

# NASA Contractor Report 158921

## Development of Graphite/Polyimide Honeycomb Core Materials

R. H. Stone

LOCKHEED-CALIFORNIA COMPANY  
Burbank, CA 91520

SEPTEMBER 1978

**NASA**

National Aeronautics and  
Space Administration

Langley Research Center  
Hampton, Virginia 23060  
N78-1187-1





PRECEDING PAGE BLANK NOT FILLED

CONTENTS

	Page
SUMMARY . . . . .	1
INTRODUCTION . . . . .	3
Polyimide Resins . . . . .	4
Fiber Reinforcements . . . . .	5
TECHNICAL APPROACH . . . . .	7
Task 1 - Graphite/Polyimide Honeycomb Core Development with F-178/T300 and NR-150A2:B2/T300 Material Selection. . . . .	7
F-178/T300 development . . . . .	8
NR-150A2:B2/T300 material and processing development . . . . .	9
NR-150A2:B2/T300 panel fabrication and testing . . . . .	12
Task 2 - Graphite/Polyimide Honeycomb Core Development with NR-150B2/HM-S and PMR-15/HM-S Material Selection. . . . .	13
Fabric development . . . . .	14
PMR-15 process development . . . . .	15
NR-150B2 process development . . . . .	16
LARC-13 bonding development . . . . .	18
Testing . . . . .	19
DISCUSSION OF RESULTS . . . . .	20
NR-150A2:B2/T300 Results . . . . .	20
PMR-15/HM-S Test Results . . . . .	23
NR-150B2/HM-S Test Results . . . . .	25
LARC-13 Test Results . . . . .	26
CONCLUSIONS . . . . .	28
REFERENCES . . . . .	30

## LIST OF TABLES

Table		Page
1	Summary of Materials . . . . .	31
2	Test Outline . . . . .	33
3	Test Outline - Optimization of Graphite/Polyimide Laminates . . .	34
4	NR-150A2:B2 Processing Development . . . . .	36
5	NR-150A2:B2/T300 Pre-Cured Skins and Test Laminates . . . . .	38
6	NR-150A2:B2 Honeycomb Panel Bonding Development . . . . .	40
7	PMR-15 Processing Development . . . . .	41
8	PMR-15/HM-S Pre-Cured Skin and Test Laminates . . . . .	44
9	NR-150B2 Processing Development . . . . .	45
10	LARC-13 Bonding Development . . . . .	50
11	Test Results - NR-150A2:B2/T300 Sandwich Panel Specimens . . . .	52
12	Test Results of NR-150A2:B2 Specimens and Loading Blocks Re-Bonded with LARC-13 . . . . .	54
13	PMR-15/HM-S Sandwich Panel Test Results . . . . .	55
14	NR-150B2/HM-S Sandwich Panel Test Results . . . . .	57

LIST OF FIGURES

Figure		Page
1	Graphite honeycomb core shear strength (RT) . . . . .	59
2	Graphite honeycomb core shear modulus (RT) . . . . .	60
3	Graphite/polyimide honeycomb core . . . . .	61
4	Diagram of heated platen . . . . .	62
5	Heated platen with lead wires . . . . .	63
6	PMR-15/HM-S laminates . . . . .	64
7	Flatwise tensile specimens bonded with LARC-13 adhesive . . . . .	65
8	Plate shear specimens showing partial core failures . . . . .	65

**DEVELOPMENT OF GRAPHITE/POLYIMIDE  
HONEYCOMB CORE MATERIALS**

**Robert H. Stone**

**Lockheed-California Company  
Burbank, California**

**SUMMARY**

The activities in this program consisted of development and evaluation of honeycomb panel constructions consisting entirely of graphite/polyimide composite materials. The graphite/polyimide materials were used in the honeycomb core webs and in precured sandwich skins. Polyimide adhesives were used for skin-core bonding. The purpose of this activity was to develop light weight sandwich constructions suitable for use in the 260°C - 316°C (500°F - 600°F) range, which could provide comparable shear strength and stiffness to metallic honeycomb constructions.

In the initial program activity, two polyimide matrix systems were evaluated: F-178, a bis-maleimide type addition polyimide developed by Hexcel Corporation; and NR-150A2:B2, a noncrosslinked aromatic condensation polyimide with thermoplastic characteristics. For the core webs, the two resins were impregnated onto a light weight bidirectional graphite fabric incorporating 1000 tow Thornel 300 yarns. For the skins, the resins were impregnated onto a standard bidirectional graphite fabric incorporating 3000 tow Thornel 300 yarns.

The F-178 polyimide proved to have a compatibility problem with the woven Thornel 300 fibers which had an epoxy sizing, and this portion of the program was discontinued. Fabrication of core and prepreg, with the NR-150A2:B2/T300 was successful. Precured skins were fabricated with this system, and bonded to the core utilizing a cell edge coating of the NR-150A2:B2 resin as the adhesive.

The NR-150A2:B2/T300 panel constructions were tested at room temperature, and at 288°C (550°F) before and after 288°C (550°F) aging. The test results indicated that the graphite honeycomb construction provides generally comparable mechanical properties to metallic honeycomb constructions. The NR-150A2:B2/T300 system appeared to be acceptable, but somewhat marginal for 288°C (550°F) use.

The next phase of the program continued the graphite/polyimide honeycomb development with two additional systems; NR-150B2, a more thermally stable version of the NR-150 type polyimides; and PMR-15, a NASA developed addition polyimide which utilizes three commercially available monomers. To avoid the compatibility problems encountered with the sized Thornel 300, and to provide a more thermally stable fiber, HM-S fibers were used in the form of bidirectional woven fabrics for both the core webs and the skins. These HM-S fabrics were a recent development. The fabrics had a PVA sizing used for weaving which was subsequently removed by heat cleaning.

Honeycomb core, prepreg, and sandwich panels were fabricated with these two systems. A recently developed polyimide adhesive, LARC-13, was utilized as the skin-core adhesive. LARC-13 was developed by NASA, and is a modification of the PMR-15 system. In both cases, the graphite/polyimide precured skins were of less than optimum quality, because of thermal mismatch with the PMR-15/HM-S system which caused resin cracks, and a void problem with the NR-150B2. Testing of the sandwich constructions indicated that 288°C (550°F) was a marginal use temperature for the PMR-15 and LARC-13 systems. The NR-150B2 system proved more satisfactory for 288°C (550°F) use.

Development and fabrication of the graphite/polyimide core was performed by Hexcel Corp., Dublin, California under the direction of Juan Chorne.

## INTRODUCTION

High temperature composite systems incorporating thermally stable graphite fibers and polyimide resins appear to have widespread applicability for advanced vehicles, such as supersonic aircraft, which require the combination of high performance, light weight, and thermal stability these materials offer. An effective means of utilizing graphite/polyimide systems is in honeycomb sandwich constructions which for many applications provides the most structurally efficient and lowest weight design concept.

The use of graphite composite as a honeycomb core material provides for the first time a nonmetallic core construction capable of matching the specific core shear strength and stiffness properties of metallic cores (Figs. 1 and 2). The most effective graphite construction to provide these properties is the use of continuous fiber reinforcements in the web oriented on the bias ( $\pm 45^\circ$ ) to the core thickness. The chief problem with aluminum honeycomb constructions have been their sensitivity to corrosion, but available nonmetallic fiberglass and nylon paper cores cannot be substituted in many of the more highly loaded components in which metallic core has been used, because of their low core shear strength and stiffness. Graphite honeycomb provides the necessary match of properties to metallic honeycomb, and with composite skins effectively eliminates corrosion as a service problem. Despite this advantage, the costs of graphite core will probably limit its use as a substitute for aluminum core in applications for the standard temperature range up to  $177^\circ\text{C}$  ( $350^\circ\text{F}$ ).

