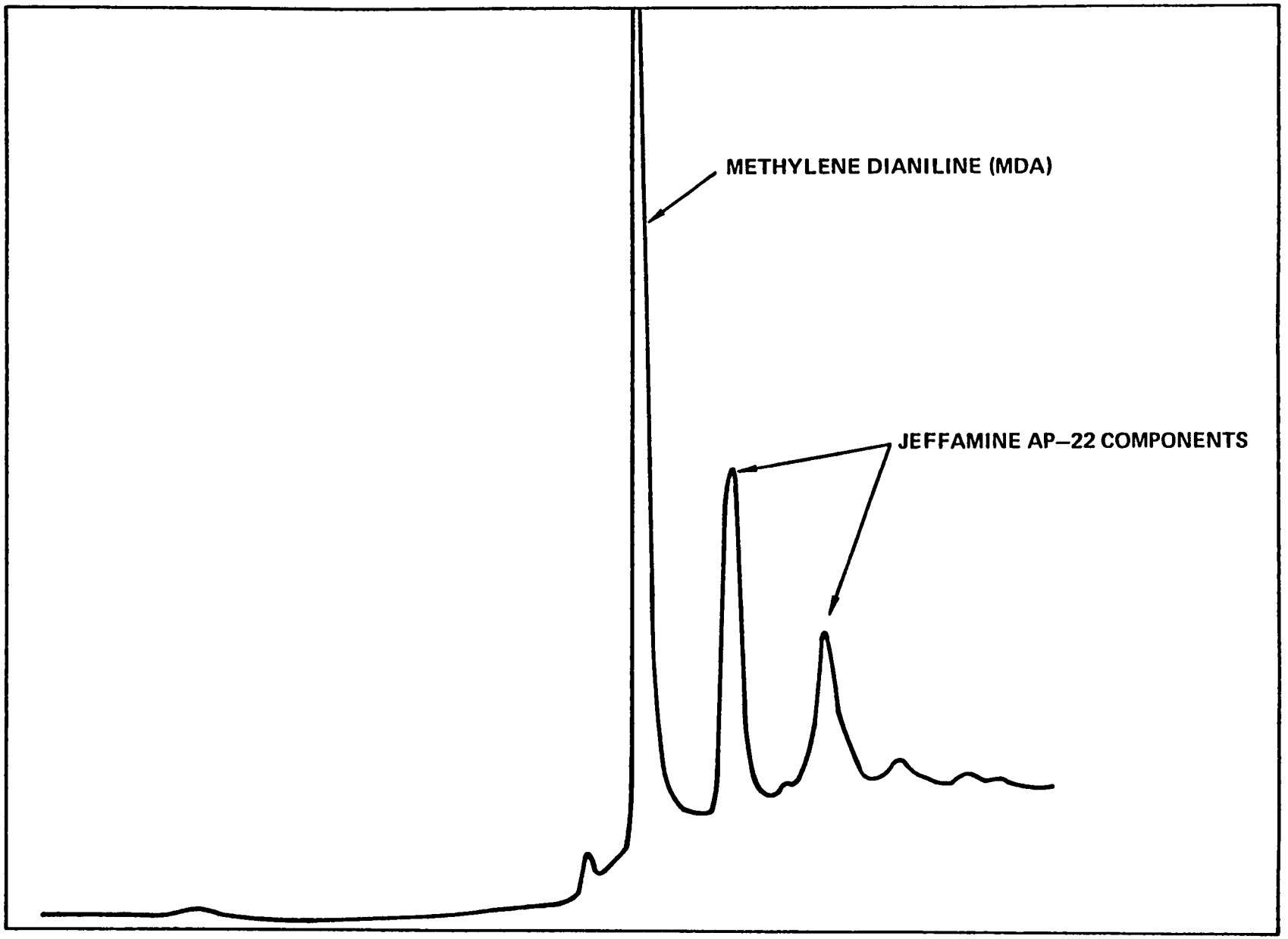


Figure 21. Ion-Pair Liquid Chromatographic Separation of Jeffamine AP-22

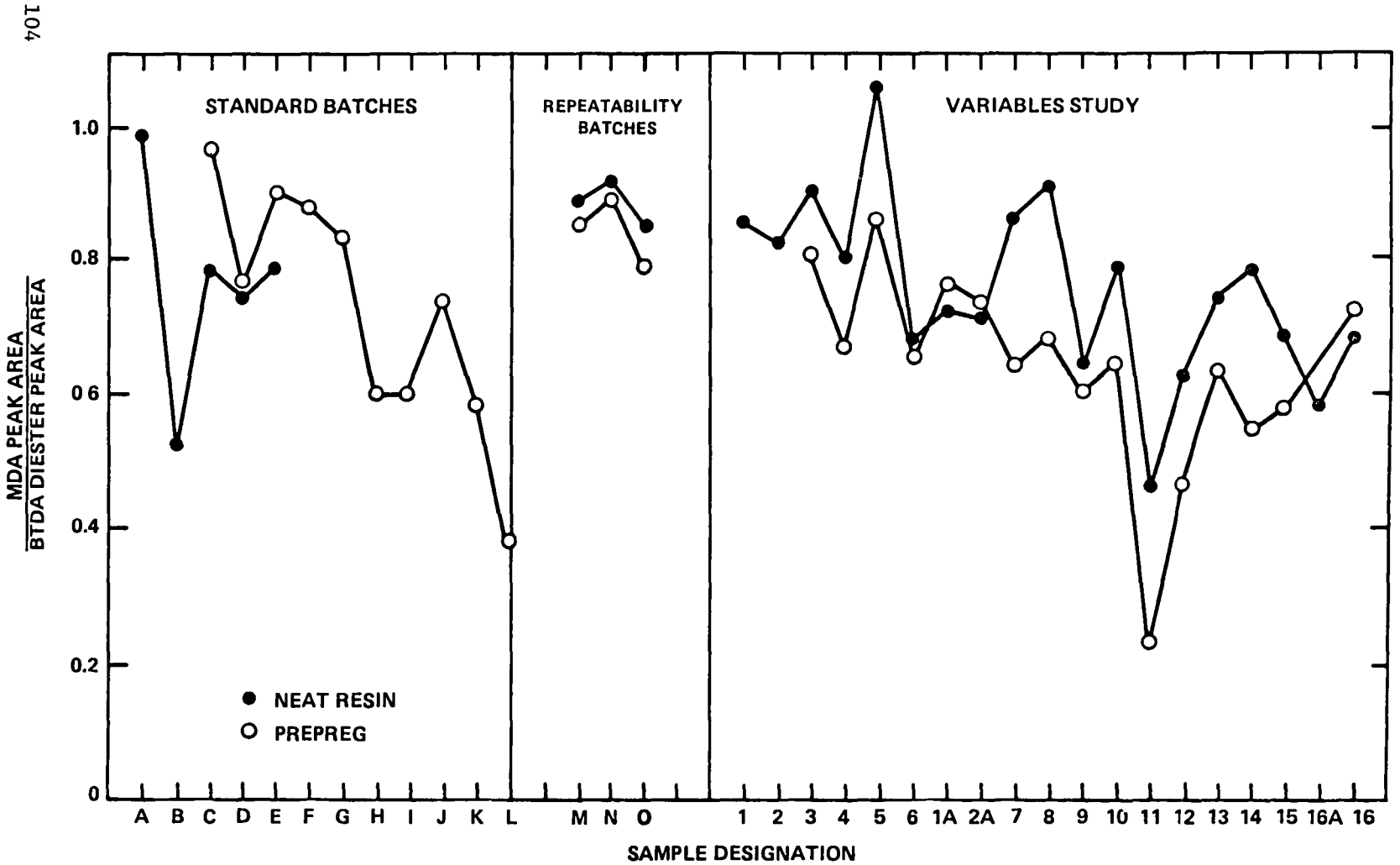
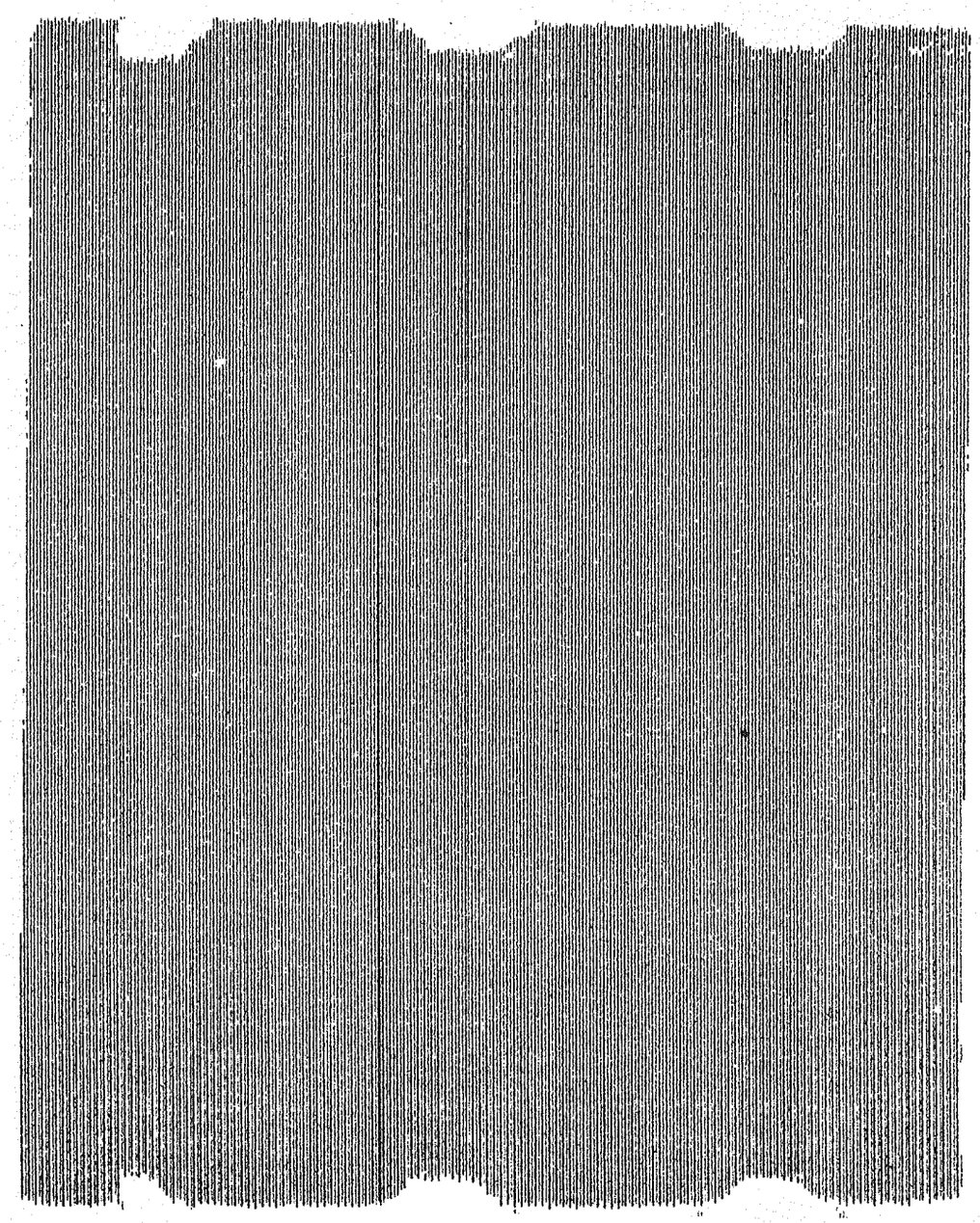
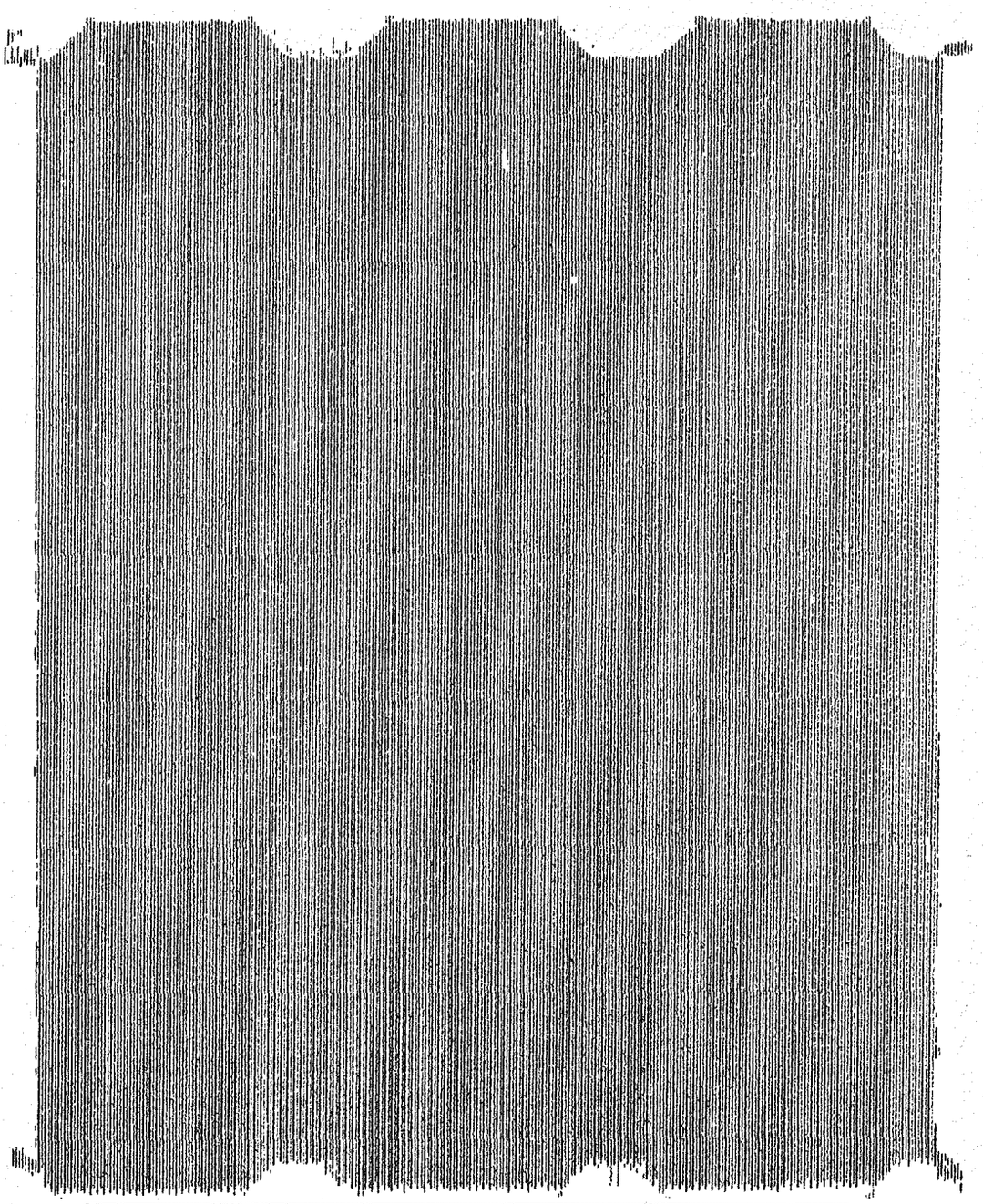


Figure 22. Relative Methylene Dianiline Concentration in LARC-160 Neat Resins and Prepregs by HPLC Analysis



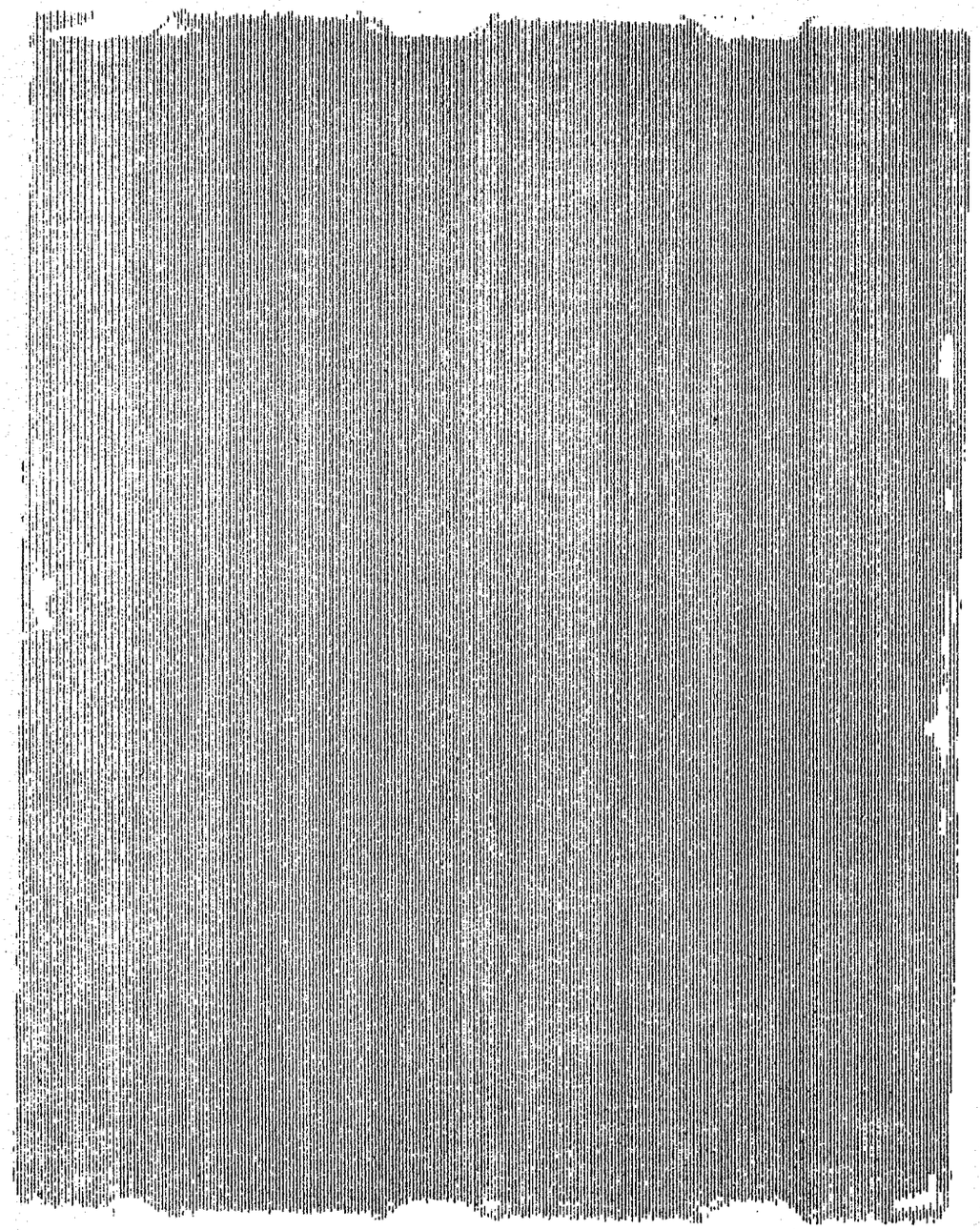
PANEL EX217	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
1 (22990)	+2%	STD	STD	STD

Figure 23. C-Scan Resin Variable No. 1



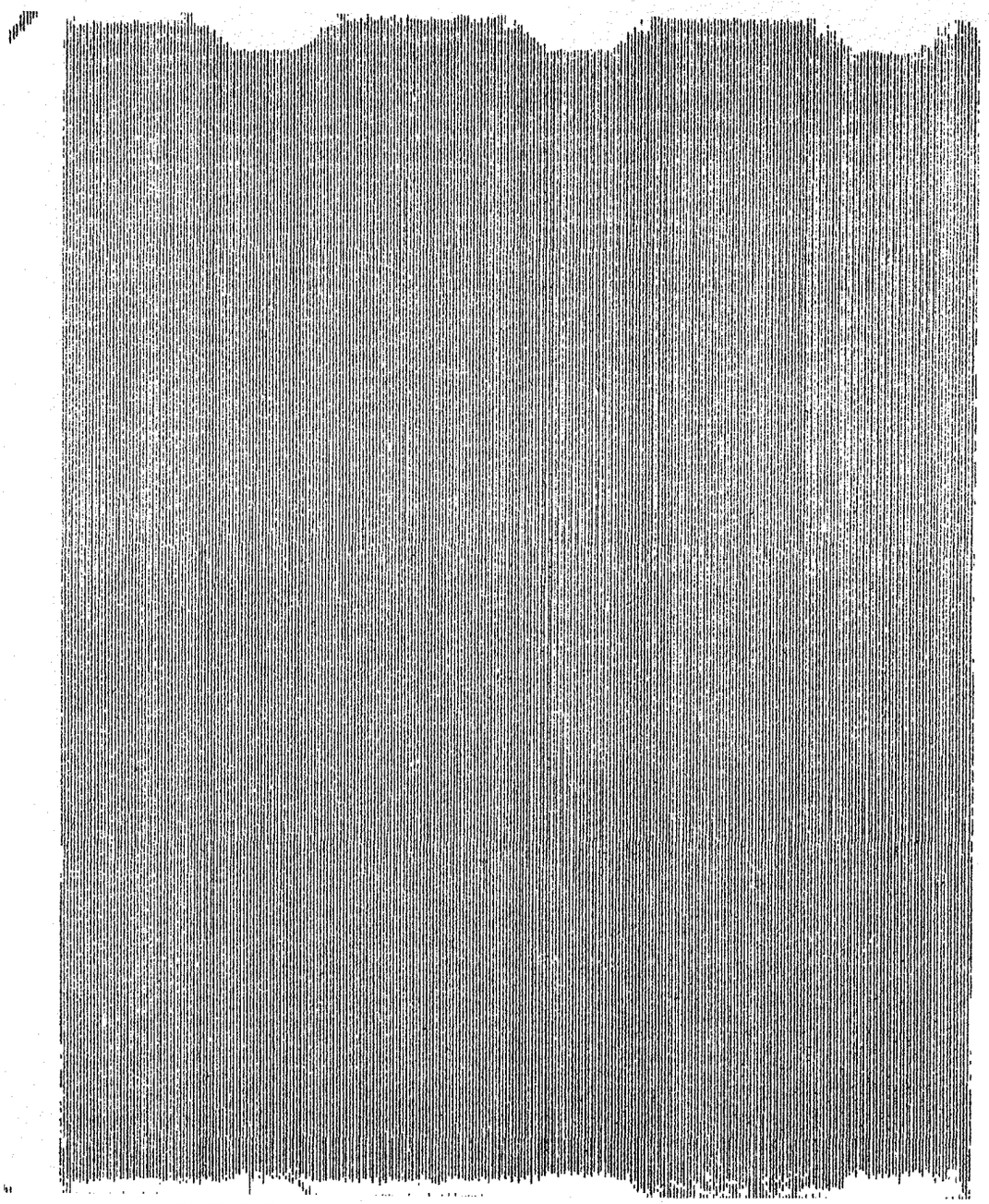
PANEL EX218	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
2 (22991)	-2%	STD	STD	STD

Figure 24. C-Scan Resin Variable No. 2



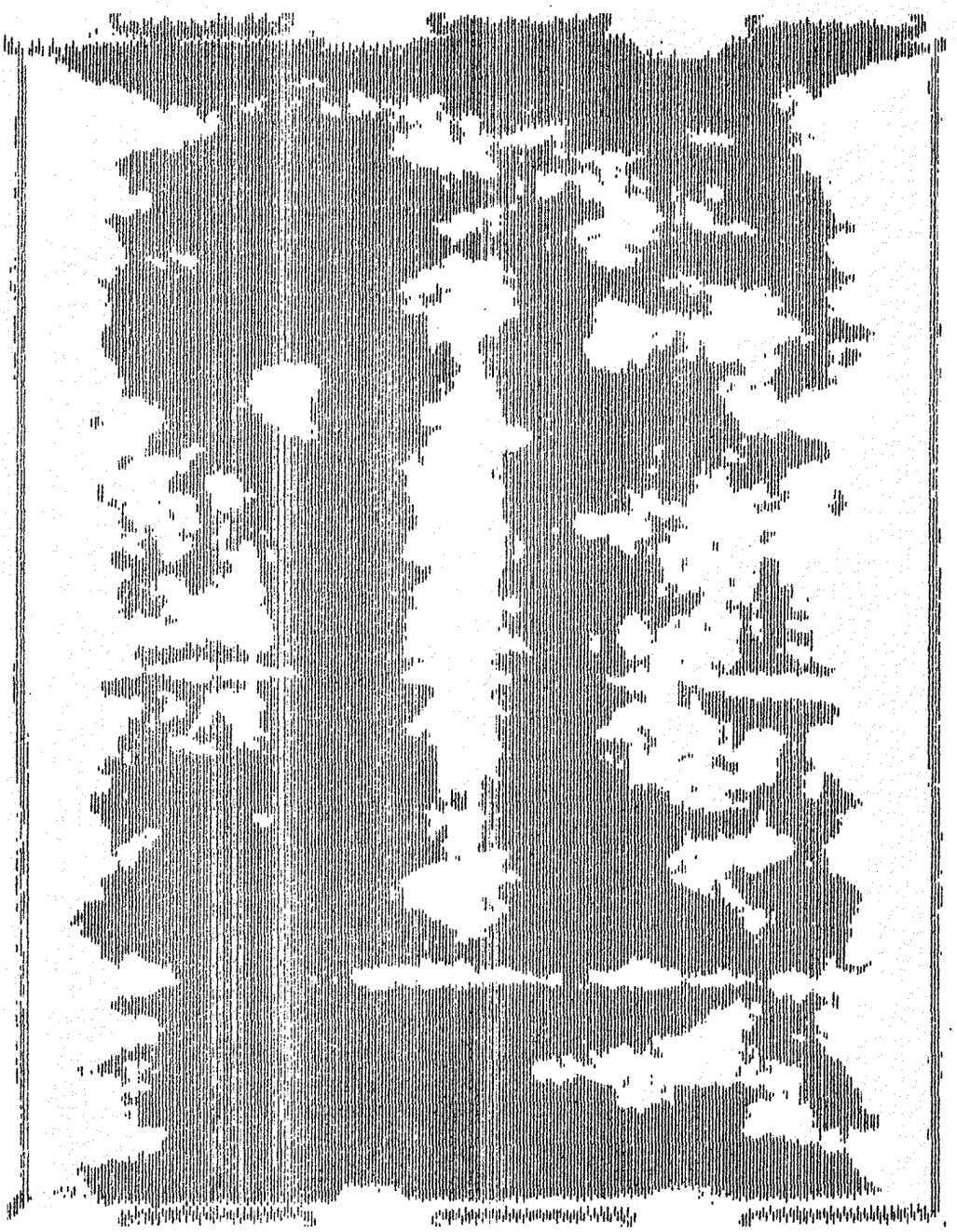
PANEL EX205	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
3 (22945)	+5%	STD	STD	STD

Figure 25. C-Scan Resin Variable No. 3



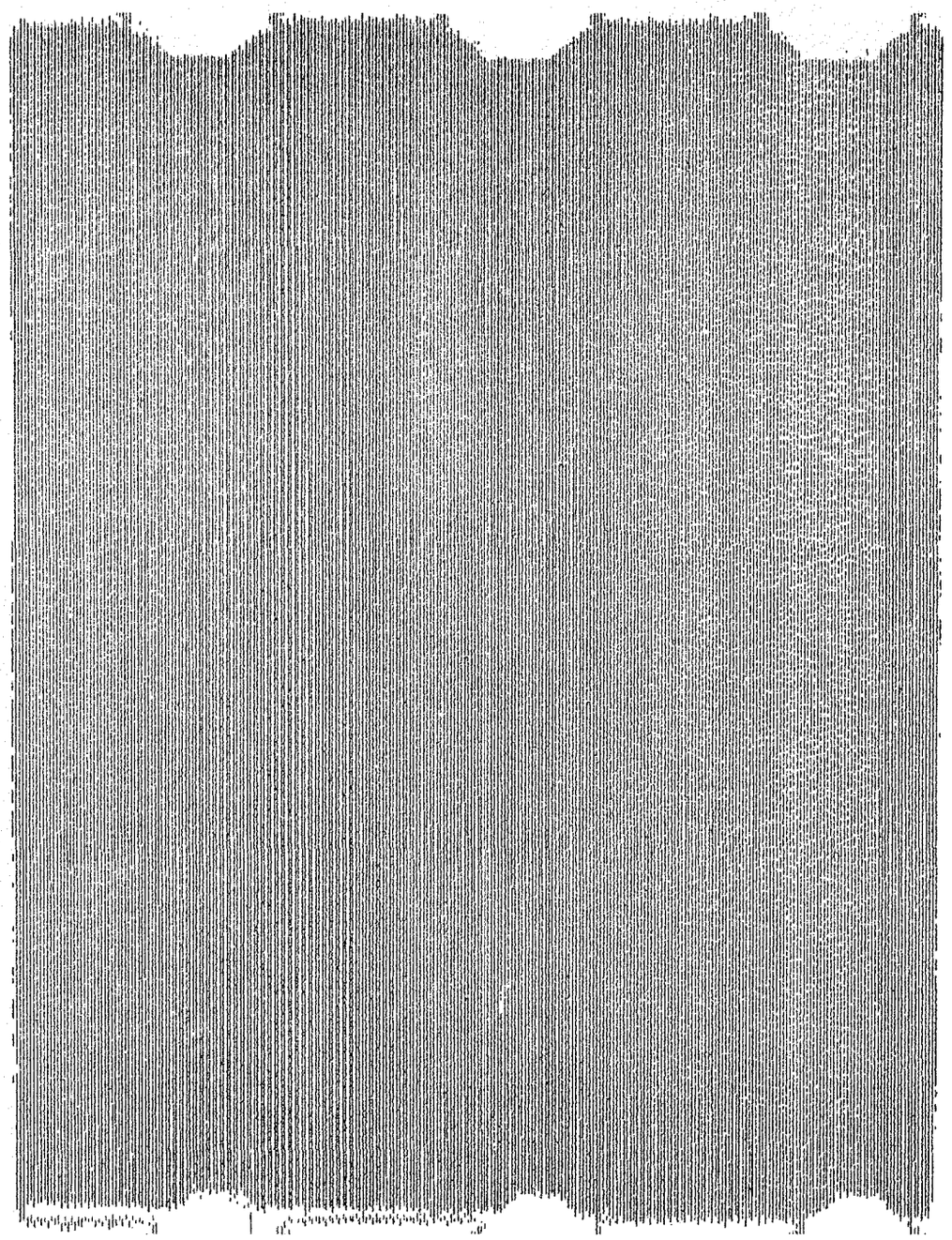
PANEL EX206	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
4 (22946)	-5%	STD	STD	STD

Figure 26. C-Scan Resin Variable No. 4



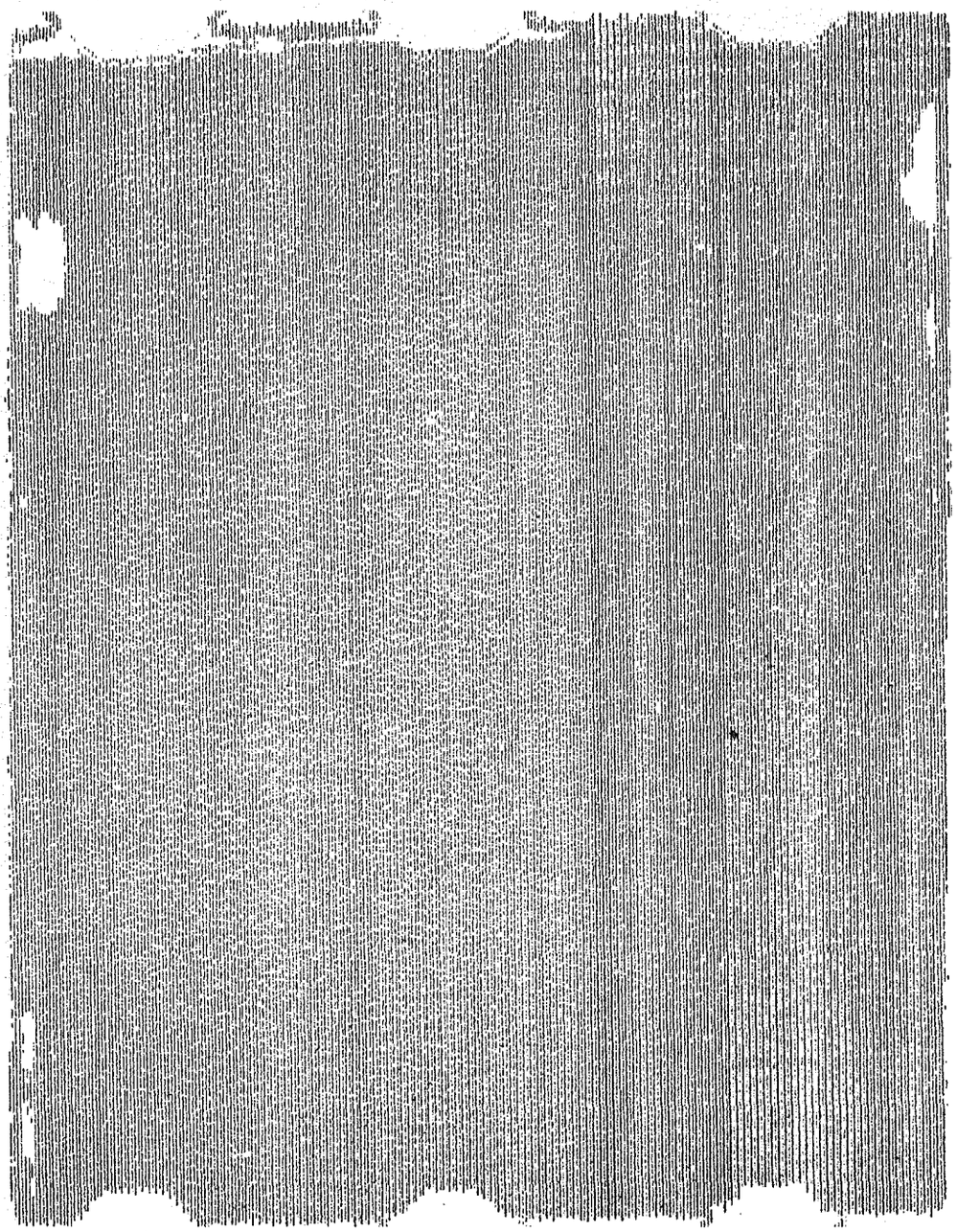
PANEL EX207	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
5 (22947)	+10%	STD	STD	STD

Figure 27. C-Scan Resin Variable No. 5



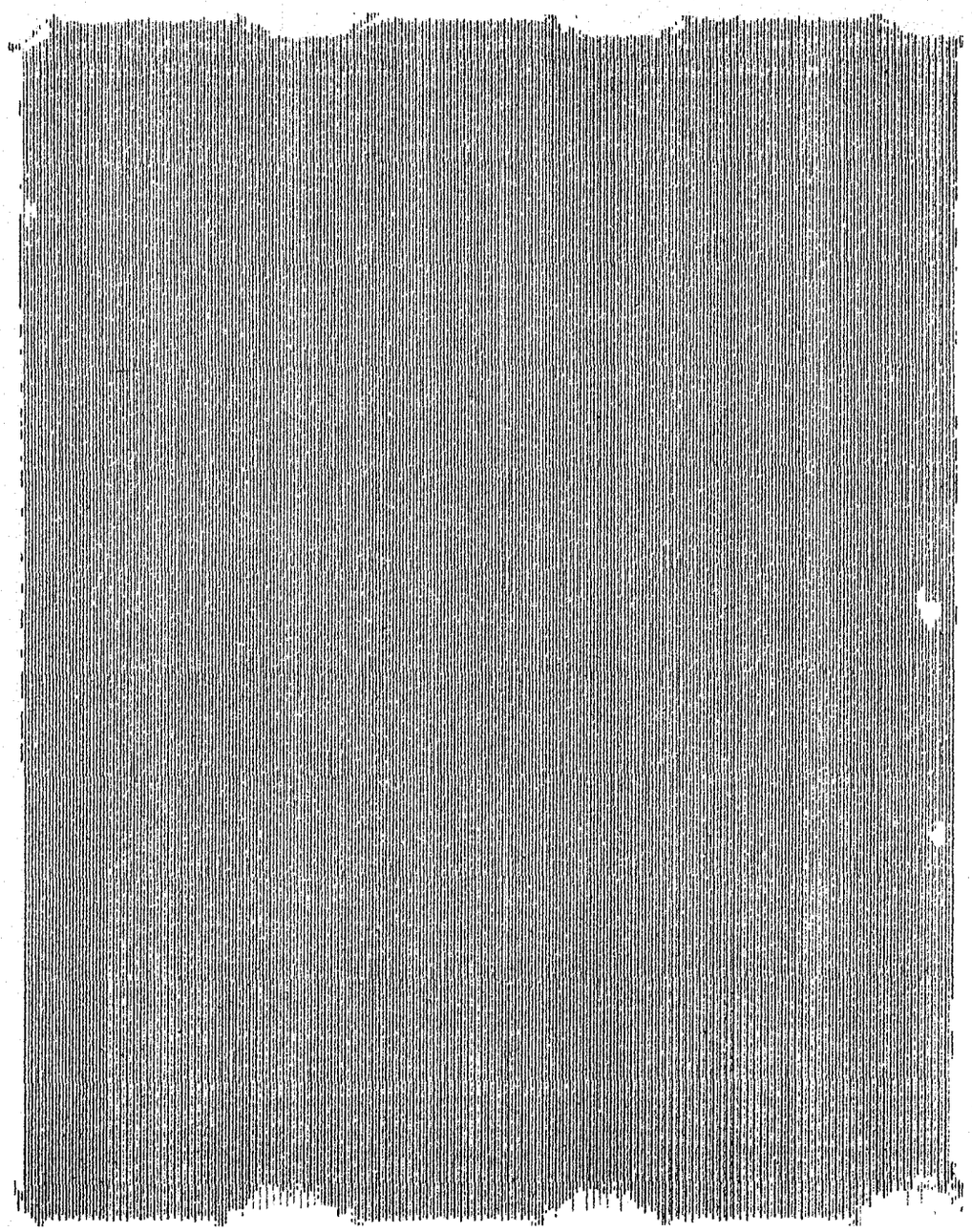
PANEL EX208	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
6 (22948)	-10%	STD	STD	STD

Figure 28. C-Scan Resin Variable No. 6



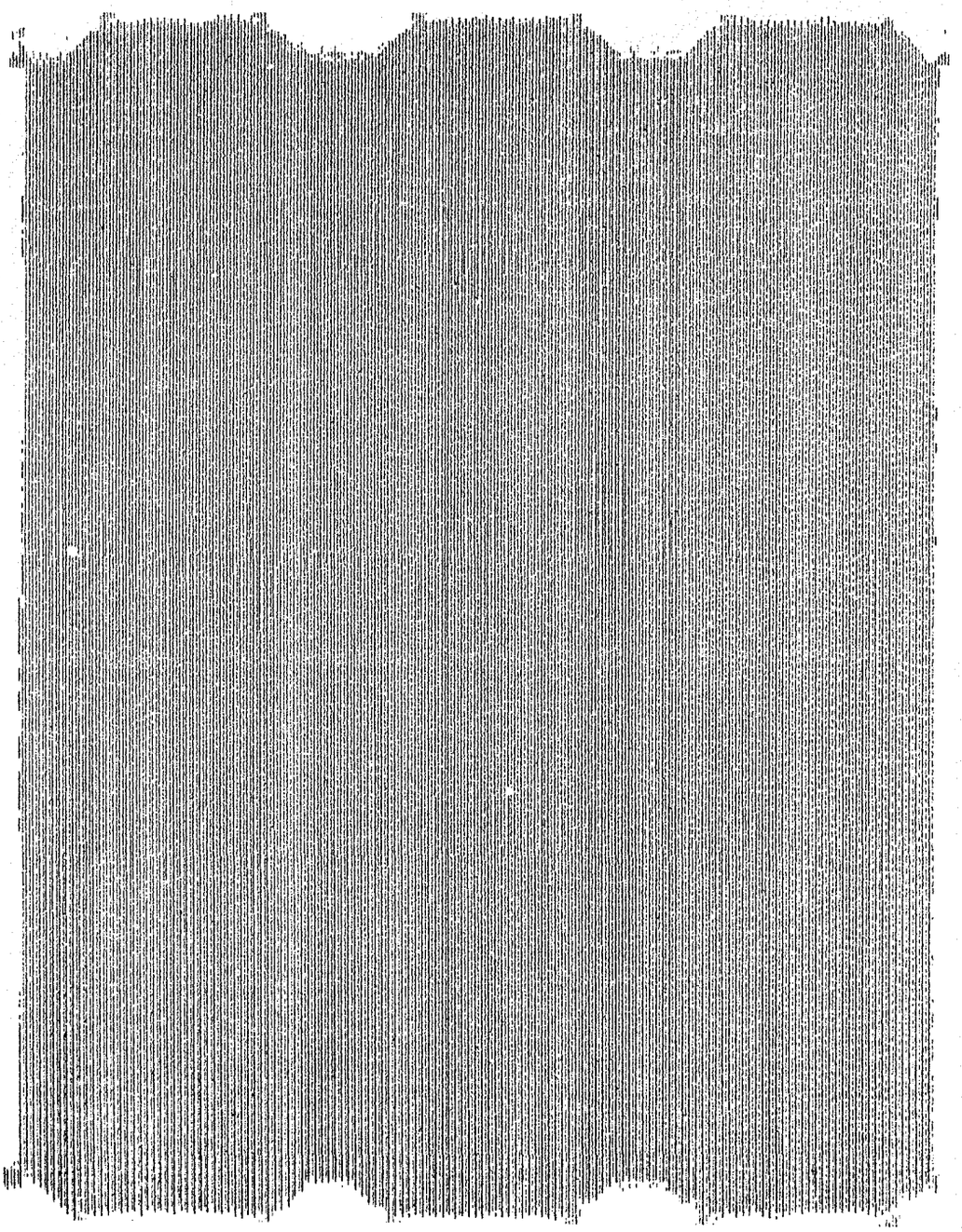
PANEL EX209	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
7 (22949)	STD	NA (+5%) BTDA (STD)	STD	STD

Figure 29. C-Scan Resin Variable No. 7



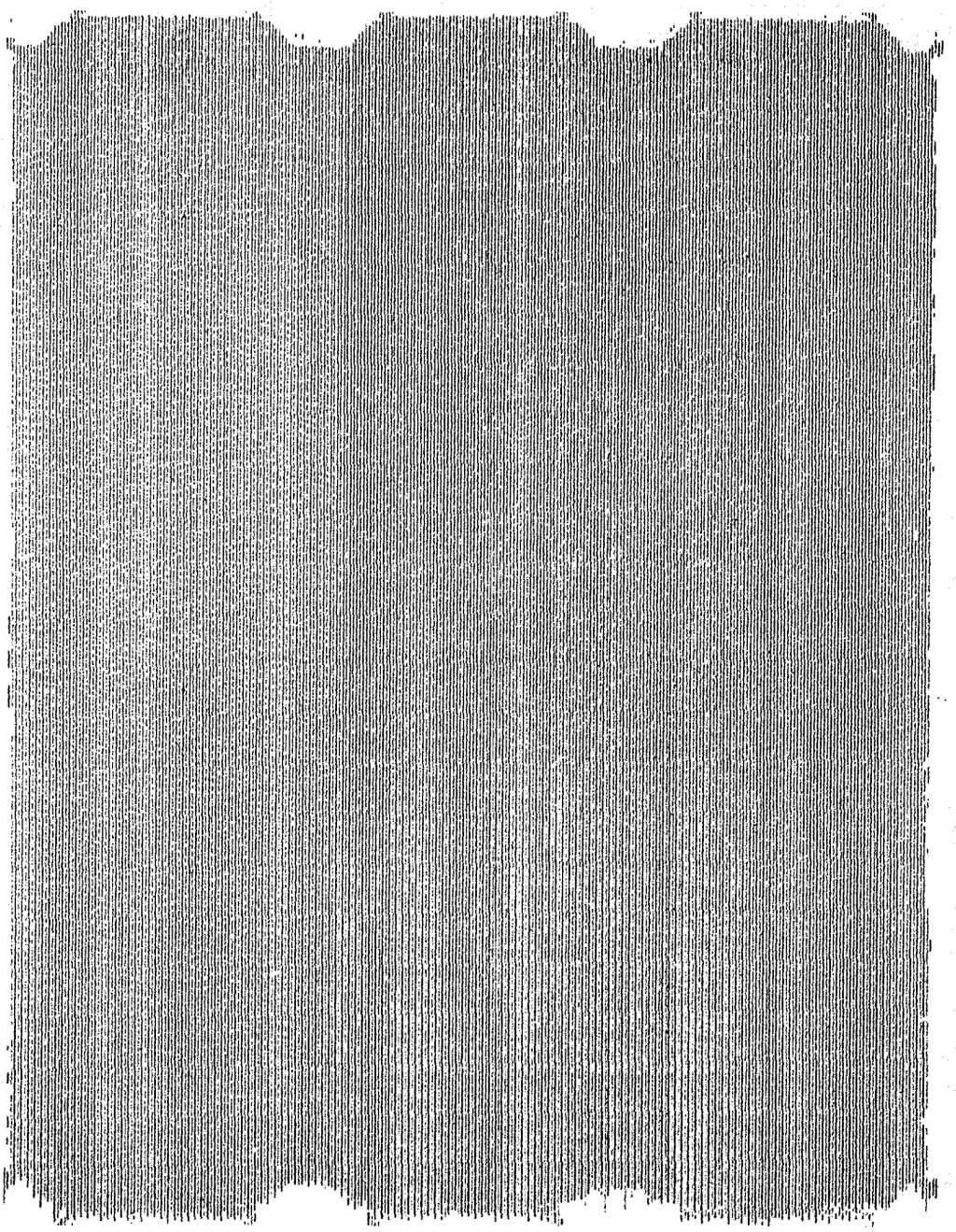
PANEL EX210	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
8 (22950)	STD	NA (-5%) BTDA (STD)	STD	STD

Figure 30. C-Scan Resin Variable No. 8



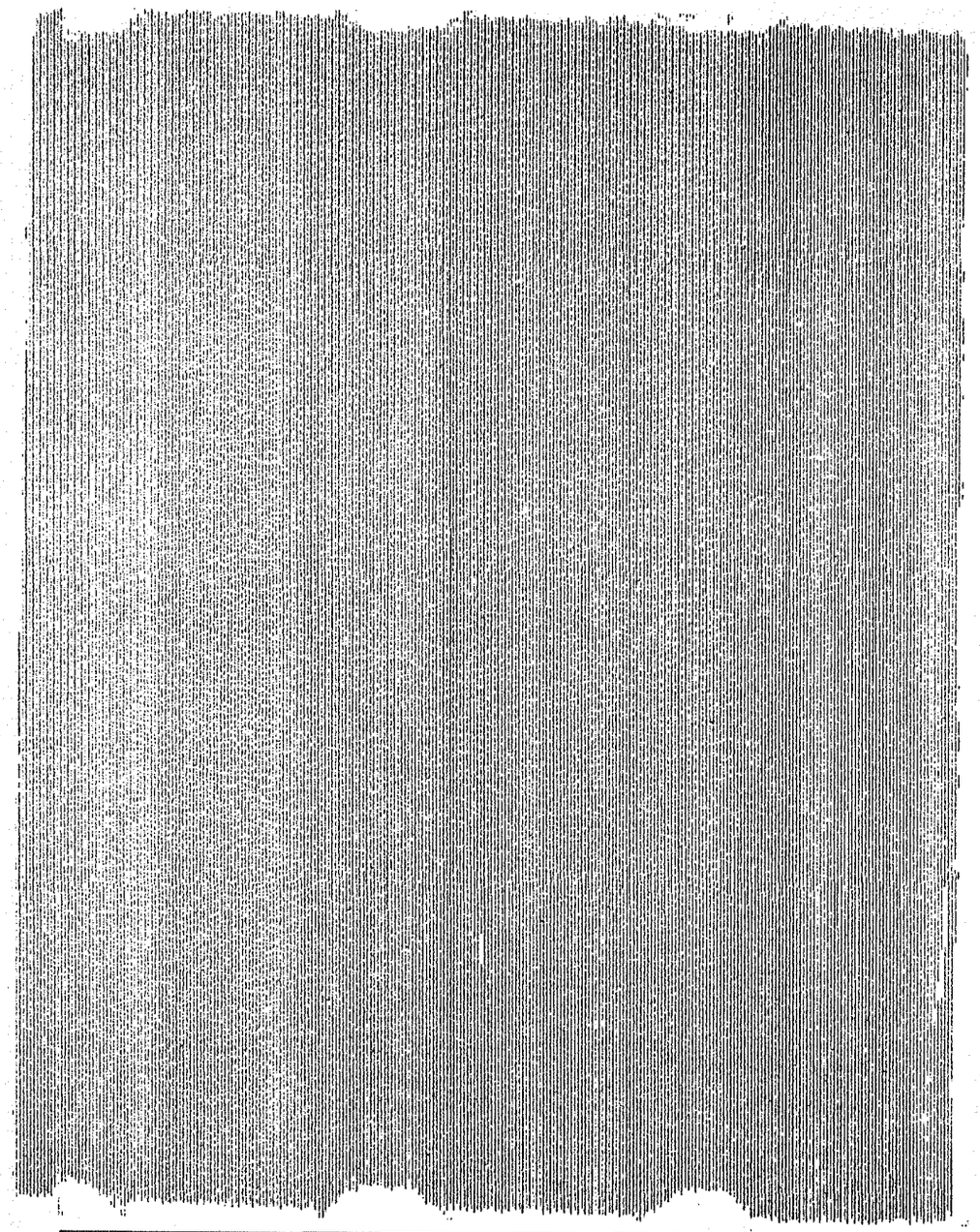
PANEL EX211	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
9 (22951)	STD	NA (STD) BTDA (+5%)	STD	STD

Figure 31. C-Scan Resin Variable No. 9



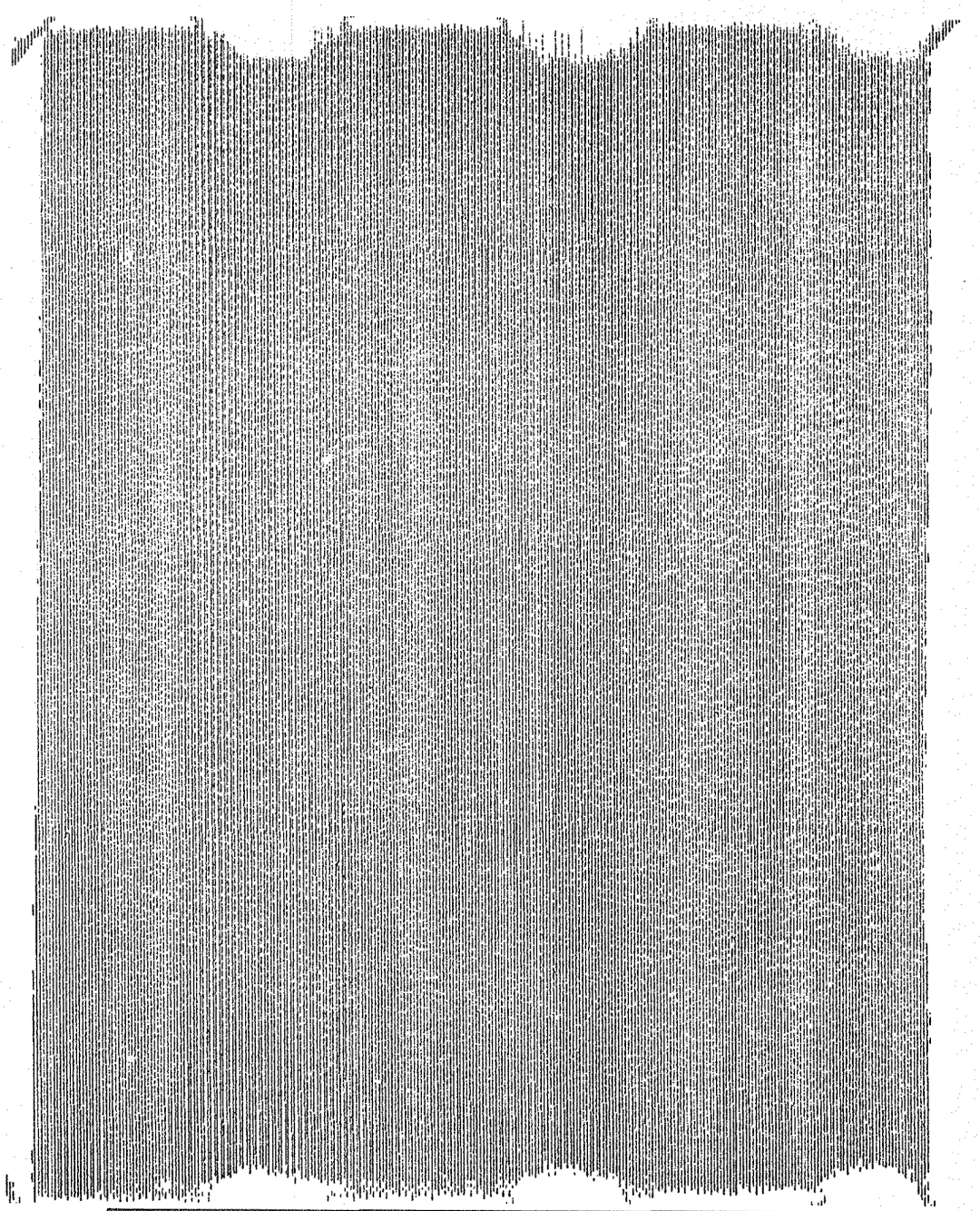
PANEL EX212	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
10 (22952)	STD	NA (STD) BTDA (-5%)	STD	STD

Figure 32. C-Scan Resin Variable No. 10



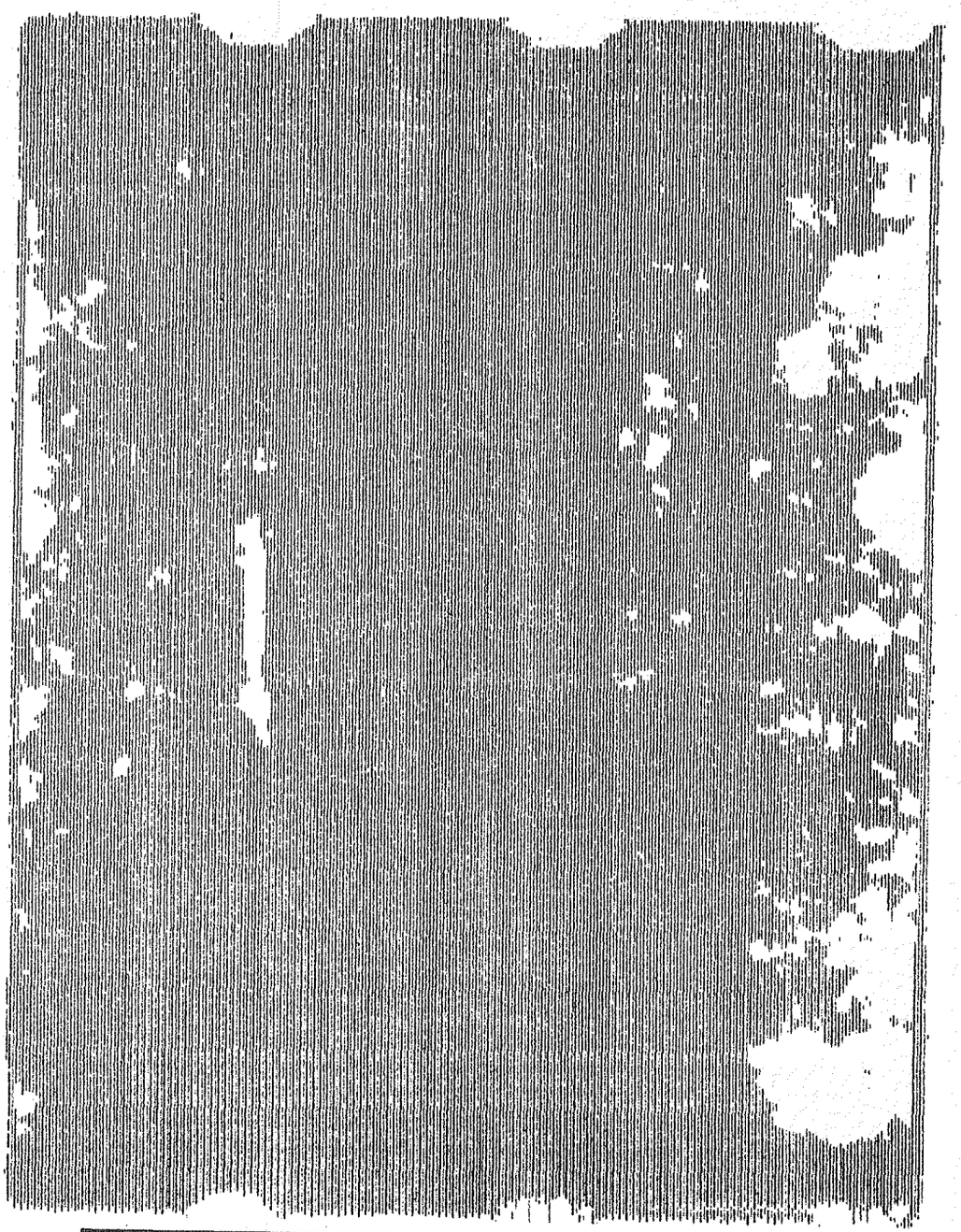
PANEL EX213	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
11 (22953)	STD	STD	2HRS @ 79C	STD

Figure 33. C-Scan Resin Variable No. 11



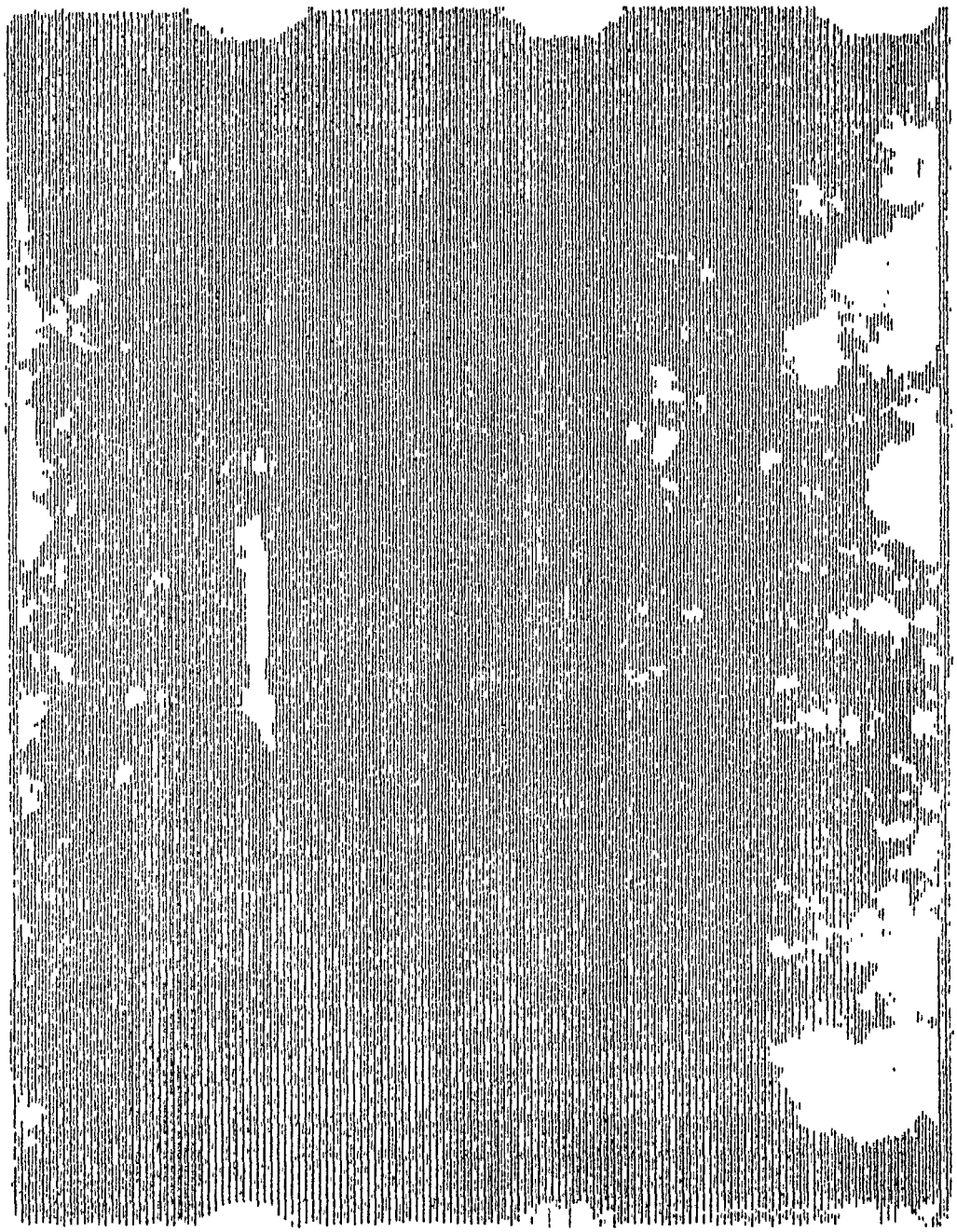
PANEL EX214	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
12 (22954)	STD	STD	2HRS @ 60C	STD

Figure 34. C-Scan Resin Variable No. 12



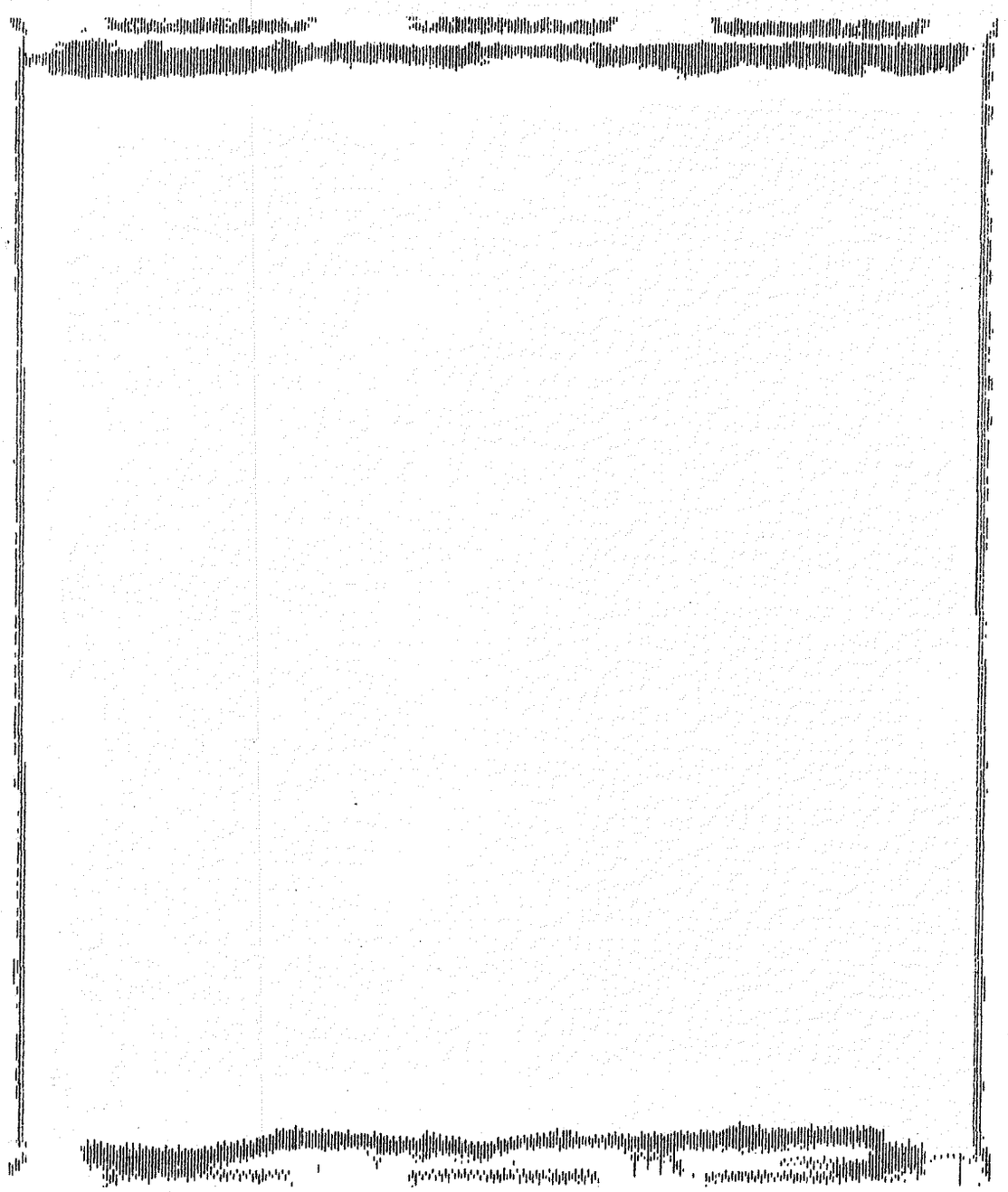
PANEL EX216	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
14 (23107)	STD ANCH-AMINE DL	STD	STD	STD

Figure 36. C-Scan Resin Variable No. 14



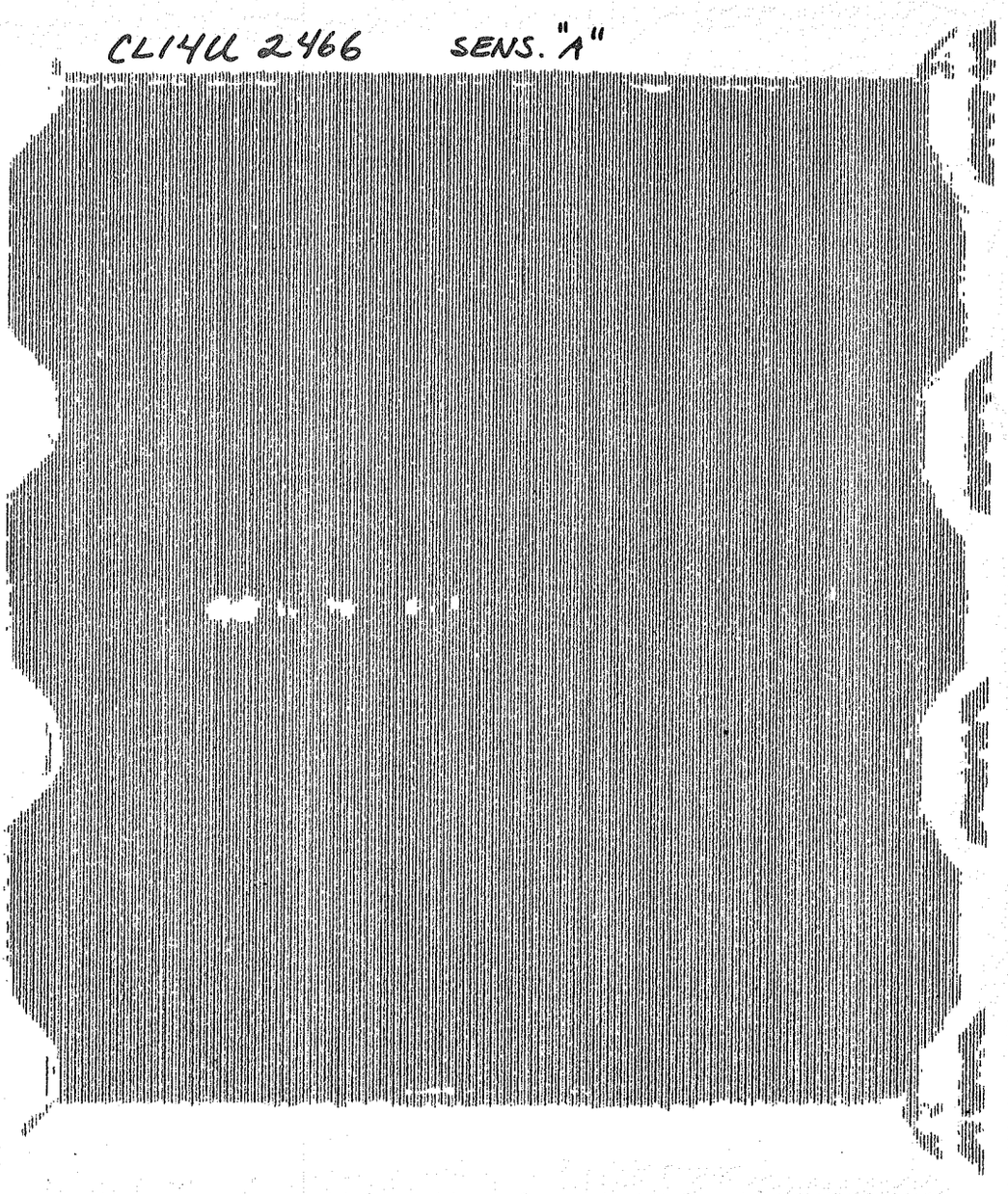
PANEL EX216	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
14 (23107)	STD ANCH-AMINE DL	STD	STD	STD

Figure 36. C-Scan Resin Variable No. 14



PANEL EX219	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME
15 (23236)	STD TONOX 22	STD	STD	STD

Figure 37. C-Scan Resin Variable No. 15



PANEL EX219	FORMULATION VARIABLES		PROCESS VARIABLES	
PREPREG RUN & (BATCH)	CONC AP-22	CONC ANHYDRIDES	COOK TIME	REFLUX TIME
16 (24266)	STD	STD	STD	STD

Figure 38. C-Scan Resin Variable No. 16

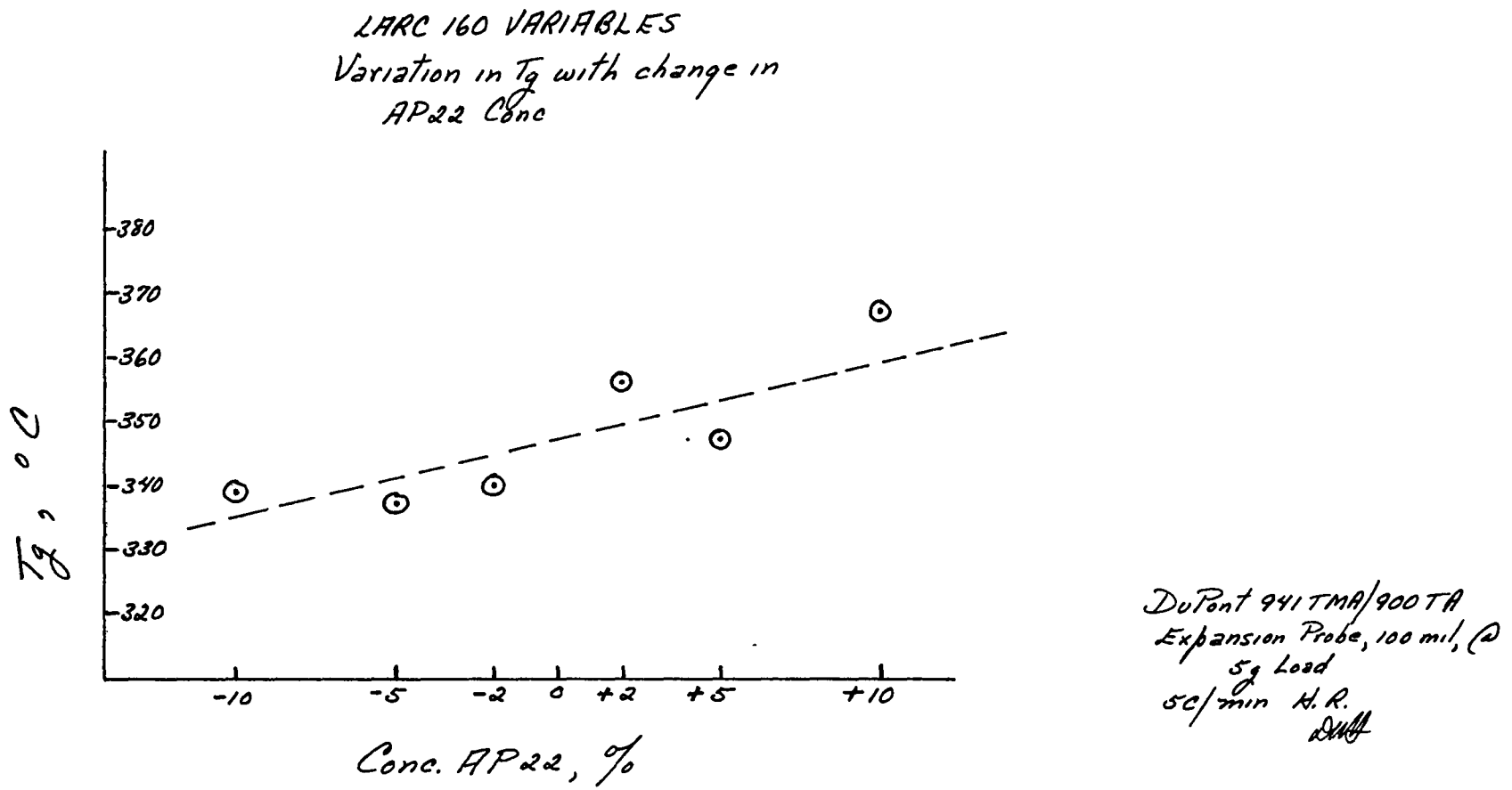


Figure 39. Variation in Tg With Change in AP22 Concentration

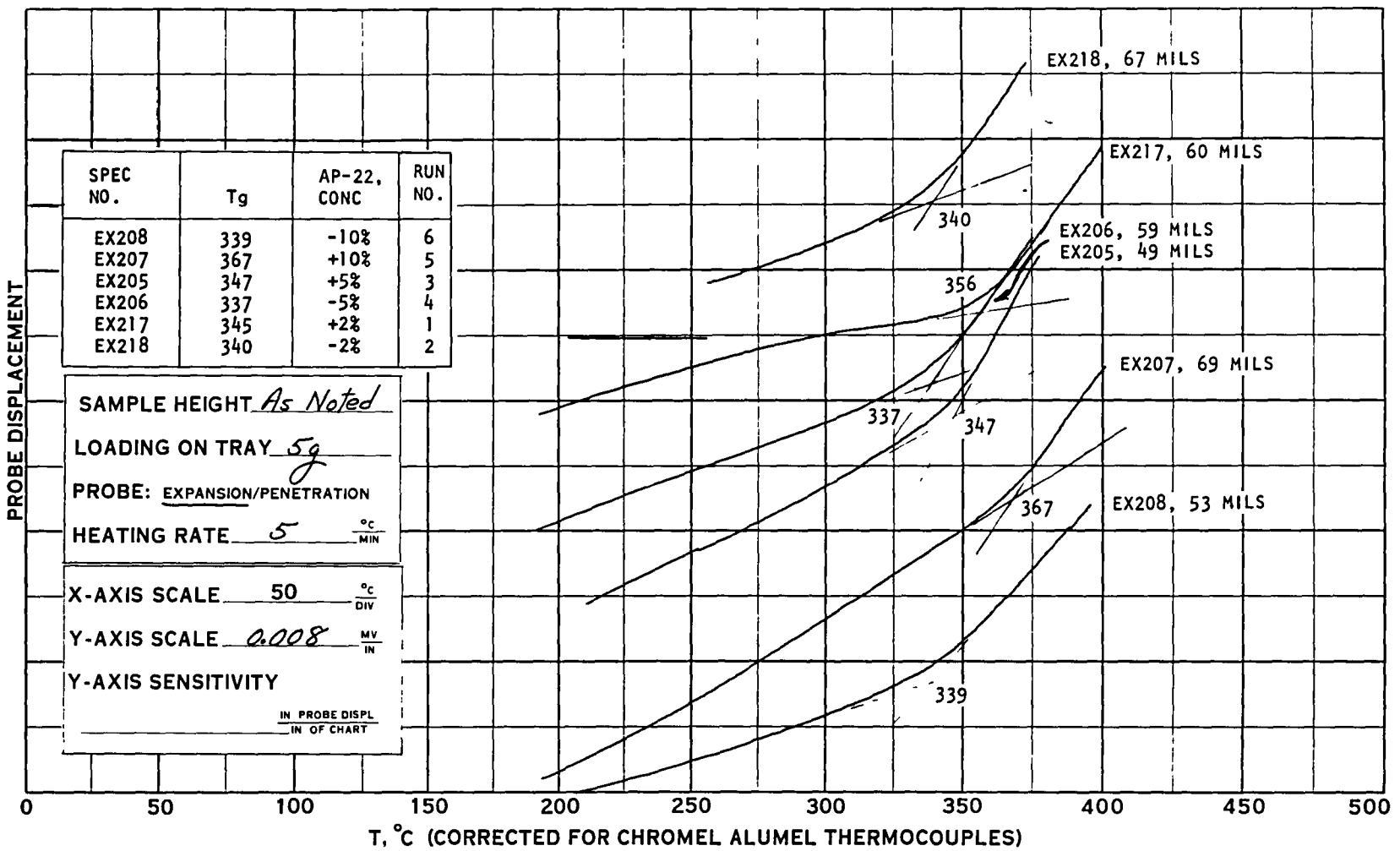


Figure 40. TMA-T_g Values, Amine Variables

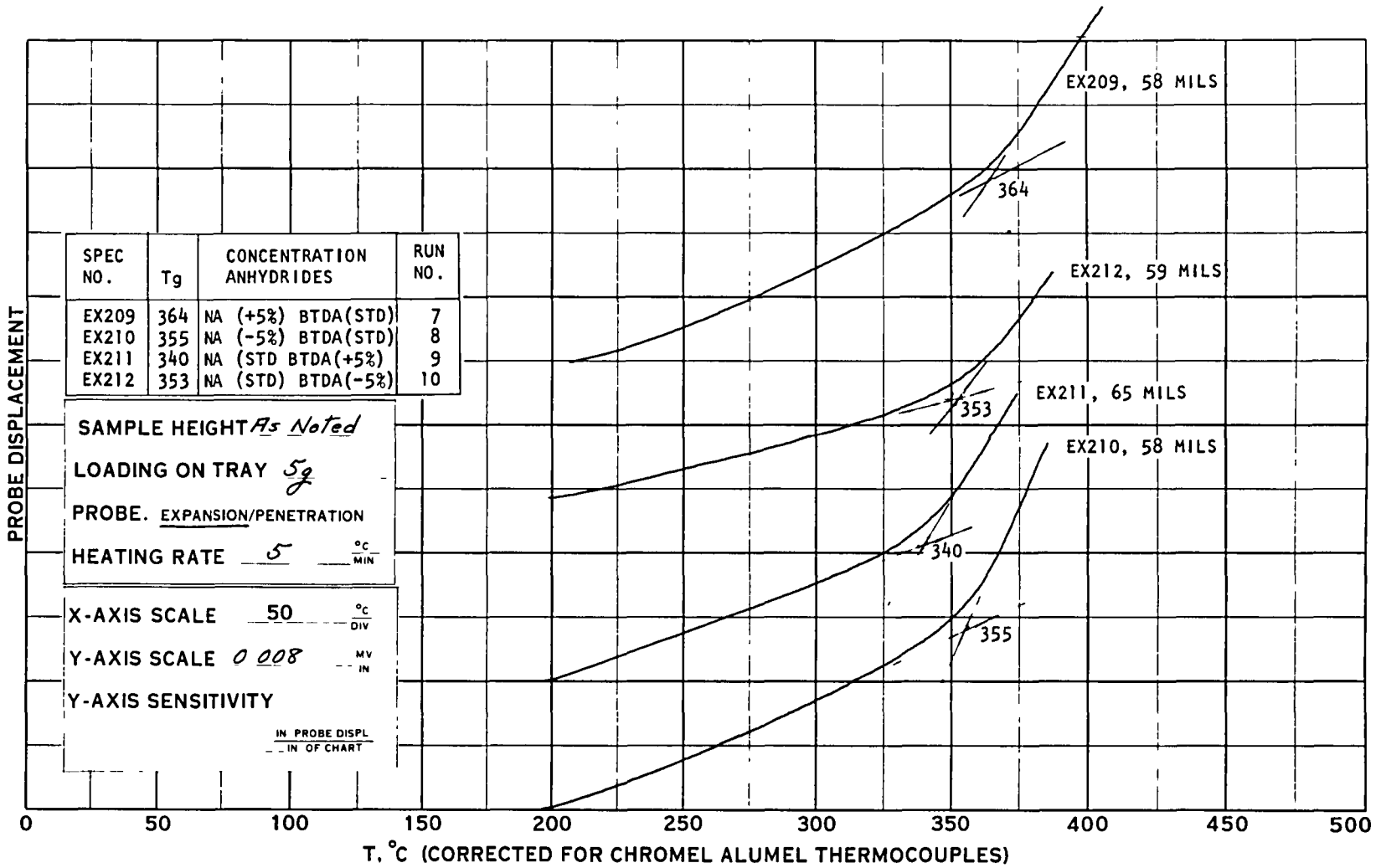


Figure 41. TMA-T_g Values NA-BTDA Variables

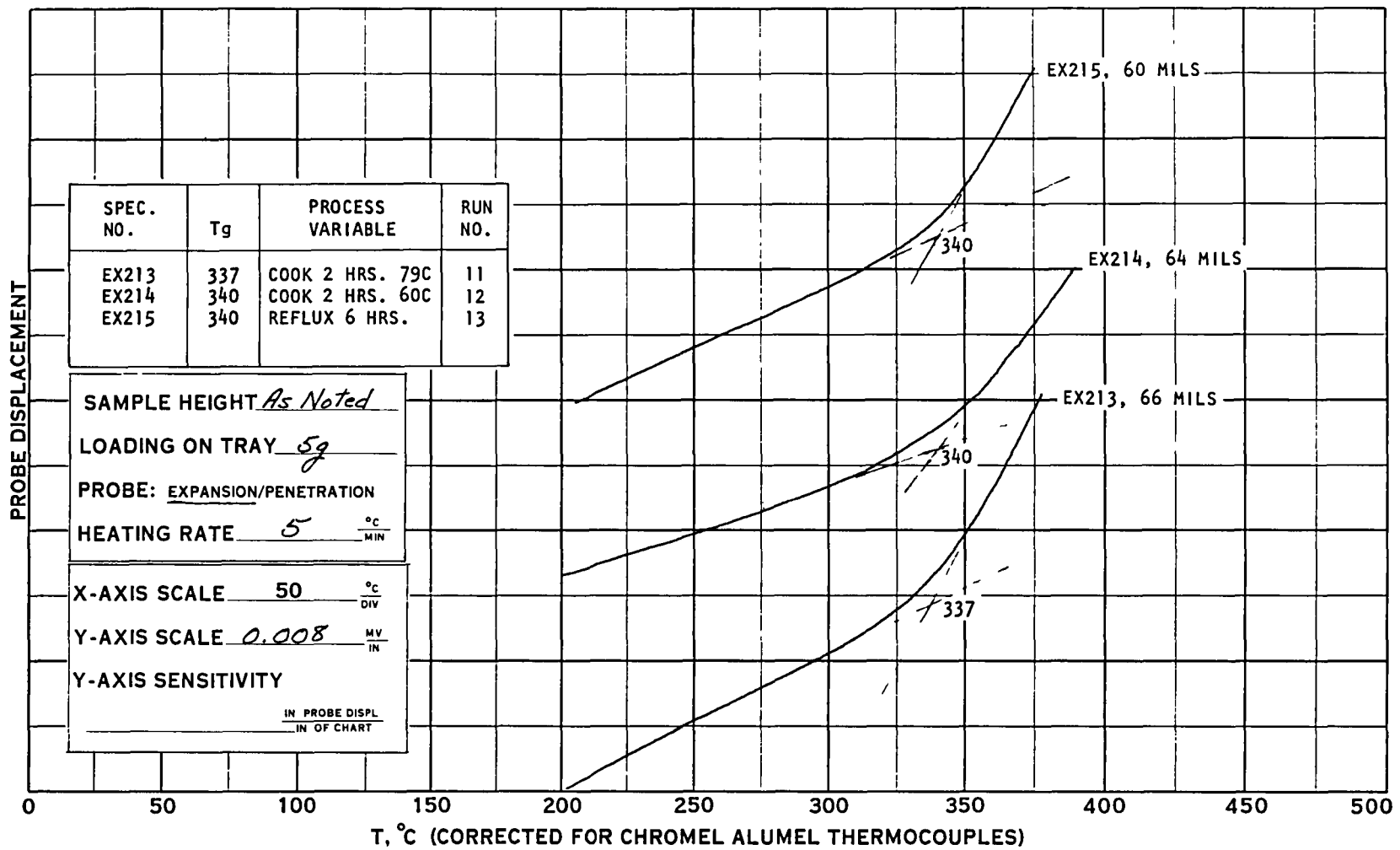


Figure 42. TMA-T_g Values Resin Processing Variables

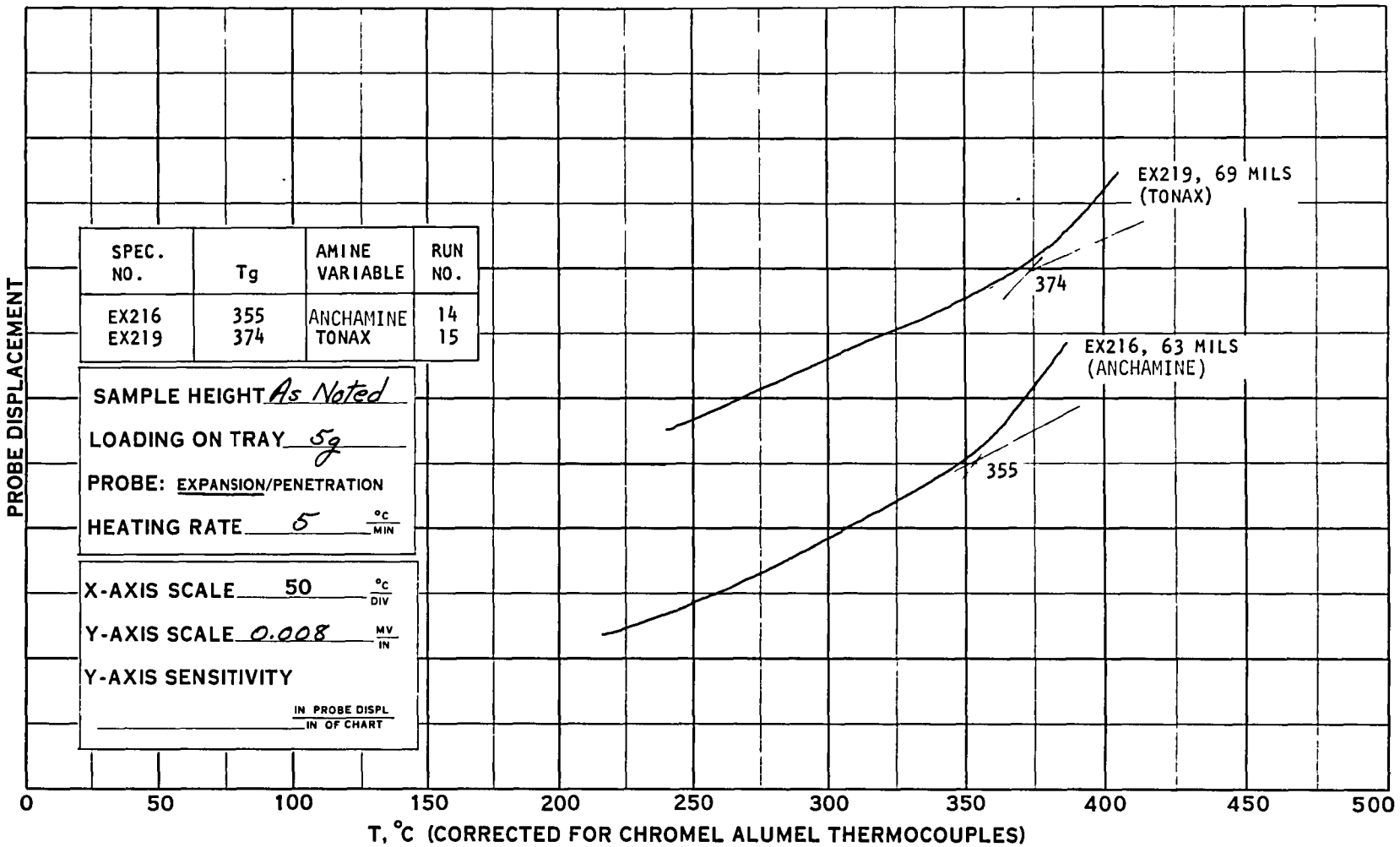


Figure 43. TMA-T_g Values Anchamine & Tonax Amines

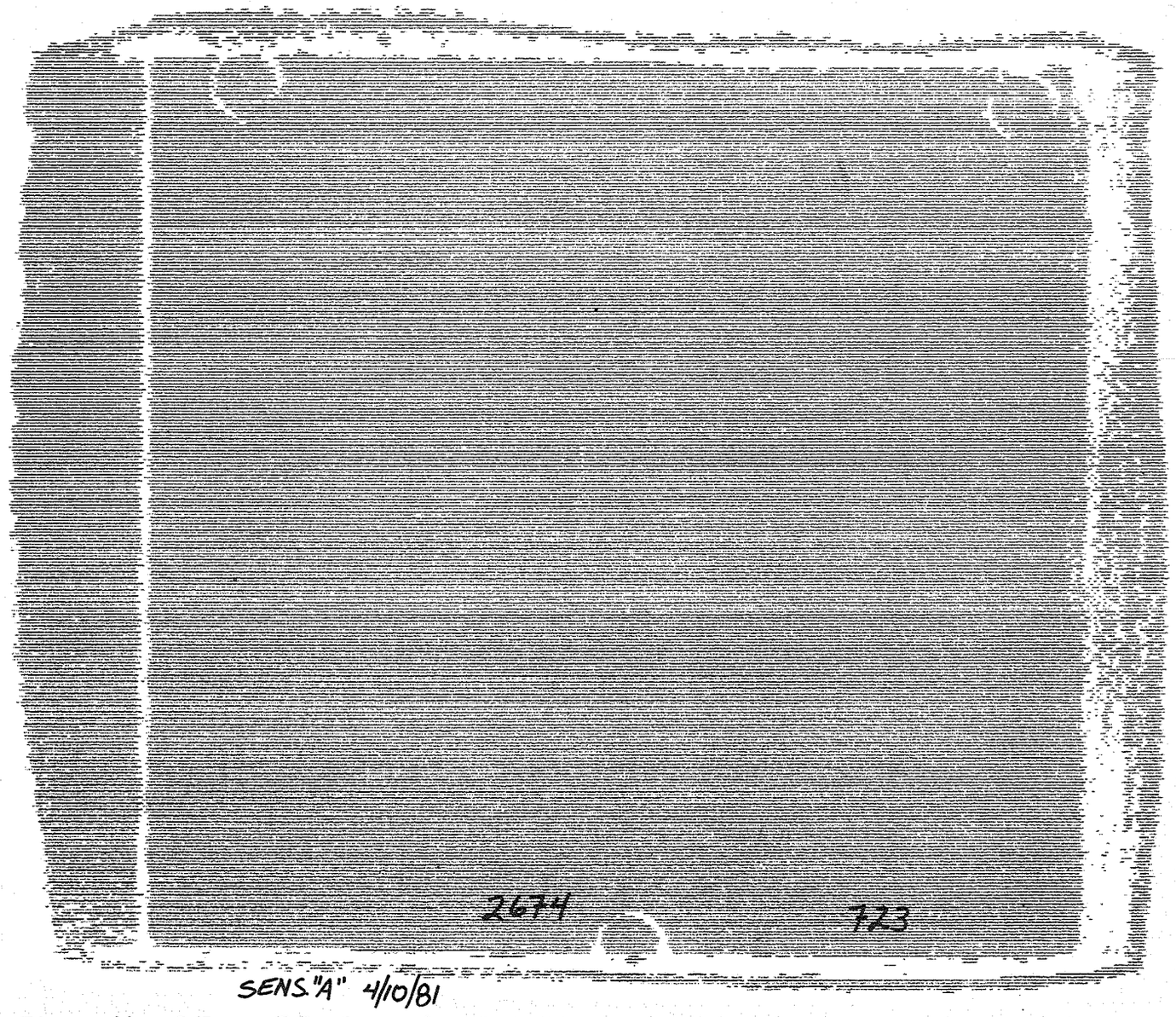


Figure 44. C-Scan of Laminate (Batch 23723) Imidizing Pressure 16.9 KN/m^2 (5in. H_g)

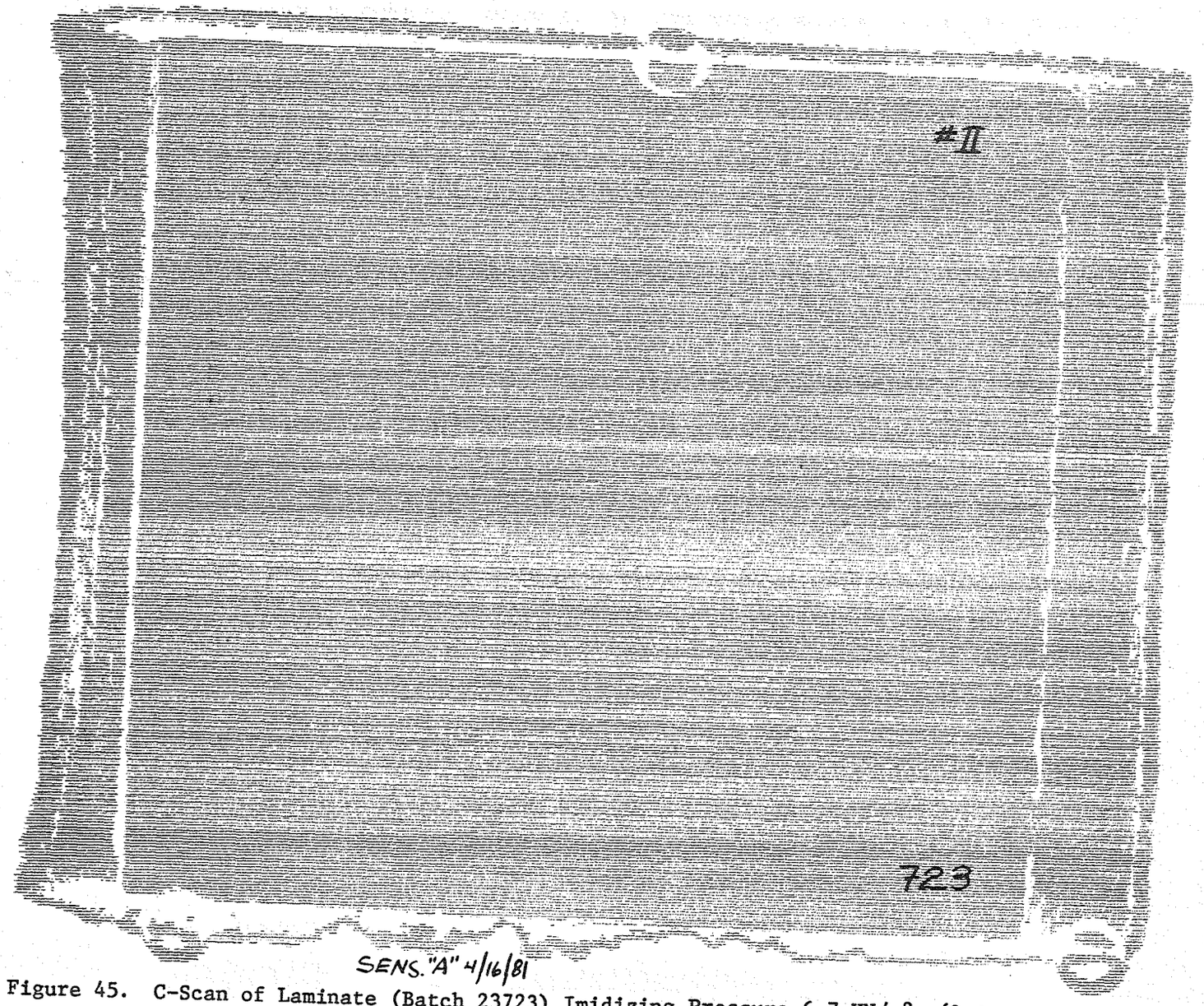


Figure 45. C-Scan of Laminate (Batch 23723) Imidizing Pressure 6.7 KN/m2 (2 in. H_g)

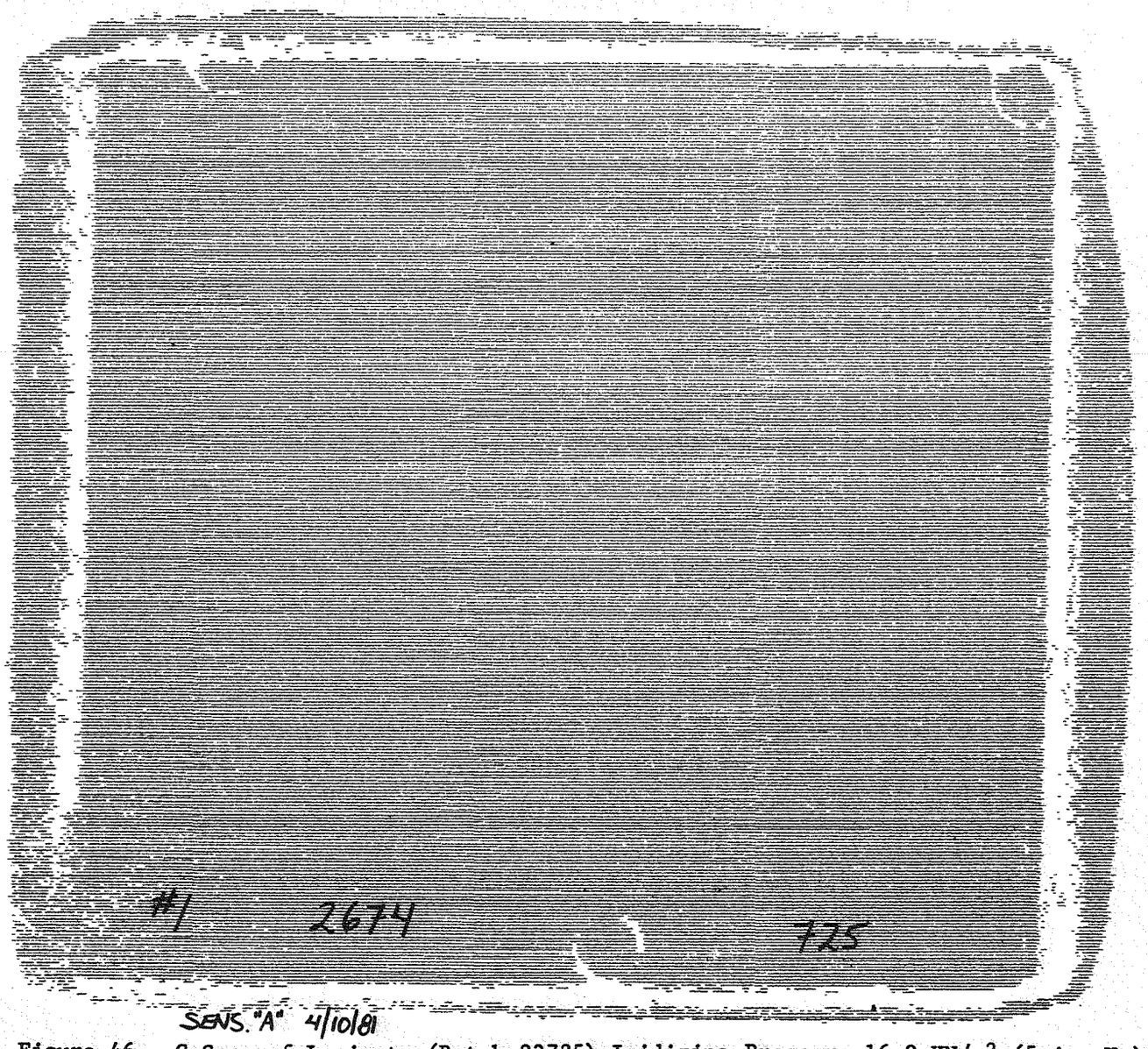


Figure 46. C-Scan of Laminate (Batch 23725) Imidizing Pressure 16.9 KN/m^2 (5 in. H_g)

3.3 TASK (c) - FABRICATION AND TEST

3.3.1 Fabrication - Mechanical Properties Specimens

Initial laminate panels to be used for mechanical properties testing were laid up and autoclave cured using the single stage in situ imidizing and cure process described in 3.2.1.1. These panels were used to obtain all postcured condition mechanical properties specified in the test matrix, Table 10. Resin flow control proved to be a problem using this cure cycle, sometimes resulting in high composite fiber volumes in the range of 64 to 68%. All laminates for specimen fabrication had essentially zero void content as determined analytically, and by NDI C-scan test.

Process optimization studies performed in Task (b) led to a two stage processing requiring an imidizing cycle where volatiles are removed to $< 2\%$ from the stacked prepreg prior to the autoclave cure. Resin flow control was maintained during imidization by low vacuum levels and a Celgard 4500 or 4510 microporous polypropylene film which allows volatile matter to escape through a perforated tooling plate while the membrane contains the low viscosity resin. Excess resin was absorbed into bleeder materials calculated to yield a laminate with a target $60 \pm 2\%$ fiber volume.

The autoclave cure was accomplished between two flat tooling plates. Since the major portion of volatile matter was removed in the imidizing cycle, the laminates were treated similarly to epoxy materials. Final laminate cure was accomplished at 287°C (500°F) for three hours or 326°C (625°F) for two hours.

This two stage process was employed in the fabrication of all laminates for mechanical properties specimens that were aged for 125 hours at 316°C (600°F). All panels had essentially zero void with fiber volumes in the 61 to 63% range. NDI C-scan recordings confirmed high quality and are shown in Figure 97 through 102. The detailed description of this two stage processing is presented in 3.2.1.2. Flexural and tensile specimens were molded from chopped fiber using the compression molds shown in Figure 96 and 103. The

molding process used is described in 3.2.5.

3.3.2 Testing - Mechanical Properties

Testing was performed in accordance with the matrix, Table 10. Three specimens for each test mode and temperature were tested in the postcured and aged, 125 hours 316°C (600°F), conditions.

3.3.2.1 Beam Test Description

Tension and compression critical beams were employed to determine $(0)_t$ tension (F_{tu}, E_t, ε_{ult}, $\mu\%$) and $(0^\circ)_t$ and $(0^\circ, \pm 45^\circ, 90^\circ)_s$ compression (F_{cn}, E_c, $\varepsilon_{ult}\mu\%$) properties. The beam designs are presented in Figure 104.

Analytical studies (Ref. 5) performed by Mr. Mark Shuart, NASA-LaRC, proved that the 352 Kg/m^3 (22 pcf) honeycomb core significantly affects the measured strength and elastic modulus properties of composite specimens. A computer program was developed by NASA-LaRC to assess the actual effect this core has on the laminate properties and to establish property adjustment factors. Bulk core properties for both aluminum, 352 Kg/m^3 (22 pcf), and 301 CRES, 639 Kg/m^3 (40 pcf), core materials were developed by Rockwell International and transmitted to NASA-LaRC for use in the computer program in developing the composite property adjustment factors. These data are shown in Table 11. Specific adjustment factors are given in the individual mechanical property data Tables 12 through 17.

During test, individual specimens were stabilized at each test temperature for 10^{+10}_{-0} minutes prior to application of stress at a head travel of 1.27-mm (0.05 in.)/minute. Data were obtained by autographic recording of axial strain gages installed on the composite specimens at the beam midpoint.

3.3.2.2 Tensile Test Description

Tensile coupons were employed to determine $(0^\circ)t$, $(90^\circ)t$, $(+45^\circ)s$ and $(0^\circ, \pm 45^\circ)s$ and $(0^\circ, \pm 45^\circ, 90^\circ)s$ tension properties. Specific properties determined were F_{tu}, E_t, ε_{ult}, $\mu(\%)$ and ν.

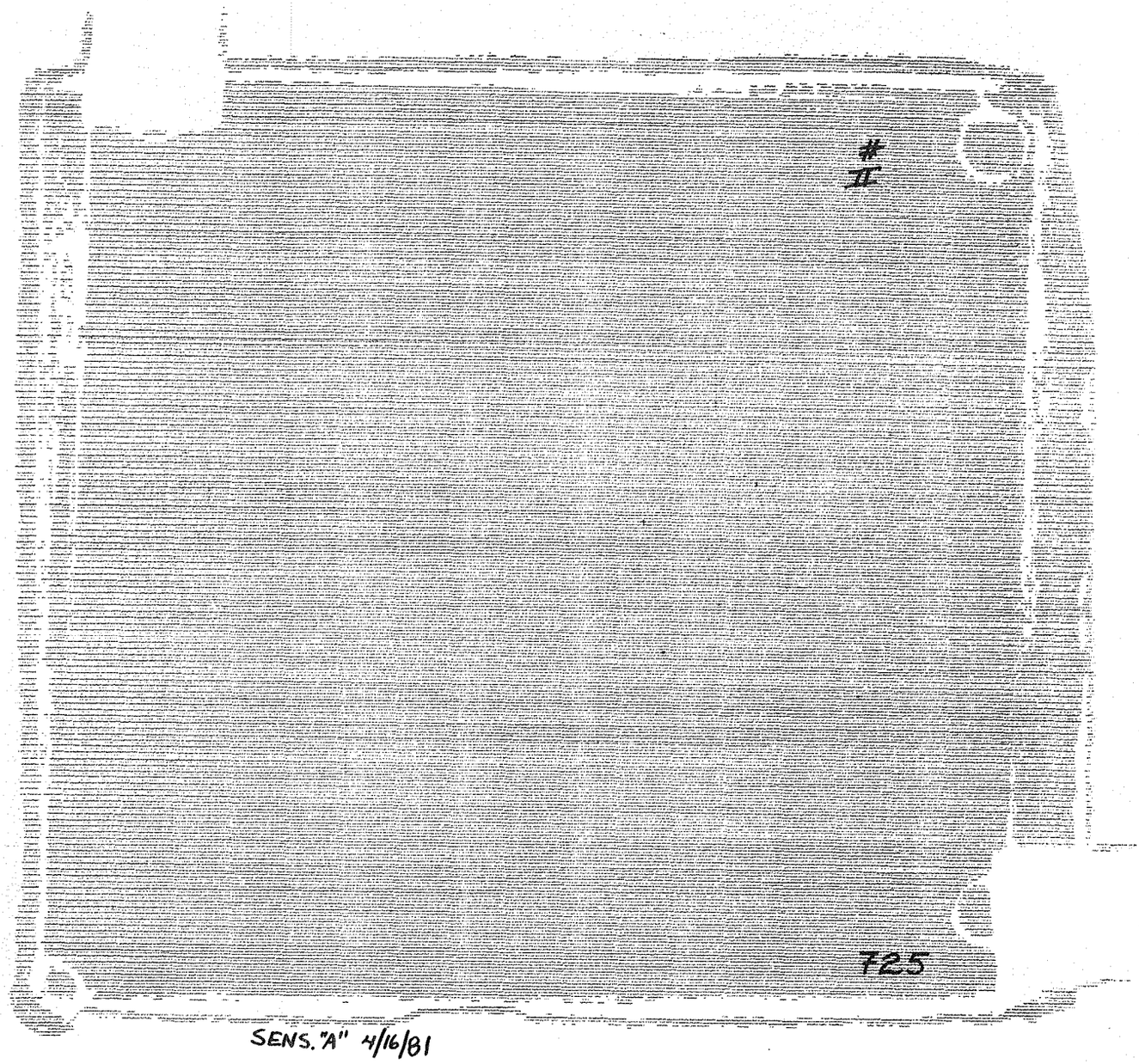


Figure 47. C-Scan of Laminate)Batch 23725) Imidizing Pressure 6.7 KN/m^2 (2 in. Hg)

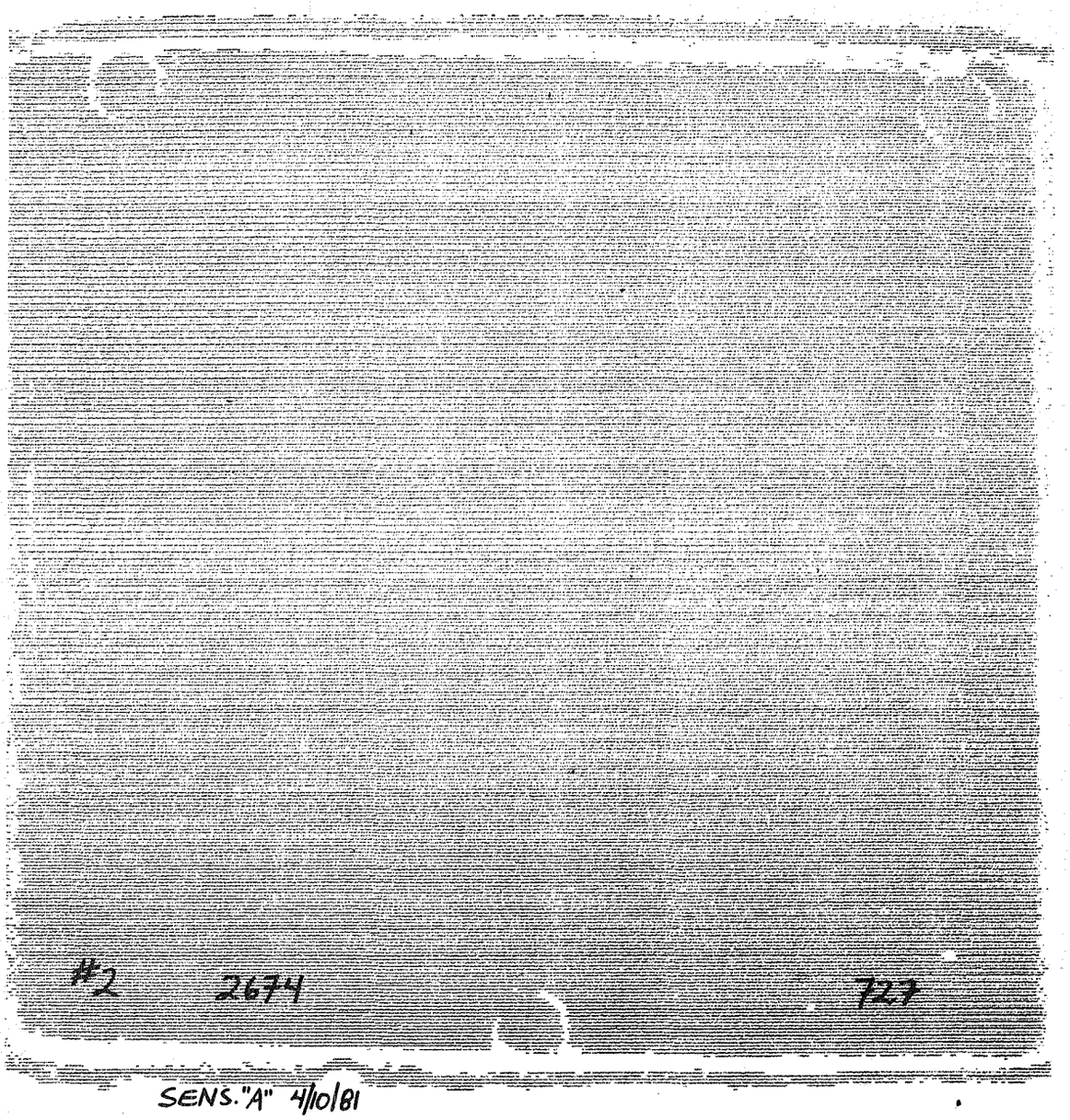


Figure 48. C-Scan of Laminate (Batch 23727) Imidizing Pressure 16.9 KN/m^2 (5 in. Hg)

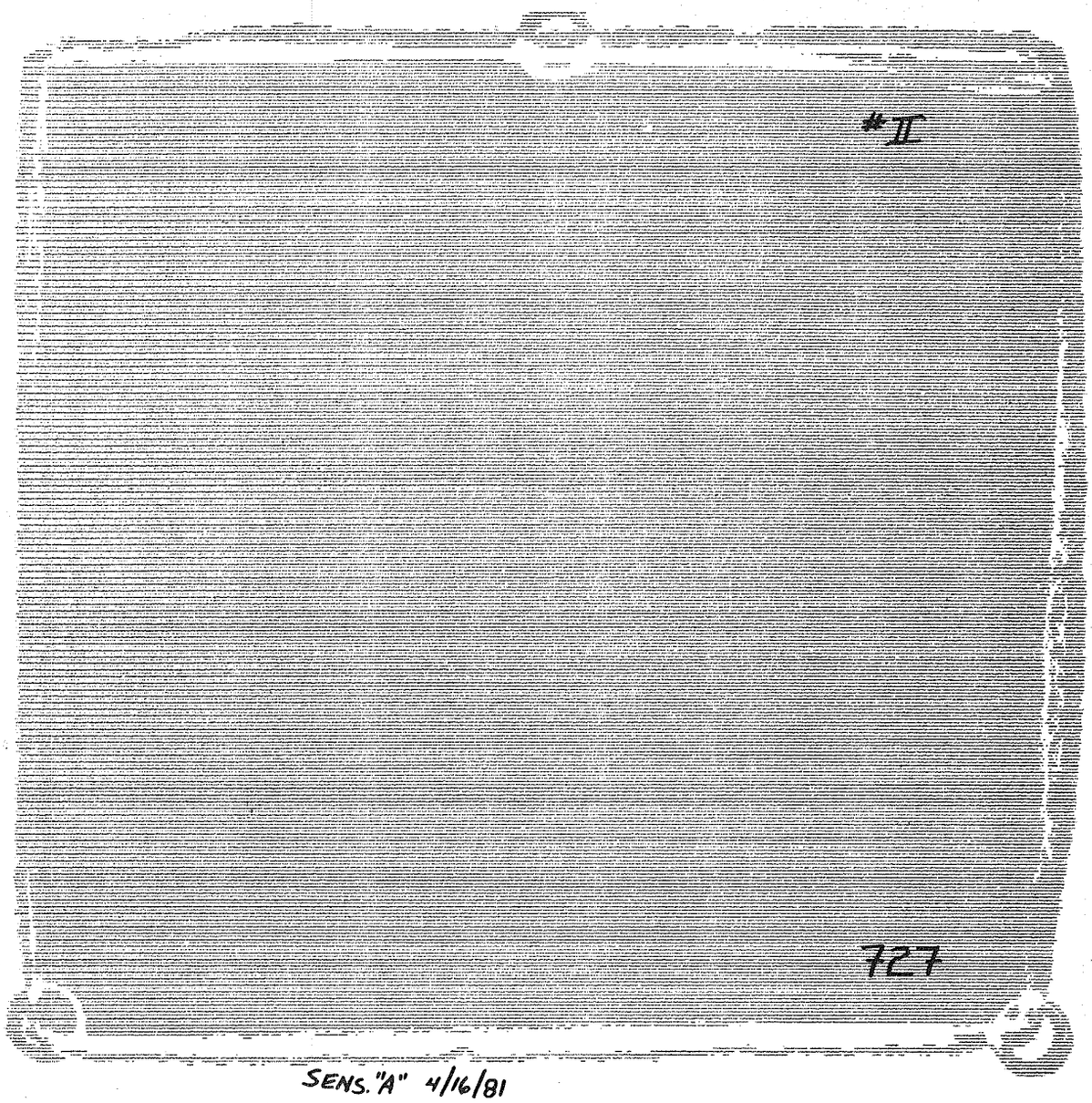


Figure 49. C-Scan of Laminate (Batch 23727) Imidizing Pressure 6.7 KN/m^2 (2 in. Hg)

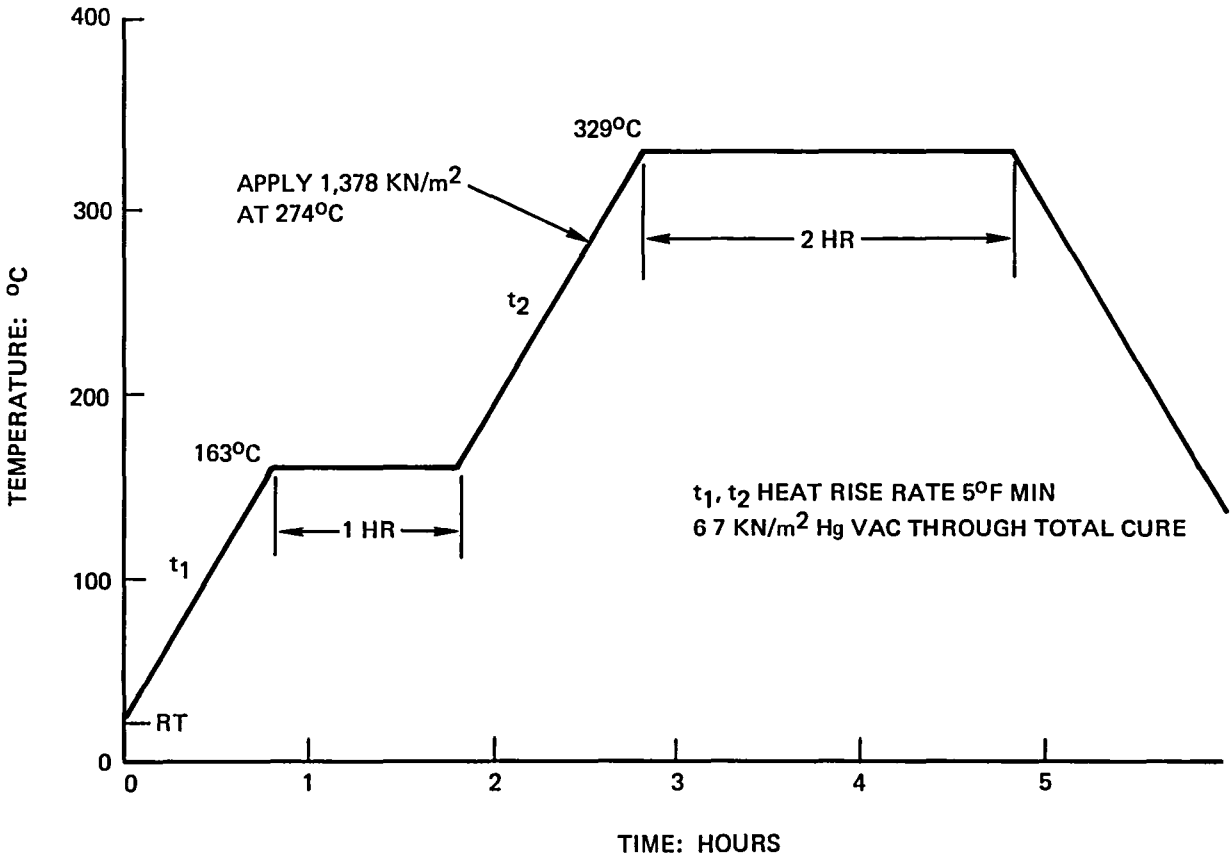


Figure 50. LARC 160 Preliminary Cure Cycle

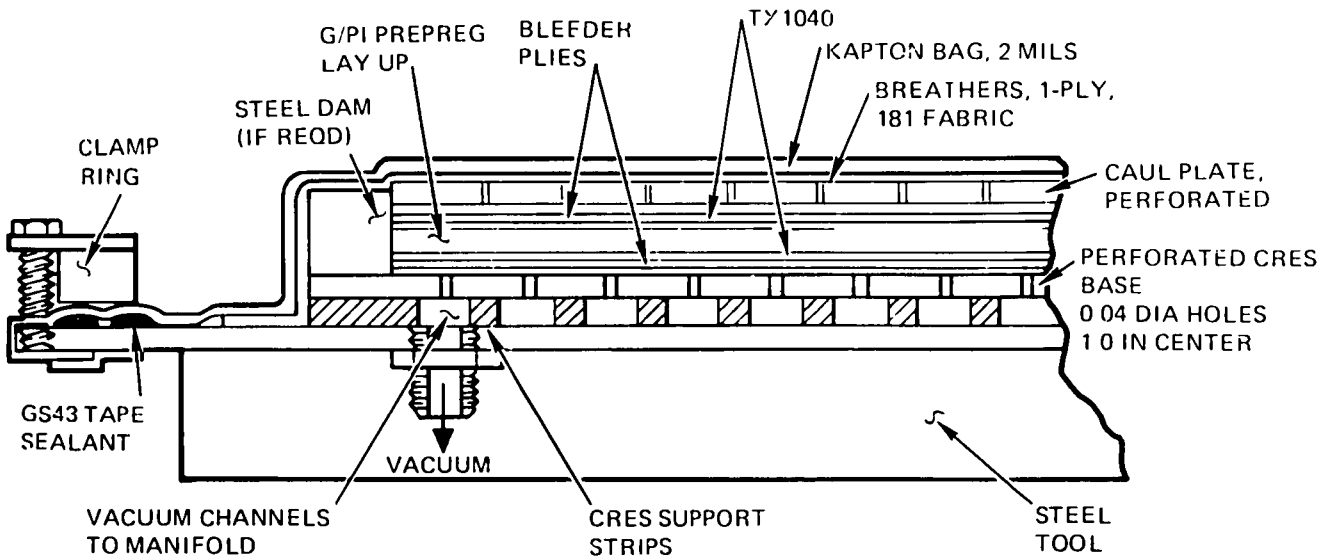
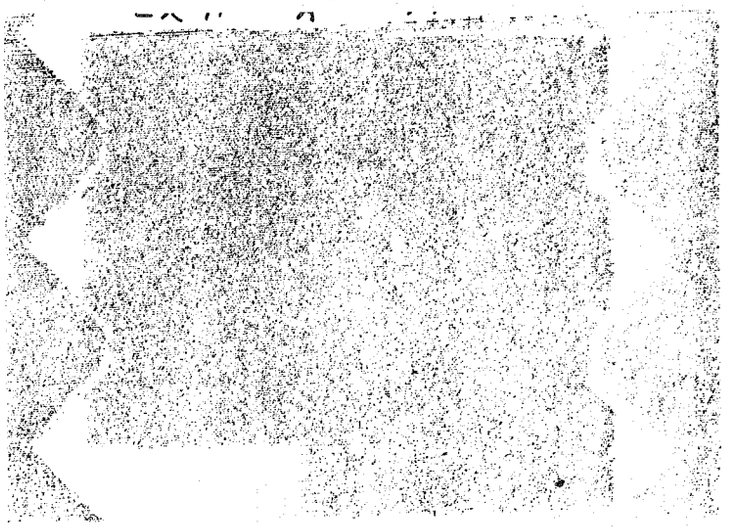


Figure 51. Flat Laminate Autoclave Tooling Concept

EX41



CRACK

COMPOSITE DESCRIPTION: EX 41

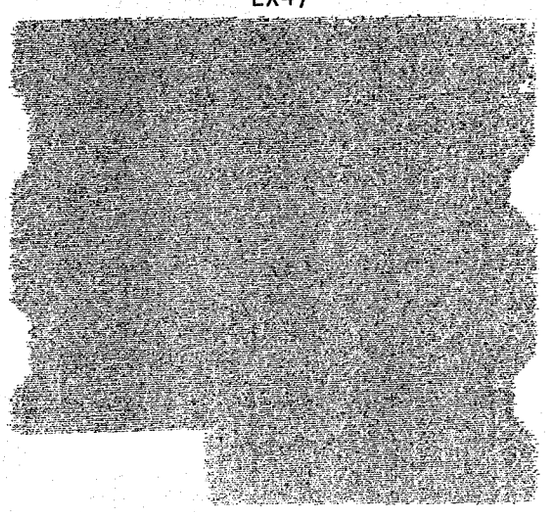
NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.03-1.79 (80-70.4)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: 163 C (325 F)
60 MINUTES (CONTROL). STANDARD
CURE AND POSTCURE CYCLE

EX47



COMPOSITE DESCRIPTION: EX 47

NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.03-1.79 (80-70.4)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: CURE PRESSURE EVALUATION
STUDY—1.0 N/M^2 (150 PSI).
STANDARD CURE TEMPERATURE AND
POSTCURE

Figure 52. C Scan Recordings of Panels EX41 and EX47 Minimum Cure Pressure Study

EX48

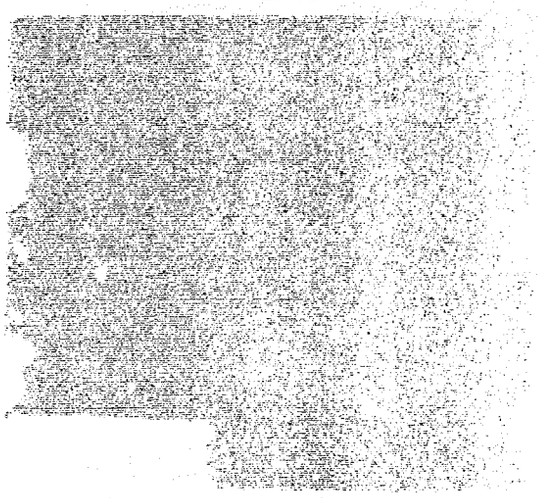
COMPOSITE DESCRIPTION: EX 48

NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.1-1.94 (83.2-76.8)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: CURE PRESSURE EVALUATION STUDY—0.689 N/M^2 (100 PSI). STANDARD CURE TEMPERATURE AND POSTCURE



EX49

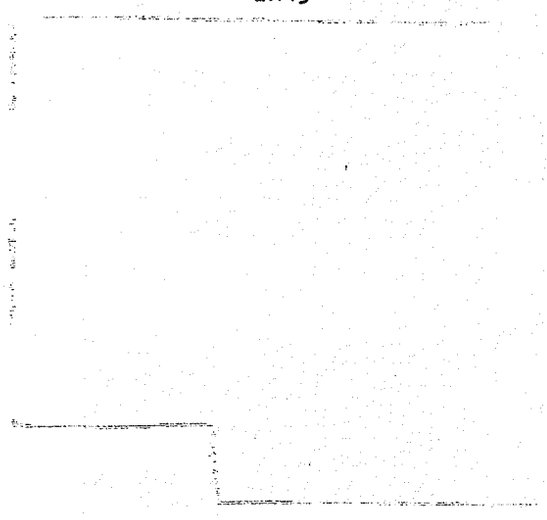
COMPOSITE DESCRIPTION: EX 49

NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.03-1.87 (76.4-73.6)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

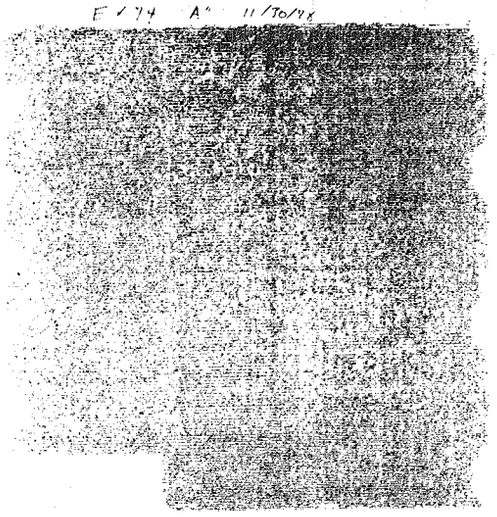
PROCESS VARIABLE: CURE PRESSURE EVALUATION STUDY—0.345 N/M^2 (50 PSI). STANDARD CURE TEMPERATURE AND POSTCURE



EXTENSIVE MICRO AND MACRO POROSITY

Figure 53. C Scan Recordings of Panels EX48 and EX49 Minimum Cure Pressure Study

EX74



COMPOSITE DESCRIPTION: EX 74

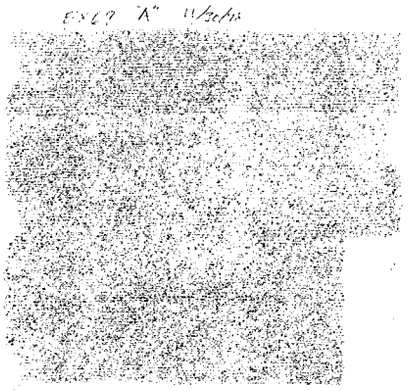
NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.1-1.9 (83.2-73.6)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: CURE TEMPERATURE EVALUATION STUDY—329 C (625 F). 1.3 N/M^2 (200 PSI). STANDARD POSTCURE

EX69



COMPOSITE DESCRIPTION: EX 69

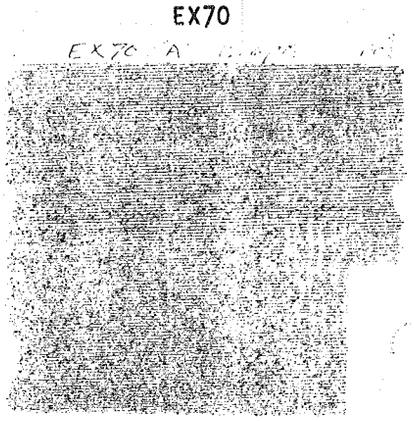
NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.1-1.87 (76.4-73.6)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: CURE TEMPERATURE EVALUATION STUDY—316 C (600 F) 13 N/M^2 (200 PSI). STANDARD POSTCURE

EX70



COMPOSITE DESCRIPTION: EX 70

NO. OF PLIES/ORIENTATION: 32/0°

THICKNESS MM (MILS): 2.3-2.2 (89.6-86.4)

PANEL SIZE CM (INCH): 10.8x12.7 (4.25x5.0)

PROCESS VARIABLE: CURE TEMPERATURE EVALUATION STUDY—302 C (575 F), 1.3 N/M^2 (200 PSI). STANDARD POSTCURE

Figure 54. C Scan Recordings of Panels EX74, EX69, and EX70 Minimum Cure Temperature Study

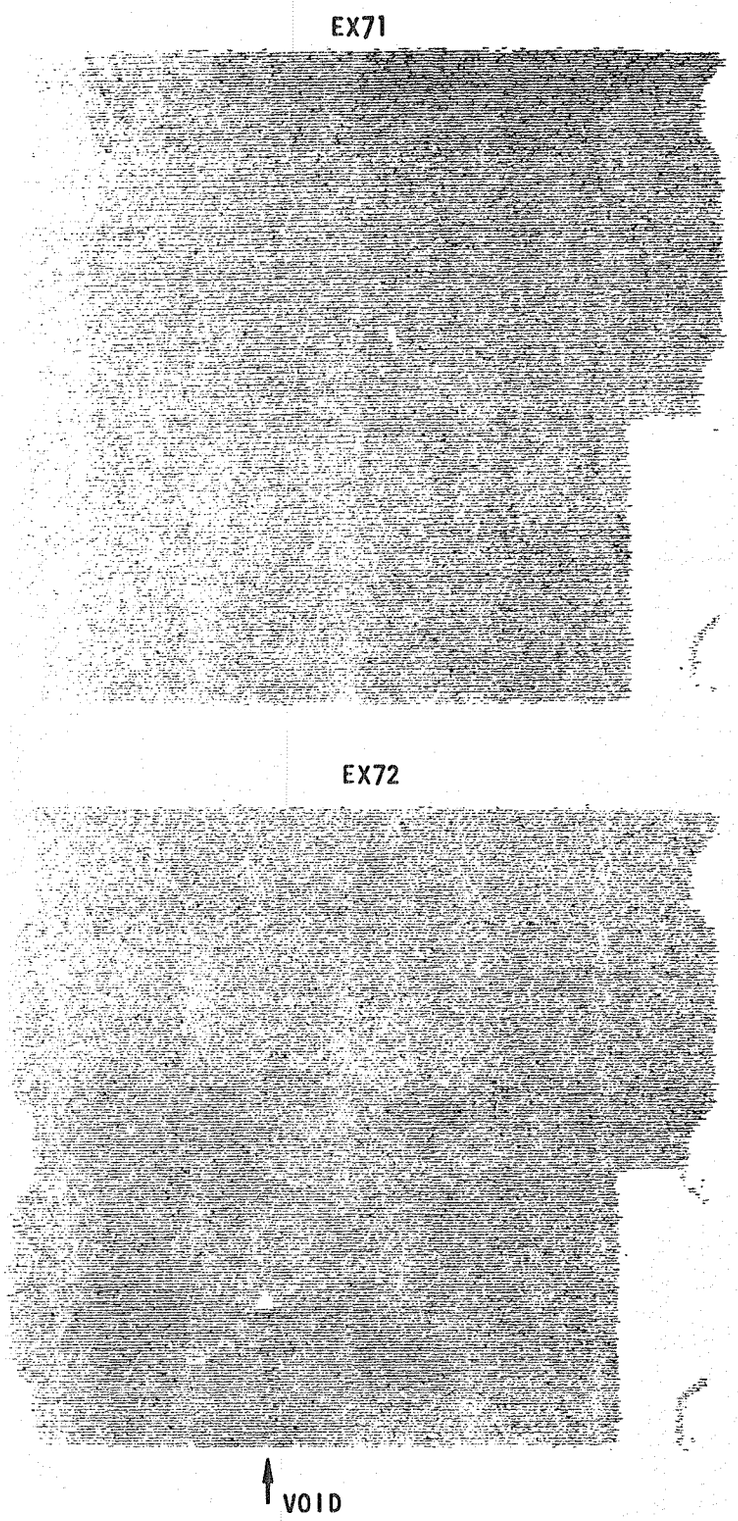


Figure 55. C Scan Recordings of Panels EX71 and EX72 Minimum Cure Temperature Study

SAMPLE:	SAMPLE HEIGHT_____	X-AXIS SCALE 50 °C/DIV	RUN NO _____
	LOADING ON TRAY_____	Y-AXIS SCALE_____ MV/IN	DATE 12-7-78
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR G O
ORIGIN:	HEATING RATE_____ °C/MIN	_____ IN PROBE DISPL / IN OF CHART	

