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THIRD-DIRECTION
REINFORCEMENT
OF CARBONIZED
COMPOSITES

BDX-613-144

August, 1970

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MASTER

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ABSTRACT

Interlaminar reinforcement in the C-direction, i. e., perpendicular to the plane of the layers, is particularly important in carbonized composites in which essentially all of the initial binder strength is lost in carbonization. Since carbon filaments are stronger than an equal volume of carbonized resin, directional strength tends to be proportional to the number of continuous filaments in that same direction. Strengths in the A- and B-directions can be increased by cross-directional layup of unidirectional filament layers; however, interlaminar strength requirements commonly necessitate some method of fiber placement perpendicular to the layers. The work described in this report consisted of three potential methods of attaining that objective: cross-weaving fibrous members between adjacent layers; stake-reinforcement in the C-direction; and molding of resin-impregnated, randomly-oriented short fibers. Of those methods, stake-reinforcement was the most promising; however, comprehensive evaluation was not completed before project termination. Each method is described, and the results of initial tests of stake-reinforced and short-fiber molded composites are given in the report.

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Section 1

SUMMARY

The purpose of this program was to obtain high tensile strengths and ablation properties of carbonized composites to requirements which are normally considered beyond the capabilities of such materials. The principal objective was to determine methods whereby composite properties--principally interlaminar and cross-directional strengths--commensurate with that purpose could be achieved.

The work was divided into three categories, each related to a different concept of reinforcement in the thickness dimension of the composite:

- Three-Dimensional Weaving (interweaving of fibrous members from each layer of woven filaments into the members of adjacent layers);
- Stake-Reinforcement (interlaminar reinforcement of cross-ply carbon roving laminated composites by inserting carbon fiber stakes through the stacked layers before press curing and carbonization); and
- Short-Fiber Moldings (molding of short lengths of randomly oriented carbon fibers in a resin binder mix to produce uniform density and equal directional strengths).

THREE-DIMENSIONAL WEAVING

The first phase of the work resulted in development of a special weaving pattern (Three-Dimension Lock Weave) which somewhat resembles a conventional basket weave. That work was described in a separate report, "Design and Development of a Three-Dimensional Interlocked Filament-Wound Composite," BDX-613-143. The method offered practical advantages for applications such as structural members; however, principally because of nonuniform density in the filament crossover areas, the concept was not particularly attractive for an ablation-resistant material. For that reason, three-dimensional weaving is not discussed in depth in this report.

REINFORCEMENT BY STAKING

Interlaminar reinforcement by inserting stakes through the thickness dimension of the composite, which proved to be the most suitable of the three methods tried for the current application, was evaluated as fully as the limits imposed by early termination of the project permitted.

Unidirectionally-oriented roving panels were fabricated from carbon rovings which had been impregnated with DEN 438/BF₃ (Dow epoxy novolac/boron trifluoride binder) before winding. Those panels were then laid up in a cross-ply pattern to provide reinforcement in the A- and B-directions. Composites of suitable thickness were press cured in a mold. Some of each group of panels were to be tested before carbonization; others were to be subjected to various numbers of vacuum reimpregnation and carbonization cycles before testing. Graphite particles were added to the DEN 438/BF₃ resin mix used for vacuum reimpregnation.

Some of the composites were reinforced in the C-direction by inserting carbon rovings through prepunched holes and cutting the ends flush with the outer laminate surfaces. These stake-reinforced panels were then cured and carbonized in the same manner as the others.

Conventional A-direction tensile test specimens and special C-direction tensile specimens of 1 square inch area bonded between aluminum blocks were machined from the panels after carbonization.

Hand punching and insertion of stakes in densities up to 500 or more stakes per square inch is an exacting and time-consuming operation. Although the first panels were staked manually, it was evident that mechanized staking would be required for a practical manufacturing process and to fabricate the number of sample panels necessary for a full evaluation of the stake-reinforcement concept. Therefore, a 17-row staking machine head was designed, and a single-section phototype version was fabricated and installed on a modified Hardinge horizontal milling machine to evaluate the design concept and to stake additional sample panels.

Early test results varied widely and did not reflect sufficient sampling to support firm conclusions; however, trends which are believed to be indicative of the general behavior of stake-reinforced composites were observed.

- An average A-direction tensile strength loss of about 50 percent was incurred in carbonized composites, as compared to that of similar composites before carbonization.
- C-direction tensile strength reductions in carbonized composites which were not staked were as much as 95 percent, compared to about 50 percent for staked samples.

- A-direction tensile strengths of cross-ply carbon roving panels were somewhat less than 50 percent of those of the unidirectional-roving samples.
- C-direction tensile strengths of cross-ply stake-reinforced carbonized composites tend to be proportional to the number of stakes, and are almost independent of the number of fiber ends in the staking material. Strengths obtained were approximately 2 pounds per stake, and were not directly related to the rated strength of 2 pounds per end for the carbon fiber.
- The C-direction strength increases obtained by staking were accompanied by appreciable losses in cross-ply strength.
- Smaller diameter stakes and closer stake spacing tended to yield higher C-direction strengths; however, larger numbers of stakes in a given cross-sectional area created greater losses in cross-ply strengths.

Breakage and bending of stakes by pressure applied to compress the stacked layers during cure was the greatest problem encountered. That problem is believed to have been responsible for much of the variation between strengths of similarly staked specimens. Incomplete impregnation and incomplete compression of the layers during the cure cycle are believed to have contributed to small-area density variations which may have affected the strengths of some of the specimens. Some small voids were observed, particularly around the stakes.

Most of the specimens tested were punched and staked manually; therefore, it is possible that staking inconsistencies might have been responsible for some of the variances in the test data. Several samples were machine punched and staked manually, but few of those had been tested when the project was terminated. Although sampling was not sufficient to support firm conclusions, first tests of machine punched specimens indicated an average increase of about 50 percent in C-direction strength (approximately 750 psi, as compared to about 500 psi for hand punched panels). Many additional panels were machine staked with a single-head prototype staking machine setup; however, those panels had not been carbonized and tested at the close of the project.

SHORT-FIBER MOLDINGS

Test results from the first randomly oriented chopped carbon fiber panels molded with DEN 438/BF₃ yielded both A-direction and C-direction tensile strengths much below those of cross-ply staked composites, both before and after carbonization. Best C-direction strengths were obtained by use of shorter fibers, while longer fibers produced higher A-direction strengths.