In the higher temperature applications, in supersonic applications where use temperatures will range up to  $316^\circ\text{C}$  ( $600^\circ\text{F}$ ), graphite core incorporating polyimide resins would be substituted for the relatively expensive titanium brazed honeycomb constructions. For these applications, graphite core would be more likely to show cost effectiveness, and for this reason emphasis has been placed on development of graphite honeycomb core for higher temperature applications.



## Polyimide Resins

An extensive amount of development work has been accomplished over the last several years on new polyimide resins for use in the range from 232°C (450°F) to 316°C (600°F), and several new polyimide systems have become available for incorporation in this program. The original condensation polyimides, while providing excellent thermal stability up to 316°C (600°F), were extremely difficult to process because of the nature of the condensation reaction which produced volatile by-products during the polymerization reaction. This also resulted in relatively high void content laminates with resultant adverse effects on mechanical properties, fatigue life, and durability.

The development of polyimide formulations which polymerized or "imidized" with addition reactions eliminated this problem and greatly improved processability of the system, but with some loss of thermal stability. The bis-maleimide type addition polyimides, of which Hexcel's F-178 is an example, provides ease of processing approaching that of epoxies and can be cured at 177°C (350°F). With proper post-cure a use temperature of 232°C (450°F) can be achieved.

For applications in the 232°C - 316°C (450°F - 600°F) range, other new systems have been developed to provide a more optimum balance of processability and thermal stability. The NR-150 polyimide systems developed by Du Pont incorporate a very thermally stable, highly aromatic polymer structure produced through a condensation reaction. (Ref. 1) The polymer chains in the NR-150 systems do not cross-link, however. This produces a noncrystalline material with thermoplastic characteristics capable of melt flow above the glass transition temperature (T<sub>g</sub>). More significantly for processing, the absence of cross-linking permits the removal of volatile condensation products and residual solvent by means of an oven post-cure.

Another new polyimide is the PMR-15 system developed by NASA. This is an addition polyimide produced through the reaction of three commercially

available monomers in a mixture with ethanol solvent. (Ref. 2) One of the monomers, a monomethyl ester of a difunctional acid (NE), provides end-caps for a low molecular weight prepolymer chain produced in the initial addition reaction. Upon further application of heat, the end-caps open to produce the cross-linking reaction which produces the final cured resin. PMR-15 provides advantages of void-free reactions, production from readily available monomeric reactants, and a high degree of thermal stability provided by the polymeric structure.

Relatively few polyimide film adhesives have become available since development of the condensation systems. An adhesive recently developed by NASA is a modification of the PMR-15 chemistry in which one of the three monomers, methylene-dianiline (MDA), is replaced with another amine system to provide improved adhesion characteristics. This system, called LARC-13, is available as a supported film adhesive.

#### Fiber Reinforcements

The Statement of Work for this program specified the use of graphite fabric reinforcement. For the honeycomb core webs, the use of woven fabric provided a distinct advantage, in that fabric prepreg with its greater integrity in the uncured form, could be adapted to the expansion process of core fabrication in which the core is expanded into the hexagonal shape after bonding the nodes in the correct pattern on the flat sheet webs. Core with graphite tape webs would have to be fabricated using the more expensive corrugation process in which the nodes are bonded after the webs have been formed into the corrugated shape.

The use of fabric severely limited the selection of graphite fiber reinforcement. At the time this program was initiated, the only readily available graphite fabric utilized Thornel 300. Thornel 300 has relatively poor thermal-oxidative stability as compared to higher modulus graphite fibers, such as HT-S or HM-S (Ref. 3), but available data (reported verbally to Lockheed) indicated that Thornel 300 provided acceptable properties up to

the 260°C - 288°C (500°F - 550°F) range when encapsulated in a thermally stable polyimide matrix. Thornel 300 is available commercially only with an epoxy sizing. This sizing, designated 309, is an uncatalyzed epoxy resin which varies in content from 0.3% to 2% on the fiber, and was the sizing used in the above mentioned specimens where acceptable elevated temperature properties were obtained. Thornel 300 was at that time available with a polyimide sizing, but only at a very high premium cost.

At the time the second task of the program was initiated, a new family of woven graphite fabrics had been developed utilizing HM-S graphite. HM-S is a high modulus  $3.86 \times 10^5$  MPa ( $56 \times 10^6$  psi nominal) fiber which because of its high processing temperature and high degree of graphitization has much greater thermal stability than Thornel 300. HM-S had previously been available only in 10,000 filament tows which were unsuitable for weaving. The recent development of 3000 and 1000 filament tows made possible the development of standard and light weight bidirectional woven fabrics suitable for skins and honeycomb core respectively. HM-S is used without sizing but with a heat cleaned surface for prepreg tapes. For the weaving operation, a PVA sizing is required, and this must be removed after weaving by heat cleaning. This is one potential problem, but a more basic drawback with HM-S is its relatively low strength, low strain to failure, and brittle characteristics as compared to the lower modulus fibers such as Thornel 300. HM-S is also a more expensive fiber, but the availability of a thermally stable fiber in fabric form appeared to outweigh potential disadvantages.

It should be noted that the Celion graphite fibers, reported to have comparable mechanical properties, weavability, and costs to Thornel 300 but with greatly improved thermal stability, were not available at the time the second task of the program was initiated. This fiber would have been an obvious choice as a fiber reinforcement for this program had it been available.

## TECHNICAL APPROACH

### Task 1 - Graphite/Polyimide Honeycomb Core Development with F-178/T300 and NR-150A2:B2/T300 Material Selection

For the initial program on graphite/polyimide honeycomb core and panel development, two recently developed polyimides were selected: Hexcel's F-178 bis-maleimide type polyimide, and Du Pont's NR-150A2:B2 polyimide. These systems are discussed more fully in Section 1 of this report. F-178 was selected to provide optimum processability for this program because of its capability of being cured at 177°C (350°F) using cure procedures comparable to epoxy cure cycles. It was recognized that F-178 would be limited in thermal stability compared to other systems.

NR-150A2:B2 was selected to provide an optimum thermal stability in the 260°C - 288°C (500°F - 550°F) range. This system is a 50:50 mixture of two resins of the NR-150 series, NR-150A2 and NR-150B2. NR-150A2 has a glass transition temperature (T<sub>g</sub>) of 280°C - 300°C (536°F - 572°F), and requires a processing temperature of approximately 343°C (650°F), providing a maximum use temperature of 260°C (500°F). NR-150B2 has a T<sub>g</sub> of 350°C - 371°C (662°F - 700°F) and a processing temperature of around 427°C (800°F), providing a maximum use temperature of 343°C (650°F). The 50:50 mixture resulted in intermediate T<sub>g</sub>, processing, and use temperatures (322°C [611°F], 371°C [700°F] and 288°C [550°F] respectively), and was selected as optimum for this program. It should be noted that the above processing temperatures are molding temperatures which are sufficiently above the T<sub>g</sub> so that melt flow of these essentially thermoplastic materials can occur. The polymerization of the material is essentially complete at 204°C (400°F).

The selected fiber reinforcement, as discussed in Section 1, was epoxy sized Thornel 300 in two fabric styles. For the core webs Style 1136 fabric, a bidirectional 34 x 33 satin weave incorporating 1000 filament tow T300 yarn was used; for the prepregs used in skin and test laminate fabrication, Style 1133 fabric, a bidirectional 24 x 23 satin weave incorporating 3000 filament

tow T300 yarn was used. These fabrics and the various resin-fiber combinations used in the program are summarized and described in Table 1.