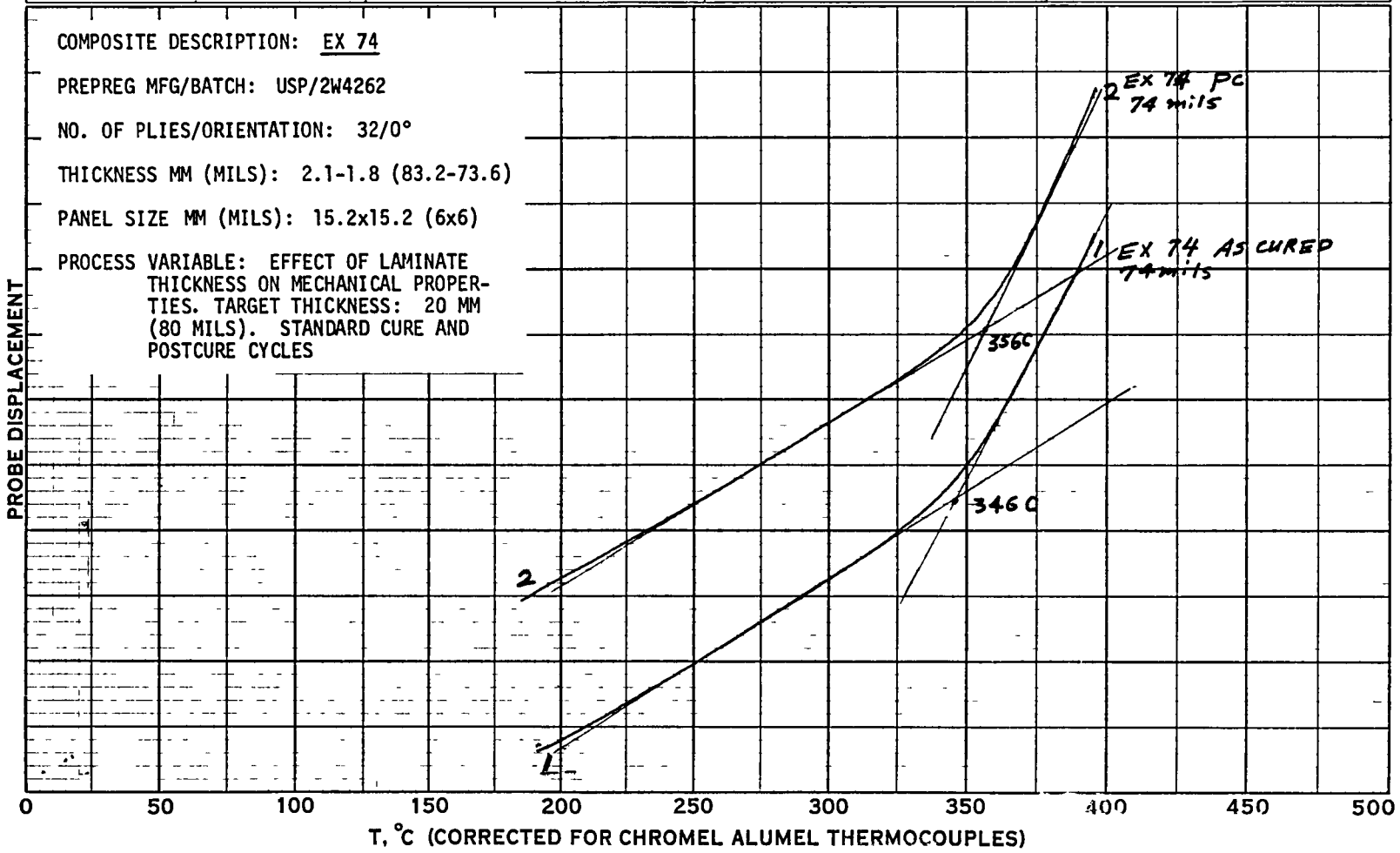


Figure 56. TMA-Tg Characteristics of Specimen EX 74

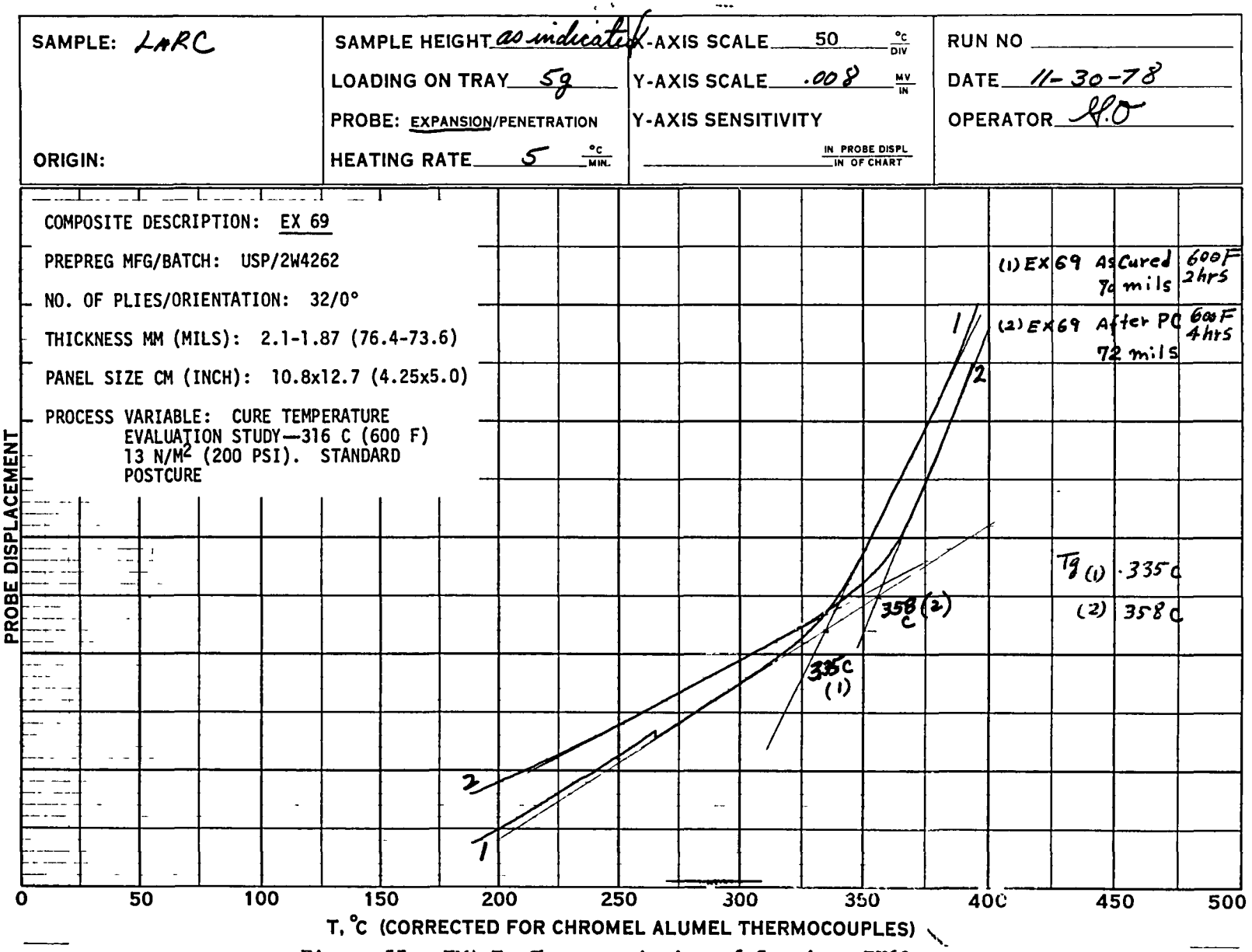


Figure 57. TMA-Tg Characteristics of Specimen EX69

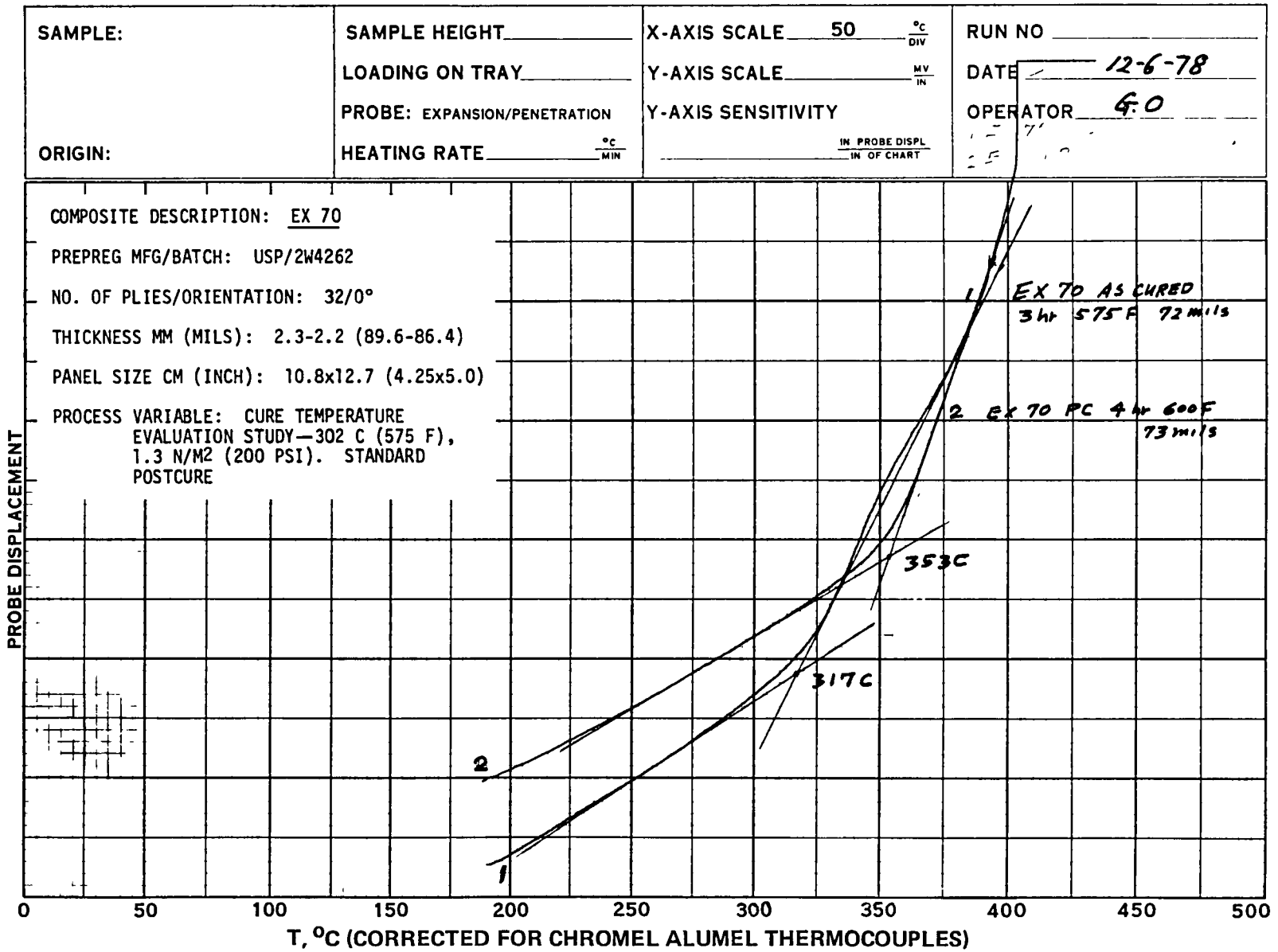


Figure 58. TMA-Tg Characteristics of Specimen EX70

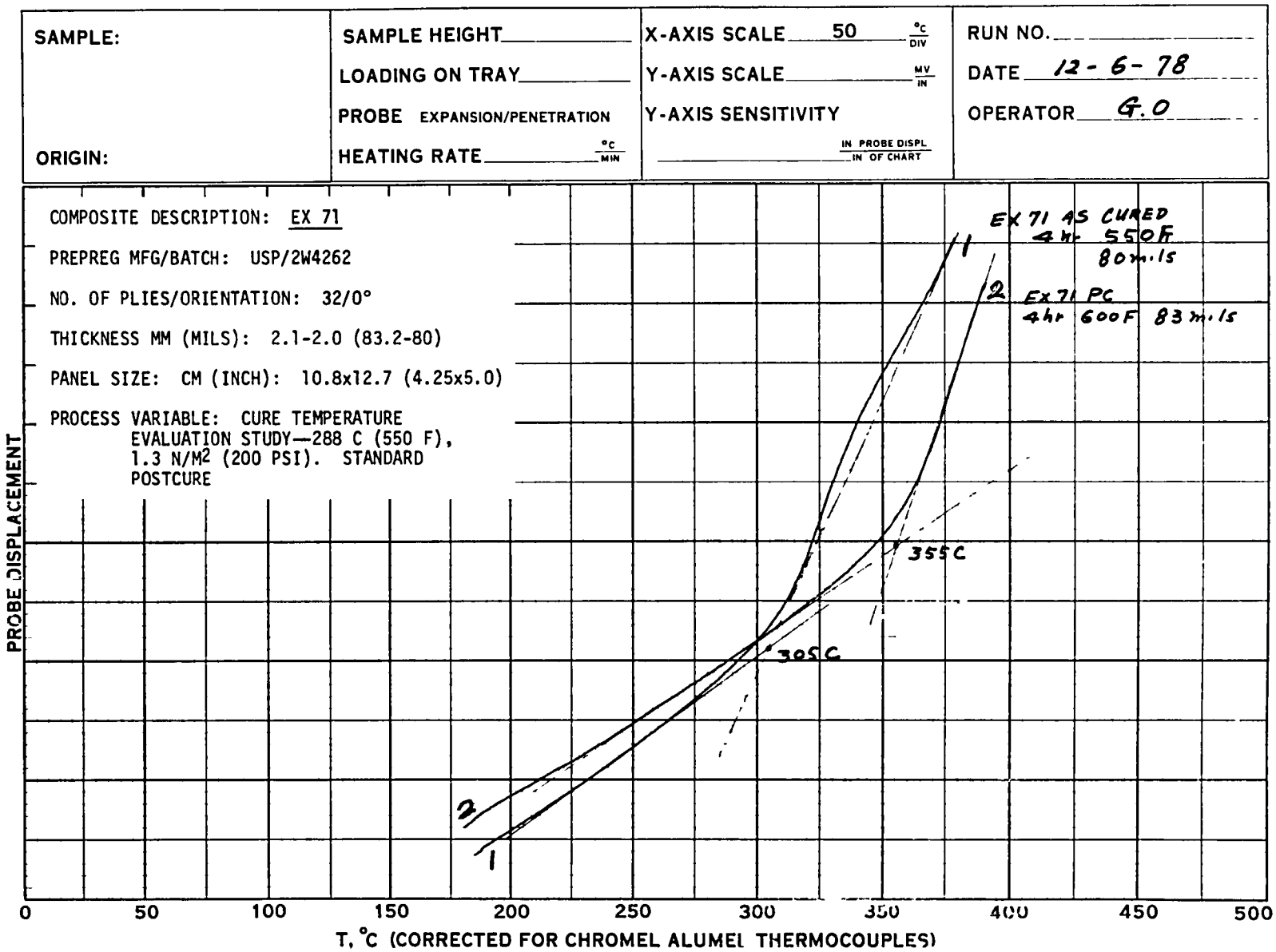


Figure 59. TMA-Tg Characteristics of Specimen EX71

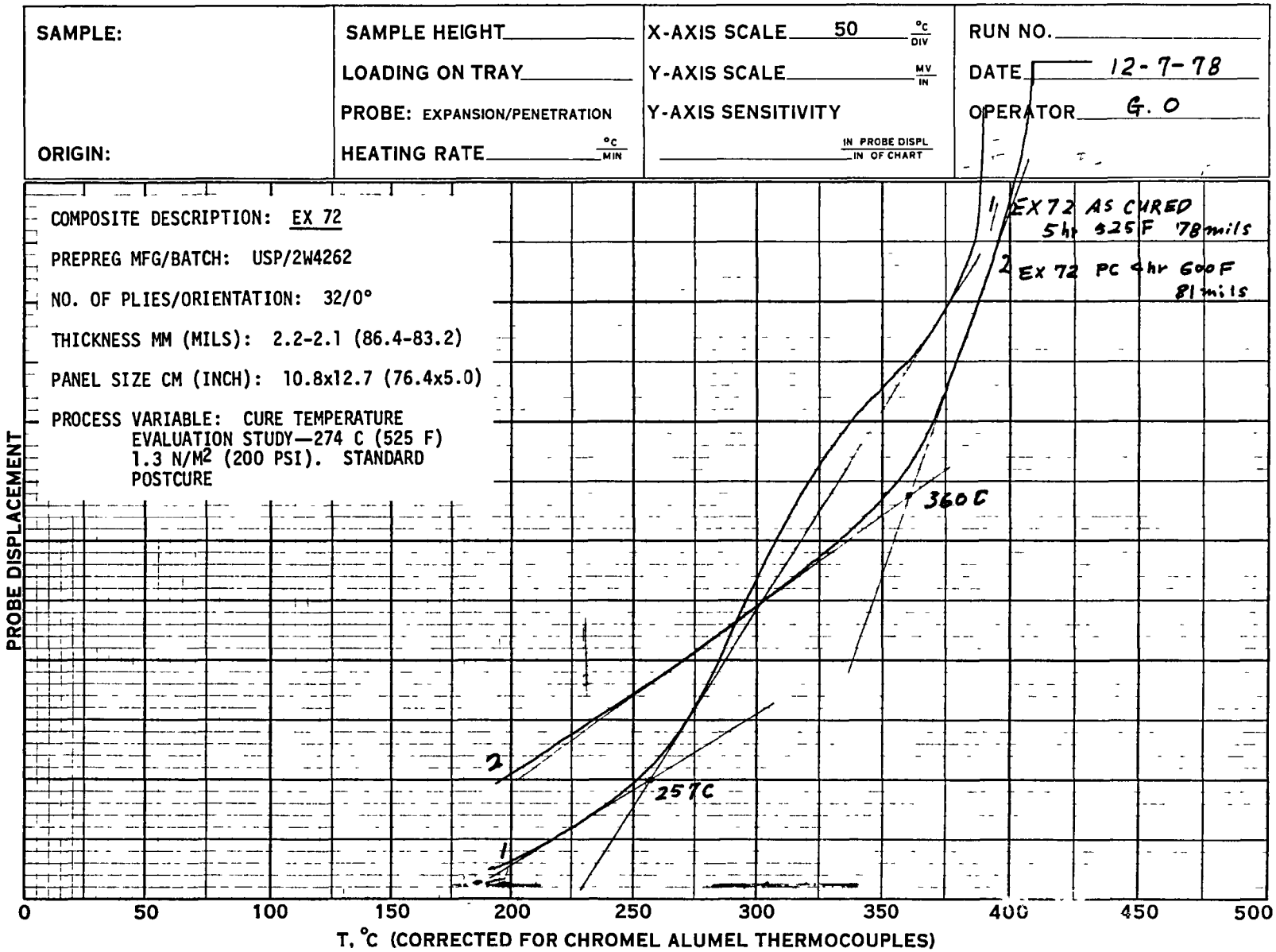


Figure 60. TMA-Tg Characteristics of Specimen EX 72

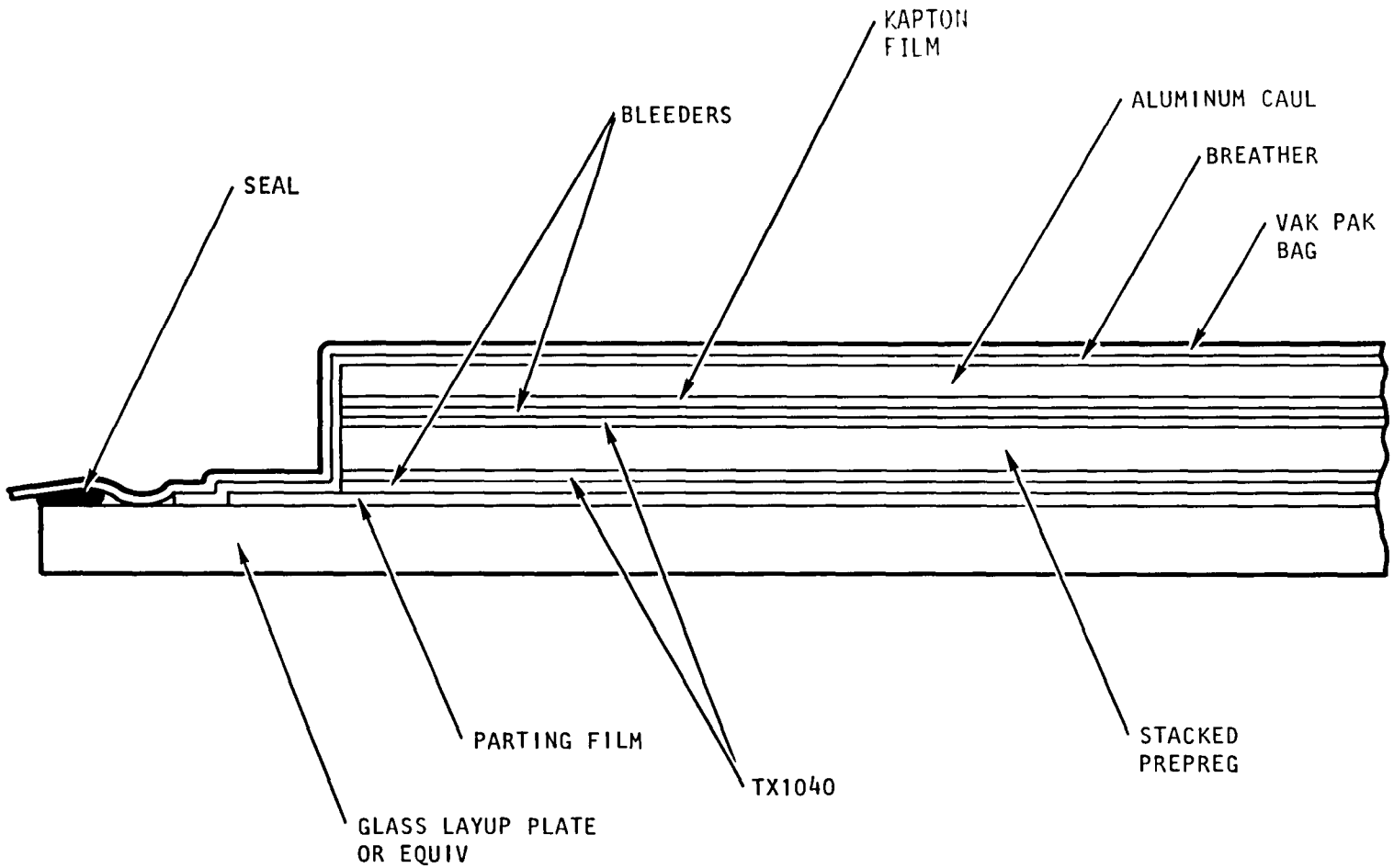


Figure 61. Typical Configuration for Debulking Celion/LARC-160 Prepreg

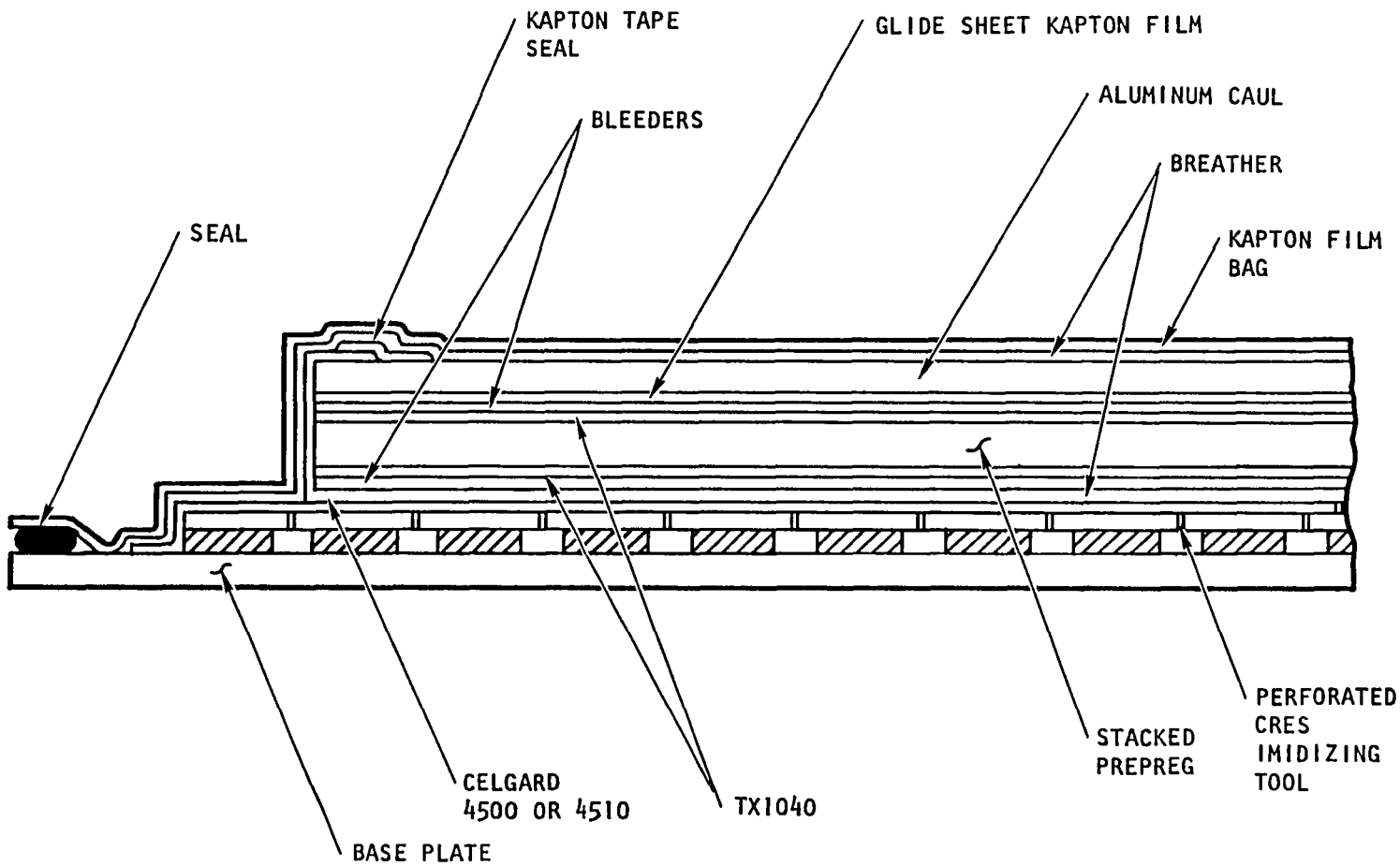


Figure 62. Typical Configuration for Imidizing Celion/LARC-160 Prepreg

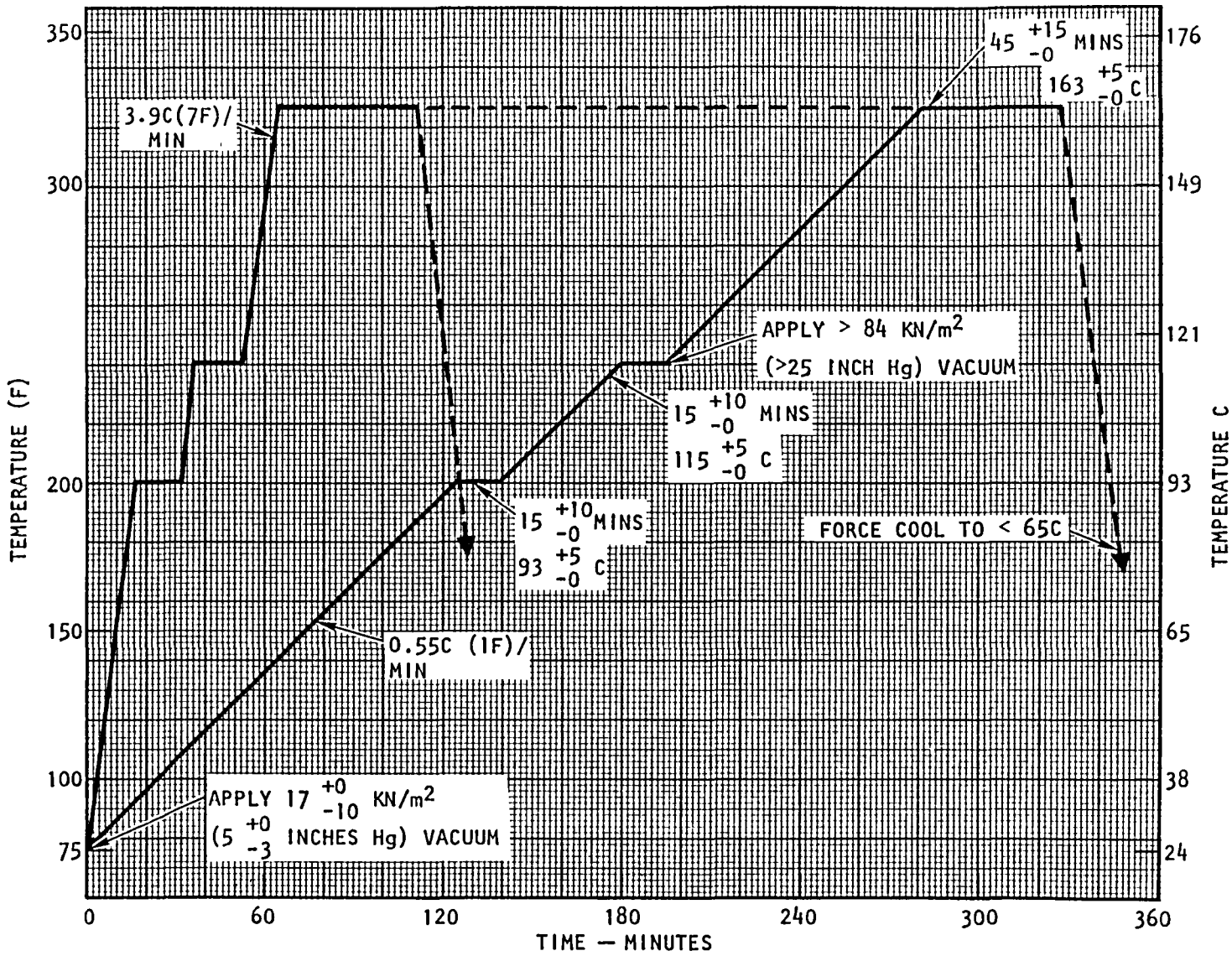


Figure 63. LARC-160/Celion Imidizing Cycle Window and Sequence of Events

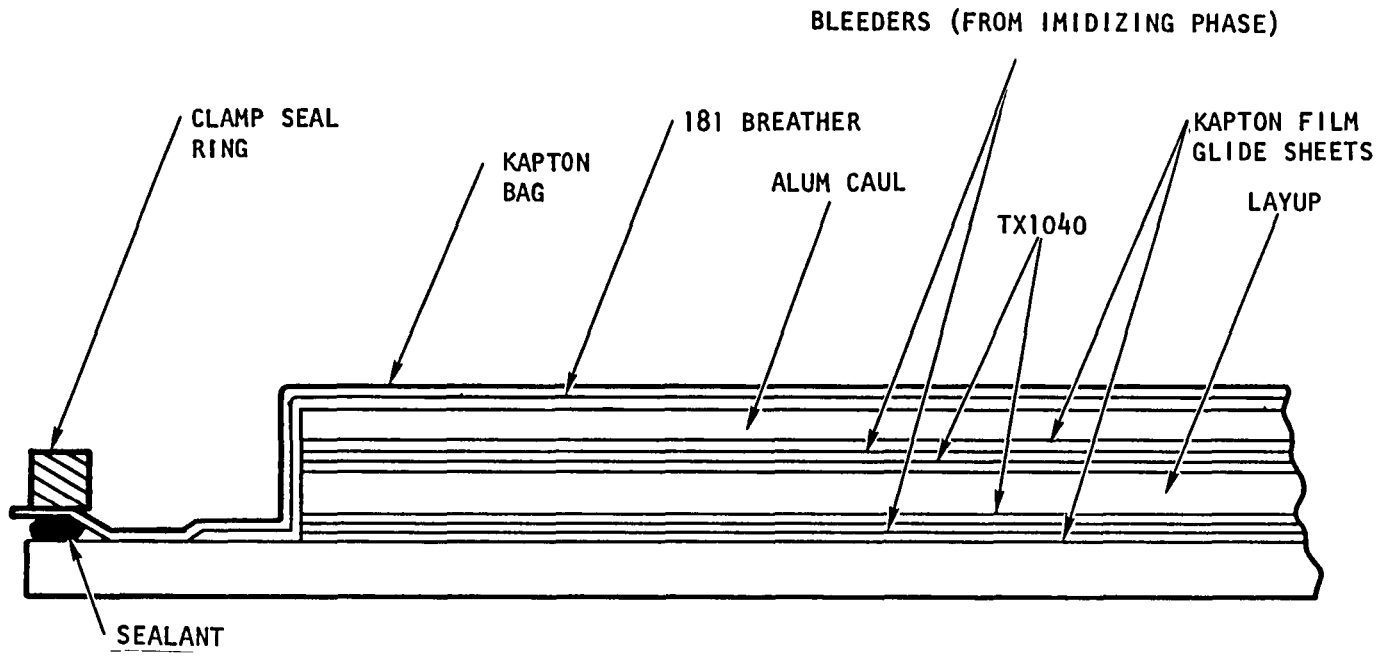


Figure 64. Typical Configuration for Curing Imidized Laminates

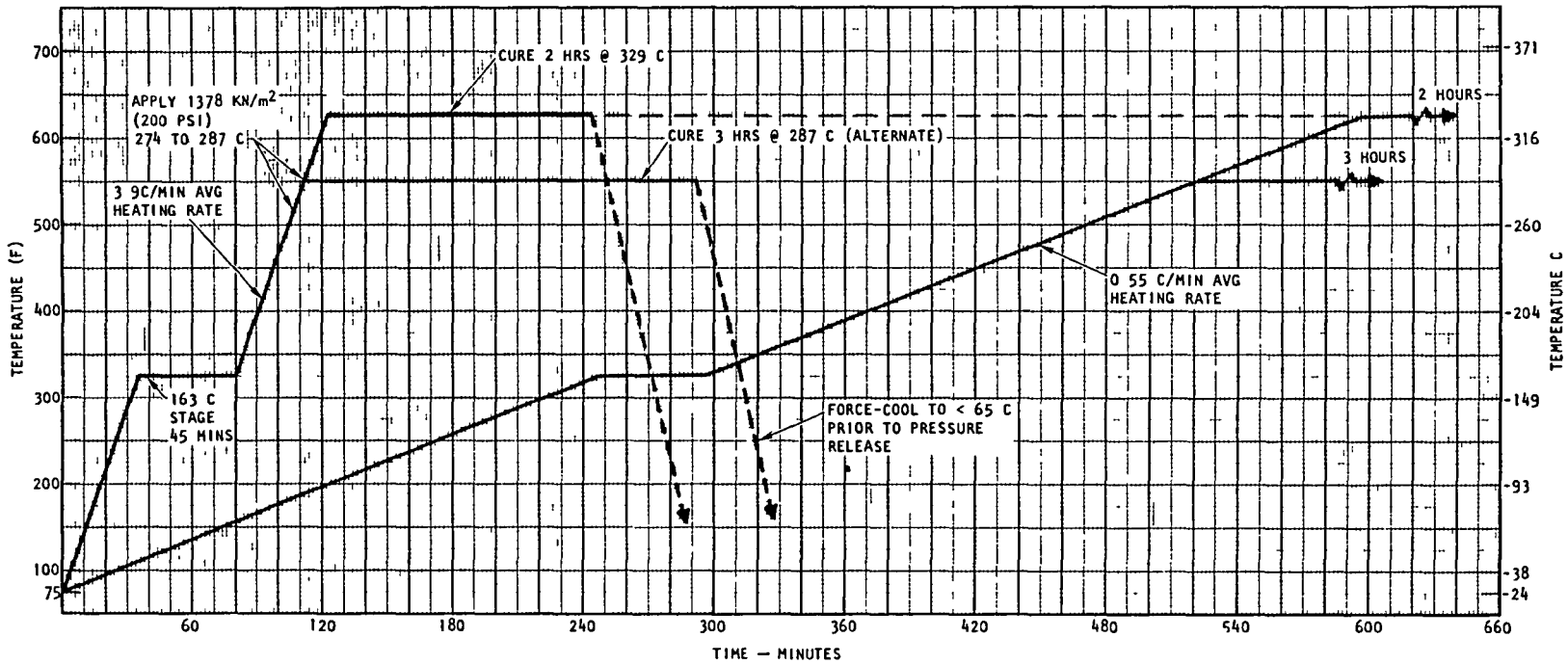


Figure 65. LARC-160/Celion Cure Cycle Window and Sequence of Events (Imidized Prepreg)

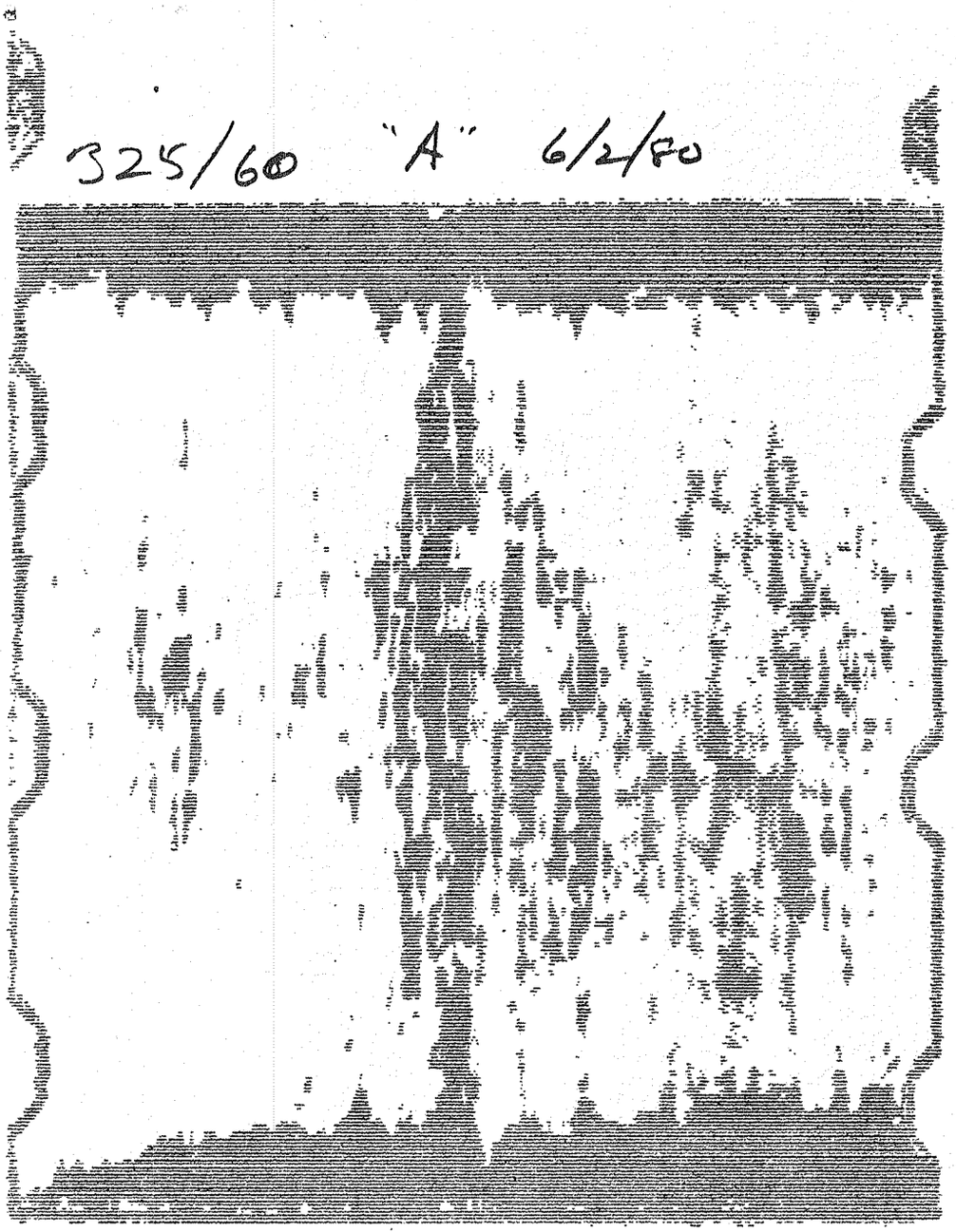


Figure 66. C-Scan of Laminate Imidized at 163 C, 60 Minutes

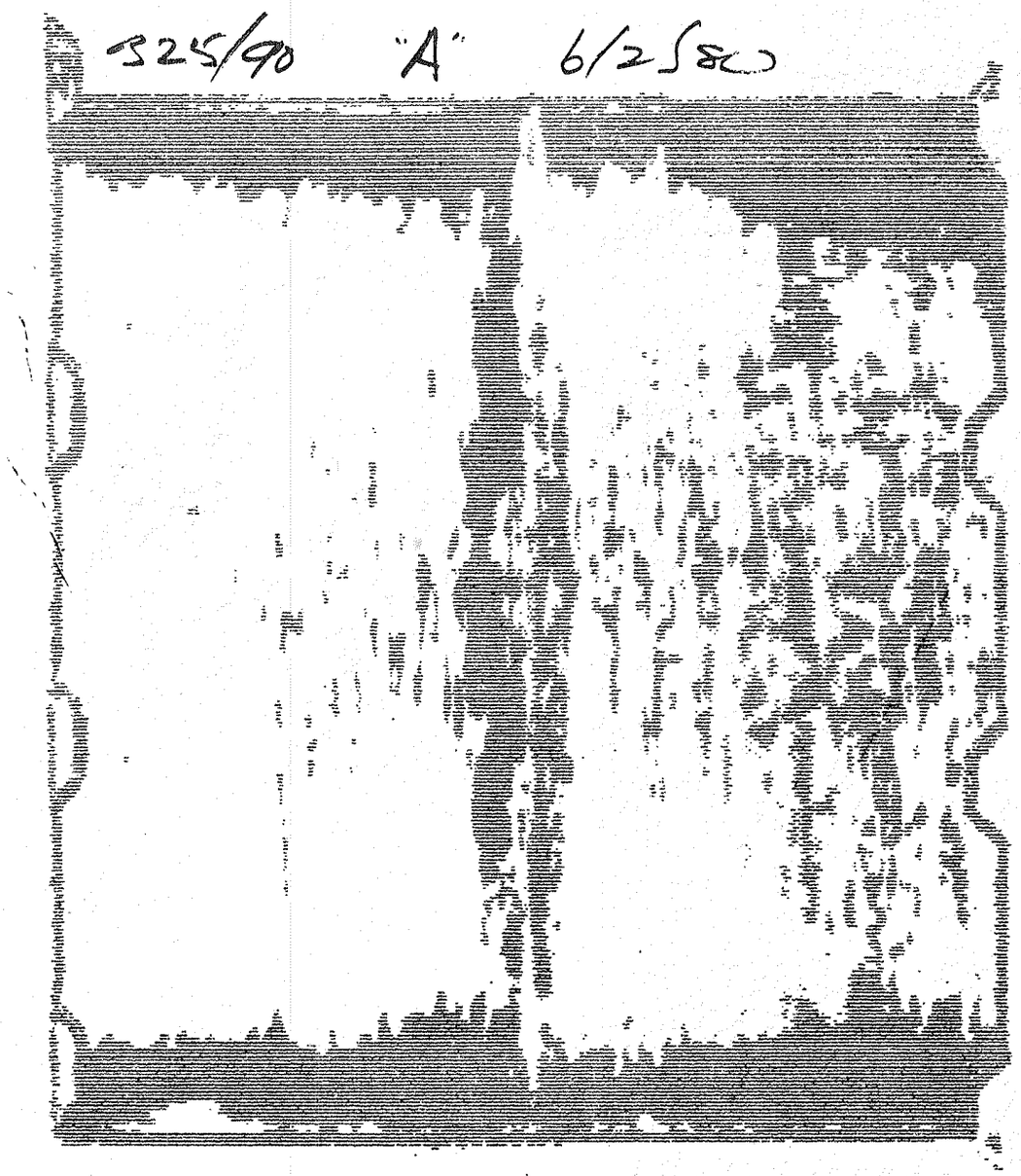


Figure 67. C-Scan of Laminate Imidized at 163 C, 90 Minutes

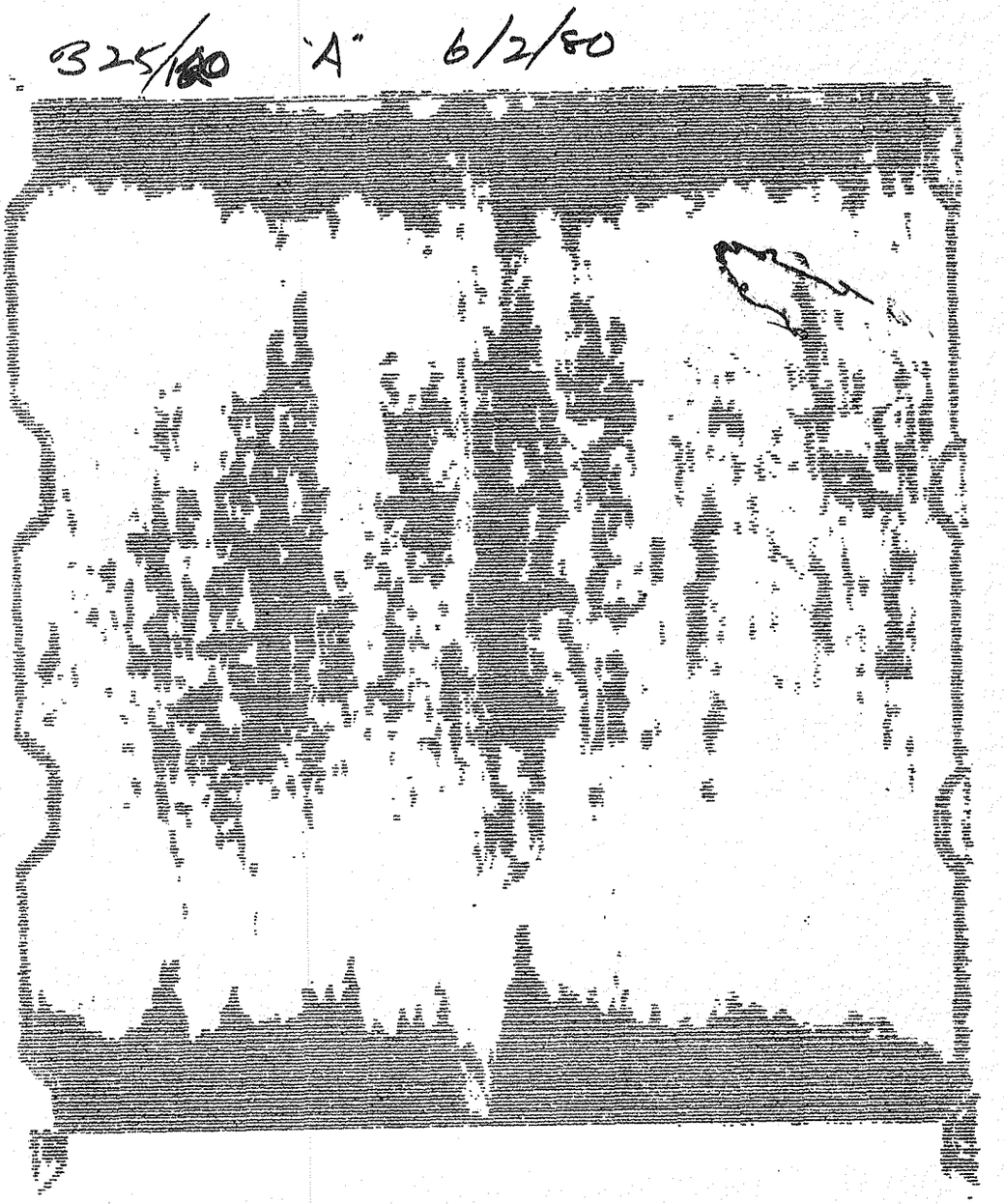


Figure 68. C-Scan of Laminate Imidized at 163 C, 120 Minutes

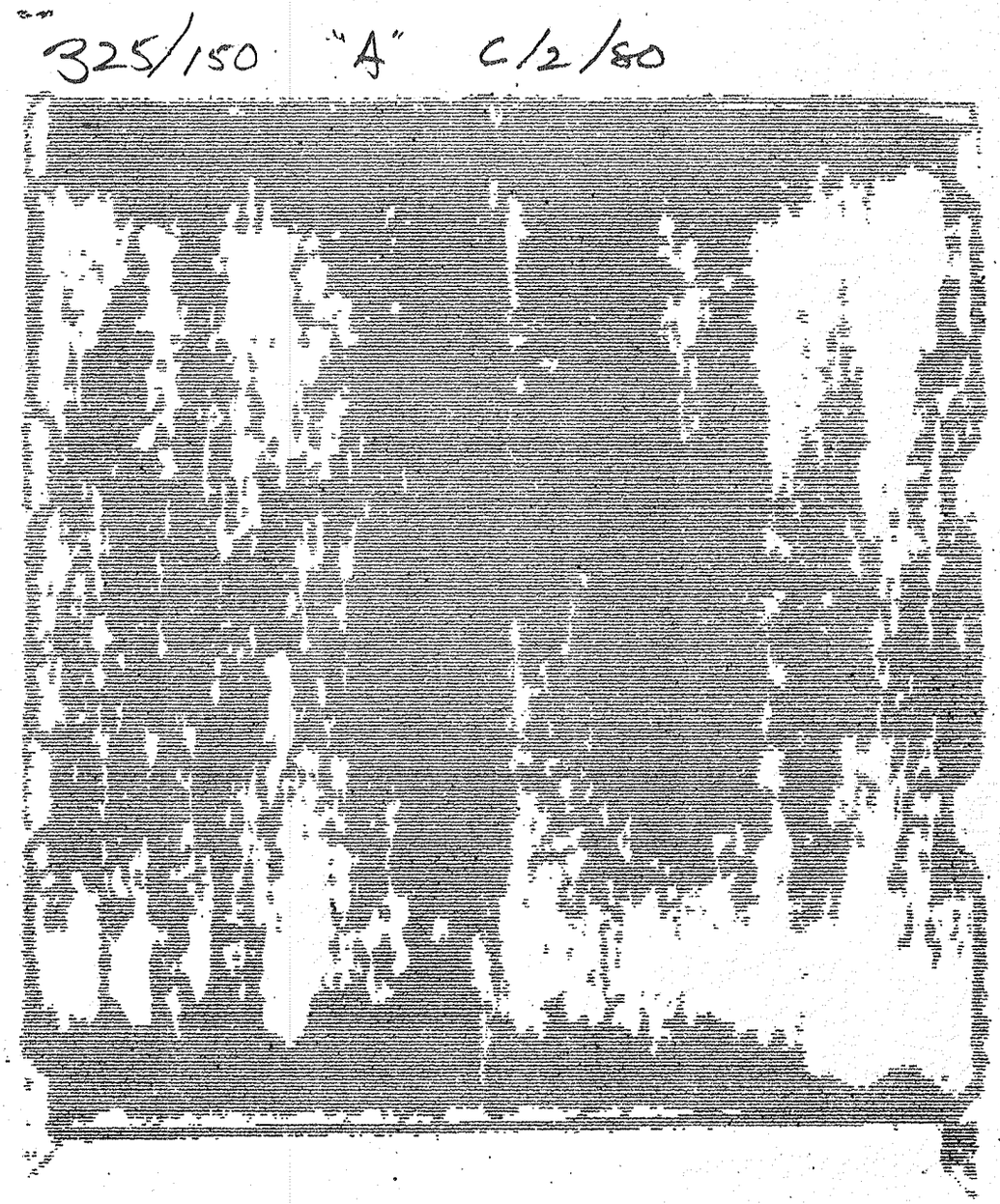


Figure 69. C-Scan of Laminate Imidized at 163 C, 150 Minutes

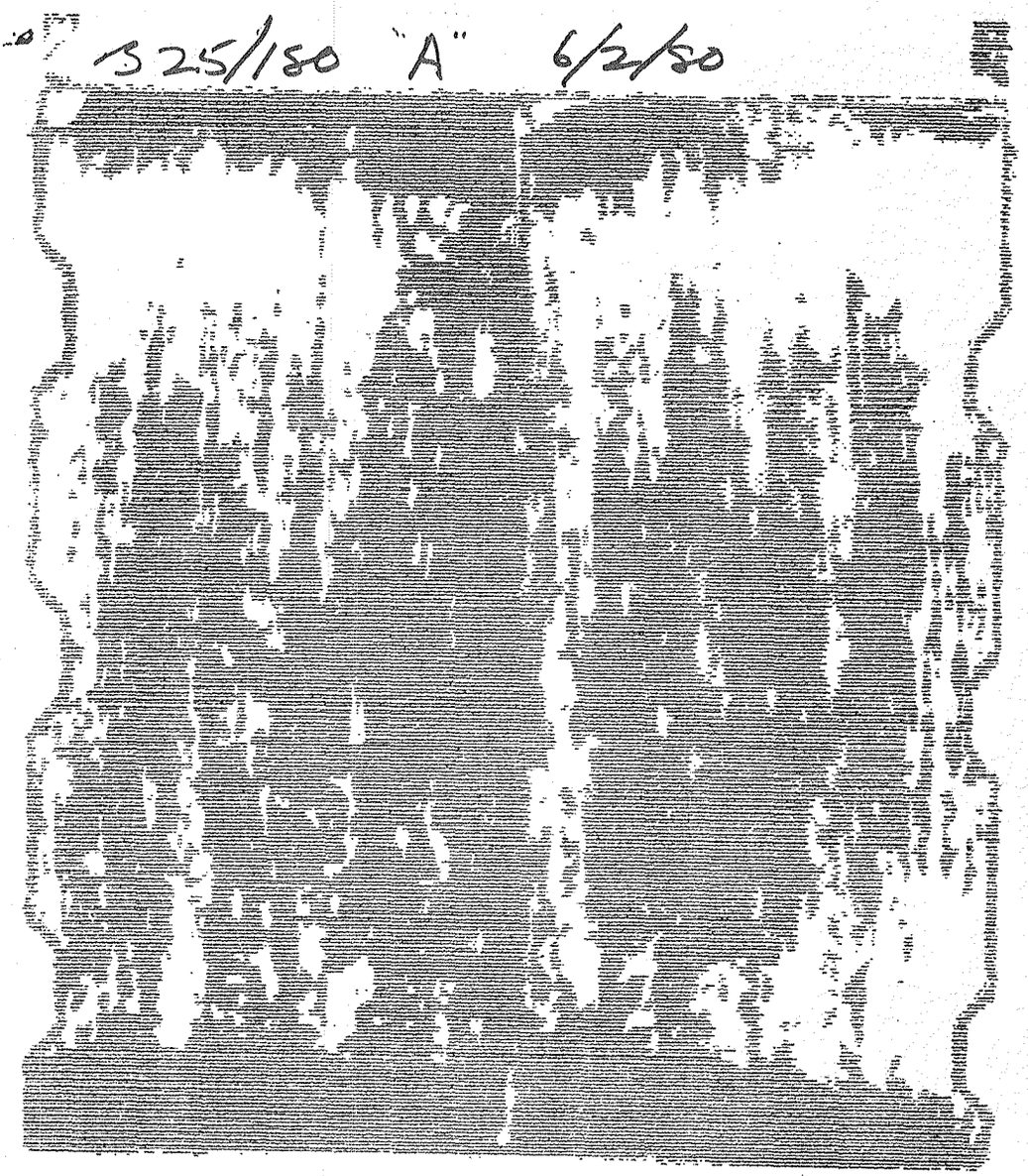


Figure 70. C-Scan of Laminate Imidized at 163 C, 180 Minutes

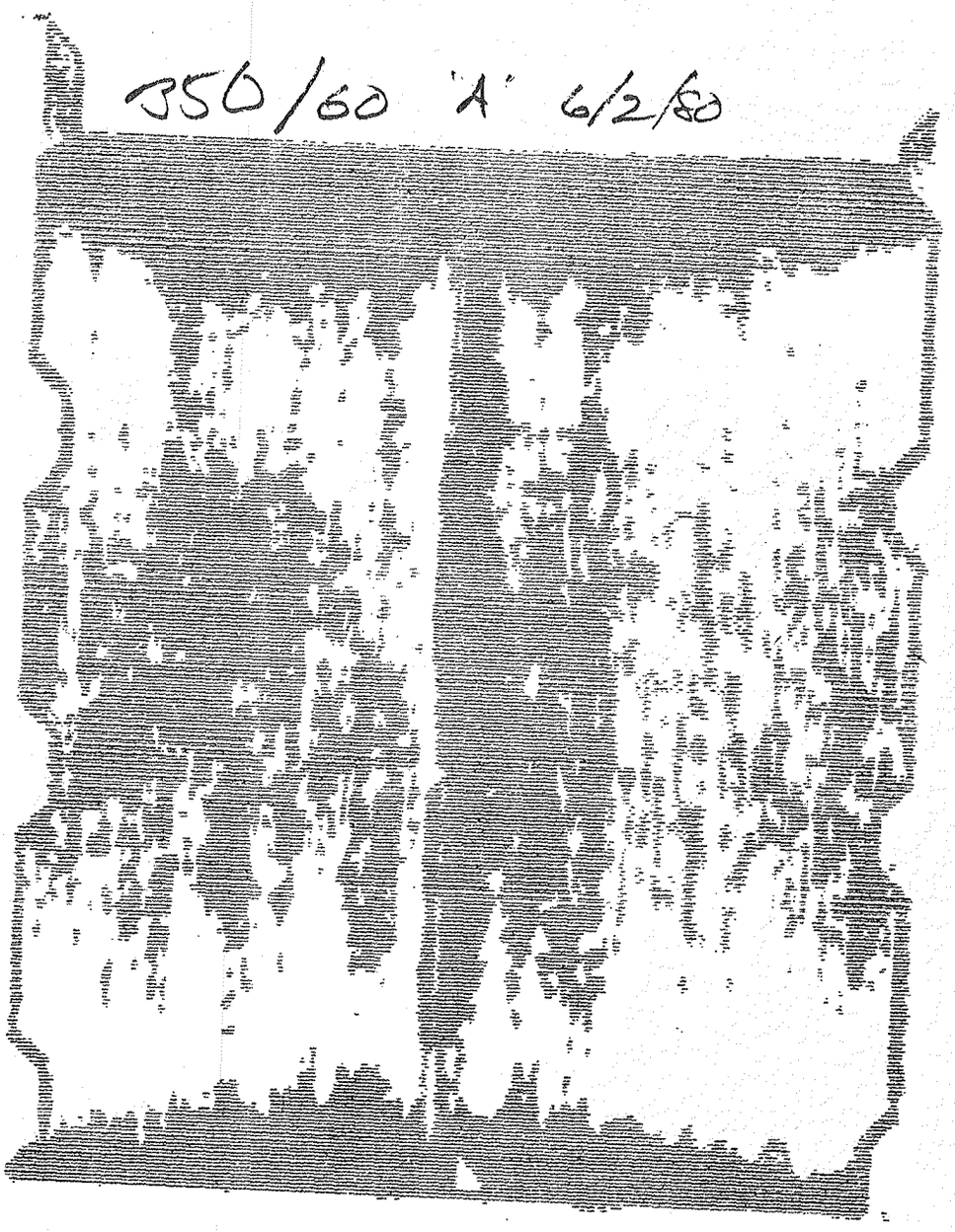


Figure 71. C-Scan of Laminate Imidized at 177 C, 60 Minutes

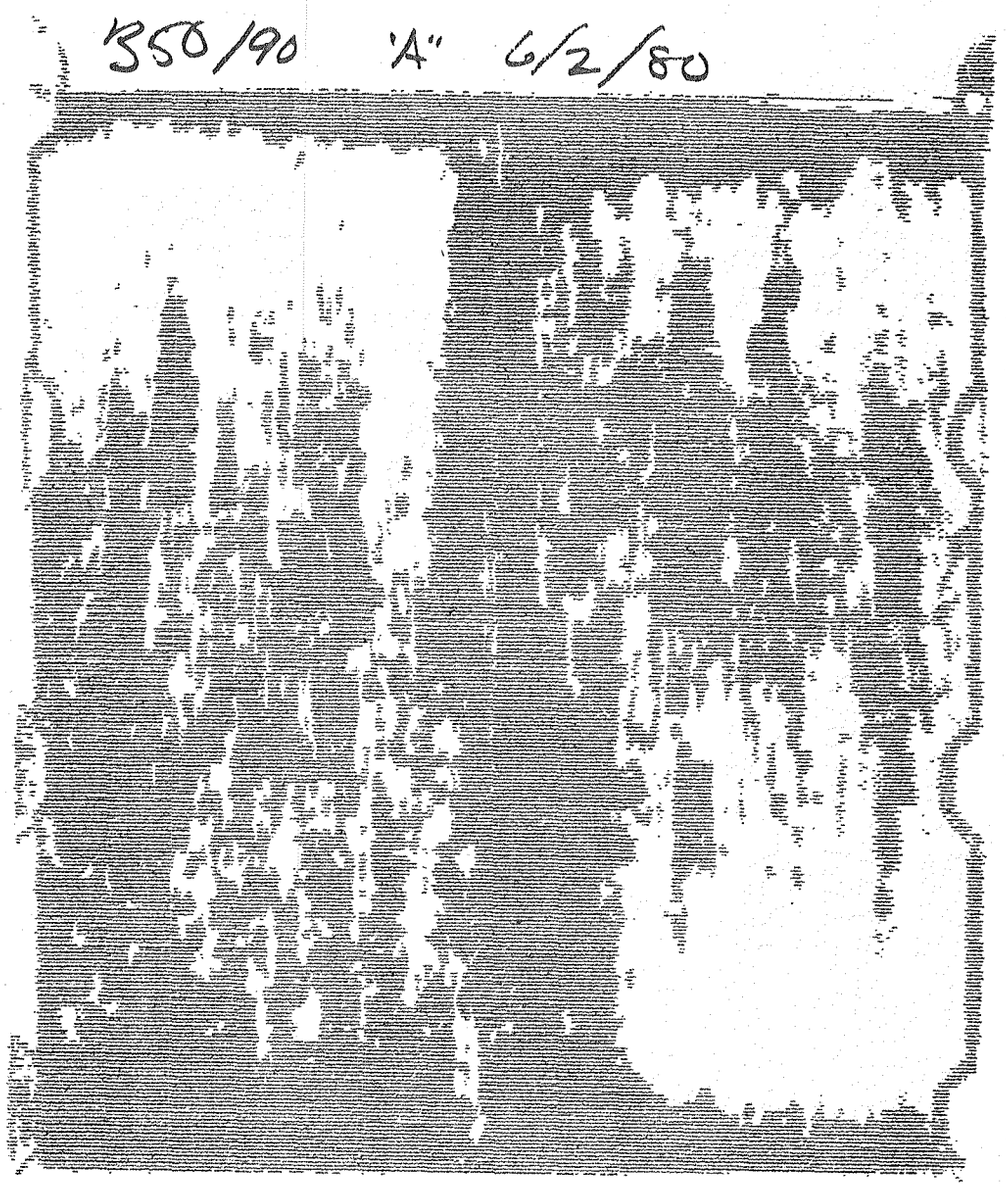


Figure 72. C-Scan of Laminate Imidized at 177 C, 90 Minutes

Figure 73. C-Scan of Laminate Imidized at 177 C, 120 Minutes

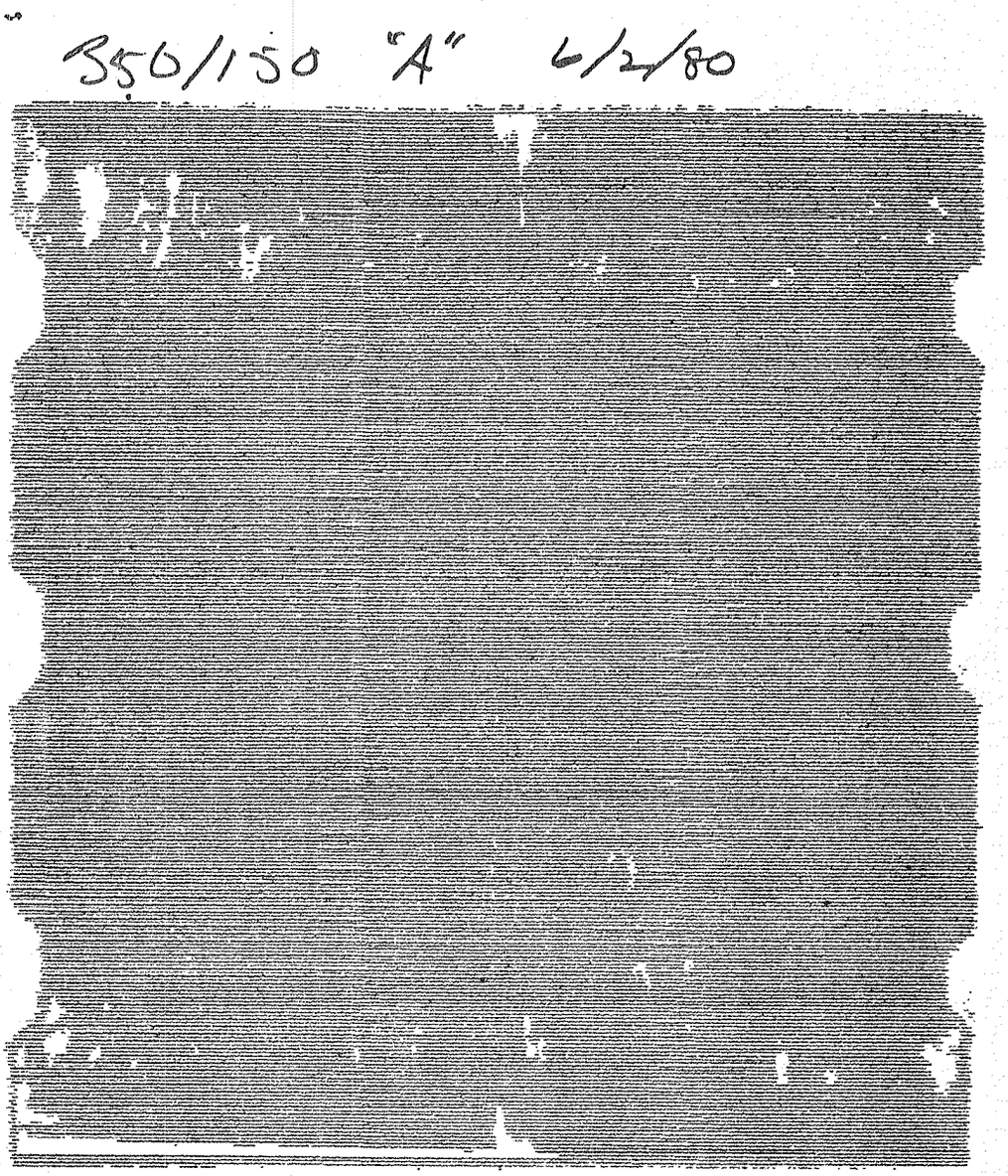


Figure 74. C-Scan of Laminate Imidized at 177 C, 150 Minutes

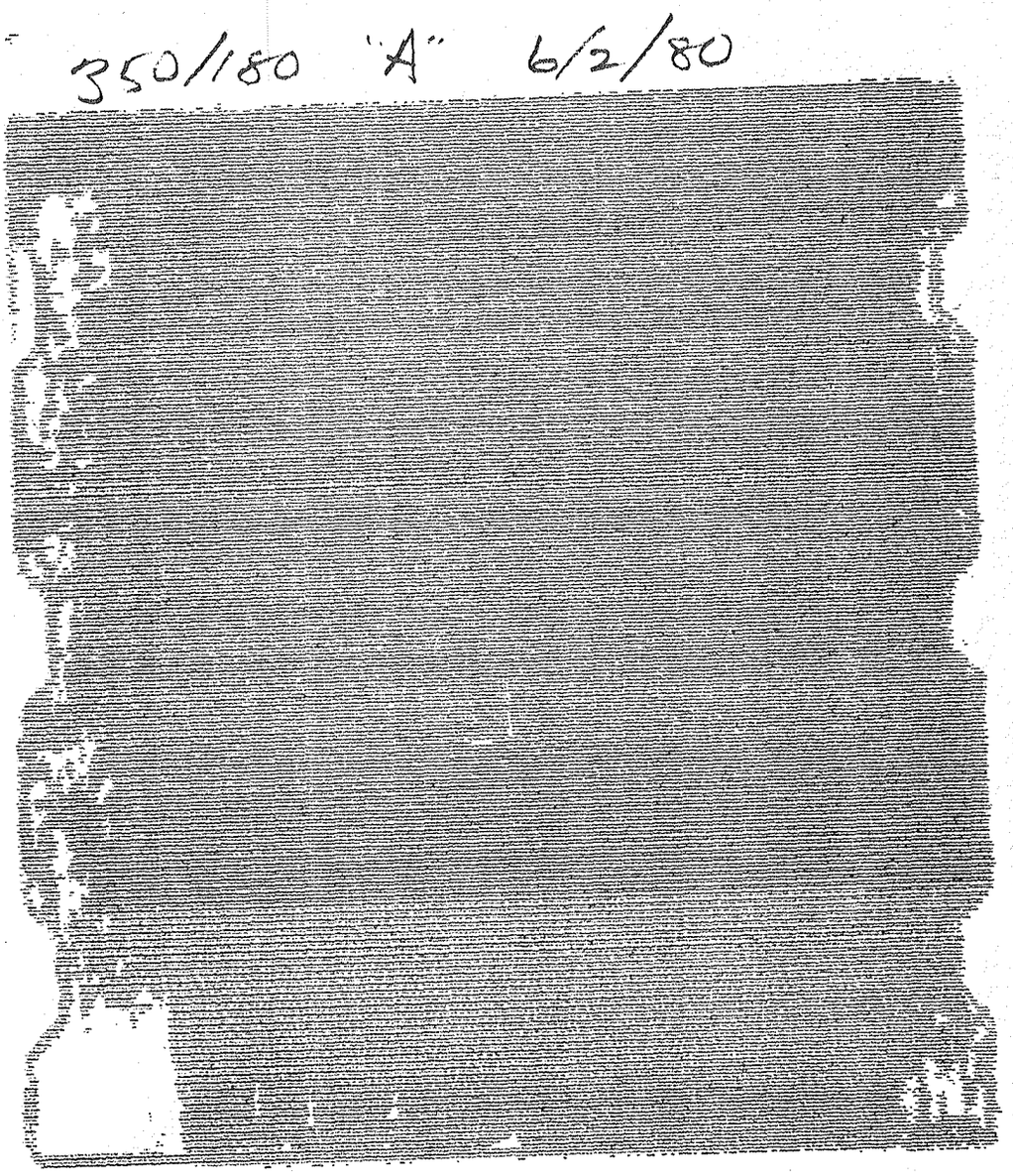


Figure 75. C-Scan of Laminate Imidized at 177 C, 180 Minutes

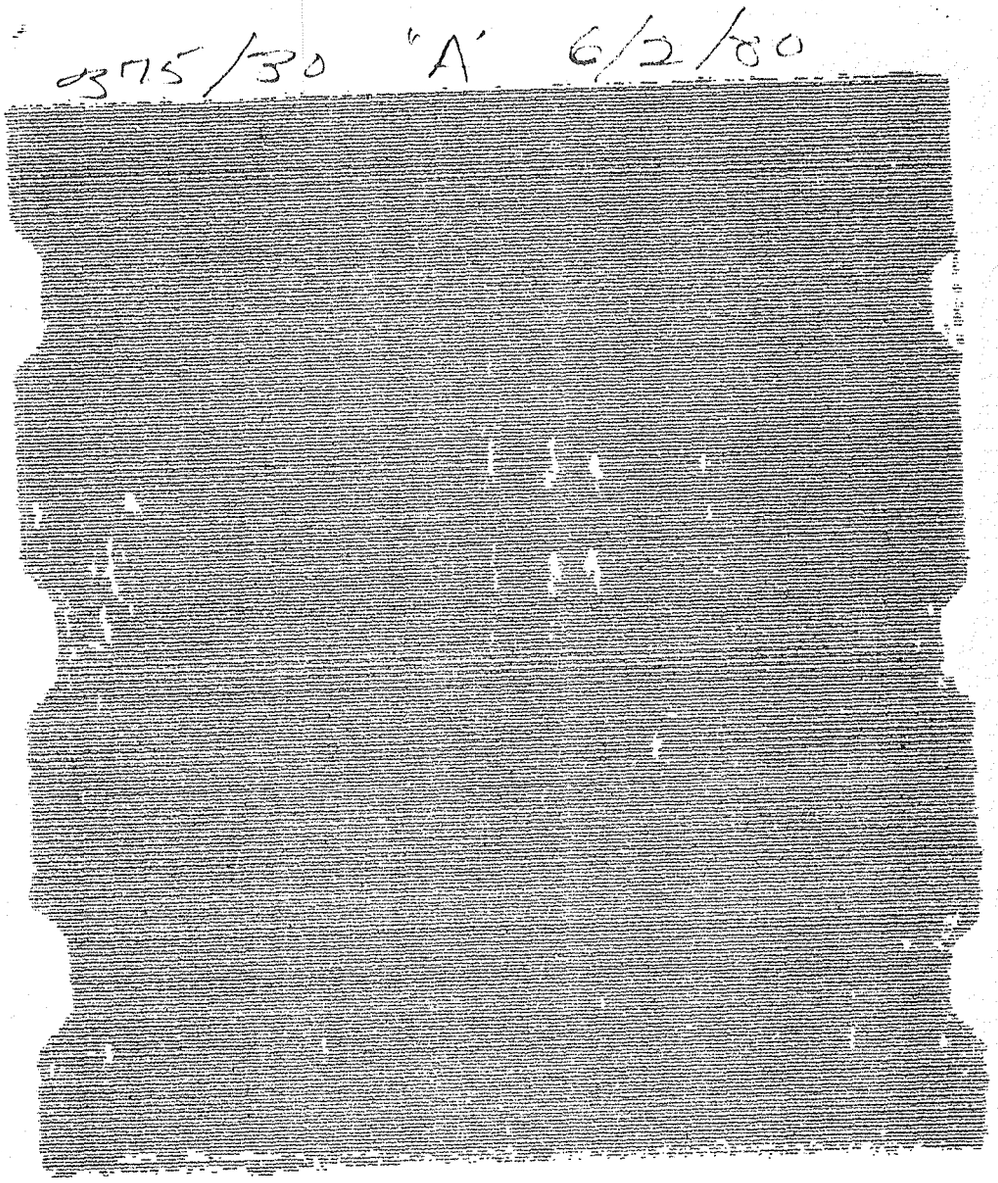


Figure 76. C-Scan of Laminate Imidized at 191 C, 30 Minutes

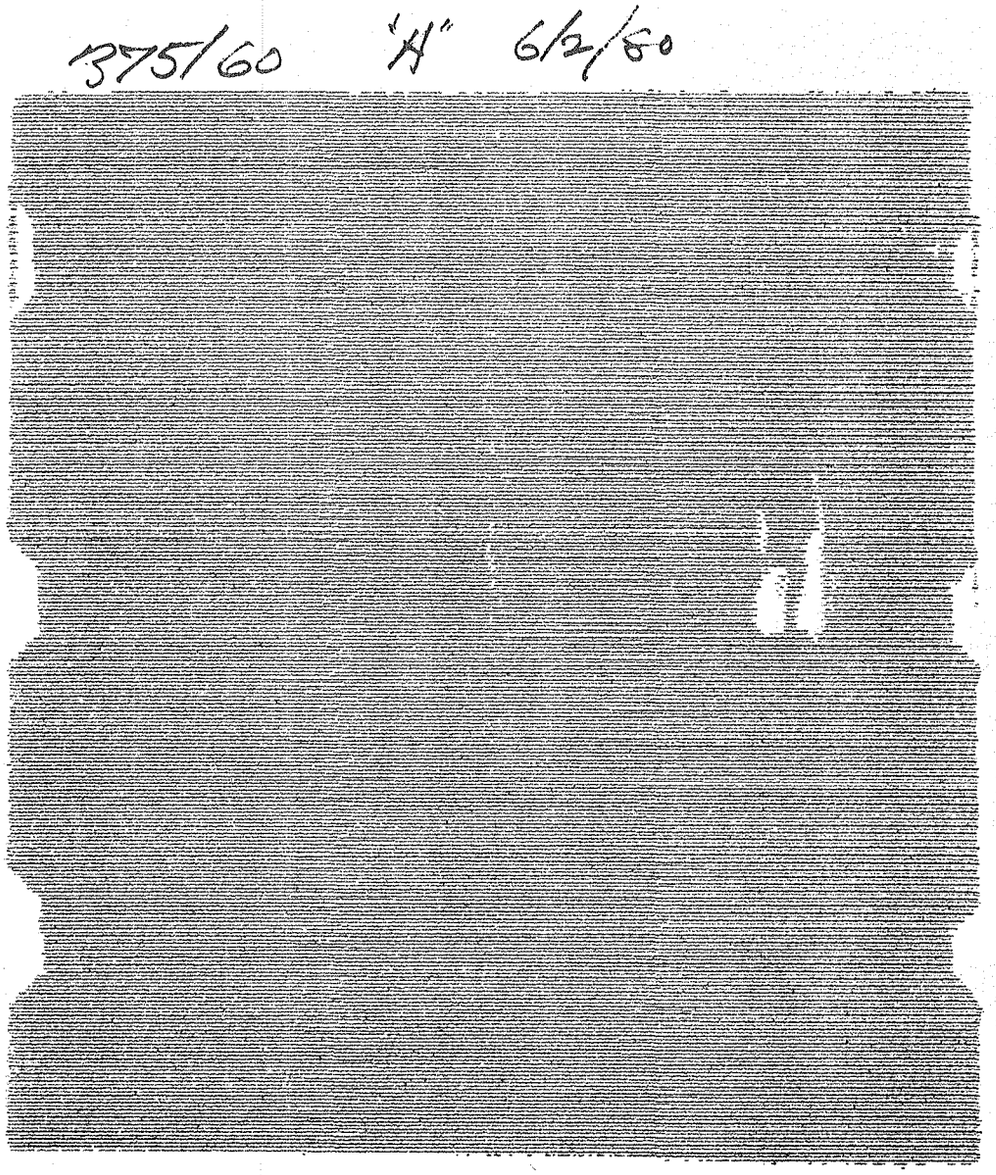


Figure 77. C-Scan of Laminate Imidized at 191 C, 60 Minutes

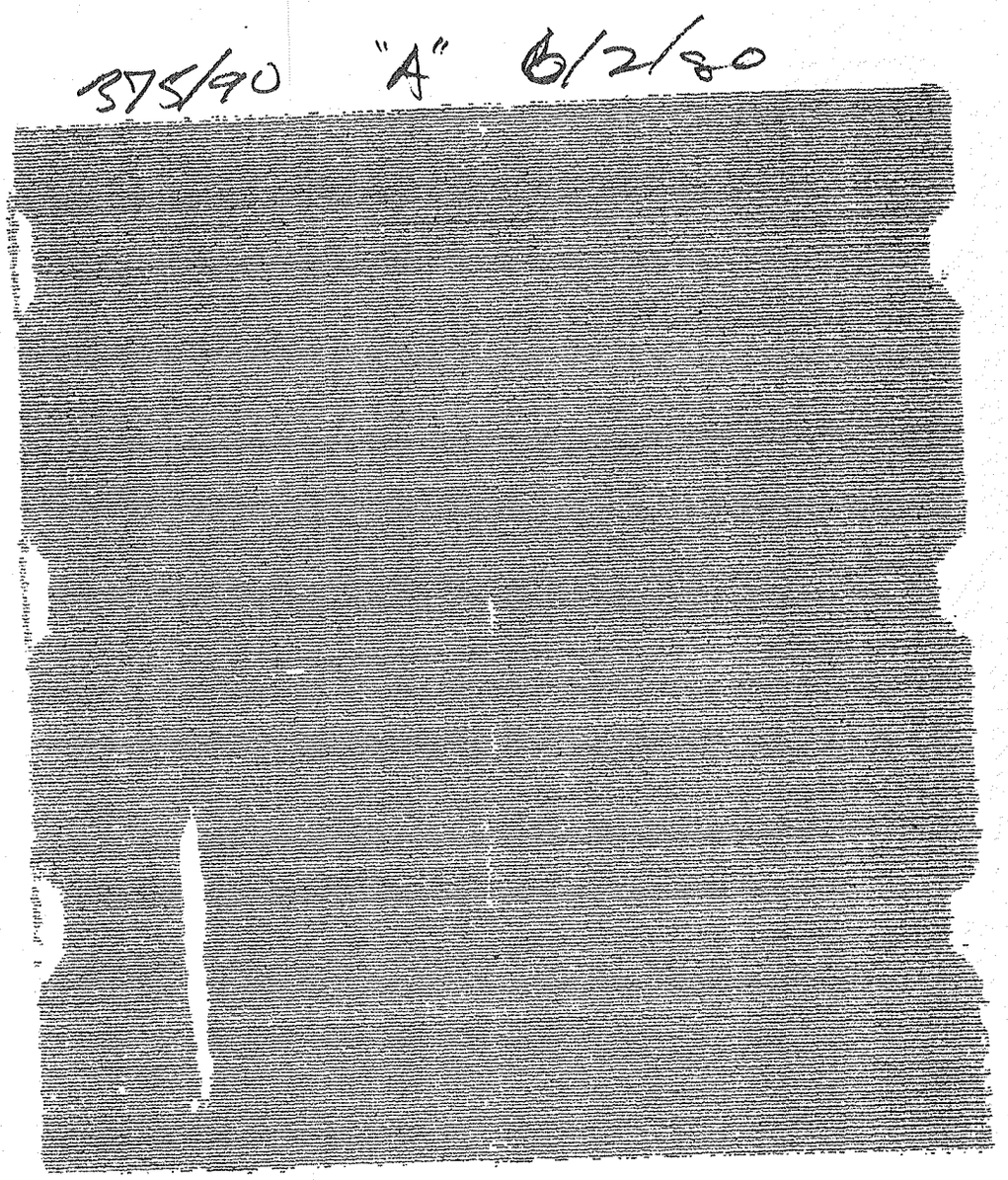


Figure 78. C-Scan of Laminate Imidized at 191 C, 90 Minutes

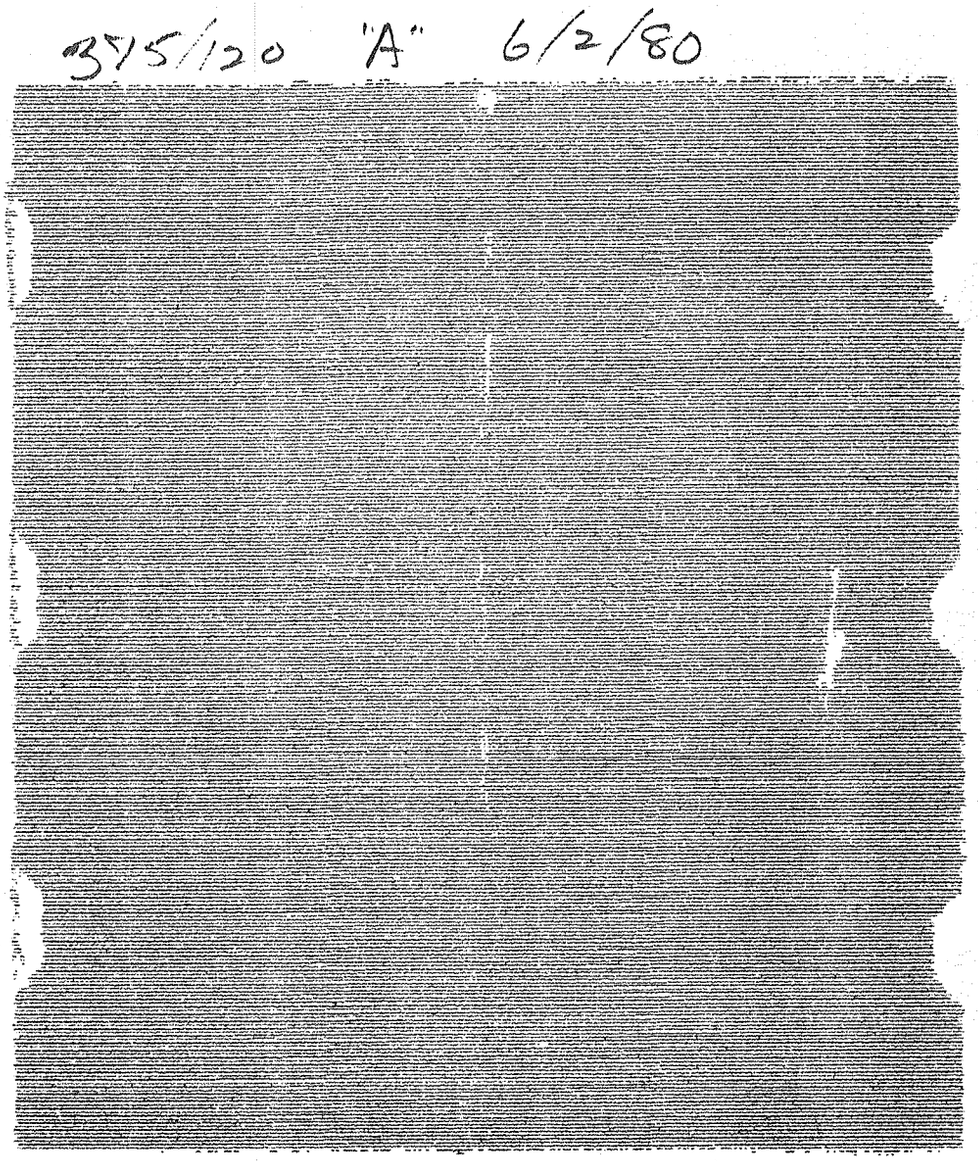


Figure 79. C-Scan of Laminate Imidized at 191 C, 120 Minutes

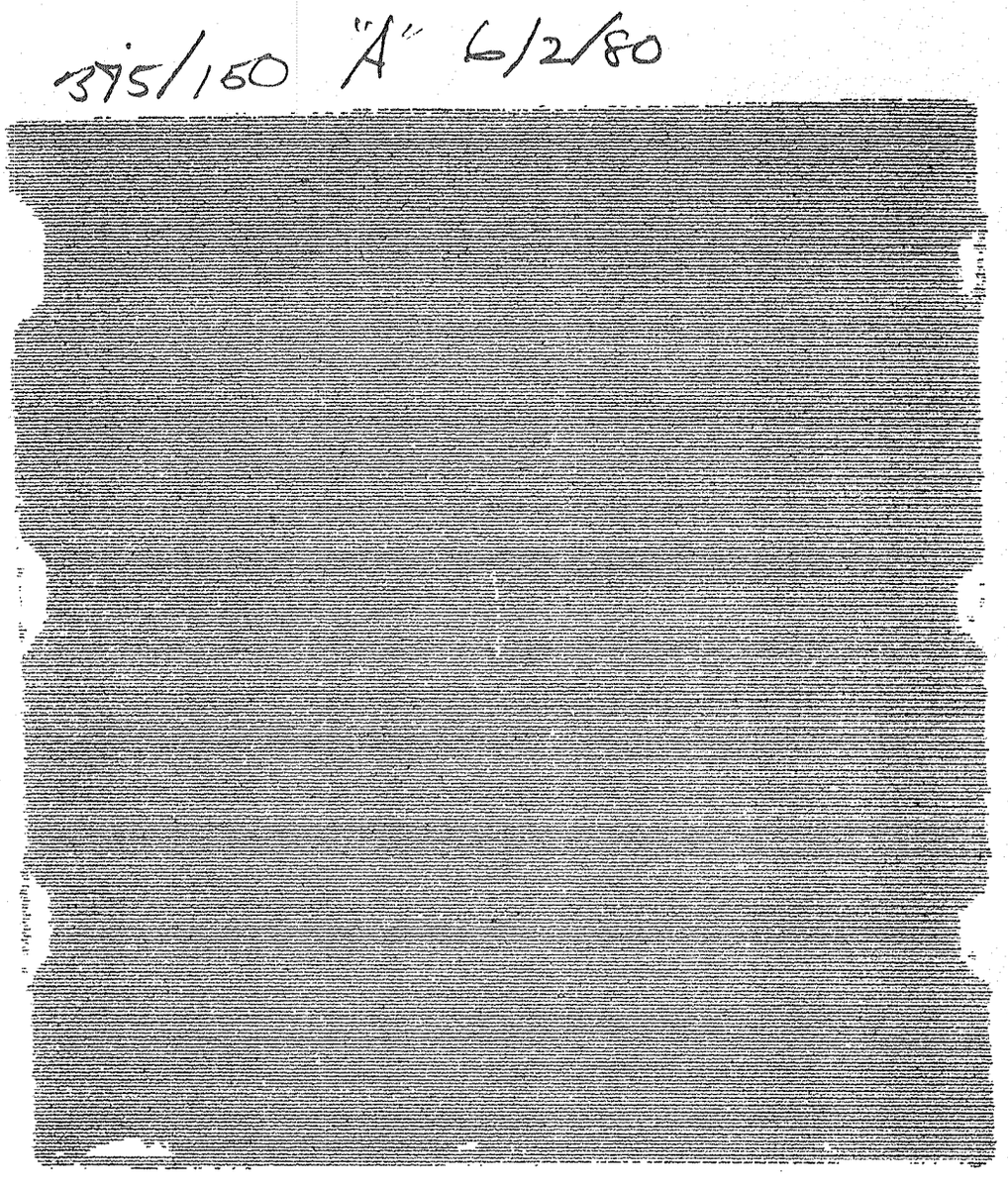


Figure 80. C-Scan of Laminate Imidized at 191 C, 150 Minutes

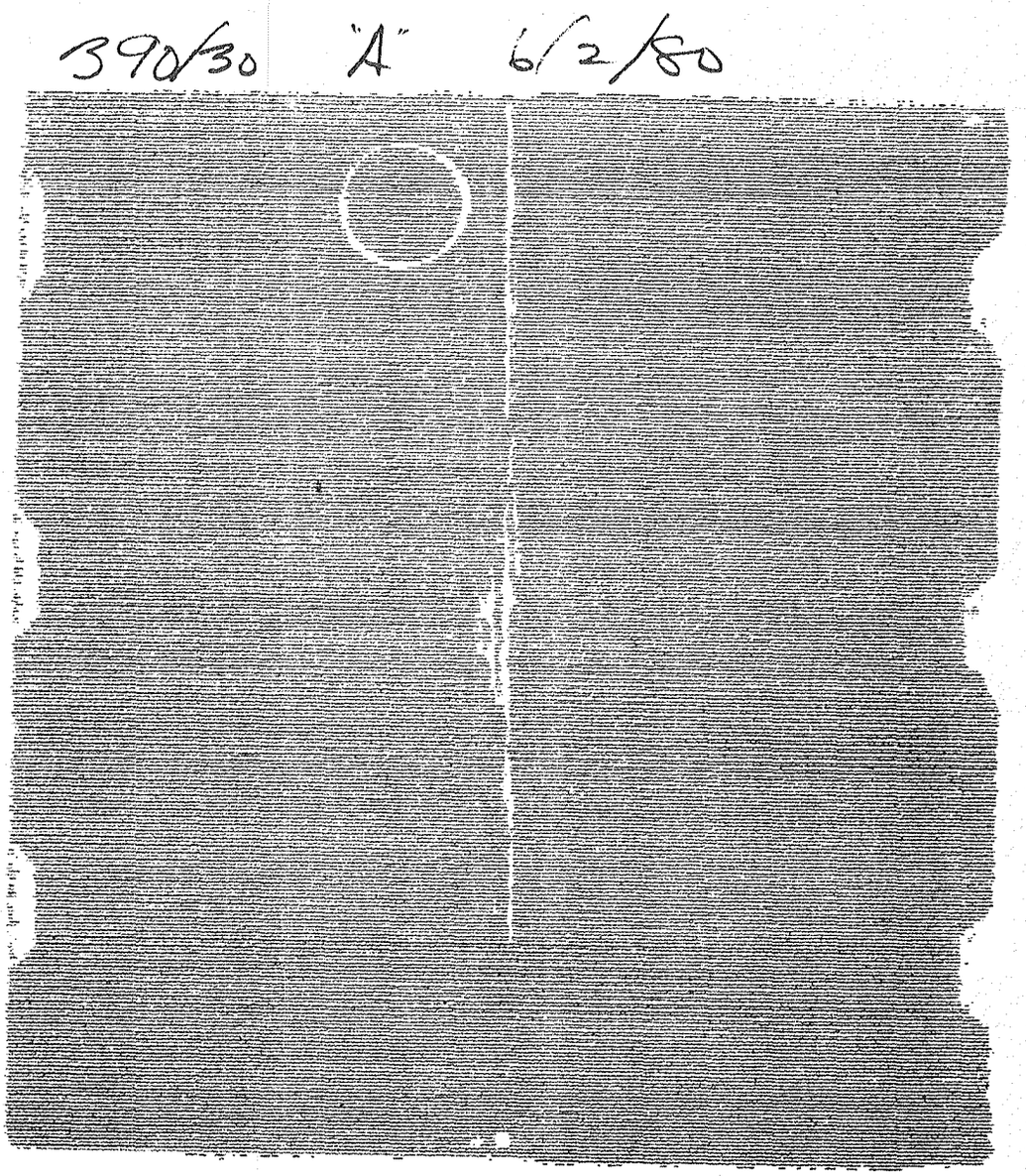


Figure 81. C-Scan of Laminate Imidized at 199 C, 30 Minutes

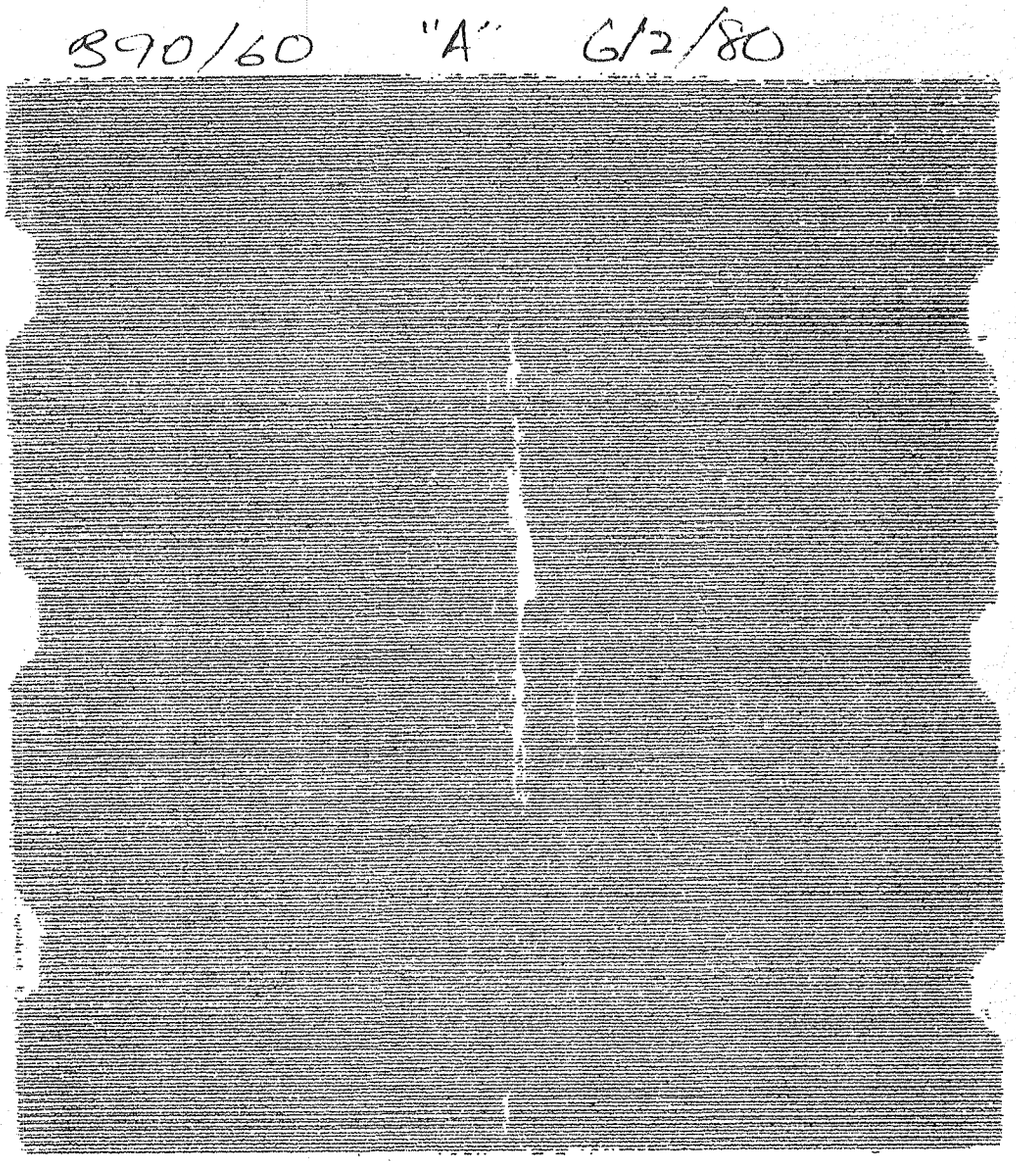


Figure 82. C-Scan of Laminate Imidized at 199 C, 60 Minutes

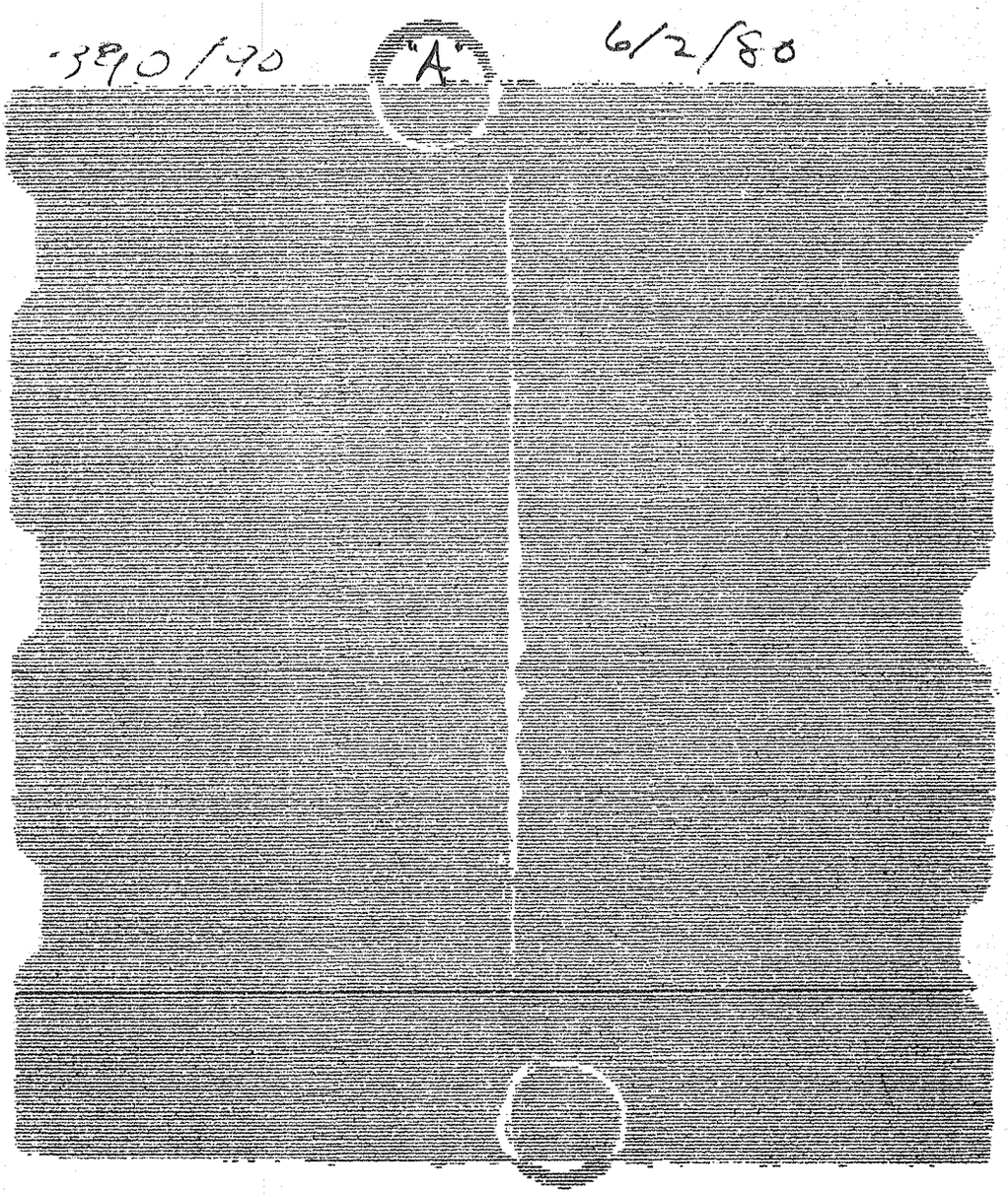


Figure 83. C-Scan of Laminate Imidized at 199 C, 90 Minutes

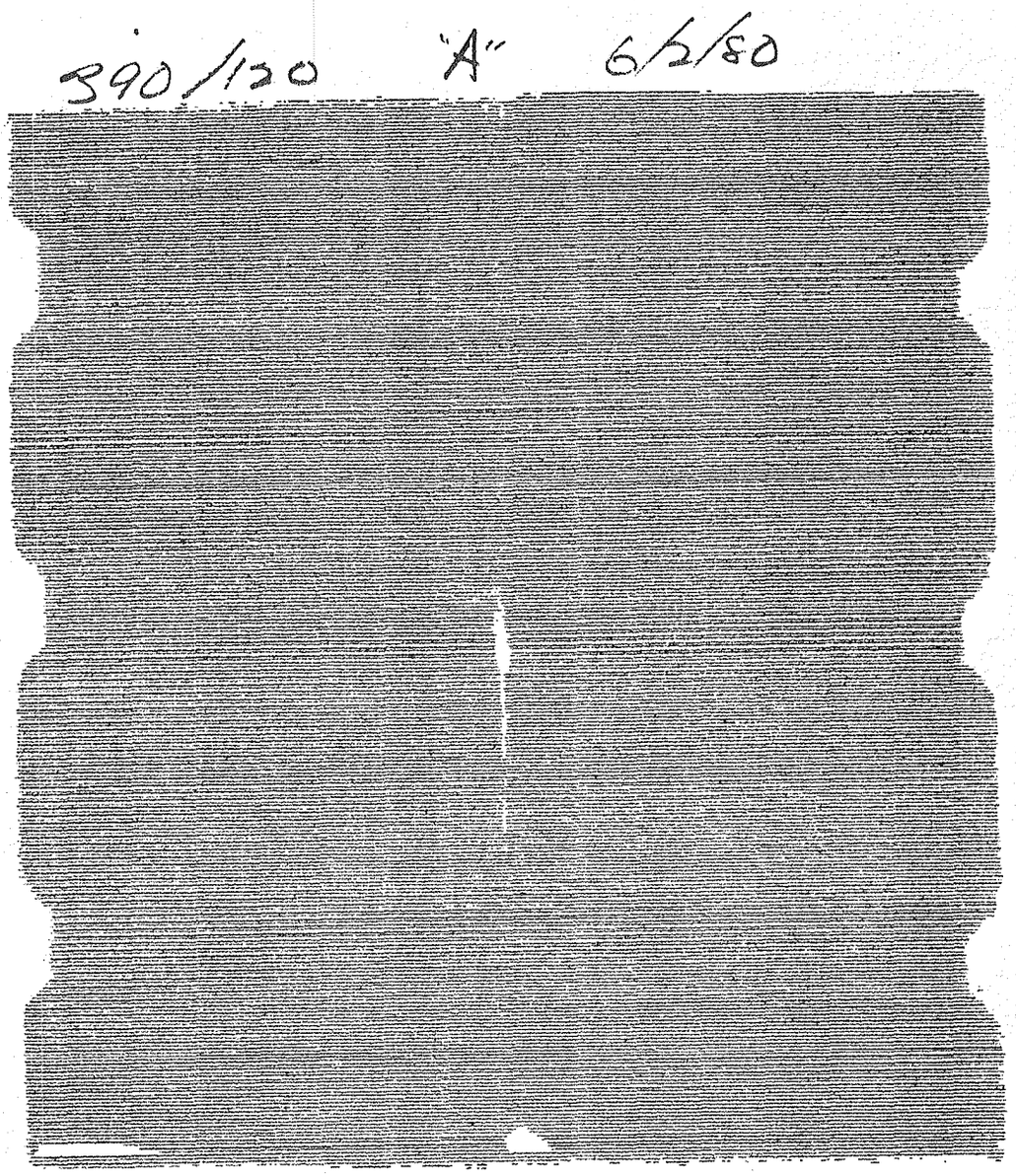


Figure 84. C-Scan of Laminate Imidized at 199 C, 120 Minutes

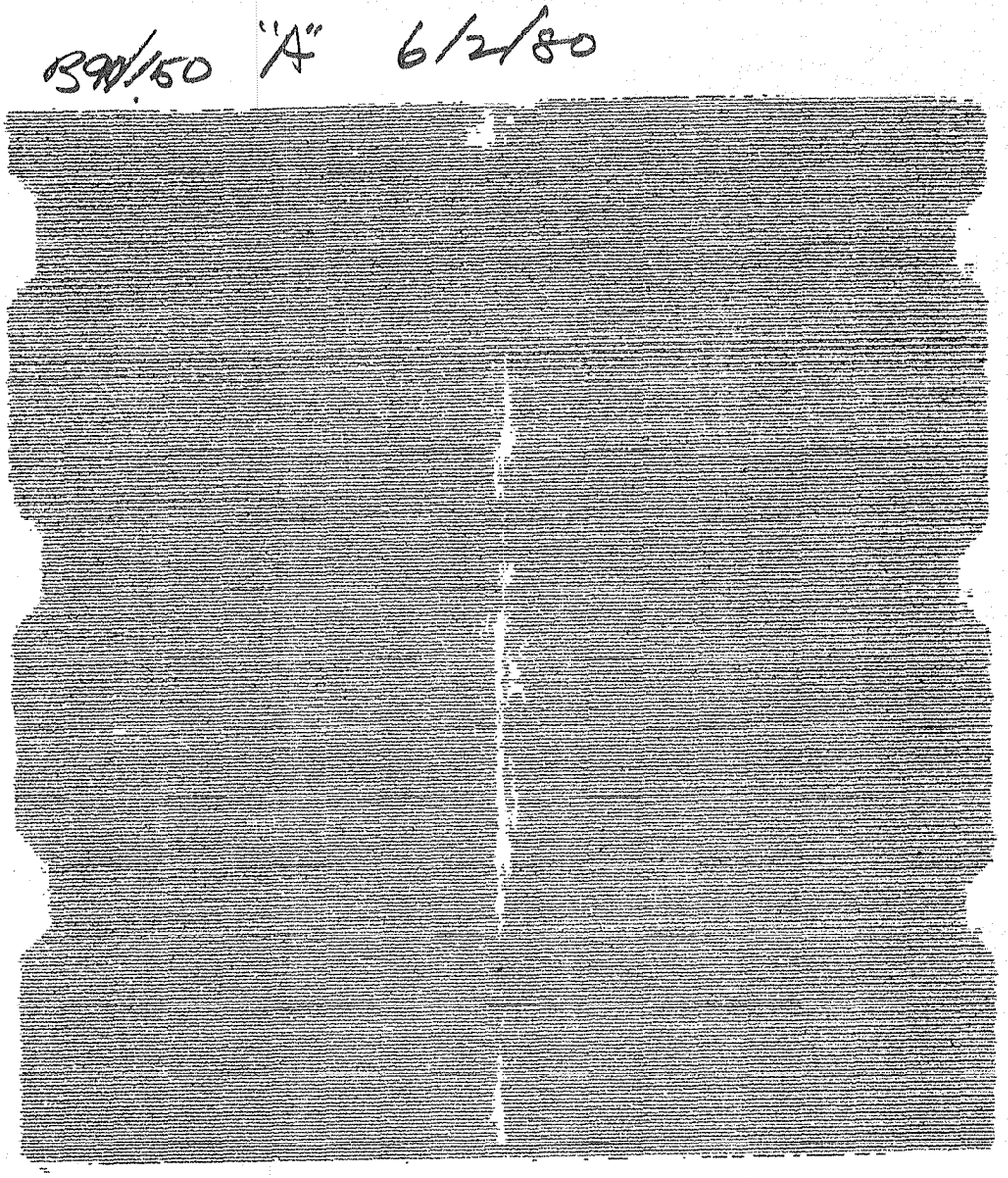


Figure 85. C-Scan of Laminate Imidized at 199 C, 150 Minutes

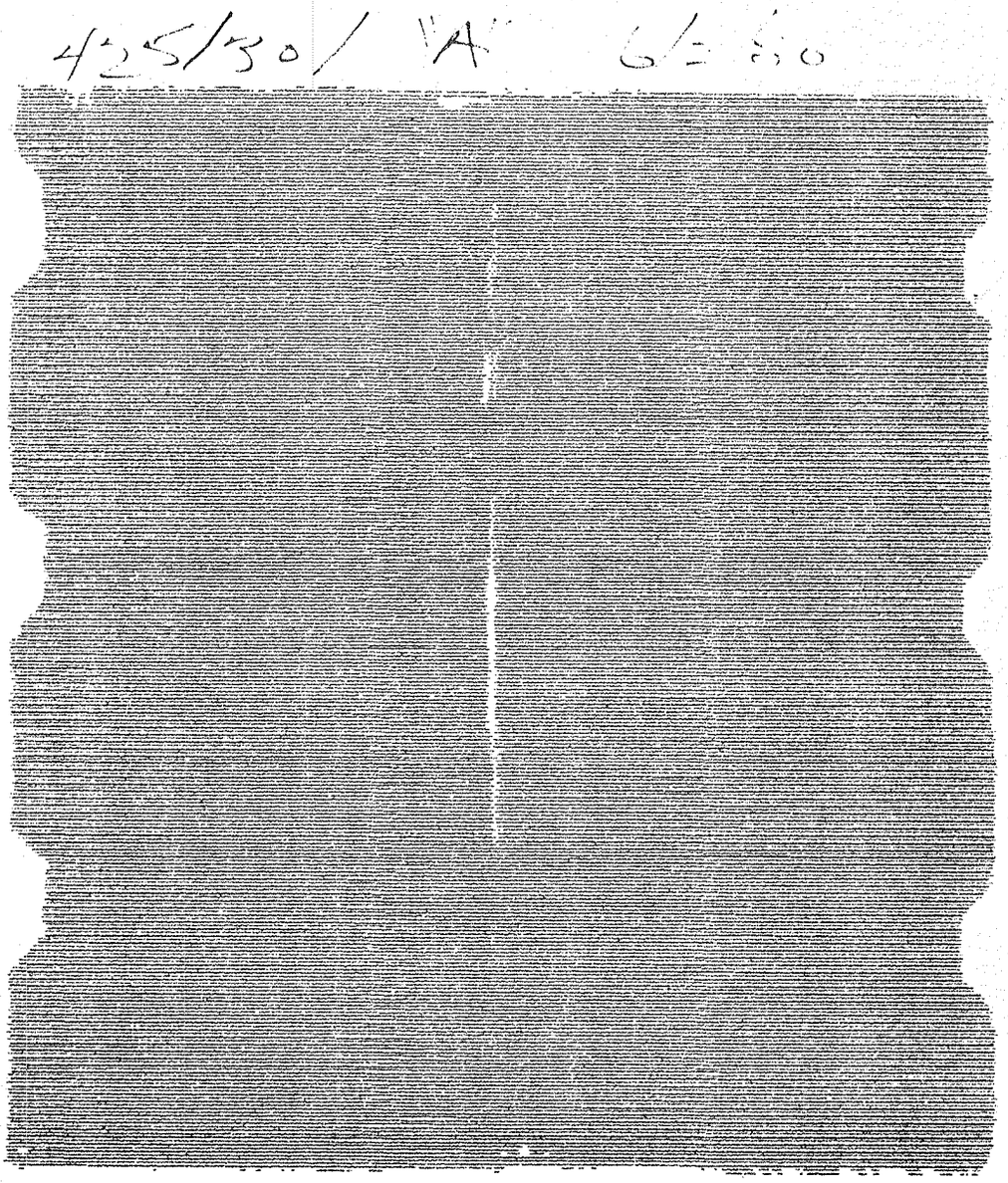


Figure 86. C-Scan of Laminate Imidized at 218 C, 30 Minutes

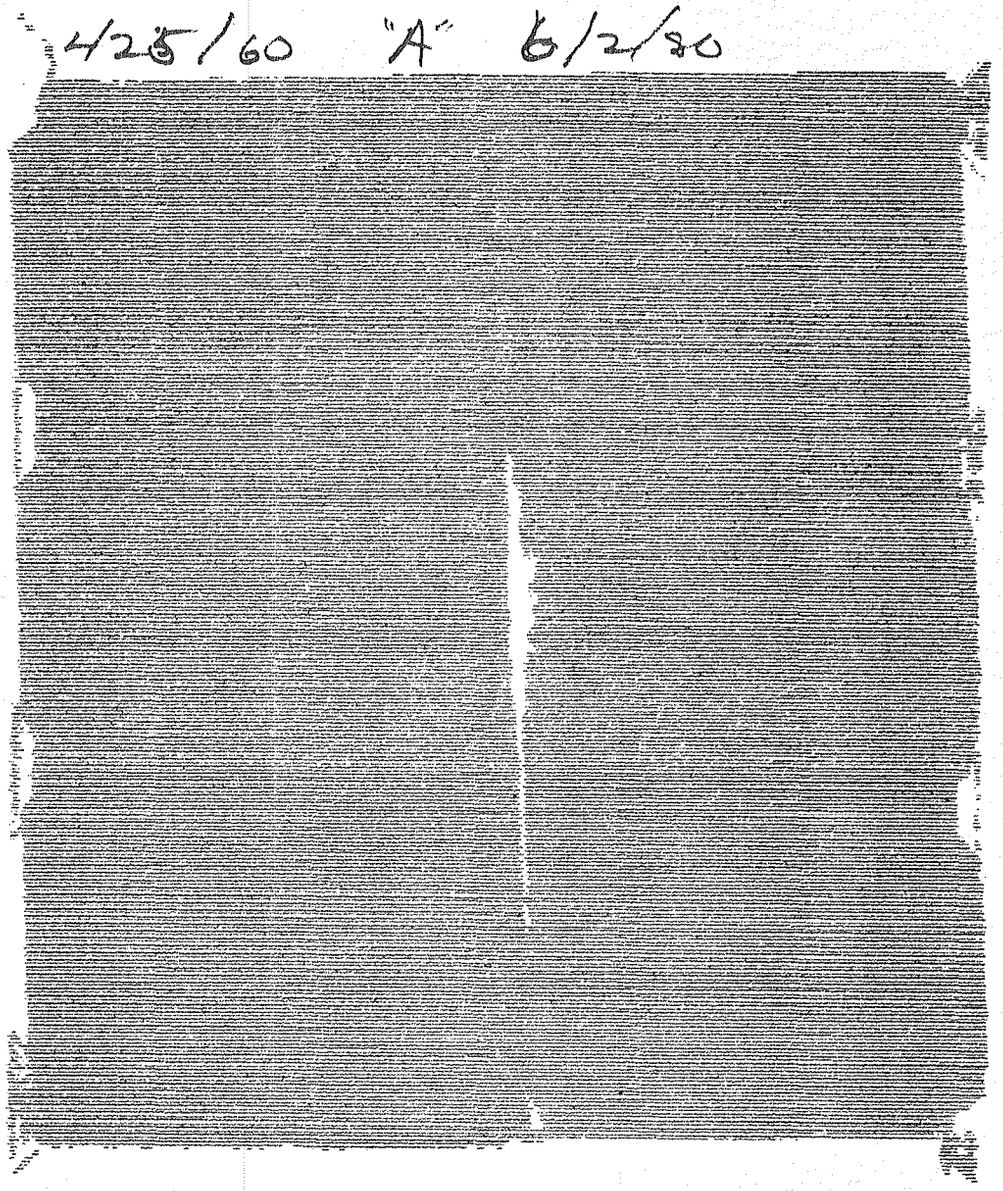


Figure 87. C-Scan of Laminate Imidized at 218 C, 60 Minutes

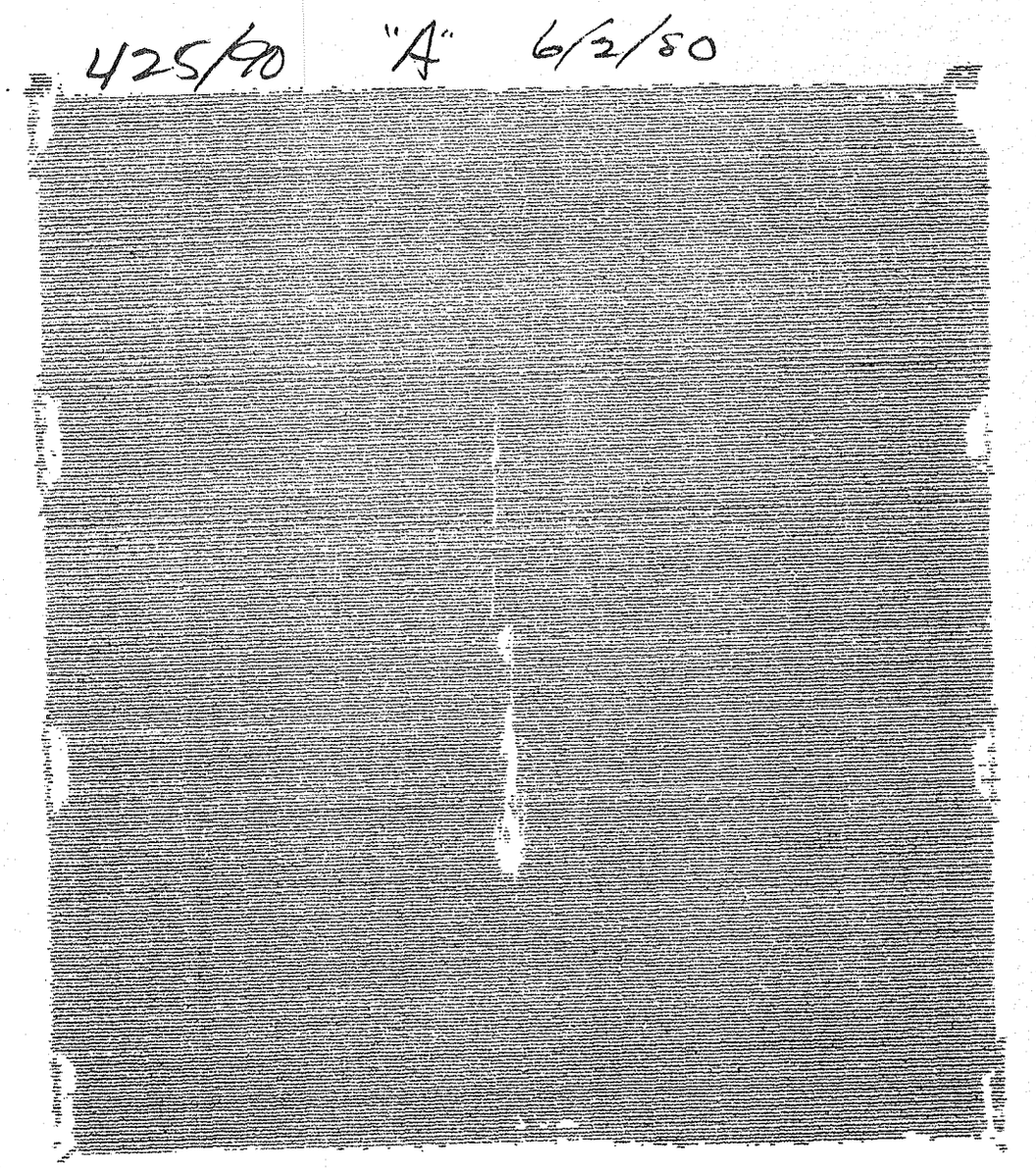


Figure 88. C-Scan of Laminate Imidized at 218 C, 90 Minutes

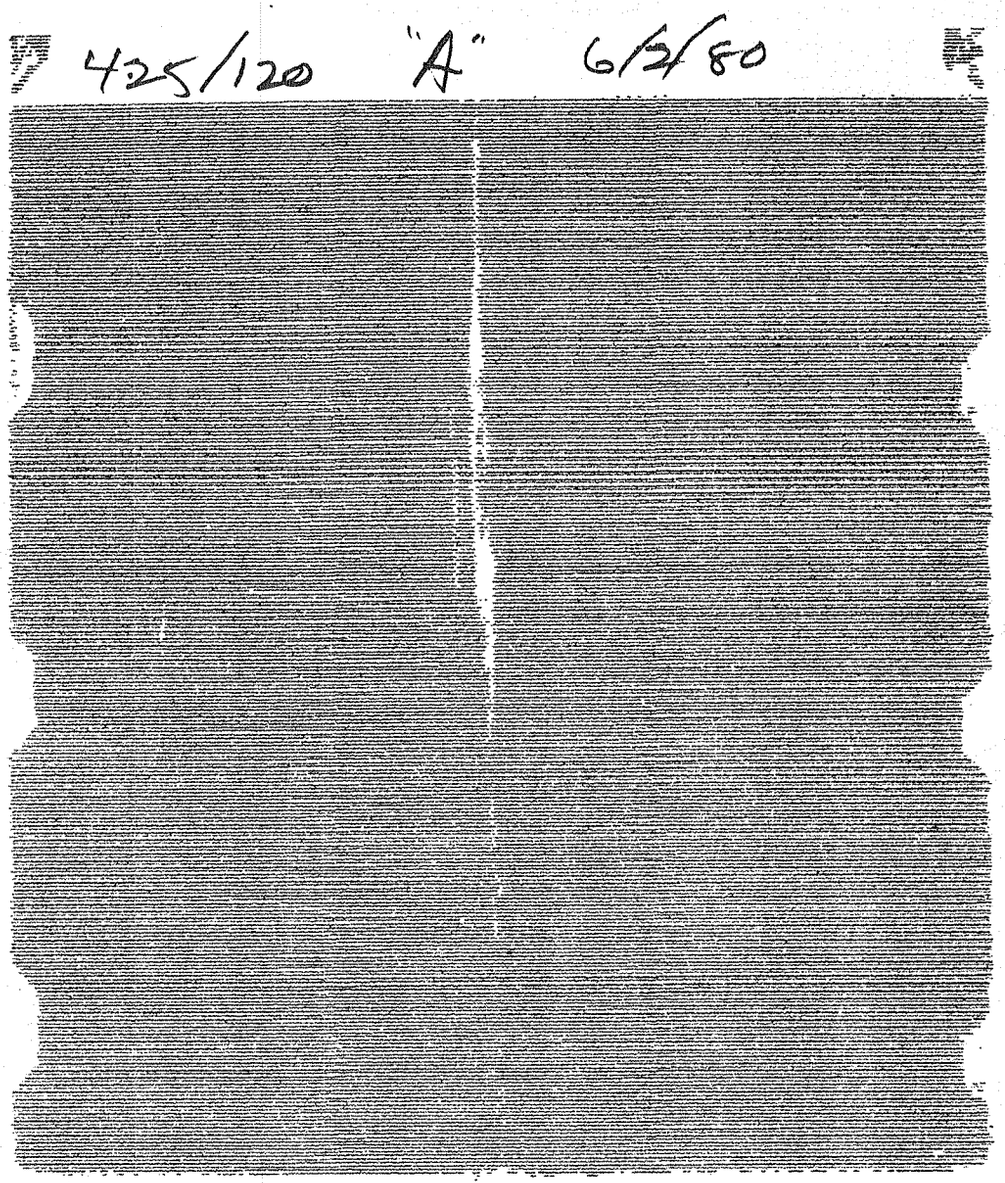


Figure 89. C-Scan of Laminate Imidized at 218 C, 120 Minutes

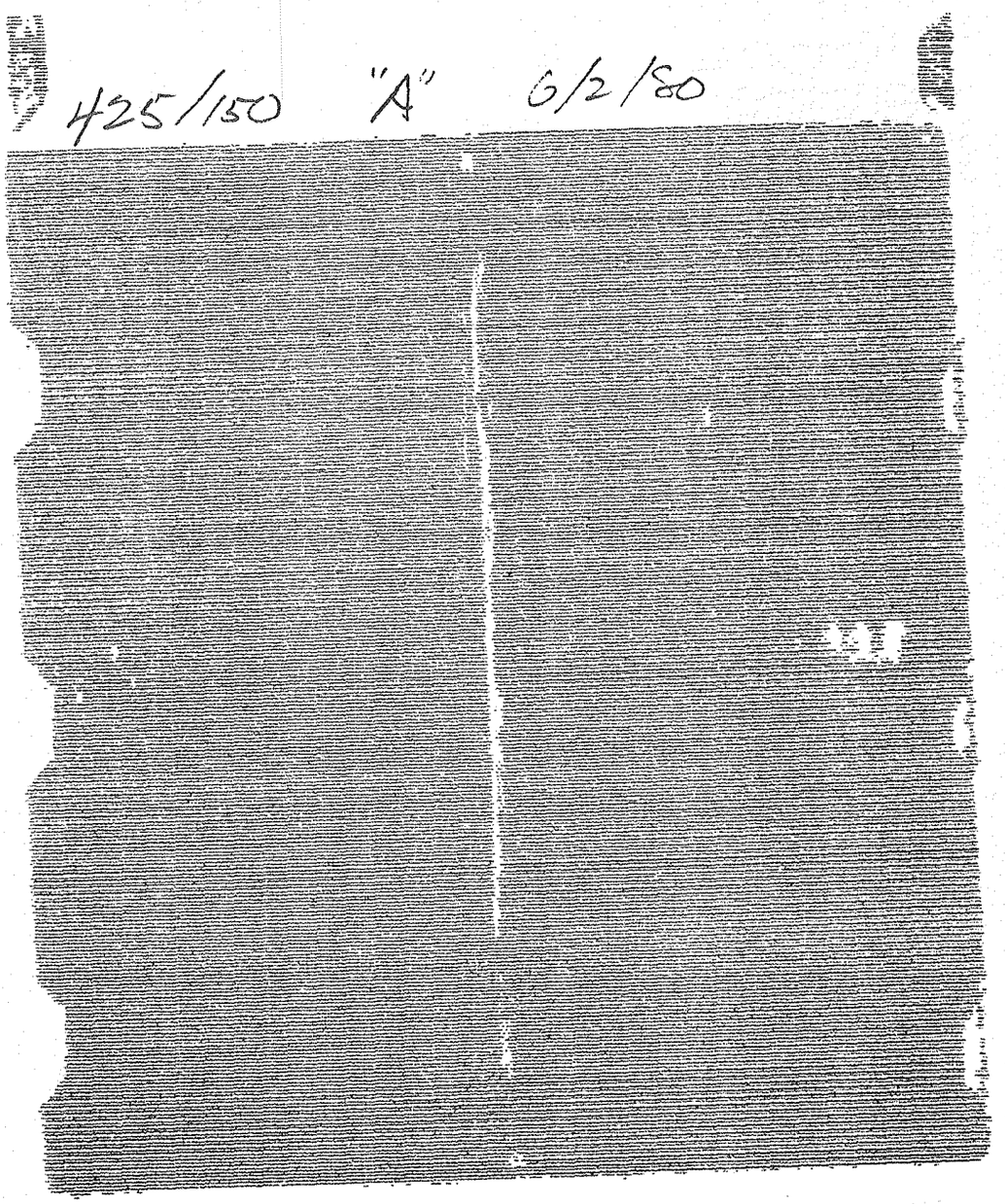


Figure 90. C-Scan of Laminate Imidized at 218 C, 150 Minutes

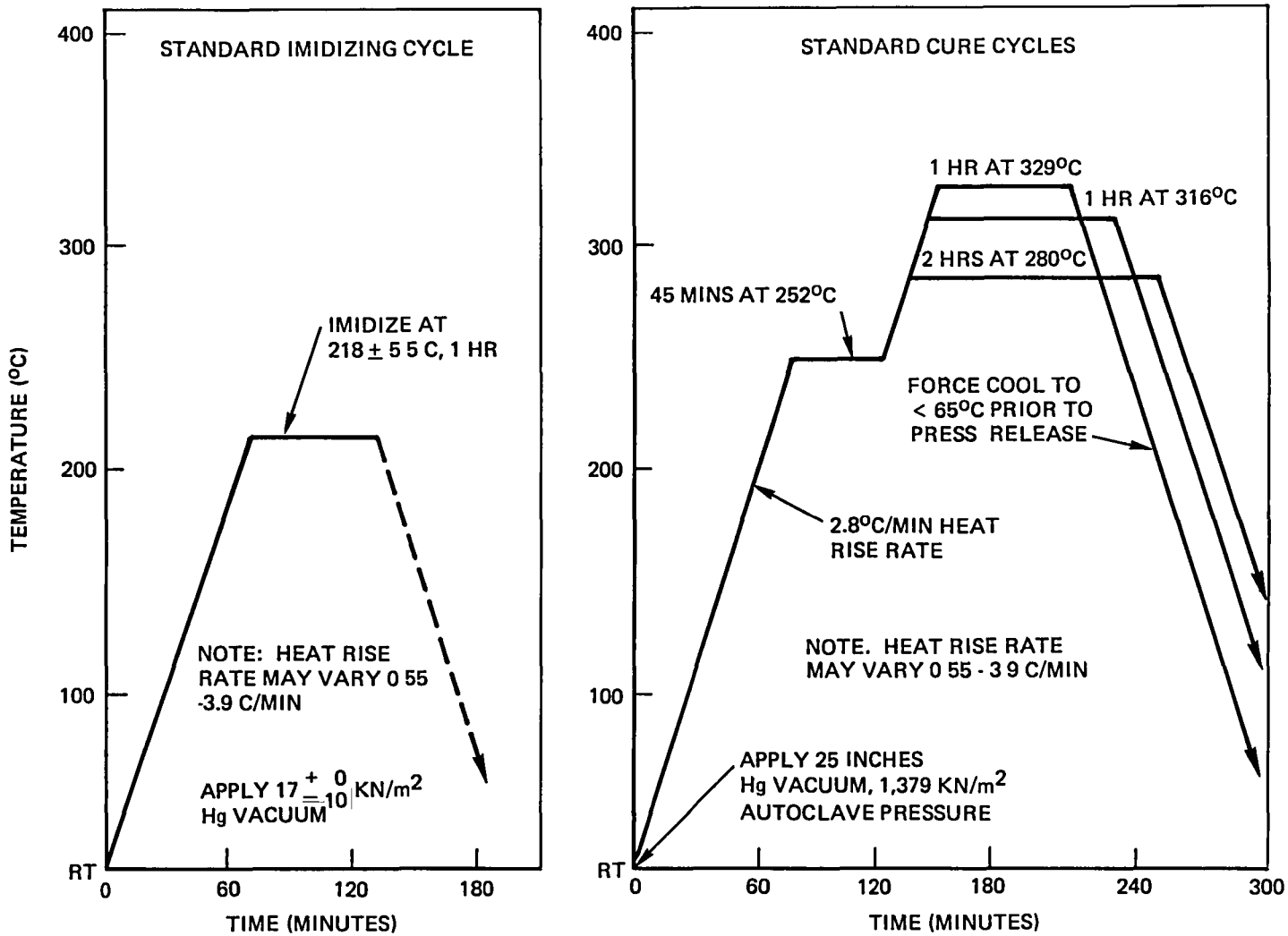


Figure 91. Improved Two Stage Processing Cycle for Celion/LARC 160

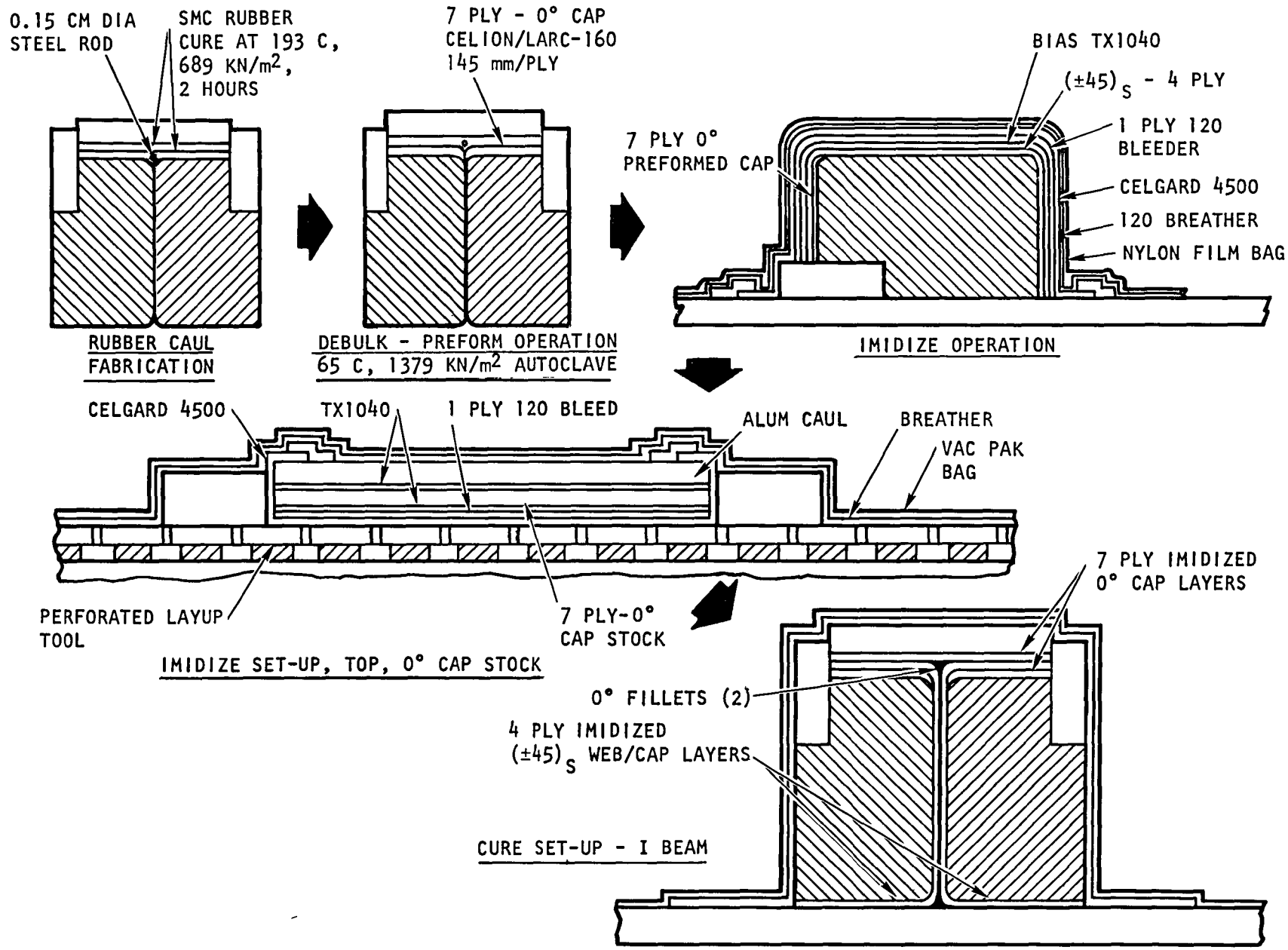


Figure 92. Layup Sequence LARC-160/Celion "I" Beam - Balanced Cap

MOLDING PRESSURE CRITERIA (1)(2)(3)

VACUUM PRESSURE LEVEL		PRESSURE TO AUGMENTER PLATE		AUGMENTED PRESSURE TO FILLET	
KN/m^2	INCHES HG	KN/m^2	PSI	KN/m^2	PSI
6.75	2	6.75	0.98	48	6.9
84.0	25	84.0	12.2	586	85
101	30	101	14.7	710	103

(1) PRESSURE AUGMENTER PLATE AREA RATIO TO MANDRELS = 7:1

(2) PROCESS PER 1.1 AND 1.2.

(3) Fillet mold is 96.5 CM (38.0 INCHES) LONG

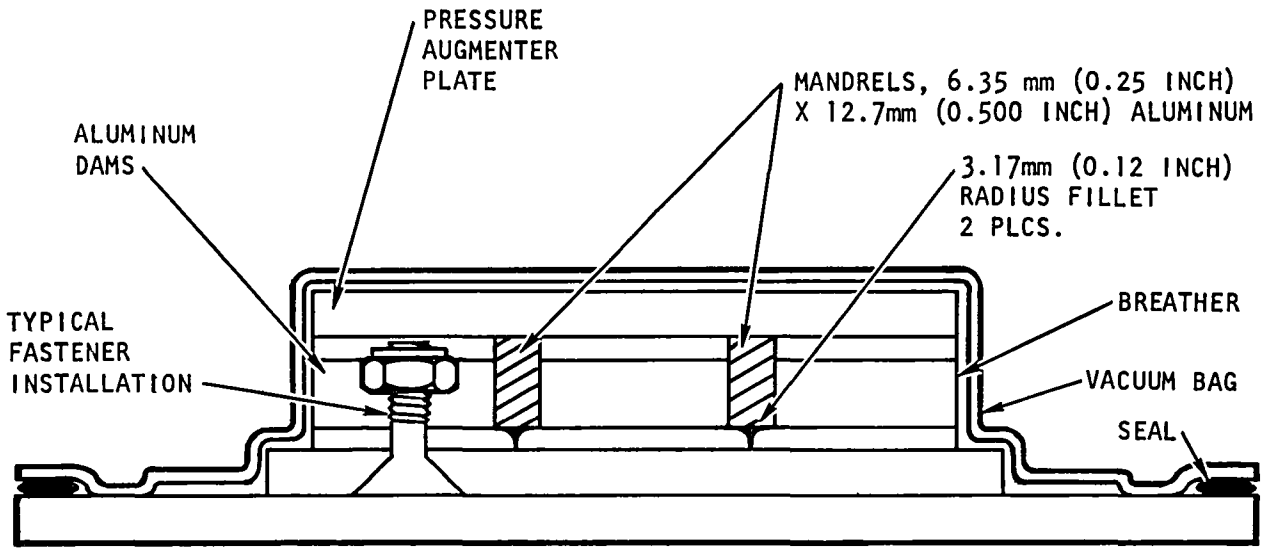


Figure 93. Fillet Radius Stock Tooling and Vacuum Bagging Arrangement - Imidizing Process

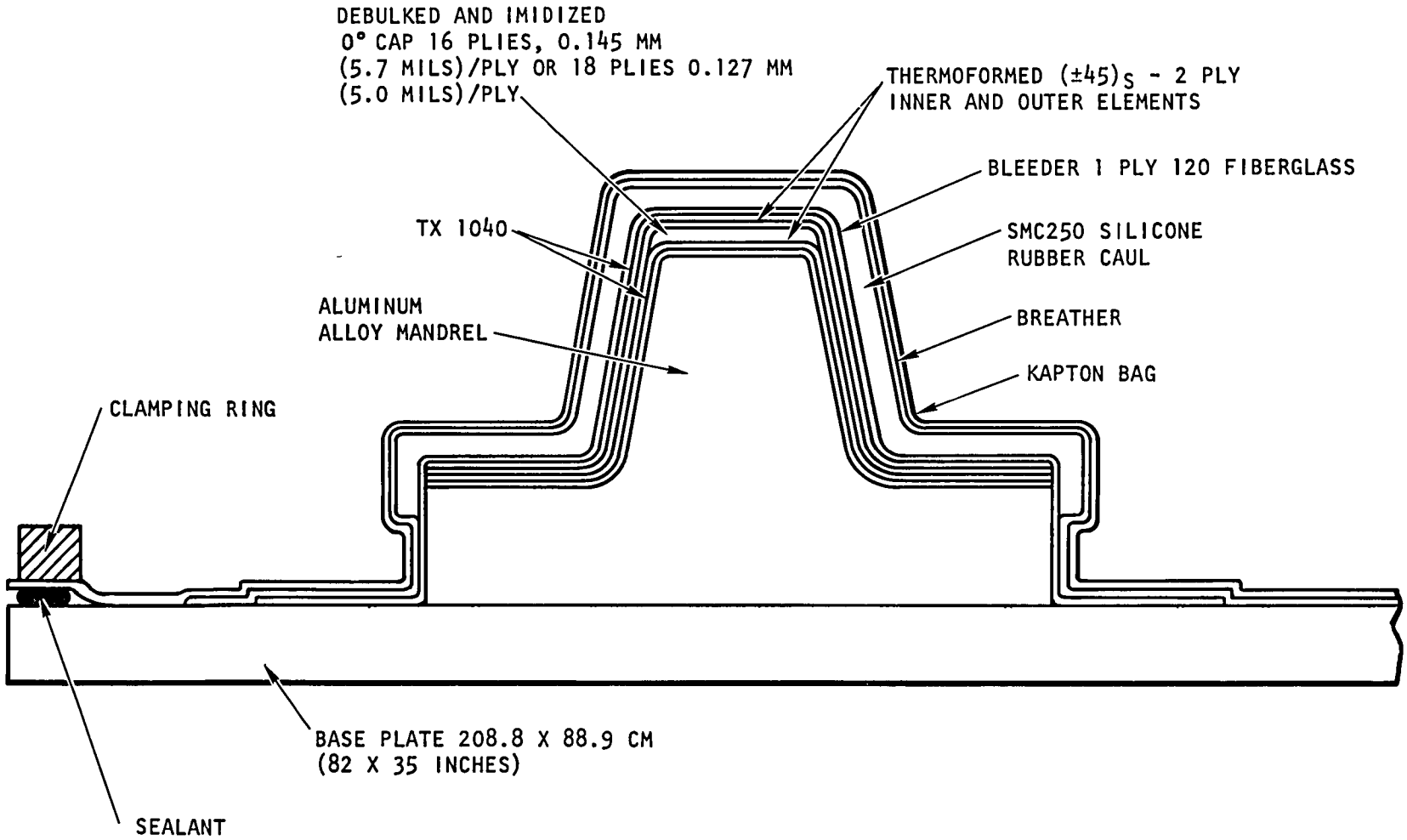


Figure 94. Layup and Tooling Process Hat-Stringer Assembly

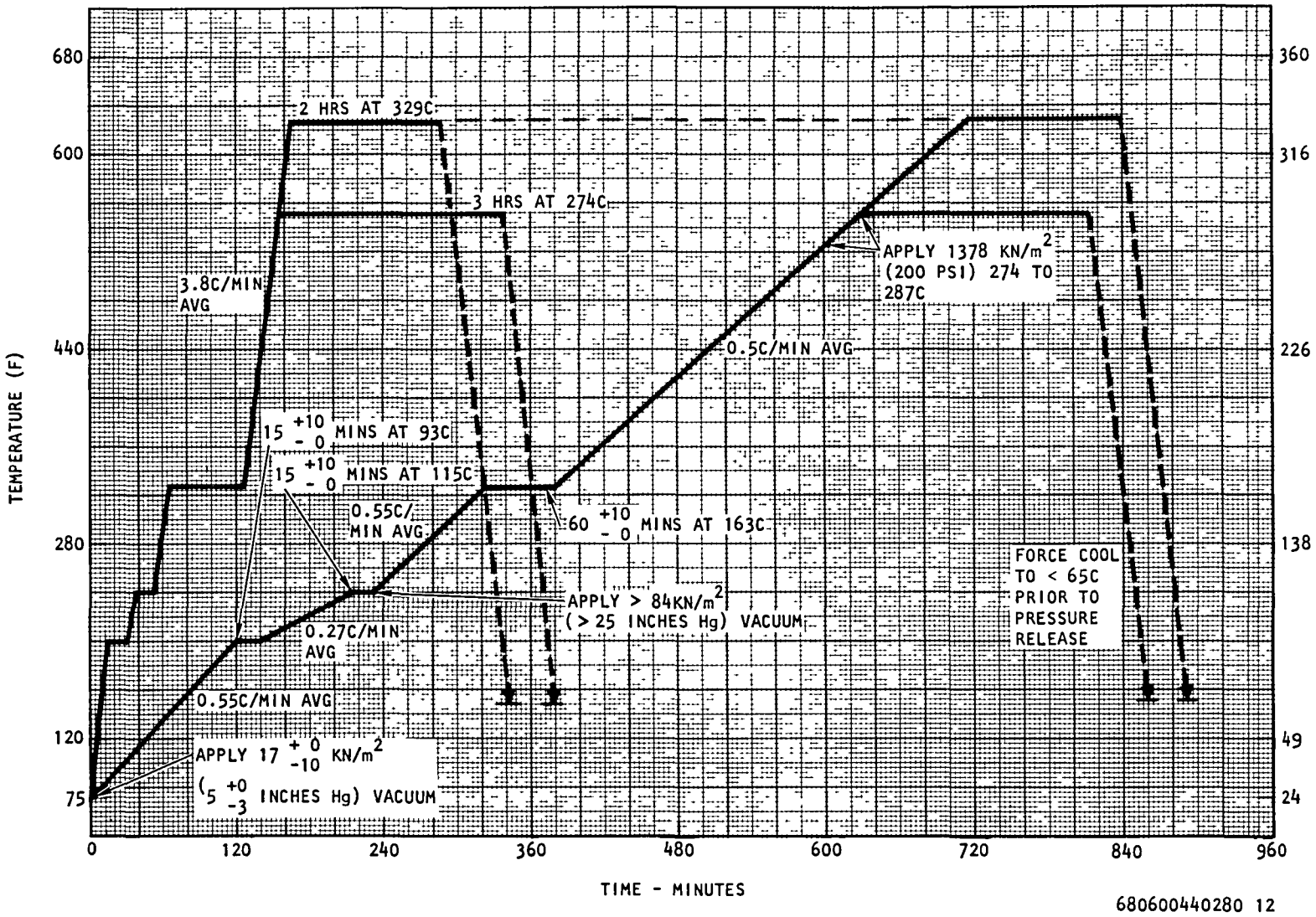


Figure 95. LARC-160/Celion In-Situ Imidizing Cure Cycle Window and Sequence of Events

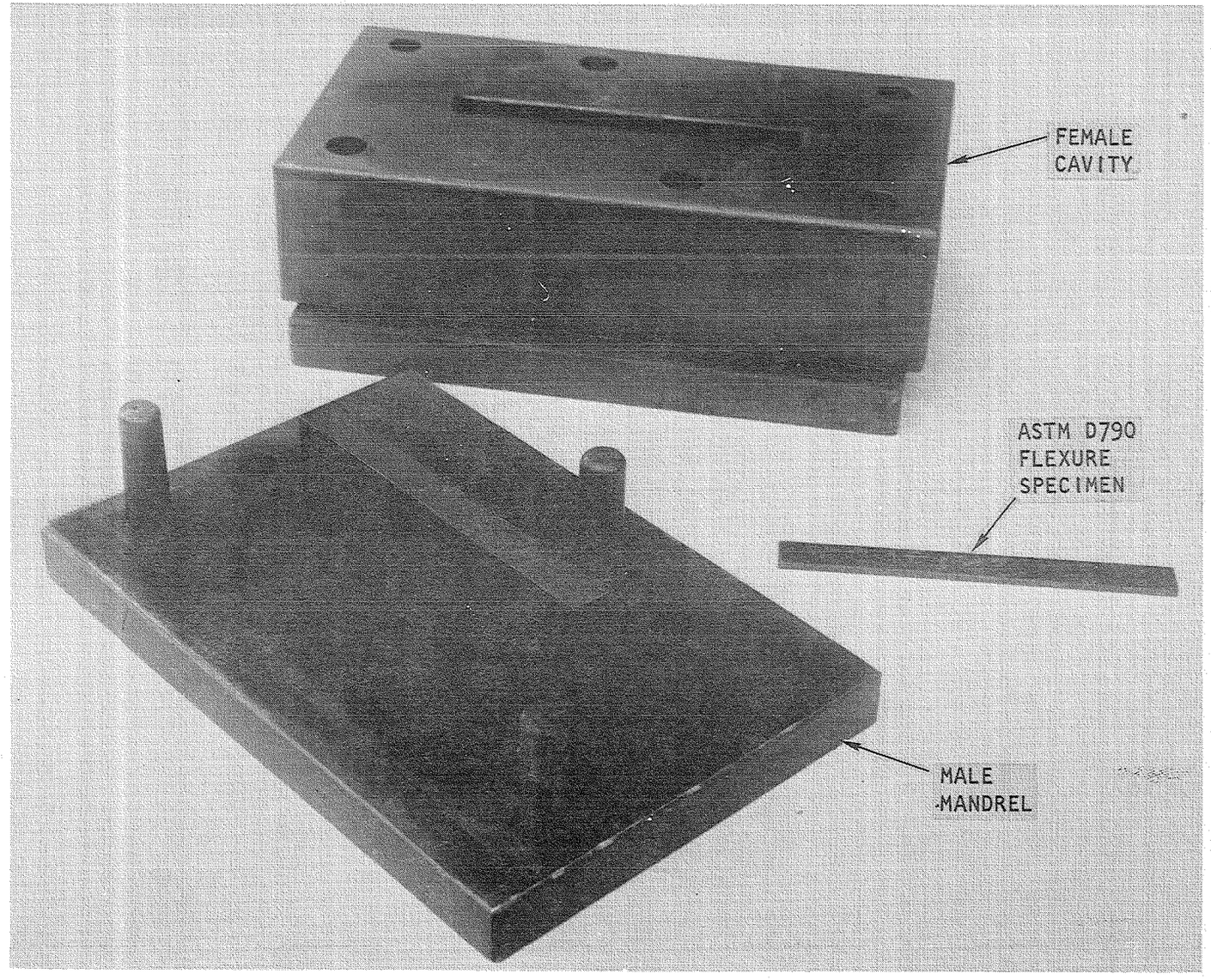


Figure 96.. ASTM D 647 Compression Mold for Celion/LARC 160 Molding Compound Flexure Specimen per ASTM D 790

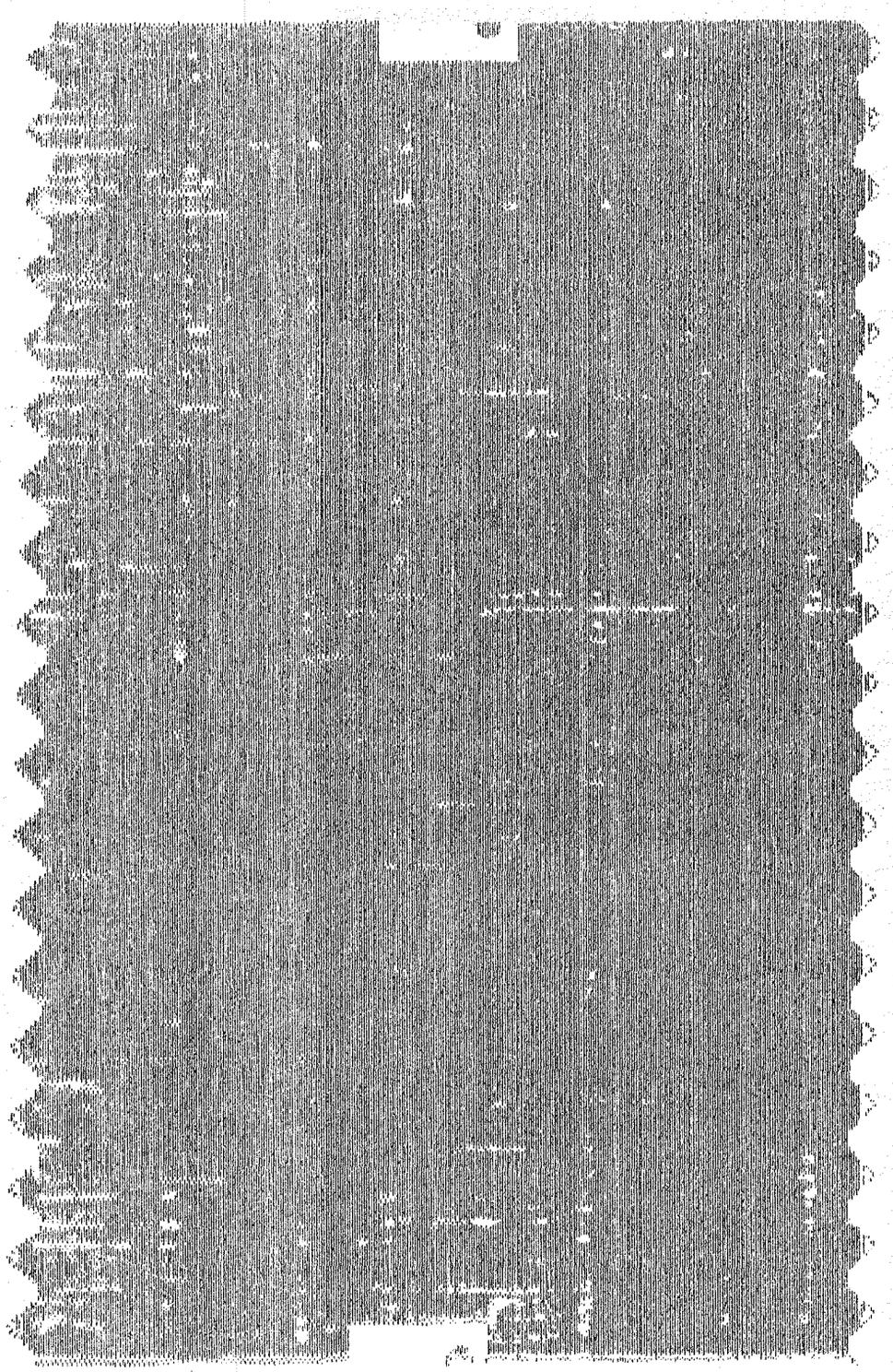


Figure 97. C-Scan Laminate EX 199 $(0)_s$ for Aged Tensile Properties

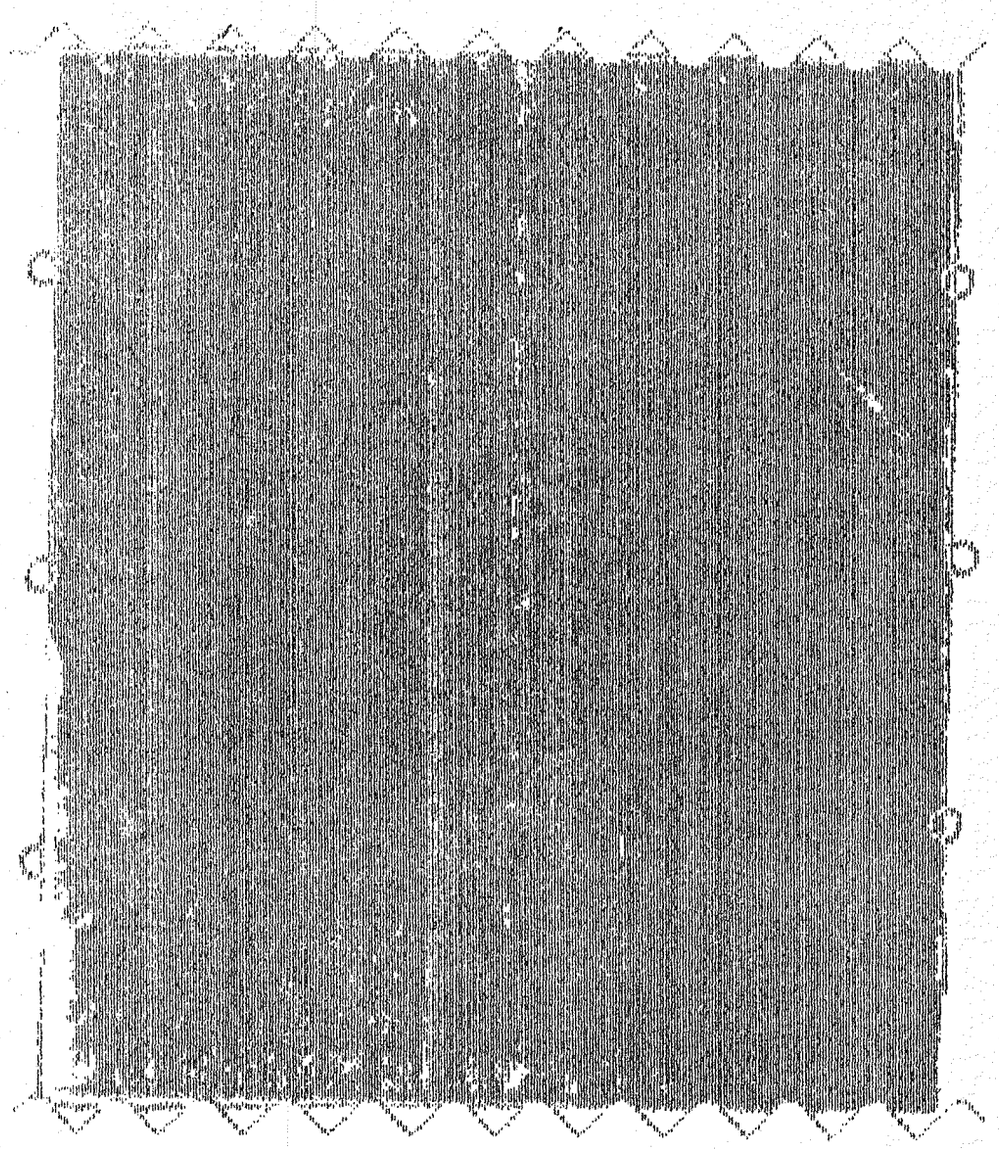


Figure 98. C-Scan Laminate EX 200 $(0,\pm45,90)_s$ for Aged Tensile Properties

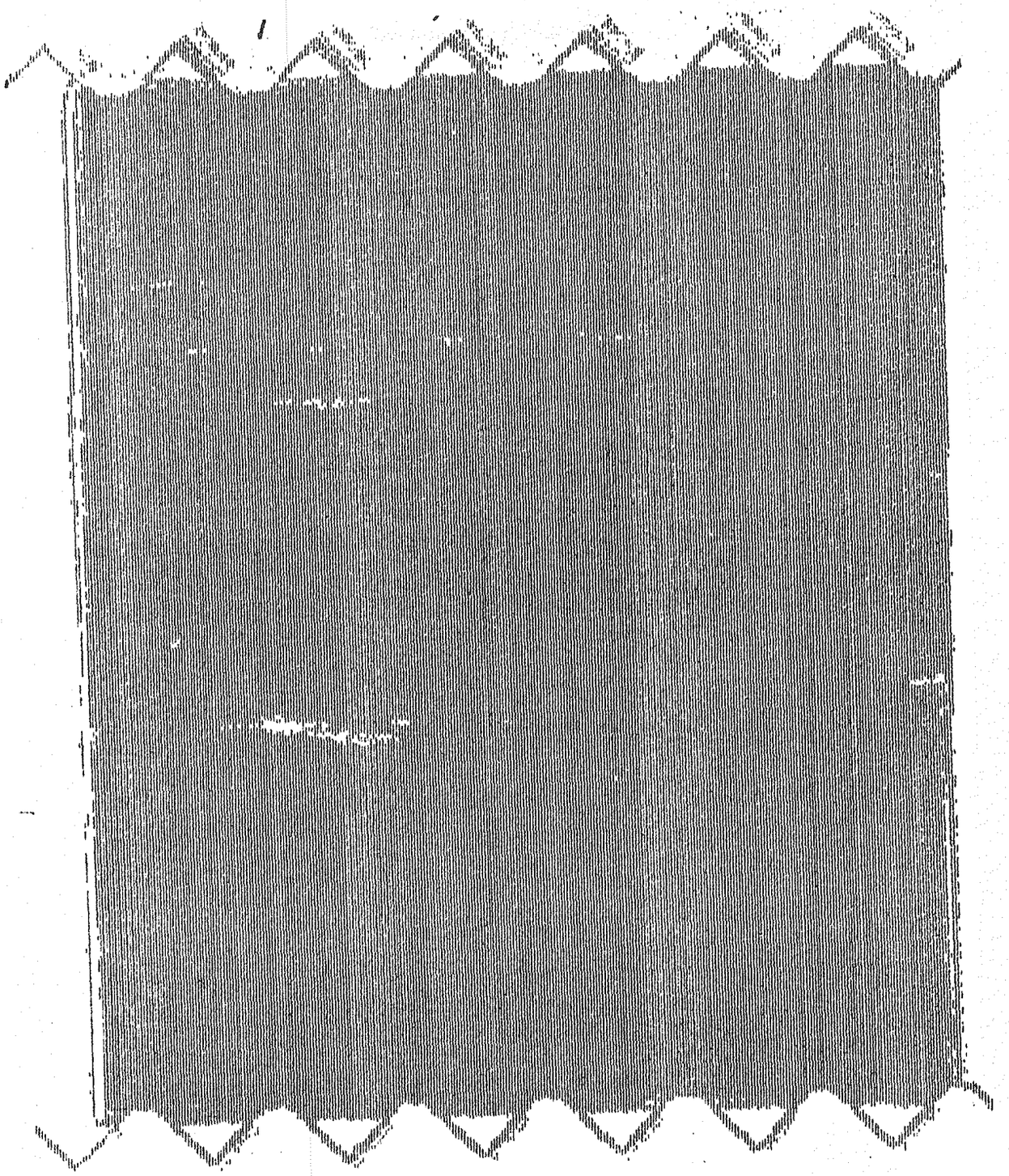


Figure 99. C-Scan Laminate EX 201 $(90)_{40}$ for Aged Tensile and Compressive Properties

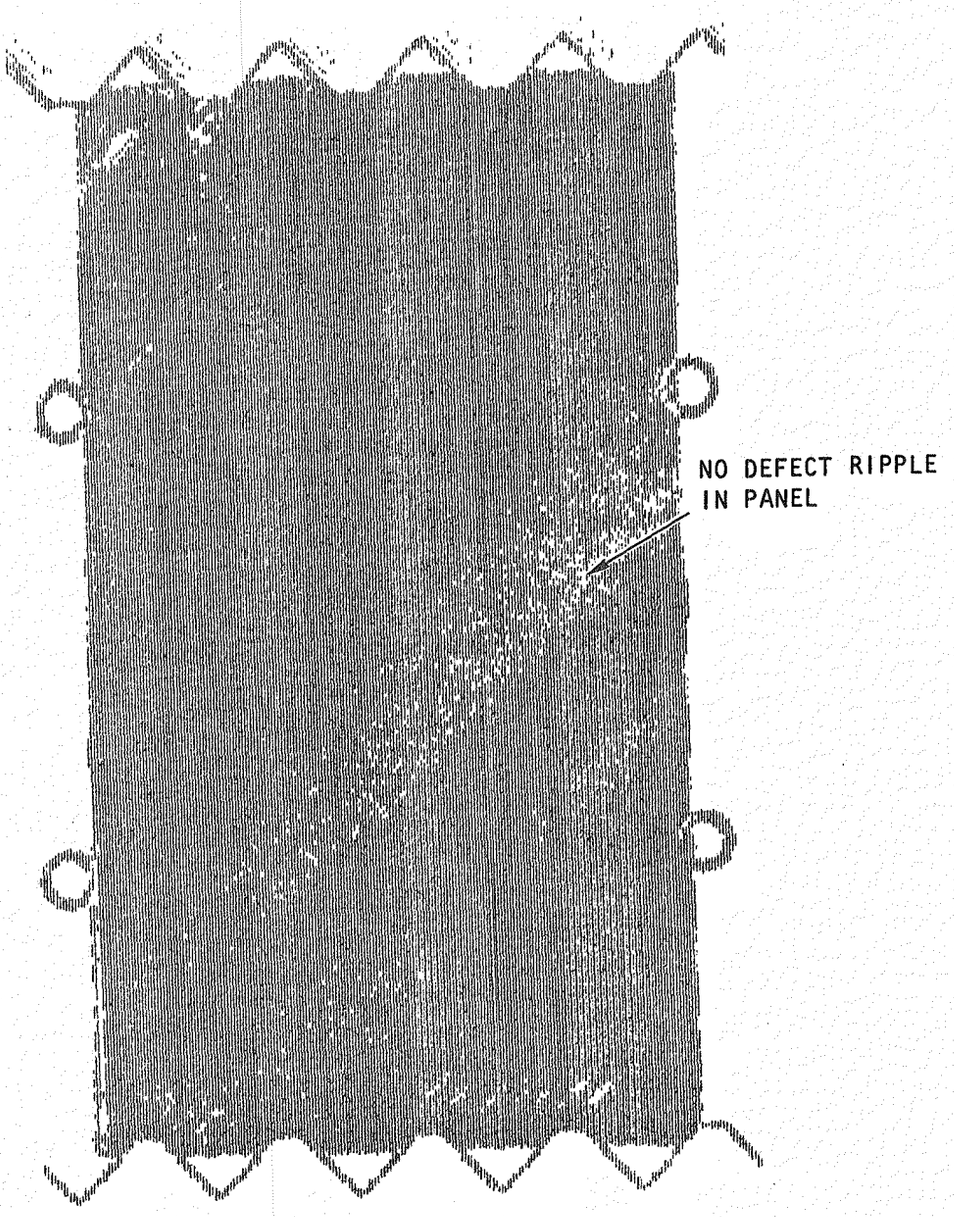


Figure 100. C-Scan Laminate EX202 $(\pm 45)_s$ for Aged Tensile Properties

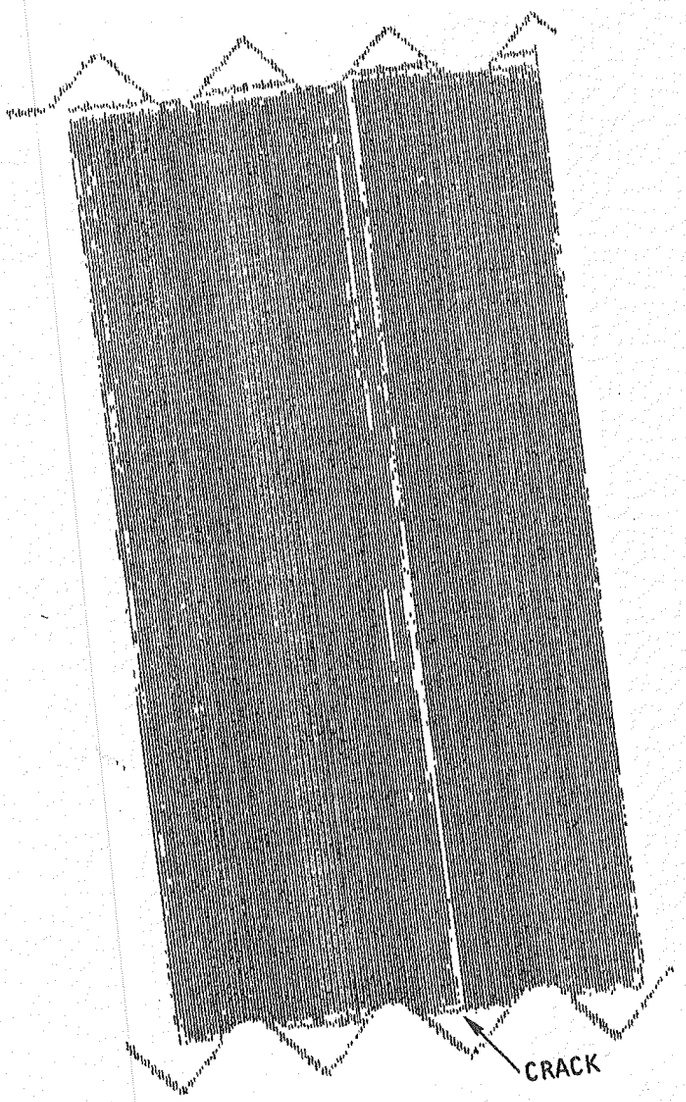


Figure 101. C-Scan Laminate EX 204 $(0)_{26}$ for Aged Short Beam Shear and Flexural Properties

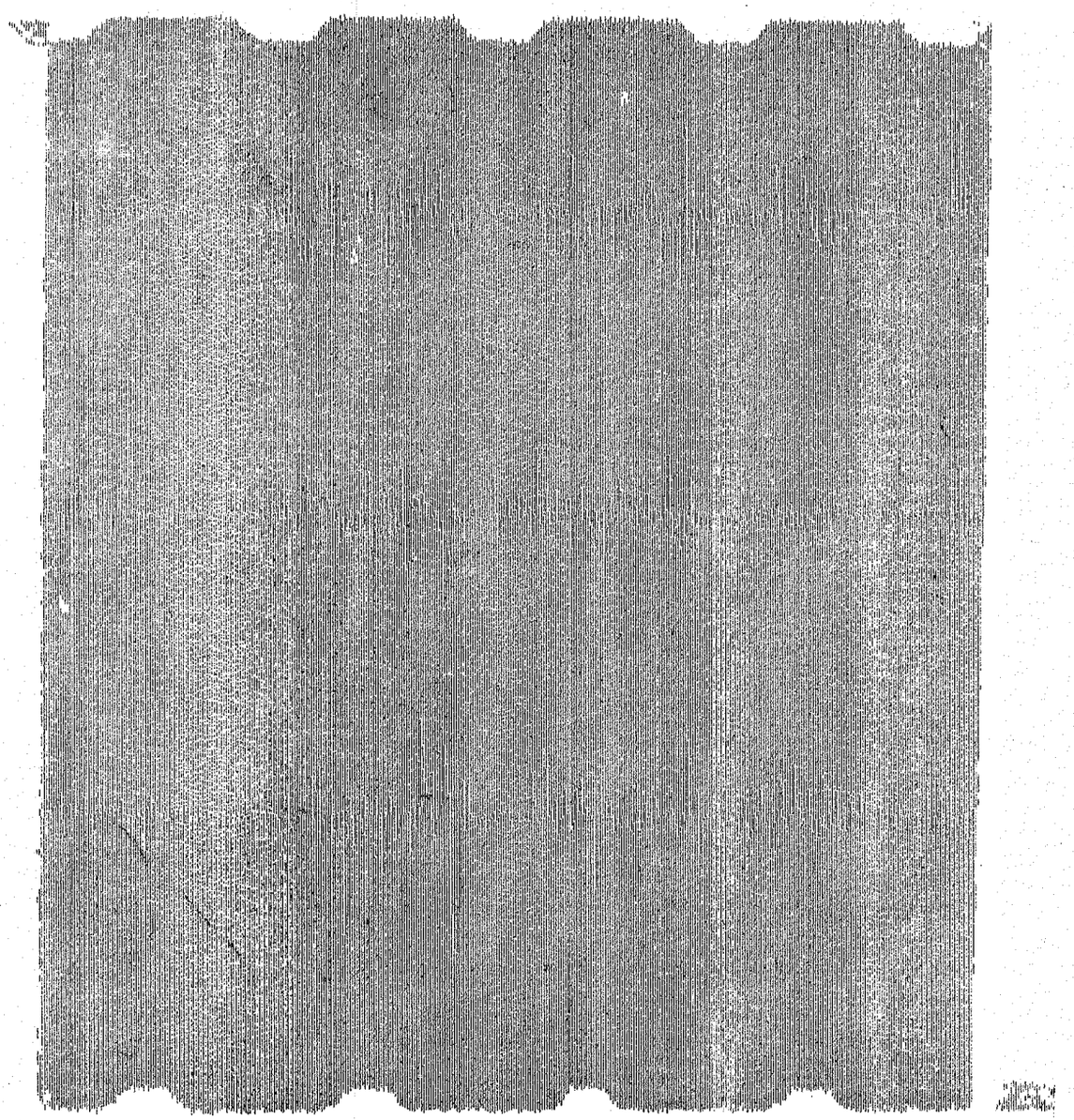


Figure 102. C-Scan Laminate EX 220 $(\pm 45)_s$ 32 Ply for Aged Compressive Properties

A801002 G-8

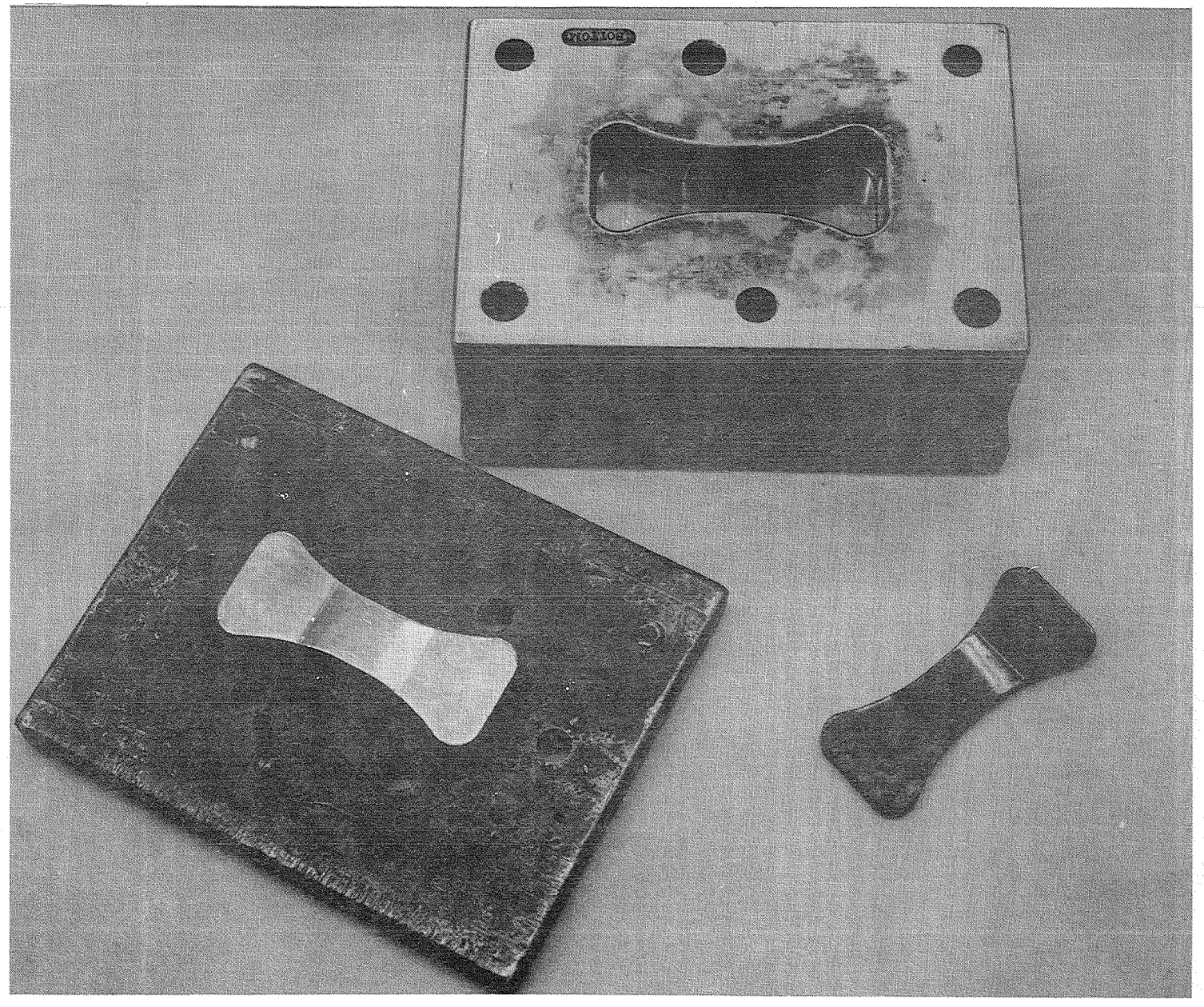
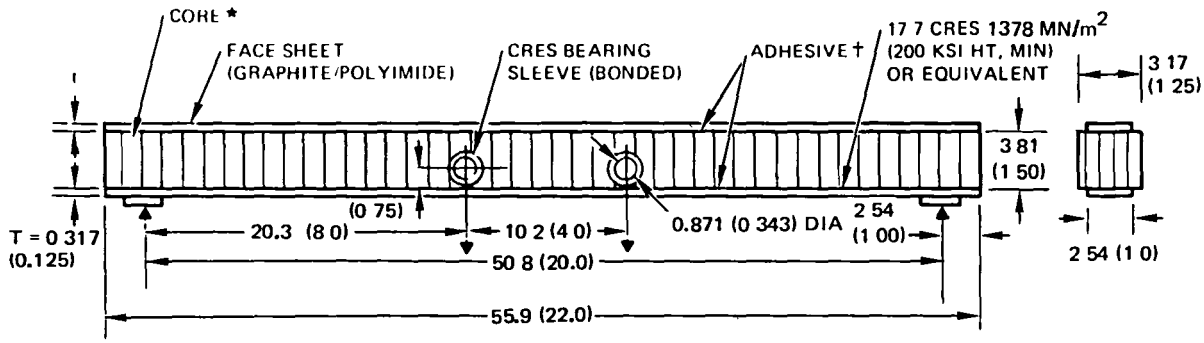


Figure 103. ASTM D647 Tensile Mold and Molded ASTM D651 Tension Coupon

FLEX SANDWICH BEAM TEST – ULTIMATE COMPRESSION STRESS ON FACE SHEET



CALCULATION OF ULTIMATE COMPRESSIVE STRESS (F_{cu}) ON COMPOSITE FACE SHEET

$$F_{cu} = \frac{4P}{W \times t\left(1.5 + \frac{T+t}{2}\right)}$$

WHERE

P = ULTIMATE FAILING LOAD

W = BEAM WIDTH

t = COMPRESSION FACE SHEET THICKNESS

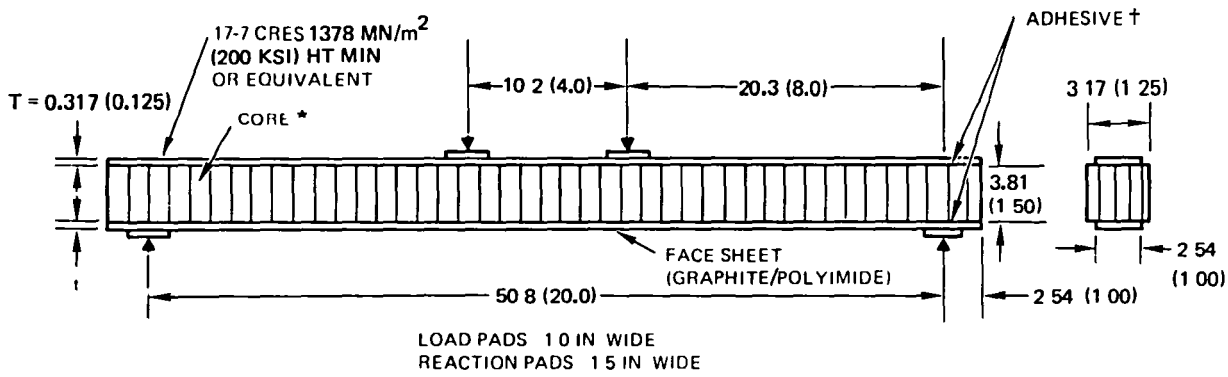
T = TENSION FACE SHEET THICKNESS

* FOR –270 F, 75 F & 400 F TESTS USE ALUMINUM HONEYCOMB CORE, 1/8-22 PCF, 5052 ALLOY

FOR 600 F TESTS, USE 321 CRES, ANN , 0 005 IN FOIL PER AMS 5510H

† FOR –270 F, 75 F & 400 F TESTS, USE 0 12 PSF FM 400 ADHESIVE FOR 600 F TESTS, USE 0 135 PSF ADHESIVE (FM34)

FLEX SANDWICH BEAM TEST – ULTIMATE TENSILE STRESS ON FACE SHEET



CALCULATION OF ULTIMATE TENSILE STRESS (F_{tu}) ON COMPOSITE FACE SHEET

$$F_{tu} = \frac{4P}{W \times t\left(1.5 + \frac{T+t}{2}\right)}$$

WHERE

P = ULTIMATE FAILING LOAD

W = BEAM WIDTH

t = TENSILE FACE SHEET THICKNESS

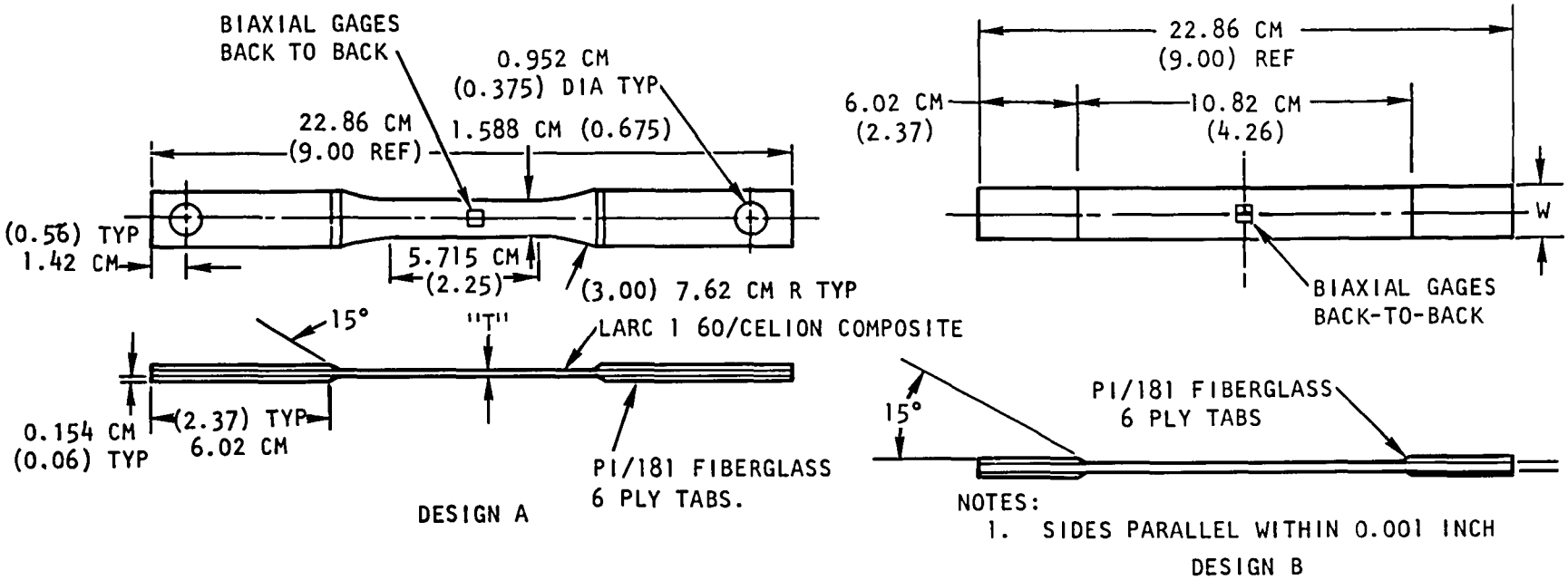
T = COMPRESSION FACE SHEET THICKNESS

* FOR –270 F, 75 F & 400 F TESTS USE ALUMINUM HONEYCOMB CORE, 1/8-22 PCF, 5052 ALLOY

FOR 600 F TESTS, USE 321 CRES, ANN , 0 005 IN FOIL PER AMS 5510 H

† FOR –270 F, 75 F & 400 F TESTS, USE 0 12 PSF FM 400 ADHESIVE FOR 600 F TESTS, USE 0 135 PSF ADHESIVE (FM34)

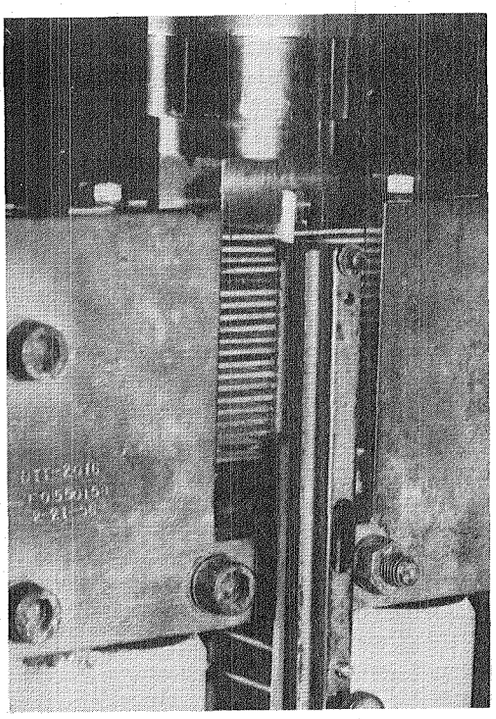
Figure 104. Composite Tension and Compression Critical Beam Specimen Designs



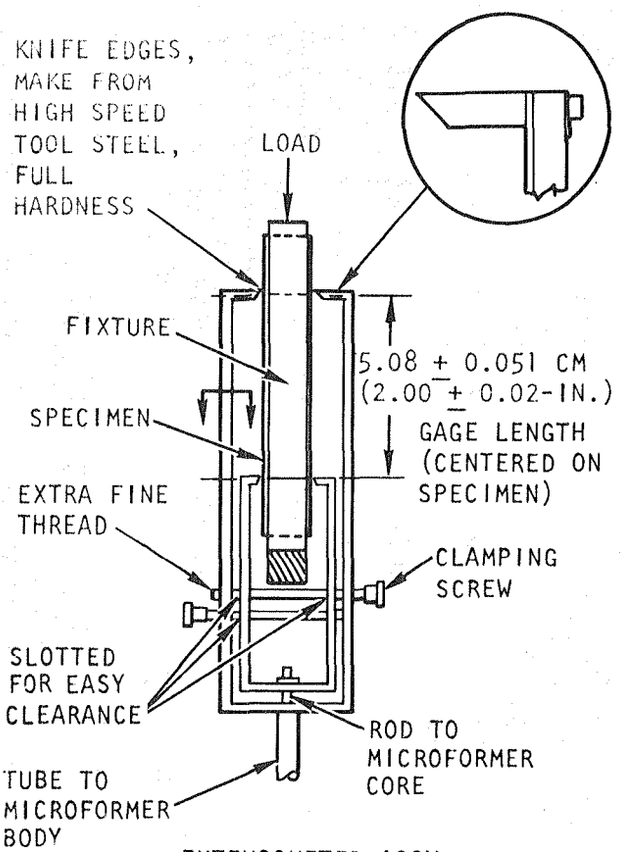
LEGEND

LAMINATE ORIENTATION	SPECIMEN DESIGN	NO. PLIES	NOMINAL THICKNESS "T" CM/ (MILS)	GAUGE SECTION WIDTH CM (INCH)	TAB ADHESIVE TEST TEMP (C)			
					-132	RT	204	316
$(0)_t$	B	5	0.032/(12.5)	1.27 (0.500)	FM400	FM400	FM400	FM34
$(90)_t$	B	32	0.20/(80.0)	2.54 (1.000)	FM400	FM400	FM400	FM34
$(\pm 45)_S$	B	4	0.0254/(10.0)	2.54 (1.000)	FM400	FM400	FM400	FM34
$(0,\pm 45,90)_S$	A	8	0.051/(20.0)	1.588 (0.625)	FM400	FM400	FM400	FM34

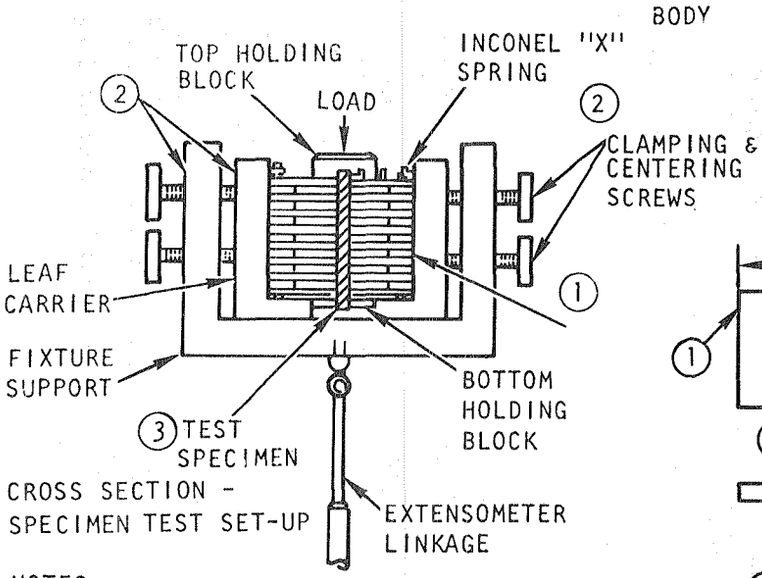
Figure 105. Tension Coupon Specimen Designs



SPECIMEN IN TEST FIXTURE



EXTENSOMETER ASSY



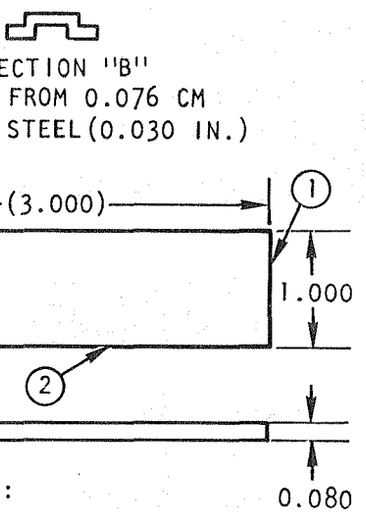
CROSS SECTION - SPECIMEN TEST SET-UP

NOTES:

(1) MAKE FROM 0.0012 CM (0.060 MILS) CRES STEEL. STACK GRIND TO IDENTICAL LENGTH & BREAK EDGES. LEAVES OF SPACERS TO HAVE LARGE CLEARANCE HOLES TO ALLOW FREE MOVEMENT.