Fiber lengths ranging from 1/4 inch to 1-1/2 inches were represented by the first limited sampling. C-direction carbonized tensile strengths of 555 psi and 580 psi were obtained from 1/2-inch fiber specimens, whereas 1-1/2-inch fibers produced 345 psi and 454 psi. Only two of each type were tested. A-direction tensile strengths of 560 psi and 3140 psi were obtained from two samples molded with 1-1/2-inch fibers, compared to an average of 2658 psi for four samples with 1-inch fibers. Because of the large difference in strengths between the first two samples, and the apparent contradiction of the averages, it must be concluded that 560 psi value was not a valid figure (probably because of density variation).

Project termination prevented testing of other panels which represented various fiber lengths and resin systems.

Section 2

BACKGROUND

PRINCIPLES

Laminated filament-wound composites are used for many applications which require relatively high strength-to-weight ratios in the direction of the filaments. Two-dimension strength can be readily obtained by cross-winding or interlacing the filaments in a simple basket-weave pattern. However, third-dimension strength (through the thickness dimension) of multilayer composites has been limited in most uses to the strength of the adhesive used to bond the adjacent layers. There are many available binder systems that are capable of providing appreciable interlaminar strength at moderate temperatures; however, the strength of the binder becomes critical in applications which involve high-temperature environments.

When filament-wound composites are carbonized to reduce weight and to increase resistance to ablation at temperatures of several hundreds or thousands of degrees Fahrenheit, conventional binder systems lose virtually all of their initial strength. Therefore, carbonized laminates require additional reinforcement in the direction perpendicular to the layers.

Ablation tests were conducted by another agency on samples of carbonized composites supplied by Bendix; however, the resulting data is not available. Representative data from room temperature tests conducted at the Bendix Kansas City facility are shown in this report.

It has been shown that third-dimension (C-direction) interlaminar strengths of carbonized composites can be increased by repetition of the impregnating and carbonizing processes, and that densification of carbon structures by processes such as chemical vapor deposition (CVD) of carbon is effective in increasing both tensile strength and resistance to ablation. However, the strengths obtained by those methods have not been sufficient to meet many requirements.

PURPOSE

The purpose of the work reported herein was to develop methods of increasing the interlaminar tensile strengths of filament-wound or fiber-reinforced composites in which the filaments or fibers have conventionally been laid in two directions only. The principal interest of this program was in C-direction reinforcement of carbonized carbon roving composites in which interlaminar tensile strength was approximately 100 psi before third-direction reinforcement. The ultimate objective was to achieve carbonized interlaminar tensile strengths of approximately 2000 psi without incurring excessive loss of strengths in the directions of the layer

rovings (A- and B-directions). Several different methods of interlaminar reinforcement were to be evaluated; however, a previously developed carbonization process was to be utilized.

APPROACH

Activities pursuant to the objective of this work encompassed three basic categories:

- Three-Dimension Lock Weave (interweaving the filaments of each layer of the composite into those of adjacent layers);
- Staking (inserting short lengths of stiff carbon fibers through the thickness of the composite prior to reimpregnation and carbonization);
- Slurry-Cast Chopped Fibers (molded as a solid, nonlaminated structure to produce uniform tensile strength in all directions).

Section 3

THREE-DIMENSIONAL LOCK WEAVE

The initial phase of the program consisted primarily of development of a special weaving pattern (3-D Lock-Weave). A report, BDX-613-143, on that work was published in January, 1967.

The 3-D Lock Weave concept consists of interweaving filaments of fibrous members from each layer of the composite into the adjacent layers in a cross-ply pattern which somewhat resembles a basket-weave. The resulting cross-orientation of fibers through the thickness dimension of stacked layers increases the interlaminar tensile and shear strengths with respect to the C-direction of the composite.

The 3-D Lock Weave is an effective method of increasing the C-direction strength of woven laminated composites; however, the open areas at the filament crossover points tend to produce regions of lesser density which may be incompletely filled during the impregnating process, thereby creating voids in the cured composite.

A further practical disadvantage of lock-weave interlaminar reinforcement lies in the difficulty of weaving such a complex pattern. Conventional weaving machines are not readily adaptable to the process, and hand weaving is very time-consuming.

Glass fiber woven tapes were used to weave the first panels produced during the early part of the weaving development, principally for the reason that tapes are more convenient to handle in a hand-weaving process. However, carbon roving panels were produced later on a hand-weaving fixture. A special fixture was designed for weaving shapes of revolution as large as 18 inches in diameter. Because of increasing interest in other methods of interlaminar reinforcement, however, the fixture was not fabricated.

After curing, the first DEN 438 resin impregnated panel had a density of 1.091 g/cc. However, radiography revealed voids within the weaving pattern that had not been completely filled during the impregnating process. The thickness of the panel had been controlled by adjustment of press force during cure; therefore, the material was not fully compacted. An impregnated and cured carbon roving panel is shown in Exhibit A-1 (see appendix for all exhibits). A similar panel is shown after carbonization in Exhibit A-2.

Although the 3-D Lock Weave offers distinct advantages in such applications as structural members, it did not appear particularly attractive as a high-strength high-temperature ablation material because of structural nonuniformity in the woven composite. For that reason, and because of the time required to produce hand-woven specimens, the 3-D Lock Weave had not been fully evaluated when the program was terminated.

Section 4

STAKING

DEFINITIONS

Staking consists of insertion of continuous stiff carbon or graphite filaments through all layers of resin impregnated multiple-layer cross-ply laminates, with the ends of the C-direction reinforcement stakes terminating at the outer surfaces of the composite. Staking is performed after layup of pre-impregnated rovings and B-stage cure, but before complete press curing and carbonization.

Carbonization (synonymous with pyrolization) is a high-temperature controlled process applied in an oxygen-free environment to convert the resin binder to carbon, thus increasing the carbon content and improving the ablation resistance and strength-to-weight ratio of the composite.

Ends is a term which designates the number of monofilaments which comprise a yarn (slightly twisted) or a roving (straight). Typically: Glass--204 monofilaments per end; Carbon or Graphite--714 monofilaments per end.

Filament as frequently used in the field of filament winding may apply to either rovings or yarns. The term, although generally understood by engineers actively engaged in the field, may cause confusion when sharp lines of distinction must be made between yarns and rovings.

DEN 438/BF₃, as used in this work, consists of Dow Chemical Company epoxy novalac (DEN 438), 100 pbw to boron trifluoride, 3 pbw. (BF₃).