Hexcel Corp. was responsible for material development in this Task, including weaving of the fabrics, prepreg fabrication, and honeycomb core development and fabrication. Lockheed was responsible for precured graphite/polyimide skin fabrication, honeycomb panel fabrication, and testing. The test plan, which was identical for both program Tasks is outlined in Table 2. Lockheed also fabricated a series of test laminates and honeycomb test specimens with each material system in this program for submittal to NASA.

F-178/T300 development. - The initial activity at Hexcel, after weaving of the fabrics, was a prepregging processing development with the two polyimide resins. Preliminary mechanical tests were run on laminates fabricated from these pre pregs, and the results indicated a compatibility problem with the F-178/T300 system. The problem was noted when room temperature flexure strengths of laminates given a 260° (500°F) post-cure (required to increase thermal stability and use temperature of the F-178 system) were observed to have a 50% reduction compared to laminates which only had the 177°C (350°F) initial cure. This indicated that short-time exposure to 260°C (500°F) seriously degraded laminate properties, and the fiber-resin interface bond involving the 309 epoxy sizing was considered to be the probable cause of this problem. Previous work at Hexcel with F-178/Thornel 300 tape pre-preg had not encountered a similar compatibility problem, and as these had been with another Thornel 300 batch it was thought that batch variations in fiber sizing formulation, content, or distribution might be the cause.

Micrographic examination of T300 fiber from different batches did indicate a variability in the amount of sizing. Flexural strengths of the NR-150A2:B2/T300 system, after cure at 399°C (750°F), were not reduced as much as the post-cured F-178 samples indicating that the problem was not caused by fiber degradation during post-cure, and that the compatibility problem was specific to F-178.

Hexcel's approach to resolving this problem included evaluation of different fiber batches, post-cure variations, resin modifications, and MEK extraction to remove the fiber sizing after weaving and prior to impregnation. Flexural specimens were taken from laminates incorporating these variables and tested at RT and 260°C (500°F). Results of this evaluation are given in Table 3, and include data on both unidirectional tape specimens and woven fabric specimens.

The test results given in Table 3 confirmed a batch variability. MEK extraction resulted in less RT flexural strength reduction after post-cure; and both post-cure variation (step-wise heat-up with maximum post-cure temperature reduced to 288°C [550°F]), and F-178 resin modification improved the RT properties. Flexure tests at 260°C (500°F), however, revealed a substantial property reductio...

Hexcel performed additional tests on modified F-178/T300 laminates under an in-house program (whose results are therefore not reported here), and was able to eliminate the loss of RT flexure strength after post-cure. However, retention of room temperature flexure strength was only 60% at 177°C (350°F) and 30% at 232°C (450°F) and the F-178/Thornel 300 was dropped from the program as the result of a mutual decision of NASA, Lockheed, and Hexcel. It should be noted that extensive data from other programs have indicated excellent properties of the F-178 at temperatures up to 232°C (450°F), and that the problems encountered in this program appear related to a specific fiber sizing/resin compatibility problem.

NR-150A2:B2/T300 material and processing development. - The program activities on development and fabrication of NR-150A2:B2/T300 honeycomb core continued, and Hexcel was successful in fabricating by the expansion process a block of this core with 0.95 cm (3/8 inch) cells and 88.1 kg/m<sup>3</sup> (5.5 lb/cu ft) density. The core was sliced into 1.27 cm (1/2 in.) segments for testing at Lockheed, and 2.54 cm (1 in.) slices for submittal to NASA, (Fig. 3).

The core segments provided to Lockheed had a cell edge coating of NR-150A2:B2 resin applied to the cell edges using a proprietary Hexcel process. The NR-150A2:B2 was applied to the cell edges unstaged but with a thixotropic additive. Preliminary flatwise tensile tests were performed with specimens incorporating precured NR-150/graphite skins which were prepared for bonding by light hand sanding. Two specimens were bonded, one of which incorporated a cocured inner ply of the NR-150A2:B2/T300 fabric prepreg. This specimen failed at 3568.2 kPa (517.5 psi) while the specimen without the cocured ply failed at 2223.6 kPa (322.5 psi). These results indicated that the combination of the cell edge NR-150 coating and a cocured prepreg ply against the core provided a satisfactory skin-core bond. The above test specimens were fabricated at Hexcel and tested by Lockheed.

The cure cycle recommended for NR-150A2:B2 at that time by Du Pont required cure temperatures in the 371°C - 427°C (700°F - 800°F) range. Lockheed's approach for skin fabrication and skin-core bonding was to develop an autoclave process. This limited cure pressure to 1379 kPa (200 psi), but preliminary work at Du Pont indicated that this was an acceptable cure pressure (Ref. 4), even though some reduction in mechanical properties had to be accepted, as compared to press cured laminates processed at much higher pressures. The laboratory autoclave at Lockheed is limited to a maximum operating temperature of 316°C (600°F), and a heated platen was developed to provide the supplemental heating required. This platen is shown in Figs. 4 and 5 and consisted of two 61 cm x 61 cm (24 in. by 24 in.) steel plates bolted together with machined cavities to accommodate cartridge heaters. Thermocouple holes were drilled from the bottom to within 0.32 cm (1/8 in.) of the top surface to provide temperature readings on the tool surface without inserting thermocouples into the laminate. The plates were bolted around the edges to provide a 55.9 cm by 55.9 cm (22 in. by 22 in.) tool surface for lay-ups.

A trial run was made with the platen to cure a NR-150A2:B2 laminate. The platen was set on a ceramic (Transite) block and covered with heavy glass cloth for insulation. The autoclave was heated to 177°C (350°F) with the

platen simultaneously heated to 200°C (392°F). With the autoclave held at 177°C (350°F), the platen was heated to a 300°C (572°F) dwell and then to the final cure temperature of 400°C (752°F) where it was held for 2 hours under 1379 kPa (200 psi) autoclave pressure. This cure cycle is outlined fully in Table 4, and used on NR-150A2:B2 fiberglass pre-preg procured specifically for cure cycle development. The heaters were operated at 150 volts, and tool surface heat-up rates of 7.2°C/min. (13°F/minute) from RT to 200°C (392°F); 3.9°C/min (7°F/min) from 200°C (392°F) to 300°C (572°F); and 2.5°C/min. (4.5°F/min.) from 300° (572°F) to 400°C (752°F) were achieved. The temperature was held to 400°C ± 5.6°C (752°F ± 10°F) for the 2 hours cure. The vacuum bag was Kapton film sealed to the platen with a one-part silicone sealant, and this bagging system held full vacuum throughout the cycle. This trial successfully demonstrated the heated platen approach for high temperature autoclave processing of the NR-150 system.

Additional trial laminates were fabricated using the same cure cycle, which was a Du Pont recommended cycle, on the fiberglass prepreg. The quality of the fiberglass laminates as determined by visual examination, resin content, density, and thickness determinations was satisfactory. This same cure cycle was then used with the NR-150A2:B2/T300 graphite prepreg, and the laminates were determined by visual observation to be excessively starved and poor quality. These conditions were the result of excessive flow. This prepreg had a volatile content in the 20 - 25% range, and this was considered to be excessive. Du Pont recommended a volatile content in the 10 - 12% range, and prepreg samples were exposed to various oven drying cycles to determine a means of reducing volatile content to this level. A cycle of 15 minutes at 143°C (290°F), with each prepreg surface exposed to circulating oven air, was found to reduce the volatile content to 12%. The roll of NR-150A2:B2/T300 prepreg was sent to a local prepregger, and was processed through their heating tower at 139°C (282°F) for 15 minutes, with the prepreg additionally experiencing a five minute heat-up and cool-down as it went through the heater. This reduced the volatiles from 23.8% to 11.7% by weight. Trial laminates were fabricated using both the Lockheed dried prepreg and the reprocessed prepreg, and the results indicated that



satisfactory laminate quality was achieved. Thickness per ply for example was 34mm (13.4 mils) per ply for the laminate made with reprocessed prepreg compared to the nominal 34.8 mm (13.7 mils) per ply. Cured resin content for this laminate was 26.7% by weight which is a relatively high but acceptable fiber loading for woven fabric laminates. Visual appearance was satisfactory, and the degree of cure was verified by determination of weight loss at 288°C (550°F). After one hour the laminate lost only 0.8% weight.

