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USAAVLABS TECHNICAL REPORT 65-66

THE EFFECT OF RESIN CONTENT AND VOIDS ON THE STRENGTH OF FIBERGLASS-REINFORCED PLASTICS FOR AIRFRAME USE

Final Report

By

Gene M. Nordby
W. C. Crisman
Charles W. Bert

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The conclusions made by the contractor are considered by this Command to be valid.

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Prepared by
University of Oklahoma Research Institute
Norman, Oklahoma

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FORT EUSTIS, VIRGINIA

ABSTRACT

As a further step toward the acceptance of fiberglass-reinforced plastics (FRP) in primary aircraft structure, the effects of resin content on laminate strength properties were investigated in depth. Functional relationships were obtained and methods of controlling resin content were compared. Uniformity in strength and resin content was observed from the point of view of replication and within-laminate conditions. Most of the data were for laminates of EPON 828-Z epoxy resin reinforced with the 181 style fiberglass fabric; however, limited data were obtained for the 120 and 909 (S-920) fabrics.

In addition, a preliminary study was made of the effects of voids in the cured laminates. A critical size was observed for large artificial voids, and the microvoid content of the pre-preg was minimized by modification of a previously developed multi-ply coating machine. Non-destructive methods of testing for voids were also reviewed.

PREFACE

This report was prepared by the University of Oklahoma Research Institute (OURI) under Phase I of U. S. Army Aviation Materiel Laboratories (USAAVLABS) Contract DA 44-177-AMC-164(T). Phase II was reported separately under the title Dynamic Elastic, Damping, and Fatigue Characterists of Fiberglass-Reinforced Sandwich Structure. The research effort is a continuation of the work done under two other USAAVLABS contracts which resulted in the following reports: Research in the Field of Fiberglass-Reinforced Sandwich Structure for Airframe Use and Strength Properties and Relationships Associated with Various Types of Fiberglass-Reinforced-Facing Sandwich Structure. The present report contains the test results, conclusions, and recommendations for research conducted on the several aspects of fabrication of fiberglass-reinforced plastics (FRP) during the period of April 9, 1964, to March 31, 1965.

This report was written by Dr. Gene M. Nordby, project director and Dean of the College of Engineering at the University of Oklahoma; Mr. W. C. Crisman, project engineer; and Dr. Charles W. Bert, research engineer and Associate Professor of Aerospace and Mechanical Engineering. Mr. Donald Hanson worked as test engineer and statistician.

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SYMBOLS

F	Fahrenheit
psi	pounds per square inch
$\bar{x}$	an arithmetic mean value
$\bar{\bar{x}}$	an arithmetic mean of differences
n	number of specimens
$\hat{s}$	estimate of standard deviation
μ	true mean value
CL	confidence level
RC	resin content by weight
W_a	weight of specimen per unit area
W_g	weight per unit area of total number plies of fabric
E_c	modulus of the composite
E_g	modulus of the glass
σ	ultimate strength

DISCUSSION

A. Introduction

Previous research work performed for USAAVLABS in the realm of fabrication of fiberglass-reinforced plastics (FRP) suitable for use in primary aircraft structure (reference 16) has shown the need for the development of consistent, reproducible mechanical properties. For example, standard deviations of 2,500 psi or more were not uncommon for ultimate strength measurements within a given laminate. Consequently, the very large number of fabrication and process variables which affect the final strength level of FRP materials must be studied and controlled if consistency is to be achieved. The research program reported herein was thus designed to obtain additional information regarding the effects of the important variables on the strength properties of the cured laminates and to determine ways of better controlling laminate resin content and uniformity.

The major research effort was prefaced by a number of preliminary investigations aimed at refinement of procedures and at establishment of bounds for several of the variables involved only indirectly in fabrication. For instance, one very important question that had to be answered was: How long can the pre-preg be kept in cold storage without adversely affecting the strength properties of the finished laminate? In addition, these preliminary studies were carefully designed to feed back information to the main research program where possible.

As a logical first step in the research, the machine previously designed (reference 16) to coat the fiberglass fabric and produce the multiply pre-preg was improved in an effort to minimize or possibly eliminate the occurrence of air voids in the pre-preg. This work was then carried further by a preliminary study of the effects of voids in the pre-preg. It was hoped that the data generated by the limited tests would reveal a critical size and/or density, should these be found important, and even suggest a measurement.

The major aspect of the research was considered to be the effect of resin content on the strength properties of the finished laminates. To ascertain the resin content effect and to determine possible ways of controlling the final resin content and uniformity of the cured laminate, a two-by-four factorial experiment in pressure and temperature was executed (pressure: 13, 30, 70, and 100 psi; Temperature: 160, 180, 200, and 230 degrees Fahrenheit). As in the previous research program for USAAVLABS, the Shell Chemical Company 828 epoxy resin activated by curing agent Z was used for the resin matrix in the laminates.

To cast further light on laminate uniformity, laminates of three types of fiberglass fabric were tested. These were the styles 120 and 181 crowfoot satin weave, Volan A finish, E-glass fabrics of the Exeter Manufacturing Company; and the style 909 high modulus weave, S-920 finish, E-glass fabric of the J. P. Stevens and Company, Inc. The number of plies of fabric used were such that all of the laminates were of approximately the same thickness.

The strength properties examined were: compressive ultimate strength and modulus; tensile ultimate strength and modulus; and flexural maximum fiber stress and flexural modulus. All tests were accomplished at room temperature.

B. Fabrication Equipment

1. Multi-Ply Coating Machine

a. Modifications

When the present research team began fabricating thin epoxy-fiberglass laminates for strength studies in October 1962, it was quickly realized from the initial work with hand-squeegeed lay-ups that if uniform, controllable and reproducible results were to be obtained, a mechanical means of impregnating and assembling or plying the wet fiberglass fabric was manditory. Consequently, a multi-ply coating machine was developed. A description of the original apparatus may be found in reference 16. The machine was a tremendous improvement in the lay-up process, especially in regard to reducing the larger type of void inclusions in the pre-preg; however, the smaller microvoids were not completely eliminated.

In the present program, as a first step in the preliminary study of void inclusions in finished laminates, several modifications were made to the machine in an effort to minimize their occurrence in the pre-preg output. It appeared that most of the tiny air voids (microvoids) were trapped in the pre-preg during the coating of the dry fiberglass fabric or during the subsequent plying of the wet fabric. This will be further discussed in section E2a. To improve the wetting process and minimize air entrainment, the original machine was modified so that the coating occurred as the fabric was drawn through a heated resin vat rather than as a result of a resin spray which was used previously. Then, the plying or laminating process was modified so as to minimize the large air bubbles that formed in front of the main pressure rollers as

the resin was squeezed out to establish the desired resin content in the pre-preg. This was done through stripping most of the excess resin from the fabric before it reached the main rollers by repositioning the smaller intermediate set of rollers which operated at very nearly the same spacing as the main rollers (see Figure 6). The existing first set of pressure rollers was moved from the vertically stacked to the horizontally stacked position for this purpose.

Other modifications were made to the machine to increase the control of the process and to decrease handling of the fiberglass. A variable-speed wind motor was installed to replace the fixed-speed motor previously used. The 2-inch-diameter take-up cylinder was replaced by an 18-inch cylinder not only to permit larger productions of pre-preg with less differential stretching of the cover films, but also to permit more uniform cooling of the material during the initial phase of B-staging, as the pre-preg was allowed to room-temperature B-stage while tightly wound on the cylinder in order to minimize air seepage between the thin polyethylene cover films. The mounts for the fiberglass supply reels were enlarged to accommodate 100-yard rolls, which virtually eliminated the need for rewinding or handling of the fabric in some cases.

The final configuration of the machine is shown in Figures 1, 2, and 3. It was found convenient for this program to rig the system for a three-layer impregnation, though the capacity of the system is much greater. The 181 and the 909 fabrics were impregnated as three single layers, while the 120 fabric was impregnated as three double layers. (The 120 style fabric is only half the thickness of the other two fabrics, thus requiring 6 plies for equivalent pre-preg thickness.) The photographs were taken of the machine threaded with the 181 style fiberglass fabric.

b. Operation

The system is operated by an electric motor (A in Figure 1) geared to the take-up cylinder (B in Figure 1) which pulls the fabric through the apparatus by winding the pre-preg. The fiberglass fabric is drawn from the supply rolls (C in Figure 1) over the first guide roller (A in Figure 2) and into the resin vat (Figure 3 and B in Figure 2). As the fabric leaves the first guide roller, the layers are separated by the three smaller guide rollers located in the bottom of the vat (A in Figure 3). Each layer of fabric (whether single-ply or double-ply as in the case of the 120

style fabric) is coated and impregnated as it moves through the resin in the vat, with the pumping action of the rollers assisting in the process.

The separated layers of resin-rich fabric are then brought together again a few inches above the vat as they enter the first set of pressure rollers (C in Figure 2). At this point, the excess resin is stripped from the three-layer unit and runs back into the vat. This process brings about further wetting and establishes the assembly of the pre-preg. From the first set of pressure rollers, the pre-preg moves through the main pressure rollers (D in Figures 1 and 2) where the final resin content is established. The 0.001-inch-thick polyethylene cover films are introduced to the outer surfaces of the pre-preg as it enters the main rollers (E in Figure 1 and D in Figure 2). The finished pre-preg is then wound onto the large take-up cylinder for room-temperature B-staging.

The resin vat is approximately 2-1/2 inches deep, and in operation the resin level is maintained at a 2-inch depth (1/2 inch above the rollers) by manually controlling the resin flow from two overhead reservoirs (F in Figure 1). Both of the reservoirs were electrically heated by strip heaters bonded to their exteriors, and the resin temperature was monitored with mercury thermometers. The vat was also electrically heated and the temperature of the resin was automatically controlled by a Barber-Colman regulating pyrometer (model 292P).

As mentioned previously, the final establishment of the pre-preg resin content takes place at the main pressure rollers; consequently, the spacing of these rollers was carefully set prior to each machine run. The adjustment screws marked E in Figure 2 were turned until the desired readings were set on the gages marked G in Figure 1. The settings were further checked by actual feeler gage measurements.

During the operation of the machine, the temperature of the resin in the vat averaged approximately 130 degrees Fahrenheit plus or minus 5 degrees. The temperature for mixing the EPON 828 epoxy resin and curing agent Z and, hence, the approximate temperature in the overhead reservoirs was usually above and within 10 degrees of that in the vat. The mixing ratio used was 100 parts resin to 20 parts curing agent by weight. From the time of mixing, the elapsed time for producing the various lengths of pre-preg used in the program was less then 15 minutes--less than half the pot life of the resin material.

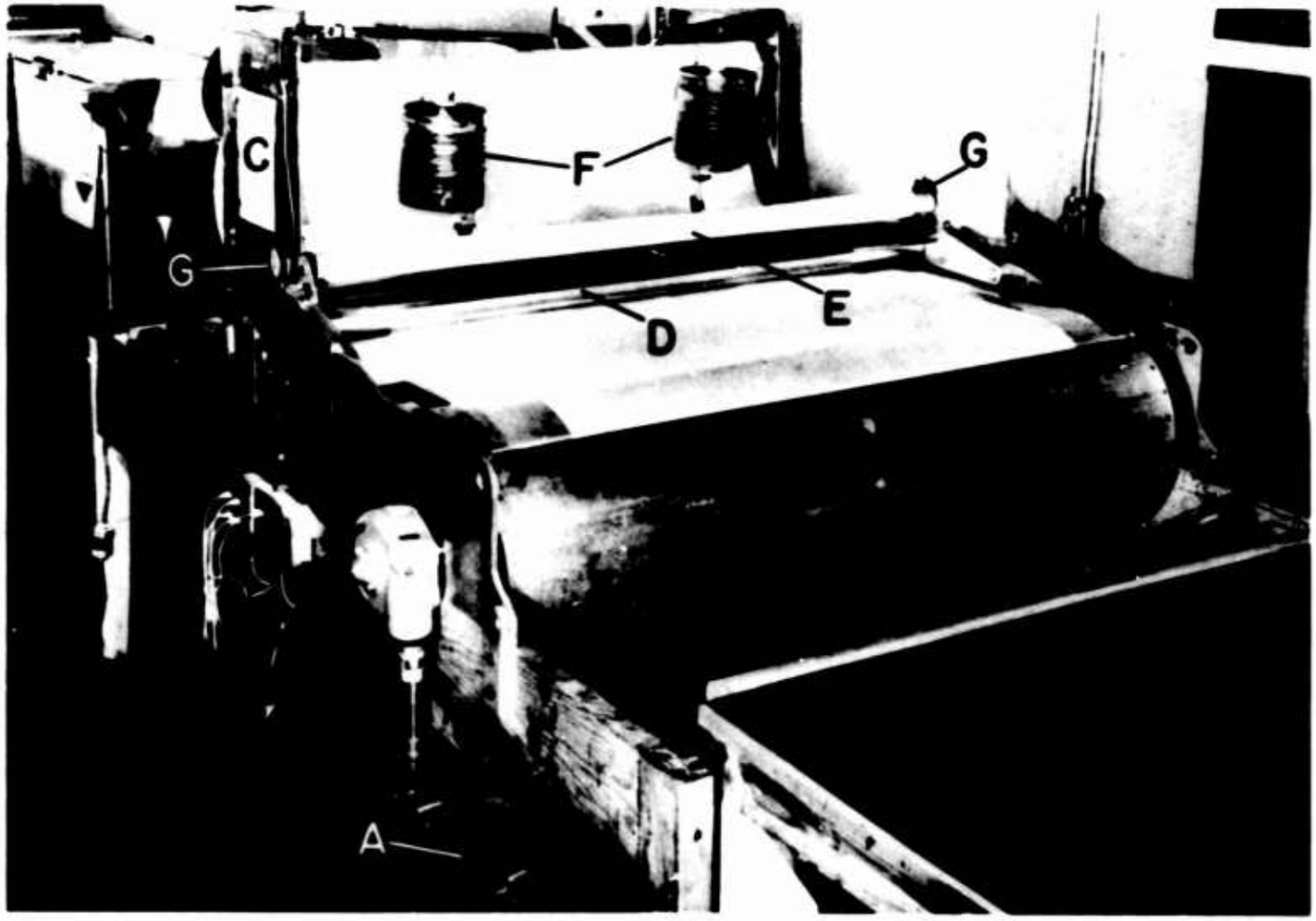


Figure 1. Overall View of Multi-Ply Coating Machine. A, Vari-Drive Wind Motor; B, 18-Inch-Diameter Take-Up Cylinder; C, Rack of Fiberglass Supply Rolls; D, Upper Main Pressure Roller (Lower Roller Not Visible); E, Upper Supply Roll of Polyethylene Film (Lower Roll Not Visible); F, Resin Reservoirs; G, Dial Gages for Monitoring Spacing of Main Pressure Rollers.

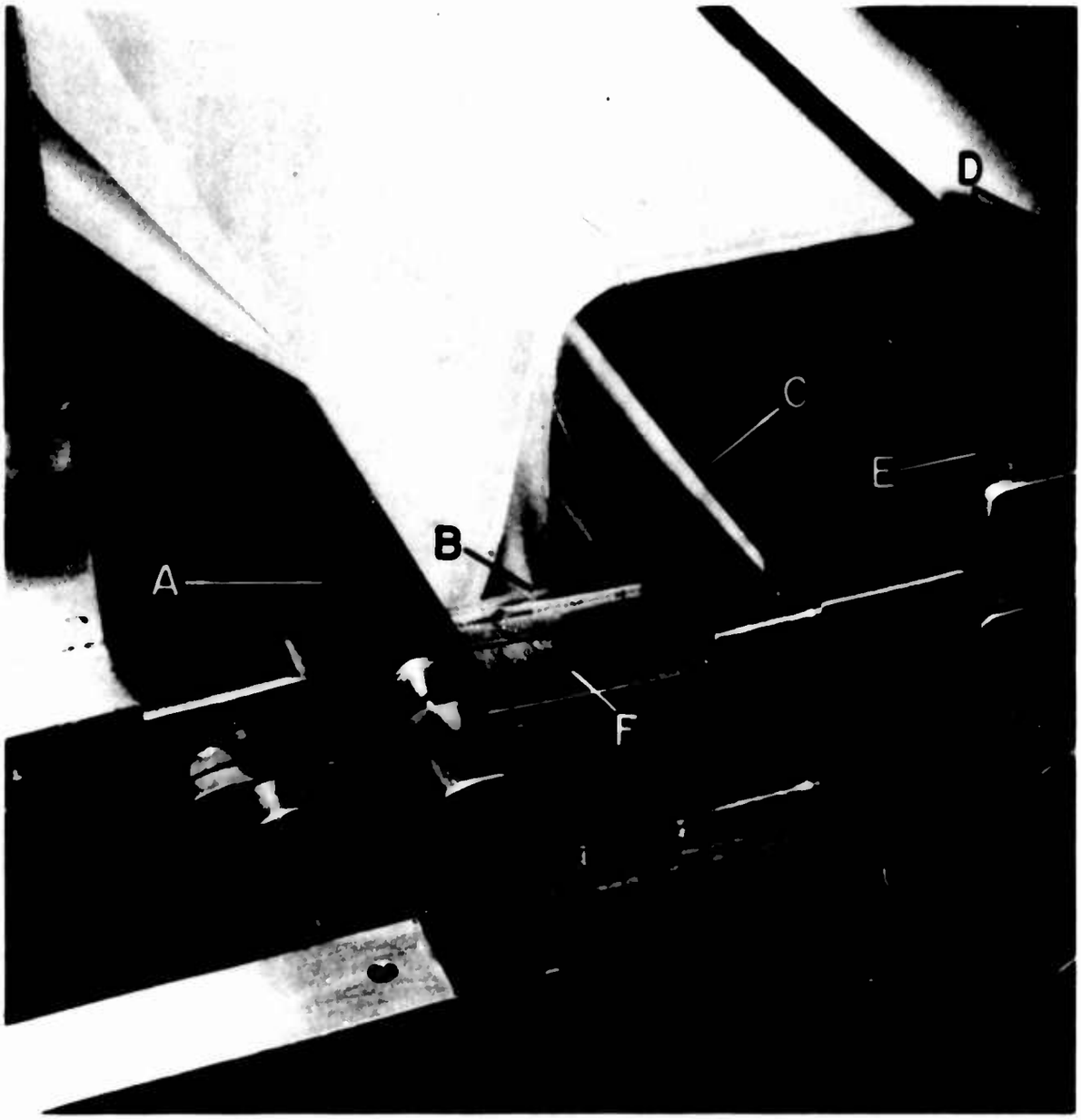


Figure 2. Detail View of Multi-Ply Coating Machine Showing Roller System. A, First Guide Roller; B, Resin Vat Area (3-Ply, 181 Fabric Shown); C, First Set of Pressure Rollers (One Roller Has Been Removed To Show Vat Area); D, Main Pressure Rollers (Edge of Polyethylene Cover Film Is Visible on Upper Roller); E, Adjustment Screw for Setting Spacing of Main Pressure Rollers (Another Is Located on the Opposite Roller Mount); F, Pin-Block Which Pins Together and Mounts Vat Assembly to Machine, One Per Side (See D in Figure 3).

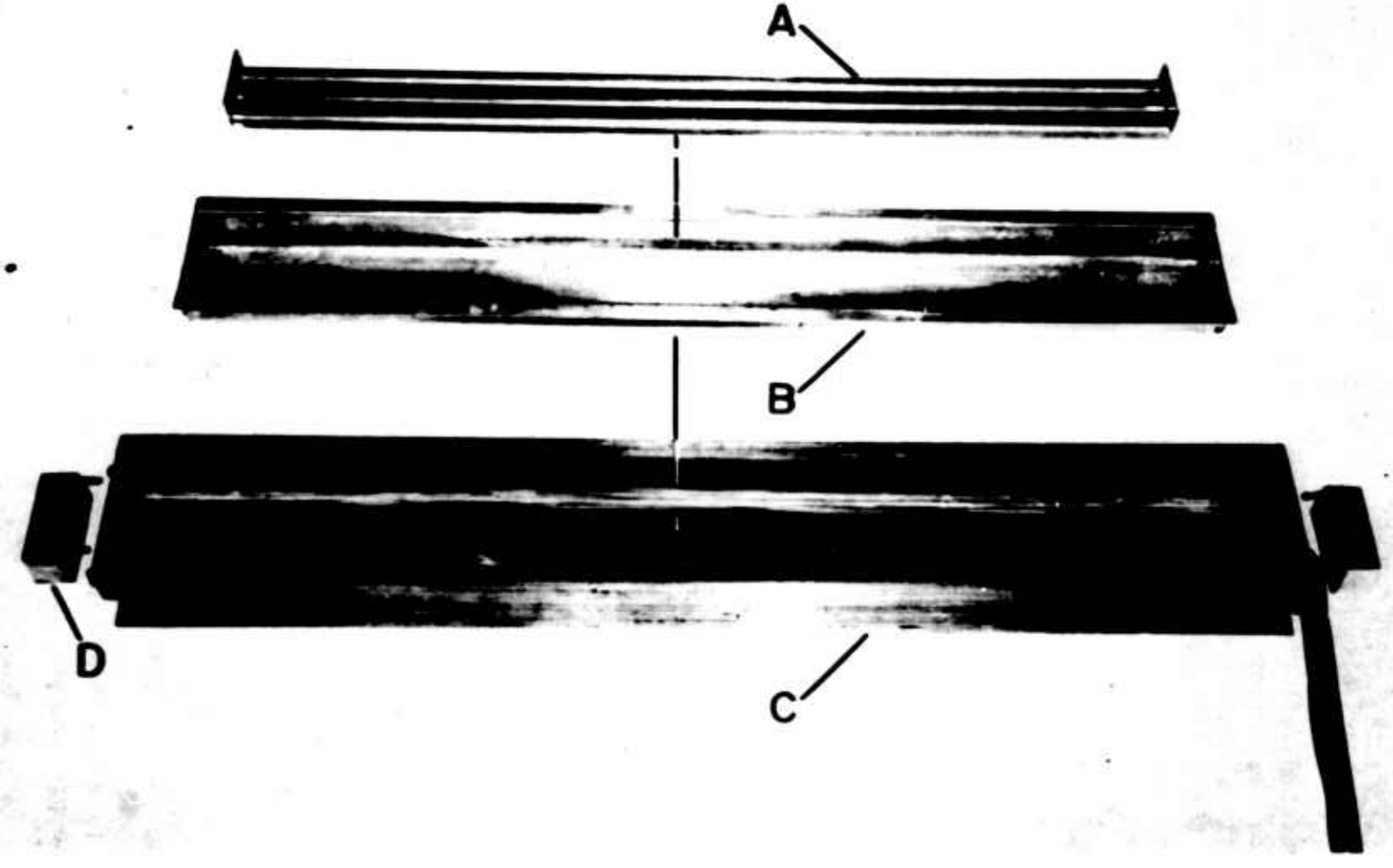


Figure 3. Detail View of Resin Vat Assembly. A, One-Inch-Diameter Rollers That Separate Fiberglass Fabric Layers Inside Vat; B, Resin Vat; C, Heating Shroud (Longitudinal Heating Strips are Visible); D, Pin-Block Which Pins Together and Mounts Vat Assembly to Coating Machine, One Per Side (Pins Permit Quick Disassembly for Cleaning).

c. Evaluation

The impregnation of the fabric brought about by immersion in the resin and assisted by the pumping action produced as the layers moved around the separator-guide rollers in the bottom of the vat plus the further circulation at the first set of pressure rollers was observed to be excellent. Moving the first set of pressure rollers (C in Figure 2) from the vertically stacked position, as they were in the original design, to the horizontally stacked position functioned well to reduce the size and number of air bubbles that had previously formed just ahead of the main pressure rollers. A small bubble still develops at the mid-span of the main roller after the production of some 25 to 50 inches of pre-preg; however, its size is greatly reduced.

The resin, being applied over the wide expanse of the fabric, cooled rapidly as it left the main pressure rollers, thus retaining the uniformity that had been established. The winding under tension onto the large take-up cylinder further maintained the uniformity and also minimized air seepage at the edges of the cover films. The large diameter of the take-up cylinder also minimized differential stretching of the cover films which fosters air seepage when the pre-preg is unrolled for cutting after B-staging on the take-up cylinder.

Thus, the repositioning of the first set of pressure rollers in conjunction with the addition of the bath coating system and the large take-up cylinder served to reduce the voids in the pre-preg to a bare minimum. With this system, the only voids visible in the pre-preg were sparsely populated beads of air less than one thousandth of an inch in diameter. Very few voids could be observed without the aid of a microscope. Microvoid contents of the finished laminates of the three fabrics are compared in section E2a.

d. Pre-Preg Uniformity

The uniformity of the pre-preg was investigated on each machine run and was found to be excellent. The resin content varied less than ± 1 per cent across both the length and the width dimensions at the 95 per cent confidence level for pre-preg productions of over 200 inches in length. The frequency distribution curves for all the productions of pre-preg used in the main research efforts were normal; however, curves for some of the preliminary coating machine runs were slightly skewed.

To illustrate the thoroughness of the pre-preg investigations, the examination of a typical production of the material will be presented in detail. The particular machine run presented is for a 181 3-ply used in the perliminary fabrication investigations. Figure 4 shows the extent of the sampling.

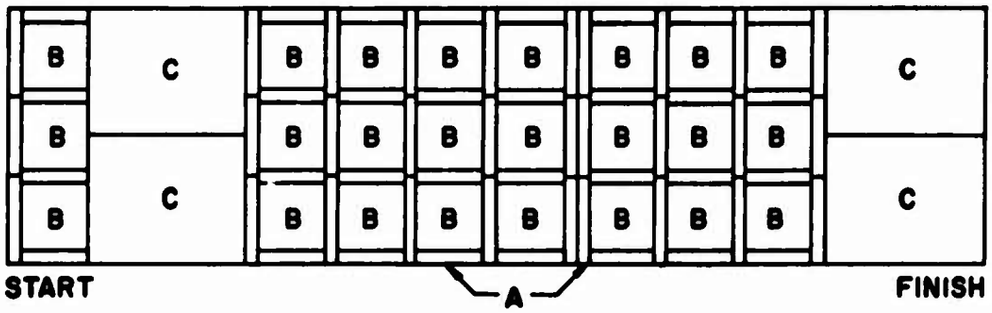


Figure 4. Typical Layout of Pre-Preg for a Resin Content Survey. A, 2-Inch-Wide Strips for Resin Content Sampling; B, 12-Inch-Square Specimens Used for Preliminary Fabrication Investigations; C, 22- by 28-Inch Specimens Used for Sandwich Facings.

The 2-inch-wide strips from the length and width dimensions were laid flat after cutting and cured in a large re-circulating hot-air oven under the weight of a 1/16-inch-thick, 22- by 28-inch aluminum caul. The cure used was 200 degrees Fahrenheit for 2 hours. After cure, the strips were rough cut into 2-inch squares with a sheet-metal shear and then ground to squareness. The grinding was done in blocks of 10 specimens, from which 4 were randomly chosen to obtain the measurements of the entire group. This scheme greatly increased the speed of measurement and introduced an error of no more than 0.10 per cent. The length and width measurements were read to the nearest 0.001 inch. The specimens were weighed individually to the nearest 0.001 gram.

Since the weights per unit area of the fiberglass fabrics were used in the resin content calculations, it was necessary to sample each fabric. This test also gave insight into the uniformity of the finished laminate (see section E1c). Twelve 14- by 15-inch rectangles were die cut from each of the three fabric styles (181, 120, and 909) and carefully weighed to the nearest thousandth of a gram on an analytical balance. Pieces were cut in a non-ordered way from the widths of the materials

(fabric widths were: 181 style, 44 inches; 120 style, 44 inches; 909 style, 38 inches) at approximately 250-inch separations along the length. The sampling was from three lots of fabric in the case of the 181 style and from a single lot for each of the 120 and 909 styles.