BASIC CONSIDERATIONS

It has been shown that (when other factors remain constant) the tensile strength of a filament-wound composite in a direction parallel to the filaments may be increased by increasing the number of filament ends per cross-sectional area in that same direction. An estimated concentration of approximately 7000 ends of carbon roving per square inch can be attained in a unidirectional (A-direction only) winding. Cross-winding (90-degree winding orientation) reduces the end count by a factor of slightly more than 1/2 in each direction. Therefore, both A-direction and B-direction strengths of cross-ply laminates are expected to be less than 1/2 of the A-direction strength of equivalent unidirectional windings.

Since all fibers within the composite occupy space, placement of a portion of the fibers in the C-direction (perpendicular to the wound or woven layers) will make less space available for A- and B-direction filaments. Therefore, interlaminar reinforcement by cross-laminar fiber orientation (staking) is possible only at the expense of reduction of strength in the A- and B-directions.

Theoretically, if an equal number of fibers were laid in each of the three directions, the A-, B-, and C-direction strengths should be essentially the same, each being less than 1/3 of the A-direction strength attainable in a unidirectional-fiber composite. There are other variables, however, such as fiber breakage, nonuniformity of impregnation and curing, and uneven stake concentration, which may influence the strength in one or more of the directions.

INITIAL STAKING EXPERIMENTS

Samples of unidirectional and 0°/90° cross-ply preimpregnated carbon rovings were fabricated to form panels from which test specimens were machined. The rovings were preimpregnated with the DEN 438/BF₃ resin system and B-stage cured. Layers were cut and stacked to form laminated panels approximately 3/4-inch thick and press cured in a mold. The panels to be used for determination of carbonized strengths were nominally subjected to four DEN 438/BF₃ vacuum reimpregnation and carbonization cycles.

Standard tensile test specimens were machined from the panels to determine A- and B-direction strengths, both before and after carbonization. C-direction tensile specimens were prepared by bonding sections of 1-square-inch area to aluminum blocks to provide support in testing, as shown in Exhibit A-3.

Additional specimens were prepared in a similar manner from hand-staked third-direction reinforced panels for a comparative evaluation of the increase in C-direction strength which might be attained by staking. The first panels were staked with 5-end preimpregnated carbon fiber stakes on 0.075-inch centerlines (approximately 180 stakes per square inch). Holes were hand punched in the stacked laminates and the stakes were manually inserted and cut flush with the outer surfaces before curing in the press. Because of the pressure exerted on the ends of the stiff stakes, some of the fibers tended to bend or break during cure. An effort was made to solve that problem by curing the staked composites between rubber blocks in the press; however, the difficulty was not completely eliminated.

Although the results of the first tests (shown in Table 1) were encouraging, it was evident that a large number of staked panels would be required to provide definitive evaluations of the many variables which might be expected to affect the tensile strengths of the composites.

Table 1. Tensile Strengths of the First Unidirectional and Cross-Ply Pluton-H Carbon Roving Laminated Composites Before and After Carbonization

Roving Direction	Test Direction	Tensile Strength (psi)	
		Before Carbonization	After Carbonization
Unidirectional A-direction only	A	24,000	12,000
	B	- - -	- - -
	C	- - -	30 to 160
Cross-Ply A & B directions	A	11,000	5,500
	B	11,000	5,500
	C	1,000	100
Cross-ply - Staked Thornel-40 10-end on 0.075 centers 180 stakes/in ²	A	- - -	- - -
	B	- - -	- - -
	C	1,500	500

To obtain the highest possible interlaminar tensile strengths of carbonized, stake-reinforced composites with minimal sacrifice of cross-ply strengths, the following factors must be considered:

- The effects of stake density (number of stakes per square inch) upon interlaminar strength and strength losses in the cross-ply directions;
- Relative merits of different carbon rovings and staking fibers;
- Effects of stake diameter and number of ends in the staking fibers;
- Comparative merits of several base resin systems, combinations thereof, and additives thereto;
- Comparison of composite strengths before and after carbonization to determine the amount of strength lost by carbonization;
- Effects of repeated reimpregnation and carbonization, as well as other prospective means of carbon densification, upon the tensile strength of carbonized composites; and the
- Relative merits of preimpregnated rovings versus dry rovings with vacuum impregnation after layup.

Hand insertion of stakes is a very tedious and time-consuming operation, and at stake densities of 500 or more stakes per square inch it becomes virtually impossible to maintain uniformity of spacing. Therefore, it was evident that hand staking of a sufficient number of panels to provide a meaningful evaluation of staking as a manufacturing process would require many months of work, and that additional time would be required to carbonize the composites, prepare the test specimens, conduct tests, and correlate the data.

Much thought was given to development of a sampling plan and calculation of theoretical parameters, based upon general knowledge and the results of the initial tests. Figure 1 shows the theoretical relationship between stake spacing and the number of fiber ends per square inch for single-end, 2-end, 5-end, and 10-end carbon fiber stakes, indicating the maximum stake density that can be attained with each stake size. For an approximately equal distribution of fibers in all three directions ($7000/3 = 2333$), Figure 1 indicates stake center spacings of about 0.067, 0.050, 0.030, and 0.020 for 10-end, 5-end, 2-end, and single-end stakes, respectively. Later tests, however, (Figure 2) indicated that C-direction strength tended to be proportional to the number of stakes, rather than to the number of ends, and was almost totally independent of the number of ends in the staking fiber after the composites were carbonized. It is therefore apparent that greater concentrations of small-diameter single-end stakes produce the highest C-direction strength, with reduced risk of fiber breakage and subsequent losses of cross-ply strengths in the A- and B-directions.

Because of the time lapse during carbonization, preparation of samples, and testing, attention was turned to development of a mechanized method of punching and staking additional panels required for further evaluation of the process. That development, conducted while awaiting test results from the second group of hand-staked panels, is described in the following section.

Figure 1. Calculated Stake Spacing

Figure 2. Theoretical C-Direction Tensile Strength Versus Number of Stakes per Square Inch--Based Upon the Rated Single-Fiber Burst Strength of Pluton-H Carbon Roving

THE STAKING MACHINE

The first effort to mechanize the staking operation consisted of design and fabrication of the air-actuated prepunch tool shown in Exhibit A-4. That tool provided holes for the reinforcing stakes, but required that the stakes be inserted manually. It was intended as a step toward development of an automatic staking machine, and was not expected to contribute directly to establishment of a production staking capability. A sample panel (top and bottom views) punched with the tool is shown in Exhibit A-5. Those photographs show a distinct irregularity in the pattern on the back side of the panel, indicating that the punch tended to bend, or wander, as it was forced through the panel. This proved to be one of the major problems in machine punching, and it became more critical when hole spacing was very small. Both sharp-point and flat-end punches were tried, with no discernible difference in performance.