NR-150A2:B2/T300 panel fabrication and testing. - These results indicated this was a satisfactory procedure for fabrication of the NR-150A2:B2/T300 precured sandwich skins and a series of test laminates for NASA. These trial runs are outlined in Table 4. A series of six laminates were fabricated for these purposes and were determined to be satisfactory in quality and appearance. These laminates are outlined and described in Table 5. These laminates all had void contents which were high compared to standards for epoxy laminates, but for the NR-150 system autoclave cured at the low end of the recommended pressure range, void contents at these levels were not unexpected. A slight warpage also occurred, probably due to a slight misalignment of the fill fibers along one edge which occurred during reprocessing. This edge was not used in any test pieces, but there still may have been some slight misalignment in the laminate. The panels could be flattened with hand pressure, so this was not considered to be a serious problem, and the laminates were determined acceptable for test use.

An additional trial honeycomb panel was fabricated using a sample of NR-150 resin coated NR-150A2:B2 fiberglass core. A ply of NR-150A2:B2 Style 181 fiberglass prepreg was cocured adjacent to the core. The precured NR-150 glass skins were sanded, solvent wiped, and brush coated with NR-150B2 resin (used since neat NR-150A2:B2 resin was not available). The bonding cycle was based on Du Pont recommendations and is given in Table 6. The results, also given in Table 6, were somewhat lower and showed more scatter than expected. The failure was in the core to cocured glass interface. Investigation revealed that the coated glass core (used in the trial to save the graphite core for test panels) had a thinner, more uneven coating than the graphite

core. An additional trial panel was fabricated using an identical procedure except for substitution of the graphite core and use of NR-15CA2:B2 graphite prepreg as the cocured inner layer. This prepreg was taken from a portion of the batch which had not been reprocessed to reduce volatiles. The results showed a definite improvement, 2282.25 kPa (331 psi) average vs. 1558.3 kPa (226 psi) average, with the same skin to core interface failure mode. Based on these results, a set of test panels were fabricated, as required to obtain the test specimens outlined in Table 2 plus an additional set of twelve 7.62 cm by 7.62 cm (3 in. by 3 in.) specimens for NASA.

Tests were then conducted in accordance with the plan outlined in Table 2. Tests were in accordance with MIL-STD-401, and the only deviation from MIL-STD-401 procedures was in sizing of the beam flexure specimens, where width limitations of the core block required less than optimum length for the "W" direction beam flexure specimens.

Two difficulties were encountered in performing these tests. The flatwise tensile loading blocks and steel plates for plate shear were bonded with FM-34 polyimide film adhesive for 288°C (550°F) tests, and all test failures occurred in the FM-34 bond rather than the test specimen. These were re-bonded using the LARC-13 adhesive in Task 2, and the tests will be discussed in the next section. The other problem was a test error in which 1/3 span loading rather than 1/4 span loading was used. (In other words, with a 15.2 cm [6 in.] span, the distance between the top loading points was 5.1 cm [2 in.] rather than the prescribed 7.6 cm [3 in.]). This did not affect ultimate strength determinations, and for modulus calculations, a revised formula was obtained from Hexcel to account for the nonstandard loading arrangement.

#### Task 2 - Graphite/Polyimide Honeycomb Core Development with NR-150B2/HM-S and PMR-15/HM-S Material Selection

The second Task was a continuation of the initial activity, with incorporation of newly developed polyimide resins and graphite fabric

reinforcements. Two additional resins were selected: NR-150B2, the more thermally stable constituent of NR-150A2:B2; and the NASA developed PMR-15, reported to have an optimum combination of processability and thermal stability. HM-S fibers, which had recently become available in weavable 3000 and 1000 tow yarns, were used in fabric form for both skin prepregs and honeycomb core webs. These material selections are discussed more fully in Section 1. This Task also evaluated a NASA developed polyimide film adhesive, LARC-13, for skin-core bonding; and this system is also discussed in Section 1. A summary of the materials used in this Task is given in Table 1.

Fabric development. - Fabrication of the HM-S woven fabrics was the first activity in this Task, and some problems were encountered by the fiber supplier, Hercules. Standard 10,000 tow HM-S is unsized, but has a heat cleaned surface which is the "S" designation. This heat cleaning operation proved difficult with the smaller tows because of breakage during the processing. Hercules was successful in providing heat cleaned 3000 tow fibers for skin and test laminate fabrication, but could not heat clean the 1000 tow yarns to be used in core fabrication. A decision was made with NASA concurrence to use the 1000 tow yarns in the untreated condition.

The weaving and prepregging operations in this Task were performed by Fiberite Corp. The 3000 tow and 1000 tow HM-S fabrics were in both cases directly comparable in weave style, weight and thickness per ply to the Thornel 300 fabrics described in the Task I Approach. A slight modification was required in the 1000 tow fabric, to the extent that a 34 warp by 28 fill count was used instead of 34 warp by 33 fill. This deviated slightly from true bi-directionality, but was equivalent in weight to the 1000 tow T300 fabric. Fiberite reported considerably more difficulty in the weaving operation, however, with HM-S yarns as compared to T300. Since the HM-S yarns are not sized, it was necessary to apply a PVA sizing to the yarns for the weaving operation. This sizing was subsequently removed by heat cleaning for one hour at 371°C (700°F). The prepregging operation at Fiberite, for the prepreg to be used in skin fabrication, was accomplished without

difficulty with both PMR-15 and NR-150B2 resins except that Fiberite reported wetting difficulties with the untreated HM fibers, which were resolved. Hexcel impregnated the 1000 tow yarns with the resins and fabricated the two graphite honeycomb core samples with relatively few difficulties, except that Hexcel lacked autoclave capabilities for the 288°C (550°F) cross-linking reaction of PMR-15 which requires positive pressure. Lockheed therefore received staged core from Hexcel imidized at 204°C (400°F), and processed the core at 288°C (550°F) in the autoclave to complete the cure. The core was restrained but not bagged during this operation, as the purpose was to provide ambient pressure greater than the vapor pressure of volatile constituents produced as intermediate reaction products. The core was then returned to Hexcel for slicing. The NR-150B2 and PMR-15 graphite core were both 96.1 kg/m<sup>3</sup> (6.0 lb/cu ft) density with 0.95 cm (3/8) in. cell size. The NR-150B2 core was coated with a cell edge adhesive coating of NR-150B2 resin, in the same manner as the NR-150A2:B2 core in Task I. However, the PMR-15 was left uncoated since the use of the LARC-13 adhesive was planned for skin-core bonding. LARC-13 adhesive was obtained as a film supported on 112 glass scrim at a weight of 0.293 kg/m<sup>2</sup> (0.06 lb/sq ft).

The next activity was processing development with the two polyimide prepreg systems, and considerably greater difficulties were encountered than anticipated in optimizing cure procedures with these systems.