(2) MAKE FROM CRES STEEL

(3) DISTANCE UNSUPPORTED BY STABILIZING LEAVES AT EACH END OF SPECIMEN SHALL NOT BE MORE THAN 0.152 CM (60 MILS)



NOTES:

(1) ENDS TO BE GROUND PARALLEL WITHIN 0.00127 CM (0.0005 IN.) & PERPENDICULAR TO LONG AXIS WITHIN 0.00127 CM/CM 0.0005 IN./IN.

(2) SIDES TO BE PARALLEL WITHIN 0.001 IN.

(3) FACES TO BE MOLDED FLAT WITHIN 0.002 IN.

SPECIMEN DESIGN

Figure 106. Compression Coupon Design and Test Set-Up

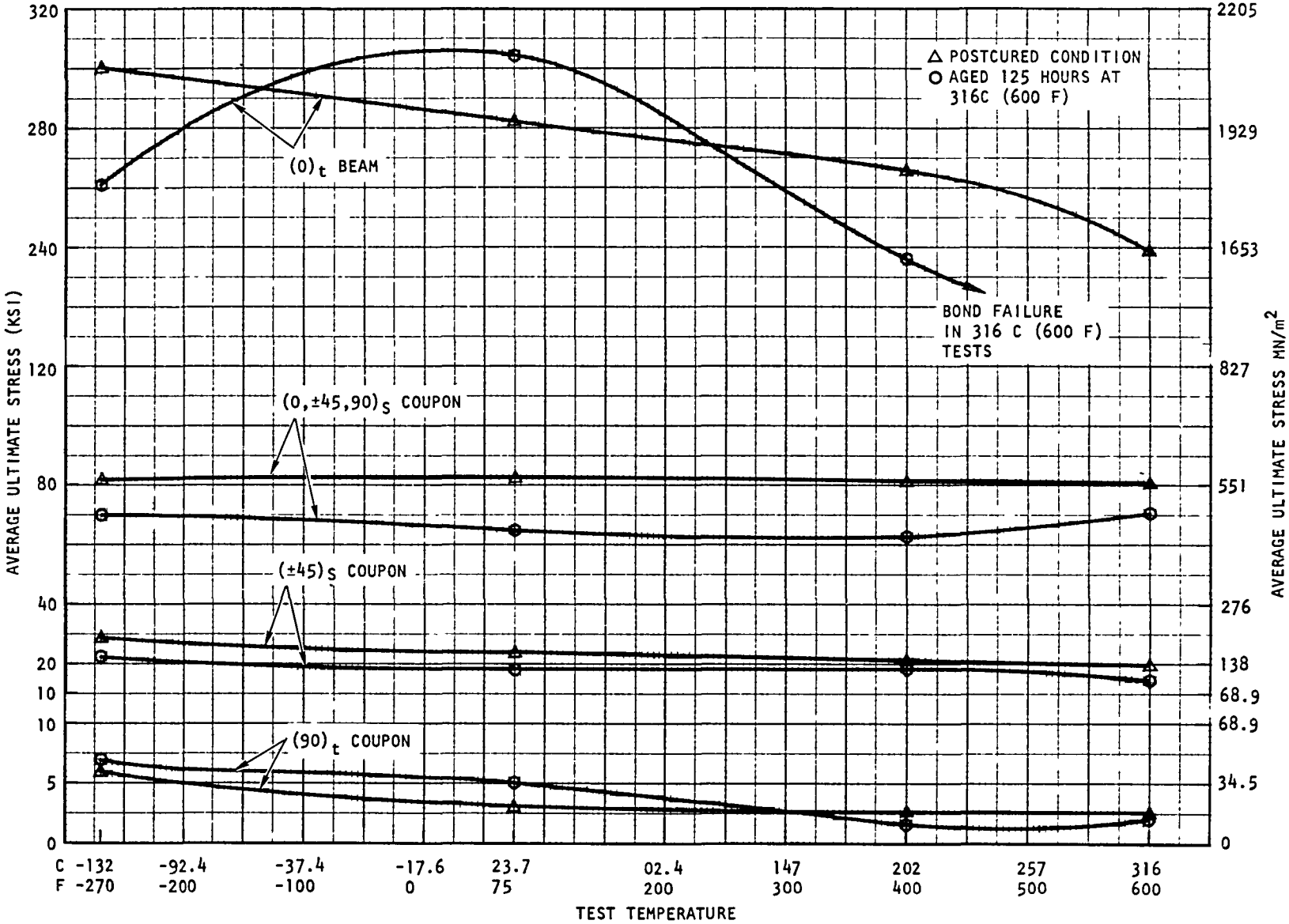


Figure 107. Tensile Properties of LARC-160/Celion Laminates Postcured and 125 Hours ⌐316 C (600 F) Aged Conditions

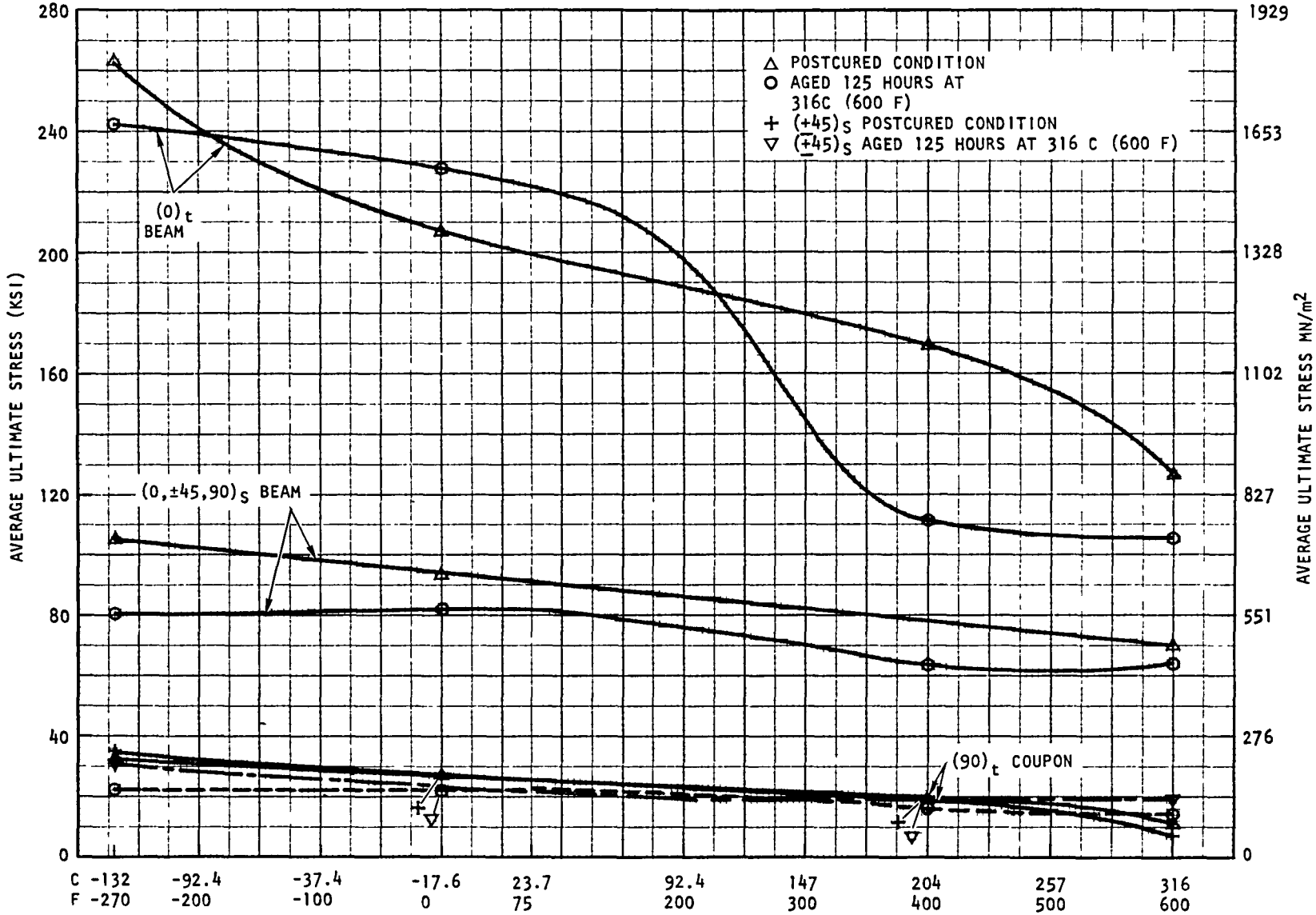


Figure 108. Compression Properties of LARC-160/Celion Laminates Postcured and 125 Hours -316 C (600 F) Aged Conditions

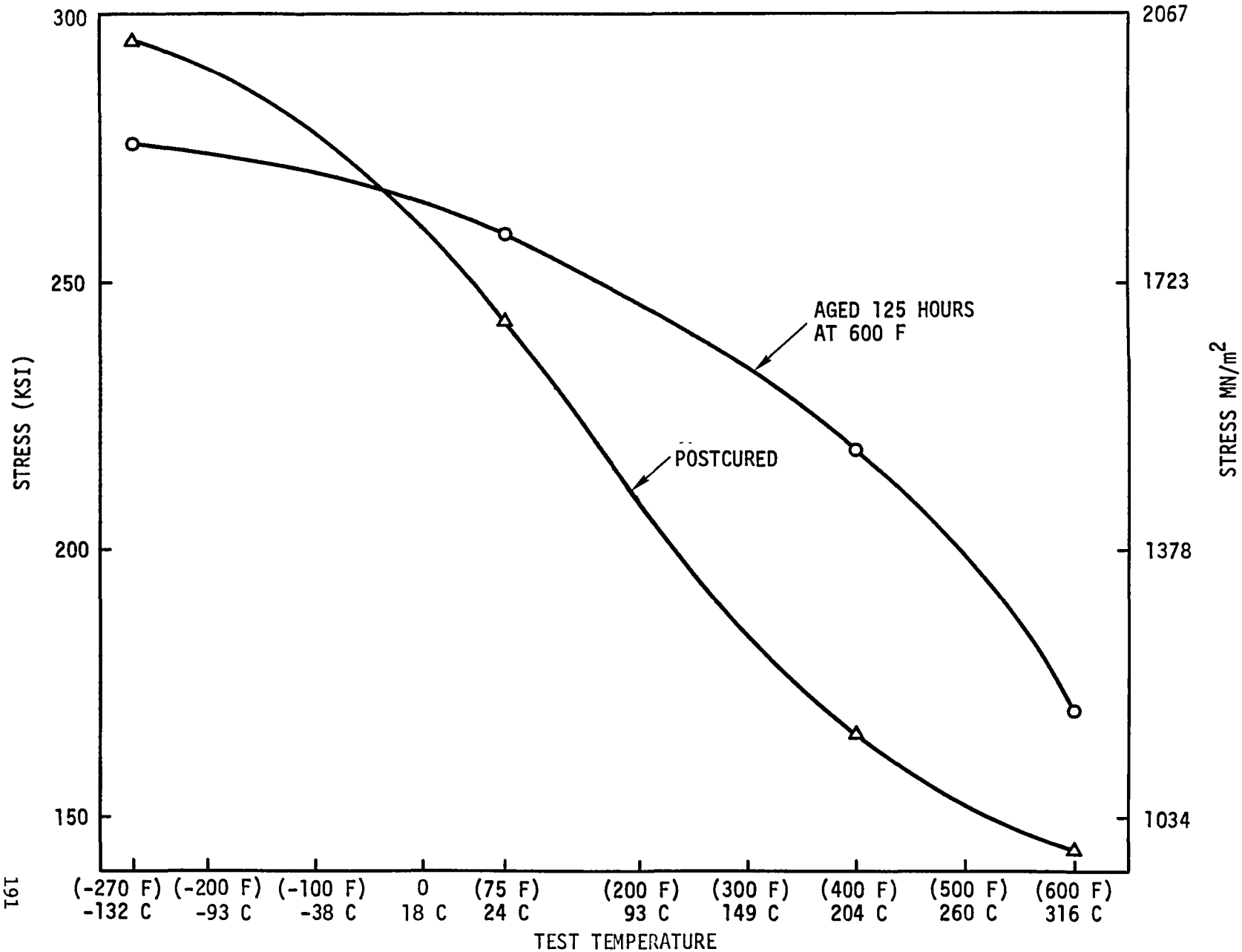


Figure 109. Flexural Strength of LARC-160/Celion 0° Laminates

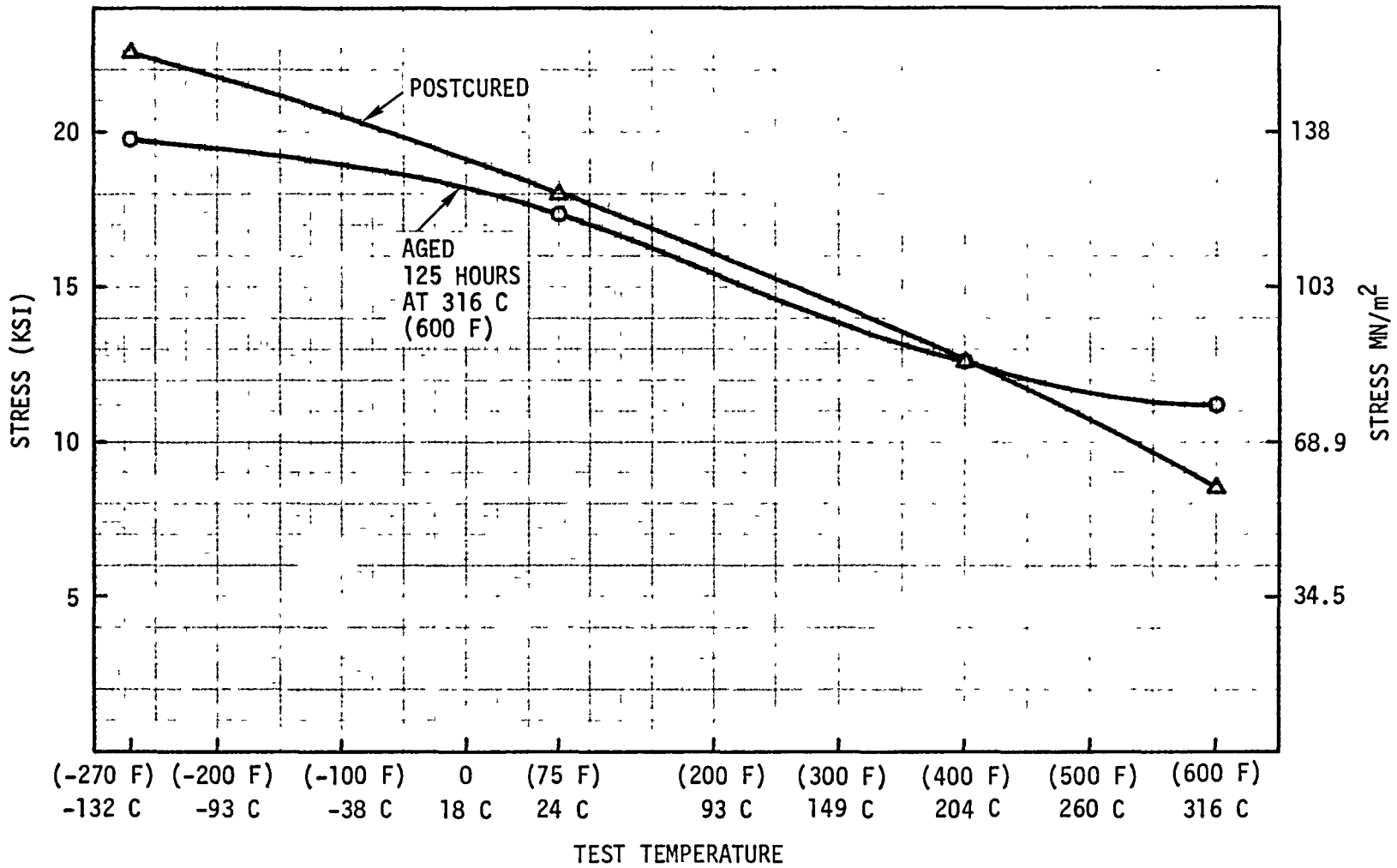


Figure 110. Short Beam Shear Strength of LARC-160/Celion Laminates

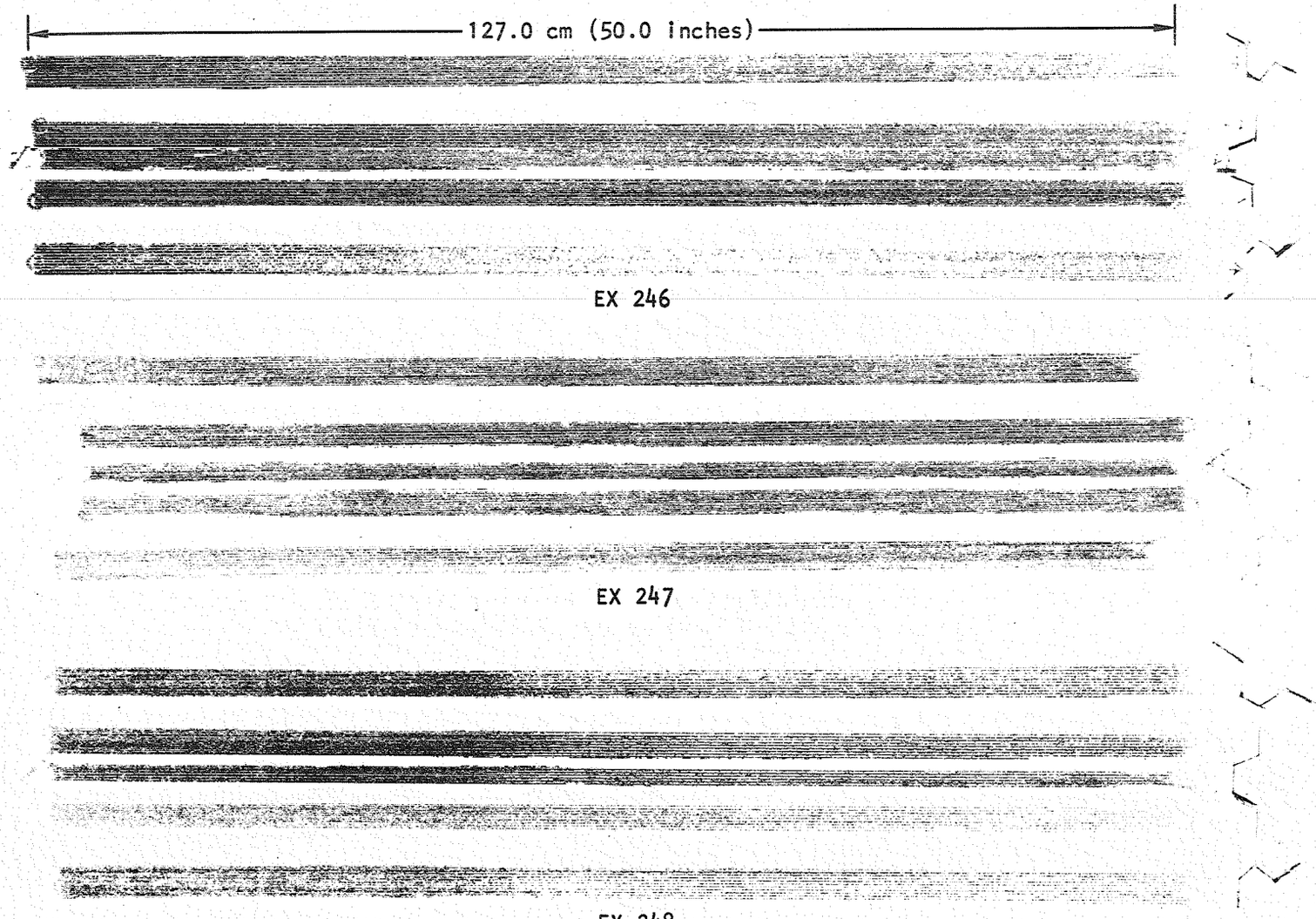
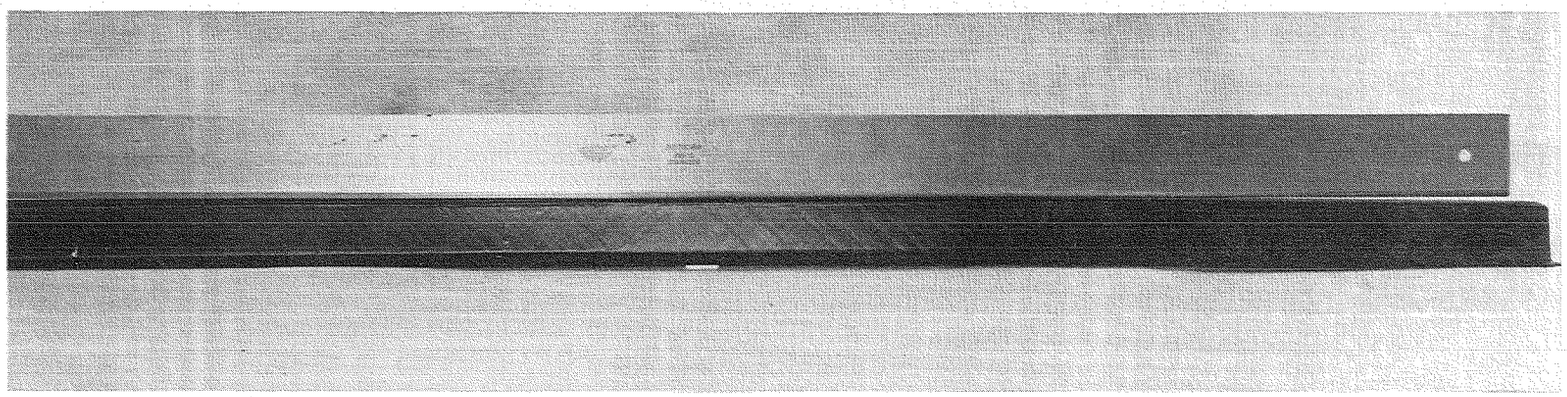


Figure 111. Typical C-Scans of "Hat" Elements



"HAT" STRINGER MOLDED ON TOOL BEFORE
REVERSE FORMING SHOWING CONCAVE WARPAGE

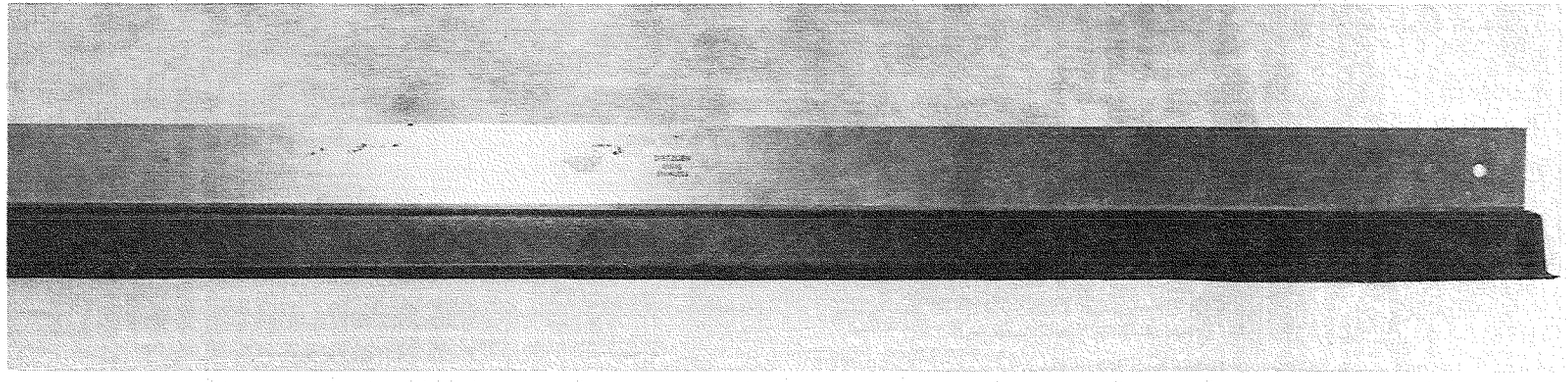


Figure 112. "Hat" Stringer Molded on Reverse Formed Tool—Showing Flat Condition

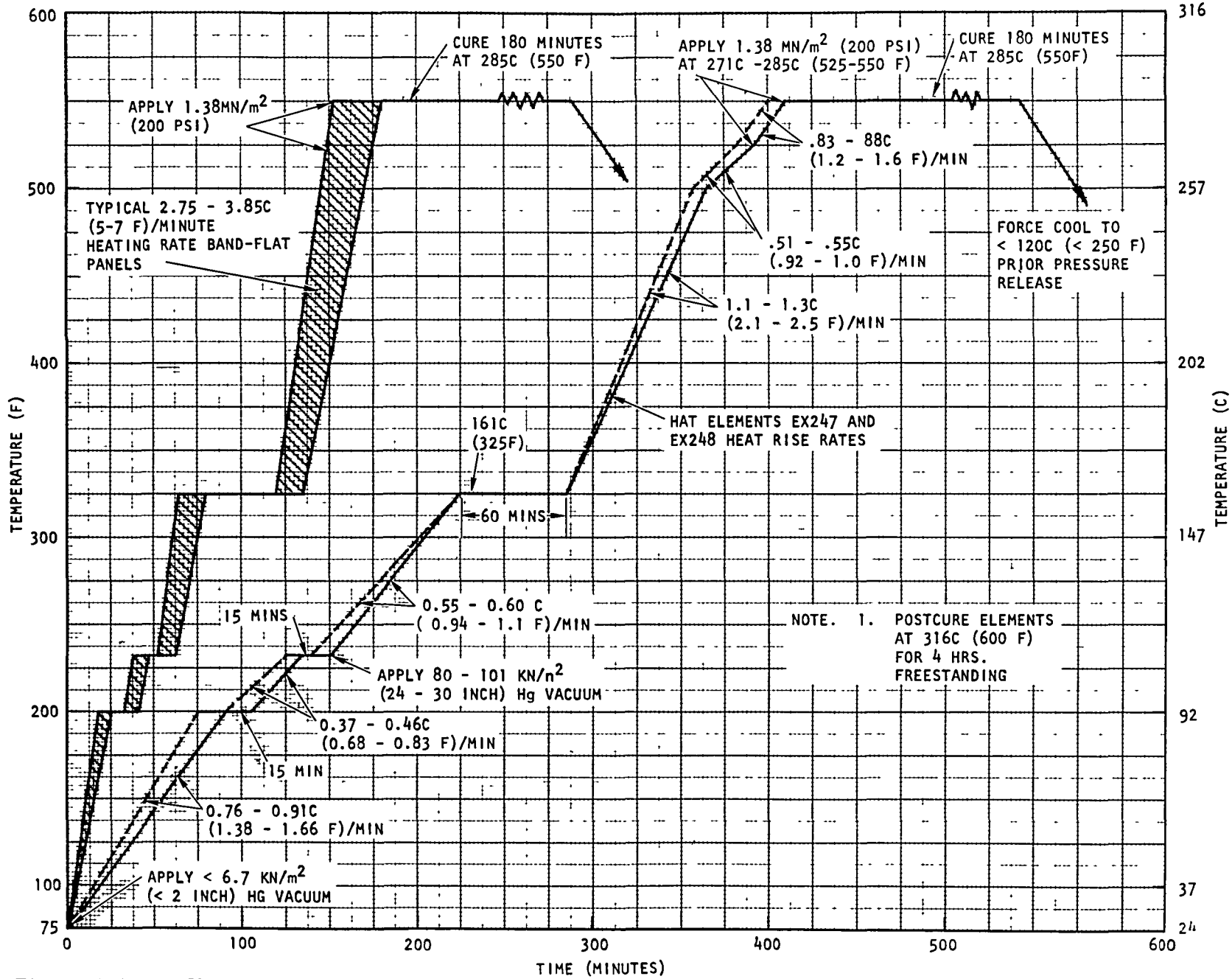


Figure 113. Influence of Tooling Mass on Cure Cycle and Heat Rise Rates - Flat Panels vs Hat Elements

A800122 C-9

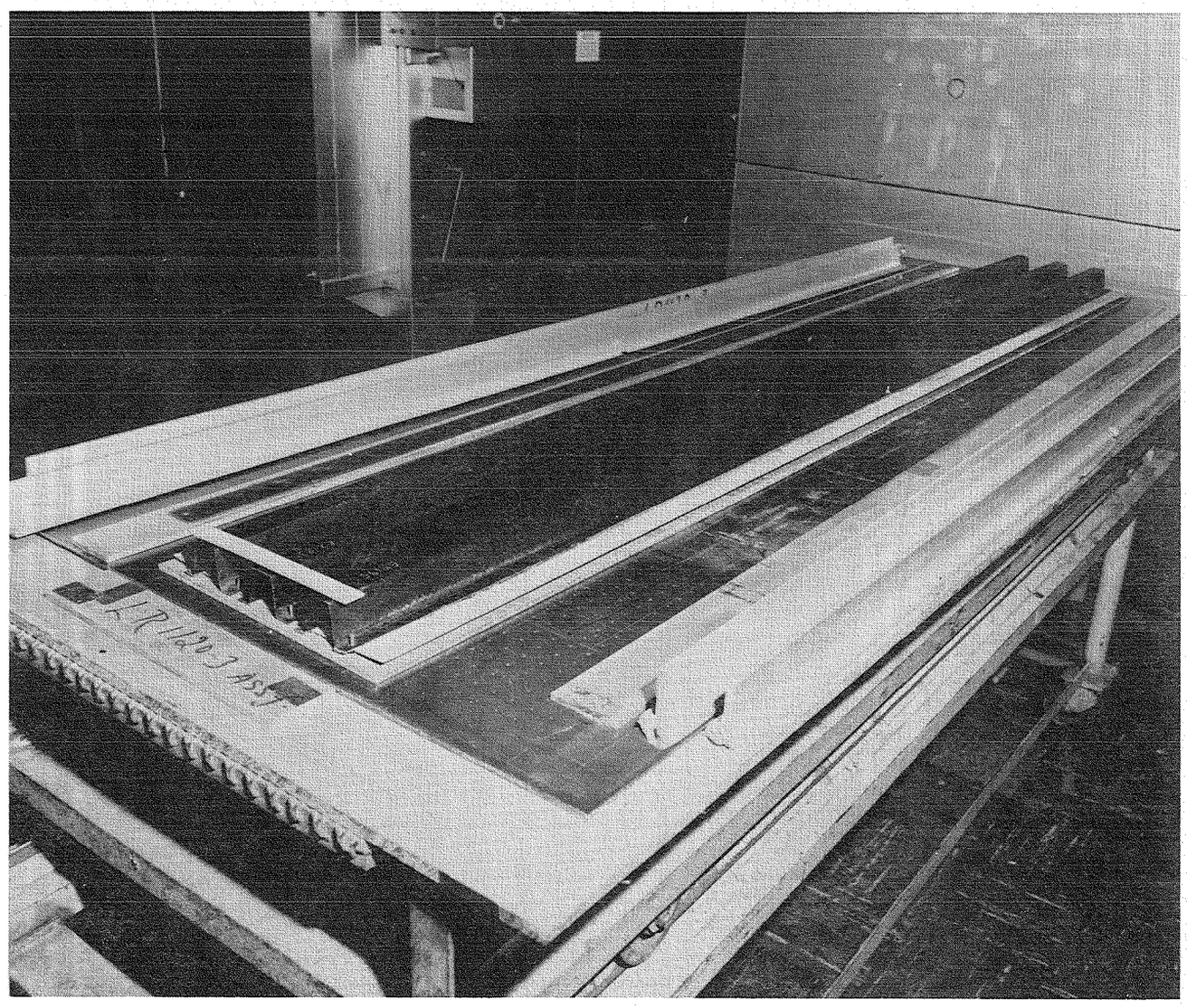


Figure 114. Hat Stringers in Position on Skin—Fit-up Operation

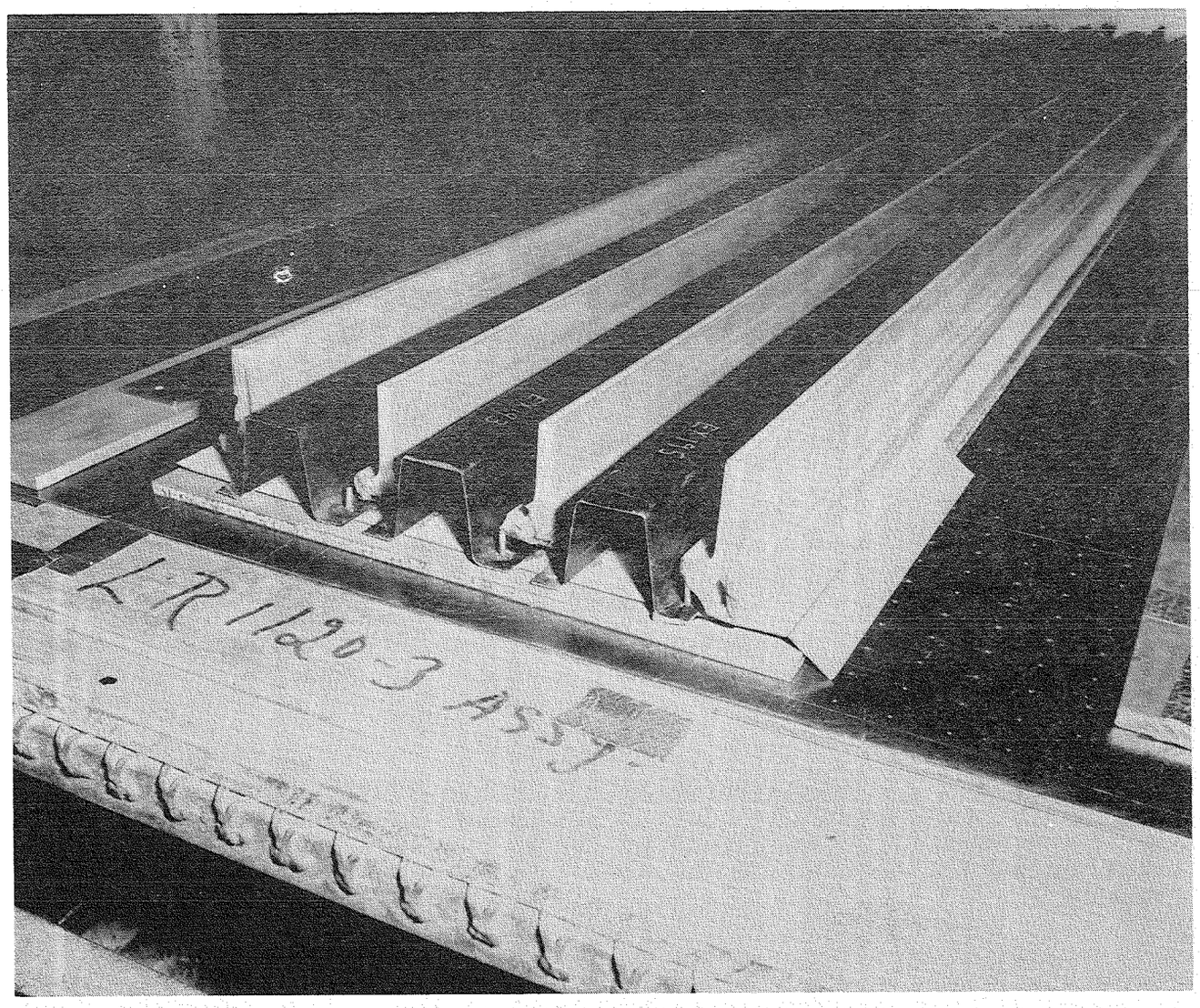


Figure 115. Hat Stringer in Position on Skin With "1" Pressure Cauls Installed

A800122 C-8

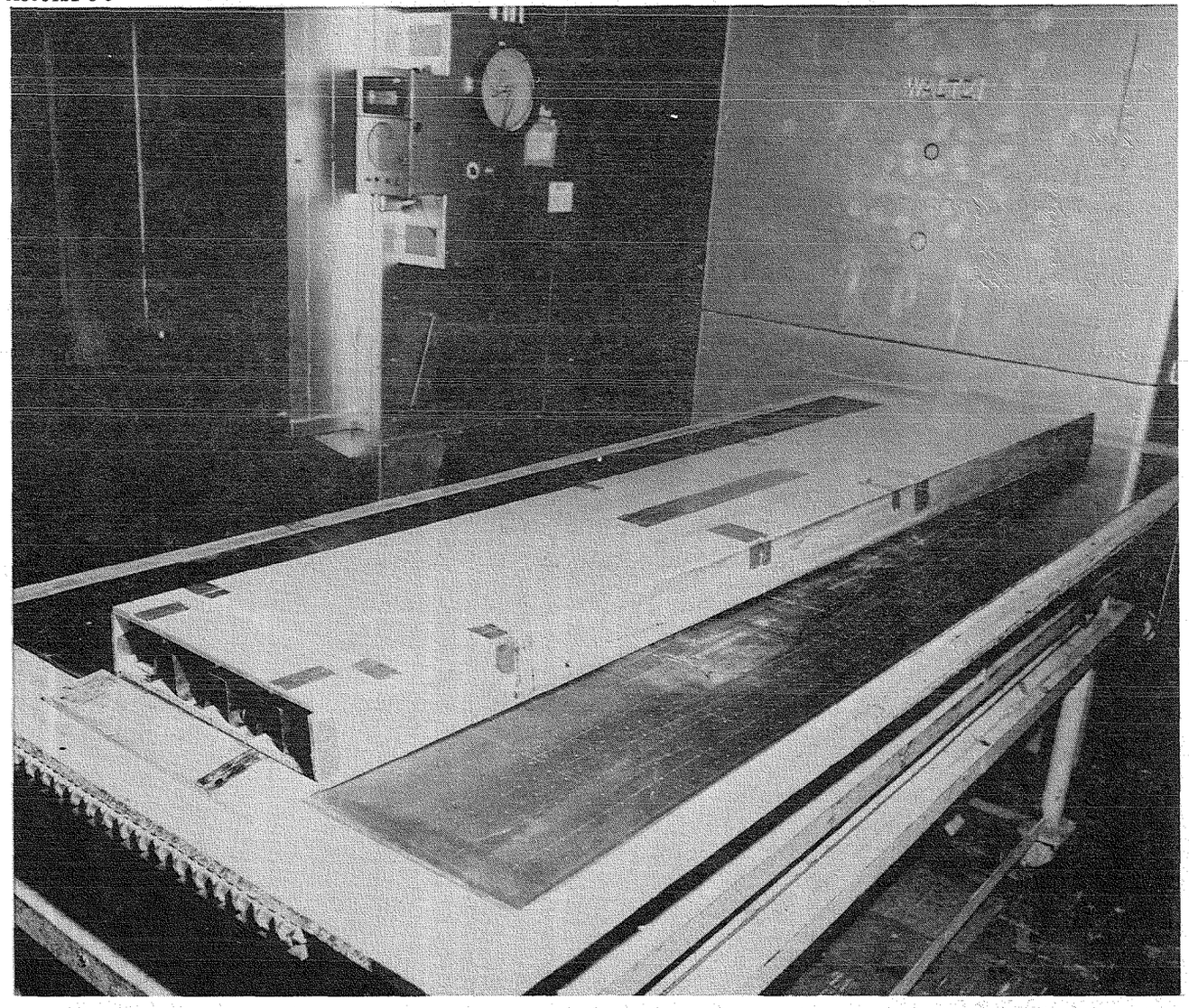
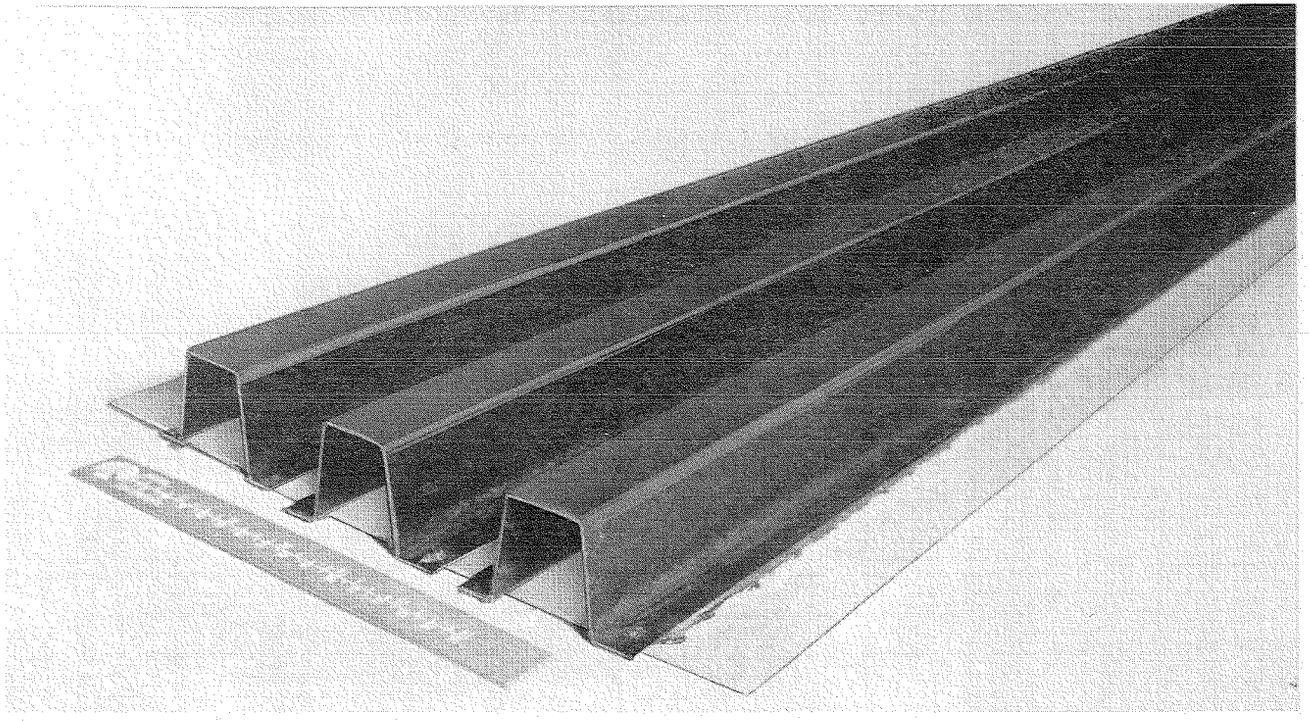


Figure 116. Pressure Augmenter Plate in Position Over "1" Pressure Cauls

A800122 C-11 C



NOTE: PANEL SIZE
29.8 X 193.0 CM
(11.75 X 76 INCHES)

A800122 C-10 C

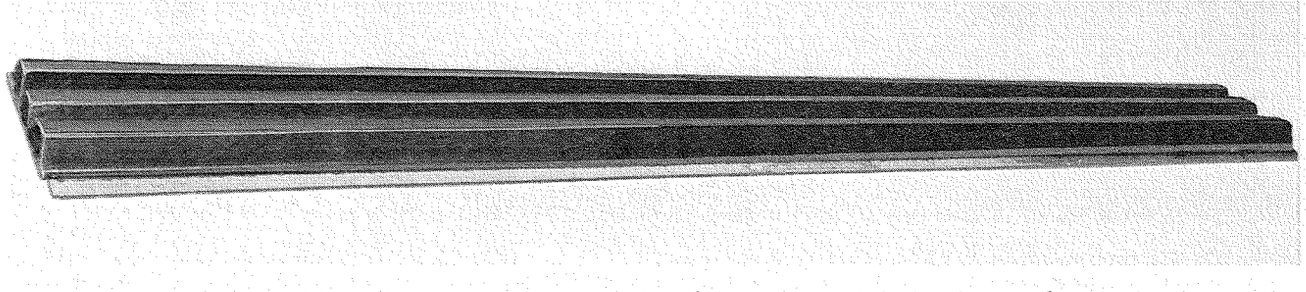


Figure 117. "Hat" Stiffened Skin/Stringer—Bonding Complete

A800122 C-12

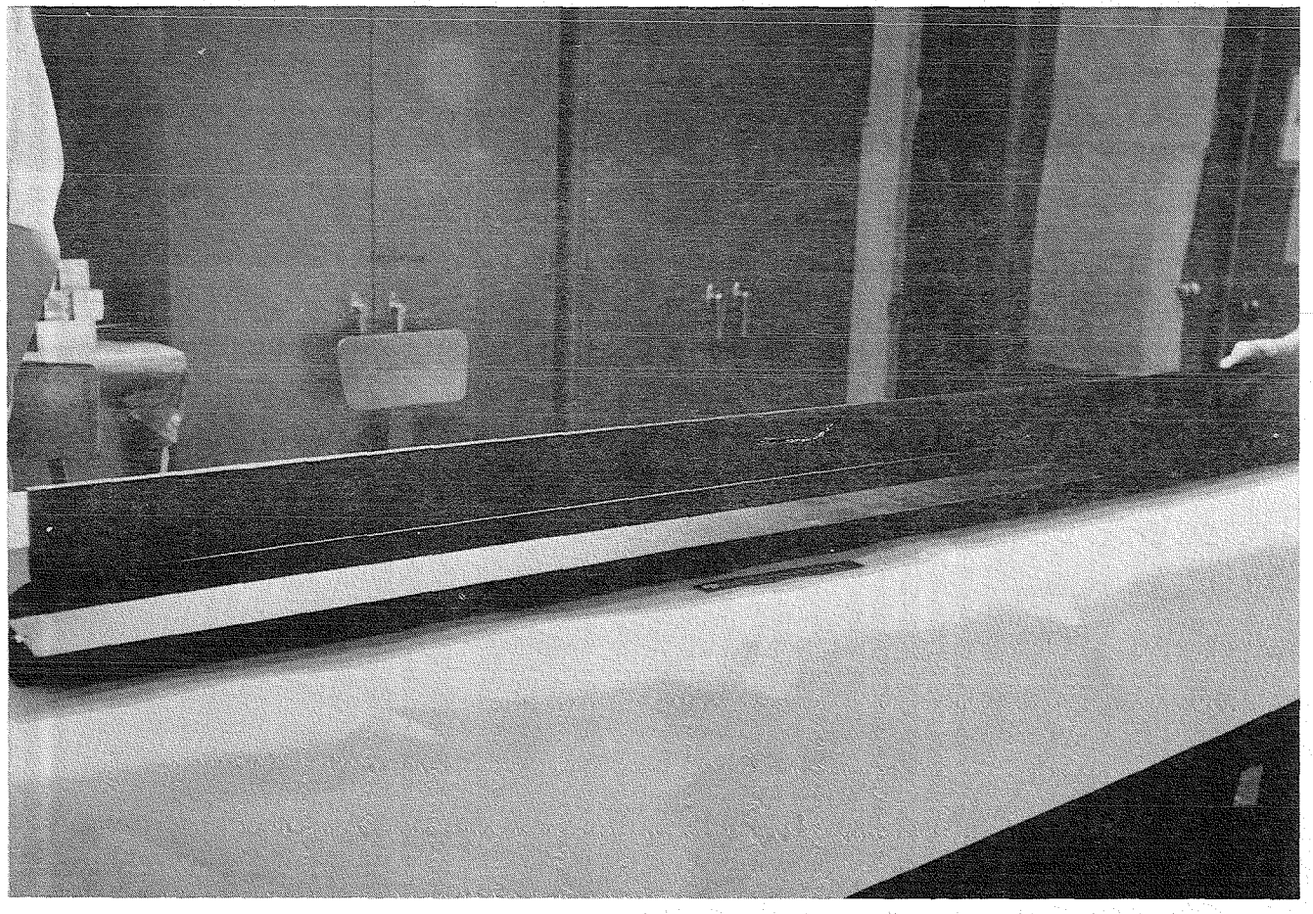


Figure 118. "Hat" Stiffened Skin/Stringer Showing Concave Skin Surface

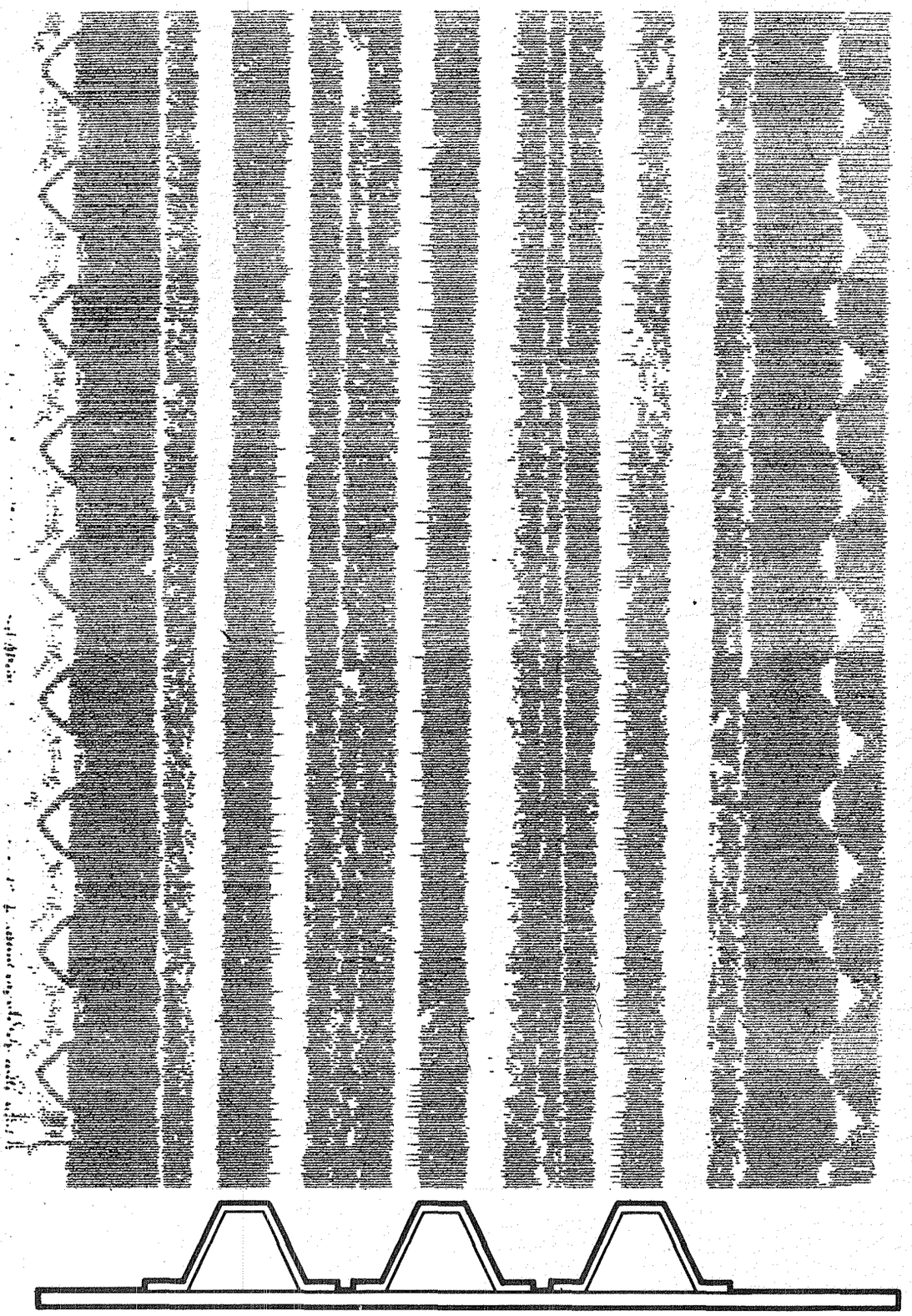
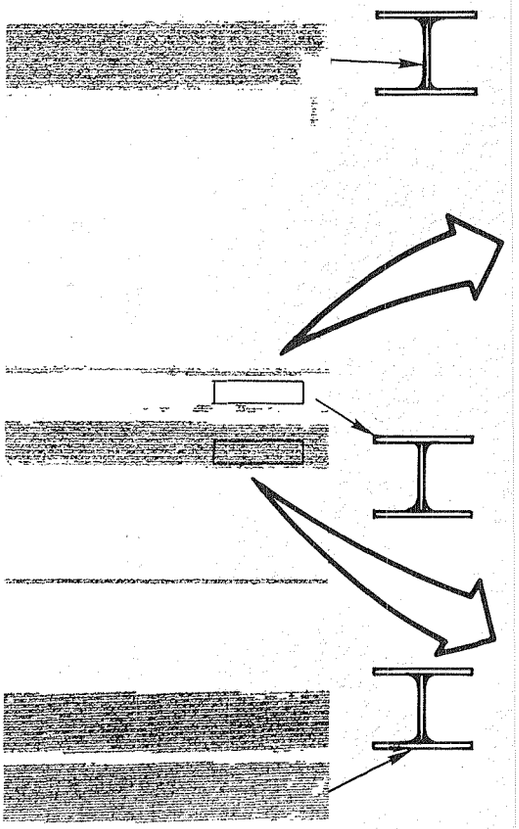
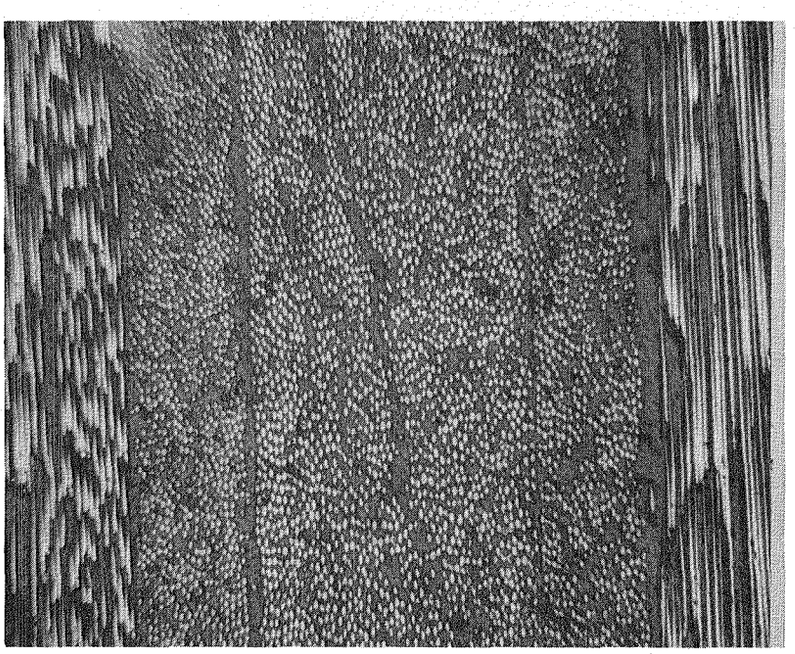
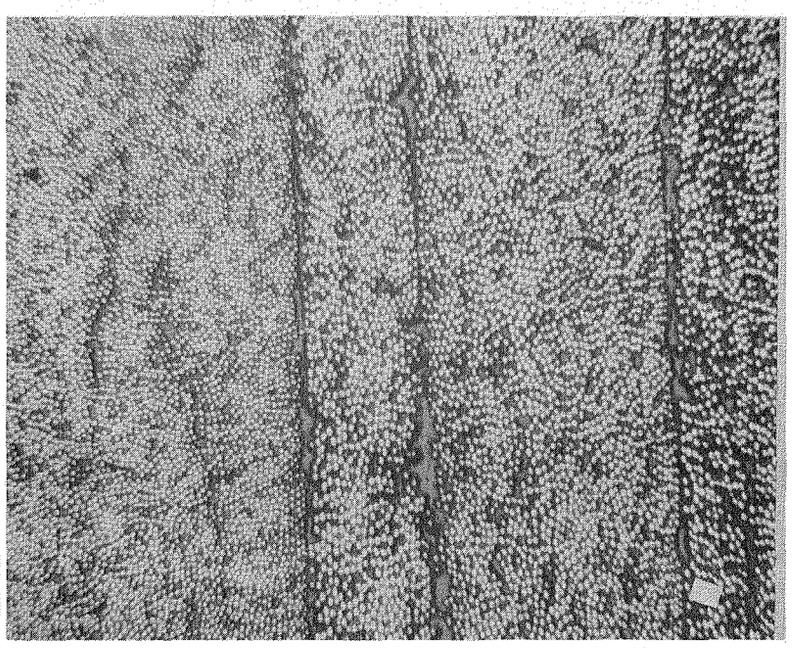


Figure 119. C-Scan of Hat-to-Skin Bond, Typical Area - 1/2 Scale





EX190 VOIDED CAP
120X



EX190 NON VOIDED CAP
120X

CAP AREA PHYSICAL PROPERTIES		
PROPERTY	VOIDED	NON VOIDED
DENSITY (G/CE)	1.467	1.581
RESIN CONTENT (%)	29.4	29.6
FIBERS VOL. (%)	58.2	62.8
VOID VOL. (%)	8.30	1.13

Figure 120. C-Scan and Photomicrograph Correlation of "I" Stringer Cap Void Characteristics

A800122 C-2

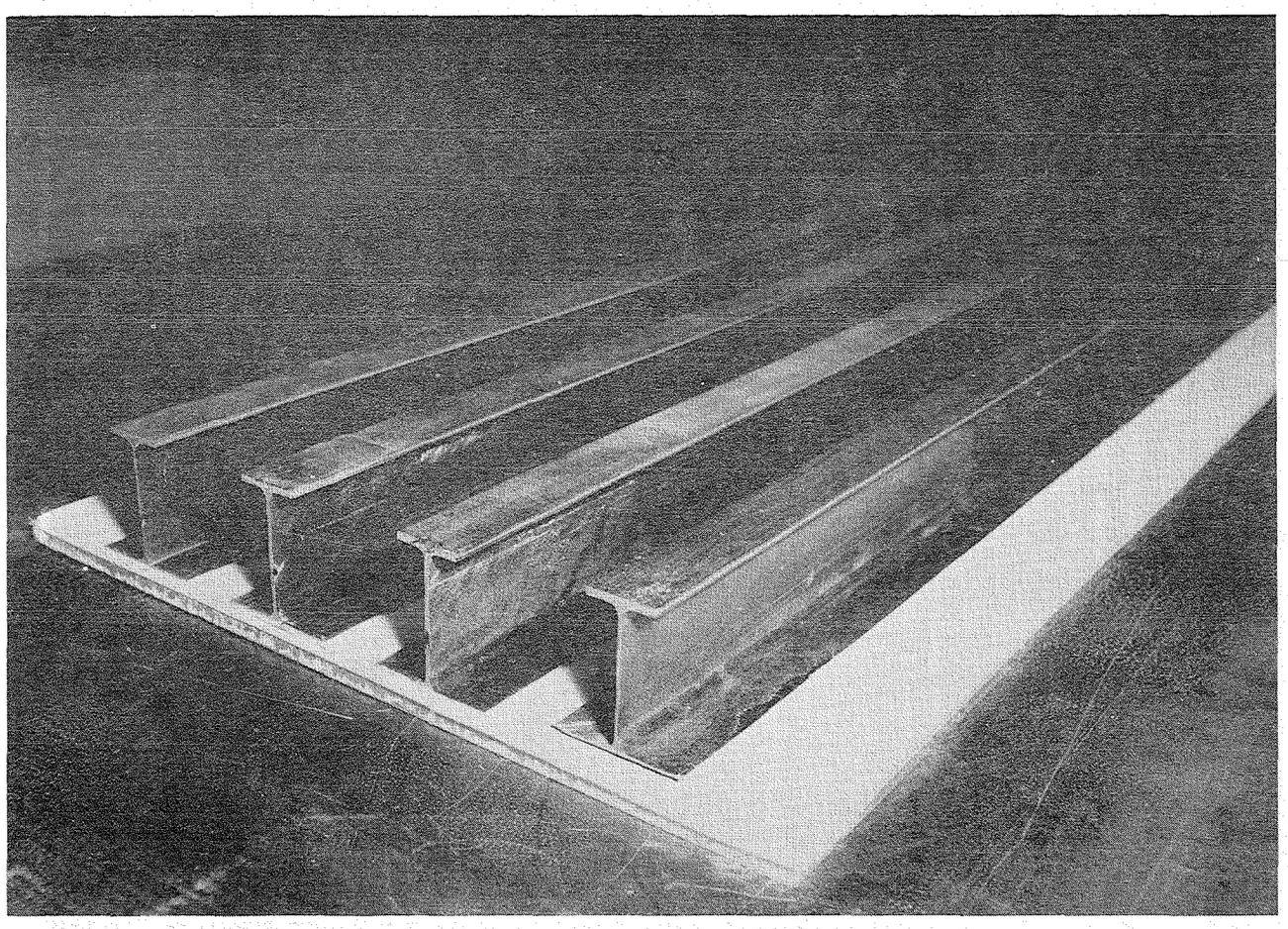


Figure 121. "I" Stringers in Dry Fit Position on Skin Assembly

A800122 C-3

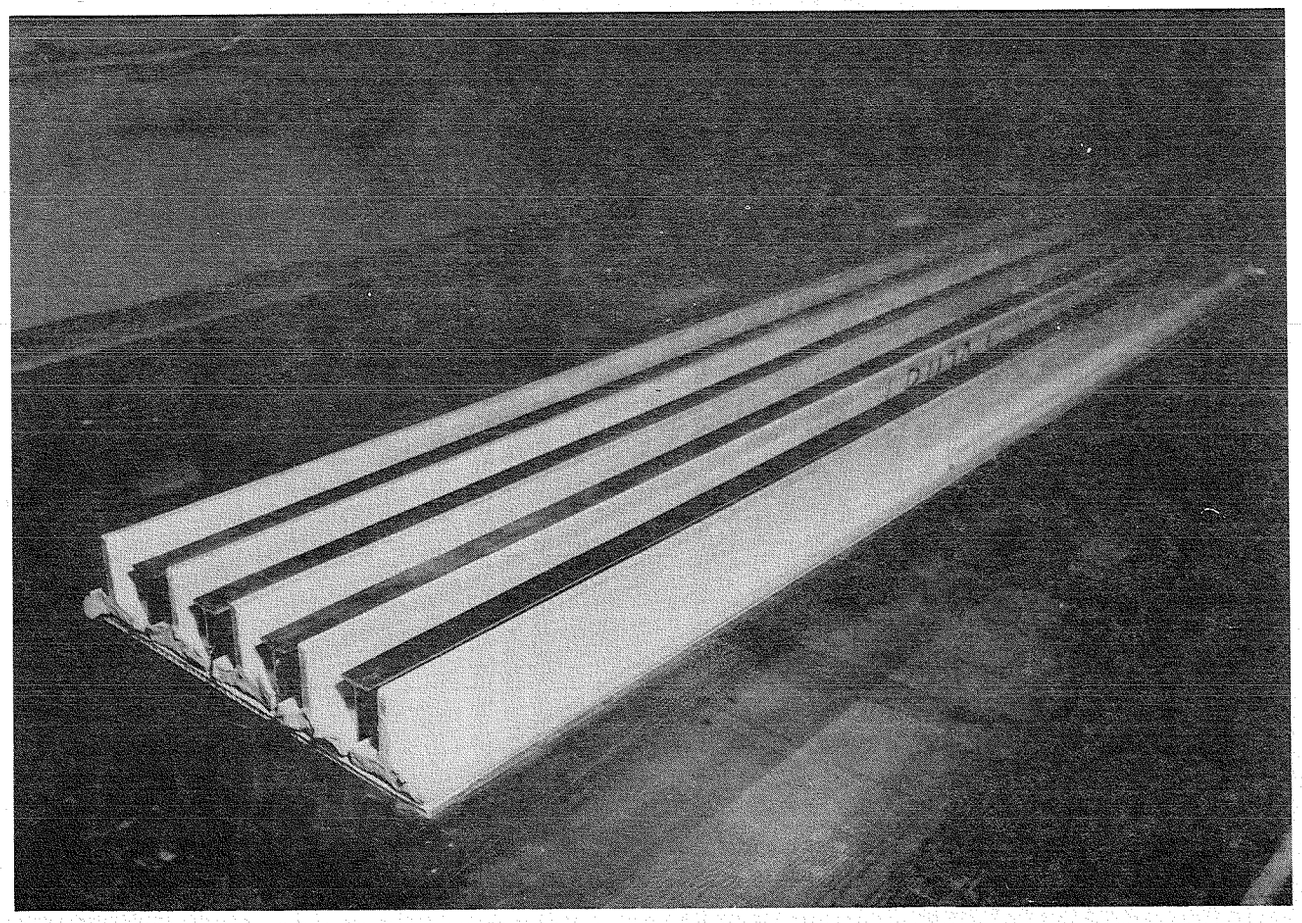


Figure 122. "I" Stringer in Bonding Position With "I" Pressure Cauls Installed

A800122 C-4

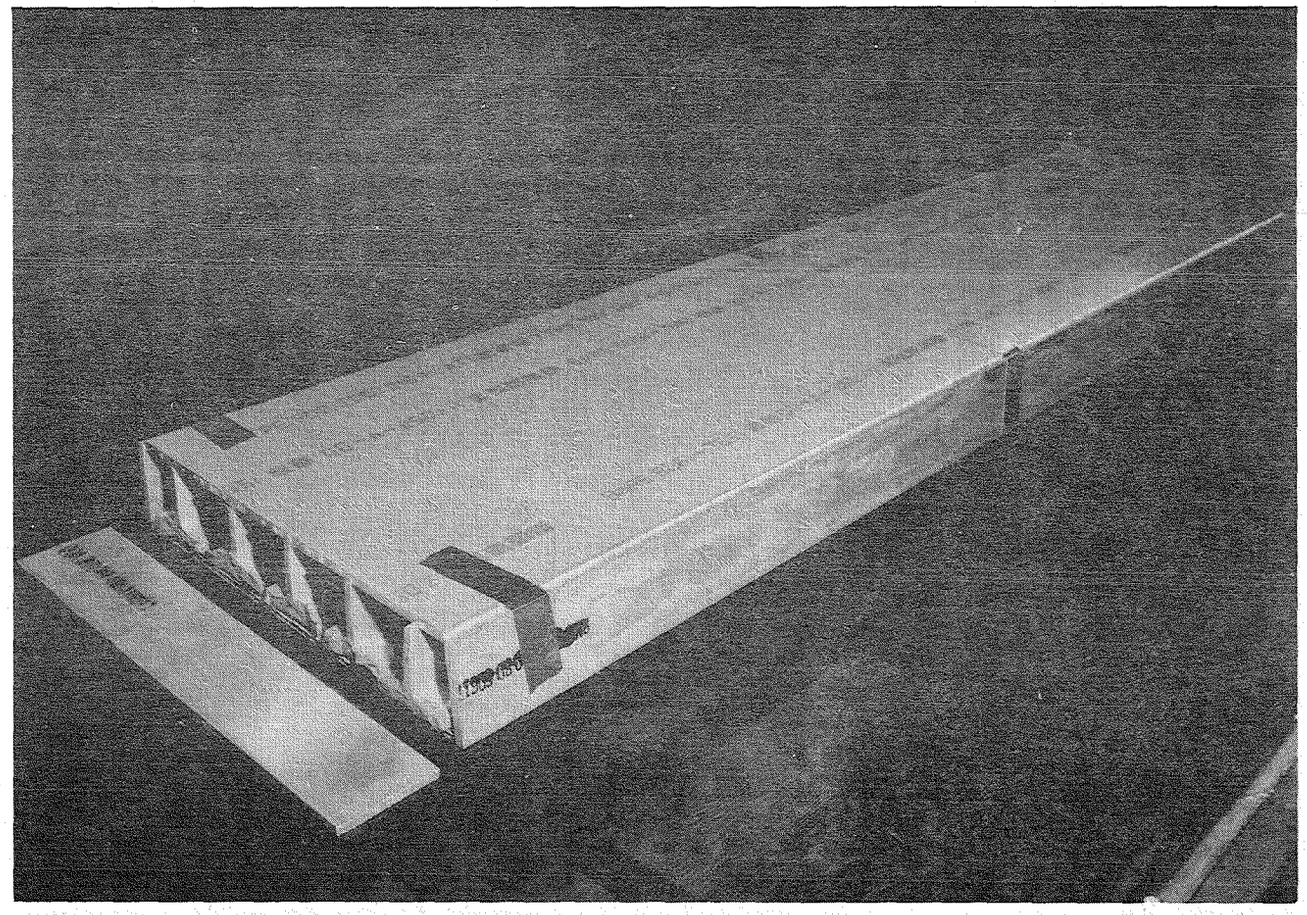


Figure 123. Pressure Augmenter Plate in Position Over "⊥" Pressure Cauls, "I" Stiffened Skin/Stringer Assembly

A800122 C-5

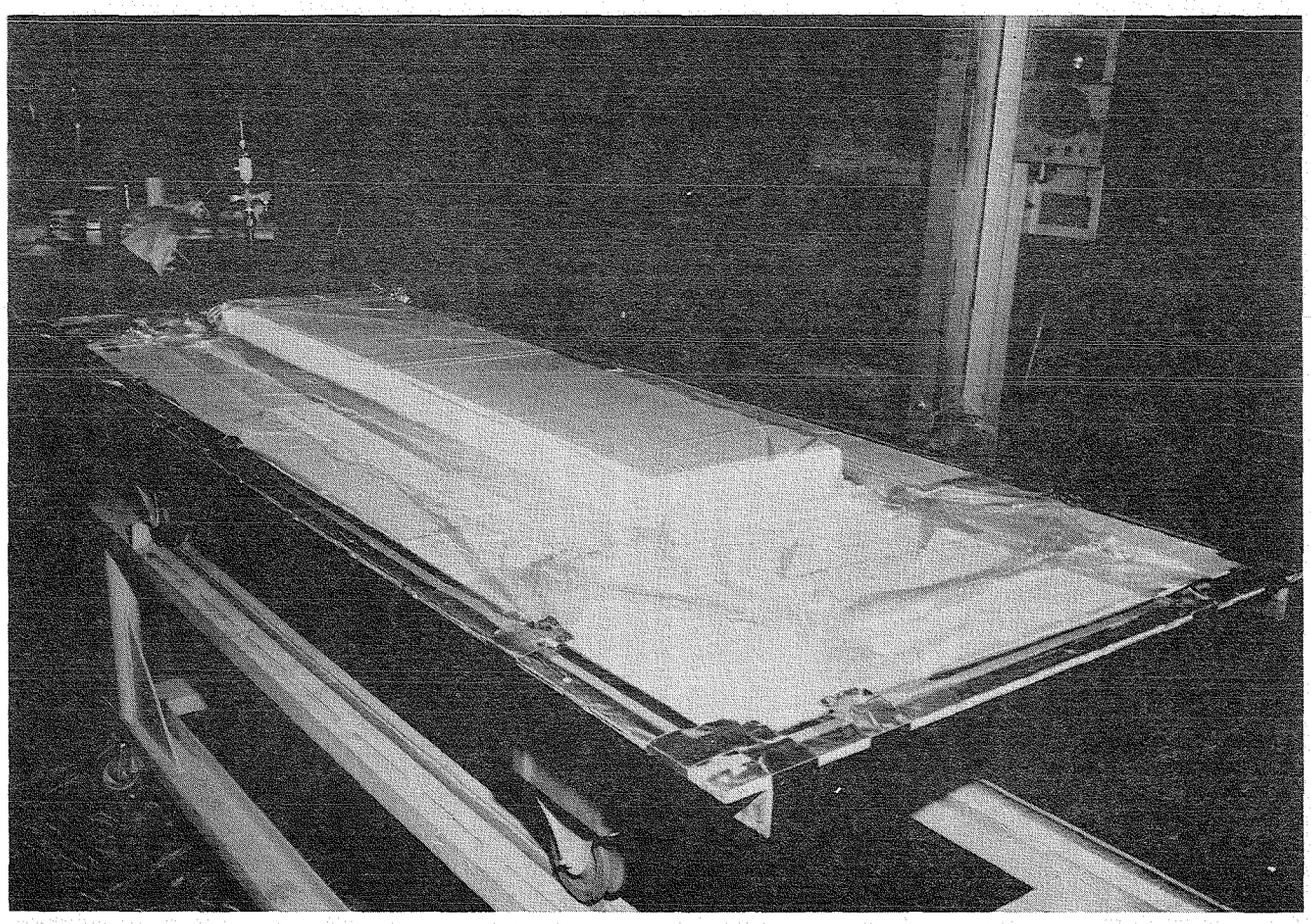
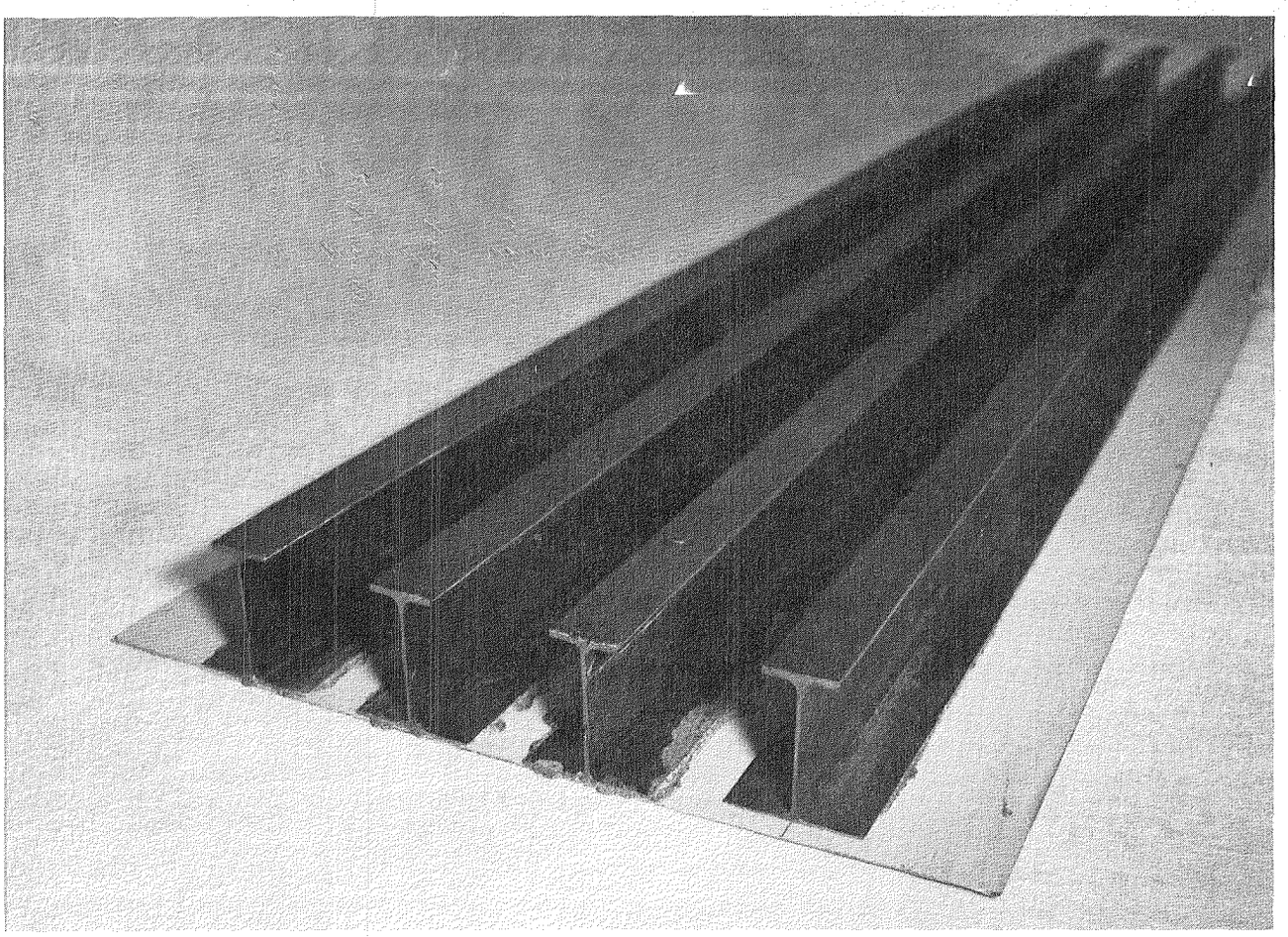


Figure 124. "I" Stringer Stiffened Skin Element in Vacuum Bag Bonding Fixture

A800125 C-2



A800125 C-1

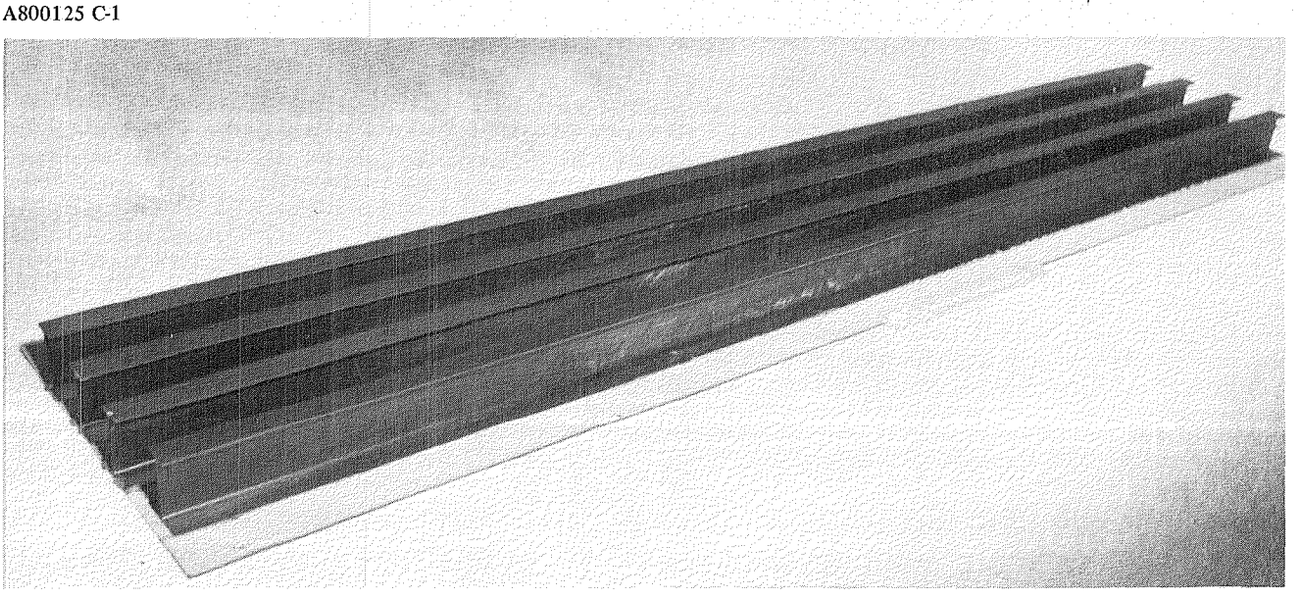


Figure 125. "I" Stiffened Skin/Stringer Panel Bonded Complete

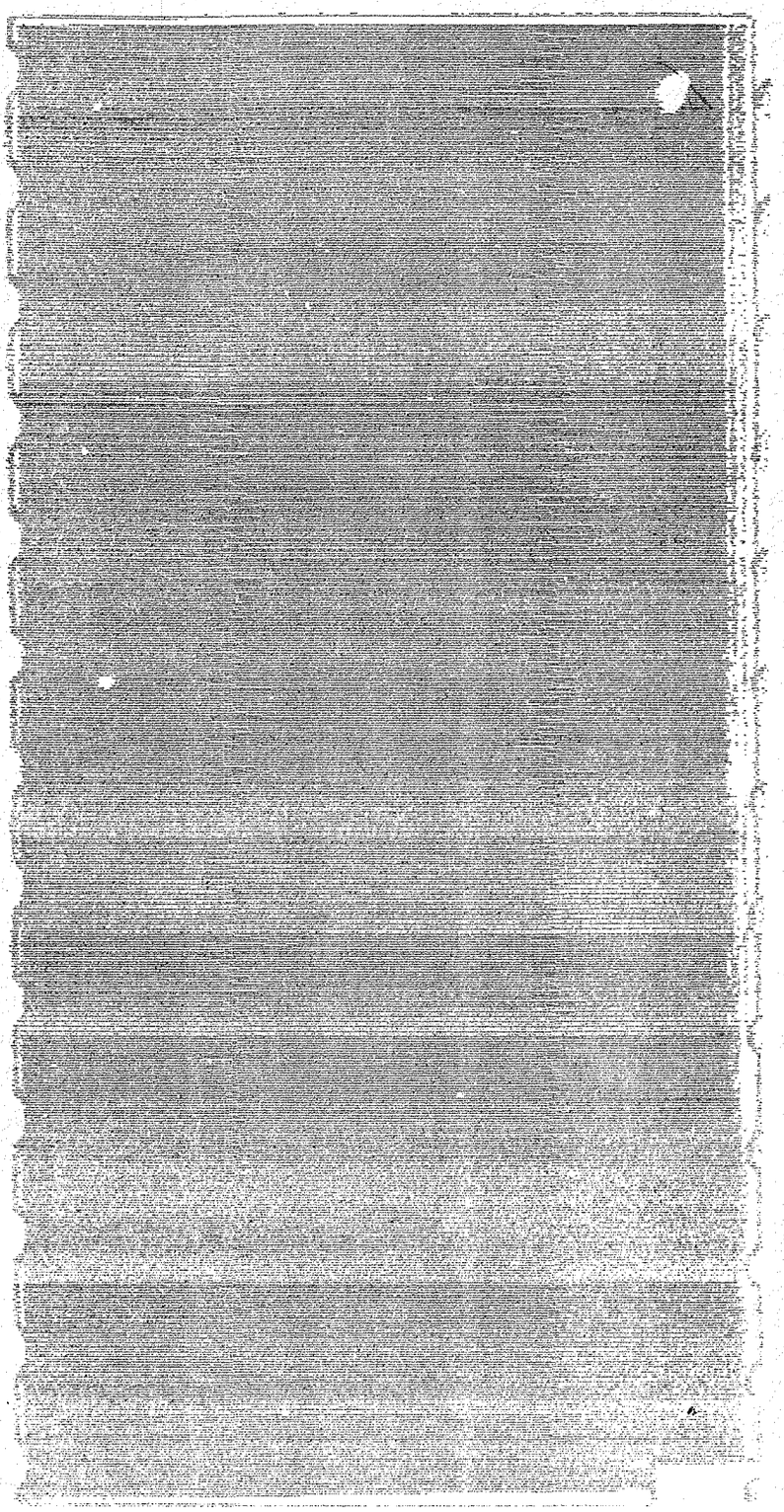


Figure 126. C-Scan of 35X34 5 Harness Satin Weave Celion Fabric/ LARC-160 Laminate 0.33 cm Thick

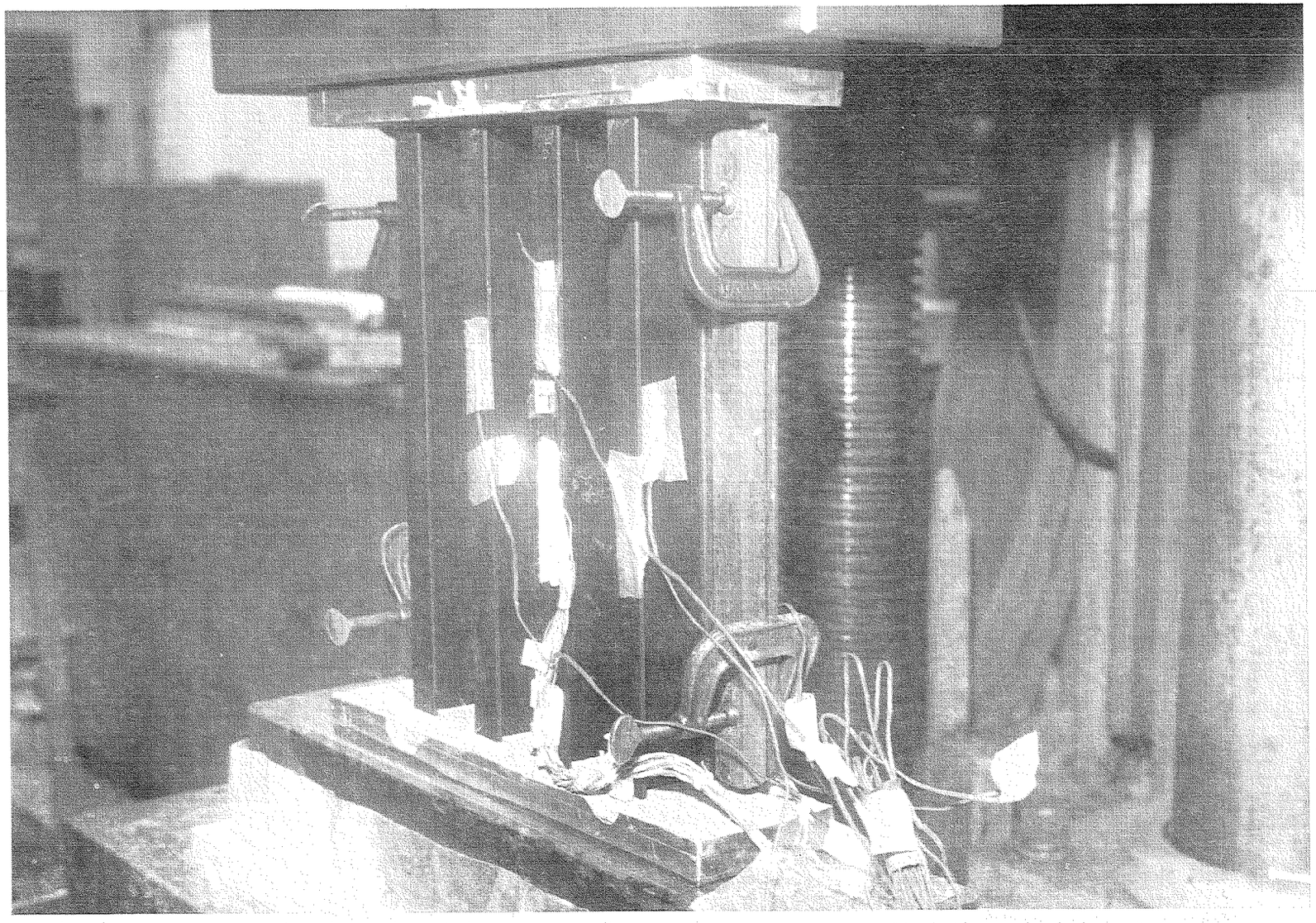


Figure 127. "I" Stringer Stiffened Skin Panel Element EX111/EX113 Being Readied for -132 C (-270 F) Test

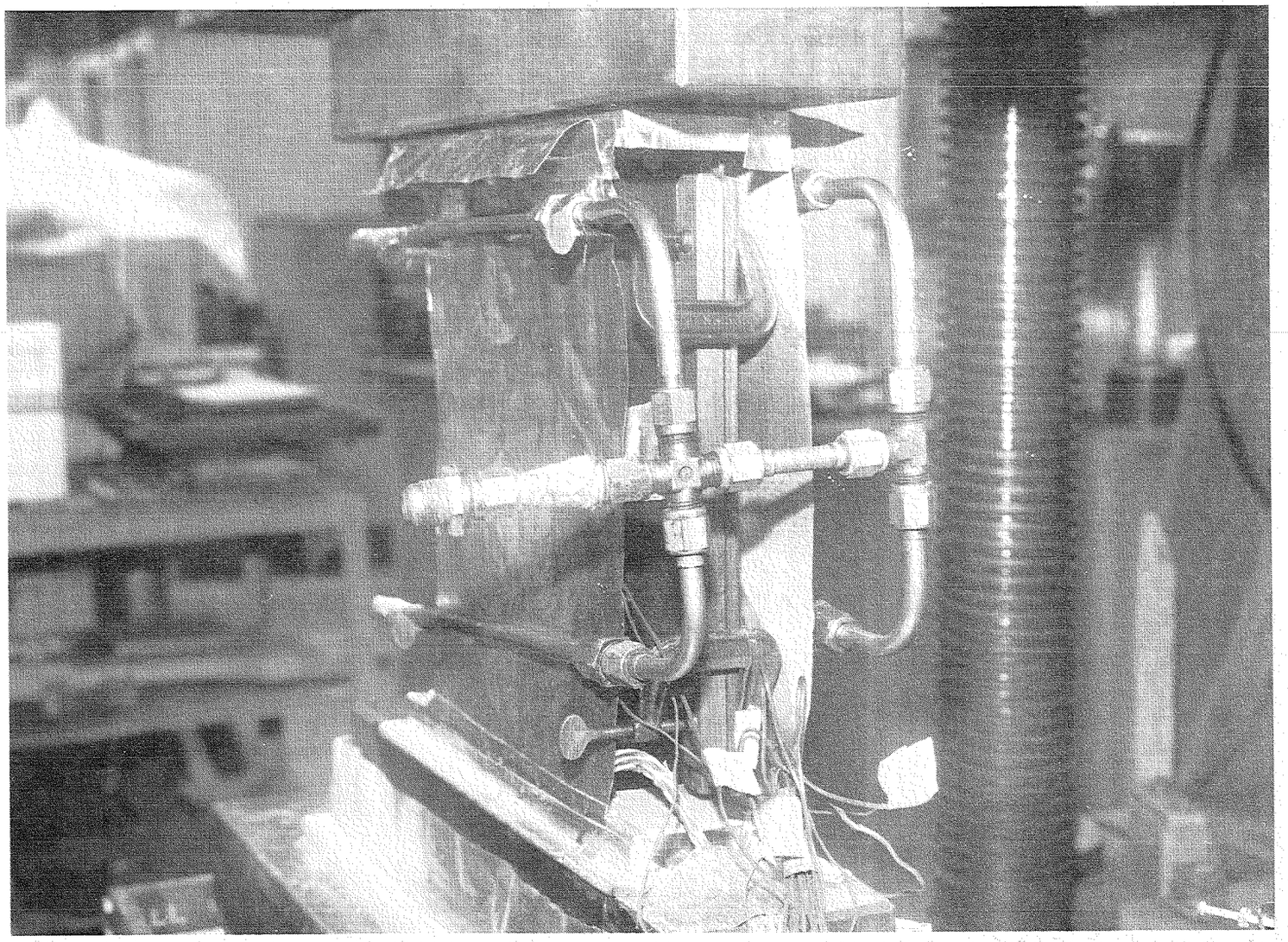


Figure 128. "I" Stiffened Skin/Stringer Panel, LN_2 Manifolds and Baffle Plates in Place

11C92-90-5C

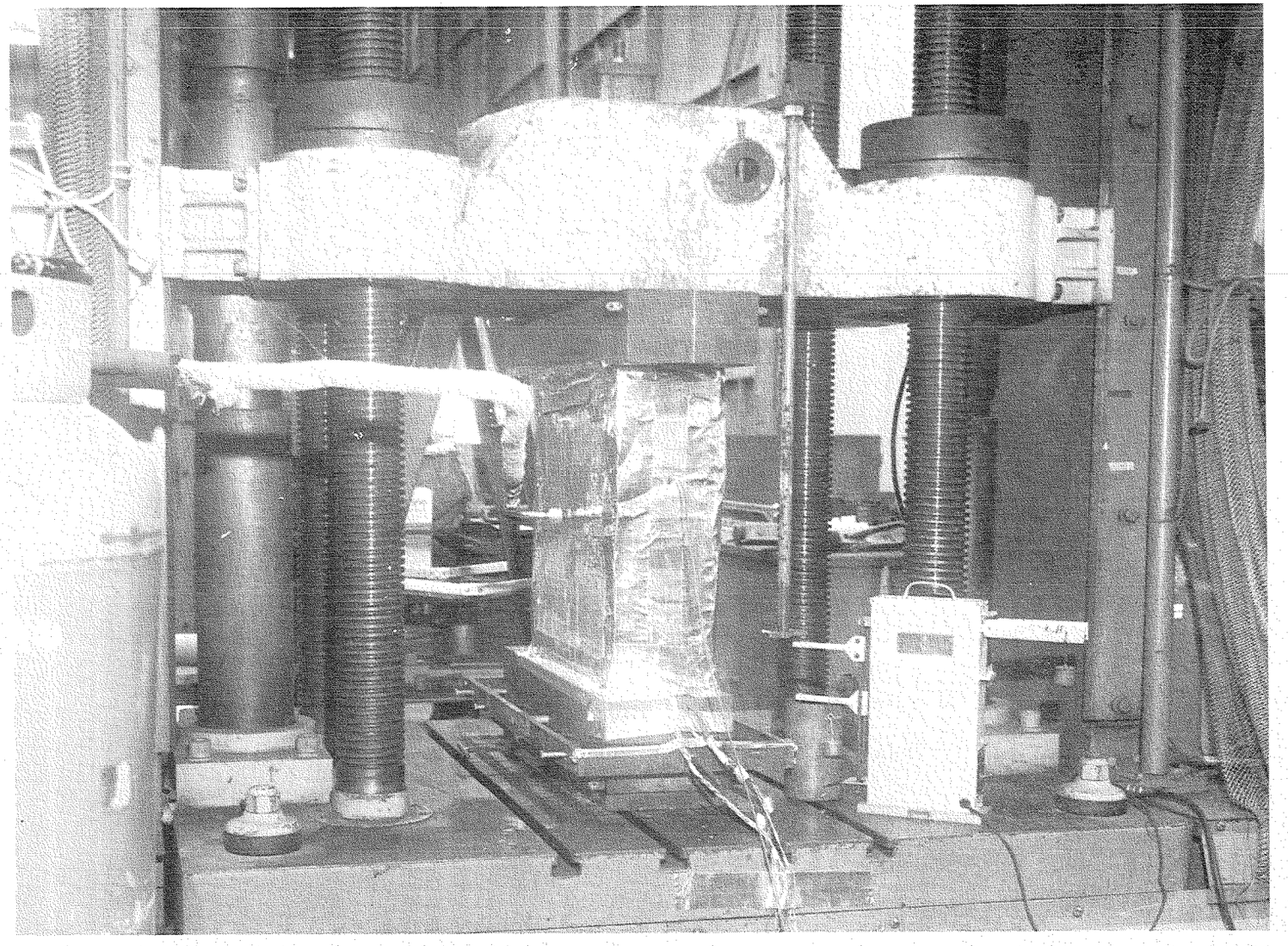


Figure 129. Typical Test Set-up for -132 C (-270 F) Compression Element Test

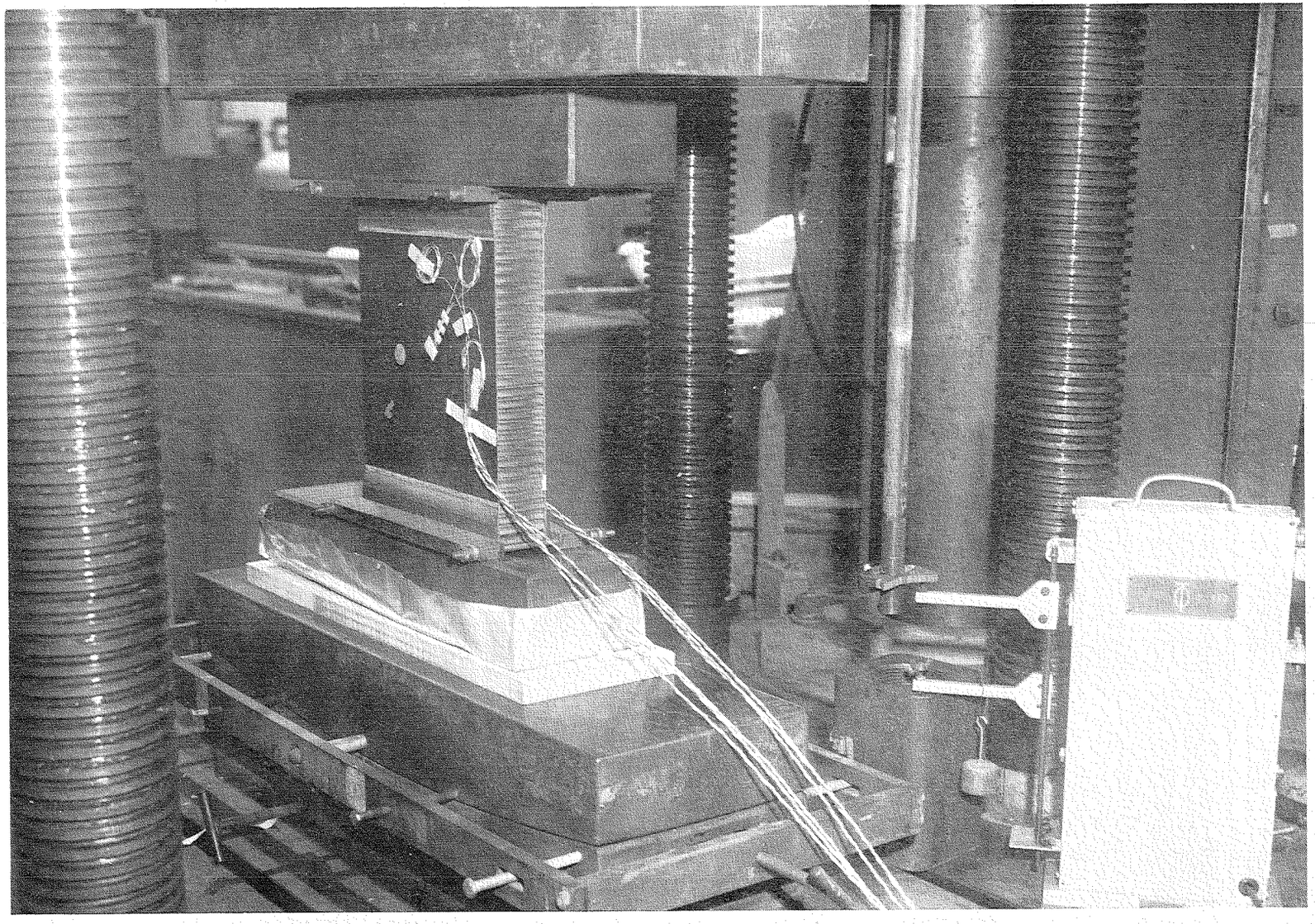
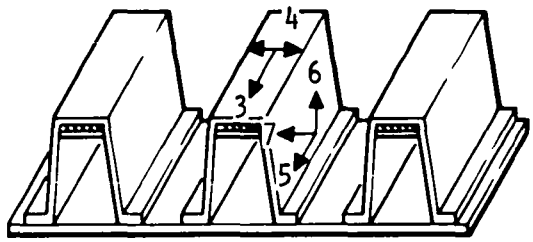
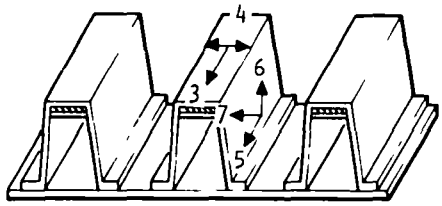


Figure 130. Sandwich Element R.T. Test Set-up



GAGES 1 AND 2 ON LOWER SKIN DIRECTLY OPPOSITE 3 AND 4

Figure 131. Load/Strain Characteristics of "Hat" Stringer Stiffened Skin Element EX109/EX110 AT-132C (-270F)



GAGES 1 AND 2 ON LOWER
SKIN DIRECTLY OPPOSITE
3 AND 4

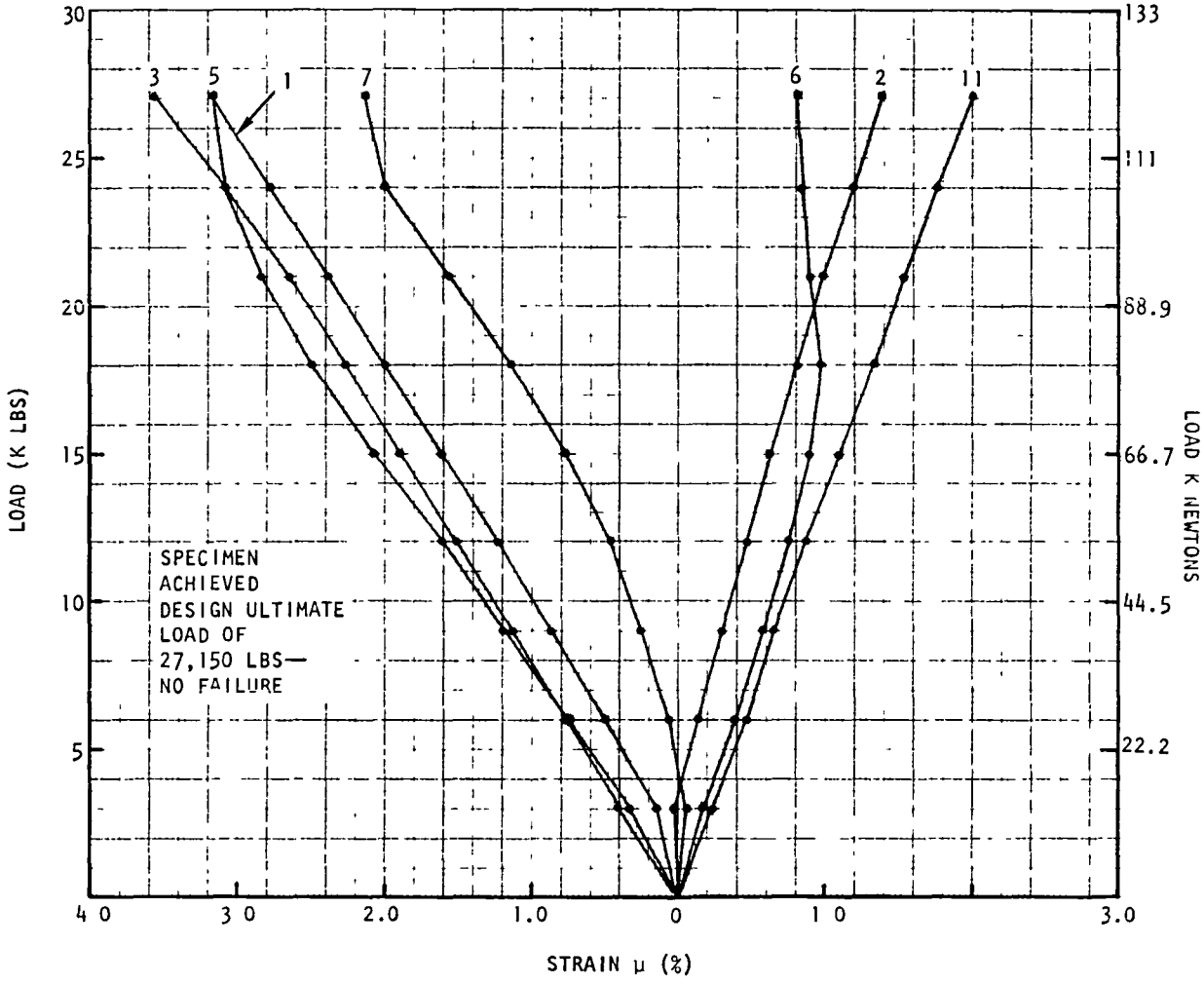
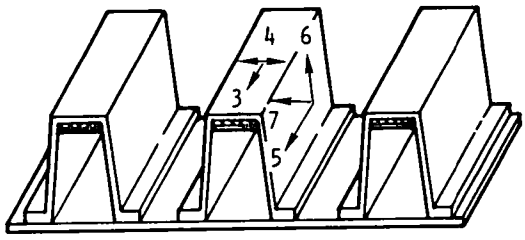


Figure 132. Compression Load/Strain Characteristics of "Hat" Stringer Stiffened Skin Element EX195-4A Aged 125 Hours at 316 C (600 F) and Tested at—132 C (-270 F)



STRAIN GAGES 1 AND 2 ON
LOWER SKIN DIRECTLY
OPPOSITE 3 AND 4

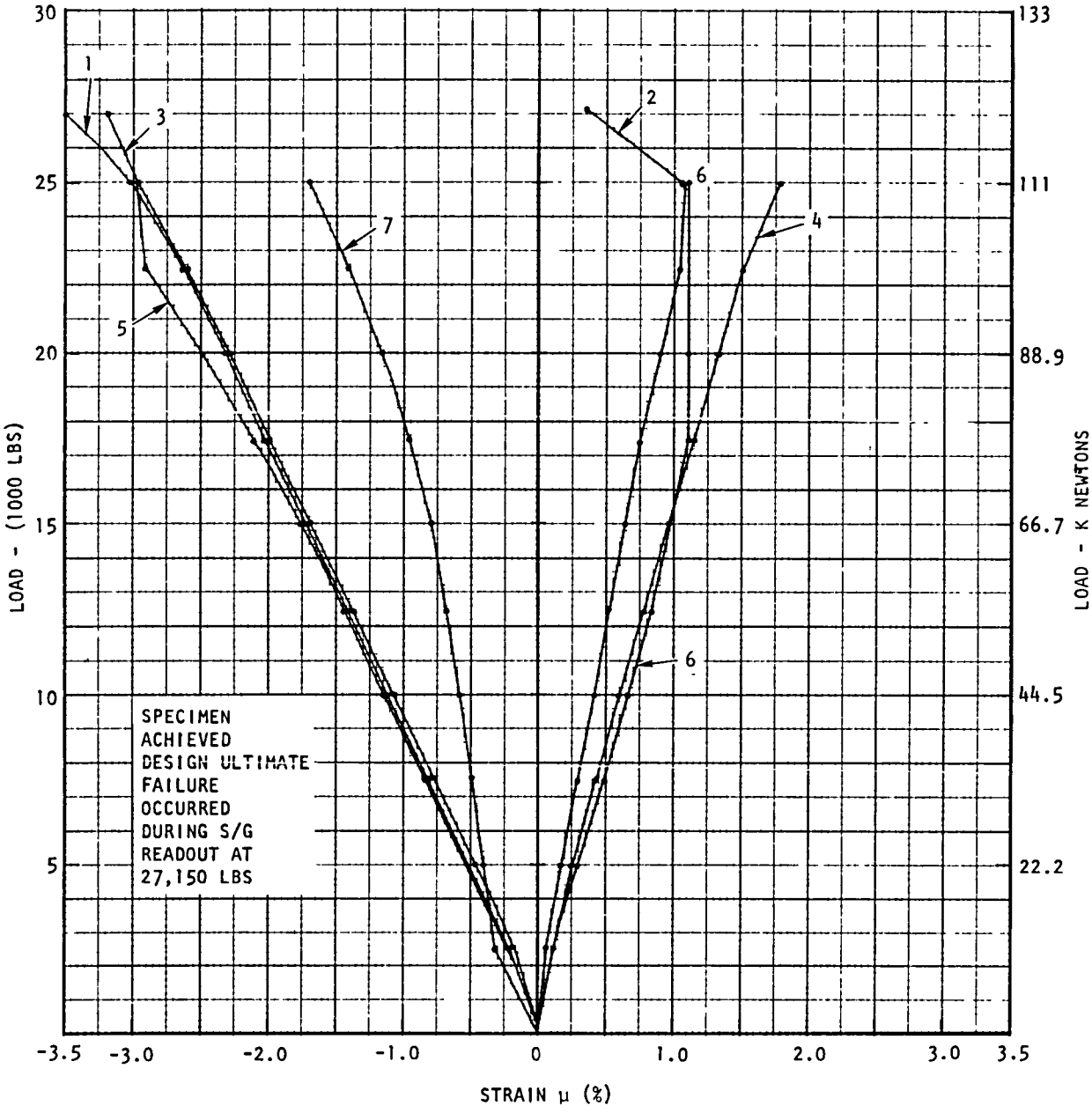
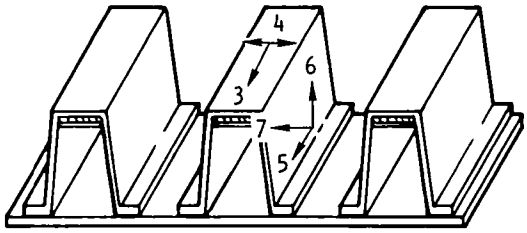


Figure 133. Compression Load/Strain Characteristics of Hat Stringer Stiffened Skin Element E109/EX110A, Postcured Condition Tested at Room Temperature



STRAIN GAGES 1 AND 2 ON
LOWER SKIN DIRECTLY
OPPOSITE 3 AND 4

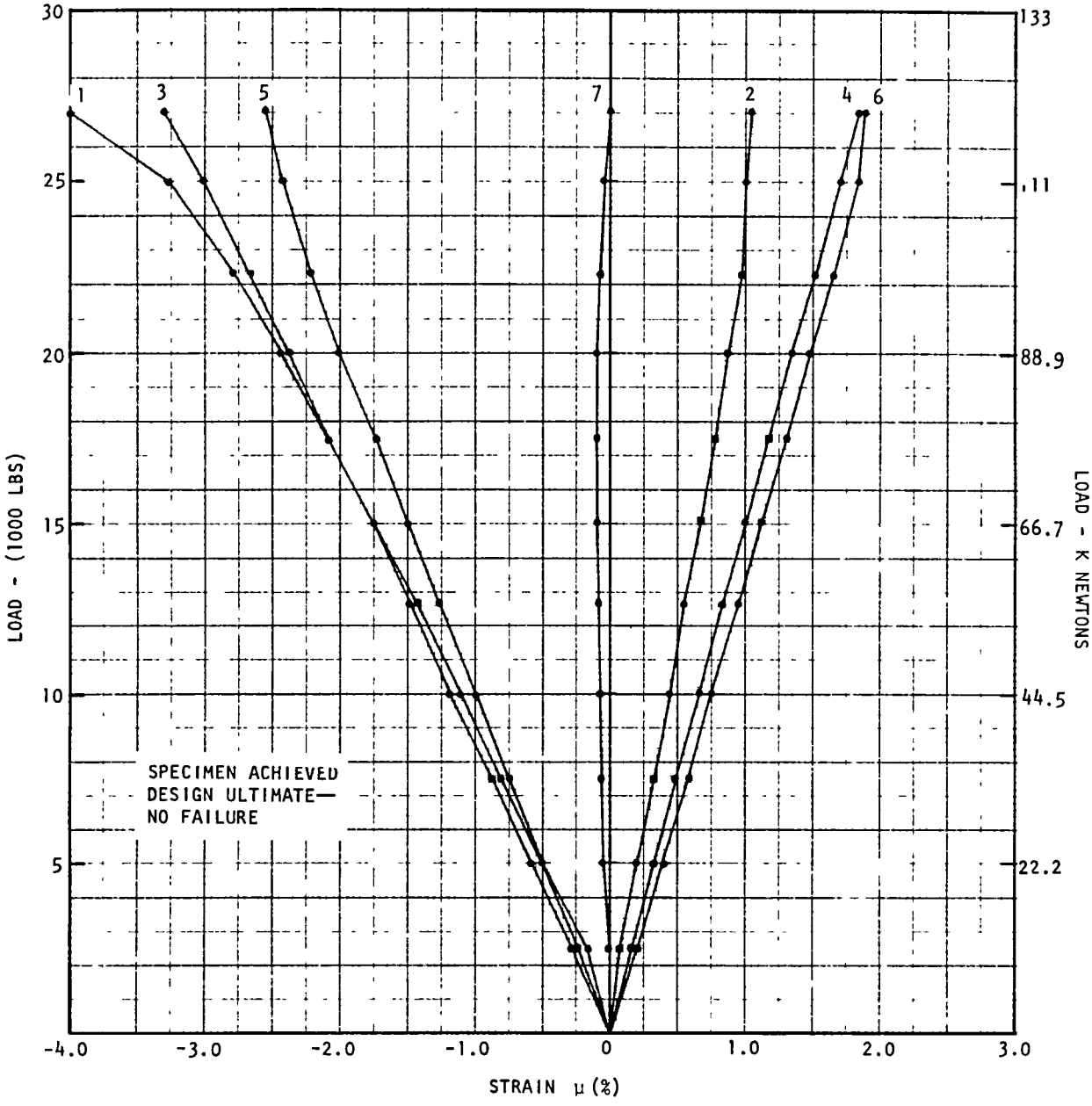
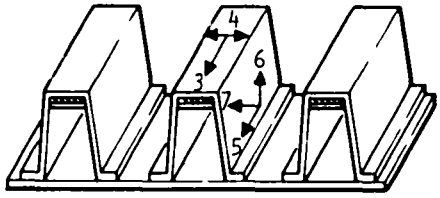


Figure 134. Compression Load/Strain Characteristics of Hat Stringer Stiffened Skin Element EX109/EX110B, Postcured Condition Tested at Room Temperature



GAGES 1 AND 2 ON LOWER
SKIN DIRECTLY OPPOSITE
3 AND 4

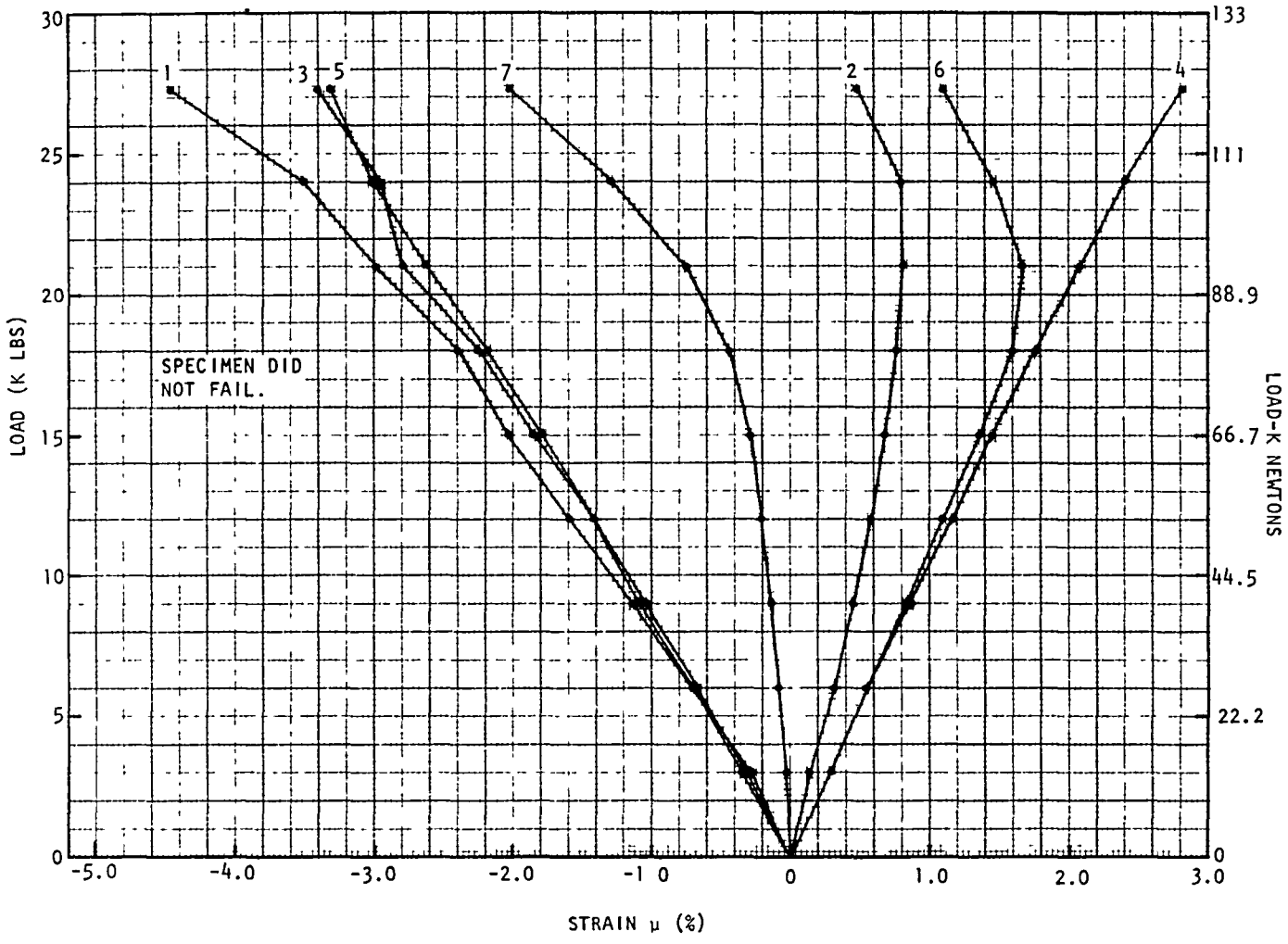
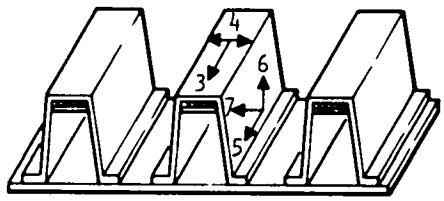
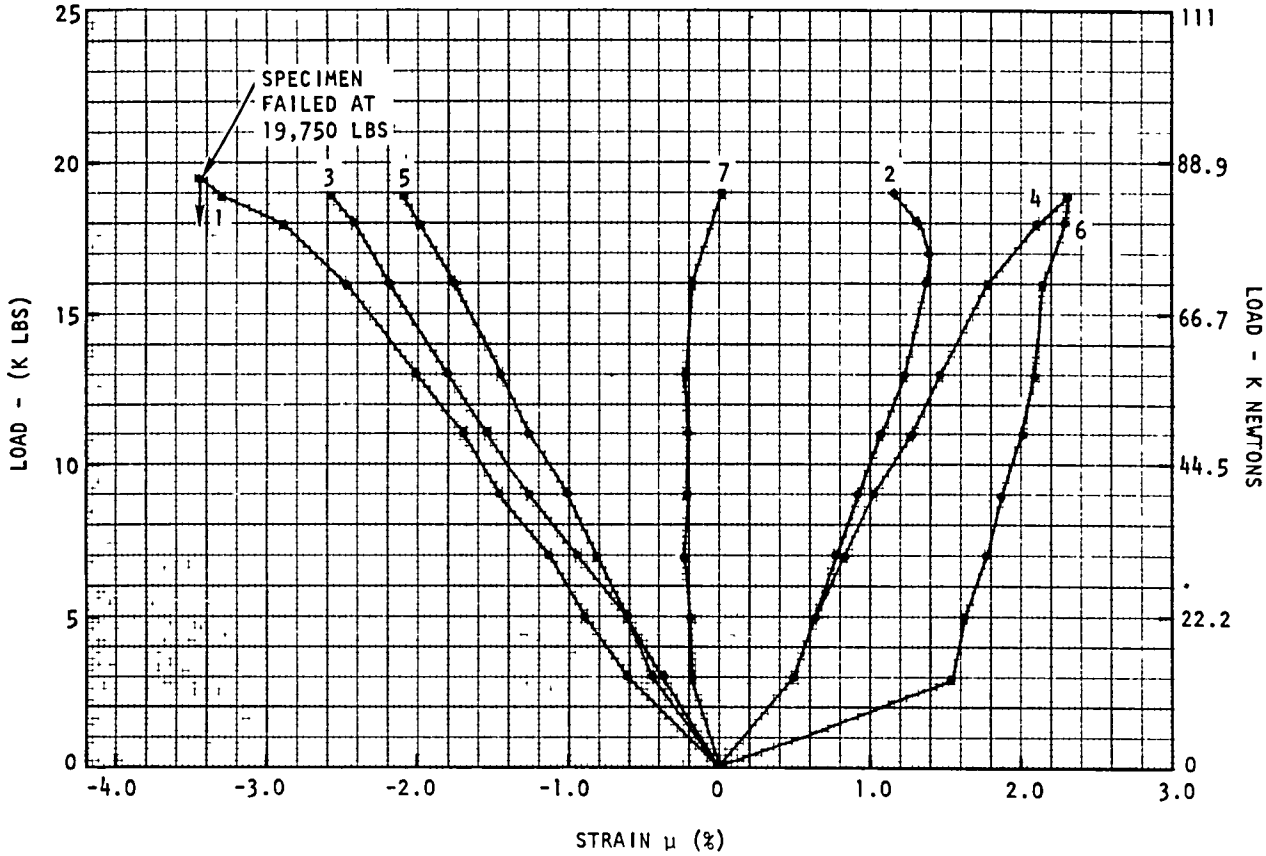


Figure 135. Load/Strain Characteristics of "Hat" Stringer Stiffened Skin Element EX195-2A Aged 125 Hours at 316 C (600 F) Tested at Room Temperature



GAGES 1 AND 2 ON LOWER
SKIN DIRECTLY OPPOSITE
3 AND 4



ıre 136. Compression Load/Strain Characteristics of "Hat" Stringer Stiffened Skin Element EX195-1PC at 316 C (600 F)

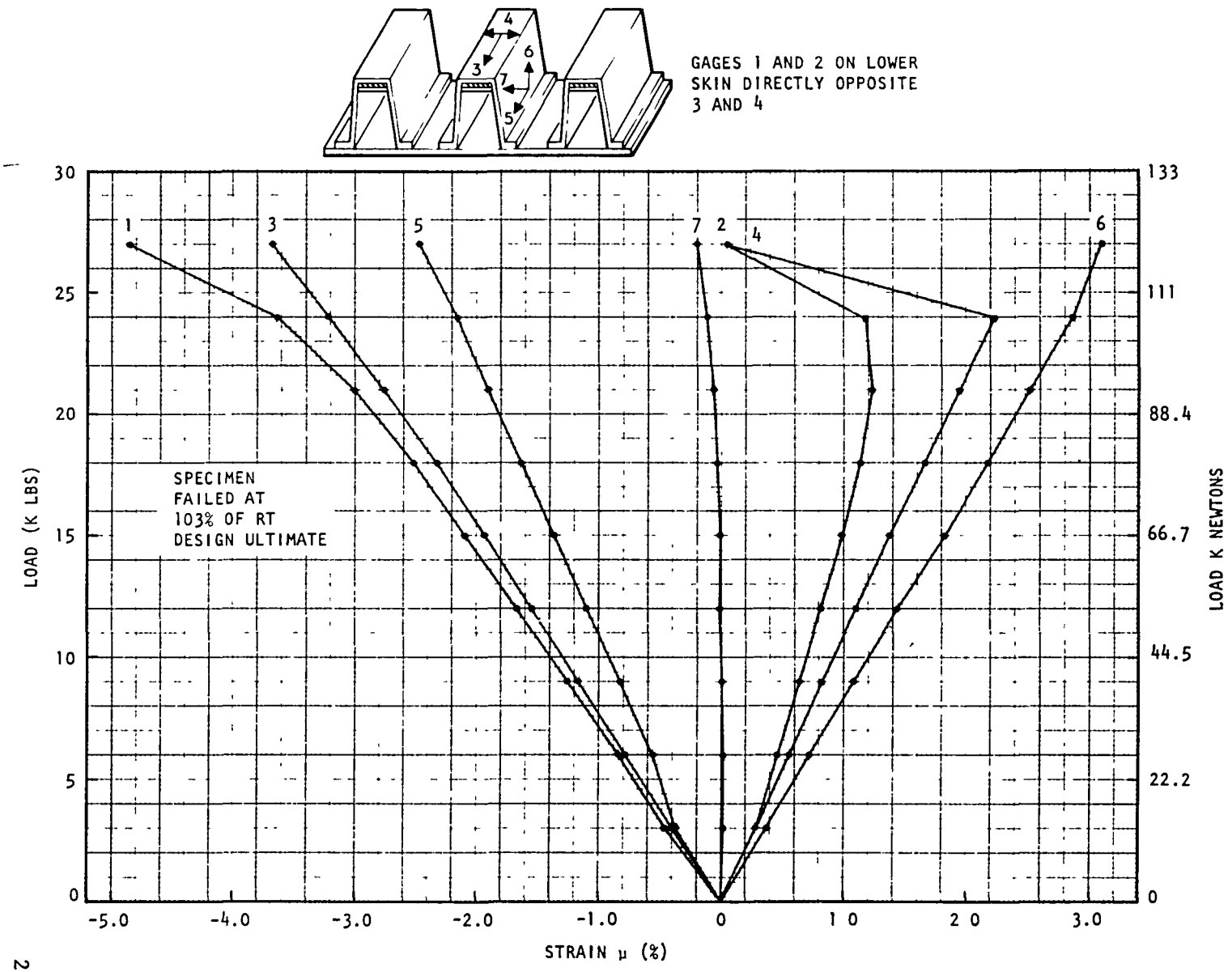
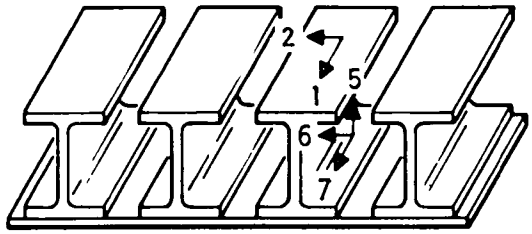


Figure 137. Compression Load/Strain Characteristics of "Hat" Stringer Stiffened Skin Element EX195-3A Aged for 125 Hours at 316 C (600 F) and Tested at 316 C (600 F)



3 AND 4 INSTALLED ON LOWER SKIN OPPOSITE 1 AND 2

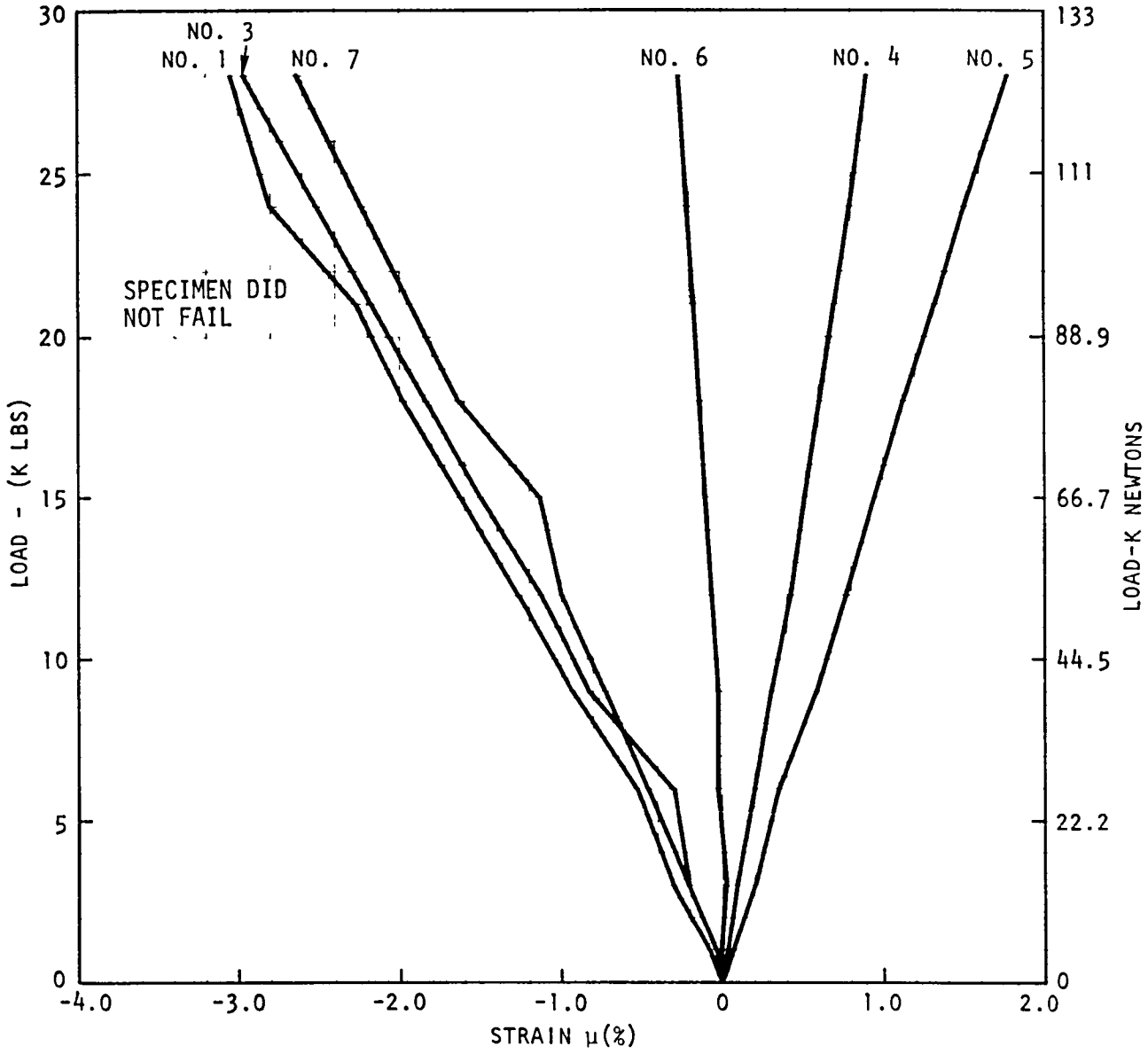
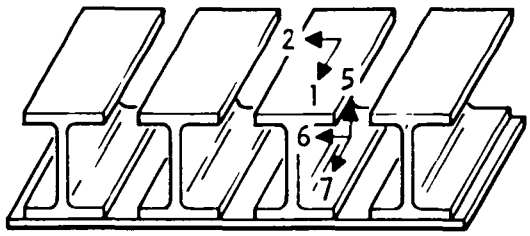


Figure 138. Load/Strain Characteristics of "I" - Stringer Stiffened Skin Element EX111/EX113 Tested at -132C (-270 °F)



STRAIN GAGES 3 AND 4 INSTALLED ON
LOWER SKIN OPPOSITE 1 AND 2

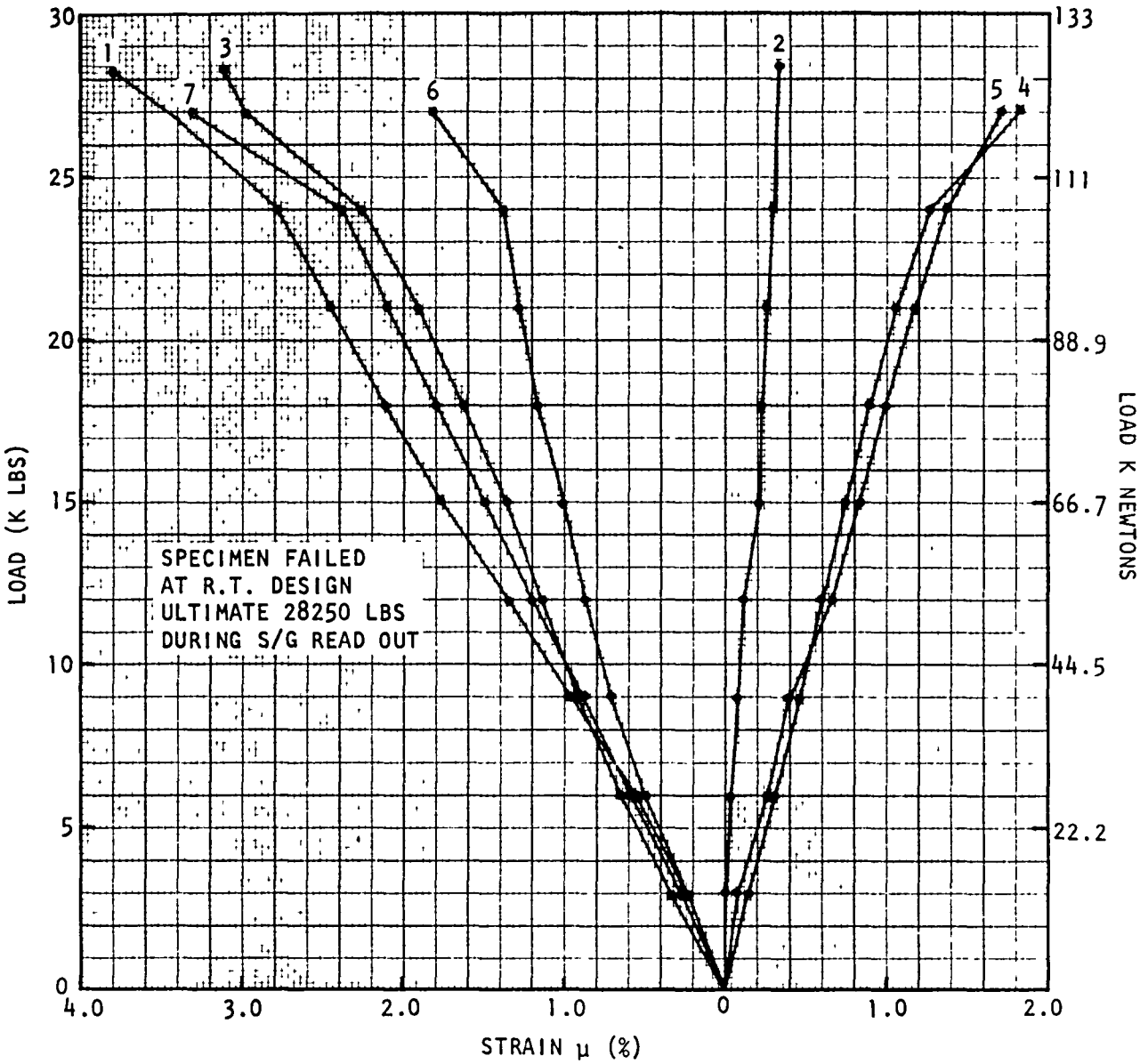
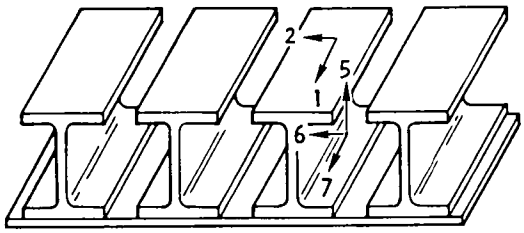


Figure 139. Compression Load/Strain Characteristics of "I" Stringer Stiffened Skin Element EX 194-4A Aged for 125 Hours at 316 C (600 F) and Tested at -132 C (-270 F)



STRAIN GAGES 3 AND 4
INSTALLED ON LOWER
SKIN OPPOSITE 1 AND 2

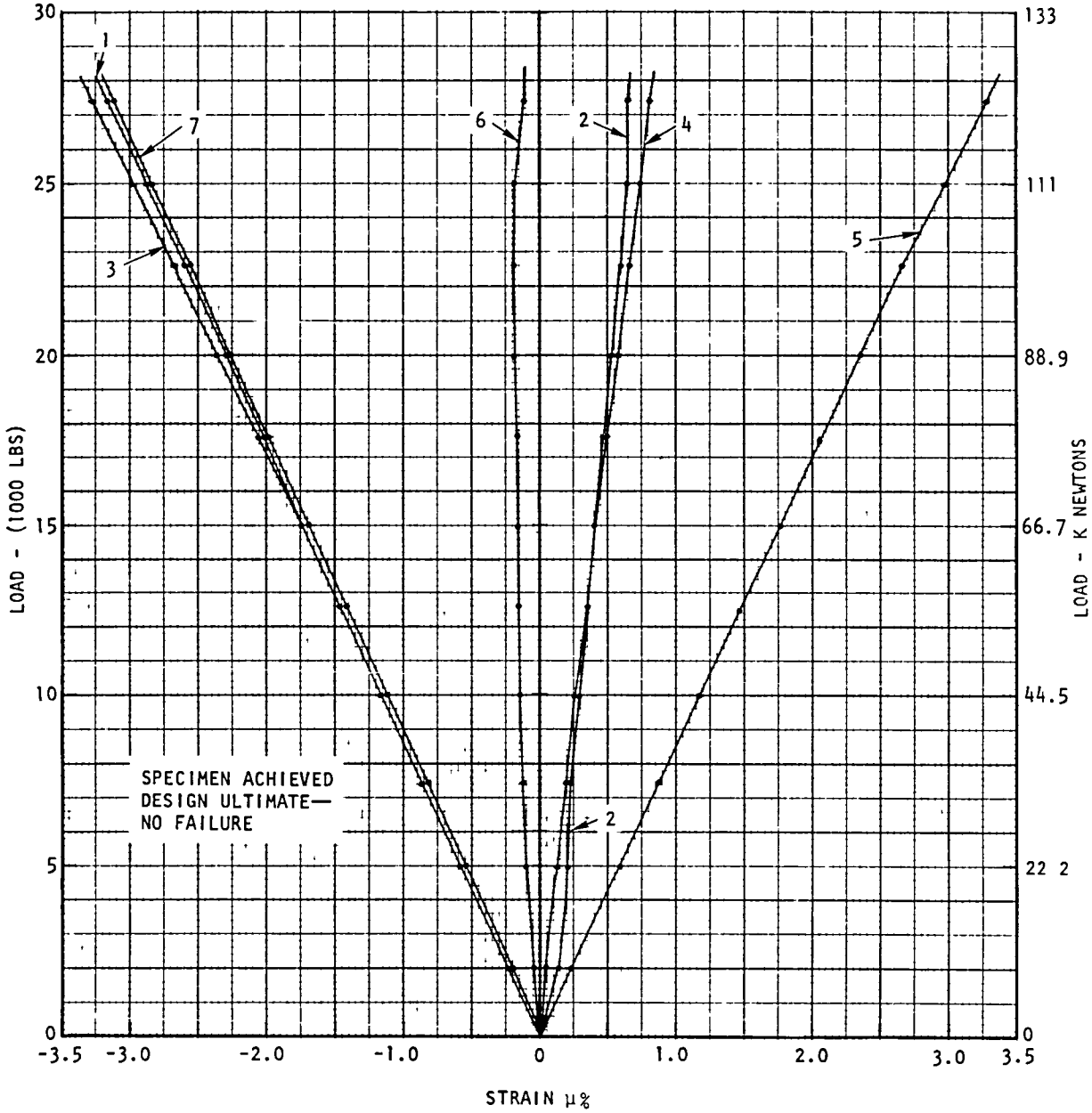
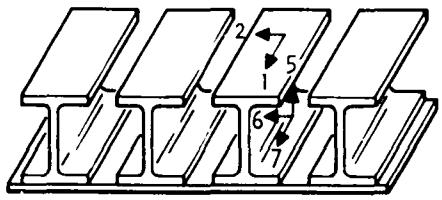


Figure 140. Compression Load/Strain Characteristics of "I"-Stringer Stiffened Skin Element EX111/EX113, Postcured Condition Tested at Room Temperature



3 AND 4 INSTALLED ON LOWER
SKIN OPPOSITE 1 AND 2

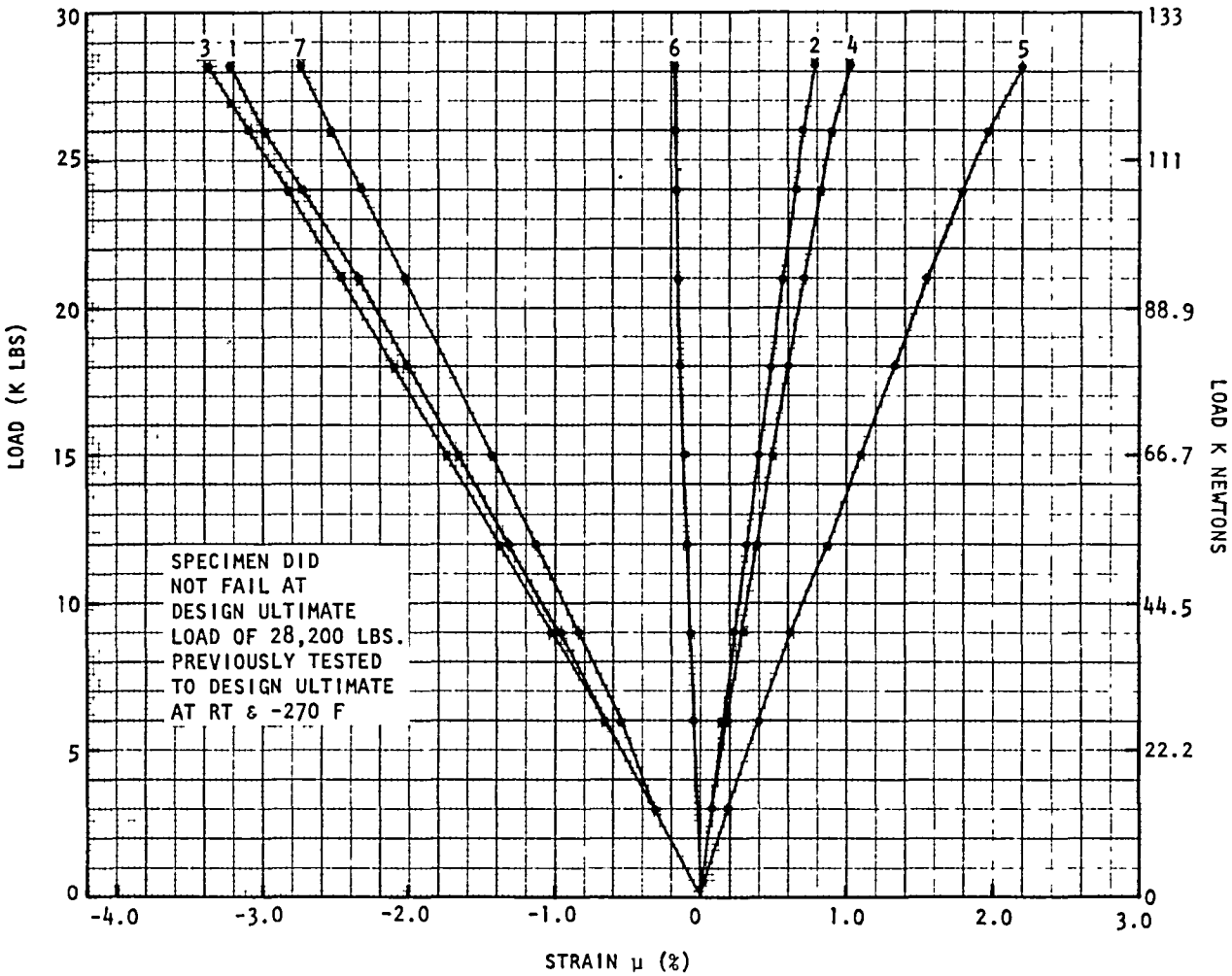
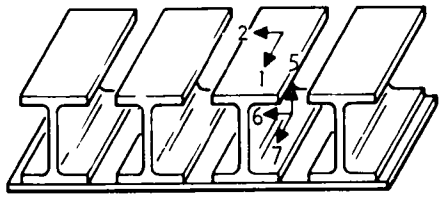


Figure 141. Compression Load/Strain Characteristics of "I" Stringer Stiffened Skin Element EX111/EX113, Postcured Condition, Tested at RT



STRAIN GAGES 3 AND 4 INSTALLED ON
LOWER SKIN OPPOSITE 1 AND 2

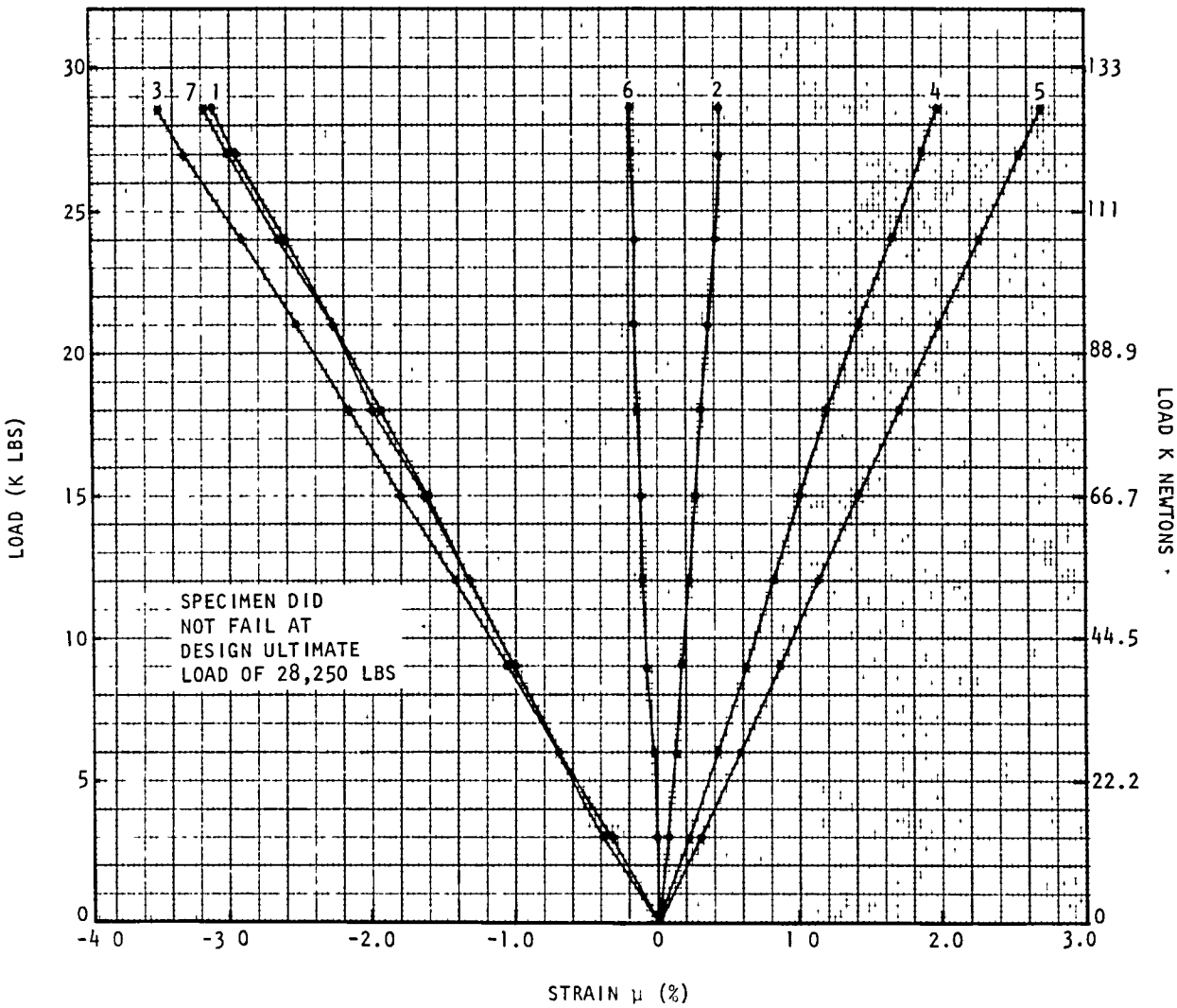
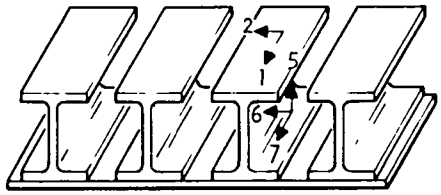


Figure 142. Compression Load/Strain Characteristics of "I" Stringer Stiffened Skin Element EX194-2A, Aged for 125 Hours at 316 C (600 F), and Tested at Room Temperature



GAGES 3 AND 4 INSTALLED ON LOWER
SKIN OPPOSITE 1 AND 2

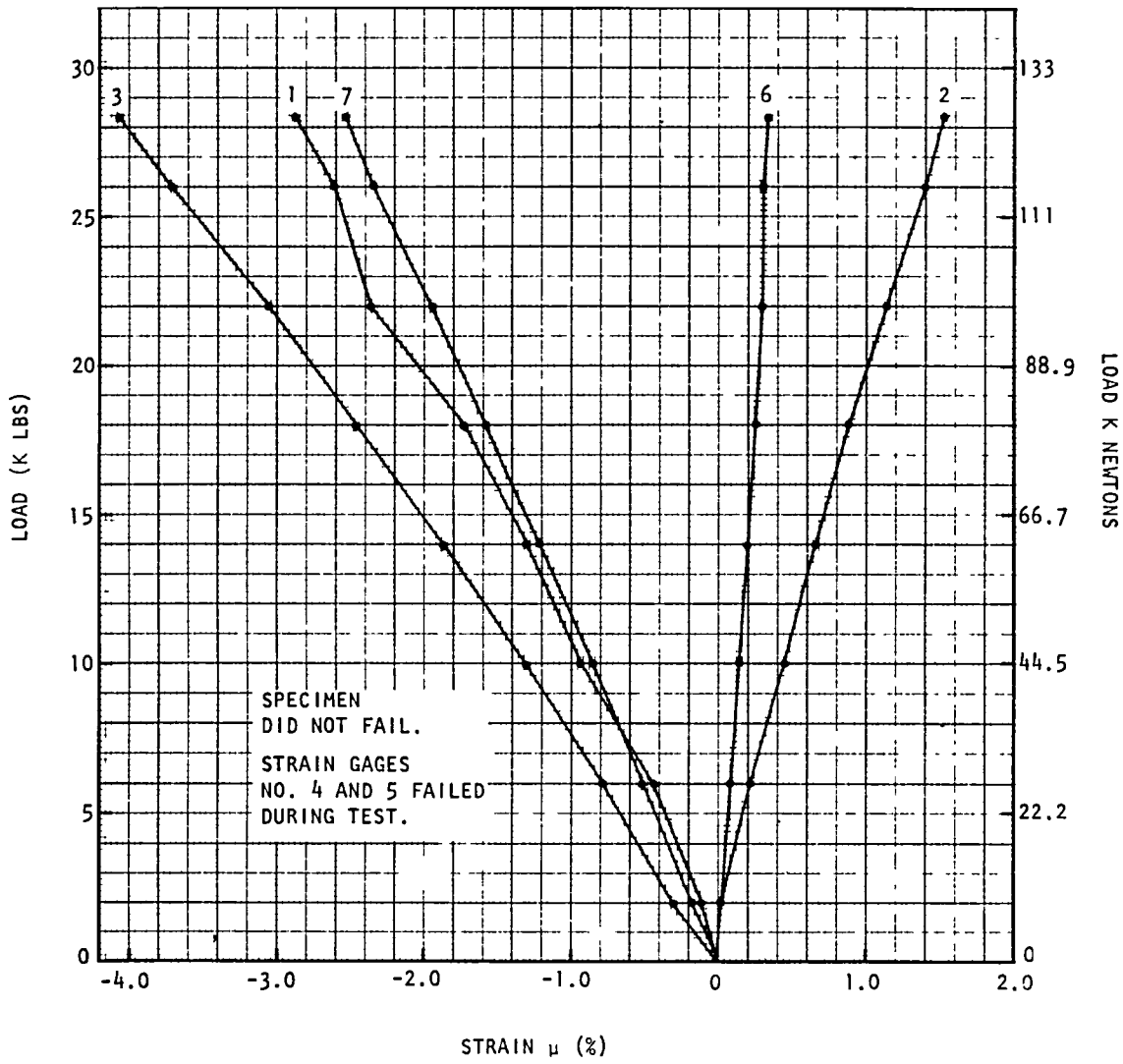
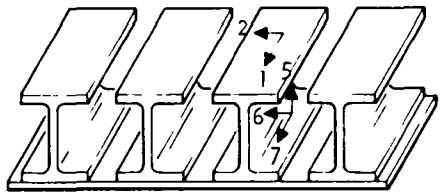


Figure 143. Load/Strain Characteristics of "I" Stringer Stiffened Skin Element EX194-1 Postcured Condition, Tested at 316 C (600 F).