The knowledge gained from use of the prototype prepunch tool was incorporated in the design of a multiple staking head to punch, shear, and insert 17 stakes in a single machine cycle. The design sketch in Figure 3 shows one of the 17 similar mechanisms of that head. The guide plate (A in Figure 3) remains in a fixed position over the laminate during the machine cycle, while the staking head indexes to three positions during that time. In position one, 17 holes are simultaneously punched through the composite; in position two, the staking material is fed into the guide plate position by the feed mechanism. As the head moves to position three, the staking material is sheared to predetermined length; and after indexing to position three, the 17 stakes are inserted into the composite by another set of punches.

Although the multiple-head design appeared feasible, and would be necessary to produce stake-reinforced structures in appreciable quantities, a mechanism of such complexity was considered too costly for the early stages of this development program. Therefore, the single-punch head shown in Figure 4 was constructed to prove feasibility of the multihead design and to facilitate production of additional stake-reinforced test panels for the evaluation program. The single-punch head was very similar to the multipunch design; however, the three-position indexing feature was eliminated and a single punch was used for both punching and inserting the stakes. Those changes necessitated altering the shearing mechanism.

The single-punch head was installed on a modified Hardinge horizontal milling machine, as shown in Exhibits A-6 through A-9. In addition to mounting the staking head on the pedestal of the machine, modifications to the Hardinge mill included additions of an air-driven cam device to provide lateral movement of the work table in uniform increments, a storage and feed fixture for the staking material and a system of

Figure 3. Design Sketch of 17-Row Automatic Staking Head

Figure 4. Sketch of Single-Stake Prototype Staking Head

programmable automatic air valves to sequence the air cylinders on the head and the work table. The work table was positioned manually for each row of stakes. The tool spindle on the machine was not used.

The single-punch staking machine is shown in operation in Exhibit A-6. The machine provided an improved method of fabricating staked test panels, with more uniform spacing, greater possible stake density, and a considerable time-saving as compared to manual staking. It is not adequate, however, for manufacturing large numbers of stake-reinforced structures.

The method of loading the staking material on the storage and feed fixture and threading the fiber into the staking head is shown in Exhibits A-7 and A-8. Thornel-40 single-end graphite fiber, in lengths up to approximately 25 feet, was used exclusively in the staking machine.

A detailed view of the punching and staking operation is shown in Exhibit A-9a, illustrating the punching portion of the machine cycle. The stake is inserted by the following stroke of the punch. A partially staked cross-ply carbon roving panel is shown in Exhibit A-9b.

STAKING EVALUATION

Most of the A-direction tensile test data was obtained from 4-inch test specimens machined from the various laminated panels. The fail sections of those specimens were machined to dimensions of $1/4$ by $1/4$ inch, or $1/16$ square inch. Because of the possibility that a small fail section might not yield true tensile strength, a few larger specimens (9 inches long, with 1-square-inch fail sections) were prepared and tested for comparison to the results obtained from the small specimens. The larger specimens are noted Special Test Specimen in the tables. The higher and more consistent tensile strengths of the larger specimens can probably be attributed to better average cross-sectional density and reduced surface effects in those specimens.

Since equal numbers of carbon rovings were laid in both A and B directions, A- and B-directional strengths were assumed to be equal. The few tests conducted tended to support that conclusion; therefore, B-direction strength tests were conducted on only a few representative panels.

C-direction tensile specimens for interlaminar strength tests were prepared by bonding sections of panels between aluminum blocks and machining the laminates for an area of 1 square inch, as shown in Exhibit A-3. Although a few of those specimens failed at the surface layer or at the bond, most of the strengths obtained were fairly consistent.

Except as otherwise noted, the test results shown in the following graphs and tables were obtained from cross-ply 15-end Pluton-H preimpregnated carbon roving laminated composites. The carbonized specimens (sometimes designated carbon/carbon) were machined from composite panels that had previously been subjected to several vacuum-impregnation/pyrolyzation cycles. DEN 438/BF₃ was used as the binder system for both preimpregnation before layup and for reimpregnation after each carbonization cycle. Cross-oriented layers of unidirectional carbon rovings were stacked to provide sufficient thickness for fabrication of test specimens. The inner-laminar-reinforced panels were staked by inserting Thornel-40 single-end or carbon roving multiple-end stakes in prepunched holes after the layers were stacked, but before the panels were press cured.

Before completion of the prototype staking machine, the panels were staked manually. Machine-staked panels were fabricated later; however, considerable time was required to carbonize the panels, prepare the specimens, and perform the tests. For that reason, test data from machine-staked panels had not become available in sufficient quantity to support a comparative evaluation of the two staking methods when the project was terminated.

To obtain the highest possible C-direction strength with the least sacrifice of A- and B-direction strengths, various numbers of staking-fiber ends, stake diameters, and stake spacings were sampled. Pluton-H rovings were used for all multiple-end stakes. Thornel-40 was used for the single-end stakes.

The results of preliminary tests listed in Table 1 provide basic strength values to which the results of later tests can be compared. The data in Table 1 indicates an A-direction strength loss of about 50 percent because of carbonization. After-carbonization C-direction strength of 500 psi for cross-ply specimens staked with Pluton-H 10-end fibers on 0.075 inch centers was approximately five times that of similar panels that were not staked. The C-direction strength loss due to carbonization was approximately 66 percent. Because of limited sampling, however, the values shown in Table 1 cannot be considered representative of the materials.

Table 2 lists A-direction strength data from a later series of tests of a unidirectional roving composite similar to that shown in Table 1. The later series reveals a much higher A-direction strength before carbonization (a five-specimen average of 41,500 psi, compared to a Table 1 figure of 24,000 psi). Carbonized A-strengths were also higher (an average of 18,500 psi, compared to 12,000 psi in Table 1), but A-strength loss due to carbonization was slightly higher (55 percent). It should be noted that the A-strengths listed in Table 2 are for carbonized specimens that failed in the clamp areas, before ultimate tensile stresses at the test sections were reached. For that reason, it may be assumed that the true A-strengths were greater than the values listed.