The dimensions of the die varied less than 0.016 inch as determined by 5 random measurements made across both the length and the width. The cuts were made with the fabric sandwiched between 1-mil-thick polyethylene covers to prevent handling and with the sides of the die parallel to the warp and weave of the fabrics. The cuts were very smooth and entirely complete in most cases. The occasional fiber that did remain only partially cut was released with scissors. After cutting in the press, the samples were carefully transferred to polyethylene sacks for weighting. The values of weight per unit area obtained and the corresponding dispersions are presented in section E1c.

The resin content was then calculated by the following equation:

$$RC = \frac{W_a - W_g}{W_a} \times 100$$

where

RC = resin content
W_a = weight of specimen per unit area
W_g = weight per unit area of the total number of plies of fabric

The frequency distribution curve for the sampling is shown in Figure 5 along with other statistical parameters. As can be seen, the distribution is very close to normal.

e. Machine Controls

Several variables were found to influence the setting and operation of the coating machine to obtain a desired resin content as well as the replication of this value. Vat temperature during machine operation, wind motor speed, and the roller spacings were observed to affect the control of resin content the most. Figure 6 is included in this discussion to indicate the control that is possible for the present type of coating machine. Needless to say, the greater the control on the operational variables, the greater the control of the

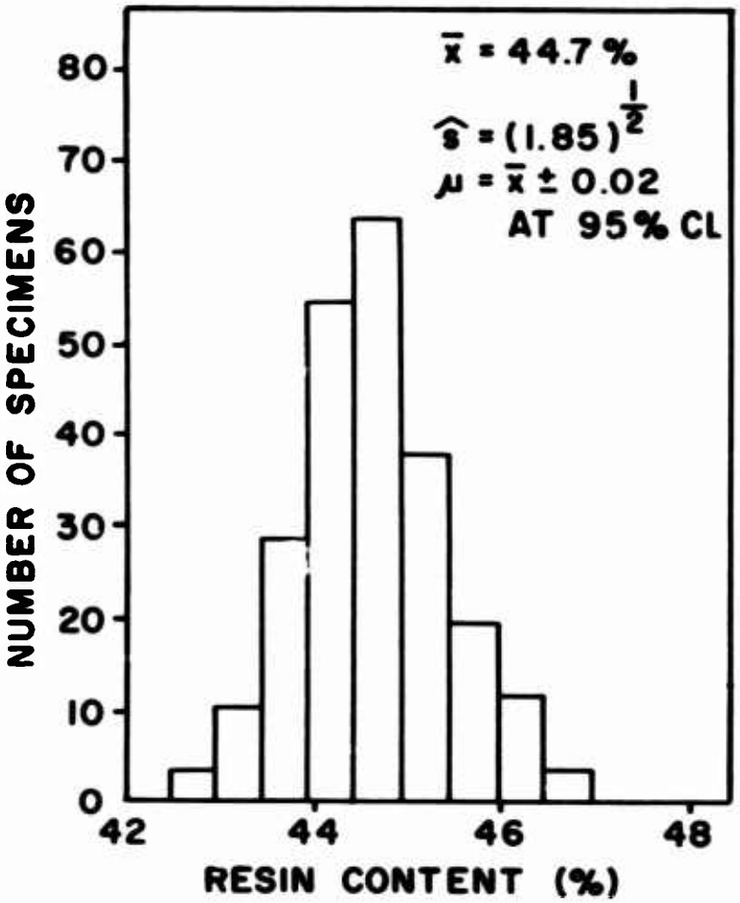


Figure 5. Typical Frequency Distribution for Machine Coated Pre-Preg. The sampling is detailed in Figure 4.

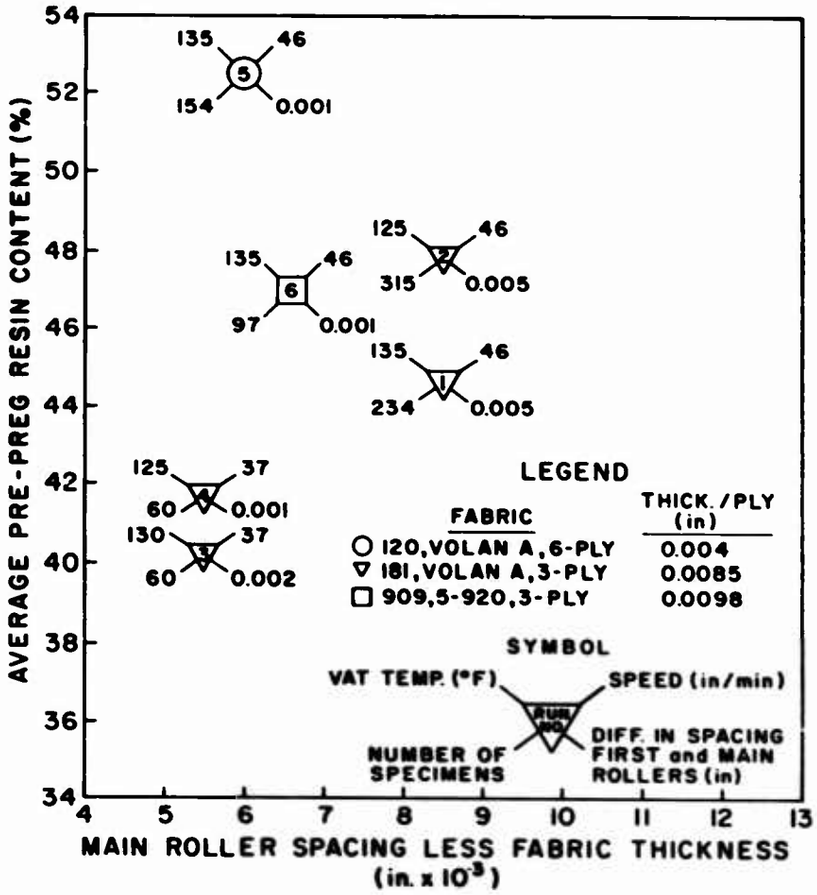


Figure 6. The Effect of Coating Machine Control Parameters on the Multi-Ply Pre-Preg Resin Content.

finished pre-preg. These variables were deliberately changed on the 181, 3-ply productions to observe the greatest run-to-run deviations to be expected. Also, these changes provided the different initial resin content values necessary to observe the control of the pre-preg on the final resin content of FRP laminates (section E1b). As can be seen, increasing the control of the vat temperature and fixing the wind motor speed, and also the relation between the roller spacings, caused the run-to-run variation of the pre-preg to approach the ±1 per cent of the within-run variation. This suggests that further elimination of the human element by automatic control of the entire process should give a high degree of uniformity and replication.

2. Laminating Press

The second significant piece of fabrication equipment was the laminating press. The hydraulic press used for molding and curing FRP laminates was designed and constructed under a previous USAAVLABS contract (reference 16). The basic press was modified in several ways for increased accuracy during the present research program. The modifications are brought out at the proper point in the description that follows.

The press is operated by an 11-inch-diameter hydraulic cylinder. Hydraulic pressure is developed by a pump and vari-drive electric motor system contained within the press console (Figure 7). There are two automatic speed control positions on the console: one for high-speed opening and closing of the press and the other for low-speed adjustment of the final platen pressure. Low platen pressure is set by a 0-200 psi hydraulic pressure gage (A in Figure 7), and high platen pressure, by a 0-1000 psi gage (B in Figure 7). The 200-psi gage was added at the beginning of the present research program. The 60-ton-capacity press is presently calibrated to approximately half capacity, or 100-psi platen pressure. An Amsler mercury compression head was used for the calibration.

The press platens were fabricated from 2-inch-thick 7075 aluminum plate. The working area of each is 24 inches wide and 28 inches long. Twenty 1/2-inch-diameter, high-strength steel bolts screwed to the back of each platen serve to transmit the compressive load from the press heads to the platens. The working surface of the platens and the ends of the bolts were ground flat and parallel to within 0.001 inch.

Each platen is electrically heated by six equally spaced Chromalox heating strips bolted to the back surface in the length direction.

Figure 7. Hydraulic Laminating Press and Controls. A, 0-200 psi Pressure Gage for Setting Platen Pressures Below 30 psi; B, 0-1000 psi Pressure Gage for Setting High Platen Pressures; C, Barber-Colman Regulating Pyrometer for Heating of Platens.

Fiberglass mat is used to insulate the heaters and the back surface from the rest of the press. Water cooling of the 20 support bolts prevents heat transfer through the bolts--each bolt is wound with two layers of 1/8-inch copper tubing. Four strategically located thermocouples are used to monitor temperature across each platen. Three of the thermocouple locations are drilled (number 34 drill) to within 1/4 inch from the working surface. The thermocouples consist of iron-constantan wire twisted and welded on the end.

The 2-inch-thick platens supported by the water-cooled steel bolts represent an improvement in the design of the original press (reference 16). Originally, the platens were thinner and were supported (backed up) by layers of Masonite and Transite, which not only insulated the platens from the rest of the press but also carried the load. Deterioration of this back-up material permitted warping of the platens. This redesign was accomplished after the preliminary investigations in fabrication but prior to the major research effort.

Electric power is supplied by a 220-volt, 3-phase circuit controlled by a model 292P Barber-Colman regulating pyrometer (C in Figure 7). This instrument has a calibrated range of 0 to 600 degrees Fahrenheit in 5-degree increments, and is effective in controlling the platen temperature within 5 degrees. Between 160 and 350 degrees Fahrenheit in the steady-state condition, the observed variation in temperature across the platens is less than 5 degrees.

C. Preliminary Investigations

1. Fabrication

In view of the large number of variables involved in the fabrication of FRP materials, it is convenient to hold several of them constant, at least as near as possible. Hence, it is necessary to know the overall effect of these variables not only for the purpose of optimization but also for the setting of realistic controls. Likewise, it is also necessary to know the effects of certain handling procedures of the raw materials between the steps in the fabrication sequence. Thus, to place the major research program on more solid footing, it was preceded by a series of preliminary investigations.

These initial investigations were also expected to suggest ways of improving the fabrication process, and further, the small compact

experiments were designed to contribute to the main objectives: the study of the effects of resin content and void inclusions on the mechanical properties.

The variables held constant in the main research effort were to be length of B-staging, condition of B-staging (under residual tension on the coating machine take-up cylinder), length of post-cure (afterback), and time of pre-cure; consequently, these were investigated first. The most important material handling operation was the cold storage of the raw pre-preg after the room-temperature B-staging; hence, the effect of cold storage on mechanical properties was also sought. Though many of these items had been examined briefly in previous research programs, they were observed in detail at this point.

During the execution of these experiments, the need for more information on surface smoothness and the effect of laminate size was also recognized. Hence, additional laminates to cover these items were included in the preliminary investigations.

Compressive strength was chosen as the criterion for evaluation in the various areas of concern. The 3-ply pre-preg of the 181 style fiberglass fabric woven by the Exeter Manufacturing Company was used exclusively for these studies. Except for the zero-hour B-stage specimens which were cut off immediately and placed in cold storage, the pre-preg was allowed to B-stage at room temperature to within 1 hour of the desired time (1/2 hour was consumed in cutting and the other in cold storage cool-down and warm-up) while wound on the coating machine take-up cylinder. It was then rolled out on a large cutting table, marked, cut into laminate specimens with scissors, and placed in cold storage at an average temperature of 19 degrees Fahrenheit. Figure 4, on page 9, stands as a typical pre-preg cutting diagram. Two separate productions of pre-preg were employed in these initial studies (runs 1 and 2).

Both the assignment of the laminate specimens to the various experiments and the establishment of the sequence of their cure were accomplished by picking them from the pre-preg cutting diagram with the aid of a random number table. To prepare for cure, the raw laminates were taken from the freezer, thawed to room temperature and stripped of their cover films, and then placed between two 1/16-inch-thick aluminum cauls coated with Dow-Corning DC-7 parting agent thinned in the ratio of 1 to 10 with XYLOL. (Note: Other parting agents were evaluated in connection with laminate surface condition and are discussed in section 4.) The assembly was subsequently centered in the previously described hydraulic

press (platens had not been modified) which was set at the desired temperature. The press was closed and the pre-cure accomplished at zero pressure followed by cure under the desired pressure for the remainder of 90 minutes. It should be noted that 90 minutes' cure was established as optimum in the previous research (reference 16). The other conditions of cure varied considerably and thus are best presented by individual experiment. Each experiment is discussed and evaluated in section 4.

There is one aspect of laminate cure that is common to all: this is the gel-time for a thin film of the resin mix at the various cure temperatures. The pre-cure of all the laminates made in the entire project was based on the same gel-time determinations; in fact, the pre-cure is usually reported as minutes before or after (+ or -) gel.

The probe method was employed in these determinations. Three-inch-square, resin-rich, 3-ply impregnations of 181 fabric lying on thin aluminum sheets were placed on the heated press platen and probed with a clean, sharp, wooden dowel until the resin string that was being pulled out broke when it reached 2 to 3 inches in length. The time between placing the specimen on the press to the breaking of the resin string was recorded as the gel-time for the particular B-staging and temperature. Figure 15, on page 28, is a plot of the data thus obtained.

2. Test Specimen Preparation

After cure in the press, each laminate was cut into test specimens using a sheet-metal shear so that their lengths were parallel to the fabric warp direction. Usually 36 specimens were obtained, from which 15 were chosen by use of random number tables and post-cured at 300 degrees Fahrenheit. In the case of the post-cure experiments, 2 samples of 15 specimens each were chosen randomly from the 36 to permit the employment of the paired sample technique of analysis. Laminate number 17 was an exception to this, in that its size (20 inches square rather then 12 inches square) permitted the taking of 6 samples of 15, across which the post-cure could be varied.

Following post-cure, the compression specimens were ground to the final 0.875-inch by 3.67-inch dimensions. They were then weighed to the nearest 0.001 gram and measured in length and width to the nearest 0.001 inch. After testing, the thickness was measured to the nearest 0.001 inch in two places, 1/2 inch on either side of the rupture. Thus, an average thickness was used in the stress area calculation.

3. Test Procedure

To prepare the specimens for testing, they were coated with a powdered molybdenum disulphide lubricant (MolyKote Z). The test coupons were then lightly clamped (screwed finger tight) in the laminate test fixture previously developed (reference 16). This fixture, shown in Figure 8, functioned to prevent buckling of the thin laminates.

The fixture with the test specimen was placed on the lower platform of the 10,000-pound-capacity Instron Testing Machine, and the top of the specimen was fitted into a tapered slot in the upper loading block attached to the crosshead. The specimen was then vertically aligned, and a wedge was inserted into the slot along the end of the specimen to provide a fixed end condition during loading. Figure 9 shows the actual test set-up with exception of the Baldwin compressometer, which was used later in the resin content study.

At a crosshead speed of 0.050 inch per minute, each specimen was loaded to failure. The failure was abrupt and the rupture was always compressive in nature as shown in Figure 10, on page 19. The maximum load recorded was used in the stress calculation. The resin content calculation for each specimen followed that described previously on page 10.

4. Results and Evaluation

The experimental values from these preliminary investigations are listed in Tables 1 and 2, on pages 20 and 21. The values shown are averages for each laminate. The data are next discussed by experiment.

a. Cold Storage

The laminates for this experiment were taken from the first production of the coating machine (run 1) and were cured under identical conditions: B-stage = 10 hours; pre-cure and cure temperature = 160 degrees Fahrenheit; pre-cure time = gel time (16 minutes) minus 4 minutes; cure pressure = 13 psi; cure time = 90 minutes; and post-cure = 2 hours at 300 degrees Fahrenheit. From Table 1, the compressive strengths of the laminates numbered 1, 2a, 3a, 4, 5a, 6a, 7, 8, 9a, 10, 11a, 12, 13, 14, and 15 with cold storage times varying from 6 days to 88 days were compared to ascertain the effect of pre-preg cold storage. The data plot of Figure 11 summarizes the findings.

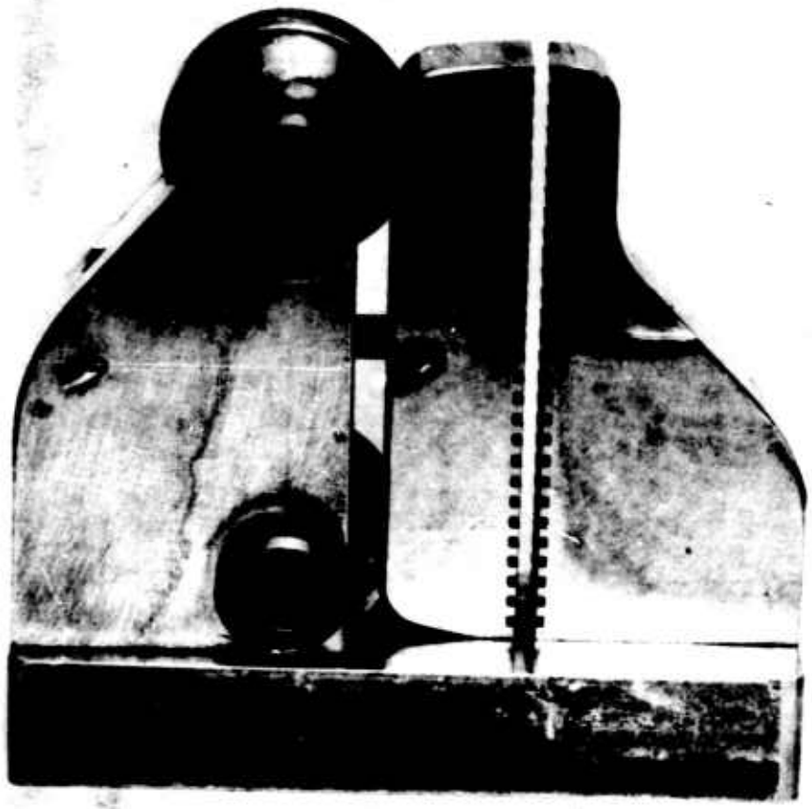


Figure 8. Thin-Laminate Compression Fixture Showing Quick-Release Cams.

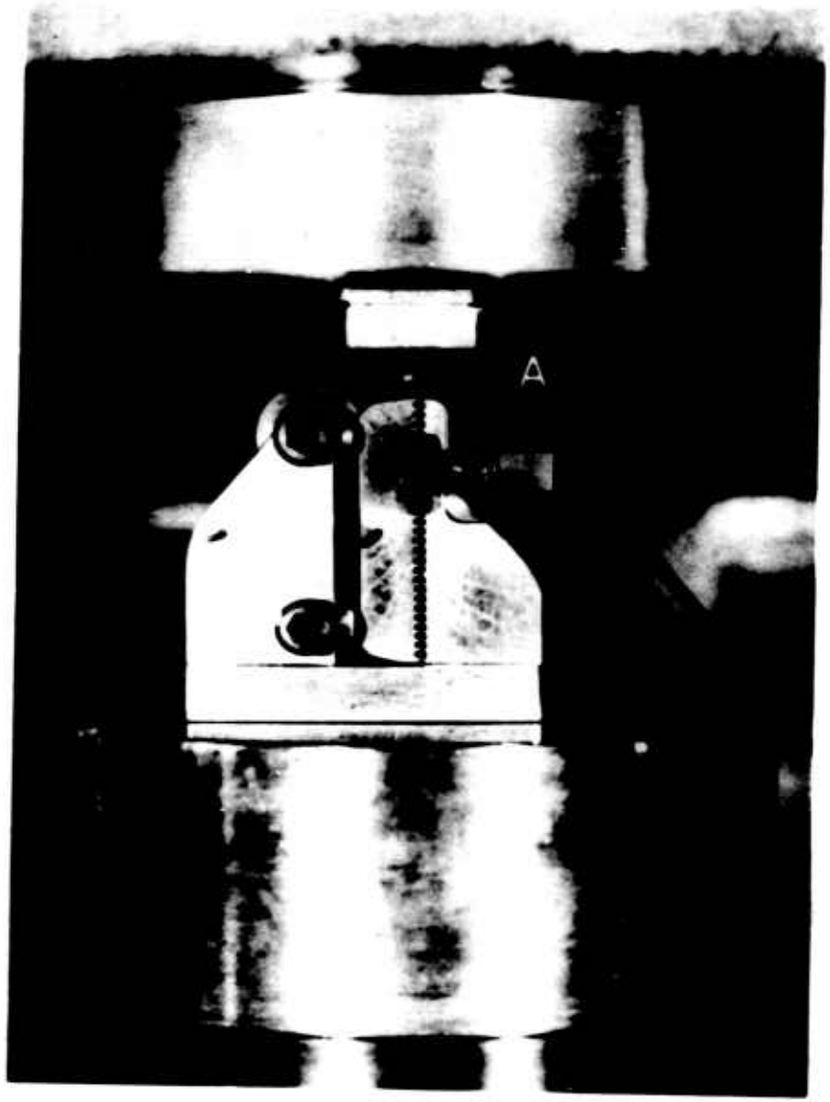


Figure 9. Set-Up for Compression Test of Thin Laminates. Pointer A indentifies the wedge grip.

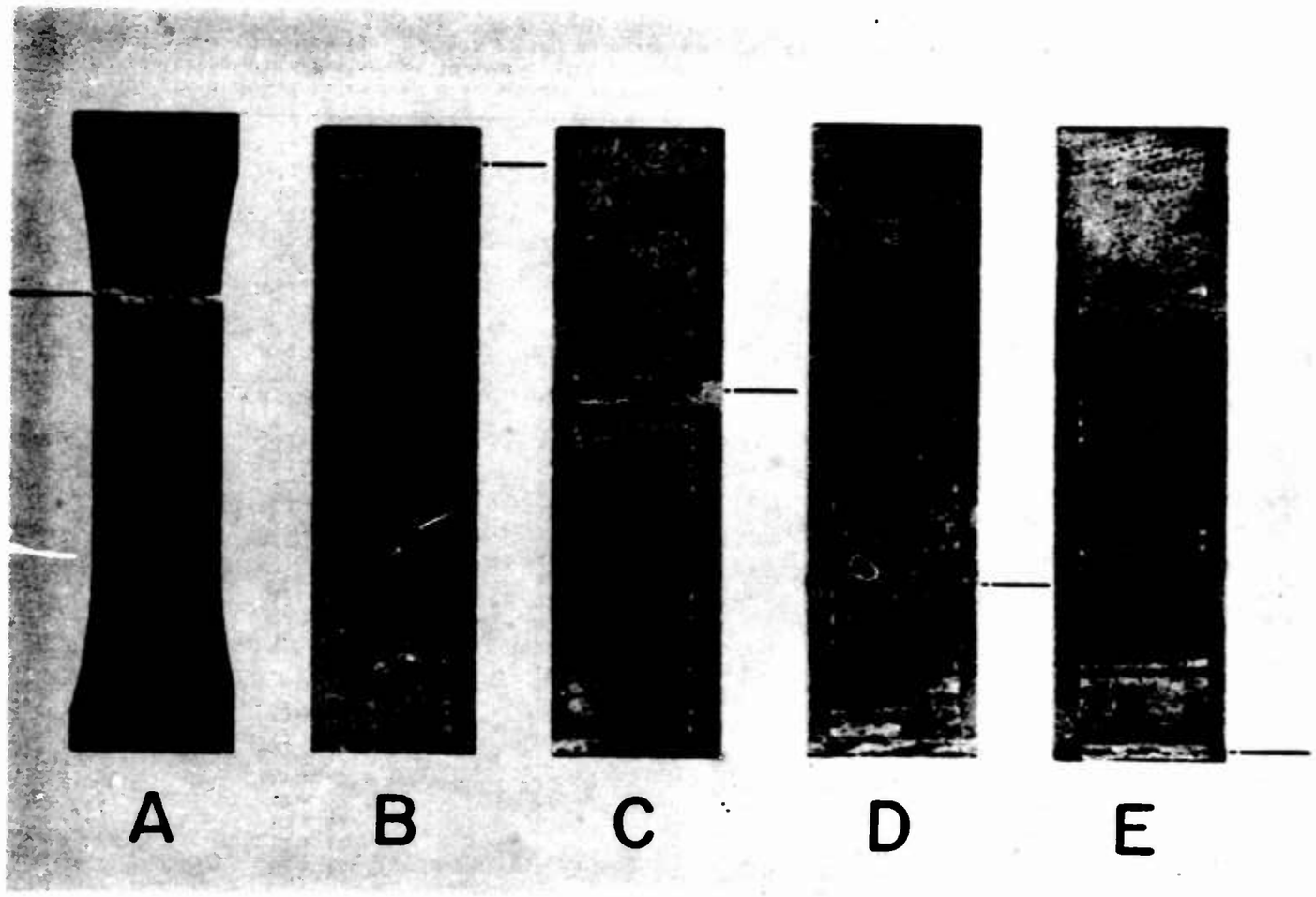


Figure 10. Laminate Compression Failures: A, Specimen Shape Initially Evaluated; B, C, D, and E, Straight-Sided Specimens Which Were Used Throughout the Test Program Showing Various Points of Failure That Occurred Within the Fixture Grips.

TABLE 1
PRELIMINARY STUDIES,
LAMINATE DATA FROM PRE-PREG 1

Laminate Number*	Laminate Size (Nominal, in. x in.)	Cold Storage (Days)**	Post-Cure (Hours)	Average Compressive Strength (psi)	Resin Content (%)	Specific Wt. (gm/in^3)
1	12 x 12	6	2	40,500	39.0	29.2
2a	12 x 12	7	2	40,900	37.3	29.2
2b	12 x 12	7	2 1/2	41,400	36.6	29.2
3a	12 x 12	7	2	40,000	41.1	28.3
3b	12 x 12	7	2 1/2	40,200	41.5	30.0
4	12 x 12	10	2	38,600	40.9	28.1
5a	12 x 12	12	2	40,700	38.7	28.8
5b	12 x 12	12	6	42,800	39.9	28.9
6a	12 x 12	19	2	38,100	36.4	29.3
6b	12 x 12	19	6	37,700	34.9	28.5
7	12 x 12	22	2	40,900	38.2	28.7
8	12 x 12	25	2	39,700	40.0	28.2
9a	12 x 12	28	2	40,000	40.3	28.2
9b	12 x 12	28	1 1/2	37,000	41.3	27.7
10	12 x 12	32	2	38,900	38.6	28.7
11a	12 x 12	35	2	40,900	42.9	27.5
11b	12 x 12	35	1	39,800	42.1	27.7
12	12 x 12	38	2	39,800	41.9	27.8
13	12 x 12	62	2	34,400	-	-
14	12 x 12	78	2	35,900	-	-
15	12 x 12	88	2	38,300	42.3	-
16	8 1/2 x 8 1/2	-	2	41,000	36.4	29.3
17a	20 x 20	-	2	41,400	40.3	28.4
17b	12 x 12	-	0	37,700	40.3	28.2
17c	12 x 12	-	1	41,500	43.2	27.7
17d	12 x 12	-	3	43,500	43.2	27.9
17e	12 x 12	-	4	42,100	41.8	27.8
17f	12 x 12	-	8	43,200	42.0	27.9
18a	12 x 12	-	2	39,200	-	-
18b	12 x 12	-	1 1/2	39,600	38.9	28.4
19a	12 x 12	-	2	41,800	40.6	28.6
19b	12 x 12	-	2 1/2	42,100	38.8	29.0
20	12 x 12	-	2	42,500	40.0	28.4

* The pre-preg was taken from coating machine run number 1 with an average resin content of 44.7% and a room temperature B-stage of 10 hours. The pre-cure and cure temperature was 160°F, the pre-cure was gel minus 4 minutes (gel = 16 min.), the cure pressure was 13 psi, and the cure time 90 minutes.