STRAIN GAGES 3 AND 4 INSTALLED
ON LOWER SKIN OPPOSITE 1 AND 2

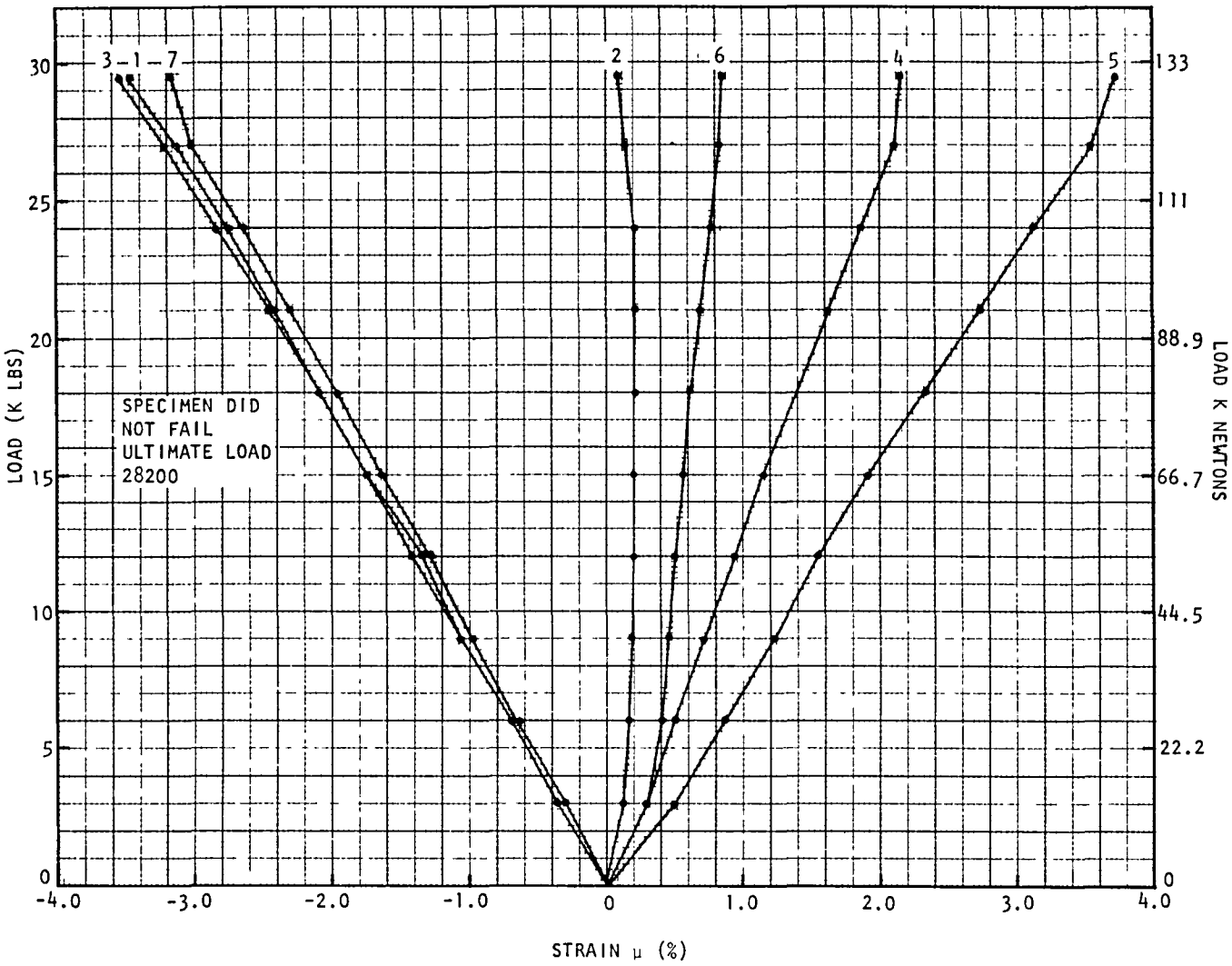


Figure 144. Load/Strain Characteristics of "I" Stringer Stiffened Skin Element EX194-3A, Aged for 125 Hours at 316 C (600 F) and Tested at 316 C (600 F).

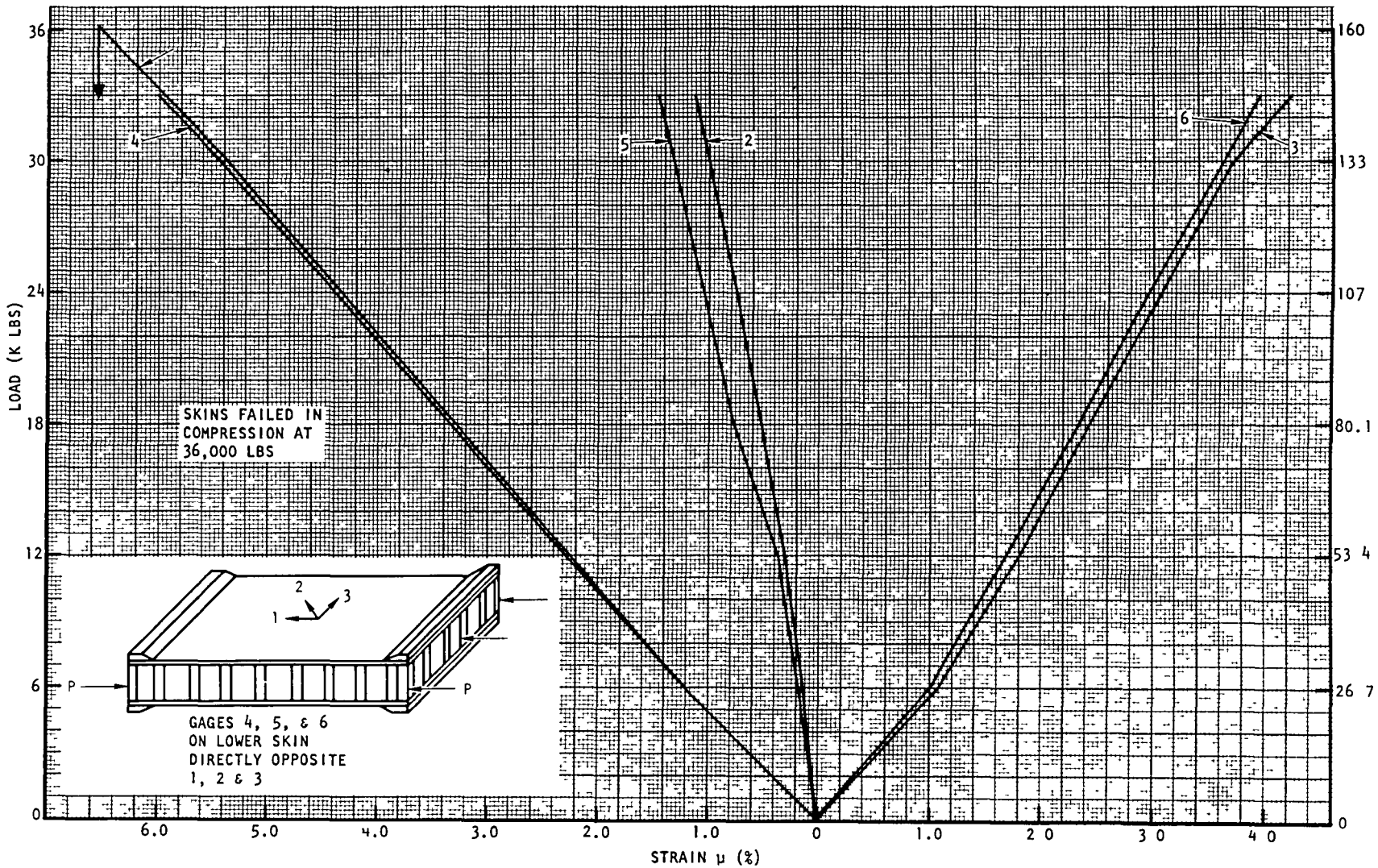


Figure 145. Load/Strain Characteristics of Sandwich Element EX241-1, Postcured Condition, Tested at -132 C (-270 F)

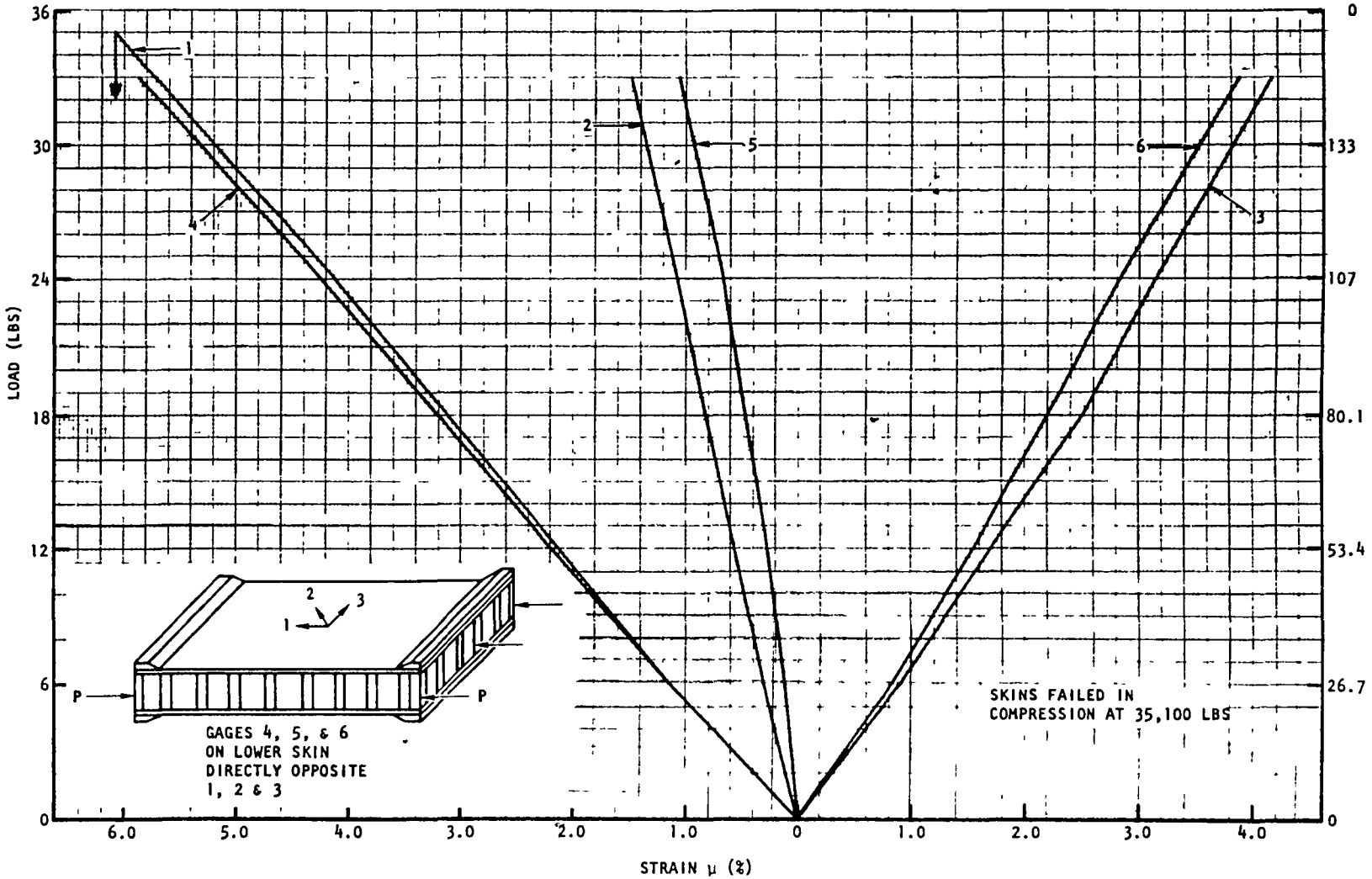


Figure 146. Load/Strain Characteristics of Sandwich Element EX241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)

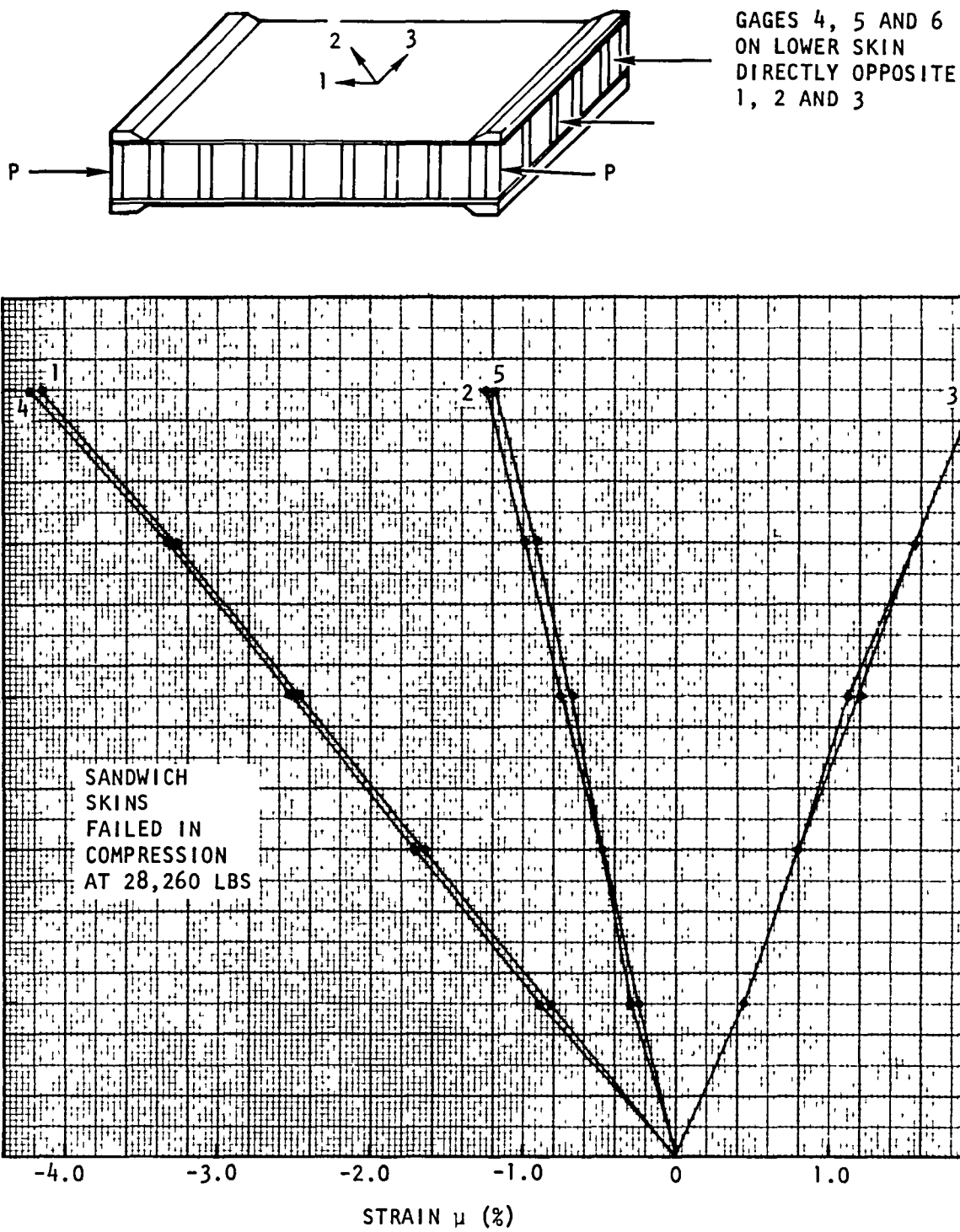


Figure 147. Load/Strain Characteristics of Sandwich Panel Element EX150-1, Postcured Condition, Tested at Room Temperature

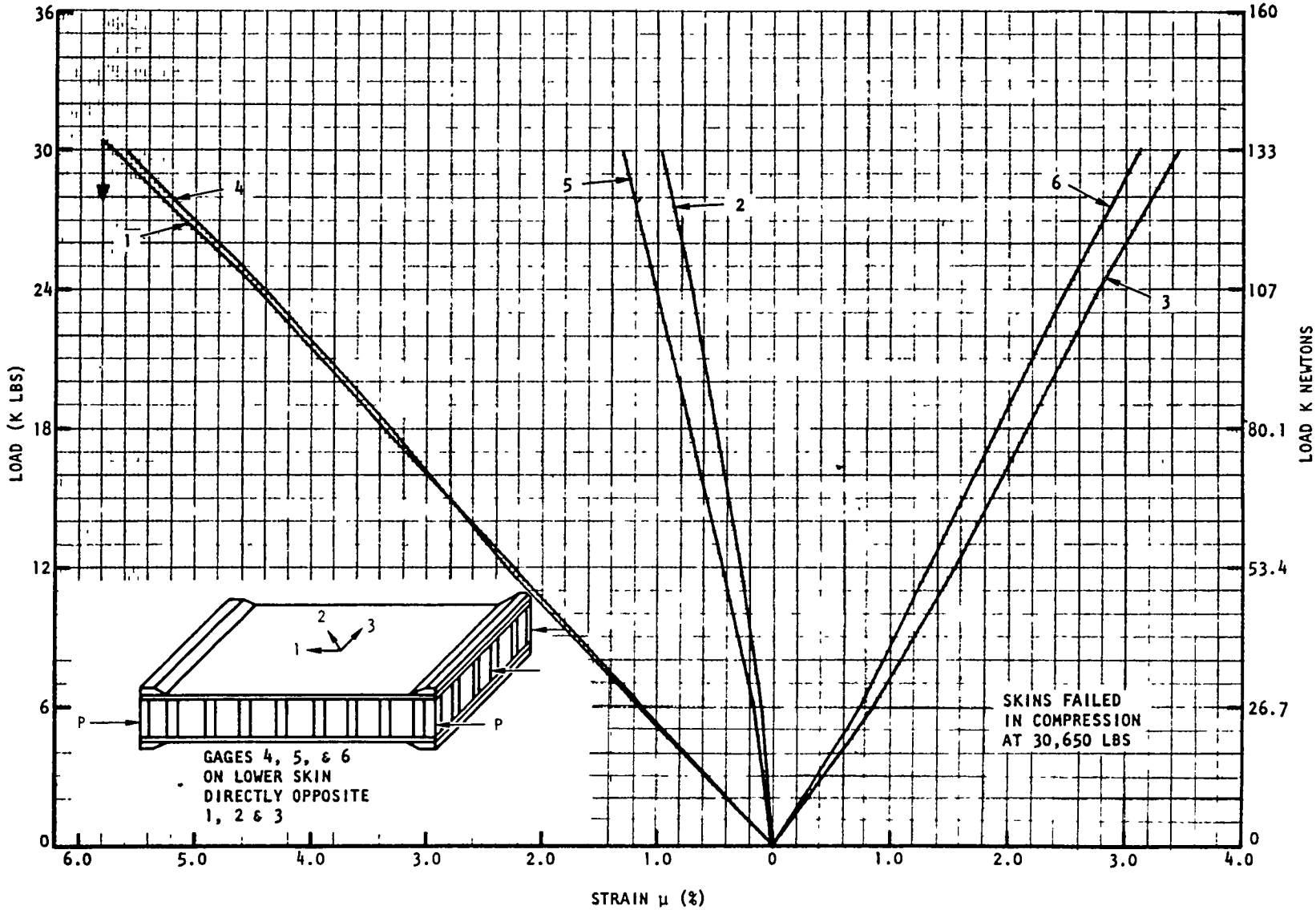


Figure 148. Load/Strain Characteristics of Sandwich Element EX241-2A Aged 125 Hours at 316 C (600 F) Tested at R.T.

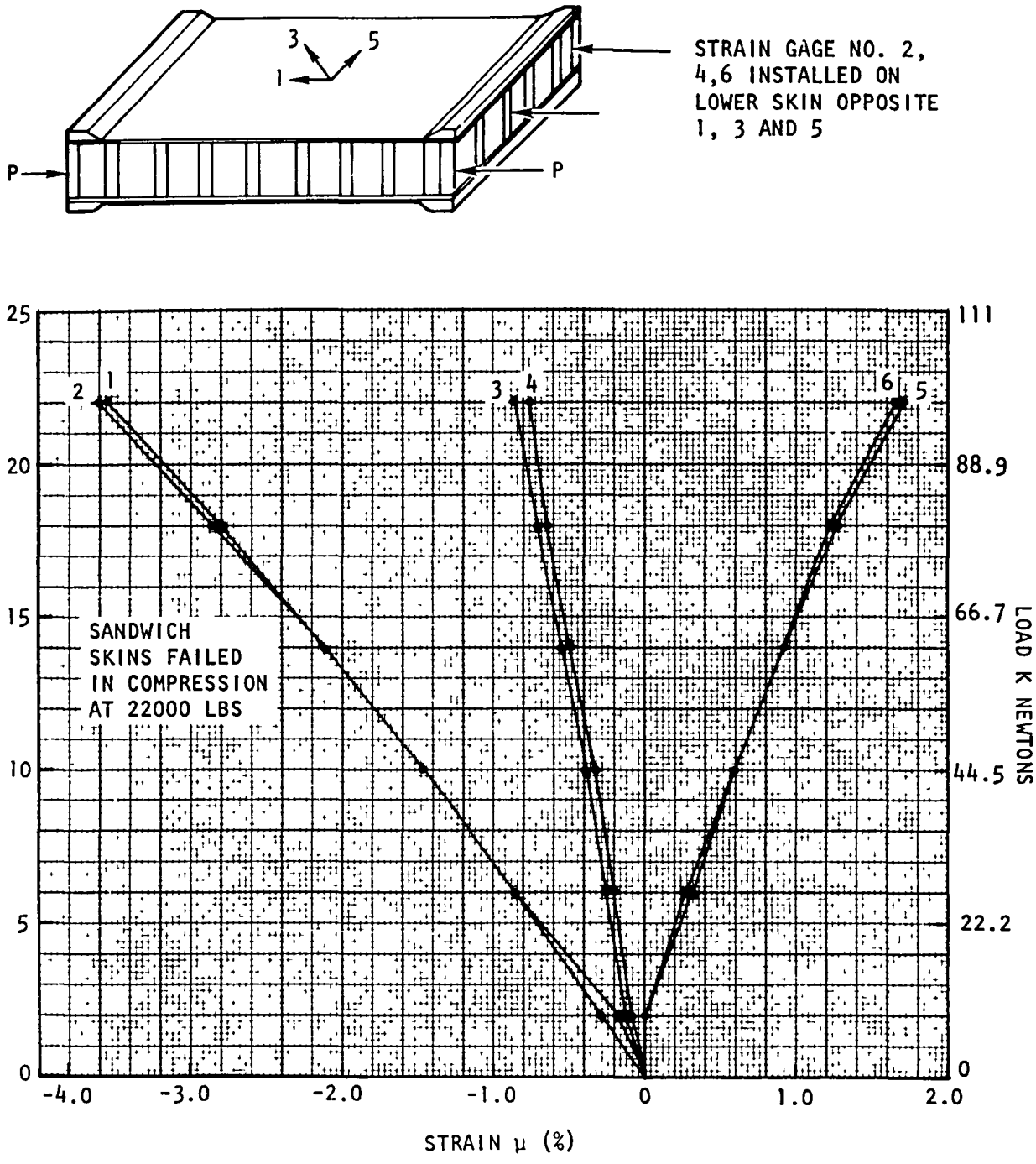


Figure 149. Load/Strain Characteristics of Sandwich Element, EX150-2 Postcured Condition, Tested at 316 C (600 F)

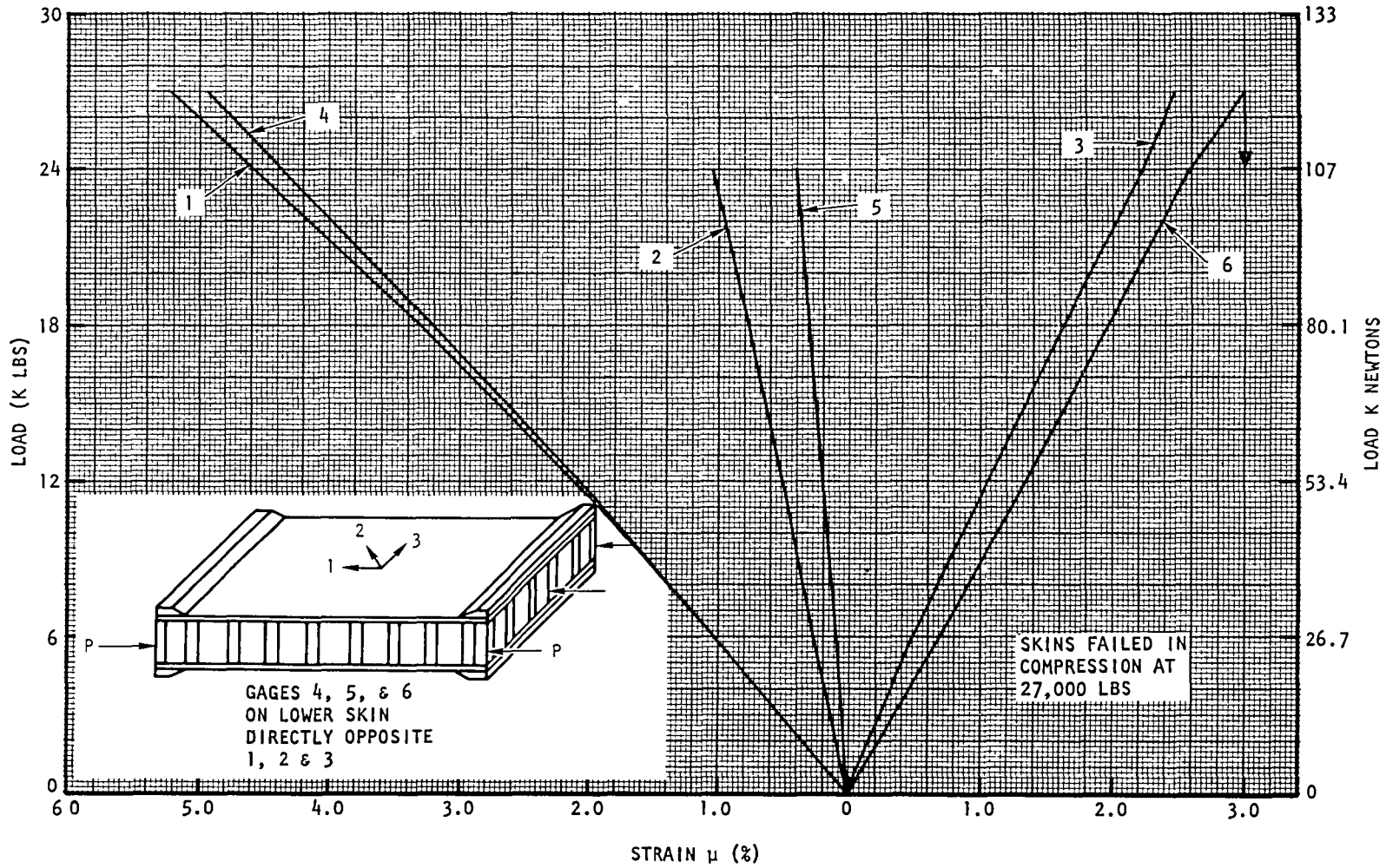


Figure 150. Load/Strain Characteristics of Sandwich Element EX241-4A Aged 125 Hours at 316 C (600 F), Tested at 316 C (600 F)

Figure 151. "Hat" Stringer Element EX109/EX110B Local Compression Failure, -132°C (-270°F) Test, Postcured.

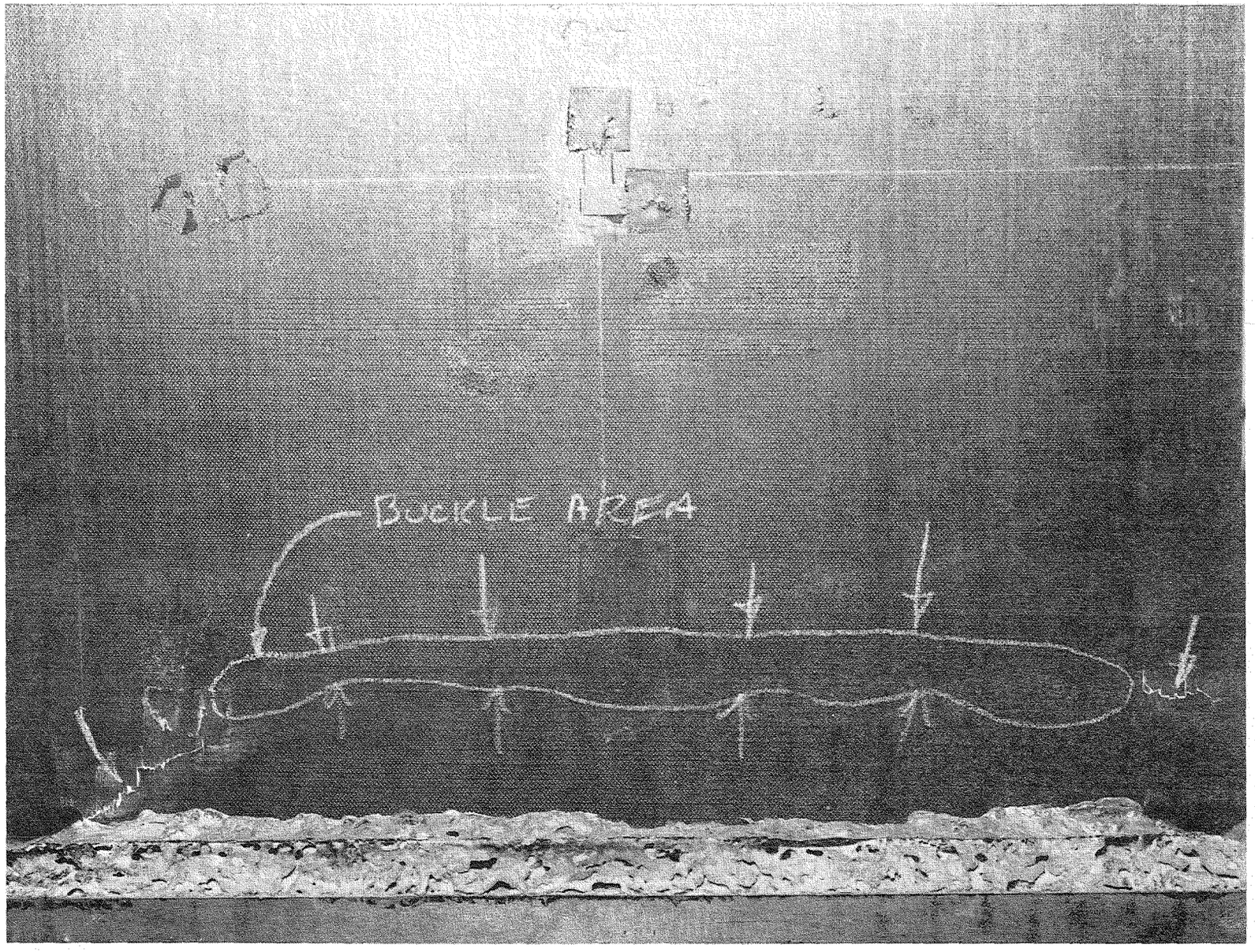


Figure 152. "Hat" Element EX195-1PC Showing Skin Compression and Buckling Failures, 316 C (600 F) Test, Postcured

A800331 G-10C

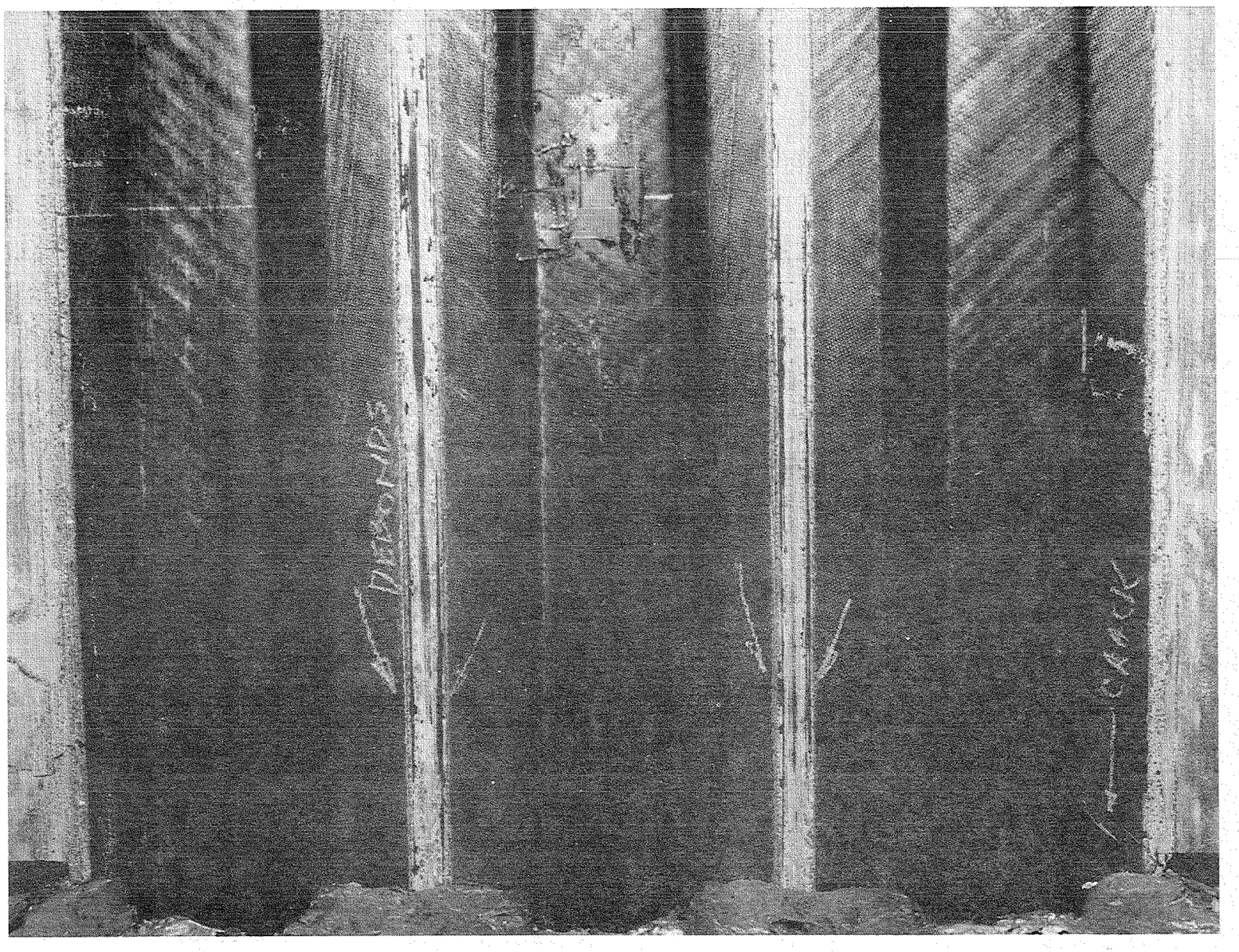


Figure 153. "Hat" Element EX195-1PC Showing Local Debonds and Flange Compression Modes, 316°C (600°F) Test, Postcured Condition.

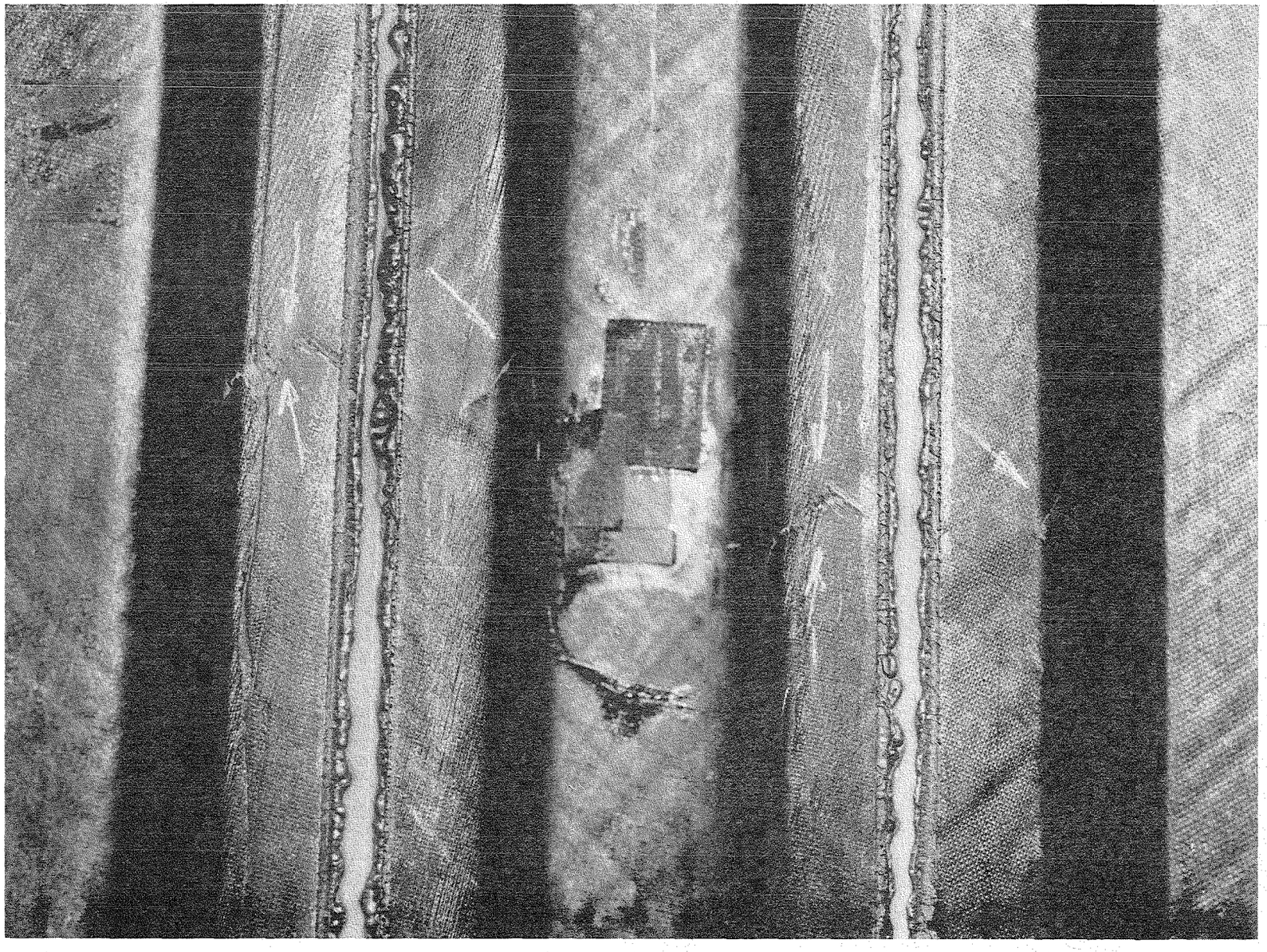


Figure 154. "Hat" Element EX195-3A Showing Local Flange Compression Modes, 316°C (600°F) Aged Condition.

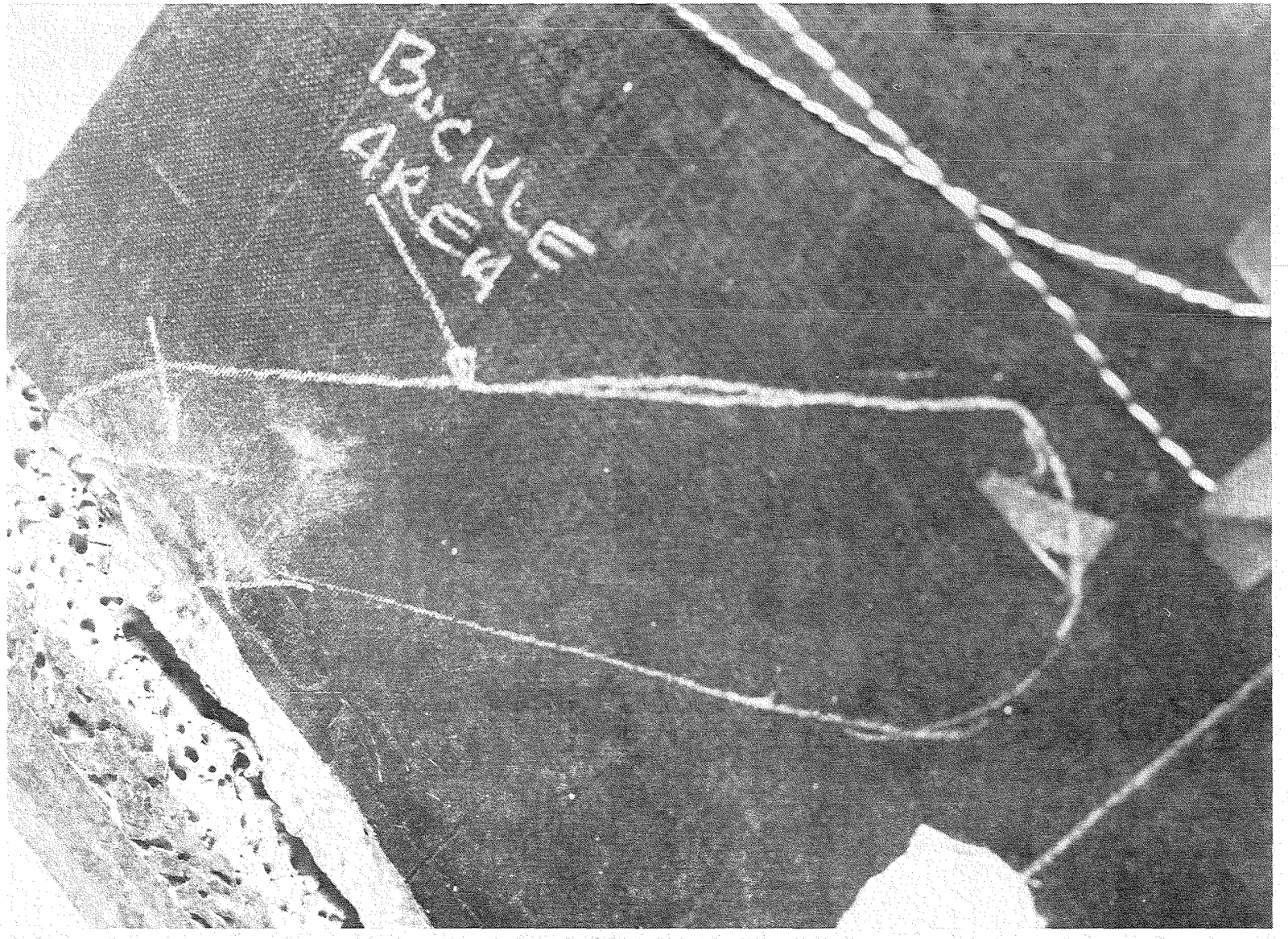


Figure 155. "Hat" Element EX195-3A, Local Skin Compression and Buckling Failure, 316°C (600°F) Test, Aged Condition.

Figure 156. "I" Element EX194-1PC Showing Local Skin Compression Failure, 316 C (600 F) Test Postcured

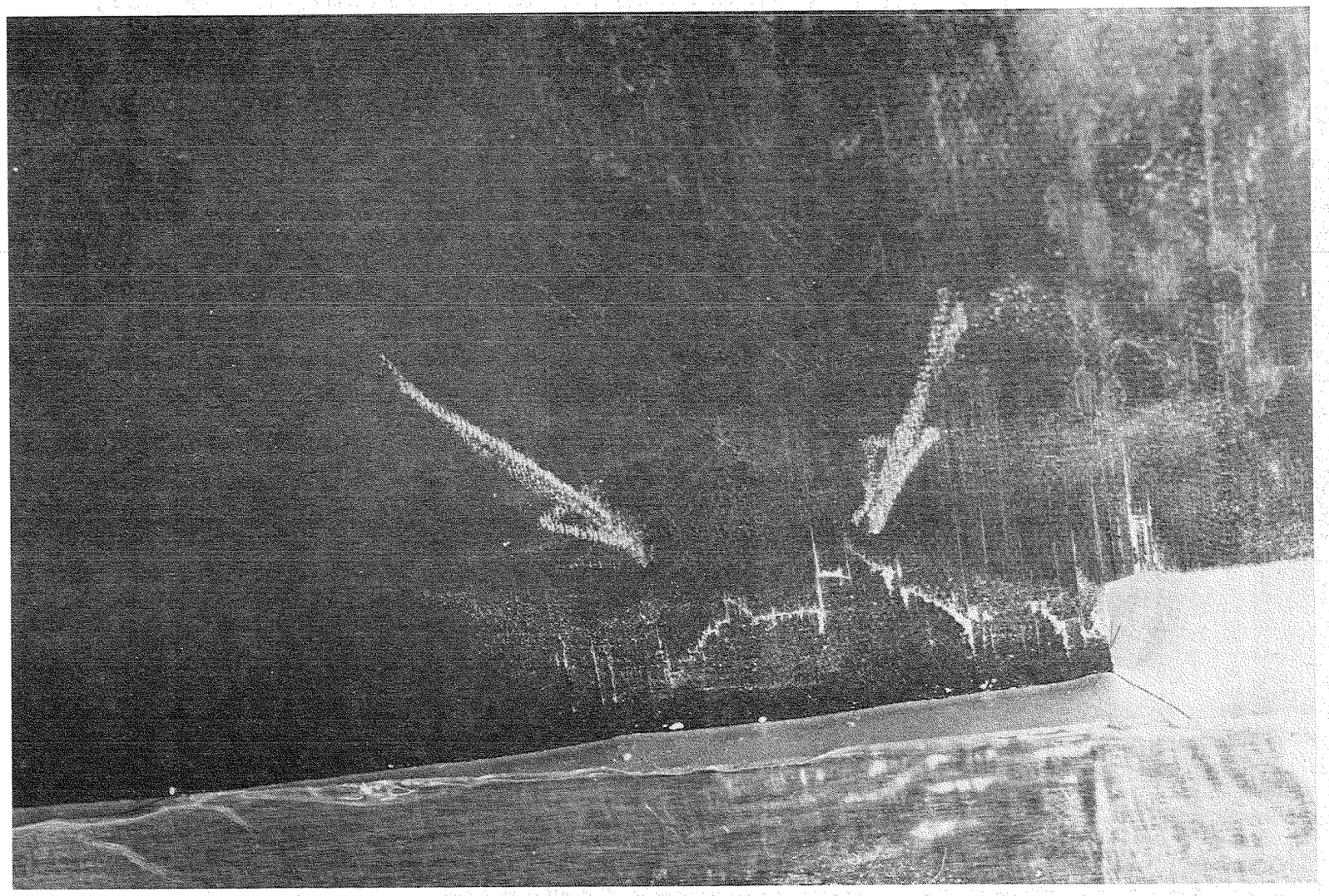


Figure 157. Skin Compression Failure "I" Stringer Element EX194-4A -132°C (-270°F) Test, Aged Condition.

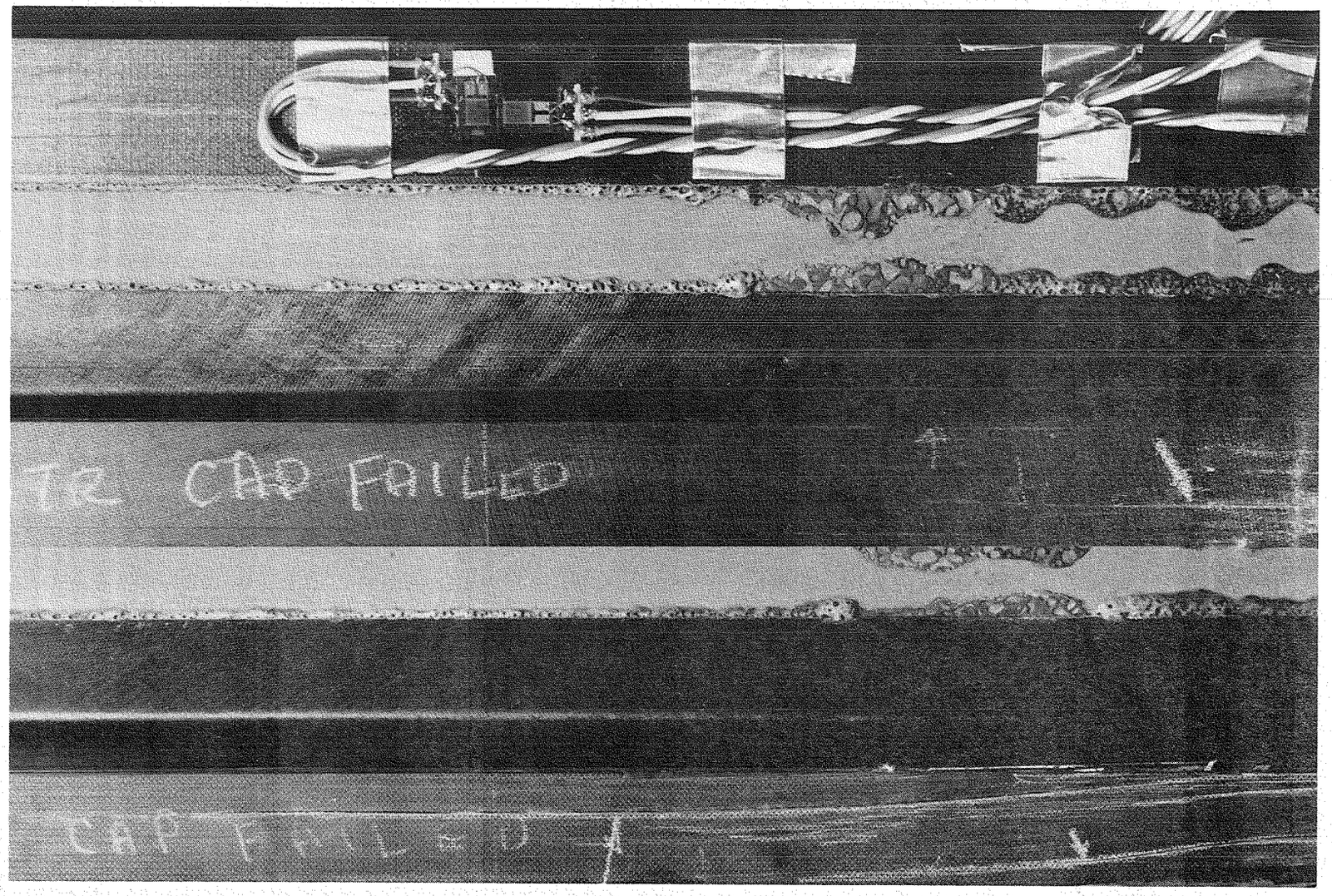


Figure 158. Cap Compression Failure, "I" Element EX194-4A, -132°C (-270°F) Test, Aged Condition

A800331 G-3C

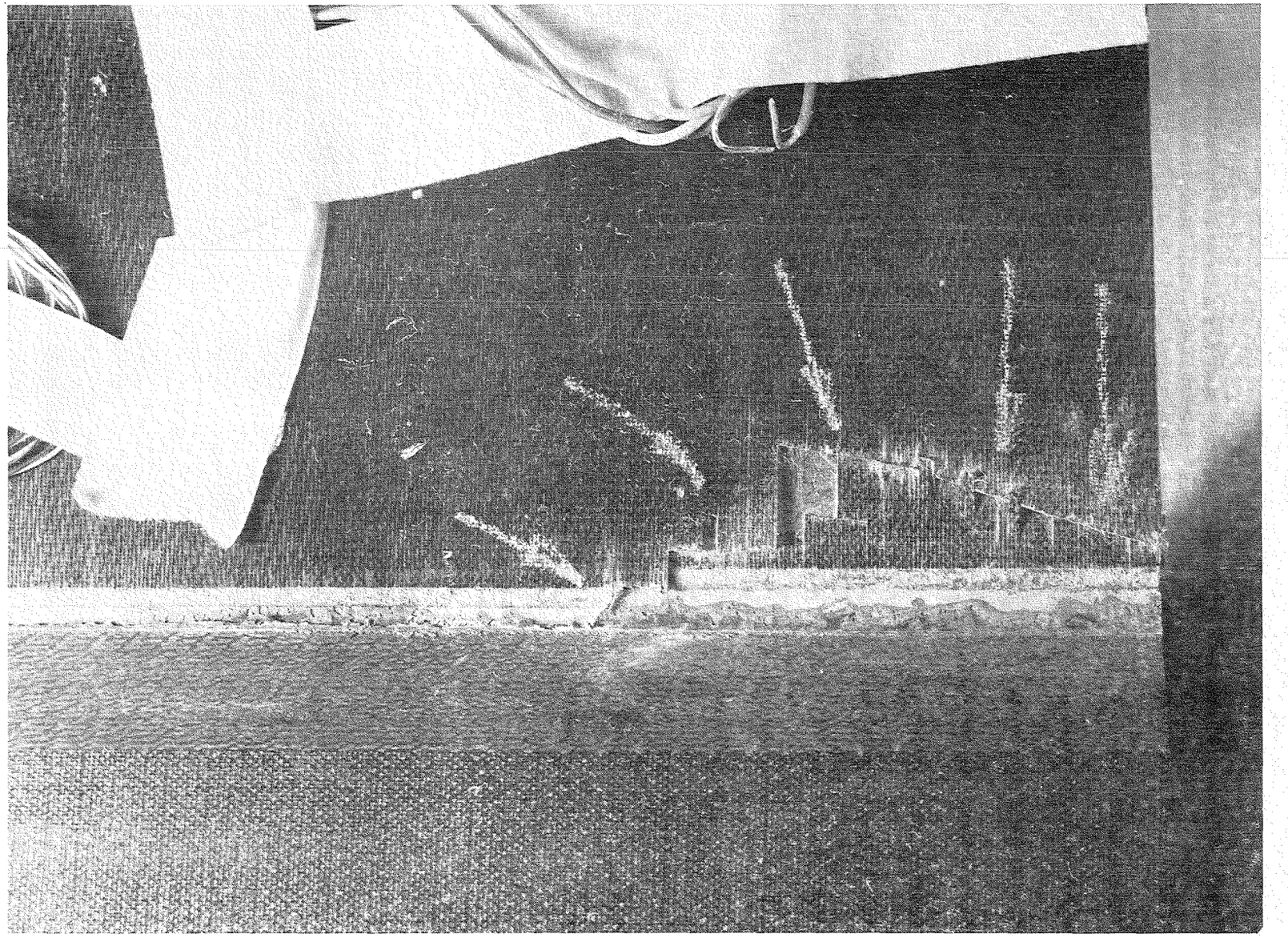


Figure 159. Local Compressive Failure—Sandwich Element EX150-1, RT Test, Side 2, Postcured.

A800331G-5C

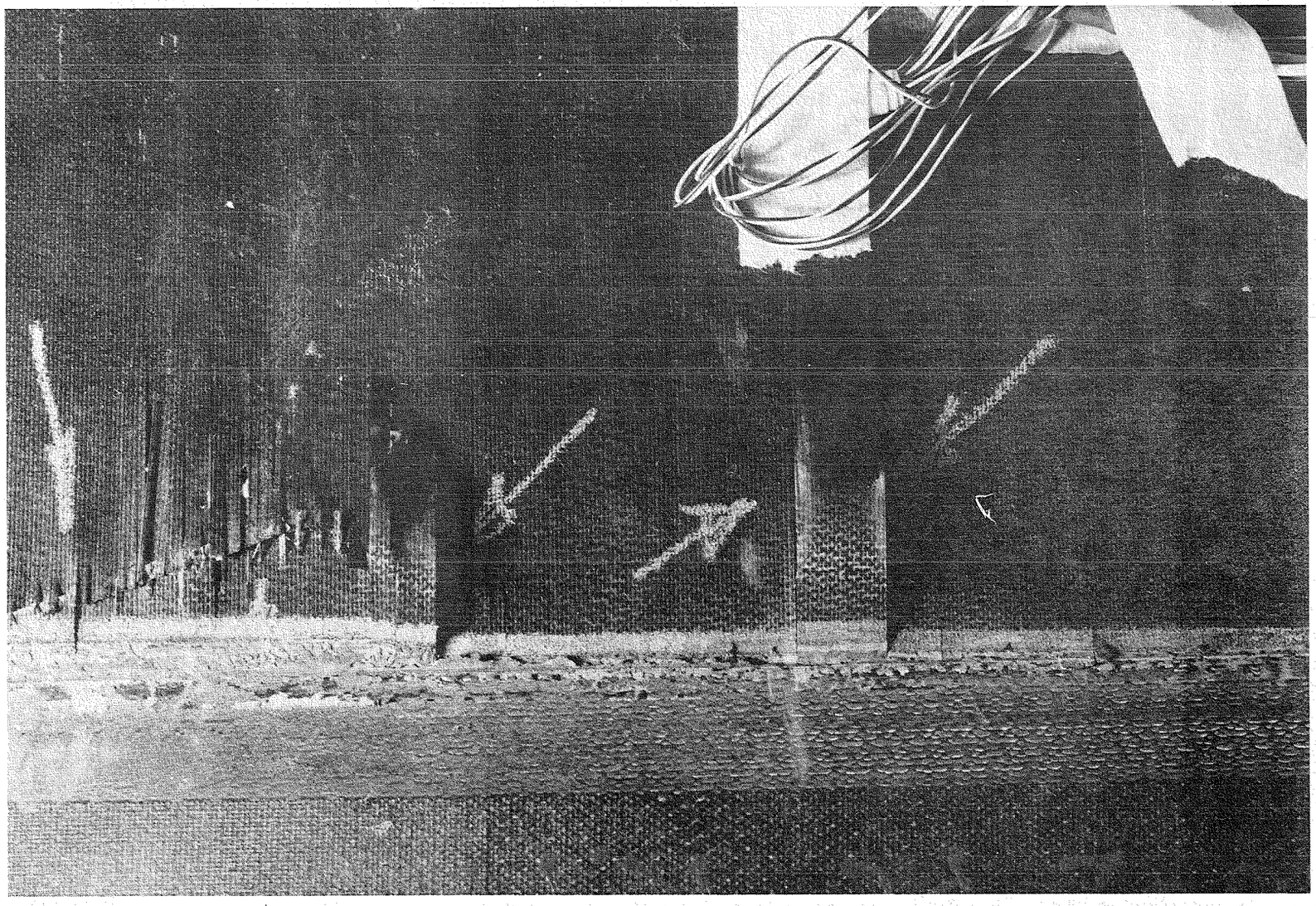


Figure 160. Compressive Failure Mode Sandwich Element EX150-1, RT Test, Side 1, Postcured.

A800331 G-8 C

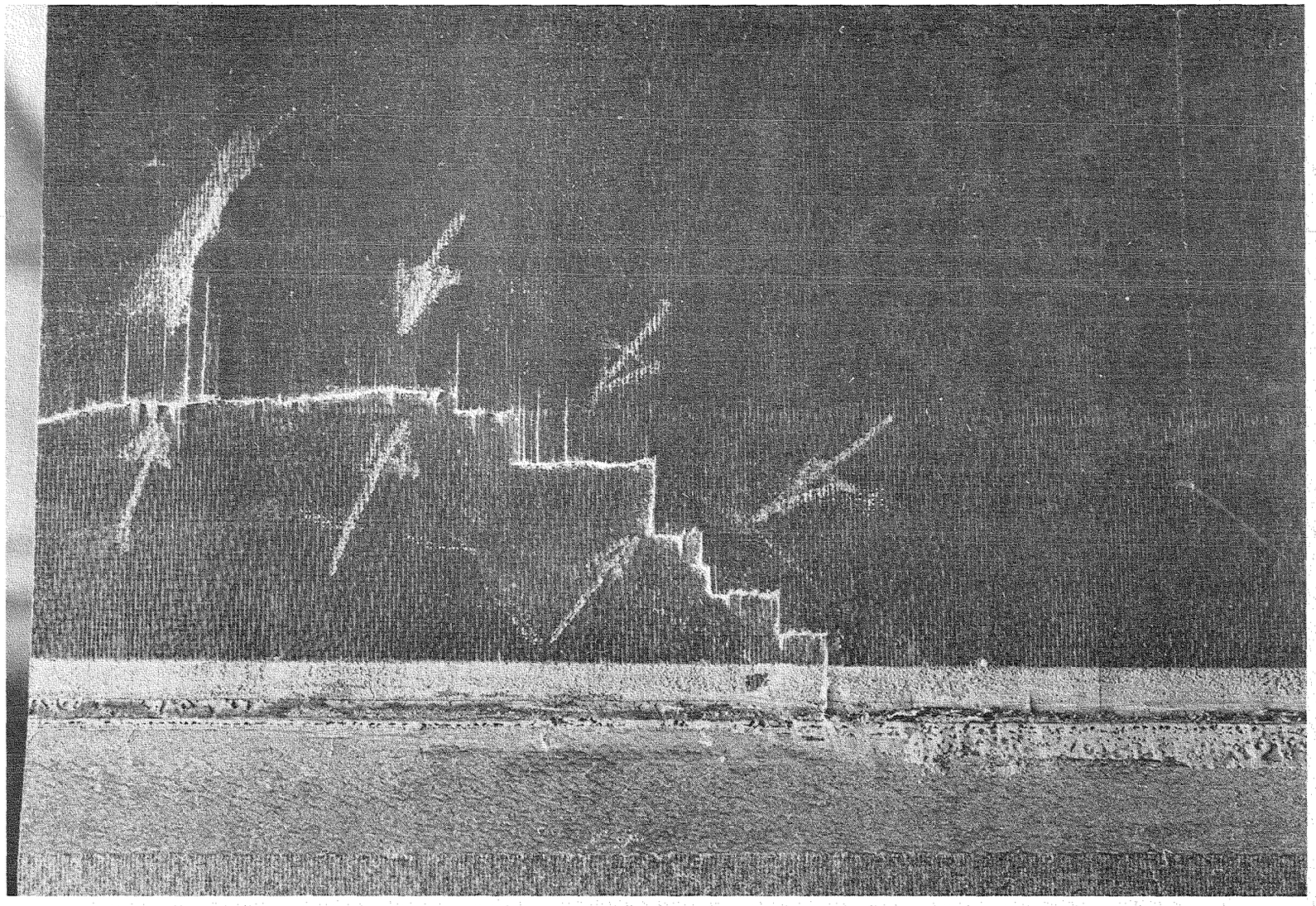
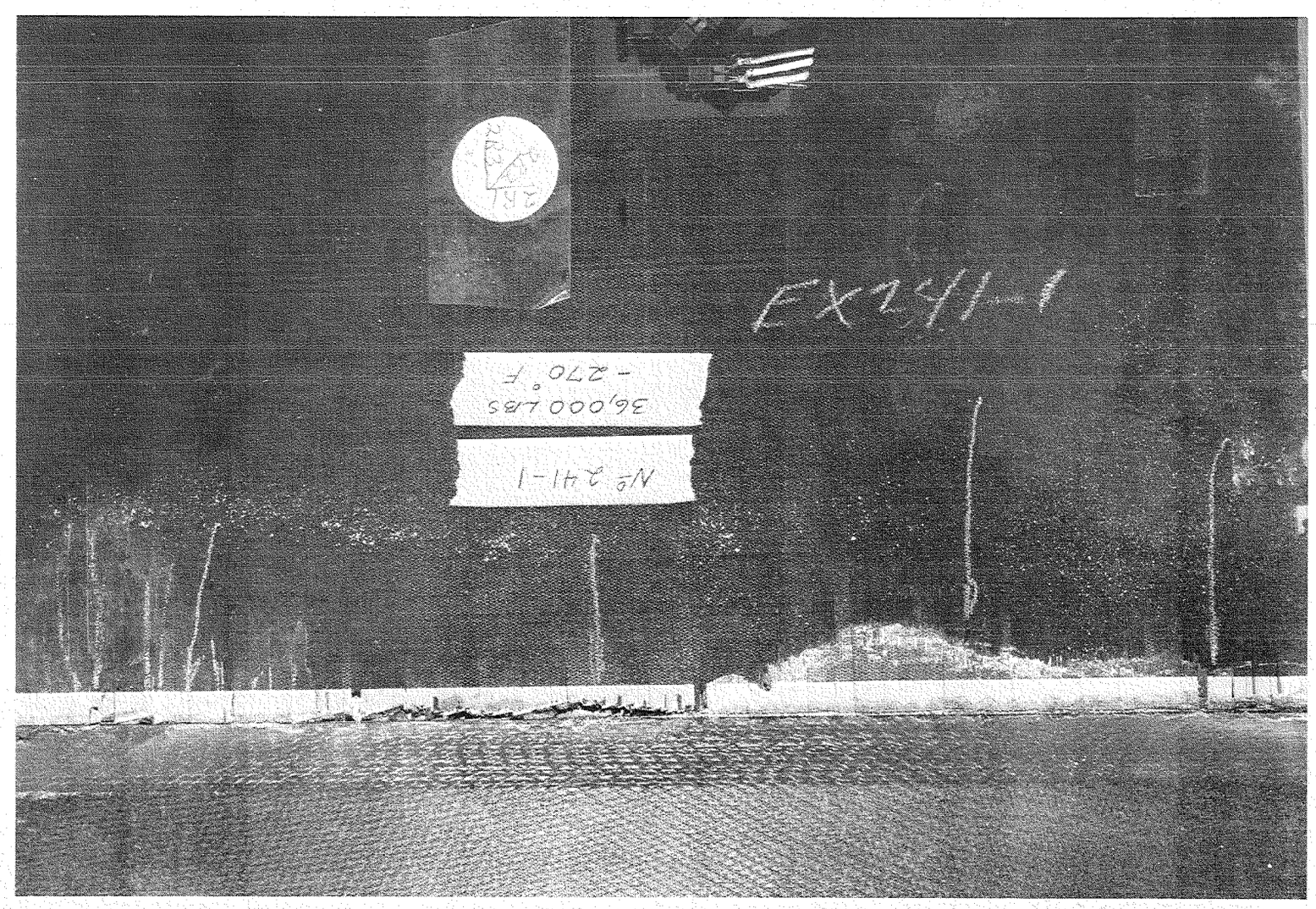
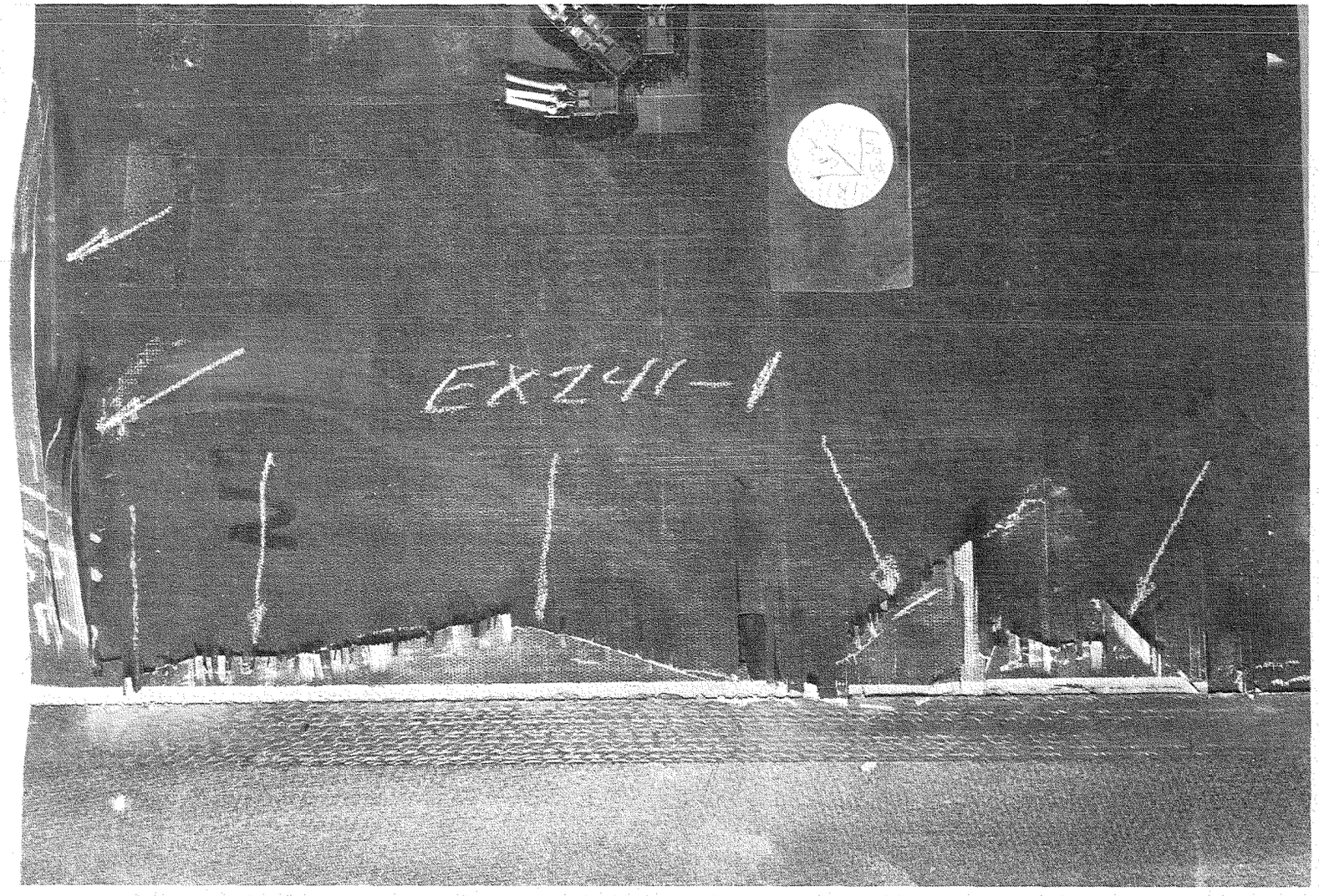


Figure 161. Local Compressive Failure Sandwich Element EX150-2, 316°C (600°F) Test, Postcured.



SIDE 1 FAILING LOAD: 160 KN (36,000 LB)

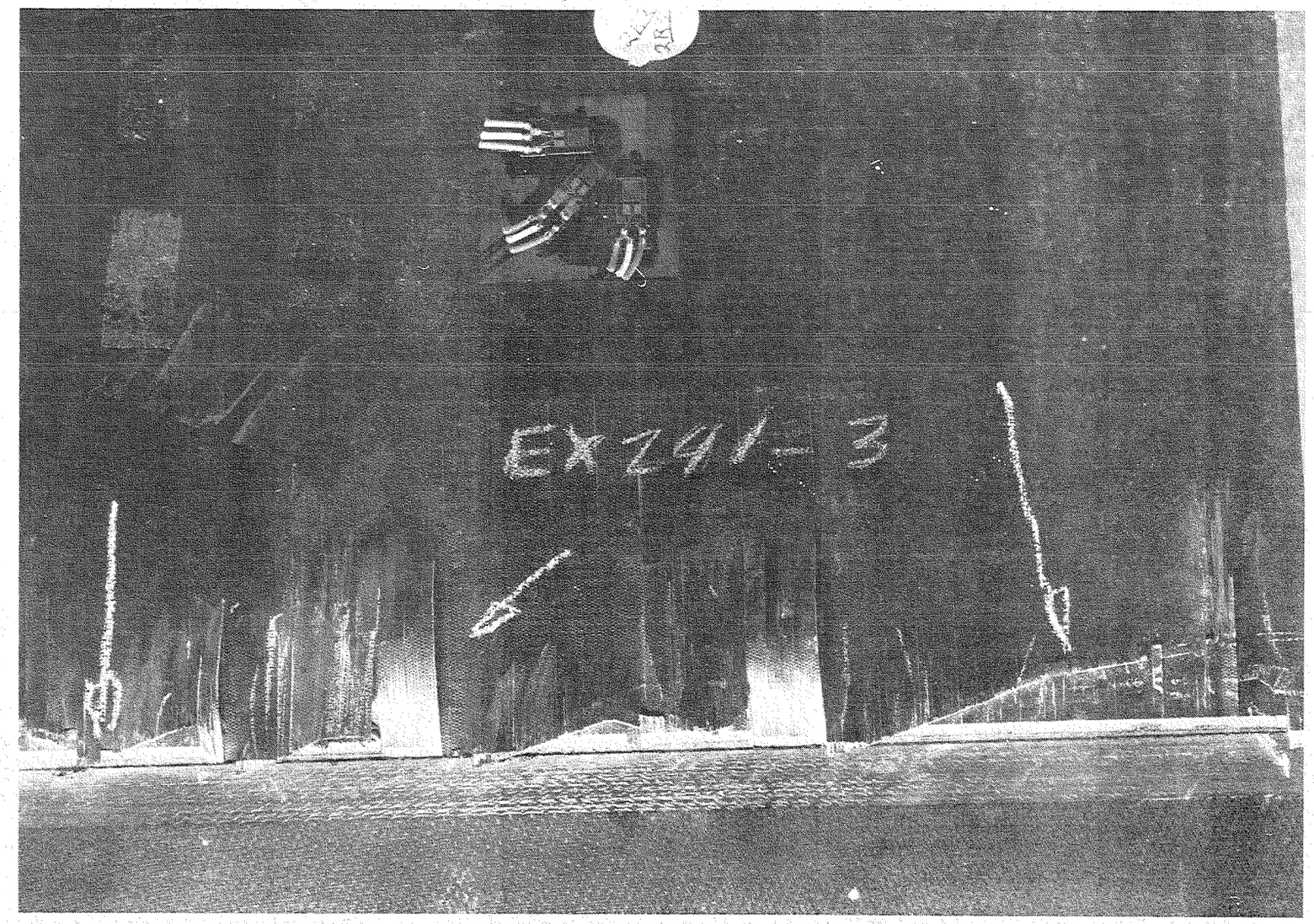
Figure 162A. Failure Modes of Sandwich Element EX 241-1, Postcured Condition, Tested at - 132 C (-220 F)



SIDE 2

FAILING LOAD: 160 KN (36,000 LB)

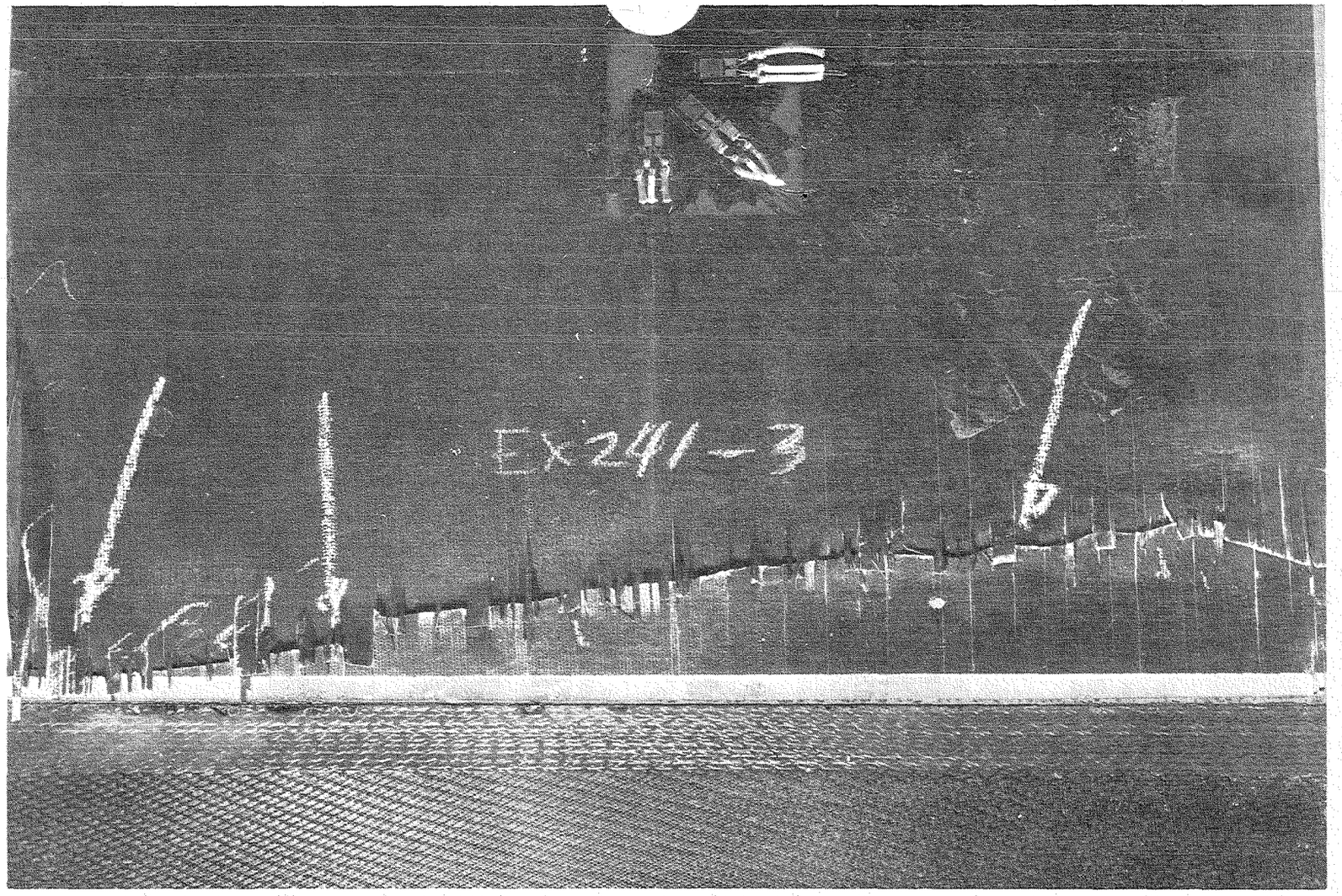
Figure 162B. Failure Modes of Sandwich Element EX 241-1, Postcured Condition, Tested at -132 C (-220 F)



SIDE 1

FAILING LOAD: 156 KN (35,100 LB)

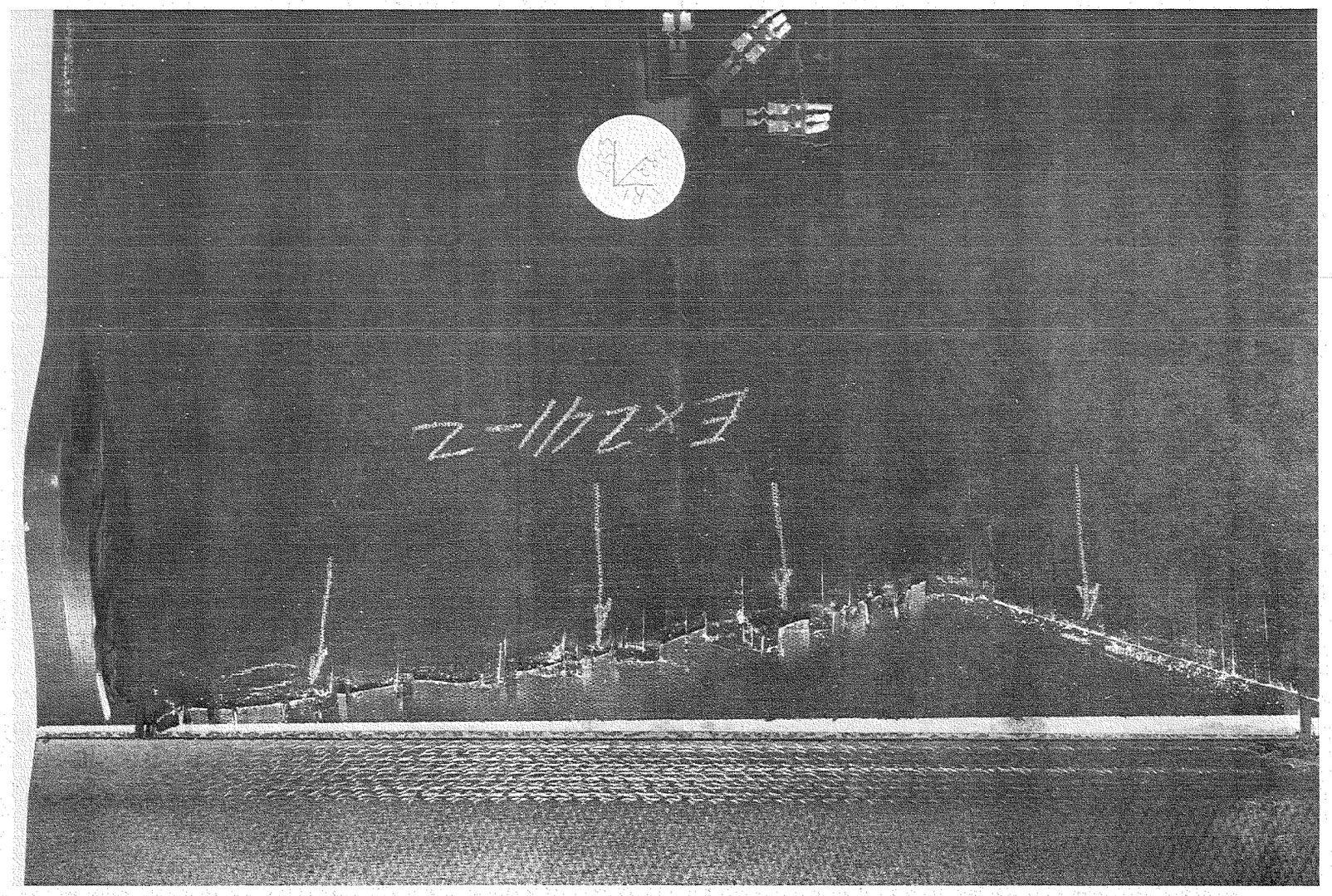
Figure 163A. Failure Modes of Sandwich Element EX 241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)



SIDE 2

FAILING LOAD: 156 KN (35,100 LB)

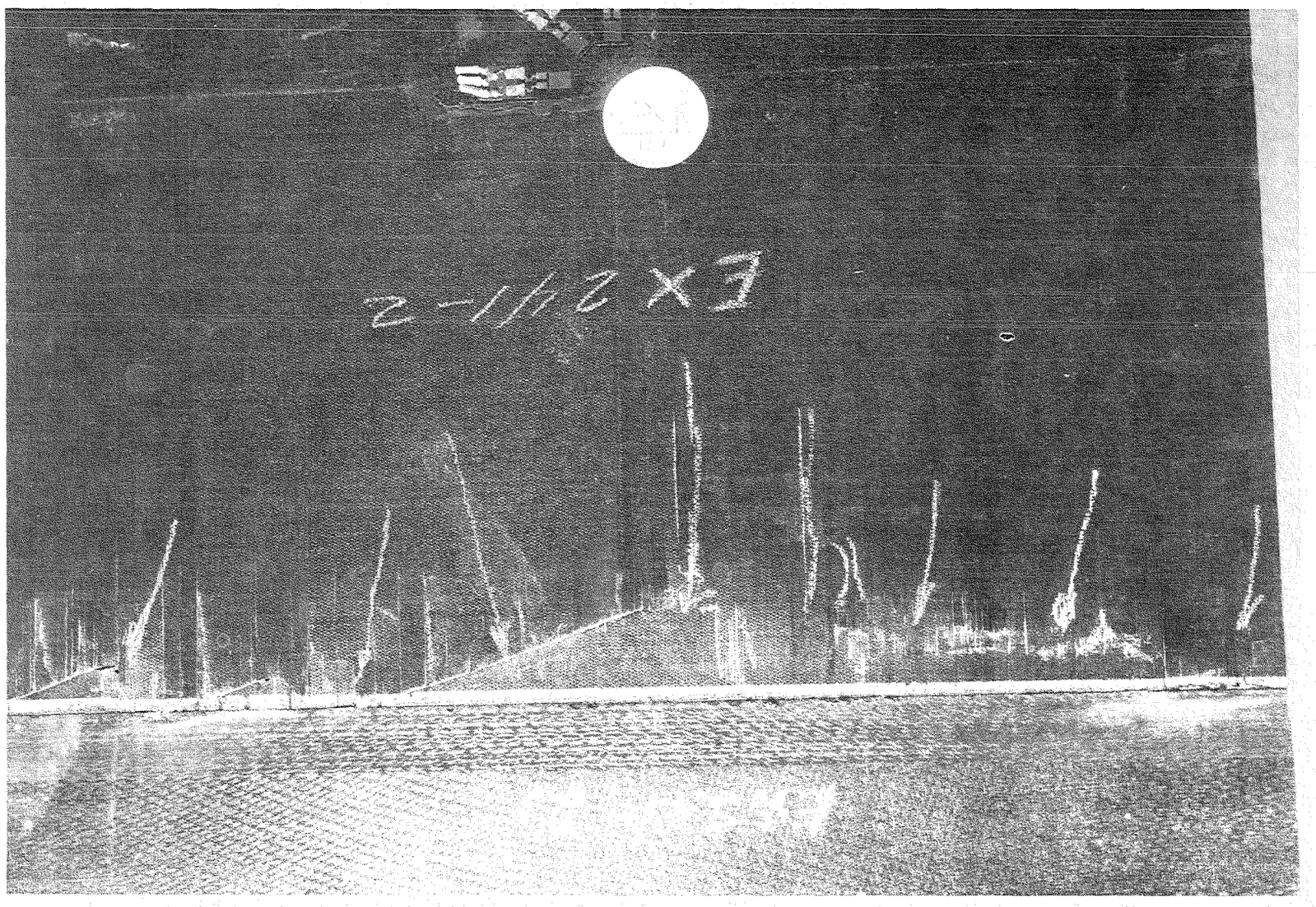
Figure 163B. Failure Modes of Sandwich Element EX 241-3A Aged 125 Hours at 316 C (600 F), Tested at -132 C (-270 F)



SIDE 1

FAILING LOAD: 136 KN (30,650 LB)

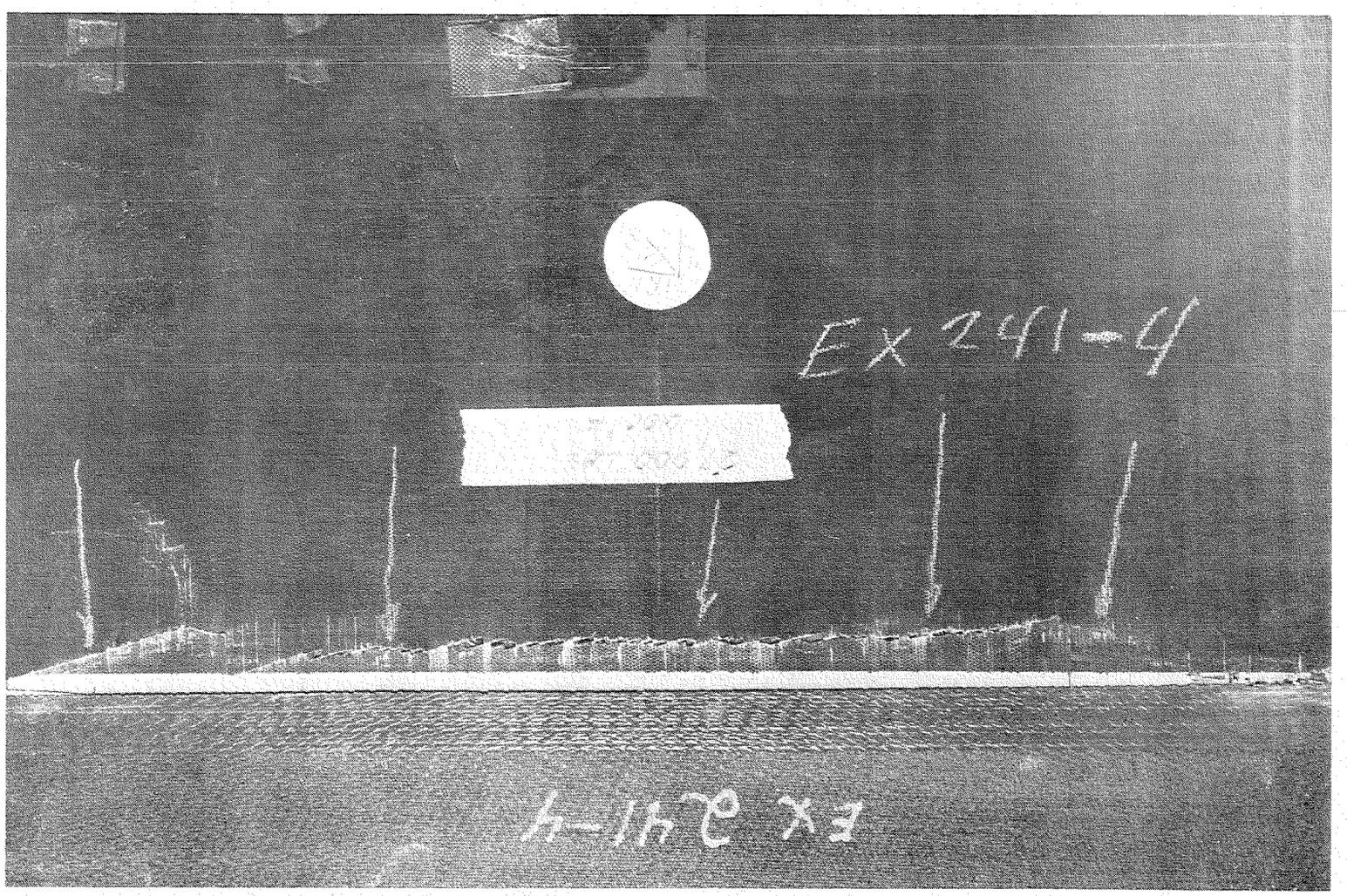
Figure 164A. Failure Modes of Sandwich Element EX 241-2A, Aged 125 Hours at 316 C (600 F), Tested at RT



SIDE 2

FAILING LOAD: 136 KN (30,650 LB)

Figure 164B. Failure Modes of Sandwich Element EX 241-2A, Aged 125 Hours at 316 C (600 F), Tested at RT

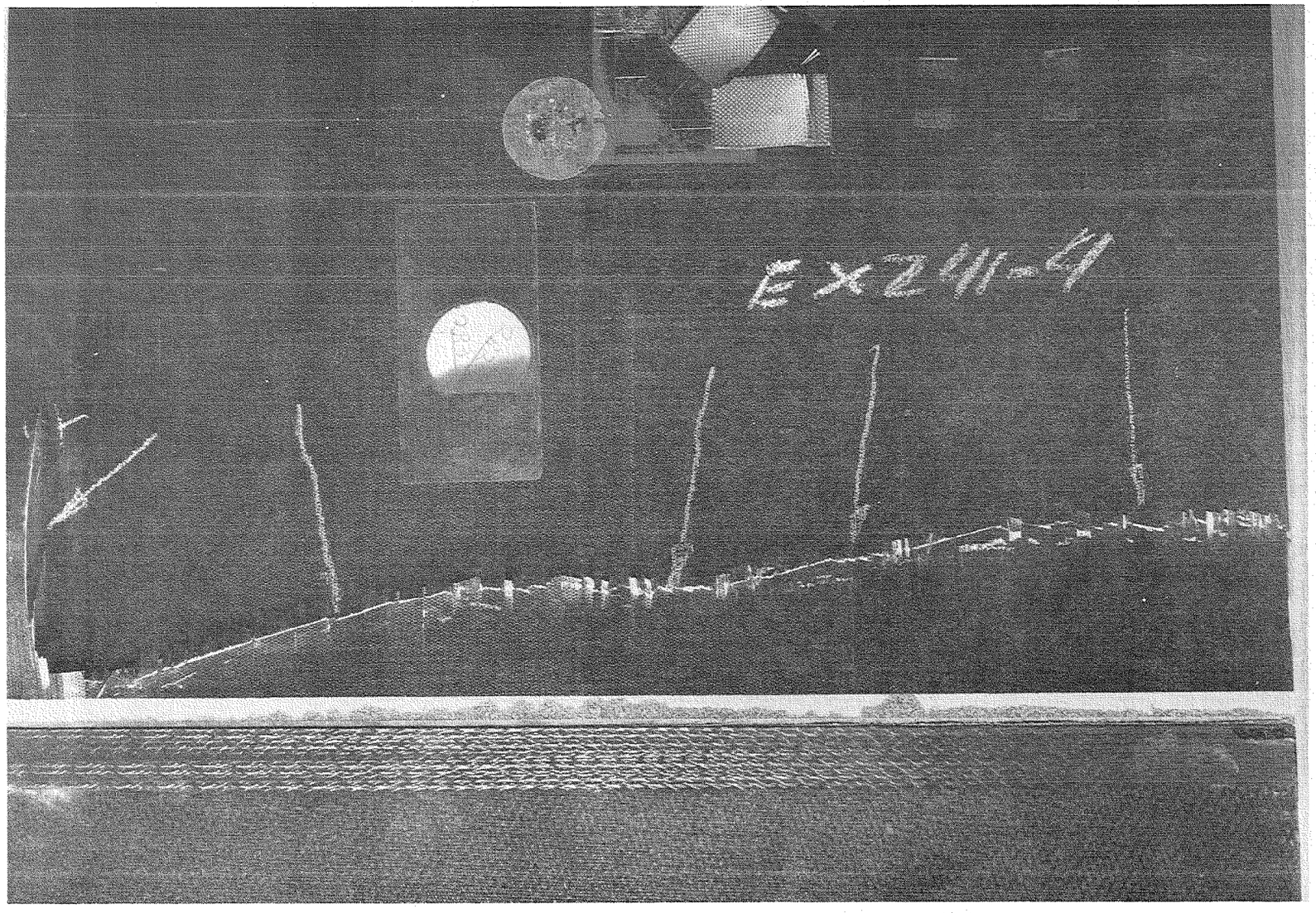


SIDE 1

FAILING LOAD: 120 KN (27,000 LB)

Figure 165A. Failure Modes of Sandwich Element EX 241-4A, Aged 125 Hours at 316 C (600 F) Tested at 316 C (600 F)

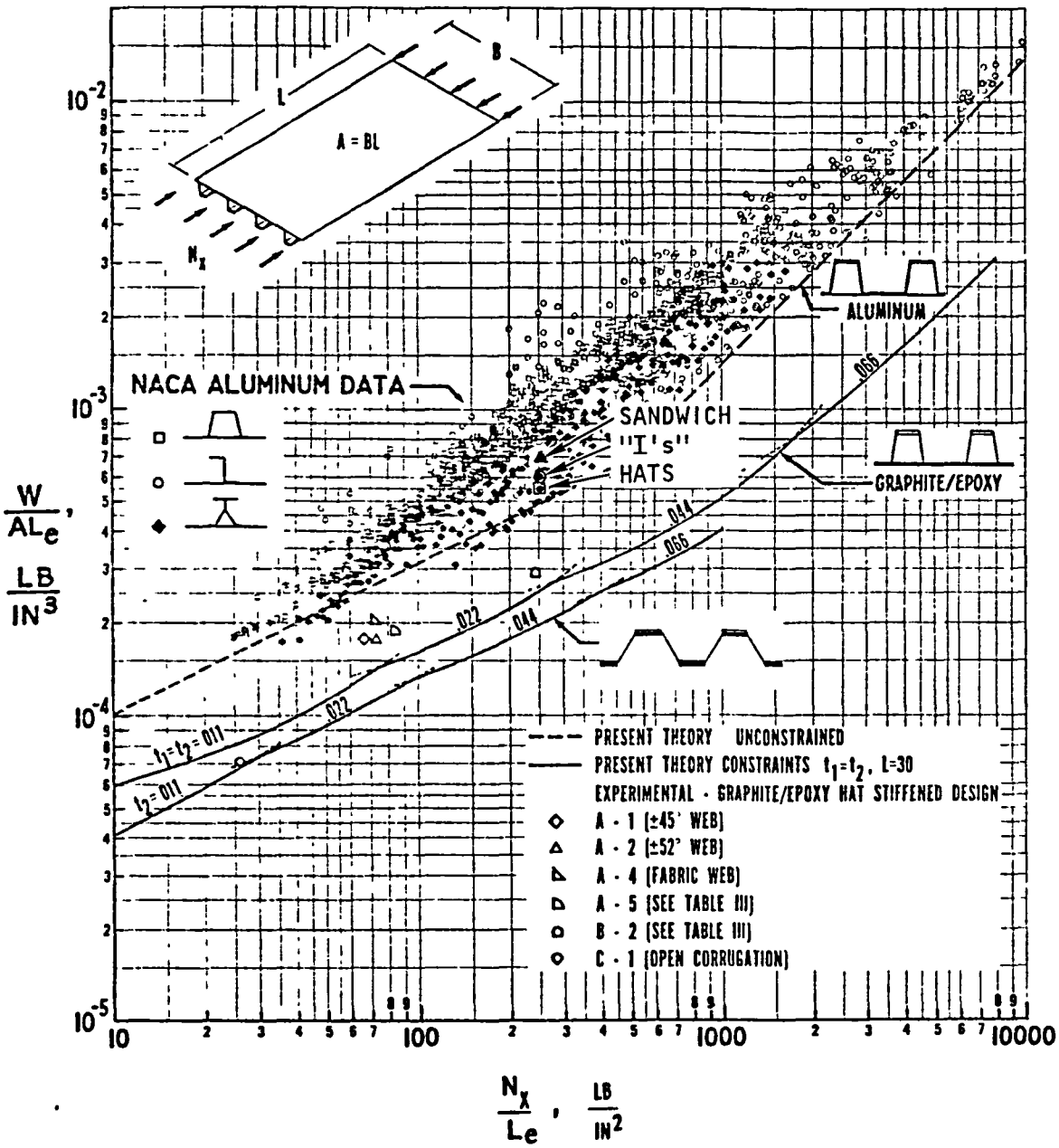
A800808 G-9



SIDE 2

FAILING LOAD: 120 KN (27,000 LB)

Figure 165B. Failure Modes of Sandwich Element EX 241-4A, Aged 125 Hours at 316 C (600 F) Tested at 316 C (600 F)



REF. "ANALYTICAL AND EXPERIMENTAL STUDY OF STRUCTURALLY EFFICIENT COMPOSITE HAT-STIFFENED PANELS LOADED IN AXIAL COMPRESSION"

JERRY G. WILLIAMS &
MARTIN M. MIKULAS, JR.
NASA-LARC
AIAA PAPER #75-754, 1975

Figure 166. Comparison of Structural Efficiencies of Compression Panels.

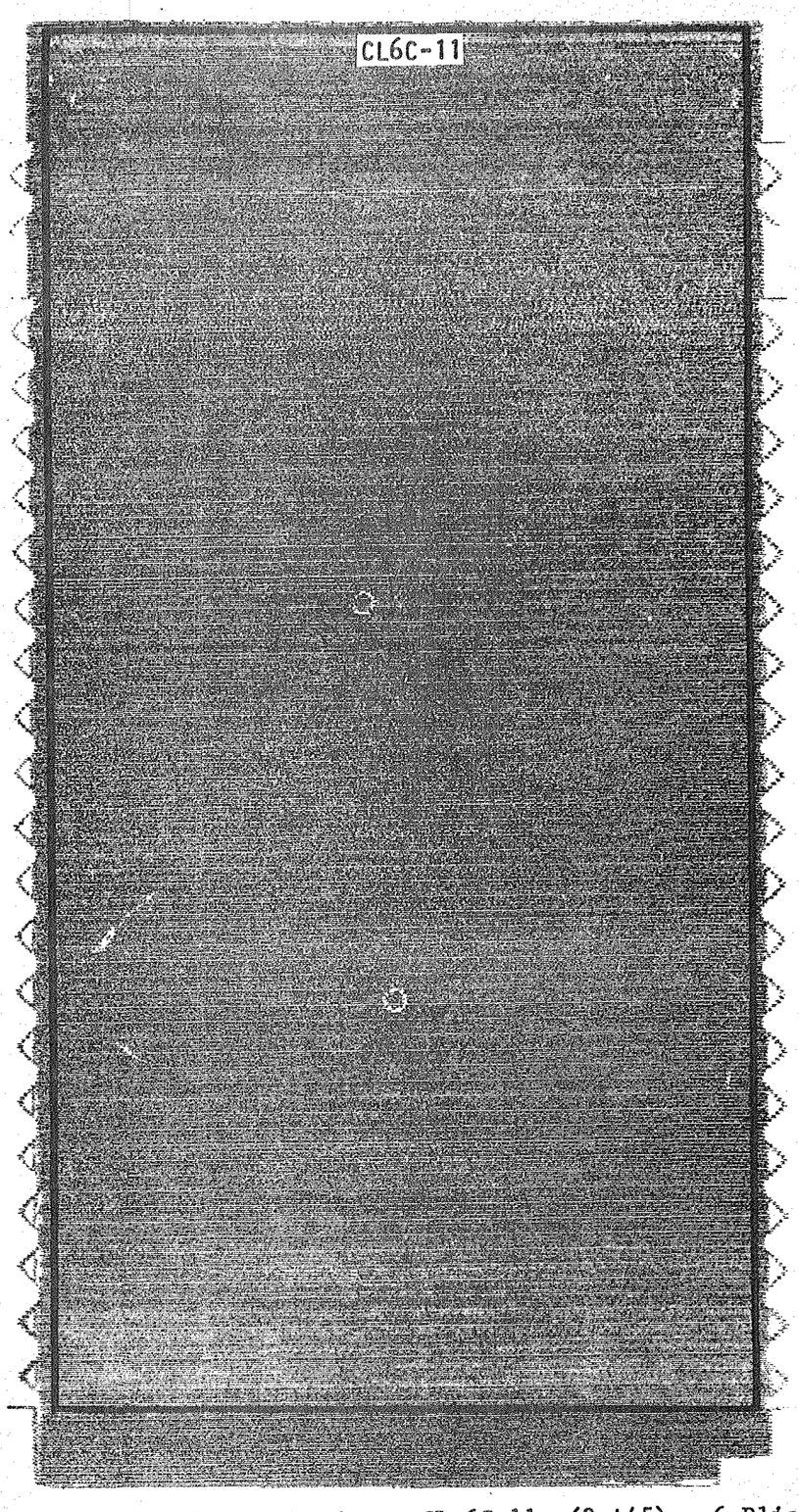


Figure 167. C-Scan Laminate CL 6C-11, $(0,\pm45)_s$, 6 Plies

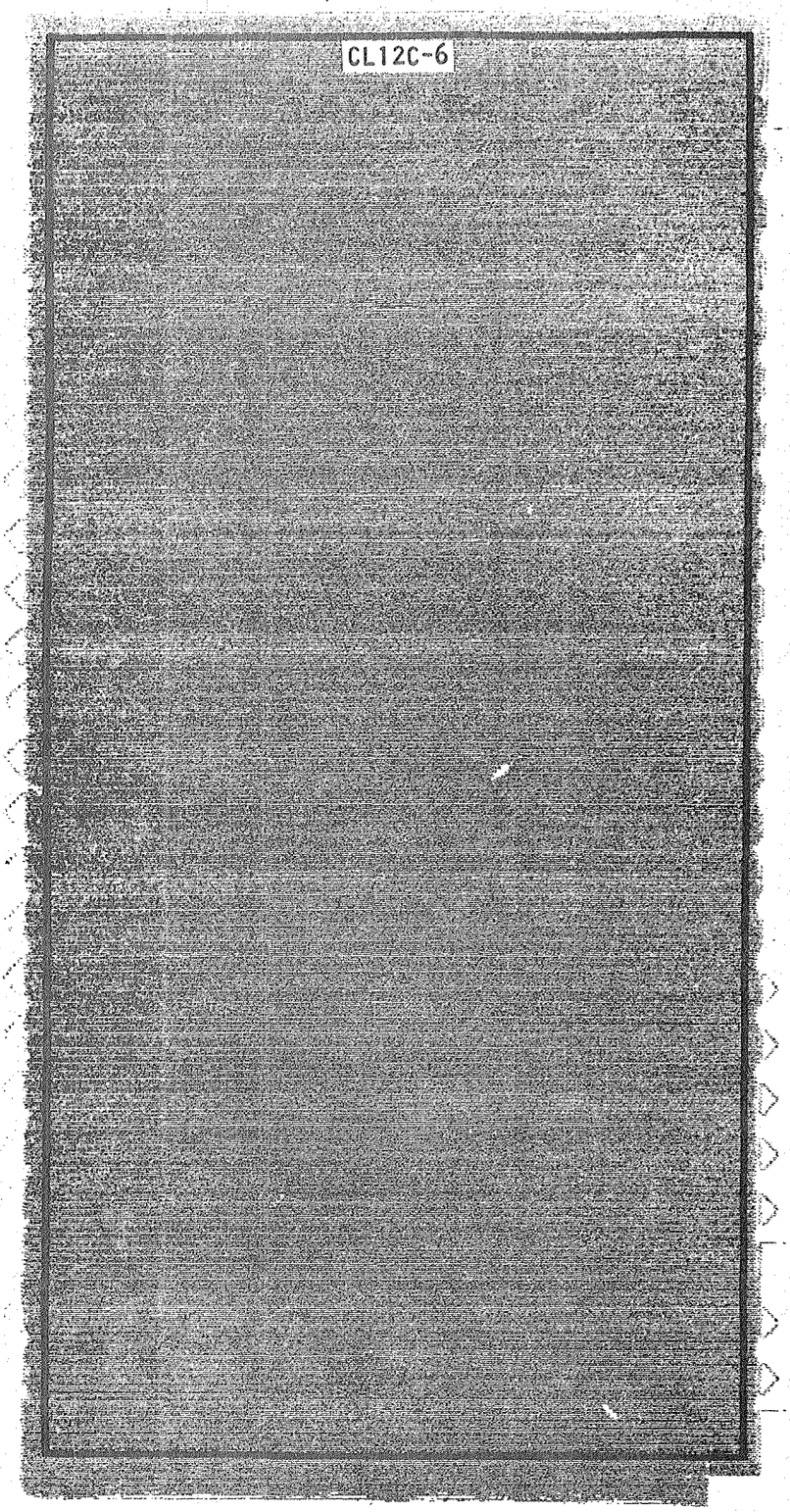


Figure 168. C-Scan Laminate CL 12C-6, $(0,\pm45)_s$, 12 Plies

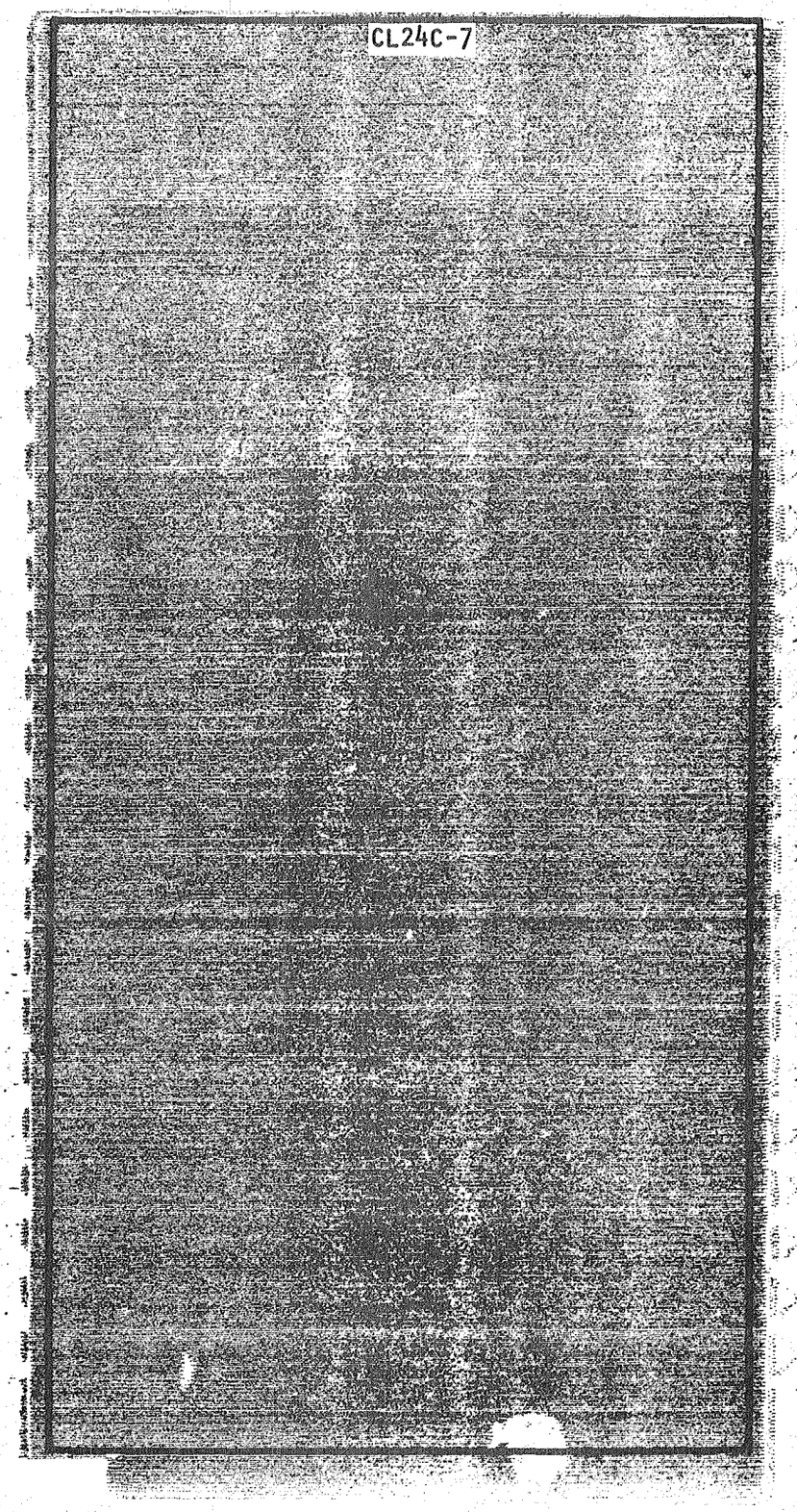


Figure 169. C-Scan Laminate CL 24C-7, $(0\pm45)_s$, 24 Plies

Figure 170. Three - 122 X 26.0 Cm Hat Stringer/Skin Panels Delivered to NASA-LaRC

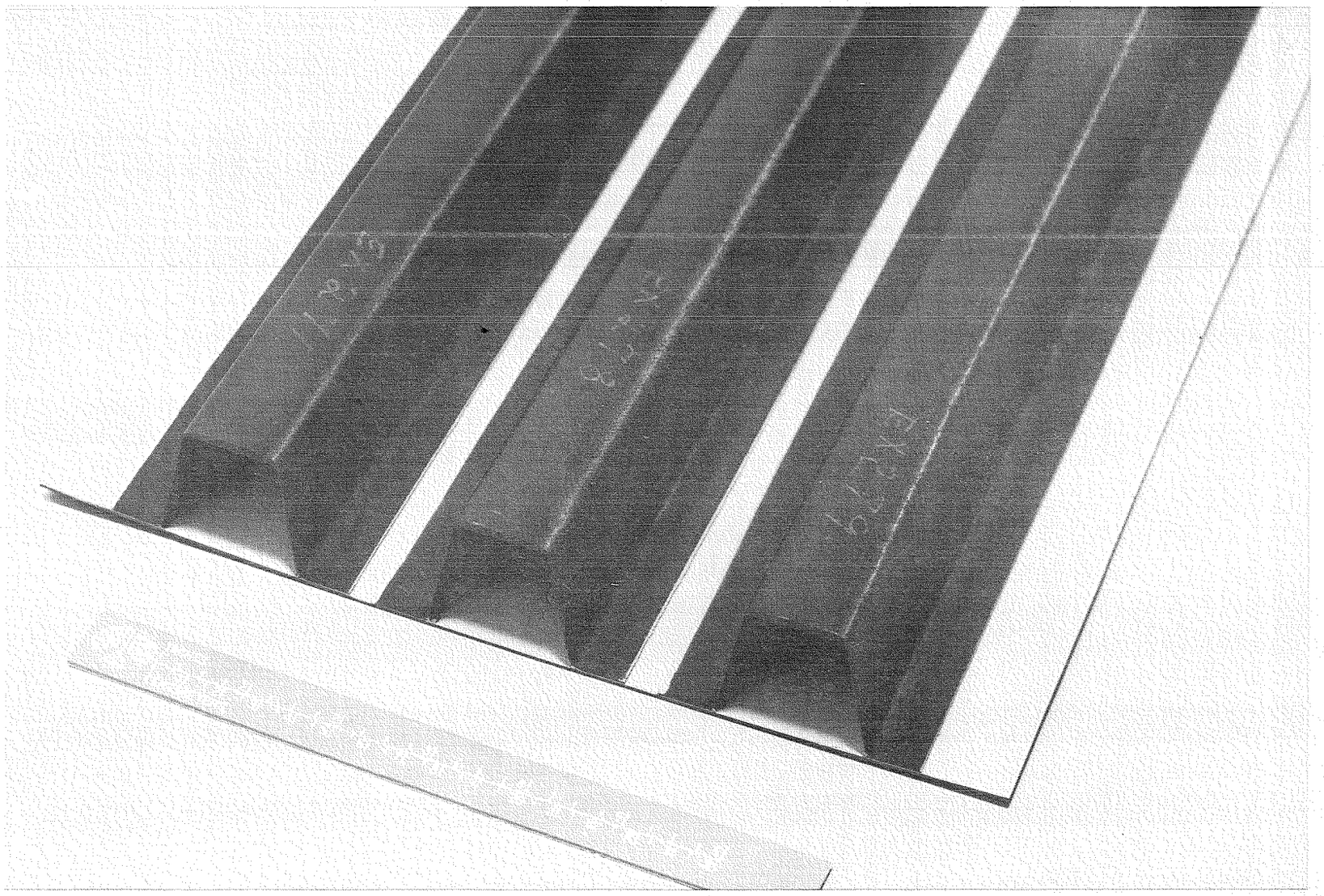


Figure 171. End Views of Hat Stringer/Skin Panels Delivered to NASA-LaRC

Figure 172. C-Scans of Stringer to Skin Bond Joints, Panels EX249/248/245 and EX279/278/277

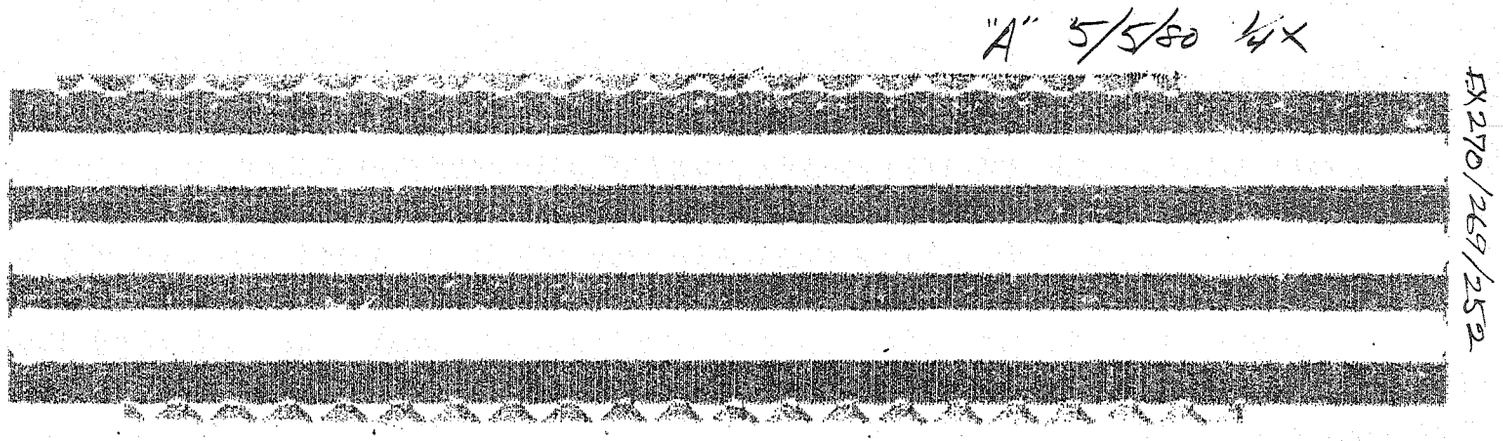


Figure 173. C-Scan of Stringer to Skin Bond Joints, Panel EX270/278/277

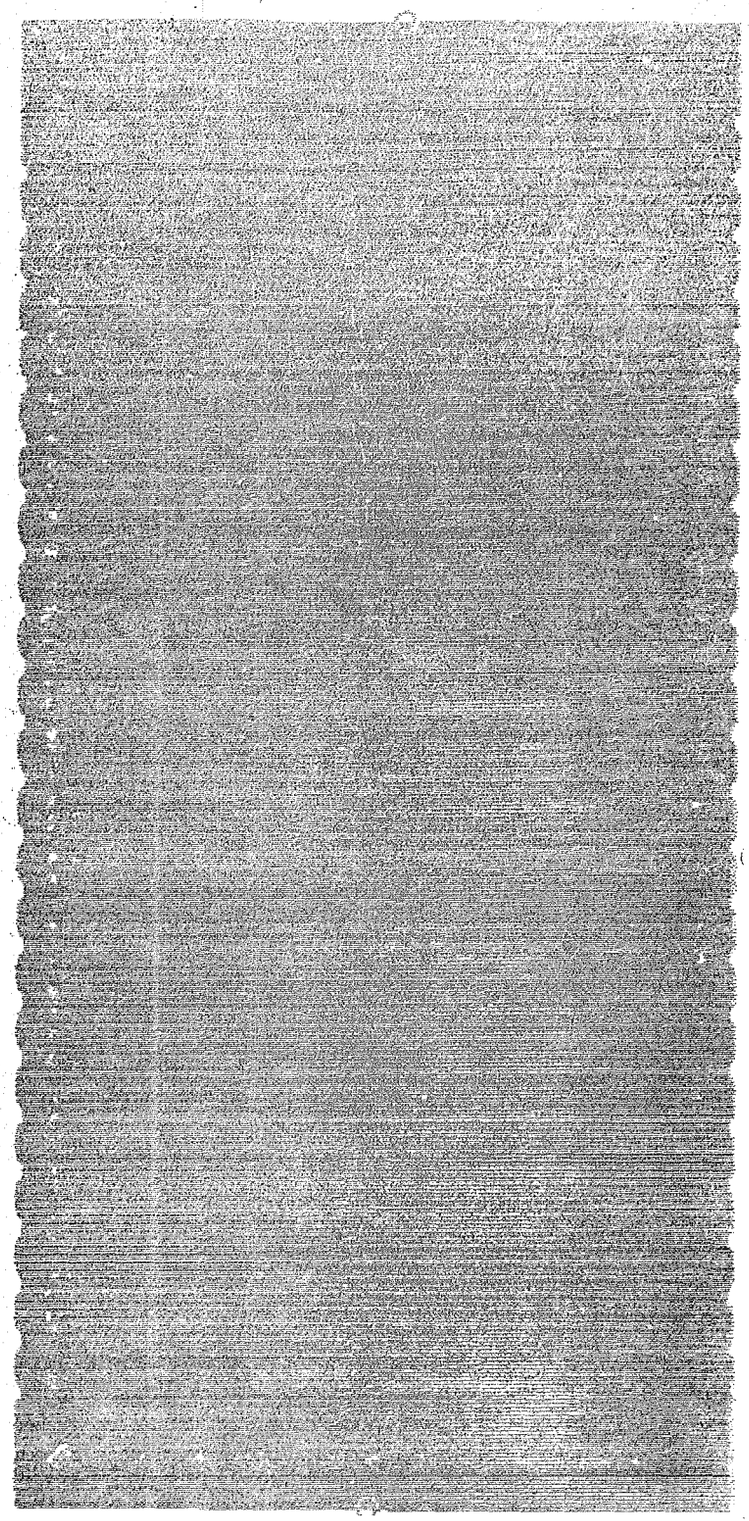


Figure 174. C-scan Laminate CL8C-18, $(0,\pm45,90)_S$, 8 Plies

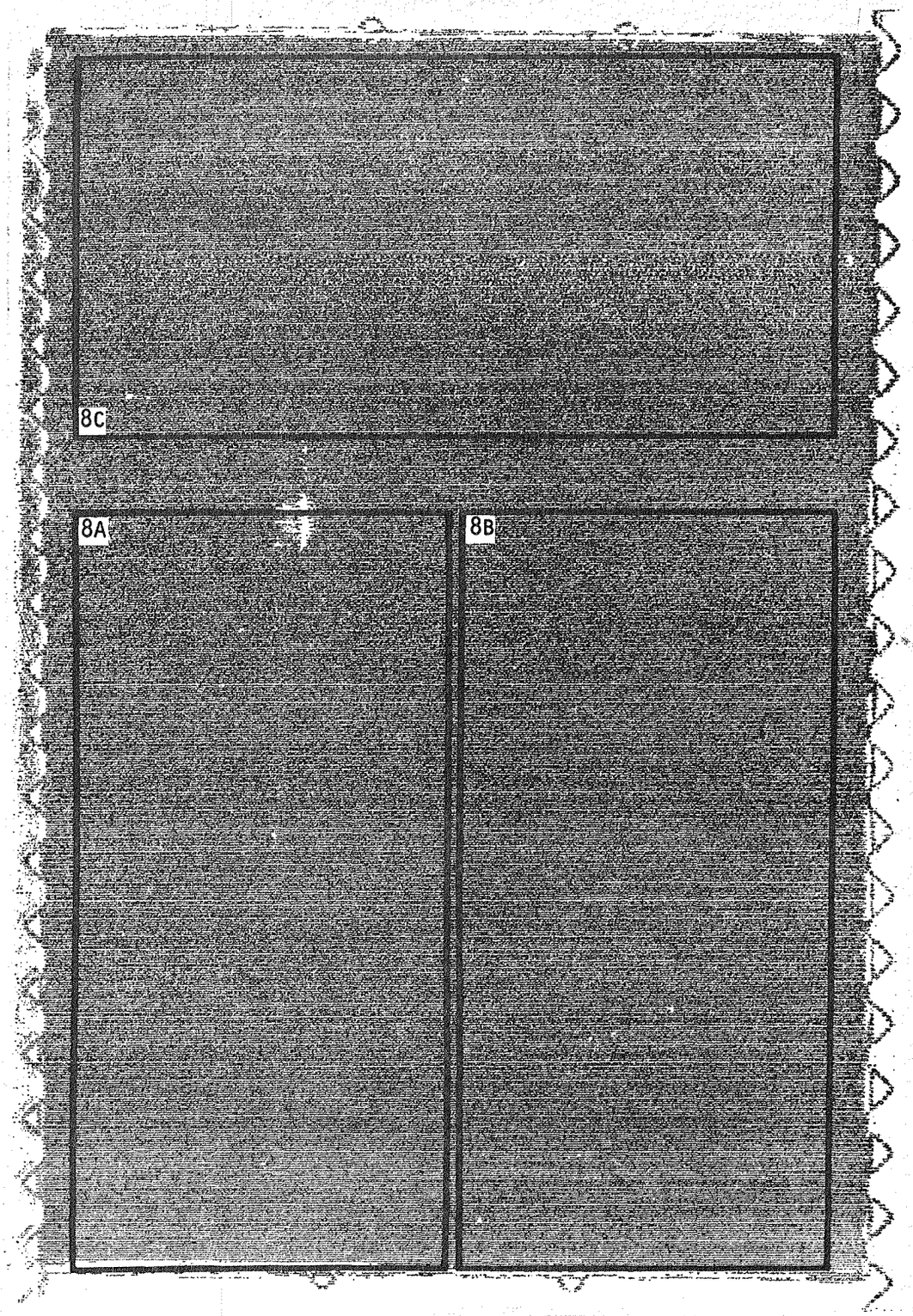


Figure 175. C-Scan Laminate C1 12C-8, $(0,90)_t$, 12 Plies, Skin for Honeycomb Panels

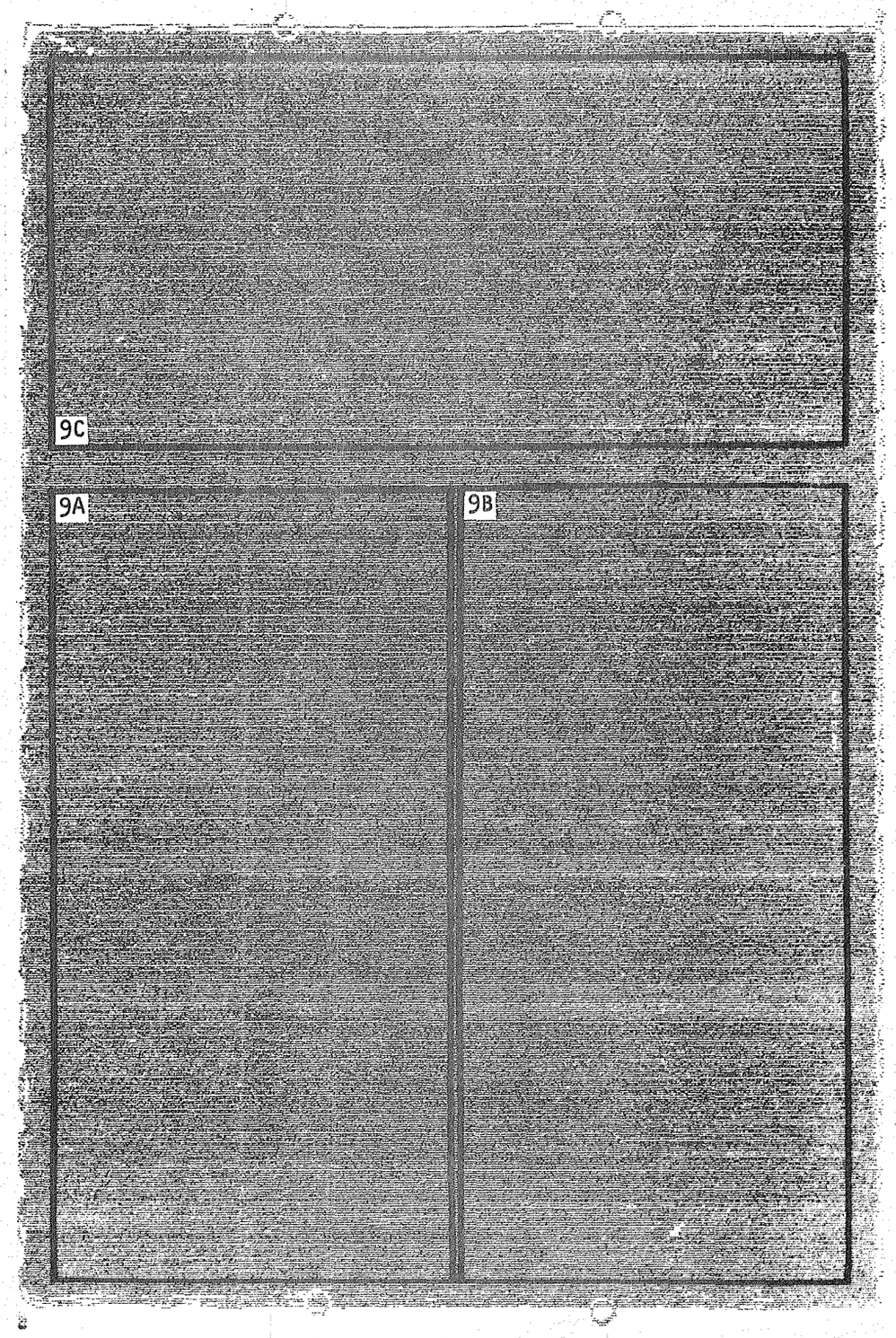


Figure 176. C-Scan Laminate CL 12C-9, $(0,90)_t$, 12 Plies, Skin for Honeycomb Panels

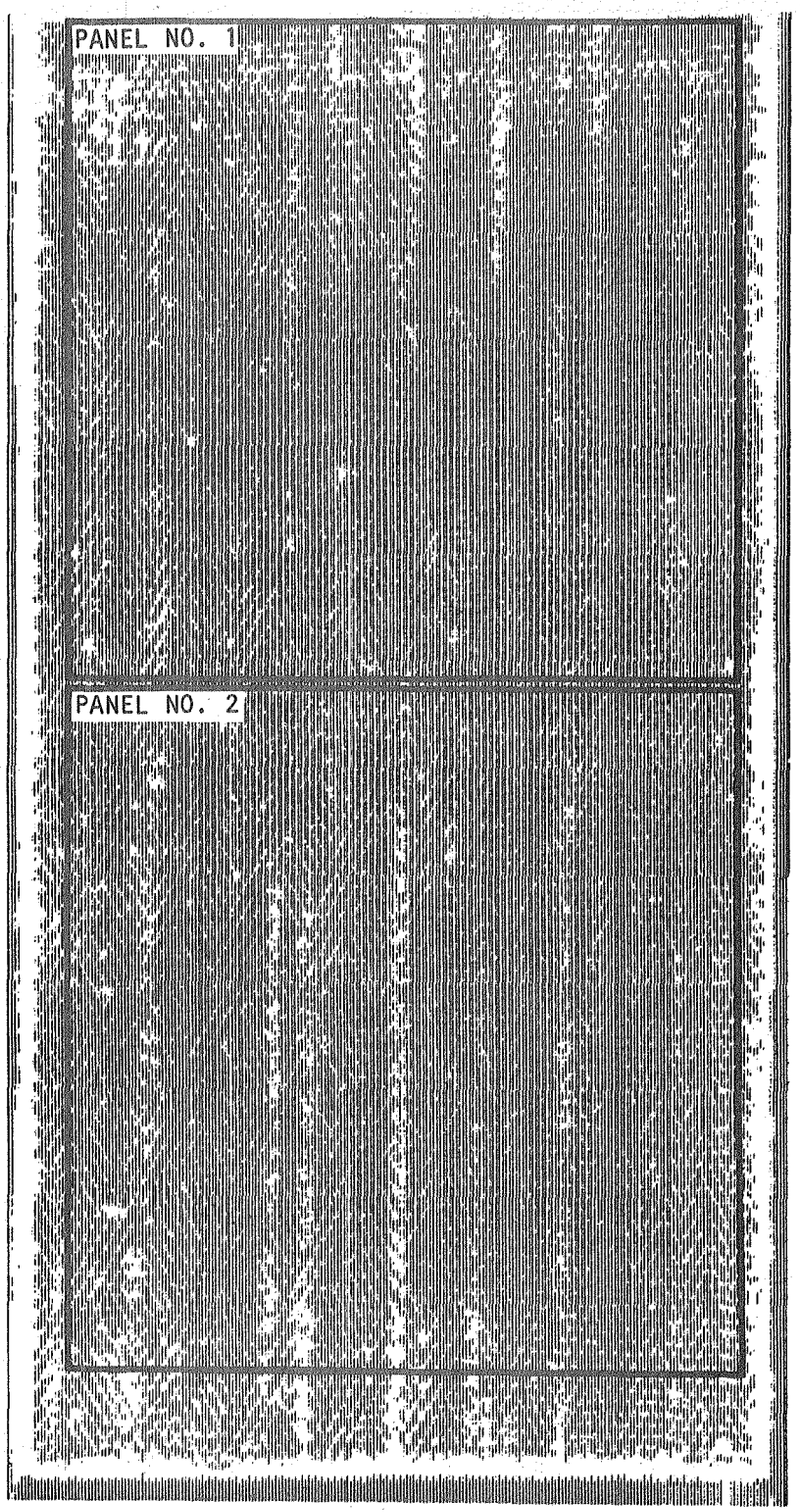


Figure 177. C-Scan Honeycomb Panel 8A with Location of 25.4 X 25.4-cm Panels No. 1 and No. 2