PMR-15 process development. - The initial trials on PMR-15 utilized PMR-15/fiberglass prepreg with recommended cure cycles obtained from the literature. These processing trials are outlined in Table 7. These laminates appeared to have excessive voids, and a modified cycle was used on the PMR-15/graphite prepreg. This provided a laminate with satisfactory appearance, low voids, and acceptable resin content. Photomicrographs revealed however, a regular pattern of transverse cracks in the resin extending across each fabric layer. (Fig. 6.) The cracks were not continuous across the laminates, and did not involve any fiber breakage. These cracks were thought to be the result of thermal stresses in the resin. PMR-15 is a relatively brittle resin, and undergoes very high processing

temperatures with a maximum 343°C (650°F) post-cure temperature. HM-S fiber is a relatively brittle fiber with low strain-to-failure, and has a more highly negative thermal coefficient of expansion than other graphite fibers such as Thornel 300. The NR-150B2 resin, which is much less brittle than PMR-15, showed the transverse cracks but only to a slight extent. It was also observed that a PMR-15/HM-S laminate which had been imidized at 204°C (400°F) but not fully cured exhibited no cracks.

The approach taken to resolve this problem was to control cool-down rates, both in the autoclave cure and oven post-cure, to 0.56°C (1°F/minute) by means of cam controls. In addition, the post-cure cycle was modified to limit the maximum temperature to 316°C (600°F) and to control heat-up rates. A stepped heat rise cycle was also used. None of these procedures was successful in eliminating the transverse cracks, and it was finally concluded that this represented an inherent incompatibility with this particular resin-fiber combination. A decision was made to proceed with skin and test laminate fabrication and testing with this system in order to evaluate the honeycomb properties and obtain comparative data on the PMR-15 resin. A summary of the PMR-15 skins and laminates is given in Table 8.

Because of the less than optimum quality of the PMR-15/graphite laminates, a decision was made to use PMR-15/fiberglass skins for those panel specimens where skin quality could affect test results. Since the primary purpose of these tests was to evaluate the graphite core properties this approach was considered an acceptable means to ensure test failures in the core. Fiberglass skins were used for short-beam flexure, flatwise tensile, and plate shear tests; but graphite skins were retained for the flatwise compression specimens.

NR-150B2 process development. - An initial trial NR-150B2/fiberglass laminate was fabricated using a Du Pont recommended cycle and appeared to be completely satisfactory. (See Table 9 for NR-150B2 processing trials.) The approach used in Task 2 processing of the NR-150 system differed from the Task 1 approach with NR-150A2:B2 in that autoclave cure temperatures were

held below the 316°C (600°F) maximum autoclave temperature so that no heated platen was required. This was based on recent Du Pont work indicating that the polymerization reaction of NR-150B2 was essentially complete at 204°C (400°F), and that higher temperatures were required only for removal of the residual n-methyl pyrrolidone (NMP) solvent and condensation products (Ref. 4). This could be accomplished in an oven under vacuum or contact pressure. This is a more practical approach for eventual part fabrication, since supplemental heating for complex shaped parts would involve tooling costs approaching matched die tools. Based on these considerations, a decision was made to process the NR-150B2 under autoclave conditions without supplemental heaters.

The initial cure cycle successfully used on the NR-150B2/fiberglass was tried on the NR-150B2/HM-S graphite prepreg, and a poor quality laminate with high porosity was obtained. A large number of cure cycle variations, described in Table 9, were tried to eliminate voids, but these were not successful. Further trials and developments were finally beyond the resources of the program, and a decision was made to fabricate and test the honeycomb panels in order to evaluate graphite core properties and obtain comparative data. This decision was influenced by the fact that the NR-150B2 prepreg used in this Task used a mixture of 3 parts ethanol to 1 part NMP. Subsequent NR-150B2 development work in other NASA programs concluded that 100% NMP was a preferable solvent system (Ref. 5). This work determined that the ethanol contributes to the void problem by reacting with one of the NR-150 components in such a manner that processing characteristics are altered. The inability to eliminate voids may also have been the results of NR-150B2 batch variables, as batch variability has been a recurring problem with this relatively new system. Another possibility is that residual PVA sizing, not fully removed from the HM-S fabric during cleaning, caused the void problem, although voids were not a problem with this same fabric combined with PMR-15. In any case, the problem did not appear responsive to cure cycle variations.

A decision was made to use NR-150B2/fiberglass skins for those honeycomb specimens where results could be affected by skin quality. This was the same approach used with the PMR-15 panels described previously; and the decision was based on the same considerations: namely the nonoptimum quality of the NR-150B2 graphite laminates, and the fact that the primary purpose of the test is evaluation of the honeycomb core and the LARC-13 adhesive rather than the laminates. Fiberglass skins were used for short beam flexure, flatwise tensile and plate shear tests; and graphite skins were used for the flatwise compression specimens. A mutual agreement was reached with NASA to delete a set of NR-150B2/graphite laminates to be supplied to NASA for tests in view of the void content problems.

LARC-13 bonding development. - The cure cycles used in fabrication of the PMR-15/HM-S and NR-150B2/HM-S test laminates and sandwich skins are indicated in Tables 7 and 9. A trial sandwich panel was fabricated with the LARC-13 as the skin-core adhesive, with precured PMR-15 glass skins and a sample of fiberglass/polyimide core. The cure cycle and flatwise tensile results are given in Table 10 and a specimen after failure is shown in Figure 7. The results compare favorably with typical epoxy flatwise tensile results, so that even with the loading block failures, these results provided a confirmation of LARC-13's acceptability as a skin-core adhesive. The bonding cycle given in Table 10 was used for all test panel fabrication with the PMR-15 skins. The NR-150B2 core as mentioned had a cell edge coating of unstaged NR-150B2 resin. The LARC-13 proved to be compatible with the NR-150B2, with a modified cycle which is also shown in Table 10.

This cycle, a compromise between recommended LARC-13 and NR-150B2 cycles, was used on two trial panels with NR-150B2/fiberglass skins and the coated NR-150B2/graphite core; one with LARC-13; and one with a cocured inner layer of NR-150B2/fiberglass pre-preg (Table 10). The results were satisfactory in both cases, but since the panels with LARC-13 showed less scatter it was decided to use LARC-13 on the NR-150B2/graphite honeycomb test panels. A subsequent trial indicated the simpler PMR-15 bond cycle with increased cure time could be used for the NR-150 panels.

Testing. - Tests were conducted on the honeycomb panels in accordance with the test plan outlined in Table 2, and as discussed in the Task 1 Approach. Some of the PMR-15 beam flexure specimens were run with an improper loading arrangement (3.8 cm [1.5 in.] instead of 7.6 cm [3 in.] between the top loading points with the 15.2 cm [6 in.] span), but this did not affect ultimate strength results. Otherwise no test problems were encountered. LARC-13 was used for plate and loading block bonds on specimens to be tested at 288°C (550°F). The standard LARC-13 cure cycle described in Table 10 was used, and the LARC-13 proved satisfactory for this purpose with most failures at 288°C (550°F) occurring in the test panel. As mentioned, some NR-150A2:B2 288°C (550°F) plate shear and flatwise tensile specimens were rebonded with LARC-13 and retested.



## DISCUSSION OF RESULTS

One of the most significant results of this program was the success achieved by Hexcel in fabrication of graphite honeycomb core incorporating the NR-150A2:B2, NR-150B2, and PMR-15 resins. These resins, or modifications of these resins, are the principal candidates for high temperature composite systems; and their adaptability to the expansion process of core fabrication provides the option of honeycomb sandwich design for high temperature components in advanced vehicles. The concept of using light weight graphite bi-directional fabric reinforcements, oriented  $\pm 45^\circ$  in the core webs, was verified. Figure 1 shows the theoretical range of specific core shear strength, and the values obtained in this program with the T300 core are seen to fall slightly below the predicted range, but above aluminum core value. The HM-S cores, which proved to have less optimum properties, were slightly below aluminum values for comparable density.