** Where values are not shown, cold storage is less than 6 days.

TABLE 2
PRELIMINARY STUDIES,
LAMINATE DATA FROM PRE-PREG 2

Laminate Number*	B-Stage (Hours)	Pre-Cure (Minutes)**	Cure, Pre-Cure Temperature (°F)	Post-Cure (Hours)	Average Compressive Strength (psi)	Resin Content (%)	Specific Wt. (gm/in^3)
21a	10 3/4	-4	160	2	39,800	39.9	28.3
21b	10 3/4	-4	160	8	41,400	36.7	29.0
22	10 3/4	-4	160	2	40,100	40.1	28.9
23	10 3/4	-4	160	2	41,200	41.2	30.4
24a	10 3/4	-4	160	2	41,500	39.8	28.2
24b	10 3/4	-4	160	4	41,700	42.1	28.1
25	17/60	-4	160	2	38,700	40.3	27.9
26	4	-4	160	2	36,700	41.7	28.3
27	6	-4	160	2	37,600	35.2	29.5
28	8	-4	160	2	39,100	39.7	28.6
29	14	-4	160	2	40,900	-	27.5
30***	16 2/3	-4	160	2	37,300	-	27.2
31	23 1/2	-4	160	2	40,500	-	26.7
32	10 3/4	-4	160	2	40,500	-	27.5
33	10 3/4	-13	160	2	39,200	37.9	28.5
34	10 3/4	-8	160	2	40,500	40.4	27.7
35	10 3/4	-1	160	2	40,300	43.3	27.4
36	10 3/4	+2	160	2	39,200	43.9	27.6
37	10 3/4	+8	160	2	40,000	43.3	27.5
38	10 3/4	-3	230	2	39,400	38.2	28.1
39	10 3/4	+2	230	2	37,200	38.8	28.7
40	10 3/4	+6	230	2	35,200	39.0	27.1
41	10 3/4	+6	230	2	37,300	38.1	37.1
42	10 3/4	-1	230	2	38,600	39.6	28.3
43	10 3/4	-1	230	2	40,000	39.6	28.1
44	10 3/4	+12	230	2	37,700	39.8	27.5
45	10 3/4	+18	230	2	37,100	40.5	27.5

* The pre-preg was taken from coating machine run number 2 with an average resin content of 47.9%. The cold storage time was less than 6 days. Laminates are 12-inch squares cured at 13 psi for 90 minutes.

** Pre-cure time in minutes before (-) and after (+) the 16 minutes gel time at cure temperature 160°F and the 6 minutes gel time at 230°F.

*** Specimens were inadvertantly sized too long.

The tests were rather closely spaced up to 38 days, and two values of laminate strength where the pre-preg has been stored above 60 days are well below the normal scatter of data. It was, therefore, concluded that it would be conservative to use pre-preg in the resin content study that had been stored 40 days or less.

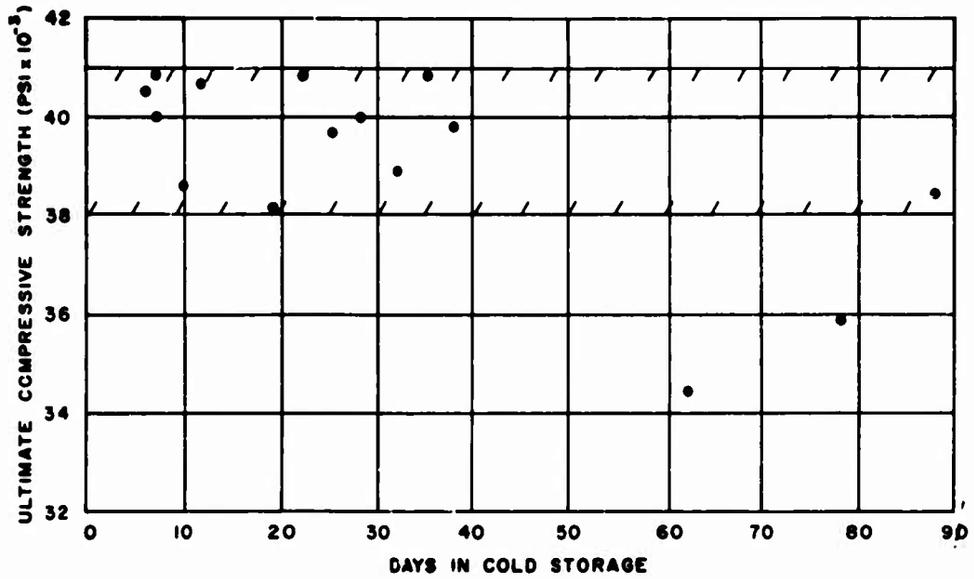


Figure 11. The Effect of Pre-Preg Cold Storage on the Compressive Strength of the Cured Laminate.

b. B-Staging

Laminates for the B-stage experiment were taken from coating machine run 2 and are numbered 21a, 22, 23, 24a, 25, 26, 27, 28, 29, 30, and 31 in Table 2. The compressive strength values for laminate 30 were rejected because the specimens were inadvertently sized longer than any of the others. The conditions of cure were: cold storage = 6 days or less; pre-cure and cure temperature = 160 degrees Fahrenheit; pre-cure time = gel time (16 minutes) minus 4 minutes; cure pressure = 13 psi; cure time = 90 minutes; and post-cure = 2 hours at 300 degrees Fahrenheit. The pre-preg B-staging was varied from less than an hour to slightly over 23 hours.

Figure 12 is a plot of the test results. For convenience, only one value, one near the average (laminate 22), was plotted for the 10-3/4-hour B-stage. Also, laminate 17b from pre-preg 2 was plotted for an estimate of the zero B-stage strength. The figure indicates that a trend does exist and that the previously used 10-hour B-staging is a good working value, in view of the data scatter and possible interactions with other fabrication variables. It should also be noted that B-staging increases the handling qualities of the pre-preg. Thus, the use of the 10-hour B-staged material was continued for the resin content study. Also, from the figure, it appears that controlling the B-staging within plus or minus 15 minutes would be sufficient.

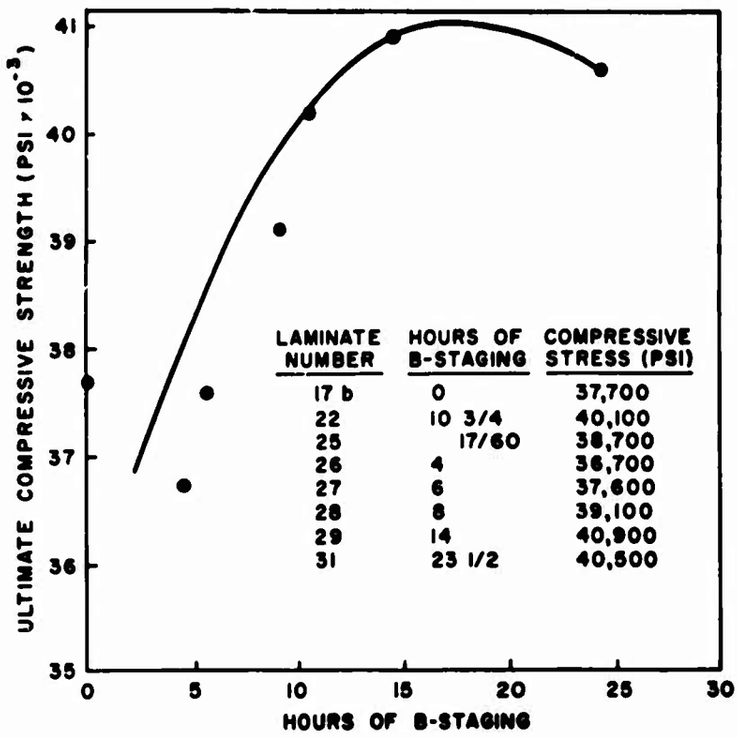


Figure 12. The Effect of Pre-Preg B-Staging on the Compressive Strength of the Cured Laminate.

c. Post-Cure

The paired-sample technique of analysis was used in the post-cure experiment. The experimental data were arranged for analysis as shown in Table 3, on the next page. Tables 1 and 2 show the common conditions of cure.

Assuming the differences to be normally distributed, the student's t test of significance was accomplished as indicated on the next page:

Mean Difference: $\bar{\bar{x}} = 263$ psi

Estimated Standard Deviation of Differences: $\hat{s} = \left[\frac{\sum(\bar{x}_i - \bar{\bar{x}})^2}{n - 1}\right]^{1/2} = 1950$ psi

Standard Error of the Mean: $\hat{s}/\sqrt{n} = 689$

$t = \bar{\bar{x}} - (\hat{s}/\sqrt{n}) = 0.38$

TABLE 3
EXPERIMENTAL DATA ARRANGEMENT
FOR ANALYSIS OF POST-CURE EFFECT

Laminate Number	Post-Cure Time at 300°F (Hours) 0	1	1 1/2	2	2 1/2	3	4	6	8	Difference of Means (psi)
17	37,700			41,400						-3,700
17		41,500		41,400						+100
12		39,800*		40,900						-1,100
18			39,600*	39,200						+400
10			37,000	40,000						-3,000
3				40,900	41,400					+500
19				41,800	42,100*					+300
4				40,000	40,200					+200
17				41,400		43,500				+2,100
17				41,400			42,100			+700
24				41,500			41,700*			+200
6				40,700				42,800*		+2,100
7				38,100				37,700		-400
17				41,400					43,200*	+1,800
21				39,800					41,400	+1,600

* The values of ultimate compressive strength indexed were chosen randomly for use in the paired-sample analysis. All values are compaired with those at 2 hours post-cure.

Thus, it is concluded that the values of compressive strength obtained from room temperature tests are not significantly different. A plot of the values for the samples obtained from laminate number 17 verifies this conclusion except at the very low values of post-cure where the strength obviously decreases (Figure 13).

Also at the short post-cure times, there was an interesting phenomenon noticed in regard to the cutting of the laminates. At zero post-cure, the laminates were observed to delaminate when cut. To obtain further information, a series of 181 specimens, post-cured at increasing times, were cut on the metal shear and the average distance of delamination from the cut edge was measured. As shown in Figure 14, on the next page, post-cure improves the room temperature interlaminar shear properties of the laminates, this being evidenced by the improved cutting characteristics. The delamination caused by cutting seems to become a minimum at about 40 minutes of post-cure, in the case of the 181 laminates.

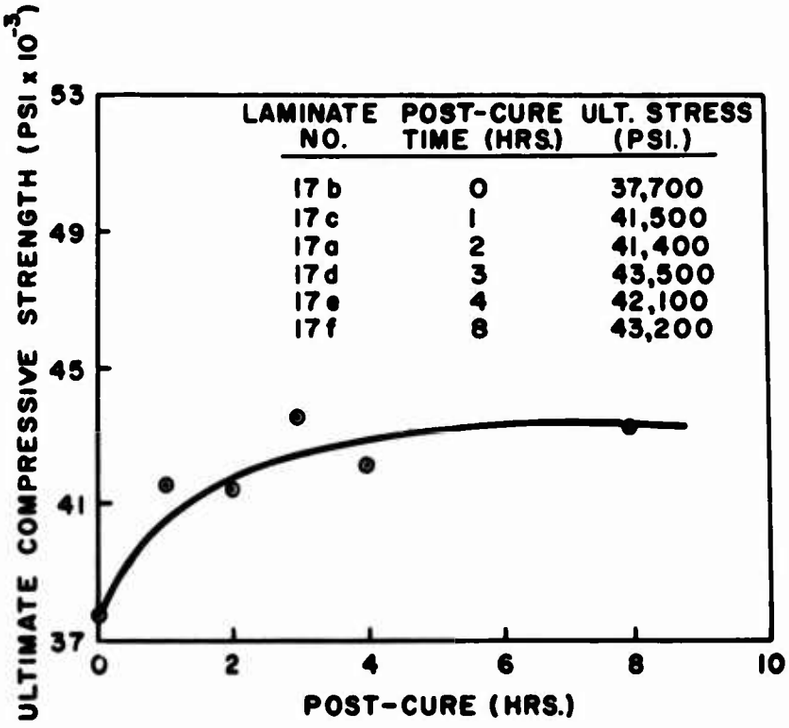


Figure 13. The Effect of Post-Cure at 300°F on the Compressive Strength of the Cured Laminate.

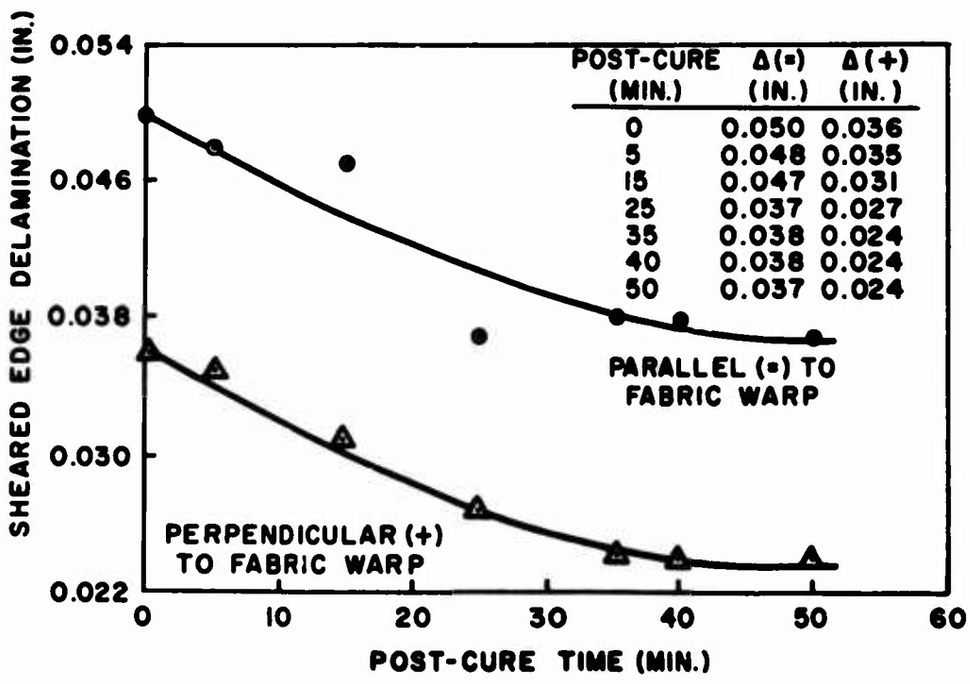


Figure 14. The Effect of Post-Cure at 300°F on the Cutting of the Cured Laminate.

Thus, on the basis of the data presented in Figures 13 and 14, the 2-hour post-cure used in previous programs was retained for the resin content study.

d. B-Staging Under Winding Tension

Three 12-inch-square specimens were cut from the pre-preg at the conclusion of coating machine run 1 and were allowed to B-stage on the flat. The other specimens for this experiment were taken from the center of the length of the pre-preg that had B-staged while wound on the coating machine take-up cylinder. Table 4 displays the data obtained, clearly showing that the values for the two situations do not differ significantly; hence, it is concluded that the B-staging on the coating machine take-up cylinder under residual winding tension does not significantly affect the strength of the finished laminates.

TABLE 4
EXPERIMENTAL DATA FOR
COATING MACHINE WINDING EFFECT

Laminate Number	B-Stage Location	Compressive Strength (psi)
1	On Machine	40,500
3a	On Machine	40,900
4a	On Machine	40,000
18a	On Flat	39,200
19a	On Flat	41,800
20	On Flat	42,500

e. Laminate Size

Observations made during laminate fabrication indicated that greater flow took place during the press cure of small laminates than occurred for full-size laminates (22 by 28 inches). Therefore, it was suspected that the size of the laminate sampled for strength properties could influence the results. To determine whether the possible effect was significant, a comparison was made of the average compressive strength of laminates of 3 sizes fabricated under the same conditions. The data are presented in Table 5. It can be seen that the differences are not significant, which is consistent with the

findings stated on page 2 of reference 24. Hence, the laminate size for the resin content study can be chosen on the basis of the desired sampling.

TABLE 5
EXPERIMENTAL DATA
FOR LAMINATE SIZE EFFECT

Laminate Number	Laminate size (Nominal, in. x in.)	Compressive Strength (psi)
3a	12 x 12	40,900
16	8 1/2 x 8 1/2	41,000
17a	20 x 20	41,400

f. Gel-Time Determination

A closer look at the method of estimating the resin gel-times is in order before discussing pre-cure effect. It was noted in the laboratory that the probe method was a rather sensitive test: actually sensitive enough to observe the resin flow develop as the temperature of the specimen increased and then subside as gellation took place. Thus, good results were expected from the tests. As can be seen from the gel-time curves in Figure 15, the consistency of the measurements is good for the wet lay-up (zero B-stage) but tends to decrease with the time of B-staging. Even so, the average values form a uniform family of smooth curves.

A plot of resin content versus pre-cure in terms of time before and after this gel point (Figure 16) suggests that the method may actually underestimate the true gel-time, in that the resin content continues to increase beyond the zero point. Had zero on the abscissa been the actual gel-point, no flow, and hence no increase in resin content, should have occurred in the laminates pre-cured beyond this point (positive values of time). Nevertheless, the method is consistent, and it is effective in tieing together all of the experiments.

g. Pre-Cure

The data used in the pre-cure experiment were obtained from compression tests of the laminates numbered 33 through 45 in Table 2. They were cured from the second production of pre-

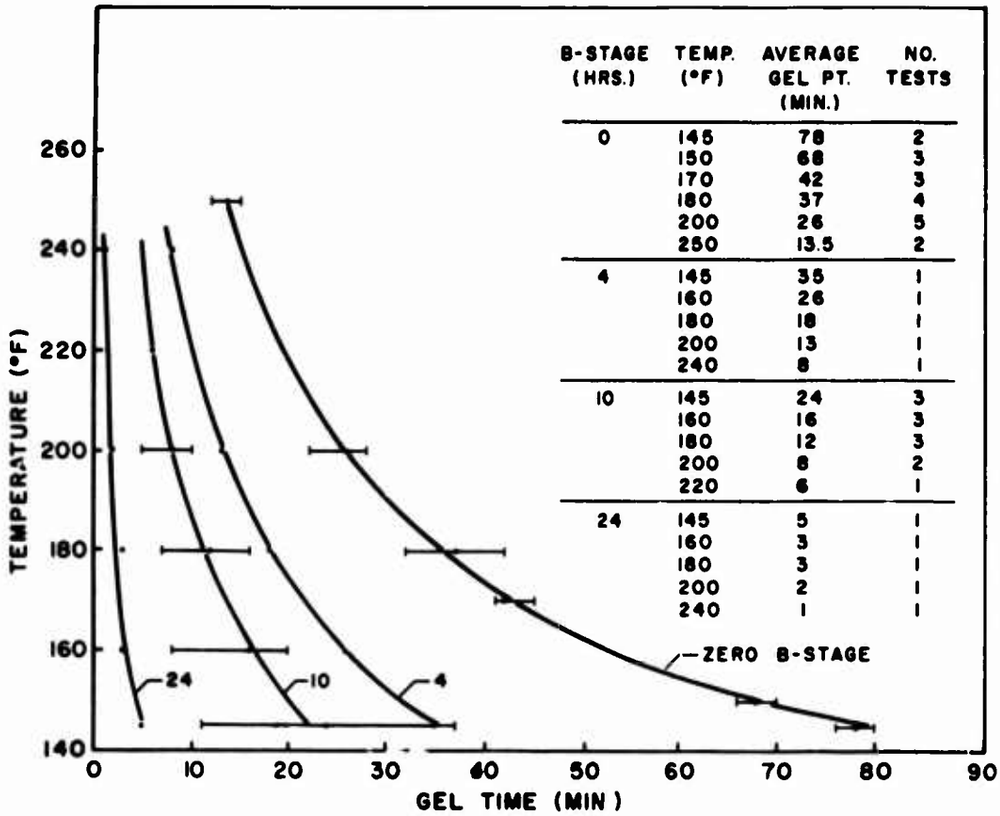


Figure 15. The Relation Between Temperature and Gel Time for EPON 828-Z Epoxy Resin. The tests were performed on 3-ply lay-ups of 181 Volan A fiberglass fabric.

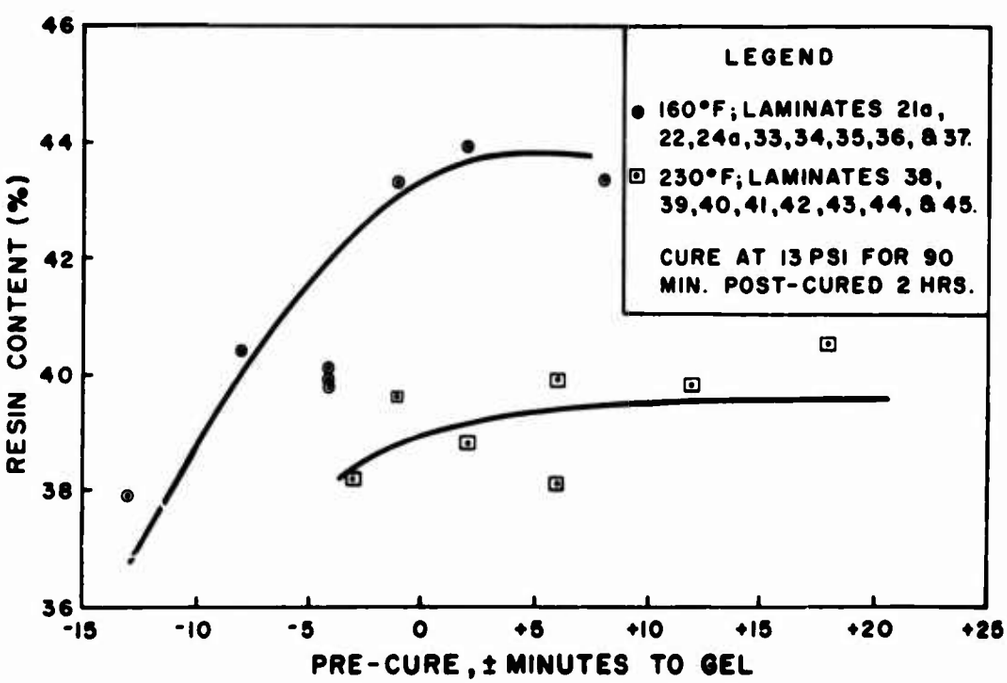


Figure 16. The Relation Between Laminate Resin Content and Pre-Cure Time. The pre-preg resin content was 47.9 per cent.

preg (run 2) under the following conditions: B-stage = 3/4 hours; cold storage = 6 days or less; pre-cure and cure temperature = 160 and 230 degrees Fahrenheit; cure pressure = 13 psi; cure time = 90 minutes; and post-cure = 2 hours. The pre-cures were varied from several minutes before to several minutes after the estimated gel time of the resin at the two temperatures, 160 and 230 degrees Fahrenheit. The results of the experiment are summarized in Figure 17, on page 30.

The plot of the data indicates that laminate compressive strength is related to the pre-cure time, with a maximum occurring in the vicinity of gel time minus 4 minutes. Though the shapes of the curves suggested by the data are similar, relative to gel-time there is a large interaction with strength. For example, the ratio of pre-cure time to gel time for the temperatures, 160 and 230 degrees Fahrenheit, is 12/16 = 75 per cent and 2/6 = 33 per cent, respectively. Since the main requirement for pre-cure in the resin content study was that it be systematic, an average value of 50 per cent of the gel time (as obtained from Figure 15, on page 28) was used for this purpose. Examination of the individual specimens fabricated at a pre-cure of 50 per cent gel also suggested that a good surface would be produced; however, this examination was only qualitative and, thus, not conclusive.

h. Surface Study

At this point, an additional study was made regarding laminate surface condition. In these preliminary investigations as well as in previous research, the surfaces of the laminates fabricated using the Dow-Corning DC-7 liquid and using Teflon sheets as a mold release were rather dull in appearance and contained numberous tiny pick-outs (examine specimens in Figure 10, on page 19). It was felt that a smoother surface was desirable for aircraft structural applications; hence, several laminates were molded using 4 candidate parting agents or mold releases. The condition of the surfaces of the laminates was observed and classified. Table 6 summarizes the findings.

Though the experiment was only qualitative in nature, the results clearly showed that a change from the previously used mold release agent was in order. Thus, the 6-mil polyvinyl alcohol (PVA) film was chosen for use in the resin content study.

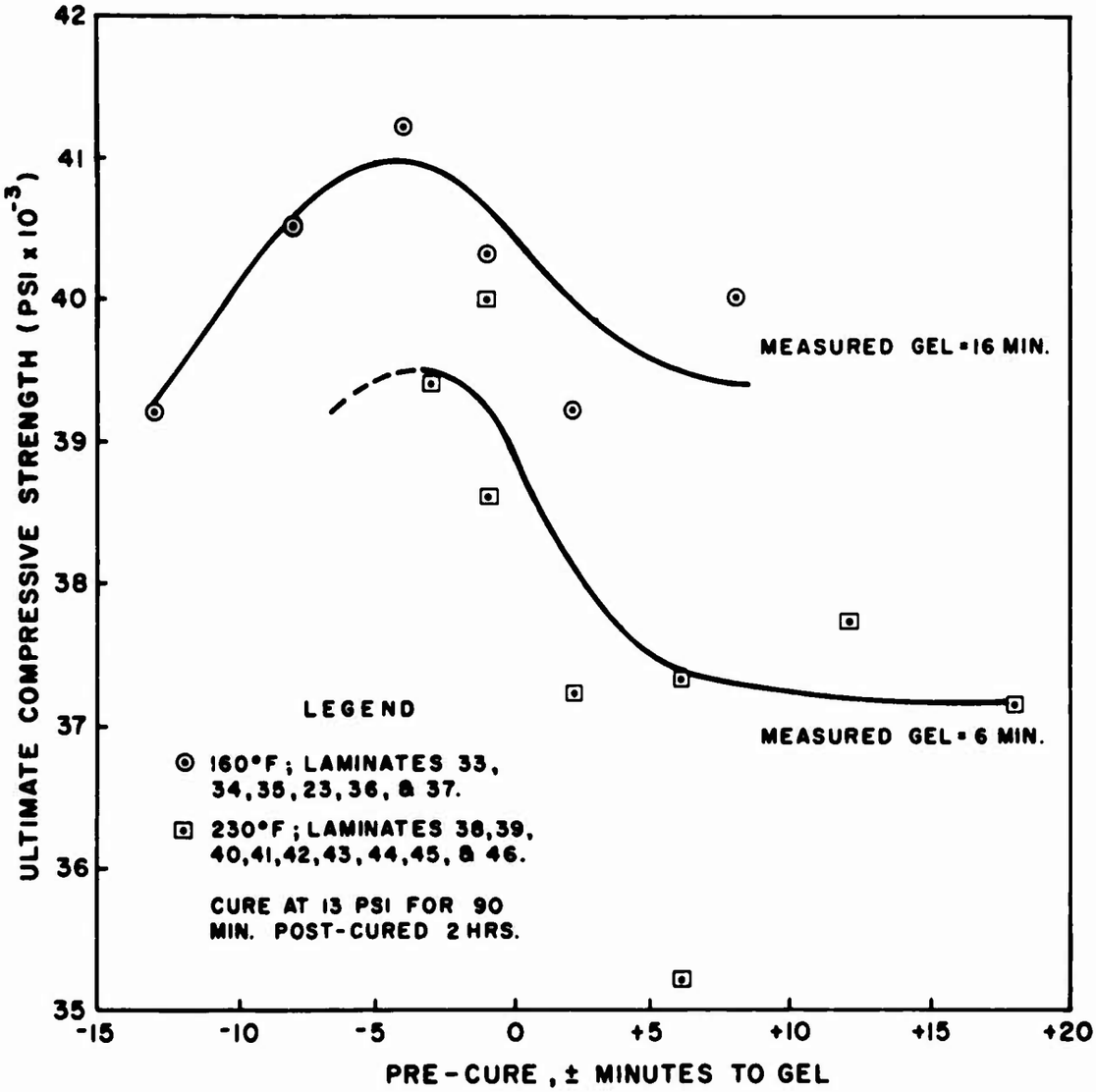


Figure 17. The Effect of Pre-Cure on the Compressive Strength of the Cured Laminate. The pre-preg resin content was 42.9 per cent.