Table 2. Tensile and Flexural Strengths and Moduli of Unidirectional Preimpregnated Pluton-H Carbon Roving Laminated Composites Before and After Carbonization

Sample Number	TENSILE		Sample Number	FLEXURAL	
	Strength (X 10 ³ psi)	Modulus (X 10 ⁶ psi)		Strength (X 10 ³ psi)	Modulus (X 10 ⁶ psi)
UNCARBONIZED					
1	42.1	- - -	-	- - -	- - -
2	38.9	- - -	-	- - -	- - -
3	41.4	- - -	-	- - -	- - -
4	40.5	- - -	-	- - -	- - -
5	38.5 * ¹	- - -	-	- - -	- - -
6	39.1 * ¹	- - -	-	- - -	- - -
7	35.8 * ¹	- - -	-	- - -	- - -
8	43.3 * ¹	- - -	-	- - -	- - -
9	45.0	- - -	-	- - -	- - -
10	35.0 * ¹	- - -	-	- - -	- - -
Valid Avg.	41.5				
CARBONIZED					
1	18.67 * ²	5.74	1	29.6	5.04
2	18.70 * ²	5.19	2	31.9	5.14
3	18.34 * ²	4.73	3	33.0	5.49
4	18.59 * ²	4.84	4	25.1 * ³	5.22
Avg.	18.50 * ²	5.12	Valid Avg.	31.5	5.22
*1 Not valid breaks--specimens failed diagonally through test section and clamp ends					
*2 Failed at clamp ends--did not reach ultimate tensile stress					
*3 Layer delamination					

Before-carbonization and after-carbonization A-strengths of cross-ply Pluton-H 15-end carbon roving composites are again compared in Table 3. Although strength values varied, an average A-direction strength loss of 42.8 percent is indicated. Comparisons of each of the three carbonized averages to each of the two uncarbonized averages follow.

<u>Carbonized</u>	<u>Uncarbonized</u>	<u>A-Strength Loss (percent)</u>
X-1	X-17	53
X-2	X-17	38
X-3	X-17	53
X-1	X-18	43
X-2	X-18	26
X-3	X-18	44
		<u>257</u>

Average loss = $257/6 = 42.833$ percent

Comparing the highest strength value from the uncarbonized specimens (24,000 psi for Panel X-17 Samples 3 and 7) to that of the lowest strength carbonized samples (10,400 psi for Panel X-1 Samples 2, 5 and 6) the strength loss was 58 percent.

In view of the many possible causes that may produce nonuniform density and strength in laminated composites, and the further variations that may occur during carbonization, the strength values shown in Table 3 are considered as consistent as can be expected.

Test results from additional carbonized 0°/90° cross-ply 15-end carbon roving composites reinforced with carbon 2-end stakes, 100 stakes per square inch, are compared to similar unstaked composites in Table 4. The unstaked panels were not tested in the C-direction. Although comparatively small (2-end) stakes were used, and the stake density was only 100 stakes per square inch, the A-strength loss due to staking averaged 58 to 65 percent.

A- and C-direction strengths of bidirectional cross-ply preimpregnated Pluton-H 15-end carbon roving stake-reinforced carbonized composites are listed in Table 5. The cause of the large differences in the data for Panel X-4, as compared to that of X-5, is not clear. However, the data for X-6 indicates that both A-direction and B-direction strengths of 20°/20° oriented composites are much higher than those of the 0°/90° layups.

Table 3. Comparative A-Direction Tensile Strengths of 0°/90° Cross-Ply Preimpregnated Pluton-H 15-end Carbon Roving Laminated Composites Before and After Carbonization

NOT CARBONIZED			CARBONIZED (Two Cycles)		
Panel Number	Sample Number	A-Direction Tensile Strength (X 10 ³ psi)	Panel Number	Sample Number	A-Direction Tensile Strength (X 10 ³ psi)
X-17	1	22.5	X-1	1	10.5
	2	23.6		2	10.4
	3	24.7		3	11.6
	4	23.3		4	11.7
	5	25.5		5	10.4
	6	22.2		6	10.4
	7	24.7		7	13.2
	8	24.6		8	<u>12.4</u>
	9	24.5		Avg.	11.3
	10	<u>24.5</u>			
	Avg.	24.1	X-2	1	14.6
X-18	1	21.6		2	14.5
	2	19.2		3	14.3
	3	20.2		4	15.5
	4	21.2		5	14.5
	5	17.1		6	14.6
	6	22.3		7	14.3
	7	18.1		8	<u>15.6</u>
	8	20.8		Avg.	14.7
	9	<u>19.1</u>			
	Avg.	19.9	X-9	1	10.7
				2	<u>11.8</u>
				Avg.	11.25

Table 4. Comparison of A-Direction Tensile Strengths of Carbonized 0°/90° Cross-Ply 15-end Carbon Roving Composites With and Without Staking, and C-Direction Strengths of Four Staked Samples

Panel Number	Sample Number	Tensile Strength (psi)	
		A-Direction	C-Direction
Carbon 2-end Stakes - 100 Stakes per Square Inch			
X-6	1	5,440	166
	2	5,190	229
			333
			252
Average		5,315	245
Without Stakes			
X-1	1	10,500	--
	2	10,400	--
	3	11,600	--
	4	11,700	--
	5	10,400	--
	6	10,400	--
	7	13,200	--
	8	12,400	--
Average		12,726	
X-2	1	14,600	--
	2	14,500	--
	3	14,300	--
	4	15,500	--
	5	14,500	--
	6	14,600	--
	7	14,300	--
	8	15,600	--
Average		14,987	
Comment: The above averages indicate that A-direction tensile strength was reduced approximately 58 to 65 percent by staking.			