The difficulties encountered with the F-178/T300 system appear to be the result of a compatibility problem with F-178 and the epoxy sizing used on Thornel 300 fibers. Hexcel's data, outlined in Table 3, showed some improvements after resin modification, but retention of mechanical properties at elevated temperature was inadequate for purposes of this program.

### NR-150A2:B2/T300 Results

The test results with the NR-150A2:B2 honeycomb panels (Table 11) indicated that the graphite/polyimide core provides significantly higher properties in compression and shear to aluminum core of the same density. Flatwise compression results at room temperature averaged 6267.6 kPa (909 psi) for 88.1 kg/m<sup>3</sup> (5.5 lb/cu ft) density graphite core compared to a reported 4757.55 kPa (690 psi) average for 83.3 kg/m<sup>3</sup> (5.2 lb/cu ft) aluminum core.

Flatwise compression specimens provided the best comparison of core properties at room temperature and 288°C (550°F). The failure mode was identical at both temperatures and was cell wall buckling. Retention of RT flatwise compression properties at 288°C (550°F) averaged 51%, indicating a significant effect of this temperature. The 3226.9 kPa (468 psi) obtained at 288°C (550°F) is a respectable crushing strength, and compares favorably with minimum crushing strength at RT of aluminum core of approximately the same density, 865 kg/m<sup>3</sup> (5.4 pcf), which is 3447.5 kPa (500 psi). (Aluminum core values are from Hexcel data). Flatwise compression is particularly useful for obtaining comparative data on core at these temperatures because it eliminates the bond line and the skin as factors in the failure load.

Several of the "L" (parallel to core ribbon) and "W" (90° to core ribbon) short-beam shear specimens at room temperature failed prematurely with a skin-core disbond at one end of the specimen due to cleavage failure. Specimens which did not fail with a disbond had shear strength values significantly superior to the disbanded specimens, and these values were comparable to shear values obtained with aluminum core of the same density. (See Fig. 1.) The cause of this problem appears to be the excessive stiffness of the 0.15 cm (0.060 in.) graphite skins at room temperature. As the specimen was deflected, the skins remained stiff and pulled away from the core producing the cleavage failure. In the 288°C (550°F) tests the skins became less stiff and were able to deflect with the panel, and proper core shear failures were obtained in all specimens. This problem was not encountered with the PMR-15 or NR-150B2 panels in Task 2, as thinner glass skins were used.

The short-beam flexure specimens at 288°C (550°F) had retentions of RT strength (based on the RT specimens which showed no debond) as follows: "L" unaged - 87%; "L" aged - 67%; "W" unaged - 87%; "W" aged - 44%. These percentages may be high, since the RT specimens which had no visible disbonds showed higher than expected deflections at about the same range as the visually disbanded specimens. This may indicate an adhesive failure which

could not be detected visually. The ultimate strength may therefore have still been below ultimate core strength at room temperature. The figures do point out, however, the greater reduction of "W" core shear properties after aging than "L" shear. The "W" short-beam flexure test applied load to the node bonds. The NR-150A2:B core used a Skybond type condensation polyimide as the node bond adhesive, which apparently lost more of its strength after aging at 288°C (550°F) than the NR-150 resin.

Short-beam flexure yield strengths are given for 288°C (550°F) tests. The "W" specimens all had yield points significantly below ultimate, as did the aged "L" specimens. The unaged "L" specimens had a yield point closer to the ultimate. Percentages of ultimate at the yield are as follows: "L" unaged - 85%; "L" aged - 64%; "W" unaged - 70%; "W" aged - 11%. This indicates 288°C (550°F) may have been close to the glass transition temperature (T<sub>g</sub>) of the core webs, which was expected to be around 302°C - 316°C (575°F - 600°F). Actual T<sub>g</sub>'s were determined to be 300°C (572°F) as received and 315°C (599°F) after 500 hours aging at 288°C (550°F). A tentative conclusion is that 288°C (550°F) is a marginal use temperature for NR-150A2:B2. T<sub>g</sub>'s were obtained by the penetration method which shows better correlation with mechanical test results than other methods.

Difficulties were encountered in the plate shear and flatwise tensile specimens due to test failures at 288°C (550°F) of the steel plates and aluminum loading blocks, which were bonded to the panels with FM-34 adhesive. These specimens were retained and subsequently rebonded to the plates and loading blocks with LARC-13. These results are shown in Table 12. The 288°C (550°F) plate shear tests, intended as core tests, produced skin-core failures with unaged 288°C (550°F) values retaining 76% of RT values. Upon retest, only one flatwise tensile specimen at 288°C (550°F) unaged failed properly in the bond-line at 27% of RT values. The flatwise tensile value is likely a more accurate indication of the effects of this temperature on the NR-150A2:B2 resin functioning as an adhesive, and indicates again that 288°C (550°F) is a marginal use temperature for NR-150A2:B2.

The RT plate shear results indicate that the NR-150A2:B2 used as a skin-core adhesive may not be providing adequate shear capability even at room temperature. The specimens failed at the skin-core bond line at an extremely low value, and provided no test results which can be related to the core. Initially, a trial specimen was made bonding the steel plates directly to the core. An acceptable bond could not be obtained, so a NR-150A2:B2 fiberglass skin (3 plies, 0.096 cm [0.038 in.] thick) was bonded to the core, using the same bonding procedures used for the graphite panels. (Cell edge adhesive, plus wet prepreg layer, plus resin coated onto the selected skin). This was the adhesive layer that provided the premature failure. The steel plates were bonded to the fiberglass skins using a room temperature curing epoxy for the room temperature test specimens and this bond did not fail.

The RT flatwise tensile results on the other hand indicate an acceptable skin-core bond was achieved for this type of loading, and this value compares favorably with flatwise tensile results obtained on conventional metal sandwich. Flatwise tensile results, while indicative of the quality of the adhesive fillet around the cell walls, are not necessarily representative of adhesive capability under other loading conditions such as shear or peel. The three room temperature specimens were bonded to the loading blocks using a room temperature curing epoxy to ensure against a loading block failure.

#### PMR-15/HM-S Test Results

Room temperature properties obtained with the PMR-15/HM-S graphite panels were quite variable in comparison to the NR-150A2:B2 and NR-150B2 panels (Table 13) and the results reflected adverse effects of the thermal cracking problem discussed previously: a relatively poor fiber-resin interface bond, and the low strength and brittle characteristics of HM-S graphite as compared to Thornel 300. The sharply lower RT flatwise compression compared to the NR-150A2:B2/T300 core probably is the result of the lower strength HM-S fiber characteristics. The values are significantly less than flatwise compression values of comparable density aluminum core,

and panels with HM-S core could be marginal or inadequate under impact and crushing loads. The PMR-15/graphite core shear strength in the "L" direction at RT, however, compared reasonably well to the other graphite cores and to comparable density aluminum core. The RT "W" core shear values are somewhat lower than the other cores which may reflect a poorer quality node bond.

Short-beam flexure and flatwise compression values tested at 288°C (550°F) without heat aging retained an acceptable percent of RT values (68.6% retention in "L" short-beam flexure, 74.4% retention in "W" short-beam flexure, and 80.7% retention in flatwise compression). A drastic drop-off occurs in these properties when tested at 288°C (550°F) after 500 hours aging at 288°C (550°F) with retentions of only 8.2% and 19.8% in "L" and "W" short-beam shear respectively. The flatwise tensile specimens aged at 288°C (550°F) disbanded during handling and could not be tested. These results strongly indicate that 288°C (550°C) is beyond the capabilities of PMR-15 resin except for very short-term applications.

Flatwise tensile results on the PMR-15 panels reflect the behavior of the LARC-13 adhesive and will be discussed in that section.