TABLE 6
MOLD RELEASE EVALUATION

Laminate Size (in. x in.)*	Parting Agent	Observations	
		Surface	Extent
6 x 6	Dow-Corning DC-7 (1:10 mix with XYLOL)	Pick-outs Non-Glossy	Patches
6 x 6	Dow-Corning DC-7 (100% Concentration)	Weave Ridges Non-Glossy	Patches
6 x 6	Partall	Weave Ridges Non-Glossy	Patches
6 x 6	Polyethylene Film (0.001 inch thick)	Film Wrinkles	Partial
7 x 5 1/2	Polyethylene Film (0.003 inch thick)	Smooth Glossy	Overall
6 x 6	Polyvinyl Alcohol Film (0.006 inch thick)	Smooth Glossy	Overall
22 x 28	Teflon Sheet (1/16 inch thick)	Pick-outs Non-Glossy	Patches
22 x 28	Dow-Corning DC-7 (1:20 mix)	Pick-outs Non-Glossy	Patches
22 x 28	Polyethylene Film (0.001 inch thick)	Film Wrinkles	Partial
22 x 28	Polyvinyl Alcohol Film (0.006 inch thick)	Smooth Glossy	Overall

* The laminates were fabricated from 10-hour B-staged pre-preg at a pre-cure of 13 minutes and a cure of 90 minutes at 13 psi and 160°F.

D. Experimental Procedure

As with the preliminary investigations, the procedures employed in the main research effort, the resin content study and the void study, are discussed according to fabrication, specimen preparation, and test procedure.

1. Fabrication

On the basis of the preliminary investigations, the fabrication of the laminates was carried out with confidence. The pre-pregs were B-staged for 10 hours, cut into laminates, and placed in cold storage as described previously on page 15. They were sized 14 by 20 inches to permit the desired sampling. In all cases the laminates were cured prior to the expiration of 40 days' cold storage.

The 3-ply 181 laminates were obtained from pre-preg run 4, the 120 laminates from run 5, and the 909 laminates from run 6. The cure procedure employed also followed that previously described on page 16. The new press platens had been installed (see page 12) and the 6-mil PVA was used as the parting agent in all cases. The pre-cure period was 50 per cent of the gel time obtained from Figure 15 and was controlled to within 1 minute. The total cure duration was 90 minutes plus or minus 1 minute. The cure pressures and temperatures employed are displayed by the data tables in the appendix for each style of fiberglass fabric. Pressure was controlled to within 3 per cent.

A special laminate 22 by 28 inches in size was prepared for the void study. The 3-ply 181 pre-preg from run 4 was used in this case. One ply was peeled from the laminate, and 1-mil-thick polyethylene discs of 1/2-, 1/4-, and 1/8-inch diameter were placed according to a scheme to be described later. The outside ply was then laid back in place without wrinkling. A great deal of care was required for this whole operation, as the resin is still tacky in the 10-hour B-staged condition. The laminate was then prepared for cure as the others. It was pre-cured 12 minutes at 160 degrees Fahrenheit and cured under 30-psi pressure for the remainder of 90 minutes.

Prior to cutting, each laminate was post-cured for 2 hours at 300 degrees Fahrenheit in a recirculating hot-air oven accurately controlled to ±10 degrees.

2. Test Specimen Preparation

The laminates were next cut into test specimens using a sheet metal type shear. In all cases, the length dimensions were cut parallel

to the fabric warp direction according to a scheme similar to the one shown in Figure 18. At least 10 specimens per laminate were taken for each type of test to be conducted (compressive, tensile, and flexure), and they were ground to squareness and to the following final dimensions in groups of 10: compression--length = 3.67 inches and width = 0.875 inch (both within 0.005 inch); tension--length = 9.37 inches (within 0.01 inch) and width = 0.750 inch (within 0.005 inch); and flexure--length = 2.0 inches (within 0.01 inch) and width = 1 inch (within 0.005 inch).

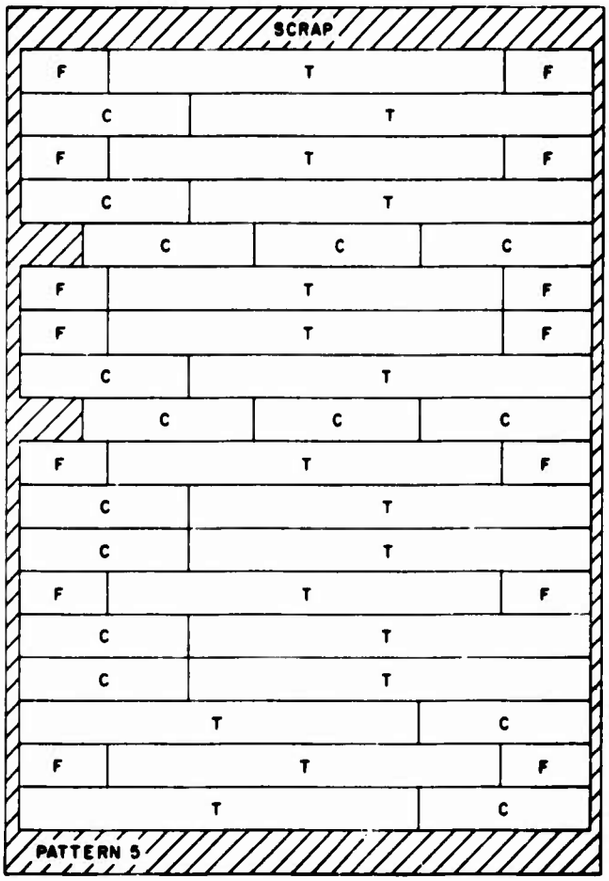


Figure 18. Typical Laminate Cutting Diagram. Types of specimens (C = compression; T = tension; F = flexure) were located in a random fashion.

After grinding, the specimens in each group of 10 were measured in length to the nearest 0.001 inch (to the nearest 0.01 inch for the tension specimens) and in width to the nearest 0.0001 inch by a sampling of 4 picked in a random fashion. The thickness of each specimen was likewise measured, but after testing, at a location 1/2 inch on either side of the rupture. The average of these two values was used in the area calculation. Following measurement, each specimen was weighed to the nearest 0.001 gram.

In regard to the specimen configurations, the flexure specimens are essentially as specified in reference 22, the compression specimens are slightly wider and longer to fit locally manufactured support fixture, and the tension specimens are of different configuration established to minimize failure at the testing machine grips. The evolution of the final configurations in the latter two cases can be seen in Figure 10, on page 19, and Figure 22, in the next section.

In regard to the tension specimens, experience with the configuration recommended in reference 22 indicated that a large number of failures at the grips and at the change in specimen width would occur. Machining the 3-inch radii which faired together the two width dimensions was time consuming in view of the many specimens to be prepared and thus was a rather expensive operation. A less expensive specimen, designed to fail in the test section, would certainly be more desirable.

Other investigators, such as Forest Products Laboratory (FPL), have enlarged the test specimen by doubling the length dimension and increasing the fairing radii to 20 inches with good success (reference 25, page 3). The Kaman Aircraft Corporation Laboratory (reference 14, page 35 and 37) developed a successful specimen in which the test section was formed by sanding a 2.50-inch radius on each side of the laminates. In this case, the failure stress was noted to be dependent on the minimum width, the optimum being less than 0.14 inch.

The approach taken in this investigation is believed to be the most practical solution. A straight-sided specimen was used which had been influenced in the test section just enough to prevent failure in the grips. Two tiny influences 0.007 inch deep were usually cut (see section E1a for deviation and further discussion) on each side of the specimens with a 1/2-inch-diameter high-speed carbide router after the final grinding operation (see Figure 22).

3. Test Procedure

Every effort was made throughout the program to prevent the magnification of spurious effects. Not only was the location of the laminates on the pre-preg, the order of their cure, and the location of the 3 types of test specimens within the laminates accomplished in a random fashion (Figure 18), but also the testing of the specimens of each type was done in a nonordered way, the latter being accomplished by shuffling each group prior to each test period.

a. Compression Tests

In view of the very smooth surface produced by the PVA parting agent, the test specimens were not coated with the MolyKote Z as described on page 17. The remainder of the test procedure was identical to that described there (part 3 of the preliminary investigation section) with the exception that a Baldwin-Wiedemann Compressometer was installed for deformation measure-

ment. (Data for modulus calculation according to reference 22 were obtained from every specimen tested.) Figure 10 shows the test set-up with the installation of the Baldwin-Wiedemann B-3M extensometer that had been converted to a compressometer for this application. The last paragraph of that section likewise completes the description of the present tests. Figure 11 also typifies the compression failures encountered here.

It should be mentioned that the B-3M (a differential-transformer type of instrument) was connected to the x-y recorder of the Instron Testing Machine which plotted directly the load versus deformation curve. The instrument was removed from the specimen at approximately 75 per cent of the failure load to prevent its damage. The slope for modulus calculation was taken from the latter part of the curve--the straight line portion. Figure 19 shows the character of the load-deformation curves obtained.

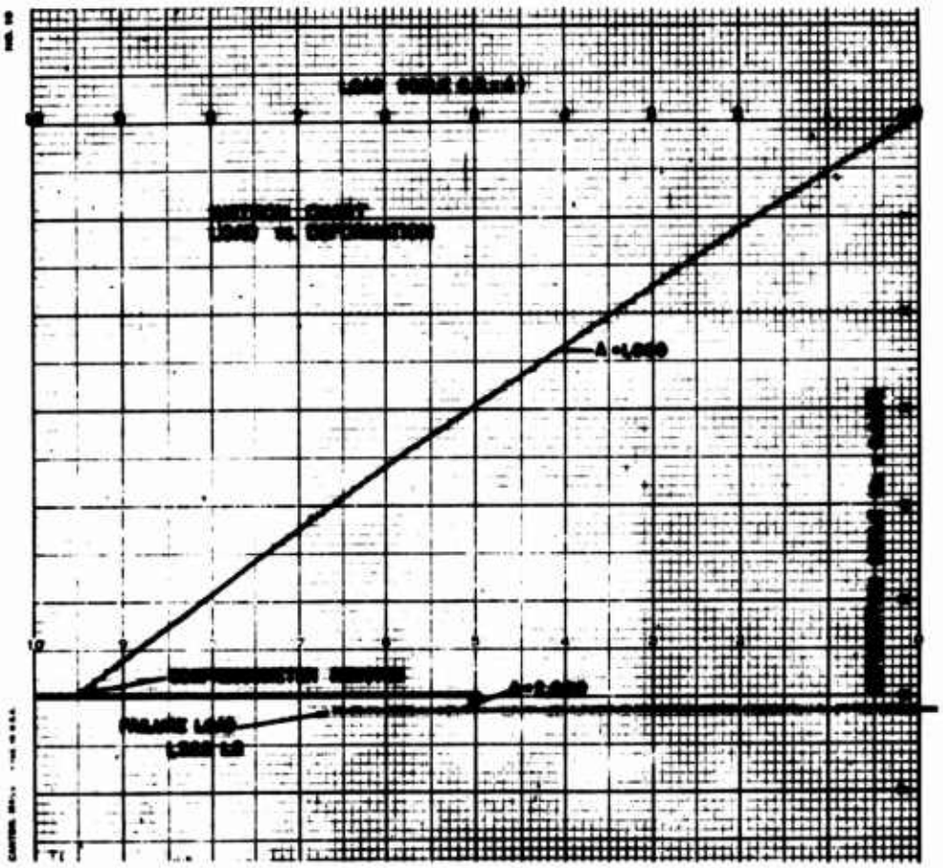


Figure 19. Typical Compression Load-Versus-Deformation Curve for 181 Laminates.

Figure 20 shows the compression test equipment in more detail. The specimen gripped in the fixture can be seen extending beyond the edges on either side. This small extension permitted the knife edges of the deformation measuring instrument to contact the specimens without the interference of the fixture. Notice that, in general, the test system was designed for volume testing without an unnecessary sacrifice of accuracy.

b. Tension Tests

The final step in the preparation of the tensile specimens was the cutting of the influences in the edges. As discussed previously in section D2, these tiny influences in the straight-sided specimens served to minimize failure in the grips.

The test coupons were placed in the self-aligning Templin grips as shown in Figure 21. As data for modulus calculations were desired in each test, the B-3M extensometer was then clamped into position with the knife edges centered vertically on the specimen influences.

The load was applied in the Instron Testing Machine at the rate of 0.050 inch per minute to failure, and the ultimate stress was computed using the maximum load. The failure was abrupt and is typified by D in Figure 22. Surface microcracks perpendicular to the load-line were usually observed to form at about 25 per cent of the ultimate load. The extensometer was removed at 75 per cent of the peak load to prevent its damage.

The load-deformation curves all possessed a definite knee at 25 to 50 per cent of the ultimate load due to extensive microcrack formation; hence, the secondary slopes were read for the modulus calculations. The resin content of each specimen was calculated as described previously.

c. Flexure Tests

The flexural tests followed closely the procedures outlined in reference 22. The two-point support with a 5/8-inch test span was placed on the lower platform of the 10,000-pound-capacity Instron Testing Machine, and the loading nose was bolted to the crosshead. The support was then moved to the position of alignment as determined by a specially designed alignment tool (D in Figure 23).

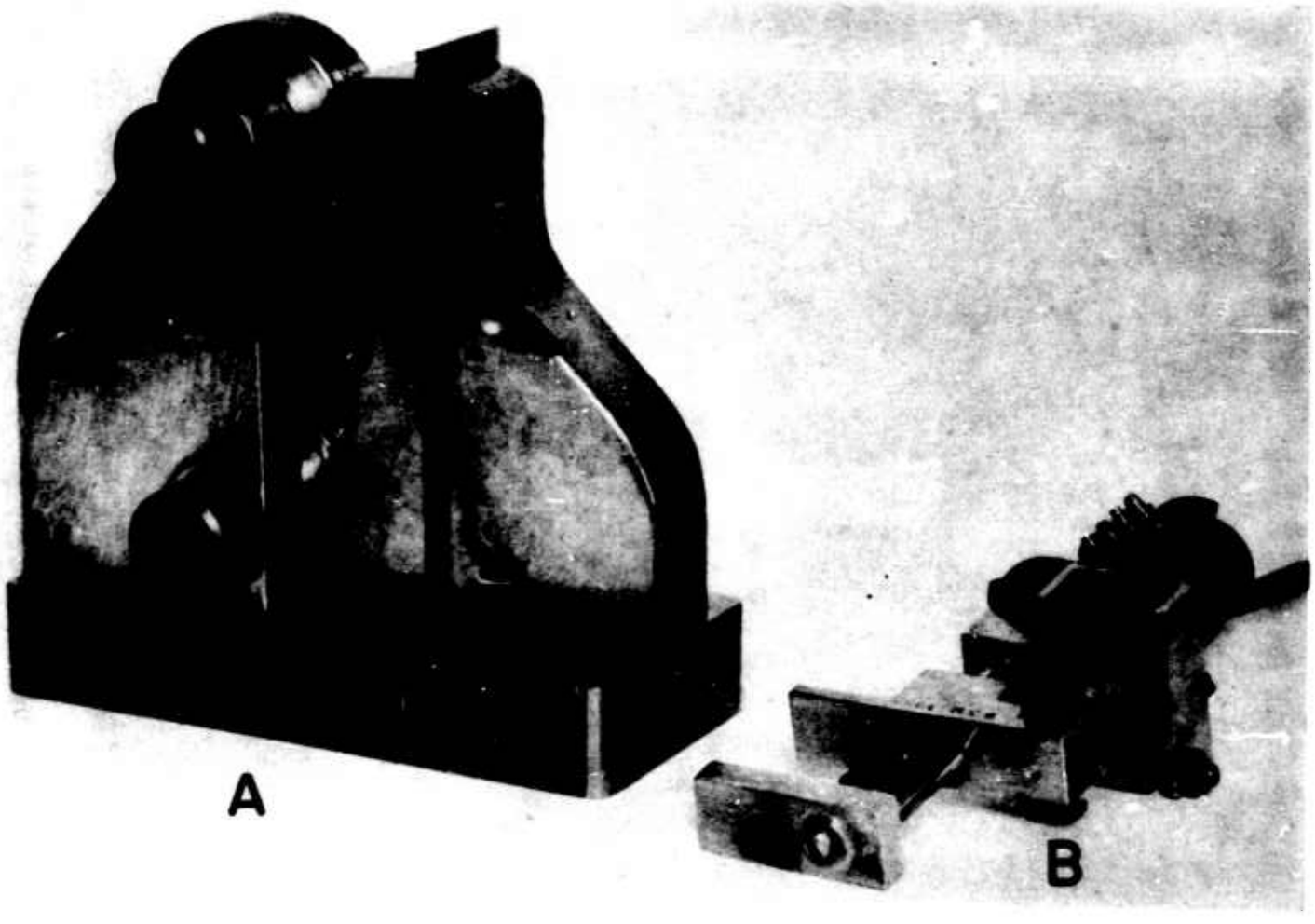


Figure 20. Thin-Laminate Compression Test Apparatus: A, Compression Fixture With Specimen Inserted; B, Baldwin-Wiedemann Compressometer Used To Monitor Deformation.

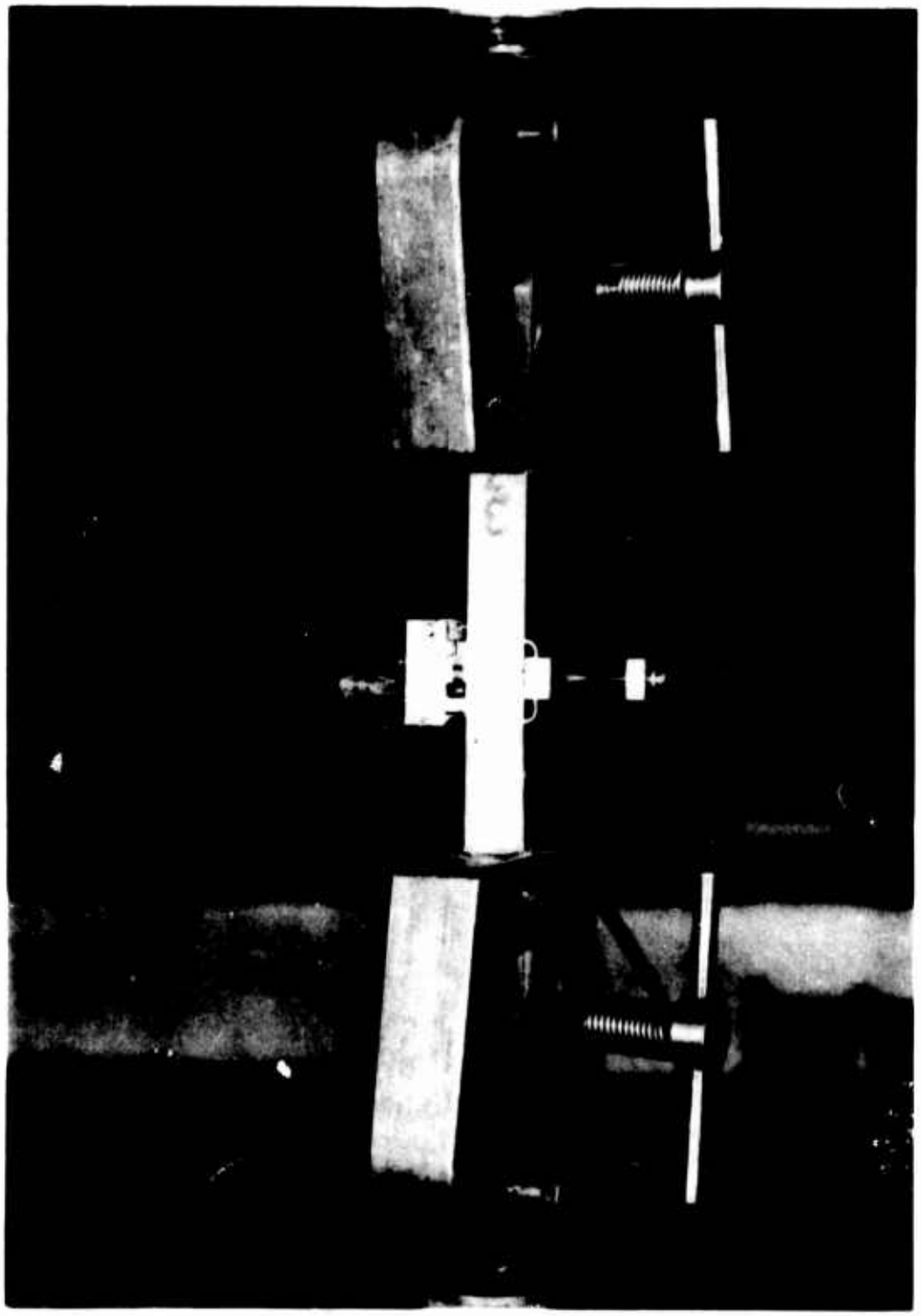


Figure 21. Set-Up for Tension Test of Thin Laminates Showing the Specimen and the Baldwin-Wiedemann Extensometer Installation.

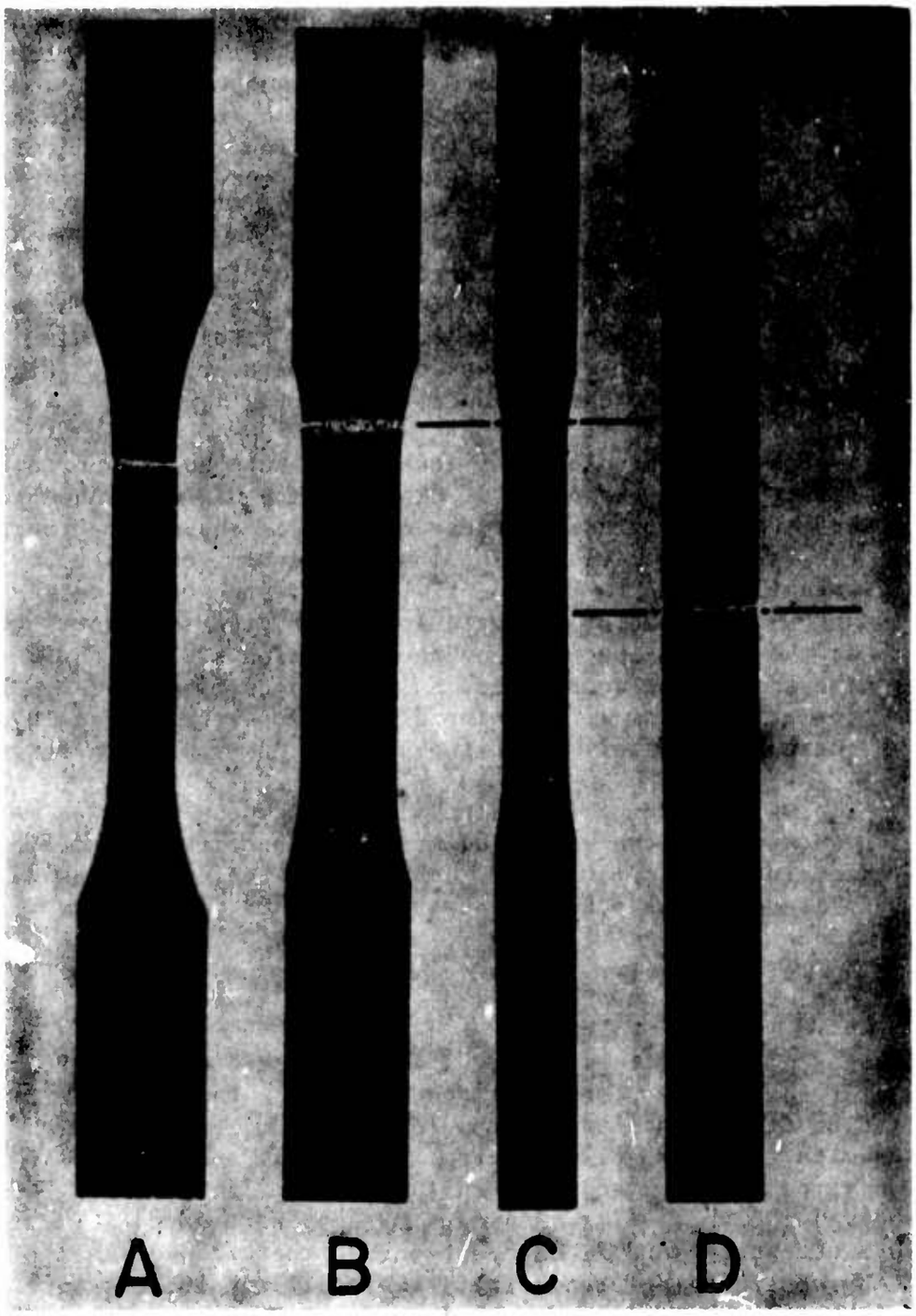


Figure 22. Laminate Tension Failures: A, B, and C, Various Shaped Specimens Evaluated in Previous Research; D, Straight-Sided Specimen with 0.007-Inch Side Influences (See Pointers) Which Is Similar to the Specimen Used in This Program.

After alignment of the fixtures, the Baldwin PD-1 deflectometer was placed in position. The flexural support had been center drilled to receive the vertical extension-shaft of the deflectometer follower to permit contact with the lower side of the specimen at mid-span. The output of the deflectometer microformer was connected to the Instron x-y recorder for automatic plotting of the load-deflection curve. These data were recorded for each specimen.

Next, the specimen was placed on top of the support and the loading nose again brought into close proximity. The alignment tool was then used to center the specimen. Thus, the alignment of the system was checked on every test. Figure 23 illustrates the completed test set-up.

A crosshead speed of 0.05 inch per minute was found to be satisfactory for these tests. The loading was continued to failure, which progressed rather quickly as the outer fibers of the specimens failed. The load-deflection curves recorded were linear up to the point where failure began (between 90 and 95 per cent of ultimate load). Microcracks were observed on the failed specimens on their tension side at the center of the span. The compression surface spots discussed by Outwater (resulting from lifting force on strands--reference 19) occurred immediately under the loading nose only, in these tests.

The usual strength-of-materials simple-beam formulas were used to calculate the values of maximum fiber stress and flexural modulus. Calculation of the specimen resin contents was accomplished as previously described.

E. Experimental Results and Evaluation

The results of the tests are tabulated in the appendix according to the purpose of the test and style of the fiberglass fabric. Table 13 is a compilation of the data from the major resin content factorial accomplished with the 181 3-ply pre-preg. Tables 14 and 15 contain the data obtained from the limited tests on laminates of the 120 and 909 fabrics, respectively. Table 16 is a summary of pertinent data (10-hour B-staged, 90-minute cure, and 2-hour post-cure) generated during a previous USAAVLABS contract (reference 16). Tables 17 and 18 contain the 181 3-ply data for the preliminary void study.