Table 5. Tensile Strengths and Moduli of Carbonized Bidirectional Cross-Ply 15-end Carbon Roving Laminates With Third-Direction Stake Reinforcement

Fiber Orientation From A-Dir	Panel Number	Sample Number	Tensile (psi)			
			Strength		Modulus	
			A-Dir.	C-Dir.	A-Dir.	C-Dir.
0°/90°	X-4	1	3,560	617	- -	- -
		2	3,960	480	- - -	- -
		3	3,050	515	- -	- -
		4	- -	576	- -	- -
		5	- -	518	- -	- -
		6	- -	570	- -	- -
		7	- -	638	- -	- -
		8	- -	491	- -	- -
		Average	3,500	550		
0°/90°	X-5	1	1,640	888*	- -	- -
		2	1,080	825*	- -	- -
		3	- -	988*	- -	- -
		4	- -	1,168*	- -	- -
		Average	1,360	990		
20° Left 20° Right	X-6	1	7,360	2,176	4.02	- -
		2	7,490	1,906	3.51	- -
		3	8,790	1,508*	3.48	- -
		4	- -	1,325*	- -	- -
		Average	7,880	2,041	3.61	- -
<p>*Adhesive bonding failure</p> <p>Comment: The above averages indicate approximately 160 percent greater A-direction tensile strength for 20°/20° fiber orientation, as compared to that of 0°/90° fibers.</p>						

The effects of various numbers of ends in the staking material and different stake spacings upon A- and C-direction tensile strengths are shown in Table 6. It has been shown that the number of ends in the staking fiber has little affect upon interlaminar strength and that C-direction strength tends to be proportional to staking density. Five-end stakes, 250/sq. in. (Panel 5, Samples 6 through 10) yielded slightly higher C-direction strength than 10-end stakes at the same stake spacing (Panel 2, Samples 3 and 4). A-direction strength tests were not performed on many of the panels; however, the values given indicate excessive loss of A-direction strength in the stake-reinforced panels.

Tensile and flexural strengths and moduli of bidirectional cross-ply 15-end Pluton-H preimpregnated carbon roving carbonized composites without stake reinforcement are shown in Table 7. Although the sampling was very limited, it appears that the A-direction strength of 45°/45° oriented laminates is inferior to that of 0°/90° layups. No C-direction tests were performed on Panels X-14 and X-15; however, the values given for Panel X-17 are well below those previously shown for similar 0°/90° uncarbonized panels. The tensile modulus and the flexural strength and modulus values listed appear fairly consistent.

The following graphs show trends which were encountered in the staking development. The test points shown around the normalized curves illustrate wide variations in the strength values of similar specimens and indicate that uniform strengths cannot be predicted, at least within the scope of the work performed. Because of cancellation of the project, test results from a large number of machine staked panels were not available. It is possible that more uniform stake spacing in those samples might have produced more uniform strengths.

Although the uncarbonized ultimate tensile strength of carbon roving is rated at two pounds per end, tests revealed that strength was reduced by carbonization to approximately two pounds per stake, regardless of the number of ends in the roving from which the stakes were made. (See Figure 2.) It therefore became apparent that greater interlaminar strength can be obtained and that a greater percentage of cross-ply strengths before staking can be retained by use of a larger number of smaller diameter stakes. That principle is illustrated by the theoretical plot shown in Figure 5. For example: A stake density of 450, 0.005-inch diameter single-end stakes would theoretically enable more than 90 percent of the initial A- and B-direction strengths to be retained. However, it is not known that this theory would hold for stakes of such small diameter. Moreover, stakes of 0.005 diameter would likely tend to bend or break during insertion. For those reasons, a compromise between maximum C-direction strength and minimum cross-ply strengths, from a practical point of view, appears to be advisable.

Table 6. Tensile Strengths of 0°/90° Cross-Ply Preimpregnated 15-end Carbon Roving Carbonized Composites--Staked and Nonstaked Values--Showing the Effects of the Number of Ends in the Staking Fiber and the Stake Spacing

Panel No.	Sample No.	Stakes Ends Density	STAKED		NOT STAKED	
			A-Tensile (psi)	C-Tensile (psi)	A-Tensile (psi)	C-Tensile (psi)
1	1	2-end 250/in ²	1417	514	7198	58.0
	2		3293	413	4518	69.0
	3		2456	425	- - -	51.3
	4		1969	390	- - -	20.0
	5		- - -	385	- - -	- - -
	Avg		2289	425	5858	49.5
2	1	10-end 64/in ²	- - -	325	5611	85.0
	2		- - -	387	5667	104.0
	Avg			356	5636	94.5
	3	10-end 250/in ²	- - -	502	5571	86.0
	4		- - -	505	6668	75.0
	5		- - -	- - -	4989	41.0
	6		- - -	- - -	5802	- - -
	Avg			503.5	6257	67.3
5	1	5-end 125/in ²	2390	287	5790	25.6
	2		4060	405	7320	44.6
	3		- - -	242	- - -	- - -
	4		- - -	250	- - -	- - -
	5		- - -	292	- - -	- - -
	Avg		3225	295	6555	35.1
	6	5-end 250/in ²	- - -	600	- - -	- - -
	7		- - -	630	- - -	- - -
	8		- - -	238	- - -	- - -
	9		- - -	585	- - -	- - -
	10		- - -	618	- - -	- - -
	Avg			534		

Table 7. Tensile and Flexural Strengths and Moduli of 45°/45° and 0°/90° Cross-Ply Preimpregnated 15-end Carbon Roving Carbonized Composites Without Third-Direction Reinforcement

Fiber Orientation From A-Dir.	Panel Number	Sample Number	Tensile			Flexural, A-Direction	
			Strength		Modulus	Strength (X 10 ³ psi)	Modulus (X 10 ⁶ psi)
			A-Dir (X 10 ³ psi)	C-Dir. (psi)	A-Dir. (X 10 ⁶ psi)		
45° Left 45° Right Parallel Repeating	X-7	1	7.86	37	- - -	- - -	- - -
		2	8.14	47	- - -	- - -	- - -
		3	6.69	56	- - -	- - -	- - -
		4	8.68	60	- - -	- - -	- - -
		Avg.	7.84	50			
0°/90°	X-14	1	11.10	- -	3.61	18.9	2.86
		2	4.08	- -	2.71	19.1	3.01
		3	9.46	- -	3.13	17.3	3.03
		4	10.16	- -	2.73	16.2	2.91
		5	9.68	- -	2.91	- - -	- - -
		Avg.	8.89		3.01	17.87	2.95
0°/90°	X-15	1	10.10	- -	3.31	- - -	- - -
		2	9.30	- -	3.37	- - -	- - -
		3	9.80	- -	3.32	- - -	- - -
		4	11.80	- -	3.26	- - -	- - -
		5	10.70	- -	3.65	- - -	- - -
		Avg.	10.34		3.38

Figure 5. Theoretical A- and B-Direction Strength Retention Versus Stake Spacing for Stakes of Different Diameters--Based Upon Experimentally Determined Carbonized Strength of Two Pounds per Stake, Independent of the Number of Ends in the Staking Fiber

Because of a reduction in the number of continuous fibers in the A- and B-directions caused by fiber displacement and breakage by stake insertion, cross-ply directional strengths can be expected to diminish as the total cross-sectional area (size and number) of the stakes is increased. That behavior is illustrated by the plot of test data in Figure 6.