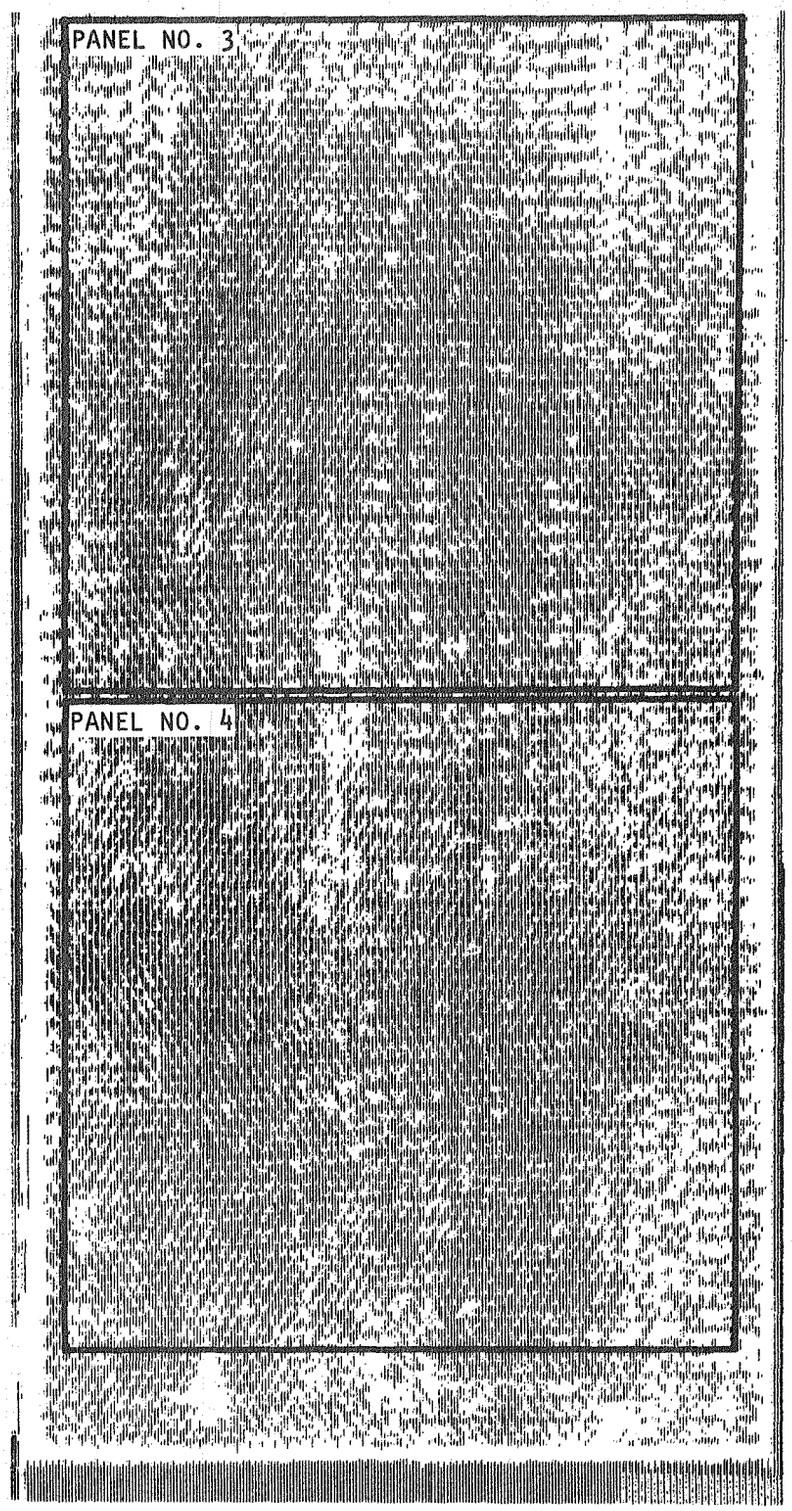


Figure 178. C-Scan Honeycomb Panel 9A with Location of 25.4 X 25.4-cm Panels No. 3 and No. 4

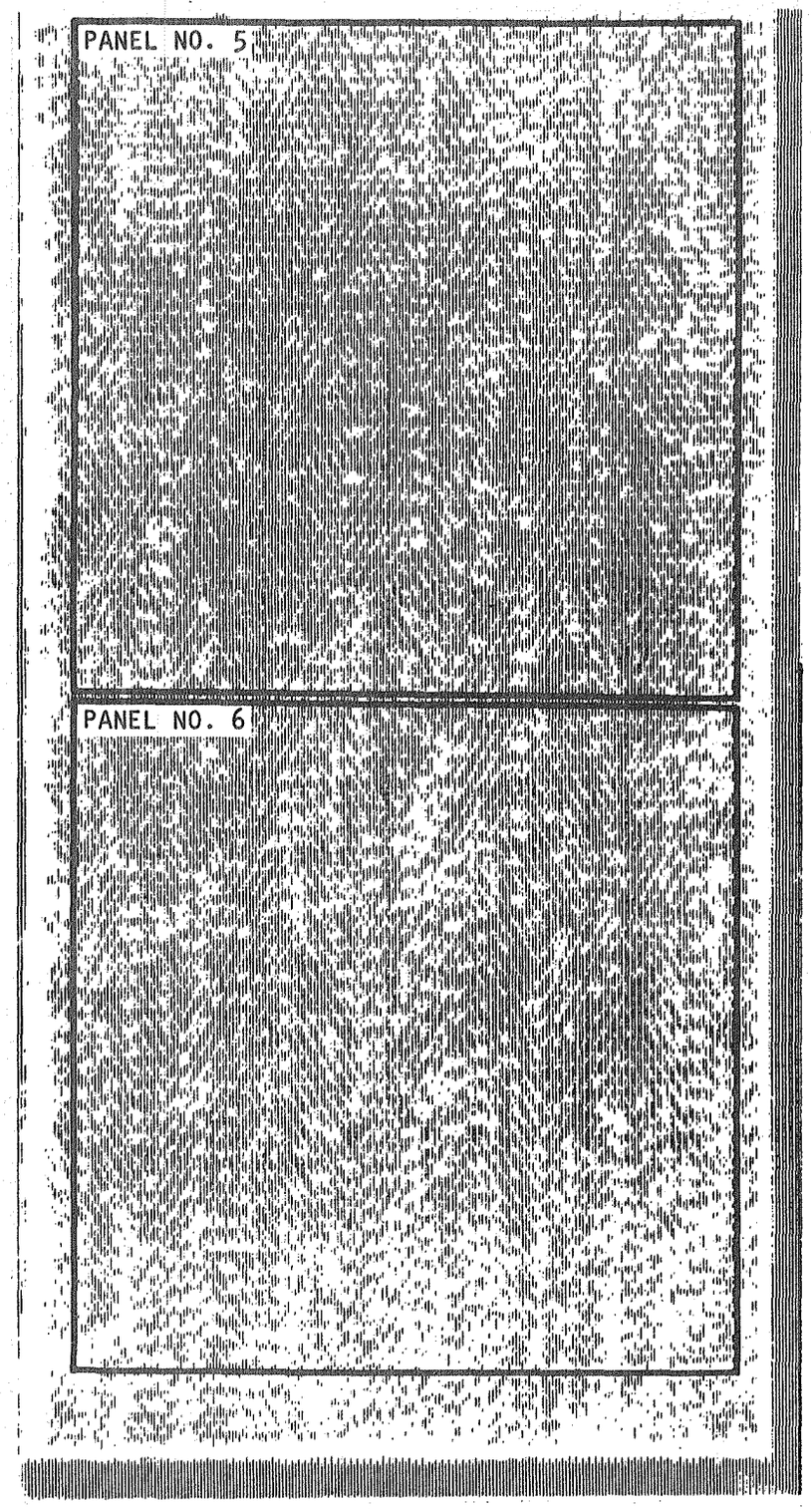


Figure 179. C-Scan Honeycomb Panel 9C with Location of 25.4 X 25.4-cm Panels No. 5 and No. 6

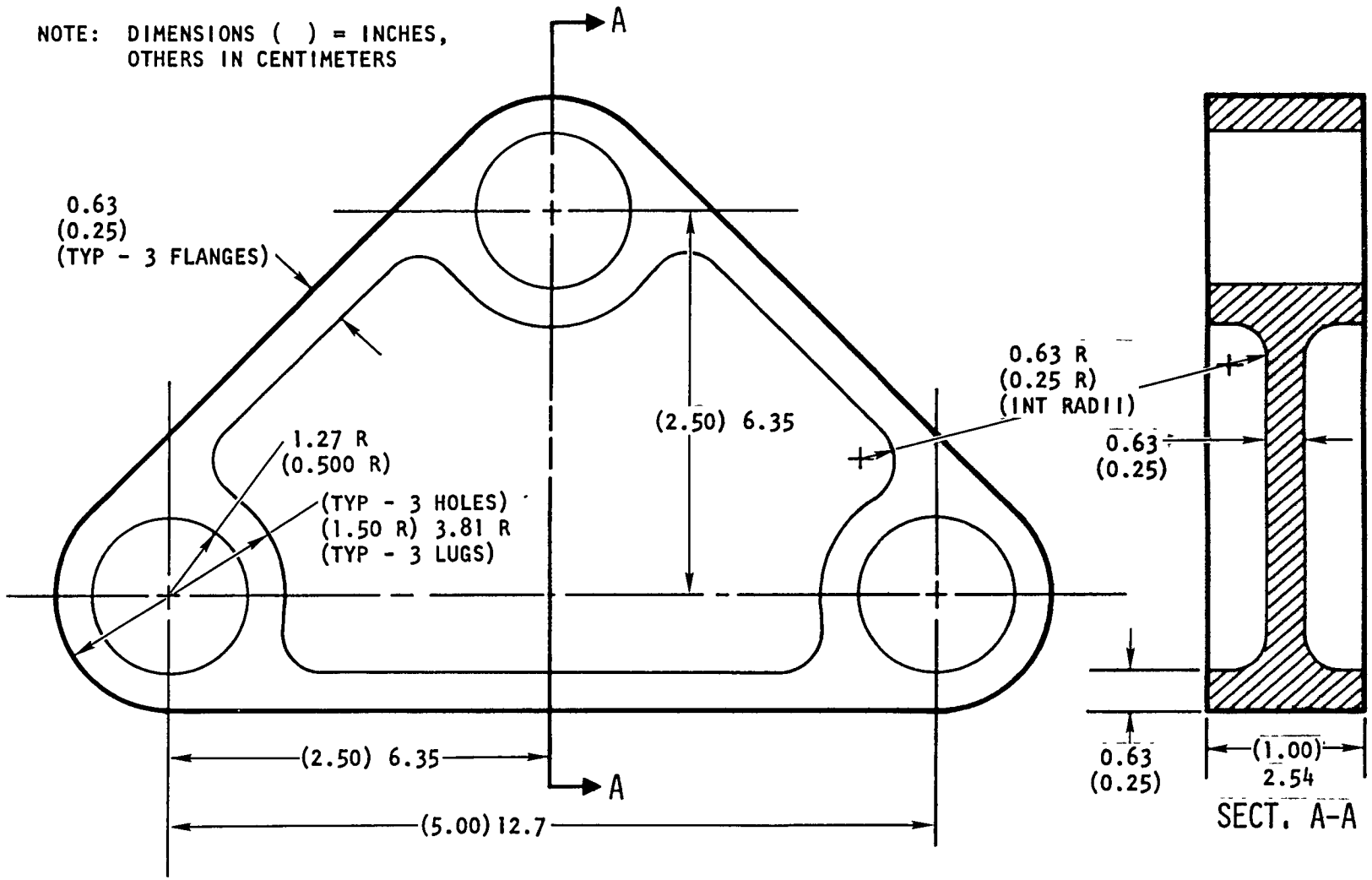


Figure 180. Molding Compound Process Demonstration Part Design

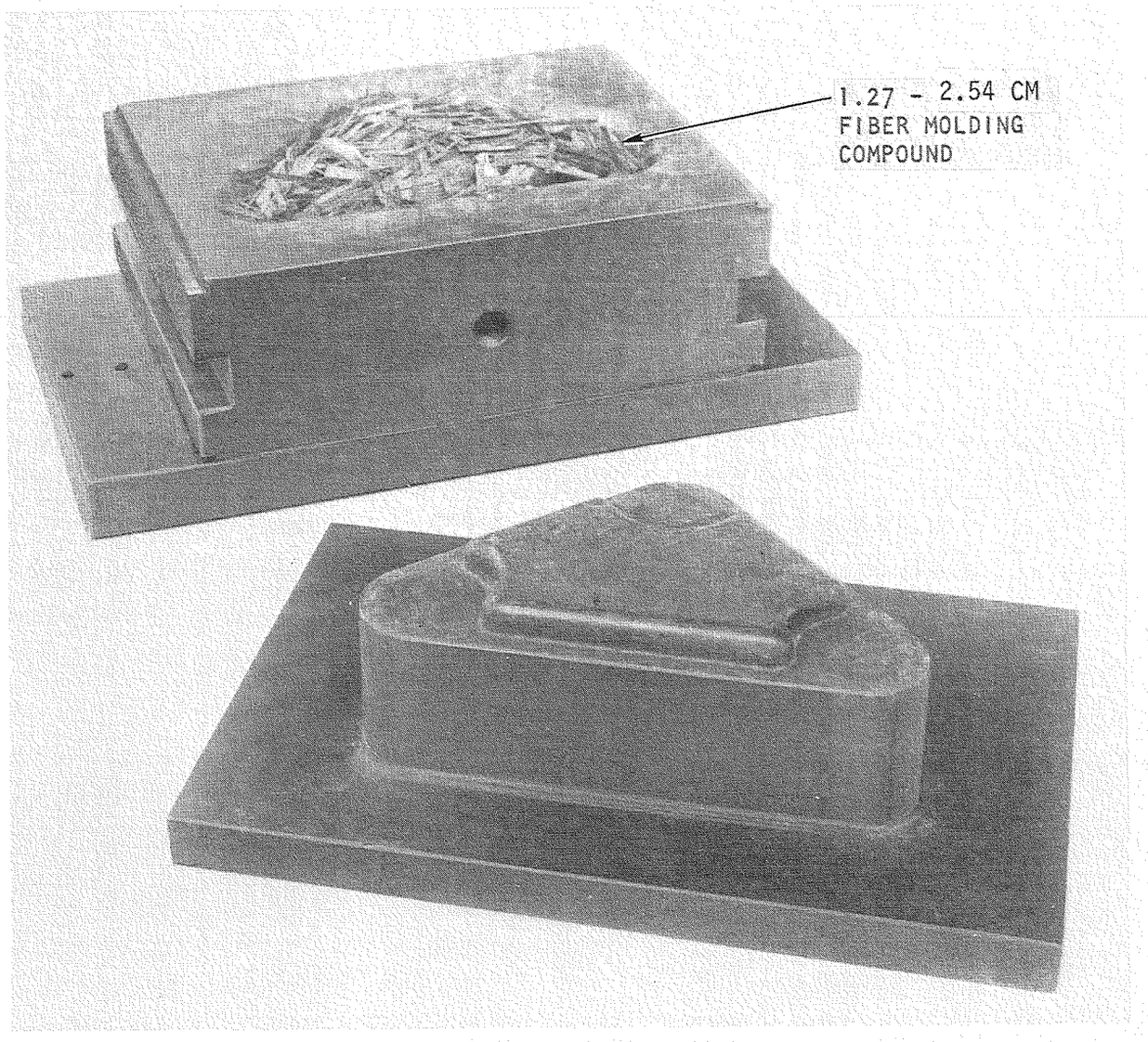


Figure 181. Celion/LARC 160 Molding Compound Complex Part Demonstration Mold

A801002 G-9

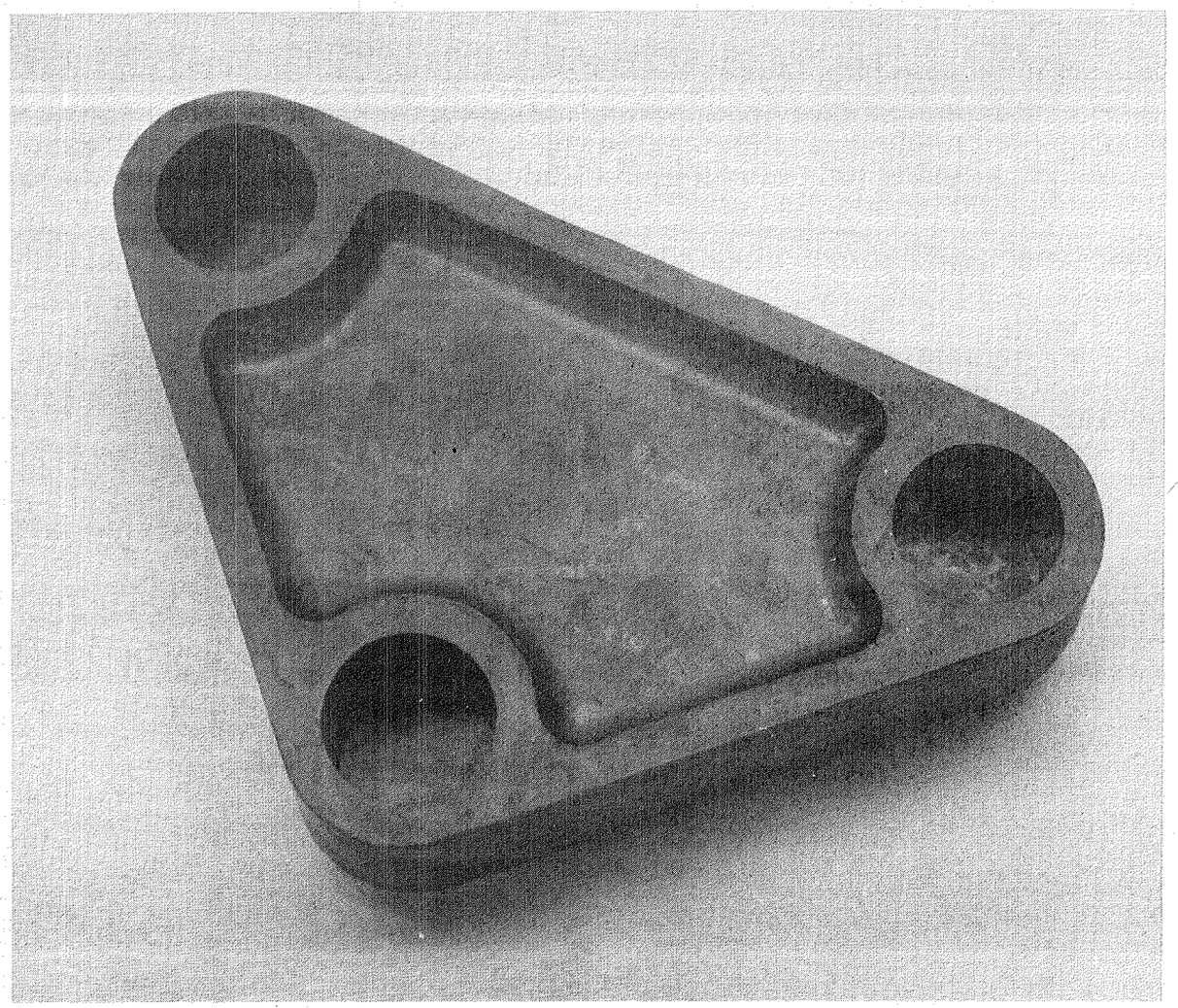


Figure 182. Complex Shaped Process Demonstration Part Showing Good Compound Flow and Mold Conformance—Imidized at 191°C (375°F), Removed From Mold Cold

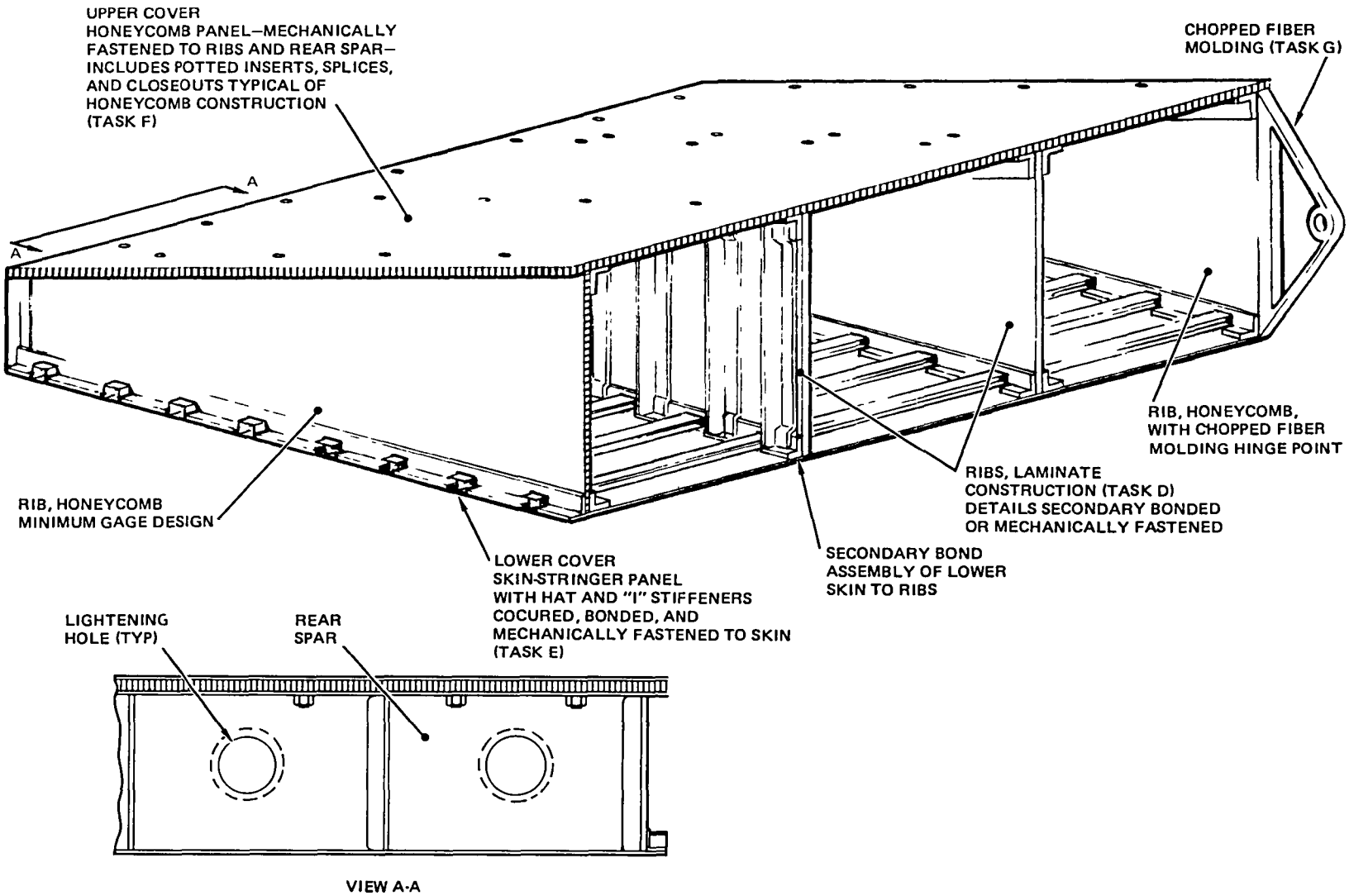


Figure 183. Representative Demonstration Component

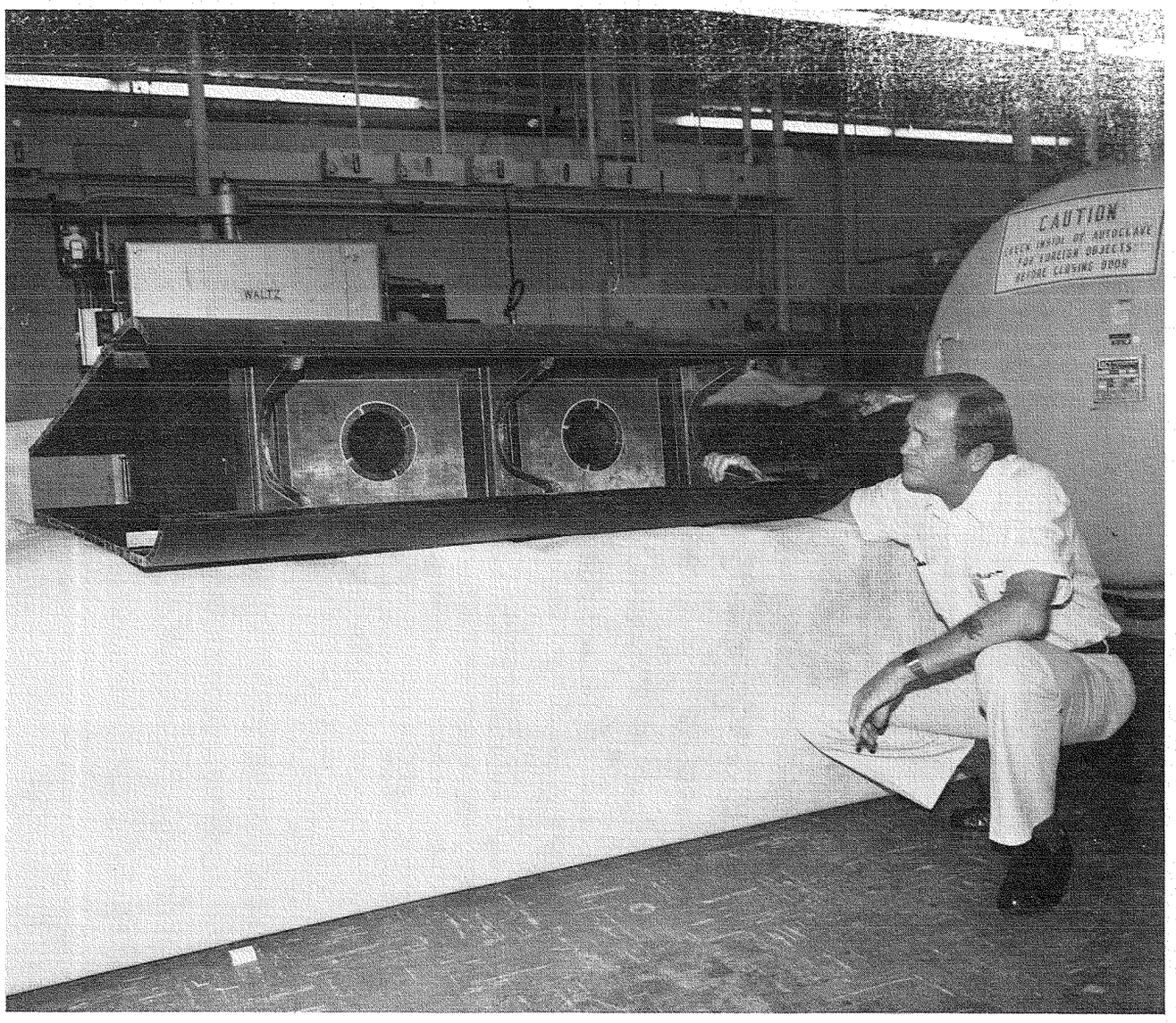


Figure 184. Completed Technology Demonstrator Segment Bonded Assembly

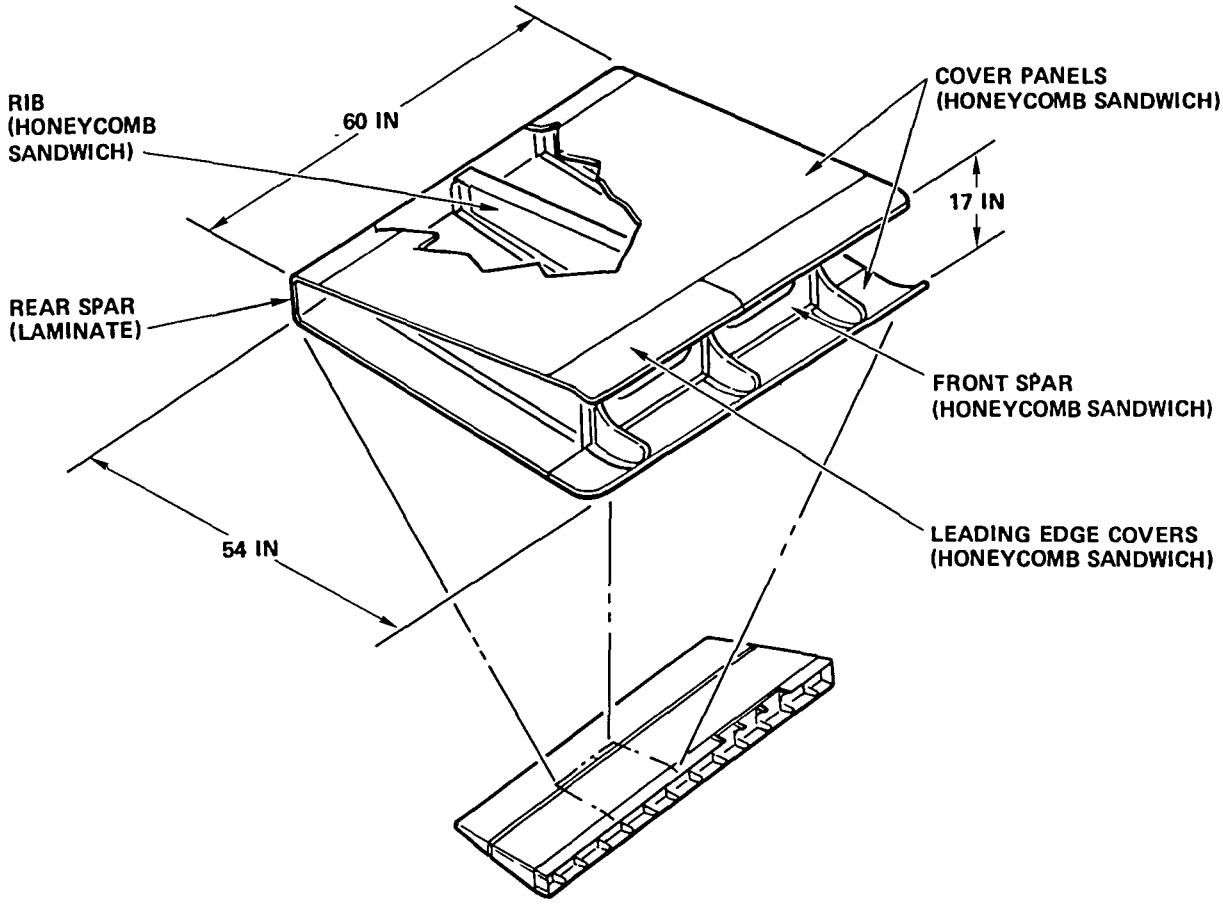
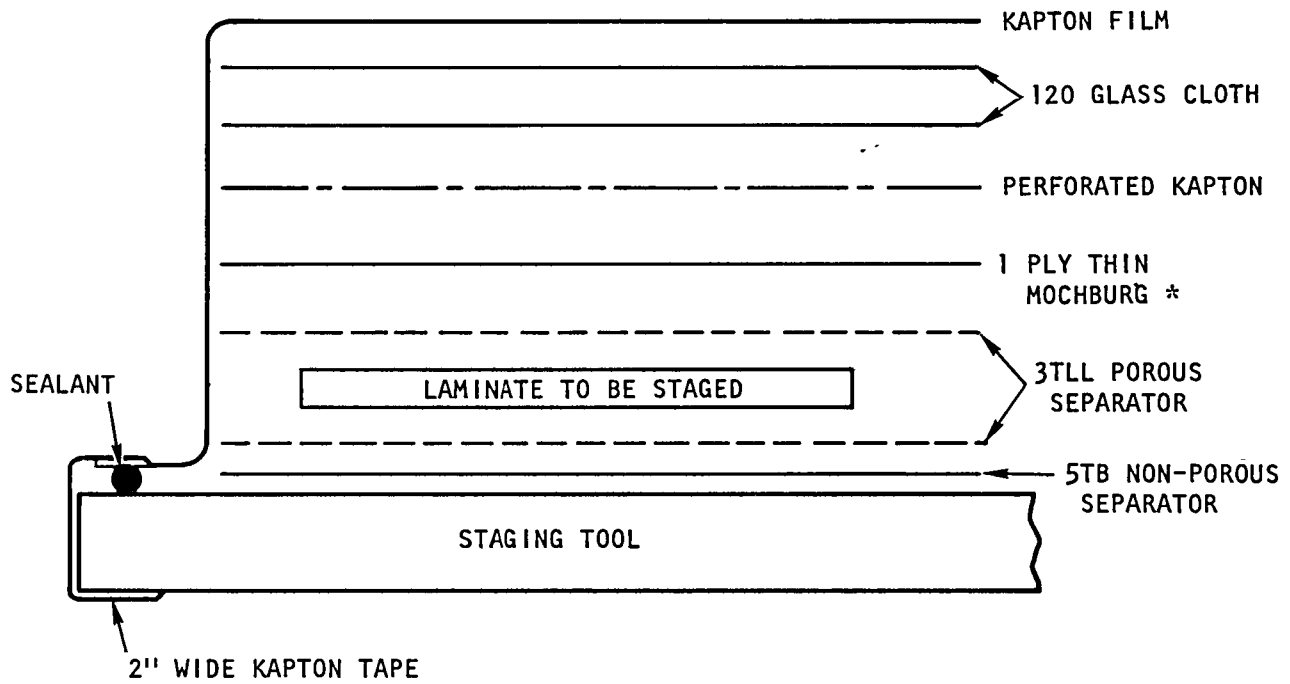
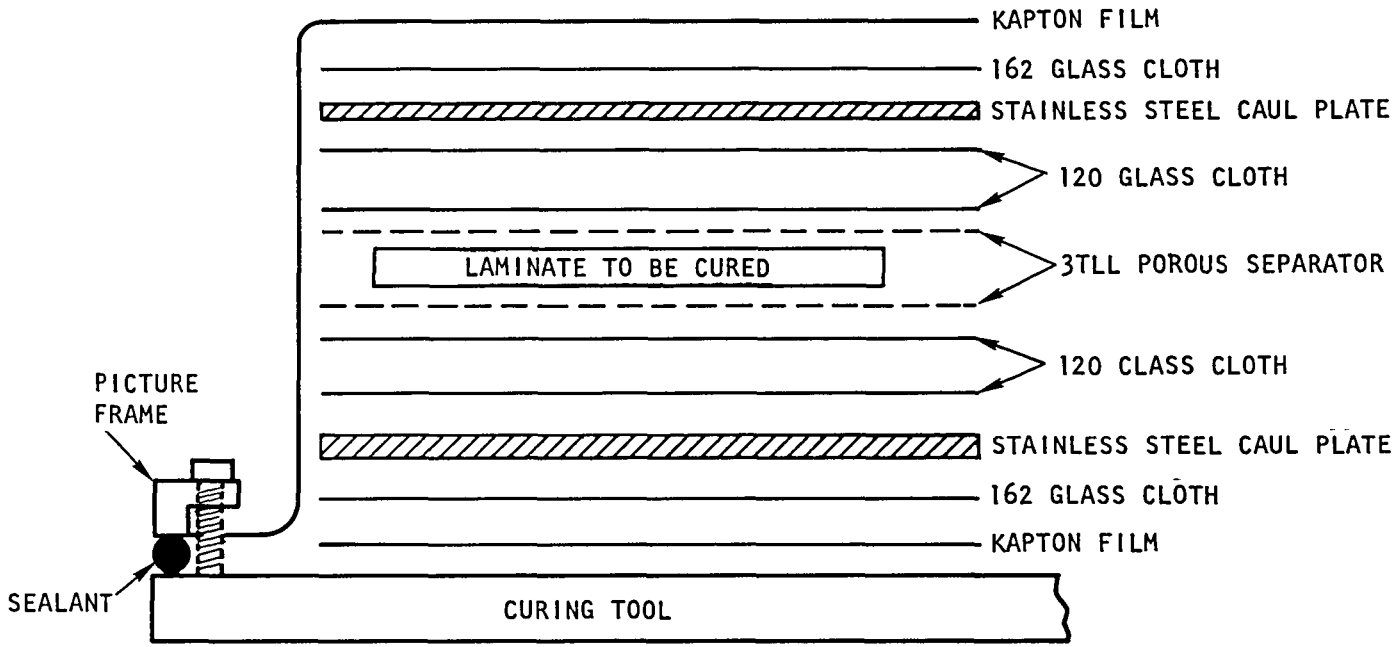


Figure 185. GR/PI Body Flap Concept and Demonstrator Segment



* THIN MOCHBURG AND/OR GLASS BLEEDERS WERE NOT USED ON LAMINATES < .38 MM (0.015 IN.)

Figure 186. Bagging Assembly for Imidizing



FOR CERTAIN LAYUPS, THE 120 CLOTH, STAINLESS STEEL CAUL PLATE AND 162 CLOTH WERE NOT USED

Figure 187. Bagging Assembly for Autoclave Curing

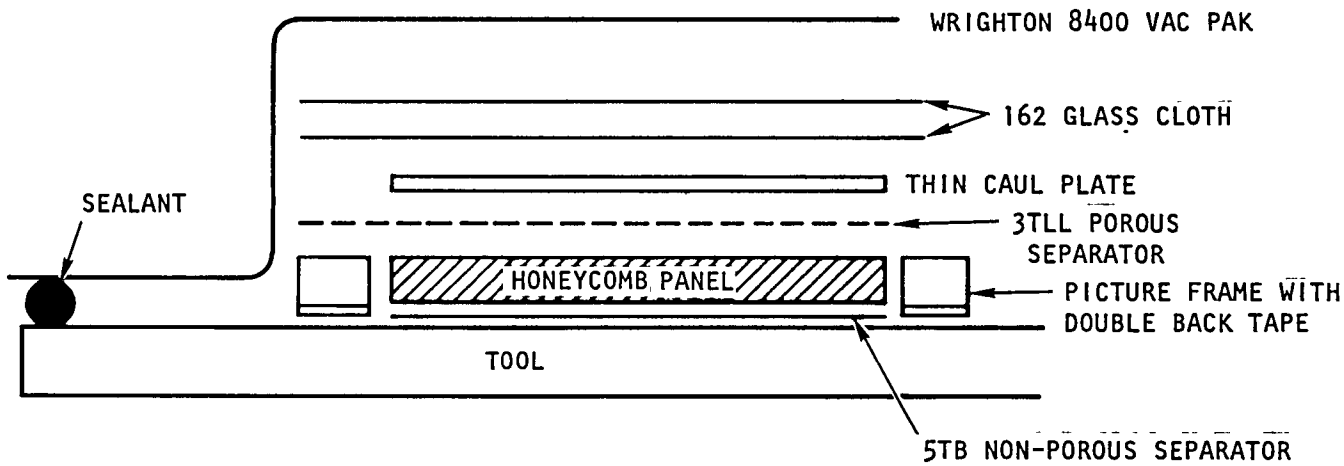


Figure 188. Bagging Assembly for Honeycomb Sandwich Panel Bonding

NASA STANDARD "A" SENSITIVITY

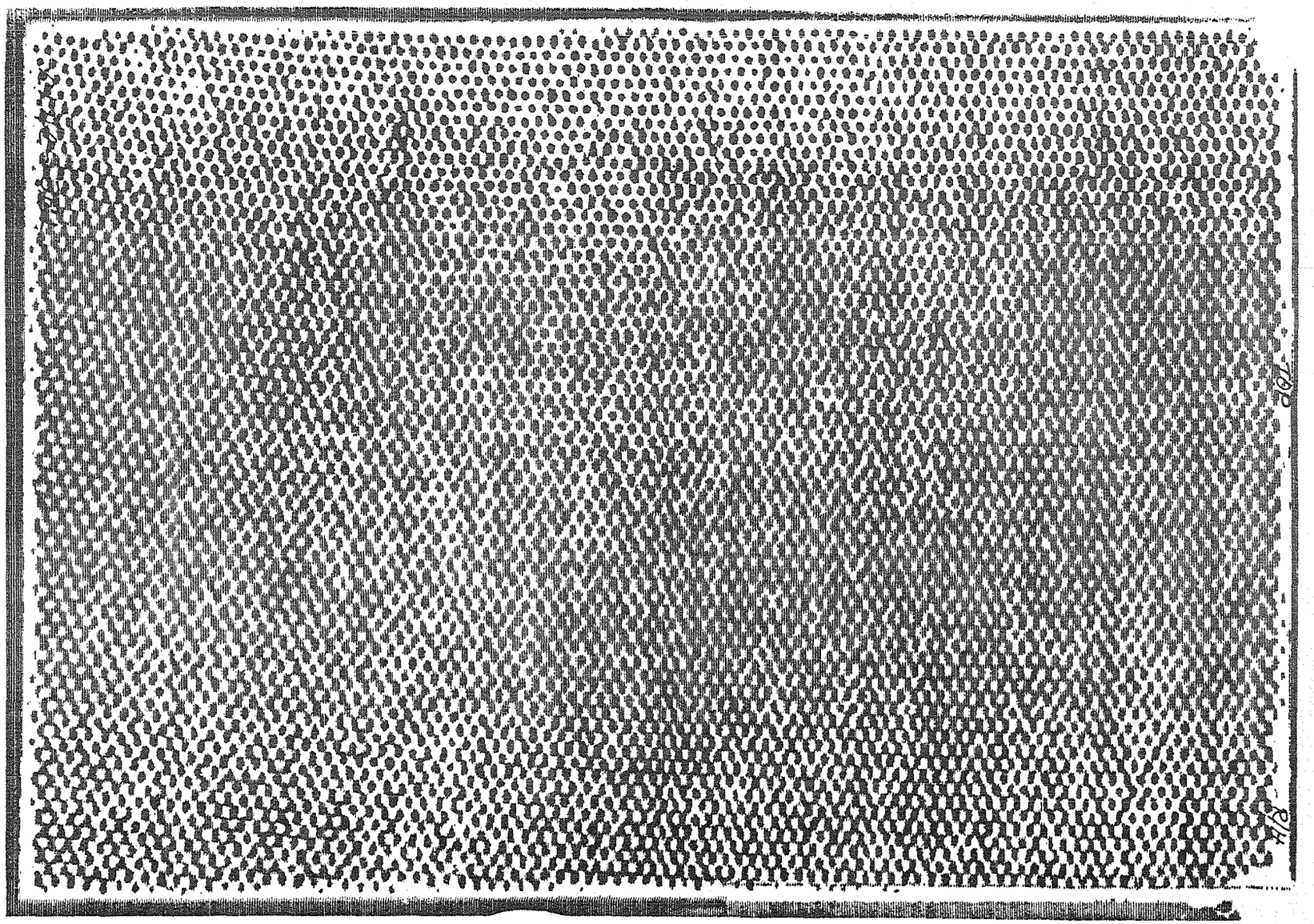


Figure 189. C-Scan of Front Spar Sandwich Panel 18.5 x 13.5

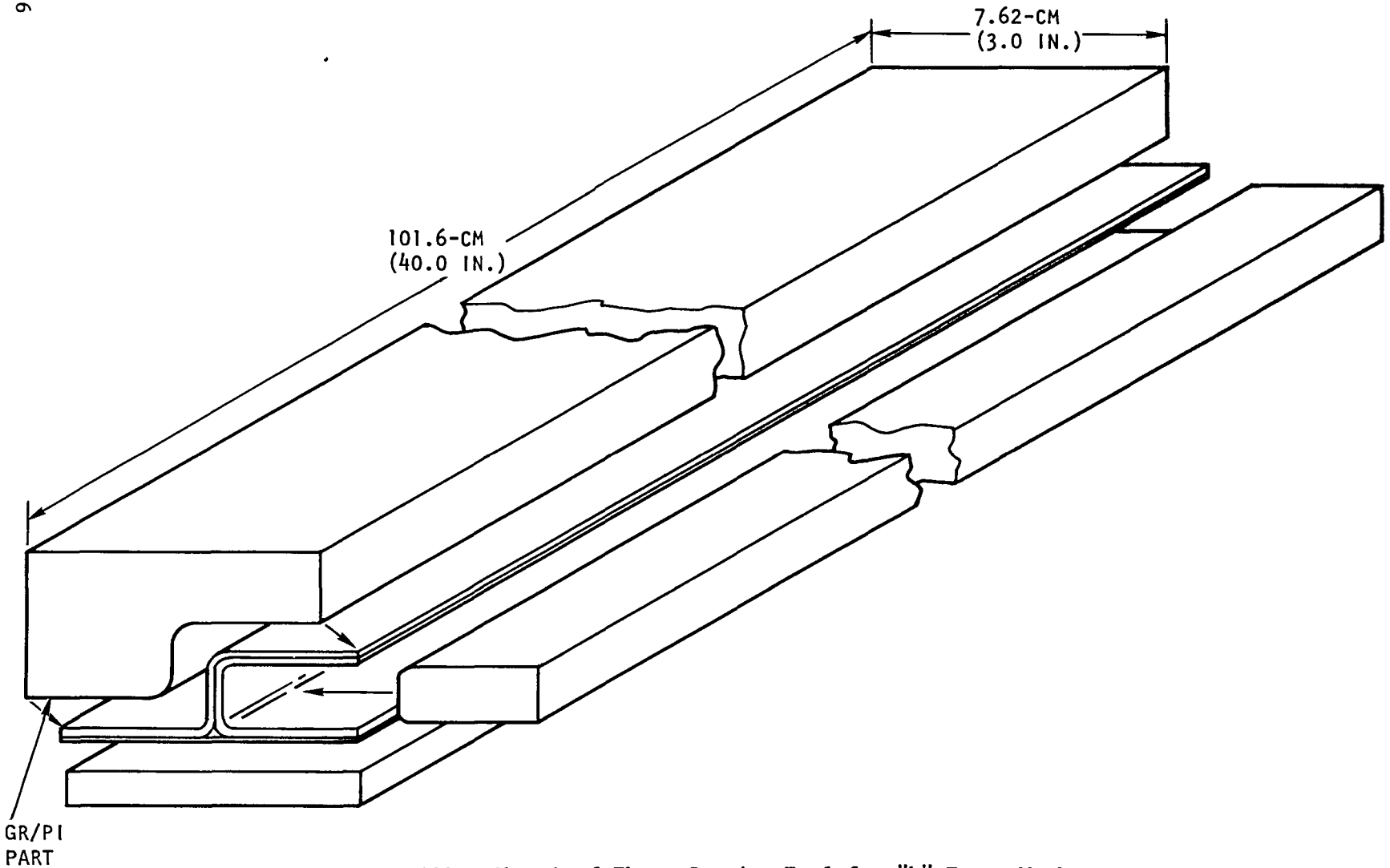


Figure 190. Sketch of Three Section Tool for "h" Frame Member

A801003 C-14

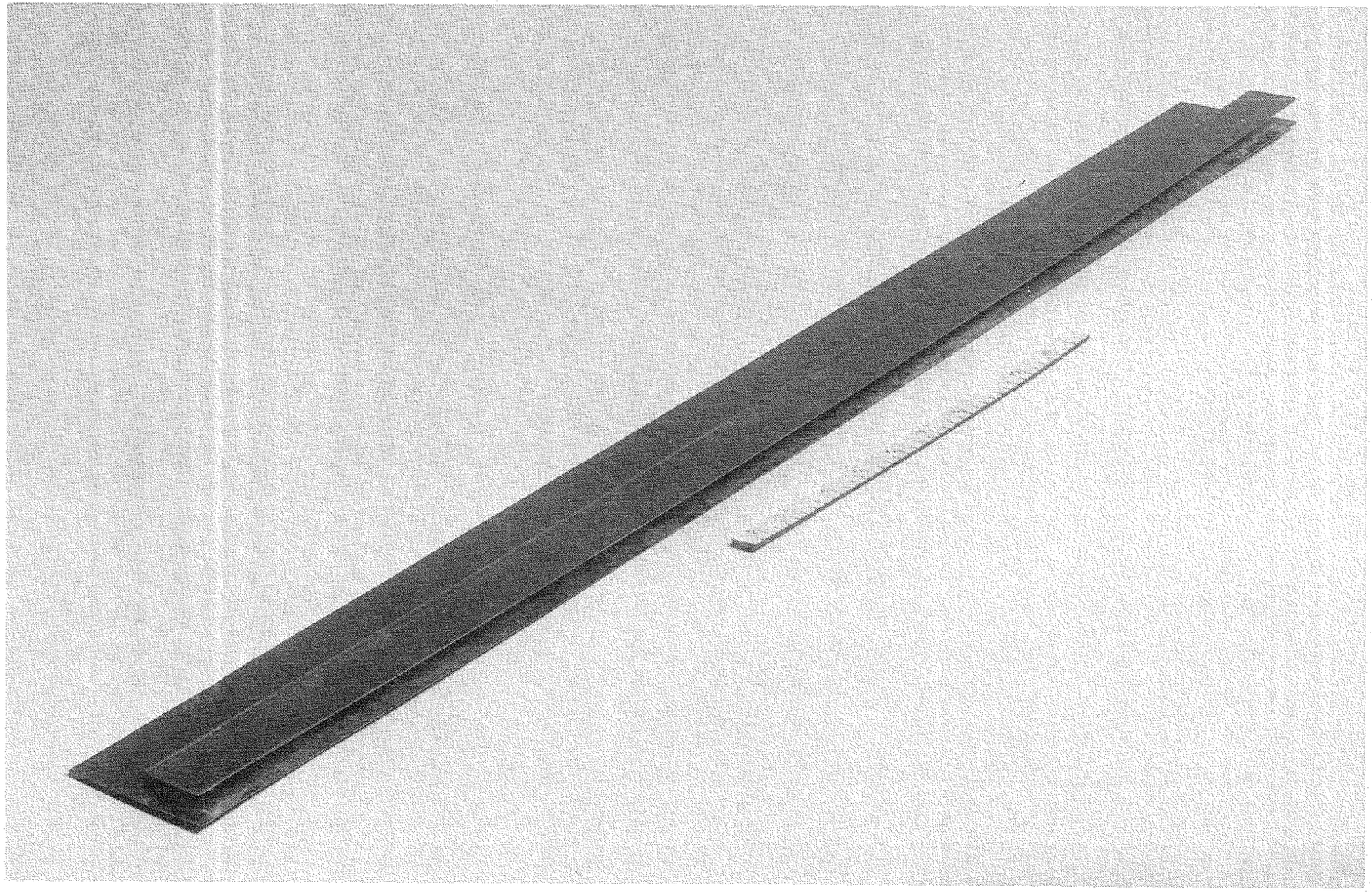


Figure 191. Cured "h" Section Before Machining

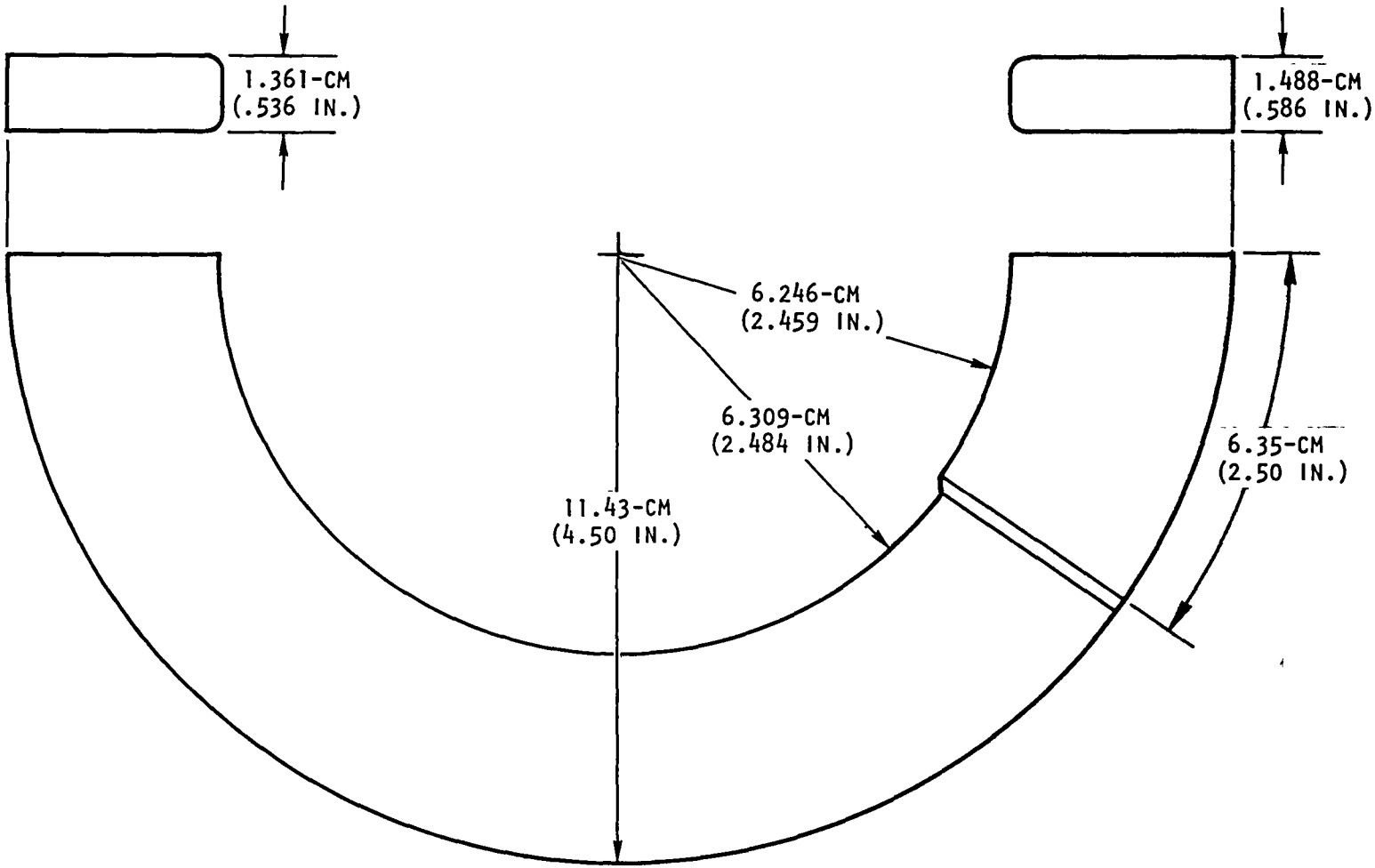


Figure 192. "U" Closeout Ring Tool

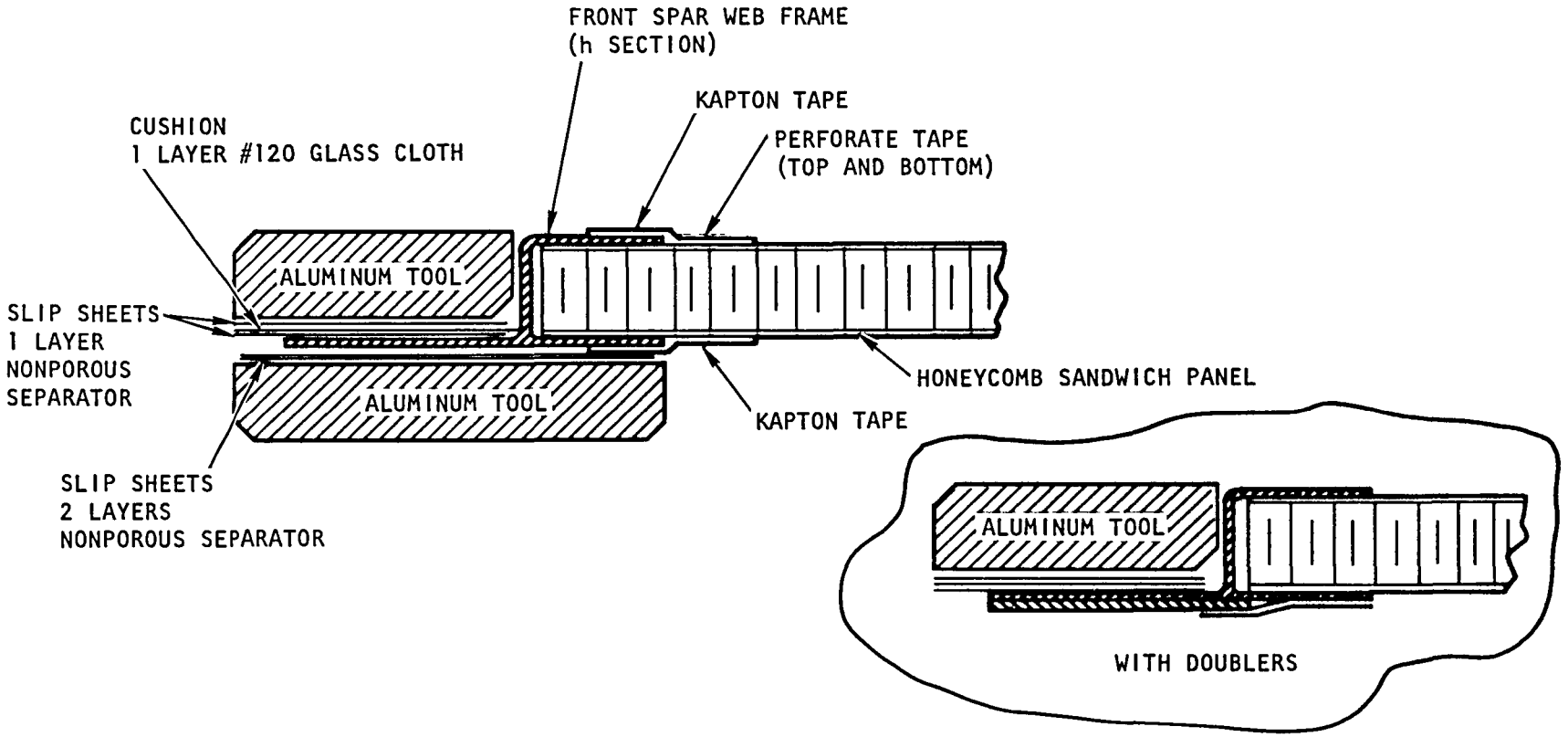


Figure 193. Bonding Configuration for TDS Front Spar Panel

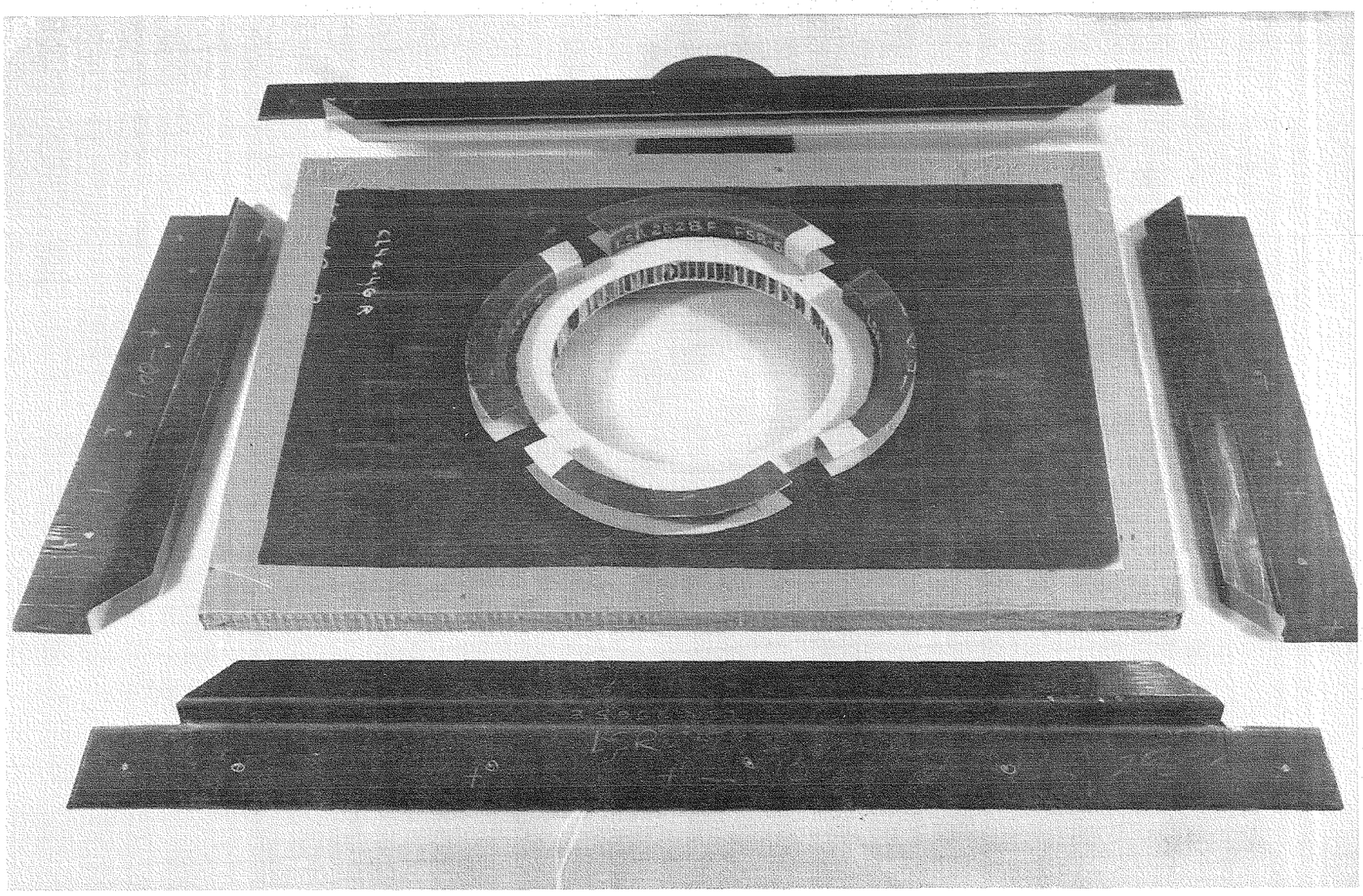


Figure 194. Elements of TDS Front Spar Panel

A810430 A-12

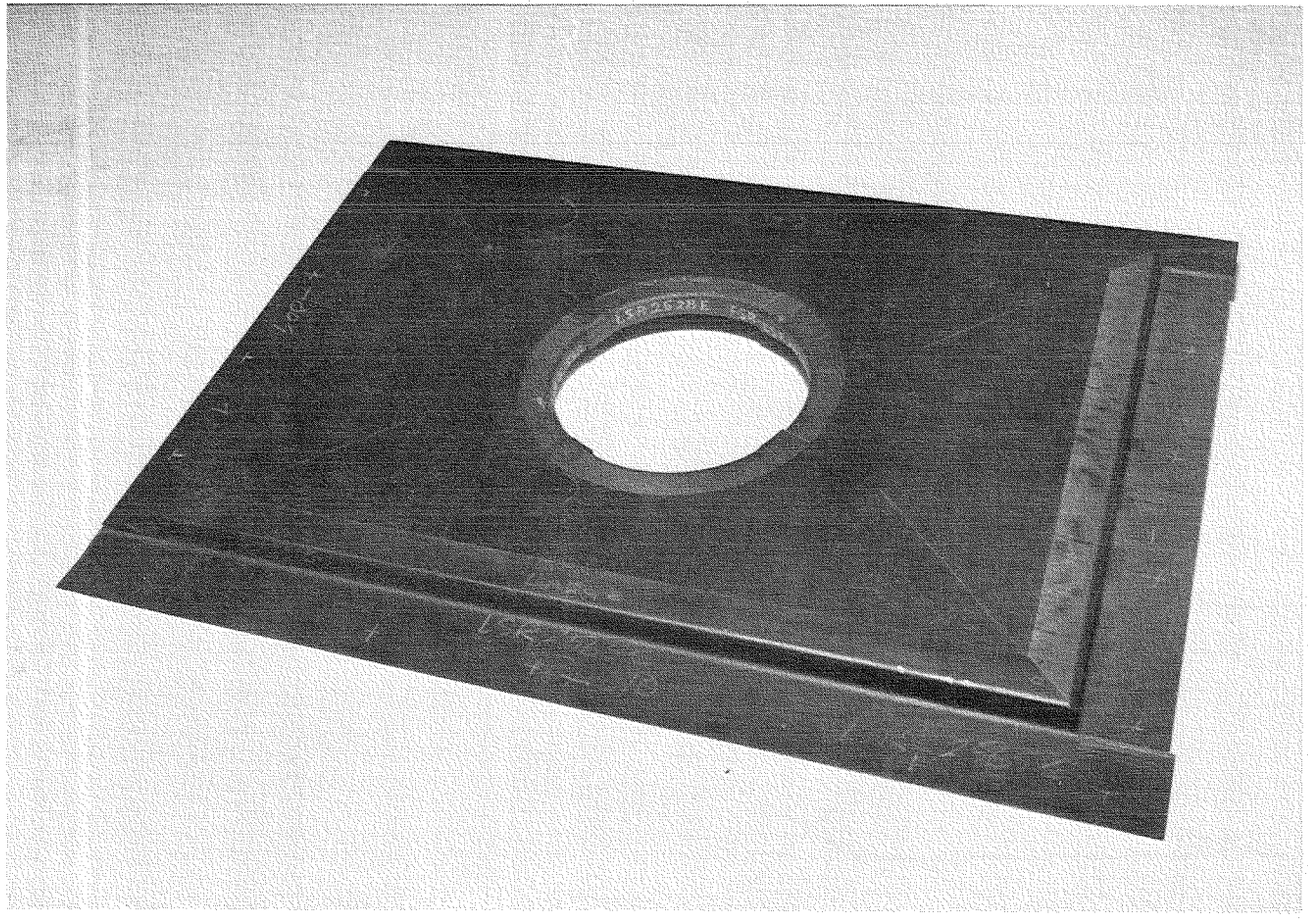


Figure 195. TDS Front Spar Panel Bonded Assembly

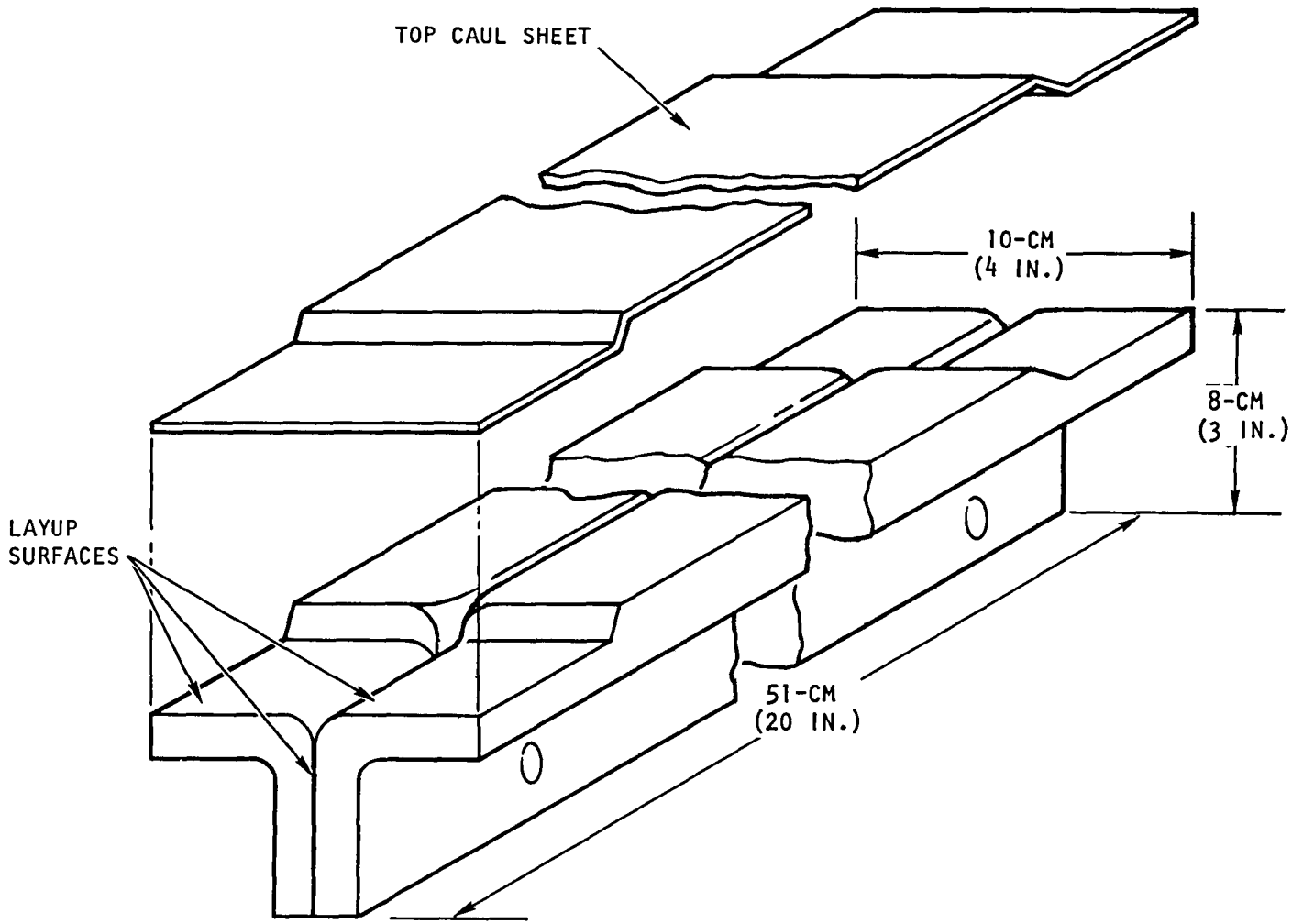


Figure 196. Typical Tool Configuration for TDS Front Spar Tee Members

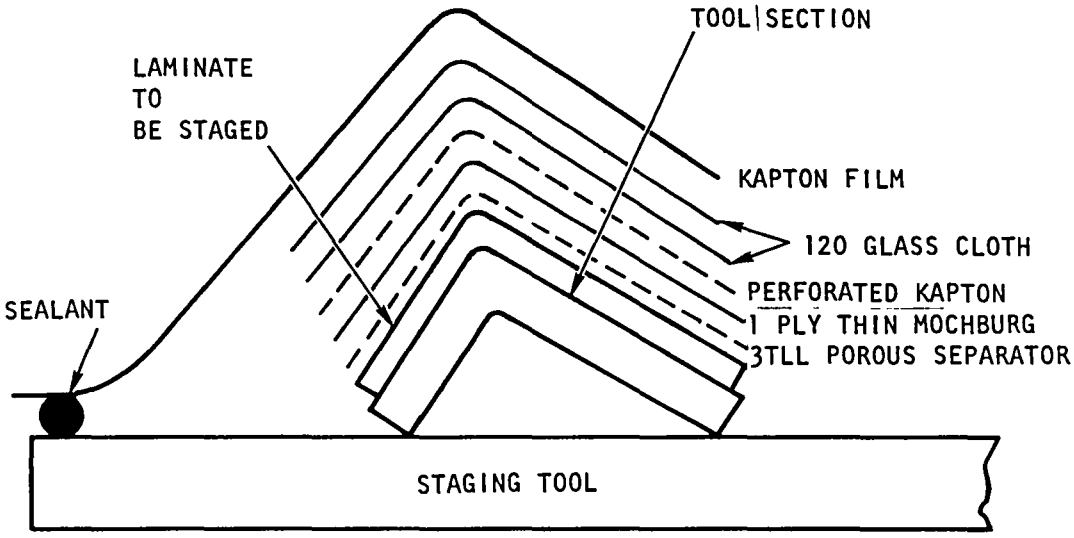


Figure 197. Imidizing Configuration for Tee Member Element

AUTOCLAVE CURE FOR TEE MEMBER (END VIEW)

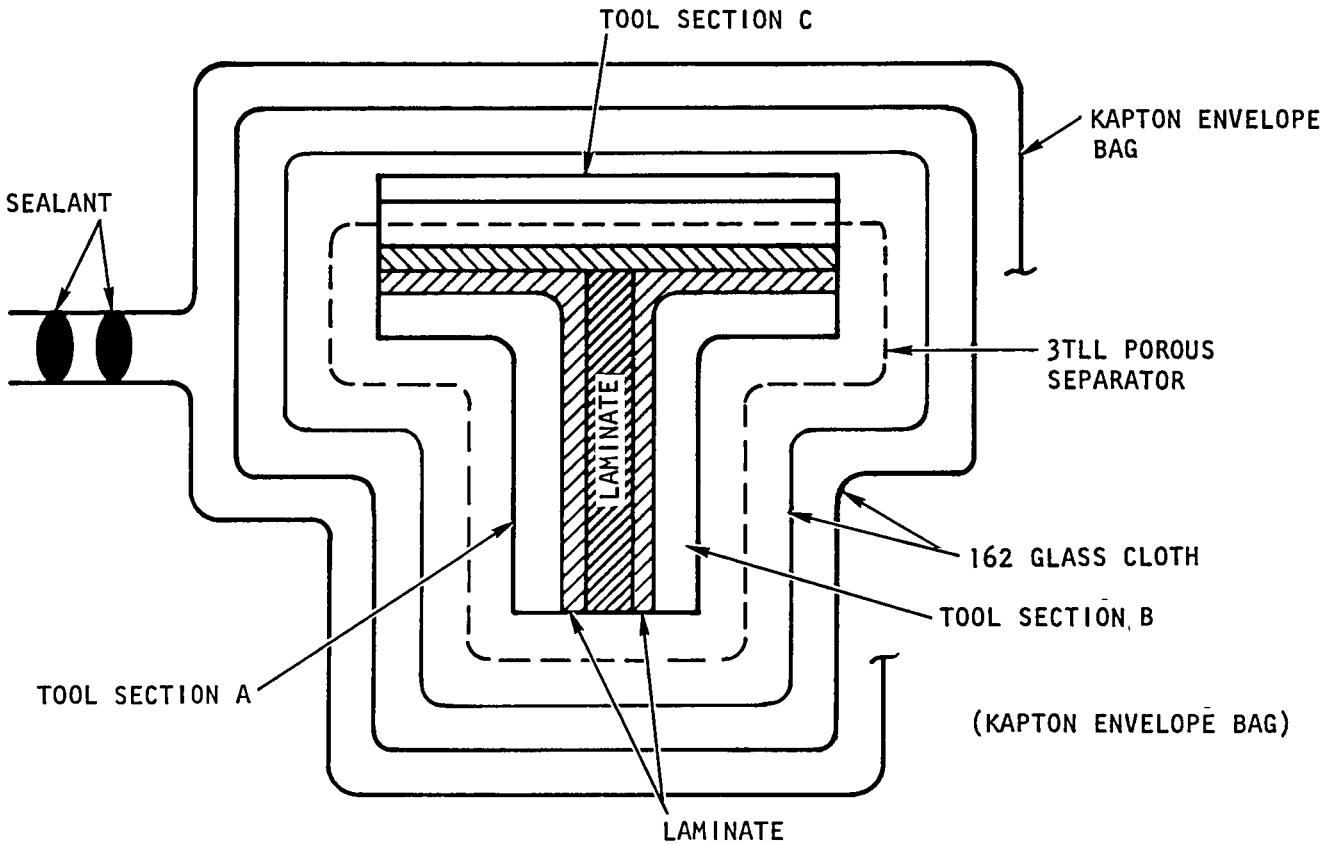


Figure 198. Tee Member Bagging for Autoclave Cure

A801023 G-11

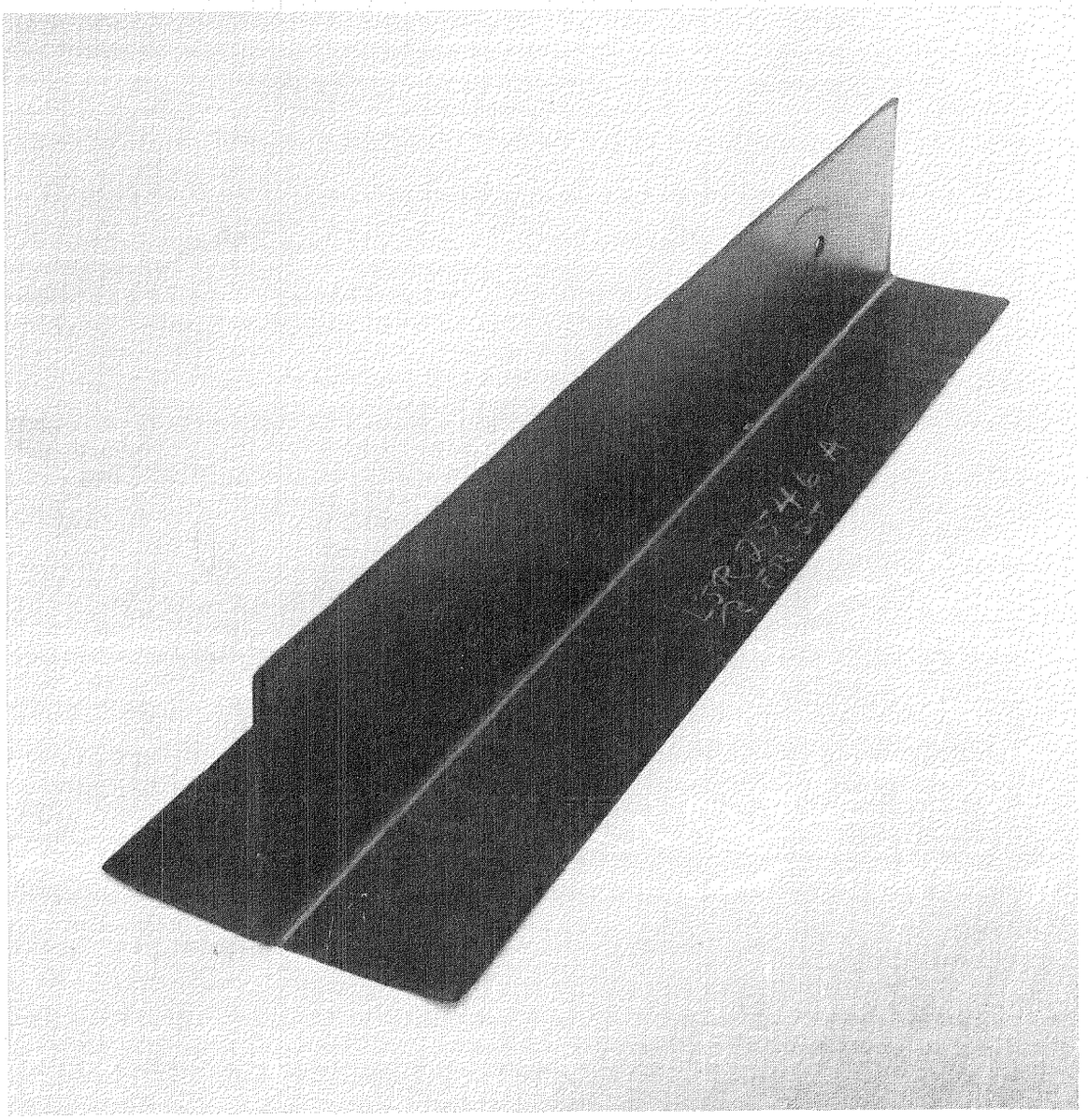


Figure 199. Cured Tee Member Before Machining

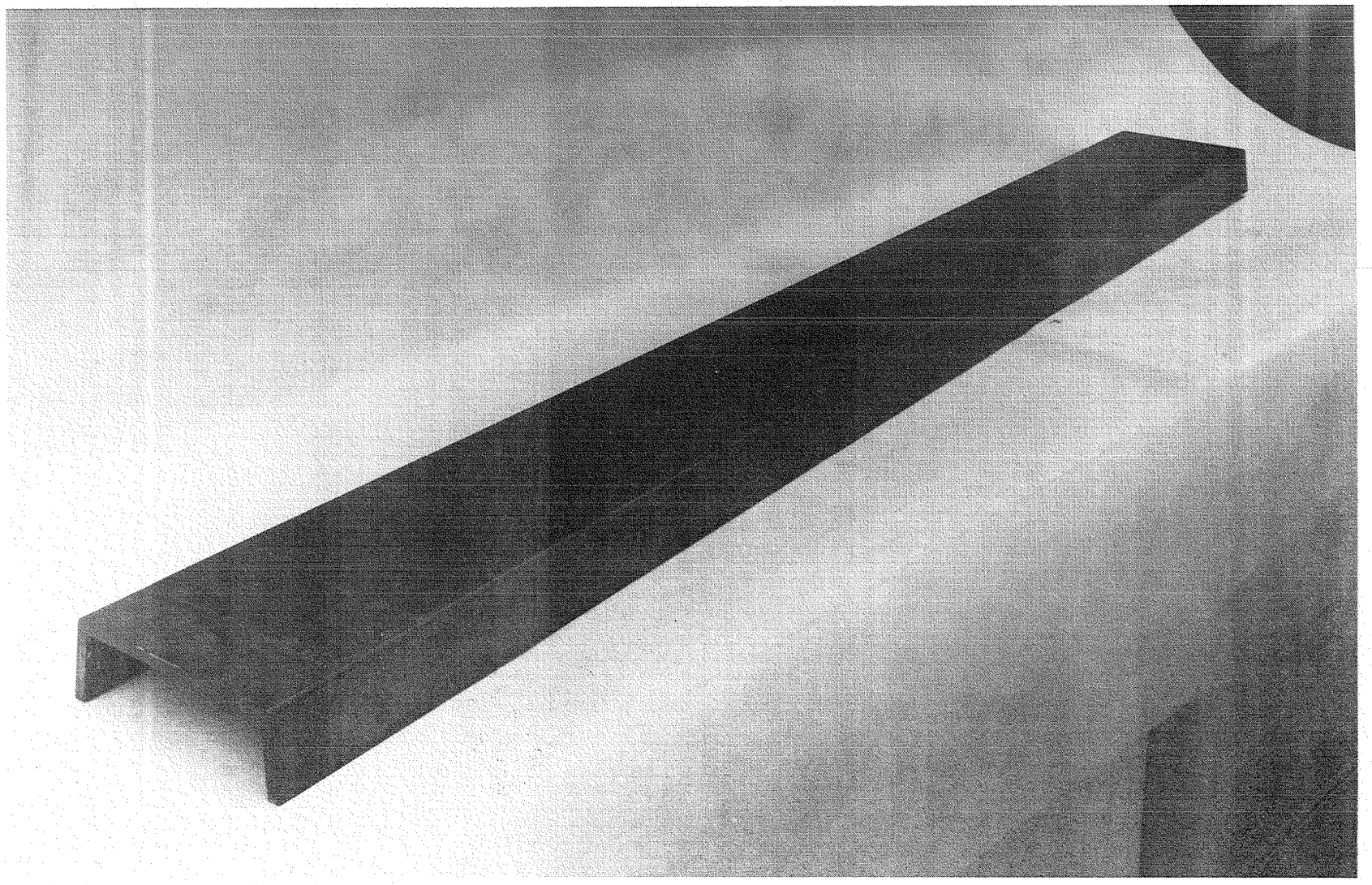


Figure 200. TDS Rear Spar Tool

A810803 A-21

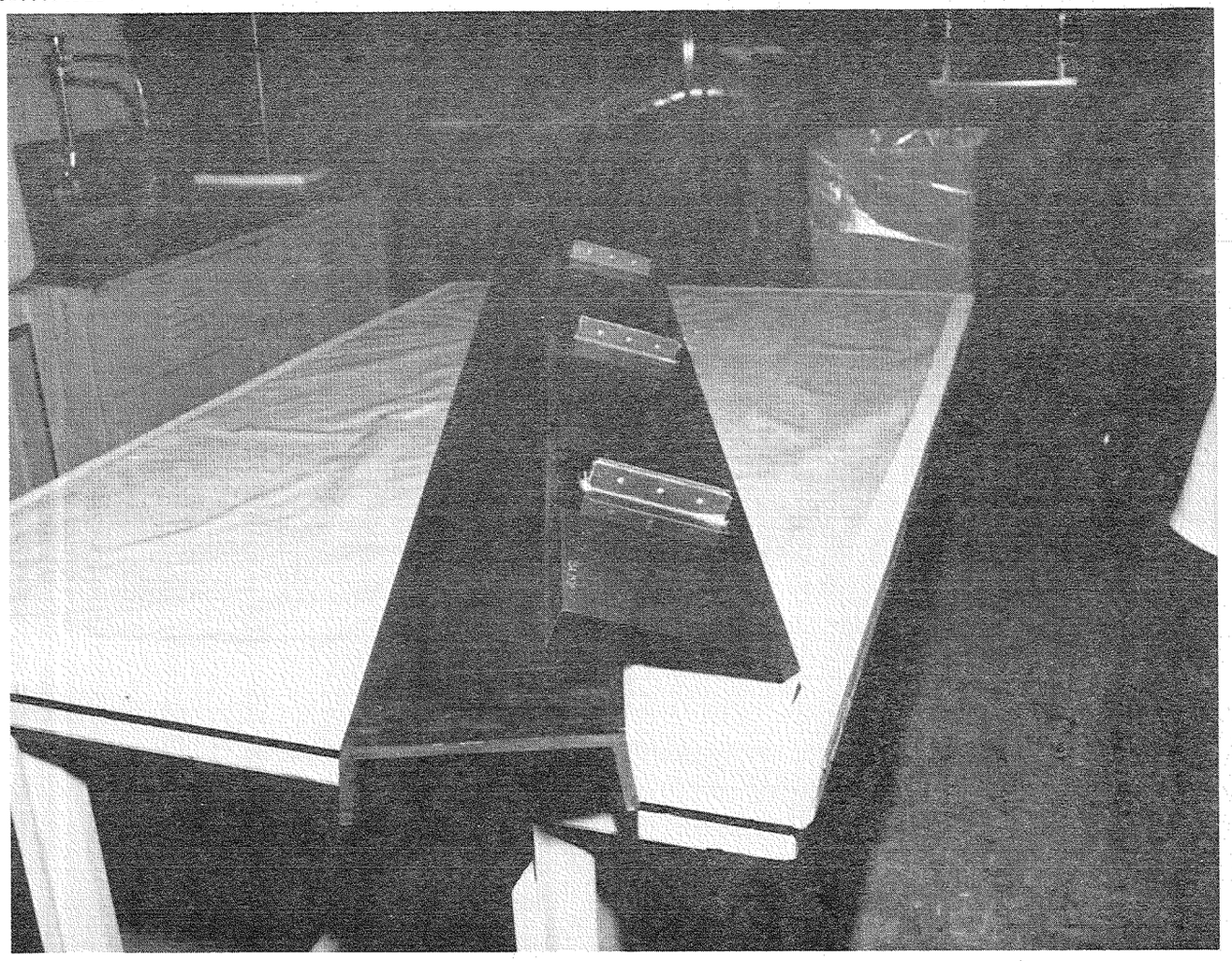


Figure 201. Load Introduction Pi Sections Bonded to the Machined TDS Rear Spar

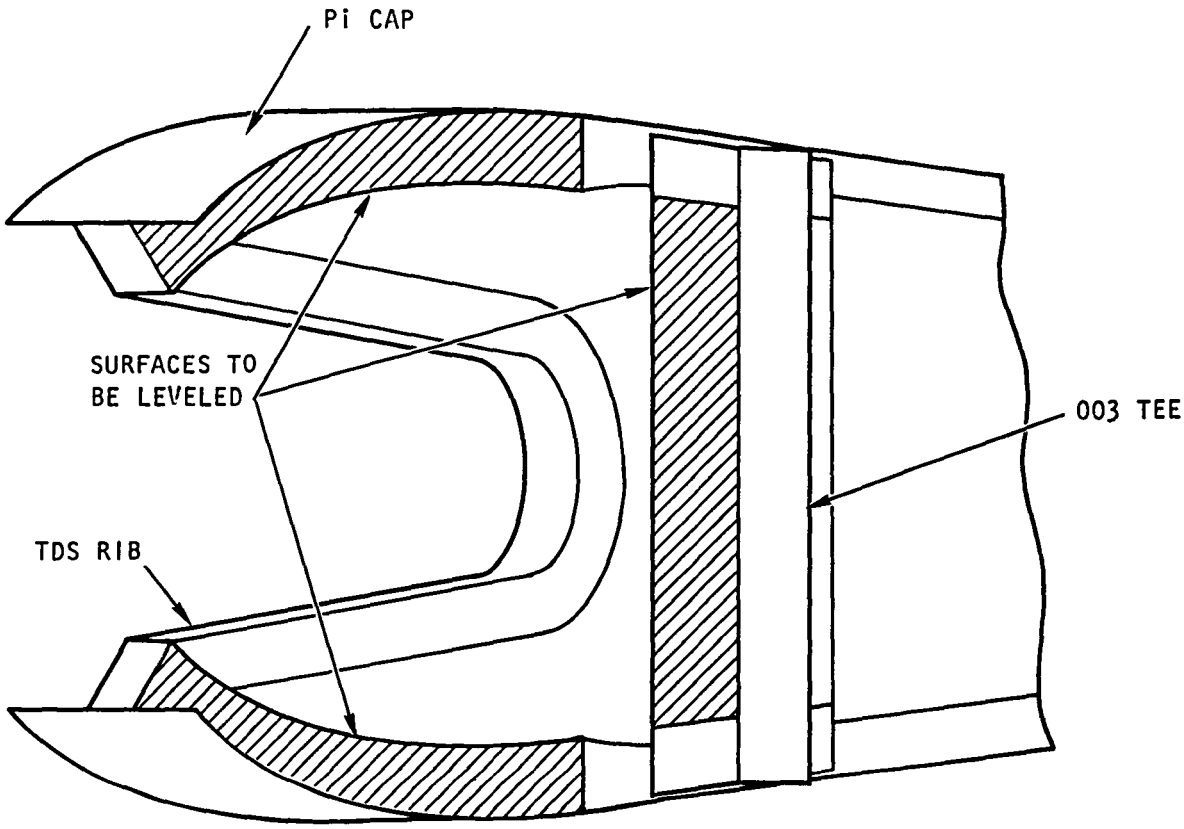


Figure 202. TDS Rib Areas Requiring Shim Bonding

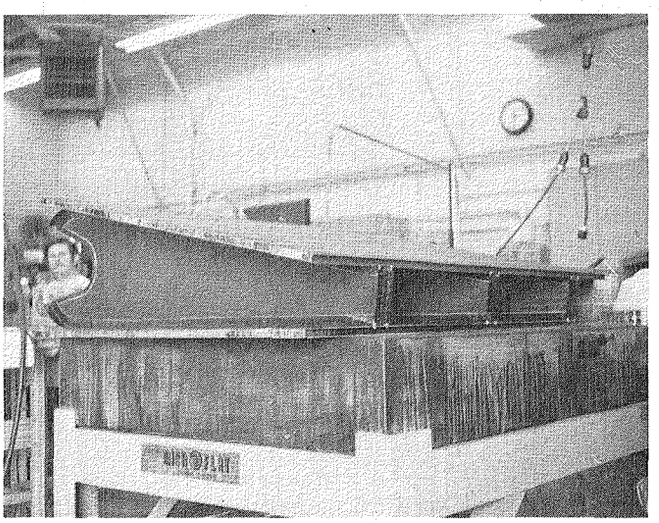


Figure 203. TDS in Rigged Condition

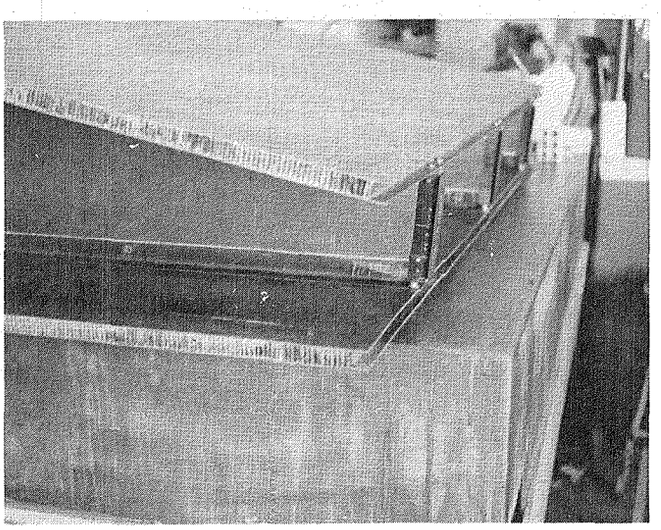


Figure 204. Mechanical Fasteners Hold Ribs to Covers at Aft End of TDS

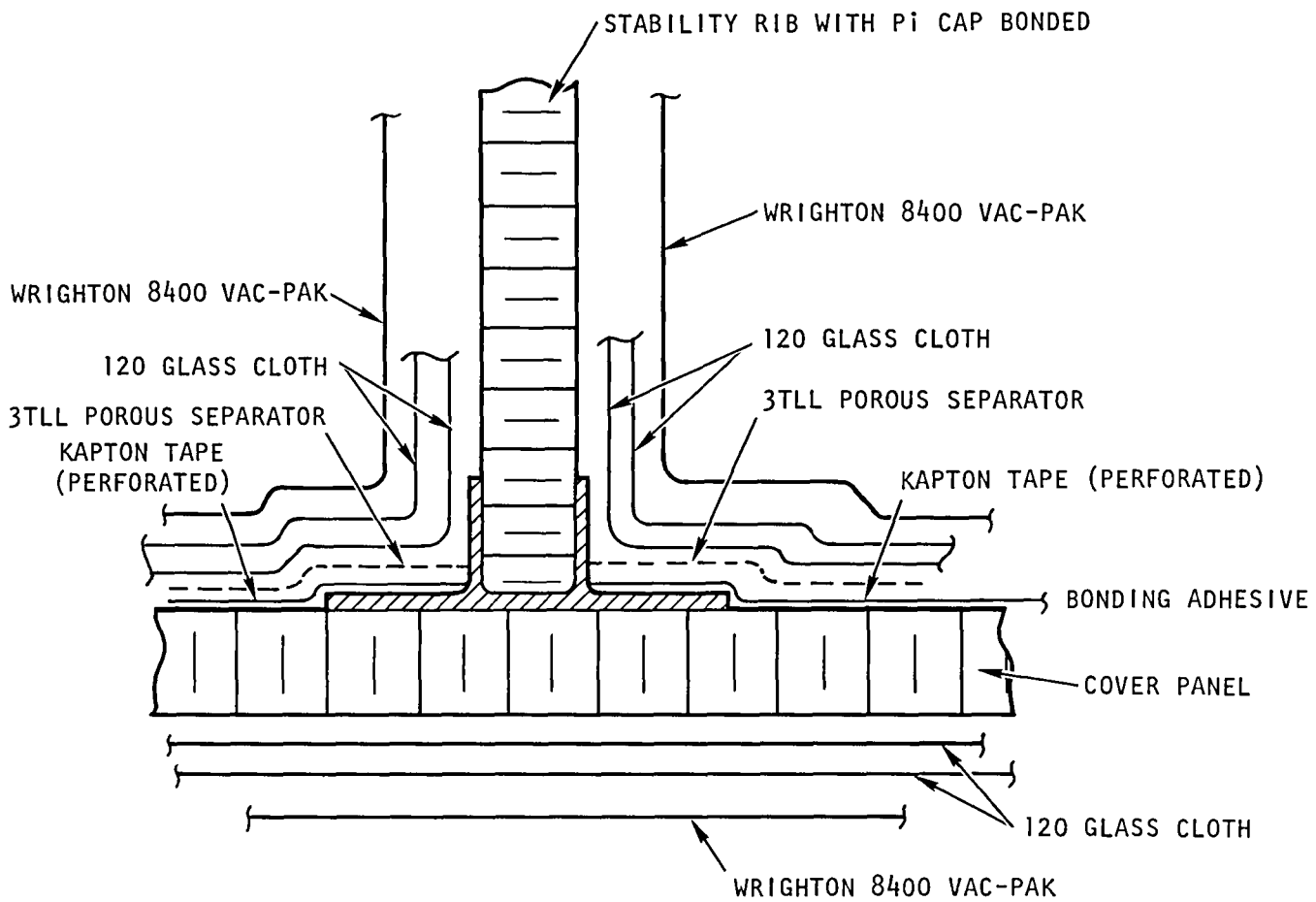


Figure 205. Bagging Configuration for Bonding Ribs to Covers

A810807 A-8

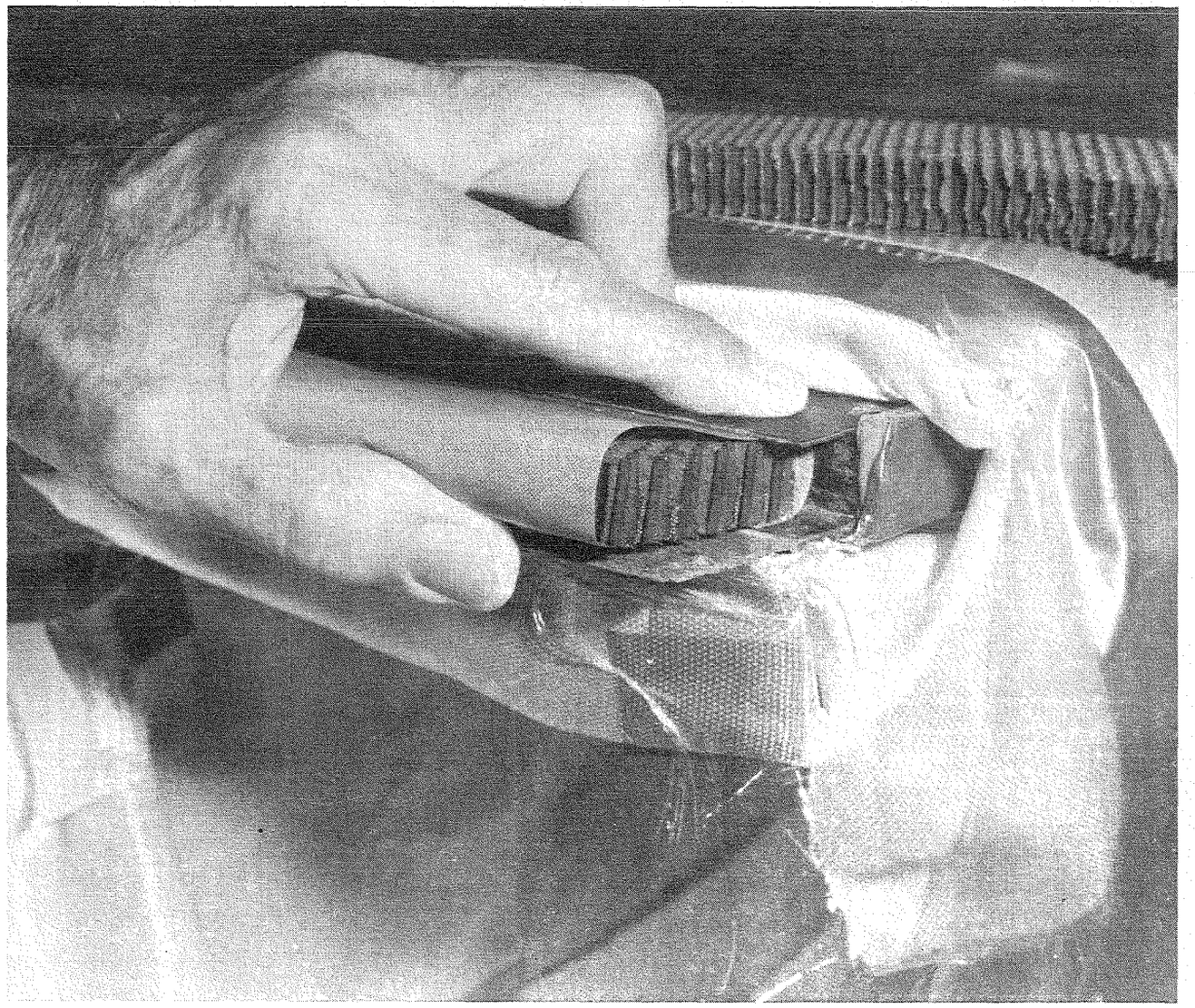


Figure 206. Honeycomb Inserted in TPS Cover Open Channel Closeout

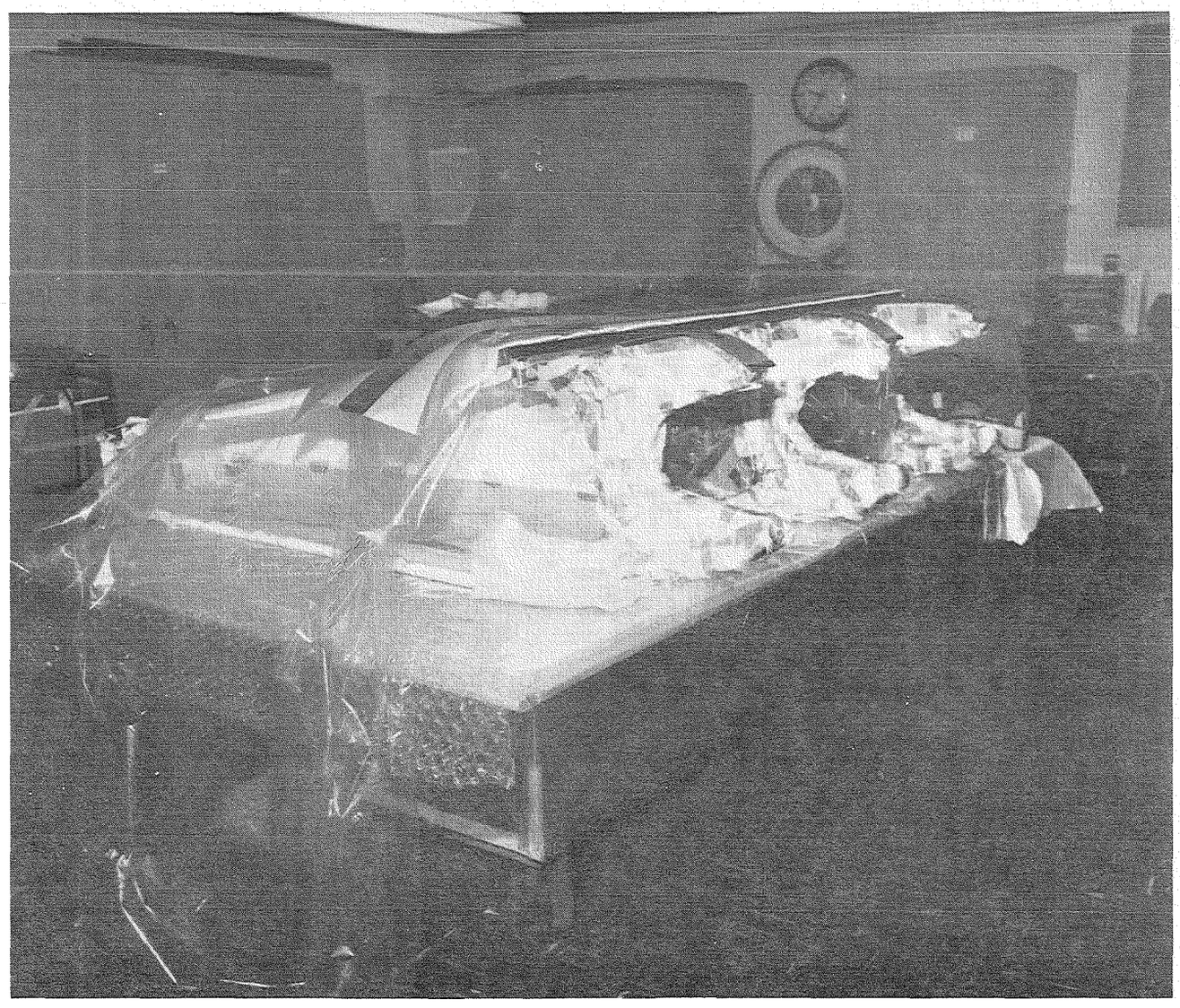


Figure 207. TDS With Breather Material in Place

A810811 G-3

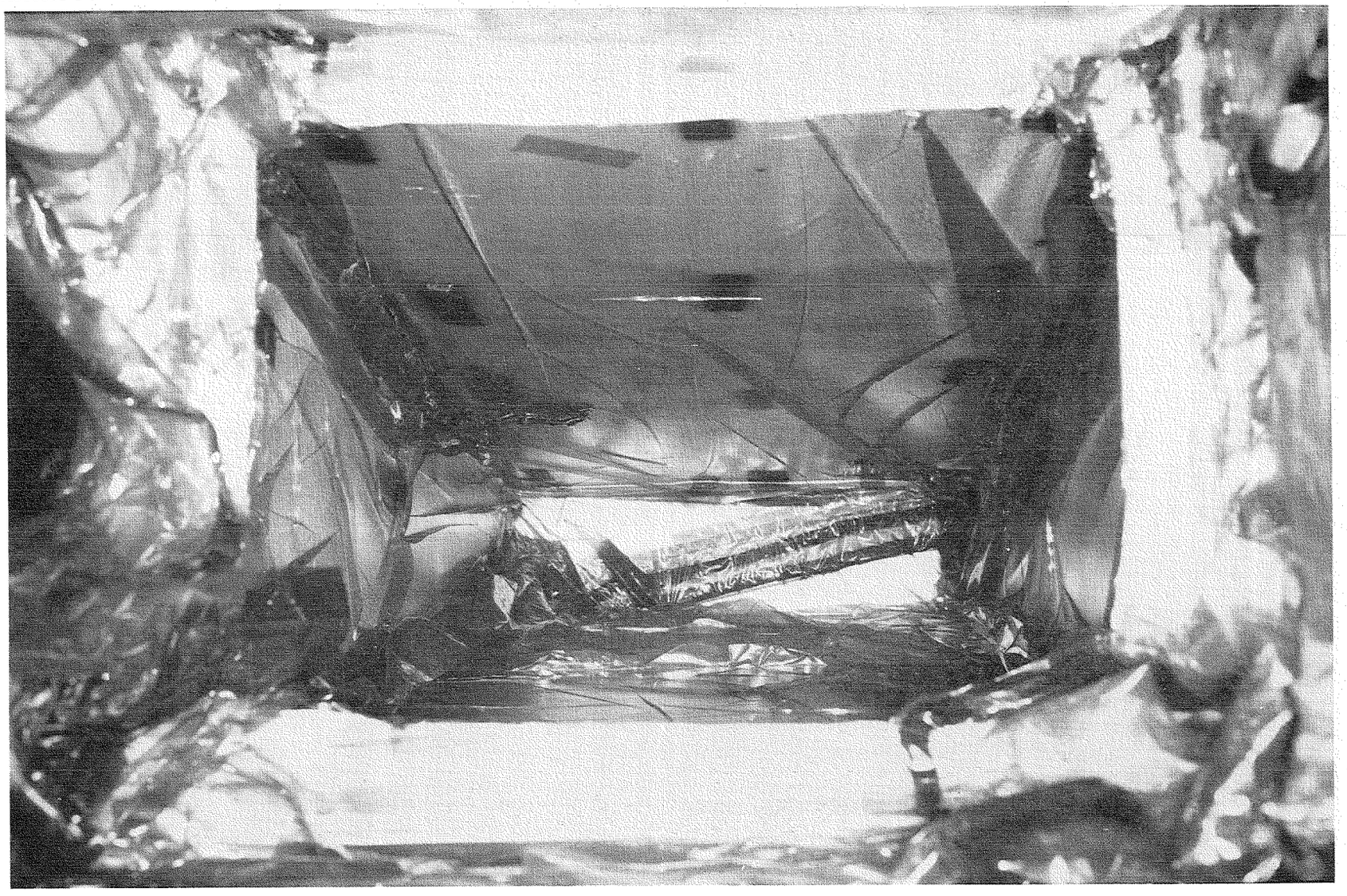


Figure 208. TDS Bagged for Bonding Cycle

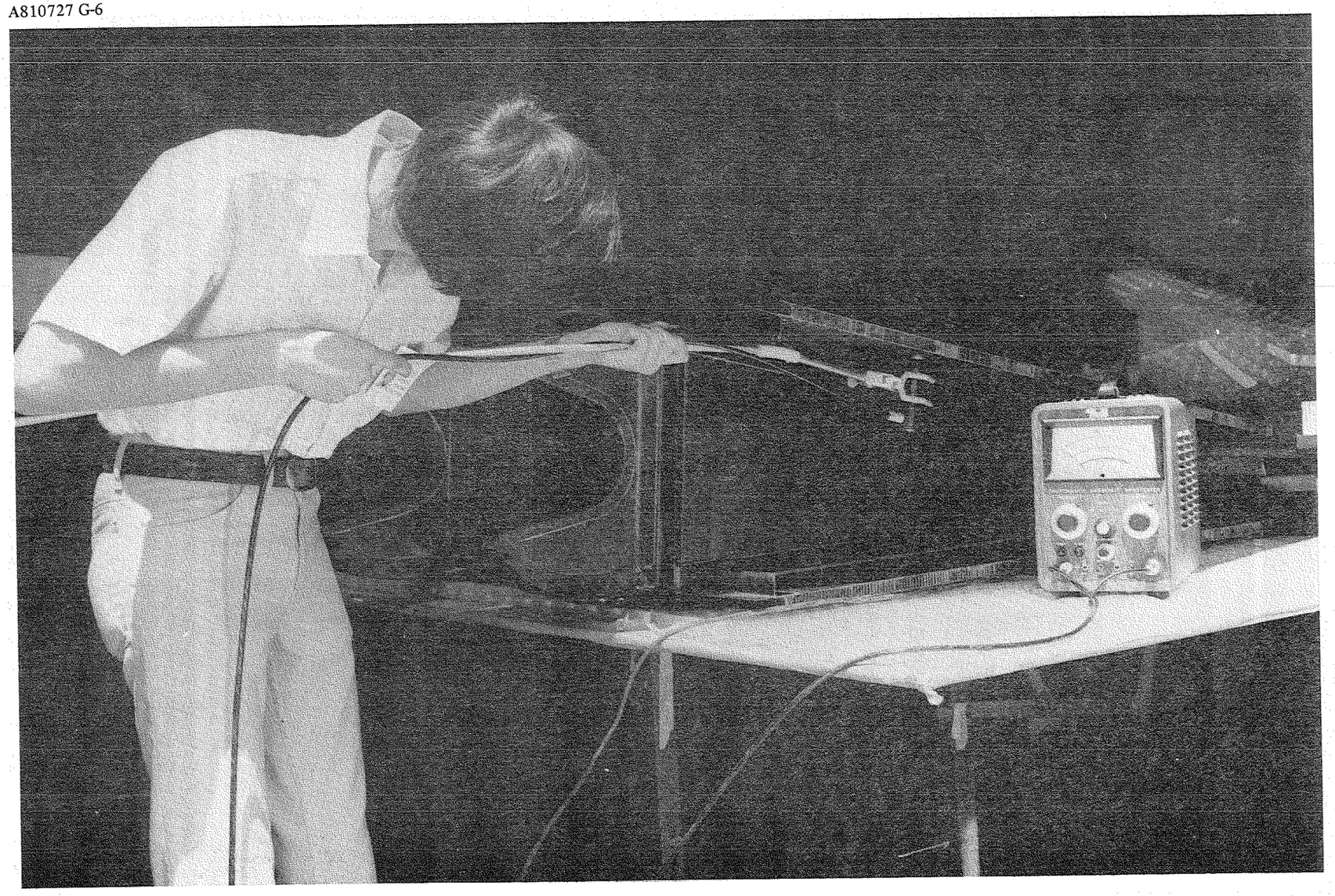


Figure 209. Harmonic Bond Testing

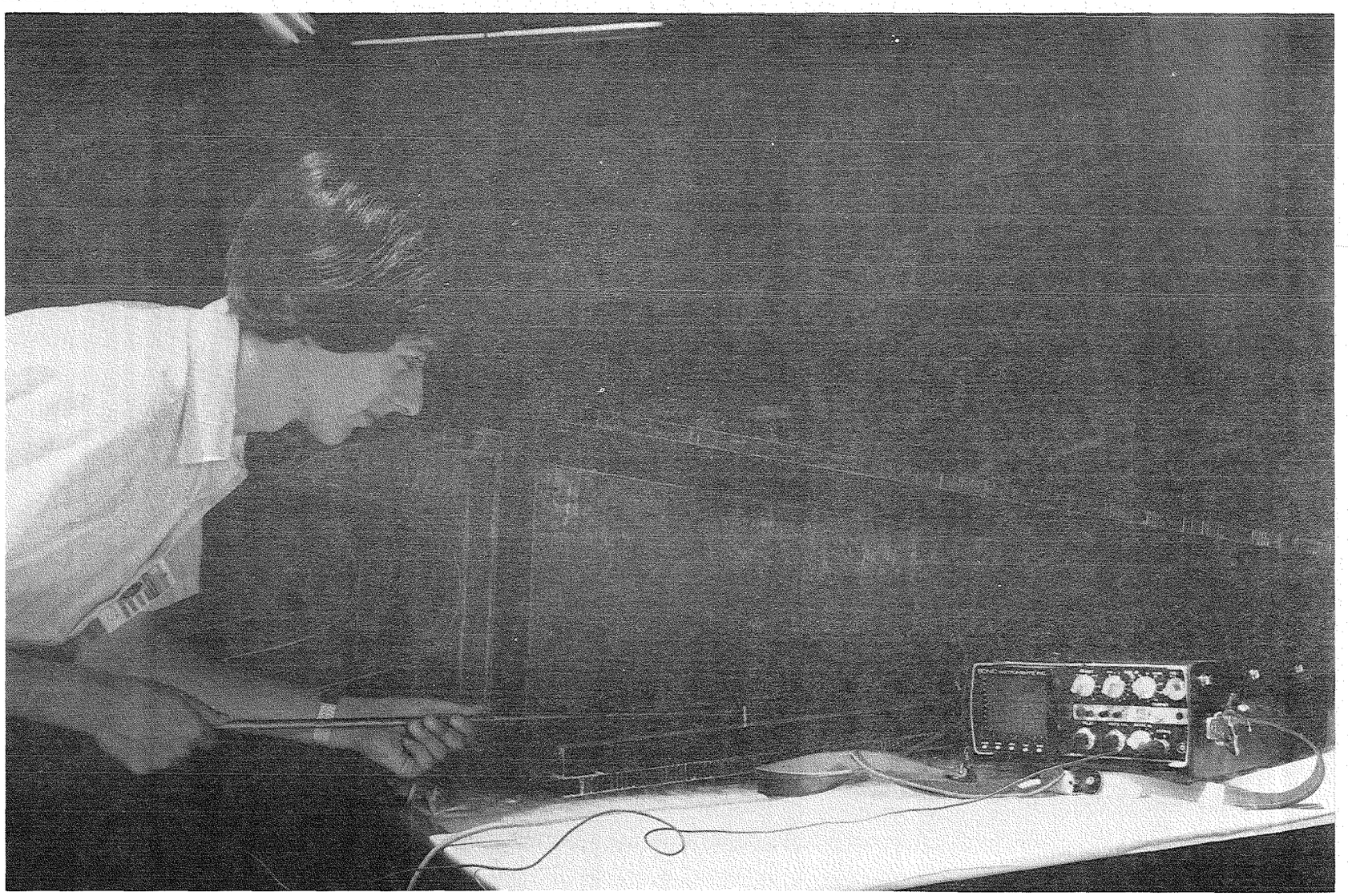


Figure 210. Ultrasonic Pulse Echo Contact Testing

A810807 A-9

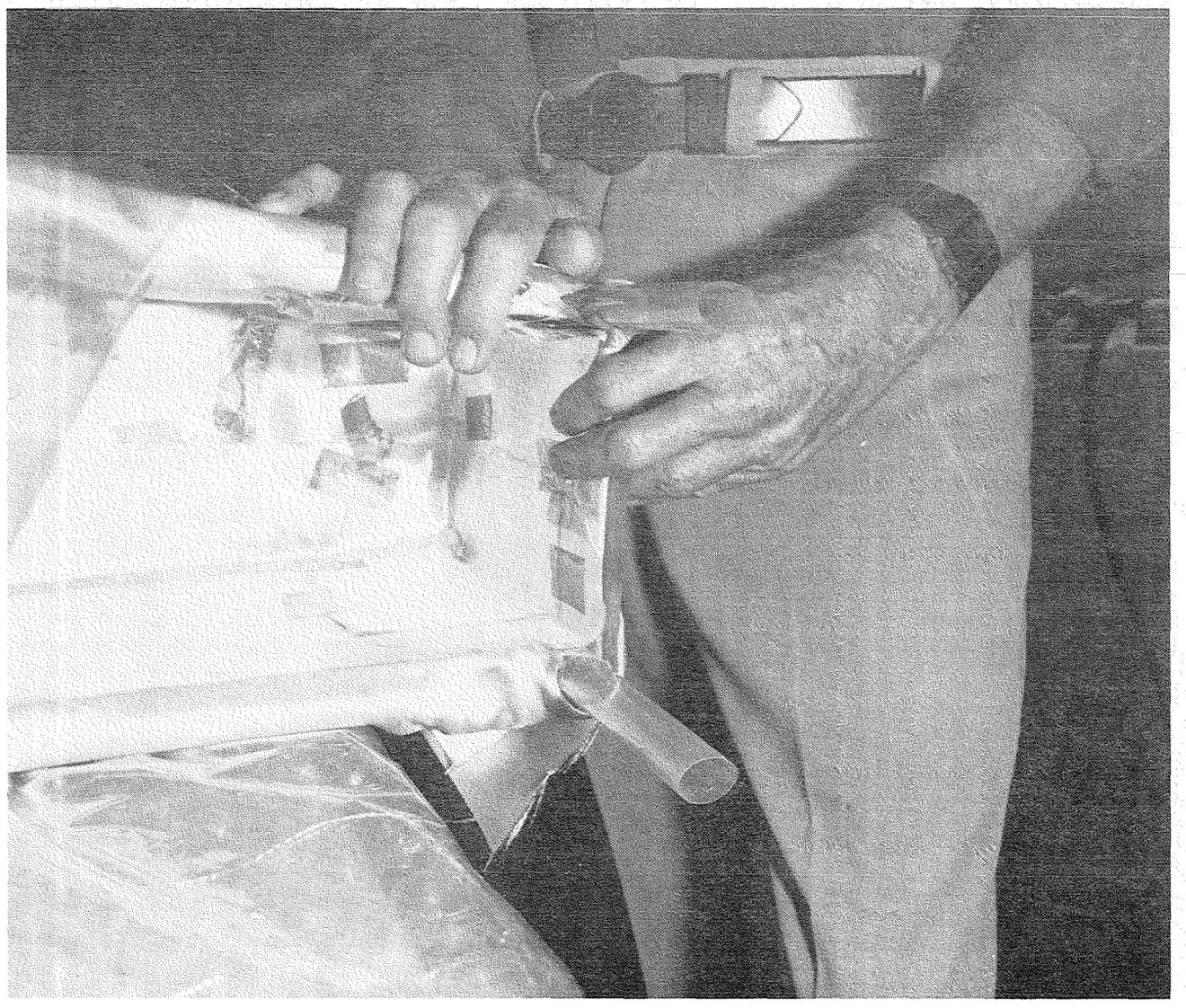


Figure 211. Teflon Tubes Inserted in Aft Open Channel Closeouts

A810807 A-38

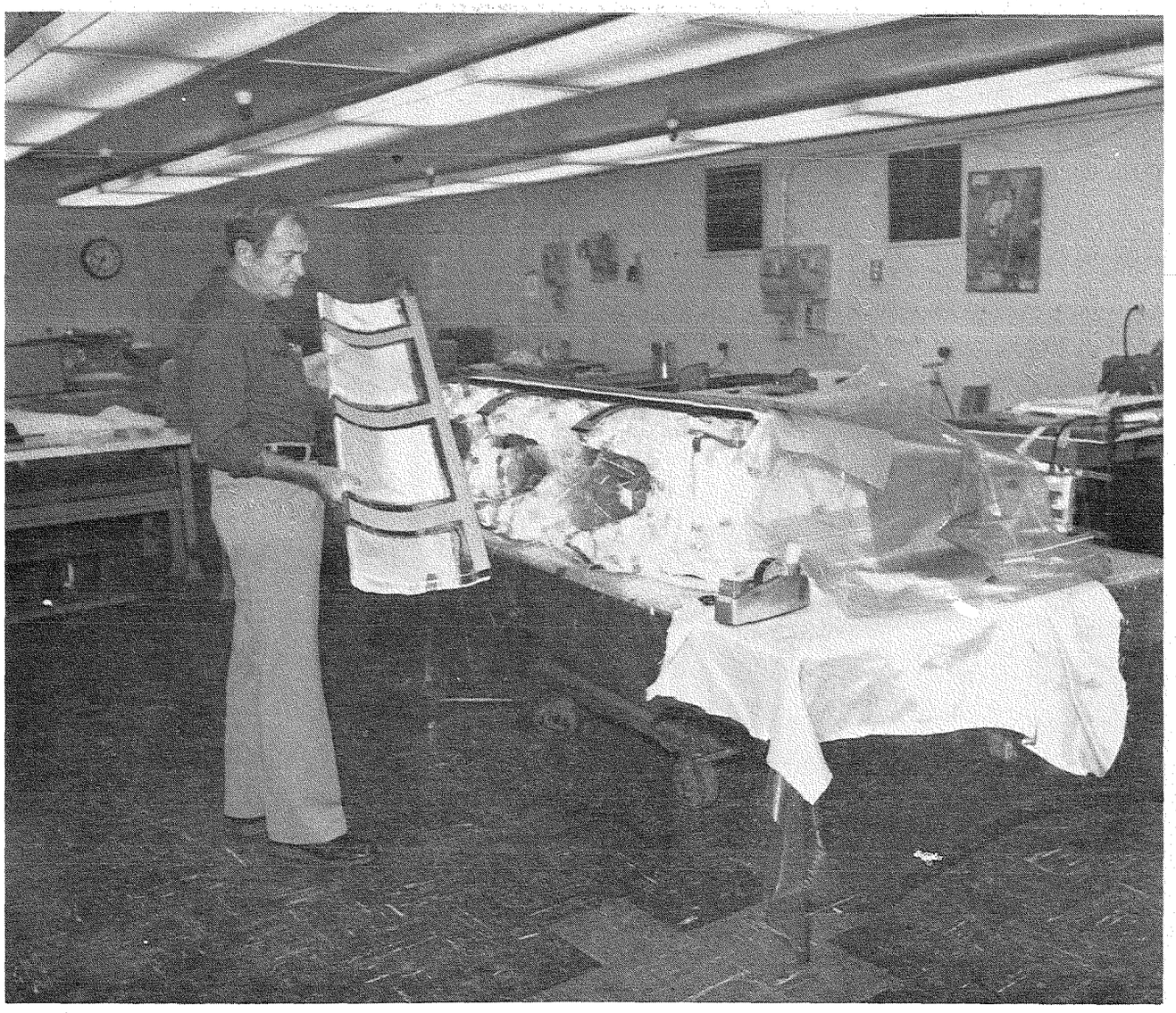


Figure 212. Lower Leading Edge Cover With Breather Material in Place

Figure 213. TDS With Lower Leading Edge Cover and Aft Spar Installed and all Breather Material in Place

A810807 A-34

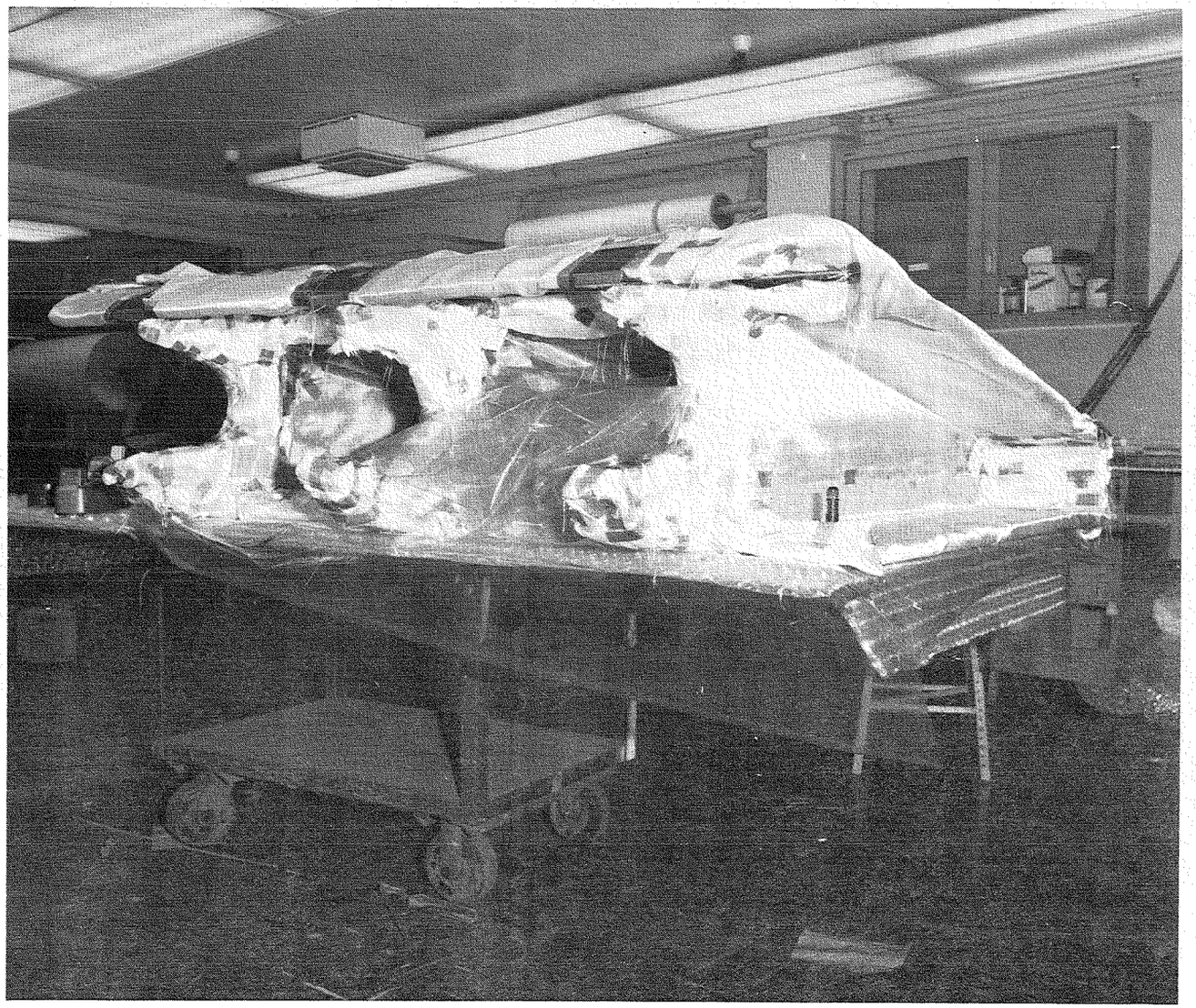


Figure 214. TDS Ready for Bagging

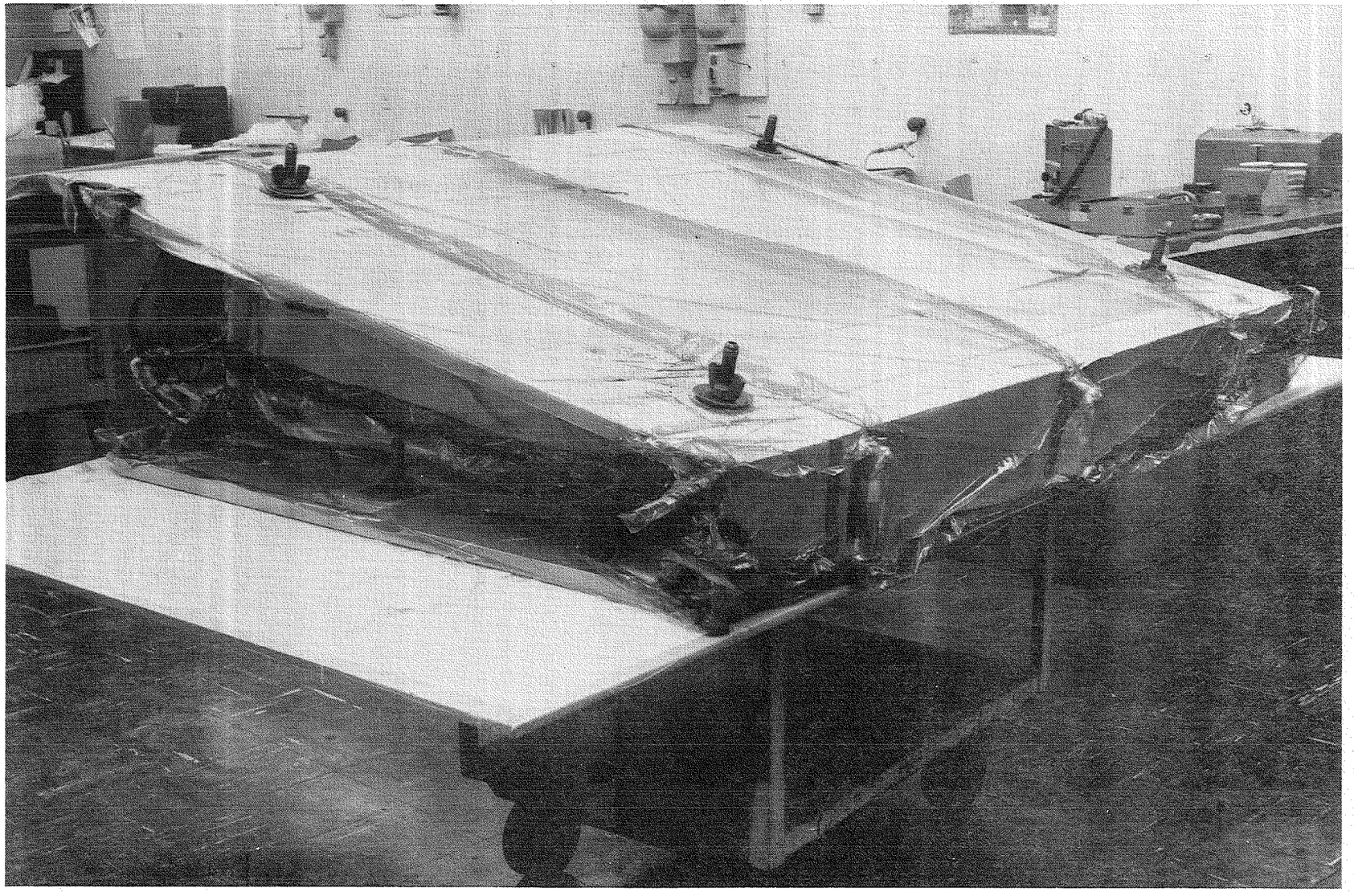


Figure 215. TDS Bagged for Bonding Cycle

A810811 G-2

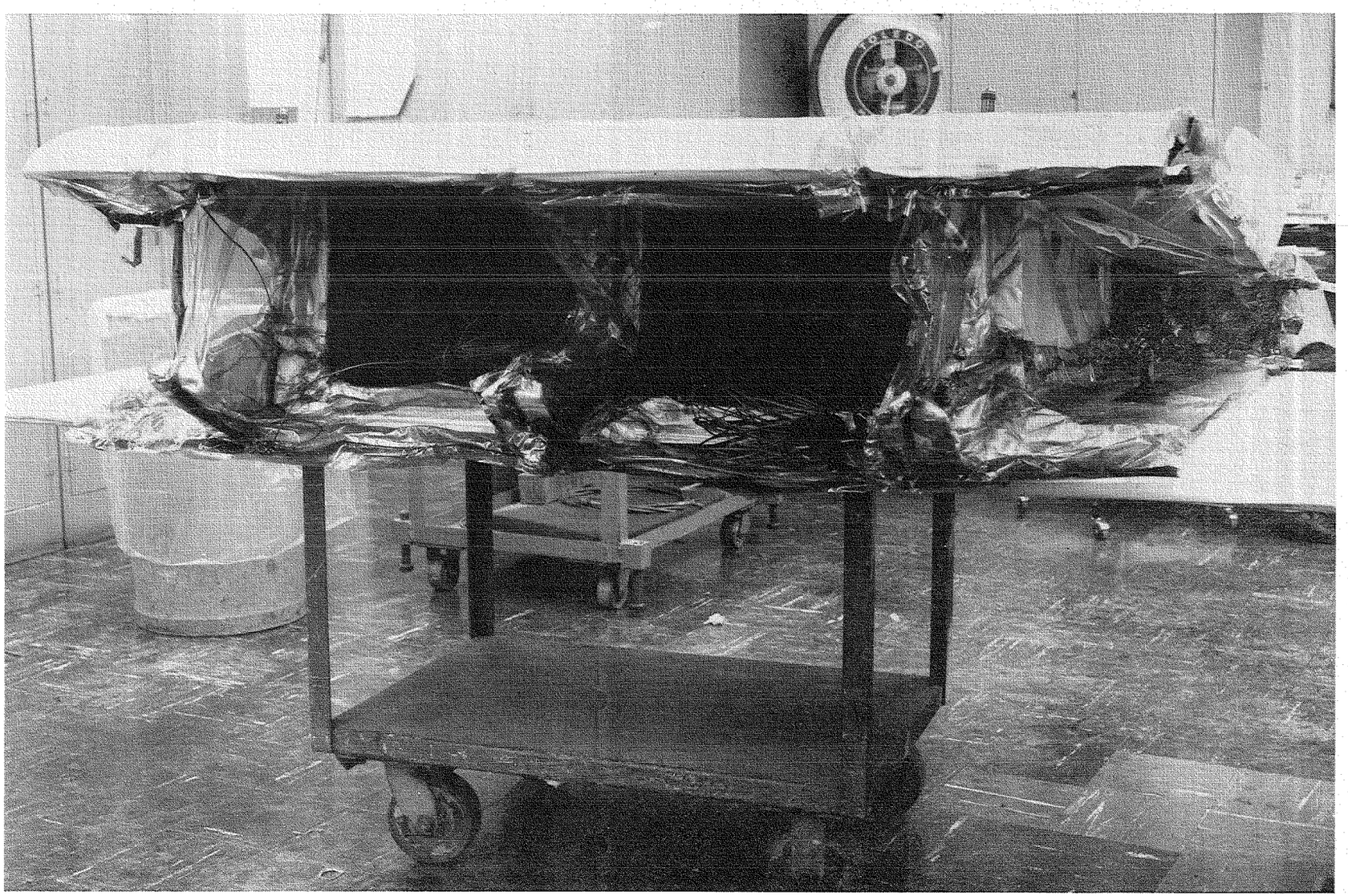


Figure 216. TDS Bagged for Bonding Cycle

A810803 A-19

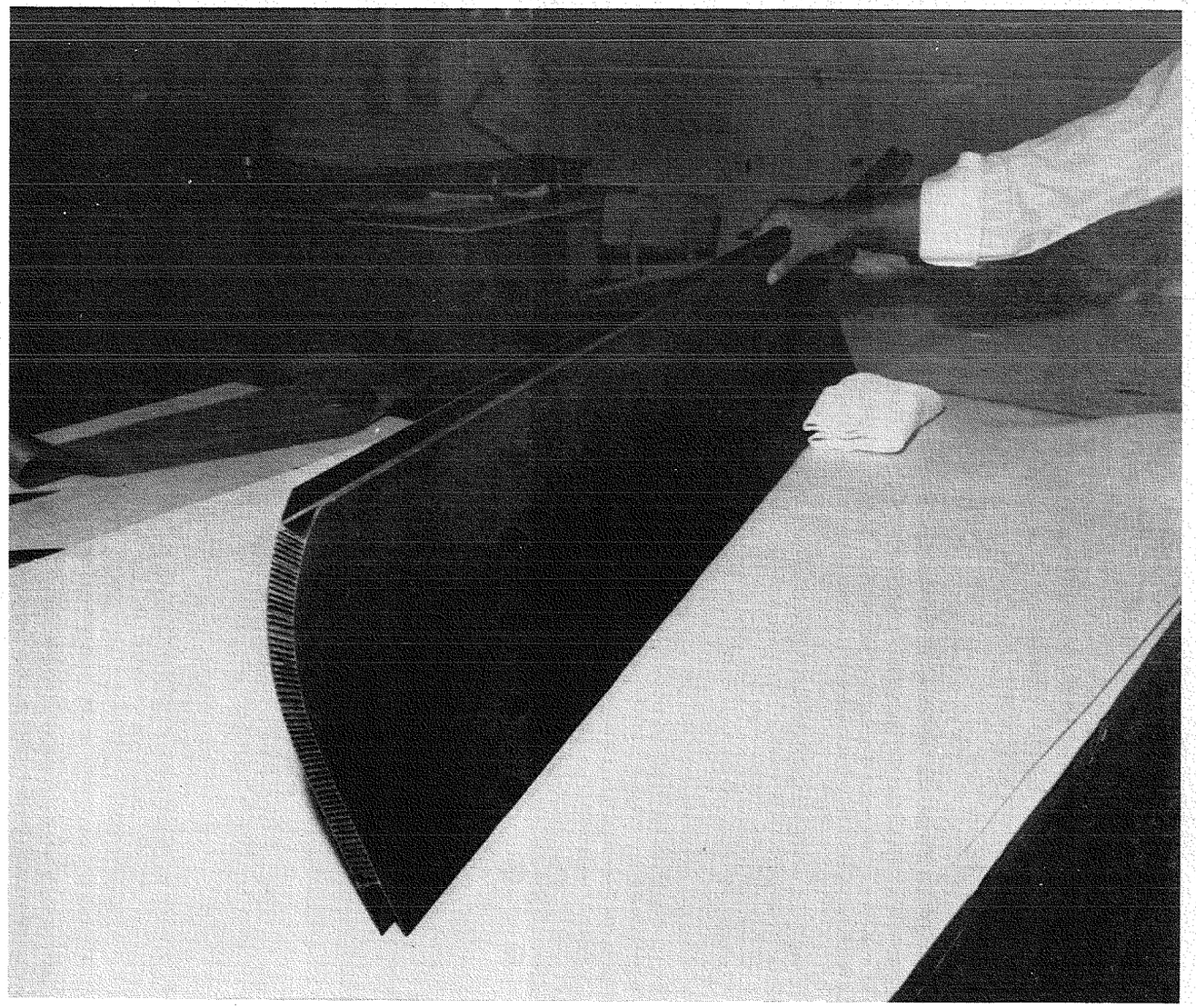


Figure 217. TDS Upper Leading Edge Cover Before Machining

A810803 A-23

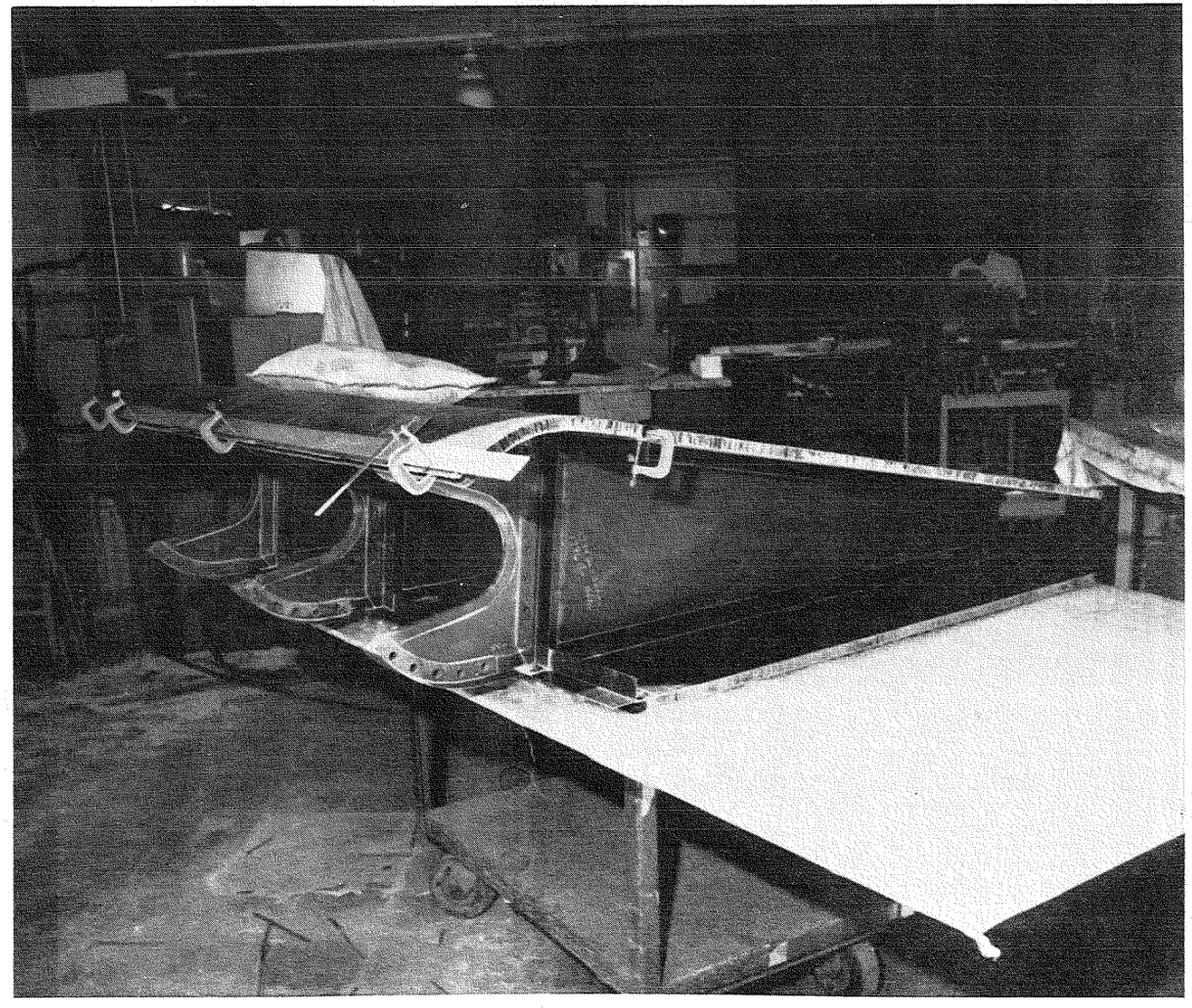


Figure 218. Partially Trimmed Upper Leading Edge Cover Clamped in Position on Bonded TDS Assembly

A810803 A-25

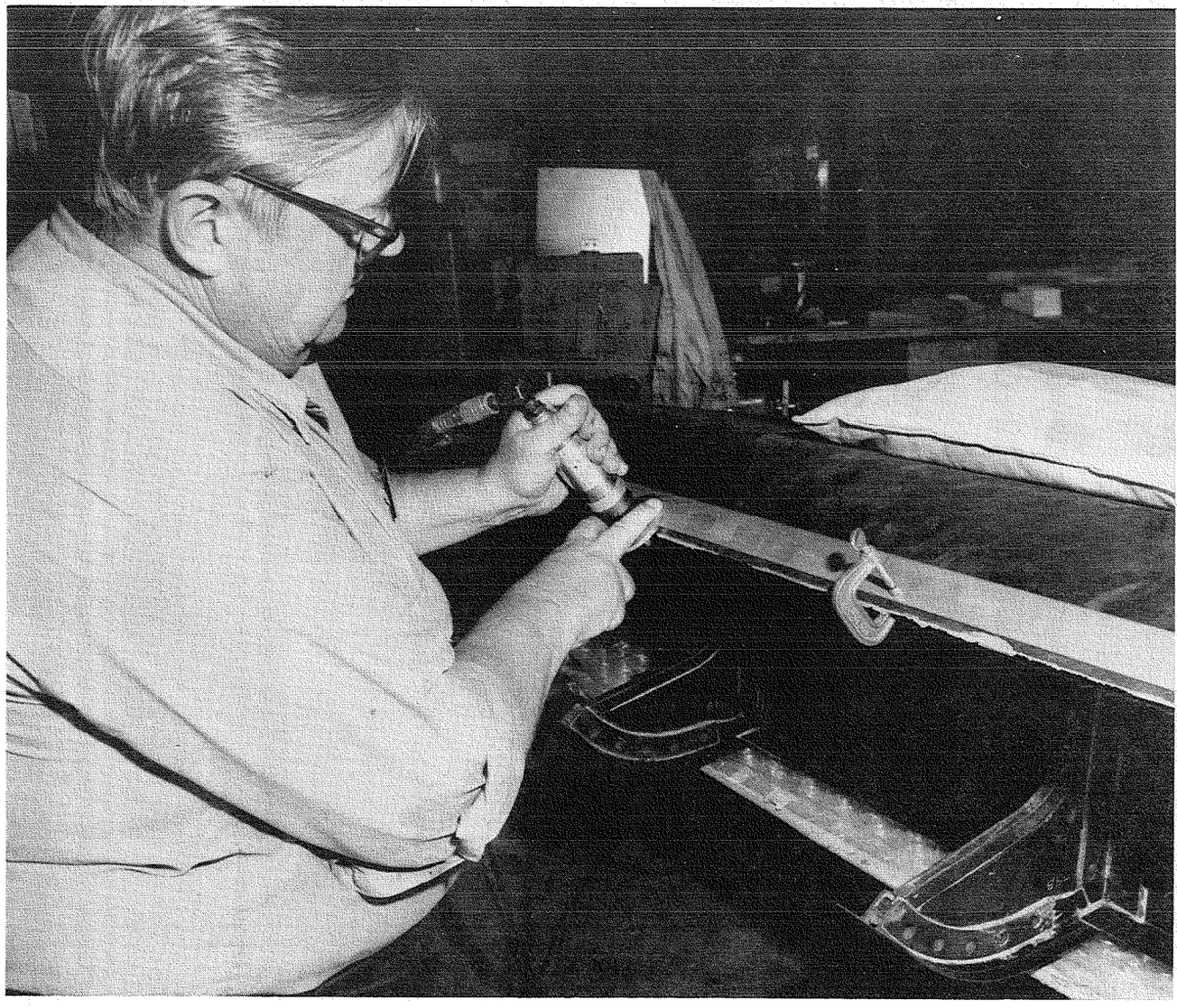


Figure 219. Final Trim of TDS Upper Leading Edge Cover

Table 1. LARC-160 Intermediate Ester Batches

Batch Code		
Standard Production Batches		
A	22367 544 Run 2	Standard Composition & Processing
B	22361 544 run 4	Standard Composition & Processing
C	22408 545 Run 1	Standard Composition & Processing
D	22746 545 Run 1	Standard Composition & Processing
E	22746 545 Run 2	Standard Composition & Processing
F	22746 545 Run 3	Standard Composition & Processing
Variables Study Batches		
1	22943	Standard Composition & Processing
2	22944	Standard Composition & Processing
3	22945	Standard Composition & Processing
4	22946	Standard Composition & Processing
5	22947	Standard Composition & Processing
6	22948	Standard Composition & Processing
1A	22990	Standard Composition & Processing
2A	22991	Standard Composition & Processing
7	22949	+5% NA, Standard Processing
8	22950	+5% BTDA, Standard Processing
9	22951	-5% NA, Standard Processing
10	22952	-5% BTDA, Standard Processing
11	22953	Standard Composition & Processing
12	22954	Standard Composition & Processing
13	22955	Standard Composition, 6 hr Reflux
14	23107	Standard Composition & Processing
15	23236	Standard Composition & Processing
16	23357	Standard Composition & Processing
Repeatability Batches		
G	23724	Standard Composition & Processing
H	23726	Standard Composition & Processing
I	23728	Standard Composition & Processing

Table 2. LARC-160 Neat Resin and Prepreg Batches

Batch Code	Neat Resin	Prepreg
A	544 22408	Not Available
B	544 22513	Not Available
C	544 22368 Cut 3	22368 Roll 1
D	544 22361 Cut 4	22361 Roll 1
E	544 22367 Cut 2	22367 Roll 1
F	Not Available	22322 Roll 1
G	544 22332 Cut 1	22332 Cut 1
H	Not Available	22999-2
I	Not Available	22999-3
J	Not Available	22999-4
K	Not Available	22999-11
L	Not Available	23091 Roll 4

Variables Study Batches

	Neat Resin	Prepreg	
1	22943	22943	+2% AP-22, Standard Processing
2	22944	22944	-2% AP-22, Standard Processing
3	22945	22945	+5% AP-22, Standard Processing
4	22946	22946	-5% AP-22, Standard Processing
5	22947	22947	+10% AP-22, Standard Processing
6	22948	22948	-10% AP-22, Standard Processing
1A	22990	22990	+2% AP-22, Standard Processing
2A	22991	22991	-2% AP-22, Standard Processing
7	22949	22949	+5% NA, Standard Processing
8	22950	22950	+5% BTDA, Standard Processing
9	22951	22951	-5% NA, Standard Processing
10	22952	22952	-5% BTDA, Standard Processing
11	22953	22953	Standard Composition, Extended Resin Cook
12	22954	22954	Standard Comp., Intermediate Resin Cook
13	22955	22955	Standard Composition, 6 hr Ester Reflux
14	22956	22956	Anchamine DL, Std. Comp. & Proc.
15	23236	23236	Tonox-22, Standard Composition & Proc.
16	23357	23427	Standard Composition & Processing

Repeatability Batches

M	23724	23723
N	23726	23725
O	23728	23727

Table 3. Experimental Conditions for HPLC Analysis of LARC-160 Resin

Column: Spectra-Physics Sperisorb ODS

Solvents: Baker HPLC water with 0.01 M KH_2PO_4 at pH=3 WITH HCL Burdick & Jackson Acetonitrile

Gradient: 10-50% acetonitrile/water, 30 min linear gradient with hold at 50% 10 min and equilibrate at initial for 10 min.