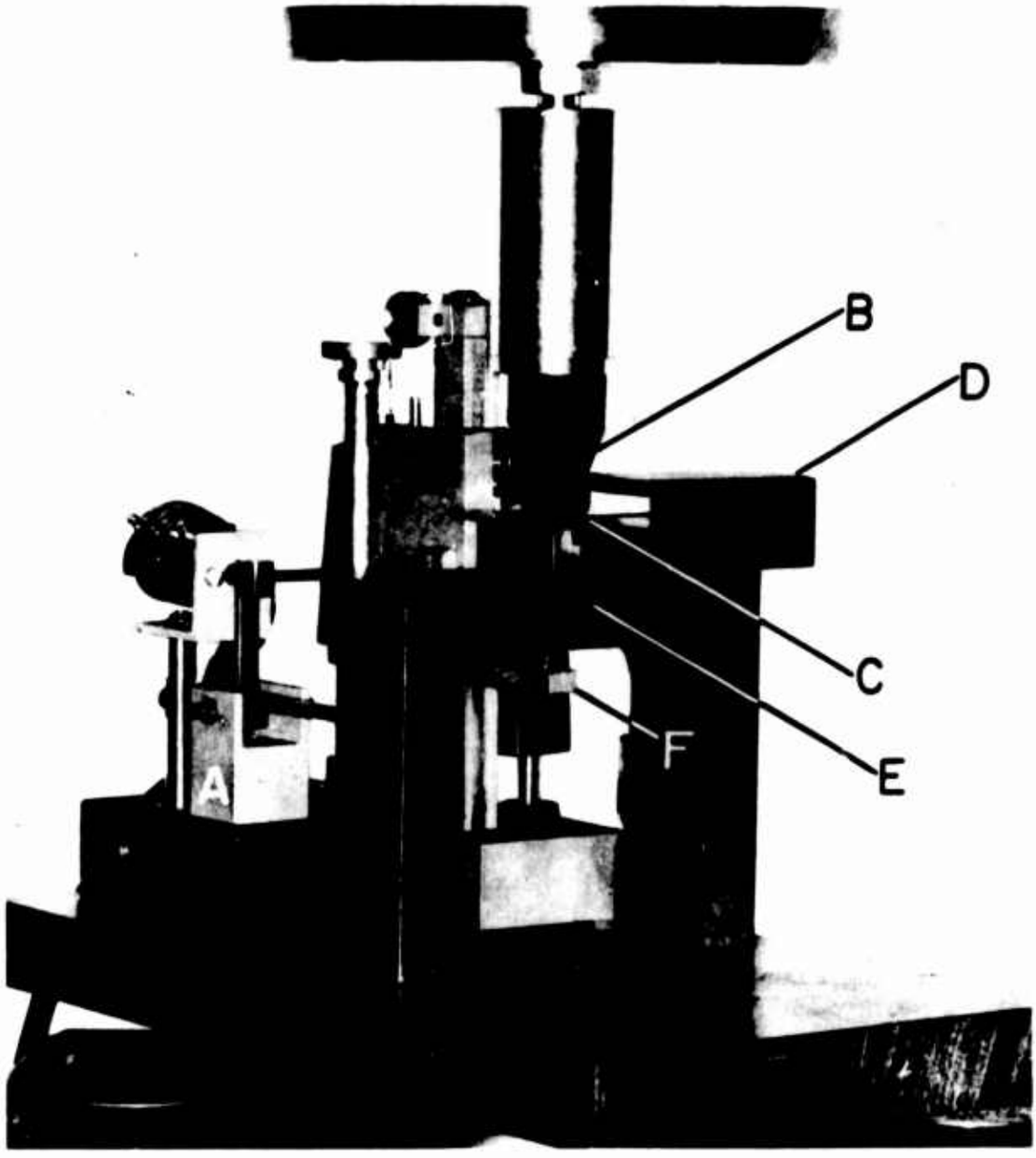


Figure 23. Set-Up for Flexural Test of Thin Laminates: A, Baldwin PD-1 Deflectometer; B, Loading Nose; C, Test Specimen; D, Alignment Tool; E, Specimen Support; F, Deflection Follower With Vertical Extension-Shaft Attached.

1. Resin Content Study

Three aspects of the epoxy-fiberglass composite are considered essential to the discussion of its suitability as a primary load carrying element in an aircraft structure. These are the basic effect of resin content, the control of resin content, and the uniformity of the material. In sections E1a, E1b, and E1c to follow, the materials fabricated are examined from these three points of view. Though void inclusions are closely related to the uniformity of the composite, the subject will be discussed separately in section E2.

a. Resin Content Effect

The initial fabrication research at this laboratory (reference 16) was aimed at detecting a correlation between the fabrication variables and the final strength properties. The trends observed at the time for the press-molded laminates were noted to be very subtle. It is believed that this was due, in part, to the non-uniform control exercised by cure pressure and temperature, as will be descussed later. However, the previous data (repeated in Table 16), when plotted against resin content, exhibit the same strong trends as will be seen to exist in the data from the present program. This indeed lends positive support to the theory that the resin content of an epoxy laminate is the major factor in determining its room temperature strength, that is, the resin content has the largest main effect.

The averages of each of the mechanical properties of the 181 laminates (values from Table 13) were plotted against the average resin content of the respective specimens to establish the strength properties/resin content patterns. Similar plots for the 120 and 909 laminates are presented in section E1c pertaining to uniformity. An overview of the 181 data (Figure 24 through 28), which also includes the data from reference 16, shows that tensile strength and modulus, flexural maximum fiber stress and flexural modulus, and compressive modulus increase as resin content is decreased, whereas compressive strength decreases. These findings are in agreement with those reported in reference 26 for 181 laminates of polyester resin, though the data were not as extensive as those presented herein. The laminates of the 120 fabric exhibit similar trends, as do the laminates of the 909 fabric, with the exception that the 909 compressive strength increases with decreasing resin content.

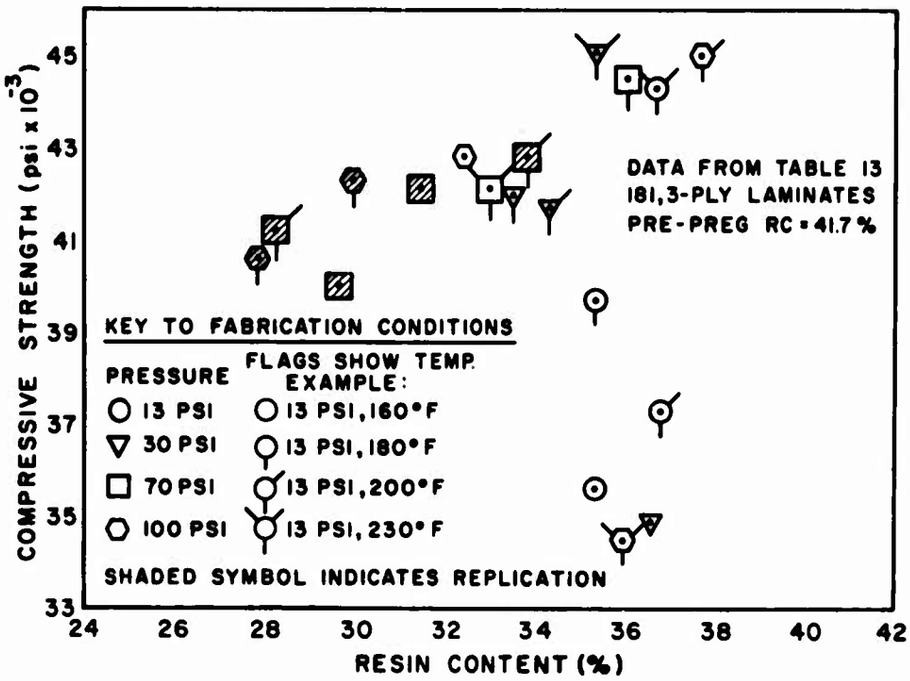
A regression analysis was made for each group of the 181 data, and the curves, along with the equations, are included in each figure. The first- and second-degree curves must be considered tentative in most cases, since the character of the peak values is not thoroughly revealed. With the additional data it would be possible to optimize the curve fit and place confidence limits on the coefficients of the equations; however, it may well be that the curves chosen are just as suitable for design. This is true because the design point for a full-scale structure is expected to be a balance between the tensile and compressive properties which would probably require utilization of the data near the center of the resin content range presented herein (say, near 30 per cent).

Since the data for the 120 and 909 laminates represent only a pilot study, the equations for these curves were not obtained. It must be noted that the 120 and 909 fabrics were included in the program mainly for a comparison of laminate uniformity; hence, only a few comparisons of absolute strength values will be possible. The conclusions that can be made will be brought out in the discussions that follow.

Compressive Strength. Within the spread of resin content examined in the total program (approximately 26 to 42 per cent), the mechanical properties still appear to be increasing; however, it can be expected that a further change in resin content would result in a peak value followed by a decrease in each property (refer to discussion of flexural strength). In fact, it is possible that the peak was approached for the compressive strength of the 181 and the 120 laminates, the values occurring at about 35 and 37 per cent, respectively (Figures 24 and 31). In the former case, this is suggested by the older 181 data from reference 16 (repeated in Table 16) plus that from laminates 1, 2a, 3a, 3b, 4, 5a, 5b, 17a, 19a, 20, 21a, 22, 23, and 24a listed in Tables 1 and 2. Other laminates could have been included from these tables, but those listed most nearly duplicate the earlier fabrication conditions and procedures.

The upper values obtained in the present 181 data are slightly higher, but no values were obtained beyond 38 per cent resin content to suggest the occurrence of the peak. Thus, with the exception of 5 low points, the current data in conjunction with the aforementioned data were used to calculate the empirical equation shown in Figure 24.

The 5 low points in the vicinity of 36 per cent resin content are widely separated and vertically aligned below a tightly



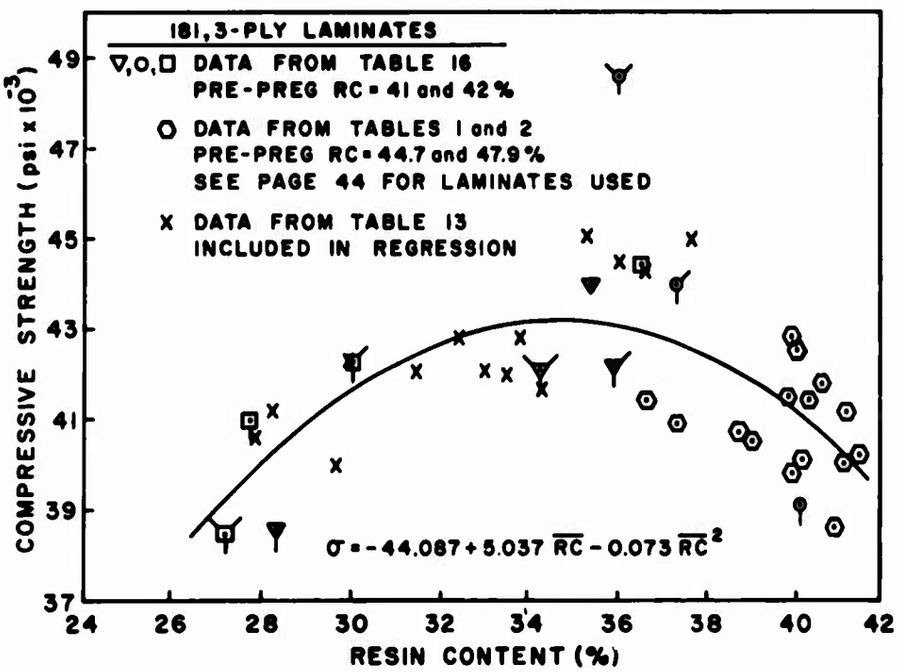


Figure 24. Compressive Strength Versus Resin Content. The regression analysis is based on the total data shown.

packed group at a much higher resin content; therefore, they should not be considered as a part of the total population of data. These extreme values are probably associated with the milling of the specimens; note that the tensile strength recorded for these laminates is not out of line.

It must be emphasized that the data from the other sources may not be completely compatible with the present data. Though the use of the older data confirms that a peak exists, it would not be expected to precisely locate the peak, and thus the second-degree curve shown is only tentative. In fact, it is suspicioned that an extension of the present program would show a peak nearer to that of the 120 laminates.

The 120 laminates have a peak value of 45,000 psi occurring at about 37 per cent resin content. However, the maximum value for the 909 laminates is seen to occur at a much lower resin content, though not specifically located by the present data (Figure 32). The high value of strength in the figure is slightly higher than the peak values of the 181 or the 120 laminates; hence, it is concluded that the maximum compressive strength for the 909 laminates will be higher than for either of the other two materials. This is evident even with the higher void content. Thus, a marked increase in compressive strength is expected with better void control (see section E2a).

That the maximum value for the 909 laminates occurs at lower resin content is not surprising, since the 909 fabric is actually more nearly a crossply than a woven fabric. The weaving involves the small-diameter binder yarns and serves to hold the large structural yarns in place with a minimum of crimping. Thus, the compressive strength/resin content pattern of the 909 laminates could be expected to approach that of filament-wound laminates (15-to 20-per cent peak; see reference 11).

In the case of the woven reinforcements, the occurrence of the maximum compressive strength at such a high resin content in comparison to the other mechanical properties suggests that different failure mechanisms are involved. Little work on the failure mechanics of fabric-reinforced composites could be found in the literature, but effort has been and is being directed to the investigation of failure in the uni-directional glass-fiber reinforced composites. Since the longitudinal strands of fabric are in essence already buckled uni-directional strands (tending toward the uni-directional in the limit), it is in order to discuss some of the more recent papers on uni-directional fiber reinforcement.

The data presented in reference 11 for compression tests of flat laminates fabricated with uni-directional filaments (laminates were wound) also exhibited the tendency to peak out, the peak possibly occurring near 15 per cent resin content. However, the test data were very limited in comparison to those reported herein. The theory advanced in that paper, and also in reference 9, attributed the compressive strength of the composite largely to lateral support of the glass filaments--in other words, to the buckling restraint of the filaments. Increasing the glass content (decreasing the resin content), it was stated, provides increased fiber support by increasing the number of "nearest neighbor" filaments up to the point where there is insufficient resin to support the fibers and carry stresses. Decreasing the resin content beyond this point causes the strength to fall off.

Broutman (reference 4) has shown that the failure mechanism is one of delamination initiated by cracks at the resin-glass interfaces. An electron photomicrograph (25,000X) showing the cross section of a specimen which had been loaded to 80 per cent ultimate stress was presented which revealed such a crack. The crack was at the edge of the highly stressed resin tricorne. Photographs taken with the aid of a light microscope (1200X) showed typical delaminations. The photographs indicated that the high-resin-content specimens there examined would support more cracking prior to failure than the low-resin-content specimens where the filaments acted as crack deflectors; hence, the low-resin-content specimens could be expected to develop the higher strengths.

Now, the much higher optimum resin contents of fabric reinforced laminates suggest that the mechanism of reinforcement for fabrics is yet somewhat different from that of uni-directional filaments. It was stated in reference 19 that, during flexural loading, lifting forces perpendicular to the laminate could cause separation of the plies and the ultimate collapse of the fibers on the compression side. These forces arise from the glass reinforcement lying in three dimensions due to the convolution of woven strands, the forces being maximum at the crossover points. It was stated that the stability of the laminations depends on restraining pressures in the resin and the rest of the laminate. Thus, it seems reasonable to assume that the failure mechanism for uniform compression of fabric-reinforced laminates would be similar in nature.

The appearance of the specimens after test lends further support to such a failure mechanism. Some of the specimens were

seen to have a few of the surface delamination spots at the strand crossover locations which were observed by Outwater (reference 19). Of course, it is possible that the formation of the few spots noted could be influenced by the bearing of the specimen against the test fixture. Nevertheless, the evidence is sufficient to encourage further research on the failure mechanism of fabric-reinforced composites.

Tensile Strength. As stated previously, the data for the ultimate tensile strength for all 3 reinforcements show an inverse relationship relative to resin content, with the optimum value of resin content probably occurring near or below 27 per cent (see flexural strength discussion). The earlier 181 data from Table 16 plotted on the same graph with the present 181 data for comparison (Figure 25) also show the same trend. The values of tensile strength of the 120 laminates are close to those of the 181 laminates, while the values for the 909 laminates are slightly higher. In each case, further research will be required to locate the peak values precisely. In parallel with the findings for the 181 laminates, the actual tensile strength at each resin content is expected to be about 30 per cent higher than shown, due to the effect of the deeper side influences as discussed next.

Since this research program was keyed mainly to making comparisons, the opportunity was taken to obtain data on the effect of stress concentrations in fabric-reinforced laminates, as very little data are given in the literature. The specimen side influences were increased from the empirically-determined optimum value of 0.007 inch (see section D2) to 0.016 inch*. The effect of this change is readily apparent from Figure 25.

The theoretical stress concentration was calculated for both of the edge conditions shown by using the value of tensile modulus obtained from Figure 28 at 30 per cent resin content. Estimates of the other needed properties such as Poisson's ratio and modulus of rupture were obtained from the literature.

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* It was also necessary to follow through with this depth increase in the case of the laminates of the 120 and 909 fabrics in order to obtain the desired uniformity comparisons.

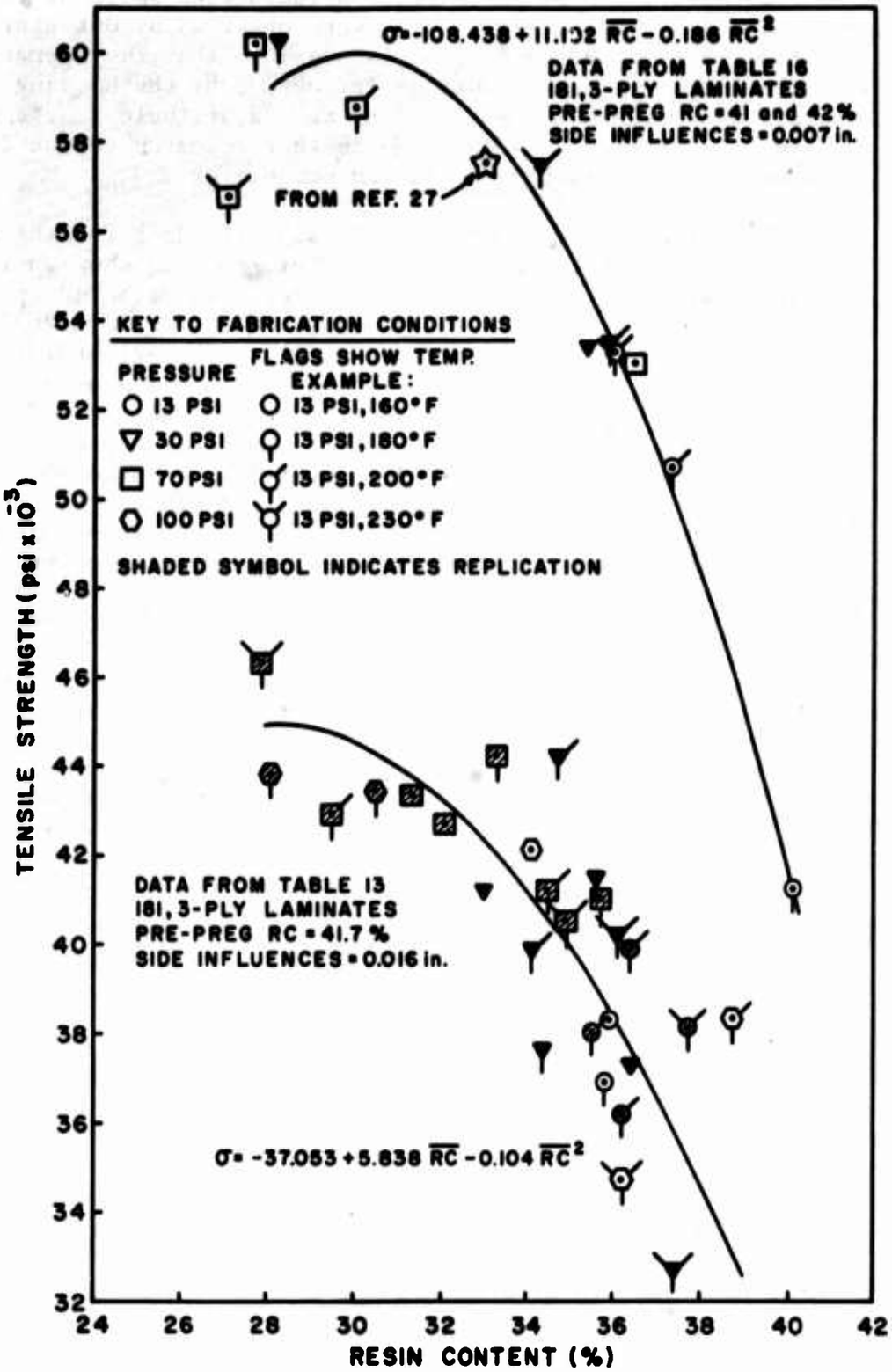


Figure 25. Tensile Strength Versus Resin Content. The lower curve indicates the effect of small stress concentrations.

Though a closed-form solution of the orthotropic elasticity equations for circular notches was not located in the literature, it was possible to approach this condition with a good approximation by using the equations developed by Smith for the hyperbolic notch (reference 23). The equations were applied by requiring the radius of the curvature of the hyperbola at its vertix to equal the radius of the router (1/4-inch, see section D2) used to influence the specimens. Actually, the approximation should be quite good, since the notch depth and the radius of curvature at the most highly loaded point are generally considered to be the most important factors affecting stress concentration (reference 15).

The value of 1.9 was calculated for the stress concentration factor in both cases of influence depth, yet the test data show that the 0.016-inch-deep influence decreased the tensile strength by a factor of 1/1.3 relative to that of the 0.007 influence. This divergence of theory and test has also been noted by other laboratories. For the case of a 1/8-inch hole in the center of the specimen, the difference that has been observed is even greater than that noted herein (3.4 compared to 1.3; see references 2 and 28). Of course, it must be remembered that the theoretical values of stress concentration are based on elastic conditions and are only partically indicative of strength comparisons at failure. Thus, it is concluded that additional research in the realm of theory and test is needed before the effects of simple stress concentrations can be accurately predicted.

At first the 0.007 cut may appear to be more influential than required for the purpose of simply precluding failure at the specimen grips during testing (section D3b), yet a comparison of the data with that obtained by the FPL investigators using the very large, gently curved specimen (reference 27) readily confirms the correctness of the empirically determined 0.007-inch depth. The value listed for the tensile strength of a 12-ply laminate of 33 per cent EPON 828-A resin was 57,500 psi, which is very close to the value obtained in the present program for the <u>thin</u> laminate.

A more thorough understanding of the fracture mechanics of FRP laminates would probably assist in the application of the theoretical values of stress concentration. The cutting of only a few of the longitudinal glass strands seems to have a dramatic effect on the strength of the material, in some cases. Since the failure of composites of uni-directional strands is

fundamental to the study of those that are fabric reinforced, mention will again be made of work performed in this area.

Reference 3 is a presentation of the results of a microscopic study of a single-ply uni-directional fiber laminate throughout the tensile test. It was noted that failure of a filament produced resin shear all along the free ends as a result of elastic recovery, causing adjacent filaments to carry greater loads. This condition was observed to be highly detrimental at the edge of the specimens. These observations were made on a model simulating the conditions in a multi-ply laminate, so that there is some question as to whether the conditions are identical; however, it does again show that more work is needed in the realm of the nature of the failure of glass-fiber reinforced composites.

Flexural Strength. The graph for the flexural data shown in Figure 26 reveals the same pattern of strength relative to resin content as was the case for the tensile test, though the scatter in the data is somewhat greater than for the tensile or the compressive test. This trend suggests that the flexural specimens may have failed in tension. Though the specimens were heard to crack at the point where the load-deflection curve became non-linear at the onset of failure, the mode of failure could not be confirmed by the laboratory observations, as the failure progressed too rapidly.

Again the peak of the curve was not defined by the present data; however, there are data in the literature (reference 5) to suggest a tentative location of 24 per cent resin content. Only 4 data points were listed for the dry processed 181-EPON 1001 laminates; therefore, the peak indicated must be considered tentative. However, this information should be valuable in guiding the future research necessary for accurate establishment of the point for the flexural loading of thin laminates.

The flexural tests were conducted to complete the picture, so to speak, for the mechanical properties of thin epoxy-fiberglass laminates. Many applications of the material will involve bending; hence, the test serves as a direct means of evaluation under bending loads. The scatter in data suggests the sensitivity of the test to small changes in material parameters and that the test may be a simple, inexpensive method of evaluation in certain investigations.

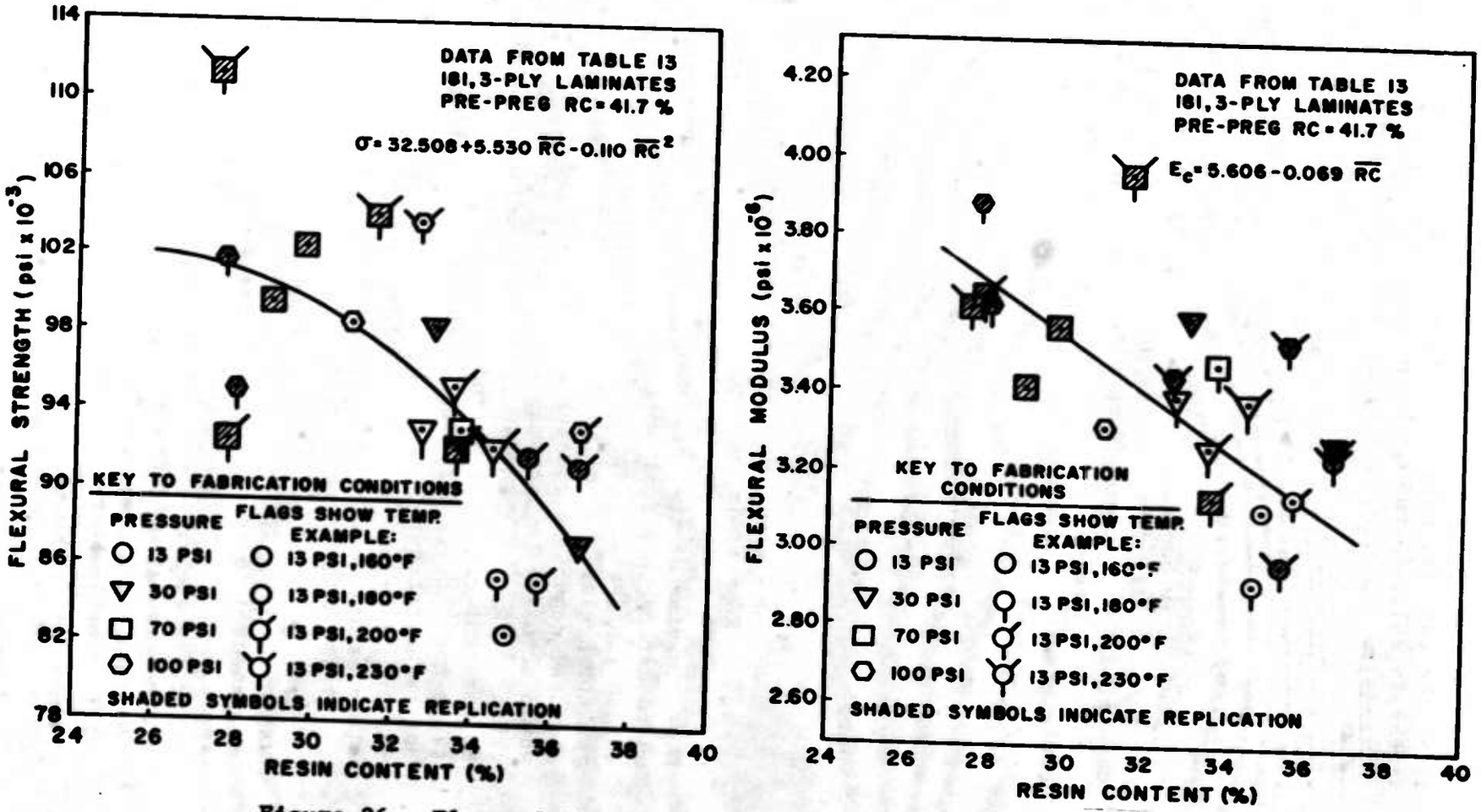


Figure 26. Flexural Strength and Modulus Versus Resin Content. The data are from Table 13.