The range of C-direction tensile strength values obtained from a typical group of staked carbonized samples is shown in Figure 7. The highest of these approached anticipated strengths; however, the loss in cross-ply strengths (shown by the dashed curve at the top of Figure 7 and fully projected in the preceding figure) was considered excessive.

A plot of interlaminar and cross-ply tensile strengths from another group of stake-reinforced carbonized samples is shown in Figure 8. Data points for 0.010 diameter single-end stakes at 450 stakes per square inch are shown for comparison. Cross-ply strength retention was higher for the small stakes; however, C-direction strengths were lower than those of the 5-end stake samples.

OBSERVATIONS

The wide ranges of cross-ply and interlaminar tensile strengths encountered in testing carbon roving laminates, both before and after staking and before and after carbonization, are believed to have been caused principally by one or more of the following characteristics of the materials and processes:

- Nonuniform density of the basic roving and resin composite;
- Nonuniform vacuum reimpregnation or curing;
- Nonuniform pressure during cure; (Since the pressure was in some instances adjusted to control panel thickness, some of the panels might have been more compacted than others.)
- Nonuniform stake spacing due to manual punching and stake insertion, or because of punch bending when punching the panels;
- Breakage or displacement of some of the cross-ply fibers when punching the panels and inserting the stakes;
- Breakage or bending of some of the stakes as they were inserted, or because of pressure applied during cure;
- Nonuniform reimpregnation after carbonization, particularly in regard to adherence to the stakes; (This was known to contribute to many surface-layer failures in C-direction staked specimens.)

- Failure of some of the stakes to penetrate all layers of the panels; and
- The granular, porous nature of carbon and carbonized materials tends to create areas of greater and lesser densities, which may be further complicated by minute quantities of foreign materials in the mix. (If such a region of low density falls within the fail section of a test specimen, that specimen will fail at a stress level far below the true tensile strength of the material. Tensile specimens with large cross-section fail areas tend to average out the effects of small irregularities in material density and the influence of surface-effect upon the ultimate tensile strength. Perhaps for those reasons, the larger specimens tested appeared to be most representative of the strengths of the composites.)

Data from selected carbonized samples reinforced with Thornel-40 single-end stakes on 0.050-inch centers are listed in Table 8. Although the test values varied considerably, the averages listed appear to be representative of the strengths that can be predicted within the scope of the work conducted. Those strengths, however, are short of the goals established at the outset of the investigation.

Figure 6. Ultimate Tensile Strengths of 0°/90° Carbon Roving Stake-Reinforced Carbonized Composites in A-, B-, and C-Directions Versus the Number of Stakes per Square Inch

Figure 7. Range of Interlaminar Tensile Strength Test Results From Carbon Roving Stake-Reinforced Carbonized Composites Versus Number of Stakes per Square Inch

Figure 8. Cross-Ply and Interlaminar Tensile Strengths of 15-end Carbon Roving Carbonized Composites With 5-end Stake Reinforcement, Data From Single-End Staked Samples Shown for Comparison

Table 8. Data From Selected 0°/90° Cross-Ply Preimpregnated Carbon Roving Carbonized Composites Reinforced With Thornel-40 Single-End Stakes on 0.050-inch Centers

Sample Number	STRENGTHS		
	A-Tensile (psi)	C-Tensile (psi)	A-Flexural (psi)
X-1	- - -	658	10,346
X-2	- - -	878	- - -
34-1	- - -	1,011	- - -
34-2	- - -	905	12,719
22-1	5,841	- - -	- - -
22-2	4,390	- - -	- - -
22-3	5,180	- - -	- - -
21-1	4,740	410	14,900
21-2	<u>4,460</u>	<u>611</u>	<u>10,900</u>
Average	4,920	745.5	12,216

Section 5

CHOPPED FIBER REINFORCEMENT

Use of randomly-oriented short lengths of carbon fibers provides a third potential method of obtaining equally distributed directional strengths in molded composites.

Masses of randomly-oriented fibers were obtained by circulating a low concentration of short carbon fibers in water through a screen in a cavity. The fibers were then dried, vacuum impregnated with a resin system, and molded into test panels. Carbon fibers ranging from one-fourth to two inches in length were molded with several different resin systems to acquire a large number of chopped-fiber panels for evaluation.

Photographs of some of the first chopped-fiber panels molded with the DEN 438/BF₃ resin system are shown in the Appendix. Exhibit A-10 shows a side view of a small panel before carbonization. An edge view of a similar panel after carbonization is shown in Exhibit A-11. The failed ends of a 9-inch stake-reinforced, carbonized tensile specimen are shown in Exhibit A-12.

Test results from tensile specimens prepared from the first group of chopped-fiber panels are shown in Table 9. A-direction tensile strengths before carbonization were less than one-half of the average A-tensile of 0°/90° cross-ply continuous roving composites shown in the preceding section. C-direction strengths, however, were slightly higher than those of the uncarbonized cross-ply panels which were not staked. Although the highest A-strength was obtained from a panel molded with 1-inch-long fibers (Panel 10-1, at 11,146 psi), the mating panel (10-2) yielded a strength of only 4640 psi (a difference of more than two-to-one). Panels 13-1 and 13-2 (1/2-inch fibers) yielded A-strengths of 6592 psi and 9520 psi, both well above the strength of Panel 10-2.

Although the two-specimen average strength of the 1/2-inch fiber specimens (Panels 13-1 and 13-2) was somewhat higher (8056 psi) than the 7893 psi average of the 1-inch fiber panels (10-1 and 10-2), the A-direction strengths of the 1/4-inch fiber panels (21-1 and 21-2) were well below those of the longer fiber panels and were as consistent as might be expected. For that reason, it is apparent that Panel 10-2 was not representative of the inherent strength of the material. The results of these first tests therefore indicate that longer fiber lengths produce greater strengths in the A- and B-directions.

Test results from the first group of carbonized chopped-fiber specimens are shown in Table 10. That limited sampling indicated that short fibers produce highest C-direction strengths in carbonized composites.