Detection: 200 nm, 0.4 aufs

Flow: 1 ml/min

Sample: 10 μl of 1.5 mg/ml solution in THF

Table 4. Experimental Conditions for Ion-Pair HPLC Analysis of LARC-160 Resin

Column: Whatman Partisil 10, ODS-2

Solvents: Baker HPLC Water with Waters PIC A Ion Pair Reagent Burdick & Jackson UV Grade Tetrahydrofuran (THF)

Gradient: 15-50% THF in water with PIC A, linear 15 min gradient with 15 min hold at 50% THF and equilibrate at initial 15 min

Detection: 254 nm, 0.4 aufs

Flow: 1 ml/min

Sample: 10 μl of 1.5 mg/ml solution in THF

Table 5. Test Matrix Effect of Resin Formulation/Process Variables

PREPREG BATCH 1.35 KG (3 LB) EA	FORMULATION VARIABLES		PROCESS VARIABLES		ANALYSES							
	CONC. AP-22	CONC. ANHYDRIDES	COOK TIME	REFLUX TIME	NA	BTDA	AP-22	INT. ESTER	RESIN	PREPREG EXTRACT	LAM	PANEL NO.
1	+2%	STD	STD	STD	✓	✓	✓	✓	✓	✓	✓	EX217
2	-2%	↕	↕	↕				✓	✓	✓	✓	EX218
3	+5%	↕	↕	↕				✓	✓	✓	✓	EX205
4	+5%	↕	↕	↕				✓	✓	✓	✓	EX206
5	+10%	↕	↕	↕				✓	✓	✓	✓	EX207
6	-10%	STD	STD	STD				✓	✓	✓	✓	EX208
7	STD	NA (+5%) BTDA (STD)	STD	STD				✓	✓	✓	✓	EX209
8	↕	NA (-5%) BTDA (STD)	↕	↕				✓	✓	✓	✓	EX210
9	↕	NA (STD) BTDA (+5%)	↕	↕				✓	✓	✓	✓	EX211
10	STD	NA (STD) BTDA (-5%)	STD	STD				✓	✓	✓	✓	EX212
11	STD	STD	2 HRS AT 70C	STD				✓	✓	✓	✓	EX213
12	STD	STD	2 HRS AT 60C	STD				✓	✓	✓	✓	EX214
13	STD	STD	STD	6 HR				✓	✓	✓	✓	EX215
14	ANCHAMINE DL STD	STD	STD	STD				✓	✓	✓	✓	EX216
15	TONOX 22 STD	STD	STD	STD				✓	✓	✓	✓	EX219
16	STD	STD	STD	STD	✓	✓	✓	✓	✓	✓		

Table 6. Prepreg and Composite Physical, Short Beam Shear and Flexural Properties, LARC-160 Resin Stoichiometry and Process Variable Program

Properties / Laminate No	Target[2] Property	EX217		EX218		EX205		EX206		EX207	
Resin/Process Variable											
1 Resin Run No	—	1		2		3		4		5	
2 Concentration AP22	—	+2%		-2%		+5%		-5%		+10%	
3 Concentration Anhydrides	—	STD		STD		STD		STD		STD	
4 Cook time	—	STD		STD		STD		STD		STD	
5 Reflux time	—	STD		STD		STD		STD		STD	
Processing Parameters[1]											
1 Type of bleeder/No Plies[6]	—	120/2T & 1B		120/2T & 1B		120/1T & B		120/1T & B		120/1T & B	
Prepreg Physical Properties											
1 Prepreg batch		22991		23107		22945		22946		22947	
2 Fiber areal weight (grams/m^2)	134±4	127		118		131		127		126	
3 Calc Thick /ply, 60% fiber vol, mm (mils)	0 131-0 124 (5 2-4 9)	0 122 (4 8)		0 112 (4 4)		0 127 (5 0)		0 122 (4 8)		0 122 (4 8)	
4 Resin solids content (%) as is	38 0 ± 3	41 2		43 0		42 1		40 4		42 8	
5 Resin solids content staged (%)[5]	32-36	32 2		31 7		28 4		30 5		34 4	
6 Volatile content as is (%)	9-14	15 6		13 8		14 3		15 2		14 5	
7 Volatile content staged (%)[5]	<2	1 13		1 11		3 04		2 65		1 97	
Composite Physical Properties[1]											
1 Specific gravity (grams/cc)	1 573-1 591	1 59		1 595		1 59		1 60		1 56	
2 Resin weight content (%)	31 05-34 71	34 0		29 1		30 8		28 2		31 8	
3 Fiber volume (%)	58-62	59 3		63 9		62 2		64 9		60 1	
4 Void volume (%)	<2	-0 18		0 97		0 74		0 92		2 3	
5 Thickness mm (mils)	—	1 42-1 60 (56-63)		1 45-1 65 (57-65)		1 16-1 37 (46-54)		1 45-1 55 (57-61)		1 63-1 75 (64-69)	
6 Thickness/ply, mm (mils)	—	0 102-0 114 (4 0-4 5)		0 104-0 117 (4 1-4 6)		0 084-0 097 (3 3-3 8)		0 104-0 112 (4 1-4 4)		0 117-0 124 (4 6-4 9	
7 Barcol hardness (ASTM 2583)	>70	72-75		72-74		72-76		74-76		64-68	
8 Weight loss in postcure (%)	<1	0 23		0 23		0 27		0 24		0 24	
9 Weight loss after 125 hrs at 316 C (600 F) (%)	<3	1 48		1 40		1 58		1 46		1 57	
10 TMA-Tg C (F) postcured 4 hrs at 316 C (600 F)	>340 (644)	356 (673)		340 (644)		347 (357)		337 (639)		367 (693)	
11 C-Scan ultra sound transmission (%)[3]											
Cured	>95	100		100		100		100		100	
Postcured 4 hrs at 316 C (600 F)	>95	100		100		100		100		100	
Composite Mechanical Properties[4]	MN/m^2 (Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
1 Flexural strength RT			(253)		(268)		(254)		(273)		(222)
			(278)		(254)		(267)		(250)		(228)
			(269)		(252)		(267)		(274)		(216)
		1840	(267)	1778	(258)	1812	(263)	1833	(266)	1530	(222)
Avg normalized strength, 60% F/V	>1571 (>228)	1861	(270)	1669	(242)	1750	(254)	1695	(246)	1530	(222)
316 C (600 F)			(161)		(132)		(160)		(155)		(131)
			(170)		(132)		(149)		(138)		(136)
			(159)		(134)		(169)		(132)		(142)
Avg		1033	(150)	916	(133)	1096	(159)	978	(142)	937	(136)
Avg normalized strength, 60% F/V	>937 (>136)	1046	(152)	860	(125)	1057	(153)	900	131	(937)	(136)
2 Flexural modulus RT	GN/m^2 (Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)
			(19 7)		(19 3)		(19 6)		(21 0)		(18 0)
			(17 3)		(19 3)		(19 9)		(18 4)		(25 2)
			(18 5)		(19 7)		(18 7)		(19 4)		(17 3)
Avg		127	(18 5)	134	(19 4)	134	(19 4)	135	(19 6)	140	(20 2)
Avg normalized modulus, 60% F/V	>124 (>18)	129	(18 7)	126	(18 2)	129	(18 7)	124	18 0	140	(20 2)
316 C (600 F)			(20 5)		(17 4)		(19 2)		(19 1)		(17 8)
			(16 5)		(16 9)		(18 1)		(18 1)		(17 4)
			(20 3)		(18 5)		(19 2)		(15 2)		(16 9)
Avg		132	(19 1)	916	(17 6)	130	(18 8)	121	(17 5)	119	(17 4)
Avg normalized modulus, 60% F/V	>124 (>18)	133	(19 3)	114	(16 5)	125	(18 1)	111	(16 1)	119	(17 4)
3 Short beam shear strength RT	MN/m^2 (Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
	>103 (>15)		(17 1)		(16 2)		(16 4)		(16 4)		(11 8)
			(17 9)		(16 2)		(17 1)		(18 4)		(12 5)
			(17 9)		(16 6)		(18 1)		(17 6)		(13 1)
Avg		121	(17 6)	112	(16 3)	118	(17 2)	120	(17 5)	86	(12 5)
316 C (600 F)	>48 (>7)		(8 9)		(6 5)		(8 4)		(6 1)		(8 8)
			(8 2)		(6 4)		(7 3)		(6 2)		(8 6)
			(6 9)		(5 9)		(7 4)		(5 8)		(8 5)
Avg		55	(8 0)	43	(6 3)	53	(7 7)	41	(6 0)	59	(8 6)

(1) The imidizing 2 stage cure cycle and tooling specified was employed in fabrication of 14 ply unidirectional laminates 17 78 x 13 59 cm (7 0 x 5 5 inches) Laminates were postcured at 316 C (600°F) for 4 hours, freestanding in an air circulating oven Prepreg and composite physical properties were calculated per Appendix A

(2) Target property values are based on Celion fiber minimum properties of 2618 MN/m^2, (380 Ksi) tensile strength and 234 GN/m^2, (34 Msi) tensile modulus using the rule of mixtures, 60% composite fiber volume Target 316 C (600 F) strength properties are based on a 60% retention of room values

(3) NDI ultrasonic through transmission tests were performed using the NASA-LaRC established "A" sensitivity standards

(4) Specimens were tested after stabilizing at 316 C (600 F) for 10 minutes

(5) Volatiles and resin solids content determined on portion of stacked laminate

(6) T = number of bleeder plies on top surface of laminate, B = number of bleeder plies on bottom surface

Table 6. Prepreg and Composite Physical, Short Beam Shear and Flexural Properties, LARC-160 Resin Stoichiometry and Process Variable Program (Cont)

Properties / Laminate No	Target[2] Property	EX208		EX209		EX210		EX211		EX212	
Resin/Process Variable											
1 Resin Run No		6		7		8		9		10	
2 Concentration AP22		-10%		STD		STD		STD		STD	
3 Concentration anhydrides		STD		NA(+5%), BTDA(STD)		NA(-5%), BTDA(STD)		NA(STD) BTDA(+5%)		NA(STR) BTDA(-5%)	
4 Cook time		STD		STD		STD		STD		STD	
5 Reflux time		STD		STD		STD		STD		STD	
Processing Parameters[1]											
1 Type of bleeder/no plies[6]	—	120/2T & 1B		181/1T & 1B		120/1B only		120/2T & 1B		120/2T & 1B	
Prepreg Physical Properties											
1 Prepreg batch	134±4	22948		22949		22950		22951		22952	
2 Fiber areal weight (grams/m²)	0 131-0 124	132		122		122		116		120	
3 Calc thick/ply 60% fiber vol, mm (mils)	(5 2-4 9)	0 124 (4 9)		0 117 (4 6)		0 117 (4 6)		0 112 (4 4)		0 112 (4 4)	
4 Resin solids content staged (%) as is	38 0 ±3	36 9		40 0		42 0		43 0		42 1	
5 Resin solids content staged (%)[5]	32-36	30 7		30 3		35 7		30 8		28 3	
6 Volatile content as is (%)	9-14	15 1		14 1		14 0		14 3		14 8	
7 Volatile content staged (%)	<2	2 92		2 09		2 79		1 58		1 34	
Composite Physical Properties[1]											
1 Specific gravity (grams/cc)	1 573-1 591	1 62		1 60		1 57		1 59		1 58	
2 Resin weight content (%)	31 05-34 71	25 0		28 8		33 3		30 7		31 1	
3 Fiber volume (%)	58-62	68 6		64 2		59 2		67 2		61 5	
4 Void volume (%)	<2	0 68		0 91		1 24		2 02		1 26	
5 Thickness mm (mils)		1 45-1 62 (57-64)		1 55-1 75 (61-69)		1 37-1 55 (54-61)		1 32-1 55 (52-61)		1 47-1 57 (58-62)	
6 Thickness/ply, mm (mils)		0 104-0 116 (4 1-4 6)		0 117-0 124 (4 4-4 9)		0 099-0 112 (3 9-4 4)		0 094-4 4 (3 7-4 35)		0 104-0 112 (4 1-4 4)	
7 Barcol hardness (ASTM 2583)	>70	1 48		1 48		1 5		1 31		1 17	
8 Weight loss in posture (%)	<1	0 24		0 22		0 25		0 23		0 23	
9 Weight loss after 125 hr at 316°C (600°F) (%)	<3	1 48		1 48		1 51		1 31		1 17	
10 TMA-Tg C, (F) postcured 4 hr at 316°C (600°F)	>340 (644)	339 (642)		364 (687)		340 (644)		340 (644)		353 (667)	
11 C-scan ultra sound transmission (%)[3]											
Cured	>95	100		100		99		100		100	
Postcured 4 hr at 316°C (600°F)	>95	100		100		100		100		100	
Composite Mechanical Properties[4]											
1 Flexural strength	MN/m² (Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)
RT			(305)		(232)		(333)		(296)		(248)
			(321)		(245)		(248)		(319)		(262)
			(274)		(233)		(270)		(283)		(260)
Avg		2067	(300)	1633	(237)	1950	(283)	2060	(299)	1770	(257)
Avg normalized strength, 60% F/V	>1571 (>228)	1808	(262)	1526	(221)	1976	(287)	1839	(267)	1728	(251)
316°C (600°F)			(144)		(144)		(167)		(180)		(148)
			(155)		(157)		(155)		(143		(134)
			(148)		(140)		(159)		(160)		(147)
Avg		1027	(149)	1013	(147)	1102	(160)	1109	(161)	985	(143)
Avg normalized strength 60% F/V	>937 (>136)	898	(130)	942	(137)	1113	(162)	987	(143)	956	(139)
2 Flexural modulus	GN/m² (Msi)	GN/m²	(Msi)	GN/m²	(Msi)	GN/m²	(Msi)	MN/m²	(Msi)	GN/m²	(Msi)
RT			(20 0)		(18 2)		(20 4)		(19 1)		(18 4)
			(18 5)		(17 9)		(20 0)		(20 3)		(22 5)
			(19 1)		(18 0)		(22 5)		(19 1)		(18 7)
Avg		132	(19 2)	124	(18 0)	145	(21 0)	134	(19 5)	137	(19 9)
Avg normalized modulus, 60% F/V	>124 (>18)	115	(16 7)	115	(16 7)	146	(21 2)	119	(17 3)	133	(19 3)
316°C (600°F)			(16 8)		(17 4)		(19 4)		(20 4)		(18 2)
			(17 6)		(16 7)		(17 9)		(19 4)		(15 7)
			(18 4)		(17 5)		(18 3)		(18 4)		(17 8)
Avg		121	(17 6)	118	(17 2)	127	(18 5)	134	(19 4)	119	(17 2)
Avg normalized modulus, 60% F/V	>124 (>18)	106	(15 3)	110	(16 0)	129	(18 7)	119	(17 3)	115	(16 7)
3 Short beam shear strength	MN/m² (Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)	MN/m²	(Ksi)
RT	>103 (>15)		(17 6)		(13 9)		(18 0)		(17 5)		(16 9)
			(17 8)		(16 3)		(17 2)		(16 3)		(16 6)
			(16 9)		(15 5)		(16 0)		(15 5)		(16 5)
Avg		120	(17 4)	105	(15 2)	118	(17 1)	113	(16 4)	115	(16 7)
316°C (600°F)	>48 (>7)		(8 1)		(8 6)		(7 0)		(8 4)		(8 5)
			(7 6)		(8 3)		(8 3)		(8 3)		(8 1)
			(7 9)		(7 9)		(8 5)		(8 5)		(8 4)
Avg		54	(7 9)	58	(8 3)	54	(7 9)	58	(8 4)	57	(8 3)

(1) The imidizing 2 stage cure cycle and tooling specified was employed in fabrication of 14 ply unidirectional laminates 17 78 x 13 59 Cm (7 7 x 5 5 inches) Laminates were postcured at 316 C (600 F) for 4 hours, freestanding in an air circulating oven Prepreg and composite physical properties were calculated per Appendix A

(2) Target property values are based on Celion fiber minimum properties of 2618 MN/m², (380 ksi) tensile strength and 234 GN/m², (34 Msi) tensile modulus using the rule of mixtures, 60% composite fiber volume Target 316°C (600°F) strength properties are based on a 60 percent retention of room values

(3) NDI ultrasonic through transmission tests were performed using the NASA-LaRC established A" sensitivity standards

(4) Specimens were tested after stabilizing at 316°C (600°F) for 10 minutes

(5) Volatiles and resin solids content determined on portion of stacked laminate

(6) T = number of bleeder plies on top surface of laminate B = number of bleeder plies on bottom surface

Table 6. Prepreg and Composite Physical, Short Beam Shear and Flexural Properties, LARC-160 Resin Stoichiometry and Process Variable Program (Cont)

Properties / Laminate No	Target[2] Property	EX213		EX214		EX215		EX216		EX219	
Resin/Process Variable											
1 Resin run no		11		12		13		14		15	
2 Concentration AP22		STD		STD		STD		STD Ancamine		STD Tonax	
3 Concentration anhydrides		STD		STD		STD		STD		STD	
4 Cook time		Extended		Intermediate		STD		STD		STD	
5 Reflux time		STD		STD		6 hr		STD		STD	
Processing Parameters[1]											
1 Type of bleeder/No Plies[6]	-	181/1T & 1B		120/2T & 1B		120/2T & 1B		120/2T & 1B		120/1T & 1B	
Prepreg Physical Properties											
1 Prepreg batch	13444	22953		22954		22955		22990		23236	
2 Fiber areal weight (grams/m^2)	0 131-0 124	115		120		121		126		133	
3 Calc Thick /ply, 60% fiber vol, mm (mils)	(5 2-4 9)	0 109 (4 3)		0 114 (4 5)		0 117 (4 6)		0 119 (4 7)		0 127 (5 0)	
4 Resin solids content (%) as is	38 0 ± 3	46 1		40 5		40 0		43 0		38 9	
5 Resin solids content staged (%)	32-36	37 9		33 9		30 3		31 9		-	
6 Volatile content as is (%)	9-14	12 7		12 9		14 8		12 0		11 7	
7 Volatile content staged (%)[5]	<2	1 62		1 36		1 64		1 39		-	
Composite Physical Properties[1]											
1 Specific gravity (grams/cc)	1 573-1 591	1 56		1 59		1 59		1 59		1 489	
2 Resin weight content (%)	31 05-34 71	36 0		30 5		31 6		29 5		32 7	
3 Fiber volume (%)	58-62	56 1		62 4		61 6		63 3		56 6	
4 Void volume (%)	<2	1 04		0 82		0 31		1 19		6 5	
5 Thickness mm (mils)		1 62-1 78 (64-70)		1 49-1 80 (59-71)		1 39-1 65 (55-65)		1 52-1 72 (60-68)		1 72-1 98 (68-78)	
6 Thickness/Ply, mm (mils)		0 117-0 127 (4 6-5 0)		0 106-0 129 (4 2-5 1)		0 099-0 147 (3 9-4 6)		0 109-0 124 (4 3-4 9)		0 124-0 142 (4 9-5 6)	
7 Barcol hardness (ASTM 2583)	>70	69-73		69-73		73-76		72-74		64-68	
8 Weight loss in postcure (%)	<1	0 27		0 21		0 26		0 25		0 23	
9 Weight loss after 125 hrs at 316 C (600 F) (%)	<3	1 73		1 45		1 34		1 37		2 12	
10 TMA-Tg C, (F) Postcured 4 hrs at 316 C (600 F)	>340 (644)	337 (639)		340 (644)		340 (644)		355 (671)		374 (705)	
11 C-Scan ultra sound tranmission (%)[3]											
Cured	>95	100		100		100		60		0	
Postcured 4 hrs at 316 C (600 F)	>95	100		100		100		70		0	
Composite Mechanical Properties[4]											
1 Flexural strength RT	MN/m^2 (Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
			(219)		(237)		(261)		(240)		(188)
			(243)		(225)		(239)		(229)		(180)
			(221)		(240)		(262)		(240)		(169)
	Avg	1557	(227)	1612	(234)	1743	(254)	1626	(236)	1233	(179)
Avg normalized strength, 60% F/V	>1571 (>228)	1665	(242)	1550	(225)	1698	(246)	1541	(224)	1307	(190)
316 C (600F)			(120)		(146)		(156)		(147)		(121)
			(119)		(155)		(144)		(138)		(110)
			(151)		(145)		(156)		(145)		(125)
	Avg	896	(130)	1027	(149)	1047	(152)	985	(143)	820	(119)
Avg normalized strength, 60% F/V	>937 (>136)	958	(139)	987	(143)	1020	(148)	934	(134)	869	(126)
2 Flexural modulus RT	GN/m^2 (Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)
			(17 7)		(20 0)		(20 0)		(18 2)		(15 6)
			(18 5)		(19 6)		(19 2)		(18 7)		(16 0)
			(18 3)		(20 3)		(18 2)		(18 7)		(15 1)
	Avg	125	(18 2)	138	(20 0)	1316	19 1	127	(18 5)	107	(15 6)
Avg normalized modulus, 60% F/V	>124 (>18)	134	(19 5)	132	(19 2)	128	(18 6)	121	(17 5)	114	(16 5)
316 C (600 F)			(15 7)		(18 3)		(19 5)		(17 5)		(16 7)
			(15 5)		(19 2)		(17 5)		(16 7)		(15 7)
			(17 7)		(17 8)		(18 2)		(17 5)		(17 2)
	Avg	112	(16 3)	127	(18 4)	127	(18 4)	119	(17 2)	114	(16 5)
Avg normalized modulus, 60% F/V	>124 (>18)	120	(17 4)	122	(17 7)	123	(17 9)	112	(16 3)	120	(17 4)
3 Short beam shear strength RT	MN/m^2 (Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
	>103 (>15)		(17 6)		(18 1)		(16 3)		(14 2)		(7 4)
			(16 8)		(16 9)		(17 4)		(12 8)		(10 3)
			(16 3)		(18 0)		(17 4)		(13 8)		(7 7)
	Avg	116	(16 9)	122	(17 7)		(17 0)	94	(13 6)	58	(8 5)
316 C (600 F)	>48 (>7)		(5 9)		(9 1)		(11 1)		(7 6)		(5 1)
			(5 9)		(9 4)		(8 6)		(6 7)		(4 5)
			(9 3)		(10 6)		(8 2)		(6 5)		(4 9)
	Avg	48	(7 0)	67	(9 7)	64	(9 3)	47	(6 9)	33	(4 8)

(1) The imidizing 2 stage cure cycle and tooling specified was employed in fabrication of 14 ply unidirectional laminates 17 78 x 13 59 cm (7 0 x 5 5 inches) Laminates were postcured at 316°C (600°F) for 4 hours, freestanding in an air circulating oven Prepreg and composite physical properties were calculated per Appendix A

(2) Target property values are based on Celion fiber minimum properties of 2618 MN/m^2, (380 Ksi) tensile strength and 234 GN/m^2, (34 Msi) tensile modulus using the rule of mixtures 60% composite fiber volume Target 316 C (600 F) strength properties are based on a 60 percent retention of room values

(3) NDI ultrasonic through transmission tests were performed using the NASA-LaRC established "A" sensitivity standards

(4) Specimens were tested after stabilizing at 316 C (600 F) for 10 minutes

(5) Volatiles and resin solids content determined on portion of stacked laminate

(6) T = number of bleeder plies on top surface of laminate, B = number of bleeder plies on bottom surface

Table 7. Minimum Molding Pressure and Temperature Investigations

Properties / Panel No	Target[5] Property		EX 41		EX 47		EX 48		EX 49		EX 74	
Processing Variable[1]												
1 Maximum cure temperature, C (F)	—		329 (625)		329 (625)		329 (625)		329 (625)		329 (625)	
2 Maximum molding pressure, MN/m^2 (psi)	—		1378 (200)		1034 (150)		689 (100)		345 (50)		1378 (200)	
Composite Physical Properties[2]												
1 Specific gravity (grams/cc)	1 561-1 579		1 618		1 60		1 54		1 49		1 613	
2 Resin weight content (%)	35 0-31 3		24 4		26 0		32 7		34 0		25 8	
3 Fiber volume (%)	58-62		69 9		67 7		60 2		60 6		68 4	
4 Void Volume (%)	<2		-0 27		0 33		0 51		6 37		-0 40	
5 Thickness mm (mils)	2 1-1 9 (83 2-76 8)		2 0-1 8 (80-70)		2 0-1 8 (80-70)		2 1-1 9 (83 2-76 8)		2 0-1 87 (76 4-73 6)		2 1-1 9 (83 2-73 6)	
6 Thickness/ply, mm (mils)	0660- 0609 (2 6-2 4)		0635- 0559 (2 5-2 2)		0635- 0559 (2 5-2 2)		0660- 0609 (2 6-2 4)		0635- 0584 (2 4-2 3		0660- 0609 (2 6-2 4)	
7 Barcol hardness (ASTM D2583)	>70		76-80		73-78		74-76		73-78		75-78	
8 Weight loss in postcure (%)	<1		0 30		0 14		0 062		0 10		0 32	
9 TMA-Tg C, (F) cured	>330 (626)		320 (608)		345 (653)		339 (642)		363 (685)		346 (655)	
Postcured 4 hrs at 316 C (600 F)	>340		331 (628)		342 (648)		346 (655)		361 (682)		356 (673)	
10 C Scan ultra sound transmission (%)[3]												
Cured	>95		100		100		99 5		0		100	
Postcured 4 hrs at 316 C (600 F)	>95		100		100		99 5		0		100	
Composite Mechanical Properties[4]												
1 Flexural strength	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
RT				(214)		(237)		(211)		(195)		(231)
				(224)		(240)		(221)		(207		(224)
				(220)		(232)		(234)		(206)		(232)
			Avg 1509	(219)	1626	(236)	1530	(222)	1399	(203)	1578	(229)
Avg normalized strength, 60% F/V	>1570	(>228)	1295	(188)	1440	(209)	1523	(221)	1385	(201)	1385	(201)
316 C (600 F)				(146)		(143)		(136)		(102)		(154)
				(145)		(158)		(124)		(99)		(147)
				(136)		(148)		(140)		(109)		(151)
			Avg 978	(142)	1027	(149)	916	(133)	710	(103)	1040	(151)
Avg normalized strength, 60% F/V	>942	(>136)	841	(122)	910	(132)	916	(133)	703	(102)	909	(132)
2 Flexural modulus	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)
RT				(19 3)		(18 8)		(17 1)		(19 1)		(18 7)
				(19 2)		(18 4)		(17 6)		(19 2)		(20 1)
				(19 2)		(18 5)		(18 5)		(20 2)		(20 0)
			Avg 132	(19 2)	128	(18 6)	122	(17 7)	134	(19 5)	132	(19 6)
Avg normalized modulus, 60% F/V	>124	(>18)	113	(16 4)	114	(16 5)	121	(17 6)	133	(19 3)	119	(17 2)
316 C (600 F)				(18 8)		(18 7)		(16 9)		(17 1)		(19 0)
				(18 4)		(19 8)		(16 2)		(18 1)		(18 6)
				(18 5)		(19 9)		(18 4)		(17 4)		(19 2)
			Avg 128	(18 6)	134	(19 5)	119	(17 2)	121	(17 5)	130	(18 9)
Avg normalized modulus, 60% F/V	>124	(>18)	110	(16 0)	119	(17 2)	118	(17 1)	119	(17 3)	114	(16 6)
3 Short beam shear strength	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
RT	>103	(>15)		(15 9)		(14 4)		(15 3)		(12 4)		(17 3)
				(17 7)		(13 9)		(14 9)		(10 7)		(16 4)
				(16 2)		(15 3)		(15 2)		(10 7)		(16 6)
			Avg 114	(16 6)	100	(14 5)	105	(15 2)	779	(11 3)	116	(16 8)
316 C (600 F)	>48	(7)		(6 93)		(7 17)		(6 98)		(6 24)		(8 17)
				(7 12)		(6 89)		(6 88)		(5 92)		(8 18)
				(7 31)		(6 75)		(6 78)		(6 79)		(8 15)
			Avg 49 1	(7 12)	47 8	(6 94)	47 4	(6 88)	43 5	(6 32)	56 2	(8 16)

[1] Laminates, 32 ply, unidirectional were autoclave molded using the two stage cycle

[2] Prepreg and composite physical properties were calculated per Appendix A

[3] NDI ultrasonic through transmission tests were performed using the NASA-LaRC established 'A sensitivity standards

[4] Specimens were tested after stabilizing at 316 C (600 F) for 10 minutes

[5] Target property values are based on Celion fiber minimum properties of 2618 MN/m^2 (34 MSI) tensile modulus using the rule of mixtures, 60% composite fiber volume Target 316 C (600 F) strength properties are based on a 60 percent retention of room temperature values

Table 7. Minimum Molding Pressure and Temperature Investigations (Cont)

Properties / Panel No	EX 69		EX 70		EX 71		EX 72	
Processing Variable[1]								
1 Maximum cure temperature, C (F)	316 (600)		302 (575)		288 (550)		274 (525)	
2 Maximum molding pressure, N/m^2 (psi)	1378 (200)		1378 (200)		1378 (200)		1378 (200)	
Composite Physical Properties[2]								
1 Specific gravity (grams/cc)	1 612		1 594		1 591		1 582	
2 Resin weight content (%)	26 2		28 5		29 4		31 3	
3 Fiber volume (%)	67 9		65 1		64 2		62 1	
4 Void Volume (%)	-0 34		-0 07		-0 16		-0 20	
5 Thickness mm (mils)	2 1-1 87 (76 4-73 6)		2 3-2 2 (89 6-86 4)		2 1-2 0 (83 2-80)		2 2-2 1 (86 4-83 2)	
6 Thickness/ply, mm (mils)	0609- 0584 (2 4-2 3)		0711- 0685 (2 8-2 7)		0660 - 0635 (2 6-2 5)		0685 - 0660 (2 7-2 6)	
7 Barcol hardness (ASTM D2583)	76-82		75-80		76-81		75-77	
8 Weight loss in postcure (%)	0 36		0 43		0 39		0 39	
9 TMA-Tg C, (F) cured	335 (635)		317 (603)		305 (581)		257 (495)	
Postcured 4 hrs at 316 C (600 F)	358 (676)		353 (667)		355 (671)		360 (680)	
10 C Scan ultra sound transmission (%)[3]								
Cured	100		100		100		99 5	
Postcured 4 hrs at 316 C (600 F)	100		100		100		99 5	
Composite Mechanical Properties[4]								
1 Flexural strength	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
RT		(230)		(216)		(227)		(197)
		(254)		(218)		(216)		(217)
		(256)		(226)		(224)		(214)
	1702	(247)	1516	(220)	1530	(222)	1440	(209)
Avg normalized strength, 60% F/V	1502	(218)	1399	(203)	1426	(207)	1392	(202)
316 C (600 F)		(173)		(136)		(154)		(156)
		(167)		(138)		(156)		(145)
		(164)		(141)		(151)		(157)
	1158	(168)	951	(138)	1054	(153)	1054	(153)
Avg normalized strength, 60% F/V	1020	(148)	875	(127)	985	(143)	1020	(148)
2 Flexural modulus	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)	GN/m^2	(Msi)
RT		(18 7)		(17 2)		(19 0)		(18 2)
		(20 2)		(17 7)		(18 1)		(18 5)
		(21 1)		(17 8)		(17 7)		(18 5)
	138	(20 0)	121	(17 6)	126	(18 3)	127	(18 4)
Avg normalized modulus, 60% F/V	122	(17 7)	112	(16 2)	118	(17 1)	123	(17 8)
316 C (600 F)		(19 2)		(17 0)		(18 2)		(17 7)
		(20 3)		(17 1)		(18 8)		(18 4)
		(21 1)		(17 0)		(18 4)		(18 6)
	139	(20 2)	117	(17 0)	127	(18 5)	125	(18 2)
Avg normalized modulus, 60% F/V	123	(17 8)	108	(15 7)	119	(17 3)	121	(17 6)
3 Short beam shear strength	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)	MN/m^2	(Ksi)
RT		(16 6)		(16 9)		(16 2)		(15 5)
		(16 7)		(15 9)		(16 9)		(14 4)
		(15 4)		(16 0		(17 5)		(14 4)
	112	(16 2)	112	(16 3)	116	(16 9)	99 9	(14 5)
316 C (600 F)		(8 52)		(8 77)		(7 73)		(8 04
		(8 36)		(8 23)		(7 84)		(7 76)
		(8 24)		(8 17)		(6 75)		(7 60)
	57 2	(8 37)	57 8	(8 39)	51 3	(7 44)	53 7	(7 80)

[1] Laminates, 32 ply, unidirectional were autoclave molded using the two stage cycle

[2] Prepreg and composite physical properties were calculated per Appendix A

[3] NDI ultrasonic through transmission tests were performed using the NASA-LaRC established A sensitivity standards

[4] Specimens were tested after stabilizing at 316 C (600 F) for 10 minutes

[5] Target property values are based on Celion fiber minimum properties of 2618 MN/m^2, (34 MSI) tensile modulus, using the rule of mixtures 60% composite fiber volume Target 316 C (600 F) strength properties are based on a 60 percent retention of room temperature values

Table 8. Test Matrix - LARC 160 Imidizing and Cure Cycle Process Improvement Study

Imidizing Temperature C (F)	Imidizing Time (Mins)				
163 (325 F)	60	90	120	150	180
177 (350 F)	60	90	120	150	180
191 (375 F)	30	60	90	120	150
199 (390 F)	30	60	90	120	150
218 (425 F)	30	60	90	120	150

Total: 25-6 X 6 - 26 ply 0° onidirectional laminates.

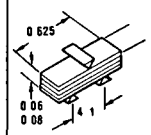
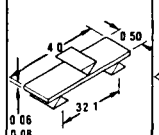
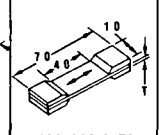
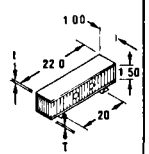
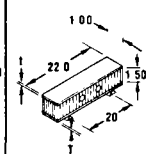
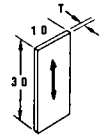
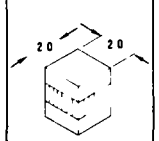
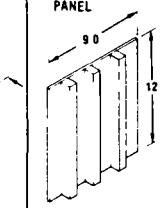
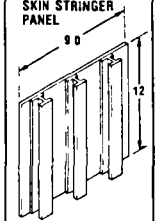
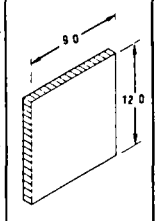
Table 9. Imidizing and Cure Cycle Process Improvement Study Observations -- Celion 3000/LARC 160 Composites[1]

Imidizing Cycle[2] Observations				Cure Cycle Observations									
Imidize Temp C (F)	Imidize Time (min)	Vol (%)	Resin Solids (%)	Resin Flow Characteristics in Cure	ε g/cc	Resin Cont (%)	Fiber Vol (%)	Void Vol (%)	Panel Thickness mm	Panel Thickness (mils)	Barcol Hardness (Cured)	C-Scan Ultrasound Transmission (%)	Remarks
163 (325)	60	1.64	34 7	high	1.530	26.3	64.4	4.61	1.34-1 63	53-64	72-75	40	Corrugated depressions top and bottom laminate surfaces parallel to fibers Fiber wash and resin flash excessive on panel sides
	90	1 52	33 0	high	1.531	25.8	64.91	4.20	1.27-1.91	20-25	72-75	40	
	120	1.48	35.0	high	1.545	26.4	64.98	3 64	1.30-1.55	51-61	72-76	40	
	150	1 68	34.0	high	1.550	26.8	64.8	3.20	1.14-1.45	45-57	72-75	60	Surface depressions have decreased, fiber wash and resin flash somewhat decreased
	180	1.43	32.6	high	1.563	27.4	64.9	2.21	1.14-1.52	45-60	68-73	50	
177 (350)	60	1.56	35 4	high	1.536	25 7	62 4	4.59	1 22-1.65	48-65	72-74	50	Corrugated depressions top and bottom surfaces parallel to fibers. Fiber wash and resin flash excessive on panel sides
	90	1.15	35.2	high	1.521	28.2	65.2	4.42	1.22-1.52	48-60	72-75	50	
	120	1.45	34 4	high	1.540	27 1	64.2	3.74	1.35-1.50	53-59	73-76	75	Laminate surfaces smooth, same fiber wash and resin flashing on sides
	150	1.06	34.6	high	1.565	28 0	64 4	1 91	1.22-1.55	48-61	74-78	95	
	180	1.14	34.2	high	1.561	28 5	63 8	2.01	1.27-1 65	50-65	73-77	95	
191 (375)	30	0 73	34 9	low	1.576	30 0	63.0	0.58	1.50-1.57	59-62	72-77	95	Very minor top and bottom surface depressions and edge fiber washing
	60	1.22	34.6	med	1.575	31.5	61.6	0.19	1.52-1.63	60-64	74-78	95 (2 splices)	Top and bottom surfaces very smooth and uniform, minor fiber washing
	90	1.24	35 0	med	1.570	33.3	59 8	-0.04	1.52-1.60	60-63	74-77	95 (1 splice)	
	120	1.17	33.8	med	1 574	33.6	59.7	-0.04	1 50-1.60	59-63	75-79	95 (1 splice)	
	150	0.92	34.9	med	1.565	35.0	58.1	-0.25	1.57-1.65	62-65	72-77	100	
199 (390)	30	1.21	35.0	med	1.577	32.3	61.0	-0.19	1.50-1.60	60-63	75-77	100	Top and bottom surfaces very smooth and uniform, minor fiber washing
	60	0.87	35.1	med	1.563	34.9	58.1	-0.10	1.60-1.65	63-65	74-76	100	
	90	1.10	33 3	low	1.579	33.7	59.8	-0.76	1.57-1.63	62-64	73-75	100	
	120	0.71	34.2	low	1 560	34.6	58 3	0.18	1.63-1.65	64-65	73-76	100	
	150	0.66	36 7	low	1.562	34.8	58.2	-0.015	1.60-1.68	63-66	70-76	100	
218 (425)	30	1.29	34.7	low	1.568	33.1	59.9	0.13	1.55-1.63	61-64	74-77	100	Top and bottom surfaces very smooth and uniform, minor fiber washing
	60	1.35	33 2	low	1.560	33.8	59 0	0.43	1.55-1.63	61-64	72-76	100	
	90	1.37	35.2	low	1.565	34.1	58.9	-0.011	1.57-1.60	62-63	74-76	100	
	120	1.15	34.0	low	1.550	35.1	56.8	-0.042	1.60-1.68	63-66	73-76	100	Top and bottom surfaces smooth but appear resin rich due to lack of flow, minor fiber wash and resin flash
	150	1.16	35.2	low	1.553	35 5	57.2	0.35	1.60-1.65	63-65	72-77	95	

[1] Celion 3000 fiber (epoxy resin sized) employed in making 30.4 cm (12.0 inches) wide, nominal 67 ±3 grams/m^2 areal fiber weight prepreg Prepreg—LARC 160 resin solids 38 ±3%, volatiles 12 ±3%.

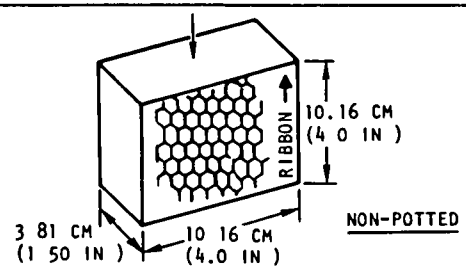
[2] One ply 120 fiberglass top and 1 ply bottom surfaces used in imidizing and cure cycles to absorb excess resin to target 60 ±2 composite fiber colume.

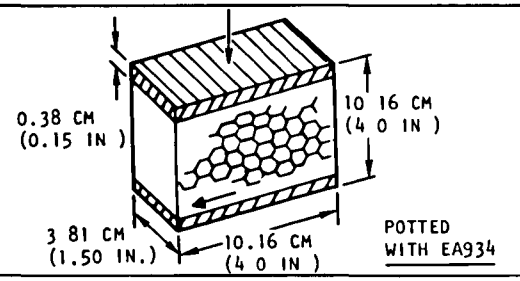
Table 10. Test Matrix Mechanical Properties and Structural Elements

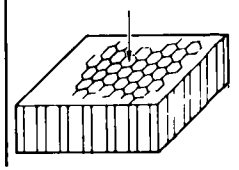
FIBER ORIENTATION	TEST TEMPERATURE °C (F)	INTERLAMINAR SHEAR  ①	FLEXURE  ①	LONGITUDINAL TENSION  FIBERGLASS/PI TABS ①	BEAM FLEXURE TENSION  ①	BEAM FLEXURE COMPRESSION  ①	COMPRESSION  ①	HONEYCOMB FLATWISE TENSION  ①	HAT STIFFENED SKIN STRINGER PANEL  ②③④	I BEAM STIFFENED SKIN STRINGER PANEL  ②③④	HONEYCOMB PANEL  ②③④
		F_{isu}	F_{fu}, E_f	$F_{fu}\ E_f\ \epsilon_{fu}\ \nu$	$F_{fu}\ E_f\ \epsilon_{fu}$	$F_{cu}\ E_c\ \epsilon_{cu}$	$F_{cu}\ E_c\ \epsilon_{cu}$	F_{fu}	$F_c\ \epsilon_{cu}$	$F_c\ \epsilon_{cu}$	$F_c\ \epsilon_{cu}$
0°	-168 (-270)	6	6	6	6	6	–	–	2	2	2
	24 (75)	6	6	6	6	6	–	–	2	2	2
	202 (400)	6	6	6	6	6	–	–	–	–	–
	316 (600)	6	6	6	6	6	–	–	2	2	2
90°	-168 (-270)	–	–	6	–	–	6	–			
	24 (75)	–	–	6	–	–	6	–			
	202 (400)	–	–	6	–	–	6	–			
	316 (600)	–	–	6	–	–	6	–			
±45°	-168 (-270)	–	–	6	–	–	6	–			
	24 (75)	–	–	6	–	–	6	–			
	202 (400)	–	–	6	–	–	6	–			
	316 (600)	–	–	6	–	–	6	–			
0 ±45 90	168 (-270)	–	–	6	–	6	–	6			
	24 (75)	–	–	6	–	6	–	6			
	202 (400)	–	–	6	–	6	–	6			
	316 (600)	–	–	6	–	6	–	6			

① 3 SPECIMENS POST CURED, 3 SPECIMENS AGED AT 316 C (600F) FOR 125 HOURS
② 1 SPECIMEN POST CURED 1 SPECIMEN AGED AT 316 C (600F) FOR 125 HOURS
③ FIBER ORIENTATION IN LAMINATE AS REQUIRED BY SPECIFIC DESIGN (REF 2ND & 3RD QUARTERLY REPORTS)
④ DESIGN LOAD > 3000 LB/IN AT ROOM TEMPERATURE

Table 11. RT Bulk Compressive Properties of 352 Kg/m^2 (22 PCF) 5052 Alloy 3.18 mm (0.125 in.) Cell Aluminum Honeycomb Core







Honeycomb Core Density	Compressive Property	Test Load Direction to Core Ribbon: Specimen ID	Parallel	Specimen ID	Perpendicular	Test Load Direction to Core Ribbon: Specimen ID	Parallel	Specimen ID	Perpendicular	Flatwise
352 Kg/m^3 (22 PCF) AL H/C	E_{cu} MN/m^2 (Ksi)	1	(13.6)	2	(7.56)	7	(16.73)	10	(6 10)	(1310)
		3	(10.8)	3	(7.20)	8	(17.64)	11	(6.03)	1 (1520)
		4	(11.20)	4	(6.59)	9	(19.89)	12	(7.67)	(1540)
		5	(11.88)	5	(8.95)		124.64 (18.09)		45.47 (6.6)	(1590)
		6	(9.82)	6	(6.20)					2 (1660)
	(AVG)		78.96 (11.46)		50.30 (7.3)					(1640)
										(1200)
										3 (1290)
										(1330)
										10,011 (1453)
	μ_c	1	-	2	-	7	1.26	10	0.72	
		3	1,15	3	0,82	8	1.15	11	0,738	
		4	1,27	4	0,69	9	1.21	12	0,751	
		5	1,19	5	0,85		(1.21)		(0.736)	
		6	1,18	6	0,80					
	(AVG)		(1.20)		(0.79)					
	F_{cu}									
96 Kg/m^3 (6 PCF) AL H/C	E_{cu} MN/m^2 (Psi)		(254.0)		(154.9)					
			(258.1)		(146.7)					
	(AVG)		1.76 (256.1)		1.04 (150.8)					
	μ_c		1.08		0.71					
			1 12		0.72					
	(AVG)		1.10		0.72					

Table 12. Tensile Properties of LARC-160/Celion Unidirectional (0)5 Oriented Composite, Postcured Condition—Beam Test

Specimen Number (4)	Test Temp. C (F) (3)	F_{tu} Test MN/m^2	F_{tu} Test (Ksi)	F_{tu} Adjusted (2) MN/m^2	F_{tu} Adjusted (2) (Ksi)	E_t Test GN/m^2	E_t Test (Msi)	E_t Adjusted (2) GN/m^2	E_t Adjusted (2) (Msi)	ε ult μ (%)	Failure Mode
EX107T-4	-168		(347)		(321)		(28.28)		(26.50)	1.22 *	Tension, 2 inches both sides of $\bar{C}_L$
-5	(-270)		(319)		(296)		(28.00)		(26.24)	1.16 *	Tension on $\bar{C}_L$
-6			(304)		(282)		(29.29)		(27.44)	1.04 *	Tension on $\bar{C}_L$
	Avg	2228	(323)	2065	(300)	197	(28.52)	184	(26.72)	1.14	
EX107T-1	RT		(300)		(278)		(27.30)		(25.58)	1.12	Tension on $\bar{C}_L$
-2			(329)		(304)		(27.80)		(26.04)	1.22	Tension, 1.5 inch off $\bar{C}_L$
-3			(284)		(263)		(25.90)		(24.27)	1.11	Tension on $\bar{C}_L$
	Avg	2097	(304)	1941	(282)	186	(27.00)	174	(25.30)	1.15	
EX107T-7	204		(296)		(274)		(25.20)		(23.61)	1.15	Tension in center area
-8	(400)		(271)		(251)		(27.70)		(25.95)	1.02	Tension in center area
-9			(262)		(243)		(23.8)		(22.31)	1.07	Tension in center area
	Avg	1076	(276)	1763	(256)	170	(24.7)	160	(23.17)	1.28	
EX107T-10	(316)		>(219)		>(206)		(26.96)		(25.45)	>0.816	Steel face to core failure
-11	(600)		(255)		(239)		(26.95)		(25.44)	0.970	Tension on $\bar{C}_L$ and steel face to core
-12			>(174)		>(163)		(22.57)		(21.31)	>0.800	Steel face to core failure
	Avg	1757	(255)	1647	(239)	176	(25.49)	166	(24.07)	0.970	

(1) Tension critical beams per the design described. Aluminum honeycomb 5052 alloy core 1/8 cell, 352 g/m^3 (22 pcf) density was employed in -168 C, RT and 204 C (-270 F, RT and 400 F) tests. CRES core, 301 alloy, 1/8 cell, 0.127 mm (0.005 inch) foil, 40 pcf density was employed in 316 C (600 F) tests.

(2) Adjusted properties were calculated from equations derived by Mr. Mark Shuart, NASA/LaRC using a computer program that considers the effect of bulk core properties on the strength and elastic modulus properties of the laminate. Adjustment factors for -168 C, RT and 204 C(-270 F, RT and 400 F) test temperature: $F_{tu} = \frac{F_{tu\ test}}{1.0792}$; $E_t = 0.937 \times E_t$ test value. For 316 C (600 F) tests: $F_{tu} = \frac{F_{tu\ test}}{1.0651}$; $E_t = 0.944 \times E_t$ test value.

(3) Tests were performed at a load rate of 0.127 Cm/minute (0.05 inch/minute) after stabilizing at the test temperature for 10 minutes.

* Projected from point of strain gage failure.

(4) Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)
1.618	25.6	68.8	-0.65	0.264	(10.4)	0.305-0.330	(12-13)	100

(5) The insitu imidizing-cure cycle specified was employed in laminate fabrication.

Table 13. Tensile Properties of LARC-160/Celion Unidirectional $(0)_5$ Oriented Composite, Aged 125 Hours at 316 C (600 F)—Beam Test

Specimen[4] Number	Test[3] Temp. C (F)	F_{t_u} Test MN/m^2	F_{t_u} Test (Ksi)	F_{t_u} Adjusted[2] MN/m^2	F_{t_u} Adjusted[2] (Ksi)	E_t Test GN/m^2	E_t Test (Msi)	E_t Adjusted[2] GN/m^2	E_t Adjusted[2] (Msi)	ε ult μ (%)	Failure Mode
EX199T-1	-168		(281)		(260)		(26.3)		(24.6)	12.5	Tensile failure on ℄
-2	(-270)		(245		(227)		(28.9		(27 0)	9 6	Tensile failure on C
-3			(286)		(265)		(24 7)		(23 1)	12 3	Tensile failure on ℄
	Avg	1867	(271)	1727	(251)	184	(26 6)	172	(24 4)	11.5	
EX199T-4	RT		(317)		(294)		(25.5)		(23 9)	13.3	Tensile failure 1 0 inch off ℄
-5			(340)		(315)		(25.3)		(23.7)	13 3	Tensile failure on ℄
-6			(331)		(307)		(24.0)		(22 5)	14.1	Tensile failure on ℄
	Avg	2269	(329)	2104	(305)	172	(24.9)	161	(23 4)	13.6	
EX199T-10	204		(259)		(243)	—*	—	—	—	—	Tensile failure on ℄
-11	(400)		(249)		(231)		(27.6)		(25 8)	9.4	Tensile failure on ℄
-12			(>257)		(>238)		(29 7)		(27 8)	>8 8	Bond failure specimen to core
	Avg	1750	(254)	1633	(237)	197	(28.7)	185	(26 8)	9.4	
EX199T-7	(316)		(>128)		(>120)		(26.7)		(25.2)	>4 5	Bond failure steel face/core
-8	(600)		(>144)		(>135)		(26.3)		(24.8	>5.5	Bond failure steel face/core
-9			(>166)		(>156)		(25.9)		(24 5)	>6.4	Bond failure steel face/core
	Avg		—	—	—	181	(26.3)	171	(24.8)	—	

(1) Tension critical beams per the design described. Aluminum honeycomb 5052 alloy core, 1/8 cell, 352 g/m^3 (22 pcf) density was employed in -168 C, RT and 204 C (-270 F, RT and 400 F) tests. CRES core, 301 alloy, 1/8 cell, 0 127 mm (0 005 inch) foil, 40 pcf density was employed in 316 C (600 F) tests.

(2) Adjusted properties were calculated from equations derived by Mr. Mark Shuart, NASA /LaRC using a computer program that considers the effect of bulk core properties on the strength and elastic modulus properties of the laminate Adjustment factors for -168 C, RT and 204 C (-270 F, RT and 400 F) test temperature

$$F_{tu} = \frac{F_{tu\ test}}{1\ 0792},\ E_t = 0\ 937 \times E_t \text{ test value} \quad \text{For 316 C (600 F) tests}$$

$$F_{tu} = \frac{F_{tu\ test}}{1\ 0651},\ E_t = 0\ 944 \times F_t \text{ test value}$$

(3) Tests were performed at a load rate of 0 127 Cm/minute (0 05 inch/minute) after stabilizing at the test temperature for 10 minutes

(4) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	Wt Loss After 125 Hours at 316 C (600 F) (%)
1 601	29 9	63 4	-0 22	0 284	(11 2)	0 279-0 318	(11-12 5)	99	3.4

(5) The 2 stage cure cycle specified was employed in laminate fabrication
*Strain gage failure

Table 14. Compressive Properties of LARC-160/Celion Unidirectional $(0)_5$ Oriented Composite Postcured Condition—Beam Test

Specimen[4] Number	Test[3] Temp C (F)	F_{cu} Test MN/m^2	F_{cu} Test (Ksi)	F_{cu} Adjusted[2] MN/m^2	F_{cu} Adjusted[2] (Ksi)	E_c Test GN/m^2	E_c Test (Msi)	E_c Adjusted[2] GN/m^2	E_c Adjusted[2] (Msi)	ε ult μ (%)	Failure Mode
EX107C-4	-168		(295)		(273)		(24.76)		(23.20)	1.42	Compression on ℄
-5	(-270)		(274)		(254)		(24.67)		(23.12)	1.34	Compression 1.0 inch off ℄
-6			(283)		(262)		(24.31)		(22.78)	1.51	Compression 2.0 inch both sides ℄
	Avg	1957	(284)	1812	(263)	164	(24.58	159	(23.03)	1.42	
EX107C-1			(223)		(207)		(22.34)		(20.93)	1.11	Compression overloading hole
-2	RT		(208)		(193)		(22.97)		(21.52)	0.976	Compression on ℄
-3			(214)		(198)		(22.20)		(20.80)	1.05	Compression on ℄
	Avg	1481	(215)	1373	(199)	155	(22.50)	145	(20.98)	1.05	
EX10C-7	204		>(102)		>(95.7)		(22.6)		(21.18)	>0.472	Composite to core bond failure
-8	(400)		(183)		(170)		(21.8)		(20.42)	0.960	Compression on ℄
-9		—	—	—	—	—	—	—	—	—	No test damaged specimen
	Avg	1261	(183)	1171	(170)	150	(21.8)	141	(204)	0.960	
EX107C-10	316		(121)		(114)		(20.88)		(19.71)	0.610	Compression on ℄
-11	(600)		(142)		(133)		(22.24)		(21.00)	0.660	Compression 1.5 inch off ℄
-12			(145)		(136)		(22.20)		(20.95)	0.660	Compression 1.75 inch off ℄
	Avg	937	(136)	(880)	(128)	150	(21.77)	141	20.55	0.643	

(1) Compression critical beams per the design described. Aluminum honeycomb 5052 alloy core 1/8 cell, 352 g/m^3 (22 psf) density was employed in -168 C, RT & 204 C (-270 F, RT & 400 F) tests, CRES Core, 301 Alloy, 1/8 cell, 0.127 mm (0.005 inch) foil 40 pcf was employed in 316 C (600 F) tests.

(2) Adjusted properties were calculated from equations derived by Mr. Mark Shuart, NASA/LaRC using a computer program that considers the effect of bulk core properties on the strength & elastic modulus properties of the laminate. Adjustment factors for -168 C, RT & 204 C (-270 F, RT & 400 F) test temperature: $F_{cu} = \frac{F_{cu\ test}}{1.0792}$; $E_c = 0.937 \times E_c$ test value. For 316 C (600 F) tests: $F_{cu} = \frac{F_{cu\ test}}{1.0651}$, $E_c = 0.944 \times E_c$ test value.

(3) Tests were performed at a load rate of 0.127 cm/minutes (0.05 inch/minute) after stabilizing at the test temperature for 10 minutes.

(4) Composite Physical Properties.

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)
1 618	25.6	68.8	-0.65	0.264	(10.4)	0.305-0.330	(12-23)	100

(5) The insitu imidizing-cure cycle specified was employed in laminate fabrication

Table 15. Compressive Properties of LARC-160/Celion Unidirectional $(0)_5$ Oriented Composite, Aged 125 Hours at 316 C (600 F)—Beam Test

Specimen[4] Number	Test[3] Temp C (F)	F_{cu} Test MN/m^2	F_{cu} Test (Ksi)	F_{cu} Adjusted[2] MN/m^2	F_{cu} Adjusted[2] (Ksi)	E_c Test GN/m^2	E_c Test (Msi)	E_c Adjusted[2] GN/m^2	E_c Adjusted[2] (Msi)	ε ult μ (%)	Failure Mode
EX199C-1	-168		(257)		(221)	—*	—	—	—	—	Compression 1 5 inch off ℄
-2	(-270)		(284)		(263)		(23 3)		(21 8)	11 7	Compression on ℄
-3			(261)		(242)		(23 6)		(22 1)	13 9	Compression on ℄
	Avg	1842	(267)	1667	(242)	162	(23 5)	151	(22.0	12 8	
EX199C-4			(198)		(184)		(21 1)		(19.8)	10.5	Compression on ℄
-5	RT		(264)		(245)		(20 7)		(19 4)	14 4	Compression 1.25 inch off ℄
-6			(257)		(238)		(21 6)		(20 2)	13 7	Compression over loading hole
	Avg	1653	(240)	1529	(222)	145	(21 1)	136	(19 8)	12 9	
EX199C-10	204		(128)		(119)	—*	—	—	—	—	Compression 1 5 inch off ℄
-11	(400)		(123)		(114)		(28 1)		(26 3)	4 60	Compression 1 0 inch off ℄
-12			(112)		(104)		(23 4)		(21 9	4 80	Compression 0 5 inch off ℄
	Avg	834	(121)	774	(112)	177	(25 8)	166	(24 1)	4 7	
EX199C-7	316		(127)		(119)	—*	—	—	—	—	Compression 1 0 inch off ℄
-8	(600)		(122)		(114)		(24 2)		(22.8)	5 40	Compression on ℄
-9			(89 1)		(83 7)		(24 3)		(22.9)	3.94	Compression 1 0 inch off ℄
	Avg	777	(113)	727	(106)	167	(24 3)	157	(22.9	4 67	

(1) Compression critical beams per the design described. Aluminum honeycomb 5052 alloy core, 1/8 cell, 352 g/m^3 (22 psf) density was employed in -168 C, RT and 204 C (-270 F, RT and 400 F) tests, CRES core, 301 alloy 1/8 cell, 0.127 mm (0 005 inch) foil 40 pcf was employed in 316 C (600 F) tests

(2) Adjusted properties were calculated from equations derived by Mr Mark Shuart, NASA/LaRC using a computer program that considers the effect of bulk core properties on the strength and elastic modulus properties of the laminate Adjustment factors for -168 C, RT and 204 C (-270 F, RT and 400 F) test temperature

$$F_{cu} = \frac{F_{cu\ test}}{1\ 0792};\ E_c = 0.937 \times E_c \text{ test value} \quad \text{For 316 C (600 F) tests}$$

$$F_{cu} = \frac{F_{cu\ test}}{1\ 0651},\ E_c = 0\ 944 \times E_c \text{ test value}$$

(3) Tests were performed at a load rate of 0 127 cm/minutes (0 05 inch/minute) after stabilizing at the test temperature for 10 minutes

(4) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	Wt Loss After 125 Hours at 316 C (600 F) (%)
1 601	29 9	63 4	-0 22	0 284	(11 2)	0 279-0 318	(11-12 5)	99	3 4

(5) The 2 stage cure cycle specified was employed in laminate fabrication.
*Strain gage failure

Table 16. Compressive Properties of LARC-160/Celion (0, ±45, 90)$_S$ Oriented Composite, Postcured Condition—Beam Test

(4)(5) Specimen Number	Test(3) Temp C (F)	F_{cu} Test MN/m^2	F_{cu} Test (ksi)	F_{cu} Adjusted(2) MN/m^2	F_{cu} Adjusted(2) (ksi)	E_c Test GN/m^2	E_c Test (msi)	E_c Adjusted(2) GN/m^2	E_c Adjusted(2) (msi)	ε ult μ (%)	Failure Mode
EX106C-4	-168		(117)		(98.4)		-		-	-	Compression on ℄
-5	(-270)		(138)		(116)		(10.43)		(9.13)	1.73	Compression overloading hole
-6			(118)		(99.2)		(10.10)		(8.83	1 14	Compression in center
	Avg	857	(124)	720	(105)	70.73	(10.27)	61.87	(8.98)	1.44	
EX106C-1			(116)		(97.5)		(10.15)		(8.88)	1.33	Compression overloading hole
-2	RT		(113)		(95.0)		(10.90)		(9.54)	1.16	Compression on ℄
-3			(95)		(79.9)		(9.50)		(8.31)	1.11	Compression on ℄
	Avg	744	(108)	626	(90.8)	70.16	(10.18)	61.39	(8.91)	1.21	
EX106C-7	204		>(68.1)		>(57.20)		(7.83)		(6.85)	>0.950	Composite-to-core bond failure
-8	(400)		>(81.8)		>(68.75)		(8.70)		(7.61)	>1.10	Composite-to-core bond failure
-9			>(85.73)		>(72.08)		(8.04)		(7.04)	>1.16	Composite-to-core bond failure
	Avg	541	(78.5)	455	>(66.01)	56.43	(8.19)	49.38	(7.17)		
EX106C-10	316		(66.1)		(59.1)		(7.99)		(7.22)	0.984	Compression on ℄
-11	(600)		>(69.4)		>(62.0)		(8.50)		(7.68)	>0 906	Composite-to-core bond failure
-12		[657]	[95.3]	[587]	[85.2]	[68.9]	[10.00]	[62.3]	[9.04]	[0.890]	[Tested in tension—tensile failure]
-13			>(80.1)		>(71.6)		(9.8)		(8.86)	>1.00	Composite-to-core bond failure
-14			(91.8)		(82.0)		10.02		9.06	1 16	Compression on ℄
	Avg	544	79.0	486	70.6	62.0	(9.05)	56.0	(8.14)	1.07	

(1) Compression critical beams per the design described. Aluminum honeycomb 5052 alloy core, 1/8 cell, 352 g/m^3 (22 pcf) density was employed in -168 C, RT and 204 C (-270 F, RT and 400 F) tests. CRES core, 301 alloy, 1/8 cell, 0.127 mm (0.005 inch) foil, 40 pcf employed in 316 C (600 F) tests.

(2) Adjusted properties were calculated from equations derived by Mr. Mark Shuart, NASA/LaRC using a computer program that considers the effect of bulk core properties on the strength and elastic modulus properties of the laminate. Adjustment factors for -168 C, RT and 204 C (-270 F, RT, and 400 F) test temperatures. $F_{cu} = \frac{F_{cu\ test}}{1.1893}$, E_c = 0.875 x E_c test value For 316 C (600 F) tests $F_{cu} = \frac{F_{cu\ test}}{1.1192}$, E_c = 0.904X E_c test value.

(3) Tests were performed at a load rate of 0.127 cm/minute (0.05 inch/minute) after stabilizing at the test temperature for 10 minutes.

(4) Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	TMA-Tg (C)
1.588	30 2	62 9	0.47	0.461	(18.1)	0.457-0 503	(18-20)	100	362

(5) The insitu imidizing-cure cycle specified was employed in laminate fabrication

Table 17. Compressive Properties of LARC-160/Celion (0, ±45, 90)$_S$ Oriented Composite, Aged 125 Hours at 316 C (600 F)—Beam Test

Specimen[4][5] Number	Test[3] Temp C (F)	F_{cu} Test MN/m^2	F_{cu} Test (Ksi)	F_{cu} Adjusted[2] MN/m^2	F_{cu} Adjusted[2] (Ksi)	E_c Test GN/m^2	E_c Test (Msi)	E_c Adjusted[2] GN/m^2	E_c Adjusted[2] (Msi)	ε ult μ (%)	Failure Mode
EX200C-1	-168		(122)		(103)	—*		—	—	—	Compression over loading hole
-2	(-2/0)		(79 1)		(66.5)		(9 60)		(8 40)	9 44	Compression over loading hole
-3			(82 5)		(69 4)		(9 10)		(7 96)	9 80	Compression 1 5 inch off ℄
	Avg	651	(94.5)	548	(79 6)	64 4	(9 35)	56 4	(8 18)	9 62	
EX200C-4			(90 1)		(75 7)		(10.50)		(9.19)	10.00	Compression on ℄
-5	RT		(102)		(85 9)		(8 50)		(7 44)	14.00	Compression outside loading hole
-6			(102)		(85.7)		(8 48)		(7 13)	13 90	Compression on ℄
	Avg	675	(98.0)	568	(82 4)	63 1	(9.16)	54.6	(7.92)	12 63	
EX200C-10	204		(77 8)		(65 4)	—*	—	—	—	—	Compression over loading hole
-11	(400)		(66 4)		(55 8)		(8.08)		(7 07)	9 80	Compression on ℄
-12			(87 5)		(73.6)		(8 13)		(7 11)	13 10	Compression over loading hole
	Avg	532	(77 2)	447	(64 9)	55 9	(8.11)	48 9	(7 09)	11 45	
EX200C-7	316		(65 7)		(58 7)	—*	—	—	—	—	Compression 1.25 inch off ℄
-8	(600)		(80 8)		(72.2)		(8 88)		(8 02)	11 40	Compression over loading hole
-9			(69 1)		(61 8)		(8 48)		(7 66)	9 80	Compression on ℄
	Avg	495	(71.8)	442	(64 2)	59 8	(8 68)	54 0	(7.84)	10 6	

(1) Compression critical beams per the design described. Aluminum honeycomb 5052 alloy core, 1/8 cell, 352 g/m^3 (22 pcf) density was employed in -168 C, RT and 204 C (-270 F, RT and 400 F) tests. CRES core, 301 alloy, 1/8 cell, 0 127 mm (0 005 inch) foil, 40 pcf employed in 316 C (600 F) tests.

(2) Adjusted properties were calculated from equations derived by Mr. Mark Shuart, NASA/LaRC using a computer program that considers the effect of bulk core properties on the strength and elastic modulus properties of the laminate. Adjustment factors for -168 C, RT and 204 C (-270 F, RT, and 400 F) test temperatures

$$F_{cu} = \frac{F_{cu\ test}}{1\ 1893},\quad E_c = 0\ 875 \times E_c \text{ test value} \quad \text{For 316 C (600 F) tests}$$

$$F_{cu} = \frac{F_{cu\ test}}{1\ 1192},\quad E_c = 0\ 904 \times E_c \text{ test value}$$

(3) Tests were performed at a load rate of 0 127 cm/minute (0.05 inch/minute) after stabilizing at the test temperature for 10 minutes

(4) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	TMA-Tg (C)	Wt Loss After 125 Hrs at 316 C (600 F) (%)
1 594	31 2	62 0	-0 21	0 465	(18 32)	0 48-0 54	(19-21 5)	100	365	3 2

(5) The 2 stage cure cycle specified was employed in laminate fabrication

*Strain gage failure.

Table 18. Summary of LARC-160/Celion Tensile Properties

Panel No. & Spec.	Fiber Orientation/ Specimen	Tensile Property(1)	Test Temperature							
			Postcured				Aged 125 Hours at 316 C (600 F)			
			-168 C (-270 F)	RT	204 C (400 F)	316 C (600 F)	-168 C (-270 F)	RT	204 C (400 F)	316 C (600 F)
EX107 (P.C.) EX199 (Aged)	$(0)_t$/ Beam	F_{tu} MN/m^2	2065	1941	1076	1647	1727	2104	1633	--
		(Ksi)	(300)	(282)	(276)	(239)	(251)	(305)	(237)	--
		E_t GN/m^2	184	174	160	166	172	161	185	171
		(Msi)	(26.72)	(25.30)	(23.7)	(24.07)	(24.4)	(23.4)	(26.8)	24.8
		ϵ ULT μ (%)	1.14	1.15	1.28	0.970	1.51	1.36	0.94	--
		ν	0.370	0.275	0.310	0.310	--	--	--	--
EX98 (P.C.) EX201 (Aged)	$(90)_t$/ Coupon	F_{tu} MN/m^2	35.6	23.0	15.8	18.1	47.3	35.1	12.1	14.0
		(Ksi)	(5.17)	(3.34)	(2.30)	(2.63)	(6.87)	(5.10)	(1.75)	(2.03)
		E_t GN/m^2	11.02	9.20	8.20	5.23	TBD	TBD	TBD	TBD
		(Msi)	1.60	1.60	(1.19)	0.759	TBD	TBD	TBD	TBD
		ϵ ULT μ (%)	0.33	0.33	0.20	0.37	TBD	TBD	TBD	TBD
		ν	0.068	0.068	0.041	0.031	TBD	TBD	TBD	TBD
EX105 (P.C.) EX202 (Aged)	$(\pm 45)_S$/ Coupon	F_{tu} MN/m^2	201	201	149	141	15.7	134	132	103
		(Ksi)	29.1	29.1	(21.6)	(20.5)	(22.8)	(19.4)	(19.1)	(14.9)
		E_t GN/m^2	27.69	27.69	20.26	17.71	TBD	TBD	TBD	TBD
		(Msi)	(4.02)	(4.02)	(2.94)	(2.57)	TBD	TBD	TBD	TBD
		ϵ ULT μ (%)	0.74	0.74	--	--	TBD	TBD	TBD	TBD
		ν	0.75	0.75	0.84	0.92	TBD	TBD	TBD	TBD
EX106 (P.C.) EX200 (Aged)	$(0,\pm 45,90)_S$ Coupon	F_{tu} MN/m^2	517	569	556	560	480	446	434	491
		(Ksi)	(74.9)	(82.5)	(80.7)	(81.3)	(69.6)	(64.7)	(63.3)	(71.3)
		E_t GN/m^2	56.03	53.28	55.14	41.96	TBD	TBD	49.61	TBD
		(Msi)	(8.13)	(7.73)	(8.00)	(6.09)	TBD	TBD	(7.2)	TBD
		ϵ ULT μ (%)	0.96	1.10	1.02	0.89	TBD	TBD	TBD	TBD
		ν	0.320	0.295	0.325	0 .300	TBD	TBD	TBD	TBD

(1) ν Poissons ratio values reported for tension beam specimens were calculated from coupon specimen data.

Table 19. Tensile Properties of LARC-160/Celion $(0)_5$ Oriented Postcured Composites(1)(2)

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu} (5)		E_t		ν_t	ε Ult μ (%)
		MN/m^2	(Ksi)	GN/m^2	(MSI)		
EX107-2-1	-168	1626	(236)		(24.5)	—	0.96A
-2-2	(-270)	1605	(233)		(22.5)	0.390	1.08 *
-2-3		1743	(253)	___	(21.6)	0.350	1.16 *
				Avg 158	(22.2)	0.370	
EX107-1-1		1709	(248)		(23.0)	—	1.05A
-1-2	RT	950	(137)		(18.4)	0.260	0.75 *
-1-3		1337	(194)	___	(21.4)	0.290	0.89 *
				Avg 145	(21.1)	0.275	
EX107-3-1	204	1633	(232)		(21.7)	—	1.17A
-3-2	(400)	1578	(229)		(21.6)	0.330	0.98*
-3-3		1357	(197)	___	(24.4)	0.290	0.81*
				Avg 156	(22.6)	0.310	
EX107-4-1	316	1357	(197)		(21.3)	—	0.90A
-4-2	(600)	1240	(180)		—	—	—
-4-3		1433	(208)	___	(24.0)	0.310	0.87*
				Avg 156	(22.7)	0.310	

(1) Coupon tensile specimen design, straight sides, 2.54 cm (1.00 inch) wide. Laminate consisted of 5 ply 0° oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness				C-Scan Transmission (%)
				Calculated		Actual Range		
				mm	(mils)	mm	(mils)	
1.618	25.6	68.8	-0.65	0.264	(10.4)	0.305-0.330	(12-13)	100

(3) Load was applied at 1.27 mm/minute (0.05 inch) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimens were loaded incrementally to allow for data aquisition.

(4) -1 specimens were tested with a 5.08 cm (2.0 inch) gage length extensometer. Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125A-A-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages.

(5) Ftu data points were not averaged since strain gaged specimens were not tested under a constant loading condition.

*** Projected from last strain gage reading**

A Actual

Table 20. Tensile Properties of LARC-160/Celion $(0)_5$ Oriented Composite, Aged 125 Hours at 316 C (600 F) (1) (2).

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu} MN/m^2	F_{tu} (Ksi)	E_t GN/m^2	E_t (MSI)	νt	ϵ Ult μ (%)
EX199TC-1	-168		207		22.1		
-2	(-270)		182			TBD	TBD
-3		———	-*				
	AVG	1340	195				
EX199TC-4			192		22 8		
-5	RT		-**			TBD	TBD
-6		———	199				
	AVG	1347	196				
EX199TC-7	204		211		24 1		
-11	(400)		-**			TBD	TBD
-12			201		———		
	AVG	1419	206				
EX199TC-8	316		-**				
-9	(600)		-**			TBD	TBD
-10		———	-**		20 8		
	AVG		———		———		

(1) Coupon tensile specimen design, straight sides, 2.54 cm (1.00 inch) wide. Laminate consisted of 5 ply 0° oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	Wt Loss After 125 Hours at 316 C (600 F) %
1 601	29.9	63.4	-0 22	0.284	(11.2)	0 279-0 318	(11-12.5)	100	3.4

(3) Load was applied at 1 27 mm/minute (0 05 inch) after specimens had stabilized at test temperature for 10 minutes Strain gaged specimen data aquisition was obtained autographically on two X, Y, Y recorders

(4) -1 specimens were tested with a 5 08 cm (2 0 inch) gage length extensometer Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages, -2 and 3 specimens tested at 204 C (400 F), type 0K-00-125A-A-350 (LEN) gages, -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages

* Damaged specimen

**Graphite/polyimide tab caused slipping in grips, damaged specimen.

Table 21. Tensile Properties of LARC-160/Celion (90)40 Oriented Postcured Composites(1) (2)

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu} (5)		E_t		ν_t	ε Ult μ (%)
		MN/m^2	(Ksi)	GN/m^2	(MSI)		
EX98-2-1	-168	24.05	(3.49)	—	(1.63)	—	0.21 A
-2-2	(-270)	47.20	(6.85)		(1.57)	0.068	0.45*
-2-3		—	—	—	—	—	—
				Avg 11.02	(1.60)	0.068	
EX98-1-1		24.87	(3.61)		(1.26)	—	0.28 A
-1-2	RT	—	—	—	—	—	—
-1-3		21.15	(3.07)		(1.41)	0.051	0.22*
				Avg 9.20	(1.34)	0.051	
EX98-3-1	204	15.16	(2.20)		(1.11)	—	0.20 A
-3-2	(400)	15.23	(2.21)		(1.28)	0.032	0.17*
-3-3		17.16	(2.49)		(1.18)	0.049	0.22*
				Avg 8.20	(1.19)	0.041	
EX98-4-1	316	24.87	(3.61)		(0.883)	—	0.43A
-4-2	(600)	14.74	(2.14)		(0.740)	0.021	0.33*
-4-3		14.81	(2.15)		(0.653)	0.041	0.35*
				Avg 5.23	(0.759)	0.031	

(1) Coupon tensile specimen design straight sides, 2.54 cm (1.0 inch) wide. Laminate consisted of 40 ply 90° oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness				C-Scan Transmission (%)	TMA-Tg (C)
				Calculated		Actual Range			
				mm	(mils)	mm	(mils)		
1.598	28.4	65.4	-0.29	2.21	86.7	1.80-2.05	75-81	100	340

(3) Load was applied at 1.27 mm/minute (0.05 inch) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimens were loaded incrementally to allow for data aquisition.

(4) -1 specimens were tested with a 5.08 cm (2.0 inch) gage length extensometer. Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125a-a-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages.

(5) Ftu data points were not averaged since strain gaged specimens were not tested under a constant loading condition.

* Projected from last strain gage reading

A Actual

Table 22. Tensile Properties of LARC-160/Celion $(90)_{40}$ Oriented Composite Aged 125 Hours at 316 C (600 F) (1) (2)

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu} MN/m^2	F_{tu} (Ksi)	E_t GN/m^2	E_t (MSI)	νt	ϵ Ult μ (%)
EX201TC-1	-168		6 90			-	
-2	(-270)		6.85	TBD	TBD	TBD	TBD
-3			-*				
	Avg	47.3	6.87				
EX201TC-4			4.80	9.64	1.4		
-5	RT		5 40	TBD	TBD	TBD	TBD
-6			5 10				
	Avg	35.1	5.10				
EX201TC-7	204		1.90	7.57	1 1		
-10	(400)		1.60	TBD	TBD	TBD	TBD
	Avg	12.1	1 75				
EX201-8	316		1.80				
-9	(600)		2.30	TBD	TBD	TBD	TBD
-11			2 00				
	Avg	14 0	2 03				

(1) Coupon tensile specimen design straight sides, 2.54 cm (1.0 inch) wide. Laminate consisted of 40 ply 90° oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	TMA-Tg (C)	Wt/ Loss After 125 Hours at 316C (600F) %
1 604	30.0	63.4	-0.45	2.27	(89.2)	2 48-2 57	(98-101)	100	359	0 86

(3) Load was applied at 1 27 mm/minute (0 05 inch) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimen data aquisition was obtained autographically using X, Y, Y recorders

(4) -1 specimens were tested with a 5 08 cm (2 0 inch) gage length extensometer. Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125a-a-350 (LEN) gages, -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages

* Damaged specimen

Table 23. Tensile Properties of LARC-160/Celion $(\pm 45)_S$ Oriented Postcured Composites[1] [2]

(4) Specimen Number	Test (3) Temperature C (F)	F_{tu} (5) MN/m²	F_{tu} (5) (Ksi)	E_t GN/m²	E_t (MSI)	ν_t	ε Ult μ (%)
EX105-2-1	-168	200	(29.1)		(3.58)	—	—
-2-2	(-270)	209	(30.4)		(4.30)	0.72	0.74*
-2-3		192	(27.9)		(4.17)	0.77	0.70*
			Avg	27.69	(4.02)	0.75	
EX105-1-1		176	(25.5)		(3.10)		—
-1-2	RT	163	(23.7)		(3.48)	0.77	—
-1-3		168	(24.7)		(3.22)	0.74	—
			Avg	22.25	(3.23)	0.76	
EX105-3-1	204	164	(23.8)		(2.53)	—	—
-3-2	(400)	149	(21.6)		(3.15)	0.75	—
-3-3		135	(19.6)		(3.15)	0.93	—
			Avg	20.26	(2.94)	0.84	
EX105-4-1	316	150	(21.8)		(2.15)	—	—
-4-2	(600)	130	(18.9)		(2.22)	0.93	—
-4-3		143	(20.7)		(3.33)	0.91	—
			Avg	17.71	(2.57)	0.92	

[1] Coupon tensile specimen design, straight sides, 2.54 cm (1.0 inch) wide. Laminate consisted of 4 ply $(\pm 45)_S$ oriented, nominal 0.064 cm (2.5 mils) per ply.

[2] Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)
1.592	29.0	64.6	-0.10	0.223	8.88	0.229-0.254	9-10	100

[3] Load was applied at 1.27 mm/minute (0.05 inches) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimens were loaded incrementally to allow for data aquisition.

[4] -1 specimens were tested with a 5.08 cm (2.0 inch) gage length extensometer. Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125A-A-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages.

[5] Ftu data points were not averaged since strain gaged specimens were not tested under a constant loading condition.

*Projected from last strain gage reading.

Table 24. Tensile Properties of LARC-160/Celion $(\pm 45)_S$ Oriented Composite, Aged 125 Hours at 316 C (600 F) (1) (2)

Specimen[4] Number	Test[3] Temperature C (F)	F_{tu} MN/m^2	F_{tu} (Ksi)	E_t GN/m^2	E_t (MSI)	νt	ϵ Ult μ (%)
EX202TC-1	-168		(23.1)	8.27	1 2		
-2	(-270)		(21 3)	TBD	TBD	TBD	TBD
-3			(24 0)				
	Avg	157	(22.8)				
EX202TC-4			(17 6)	14.5	2 1		
-5	RT		(18 7)	TBD	TBD	TBD	TBD
-6			(21.9)				
	Avg	134	(19.4)				
EX202TC-7	204		(19.2)	16.5	2 4		
-11	(400)		-*	TBD	TBD	TBD	TBD
-12			(19.0)				
	Avg	132	(19 1)				
EX202-8	316		(14 0)	-			
-9	(600)		(15 0)	TBD	TBD	TBD	TBD
-10			(15.7)	12.4	1.8		
	Avg	103	(14.9)				

(1) Coupon tensile specimen design, straight sides, 2.54 cm (1.0 inch) wide Laminate consisted of 4 ply $(\pm 45)_S$ oriented, nominal 0.064 cm (2 5 mils) per ply

(2) Composite Physical Properties

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	Thickness Calculated (mils)	Thickness Actual Range mm	Thickness Actual Range (mils)	C-Scan Transmission (%)	Wt Loss After 125 Hours at 316 C (600 F) %
1.580	33.6	59.26	-0.10	0.238	(9.36)	0 203-0.254	(8-10)	100	5 2

(3) Load was applied at 1 27 mm/minute (0.05 inches) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimen data acquisition was obtained autographically using two X, Y, Y recorders

(4) -1 specimens were tested with a 5.08 cm (2 0 inch) gage length extensometer Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125A-A-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages

* Damaged specimen

Table 25. Tensile Properties of LARC-160/Celion (0, ±45, 90)S Oriented Postcured Composite(1) (2)

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu} (5) MN/m^2	(Ksi)	E_t GN/m^2	(MSI)	γ_t	ε Ult μ (%)
EX106-2-1	-168	581	(84.3)		(8.67)	—	0.99A
-2-2	(-270)	393	(57.0)		(7.55)	0.310	0.79*
-2-3		576	(83.6)		(8.18)	0.330	1.11*
			Avg	56.03	(8.13)	0.320	
EX106-1-1		619	(89.9)		(7.55)	—	1.22*A
-1-2	RT	558	(81.0)		(7.76)	0.280	1 08*
-1-3		528	(76.7)		(7.89)	0.310	1.01*
			Avg	53.28	(7.73)	0.295	
EX106-3-1	204	634	(92.0)		(7.79)	—	1.20A
-3-2	(400)	544	(79.0)		(8.04)	0.310	0.99*
-3-3		490	(71.1)		(8.18)	0.340	0.86*
			Avg	55.14	(8.00)	0.325	
EX106-4-1	316	604	(87.6)		(7.82)	—	0.90A
-4-2	(600)	573	(83.1)		(5.20)	0.320	0.96*
-4-3		504	(73.2)		(5.25)	0.280	0.83*
			Avg	41.96	6.09	0.300	

(1) Coupon tensile specimen design necked down test section. Laminate consisted of 8 ply (0, ±45, 90)S oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties:

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness Calculated mm	(mils)	Actual Range mm	(mils)	C-Scan Transmission (%)	TMA-Tg (C)
1.588	30.2	62.9	0.47	0.461	(18.1)	0.457-0.503	(18-20)	100	365

(3) Load was applied at 1.27 mm/minute (0.05 inch) after specimens had stabilized at test temperature for 10 minutes. Strain-gaged specimens were loaded incrementally to allow for data aquisition.

(4) -1 specimens were tested with a 5.08 cm (2.0 inch) gage length extensometer. Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type WK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type OK-00-125A-A-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages.

(5) Ftu data points were not averaged since strain gaged specimens were not tested under a constant loading condition.

*** Projected from last strain gage reading**

A Actual

Table 26. Tensile Properties of LARC-160/Celion (0, 145, 90)S Oriented Composite, Aged 125 Hours at 316 C (600 F) (1) (2)

Specimen (4) Number	Test (3) Temperature C (F)	F_{tu}		E_t		γ_t	ϵ Ult μ (%)
		MN/m^2	(Ksi)	GN/m^2	(MSI)		
EX200TC-1	-168		(71.2)	53.1	7 7		
-2	(-270)		(70.8)	TBD	TBD	TBD	TBD
-3			(66.8)				
	Avg	480	(69.6)				
EX200TC-4			63.1	47.5	6.9		
-5	RT		70.9	TBD	TBD	TBD	TBD
-6			60.0				
	Avg	446	64.7				
EX200TC-7	204		63.3	49.6	7.2		
	(400)			TBD	TBD	TBD	TBD
	Avg	434	63.3		7.2		
EX200TC-8	316		70.9				
-9	(600)		68.1	TBD	TBD	TBD	TBD
-10			74.8	46.1	6.7		
	Avg	491	71.3				

(1) Coupon tensile specimen design necked down test section. Laminate consisted of 8 ply (0, ±45, 90)S oriented, nominal 0.064 cm (2.5 mils) per ply.

(2) Composite Physical Properties·

Density (grams/cc)	Resin Content (%)	Fiber Vol (%)	Void Vol (%)	Thickness				C-Scan Transmission (%)	TMA-Tg (C)	Wt. Loss After 125 Hours at 316 C (600 F) %
				Calculated		Actual Range				
				mm	(mils)	mm	(mils)			
1.594	31.2	62.0	-0.21	0.465	(18.32)	0.48-0.54	(19-21.5)	100	365	3.2

(3) Load was applied at 1.27 mm/minute (0.05 inch) after specimens had stabilized at test temperature for 10 minutes. Strain gaged specimen data acquisition was obtained autographically on 2 X, Y, Y recorders.

(4) -1 specimens were tested with a 5.08 cm (2.0 inch) gage length extensometer Specimens -2 and 3 tested at -168 C and 316 C (-270 F and 600 F) employed type OK-00-125AD-350 gages; -2 and 3 specimens tested at 204 C (400 F), type)K-00-125A-A-350 (LEN) gages; -2 and 3 specimens tested at RT, type CEA-00-125UT-350 gages.

Table 27. Summary of LARC-160/Celion Compression Properties

Panel No.	Fiber Orientation/ Specimen	Compression Property	Test Temperature: Postcured -168 C (-27°F)	Postcured RT	Postcured 204 C (400 F)	Postcured 316 C (600 F)	Aged 125 Hours at 316 C (600 F): -168 C (-27°F)	Aged 125 Hours at 316 C (600 F): RT	Aged 125 Hours at 316 C (600 F): 204 C (400 F)	Aged 125 Hours at 316 C (600 F): 316 C (600 F)
EX107 (P.C.) EX199 (Aged)	$(0)_t$/ Beam	E_{CU} MN/m² (Ksi)	18.2 (263)	1373 (199)	1171 (170)	880 (128)	1667 (242)	1529 (222)	774 (112)	727 (106)
		E_C GN/m² (Msi)	159 (23.03)	155 (20.98)	141 (20.4)	141 (20.55)	151 (22.0)	136 (19.8)	166 (24.1)	157 (22.9)
		ε ULT μ(%)	1.42	1.05	0.960	0.643	1.28	1.29	0.47	0.47
EX98 (P.C.) EX201 (Aged)	$(90)_t$/ Coupon	E_{CU} MN/m² (Ksi)	231 (33.5)	175 (25.4)	138 (201)	92.3 (13.4)	163 (23.7)	157 (22.8)	122 (177.7)	103 (15.0)
		E_C GN/m² (Msi)	11.8 (1.71)	9.3 (1.35)	7.02 (1.02)	5.79 (0.841)	11.3 (1.65)	8.98 (1.30)	7.37 (1.07)	7.19 (1.04)
		ε ULT μ(%)	2.03	2.17	2.44	2.80	1.64	1.43	1.86	1.83
EX105 (P.C.) 220 (Aged)	$(\pm45)_S$/ Coupon	E_{CU} MN/m² (Ksi)	242 (35.1)	182 (26.4)	130 (18.9)	63.5 (9.22)	208 (30.2)	157 (22,8)	138 (20.0)	125 (18.2)
		Ec GN/m² (Msi)	19.9 (2.90)	15.8 (2.29)	107 (1.55)	10.1 (1.47)	20.8 (3.02)	17.2 (2.50)	14.1 (2.05)	12.7 (1.85)
		ε ULT μ(%)	1.50	3.00	4.17	2,27	1.60	1.47	-	-
EX106 (P.C.) EX200 (Aged)	$(0,\pm45,90)_S$/ Beam	E_{CU} MN/m² (Ksi)	720 (105)	626 (90.8)	>455 >(66.01)	486 (70.6)	548 (79.6)	568 (82.4)	447 (64.9)	442 (64.2)
		E_C GN/m² (Msi)	61.87 (8.98)	61.39 (8.91)	49.38 (7.17)	56.0 (8.14)	56.4 (8.18)	54.6 (7.92)	48.8 (7.09)	54.0 (7.84)
		ε ULT μ(%)	1.44	1.21	>1.10	1.07	0.96	1.26	1.15	1.06

Table 28. Compressive Properties of $(90)_{40}$ and $(\pm 45)_S$ Oriented Fiber LARC-160/Celion Composites

Panel No / Orientation/ (plies)	Condition (1)	-168°C (-270°F) (2)					RT					204°C (400°F) (2)					316°C (600°F) (2)				
		F_{cu}		F_c		ε ult (μ)	F_{cu}		E_c		ε ult (μ)	F_{cu}		E_c		ε ult (μ)	F_{cu}		F_c		ε ult (μ)
		MN/m^2	(Ksi)	GN/m^2	(Msi)	(%)	MN/m^2	(Ksi)	GN/m^2	(Msi)	(%)	MN/m^2	(Ksi)	GN/m^2	(Msi)	(%)	MN/m^2	(Ksi)	GN/m^2	(Msi)	(%)
EX98			(31 2)		(1 72)	1 86		(23 1)		(1 35)	1 96		20 7		1 00	2 46		13 1		0 826	3 00
(90)/	①		(35 6)		(1 76)	2 13		(27 3)		(1 39)	2 32		20 1		1 01	2 48		13 7		0 837	2 50
(40)			(33 8)		(1 64)	2 11		(25 7)		(1 32)	2 23		19 4		1 04	2 37		13 3		0 860	2 90
	Avg	231	(33 5)	11 8	1 71	2 03	175	(25 4)	9 3	(1 35)	2 17	138	20 1	7 02	1 02	2 44	92 3	13 4	5 79	0 841	2 80
EX201			(25 2)		(1 64)	1 68		(14 9)		(1 29)	1 19		--		--	--		(14 2)		(1 06)	1 61
(90) /	②		(21 5)		(1 64)	1 66		(18 9)		(1 30)	1 55		--		--	--		(14 6)		(1 01)	1 89
(40)			(24 4)		(1 67)	1 59		(19 8)		(1 30)	1 55		(17 7)		1 07	1 36		(16 1)		(1 06)	2 01
	Avg	163	(23 7)	11 3	(1 65)	1 64	157	(22 8)	8 98	(1 30)	1 43	122	(17 7)	7 37	1 07	1 86	103	15 0	7 19	(1 04)	1 83
EX91			37 7		2 93	1 51		25 3		2 23	2 91		17 8		1 47	3 49		8 49		1 66	1 89
$(+45)_S$/	①		37 0		2 84	1 76		25 9		2 46	2 30		19 0		1 40	4 50		9 35		1 11	2 58
(32)			30 5		2 94	1 22		27 9		2 18	4 06		19 8		1 78	4 53		9 82		1 64	2 37
	Avg	242	35 1	19 9	2 90	1 50	182	26 4	15 8	2 29	3 00	130	18 9	107	1 55	4 17	63 3	9 22	10 1	1 47	2 28
EX220			(28 8)		(3 18)	1 17		(22 1)		(2 56)	1 41		(18 6)		(2 07)	--		(19 7)		(1 77)	--
$(\pm 45)_S$/	②		(32 8)		(2 80)	2 45		(23 6)		(2 52)	1 54		(20 3)		(2 07)	--		(16 7)		(1 73)	--
(32)			(29 1)		(3 08)	1 18		(22 7)		(2 48)	--		(21 2)		(2 01)	--		(18 1)		(2 05)	--
	Avg	208	30 2	20 8	(3 02)	1 60	157	(22 8)	17 2	2 50	1 47	138	20 0	14 1	(2 05)		125	(18 2)	12 7	1 85	

Composite Physical Properties (4)	Target properties	EX98	EX91	EX201	EX220
1 Specific gravity (grams/cc)	1 561-1 579	1 598	1 558	1 604	1 589
2 Resin weight content (%)	35 0-31 3	28 4	34 7	30 0	32 5
3 Fiber volume (%)	58-62	65 4	58 1	63 4	59 4
4 Void Volume (%)	<2	-0 29	0 29	0 45	0 90
5 Thickness mm (mils)		1 90-2 05 (75-81)	2 00-2 13 (79-84)	2 48-2 57 (98-101)	1 98-2 08 (78-82)
6 Thickness/ply, mm (mils)	0 0660-0 0609 (2 6-2 4)	0 047-0 051 (1 87-2 0)	0 063-0 067 (2 47-2 63)	062- 064 (2 45-2 52)	062- 065 (2 43-2 56)
7 Barcol hardness (ASTM D2583)	>70	75-78	76-79	72-78	73-78
8 Weight loss in postcure (%)	<1	0 15	0 31	--	00 73
9 Weight loss after 125 hrs at 316°C (%)	<1	--	--	0 86	--
10 TMA-Tg C, (F) cured	>330 (626)	330 (626)	352 (666)	--	--
Postcured 4 hours at 316°C (600°F)	>340 (644)	340 (644)	332 (630)	--	349 (660)
Aged 125 hours at 316°C (600°F)	>340 (644)	--	--	359 (678)	
11 C-scan ultra sound transmission (%) (3)					
Cured	>95	100	100	100	100
Postcured 4 hours at 316°C (600°F)	>95	100	100	100	100

(1) Condition ① Postcured 4 hours at 316°C (600°F), ② aged 125 hours at 316°C, (600°F) for 125 hours

(2) Specimens were tested after stabilizing at test temperature for 10 minutes at a load rate of 1 27 mm (0 05 inch)/minute in the test fixture shown in the Third Quarterly Report

(3) NDI ultra sonic through transmission tests were performed using the NASA-LARC established "A" sensitivity standards

(4) Insitu cure cycle used for EX91 & E98 laminates, two stage cycle used for EX201 and E220 laminates

Table 29. Flexural Properties of 0° Oriented Fiber LARC-160/Celion Laminates

Panel No / No. of Plies	Condition[1]	-168°C (-270°F)				RT				204°C (400°F)				316°C (600°F)			
		F_{fu}		E_f		F_{fu}		E_f		E_{fu}		E_f		F_{fu}		E_f	
		MN/m^2	(ksi)	GN/m^2	(msi)	MN/m^2	(ksi)	GN/m^2	(msi)	MN/m^2	(ksi)	GN/m^2	(msi)	MN/m^2	(ksi)	GN/m^2	(msi)
EX225/ 26	①	-	(296)		(18 9)		(238)		(17 3)		(143)		(19 7)		(161)		(18.4)
		-	(293)		(17.8)		(248)		(18 3)		(152)		(21 4)		(137)		(19.6)
		-	(295)		(18.1)		(241)		(18 0)		(203)		(19 1)		(134)		(19 6)
	Avg	2033	(295)	126	(18.3)	1674	(243)	123	(17.9)	1144	(166)	138	(20.1)	992	(144)	132	(19.2)
EX204/ 26	②		(304)		(19.6)		(254)		(18.0)		(210)		(19.0)		(161)		(17 7)
			(272)		(21 6)		(255)		(19.1)		(220)		(20.1)		(174)		(18 7)
			(255)		(22 2)		(267)		(19 0)		(227)		(18 3)		(176)		(19.4)
	Avg	1902	(276)	145	(21.1	1785	(259)	129	(18.7)	1509	(219)	132	(19.1)	1171	(170)	128	(18.6)

Composite Physical Properties[4]	Target Properties	EX225	EX204
1. Specific gravity (grams/cc)	1 561-1.579	1.581	1.608
2. Resin weight content (%)	35.0-31.3	30.4	30 4
3. Fiber volume (%)	58-62	62 2	63.2
4 Void volume (%)	< 2	0.87	-0.83
5. Thickness mm (mils)	1 716-1 583 (67.6-62 4)	1.47-1.62 (58-64)	1.37-1.54 (54-61)
6. Thickness/ply, mm (mils)	0 0660-0.0609 (2.6-2 4)	0.056-0.Q62 (2.23-2.46)	0.059-0.053 (2.08-2 35)
7 Barcol hardness (ASTM D2583)	> 70	72-78	73-74
8. Weight loss in postcure (%)	< 1	0.22	-
9. Weight loss after 125 hr at 316°C (°F)		-	1.03
10. TMA-Tg °C, (°F) cured	> 330 (626)	-	-
Postcured 4 hours at 316°C (600°F)	> 340 (644)	353 (667)	-
Aged 125 hours at 316°C (600°F)	> 340 (644)	-	365 (689)
11. C-scan ultra sound transmission (%)[3]			
Cured	> 95	100	100
Postcured 4 hours at 316°C (600°F)	> 95	100	100

[1] Condition ① Postcured 4 hours at 316°C (600°F), ② aged 125 hours at 316°C (600°F)

[2] Specimens were tested after stabilizing at test temperature for 10 minutes at a load rate of 1 27 mm (0 05 inch)

[3] NDI ultra sonic through transmission tests were performed using the NASA-LaRC established "A" sensitivity standards.

[4] The two stage cycle was employed in laminate fabrication

Table 30. Short Beam Shear Properties of 0° Oriented Fiber LARC-160/Celion Laminates

Panel No / No of Plies	Condition (1)	-168°C (-270 F)		RT		204 C (400°F)		316 C (600°F)	
		F_{su}		F_{su}		F_{su}		F_{su}	
		MN/m²	(ksi)	MN/m²	(ksi)	MN/m²	(ksi)	MN/m²	(ksi)
EX204/ 26	②		(19 0) (19 7) (20 9)		(17.1) (17.6) (17 5)		(12 6) (12.8) (12 6)		9 3 9 3 8 8
	Avg	137	(19.9)	120	(17.4)	86 8	(12 6)	63 4	(9.2)
EX225/ 26	①		(21 1) (23.9) (22 9)		(17 9) (18 3) (17 7)		(12 8) (12.9) (12 0)		(8.9) (8 5) (8.1)
	Avg	156	(22 6)	124	(18 0)	86 8	(12 6)	58.5	(8.5)

Composite Physical Properties (4)	Target Properties	EX225	EX204
1 Specific gravity (grams/cc)	1.561-1 579	1.581	1.608
2. Resin weight content (%)	35.0-31.3	30 4	30.4
3. Fiber volume (%)	58-62	62 2	63.2
4 Void volume (%)	<2	0.87	-0 83
5. Thickness mm (mils)	1.716-1 583 (67 6-624)	1 47-1.62 (58-64)	1 37-1.54 (54-61)
6. Thickness/ply, mm (mils)	0.0660-0 0609 (2.6-2 4)	0.059-0 062 (2.23-2.46)	0.059-0 053 (2.08-2.35)
7. Barcol hardness (ASTM D2583)	>70	72-78	73-74
8. Weight loss in postcure (%)	<1	0.22	—
9. Weight loss after 135 hours at 316°C (%)		—	1 03
10. TMA-Tg C, (F) cured	>330 (626)	—	—
Postcured 4 hours at 316°C (600°F)	>340 (644)	353 (667)	—
Aged 125 hours at 316°C (600°F)	>340 (644)	—	365 (689)
11. C-scan ultra sound transmission (%)(3)			
Cured	>95	100	100
Postcured 4 hours at 316°C (600°F)	>95	100	100

(1) Condition ① Postcured 4 hours at 316°C (600°F), ② aged 125 hours at 316°C (600°F)

(2) Specimens were tested after stabilizing at test temperature for 10 minutes at a load rate of 1 27 mm (0 05 inch)/minute

(3) NDI ultrasonic through transmission tests were performed using the NASA-LaRC established "A" sensitivity standards

(4) The two state cycle specified was employed in laminate fabrication

Table 31. Tensile and Flexural Properties (Average) of Celion/LARC-160 Chopped Unidirectional Tape Molding Compounds

Batch	Areal Weight gm/m^2	Fiber Length cm(in.)	Tensile Properties (1)		Flexural Properties (2)			
			22 C (75 F)	316 C (600 F)	22 C (75 F)		316 C (600 F)	
			F_{tu} (3) MN/m^2 (ksi)	F_{tu} (3) MN/m^2 (ksi)	F_{fu} (3) MN/m^2 (ksi)	E_f GN/m^2 (Msi)	F_{fu} (3) MN/m^2 (ksi)	E_f GN/m^2 (Msi)
A	66.8	1.27 to 2.54 (0.5 to 1.0)	194 (28.1)	184 (26.7)	586 (85.1)	74.4 (10.8)	497 (72.2)	72.3 (10.5)
B	153.7	1.27 (0.5)	116 (16.9)	89.6 (13.0)	245 (35.6)	37.9 (5.5)	196 (28.5)	35.1 (5.1)
C	60.4	1.27 (0.5)			484 (70.3)	73.7 (10.7)	278 (40.3)	55.8 (8.1)
D	67	2.54 (1.0)			855 (124)	82.7 (12)	537 (77.9)	73 (10.6)

(1) Individual tensile coupons per ASTM D 651 were net molded at 13.78 MN/m^2 (2,000 psi) 316 ±5 C (600 ±10 F) for 1 hour. Pressure was applied when part reached 204 C (400 F). Parts were force cooled under pressure to 66 C (150 F).

(2) Individual flexure coupons per ASTM D 790 were net molded at 13.78 MN/m^2 (2,000 psi) 316 ±5.5 C (600 ±10 F). Pressure was applied when part reached 204 C (400 F). Parts were force cooled under pressure to 66°C (150 F).

(3) Load was applied at 0.127 cm (0.05 inch)/minute after stabilizing at 316 C (600 F) for 10 ±5 minutes.

Table 32. Structural Element Weight Losses After Aging at 316 C (600 F) for 125 Hours.

Configuration	Specimen No.	Initial Wt. (grams)	Wt. Loss %
"HAT"	EX195-2A	349.1	1.43
	EX195-3A	348.0	1.39
	EX195-4A	343.8	1.45
"I"	EX194-2A	399.6	1.25
	EX194-3A	398.2	1.36
	EX194-4A	395.1	1.27
Sandwich	EX241-2A	—*	0.75
	EX241-3A	—	0.72
	EX241-4A	—	0.72

*Not comparable—doublers are bonded to panel ends.

Table 33. Structural Element Potting Materials and Processes.

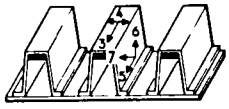
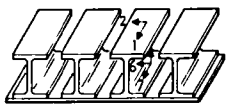
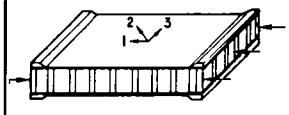
Test Temperature	Potting Material	Process[1]
-168 C (-270 F)	Filled epoxy paste, EA934, Hysol Corporation	1. Mix, pot & cure at R.T, 4 hours. 2. Post cure at 121 C (250 F) for 2 hours.
RT	Filled epoxy paste, EA911-11, Hysol Corporation	1. Mix, pot and cure at R.T., 8 hours minimum
316 C (600 F)	Aluminum filled polyimide resin, BR 34B-18, American Cyanamid Corporation	1. Modify base material by adding 35% - 0.8 mm (1/32 in) fiberglass milled fibers. Mix on paint shaker for 30 minutes minimum. 2. Pot specimen ends with Compound approximately 10.16 mm (0.40 inch) deep. 3. Place in oven and raise temperature R.T. to 177 C (350 F) at < 1.1 C (2 F)/min. 4. Raise temperature 177 C (350 F) to 316 C (600 F) at < 1.7 C (3 F)/min. 5. Post cure at 316 C (600 F) for 2 hours.

(1) Alignment of all specimens during cure was maintained within machined tolerance by clamping to right angle fixtures.

Table 34. Structural Element Target Loads

Specimen Design	Design Ult. Load, KN/cm (lbs/inch) Test Temperature C (F)		
	-168 (-270 F)	RT	316 C (600 F)
"Hat" Stringer	528 (3016)	528 (3016)	319 (1819)
"I" Stringer	542 (3016)	542 (3096)	325 (1858)
Sandwich	805 (4600)	805 (4600)	483 (2760)

Table 35. Results of Compression Tests on "Hat" and "I" Stiffened Skin and Sandwich Panel Structural Elements(1)

Element Configuration	Condition(2)	Element No	Test Temperature C	Test Temperature (F)	Ultimate Load KN	Ultimate Load (LBS)	Remarks
 STRAIN GAGES 1 AND 2 INSTALLED ON LOWER SKIN DIRECTLY OPPOSITE 3 AND 4	①	EX109/EX110A	24	(75)	120 8	(27,150)	Achieved design ultimate, compressive failure of hat caps with transfer thru webs occurred during strain gage readout at 27,150 lbs Skin and bond failures were secondary
		EX109/EX110B	24	(75)	120 8	(27,150)	Achieved design ultimate - no failure Specimen was then fatigue tested 5% to 67% of design ultimate, compression/compression load to 265,000 cycles - no failure
		EX109/EX110B	-168	(-270)	116 8	(26,250)	Skin compression failure - load dropped to 19,500 lbs Retest of EX109/EX110B specimen tested at RT Failed at 97% of design ultimate
		EX195-1 PC	316	(600)	87 84	(19,750)	Skin compression failure, bottom 2 corners followed by buckling through bottom-center Minor bond failure, center stringer under skin buckle 73% of RT design ultimate
	②	EX195-2A	24	(75)	120 8	(27,150)	Achieved design ultimate - no failure
		EX195-4A	-168	(-270)	120 8	(27,150)	Achieved design ultimate - no failure
		EX195-3A	316	(600)	124 3	(27,950)	Skin compression failure inboard of 1 bottom corner followed by diagonal skin buckling toward center of panel Minor debond under center stringer Failed at 103% of RT design ultimate
 STRAIN GAGES 3 AND 4 INSTALLED ON LOWER SKIN OPPOSITE 1 AND 2	①	EX111/EX113	24	(75)	125 4	(28,187)	Achieved design ultimate - no failure
		EX111/EX113	-168	(-270)	125 4	(28,187)	Achieved design ultimate - no failure Retest of EX111/EX113 tested at RT
		EX111/EX113	24	(75)	125 4	(28,200)	Achieved design ultimate - no failure Retest of EX111/EX113 tested at RT and -270 F
		EX194-1 PC	316	(600)	125 4	(28,187)	Achieved design ultimate - minor skin compression failure in 1 corner Did not cause drop in load No debonds
	②	EX194-2A	24	(75)	125 4	(28,187)	Achieved design ultimate - no failure
		EX194-4A	-168	(-270)	125 7	(28,250)	Achieved design ultimate Compression failure of skin starting at 1 upper corner extends inboard 1 inch Two stringer caps and webs also failed in compression with caps splitting axially No debonds
		EX194-3A	316	(600)	125 4	(28,187)	Achieved design ultimate - no failure
 STRAIN GAGES 4, 5, AND 6 INSTALLED ON LOWER SKIN OPPOSITE 1, 2, AND 3	①	EX150-1	24	(75)	125 7	(28,260)	Achieved required 0 53 MN/m (3000 lbs/inch) compression load Skin compression failures occurred along edges of bottom doublers No debonds
		EX241-1PC	-168	(-270)	160 1	(36,000)	Achieved 133% RT design ultimate Compression failure both skins next to doubler
		EX150-2	316	(600)	97 86	(22,000)	Skin compression 1 side only, top corner, 1 12 inch above doubler at edge, extends 3 0 inches inboard Achieved 81% of RT requirement of 0 53 MN/m (3000 lbs/inch) No debonds
	②	EX241-2A	24	(75)	136 3	(30,650)	Achieved 113% RT design ultimate Compression failure both skins next to doubler, also core shear failure after skin failure due to instability
		EX241-3A	-168	(-270)	156 1	(35,100)	Achieved 130% RT design ultimate Compression failure both skins 1 0 inch above doubler and next to doubler
		EX241-4A	316	(600)	120 1	(27,000)	Achieved 100% RT design ultimate Compression failure 1 skin next to doubler and 1 skin 1 0 inch above doubler

Condition 1 Postcured 4 hours at 316 C (600 F), 2 aged 125 hours at 316 C (600 F)

Table 36. Physical Properties of Celion/LARC-160 Composite Laminates Delivered to NASA-LaRC, Tasks (d) and (f)[1]

Properties / Panel No./No. Plies-Orientation	Target Property	CL6C-11/ 6-($\pm$45,90)$_S$	CL12C-6/ 12-($\pm$45,90)$_S$	CL24C-7/ 24-($\pm$45,90)$_S$	CL12C-8/ 12-(0,90)	CL12C-9/ 12-(0,90)
Composite Physical Properties[1]						
1. Specific gravity (grams/cc)	1.561-1.579	1.565		1.587	1.567	1.560
2. Resin weight content (%)	35.0-31.3	34.5		30.3	31.7	35.2
3. Fiber volume (%)	58-62	57.91		62.5	60.5	57.1
4. Void Volume (%)	<2	0.56		0.51	1.32	0.65
5. TMA-Tg C, (F)						
Postcured 4 hrs at 316 C (600 F)	>340 (644)	361 (682)	348 (658)	344 (651)	341 (646)	344 (651)
6. C-Scan ultra sound transmission (%(						
Cured	>95	98	99	97	98	100
Postcured 4 hrs at 316 C (600 F)	>95	98	99	97	98	100

[1] Prepreg physical properties are as follows· Fiber areal weight: 127 grams/m^2; calculated thickness/ply: 0.122 mm (4.80 mils); resin solids content: 37.4%; volatile content: 12.5%

APPENDIX A

This appendix contains three documents which were used during the course of the program to assure prepreg quality. Appendix A1 presents the questionaire which was utilized as a preliminary screening device for potential prepreg suppliers. The flysheet attachment, Appendix A2, was submitted with each purchase order for prepreg materials. Appendix A3 presents test procedures and defines calculations which were imposed by the flysheet requirements.

QUESTIONS

1. Have you ever produced graphite/LARC-160 prepregs?

   What graphite filaments were utilized?

   a. HTS
   b. HTS-II
   c. Celion

3. Is your process proprietary?

4. As a producer of LARC-160 prepregs, how do you obtain the LARC-160 varnish?

   a. Buy from supplier
   b. Produce yourself

5. If you produce the LARC-160 varnish yourself, which method do you use to maintain accurate ratio control of BTDE, AP22 and NE?

   a. By dispensing the initial amounts of BTDS, AP22 and NA in solid form for each batch

   b. By the use of commercial alcohol solutions of BTDE and NE

   c. By the use of alcohol solutions of BTDE and NE which you have produced yourself

   d. Other

6. Do you use methyl or ethyl ester of BTDE and NE for your LARC-160 varnish?

   a. Methyl ester
   b. Ethyl ester

7. If you use the commercial LARC-160 varnish, what solids concentration do you buy?

   a. 99% solids
   b. 85% solids
   c. 66% solids
   d. Other - explain why

8. In the production of your LARC-160 prepregs, do you use a hot melt process or a solvent process?

   a. 99% solids (hot melt)
   b. 85% solids
   c. 66% solids
   d. Others - explain why

9. How do you assure high purity of the LARC-160 varnish for prepregging?

   a. Use high purity ingredients

   b. Store the LARC-160 alcohol solutions at a low temperature such as 40°F and for less than one month

   c. Use the varnish in solids form (99%) only

   d. Other

10. If you buy ingredients for preparation of the LARC-160 varnish, do you obtain certification on the assay of these ingredients from the suppliers?

    a. Methyl and/or ethyl alcohol
    b. BTDA, NA and AP22
    c. BTDE and NE

11. What facilities do you have for producing LARC-160/Graphite Fiber Prepregs?

    a. Hot melt coater
    b. Solvent coating facility
    c. Continuous fabric or tape coating facility

12. What type of LARC-160 prepregs can you produce?

    a. Continuous unidirectional tape up to 12 inches wide

    b. Continuous unidirectional tape up to 24 inches wide

    c. Up to 50 inch wide fabric

    d. Up to 60 inch wide fabric

13. What in-process quality control tests do you conduct on your LARC-160 prepregs?

    a. Resin content
    b. Volatiles
    c. Resin flow
    d. Other - explain why

14. What quality control tests do you conduct on your finished prepreg?

    a. Resin content
    b. Volatiles content
    c. Fiber content
    d. Interlaminar shear test
    e. Compression test
    f. Gel time
    g. Resin flow
    h. Flex and flex modulus
    i. Other - explain why

15. What documentation do you provide on your prepreg materials?

    a. Purchase orders and certification of all basic raw materials

    b. Date of prepration and batch number of the monomer reactants

    c. Specifications for prepregging and test methods

    d. Date of prepregging and batch number of prepreg

16. Can you produce LARC-160 molding compounds?

    a. Chopped roving
    b. Chopped fabric
    c. Other - explain

17. Can you produce LARC-160 adhesives?

CELION/LARC-160 PREPREG REQUIREMENTS

1. Graphite/polyimide prepreg tape, continuous, 6 to 12 inches wide, slit net. Supply to the following target requirements:

   a. Resin type: LARC-160

   b. Fiber type: Celion 6000, NR150-B2G sized

   c. Resin solids (%): $37\ ^{+3}_{-3}$

   d. Volatile content (%): 12 ± 3 at 600°F, 30 minutes

   e. Fiber areal weight (gram/m^2): 153 ± 3

   f. Graphite fiber tensile strength (ksi: 400 min.)

2. Supplier to perform all resin mixing operations. Detailed mixing procedures to be supplied with material shipment.

3. Prepreg materials and required resin samples shall be packed with dry ice for shipment. All containers shall be plainly labeled to indicate dry ice shipping conditions.

4. Supply all available resin, resin precursor, and fiber physical properties certified by Celanese and batch numbers. Supplier will provide duplicate sample quantities of resin precursor materials, intermediate esters, and neat resin. One set of samples will be sent with each prepreg batch. The second set of samples will be sent directly to:

   Science Center
   1049 Camino Dos Rios
   Thousand Oaks, CA 91360

   Attention: Paul J. Dynes, A12

   Samples sent to the Science Center shall be labeled to note contents (e.g. analytical samples) and storage requirements.

   Frequency of sample submission and sample size are as follows:

   a. Precursor materials - 300 gm each of BTDA, NA, and AP22 and 1 liter of Fotocol or equivalent will be submitted with the resin batch. Where the precursors are from a lot used in formulating prior resin batches, these samples will not be required. However, the precursor lot number and prior resin batch number shall be noted.

b. Intermediate esters and neat resin - 10 gm each of the intermediate ester and neat resin will be submitted for each resin batch formulated. These samples will be shipped under temperature controlled conditions commensurate with those used for shipping the prepreg tape representative of the resin batch.

5. Test conditions and calculations for prepreg target requirements are contained in LTR 2433-4462 which is in supplier's possession.

6. Two (2) copies of the following will be furnished to Rockwell for each lot or batch of material processed:

   a. Graphite Yarn Q.C. Summary (Celanese)

   b. Tensile Strength and Young's Modulus of Graphite Fibers (Celanese)

   c. Mix Order and Specifications

   d. Collimated Tape Traceability

7. The prepreg tape batch will not be accepted by Rockwell unless samples of the intermediate ester and neat resin specified in 4(b) have been received by both the Space Systems Group (Downey) and the Science Center prior to prepreg shipment or unless these samples accompany the prepreg shipment to the Space Systems Group and evidence of sample submittal to the Science Center is documented.

   Documentation items noted in 6 will be submitted no later than fifteen (15) days after prepreg shipment.

Space Systems Group Rockwell International

Laboratory Test Report LTR 2433-4462

GRAPHITE/POLYIMIDE PREPREG AND COMPOSITE PHYSICAL PROPERTY TESTING PROCEDURES

March 1978

Authored by

J. S. Jones
Responsible Test Engineer

Approved by

S. Kritzer, Supervisor
Non-Metallic Materials & Processes
Laboratories and Test

J. H. Diaz, Manager
Materials & Processes Laboratories
Laboratories and Test

I PREPREG AND COMPOSITE CONSTITUENT CALCULATIONS * FOR POLYIMIDE RESIN/GRAPHITE FIBER MATERIALS

The following procedures define calculations to be employed in establishing graphite/polyimide prepreg and composite constituents. Example calculations for a typical PMR15 I/HTS II prepreg and composite, 60% fiber volume are given.

1.0 Resin and Fiber Densities

1.1 Establish resin ρ_r = 1.32 grams/cc Vendors Certification

1.2 Establish Fiber ρ_f = 1.65 grams/cc

2.0 Establish Theoretical Density of Composite, 0 Void Assumed

$$\rho_c = (VF_f \times \rho_f) + (Vr_f \times \rho_r)$$

Where: ρ_c = density of composite
VF_f = desired fiber volume fraction
ρ_f = density of fiber, grams/cc
Vr_f = desired resin volume fraction
ρ_r = density of resin, gram/cc

Example: $\rho_c = (.60 \times 1.65) + (.40 \times 1.32)$

$(.990 + .528)$

ρ_c = 1.518 grams/cc

3.0 Establish Resin Solids (%) and Fiber Weight (%) for a Desired 60% Composite Fiber Volume, 0 Void Content

$$R.S.\ (\%) = \frac{(Vr_f \times \rho_r)}{(Vr_f \times \rho_r) + (Vf_f \times \rho_f)} \times 100$$

Where: R.S. (%) = resin solids weight (%)
Vr_f = desired resin volume fraction
Vf_f = desired fiber volume fraction
ρ_r = actual resin density (g/cc)
ρ_f = actual fiber density (g/cc)
FW (%) = fiber weight (%)

Example: $R.S.\ (\%) = \frac{(.40 \times 1.32)}{(.40 \times 1.32) + (.60 \times 1.65)} = \frac{.528}{1.518} = .3478$

$.3478 \times 100 = 34.78\%$

FW (%) = $100 - 34.78 = 65.22\%$

* These procedures have been programmed on a Hewlett Packard computer, Model 9820A, to facilitate rapid data acquisition.

4.0 Establish Fiber Weight Percent of Prepreg

4.1 Test Procedure

1) Prepare nominal 3-inch square specimens of prepreg. For handling convenience the specimen may be cut (before weighing) into several narrow strips. Remove release paper before analyzing.

2) Determine the area of each nominal 3 x 3 inch specimen to an accuracy of 0.01 square inch. Record as "AF"(Use in step 7).

3) Obtain a clean dry extraction thimble and weigh to the nearest 0.1 mg and record as W_1.

   Note: may be purchased from Van Waters & Rogers Company as fritted glass extraction thimbles, medium E.C. 35 x 90 mm, Catalog No. 27743-120.

4) Place specimen in thimble (step 2) and weigh to the nearest 0.1 mg. Record as W_2.

5) Place thimble and specimen in beaker and add enough solvent to cover the extraction thimble, and let set at room temperature with intermittent agitation for 30 minutes. The solvent used shall be selected on the basis of being able to dissolve the resin completely under the conditions of the test. Normally methyl ethyl ketone is suitable.

6) Remove extraction thimbles with specimen from beaker of solvent and drain. Discard used solvent and rinse beaker with fresh solvent.

7) Place extraction thimbles inside beaker and cover with fresh solvent. Repeat steps 5 and 6 until solvent is visually clean and fibers stand apart.

8) Following the last extraction, remove extraction thimble with specimen from beaker of solvent and place in a rubber crucible holder on a vacuum filter flask capable of maintaining a vacuum of at least five inches of mercury. Drain free of solvent and rinse once more with fresh solvent.

9) Dry for 30 minutes at 300 - 320F in a mechanical convection oven or, alternatively, for 15 minutes at 130 - 150F in a vacuum oven.

10) Remove from oven and cool to room temperature in a desiccator.

11) Weigh each extraction thimble and specimen to nearest 0.1 mg.

12) Record test specimen weight as W_3.

4.2 Calculations and Report of Results

Calculate fiber content as follows:

$$\text{Weight \% fiber} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

Where: W_1 = weight of extraction thimble, grams
W_2 = weight of extraction thimble plus specimen, in grams
W_3 = weight of extraction thimble plus test sample after extraction, in grams

Example: $\frac{33.20352 - 32.61985}{33.63455 - 32.61985} = \frac{.58367}{1.01420} = .5752$

$.5752 \times 100 = 57.52\%$

5.0 Establish Total Volatile Weight Percent of Prepreg

5.1 Test Procedure

1) Obtain clean, dry aluminum weighing dish (expendable).

2) Prepare specimens of a size not to exceed four square inches. The sampling plan shall permit assessment of distributional uniformity within the prepreg tape.

Note: release paper must be removed prior to analyzing. Any resin adhering to the release paper will be lost to the test.

3) Weigh each aluminum dish to the nearest 0.1 mg and record as W_2.

4) Place a test specimen in each dish and weigh to the nearest 0.1 mg. Record as W_1.

5) Remove volatiles by heating the dish and the test specimens in a pre-heated air-circulating oven at 600F for 30 minutes.

6) Remove dish and test specimens from oven and cool to room temperature in a desiccator.

7) Weigh each dish and testspecimens to the nearest 0.1 mg and record as W_3.

5.2 Calculations and Report of Results

1) Calculate the volatiles content according to the following equation:

$$\text{Volatiles content, percent by weight} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

Where: W_1 = weight of dish plus test specimen in grams, before volatiles removal.

W_2 = weight of dish in grams

W_3 = weight of dish plus test specimen after volatiles removal

2) Report results in percent by weight to the nearest .01 percent.

Example: $\frac{10.4770 - 10.3461}{10.4770 - 8.9642} = \frac{.1309}{1.5128} = .0865$

$.0865 \times 100 = 8.65\%$

6.0 Establish Prepreg Tape Resin Solids Weight Percent

$$\text{R.S. (\%)} = \left(1.0 - \frac{FW_f}{1.0 - VW_f}\right) \times 100$$

Where: R.S. (%) = resin solids in prepreg
FW_f = fiber weight fraction of prepreg per step 4.0
VW_f = volatile weight fraction of prepreg per step 5.0

Example: $\text{R.S. (\%)} = 1.0 - \frac{.5752}{1.0 - .0865} = \frac{.5752}{.9135} = .6296$

$1.0 - .6296 = .3704$

$.3704 \times 100 = 37.04\%$

7.0 Establish Fiber Areal Density

$$Ad\ (g/m^2) = \frac{Wf}{Af} \times 10.76$$

Where: Ad = fiber areal density (g/m^2)
Wf = total weight of fiber in prepreg sample per 4.0 above (grams)
Af = fiber area per step 4.0 (ft^2)
10.76 = conversion factor, g/ft^2 to g/m^2

Example: $Ad = \frac{0.872}{.0625} = 13.95$ grams/ft^2

conversion to grams/meter^2 = 13.95 x 10.76 = 150.1 gram/m^2

8.0 Establish Predicted Thickness per Ply of Cured Composite, 0 Void Assumed, 60 percent Fiber Volume

$$Tp = \frac{Ad}{Wf_f \times \rho c \times (25.4)}$$

Where: Tp = thickness per ply of composite (mils)
Ad = fiber areal density, grams/meter^2 (per step 7.0)
Wf_f = fiber weight, fraction (per step 3.0)
ρc = theoretical density of composite (per step 2.0)
25.4= conversion factor, inch to m m

Example: $Tp = \frac{150.1}{.6522 \times 1.518 \times (25.4)} = \frac{150.1}{25.147} = 5.97$ mils/ply

9.0 Establish Prepreg Fiber Areal Density Based on Desired Cured Ply Thickness of Composite with a 60% Fiber Volume

$$Ad = Tp\ (Wf_f)\ (\rho c)\ (25.4)$$

Where: Ad = fiber areal density (grams/m^2)
Tp = thickness/ply composite (mils)
Wf_f = desired fiber weight fraction (per step 3.0)
ρc = theoretical density of composite, gram/cc (per step 2.0)
25.4= conversion factor, inch to m m

Example: 5.967 (.6522) (1.518) (25.4) = 150.05 grams/meter^2

10.0 Establish Specific Gravity of Composite

10.1 Apparatus

1) Analytical Balance - a balance with a precision within 0.1 mg, accuracy within 0.05 percent relative (that is, 0.05 percent of the weight of the specimen in air), and equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

2) Wire - a corrosion-resistant wire, Awg No. 36 or finer, for suspending the specimen.

3) Immersion Vessel - a beaker or other wide-mouthed vessel for holding the water and immersed specimen.

4) Thermometer - a thermometer with an accuracy of ±0.1C (±0.18F) is required.

10.2 Materials

1) Water - the water shall be substantially air-free, distilled or demineralized water.

10.3 Test Procedure

1) Cut the required number of test specimens of any convenient size, weighing from 1 to 2 grams each. Edges must be smooth, square, and surfaces clean for accurate density determination.

2) Weigh the specimen in air to the nearest 0.1 mg or 0.05 percent relative, whichever is greater.

3) Attach to the balance a piece of fine wire sufficiently long to reach from the hook above the pan to the support for the immersion vessel. Attach the specimen to the wire such that it is suspended about 1 inch above the vessel support.

4) Mount the immersion vessel on the support, and completely immerse the suspended specimen in water (10.2) at a temperature of 23 ± 2C. The vessel must not touch wire or specimen. Remove any bubbles adhering to the specimen and wire. Usually these bubbles can be removed by rubbing them with another wire. Weigh the suspended specimen to the required precision. Record this weight as b (the weight of the specimen, and the partially immersed wire in liquid). Weigh rapidly in order to minimize absorption of water by the specimen.

5) Weigh the wire in water with immersion to the same depth as used in the previous step. Record this weight as w (weight of the wire in liquid).

10.4 Calculations

1) Calculate the specific gravity of the composite as follows:

$$\text{Sp gr (g/cc)} = \frac{a\,(\rho_w)}{a + w - b}$$

Where: a = apparent weight of specimen, without wire in air
b = apparent weight of specimen, completely immersed and of the wire partially immersed in liquid
w = apparent weight of partially immersed wire.
ρ_w = density of water at water temperature

11.0 Establish Resin Content of a Graphite/Polyimide Composite Using Acid Digestion Techniques

11.1 Test Procedure

1) Use the same composite specimen employed in determining specific gravity per 10.0 above.

2) Obtain a clean dry extraction thimble or clean the thimble in a beaker containing HNO_3 for a minimum of 1 hour at 200 ± 10F. Wash with distilled water, dry in oven at 250 ± 10F, desiccate and cool.

Note: may be purchased from Van Waters & Rogers Co. as fritted glass extraction thimbles, medium E.C. 35 X 90 mm, Catalog No. 27743-120.

3) Weigh each extraction thimble to the nearest 0.1 mg and record as "W_1".

4) Dry specimen used in density determination (step 10.0) and place in clean extraction thimble and weigh to the nearest 0.1 mg. Record as "W_2".

5) Place thimble and specimen in a beaker fitted with a raised platform and a magnetic stirring bar, and add concentrated H_2SO_4 until the specimen is covered. Bring slowly to a boil and hold for 30 minutes.

6) Remove thimble and decant spent H_2SO_4. Replace thimble in same beaker and repeat step 6.

7) Continue boiling until fibers are completely separated and the resin is completely decomposed. This is determined by visual examination and may require additional H_2SO_4.

Note: Complete digestion is indicated when the test specimen changes its appearance from a unitized mass to loose, soft fibers which have a tendency to sink to the bottom of the thimble.

8) After digestion, place on a magnetic stirrer, stir slowly and allow to cool below 300F.

9) While stirring, carefully add 10% H_2O_2 to the hot solution.

Caution: Allow the H_2O_2 to run down the side of the beaker. Add very slowly.

10) Continue adding H_2O_2 until the acid solution turns a transparent clear color. If a clear color is not obtained, the test is invalid and should be repeated.

11) Allow acid to digest three more minutes.

12) Remove extraction thimble from the acid, drain, place in a rubber crucible holder on a vacuum filter flask and wash fibers with distilled water until free of acid.

13) Remove thimble containing fibers and dry in an oven maintained at 300 ± 10F for a minimum of 30 minutes. Cool in a desiccator and weigh to the nearest 0.1 mg. Record weight as "W_3".

11.2 Calculations and Report of Results

Calculate resin content according to the following equation:

$$\text{Resin content weight, \%} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

Where: W_1 = weight of extraction thimble plus test specimen before acid digestion in grams
W_2 = weight of extraction thimble in grams
W_3 = weight of extraction thimble plus specimen after acid digestion in grams.

Example: $\frac{33.61985 - 33.27205}{33.61985 - 32.61985} = .34780$

.3478 x 100 - 34.78%

13.0 Establish Void Volume Percent of Composite

$$V_v = 1 - \rho_c \left(Wf_f/\rho_f\right) + \left(Wr_f/\rho_r\right) \times 100$$

Where: V_v = void volume percent, composite
ρ_c = actual density of composite (gram/cc) determined per step 10.0
ρ_r = density of resin (gram/cc) per step 1.1
Wf_f = fiber weight fraction (1.0 - Wr_f)
ρ_f = density of fiber (gram/cc) per step 1.2
Wr_f = resin solids weight fraction per step 11.0 or 12.0

Example: $V_v = 1.518 \left(\frac{.6522}{1.65}\right) + \left(\frac{.3478}{1.32}\right)$

$(.3953) + (.2635)$

$1.518 \quad (.6587) = 1.00$

$1 - 1.00 = 0.00\%$ void

14.0 Establish Fiber Volume Percent of Composite

$$V_f = \frac{Wf_f}{\rho_f} \times (\rho_c) \times 100$$

Where: V_f = fiber volume percent of composite
Wf_f = fiber weight fraction per step 13.0
ρ_f = density of fiber per step 1.2
ρ_c = density of composite (actual per step 10.0)

Example: $V_f = \left(\frac{.6522}{1.65}\right) \times (1.518) = .6000$

$.6000 \times 100 = 60.00\%$

12.0 Establish Resin Content and Coke Number (char yield) of Cured Polyimide Laminates by Thermogravimetric Analysis (TG).

Test Procedure

1) Prepare a cured sample of polyimide resin. The resin shall be free of graphite fiber by prior extraction if taken from a prepreg sample, with appropriate solvent followed by filtration and then solvent evaporation. TG sample shall be taken from specific gravity sample.
2) Determine the coke number (char yield) by carrying out a TG run to 800C in N_2 on the cured neat resin. The balance and control/readout system shall be operated per the applicable instrument manual. Initial sample weight shall be from 10 to 20 mg.

3) The coke number is calculated as follows:

$$\text{coke number } (Cn) = \frac{W_r}{W_i}$$

Where: W_i = initial sample weight
W_r = weight remaining at 800C
W_v = volatile weight loss to 300C (if present)

4) A sample of cured laminate weighing 20 to 30 mg is then run by TG to 800C in N_2. Calculation of resin content is as follows:

$$\text{Wt \% resin} = \frac{(W_r - W_v)}{(W_i - W_v)(C_n)} \times 10^2$$

Where: W_r = total weight loss
W_v = volatile weight loss to 300C (if present)
W_i = initial weight
C_n = coke number

Note: 1) Twenty to 30 mg initial sample weight is only a guide: Samples of greater weight may be required if graphite content and coke number are high.

2) This method is based on the assumption that graphite present in the laminate loses no weight to 800C in N_2.

APPENDIX B

Two specifications, material (Appendix B1) and process (Appendix B2), were prepared to comply with the requirements of Task (a). The ultrasonic inspection procedure for adhesive bonded assemblies (Appendix B3), in effect since October 1974, was utilized where production facilities were necessary for NDI of sandwich structure.

PREPARED BY	CODE IDENT NO 03953	NUMBER MB0130-152
R. L. Long SL48	Space Division Rockwell International 12214 Lakewood Boulevard Downey California 90241	TYPE Material
APPROVALS	**SPECIFICATION**	DATE 11-13-81
J. M. Smith 11/19/81	WP I.D. 0004S	SUPERSEDES SPEC DATED 6-18-81
W. W. Kerr 11/23/81		REV LTR A / PAGE 1 of 25

TITLE

Total Pages 27

Graphite/Polyimide Resin Prepreg - 600°F Applications

TABLE OF CONTENTS

E.O. M927153

( )Indicates Change

FORM M 131—H—1 REV 5-73

CONTENTS

CONTENTS (Cont'd)

1.0 SCOPE

1.1 Description. This specification establishes the requirements for unidirectional tape and woven graphite fiber fabric impregnated with condensation-reacting polyimide resins suitable for fabrication of structures for use at temperatures up to 316C (600F).

1.2 Classification. The preimpregnated materials shall be of the following classes, types and grades:

1.2.1 The type shall specify the chemical nature of the matrix resin system as follows:

Type I — Nonhalogenated system with diamine to tetra-amine mixtures, identified as LaRC 160

Type II — Nonhalogenated system, identified as PMR-15

1.2.2 The class shall specify graphite fiber strength and modulus properties.

Class 1 - Graphite prepreg made from high strength fibers having a minimum strength of 2.76 GN/m^2 (400 ksi) and modulus 230 GN/m^2 (33 msi).

Class 2 - Graphite prepreg made from high modulus fiber having a minimum strength of 2.20 GN/m^2 (320 ksi) and modulus 345 GN/m^2 (50 msi).

Class 3 - Graphite prepreg made from fiber having a modulus in excess of 483 GN/m^2 (70 msi).

1.2.3 The grade shall specify the graphite form

Grade U — Unidirectional tape

Grade B — Broad Goods, Woven fabric

1.3 Form - The graphite tape preimpregnated material is to be furnished in the required widths on a core which shall not be deformed by the material weight.

2.0 APPLICABLE DOCUMENTS. The latest issues of the following documents form a part of this specification to the extent specified herein. In case of conflict between these documents and this specification, this specification shall prevail.

Test Method	Methods of Testing
FED-STD-406	Plastics: Methods of Testing
MIL-B-117	Bags, Sleeves, and Tubing - Interior Packaging
MIL-G-83410	Graphite Fiber Resin Inpregrated Tape and Sheet, for Hard Layup

0004S

ASTM D 638	Tensile Properties of Plastics
ASTM D 790	Flexural Properties of Plastics
ASTM D 792	Specific Gravity and Density of Plastics by Displacement
ASTM D 2344	Apparent Horizontal Shear Strength of Reinforced Plastics by Short-Beam Shear Method

3.0 REQUIREMENTS

3.1 Workmanship

3.1.1 Uniformity. The material shall be uniform in quality and condition, and clean and free from foreign materials, and shall not have characteristics which are detrimental to fabrication, appearance, or performance. These defects shall be acceptable only to the limits given in 3.4 and 3.5.

3.1.2 Defects. Material containing defects shall be allowed if each defect is flagged, and replacement yardage is added to the roll for every defect occuring in that roll.

3.2 Resin Properties. A one-pint sample shall be taken from each batch of neat resin used to perform the prepreging of the material to this specification and shall be sent with the prepreg order represented. The resin sample shall be identified with cook batch, filming batch and other processing identification as applicable.

3.2.1 Infrared Spectrography. An infrared spectrogram shall be made on a sample of the neat resin from each batch of prepreg per 4.5.2.2 and a copy of this spectrogram shall be transmitted as supporting data to the prepreg certification.

3.2.2 High Pressure Liquid Chromatography. Liquid Chromatographic analysis shall be made on neat resin sample from each batch of prepreg per 4.5.2.3 and a copy of all chromatograms shall be transmitted as supporting data to the prepreg certification.

3.3 Fiber Properties. Fiber properties shall be determined by the fiber manufacturer and the information transmitted to the prepreger by certification for ultimate transmission to Rockwell International as specified herein. A 50 gram sample from each batch of fiber used in the prepreging operation shall be supplied to Rockwell International with the prepreg order represented. Fibers used shall exhibit the properties shown in Table I. The prepreg supplier shall certify in writing that the fibers meet the values of Table I. The actual test values and fiber lots may be obtained from the fiber manufacturer.

0004S

3.3.1 Specific Gravity. The specific gravity of each lot of fiber shall be measured per 4.5.3.1 and shall be transmitted as supporting data to the prepreg certification.

3.3.2 Mechanical Properties. Fiber room temperature tensile strength, tensile modulus and ultimate strain shall be measured per 4.5.3.2.

3.3.3 Finish. Fiber sizing shall be polyimide. Epoxy sizing may be substituted with engineering approval prior to shipment. Fiber certification shall include a statement of sizing material, and quantity expressed as weight addition.

3.3.4 Thermal Oxidation Resistance. Fiber weight loss shall not exceed 1.50 percent when exposed to 600°F in air for 168 hours per 4.5.3.3.

Table 1 Strength and Weight Loss Properties of Graphite Fibers

Properties	Graphite Fiber		
	Class I	Class II	Class III
Modulus, GPa (msi), min.	230 (33)	320 (50)	520 (70)
Tensile Strength, MPa (ksi), min.	2760 (400)	2200 (320)	1860 (270)
Density, g/cc (lb/cu in), ± .04	1.77 (.0639)	1.90 (.0686)	2.07 (.0748)
Weight Loss After 125 Hours at 316C (600°F), % W_1/W_2, max.	1.5	1.5	1.5
Weight Per Unit Length of Tow, Kg/m (lb/in) X 10^{-6}, ± 10%	780 (44)	760 (43)	800 (45)

Definitions

GPa = Gigapascals
msi = Million pounds per square inch
MPa = Megapascals
KSI = Thousand pounds per square inch
g/cc = grams per cubic centimeter
lb/cu in = pounds per cubic inch
% W_1/W_2 = Weight lost over original weight times a hundred gives weight loss in percent
kg/m = kilograms per meter
lb/in = pounds per inch

0004S

3.4 Prepreg Properties. The prepreg material furnished to this specification shall be of quality workmanship. It shall be essentially free from crimped fibers, gelled resin particles, twists, fiber balling, unwetted fibers and dry or boardy areas. Individual tows shall be parallel to the tape or sheet centerline. Indications of impurities, dry areas, areas of nonuniformity, incomplete impregnation, gelled resin, hard spots, or localized color difference in impregnated cloth shall be marked by tape as nonconforming area. The physical properties shall be as shown in Table 2.

Table 2 Physical Properties of the Uncured Prepreg

Property	Requirement	Test Method Paragraph
Volatiles, Weight percent	12±3	4.5.4.3
Resin Solids, Weight percent	38±3	4.5.4.4
Gel Time at 204°C (400°F) Minutes	0.5 to 2.0	4.5.4.5

3.4.1 Fiber Content. The prepreg as specified is intended to produce a cured laminate with a 60 ± 2 percent fiber content by volume per 4.5.4.1.

3.4.2 Fiber Wetting. The filaments shall be completely wetted by the resin; no cured resin particles are permitted when determined visually using magnification as necessary.

3.4.3 Alignment. The filament bundles in the tape shall be parallel to the longitudinal direction of the prepreg within an angle of one degree when examined visually using appropriate aids to measure angular alignment.

3.4.4 Gaps. Any gaps within or between tows in unidirectional tape shall comply with the following when determined visually using adequate scales.

(a) No gap shall exceed 0.010 inch in width.

(b) The length of any gap shall not exceed 4 inches.

(c) Gaps in line with each other and no more than one inch apart shall be considered as one gap, regardless of number.

(d) Gaps with excessive width or length will be considered defective and must be replaced as described in 3.1.2.

3.4.5 Splices. Splices shall be determined visually using magnification as necessary.

0004S

3.4.5.1 Prepreg Splices. Prepreg splices are permitted on any roll of tape where processing is continuous without change in fiber or resin batch. Such splice must be marked by tape as a nonconforming area per 3.1.2.

3.4.5.2 Fiber Splices. Splices of filament bundles shall be kept as short as possible. Adhesive used for bonding filement bundles shall be compatable with the resin system and shall be identified and transmitted as supporting data to the prepreg certification. Where possible, fiber splices shall be flagged during prepreg production.

3.4.6 Width. The prepreg width shall be as specified on the purchase order. Width tolerance for unidirectional tape shall be ± 0.125 inch.

3.4.7 Edges. Maximum acceptable waviness of any 24-inch length of tape shall be 0.030 inch from the edge when measured with appropriate straight edge.

3.4.8 Length. The length of each individual roll of prepreg shall be provided together with sequence in production and batch identification as supporting data to the prepreg certification. The maximum length of prepreg on any single roll shall be as specified in the purchase order.

3.4.9 Areal Weight. Variation from the nominal areal weight specified on the purchase order shall not exceed ± 5 percent.

3.4.10 Storage Life. The prepreg material shall have a storage life of a least nine months when stored in the original package at temperatures of 255°K (0°F) or below. Storage life is defined as the length of time, starting with the date of manufacture, during which the material continues to meet all the requirements of this specification.

3.5 Laminate Properties. The following requirements shall apply when laminates are cured to the procedure specified in 4.5.5.1.

3.5.1 Cured Ply Thickness. The average thickness per ply of the laminate shall be as specified in the purchase order.

3.5.2 Physical Properties. The physical properties of the cured laminate shall conform to the requirements specified in Table 3.

0004S

Table 3. Physical Properties of Type I and Type II Cured Laminates

Property	Requirement
Specific Gravity	1.57 ± .03
Resin Solids Content, Weight percent	31.0 ± 3
Fiber Content, Volume percent	60 ± 2
Glass Transition Temperature, °C (°F) minimum (3 specimens)	316 (600)

3.5.3 Mechanical Properties. The mechanical properties of unidirectional laminates cured per procedures defined in 4.5.5.1 shall meet the requirements in Table 4.

Table 4. Mechanical Properties of Unidirectional Tape Laminates

Property	TEST TEMP. °C(°F)	Graphite Fiber		
		Class I	Class II	Class III
Flexural Strength (ultimate)				
MPa (ksi)	RT	1571 (228)	1515 (220)	1394 (202.2)
Mpa (ksi)	316(600)	942 (126)	757 (110)	900 (130.6)
Flexural Modulus				
GPa (msi)	RT	124 (18)	117 (17.0)	118 (17.2)
GPa (msi)	316(600)	124 (18)	103 (15.0)	108 (15.6)
Short Beam Shear				
MPa (ksi)	RT	103 (15)	96 (14.0)	95 (13.8)
MPa (ksi)	316(600)	48 (7)	41 (6.0)	50 (7.2)

Note: All values are minimum for the average of the specimens tested with no individual value less then 80 percent of the value listed here. Where the number of specimens is not otherwise specified, five replicates shall be fabricated and tested.

0004S

4.0 QUALITY ASSURANCE

4.1 Qualification

4.1.1 Requirements. Material submitted for qualification to this specification shall be tested to all requirements of this specification. In addition, each prepreg manufacturing facility must be qualified individually. The adequacy of the manufacturing facility may be verified, as deemed necessary, by company representatives, by a survey of such facilities. All requests for qualification shall be directed to the company's Material Department which will request data and samples when desired for qualification purposes.

4.1.1.1 Qualification Samples. The qualification samples shall consist of one representative production sample roll (at least 1.5 kg (3.5 lbs.)) of the particular type from each of a minimum of three separate resin mixes. Each type must be qualified individually.

4.1.1.2 Certified Test Report. The qualification sample submitted for approval shall be accompanied by a certified test report in duplicate which shows that the sample meets the prepreg physical, chemical and composite property requirements of this document. The supplier qualification report shall contain the following:

(1) Supplier product designation.

(2) Prepreg type in accordance with this document.

(3) Test results including individual test values

4.1.1.3 Test Facilities. All suppliers shall have test facilities, or access to test facilities required to test, in accordance with this document, including chemical characterization requirements. The adequacy of test facilities may be verified, as deemed necessary, by a survey by company representatives of such facilities.

4.1.1.4 Demonstration Tests. Qualification testing shall consist of a demonstration of the conformance of the sample supplied in accordance with 4.1.1.1 to all requirements of this document.

4.1.2 Changes. If there is any change in formulation of the material originally qualified to this specification, a new manufacturer's designation shall be assigned and the material shall be resubmitted for qualification.

4.2 Acceptance

4.2.1 Definitions. For purposes of sampling, inspection and maintenance of records the following definitions shall apply.

a. Fiber Lot - Fiber that is produced in one single continuous operation and is separately identified by the supplier.

0004S

b. Resin Batch - The quantity of resin compounded in one operation and blended in one mixer and separately identified as such by the supplier.

c. Roll - Any sub-section of a prepreg lot.

d. Prepreg Lot - That quantity of prepreg which is produced as a continuous product an one apparatus from one resin batch and preferrably from one single fiber lot. When it is necessary to utilize more than one fiber lot, certification data shall document related percents of fiber lots used and placement within the product form.

4.2.2 Testing. Acceptance shall be based upon supplier certification that the prepreg will meet specified requirements for the neat resin (ref. 3.2), fiber (ref. 3.3), prepreg (ref. 3.4) and the following tests specified for the cured laminate (ref. 3.5).

Flexural strength at room temperature and 316C (600°F), when tested per ASTM D 790.

Interlaminar shear strength at room temperature and 316C (600°F) when tested per ASTM D 2344.

4.3 Certification. A certified report from the supplier shall accompany each shipment stating conformance to the requirements of this specification. This report shall include this specification number, type and class, purchase order number, batch number, roll number and footage in each roll, manufacturer's designation and date of manufacture.

4.4 Responsibility for Inspection and Testing

4.4.1 Supplier. The supplier is responsible for the performance of all inspection and testing specified herein and may (with the approval of Rockwell International) use his own facilities or those of a commercial laboratory. Rockwell reserves the right to perform or witness any of the inspection and testing set forth in this specification where such are deemed necessary to ensure compliance with specification requirements.

4.4.2 Inspection Records. The supplier's inspection records of examination and tests for conformance to the requirements of this specifiction shall be kept complete and available to Rockwell International upon request.

4.5 Test Methods

4.5.1 Standard Conditions. Unless otherwise specified herein, all room temperature tests shall be conducted at a temperature of 25 to 27°C (75 to 79°F), and relative humidity of 70 percent maximum.

4.5.2.1 Infrared Spectrogram. The infrared spectrographic analysis shall be conducted on a sample of neat resin obtained per 3.2. Extract the resin with C.P. acetone or other appropriate solvent. Deposit the resin on a salt plate such that a film will be formed after solvent evaporation. Obtain the spectra using applicable infrared procedures.

0004S

4.5.2.2 High Pressure Liquid Chromatography. This or an equivalent procedure may be used. From a sample of neat resin obtained per 3.2 prepare the sample solutions and inject into the instrument with the liquid chromatograph containing the following columns and settings.

Test 1. Experimental Conditions for High Pressure Liquid Chromatographic (HPLC) Analysis of 3, 3', 4, 4'- benzophenonetetracarboxylic acid dimethylester (BTDE), 5 - norboreze - 2, 3 - dicarboxylic acid monomethylester (NE) and Reactions Products in Type I and Type II Polymide Resins.

Column: Spectra-Physics Sperisorb ODS

Solvents: Baker HPLC water with 0.01 M dihydrogenorthophosphate (KH_4PO_4) at pH-3 Burdick & Jackson Acetonitrile

Gradient: 10 to 50 percent acetonitrile/water, 15 minutes linear gradient with hold at 50 percent 10 minutes and equilibrate at initial for 10 minutes.

Direction: 200 nanometers (nm). 0.4 aufs

Flow: 1 milliliter (ml)/minute

Sample: 10 micrograms (ul) of 1/5 milligrams per milliliter (mg/ml) of solution

Test 2. Experimental Conditions for Ion-Pair Liquid Chromatographic Analysis of Amine Components in LaRC-160 and PMR-15 Polymide Resins.

Column: Whatman Partisil 10, ODS-2

Solvents: Baker HPLC water with Waters PIC A Ion Pair Reagent
Burdick & Jackson ultraviolet (UV) Grade Tetrahydrofurane (THF)

Gradient: 15 to 50 percent THF in water with PIC A, linear 15 min gradient with 15 min hold at 50 percent THF and equilibrate at initial 15 minutes.

Detector: SP-770, 254 nm, 0.4 aufs

Flow: 1 ml/minute

Sample: 10 ul of 1/5 mg/ml solution in THF

4.5.2.3 Record Retention. Copies of all test data and chromatograms shall be retained for a period of three years and shall be made available to Rockwell upon request.

4.5.3 Fiber The prepreg supplies may use certification properties supplied by the fiber manufacturer. If such certification is not available, the following procedures shall apply.

0004S

4.5.3.1 Specific Gravity

(1) Equipment

(a) Pycnometer, model 930, Beckman or equivalent
(b) Oven, vacuum
(c) Balance, analytical, minimum sensitivity 0.001 gram
(d) Inert gas (helium preferred) bottled with regulator.
(e) Vacuum source

(2) Obtain a representative sample of approximately five (5) grams.

(3) Condition the sample in a vacuum oven operating at full vacuum and 275 ± 5°F for 20 to 30 minutes.

(4) Cool in a desiccator and weigh to the nearest 0.001 gram.

(5) Calibrate the equipment just prior to use.

(6) Adjust gas pressure regulator on the helium tank to a pressure no greater than 2 psi. Turn vacuum supply on.

(7) With purge and coupling valves open, rotate both handwheels in a counter-clockwise direction to the extreme position. Turn the measuring wheel until starting number is reached (located on a plate affixed to the side of the case above the measuring handwheel).

(8) Insert previously conditioned and weighed specimen into the sample cup and insert sample cup into position and secure it firmly.

(9) Open purge and coupling valves.

(10) Open vacuum valve and allow 10 seconds for system to evacuate, then close valve.

(11) Open gas (helium) valve and allow 5 seconds for pressure equilibrium, then close gas valve.

(12) Open vent valve for 5 seconds to allow for pressure equilibrium, then close vent and purge valves.

(13) Wait for 10 seconds then loosen the coupling valve (this should be rotated a couple of times).

(14) Wait for 10 seconds then, turn both handwheels clockwise simultaneously until the reference wheel stops. Apply a minimum amount of pressure after the wheel initially stops. Keep the pointer on the scale during this process.

(15) Wait for 10 seconds and adjust pointer by image alignment to the zero mark with the measuring handwheel.

(16) Open coupling valve. Read specimen volume on counter directly in cubic centimeters.

0004S

(17) Repeat steps 7 through 16 until consistent readings are obtained on each specimen.

(18) Calculations:

$$\text{Density} = \frac{\text{weight of dried specimen, grams}}{\text{volume reading obtained, cubic centimeters}}$$

4.5.3.2 Mechanical Properties of Fiber

Specimen Preparation

Materials Required:

3 pieces - Stainless Steel plates 22 X 1/16-inch (sharp edges rounded)
4 pieces - Stainless Steel plate 22 X 12 X 1/4-inch (to take 150°C) (238°F))
Air circulating oven, maximum temperature 200°C (328°F)
4 - 3-inch Boston No. 4 clamps.
Mochberg bleeder cloth No. CW-1850.

(1) Coat 1 stainless steel plate on one side with

(a) 2 heavy coats of Frekote #33 release spray and

(b) 2 coats of FM-122 release spray.

(2) Coat 2nd and 3rd stainless steel plates on one side only. Same as step 1 (a) and (b).

(3) Dress drum and work table as follows: (Drum is 12-inch long, 9-inch diameter rotating cylinder.)

(a) Cut a 32-inch piece of release paper.

(b) Tape one end of the paper to the drum (30-inch circumference drum used) and wrap around as tightly as possible.

(c) Tape the two ends of release paper together.

(d) Cut a 32-inch piece of bleeder paper.

(e) Tape the bleeder paper to the release paper and wrap around the drum as tightly as possible.

(f) Cut a 40-inch length of release paper and 40-inch length of bleeder paper.

(g) Place the bleeder paper on top of the release paper and tape to the stainless steel table.

(h) Mark off the top of the bleeder paper into 10-inch wide sections.

(i) Cut 2-inch and 1-inch masking tape and fasten loosely to the table's edge.

0004S

(4) Tape a strip of 2-inch masking tape (sticky side up) across the full width of the drum.

(5) Attach one end of the sample to 2 inch masking tape 1/2-inch from the left edge of bleeder cloth.

(6) Rotate drum at desired speed until one wrap of sample is achieved.

(7) Cut sample and attach end to face up masking tape. Identify sample.

(8) Place next sample 1/2-inch to the right of previous sample and repeat steps 5 thru 7.

(9) Once around the drum is generally enough material for 1 (one) strand tensile samples.

(10) Impregnate samples with pre-mixed X506 resin.

(11) Place bleeder cloth over sample and hold ten minutes.

(12) Press very lightly with roller to bleed off excess resin.

(13) Remove the bleeder cloth.

(14) Cut strand at 2-inch masking tape and unwind.

(15) Place each impregnated strand on prepared work table and identify.

(16) Repeat Steps 14 and 15 for each strand.

(17) Place 20-inch sample across 1/16 inch plate.

(18) Repeat step 17 for remaining strands, maintaining approximately 1/2-inch spacing between samples.

(19) Sandwich sample plate with remaining Stainless Steel plates (coated side to the samples). Note: Do not slide plates against each other because sample could be damaged.

(20) Place the four Boston No. 4 clamps equidistant around the perimeter of the sandwiched assembly.

(21) Place the sample assembly into a preheated air circulated oven set @ 150°C (302F). The assembly should be horizontal and supported to permit air circulation on all sides.

(22) Hold in oven for two hours.

(23) Remove sample from oven and allow to cool to room temperature.

(24) Place in freezer for approximately 15 minutes to ease separation.

(25) Carefully break open assembly and cut cured samples to an 18-inch length and identify.