As is the case for many aircraft materials, the maximum fiber stress (modulus of rupture) for epoxy laminates is higher than the tensile strength, the ratio being approximately 1.8.

Moduli. The modulus data for the 181 laminates are plotted in Figures 27 and 28, and those for 120 and 909 laminates, in Figures 31 and 32, respectively. All three of the moduli (compressive, tensile, and flexural) are seen to increase with decreasing resin content over the range explored. In the test range of resin content, this agrees in trend with the theoretical relationship developed by Outwater (reference 18) for the case of uni-directional fibers in tension in which the fibers were assumed to carry all the load. For the approximate specific gravities of fiberglass and epoxy resin (2.6 and 1.17, respectively), the equation becomes:

$$E_c = \frac{E_g}{\frac{2.22}{1-RC} - 1.22}$$

where

E_c = modulus of the composite
E_g = modulus of the glass
RC = resin content

Thus, in view of its derivation, this equation could be expected to define the ideal or limiting condition for fabric-reinforced laminates. For example, using a modulus of 10.5×10^6 psi for E-glass, a resin content of 28 per cent would indicate an ideal composite modulus of 5.6×10^6 psi (note: the resin would actually be carrying about 5 per cent of the load). In other words, the thin fabric-reinforced laminate in only about 80 per cent efficient in this regard. Because of the assumptions made in its derivation, the theoretical equation could not be expected to apply at very high or low values of resin content; however, it is noted that its slope and the actual slope established by the test data are nearly equal in the 27 to 37 per cent range of resin content.

As in the case of the strength properties, modulus could also be expected to reach a maximum value and not necessarily at the same resin content. An optimum resin content may also exist for each type of loading; however, the precise locations are not revealed by the present data. Since very low resin contents did not appear in the present program, special effort will probably be required to produce the low values needed to locate the peaks.

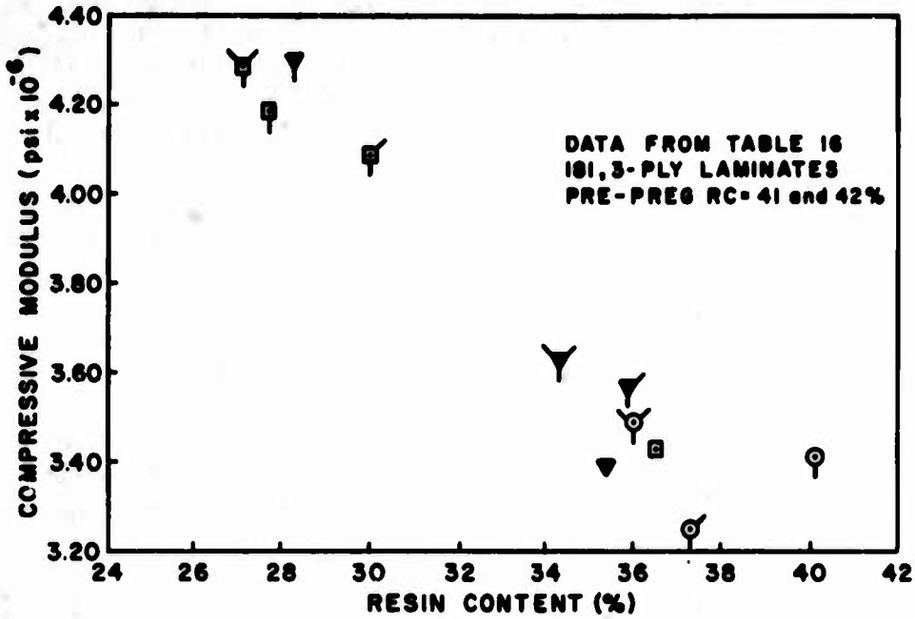
For a given resin content, the values of moduli for the 120 laminates are slightly lower than the corresponding values for the 181 laminates. This is likewise the case for the 909 laminates, though such a comparison is not justified, as the maximum for this material will probably occur at a much lower resin content than that of the 120 and 181 laminates. Thus, more extensive testing will be required before a thorough comparison of the moduli can be made.

The values of compressive modulus for the 181 laminates are seen to form approximately the same trend as the older data; therefore, the total data picture was used to obtain the empirical equation with the exception of the two widely dispersed points at the lower resin content (Figure 27). A slight difference in the two sets of data does appear when the values listed in reference 16 are plotted directly, due to the use of a more accurate gage length in the present program (1.15 rather than 1.03 inches). The small correction was made when the data were transcribed to Table 16.

The tensile moduli recorded (secondary values) consistently appear lower than those for compression or flexure. This difference can also be seen in the FPL data when the average or secondary tensile modulus is considered (reference 27). No doubt, surface or interlaminar crazing accounts for the loss in the tensile modulus after the proportional limit has been exceeded. In the present research, the surface microcracks could be observed to form at about 25 per cent of the ultimate strength; hence, the secondary value of modulus is obviously the most valuable for design purposes. It is concluded that resins of higher yield strengths will be required if the full potential of the glass modulus is to be approached.

Except for 6 extreme values on the periphery of the data pattern, the totality of the 181 data presented was used to obtain the empirical equation for the tensile modulus (Figure 28).

The values obtained for flexural modulus of the 181 laminates are slightly lower in magnitude than the compressive moduli, yet higher than those for tension. Thus, the flexural modulus seems to be a balance between the compressive and tensile moduli. The change in modulus is probably linked to early microcrack formation. Though the test apparatus would not permit observation of the tension (lower) side of the specimens for microcrack formation during the test, microcracks were later found to be present in the center of the failed specimens. The graph of the flexural modulus is included with the flexural strength in Figure 26.



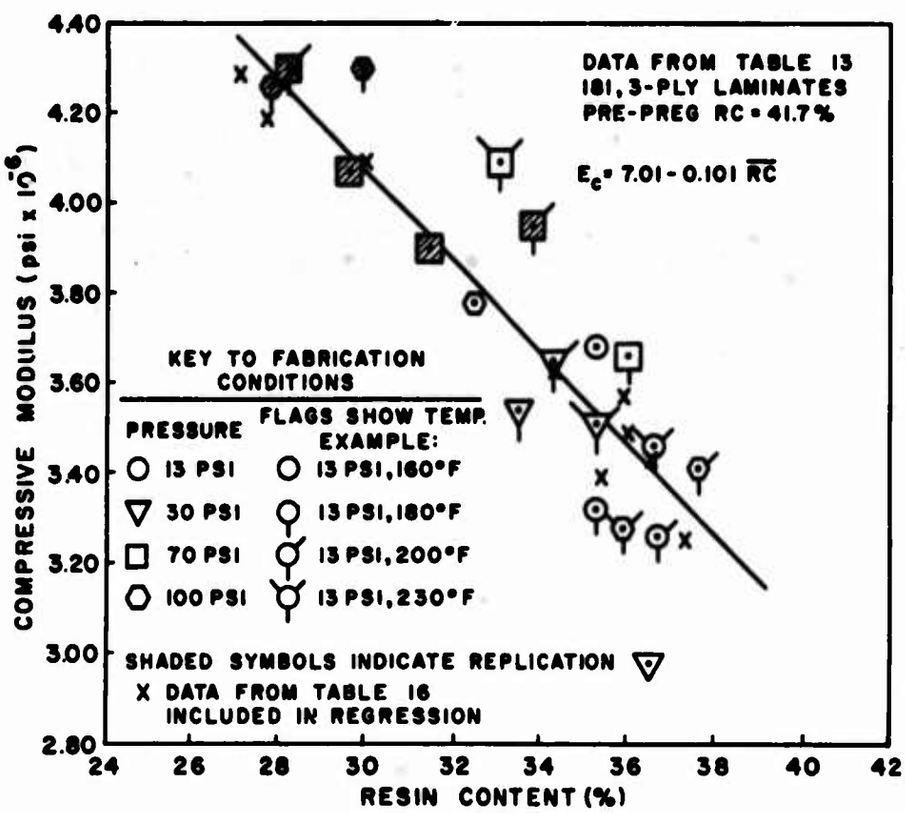


Figure 27. Compressive Modulus Versus Resin Content. The regression analysis is based on the total data shown.

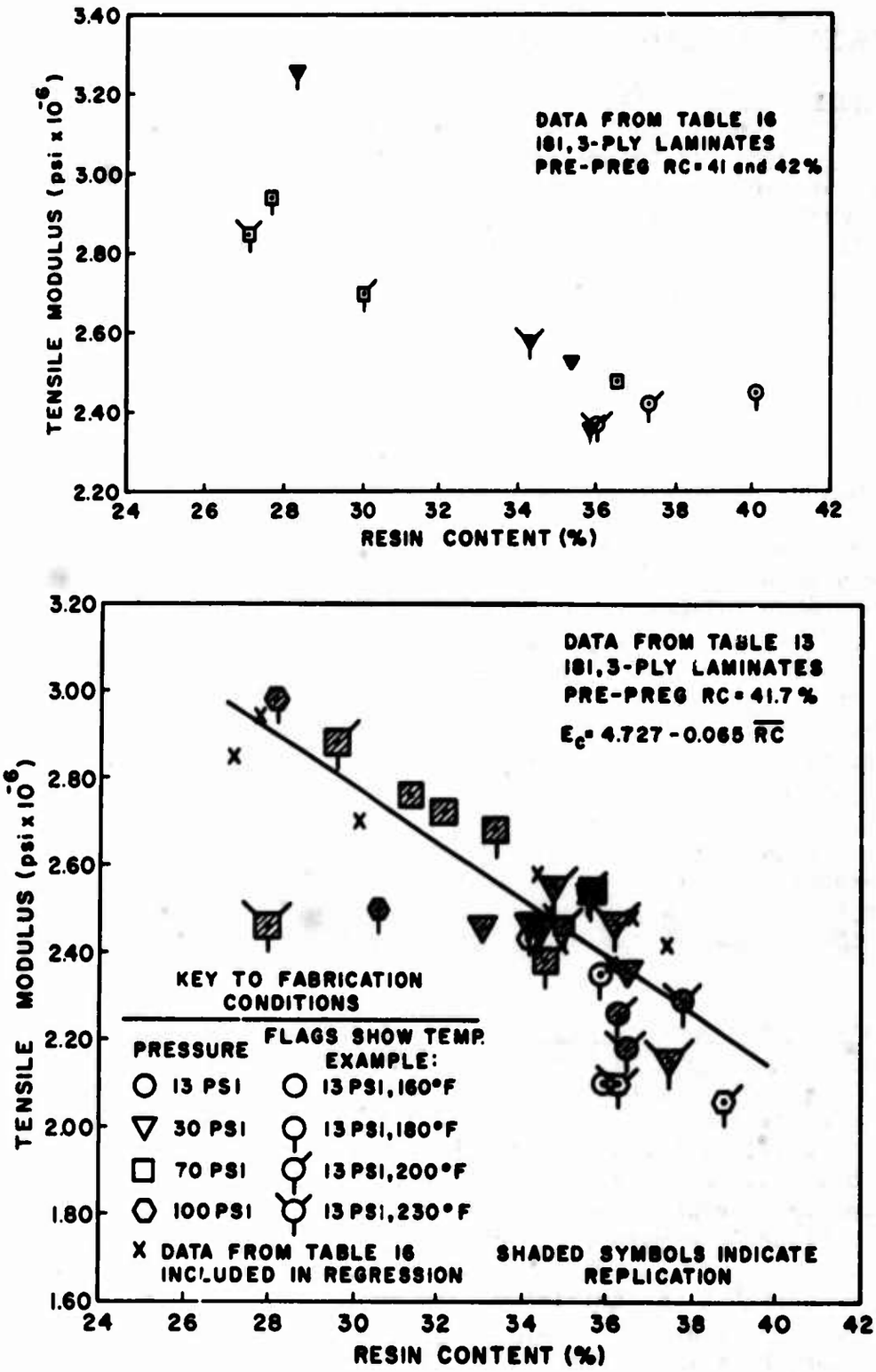


Figure 28. Tensile Modulus Versus Resin Content. The regression analysis is based on the total data shown.

b. Resin Content Control

Pressure Effect. With the knowledge of the strength properties/resin content pattern in hand, it is logical to inquire of the procedures to achieve a given resin content. The first means of control that will be examined is that offered by the cure cycle. Figure 29 is a 3-dimensional plot of the averages of all 181 laminate resin contents obtained at a given cure condition, against the respective cure pressures and temperatures. It is seen that the cure pressure is effective in uniformly decreasing resin content except at the higher temperatures. Increasing the temperature at a given pressure has little influence on the resin content until the 100-psi pressure level is reached, where it increases slightly. In this case, undoubtedly the gellation is occurring rapidly and restricting the flow.

The previous OURI data listed in Table 16 also exhibit this cure pressure-resin content trend, although the data show a much greater dispersion. It should be mentioned that with the aid of the findings from the preliminary investigations, it was possible to place tighter controls on the fabrication in the present research. Forest Products Laboratory likewise observed that increasing the cure pressure would decrease resin content. However, this was for polyester laminates (reference 29), and much less data were obtained in that experiment.

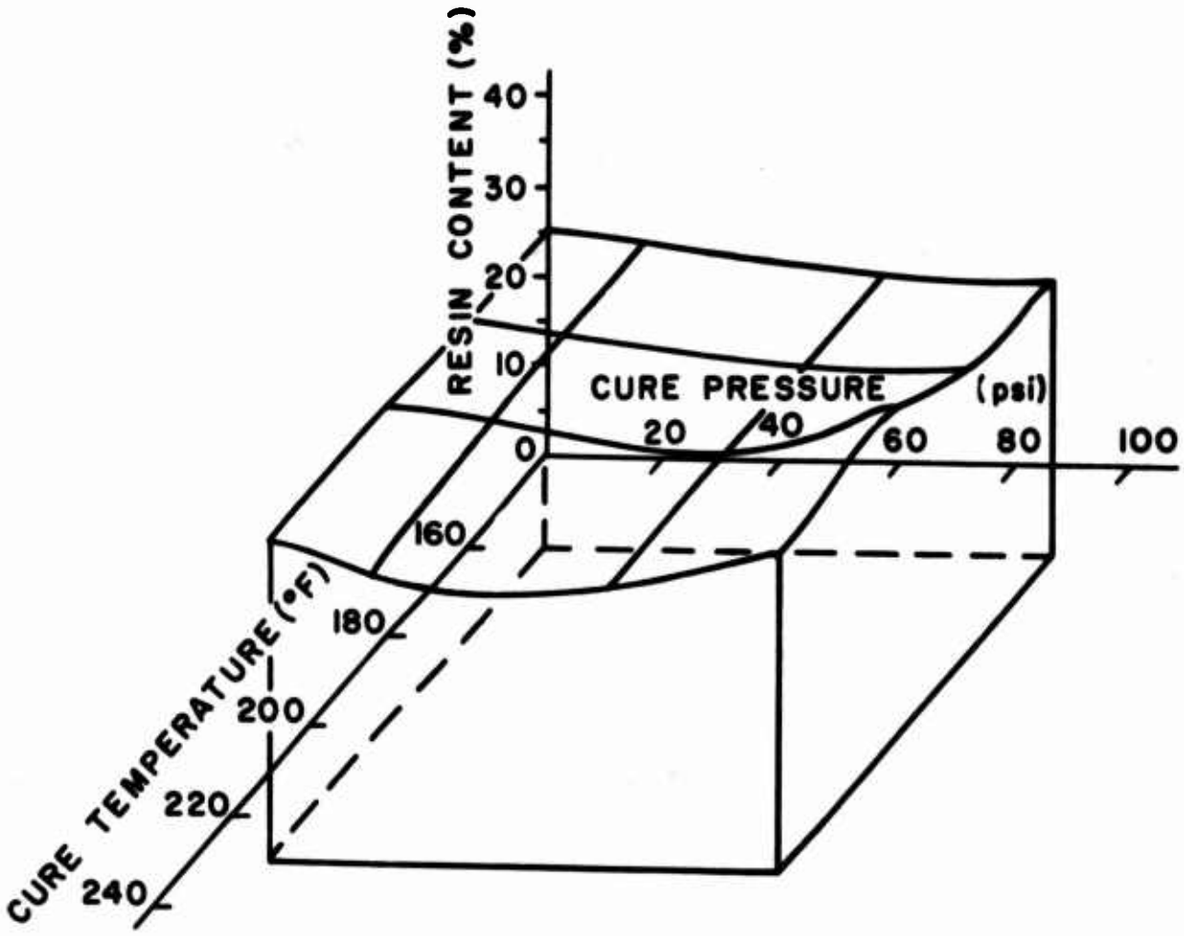
Resin content-pressure cross sections of the resin content-pressure-temperature surfaces are included in Figure 30 to show the effect of pressure as a control parameter for the 120 and the 909 fabrics as compared to the 181 fabric reinforcement.

Initial Resin Content Effect. The program was designed so that it was also possible to obtain a preliminary indication of the effect of initial resin content (the resin content of the pre-preg) on the resultant or final value. At a common cure condition of 13-psi pressure and 160-degrees-Fahrenheit temperature, the final resin contents resulting from the different 181 pre-preg productions used in the preliminary investigations and the main resin content program may be compared.

It is seen that the initial values of 44.7 and 47.9 per cent in the preliminary studies and the initial value of 41.7 per cent for the main program (Table 13) resulted in the following average final contents: 39.6 per cent, considering laminates 1, 2a, 2b, 3a, 3b, 4, 5a, 17a, 19a, and 20; 40.2 per cent,

AVERAGE RESIN CONTENT

CURE PRES. (psi)	CURE TEMP. (°F) 160	180	200	230
13	35.4	35.3	36.0	36.6
30	34.8	34.1	34.2	35.8
70	30.5	34.7	31.2	31.0
100	32.5	28.8	37.7	35.2



181, 3-PLY LAMINATES
PRE-PREG RC = 41.7 %
PRE-CURE = 50% GEL TIME
CURE TIME = 90 MIN.

Figure 29. The Effect of Cure Pressure and Temperature in Controlling Laminate Resin Content. The data employed are average values for each fabrication condition listed in Table 16.

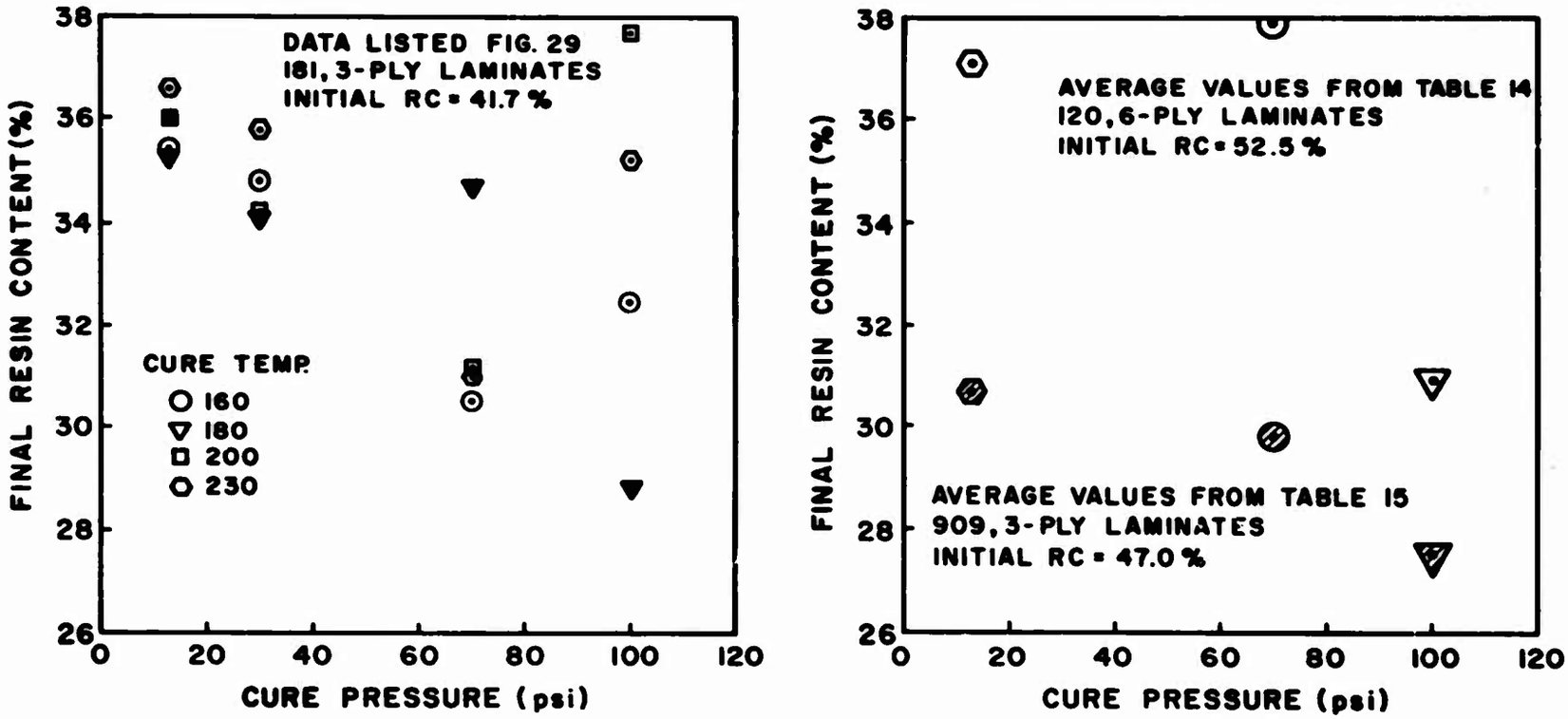


Figure 30. Cure Pressure as a Control of the Final Resin Content.

considering laminates 21a, 22, 23, and 24a; and 35.4 per cent, considering the main program. Thus, there is evidence of the initial resin content directly influencing the final value in the case of the simple flat panels; that is, decreasing the initial resin content would be expected to decrease the final value. The conclusion should not be altered when it is noted that there were slight differences in cure conditions of the laminates compared, such as laminate size and pre-cure time.

The values of flow for the 13-psi, 160-degree cure condition are 5.1, 7.7, and 6.3 per cent, respectively, which are surprisingly close together. A flow comparison may also be obtained for the three fabric reinforcements. Considering the cure condition of 13 psi and 230 degrees Fahrenheit, the flows for the 181, 120, and 909 laminates are 5.1, 15.4, and 16.3 per cent, respectively.

These data show that the flow tends to increase as the initial resin content increases. The type of fabric apparently does not affect the process greatly, as two types of fabric are represented and both have high flows. This comparison suggests that there is a limit to the control of resin content that may be effected in the free edge-flow condition by the initial resin content. However, it is anticipated that the control power of initial resin content will increase with the increased flow restriction that will probably be brought about in large closed structures.

Pre-Cure Effect. Another control of the resin content that was observed in the preliminary investigation is the pre-cure time. Figure 17 shows the uniformity that can be expected in resin content, especially at the low cure pressure and temperature. The effect on the strength properties of the resin content established in this manner follows that where the cure pressure and temperature were the controlling factors, though, there is considerable scatter in the compressive strength values, especially at the higher temperature.

In Conclusion, it is evident that strong interactions exist between the various resin content control factors. It is probable that several test panels would have to be made in order to achieve the optimum for a desired strength property and simultaneously achieve a desired uniformity and void condition. (The latter two factors are discussed in the following sections.) Since the other variables so greatly affect uniformity, possibly the pre-preg resin content will prove to be the most desirable control of laminate resin content, especially when the flow is restricted by boundary conditions.

c. Uniformity

Fiberglass Fabrics. The investigation of laminate uniformity was begun with the reinforcements, the fiberglass fabrics. As described in section B1d, 12 samples were die cut from each fabric and accurately weighed. The weights per unit area for the 181, 120 and 909 fabrics were found to be 0.1904, 0.0667, and 0.2008 grams per square inch with standard deviations of 0.0012, 0.0004, and 0.0011 grams per square inch, respectively. Thus, the 120 style fabric is seen to be the most uniform, with the 181 and 909 fabrics tieing for second place. It was noted that the test values are from 2 to 6 per cent lower than the values commonly published by the weavers.

Laminates. To give insight into the uniformity of the cured laminates at the various conditions of fabrication, partial replication was accomplished for the resin content factorial involving the 181 laminates and for the pilot studies of the 120 and 909 laminates. The replicates and each fabrication condition are identified in the graphs of the strength properties with the aid of symbols. The graphs for the 181 laminates were presented in the discussion of the main resin content study (Figures 20 through 28 in section E1b). The graphs for the 120 and 909 laminates are grouped in Figures 31 and 32 on the following pages. An indication of the within-laminate uniformity of the resin contents can be obtained from the standard deviations presented in Tables 7, 8, and 9, on page 64. These three materials were fabricated at nearly the same thickness for this analysis. For example, the thicknesses of the 181, 120, and 909 laminates at 70 psi and 160 degrees are 0.027, 0.023, and 0.030 inch, respectively.

On the basis of the 181 data obtained, the cure condition of 70 psi and 160 degrees stands out as the most uniform in regard to both strength properties and the resin content. Though complete replication was not accomplished for the conditions at 13 psi/200 degrees and 13 psi/230 degrees, these also exhibit good uniformity. Except for the tensile modulus, the uniformity at 100 psi and 180 degrees also appears good.