Table 9. Data From First Noncarbonized Chopped-Fiber Panels

Panel Number	Fiber Length	Tensile Strength (psi)	
		A-Direction	C-Direction
22-1	1 in.	- - - -	1,184
22-2	1 in.	- - - -	1,512
21-1	1/4 in.	4,820	- - -
21-2	1/4 in.	4,470	- - -
13-1	1/2 in.	6,592	- - -
13-2	1/2 in.	9,520	- - -
10-1	1 in.	11,146	- - -
10-2	1 in.	4,640	- - -

Table 10. Data From First Carbonized Chopped-Fiber Panels

Panel Description	A-Direction		C-Direction	
	Tensile (psi)	Flexural (psi)	Tensile (psi)	Flexural (psi)
FD-18 (1.27 g/cc) 1/2 in. fibers	- - - - - -	- - - - - -	555 580	8,936 9,678
FD-20 (1.26 g/cc) 1 in. fibers	- - - - - -	- - - - - -	454 345	7,779 11,462
FD-23 (1.18 g/cc) 1 in. fibers	2,720 1,830 2,770 3,430	8,546 5,671 - - - - - -	- - - - - - - - - - - -	- - - - - - - - - - - -
FD-26 (1.09 g/cc) 1-1/2 in. fibers	560 3,140	7,457 - - -	- - - - - -	- - - - - -

Many other chopped-fiber panels were fabricated; however, because of the time required to carbonize the panels and process the specimens, test data had not been received when the project was terminated.

The additional panels that were fabricated for carbonization, but were not tested, are listed in Table 11. Various binder formulations and fiber lengths were included to provide a comprehensive evaluation of the chopped-fiber molding process.

The resins and additives listed in Table 11 are identified as follows:

RT--Allied CP424 roofing tar;

PR 275--3M polymerizable coal tar pitch;

DEN 438--Dow epoxy novolac resin system; and

Durez--Hooker Chemical phenolic resin.

All of the panels listed in Table 11 were submitted for carbonization except Panels FD-29 and FD-36, which were scrapped because of poor impregnation. Panels FD-30 and FD-33 were also poorly impregnated, but to a lesser degree.

Sufficient information was not available at the close of the project to support valid conclusions. Appreciable C-direction tensile strengths (555 to 580 psi) were attained for carbonized chopped-fiber composites in the first test; however, A-direction tensile strengths were very low compared to those of cross-ply staked panels. (See Section 4.) Based on present information, it appears that molded chopped-fiber composites are inferior in strength to cross-ply stake-reinforced structures.

Table 11. Additional Chopped-Fiber Panels--Fabricated, but Not Tested

Panel Number	Resin	Additive	Fibers (in.)
FD-29	Durez, 50 pbw	RT, 50 pbw	2
FD-30	Durez, 60 pbw	PR 275, 40 pbw	2
FD-31	Durez, 75 pbw	PR 275, 25 pbw	2
FD-32	DEN 438, 20 pbw	RT, 80 pbw	2
FD-33	Durez, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	2
FD-34	DEN 438, 20 pbw	RT, 40 pbw - PR 275, 40 pbw	2
FD-35	DEN 438, 30 pbw	RT, 70 pbw	2
FD-36	Durez, 30 pbw	RT, 70 pbw	2
FD-37	DEN 438, 20 pbw	PR 275, 80 pbw	2
FD-38	Durez, 25 pbw	RT, 60 pbw - PR 275, 15 pbw	2
FD-39	DEN 438, 70 pbw	RT, 30 pbw	1
FD-40	DEN 438, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-41	DEN 438, 30 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-42	DEN 438, 40 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-43	DEN 438, 50 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-44	Durez, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-45	Durez, 30 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-46	Durez, 40 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-47	Durez, 50 pbw	RT, 75 pbw - PR 275, 5 pbw	1
FD-48	DEN 438, 40 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-49	Durez, 50 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-50	DEN 438, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-51	DEN 438, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-52	DEN 438, 50 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-53	Durez, 20 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-54	Durez, 30 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-55	Durez, 40 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4
FD-56	Durez, 30 pbw	RT, 75 pbw - PR 275, 5 pbw	1/4

Section 6

COMMENTS

Although limited sampling and incompleteness of tests because of project termination prevented full evaluation of the reinforced composites, trends that provide general indications of materials strengths were observed.

Of the interweaving, staking, and short-fiber molding methods tried, stake-reinforced cross-ply carbon roving panels produced the highest interlaminar and surface-direction strengths. The principal disadvantages of stake-reinforcement are excessive losses of A- and B-direction strengths because of stake insertion and the time required to stake structures of appreciable size. Although the stake-reinforced samples tested revealed wide strength variations between similar specimens, most of the specimens tested were from manually staked panels. It is believed that test results from the later machine-staked panels would have yielded greater uniformity.

It is believed that more extensive sampling and development of processes (especially the machine-staking process) would produce more uniform strengths and densities, although it is not apparent that strength and ablation properties in keeping with the objectives of the program could be attained.

APPENDIX

PHOTOGRAPHIC ILLUSTRATIONS OF MATERIALS,
PROCESSES, AND FACILITIES

P-52140

Exhibit A-1. A Hand-Woven Three-Dimension Interlock Panel

P-52139

Exhibit A-2. Woven Panel After Press Cure, Vacuum
Impregnation, and Carbonization

P-52134

Exhibit A-3. Innerlaminar Tensile Strength Test Specimen Mounting

P-52131

Exhibit A-4. The Experimental Prepunch Tool

P-52144 (above)

P-52143 (below)

Exhibit A-5. Top and Bottom Views of a Prepunched Panel

P-53404

Exhibit A-6. The Prototype Staking Machine Setup

P-53405

Exhibit A-7. Loading the Staking Fiber

P-53406

Exhibit A-8. Threading the Staking Fiber Through the Head

P-53403

Exhibit A-9a. Closeup of the Staking Operation

P-53407

Exhibit A-9b. Edge of a Partially Staked Panel

P-52135 (above)

P-52133 (below)

Exhibit A-10. Failed Stake-Reinforced Tensile Specimens
(4-inch above, 9-inch below - not to same scale)

P-52147

Exhibit A-11. A Failed Innerlaminar Bond in a Staked Panel

P-52146

Exhibit A-12. A Molded Chopped-Fiber Panel

A-13

P-52167

Exhibit A-13. Enlarged End View of a Chopped-Fiber Panel

P-52168

Exhibit A-14. A Magnified View of a Section Across the Ends
of a Failed Chopped-Fiber Tensile Specimen
(The entire areas of the ends are not shown)