0004S

Specimen Test

Apparatus:

Tension testing Machine - a testing machine having a constant-rate-of-crosshead movement with a stationary and a movable member.

Grips - grips for holding the test specimen between the fixed and movable members should be of the self-aligning type with a pneumatic-hydraulic action capable of applying 3000 pounds of pressure to the jaws.

Jaws: 2 by 1-inch rubber faced jaws are recommended; with edge markings indicating their center.

Specimen: Cured impregnated tows ten inches in length.

Testing Procedure:

(1) Set speed of testing machine at 1.27 millimeters (0.05 in.)/minute.

(2) Set the load scale range as follows:

3K = 200 pounds
6k = 500 pounds

(3) Set the jaws at an effective gage length of 127 millimeters (5 inches) apart.

(4) Adjust recorder speed to lay curve at approximately 45°.

(5) Sandwich one end of the specimen between aluminum oxide cloth (320 grit) and place in upper jaws, aligning it with centering marks on jaws.

(6) Repeat Step 5 for lower jaws.

(7) Apply approximately 2000 pounds of pressure to jaws. Majority of specimens should break within the 5-inch gauge length, if slippage occurs apply more pressure, if specimens continually break at jaws reduce pressure.

(8) Manually remove any positive or negative load applied to specimen by jaws.

(9) Attach 2-inch extensometer to specimen and connect to Instron chart drive so that the abcissa of the stress-strain curve is fractional strain, directly.

(10) Start test.

(11) Test all specimens with extensometer for modulus determination and use breaking load to calculate strength.

0004S

Calculations

Cross-sectional area, square inches = .172 X 10^{-6} X Denier/Density

Denier = original yarn denier of specimen
Density = fiber density grams per cubic centimeter (g/cm^3)

Modulus, psi = (Load)/(Area X strain)

Strength, psi = (Load)/(Area)

Strain, in/in = (Deflection)/(Gauge length)

4.5.3.3 Thermal Oxidation Resistance

(1) Weigh a clean dry 2-inch diameter 1/2-inch high wall aluminum weighing dish. Record the weight as W_1.

(2) Put on polyethylene gloves.

(3) Roll approximately 3 grams of clean dry fiber into a roll so that it fits snugly into the weighing dish. Bend the wall of the dish in slightly to hold the coil of fiber in position.

(4) Weigh the dish and fiber. Record the weight as W_2.

(5) Place the weighing dish containing the fiber in a hot block oven heated to 316C (600 ± 10°F) or equivalent. There should be only a slight air flow.

(6) Leave for 130 ± 10 minutes.

(7) Remove from the oven, cool in a desiccator and weigh. Record weight as W_3.

Note. This should be the start - weight where any combined moisture, sizing or other non fiber contaminates are driven off leaving a pyrolytically clean fiber.

(8) Place the weighing dish with the specimen back in the oven heated to 316°C (600°F) and heat for 168 ± 1 hours.

(9) Remove the weighing dish with the specimen from the oven. Place in a desiccator to cool. Weigh and record the weight as W_4.

0004S

(10) Calculations:

$$\text{Percent Weight Loss} = \frac{(W_3 - W_4) \times 100}{W_3 - W_1}$$

W_1 = weight of weighing dish

W_2 = weight of weighing dish and non-pyrolytically cleaned fiber (includes moisture and sizing)

W_3 = weight of weighing dish and pyrolytically cleaned fiber (moisture and sizing free)

W_4 = weigh of weighing dish and fiber thermally oxidized for 168 hours at 600°F in air.

(11) Test shall be conducted on three specimens with the average and minimum recorded. All specimens shall meet the requirements specified in 3.3.

4.5.4 Prepreg

4.5.4.1 Fiber Content. This or an equivalent procedure may be used.

(1) Prepare nominal 3-inch square specimens of prepreg. For handling convenience the specimen may be cut (before weighing) into several narrow strips. Remove release paper before analyzing.

(2) Determine the area of each nominal 3 X 3-inch specimen to an accuracy of 0.01 square inch. Record as fiber area (A_f). This value will also be used in determining the fiber areal weight.

(3) Obtain a clean dry extraction thimble and weigh to the nearest 0.1 milligram and record as W_1.

NOTE: May be purchased from Van Waters & Rogers Company as fritted glass extraction thimbles, medium E.C. 35 X 90 millimeters, Catalog No. 27743-120.

(4) Place specimen in thimble (step 2) and weigh to the nearest 0.1 milligram. Record as W_2.

(5) Place thimble and specimen in beaker and add enough solvent to cover the extraction thimble, and let set at room temperature with intermittent agitation for 30 minutes. The solvent used shall be selected on the basis of being able to dissolve the resin completely under the condtions of the test. Normally methyl ethyl ketone is suitable.

(6) Remove extraction thimbles with specimen from beaker of solvent and drain. Discard used solvent and rinse beaker with fresh solvent.

(7) Place extraction thimbles inside beaker and cover with fresh solvent. Repeat steps 5 and 6 until solvent is visually clean and fibers stand apart.

0004S

(8) Following the last extraction, remove extraction thimble with specimen from beaker of solvent and place in a rubber crucible holder on a vacuum filter flask capable of maintaining a vacuum of at least five inches of mercury. Drain free of solvent and rinse once more with fresh solvent.

(9) Dry for 30 minutes at 150 to 160C (300 to 320F) in a mechanical convection oven or, alternatively, for 15 minutes at 55 to 65C (130 to 150F) in a vacuum oven.

(10) Remove from oven and cool to room temperature in a desiccator.

(11) Weigh each extraction thimble and specimen to nearest 0.1 milligram.

(12) Record test specimen weight as W_3.

(13) Calculations and Report of Results

Calculate fiber content as follows:

$$\text{Weight percent fiber} = \frac{(W_3 - W_1) \times 100}{W_2 - W_1}$$

Where: W_1 = weight of extraction thimble, grams

W_2 = weight of extraction thimble plus specimen, in grams

W_3 = weight of extraction thimble plus test sample after extraction, in grams

4.5.4.2 Areal Weight

$$A\ (g/M^2) = \frac{W_f \times 1550}{A_f}$$

Where: A = fiber areal weight (grams per square meter)

W_f = total weight in grams (g) of fiber in the prepreg sample, $W_3 - W_1$.

A_f = fiber area in square inches (in^2) from 4.5.4.1(2).

1550 = conversion factor of grams per square inch (g/in^2) to grams per square meter (g/M^2)

4.5.4.3 Volatiles Content

(1) Obtain samples of the prepreg weighing 2.0 to 4.0 grams.

(2) Remove the release paper backing from the specimen. Place in a previously weighed dish and weigh to the nearest milligram.

0004S

(3) Place the sample and dish in an air circulating oven pre-heated to 232°C (450°F) and hold at constant temperature for 30 minutes.

(4) Remove dish from the oven and weigh.

(5) Calculate volatile content as follows:

W_1 = weight of dish

W_2 = weight of dish plus prepreg sample before heating

W_3 = weight of dish plus prepreg sample after heating

$$\text{Volatile Weight percent} = \frac{W_2 - W_1}{W_2 - W_1}$$

4.5.4.4 Prepreg Resin Content - Dry Analysis Method. Calculate dry resin content using data obtained in the preceeding paragraphs.

Percent Resin Content, Wet = 100 - weight percent Fiber - weight percent Volatiles

$$\text{Percent Resin Content, Dry} = \frac{\text{weight percent Wet Resin}}{\text{weight percent fiber + weight percent Wet Resin}} \times 100$$

4.5.4.5 Gel Time

Apparatus:

(1) Fisher-Johns melting point apparatus or equivalent to read ±1°C (1.8°F) of the specified temperature.

(2) Thickness No. 2 cover glasses (18 millimeters) or equivalent.

(3) Timer

(4) Wooden picks

Procedure:

(1) Preset the melting point apparatus to 204°C (400°F).

(2) Insert a 1/4 inch by 1/4 sample between two cover glasses and place in the melting point apparatus.

(3) Start the timer and probe the specimen with a wooden pick.

(4) Resin gel is evidenced when no resin movement is seen when moderate pressure is applied to the specimen. At this point stop the timer and report the gel time to the nearest 0.1 minute.

0004S

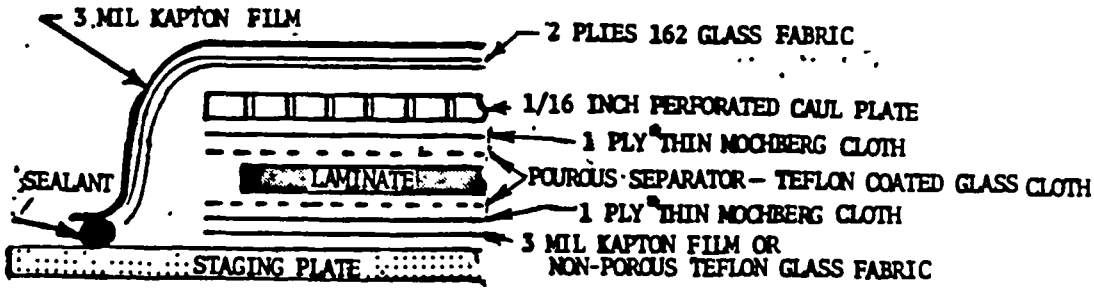
4.5.5 Cured Laminate

4.5.5.1 Preparation of Cured Laminate. Lay up the laminates necessary to produce the specimens required by the tests in this specification. After assembly, checkout the bag and seal system to be used to cure these specimens by applying full vacuum. Correct any degradation. Release the vacuum and commence the appropriate process as described below:

Staging Procedure

Types I & II Material

(1) Make lay-up for staging as shown in Figure 1.
(2) Install lay-up assembly in air circulating oven.
(3) Apply 2 to 15 inches of mercury vacuum and raise the temperature at (1-3°C) (2 to 5°F) per minute to 218 ± 3°C (425 ± 5°F).
(4) Hold at temperature for 30 minutes minimum.
(5) Reduce the temperature to 65°C (150°F) or lower before releasing vacuum or removing from oven.
(6) Remove laminate from lay-up and inspect



* NOTE- THE AMOUNT OF MOCHBERG CLOTH AND VACUUM APPLIED DURING STAGING SHALL BE ADJUSTED AS REQUIRED TO ACHIEVE A CURED LAMINATE FIBER VOLUME OF 60% BASED UPON CERTIFIED PREPREG VOLATILE AND RESIN CONTENT.

FIGURE 1. Layup Assembly for Staging Type I & II Materials

0004S

Curing Procedure

Types I & II Material

(1) Assemble layup for curing as shown in Figure 2.
(2) Install in autoclave and attach vacuum and instrumentation lines.
(3) Apply full vacuum of 635 mm (25in) Hg minimum to layup and increase autoclave pressure to 1378 + 65-0 Kilopascals (KPa) (200 pounds per square inch (psi)). Shut off vacuum, and verify leak rate less than 25 millimeter (mm) (1 inch) mercury per minute. Correct if necessary.
(4) Reapply full vacuum and hold autoclave pressure at 1378 KPa (200 psi).
(5) Increase temperature to 246 ± 3°C (475 ± 5°F) at a rate of 3-4°C (5-7°F) per minute as shown in Figure 3.
(6) Hold at 245°C (475°F) for 30 minutes.
(7) Increase temperature to 329 ± 3°C (625 ± 5°F) at rate of 3-4°C (5-7°F) per minute.
(8) Hold at temperature for 3 hours.
(9) Reduce temperature to 65°C (150°F) or lower before releasing pressure, vacuum or removing lay-up from the autoclave.

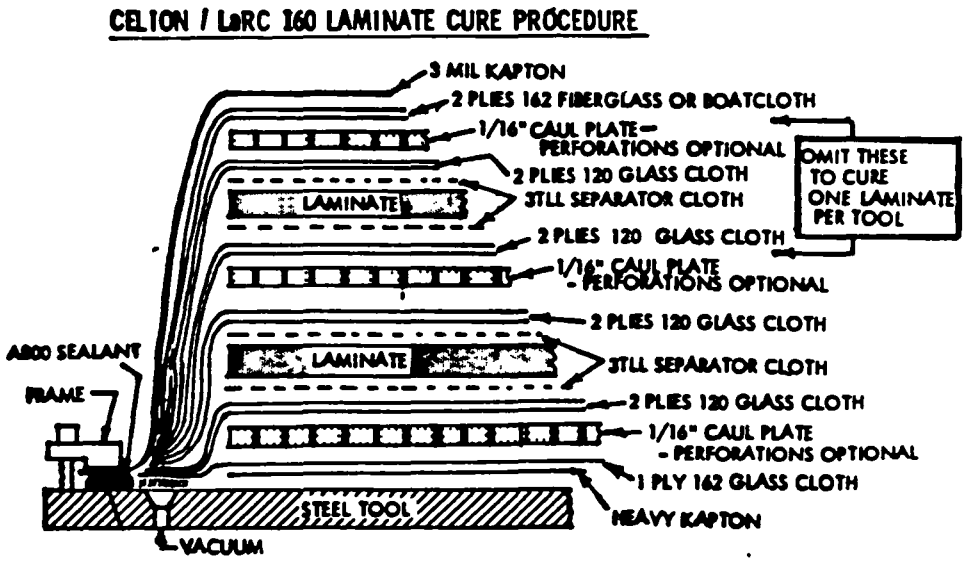


FIGURE 2. Layup Assembly for Curing Types I and II Materials

0004S

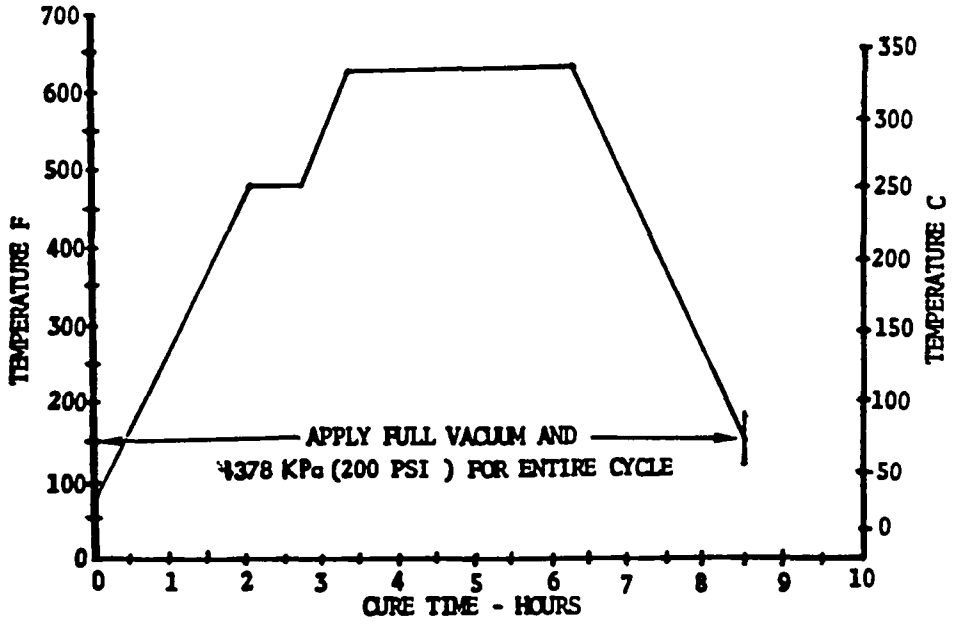


FIGURE 3. Cure Cycle for Curing Types I and II Materials

0004S

Postcuring Procedure

Types I & II Material

These materials normally exhibit transition temperatures (Tg) in excess of 316°C (600F) after curing per preceding procedure. If it is found necessary to postcure laminates, the following procedure shall be utilized.

(1) Install laminate in air circulating oven in free standing condition.
(2) Increase temperature to 316 ± 9°C (600 ± 5°F)
(3) Hold at 316 ± 9°C (600 ± 5°F) for 16 hours
(4) Reduce temperature to 65°C (150°F) or lower before removing from oven.

4.5.5.2 Cured Ply Thickness. The thickness of the cured laminate shall be measured to the nearest 0.025 millimeter (0.001 inch) in at least five representative locations using a doubleball 3-millimeter (1/8 R) micrometer or ultrasonic gage. The thickness per ply shall be computed by averaging the five readings and dividing by the number of plies.

4.5.5.3 Specific Gravity. The specific gravity shall be determined in accordance with Federal Test Method No. 406, Method 5011.

4.5.5.4 Fiber Content. Either the acid digestion or the hydrazine digestion method shall be used.

Caution: Appropriate safety precautions must be observed using either method.

Acid Digestion

(1) The composite specimens used for specific gravity determinations per 4.5.5.3 may be used.
(2) Obtain a clean dry extraction thimble or clean the thimble in a beaker containing nitric acid for a minimum of one hour at 149 ± 5.5°C (300 ± 10F). Wash with distilled water, dry in oven at 121 ± 55°C (250 ± 10F), desiccate and cool.
Note: May be purchased from Van Waters & Rogers Co. as fritted glass extraction thimbles, medium E.C. 35 X 90 millimeters, Catalog No. 27743-120.
(3) Weigh each extraction thimble to the nearest 0.1 milligram and record as "W_1".
(4) Dry the specimens used in the specific gravity determination and place each of three in a clean extraction thimble and weigh to the nearest 0.1 milligram. Record as W_2.
(5) Place thimble and specimen in a beaker fitted with a raised platform and a magnetic stirring bar, and add concentrated sulfuric acid until the specimen is covered. Bring slowly to a boil and hold for 30 minutes.
(6) Remove thimble and decant spent sulfuric acid. Replace thimble in same beaker and repeat Step 5.

0004S

(7) Continue boiling until fibers are completely separated and the resin is completely decomposed. This is determined by visual examination and may require additional sulfuric acid.
Note: Complete digestion is indicated when the test specimen changes its appearance from a unitized mass to loose, soft fibers which have a tendency to sink to the bottom of the thimble.

(8) After digestion, place on a magnetic stirrer, stir slowly and allow to cool below 300F.

(9) While stirring, carefully add 10 percent by volume hydrogen peroxide to the hot solution.
Caution: Allow the hydrogen peroxide to run down the side of the beaker. Add very slowly.

(10) Continue adding hydrogen peroxide until the acid solution turns a transparent clear color. If a clear color is not obtained, the test is invalid and should be repeated.

(11) Allow acid to digest three more minutes.

(12) Remove extraction thimble from the acid, drain, place in a rubber crucible holder on a vacuum filter flask and wash fibers with distilled water until free of acid as shown by neutral indication of pH paper.

(13) Remove thimble containing fibers and dry in an oven maintained at 300 ± 10F for a minimum of 30 minutes. Cool in a desiccator and weigh to the nearest 0.1 milligram. Record weight as "W_3".

(14) Calculate fiber content according to the following equation:

$$\text{Fiber content, weight percent} = \frac{W_3 - W_2}{W_1 - W_2} \times 100$$

Where: W_1 = weight of extraction thimble plus test specimen before acid digestion in grams

W_2 = weight of extraction thimble in grams

W_3 = weight of extraction thimble plus specimen after acid digestion in grams.

Hydrazine Digestion

(1) A cured laminate with a nominal size of 2.5 X 1.75 centimeters (one by one-half inch) and a nominal weight of .5 to .8 gram shall be weighed to the nearest 0.1 milligram. Record weight as W_1.

(2) Place the sample in a 400 millilliters wide mouth Erlenmeyer flask & add approximately 40 millilliters of reagent grade anhydrous hydrazine or hydrazine hydrate.
Caution: All operations involving hydrazine shall be performed in a fume hood with an airflow adequate to prevent any fumes escaping. The operator shall wear adequate rubber gloves to prevent any contact of the hydrazine to the skin. The hood shall be configured to preclude dropping of rust, dust, condensed liquids or other contaminant into the flask or on the work area.

0004S

(3) Heat until the material is completely degraded as indicated by the separation of fibers free of any polyimide resin. Do not allow the mixture to go to dryness.
(4) Obtain a clean dry coarse grade fritted glass filter and weigh to the nearest 0.1 milligram. Record weight as W_2.
(5) Filter the digested material through the weighed, coarse grade, fritted glass filter.
(6) Rinse the fibers with ethanol until the washings are colorless.
(7) Oven dry at 90 to 100°C (194 to 212°F).
(8) Cool in a desiccator and weigh the filter and contents to the nearest 0.1 milligram. Record weight as W_3.
(9) Calculate fiber content according to Step 14 of acid method.

4.5.5.5 Fiber Content, Volume Percent. Calculate the fiber volume according to the following formula:

$$\text{Fiber volume, percent} = \frac{W^{f/f}}{\Sigma f} \times \Sigma c \times 100$$

Where: $W^{f/f}$ = fiber weight fraction per 4.5.5.4.

Σf = density of fiber per 4.5.3.1.

Σc = density of composite per 4.5.5.3.

4.5.5.6 Glass Transition Temperature. The glass transition temperature of the cured composite shall be determined using a DuPont 941 TMA model/900 Thermal Analyzer or equivalent. The heating rate shall be 5 + 0.5°C per minute. Measurements will be made on a 100 mil diameter expansion probe under a 5-gram load. Tests shall be conducted on laminates that have been cured per 4.5.5.1.

4.5.5.7 Short-Beam Shear. The interlaminar shear strength shall be determined per MIL-G-83410, with a minimum half-hour soak at each test temperature prior to loading.

4.5.5.8 Longitudinal Flexural Test. The flexural strength and modulus shall be determined per MIL-G-83410, with a minimum half-hour soak at each test temperature prior to loading.

5.0 PREPARATION FOR DELIVERY

5.1 Packaging. Prepreg material shall be rolled on a reel not less than 8 inches in diameter. A nonadherent paper or Mylar separator of a contrasting color shall be used on one side of the material to prevent the layers of material from sticking to each other. Each roll or rolls of prepreg shall be heat-sealed in an evacuated, moisture-proof plastic bag.

5.2 Identification. Each roll of prepreg shall be permanently marked with the following data:

Material: Graphite/Polyimide Resin Prepreg - 600°F Applications

Rockwell International Specification: MB0130-152 Type:_____Class _____

Manufacturer's Name and Product Identification: _____

Batch No.: _____Date of Manufacture: _____

Roll No.: _____Linear Feet: Gross: ____,Net Adjusted:____

Storage Temperature, Max. 0°F Shelf Life: 9 months at 0°F

5.3 Packing. Units packaged as specified in 5.1 shall be packed in exterior type shipping containers in a manner that will allow solid carbon dioxide to be packed in sufficient quantities to maintain a material temperature of 0°F maximum if refigeration is required during transit. Shipping containers shall comply with carrier regulations applicable to the mode of transportation and shall be so constructed as to assure safe delivery and acceptance at the destination.

5.4 Marking of Shipment. Each shipping container shall be marked with the following information:

Material: Graphite/Polyimide Resin Prepreg - 600°F Applications

Purchase Order No.: _____

Manufactur's Name and Product Identification _____

Storage Temperature, Max. 0°F Shelf Life: 9 months at 0°F

STORE AT 0°F

6.0 NOTE

6.1 Intended Use. Material procured in accordance with this specification, when molded using indicated laminating methods, is suitable for use in airframe, aerospace, and similarly related primary structural components where high stiffness and strength-to-weight ratios are required.

0004S

APPENDIX B2

PREPARED BY	CODE IDENT NO 03953	NUMBER
R. L. Long	Space Division Rockwell International 12214 Lakewood Boulevard Downey California 90241	MA0105-328
APPROVALS		TYPE Process
		DATE 1-13-82
		SUPERSEDES SPEC DATED 6-12-81
		REV LTR A — PAGE 1 of 11

TITLE

Total Pages 12

FABRICATION OF LaRC 160 POLYIMIDE/GRAPHITE COMPOSITES

E.O.

FORM M 131—H—1 REV 5-73

PARAGRAPH TABLE OF CONTENTS PAGE

0005S-1

1.0 SCOPE

1.1 Description. This specification establishes the materials to be used and the procedures to be followed for fabricating composites from LaRC 160 and PMR-15 polyimide resin systems reinforced with graphite fibers. This specification is intended for use in fabricating parts capable of structural application in a 316C (600F) environment.

2.0 APPLICABLE DOCUMENT AND MATERIALS

The latest issue of the following documents form a part of this specification to the extent specified herein. In case of conflict between these documents and this specification, this specification shall prevail.

2.1 Documents

FED-STD-406	Plastics: Methods of Testing
MA0110-306	Environmentally Controlled Areas
MT0302-001	Test Method Standards for Advanced Composite Materials
MT0501-510	Inspection, Ultrasonic
MT0302-502	Process Control for Fabrication of Reinforced Plastic Parts

2.2 Materials

MB0130-152,	Graphite/Polyimide Resin Prepreg. - 600°F Applications

3.0 REQUIREMENTS

3.1 Safety Requirements

3.1.1 Supervision shall inform all personnel working to this specification of the hazards involved and the necessary precautions required when handling the chemicals used in the procedures of this specification.

3.1.2 Skin contact with the liquid chemicals and solutions required by this specification shall be prevented by wearing protective equipment (gloves, aprons, etc.).

3.1.3 Face shields or safety goggles shall be worn during the handling of solvents and liquid chemicals.

3.1.4 Solvents and chemical solutions required by this specification shall be handled only in areas approved by Industrial Safety. Operating department supervision, with Facilities and Industrial Engineering, shall ensure that local exhaust ventilation and/or general air circulation is adequate to prevent employee exposure to vapors or mists in concentrations above the Threshold Limit Values. (Threshold Limit Values can be obtained from Industrial Safety.)

3.1.5 Information or assistance on any aspects of safety associated with this specification shall be obtained from Industrial Safety.

3.2 Material Storage and Handling. Adhesive and preimpregnated material shall be stored in accordance with the storage-life requirements of the applicable material specifications and monitored per MT0302-001.

3.2.1 Materials that have exceeded the storage life shall be withheld from use pending retest to the acceptance requirements of the applicable specifications. Acceptable materials shall be returned to storage.

3.2.2 When not in use, the material shall be stored in a heat-sealed, moisture proof plastic bag. Allow refrigerated material to warm to at least 16°C (60°F), but less than 32°C (90°F), prior to opening container.

3.2.3 The total cumulative time that MB0130-152 prepreg material may be between zero and 24°C (75°F) during storage before the staging operation is started shall not exceed 168 hours.

3.3 Layup

3.3.1 Layup shall be conducted in an area classified per MA0110-306 as GHA, Condition C, Level III. The air supplied to this area shall be filtered through an industrial grade (or better) air filter.

3.3.2 The part layup shall consist of the required number and orientation of plies of prepreg as shown on the Engineering Drawing. Material with different manufacturer's prepreg designations shall not be intermixed in the same part unless so directed in engineering documentation.

3.3.3 Each ply shall be continuous in the length direction; orientation of the prepreg shall not deviate more than two degrees from the specified direction.

3.3.4 Gaps between laterally adjacent tapes shall be no wider than 0.030 inch; tape edges in adjacent plies of the same orientation shall be staggered at least 1.0 inch.

3.3.5 Layups containing details to be co-cured into the part shall have the details prepared for processing in accordance with Engineering Drawing.

3.3.6 Heat may be applied locally to assist this operation using a hand held pressing iron or heat gun with temperatures up to 93C (200F).

3.3.7 Debulking under pressure without application of heat is permitted. If heat is used during debulking, any process control panel that is required by engineering drawings shall be debulked concurrently with the part using a proportionate number of plies and bleeders at each step. No more than two heat debulking cycles performed at 93 ± 6°C (200 ± 10°F) for 20 ± 5 minutes, shall be applied prior to the final curing cycle.

0005S-3

3.4 Assembly. Lay up the preimpregnated composite material to the configuration shown on the Engineering Drawing.

3.5 Laminate Cure Procedure

(1) Assemble layup for curing as shown in Figure 1.
(2) Install in autoclave and attach vacuum and instrumentation lines.
(3) Apply miminum vacuum of 68.5 centimeters (27 inches) of mercury and autoclave pressure of 1378KPa (200 Psi).Shut off vacuum and verify leak rate less than 2.5 centimeters (1 inch) of mercury per minute. Correct any leaks.
(4) Apply vacuum in the range of 2 to 15 inches of mercury. The actual level to be determined from prepreg evaluation tests conducted as part of Quality Assurance receiving inspection.
(5) Raise the air temperature at 3-4°C (5 to 7°F) per minute to 218 ± 3°C (425±5°F) and hold at that temperature for 60±2 minutes
Note: Air temperature and dwell time may need to be increased if heavy tooling is used. Actual conditions shall be predetermined to achieve a minimum laminate temperature of 210C (410°F).
(6) Reapply full vacuum and maintain autoclave pressure at 1378KPa (200 psig).
(7) Increase temperature to 329 ± 3°C (625 ± 5°F) at a rate of 3-4°C (5-7°F) per minute.
(8) Hold at temperature for 3 hours.
(9) Reduce the temperature to 65°C (150°F) or lower before releasing vacuum or removing from oven.

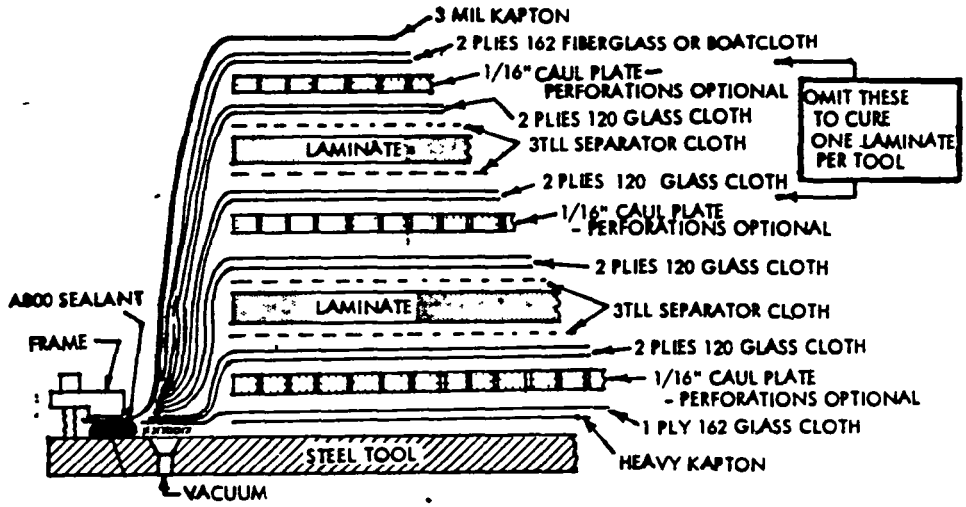


FIGURE 1. Layup Assembly for Staging Laminates

0005S-4

3.6 Postcure Procedure. These materials normally exhibit glass transition temperature (Tg) in excess of 316°C (600F) after curing per preceding procedure. If it is found necessary to postcure laminates, the following procedure shall be utilized.

(1) Install laminate in air circulating oven in free standing condition.
(2) Increase temperature at a rate of 4-5°C (8-10°F) per minute to 316 ± 3°C (600 ± 5°F)
(3) Hold at 316 ± 3°C (600 ± 5°F) for 16 hours.
(4) Reduce temperature to 65°C (150°F) or lower before removing from oven.

3.7 Structure Requirements. Cured composites shall conform to the requirements in Table I, and the dimensional requirements of the applicble Engineering Drawings, and shall meet the requirements in 3.7.1 through 3.7.6.

3.7.1 Fractures. A fracture is defined as a visible break in the fiber reinforcement. There shall be no defects of this nature.

3.7.2 Blisters and Unbonded Areas. A blister is defined as a local increase in thickness usually caused by the formation of trapped gas generating pockets between plies during cure. These gas pockets may not increase the thickness locally but may appear as unbonded areas rather than as blisters. There shall be no defects of this nature.

3.7.3 Delaminations. A delamination is defined as an area where plies at the trimmed edge of the laminate have become separated for any reason, i.e., machining with dull tools such as drilling, sawing, etc. There shall be no defects of this nature.

3.7.4 Bridged Plies. Bridged plies are defined as areas in which the plies have insufficient or no contact with the inside radius of the mold surface or with the preceding plies. There shall be no defects of this type.

3.7.5 Wrinkles. A wrinkle is defined as a raised fold in one or more layers of the laminate. Defects of this nature are not allowed unless identified on the Engineering Drawing.

3.7.6 Foreign Objects. There shall be no inclusions of materials not specifically called for in the applicable material specifications, process specifications, or Engineering Drawings.

3.8 Ultrasonic Inspection. An autographic recording shall be made of the ultrasonic inspection of all cured parts and shall be kept as a permanent record. This inspection shall be conducted per 4.3.1.

3.9 Specific Gravity. The specific gravity of the laminates shall be between 1.56 and 1.58 when determined per 4.3.2.

3.10 Glass Transition Temperature. The glass transition temperature of the cured assembly shall exceed 316C (600°F) when measured per 4.3.3.

3.11 Fiber Content. The fiber content shall be between 58 and 62 volume percent when determined per 4.3.5.

3.12 Void Content. The void content shall be no greater than 2 volume percent when determined per 4.3.6.

3.13 Mechanical Properties. The mechanical properties shall meet the requirements of Table 1.

Table I. Mechanical Properties

PROPERTY	TEST TEMPERATURE C (F)	TEST VALUE	TEST METHOD PARAGRAPH
Flexural Strength, F^{fu}	21 (70)	1515MPa (220 KSI)	4.3.7
F^{X}	316 (600)	757MPa (110 KSI)	
Flexural Modulus, E^{f}	21 (70)	117GPa (17 MSI)	
E^{X}	316 (600)	103GPa (15 MSI)	
Short-Beam Shear, F^{ilsu}	21 (70)	96MPa (14 KSI)	4.3.8
F^{X}	316 (600)	41MPa (6 KSI)	

NOTES:

(1) All values shown are minimum requirements for the average of specimens tested. No individual value shall be less than 80 percent of the minimum average requirement. Where the number of specimens is not otherwise specified, five (5) replicates shall be fabricated and tested.

(2) Elevated temperature tests shall be preceded by a 30 ±2 minute soak at test temperature.

4.0 QUALITY ASSURANCE

4.1 Process Qualification.

4.1.1 The fabrication process shall be qualified for each combination of materials used prior to fabrication of production parts. Qualification shall consist of documented evidence the process is capable of producing a part that will meet all the requirements of this specification. Any change in procedure or materials will require requalification.

4.2 Product Acceptance.

4.2.1 Product acceptance shall be based upon evidence of compliance with the requirements of this specification.

4.2.2 When required by engineering drawing, a minimum of four specimens shall be tested for each of the following properties to the requirements in Table I.

Room Temperature Test

Flexural strength
Flexural modulus
Short-beam shear

After One-Half Hour at 316C (600F)

Flexural strength
Flexural modulus

4.2.3 The specific gravity shall be determined from the trim of the part where trim is available or from the process control coupon, and these values shall meet the requirements of 3.9.

4.2.4 When a question arises as to an excessive variation of resin content on the part, resin content determinations on the part trim or a process control coupon shall be made at the request of Quality Control and shall meet the requirements of 3.11, 3.12, and 3.13.

NOTE: A process control coupon is a laminate fabricated with the same materials subjected to the same cure cycle as the part represented. The coupon shall have a size sufficient to supply all the specimens required by this specification.

4.3 Test Methods.

4.3.1 Ultransonic Inspection. The cured laminates shall be ultrasonically inspected per MT0501-510.

4.3.2 Specific Gravity. The specific gravity shall be determined in accordance with Federal Test Method No. 406, Method 5011.

4.3.3 Glass Transition Temperature. The glass transition temperature of the cured composite shall be determined using a DuPont 941 TMA mode/900 Thermal Analyzer, or equivalent. The heating rate shall be 5 ± 0.5°C (9 ± .9°F) per minute. Measurements will be made on a 100-mil-diameter expansion probe under a 5-gram load.

4.3.4 Fiber Content. The fiber content of the cured laminate shall be determined by either the acid digestion or hydrazine digestion method as follows·

Acid Digestion

CAUTION: All operations involving acid digestion shall be performed in a fume hood having adequate airflow and other safeguards to prevent fumes from escaping. The operator shall wear adequate protective clothing to prevent contact between the acid or other reactants with the skin.

1. The composite specimens used for specific gravity determinations described in 4.3.2 shall be used.

2. Obtain a clean, dry extraction thimble or clean the thimble in a beaker containing nitric acid for a minimum of one hour at 149 ± 6°C (300 ± 10°F). Wash with distilled water, dry in oven at 121 ± 6°C (250 ± 10°F), desiccate and cool.

   NOTE: May be purchased from Van Waters & Rogers co. as fritted glass extraction thimbles, medium E.C. 35X90 millimeters.

3. Weigh each extraction thimble to the nearest 0.1 milligram and record as "W_1"

4. Dry the specimens used in the specific gravity determination and place each of three in a clean extraction thimble and weigh to the nearest 0.1 milligram. Record as "W_2"

5. Place thimble and specimen in a beaker fitted with a raised platform and magnetic stirring bar, and add concentrated sulfuric acid until the specimen is covered. Bring slowly to a boil and hold for 30 minutes.

6. Remove thimble and decant spent sulfuric acid. Replace thimble in same beaker and repeat Step 5.

7. Continue boiling until fibers are completely separated and the resin is completely decomposed. This is determined by visual examination and may require additional sulfuric acid.

   NOTE: Complete digestion is indicated when the test specimen changes its appearance from a united mass to loose, soft fibers which have a tendency to sink to the bottom of the thimble.

8. After digestion, place on a magnetic stirrer, stir slowly and allow to cool below 149°C (300°F).

9. While stirring, carefully add 10 percent hydrogen peroxide to the hot solution.

   CAUTION: Allow hydrogen peroxide to run down the side of the beaker. Add very slowly and incrementally.

10. Continue adding hydrogen peroxide until the acid solution turns a transparent, clear color. If a clear color is not obtained, the test is invalid and should be repeated.

11. Allow acid to digest three more minutes.

12. Remove extraction thimble from the acid, drain, place in a rubber crucible holder on a vacuum filter flask, and wash fibers with distilled water until free of acid as indicated by neutrality indication of pH paper. Denatured alcohol may be used to expedite drying after acid removed.

13. Remove thimble containing fibers and dry in an oven maintained at 149 ± 6°C (300 ± 10°F) for a minimum of 30 minutes. Cool in a desiccator and weigh to the nearest 0.1 milligram. Record weight as "W_3".

14. Calculate the fiber content according to the following equation:

$$\text{Fiber content, weight percent} = \frac{W_3 - W_2}{W_1 - W_2} \times 100$$

Where W_1 = weight of extraction thimble plus test specimen before acid digestion in grams

W_2 = weight of extraction thimble in grams

W_3 = weight of extraction thimble plus specimen after acid digestion in grams

Hydrazine Digestion

CAUTION: All operations involving hydrazine shall be performed in a fume hood with an airflow edequate to prevent any fumes escaping. It shall be configured to prevent dropping of any solid such as rust or dust or dripping of any condensed liquid contaminate onto the work area. The work area shall be free of all debris and dust. The operator shall wear adequate rubber gloves and other protective clothing to prevent any contact of the hydrazine to the skin.

1. A cured laminate with a nominal size of 1 by 1/2 inch and a nominal weight of 0.5 to 0.8 gram shall be weighed to the nearest 0.1 milligram. Record weight as "W_1".

2. Place the sample in a 400-milliliters wide mouth Erlenmyer flask and add approximately 50 milliliters of reagent grade anhydrous hydrazine or hydrazine hydrate.

3. Heat until the material is completely degraded as indicated by the separation of fibers free of any polyimide resin. Do not allow the mixture to go to dryness.

4. Obtain a clean, dry coarse grade fritted glass filter and weigh to the nearest 0.1 milligram. Record weight as "W_2".

5. Filter the digested material through the weighed, coarse-grade, fritted glass filter.

6. Rinse the fibers with ethanol until the washings are colorless.

7. Oven dry at 90 to 100°C (194 to 212°F).

8. Cool in a desiccator and weigh the filter and contents to the nearest 0.1 milligram. Record weight as "W_3.

9. Calculate fiber content as shown in Step 14 of Acid Method:

4.3.5 Fiber Content, Volume Percent. Calculate the fiber volume according to the following formula:

$$\text{Fiber volume, percent } (V\%) = \frac{(W\%)_f}{\rho_f} x_c\rho \times 100$$

Where $(W_f)_f$ = fiber weight fraction per 4.3.4

ρ_f = specific gravity of fiber per vendor certification on the batch of material used

ρ_c = specific gravity of composite per 4.3.2

4.3.6 Void Content. Calculate the void volume percent according to the following formula:

$$\text{Percent Void Volume} = \frac{100}{\rho_c} - \left[\frac{(WT\%)_r}{\rho_r} + \frac{(Wt\%)_f}{\rho_f} \right]$$

Where: $(Wt\%)_r$ = resin weight percent per 4.3.4

$(W_f)_f$ = fiber weight fraction per 4.3.4

ρ_c = specific gravity of composite per 4.3.2

ρ_r = specific gravity of resin per vendor certification on the batch of material used

ρ_f = specific gravity of fiber per vendor certification on the batch of material used

4.3.7 Flexure Test. The flexure strength and modulus shall be determined in accordance with MT0302-001 with a minimum half-hour soak at each test temperature prior to loading.

4.3.8 Short-Beam Shear. The interlaminar shear strength of the laminate shall be determined in accordance with MT0302-001, with a minimum half-hour soak at each test temperature prior to loading.

4.4 Records. Records of the entire fabrication process and the control thereof shall be maintained for the purpose of demonstrating process control.

5.0 PREPARATION FOR DELIVERY

This section is not applicable to this specification.

6.0 NOTES

This section is not applicable to this specification.

00055-11

PREPARED BY		MPP NO	
C. C. Kammerer		501MT510M03	
APPROVALS		REV LTR: New	PAGE 1 of 6
	MATERIAL PROCESSING PROCEDURE	DATE: 10-11-74	
		SUPERSEDES MPP DATED	
		AUTHORIZING MPS: MT0501-510	

ERS 66 CONFIGURATION

QUALITY PROCESS PROCEDURE

TITLE

ULTRASONIC INSPECTION OF ADHESIVE BONDED ASSEMBLIES

REVISION RECORD

REV NO CHG LTR	DESCRIPTION/AUTHORIZATION	APPROVAL/DATE

EO 839733

FORM 3916 S REV 3-73

1. SCOPE

1.1 This procedure describes the methods to be used when performing ultrasonic through-transmission inspection of voids in adhesive bonded honeycomb structures, to the requirements of MT0501-510 and MT0501-508.

2.0 APPLICABLE DOCUMENTS/MATERIALS

2.1 501MT510M08 Ultrasonic Pre-Inspection Preparation

3.0 GENERAL NOTES/SAFETY REQUIREMENTS

3.1 Personnel Qualification and Certification - All personnel performing inspection operations to this procedure will require both "certification" and "qualification" as follows:

3.1.1 Certification: All inspectors will be currently "certified" to Space Division Training Course No. 182AI/J.

3.1.2 Qualification: All inspectors will, in addition to certification, be qualified in the ultrasonic inspection process (TCN 192AI) to be used.

3.2 All test equipment shall be used with safety precautions suggested by suppliers

3.3 Industrial Safety approved ventilation must be available and operating in all facility areas where chemicals in this procedure are used.

3.4 Personnel must wear impervious plastic gloves and protective goggles during use of chemicals.

3.5 Reference Standards: Ultrasonic inspection shall be performed upon production parts only when standards with known size defects and cross sections are available which represent the part configuration.

3.6 Equipment Maintenance: Ultrasonic equipment shall be maintained in operating condition, and shall be the responsibility of Inspection supervision.

3.7 Reference Standard Maintenance: Adhesive bonded reference standards shall be maintained with edge seals free of holes and fractures. Repairs will be made, when required, with permanent sealing material such as polysulfide sealants.

3.8 Electronic Instrumentation: All electronic instrumentation shall be allowed to warm up prior to inspection for a period of time (not less than 15 minutes) recommended by the manufacturer of the instrument.

3.9 Equipment Calibration: Ultrasonic instrumentation shall be calibrated in accordance with procedures established by the Quality Engineering functional group responsible for the ultrasonic inspection process.

3.10 The following definitions are included to assist in understanding the procedure:

Squirter: A clear plastic assembly housing an ultrasonic transducer, a nozzle, a water input fitting, and a nozzle insert.

Nozzle: An adjustable plastic tube which can be adjusted for optimum laminar flow of water through the nozzle insert.

Nozzle Insert: An orifice control plastic insert in the nozzle end to provide water stream diameters of 1/8, 5/32, 3/16, 7/32, 1/4, 5/16 and 3/8 inch.

Attenuation: The loss of propagation of ultrasound in a material. The amplitude measure of attenuation will be expressed in decibels (db).

Water Noise: Dissolved air and turbulent water flow will create ultrasonic reflective noise responses (water noise) on the ultrasonic transceiver video display cathode ray tube (CRT).

Video: Ultrasonic signal responses displayed visually on the face of the transceiver (CRT).

Gated Video: A portion of the overall video that is selected for C-scan recording to show the presence or absence of defects.

Reference Standards: A test panel that duplicates configuration and fabrication of the hardware item to be inspected with built-in controlled size defects.

4.0 ACCEPT/REJECT CRITERIA

4.1 The bonded honeycomb structures accept/reject criteria shall be determined by the requirements of the applicable engineering drawing and/or as referenced by the planning documentation.

5.0 PROCEDURE

5.1 Summary - The following steps shall be performed in sequence.

Step 1. Enter required parts data on inspection forms.

Step 2. Prepare the part for ultrasonic inspection.

Step 3. Position reference standard and part into holding fixtures.

Step 4. Set up for reference standard scanning.

Step 5. Set up for production part scanning.

Step 6. Identify indications.

Step 7. Evaluate defects.

Step 8. Complete inspection records.

Step 9. Clean and dry production part.

5.2 Detailed Steps

Step 1. Enter required parts data on inspection forms.

1A Check the Manufacturing Order to see that all previous operations have been completed.

1B Review other documentation, such as drawings, photographs, etc., to verify location of closeouts and metal inserts.

Step 2. Prepare the part for ultrasonic inspection.

2A Assign a control number to the technique control record.

2B Mask and/or seal parts as necessary to prevent water entering areas that may be necessary to keep dry. Use method presented in 501MT510M08.

2C Attach any tooling that might be necessary to support the part or standard in the inspection system.

Step 3. Position reference standard and part into holding fixture.

3A Select the holding fixture for the configuration to be inspected. For example, flat parts can be clamped vertical between squirters.

Step 4. Set up for reference standard scanning.

4A Turn instrumentation to ON position, and allow to warm up for a minimum of 15 minutes.

4B Position squirter to scan position and adjust water controls for abundant flow to force air bubbles out of the system.

4C Reduce water flow until a smooth stream is obtained, free of spirals.

4D Observe CRT of ultrasonic detector for reflective amplitude response from surface reflection, back surface and/or reflector surfaces.

4E Move transducers to a reference standard void and observe signal response. Little or no response will be indicated on the CRT.

4F If amplitude of signal response over the void is greater than 10% of total signal, increase reject until 10% or less is obtained.

4G Return transducers to a well bonded area and observe signal response. If signal response does not return to saturation, increase gain until saturation is achieved, then repeat steps 4E, 4F, and 4G.

4H If the saturation and 10% of saturation signal differential is not obtainable, different transducer frequency and squirter nozzle insert sizes shall be used until separation is obtained.

4I Adjust gate controls until gated video includes only the through-transmission signal response.

4J Adjust bridge controls to set scan limits for C-scan of the reference standard.

4K Scan approximately one inch of the reference standard with the void included.

4L Measure the width dimensions of the C-scan recorded void. If it is not the dimension of the standard defect, increase or decrease the receiver gain and repeat the one-inch scan.

Step 5. Set up for production part scanning.

5A Move bridge controls for a C-scan of the part. If possible, part scanning shall include the standard simultaneously with the part. If not possible, a one-inch scan of the standard shall be completed at the end of the part C-scan.

5B With scan speed reduced to zero, push scan switch to ON and advance speed control until operation speed is obtained (not to exceed 20 inch/sec.).

Step 6. Identify indications.

6A All C-scan recorded discontinuities shall be identified by comparison to drawings illustrating location of closeouts or other areas that would cause signal loss.

6B Mark areas on the recording that are caused by internal changes in cross section that are not necessarily voids.

Step 7. Evaluate defects.

7A Evaluation of discontinuities will be in accordance with the applicable process specifications or Engineering drawing requirements for acceptance criteria.

7B Discontinuities that may be present shall be further evaluated with manual A-scan techniques.

7C Discontinuities larger than allowable that have been verified by A-scan techniques shall be labeled defects on the C-scan recording.

7D Defects in sandwich structures shall be further evaluated to provide information relative to location.

7E Specific techniques for location of defects within the cross-section shall be with pulse echo contact, sonic test system, and/or coin tapping.

7F Mark location of defect upon part surface.

Step 8. Complete inspection records.

8A Accept parts free from defects and stamp inspection records and work order.

8B Squawk defective debonds or delaminations in accordance with work order procedures.

8C Complete ultrasonic technique record with the required information relative to transducer and receiver frequencies, water nozzle insert size, etc..

Step 9. Clean assemblies with clean water and wipe dry.

9A All masking and/or sealing material that may have been used shall be removed in accordance with 501MT510M08.

9B Oven drying when necessary or required, shall be in accordance with 501MT510M08.

SHUTTLE

PREPARED BY D/044-220-041-FA91 C. J. Adams, Ext. 2545	CODE IDENT. NO. 03953	DOCUMENT NUMBER: 501MT510M03	REV: A	SEQ: 01
APPROVALS Mfg.	Space Division Rockwell International	DOCUMENT TYPE: QUALITY		
M&P ENG. 4/23/75		AUTHORITY: G.O. 40039		
Q.A.	AMENDMENT Ultrasonic Inspection of Adhesive Bonded Assemblies	DATE: 4/23/75	PAGE 1 OF 1	
N.D.E.	ERS 82	SUBJECT: Change		

This amendment forms a part of Quality Processing Procedure 501MT510M03, New, dated 11 October 1975.

IS Step 3 Position reference standard and part into holding fixture.

3A Select the holding fixture for the configuration to be inspected, for example, flat parts can be clamped vertical between squirters.

CAUTION

Where interim improvised clamping devices are used in lieu of approved holding fixtures, restraints must be provided to prohibit inadvertent hardware damage.

WAS Step 3 Position reference standard and part into holding fixture.

3A Select the holding fixture for the configuration to be inspected, for example, flat parts can be clamped vertical between squirters.

OFFICIALLY RELEASED
APR 29 1975
4:12 PM
ENGINEERING

FORM 3945-E REV 2-74

3568 SHUTTLE

PREPARED BY: L. Owen 044-220 AD50 2-5146	CODE IDENT. NO. 03953	DOCUMENT NUMBER: 501MT510M03	REV: A SEQ: 02
APPROVALS: QA; ENG; MFG; PROJ 7 26 77	Space Division Rockwell International	DOCUMENT TYPE: QUALITY	
		AUTHORITY: GO 40039	
	AMENDMENT	DATE: 08-04-77	PAGE 1 OF 1
	Ultrasonic Inspection of Adhesive Bonded Assemblies	SUBJECT: CHANGE	

This amendment forms a part of Quality Process Procedure 501MT510M03, Revision NEW, dated October 11, 1974.

IS

3.1.1 Certification: .... Space Division Training Course No. 182 AI/AJ.

3.1.2 Qualification: All inspectors will be currently qualified to Space Division On-The-Job Qualification (OJQ) Course No. 888 AO/AP.

WAS

3.1.1 Certification: .... Space Division Training Course No. 182 AI/J.

3.1.2 Qualification: All inspectors will, in addition to certification, be qualified in the ultrasonic inspection process (TCN 182 AI) to be used.

OFFICIALLY RELEASED
AUG - 5 1977
8:32 AM
ENGINEERING

FORM 3945-E REV 2-74

812

SHUTTLE

PREPARED BY C. C. Kammerer D/344-230 3108	CODE IDENT. NO. 03953	DOCUMENT NUMBER 501MT510M03	REV A / SEQ 03
APPROVALS QE Safety	Rockwell International Space Systems Group	DOCUMENT TYPE Quality	
M&P 6/5/80	AMENDMENT	AUTHORITY GO 40039	
Mfg.	ULTRASONIC INSPECTION OF ADHESIVE BONDED ASSEMBLIES	DATE May 5, 1980	PAGE 1 OF 1
Proj.Eng. 6/9/80		SUBJECT Change	

This Amendment forms a part of Quality Process Procedure 501MT510M03, Revision New, dated October 11, 1974.

Page 6 — 9B

IS — All parts subjected to water squirter inspection shall be dried as soon as practical (not to exceed 16 hours) after ultrasonic inspection

9C

IS — Oven drying when necessary or required, shall be in accordance with 501MT510M08.

Page 6 — 9B

WAS — Oven drying when necessary or required, shall be in accordance with 501MT510M08.

RECEIVED
1980 JUN 18 AM 10
OFFICIAL FILES

FORM 3945 E REV 1-79

SHUTTLE

PREPARED BY	CODE IDENT. NO. 03953	DOCUMENT NUMBER	REV	PRO
J. Mamon D/344-230 2-3108		501MT510M03	A	04
APPROVALS	Space Division Rockwell International	DOCUMENT TYPE		
QE / Mfg. Eng.		Quality		
M&P	QUALITY PROCEDURE	AUTHORITY		
	AMENDMENT	CO 40111		
Proj. Eng.	ULTRASONIC INSPECTION	DATE	PAGE 1	
	OF	8-10-81	OF 1	
Safety	ADHESIVE BONDED ASSEMBLIES	SUBJECT		
		CHANGE		

ERS 68 CONFIGURATION

This amendment forms a part of Quality Process Procedure 501MT510M03, revision new, dated October 11, 1974.

Page 6 9B

IS All parts subjected to water squirter inspection shall be dried as soon as practical (not to exceed 72 hours) after ultrasonic inspection.

WAS All parts subjected to water squirter inspection shall be dried as soon as practical (not to exceed 16 hours) after ultrasonic inspection.

OFFICIALLY RELEASED
1981 SEP -1 PM 1:40

FORM 3945-E REV 2-78

APPENDIX C

This appendix contains the stress/strain curves for testing of beam and coupon specimens in tension and compression.

TENSILE COUPON

CURVES

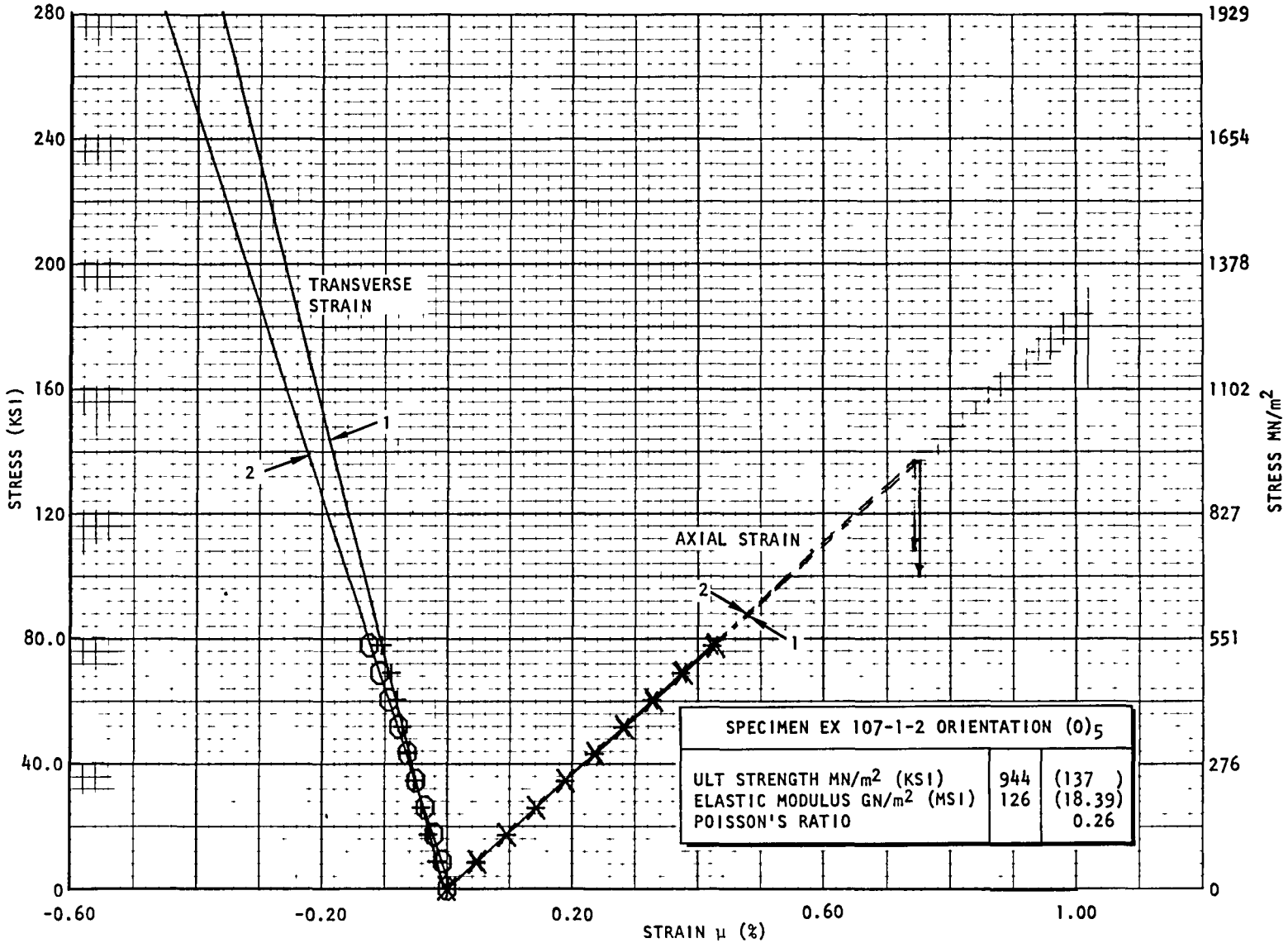


Figure C-1. LARC-160/Celion $(0)_5$ Stress/Strain Curves at RT

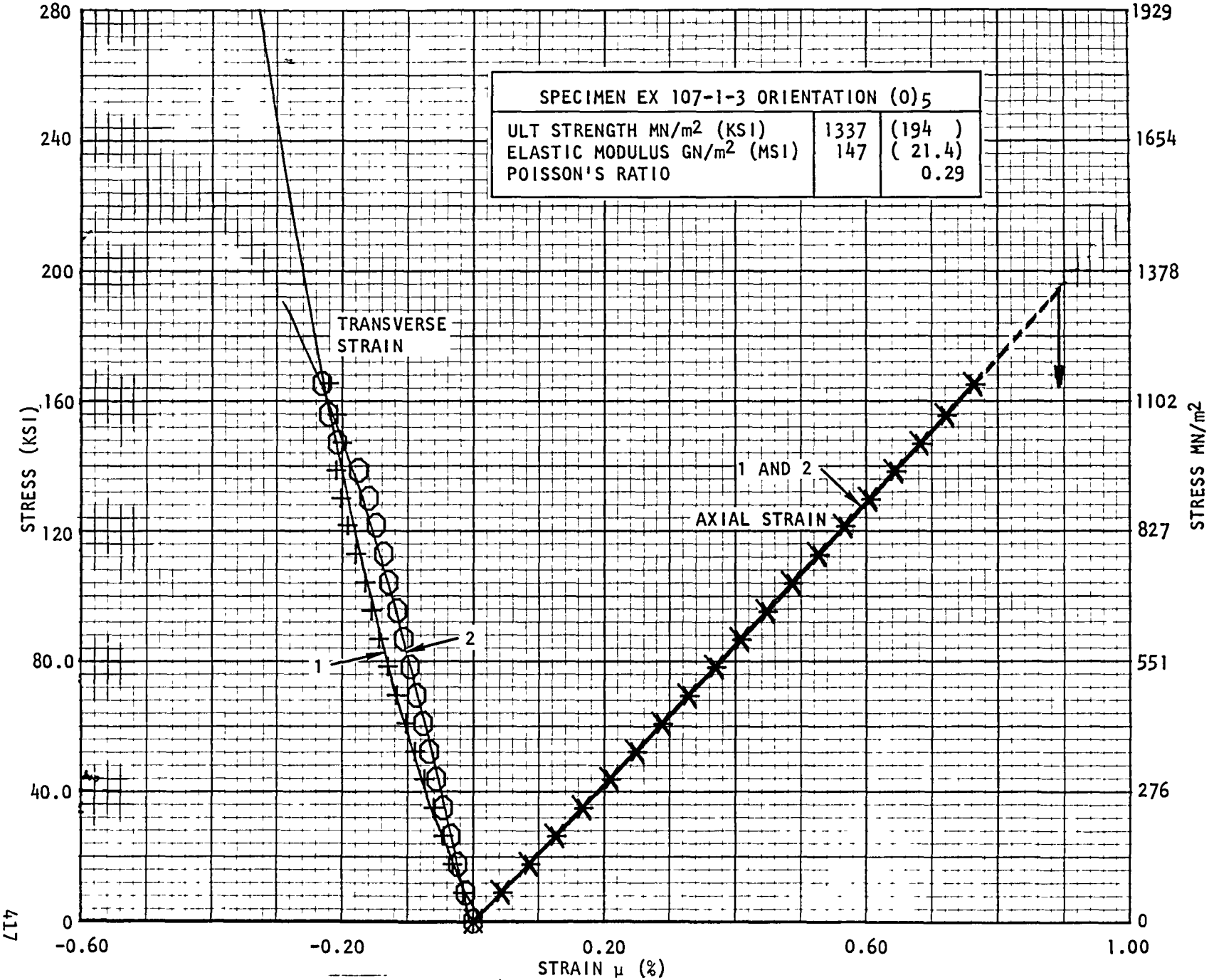


Figure C-2. LARC-160/Celion $(0)_5$ Stress/Strain Curves at RT.

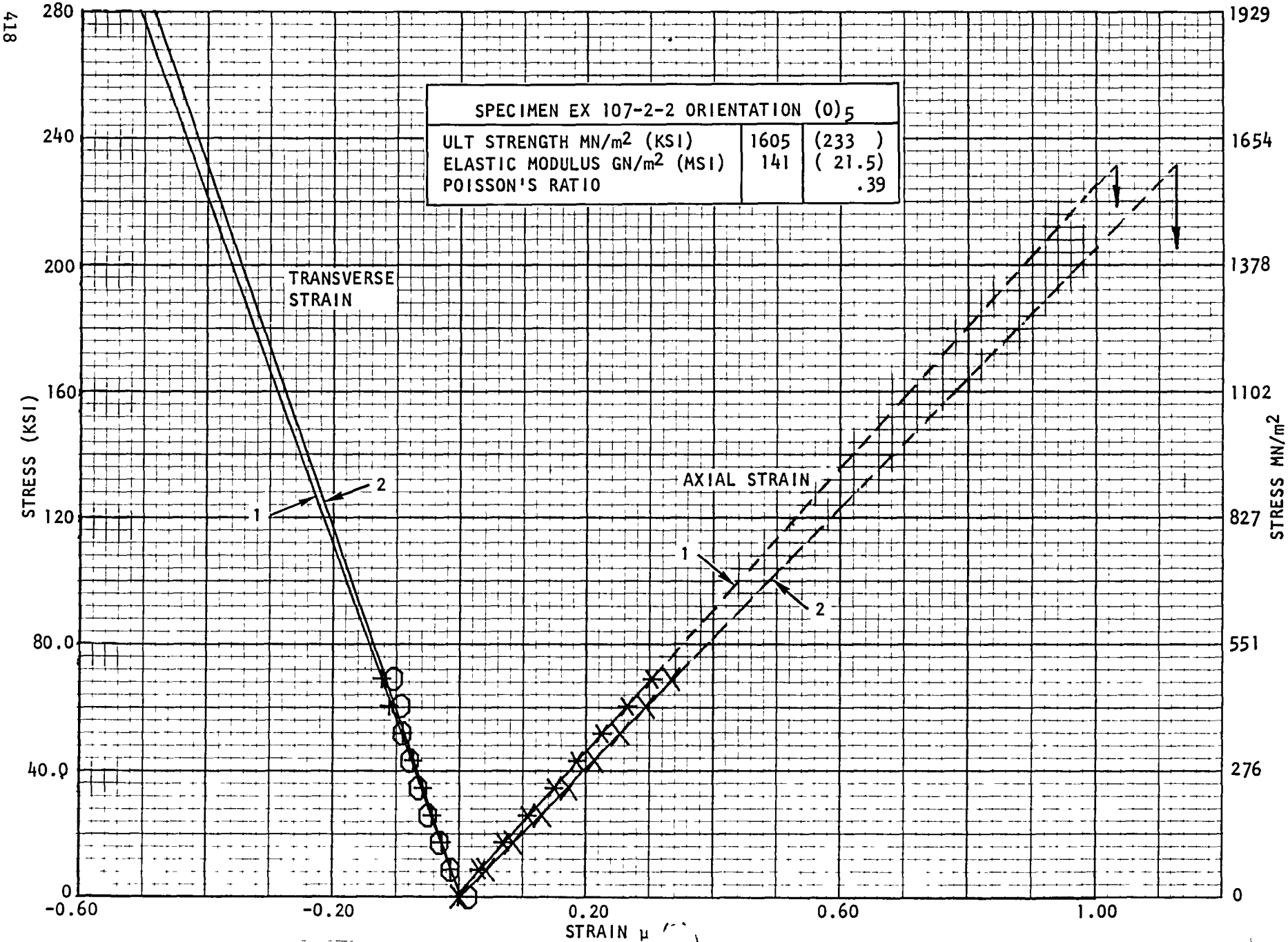


Figure C-3. LARC-160/Celion $(0)_5$ Stress-Strain Curves at -132 C (-270 F)

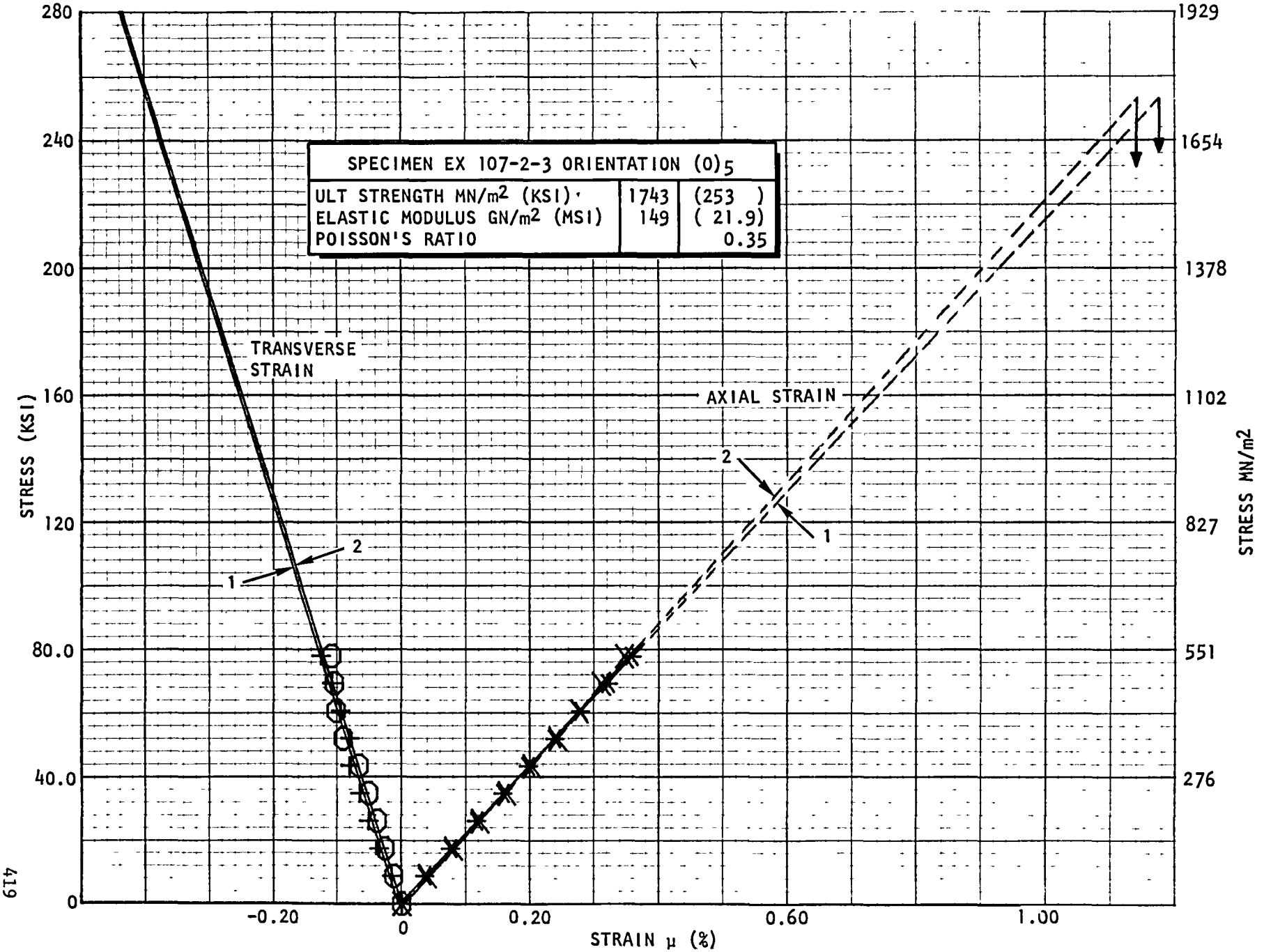


Figure C-4. LARC-160/Celion $(0)_5$ Stress/Strain Curves at -132 C (-270 F)

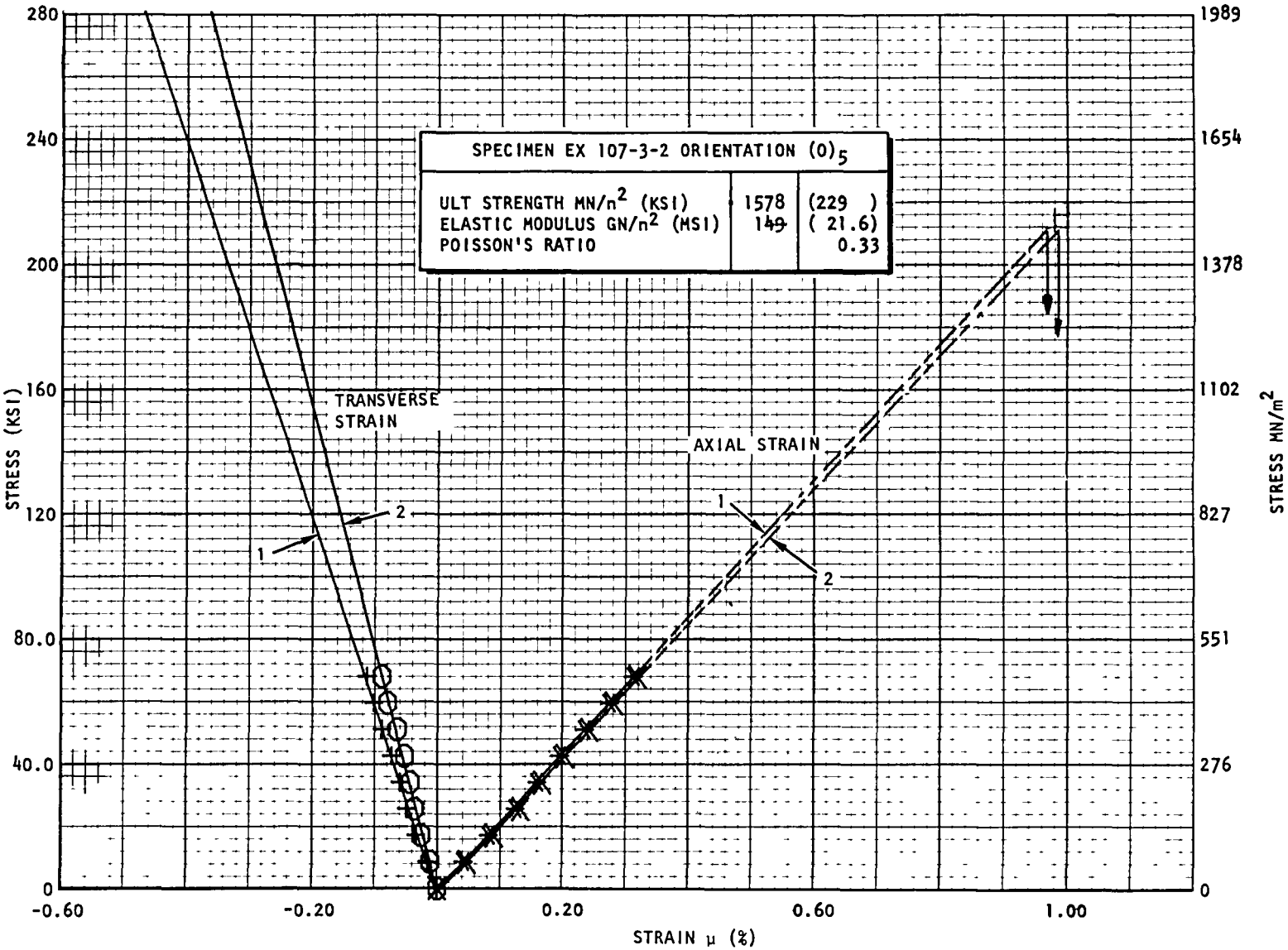


Figure C-5. LARC-160/Celion $(0)_5$ Stress/Strain Curves at 204 C (400 F)

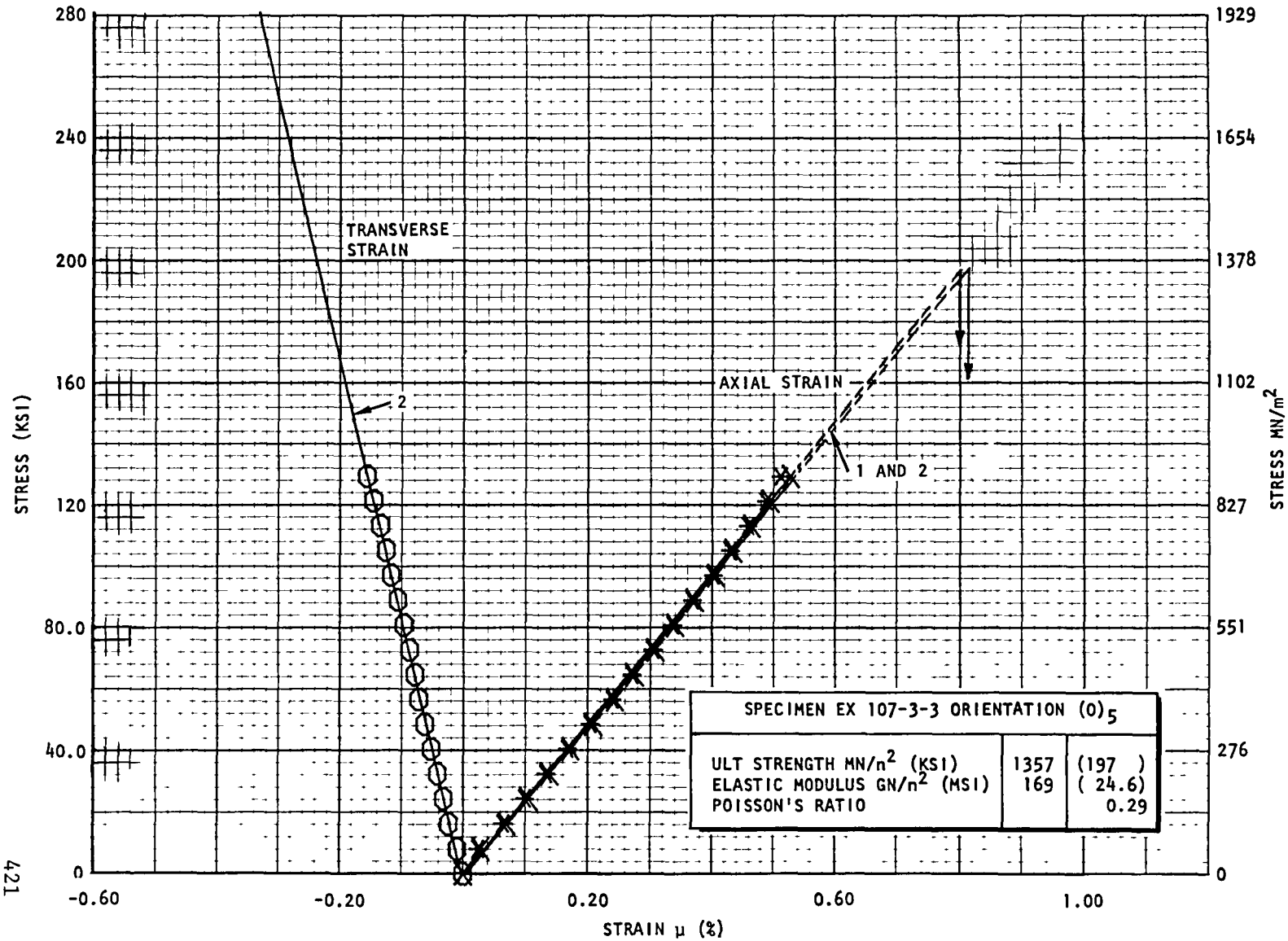


Figure C-6. LARC-160/Celion $(0)_5$ Stress/Strain Curves at 204 C (400 F)

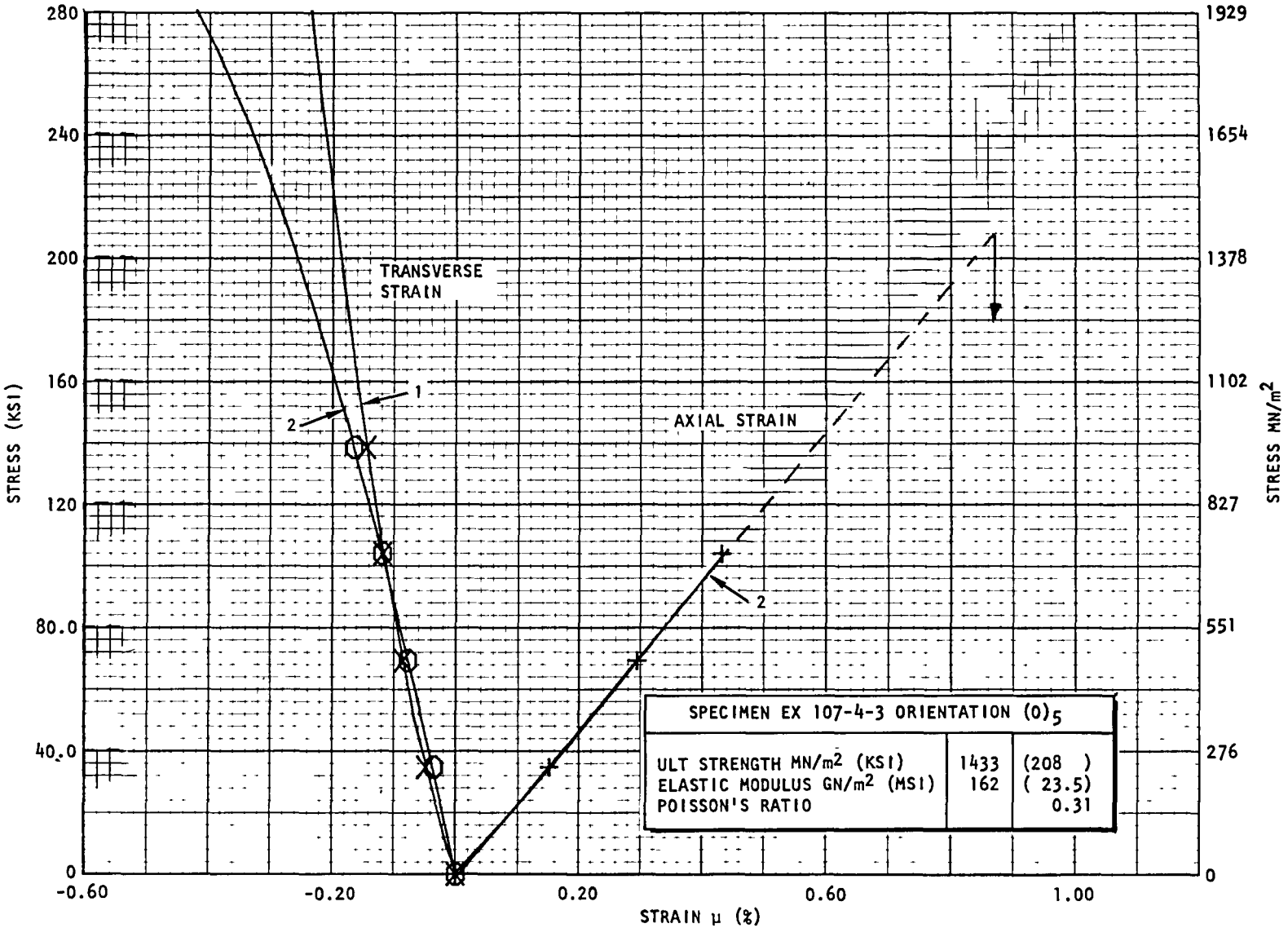


Figure C-7. LARC-160/Celion $(0)_5$ Stress/Strain Curves at 316 C (600 F)

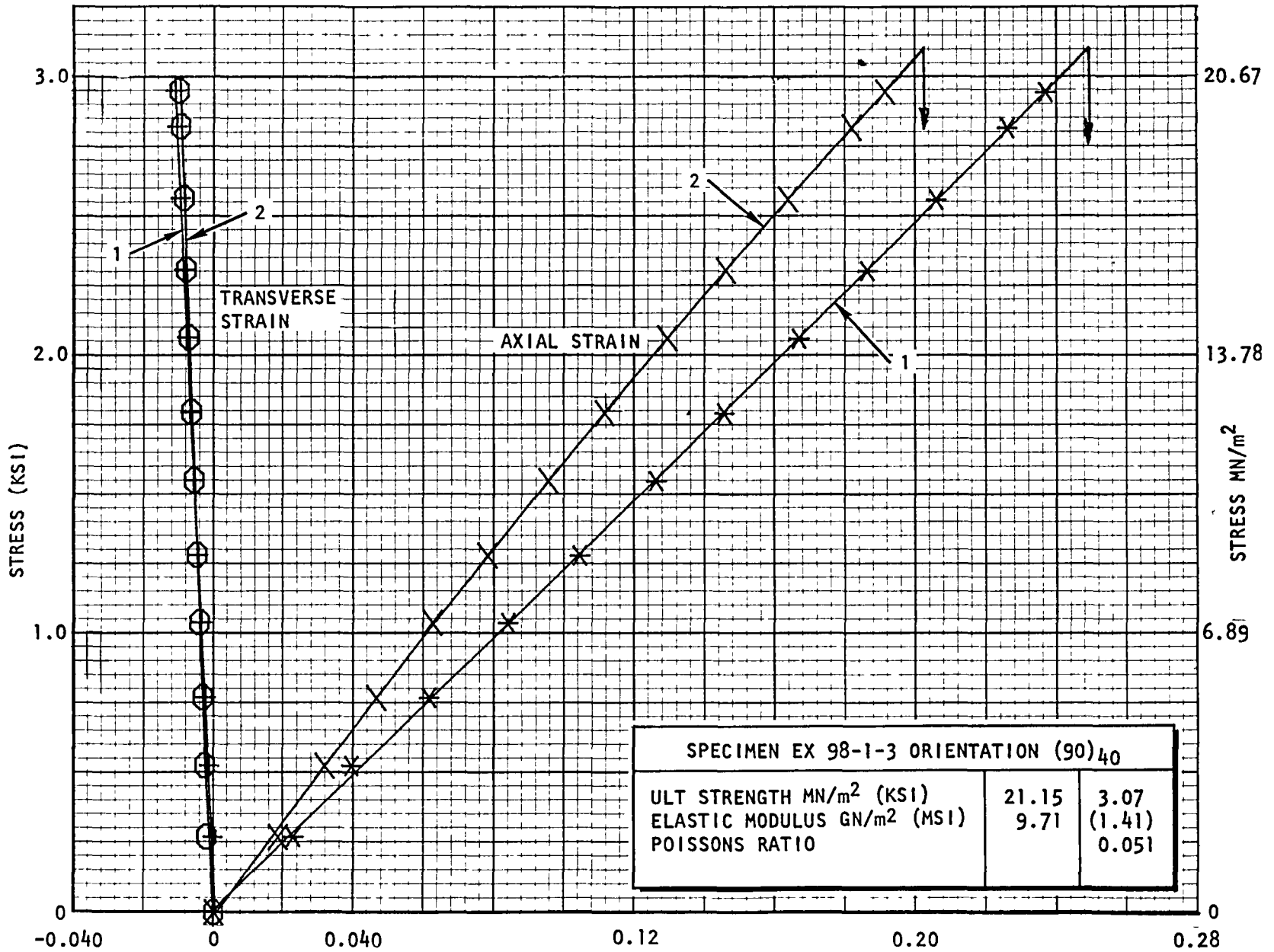


Figure C-8. LARC-160/Celion 90° Stress/Strain Curves at RT

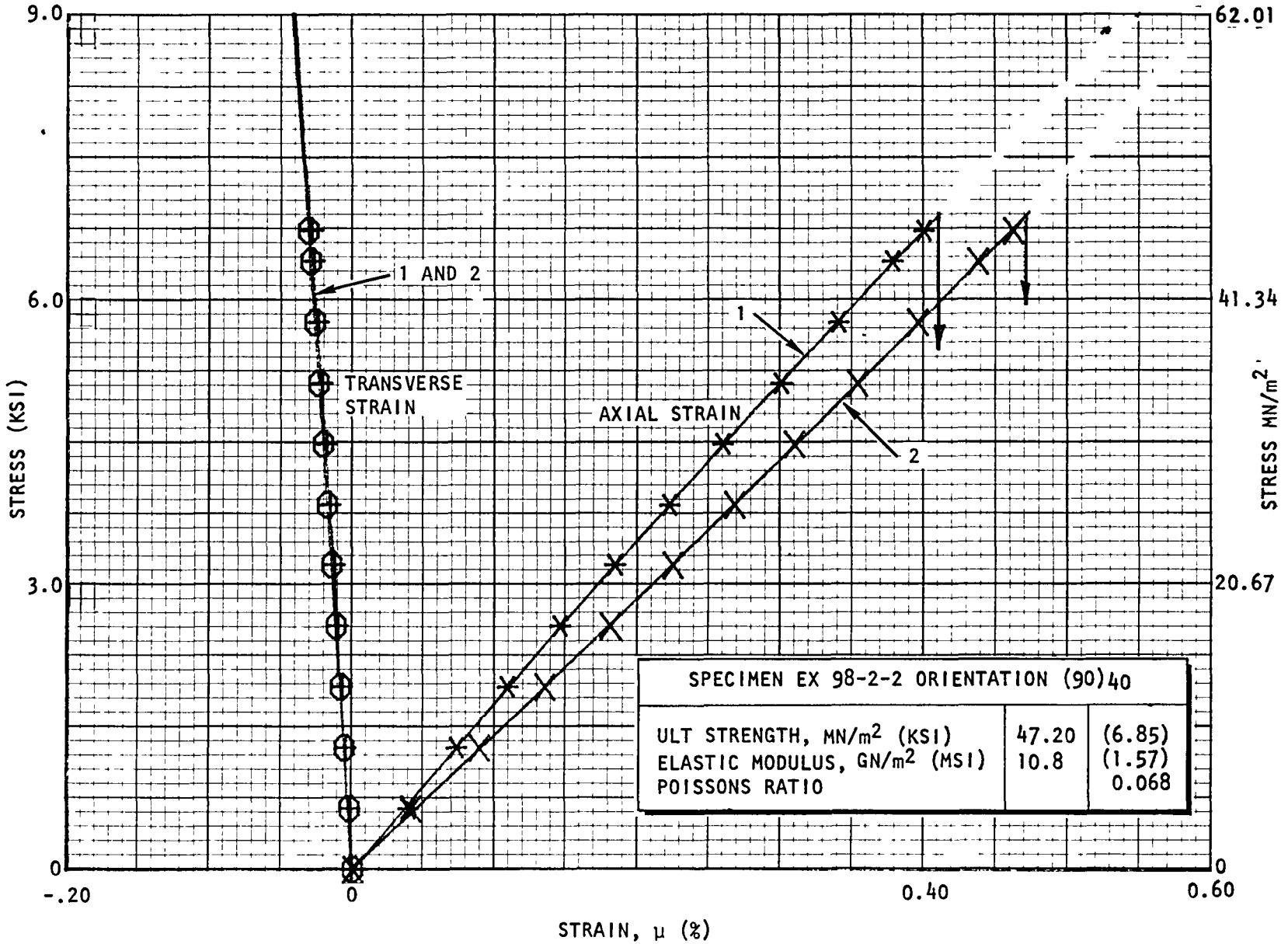


Figure C-9. LARC-160/Celion 90° Stress/Strain Curves at -132 C (-270 F)

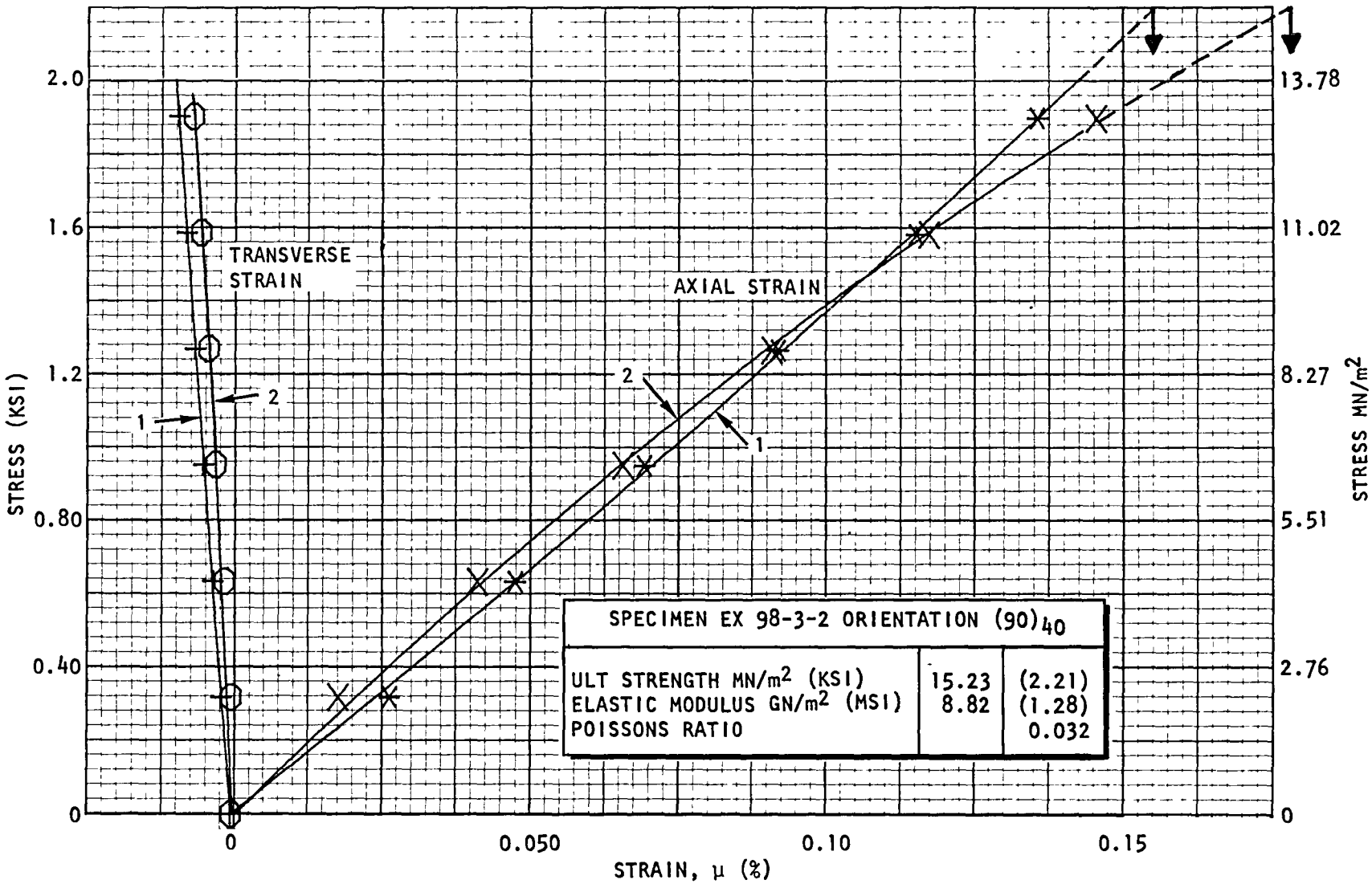


Figure C-10. LARC-160/Celion 90° Stress/Strain Curves at 204 C (400 F)

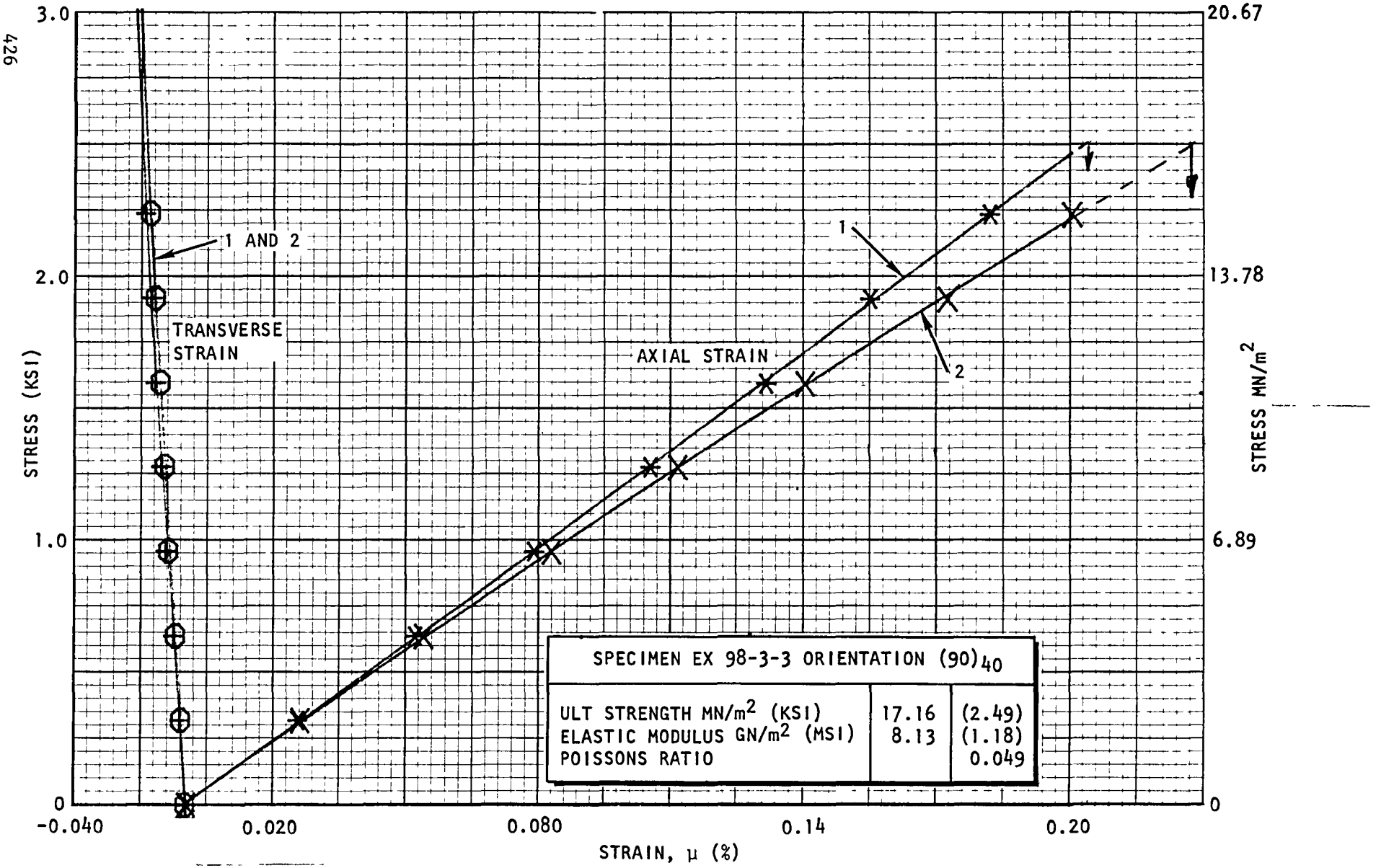


Figure C-11. LARC-160/Celion 90° Biaxial Stress/Strain Curves at 204C (400 F)

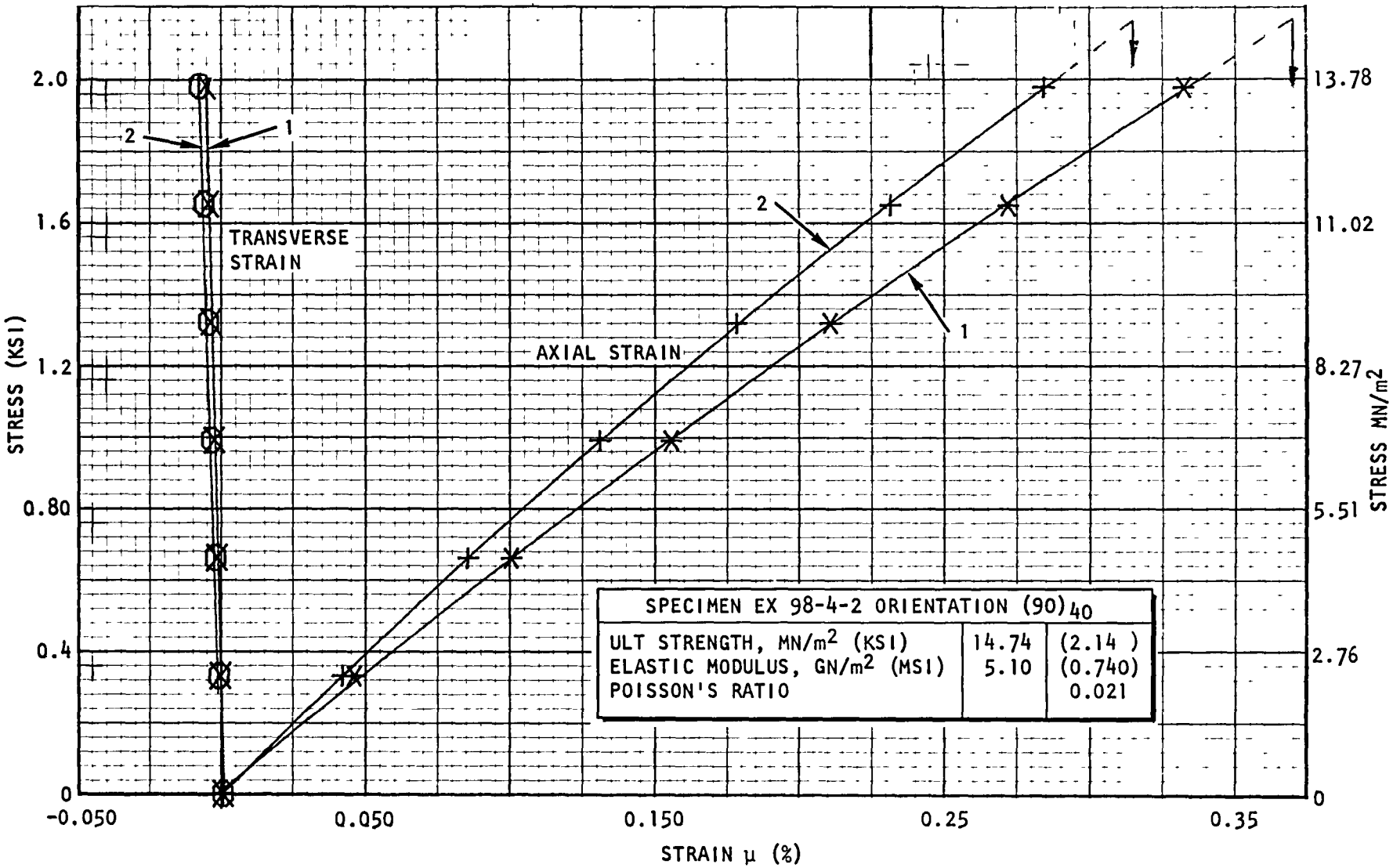


Figure C-12. LARC-160/Celion 90° Stress/Strain Curves at 316 C (600 F)

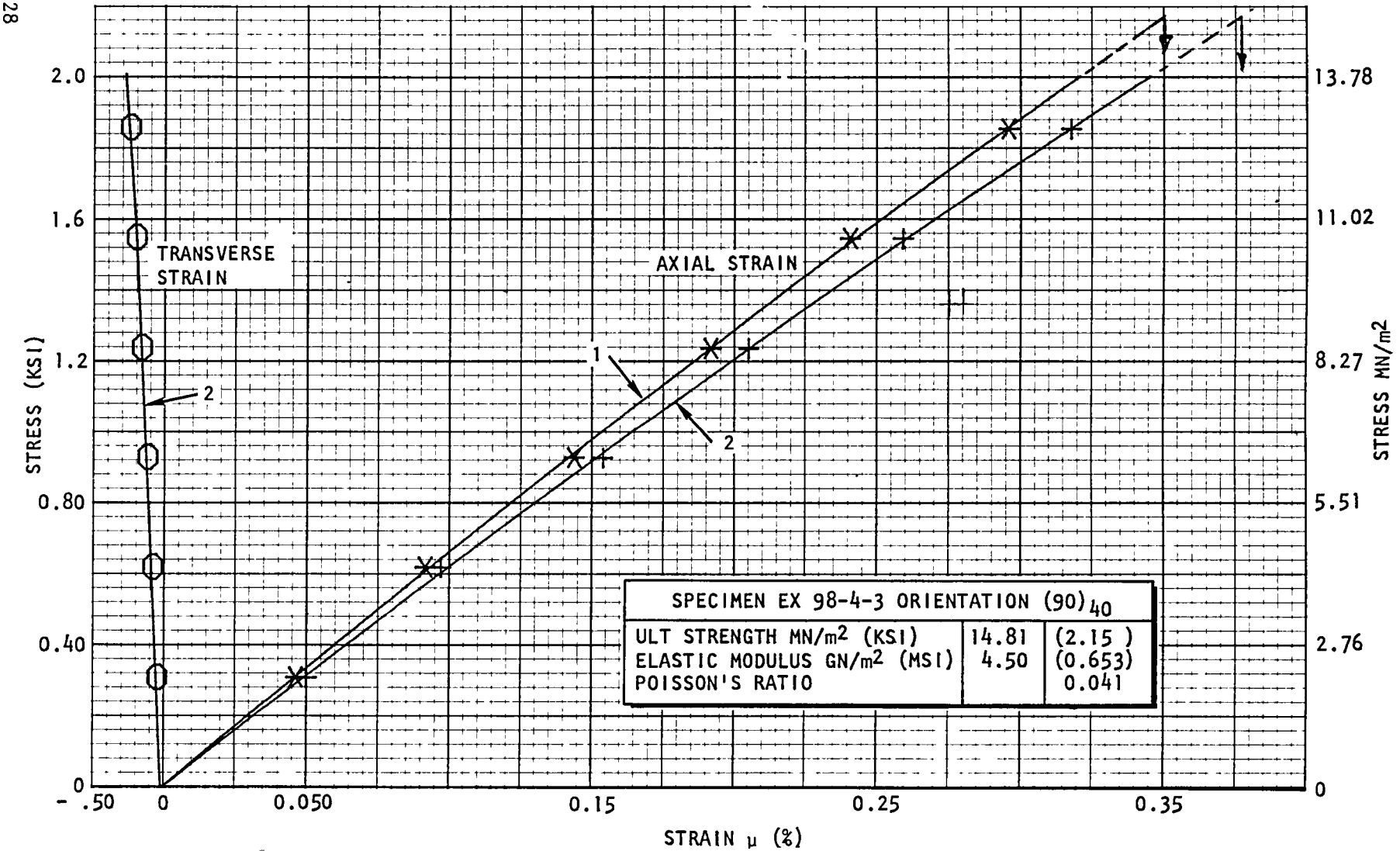


Figure C-13. LARC-160/Celion 90° Stress/Strain Curves at 316 C (600 F)

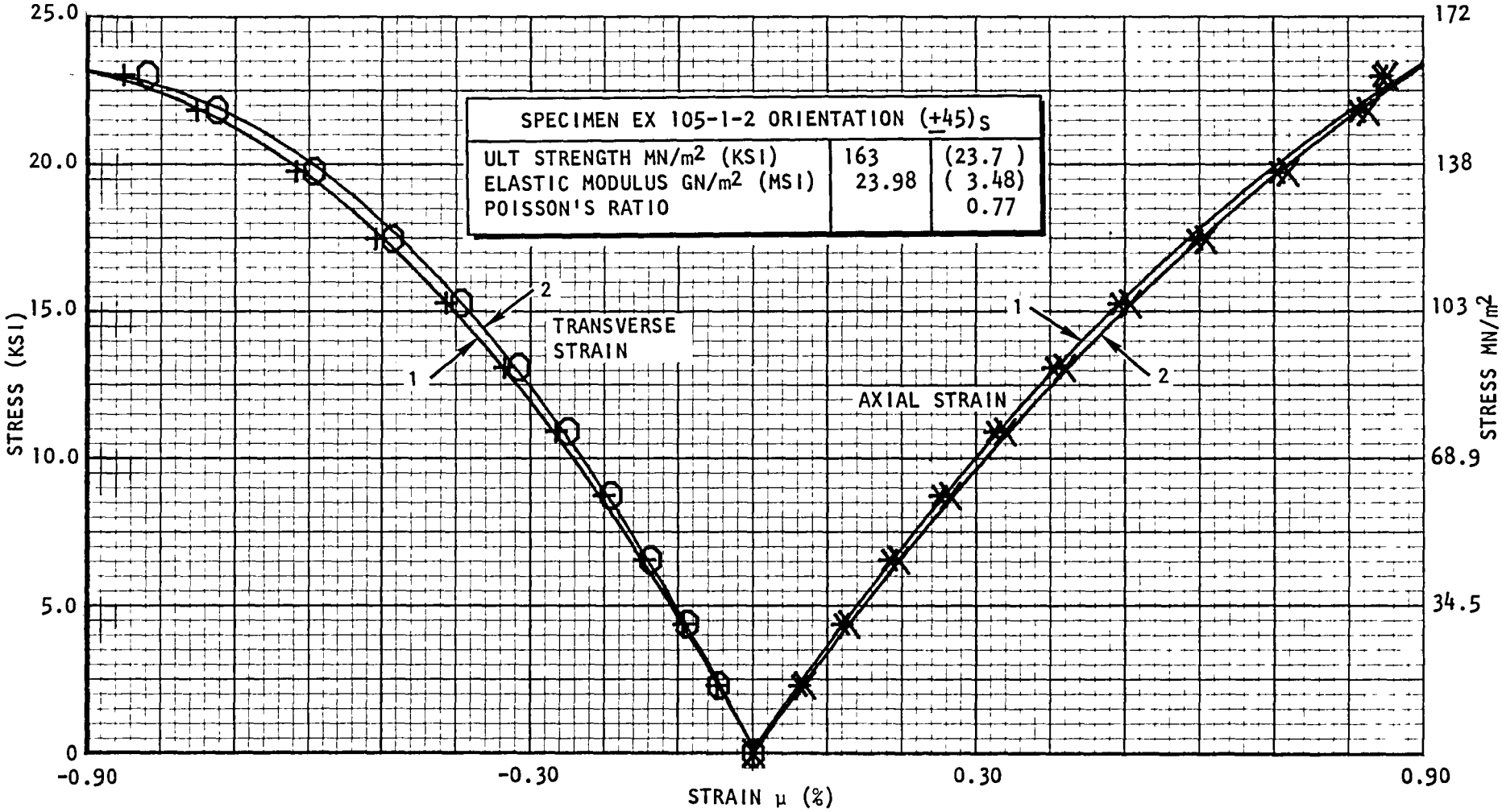


Figure C-14. LARC-160/Celion (±45)S Stress/Strain Curves at RT.

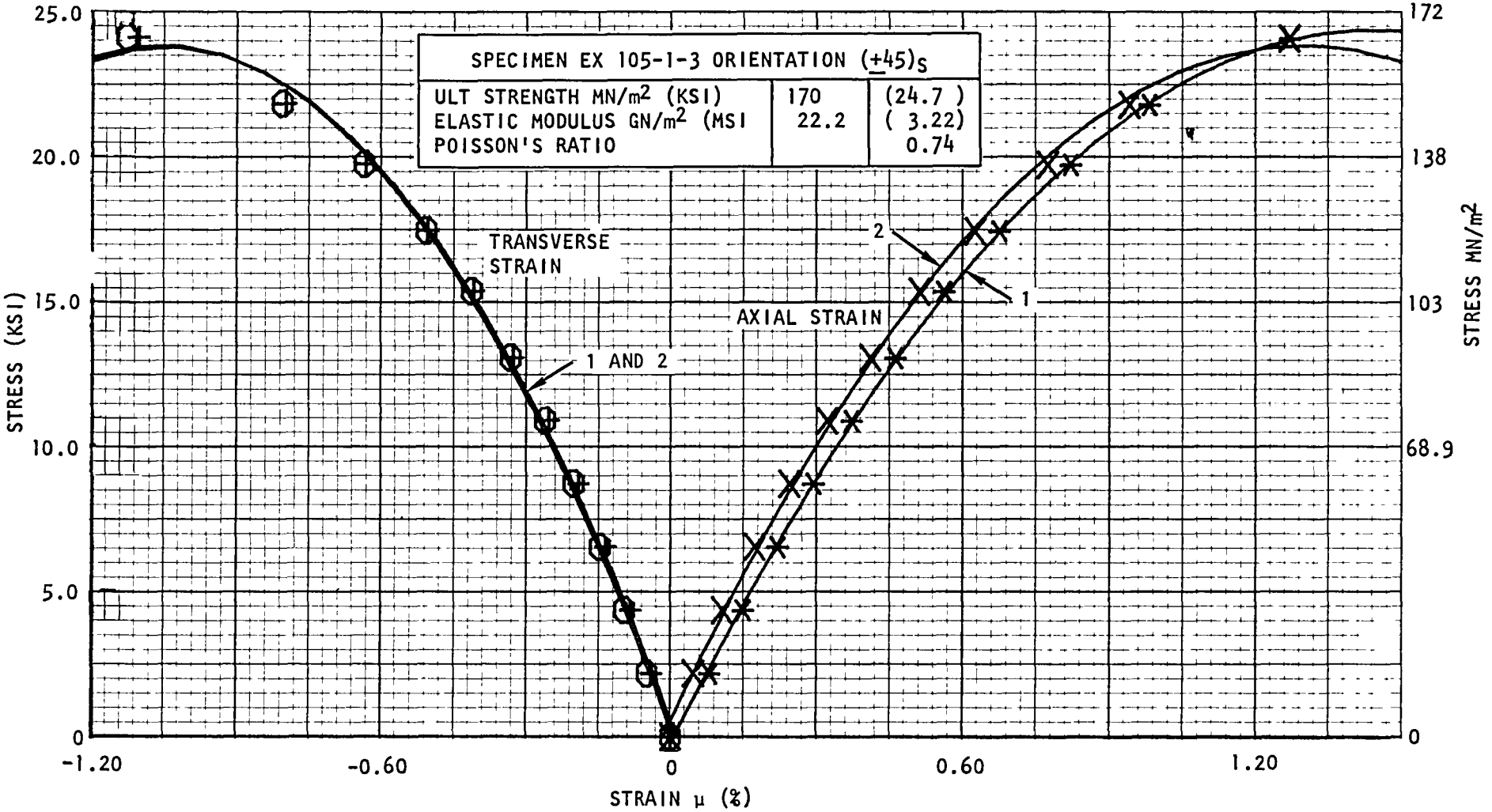


Figure C-15. LARC-160/Celion $(\pm 45)_S$ Stress/Strain Curves at RT.

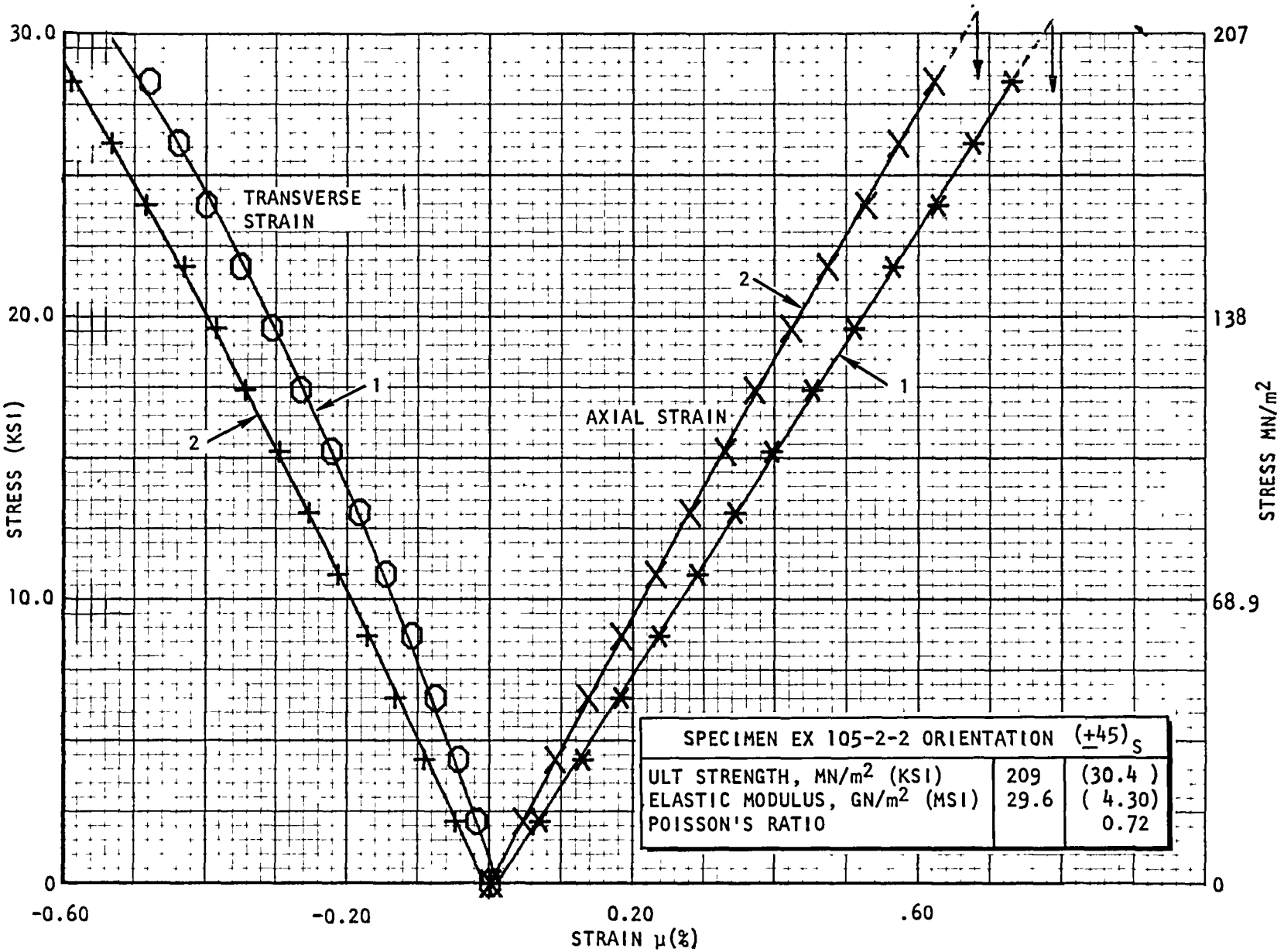


Figure C-16. LARC-160/Celion $(\pm 45)_S$ Stress/Strain Curves at -132 C (-270 F)

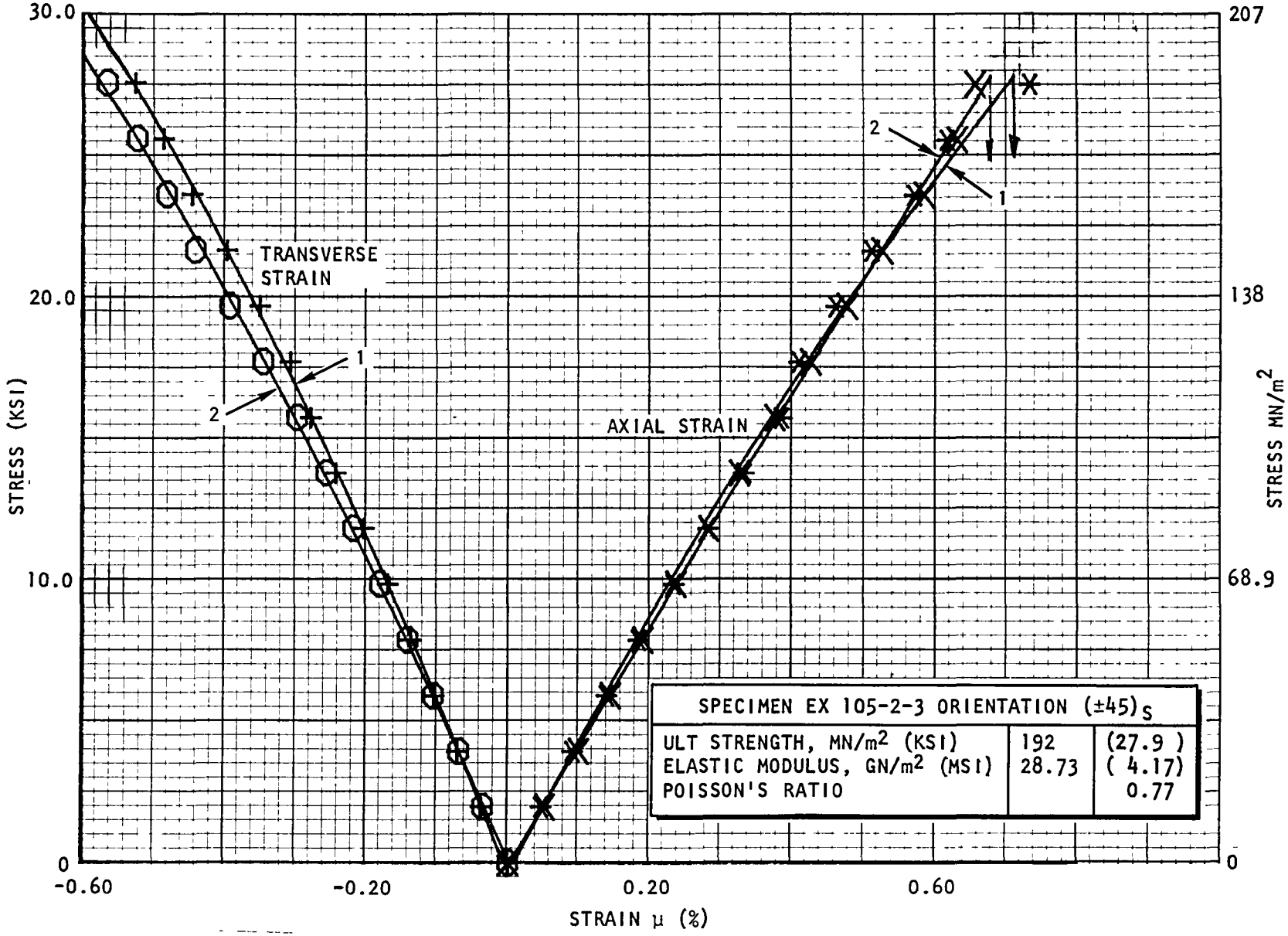


Figure C-17. LARC-160/Celion $(\pm 45)_S$ Stress/Strain Curves at -132 C (-270 F)

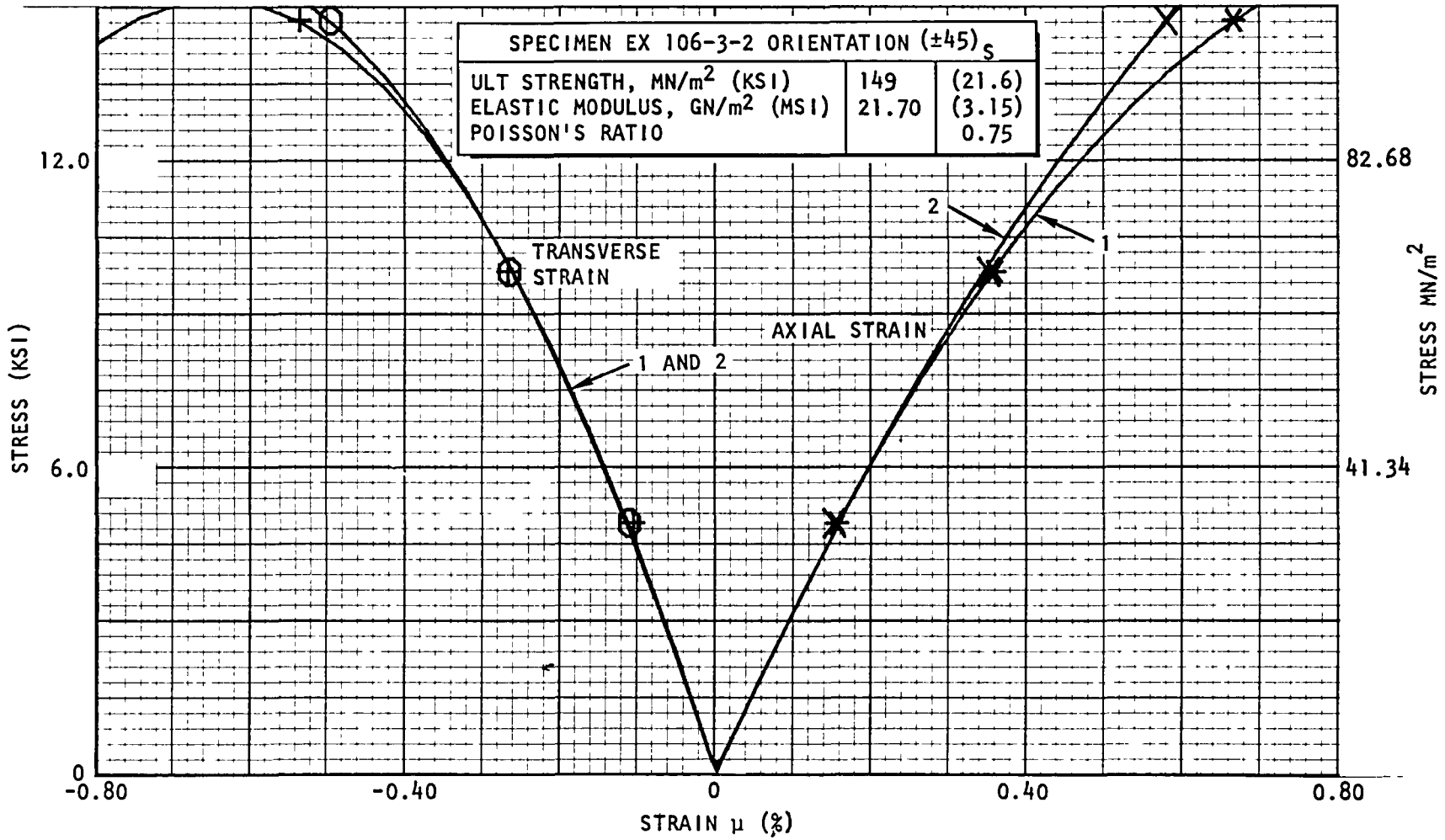


Figure C-18. LARC-160/Celion (±45)S Stress/Strain Curves at 204 C (400 F)

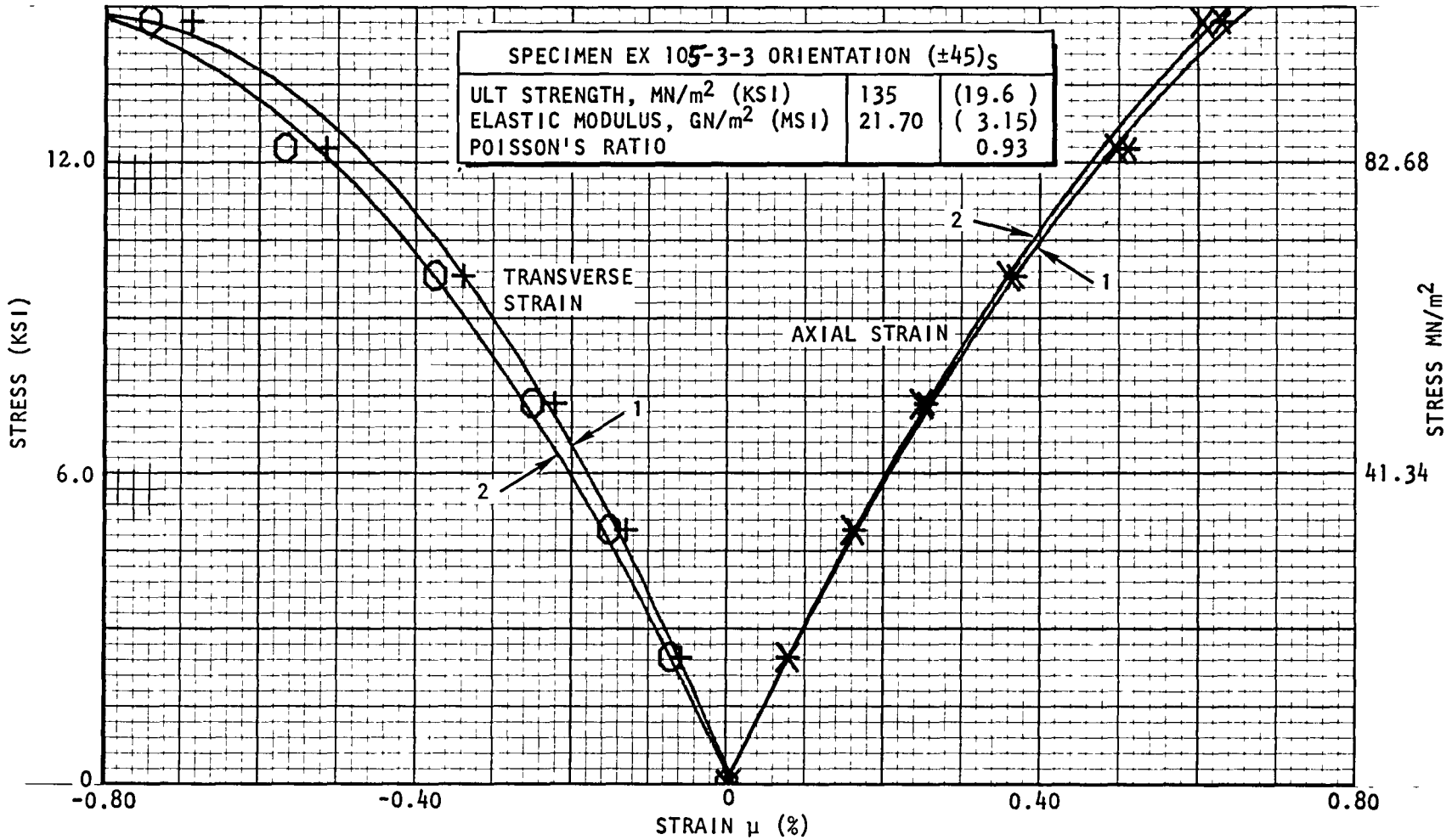


Figure C-19. LARC-160/Celion (±45)S Stress/Strain Curves at 204 C (400 F)

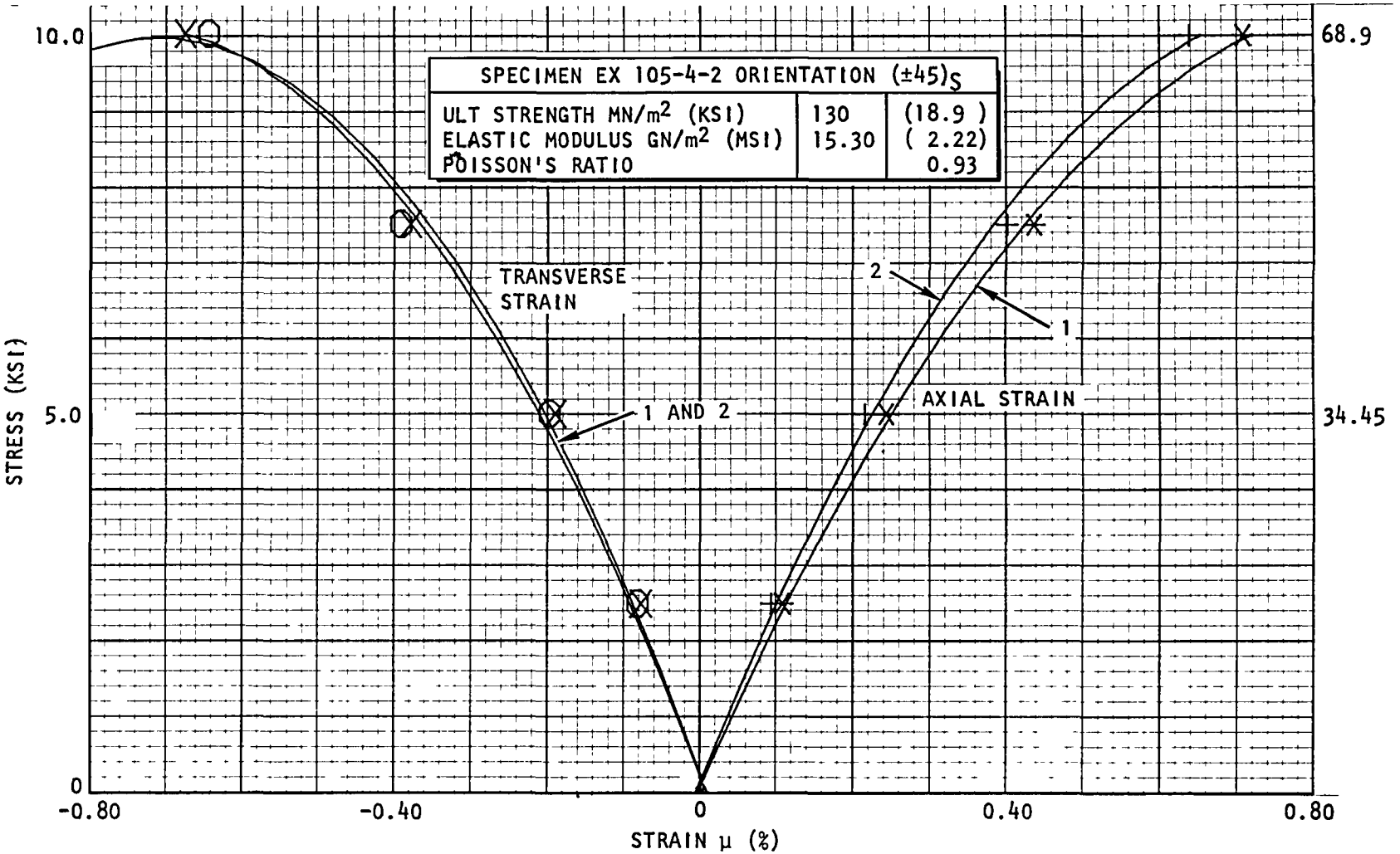


Figure C-20. LARC/Celion $(\pm 45)_S$ Stress/Strain Curves at 316 C (600 F)

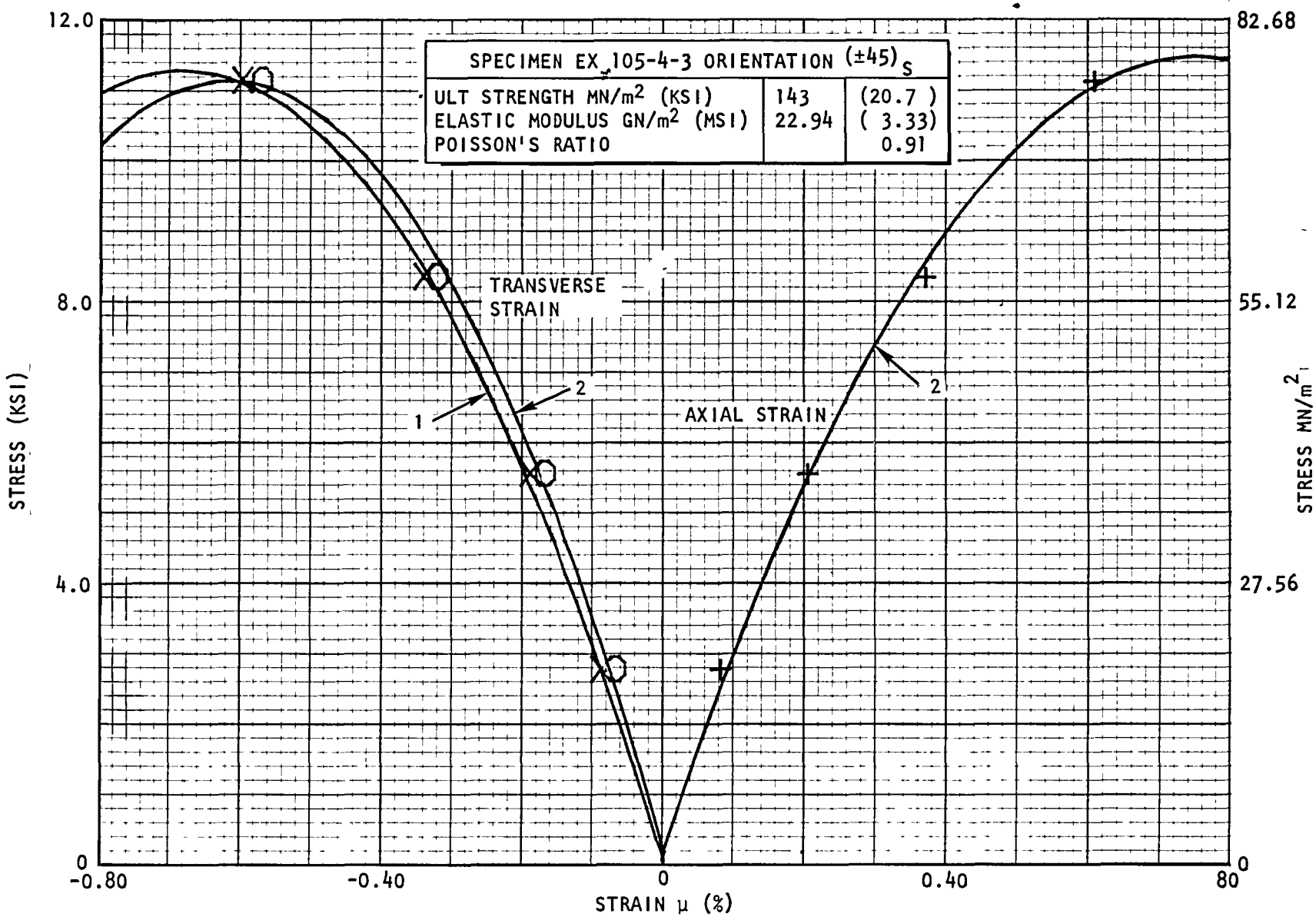


Figure C-21. LARC-160/Celion $(\pm 45)_S$ Stress/Strain Curves at 316 C (600 F)

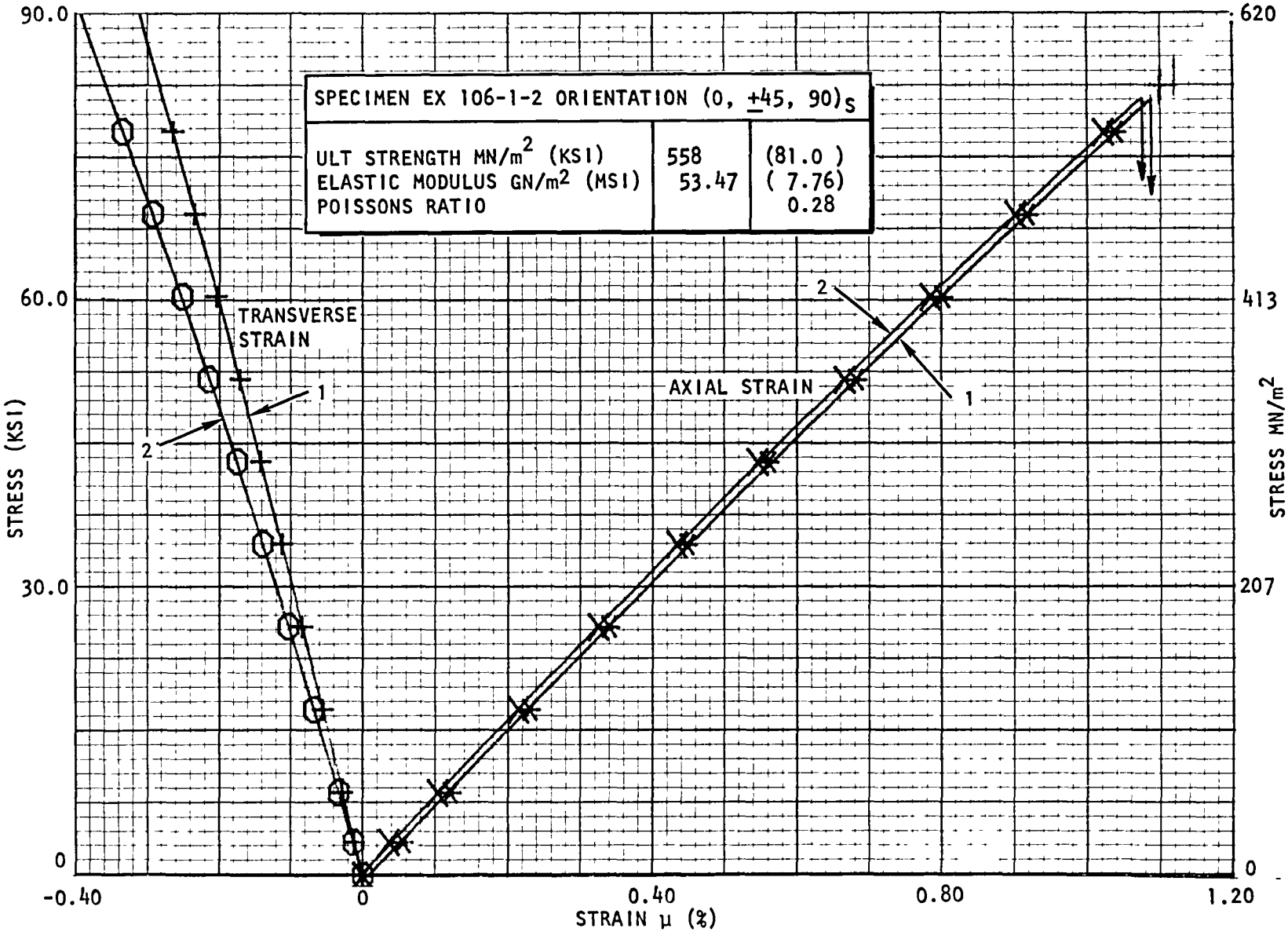


Figure C-22. LARC-160/Celion (0, ±45, 90)S Stress/Strain Curves at RT

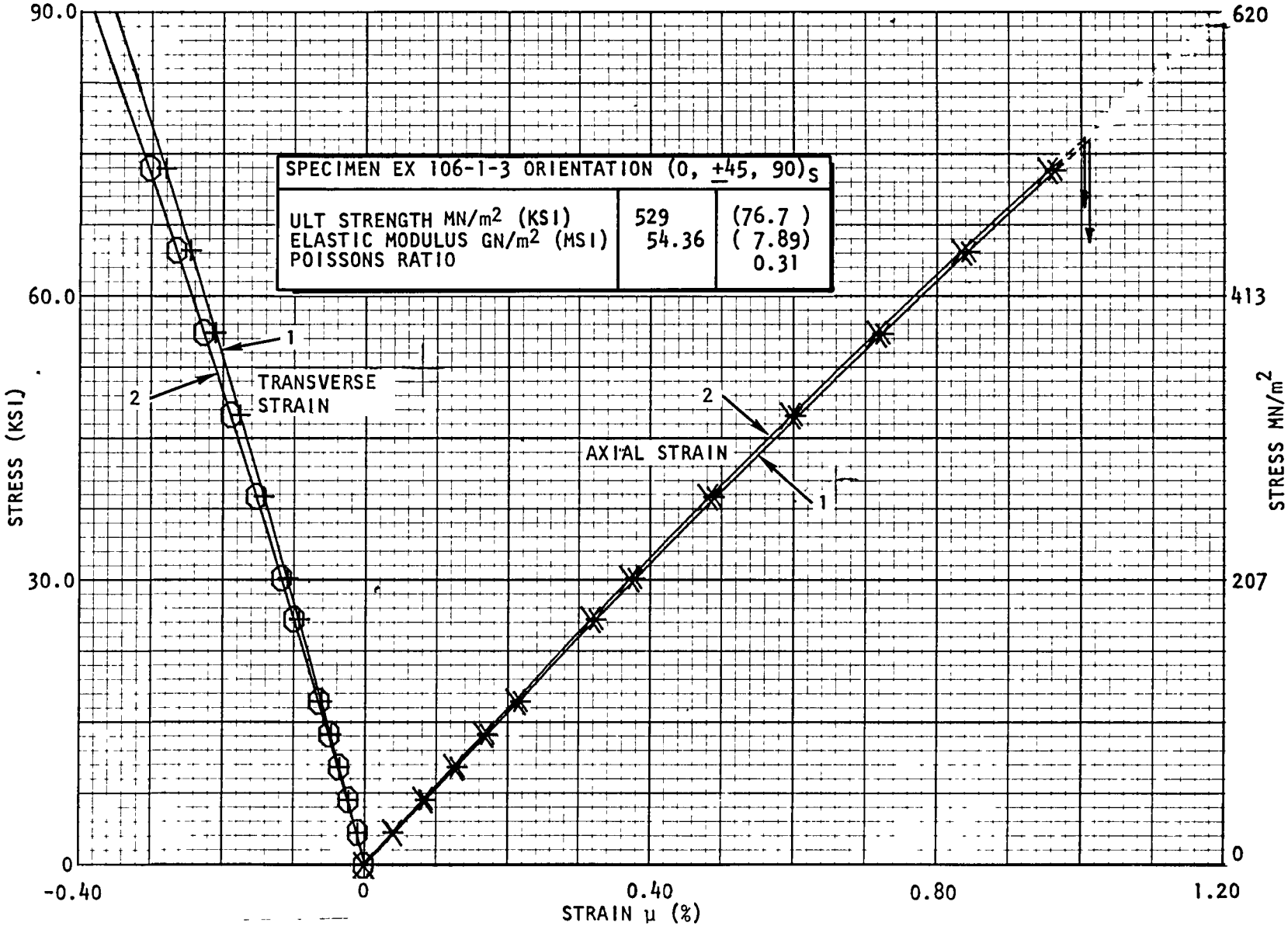


Figure C-23. LARC-160/Celion (0, ±45, 90)S Stress/Strain Curves at RT

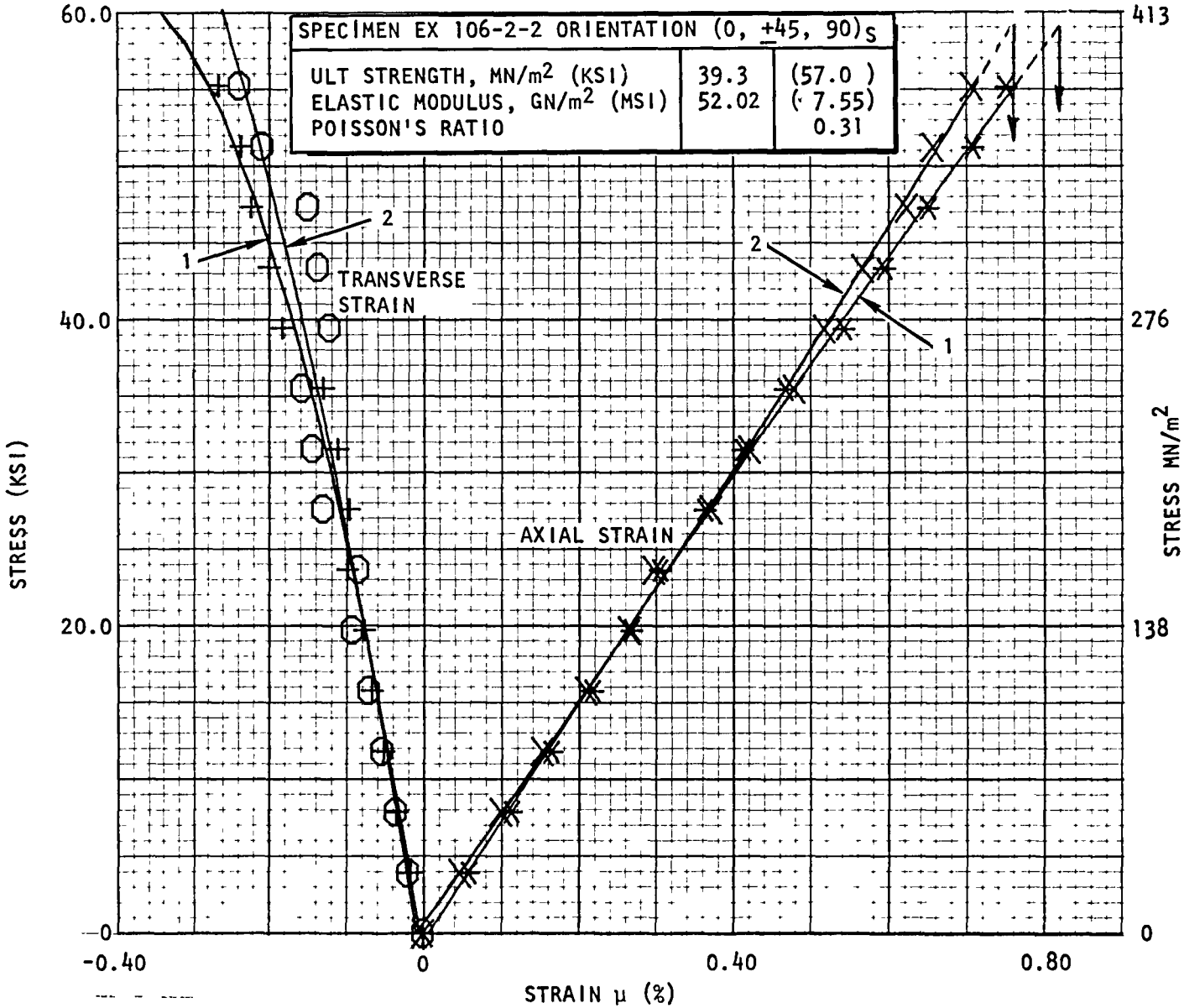


Figure C-24. LARC-160/Celion (0, ±45, 90)$_S$ Stress/Strain Curves at -132C (-270 F)

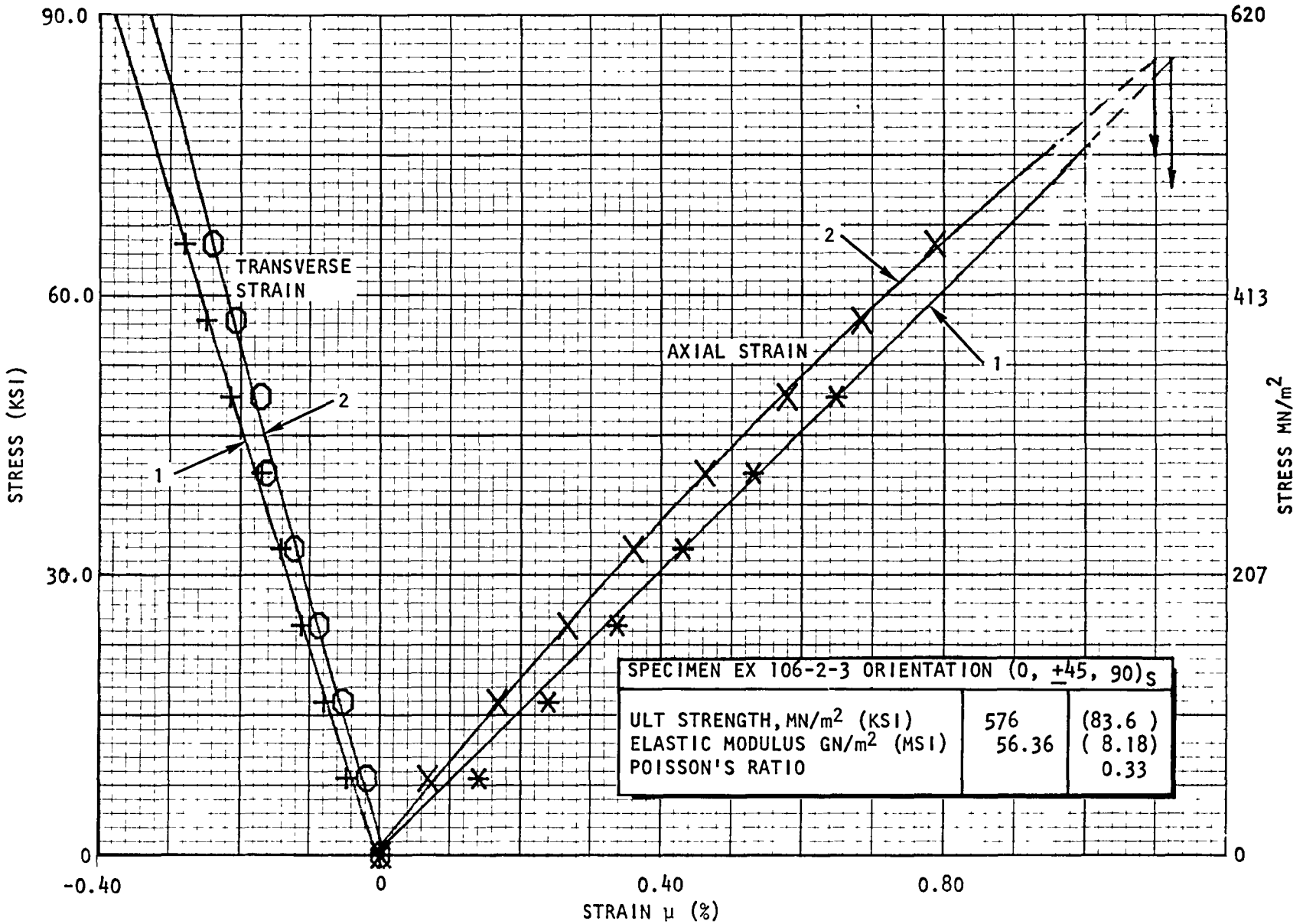


Figure C-25. LARC-160/Celion $(0, \pm 45, 90)_S$ Stress/Strain Curves at -132 C (-270 F)