In most cases, the values of strength and resin content at the cure conditions marked with an underline in the table of standard deviations (Table 7) are not as consistent as, or are very poor in uniformity in comparison to, the conditions previously mentioned. The conditions 70 psi/200 degrees and 70 psi/230 degrees are the poorest of all for the 181 laminates. In the latter case, the resin content standard deviations did not in-

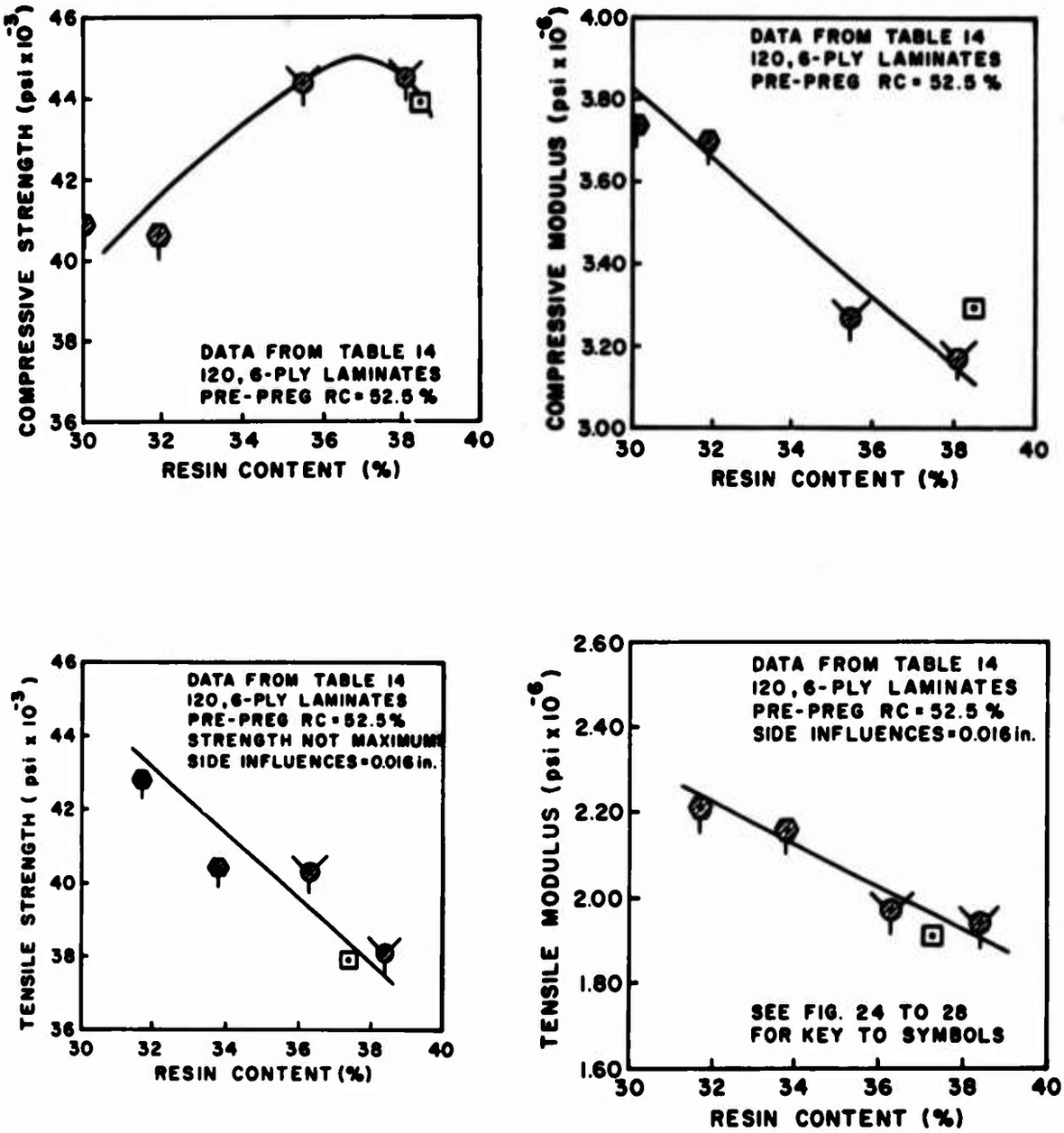


Figure 31. The Effect of Resin Content on Strength Properties of Laminates Reinforced With 120 Fabric, Volan A Finished.

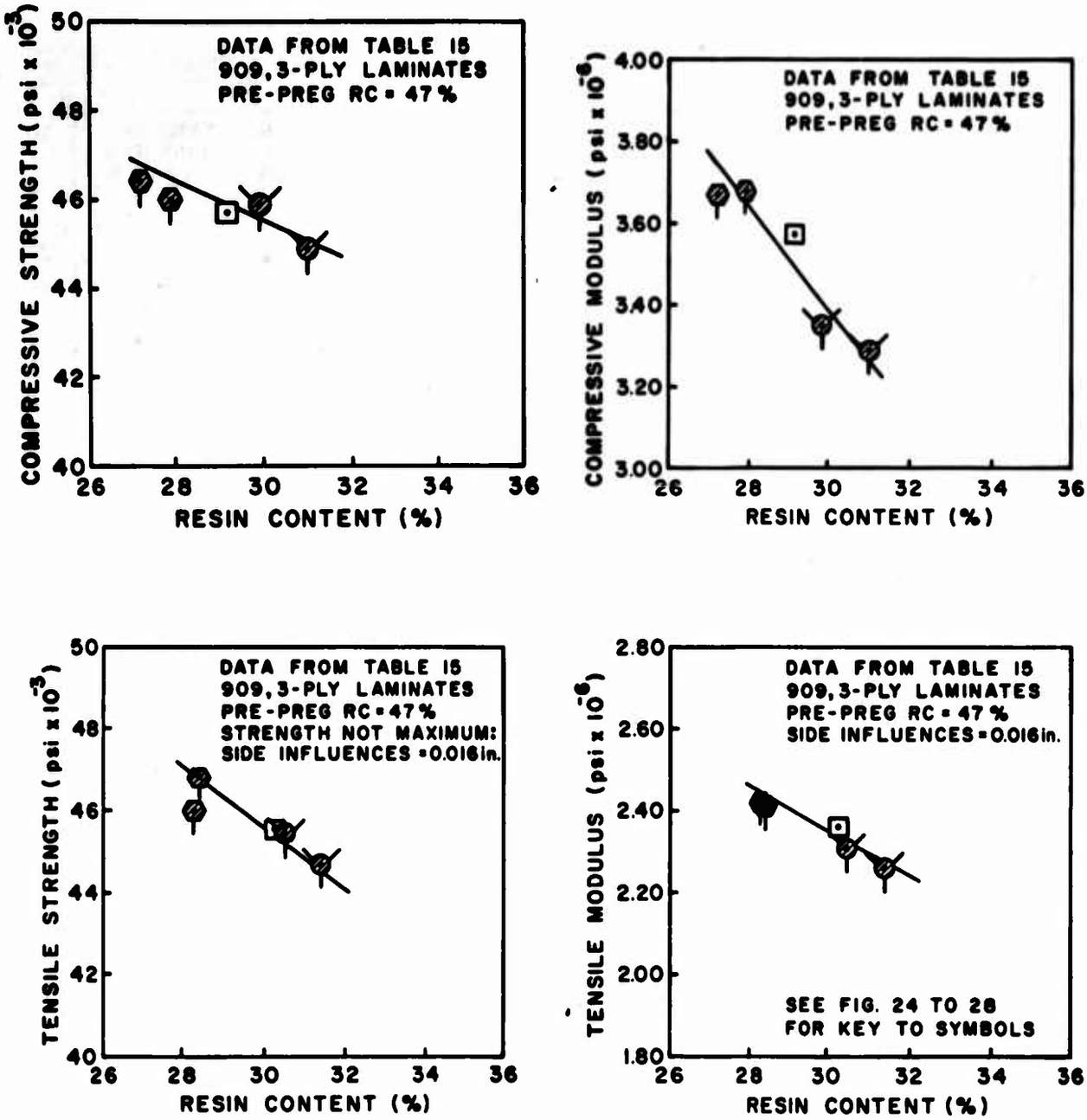


Figure 32. The Effect of Resin Content on Strength Properties of Laminates Reinforced With 909 Fabric, S-920 Finished.

dicate the poor uniformity; this is the only case where the criterion was noted to fail.

The 120 laminate data show the replication of this 6-ply to be about the same at 13 psi/230 degrees as at 100 psi/180 degrees. This is also borne out by the resin content standard deviations (Table 8). In contrast, the 909 laminate data reveal a superior fabrication condition regarding replication. The 13 psi/230 degrees condition appears very good, but that at 100 psi/180 degrees is better in all the cases cited. However, the best within-laminate uniformity was obtained at the former condition (Table 9).

A comparison of the uniformity of all three materials at the three fabrication conditions investigated reveals the superiority of the 3-ply 909 laminate in regard to both replication and within-laminate resin content uniformity. It would be difficult to rank the 3-ply 181 and the 6-ply 120 laminates other than to say that both are not as uniform as the 909 by these two criteria.

Also, the standard deviations for the tensile and compressive strengths were compared at the 70 psi/160 degree cure condition for all three materials. The values calculated were 2.81 and 2.21, 2.72 and 2.33, and 3.74 and 3.78 for the 181, 120, and 909 laminates, respectively. In view of the fiberglass fabric uniformity and the resin content uniformity, these strength uniformity measures take on added significance. In the case of the 909 laminates, the strength standard deviations are not the lowest but the highest. This drop in uniformity of the finished product is probably due to the high void content (see next section). It was anticipated that the 120 laminates would appear superior at this point, as has been indicated by other laboratories (reference 14); yet there is little distinction between the strength uniformities of the 120 or 181 laminates.

It was noted in reference 14 (page 26) that the uniformity in laminate strength was a function of number of plies and not the thickness of the fabric. The findings in the present research do not confirm this, as is stated above. It is believed that the number of specimens was smaller than for the data presented herein. Even with the number of specimens used here, occasionally a distribution would not be thoroughly normal; therefore, comparisons made on the basis of less than 10 specimens should be made with caution.

It is interesting to note that the slope of the tensile strength versus resin content curve is steeper for the 120

TABLE 7
STANDARD DEVIATIONS,
181-LAMINATE RESIN CONTENT

Cure Pressure	Cure Temperature (°F) 160	180	200	230
13 psi	1.18	2.36	1.67	1.56
	-	-	2.30	2.05
30 psi	1.28	0.98**	1.65	0.55
	5.34*	1.74	1.44	1.75
70 psi	2.40	3.62	3.67	2.59
	2.44	2.10	2.06	2.27
100 psi	4.68	2.94	2.33	0.88
	-	2.42	-	2.69

* Standard deviations are in per cent. Replicate laminates are in the lower position as listed in Table 13.

** Fabrication conditions giving poorest replication are underlined.

TABLE 8
STANDARD DEVIATIONS,
120-LAMINATE RESIN CONTENT

Cure Pressure	Cure Temperature (°F) 160	180	230
13 psi	-	-	1.78
	-	-	2.38*
70 psi	2.72	-	-
	-	-	-
100 psi	-	2.35	-
	-	2.34	-

* Standard deviations are in per cent. Replicate laminates are in the lower position as listed in Table 14.

TABLE 9
STANDARD DEVIATIONS,
909-LAMINATE RESIN CONTENT

Cure Pressure	Cure Temperature (°F) 160	180	230
13 psi	-	-	1.14
	-	-	1.39*
70 psi	1.40	-	-
	-	-	-
100 psi	-	1.55	-
	-	1.98	-

* Standard deviations are in per cent. Replicate laminates are in the lower position as listed in Table 15.

laminate than for the 181, at the higher resin contents. However, no trend in uniformity with resin content could be seen within the scope of the pilot study. This can be seen by comparing the ranges of the data given in the tables.

2. Preliminary Void Study*

The study of voids in facing laminates was initiated in view of the application to large sandwich structures in which the probability of their occurrence is increased. The results of testing laminates containing large artificial voids between the plies of the fiberglass reinforcement are discussed in section a along with the effect of much smaller air voids of higher concentration or density. The definition of void concentration and the effect of size are also considered in regard to the existence of critical limits. Section b is devoted to possible methods of detection.

a. Effect of Voids

Large Artificial Voids. In the fabrication of large structures, the introduction of macroscopic voids is a constant menace; therefore, a preliminary investigation was made to observe the effect of large voids on strength properties. As described under fabrication (section D1), the large voids were introduced interlaminarly in the form of 1-mil-thick polyethylene discs. Three sizes of discs were used which had diameters of 1/2, 1/4, and 1/8 inch, and these were located in the specimens as summarized in Figure 33.

In the case of the compression tests, the voids were arranged to encourage laminate shear should this mode of failure be critical; however, as the photograph of the failed specimens shows, the rupture was always compression in nature and followed the section with the greatest width of void. Similar placement patterns were used for the flexure specimens; but they continued to fail at the mid-span, the location of the maximum bending moment, regardless of the void arrangement.

The best correlation of the data from these limited tests was found by plotting strength against void concentration defined

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* The effort to minimize air voids in the pre-preg used throughout the program through modification of the laboratory coating machine is presented and discussed in section B1.

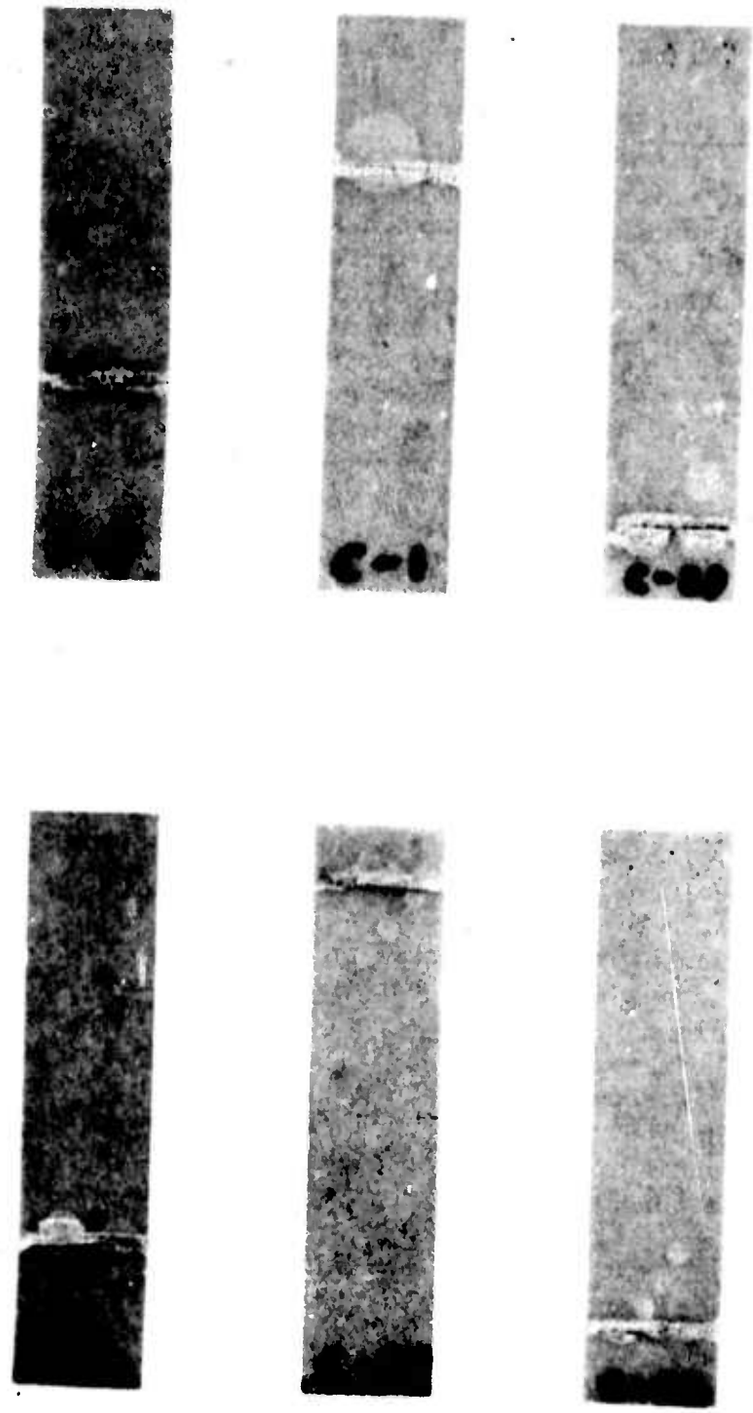

Figure 33. Typical Compression Failures of Specimens Containing Artificial Interlaminar Voids: A, Void Free; B, 1/2-Inch-Diameter Void; C and D, 1/4-Inch-Diameter Voids; and E and F, 1/8-Inch-Diameter Voids. Other arrangements of the 1-mil-thick polyethylene voids were tested, but these typify the failures encountered.

as void diameter divided by specimen width or loaded width. The results of the compressive tests are shown in Figure 34. There appears to be no measurable degradation of strength until the diameter of this type of void exceeds 1/4 inch. The decrease in strength is then seen to be more pronounced when a given concentration is produced by a number of small voids rather than one large void. This conclusion is based on the data for voids whose diameters add to 1/2 inch or 57.2 per cent concentration. In regard to compressive modulus, it can be seen from Table 17 that the voids have not greatly influenced this property.

The flexural data were plotted in a like manner as shown by Figure 35. The findings in this case were not as conclusive, however, because of the larger scatter. Nevertheless, the 1/2-inch void specimens are definitely low in strength, as was the case for the compression tests.

The excessive scatter is attributed to the voids that were not precisely at the specimen mid-span (see rupture data column in Table 18). The voids floated off location during laminate cure, making it difficult to obtain the desired specimens. Hence, on the basis of the present data, no assertion can be made regarding the effect on flexure strength of the number of voids at a given ratio of diameter to loaded length, as was noted for compression. Neither is the critical point for the ratio as distinct. Similar to compression, no trend in modulus appears to exist either.

The decrease in strength noted in these tests is probably due to the discs of parting film having prevented the resin from supporting the fiberglass yarns against buckling. It is believed that natural voids (air voids) will have the same effect; for this reason, the research should be extended to include tests of laminates possessing large air voids that may be encountered in the fabrication of full-size structures. This work should also assist in establishing a more complete and inclusive definition of voids as regards the effect of their number as well as the ratio of the accumulative void diameter to loaded length.

Microvoids. Most of the laminates fabricated for the present research program were noted to contain microvoids. On the basis of calculated specific gravity of the specimens, the void contents of the 181, 120, and 909 laminates were calculated according to reference 21 and recorded in Tables 10, 11, and 12, respectively. For the three fabrication conditions examined in the pilot studies of the 120 and 909 laminates.

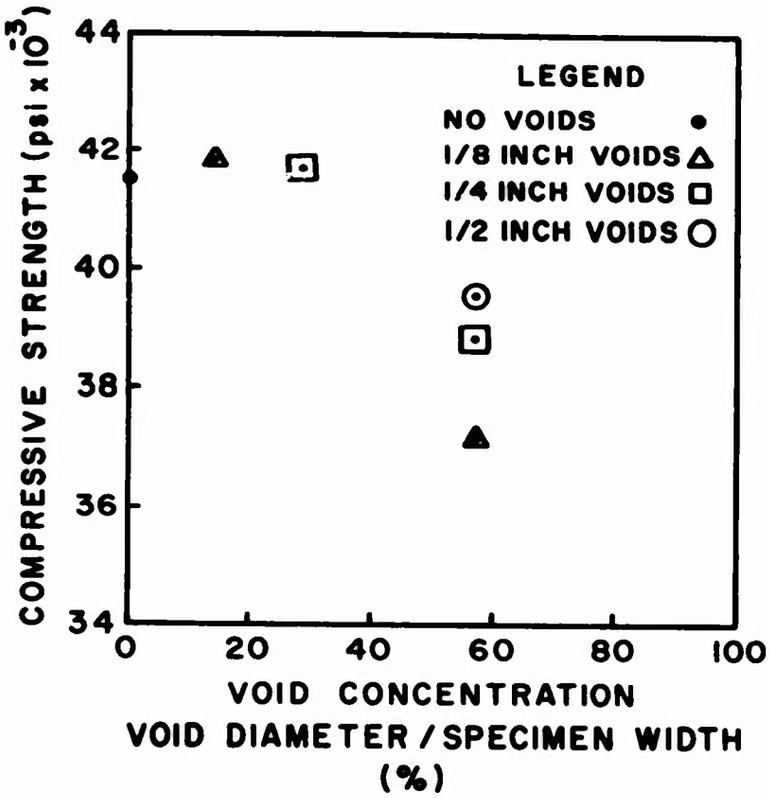

Figure 34. The Effect of Large Interlaminar Voids on Compressive Strength. The data are from Table 17.

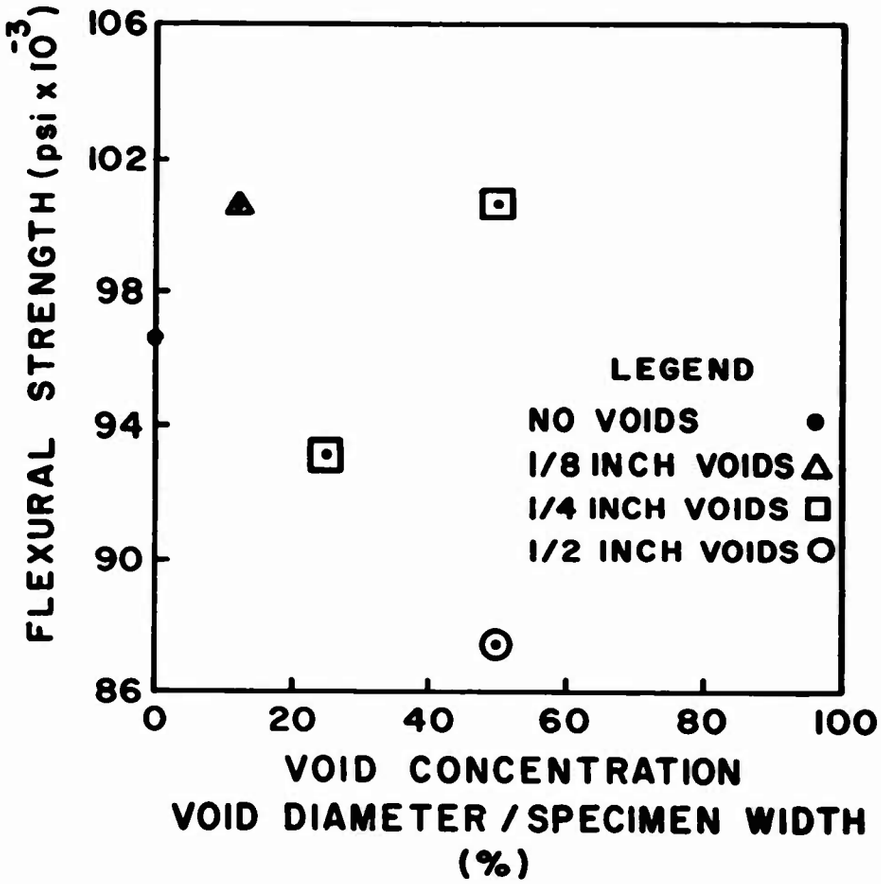

Figure 35. The Effect of Large Interlaminar Voids on Flexural Strength. The data are from Table 18.

TABLE 10
VOID CONTENT, 181-LAMINATES

Cure Pressure	Cure Temperature (°F) 160	180	230
13 psi	-	-	2.34
	-	-	2.77*
70 psi	3.17	-	-
	2.69	-	-
100 psi	-	1.46	-
	-	2.64	-

* Void contents by per cent volume were calculated according to reference 21. Replicate laminates are in lower position as listed in Table 13.

TABLE 11
VOID CONTENT , 120-LAMINATES

Cure Pressure	Cure Temperature (°F) 160	180	230
13 psi	-	-	2.12
	-	-	1.51*
70 psi	2.03	-	-
	-	-	-
100 psi	-	4.33	-
	-	1.74	-

* Void contents by per cent volume were calculated according to reference 21. Replicate laminates are in lower position as listed in Table 14.

TABLE 12
VOID CONTENT, 909-LAMINATES

Cure Pressure	Cure Temperature (°F) 160	180	230
13 psi	-	-	9.35
	-	-	8.06*
70 psi	6.16	-	-
	-	-	-
100 psi	-	7.91	-
	-	8.00	-

* Void contents by per cent volume were calculated according to reference 21. Replicate laminates are in lower position as listed in Table 15.

the 909 had the highest void content and the 120 the least. From the appearance of the pre-preg, it is concluded that high void content of the 909 laminates was introduced during the resin impregnation process.

These voids undoubtedly made some contribution to the level of uniformity noted; but, most important, their presence is believed to have prevented the attainment of the optimum strength that the resin and glass fabric reinforcement are capable of producing. Several recent papers have established this fact for filament windings and have cast some light on the mechanism involved.

Paul and Thompson (reference 20) have photographed the microvoids present in filament would rings and have classified the types observed. The classification consisted of three categories: general, interstitial, and planar. The general voids were approximately circular in cross section, ranging from 2 to 10 filament diameters in size, and extended from just a few to several hundred filament diameters in length in the filament axis direction, containing few or no filaments within them.

The interstitial voids arise from the failure of the resin to completely penetrate the crevices of the filaments that are tightly bonded together. They were usually found in groups, frequently forming a star-shaped pattern, and ran along the filament axes.

Planar voids were oblong in cross section and occurred at discontinuities such as fiber crossovers. It was stated that the void may or may not run along a fiber, depending on how it was formed. Handling during fabrication and resin run-out during cure were listed among the possible causes of formation. Typical void sizes ranged from 20 to 50 filament diameters for the rings examined. Much larger voids were anticipated for the more complex windings, which makes the planar void the most serious type.

Photographs were also presented showing voids to be the origin of stress concentrations. Stress concentration and bonding area reduction at the resin-glass interface were listed as the major causes of failure. These conclusions are borne out, at least in part, in Fried's recent paper (reference 10).

Fried showed that both compressive and interlaminar shear strength are definitely related to void content as determined by conventional burnout tests (reference 10). The theory of

compressive failure for uni-directional filament reinforcement was reviewed to show how voids can interfere with the resin's function of supporting the filaments against buckling through premature debonding at the resin-glass interface.

Most investigators conclude that by far the most important cause of microvoids is air entrainment at the time of impregnation. Paul and Thompson obtained void-free test rings by placing the winding mechanism in a moderate vacuum, on the order of 1 cm. mercury pressure obsolute. Bascom (reference 1) has shown that microvoids in filament windings may be reduced by increasing the wettability of the fiberglass yarn. However, impregnation in a controlled pressure environment (probably a vacuum) is believed to be the method that holds the greatest promise for the immediate future.

These recent research papers on voids are valuable contributions; however, much is yet tobe learned. In particular, a better method of expressing microvoid content is needed. The conventional burnout method has proven valuable but does not provide information on the character of the voids (refer to section E2b following). The matter of void size and type should be considered throughout the size spectrum from the microscopic to the macroscopic.

Fatigue. A review of recent literature revealed no noteworthy research directly linking fatigue strength to void concentration in FRP laminates. The authors were able to make limited observations of the effect of voids, recently, during the fatigue testing of sandwich specimens (reference 17). The same laminate material as used for the resin content study (Table 13, 30 psi and 160° F) was used for the facings. Since the loading was the pure bending (no shear) of a beam specimen, the facings were the main elements being tested, and thus the data can be considered in the present context.

The voids that could be observed with a 100-power microscope were on or near the laminate surface and were on the order of 1 to 5 mils in diameter. These air bubbles did not seem to influence the microcrack pattern; i. e., they were not the origin of the surface cracks that were always present in the failed specimens. Although sometimes cracks terminated in voids or passed through them, there were many voids which were bypassed by cracks. The voids were only faintly visible to the naked eye, and the surface of the specimens could usually be described as smooth and glossy. Some specimens exhibited more air voids than others, but there did not seem to be a

relationship between the fatigue life and the number of voids. On the contrary, some of the specimens with the greatest number of voids had the longest fatigue life.

No doubt, many of the phenomena occurring in the static tests will carry over into the realm of fatigue, but this does not preclude the need for fatigue testing. It points out more strongly that further research must be done regarding the effect of voids on all the mechanical properties of FRP laminates. The observations listed here are very limited and can only be considered preliminary, yet they do suggest the variable role that voids play as their size varies from the small to the large, in addition to the effect of their character--their classification or type and method of formation. The knowledge of the strength-changes through the size spectrum would permit the setting of practical, acceptable rejection limits for aircraft structural components. Such quality control is vital in view of the high safety standards required in aircraft structures.

b. Detection of Voids

Laminates. Not only the characteristics of the voids that can be expected to occur in a given shell structure fabricated a given way (type or class of void, how it is produced, etc.) must be known, but also the concentration, to adequately assess severity in order to determine whether critical rejection limits have been exceeded. Hence, a review of the recent papers on applicable non-destructive methods of testing are presented to guide future research.

Acoustical methods offer considerable promise regarding void detection in FRP laminates, and several companies are presently marketing test equipment. Two cases in particular can be cited where through transmission of ultrasonic beams was successfully employed to relate void information to reflected and transmitted sound energy (references 12 and 13). In both cases, however, emersion of the part being examined was required for adequate coupling of the transducer to the part. This is not considered desirable for large aircraft structures; yet, as mentioned in reference 13 and confirmed by the present authors with equipment available in the OURI lab, considerable error is associated with contact coupling. Further, even with contact coupling, thorough wetting of the surface is necessary. Contract coupling also presents scanning problems. In short, the acoustical system needs further refinement for application to large or complicated aircraft components.