Modulus values were calculated from the short-beam flexure data using MIL-STD-401 formulas but no reasonable values could be obtained. Core modulus determinations from sandwich beam flexure are extremely sensitive to slight errors in strain measurement, and plate shear is generally recommended for core shear modulus determination. Unfortunately, Lockheed did not have a proper extensometer set-up for plate shear strain measurements and development of this capability was beyond the scope of the program. Thus, only plate shear strength was obtained, and the core shear strengths obtained were somewhat lower than obtained in beam flexure. This reduction is probably caused by the partial skin-core disbond noted in the failed specimens.

## NR-150B2/HM-S Test Results

The NR-150B2/HM-S core had comparable room temperature beam flexure and flatwise compression properties as the PMR-15/HM-S core. (See Table 14 for NR-150B2 test results.) The ultimate RT core shear strength as determined by "L" beam flexure and by plate shear was somewhat lower than the NR-150A2:B2/T300 core and comparable density aluminum core, reflecting the lower strength characteristics of the HM-S fiber. The NR-150B2/HM-S core like the PMR-15/HM-S core, had RT flatwise compression values significantly less than the NR-150A2:B2/T300 core and aluminum core of comparable density. As previously discussed, these low values are attributed to the low strength brittle characteristics of HM-S graphite as compared to Thornel 300, and would indicate a serious structural limitation for graphite core made with this fiber. (Ref. Table 14)

Retention of short-beam flexure and flatwise compression properties at 288°C (550°F) without aging was comparable or slightly superior to the PMR-15 core and the NR-150A2:B2 cores (84%, 75%, and 85% for "L" beam flexure, "W" beam flexure, and flatwise compression respectively). The NR-150A2:B2 core had less retention of flatwise compression at 288°C (550°F) than the other two cores but the absolute 288°C (550°F) flatwise compression value of the NR-150A2:B2 core was still substantially higher than 288°C (550°F) flatwise compression of the cores incorporating HM-S fiber.

After 500 hours aging at 288°C (550°F), the NR-150B2 core had excellent retention of RT properties (80% and 72% for "L" and "W" short-beam shear respectively), comparable to 288°C (550°F) retentions of the aged NR-150A2:B2/T300 core and greatly superior to 288°C (550°F) retention of the aged PMR-15 core. Considering the indications that the NR-150B2/HM-S system used may have been less than optimum with batch variability problems and poor resin-fiber interface, these results are strong indications that the NR-150B2 system has excellent characteristics for long-term 288°C (550°F) service.

Flatwise tensile results are dependent on characteristics of the LARC-13 polyimide adhesive, and are discussed in the next section.

#### LARC-13 Test Results

The trial panels bonded with the LARC-13 film adhesive at  $9.29 \text{ kg/m}^2$  [0.06 lb/sq ft], supported on 112 glass scrim, gave acceptable flatwise tensile test results which compared favorably with typical epoxy flatwise tensile values (Table 10). When the LARC-13 was used to bond skins on the NR-150B2 core coated with an unstaged cell edge layer of NR-150B2 resin, increased flatwise tensile values were obtained (2899.3 kPa [420.5 psi] vs. 1999.55 kPa [290 psi]) for values on uncoated PMR-15 core. This demonstrated that LARC-13 could be effectively cured with NR-150B-2.

LARC-13 was also used to rebond aluminum loading blocks and steel plates onto NR-150A2:B2 flatwise tensile and plate shear specimens which had failed at 288°C (550°F) in the loading blocks and plates. These had been bonded with FM-34 condensation polyimide adhesive. Loading block failures still occurred on two out of three 288°C (550°F) flatwise tensiles (Table 12), but the plate shear-tests at 288°C (550°F) all failed in the specimen (Fig. 8). These specimens indicated that LARC-13 may be adequate but somewhat marginal for short-term 288°C (550°F) exposures.

The flatwise tensile results obtained on the PMR-15 and NR-150B2 honeycomb panel specimens (Tables 13 and 14) showed excellent adhesion at room temperature. At 288°C (550°F) the unaged NR-150B2 specimens fell off drastically in strength with two loading block failures out of the three specimens. (Loading blocks were also bonded to the specimens with LARC-13). However, the PMR-15 flatwise tensiles, with LARC-13 bonded loading blocks, retained a reasonably good 53% of RT values. A common difficulty with flatwise tensile testing is inadvertent introduction of eccentric loads which produce peel failures on the specimen. This may account for the failures of LARC-13 aluminum loading block bond at 288°C (550°F) on one set of

specimens but not the other. LARC-13 obviously is lacking in peel strength at 550°F. After 500 hours aging at 288°C (550°F), the NR-150B2 specimens disbonded during handling and the PMR-15 specimens retained less than 3% of RT values, indicating LARC-13 has no aging capability at 288°C (550°F).

In summary, LARC-13 appears to have excellent adhesion characteristics for skin-core bonding, but 288°C (550°F) is beyond its useful temperature range, except for short-term applications.



## CONCLUSIONS

The demonstration by Hexcel of the feasibility of producing honeycomb core with light weight graphite fabrics and polyimide resins was the most significant result of this program. This product is now an available structural material for high temperature applications with two of the most promising polyimide systems, NR-150B2 and PMR-15 as matrix materials. The core shear test results also confirmed Hexcel's predictions; and verified that graphite honeycomb core, with continuous fiber reinforcements oriented  $\pm 45^\circ$  to the web, provides specific shear properties comparable or superior to metallic honeycomb. This is the first nonmetallic honeycomb to approach metal cores in shear properties, and graphite core can be used with composite skins in structural applications where metallic core would create serious corrosion and thermal mismatch problems.

Of the three polyimide systems evaluated in the program, PMR-15 appears to be marginal for short-term 288°C (550°F) use, and is not acceptable for long-term 288°C (550°F) applications. NR-150B2 appears to have excellent 288°C (550°F) capabilities, while the NR-150A2:B2 is acceptable for 288°C (550°F) but has less thermal stability than the NR-150B2.

The epoxy sized Thornel 300 fibers reinforcements used in the first task appear to be acceptable for high temperature applications when combined with suitable polyimide resins, despite the compatibility problems encountered with F-178. Neither the fiber or the sizing appeared to adversely affect elevated temperature properties with the NR-150A2:B2 system.

The higher modulus HM-S fibers also appear to have acceptable thermal stability combined with the PMR-15 and NR-150B2 systems. Both HM-S and Thornel 300 fibers provided core shear properties slightly less but reasonably close to predicted values. However the sharply reduced flatwise compression values of the HM-S graphite core indicates that high modulus graphites would not provide sufficient crushing strength to the panel to be

practical for structural use. Thornel 300 and similar intermediate modulus fibers, despite theoretically lower thermal stability, appear to be the best selection for graphite core reinforcement.

Problems encountered with skin laminate quality of PMR-15/HM-S indicate that thermal mismatch is a factor that needs to be carefully considered in combining a brittle, high temperature curing polyimide with high modulus graphites. The problems with the NR-150B2 are indicative of batch variability and/or storage stability problems. These problems may have been related to the use of ethanol in the solvent mixture, which has now been discontinued.

This program represented one of the first uses of woven high modulus HM-S fibers. Despite heat cleaning and weaving difficulties this appears to be a promising product. There is a possibility that residual PVA sizing contributed to the problems with the cured laminates, and application and removal of sizing is an area requiring further studies with these new fabrics.

LARC-13 polyimide adhesive appears to provide satisfactory adhesive characteristics, with excellent processing characteristics, and the system appears well adapted for skin-honeycomb core bonding, and for cocuring with other polyimide systems. 288°C (550°F) appears to be a marginal use temperature, however, even for short-term applications.