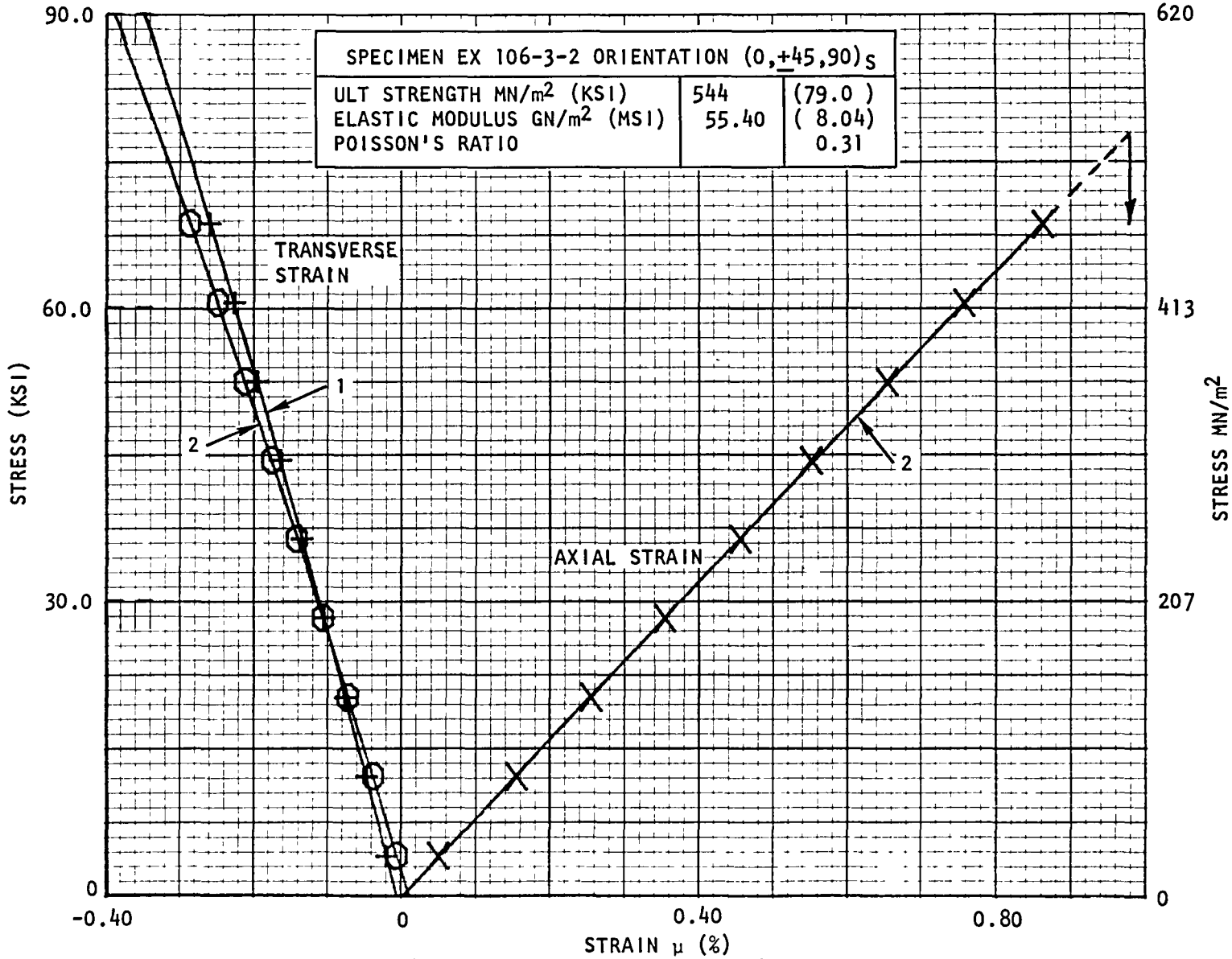


Figure C-26. LARC-160/Celion (0, ±45, 90)$_S$ Stress/Strain Curves at 204 C (400 F)

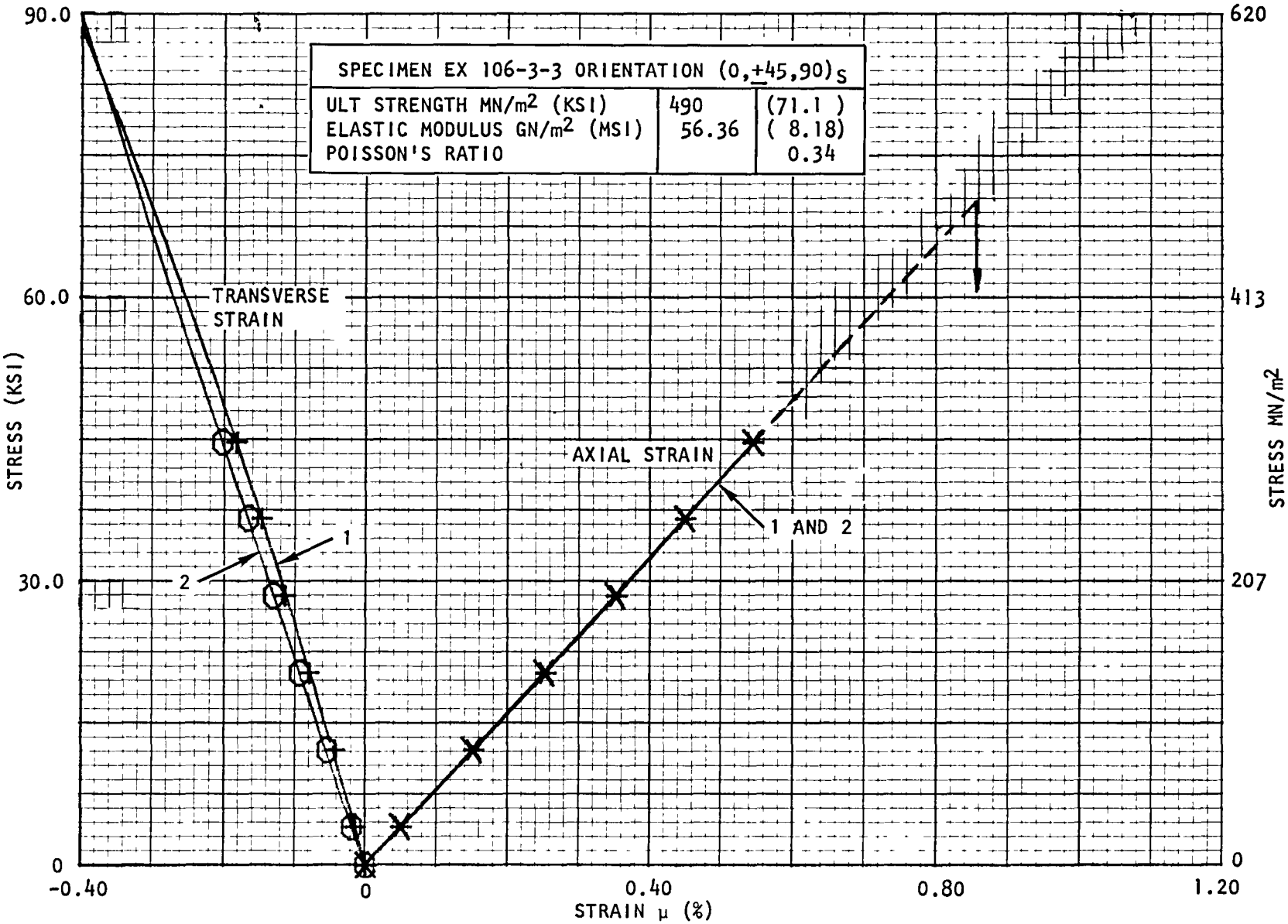


Figure C-27. LARC-160/Celion (0, ±45, 90)$_S$ Stress/Strain Curves at 204 C (400 F)

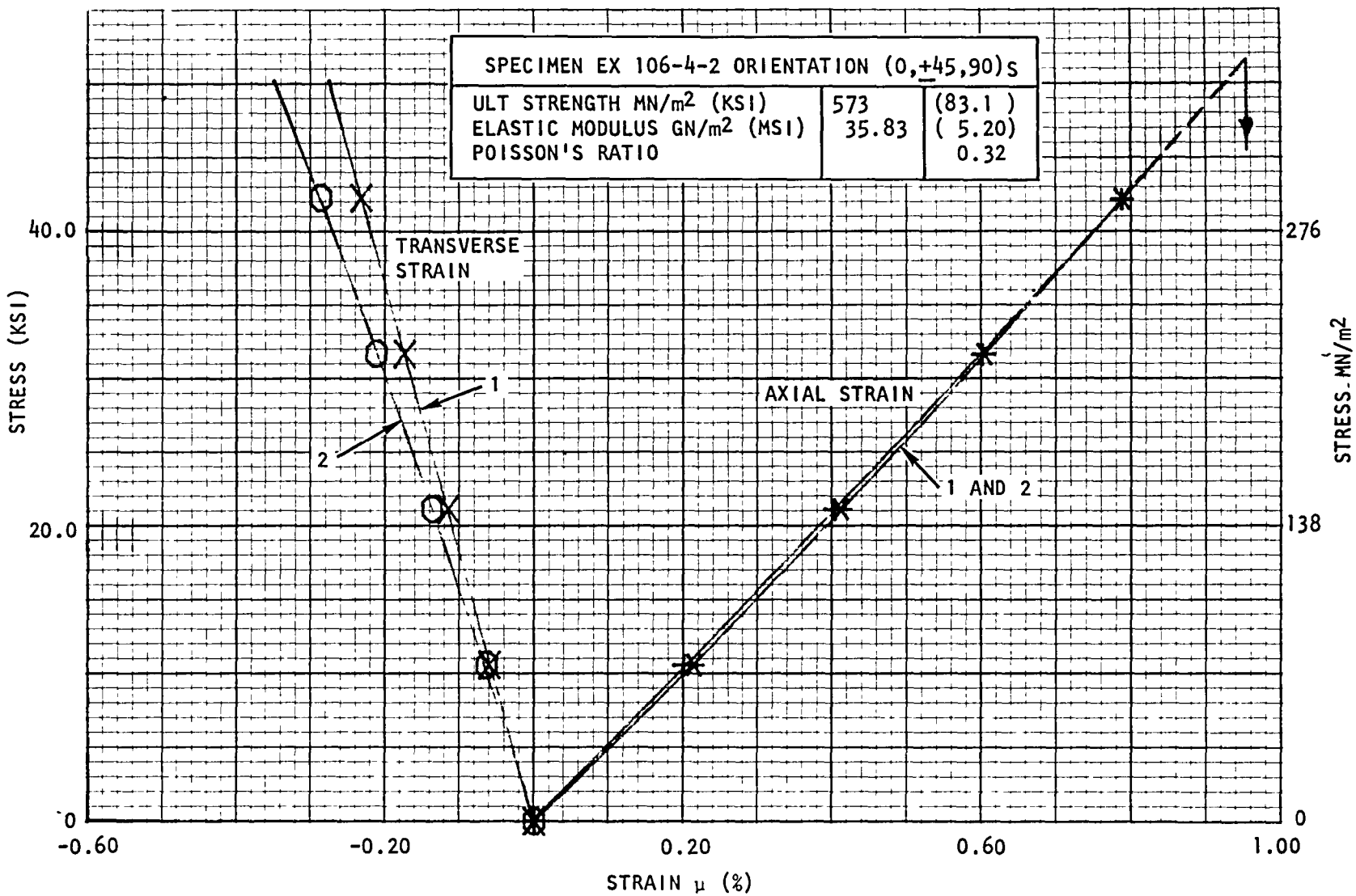


Figure C-28. LARC-160/Celion (0, ±45, 90)S Stress/Strain Curves at 316 C (600 F)

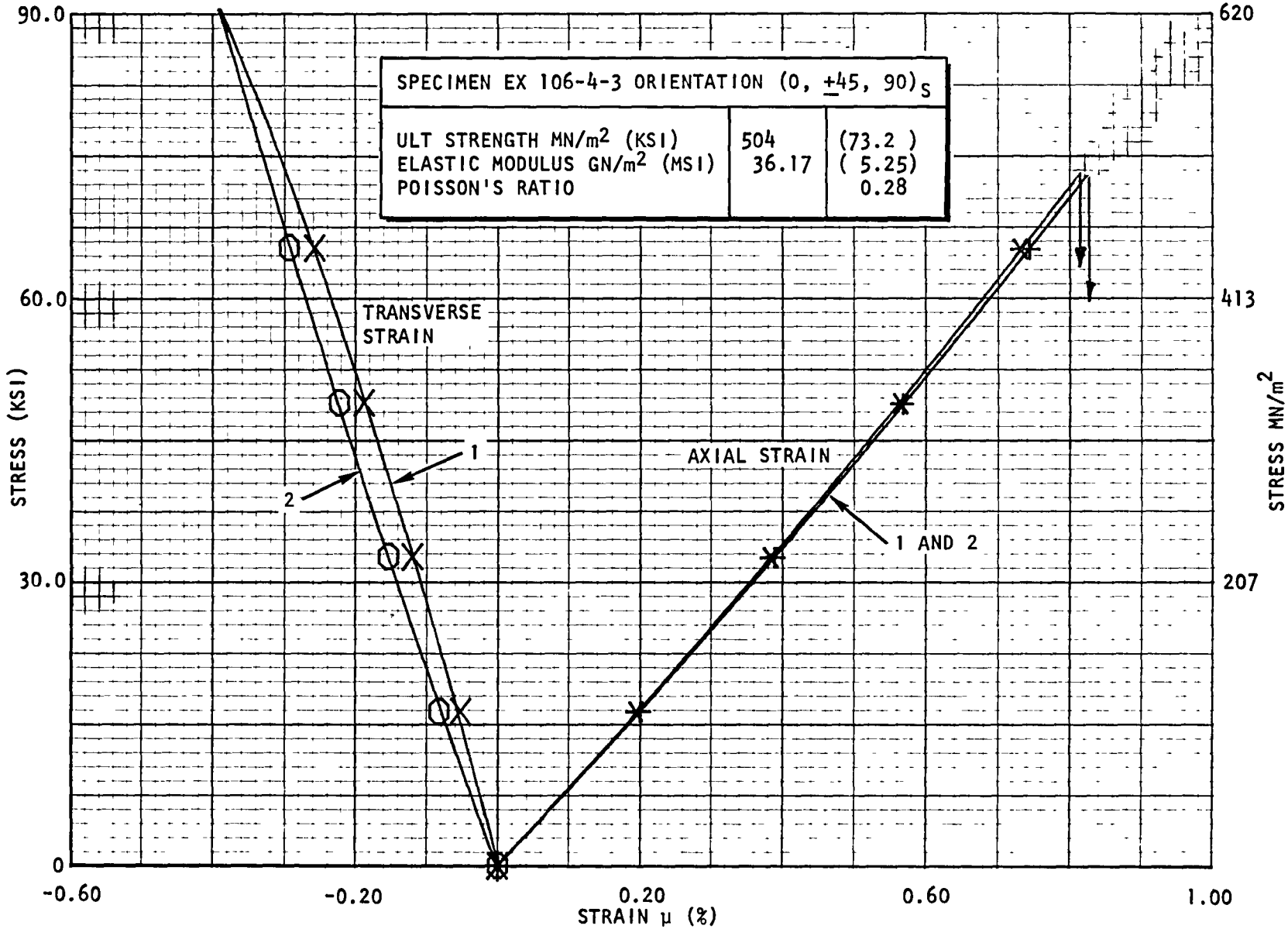


Figure C-29. LARC-160/Celion (0, ±45, 90)S Stress/Strain Curves at 316 C (600 F)

TENSION BEAM

CURVES

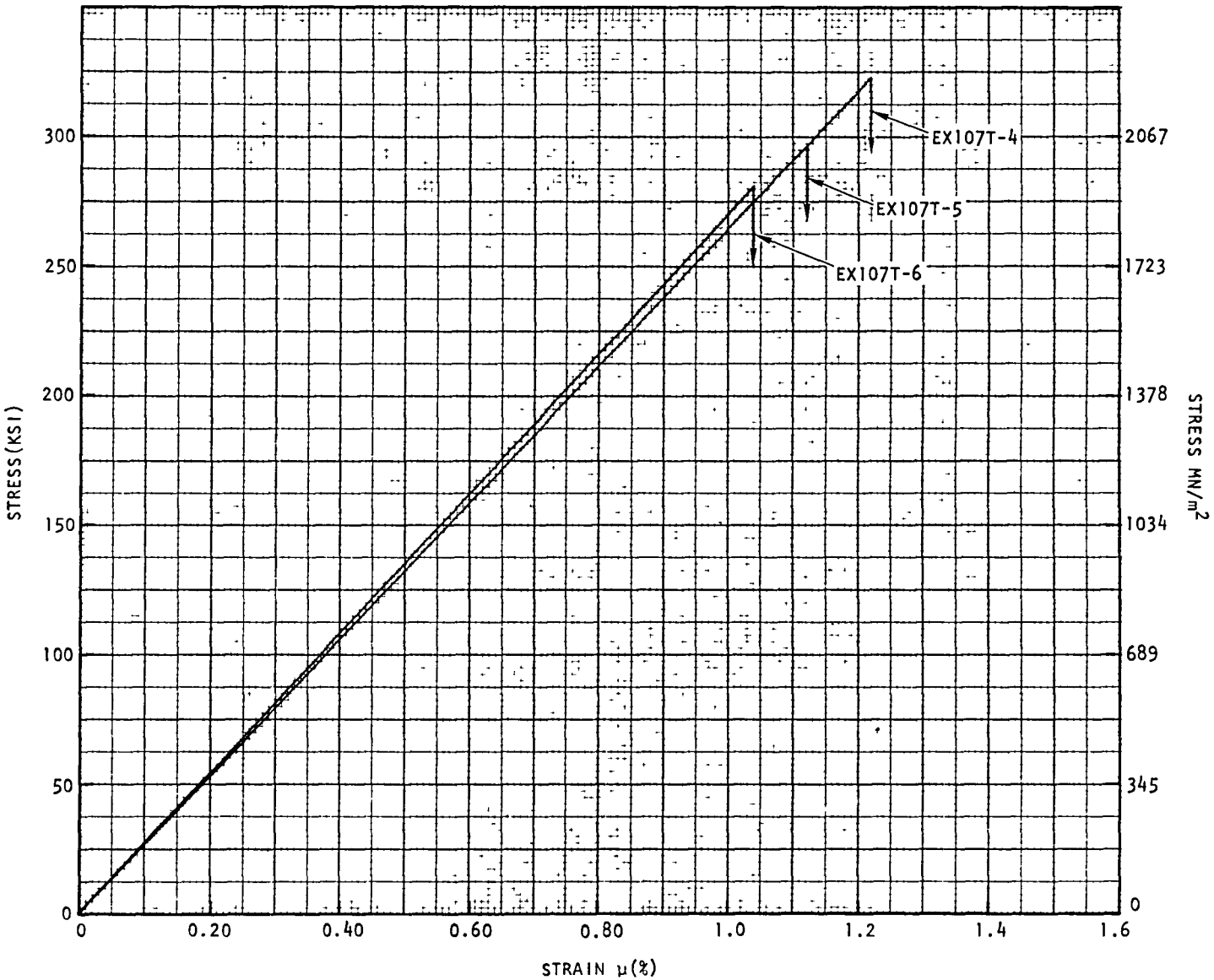


Figure C-30. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at—132 C(-270 F) Beam Test

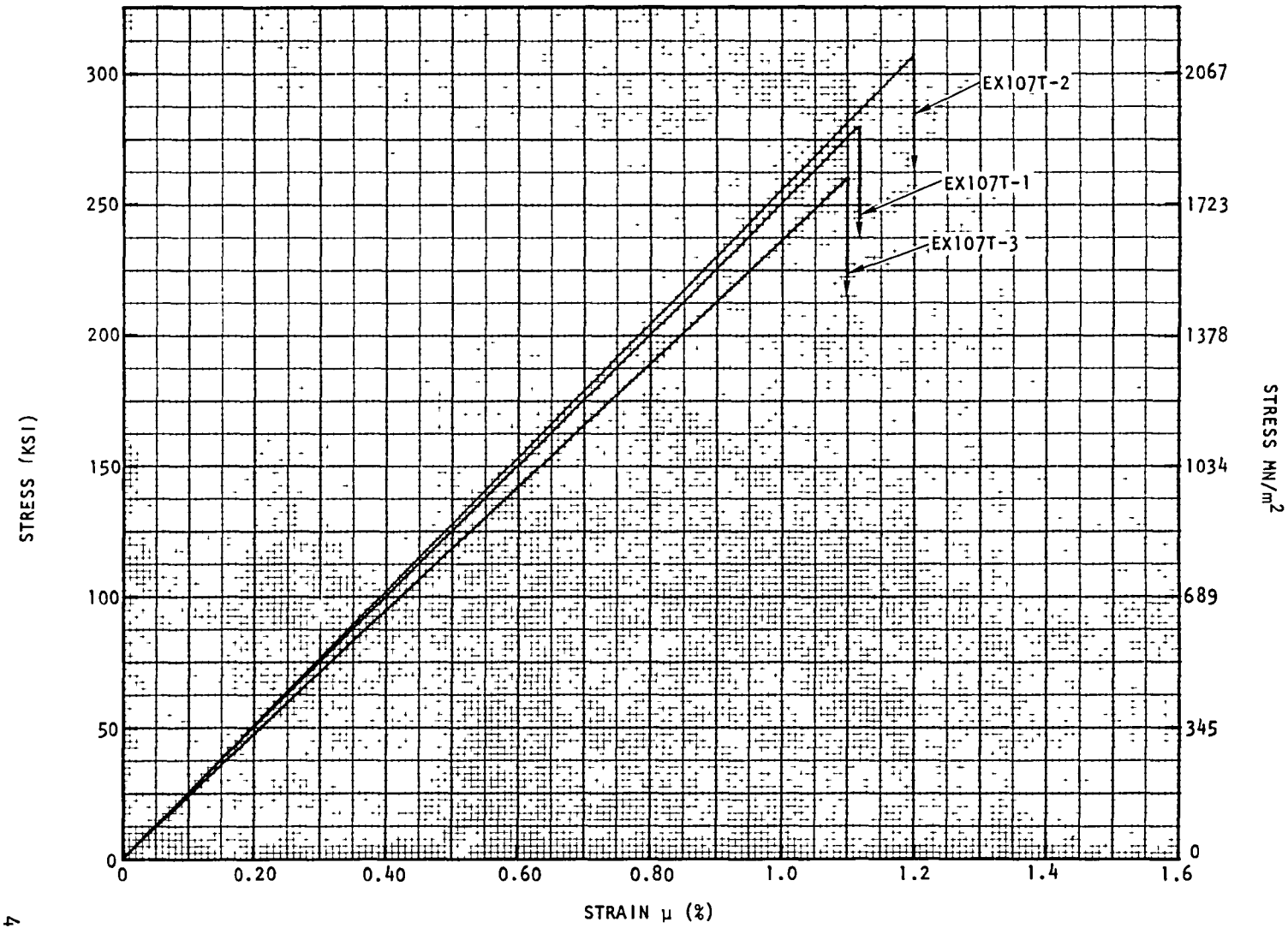


Figure C-31. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at RT—Beam Test

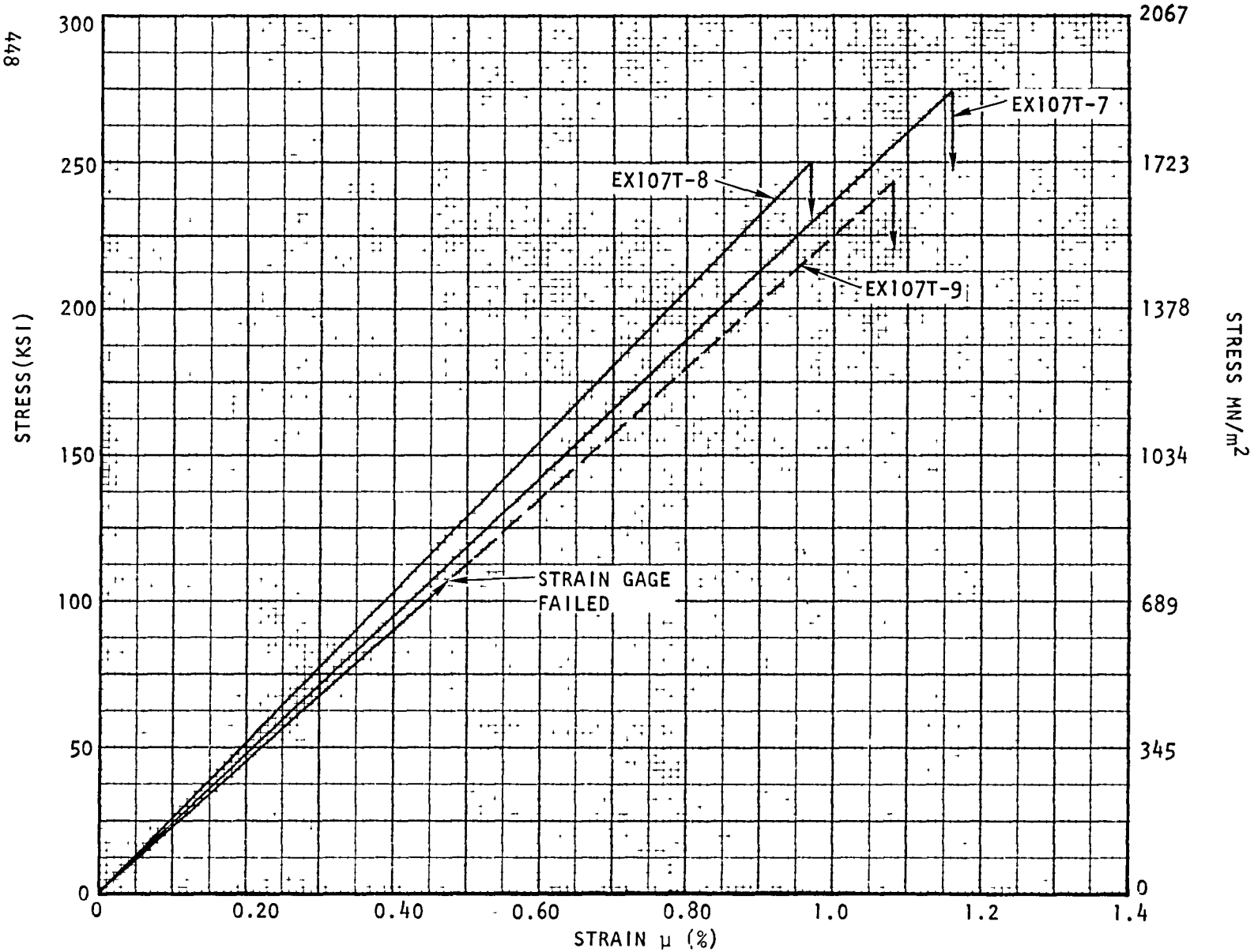


Figure C-32. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/celion Laminates at 204 C(400 F)—Beam Test

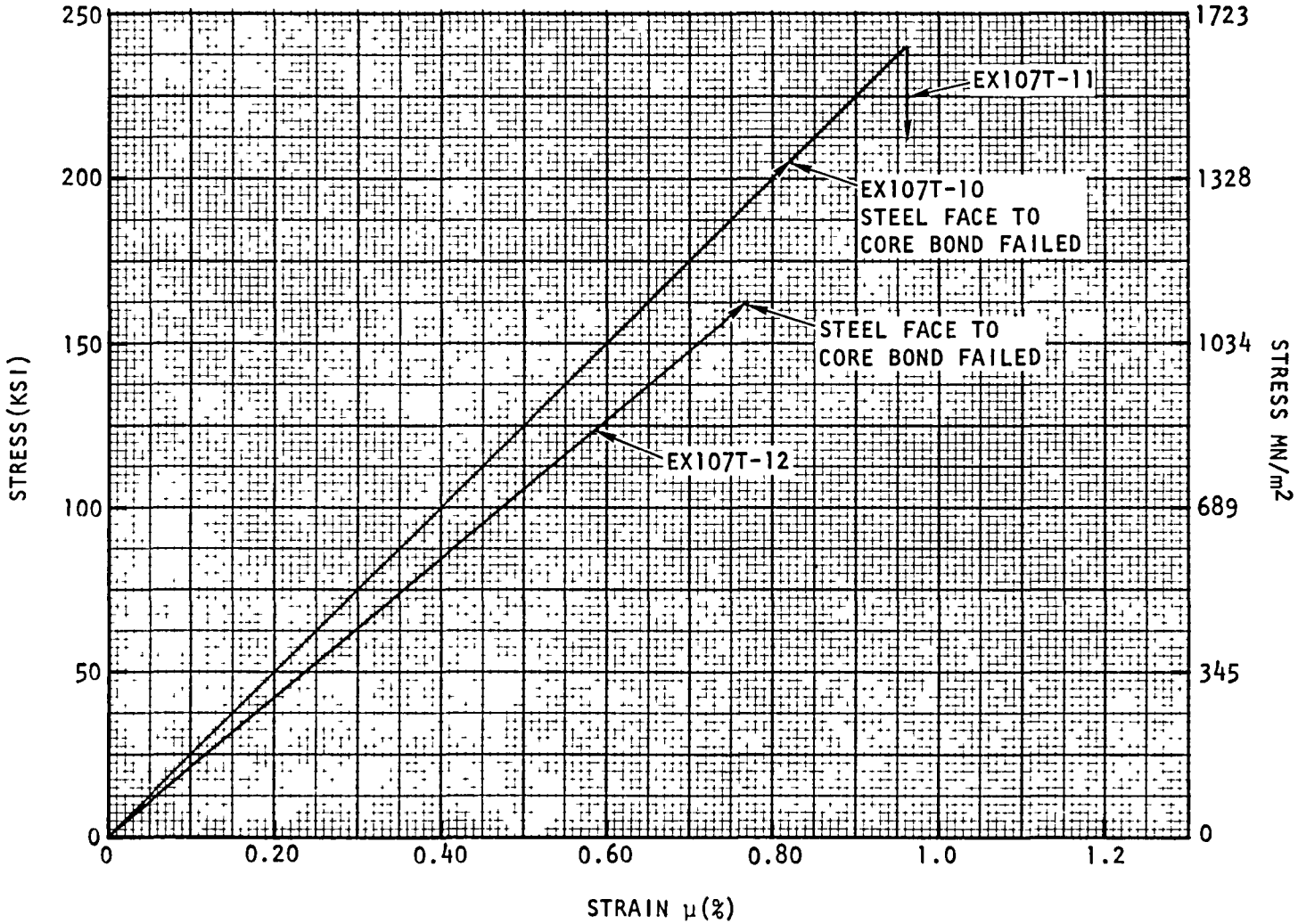


Figure C-33. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at 316 C (600 F) - Beam Test

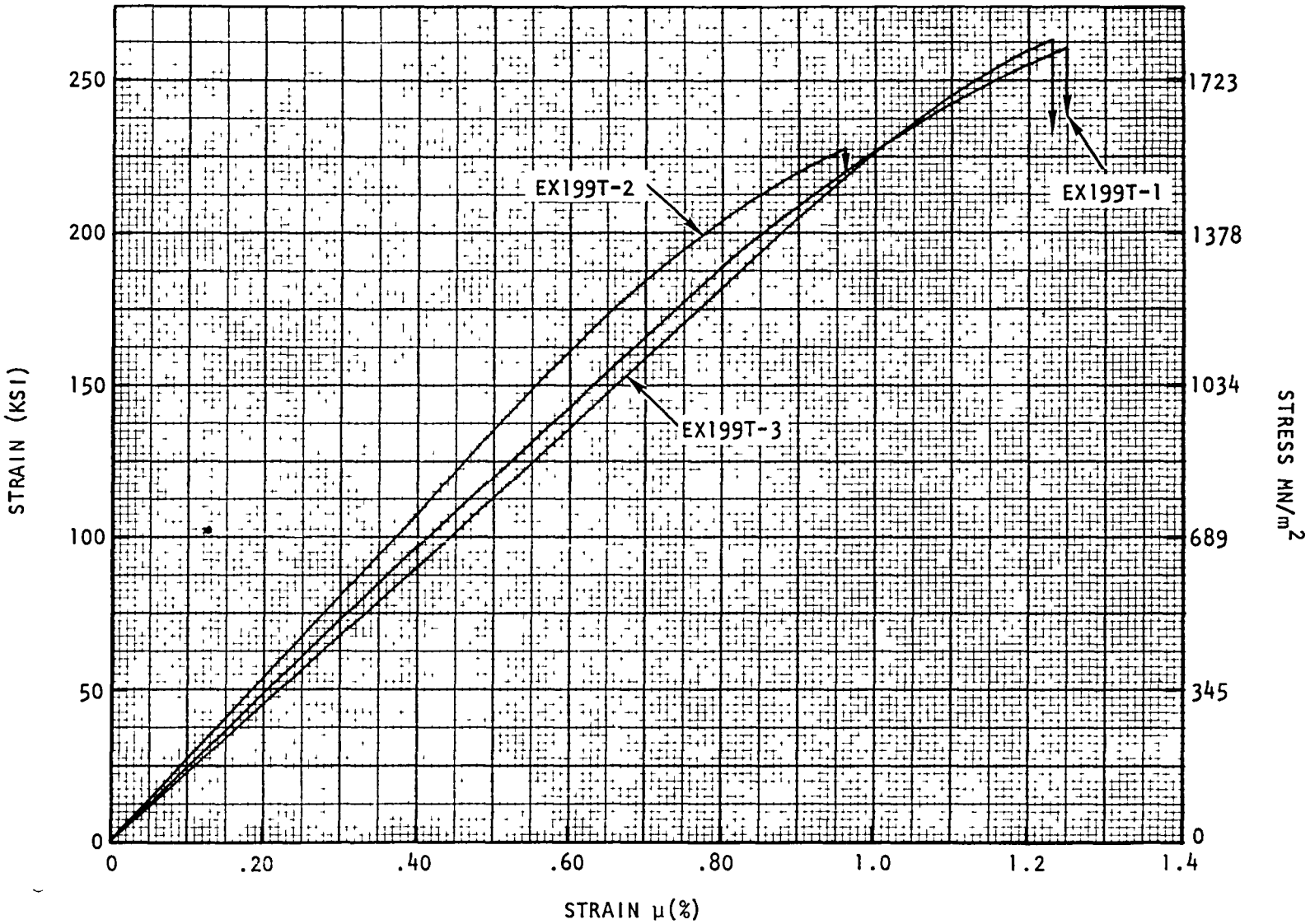


Figure C-34. Tensile Stress/Strain Characteristics of $(0)_5$, Parallel LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F), Beam Test at - 132 C (-270 F)

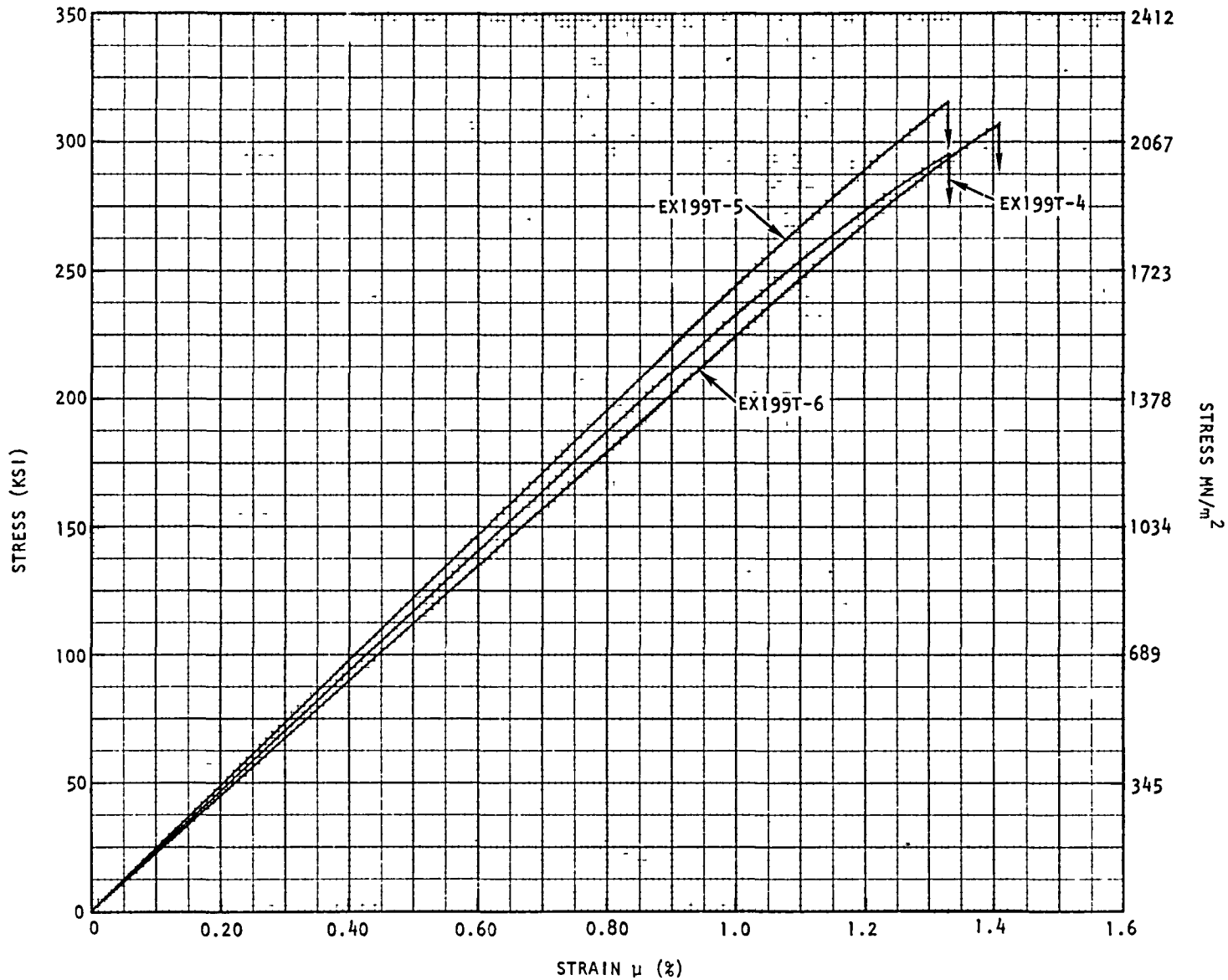


Figure C-35. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel Oriented LARC-160/Celion Laminates Aged 125 Hours at 316 C (600 F), Beam Test at RT

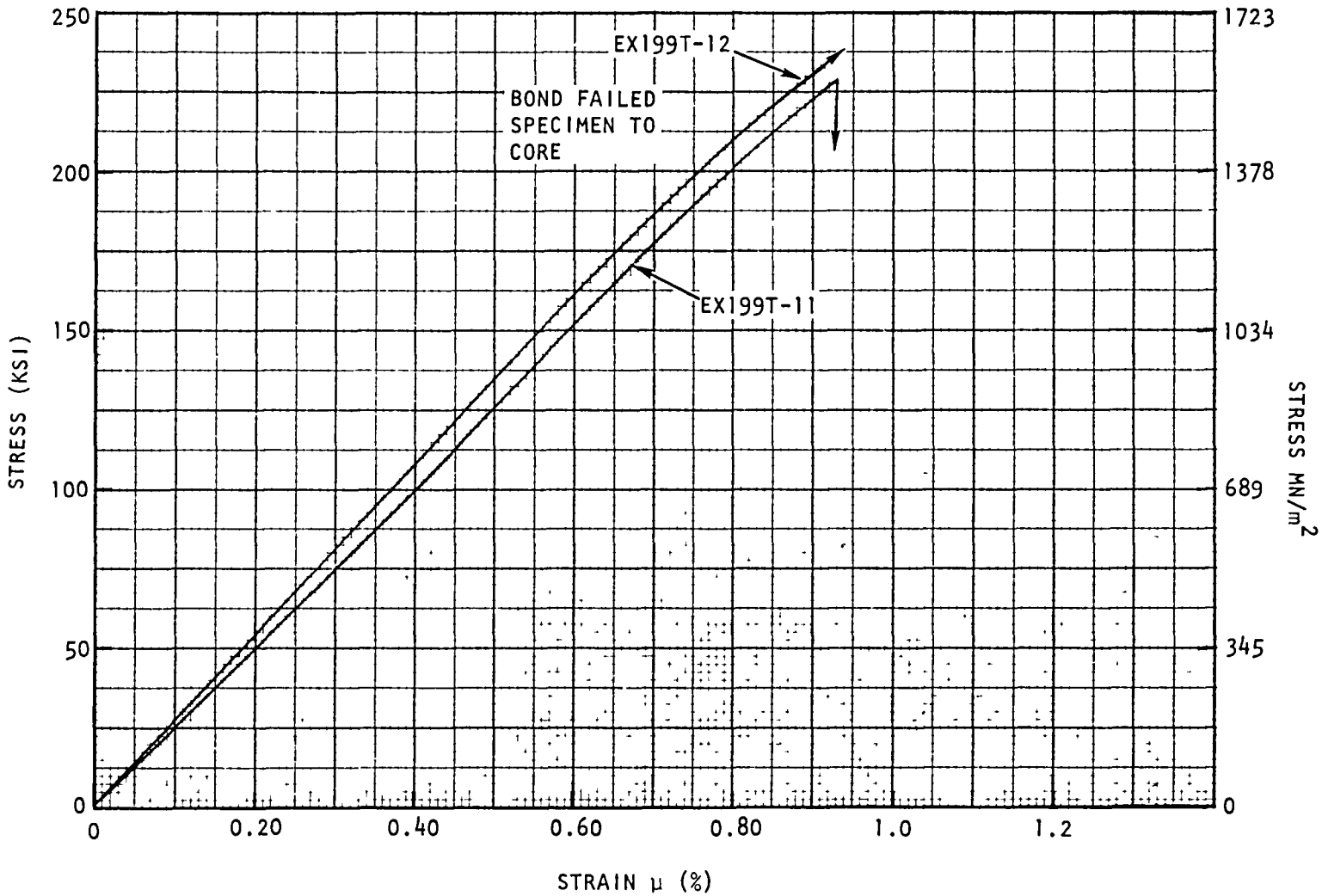


Figure C-36. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel Oriented LARC-160/Celion Laminates Aged 125 Hours at 316 C (600 F), Beam Test at 204 C (400 F)

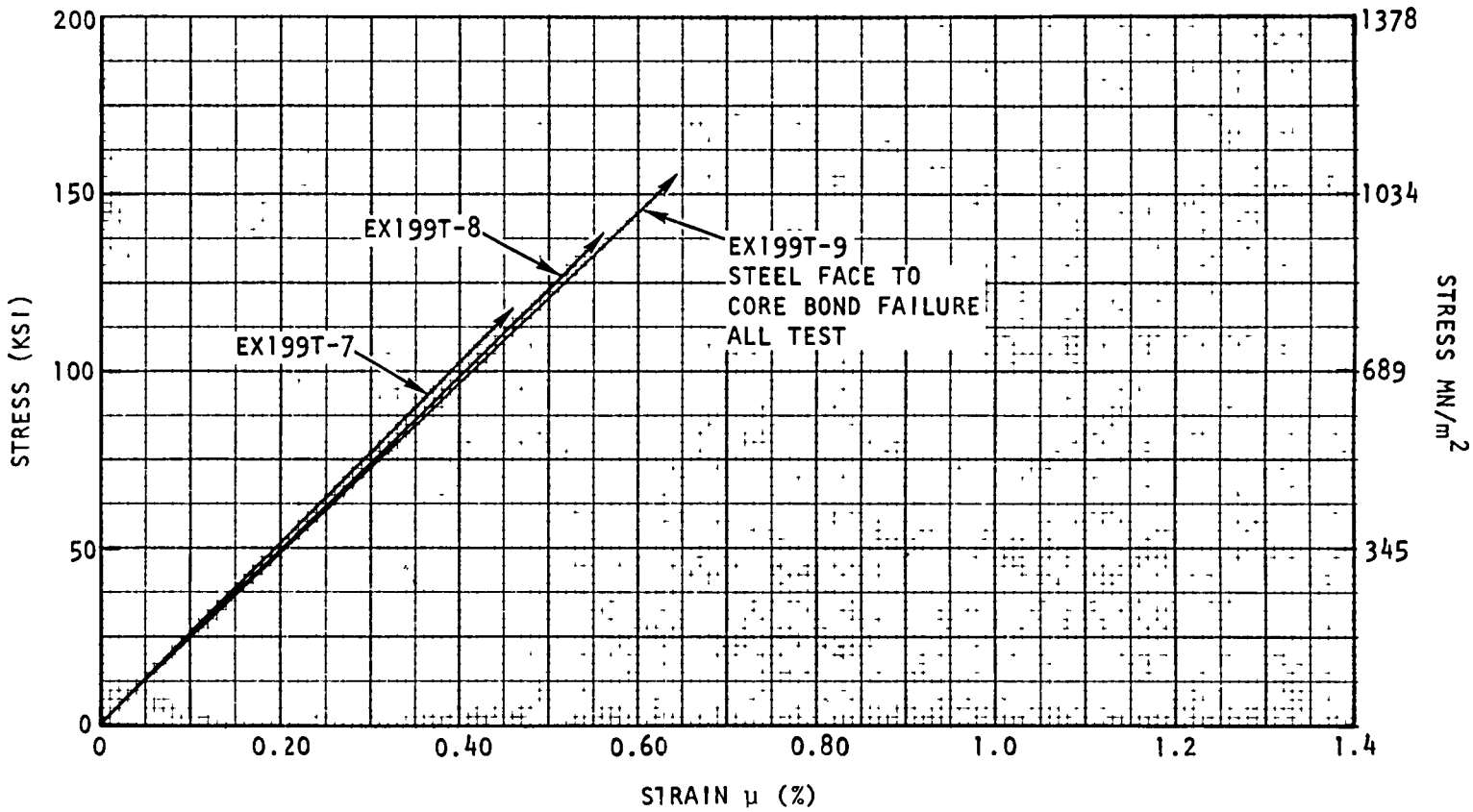


Figure C-37. Tensile Stress/Strain Characteristics of $(0)_5$ Parallel Oriented LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F), Beam Test at 316 C (600 F)

COMPRESSION BEAM

CURVES

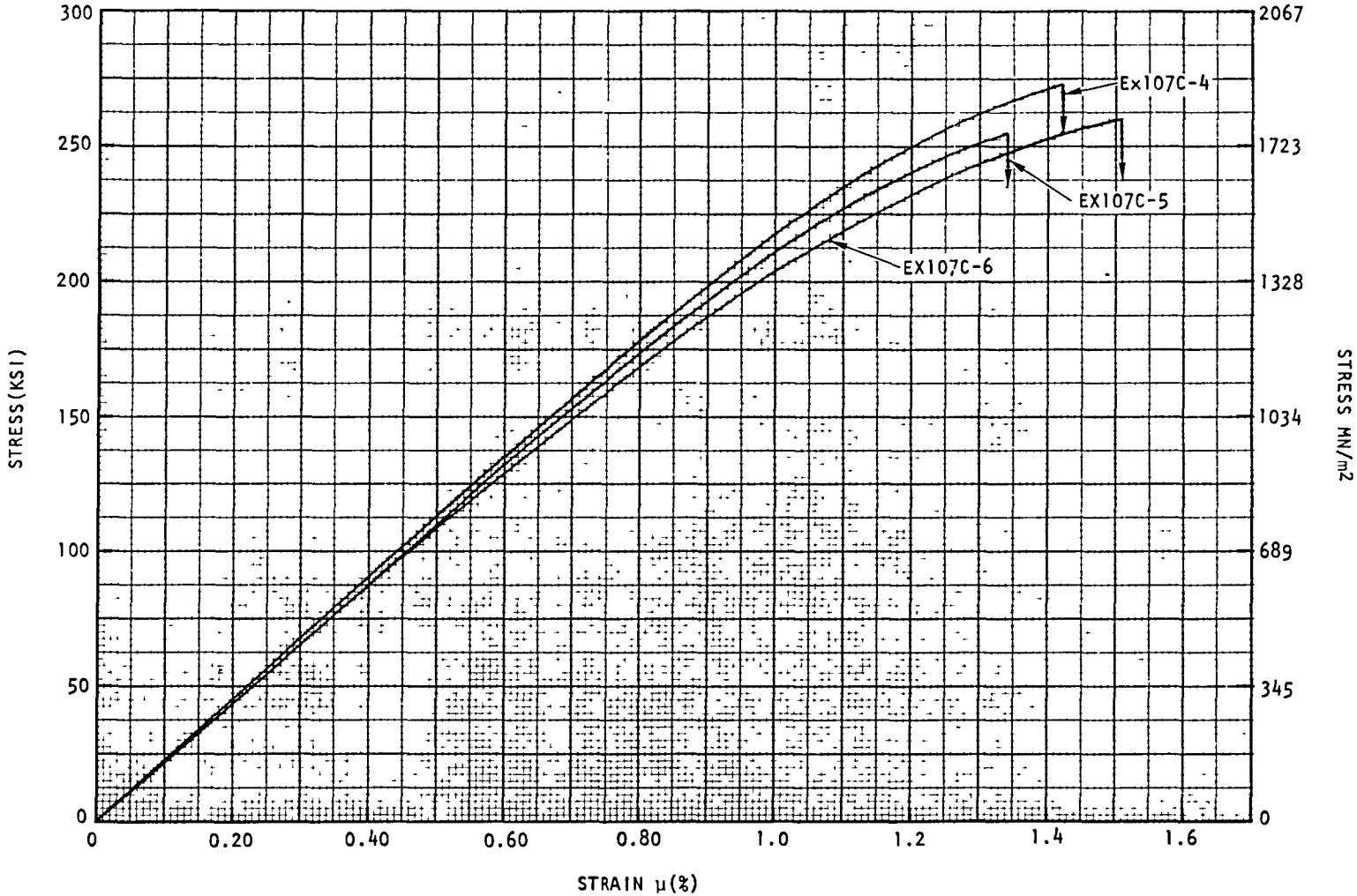


Figure C-38. Compression Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at—132 C(-270 F)—Beam Test

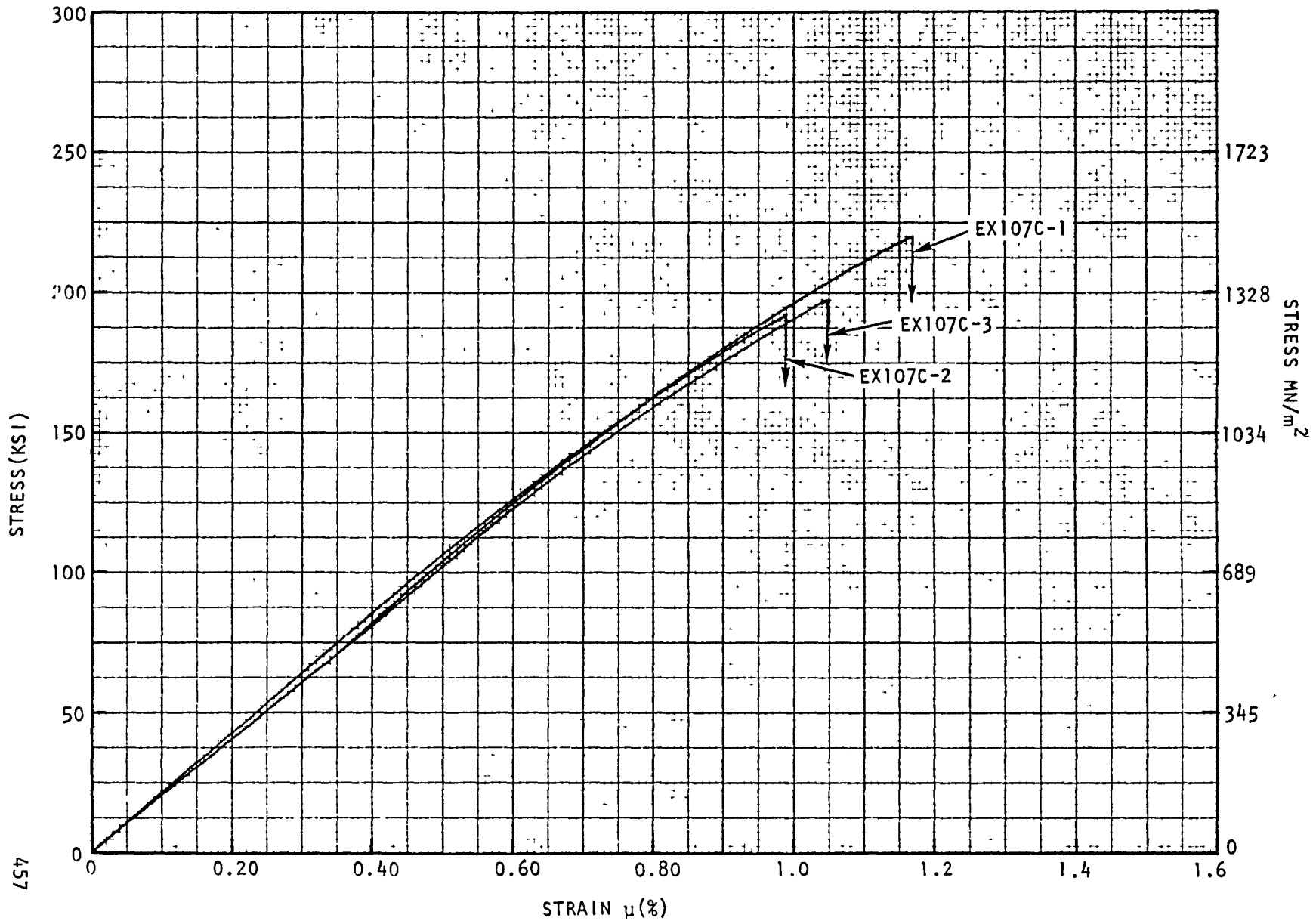


Figure C-39. Compression Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at RT—Beam Test

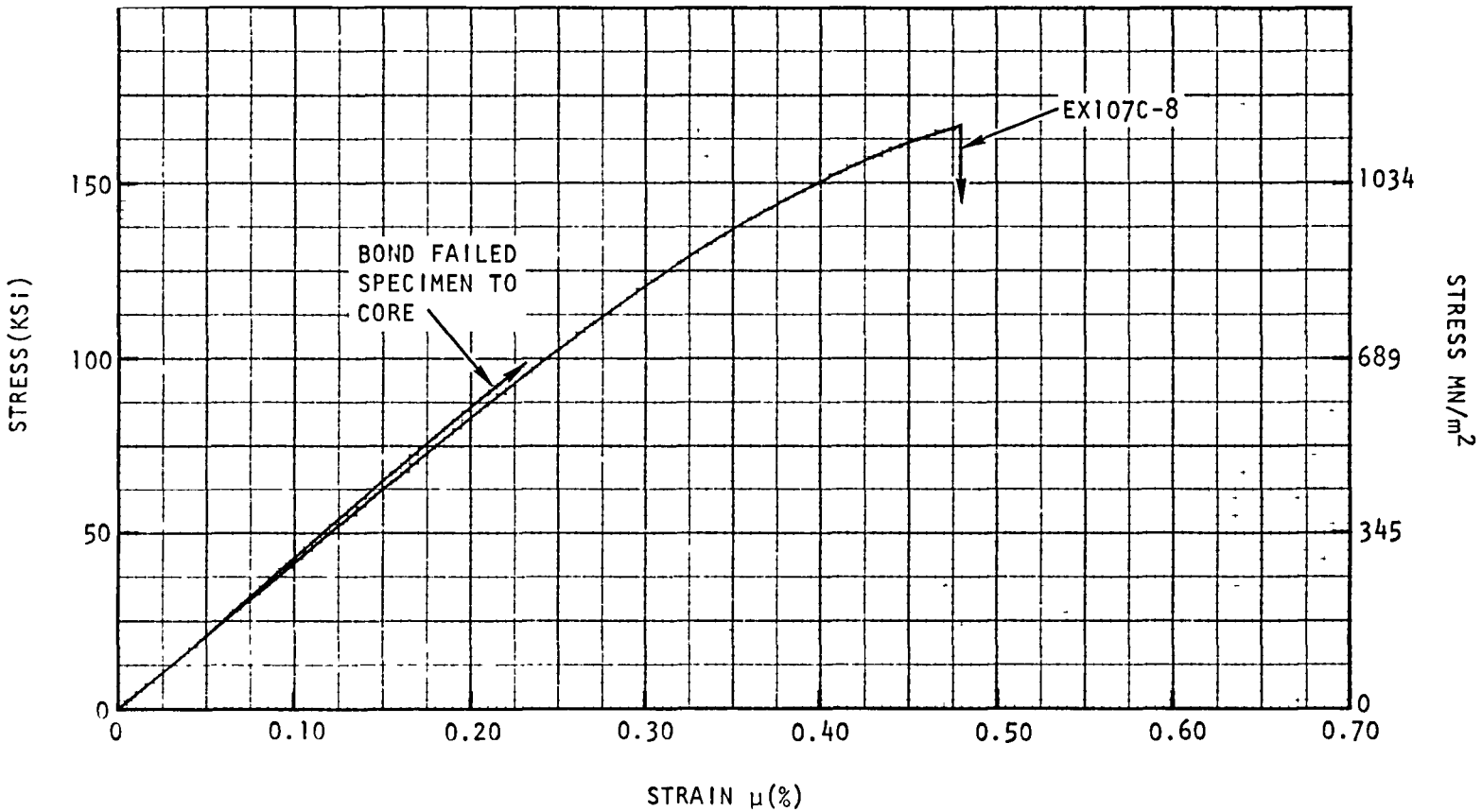


Figure C-40. Compressive Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at 204 C(400 F) Beam Test

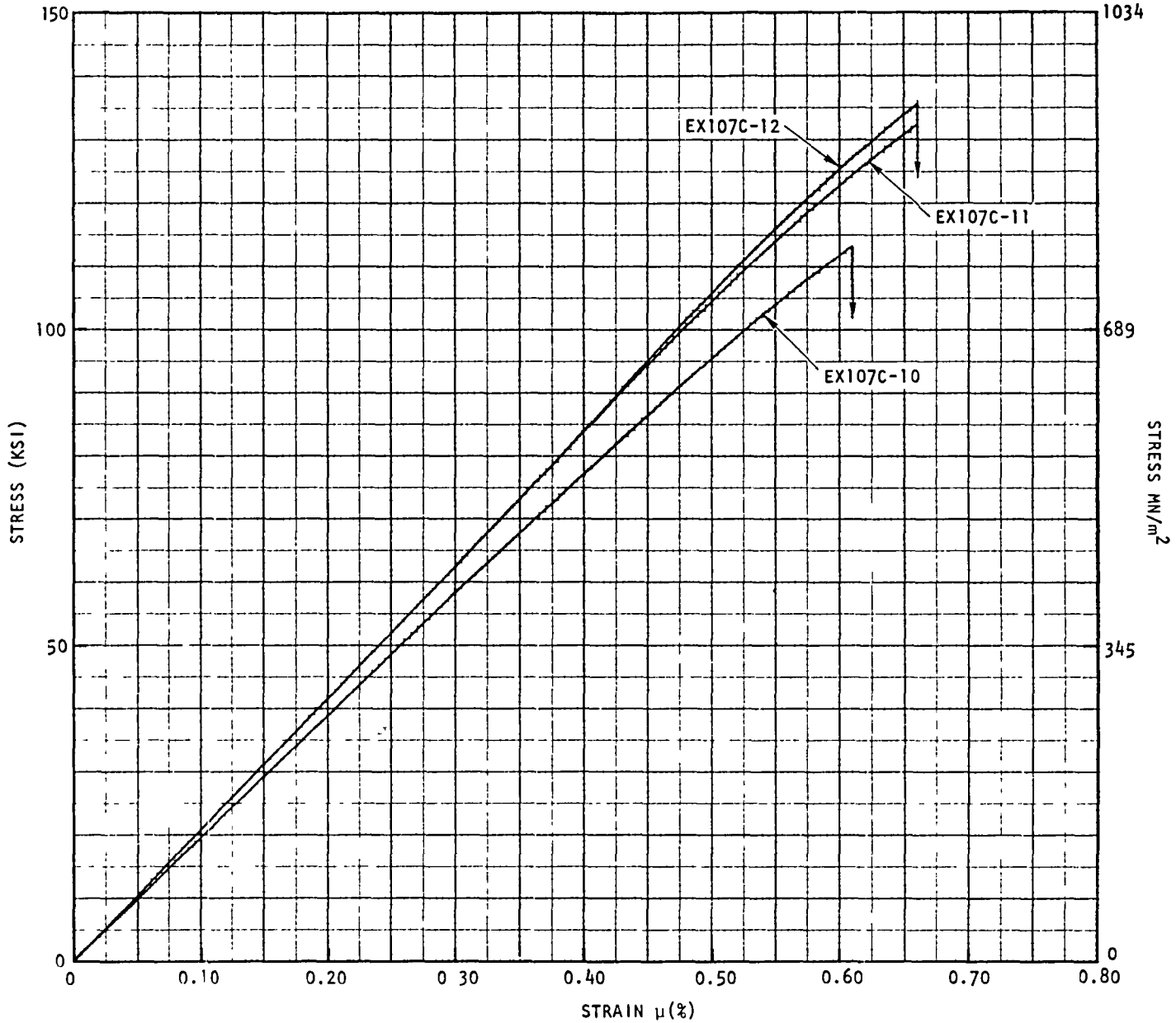


Figure C-41. Compression Stress/Strain Characteristics of $(0)_5$ Parallel LARC-160/Celion Laminates at 316 C (600 F)—Beam Test

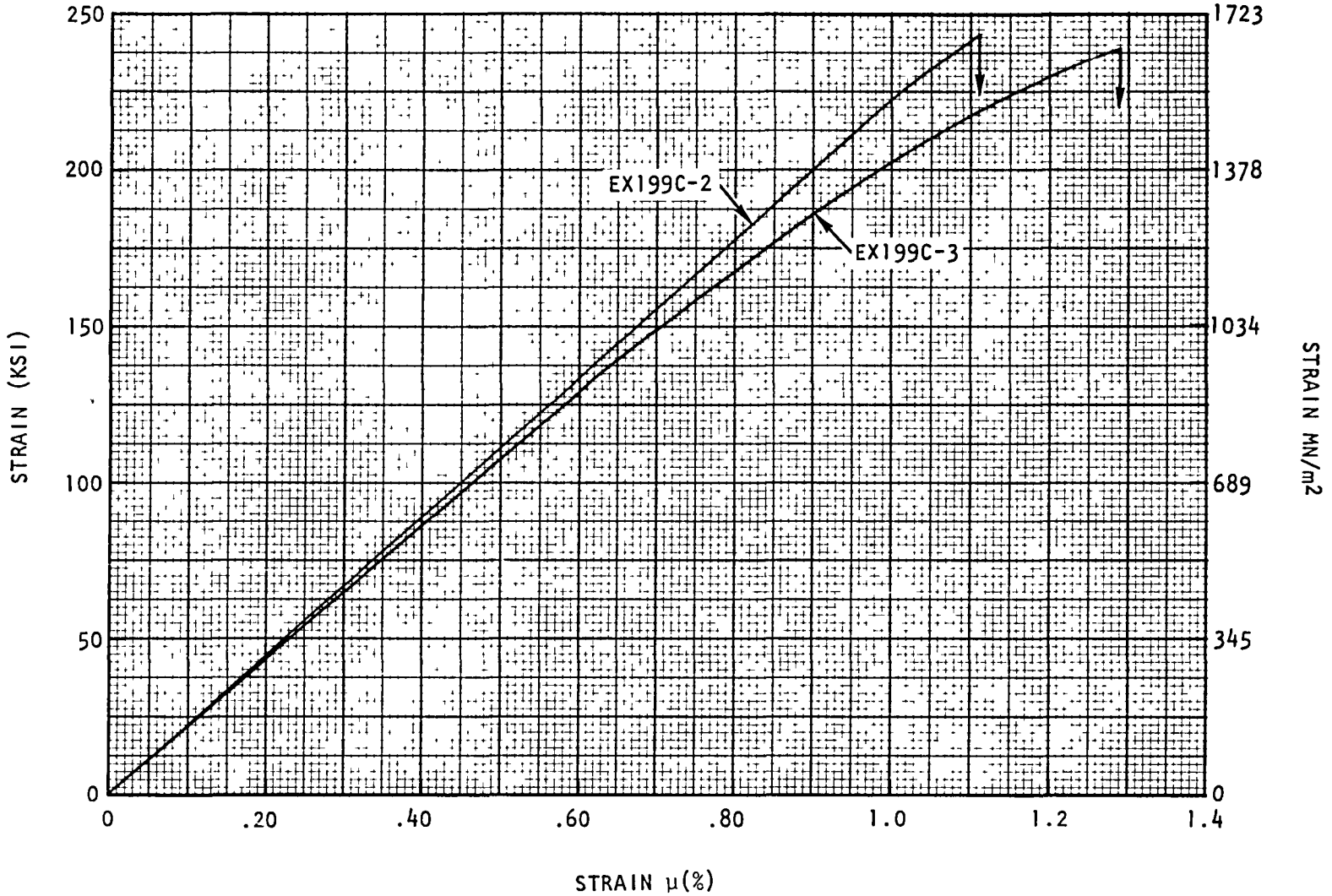


Figure C-42. Compression Stress/Strain Characteristics of $(0)_5$, Parallel LARC-160/Celion Laminates aged 125 Hours at 316 C (600 F), Beam Test at - 132 C (-270 F)

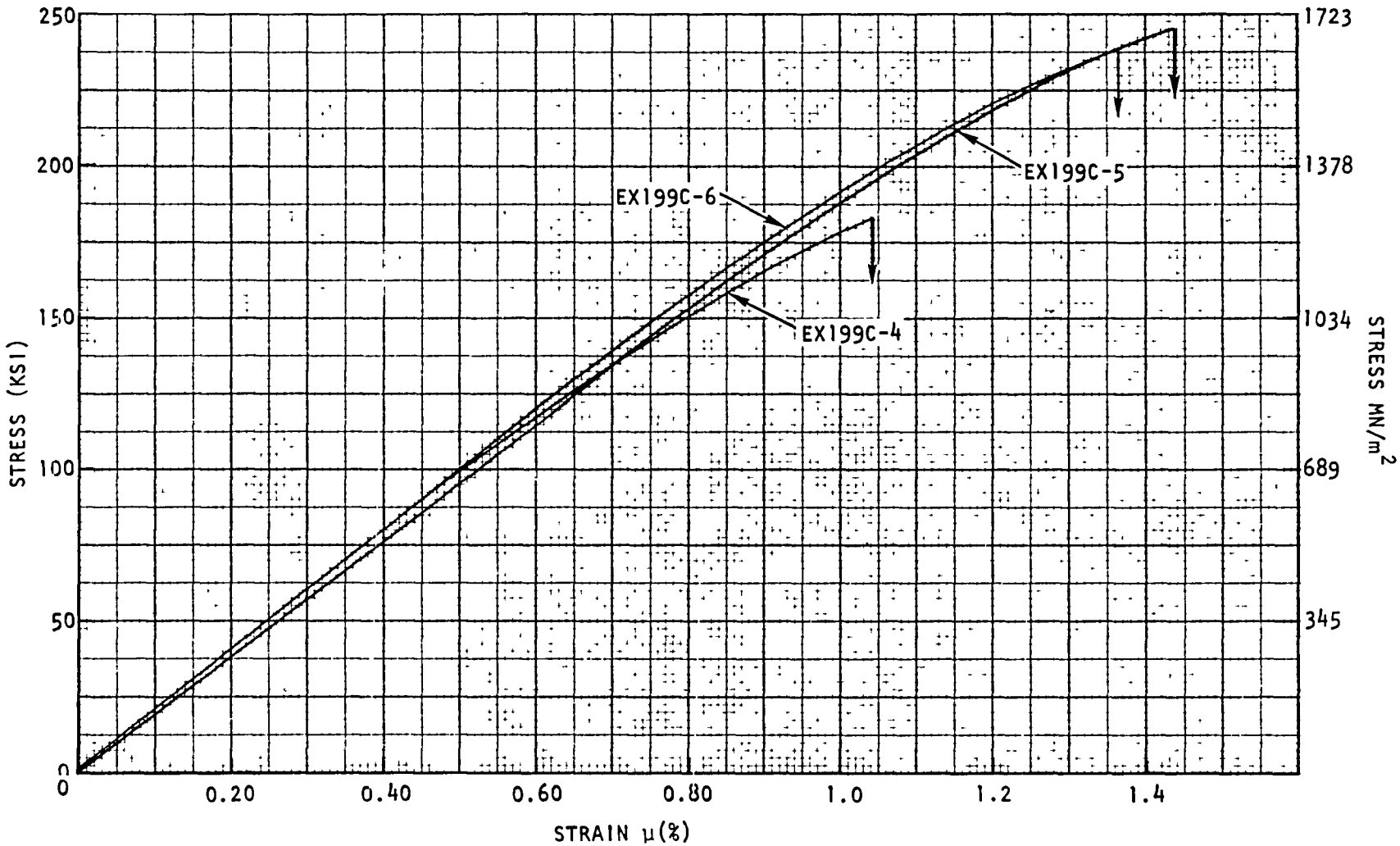


Figure C-43. Compression Stress/Strain Characteristics of $(0)_5$, Parallel Oriented LARC-160/ Celion Laminates Aged 125 Hours at 316 C (600 F), Beam Test at RT

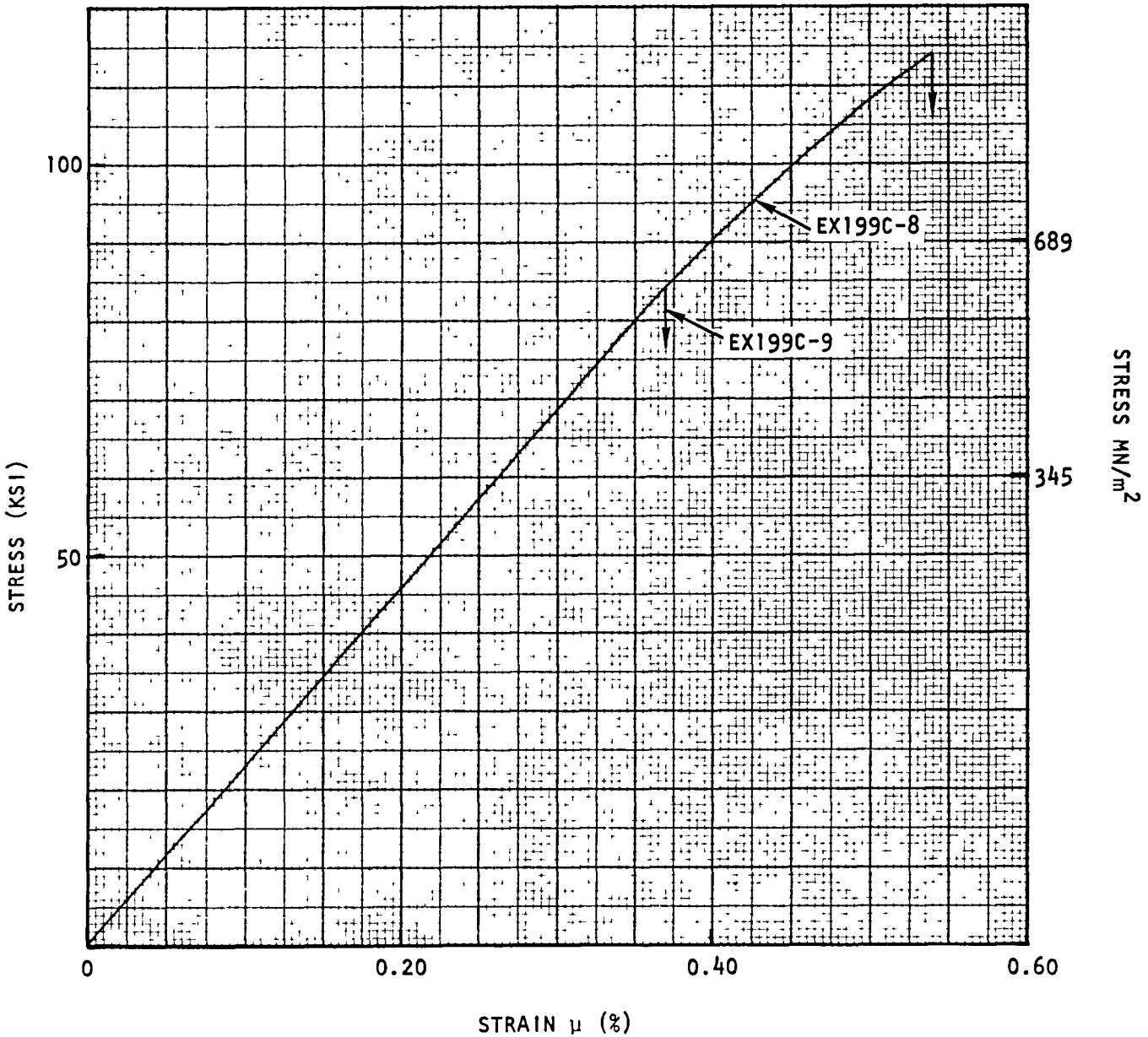


Figure C-44. Compression Stress/Strain Characteristics of $(0)_5$ Parallel LARC160/ Laminates Aged for 125 Hours at 316 C (600 F) - Beam Test at 316 C (600 F)

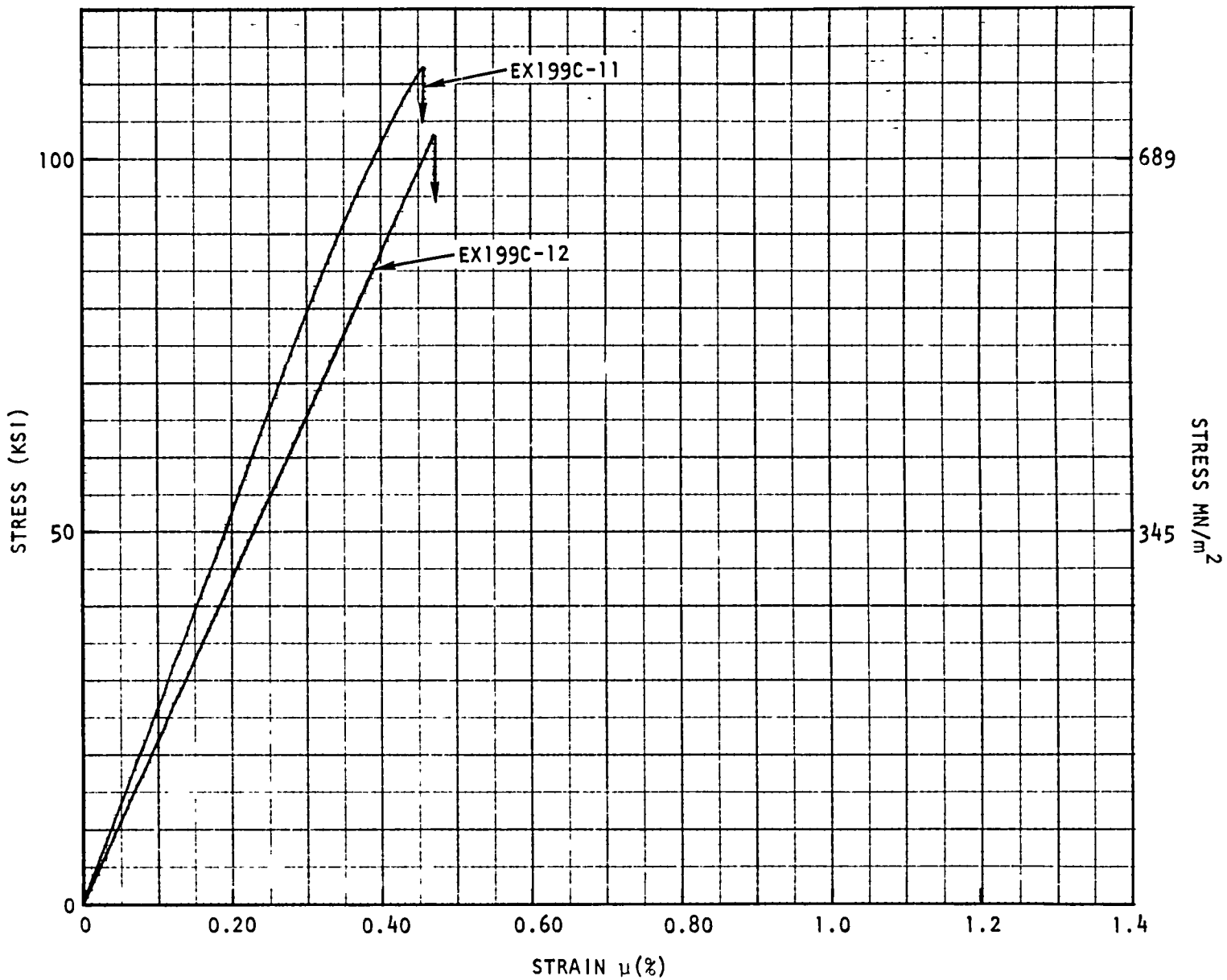


Figure C-45. Compression Stress/Strain Characteristics of $(0)_5$, Parallel Oriented LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F), Beam Test at 204 C (400 F)

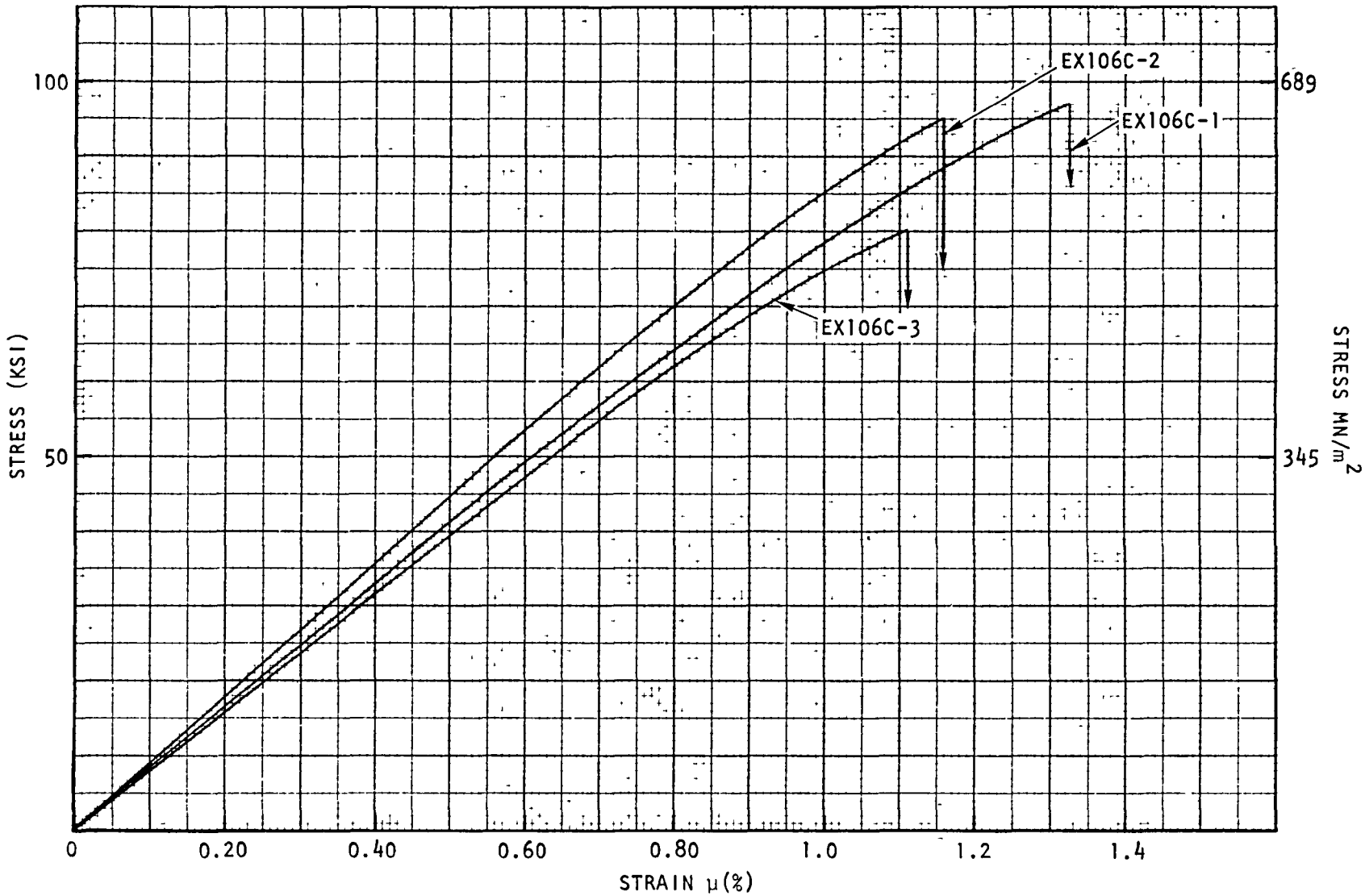


Figure C-46. Compression Stress/Strain Characteristics of $(0, \pm 45, 90)_S$ LARC-160/Celion Laminates at RT—Beam Test

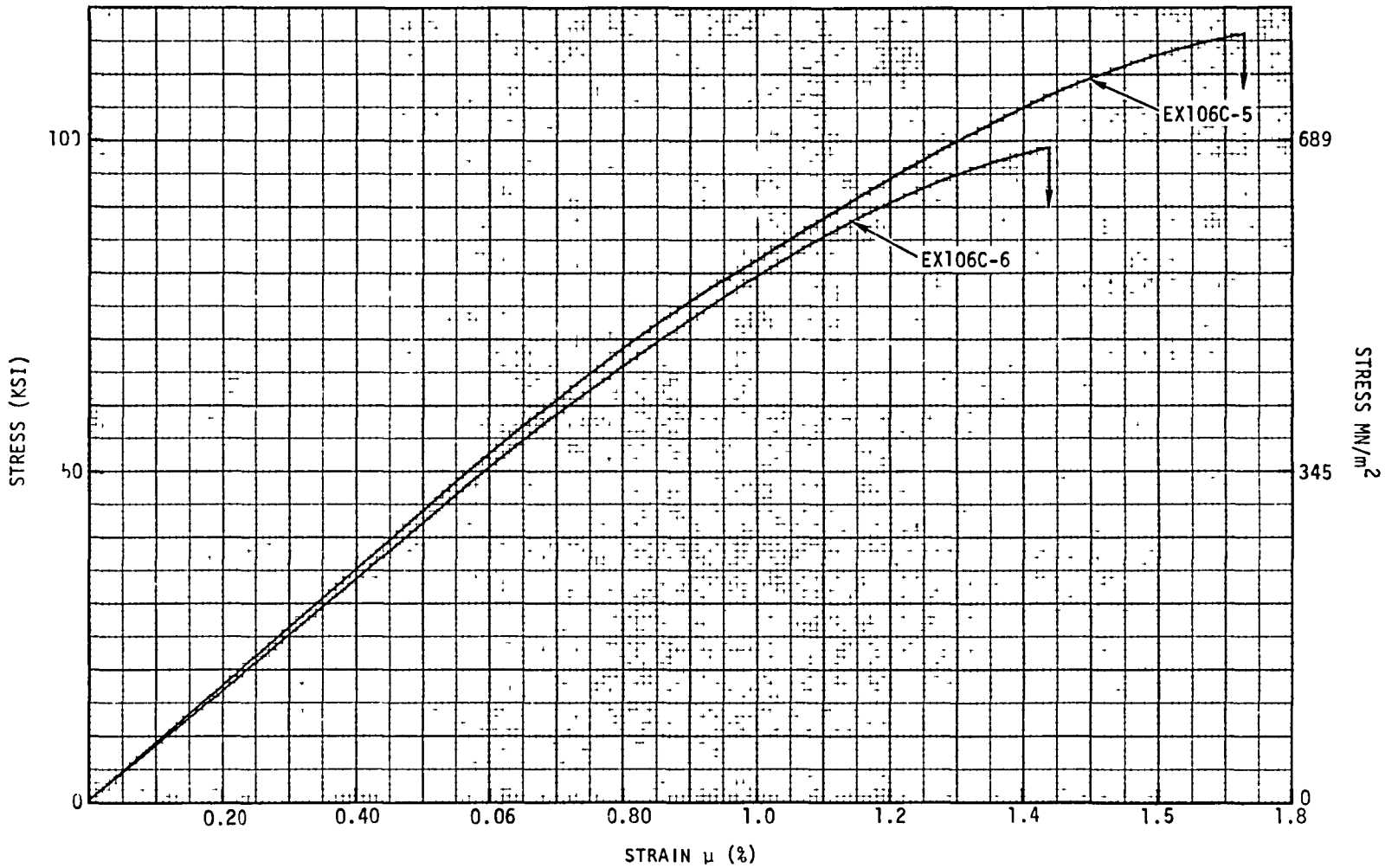


Figure C-47. Compression Stress/Strain Characteristics of $(0,\pm45,90)_S$ LARC-160/Celion Laminates at—132(-270 F)—Beam Test

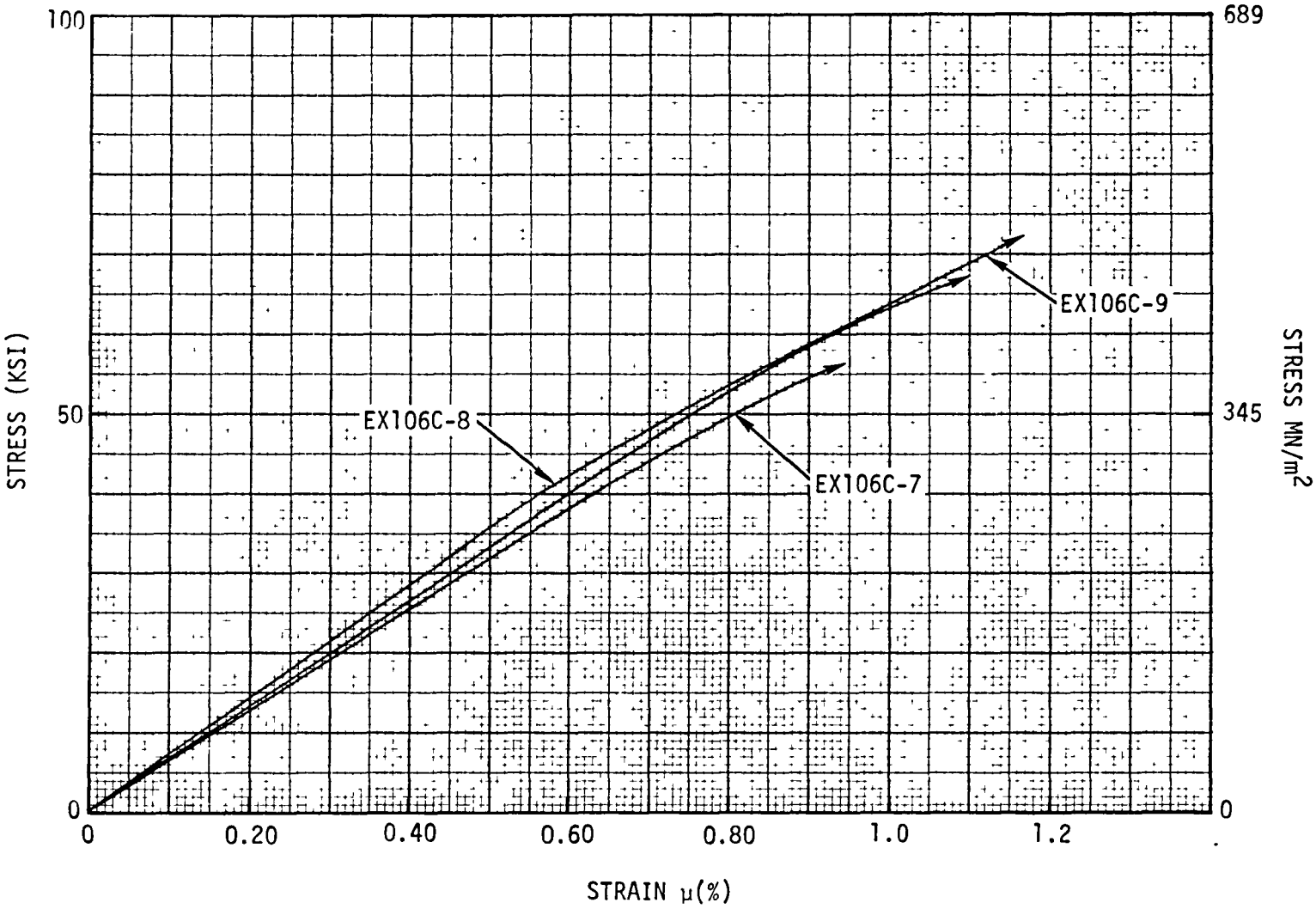


Figure C-48. Compression Stress/Strain Characteristics of $(0,\pm45,90)_S$ LARC-160/Celion Laminates at 204 C (400 F)—Beam Test

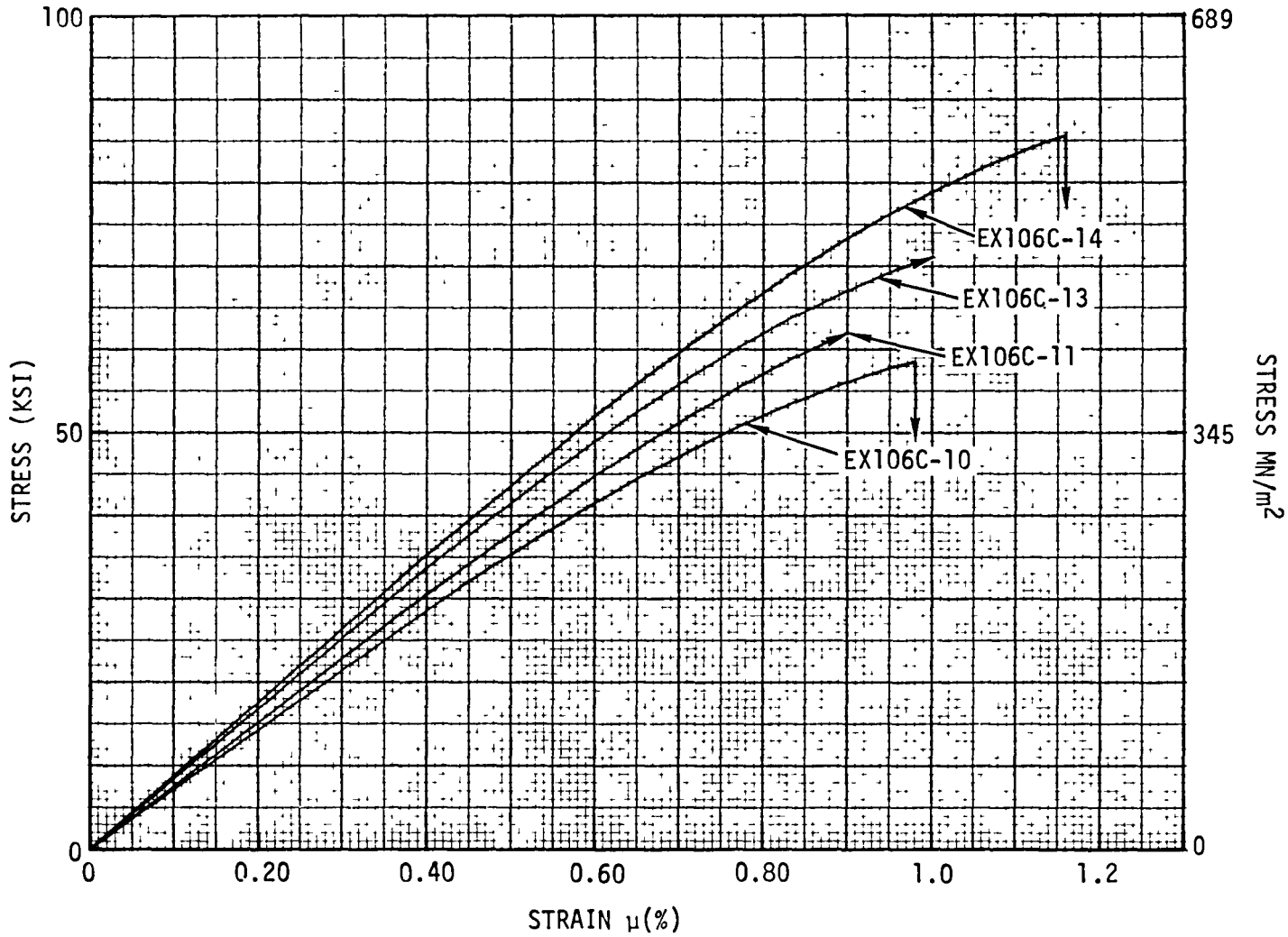


Figure C-49. Compression Stress/Strain Characteristics of $(0,\pm45,90)_S$ LARC-160/Celion Laminates at 316 C (600 F)—Beam Test

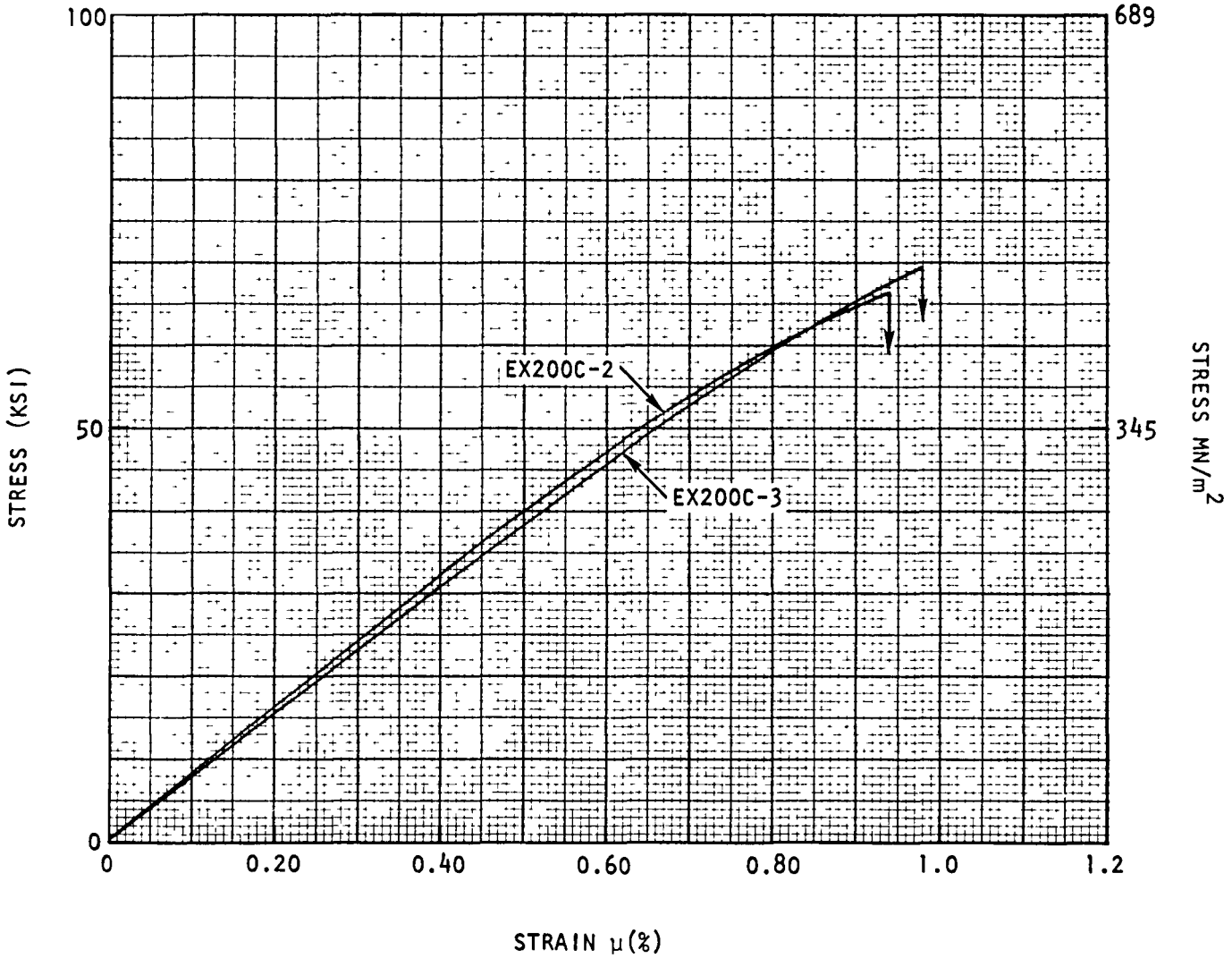


Figure C-50. Compression Stress Strain Characteristics of $(0, \pm 45, 90)_S$ LARC-160/Celion Laminated Aged for 125 Hours at 316 C (600 F), Beam Test at -132 C (-270 F)

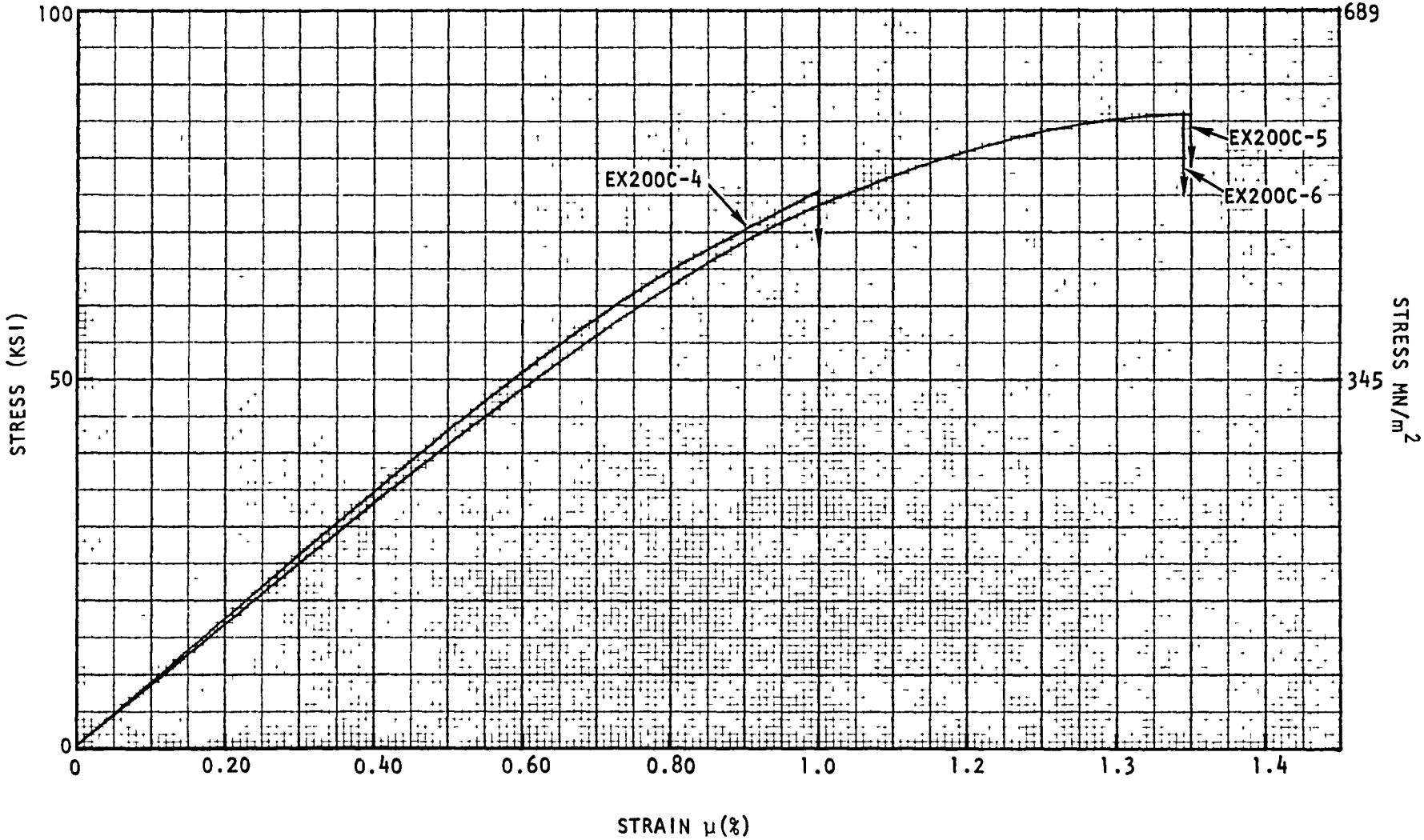


Figure C-51. Compression Stress/Strain Characteristics of (0, ±45, 90)S LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F) Beam Test at RT

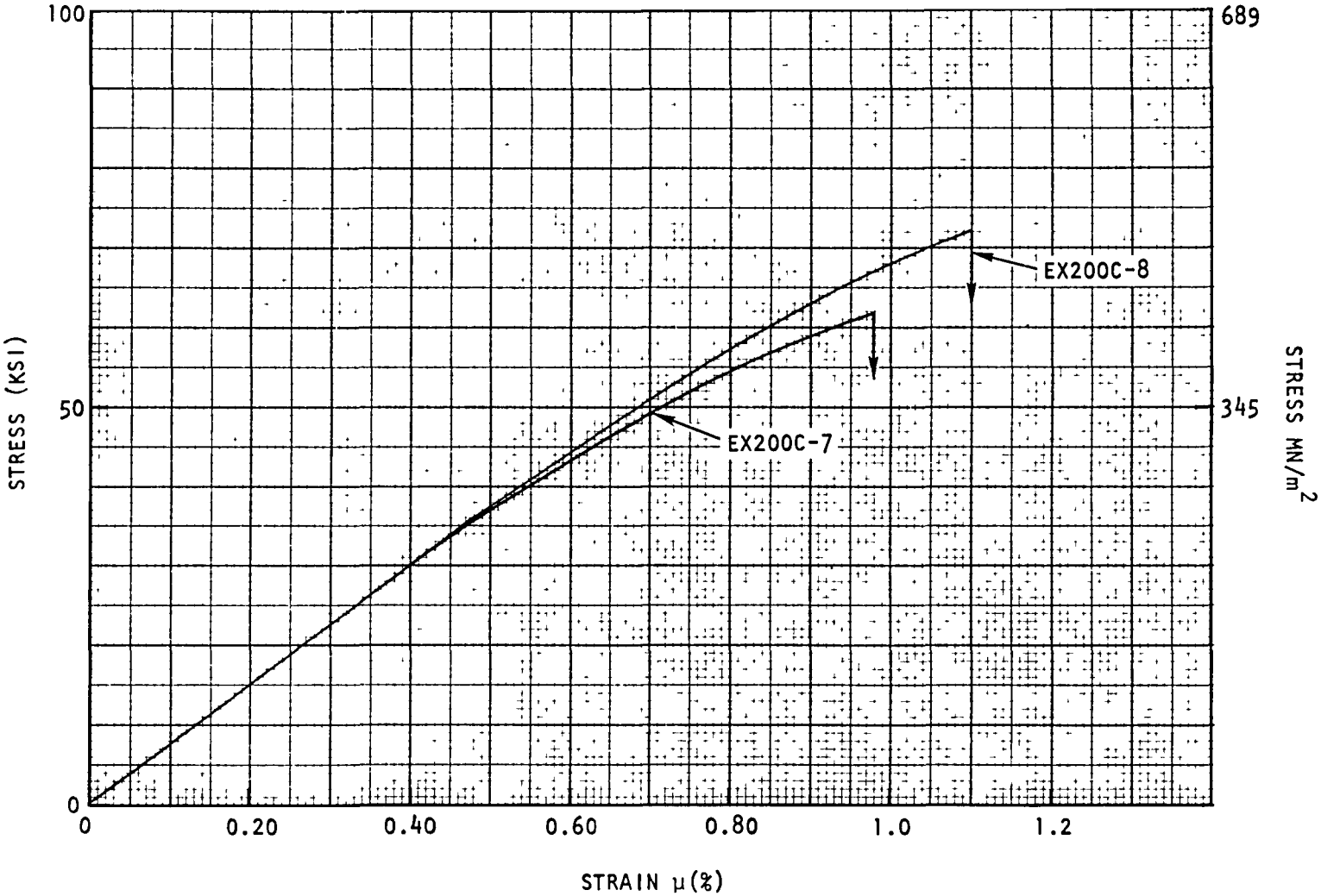


Figure C-52. Compression Stress/Strain Characteristics of (0, ±45, 90)$_S$ LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F), Beam Test at 316 C (600 F)

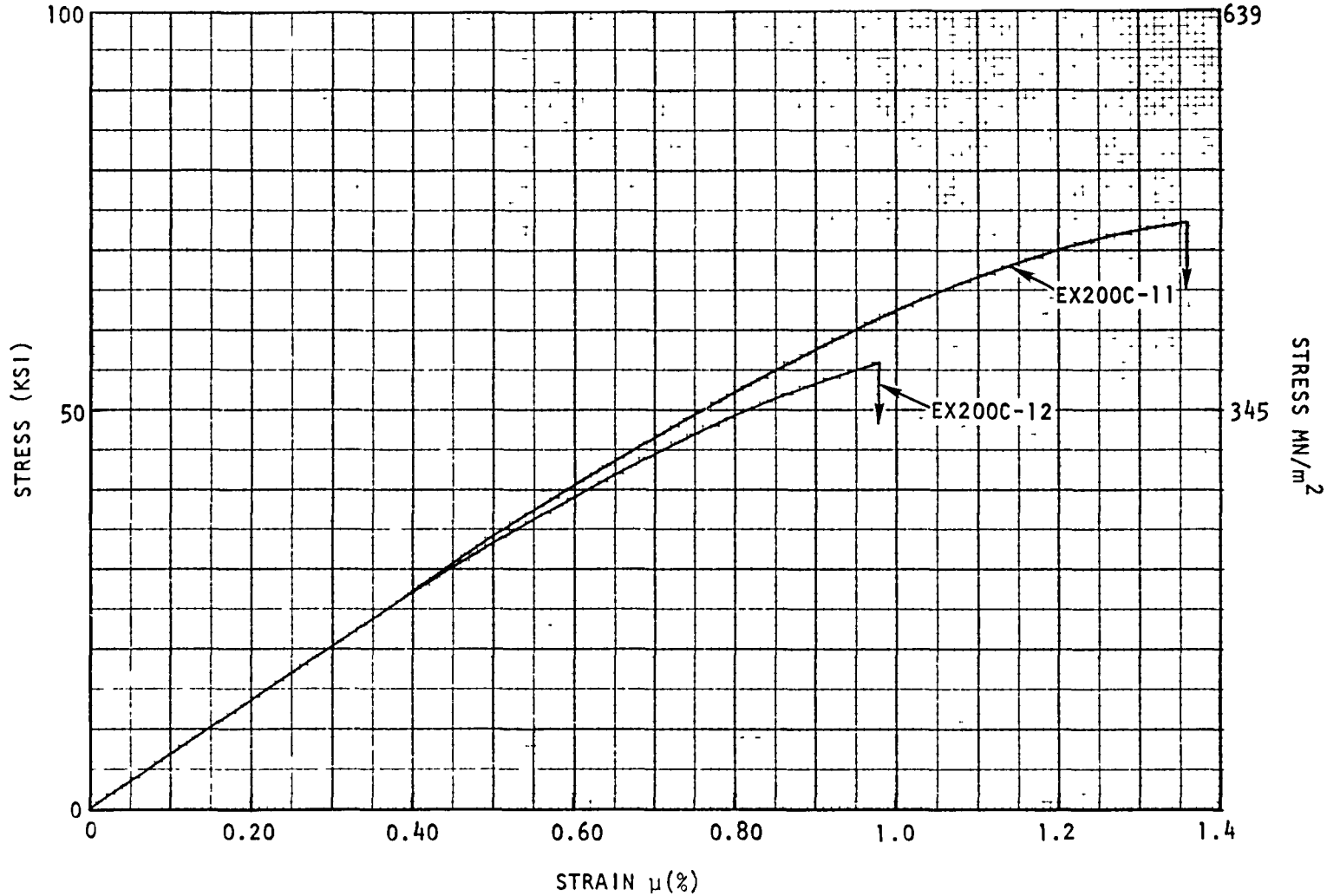


Figure C-53. Compression Stress/Strain Characteristics of $(0, \pm 45, 90)_S$ LARC-160/Celion Laminates Aged for 125 Hours at 316 C (600 F), Beam Test at 204 C (400 F)

APPENDIX D

This appendix presents design requirements for hat-section and "I" stiffened panels, design assumptions for optimization of panel configuration, analysis, and supportive calculations.

PANEL DESIGN REQUIREMENTS

Test panels were designed in accordance with requirements specified by NASA/LaRC.

(1) Panel Design Load: .53 MN/m (3000 lb/in.) nominal, .49 to .56 MN/m (2800 lb to 3200 lb/in.)

(2) Panel Skin: 8-ply isotropic layup $(0, \pm 45, 90)_s$

(3) Stringers: 3 minimum

(4) Panel Dimensions and Fabrication Concepts:

(a) 22.86 cm (9 in.) wide X 30.48 cm (12 in.) long panels
(16) Hat stiffened - 2 each, bonded, bolted, cocured
(16) "I" stiffened - 2 each, bonded, bolted, cocured

(b) 22.86 cm (9 in.) wide X 121.9 cm (48 in.) long of best configurations fabricated and tested above.

(5) Panel failure mode: 121.9 cm (48 in.) long panels - Euler buckling
30.48 cm (12 in. long) panels - local crippling or buckling

(6) Minimum Weight Design

(7) Test at -121C (-250F), room temperature, 204C (400F), and 316C (600F) after postcure and 125 hours exposure at 316C (600F)

DESIGN ASSUMPTIONS

In addition to the above design requirements, the following assumptions were made:

(1) Panel components are to be made of symmetric layups in order to avoid material warpage, which could severly degrade panel strength.

(2) Tape with the following cured and postcure properties will be used;

E_x = 148.1 GN/m^2 (21.5 Msi) compression

E_y = 9.85 GN/m^2 (1.43) Msi) compression

G_{xy} = 5792 MN/m^2 (840 Ksi)

V_{xy} = .25

V_{yx} = .05

Specific gravity, 1.57 g/cc

Fiber volume, 60%

(3) Long panel tests will be made with simply supported ends and with non-local load transfer into the panel to as great a degree as possible.

(4) $\pm$ 45 plies will be used in the web and the top of the hat or "I" section will be reinforced with 0° plies. Past investigations (Agarwal and Davis, 1974, "Minimum-Weight Designs for Hat Stiffened Composite Panels Under Uniaxial Compression," NASA TND-7779) indicate that this results in the most efficient stringer-stiffener panel design. This assumption was verified during the design process.

ANALYSIS

Panel analysis was made utilizing the AC-3 computer program and other routines written for the HP9825 for the determination of section properties following the guidelines of the Rockwell Structures Manual.

The design and optimization methodology was as follows:

(1) Select number of stiffeners.

(2) Select tape thickness.

(3) Select web layup - This is influenced by the assumption that symmetric laminates are to be used in panel subelements. Thus, the web of the "I" sections, which is formed from two "C" channels, must be increased or decreased by 4 plies at a time. Similarly, the legs of the hat section must each be increased or decreased by 2 plies at a time.

(4) Select hat or "I" section width - This choice may be modified at a later time, especially in the case of the "I" section, in order to prevent local crippling.

(5) Select hat height.

(6) Sufficient 0° ply reinforcement is added to the flange of the "I" section or top of the hat section to give the required Polar moment of inertial, I, for Euler buckling of the 122 cm (48 in.) long panels. The total load on the column will be 120.096 N (27,000 lb.) nominal 112,090 N (25,200 lb.) - 128,102 N (28,800 lb.), thus, the required I is $2.7 \times 10^{-7} m^4$ ($.6635\ in^4$) nominal ($2.5 \times 10^{-7} m^4$) $-2.9 \times 10^{-7} m^4$ ($.7077\ in^4$).

This is based on an effective modulus of elasticity E_{eff} = 65.5 GN/m^2 (9.5 Msi), which is equal to the experimentally determined compression modulus of the 8-ply "isotropic" laminate.

In order to calculate I, the areas of the various panel subelements are first converted to effective areas:

$$A_{eff_i} = A_i \frac{E_i}{E_{eff}}$$

Section properties are then calculated in accordance with procedures described in the Rockwell Structures Manual.

(7) Parametric plots are generated displaying weight vs. hat/ "I" section height for various tape thicknesses and configurations. The least weight configurations are then analyzed for local crippling.

(a) The b/t for the panel subelements is generally large enough such that they may be molded as simply supported long plates in axial compression.

(b) The formula for the local crippling of simply supported laminated plates is given in the Advanced Composite Design Guide:

$$N_{x,cr} = \frac{2\pi_2}{b^2} \left(\sqrt{D_{11} D_{22}} + D_{12} + D_{66} \right), \text{ lbs/in}$$

or

$$\sigma_{cr} = \frac{2\pi^2}{b^2 t} \left(\sqrt{D_{11} D_{22}} + D_{12} + 2 D_{66} \right), \text{ lbs/in}^2$$

where D_{11}, D_{12}, D_{22}, and D_{66} are the flexural rigidities of the laminate. These are calculated using the AC-3 Point Stress Analysis computer program, which is also described in the Advanced Composites Design Guide.

(c) The flange of the "I" section is molded as a plate with 3 sides simply supported and one side free. The corresponding cripping stress is given by:

$$\sigma = \frac{t^2}{b^2} Gxy$$

(8) The local crippling analysis places cut-off points on the parametric curves of weight vs hat/"I" height.

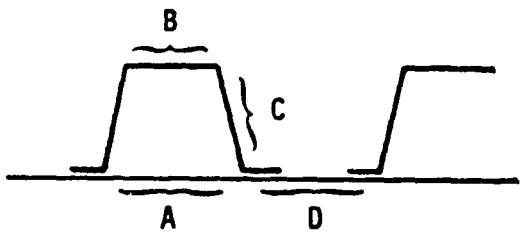
Least weight practicable designs are chosen from the curves and one is chosen subject to manufacturing constraints. The most notable fabrication problem is found in the mechanically fastened configurations in which it is necessary to provide a wide lap for tool clearance and sufficient fastener edge distance.

SUPPORTIVE CALCULATIONS FOR PANEL DESIGNS

The following provides supportive correlation with previous research and calculations pertinent to panel section properties.

(a) Local Buckling

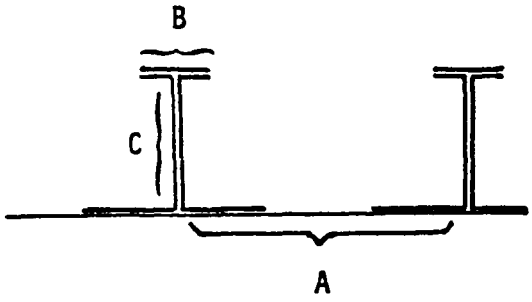
Shown below are crippling stress levels for subelements of the hat and "I" sections. Also shown are the predicted stress levels in the subelements at the Euler limit load of the long column and column loads necessary to precipitate local crippling.



Euler Buckling Load,
L = 1.22 m (48 in.)
P = 112,588 N (25,312 lb.)

Predicted:

	Crippling Stress	Stress at Euler Limit	Column Load for Local Failure
A.	163.8 MN/m^2 (23,760 psi)	154.1 MN/m^2 (22,350 psi)	120,763 N (27,150 lb.)
B.	731.4 MN/m^2 (106,077 psi)	345.7 MN/m^2 (50,131 psi)	238,235 N (53,560 lb.)
C.	53.2 MN/m^2 (7,720 psi)	48.2 MN/m^2 (6,990 psi)	124,322 N (27,950 lb.)
D.	163.8 MN/m^2 (23,760 psi)	154.1 MN/m^2 (22,350 psi)	120,763 N (27,150 lb.)



Euler Buckling Load
L = 1.22 m (48 in.)
P = 123,975 N (27,872 lb.)

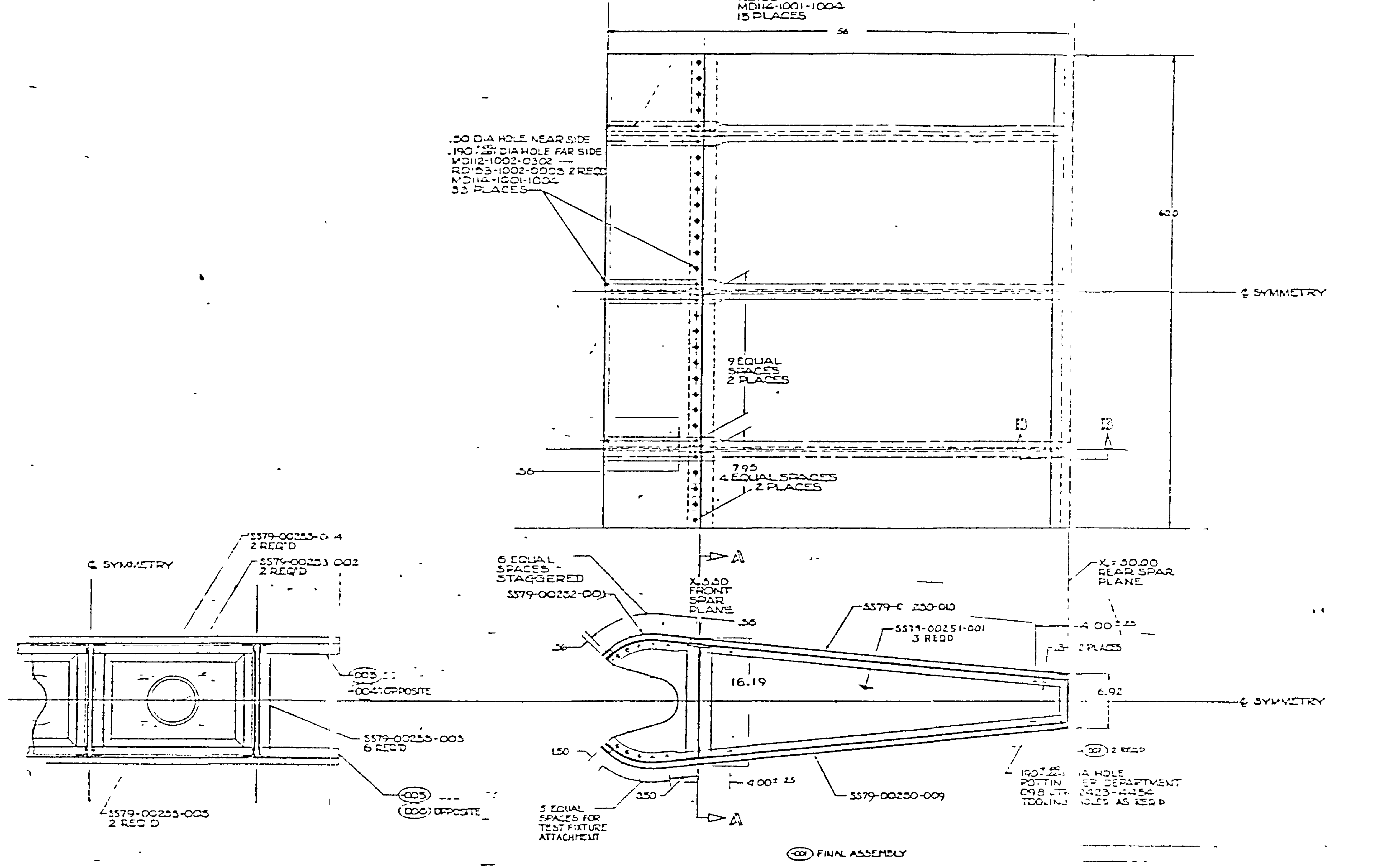
Predicted:

Crippling Stress	Stress at Euler Limit	Column Load for Local Failure
175.2 MN/m^2 (25,412 psi)	173.3 MN/m^2 (25,128 psi)	125,376 N (28,187 lb.)
433.6 MN/m^2 (62,880 psi)	392.1 MN/m^2 (56,869 psi)	137,078 N (30,818 lb.)
212.8 MN/m^2 (30,863 psi)	54.7 MN/m^2 (7,938 psi)	480,012 N (108,366 lb.)

APPENDIX E

This appendix contains the Engineering Design drawings which define the TDS. The drawing list is as follows:

SS79-00249 2 Sheets	Technology Demonstration Segment - Graphite/Polyimide, Shuttle Body Flap
SS79-00250	Cover - Body Flap Segment
SS79-00251 2 Sheets	Rib-Stability Technology Demonstration Segment, Graphite/Polyimide
SS79-00252	Leading Edge Panels - Body Flap Segment
SS79-00253 3 Sheets	Spar Webs - Body Flap Segment



10 INSTALL FASTENERS PER MA0101-301 CLASS I

(9) DRILL 9/16 DIAMETER HOLE AND INSTALL SL6149-3-770 TYPE B INSERTS WITH POTTING COMPOUND PER DEPARTMENT 098 LTR-2223-4451

(8) BOND WITH MB0120-032 [illegible] ADHESIVE SYSTEM [illegible] PER D/098 LTR 2429-445[illegible]

(7) ULTRASONIC INSPECT PER MT0501-010

(6) INSPECT LAMINATE MATERIALS PER LTR 2433-4462

(5) FABRICATE GR/PI LAMINATES PER PROCESS SPECIFICATION MA0102-333A, CURE CYCLE 5C079-333 & SPECIFIC PROCESS PER D/098 LTR 2429-4454

(4) GRAPHITE/POLYIMIDE TAPE 0.0025 THICK CELION 6000/LARC 160 PER MB0130-152A

3. LAMINATE ORIENTATION TOLERANCE: ± 2°

2. DIMENSIONAL LENGTH TOLERANCE: ± 0.03
ANGULAR TOLERANCE: ± 0° 30'

1. DIMENSIONS ARE IN INCHES

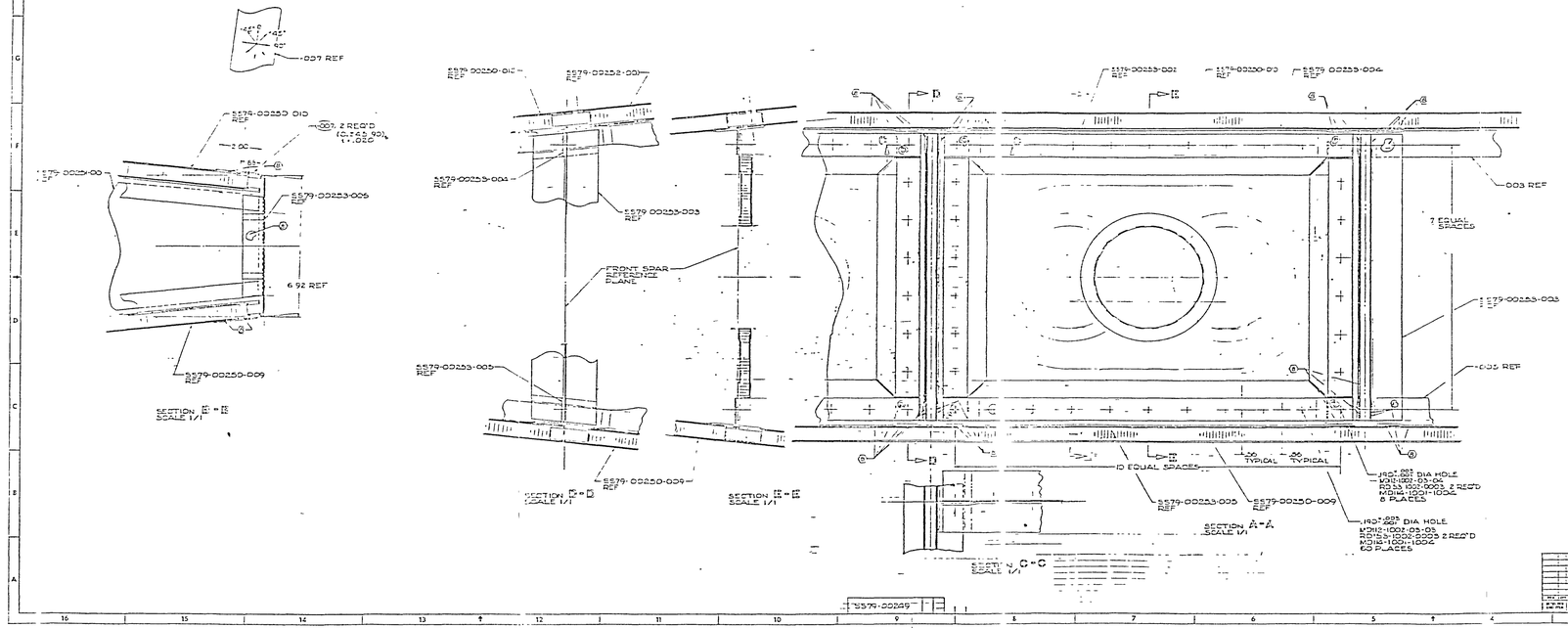
NOTES: UNLESS OTHERWISE NOTED

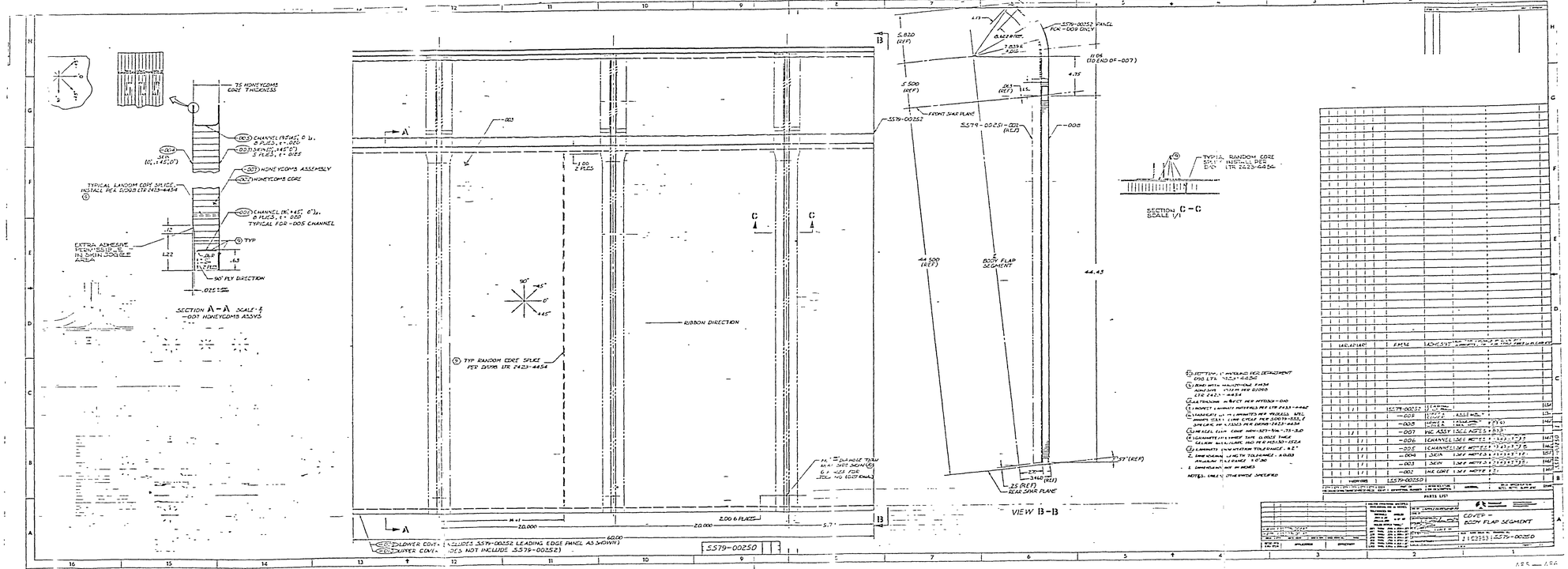
QTY REQD	PART OR IDENTIFYING NUMBER	NOMENCLATURE OR DESCRIPTION	MATERIAL	BULK SPECIFICATION
217	RD153-1002-0003	WASHER		
116	MD114-001-1004	NUT		
160	MD112-1002-03-03	SCREW		
8	MD112-1002-03-04	SCREW		
15	MD112-3002-03-43	SCREW		
33	MD112-1002-03-02	SCREW		
15	SL6149-3-770 TYPE A	INSERT	[illegible]	
1	5579-00253-006	SPAR		
2	5579-00241-007	STRAP	(4)(5)(6)(7)	
1	5579-00247-006	TEE		[illegible] 5579-00253-006
1	5579-00247-005	TEE		[illegible] 5579-00253-005
1	5579-00247-004	TEE		[illegible] 5579-00253-006
1	5579-00247-003	TEE		[illegible] 5579-00253-006
2	5579-00253-005	TEE		
2	5579-00253-004	TEE		
6				
2	5579-00253-002	WEB		
1	5579-00252-001	PANEL		
3	5579-00251-005	RIB ASSEMBLY		
1	5579-00250-009	COVER		
1	5579-00250-008	COVER		
1	-002	BOND ASSY (7)(8)		
002/-001	5579-00249	SEGMENT		

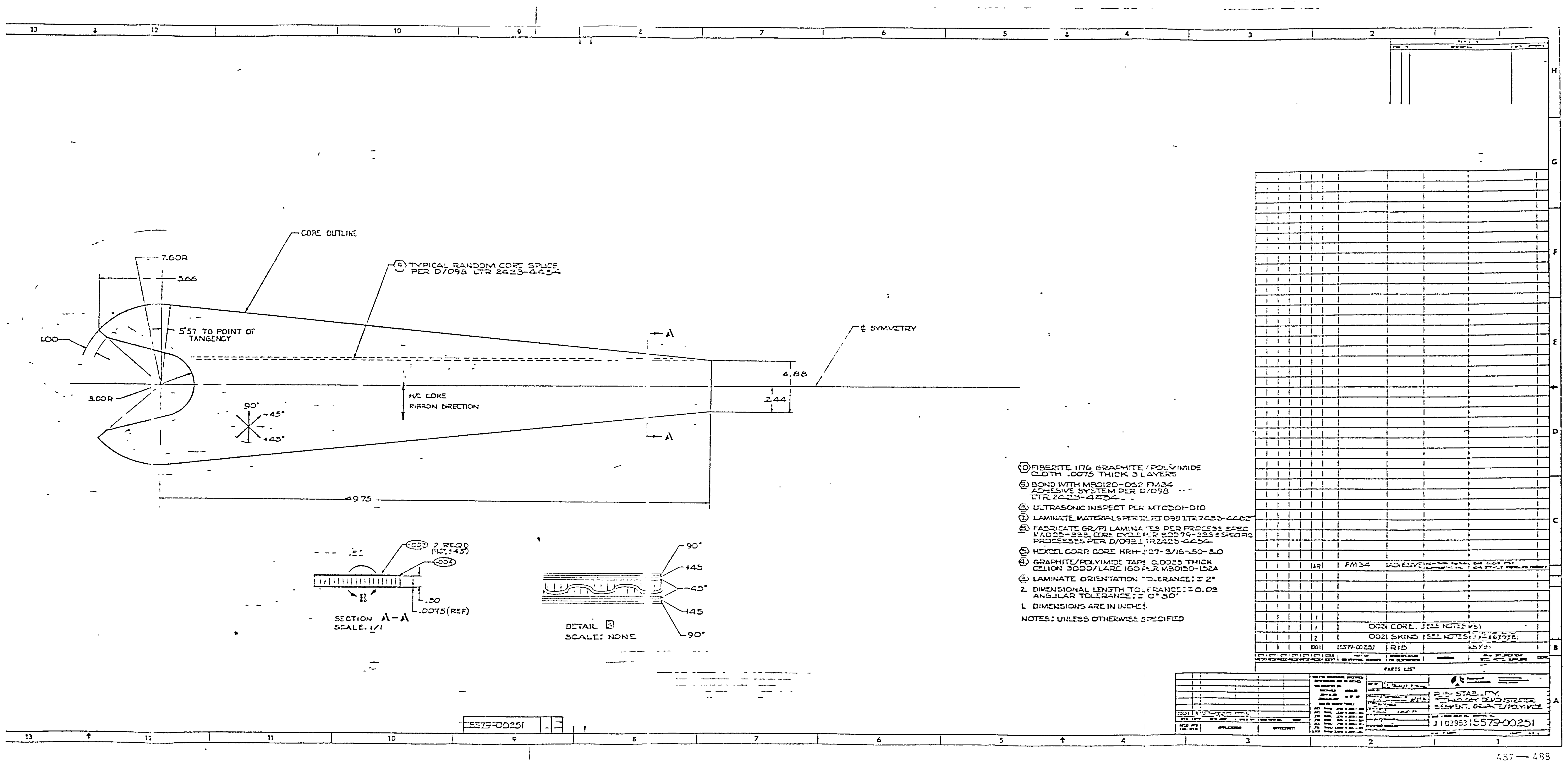
PARTS LIST

TECHNOLOGY DEMONSTRATOR SEGMENT
GRAPHITE/POLYIMIDE

5579-00249

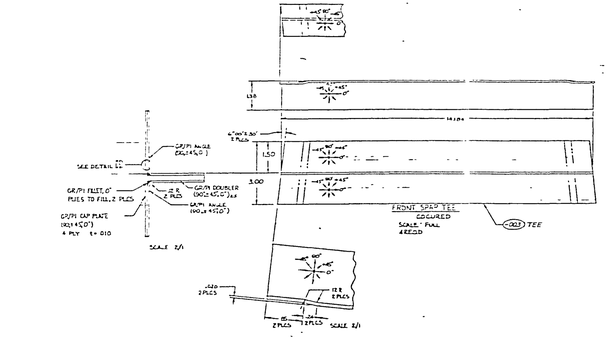

.037 REF
5579-00250-010 REF
.007 2 REQ'D (0.765 .901) (1.020
2.00
5579-00250-00 REF
5579-00253-006 REF
6.92 REF
5579-00250-009 REF
SECTION E-E SCALE 1/1
5579-00250-010 REF
5579-00252-001 REF
5579-00253-004 REF
5579-00253-003 REF
FRONT SPAR REFERENCE PLANE
5579-00253-005 REF
5579-00250-009 REF
SECTION D-D SCALE 1/1
SECTION F-F SCALE 1/1
5579-00253-001 REF
5579-00250-00 REF
5579-00253-004 REF
.003 REF
7 EQUAL SPACES
5579-00253-003 REF
.005 REF
10 EQUAL SPACES
.00 TYPICAL
.00 TYPICAL
5579-00253-005 REF
5579-00250-009 REF
.190+.007 -.002 DIA HOLE MS21042-03-06 RD33.1500-0003 2 REQ'D MS116-1001-1006 8 PLACES
SECTION A-A SCALE 1/1
.190+.007 -.002 DIA HOLE MS21042-03-05 RD33.1500-0003 2 REQ'D MS116-1001-1006 60 PLACES
SECTION C-C SCALE 1/1
5579-00249
PARTS LIST
STRUCTURE ASSEMBLY
SCALE 1/1
5579-00249

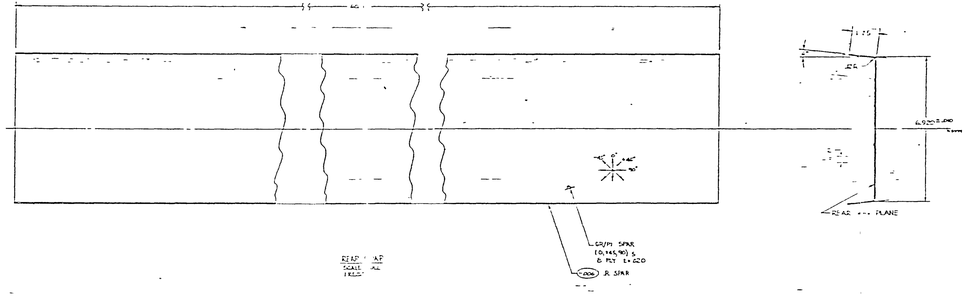

SECTION A-A SCALE: 8
-007 HONEYCOMB ASSYS
TYPICAL RANDOM CORE SPLICE INSTALL PER BOEING STR 2423-4454
EXTRA ADHESIVE TO INSIDE & TAIL SKIN DOUBLER AREA
90° FLY DIRECTION
.025 THK
.75 HONEYCOMB CORE THICKNESS
-003 HONEYCOMB ASSEMBLY
-007 HONEYCOMB CORE
TYP
TYP RANDOM CORE SPLICE PER BOEING STR 2423-4454
RIBBON DIRECTION
-003
-005
-006
-008
SECTION C-C SCALE 1/1
VIEW B-B
5579-00252 PANEL FOR -005 ONLY
5579-00251-005 (RLD)
FRONT SPAR PLANE
REAR SPAR PLANE
BOOK FLAP SEGMENT
44.500 (REF)
44.43
57" (REF)
5.820 (REF)
5.500 (REF)
11.06 (TO END OF -007)
4.75
.063
.25 (REF)
3.440 (REF)
2.00 6 PLCS
20.000
60.00
5.7
5579-00250
-003 DOES NOT INCLUDE 5579-00252)
INCLUDES 5579-00252 LEADING EDGE PANEL AS SHOWN)
PARTS LIST
COVER - BOOM FLAP SEGMENT
5579-00250


CORE OUTLINE
(5) TYPICAL RANDOM CORE SPLICE PER D/098 LTR 2423-4454
7.60R
3.66
5.51 TO POINT OF TANGENCY
1.00
3.00R
℄ SYMMETRY
4.88
2.44
H/C CORE RIBBON DIRECTION
90°
+45°
-45°
49.75
SECTION A-A
SCALE: 1/1
.30
.0075 (REF)
DETAIL B
SCALE: NONE
90°
+45
-45°
+45
-90°
(10) FIBERITE 1176 GRAPHITE / POLYIMIDE CLOTH .0075 THICK 3 LAYERS
(9) BOND WITH MB0120-052 FM34 ADHESIVE SYSTEM PER D/098 LTR 2423-4454
(8) ULTRASONIC INSPECT PER MTC501-010
(7) LAMINATE MATERIALS PER D/098 LTR 2423-4462
(6) FABRICATE GR/PI LAMINATES PER PROCESS SPEC MAC 25-333, CORE CYCLE PER 50079-295 & SPECIFIC PROCESSES PER D/098 LTR 2423-4454
(5) HEXCEL CORP. CORE HRH-327-3/16-50-5.0
(4) GRAPHITE/POLYIMIDE TAPE 0.0025 THICK CELION 3000/LARC 160 PER MB0150-152A
(3) LAMINATE ORIENTATION TOLERANCE: ± 2°
2. DIMENSIONAL LENGTH TOLERANCE: ± 0.03 ANGULAR TOLERANCE: ± 0° 30'
1. DIMENSIONS ARE IN INCHES
NOTES: UNLESS OTHERWISE SPECIFIED
0031 CORE
0021 SKINS SEE NOTES (3)(4)(6)(7)(8)
001 1S579-00251 RIB
PARTS LIST
PITCH STABILITY TECHNOLOGY DEMONSTRATOR SEGMENT, GRAPHITE/POLYIMIDE
J 102953 1S579-00251
1S579-00251


-002 DETAIL
½ SCALE
SECTION A-A
SECTION B-B
-004 DETAIL
½ SCALE
SECTION C-C
-003 DETAIL
SECTION D-D
TDS RIB ASSEMBLY
TANGENT POINT
LAMINATE SCHEMATIC
53.820
50.000
5579-00251-001 1 REQD
11 FIBERITE W/ GRAPHITE/POLYIMIDE CLOTH .0075 THICK 3 LAYERS
10 POTTING COMPOUND PER D/098 LTR 2423-4454
9 BOND WITH MBO120-062 FM34 ADHESIVE SYSTEM PER D/098 LTR 2423-4454
8 ULTRASONIC INSPECT PER MTO501-010
7 INSPECT LAMINATE MATERIALS PER LTR 2433-4462
6 FABRICATE GR/PI LAMINATES PER PROCESS SPEC MAO106-333 CURE CYCLE PER 500TT-333, & SPECIAL PROCESSES PER D/098-2423-4464
5 HEXCEL CORP CORE HRH-327-3/16-7.5-3.0
4 GRAPHITE POLYIMIDE TAPE 0.0025 THICK CELION 6000/LARC 160 PER MB0130-152A
3 LAMINATE ORIENTATION TOLERANCE: ± 2°
2. DIMENSIONAL LENGTH TOLERANCE ± 0.03 ANGULAR TOLERANCE: ± 0°30
1. DIMENSIONS ARE IN INCHES
NOTES, UNLESS OTHERWISE SPECIFIED
5579-00251-005 ASSEMBLY
2 5579-00253-003 TEE
1 -004 FRONT RIB CLOSE-OUT SEE NOTES (3)(6)(7)(8)(11)
1 -003 REAR SPAR ATTACH SEE NOTES (3)(4)(6)(8)
2 -002 UPR & LWR RIB CAPS SEE NOTES (3)(4)(6)(7)(8)
1 5579-00251-001 RIB CORE ASSEMBLY
PARTS LIST
RIB ASSEMBLY FOR TECHNOLOGY DEMONSTRATION SEGMENT
J 03953 5579-00251
5579-00251


TYPICAL RANDOM CORE SPLICE (9) INSTALL PER D/098 LTR 2423-4454
RIBBON DIRECTION
TYP FOR -003 & -004
60.00
-005 CHANNEL CO-CURED LAMINATE SCHEMATIC
24 PLIES
8 PLIES
BODY FLAP SEGMENT MOLD LINE
1.13
8.622 R
6.40
5.500
(-005) CHANNEL 2 REQD
(-002) HONEYCOMB CORE
(-003) SKIN (0°, ±45, 0°)
(-004) SKIN (0°, ±45, 0°)
-005 REF
FRONT SPAR PLANE
8.096
.7503 .006 HONEYCOMB CORE THICKNESS CONSTANT
5°53'
(9) BOND WITH MB0120-062 FM34 ADHESIVE PER D/098 LTR 2423-4454
(8) ULTRASONIC INSPECT PER MT0501-010
(7) INSPECT LAMINATE MATERIALS PER LTR 2423-4462
(6) FABRICATE LAMINATES PER MAT'L SPEC MB105-333 CURE CYCLE PER S0079-333, & SPECIFIC PROCESSES PER D/098-2423-4454
(5) HEXCEL CORP. HRH-327-3/16-.75-5.0 TO 5.6 PCF
(4) GRAPHITE/POLYIMIDE TAPE 0.0025 THICK CELION 6000/LARC 160 PER MB0130-152A
(3) LAMINATE ORIENTATION TOLERANCE ± 2°
2. DIMENSIONAL LENGTH TOLERANCE ± 0.03 ANGULAR TOLERANCE ± 0.30
1. DIMENSIONS ARE IN INCHES
NOTES - UNLESS OTHERWISE SPECIFIED
AR | FM34 | ADHESIVE
2 | -005 | CHANNEL | SEE NOTES
1 | -004 | SKIN | SEE NOTES
2 | -003 | SKIN | SEE NOTES
1 | -002 | H/C CORE | SEE NOTE
SS79-00252 | SEE NOTE
PARTS LIST
LEADING EDGE PANELS - BODY FLAP SEGMENT
SS79-00252


GR/PI ANGLE (90,45,0)
SEE DETAIL 'D'
GR/PI FILLET, 0" FILES TO FILL, 2 PLCS
GR/PI DOUBLER (90,45,0) t=.040
GR/PI CAP PLATE (90,45,0) 4 PLY t=.010
GR/PI ANGLE (90,45,0)
SCALE 2/1
FRONT SPAR TEE COGURED
SCALE: FULL 4 REQD
-003 TEE
90° +45° -45° 0° 0° -45° +45° 90°
WEB ON CAP
DOUBLER ON RADIUS
DETAIL 'D'
SCALE: NONE LAMINATE SCHEMATIC
SCALE 2/1
GR/PI CAP PLATE (90,45,0) 4 PLY t=.010
-004 TEE
SEE DETAIL 'D'
GR/PI FILLET, 0" FILES TO FILL, 2 PLCS
GR/PI ANGLE (90,45,0)
GR/PI DOUBLER (90,45,0) t=.040 4 PLY
GR/PI ANGLE (90,45,0)
SECTION C-C
-004 TEE, 2 REQD
SCALE 2/1
SEE LAMINATE SCHEMATIC, SHT 3
RIBS
C
D
LH STIFF
C
-004 TEE
D
-005 TEE
GR/PI ANGLE (90,45,0) & (45,90,*)
-002 TEE
FRONT SPAR PLANE
FRONT SPAR CAP
-004 - 2 REQD
-005 - 2 REQD
SCALE 1/4
GR/PI FILLET, 0" FILES TO FILL
GR/PI ANGLE (90,45,0)
GR/PI PLATE (90,45,0),t=.010
SECTION D-D
-005 TEE, 2 REQD, SCALE 2/1
IDENTICAL TO -004 EXCEPT AS NOTED.
SEE LAMINATE SCHEMATIC, SHT 3
CORE SPLICE PER SDDB LTP 2423-4454
GRAIN DIRECTION
6.50 DIA
5.00 DIA
5.25 DIA
-45° 0° +45° 90°
-002 FRONT SPAR PANEL
-003 PANEL ASSY
-009 FRAME
-011 RING
-012 HONEYCOMB ASSY
-013 HONEYCOMB CORE
-014 SEINE(5.45,90°)
-015 DOUBLER
-016 DOUBLER
SEE ALTERNATE CLKS EPT, SHT 3
SECTION A-A
-009 I-D10 FRAME SECTION, COGURED
SCALE 1/1
SEE LAMINATE SCHEMATIC, SHT 3
DETAIL 'B'
SCALE 2/1
-011 RING
SEE LAMINATE SCHEMATIC, SHT 3
SPAR WEBS - BODY FLAP SEGMENT
J103953 | 55T9-00253
55T9-00253
493 — 494



REAR SPAR
SCALE: ALL

55-71-00253 | 2

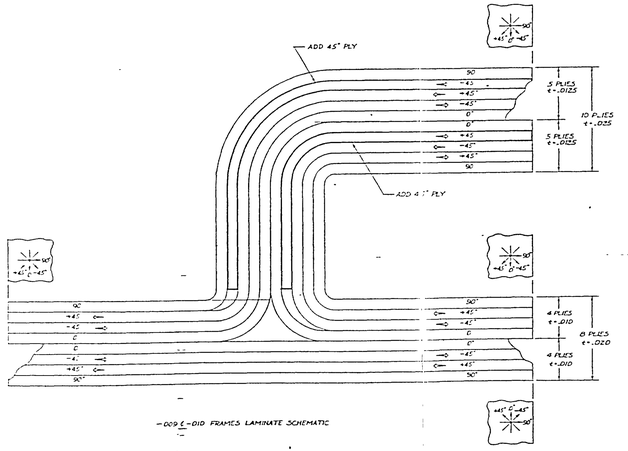
ALTERNATE CONCEPT FOR -011 RING

.625

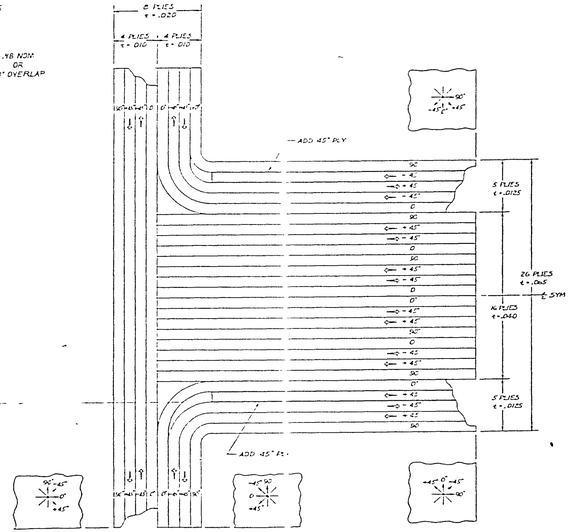
.100 TYP

.98 NOM. OR 10° OVERLAP

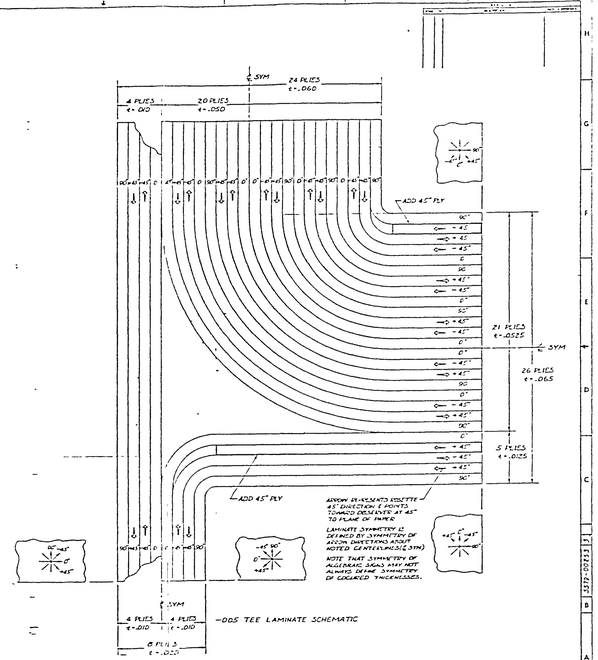
OVERLAPPING CHANNEL SEGMENTS



-009 & -010 FRAMES LAMINATE SCHEMATIC



-003 & -004 TEES LAMINATE SCHEMATIC



-005 TEE LAMINATE SCHEMATIC

1 Report No	2 Government Accession No	3 Recipient's Catalog No
NASA CR 165809		

4 Title and Subtitle	5 Report Date
Development and Demonstration of Manufacturing Processes for Fabricating Graphite/LARC-160 Polyimide Structural Elements	December 1981
	6 Performing Organization Code

7 Author(s)	8 Performing Organization Report No
R. K. Frost J. S. Jones P. J. Dynes D. H. Wykes	
	10 Work Unit No

9 Performing Organization Name and Address	11 Contract or Grant No
Rockwell International 12214 Lakewood Blvd. Downey, Ca. 90241	NAS1-15371
	13 Type of Report and Period Covered
12 Sponsoring Agency Name and Address	Contractor Report
National Aeronautics and Space Administration Washington, D.C. 20546	14 Sponsoring Agency Code

15 Supplementary Notes

Langley Technical Monitor: Robert M. Baucom

Final Report

16 Abstract

This program was conducted to develop and demonstrate manufacturing technologies for the structural application of Celion graphite/LARC-160 polyimide composite material. The program consisted of two parts: Process Development and Fabrication of Demonstration Components. Process development included establishing quality assurance of the basic composite material and processing, nondestructive inspection of fabricated components, developing processes for specific structural forms, and qualification of processes through mechanical testing. In the second part of the program, demonstration components were fabricated using the processes developed in part one. The demonstration components consisted of flat laminates, skin/stringer panels, honeycomb panels, chopped fiber compression moldings, and a Technology Demonstrator Segment (TDS) representative of the Space Shuttle aft body flap. The TDS is a full-size testable component and will be subjected to mechanical loading at room temperature and 260°C; simulated fatigue, 400 cycles between 5-100% of limit load at 260°C; and thermal cycling, 125 cycles between -107°C and 316°C. TDS test results will be reported in a separate final report.

17 Key Words (Suggested by Author(s))	18 Distribution Statement
LARC-160 Polyimide Resin, Graphite/Polyimide Celion/LARC-160, Polyimide Processing Polyimide Characterization Polyimide Fabrication Polyimide Adhesive Bonding	Unclassified - Unlimited

19 Security Classif (of this report)	20 Security Classif (of this page)	21 No of Pages	22 Price
Unclassified	Unclassified	498	

End of Document