Measurement of the laminate dielectric constant is another possible method of detecting void content and locating points of high concentration. In reference 7, the Delsen Corporation D/K meter was used to make dielectric measurements for laminates in contact with liquids during studies of the effect of exposed glass fibers, resin viscosity, and laminating conditions. It is reasonable to think that the application could also be made to the study of voids. The effect of voids in the dielectric properties of radomes is well known and has been discussed by many authors; hence, further research may well prove dielectric measurement an effective tool.

Examination by X-ray techniques may also be worth consideration. Though no papers directly related to FRP materials were located in the literature, this method should be given consideration. The merits of light techniques should not be overlooked, either. It was shown in reference 20 that void-free laminates were highly transparent: a photograph showing how printing could be read through a 1/4-inch-thick laminate was presented. Optical clarity or measurement of transmitted light could easily be employed quantitatively. Actually, visual examination may prove to be the most practical way of dealing with the larger voids.

Sandwich Structures. A most important aspect of sandwich integrity is the core-to-facing bond. In view of coupling problems, it is doubtful that ultrasonic inspection methods would be adequate to detect unbonded areas, especially for honeycomb cores; however, several other methods have been suggested.

Visual inspection of panels immediately after removal from the fabrication environment has proven effective in some cases, but is not generally considered dependable. The use of lights has also been rejected by most fabricators for the lack of dependability. One of the simplest and most effective methods that has been used is the tapping of the completed part. A completely bonded area will produce a clear tone, while an unbonded area usually produces a lower quality tone or thud. Unfortunately, this method will not permit location of areas of poor or weak bond.

Proof loading is another technique that has been employed. The proof loader is usually designed to place a portion of the facing in flatwise tension by means of a small vacuum chamber sealed to the facing. It is readily seen that this method can be effective, even though large structures may involve difficulties.

Thus, it is concluded, from this brief review of the literature available, that additional research will be required to adequately inspect large FRP sandwich structures for bond integrity. Inspection techniques may have to be tailored to fit the given structural component; hence, some of the work can be a part of any future fabrication program.

F. Conclusions and Recommendations

The major conclusions and recommendations drawn from the research are as follows:

1. As determined by extensive sampling, a multi-layer pre-preg by machine coating can be produced with resin content varying less than ±1 per cent across both the length and the width dimensions at the 95 per cent confidence level.

2. Resin temperature, fabric speed, and roller spacing effected the greatest control on the pre-preg resin content. Automation of the coating machine operation is anticipated to increase the replication to the level of the within-run uniformity of the pre-preg and to permit production on a volume basis.

3. Mechanization of the coating and laminating process has eliminated the larger type of air voids from the pre-preg and minimized the occurrence of microvoids. Calculation of the void content of the 3 types of finished laminate revealed that the 120 laminate had the lowest value and the 909 laminate the highest. This was also confirmed by the appearance of the pre-preg. It is recommended that control of the pressure environment during the impregnation process be investigated as a means of eliminating the microvoids. Negative pressure (vacuum) should especially be investigated.

4. Limited compressive and flexural tests of laminates with large artificial interlaminar voids indicate that there is a critical size relative to strength. Preliminary studies at other laboratories suggest that microvoids are also detrimental to laminate strength; therefore, the effect of both large and small void inclusions on laminate strength should be investigated in detail. Such research should include fatigue as well as static strength.

5. The search for a practical and reliable means of non-destructive testing of FRP laminates should be continued. Future research should encompass acoustical methods, dielectric measurements, X-ray techniques, and light techniques, to mention a few of the possible avenues of approach. This research should also include investigation of methods of determining the integrity of core-to-facing bonds in structural sandwich.

6. The cold storage of EPON 828-Z epoxy pre-preg at 19 degrees Fahrenheit or below for 40 days or less can be considered conservative, in that no degradation of strength was observed under these conditions.

7. A post-cure of at least 1 hour at 300 degrees Fahrenheit is recommended for improved cutting characteristics and full development of room temperature strength for the materials discussed in this report.

8. The B-staging of the pre-preg under the residual winding tension of the coating machine and the size of the laminate up to 22 by 28 inches are expected to have but a minor effect on the strength properties of the epoxy-fiberglass laminates when the cure is under the free edge-flow conditions.

9. For the 3-ply laminates of the 181 style fiberglass fabric, the tensile strength and modulus, the compressive modulus, and the flexural strength and modulus are decreasing functions of resin content between 27 and 40 per cent. The resin content for the maximum values of these strength properties is anticipated to occur near or below 27 per cent. It is recommended that this program of research be continued to the extent that the lower resin contents are achieved and the maximum values of the strength properties are located conclusively.

10. In conjunction with paragraph 9, the compressive strength for the 181 laminates increases from the 27 per cent resin content point and reaches a maximum near 35 per cent.

11. The same trends noted for the 181 laminates also exist for the 6-ply laminates of the 120 style fiberglass fabric in regard to tensile and compressive strength and moduli. The peak in the compressive strength relation is approximately the same in value and occurs at 37 per cent resin content. The values of modulus obtained in this pilot study were lower than those for the 181 laminates, but this condition should be confirmed by more extensive testing.

12. The trends in the mechanical properties of the 3-ply laminates of the J. P. Stevens style 909, S-920 finished, fiberglass fabric were essentially the same as noted for the other two reinforcements with the exception of the compressive strength, which decreased rather than increased with increasing resin content. The peak in compressive strength will probably occur between 20 and 26 per cent resin content in comparison to the previous values of 35 and 37 per cent. It is expected that at optimum resin content the properties of 909 laminates will be greater than those

for the 181 or 120 reinforcement; however, it is recommended that this pilot study of the 909 reinforcement be supplemented with additional test data before it is extensively used in the design of aircraft structures.

13. The limited analysis that could be made in this program indicates the need of a more accurate way of computing the effect of stress concentrations on thin FRP laminates. It is recommended that further study be made of the failure mechanics of this composite material as a means of better understanding the effect of stress concentrations and for the purpose of improving the strength properties.

14. Cure pressure, pre-preg resin content, and time of pre-cure were noted to exercise control over the final resin content of laminates cured in a press under free edge-flow conditions. Pre-preg resin content will probably become the predominate factor in large, closed structures.

15. From the point of view of the strength properties and resin content, the cure condition of 70-psi pressure and 160 degrees Fahrenheit temperature gave the best uniformity relative to replication and within-laminate condition for the laminates reinforced with 181 fiberglass fabric. Therefore, it is recommended that this condition be considered for use in the fabrication of structural shapes from EPON 828-Z epoxy resin and 181 fiberglass fabric.

16. Of the three fiberglass fabrics examined in this program, the 120 style was found to be the most uniform in weight per unit area.

17. A comparison of the uniformity of laminates of all three fabrics (181, 120, and 909) revealed the superiority of the 909 in regard to replication of average strength and resin content and in regard to within-laminate resin content uniformity. This material, however, showed the lowest within-laminate uniformity of strength properties, probably because of its high void content.

18. The completion of the present research program, which is part of the overall effort to determine the optimum employment of raw materials and fabrication processes to develop maximum stress levels, both static and dynamic, has advanced the state of the art to the point where the evaluation of structural shapes can be investigated with confidence. Therefore, it is recommended that this line of research be extended to the design, fabrication, and testing of curved panels and beams of simple geometry leading ultimately to the design of an aerodynamic structure.

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			39.9	94.3	3.37				31.0	89.1	3.02			
			33.0	98.2	3.59	-	-	-	33.5	95.2	3.26	-	-	-
			33.8	103.1	4.19				35.1	103.4	3.53			
		Flexure												
			32.8	76.8	2.34	30.3	87.8	2.45				32.0	84.1	3.21
			36.7	87.2	3.28	32.7	93.0	3.39	-	-	-	34.5	92.2	3.38
			39.5	93.4	4.15	34.7	96.6	3.63				37.1	98.5	3.63
			28.7	35.6	3.42	29.4	35.9	3.22	22.9	33.8	3.52	27.9	35.2	3.78
			31.4	42.1	3.90	36.0	44.5	3.66	28.2	41.2	4.30	33.0	42.1	4.09
			32.9	47.0	4.33	38.8	50.7	4.36	34.2	49.3	4.98	37.9	47.9	4.45
		Compression												
			25.7	35.5	3.78				29.2	33.9	3.37			
			29.6	40.0	4.07	-	-	-	33.8	42.8	3.95	-	-	-
			34.2	46.9	4.47				38.5	46.7	4.39			
			28.5	38.7	2.49	29.4	33.6	2.22	24.2	39.9	2.73	30.2	35.4	2.30
			32.1	42.7	2.72	35.7	41.0	2.54	29.5	42.9	2.88	34.9	40.5	2.46
			34.2	46.7	3.04	38.3	50.0	2.94	32.9	46.3	3.14	37.3	45.7	2.59
70 psi		Tension												
			28.4	37.7	2.58	29.3	39.6	2.54	31.1	35.3	2.26	23.0	43.3	2.30
			31.3	43.3	2.76	33.3	44.2	2.68	34.5	41.2	2.38	27.9	46.3	2.46
			33.8	49.1	3.08	35.7	47.7	2.94	36.9	47.8	2.50	31.5	52.0	2.62
			26.4	90.8	3.20	29.7	82.2	3.03	20.5	84.6	3.35	29.0	98.6	3.59
			29.7	102.4	3.56	33.7	93.1	3.47	27.8	92.5	3.64	31.5	104.0	3.96
			31.3	115.1	3.93	36.7	104.7	3.89	33.0	99.5	4.20	34.2	111.9	5.27
		Flexure												
			25.5	88.8	3.26				30.0	88.6	2.93	23.0	107.0	2.56
			28.9	99.5	3.41	-	-	-	33.6	92.1	3.13	27.5	111.3	3.61
			32.0	104.7	3.75				35.6	100.1	3.50	29.4	117.7	3.91
			25.5	37.3	3.11	24.3	37.8	3.87	32.6	39.8	3.15	34.4	31.7	2.81
			32.4	42.8	3.78	29.9	42.3	4.30	37.6	45.0	3.41	35.9	34.5	3.28
			37.6	47.5	4.57	34.6	45.0	4.78	41.5	48.7	3.85	37.5	37.8	3.46
		Compression												
						24.9	34.6	3.94						
			-	-	-	27.8	40.6	4.24	-	-	-	-	-	-
						30.2	50.4	4.88						
			26.4	33.3	2.18	25.2	37.6	2.28	35.4	34.9	1.87	35.1	30.5	1.93
			34.1	42.1	2.43	30.5	43.4	2.50	38.7	38.3	2.06	36.2	34.7	2.10
			37.5	47.7	2.98	34.5	49.2	2.91	41.1	42.3	2.28	37.2	41.8	2.53
100 psi		Tension												
						23.5	39.1	2.80				30.9		
			-	-	-	28.1	43.8	2.98	-	-	-	35.6	-	-
						30.5	48.4	3.30				38.4		
			22.4	82.6	2.56	25.3	93.6	3.57	33.4	87.7	2.93	33.6		2.32
			30.9	98.5	3.31	27.7	101.7	3.88	36.7	93.2	3.27	35.5	-	2.53
			37.6	108.4	3.89	31.7	109.8	4.17	39.0	98.0	3.52	36.3		2.77
		Flexure												
							86.6	3.21				29.3	96.3	3.23
			-	-	-	28.0**	95.0	3.62	-	-	-	32.6	103.7	3.44
							100.1	3.98				36.1	112.8	3.87

* All entries are tabulated vertically in the following order: low, average, and high. Strength reported in psi x 10^{-3}, modulus reported in psi x 10^{-6}, and resin content (RC) in %. Values for the replicate laminates are placed below those for the original (this scheme is indicated by the letter R at the upper left of the table). The pre-preg was taken from coating machine run 4 with an average resin content of 41.7%.

** The resin content values indexed are the average for the respective laminates.

TABLE 13
RESIN CONTENT STUDY--
181, 3-PLY LAMINATE TEST DATA

Cure Pressure	Strength Property	Cure Temperature and Pre-Cure Time											
		160°F, 8 Minutes			180°F, 5 Minutes			200°F, 4 Minutes			230°F, 3 Minutes		
		RC*	Strength*	Modulus*	RC*	Strength*	Modulus*	RC*	Strength*	Modulus*	RC*	Strength*	Modulus*
13 psi	Compression	32.9	32.2	3.37		33.5	3.04				32.8	39.1	3.24
		35.3	35.6	3.68	35.3**	39.7	3.32	-	-	-	36.6	44.3	3.46
		37.4	43.1	3.94		44.1	3.65				38.5	48.6	3.98
								32.4	34.1	2.93			
		R	R	R	-	-	-	36.7	37.3	3.26	-	-	-
								39.4	42.0	3.60			
	Tension	34.1	33.1	2.01	32.0	32.4	2.18	31.7	33.3	2.38	33.2	36.3	2.01
		35.9	38.3	2.10	35.8	36.9	2.35	35.5	38.0	2.54	36.4	39.9	2.18
		37.4	41.4	2.38	37.4	41.5	2.66	37.4	42.2	2.69	38.0	45.3	2.40
									33.9	2.19	35.6	34.3	2.22
		R	R	R	-	-	-	36.2**	36.2	2.26	37.7	38.1	2.29
									39.4	2.43	39.6	41.1	2.42
	Flexure	33.0	79.5	3.01	30.0	75.3	2.62				32.9	82.7	2.77
		34.9	82.6	3.10	34.7	85.5	2.91	-	-	-	35.4	91.8	2.96
		36.4	85.1	3.48	37.2	93.2	3.33				36.9	99.3	3.12
								32.1	83.5	2.54	34.8	84.1	2.64
		R	R	R	-	-	-	35.7	85.3	3.14	36.7	91.3	3.24
								39.3	94.8	3.55	39.3	98.0	3.36
30 psi	Compression							30.4	34.0	3.45			
		-	-	-	-	-	-	34.3	41.7	3.65	-	-	-
								36.2	46.7	3.98			
			30.4	2.49	30.0	39.9	3.24				31.9	37.9	3.30
		36.5**	34.9	2.98	33.5	42.0	3.54	-	-	-	35.3	45.1	3.51
			40.5	3.47	37.1	45.9	4.11				37.2	50.1	3.84
	Tension	30.5	37.8	2.32	34.0	36.4	2.34	31.9	36.2	2.28	36.4	30.4	2.05
		33.0	41.2	2.46	35.6	41.5	2.54	34.1	39.9	2.47	37.4	32.7	2.16
		35.1	47.4	2.73	37.2	45.2	2.70	36.1	45.5	2.76	38.0	35.3	2.41
		27.2	29.1	1.94	31.6	33.4	2.37	31.4	39.6	2.40	33.6	35.3	2.30
		36.4	37.3	2.36	34.4	37.6	2.46	34.7	44.2	2.55	36.1	40.2	2.47
		41.6	45.1	3.19	36.2	43.0	2.66	37.1	50.9	2.63	38.3	44.7	2.86
		30.9	94.3	3.37				31.0	89.1	3.02			

TABLE 14
RESIN CONTENT STUDY--
120, 6-PLY LAMINATE TEST DATA

Cure Pressure	Strength Property	Cure Temperature and Pre-Cure Time: 160°F, 8 Minutes RC*	Strength*	Modulus*	180°F, 5 Minutes RC	Strength	Modulus	230°F, 3 Minutes RC	Strength	Modulus
								35.9	40.3	2.84
	Compression	-	-	-	-	-	-	38.1	45.5	3.17
								41.3	49.7	3.57
								29.2	34.8	2.96
13 psi		-	-	-	-	-	-	35.5	44.4	3.27
								40.1	49.5	4.12
								31.9	36.5	1.80
	Tension	-	-	-	-	-	-	36.3	40.3	1.97
								38.4	45.2	2.18
								35.0	32.5	1.71
		-	-	-	-	-	-	38.4	38.1	1.94
								41.5	43.2	2.18
		34.3	38.9	2.97						
	Compression	38.5	43.9	3.29	-	-	-	-	-	-
		41.2	49.5	3.90						
70 psi		R	R	R	-	-	-	-	-	-
		30.7	33.3	1.72						
	Tension	37.3	37.9	1.91	-	-	-	-	-	-
		41.3	40.4	2.04						
		R	R	R	-	-	-	-	-	-
					27.9	33.0	3.32			
	Compression	-	-	-	31.9	40.6	3.70	-	-	-
					34.3	49.8	4.04			
					25.4	33.6	3.37			
100 psi		-	-	-	30.1	40.4	3.74	-	-	-
					34.2	45.6	4.22			
					28.4	38.3	2.06			
	Tension	-	-	-	31.7	42.8	2.21	-	-	-
					34.1	47.9	2.38			
					29.1	39.4	2.02			
		-	-	-	33.8	40.4	2.16	-	-	-
					36.0	46.6	2.36			

* All entries are tabulated vertically in the following order: low, average, and high. Strength is reported in psi x 10^{-3}, modulus is reported in psi x 10^{-6}, and resin content (RC) in %. Values for the replicate laminates are placed below those for the original (this scheme is indicated by the letter R in the center left of the table). The pre-preg was taken from the coating machine run 5 with an average resin content of 52.5%.

TABLE 15
RESIN CONTENT STUDY--
909, 3-PLY LAMINATE TEST DATA

		Cure Temperature and Pre-Cure Time								
		160°F, 8 Minutes			180°F, 5 Minutes			230°F, 3 Minutes		
Pressure	Property	RC*	Strength*	Modulus*	RC	Strength	Modulus	RC	Strength	Modulus
								28.7	40.9	3.09
	Compression	-	-	-	-	-	-	31.0	44.9	3.29
								32.4	50.8	3.51
								27.9	42.1	3.09
13 psi		-	-	-	-	-	-	29.9	45.9	3.35
								33.1	50.4	3.96
								29.6	40.8	2.13
	Tension	-	-	-	-	-	-	31.4	44.7	2.26
								33.0	49.2	2.36
								28.5	38.7	2.14
		-	-	-	-	-	-	30.5	45.4	2.31
								32.0	50.0	2.56
		26.3	39.3	3.36						
	Compression	29.2	45.7	3.57	-	-	-	-	-	-
		30.8	52.8	3.97						
70 psi		R	R	R	-	-	-	-	-	-
		27.5	40.6	2.27						
	Tension	30.3	45.5	2.36	-	-	-	-	-	-
		32.1	48.2	2.52						
		R	R	R						
					24.8	43.6	3.39			
	Compression	-	-	-	27.2	46.4	3.67	-	-	-
					31.2	48.1	3.94			
					26.0	42.9	3.27			
100 psi		-	-	-	27.9	46.0	3.68	-	-	-
					29.8	51.8	4.12			
					25.6	41.6	2.33			
	Tension	-	-	-	28.3	46.0	2.42	-	-	-
					29.9	49.1	2.59			
					24.8	41.1	2.31			
		-	-	-	28.4	47.8	2.41	-	-	-
					30.1	51.1	2.62			

* All entries are tabulated vertically in the following order: low, average, and high. Strength is reported in psi x 10^{-3}, modulus is reported in psi x 10^{-6}, and resin content (RC) in %. Values for the replicate laminates are placed below those for the original (this scheme is indicated by the letter R in the center left of the table). The pre-preg was taken from coating machine run 6 with an average resin content of 47.0%.

TABLE 16
RESIN CONTENT STUDY--
PREVIOUS 181, 3-PLY LAMINATE TEST DATA

Cure Pressure	Strength Property	Cure Temperature and Pre-Cure Time: 160°F, 13 Minutes RC*	Strength*	Modulus*	180°F, 5 Minutes RC	Strength	Modulus	200°F, 3 Minutes RC	Strength	Modulus	230°F, 1 Minute RC	Strength	Modulus
13 psi	Compres.	-	-	-	-	32.1	2.89	-	37.9	2.99	-	39.2	3.27
					40.1B	39.1	3.41	37.3A	44.0	3.25	36.0A	48.6	3.49
					-	44.2	4.29	-	47.1	2.63	-	47.0	3.66
	Tension	-	-	-	-	-	-	-	47.0	2.19	-	48.1	2.33
					40.1B	41.2	2.45	37.3A	50.7	2.42	36.0A	53.3	2.37
					-	-	-	-	56.1	2.56	-	60.1	2.40
30 psi	Compres.	-	34.9	2.98	-	33.6	4.14	-	34.1	3.07	-	39.9	3.29
		35.4A	44.0	3.39	28.3X	38.6	4.30	35.9A	42.2	3.57	34.3B	42.1	3.63
		-	48.4	3.78	-	42.1	4.51	-	46.7	4.01	-	45.2	4.00
	Tension	-	51.6	2.43	-	55.5	2.98	-	52.0	2.32	-	54.6	2.52
		35.4A	53.4	2.53	28.3X	60.3	3.26	35.9A	53.5	2.36	34.3B	57.5	2.58
		-	55.0	2.61	-	61.5	3.81	-	56.4	2.42	-	60.8	2.64
70 psi	Compres.	-	39.2	2.99	-	38.8	3.85	-	37.9	3.70	-	31.1	3.72
		36.5A	44.4	3.43	27.7X	41.0	4.19	30.0X	42.3	4.09	27.1A	38.5	4.29
		-	49.0	3.77	-	44.4	4.66	-	45.6	4.61	-	42.4	4.62
	Tension	-	50.5	2.36	-	55.6	2.85	-	56.4	2.56	-	54.0	2.73
		36.5A	53.0	2.48	27.7X	60.2	2.94	30.0X	58.8	2.70	27.1A	56.8	2.85
		-	58.0	2.67	-	63.9	3.00	-	60.8	2.99	-	60.2	3.01

* Values were obtained from reference 16 and are tabulated vertically in the following order: low, average, and high. Strength is recorded in psi x 10^{-3} and modulus in psi x 10^{-6}. The laminates were fabricated from 10-hour B-staged pre-preg with an initial resin content (RC) of A = 41% and B = 42% (X placed after an RC value indicates the pre-preg value was not recorded). Teflon sheets were used as a parting agent. Resin content was obtained by weighing the entire laminate from which each set of compression and tension specimens was taken. The tension specimens were influenced 0.007 inch on their sides.

TABLE 17
COMPRESSION DATA,
PRELIMINARY VOID STUDY

Specimen Number	No Voids Strength psi x 10^{-3}	No Voids Modulus psi x 10^{-6}	1/2-Inch Voids* Strength psi x 10^{-3}	1/2-Inch Voids* Modulus psi x 10^{-6}	1/4-Inch Voids* Strength psi x 10^{-3}	1/4-Inch Voids* Modulus psi x 10^{-6}	Rupture Data**	1/8-Inch Voids* Strength psi x 10^{-3}	1/8-Inch Voids* Modulus psi x 10^{-6}	Rupture Data**
C 9	42.7	2.87								
C 10	43.1	2.94								
C 11	40.9	3.07								
C 12	39.3	2.95								
Average	41.5	2.96								
C 1			38.8	3.43						
C 2			37.3	3.36						
C 3			41.1	3.41***						
C 4			41.3	3.38						
		Average	39.6	3.40						
C 13					36.5	3.14	1/2 D			
C 14					44.8	3.37	1/2 D			
C 15					43.9	3.15	1/2 D			
				Average	41.7	3.22				
C 5					38.7	3.28	2/2			
C 6					39.4	2.99	0/2			
C 7					37.9	2.97	0/2			
C 8					34.3	2.96	2/2			
C 25					33.8	2.73	0/3			
C 26					39.0	3.01	0/3			
C 27					42.0	3.16	2/3			
C 28					42.9	3.10	0/3			
C 29					41.1	3.01	2/3			
				Average	38.8	3.02				
C 21								44.3	3.17	0/4 D
C 22								36.7	3.26	1/4 D
C 23								44.0	3.59	0/4 D
C 24								42.0	3.14	1/4 D
							Average	41.8	3.29	
C 17								35.1	3.03	0/4
C 18								37.5	3.54	4/4
C 19								36.4	2.97	4/4
C 20								34.1	3.20	4/4
C 30								38.4	2.92	4/7
C 31								45.0	3.05	4/7
C 33								39.9	3.06	0/7
C 34								36.0	-	0/7
C 182								32.2	3.17	4/4
							Average	37.2	3.12	

* Voids were 1-mil-thick polyethylene discs arranged as follows: (1) 1/2-inch-diameter void (one per specimen); (2) 1/4-inch-diameter voids (. , ·. , ·:); (3) 1/8-inch-diameter voids (·····, ·····, ·····). Voids were placed under one ply of the 3-ply, 181 laminate and located vertically on the specimens in a random manner.

** First number is number of voids that the rupture passed through. The second number is the number of voids in the specimen, where the letter D is used to identify the single diagonal arrangement. The rupture was always normal to the edges of the specimen.

*** Rupture did not pass through void.

TABLE 18
FLEXURAL DATA,
PRELIMINARY VOID STUDY

Specimen Number	No Voids Strength psi x 10^{-3}	No Voids Modulus psi x 10^{-6}	1/2-Inch Voids Strength psi x 10^{-3}	1/2-Inch Voids Modulus psi x 10^{-6}	1/2-Inch Voids Rupture Data*	1/4-Inch Voids Strength psi x 10^{-3}	1/4-Inch Voids Modulus psi x 10^{-6}	1/4-Inch Voids Rupture Data*	1/8-Inch Voids Strength psi x 10^{-3}	1/8-Inch Voids Modulus psi x 10^{-6}	1/8-Inch Voids Rupture Data*
F 9	95.1	3.61									
F 10	105.7	3.85									
F 11	98.7	3.54									
F 12	108.9	4.13									
a	89.8	3.59									
c	97.9	3.75									
e	103.1	3.89									
f	106.8	4.15									
i	91.2	3.39									
j	96.3	3.37									
k	96.9	3.72									
l	84.1	3.07									
o	86.6	3.50									
p	90.7	3.52									
Average	96.6	3.65									
F 1			75.3	3.06	1/0.49						
F 2			93.0	3.53	1/0.37						
F 3			71.9	2.86	1/0.44						
F 4			109.4	3.78	1/0.42						
		Average	87.4	3.31							
F 13						99.9	3.95	1/0.22			
F 15						92.0	3.29	1/0.10			
F 25						94.3	3.43	1/0.25			
F 28						100.9	4.57	1/0.19			
					Average	93.1	3.78				
F 5						100.9	3.86	2/0.43			
F 6						101.6	4.05	2/0.20			
F 7						92.2	3.74	2/0.43			
F 8						107.6	4.17	2/0 10			
					Average	100.6	3.96				
F 21									101.6	4.21	1/0.125
F 22									100.5	3.85	1/0.125
F 23									104.2	3.67	1/0.125
F 30									95.6	4.22	1/0.100
F 32									95.4	3.44	1/0.125
F 34									105.8	3.94	1/0.125
								Average	100.5	3.89	

* The voids (1-mil-thick polyethylene discs) were not always located exactly at the mid-span of the specimen; hence, the number of voids encountered followed by the approximate void width in the rupture was recorded. During testing, the specimens were oriented so that the voids were nearest the bottom or tension surface (under one of the 3 plies of fabric).

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As a further step toward the acceptance of fiberglass-reinforced plastics (FRP) in primary aircraft structure, the effects of resin content on laminate strength properties were investigated in depth. Functional relationships were obtained and methods of controlling resin content were compared. Uniformity in strength and resin content was observed from the point of view of replication and within-laminate conditions. Most of the data were for laminates of EPON 828-Z epoxy resin reinforced with the 181 style fiberglass fabric; however, limited data were obtained for the 120 and 909 (S-920) fabrics.

In addition, a preliminary study was made of the effects of voids in the cured laminates. A critical size was observed for large artificial voids, and the microvoid content of the pre-preg was minimized by modification of a previously developed multi-ply coating machine. Non-destructive methods of testing for voids were also reviewed.

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