## REFERENCES

1. Gibbs, Hugh H.: Latest Developments in NR-150 Polyimide Binders. Paper presented at 19th National SAMPE Symposium, 1974.
2. Serafini, T. T. and Vanucci, R. D.: Tailor Making High Performance Graphite Fiber Reinforced PMR Polyimides. NASA TMX-71616, 1975.
3. McMahon, Paul E.: Oxidative Resistance of Carbon Fibers and Their Composites, Celanese Research Co., 1977.
4. Gibbs, Hugh H.: Status Report NR-150 Polyimide Binders and Adhesives. Paper presented at 21st National SAMPE Symposium, April 1976.
5. Develop and Demonstrate Manufacturing Processes for Fabricating Graphite Filament Reinforced Polyimide (Gr/PI) Composite Structural Elements. Quarterly Report No. 1, March 8 - June 8, 1977, CASD-NAS-776-318-1, General Dynamics, Convair Div.

TABLE 1 - SUMMARY OF MATERIALS


Resin	Reinforcement Form	Prepreg	Core Description	Adhesive
<p>- F-178  (Hexcel Corp.)</p> <p>- NR-150A2:B2 (Du Pont)</p>	<p>Thornel 300 yarns (Union Carbide), 3000 filaments per tow, sized with 309 epoxy, woven into Style 1133 fabric - 24 x 23 8 harness satin weave, 0.33 mm, (13 mils)/ply nominal (woven by Hexcel)</p>	<p>NR-150A2:B2/T300 Style 1133 fabric prepreg used for skins and test laminates. 34% resin content by weight. 23.8% vols., reprocessed prior to laminate fab. to 11.8% (Impregnated by Hexcel)</p>	<p>—</p>	<p>NR-150A2:B2 resin, unstaged, applied to core as cell edge adhesive (Hexcel proprietary process)</p>
<p>- PMR-15 (NASA dev. system)</p> <p>-NR-150B2 (Du Pont)</p>	<p>Thornel 300 yarns (Union Carbide), 1000 filaments per tow, sized with 309 epoxy, woven into Style 1136 fabric - 34 x 33 satin weave, 0.127 mm, (5 mils)/ply nominal (woven by Hexcel)</p>	<p>—</p>	<p>0.95 cm (3/8 in.) cells 88.1 kg/m<sup>3</sup> (5.5 lb/cu ft) density</p>	<p>NR-150B2 resin unstaged, applied as cell edge adhesive NR-150B2 core only.</p>
<p>- PMR-15 (NASA dev. system)</p> <p>-NR-150B2 (Du Pont)</p>	<p>HM-S yarns (Hercules), 3000 filaments/tow, surface heat cleaned, PVA sizing applied, woven into 24 x 23 8 harness satin weave 0.33 mm (13 mils)/ply nominal, PVA sizing removed by heat cleaning, (woven by Fiberite).</p>	<p>- PMR-15/HM-S, 0.33 mm (13 mil) fabric prepreg: 36.6% resin content by wt. 12.0% vols, 21.6% flow at 100 psi.</p>	<p>—</p>	<p>NR-150B2 resin unstaged, applied as cell edge adhesive NR-150B2 core only.</p>

TABLE 1 - SUMMARY OF MATERIALS (Concluded)

Resin	Reinforcement Form	Prepreg	Core Description	Adhesive
<p>- PMR-15 (NASA dev. system)</p> <p>-NR-150B2 (Du Pont) (Cont'd)</p>	<p>HM-S yarns (Hercules), 3000 filaments/tow, surface heat cleaned, PVA siz- ing applied, woven into 24 x 23 8 harness satin weave 0.33 mm (13 mils)/ ply nominal, PVA sizing removed by heat cleaning, (woven by Fiberite).</p>	<p>- NR-150B2/HM-S 0.33 mm (13 mil) fabric prepreg: 36% resin content by wt.</p>	<p>---</p>	<p>LARC-13 sup- ported film adhesive, 0.293 kg/m<sup>2</sup> (0.06 lb/sq ft) on 112 glass resin used for bond- skins to both cores.</p>
	<p>HM yarns (Hercules), 1000 filaments/tow, surface untreated, PVA sizing applied, woven into 34 x 28 satin weave, 0.127 mm (5 mils)/ cured ply nominal PVA sizing removal by heat cleaning (woven by Fiberite)</p>	<p>---</p>	<p>0.95 cm (3/8 in.) cells, 96.1 kg/m<sup>3</sup> (6.0 lb/cu ft) density - (for both PMR-15 and NR-150B2 honey- comb cores).</p>	<p>---</p>

△ F-178 deleted from program, and prepreg and core were never received with this material.

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 2 - TEST OUTLINE

Test	MIL-STD-401 Test Method Para. No.	Specimen Dimensions	Test Temperature $\Delta$	Aging	No. of Specimens			
					F178 Panels	MR-150A2/B2 (50:50) Panels	FRB-15 Panels	MR-150B2 Panels
Short-beam shear strength "L" direction	5.2.4	17.8 cm L x 5.08 cm W x 1.27 cm (7 in. L x 2 in. W x 0.500 in.)	RT	-	3	3	3	3
			ET	-	3	3	3	
			ET	After 500 hrs. at ET	3	3	3	3
Short-beam shear strength "W" direction	5.2.4	17.8 cm W x 5.08 cm L x 1.27 cm (7 in. W x 2 in. L x 0.500 in.)	RT	-	3	3	3	3
			ET	-	3	3	3	
			ET	After 500 hrs. at ET	3	3	3	3
Flatwise Compression	5.2.1(a)	7.62 cm x 7.62 cm x 1.27 cm (3 in. x 3 in. x 0.500 in.)	RT	-	3	3	3	
			ET	-	3	3	3	
Flatwise Tensile	5.2.3	5.08 cm x 5.08 cm x 1.27 cm (2 in. x 2 in. x 0.500 in.) Loading blocks bonded to panel	RT	-	3	3	3	
			ET	-	3	3	3	
			ET	After 500 hrs. at ET	3	3	3	
Plate shear strength and modulus "L" direction	5.1.5	15.24 cm x 5.08 cm x 1.27 cm (6 in. L x 2 in. W x 0.500 in.) - Loading blocks bonded to core panel	RT	-	3	3	3	
			ET	-	3	3	3	

$\Delta$  ET is 288°C (550°F).  
L = core ribbon direction  
W = 90° to core ribbon direction

TABLE 3 - TEST OUTLINE - OPTIMIZATION OF GRAPHITE/POLYIMIDE LAMINATES

Resin	Thornel 300 $\Delta$ Fiber Batch	Reinforcement		Processing Variables	Flexure Str. Results MPa (Ksi.)	
		Un. Tape	Woven Fabric		RT	260°C (500°F)
F-178	Lot 65-2 Pkg. A588 3k filaments/tow (batch used for skins and test laminates)	X		No post-cure. 177°C (350°F) cure only	1894.1 (274.7)	--
				Post-cure 8 hr at 260°C (500°F)	839.8 (121.8)	--
				No post-cure	621.9 (90.2)	--
			X	Post-cure 8 hr at 260°C (500°F)	329.6 (47.8)	--
				Post-cure 8 hr at 260°C (500°F). T300 treated with MEK 2 hr at 49°C (120°F) prior to impregnation	468.9 (68)	--
			X	Post-cure 8 hr at 260°C (500°F)	641.2 (93)	--
			X	Post-cure 3 hr at 232°C (450°F), after step-wise heat-up to 232°C (450°F)	1965.1 (285)	594.3 (86.2) 744.7 (108.0) AVG. = 669.5 (97.1)
			X	Post-cure 3 hr at 232°C (450°F), after step-wise heat-up to 232°C (450°F)	1903.0 (276)	573.7 (83.2) 653.6 (94.8) 635.7 (92.2) AVG. = 621.2 (90.1)
				Post-cure 8 hr at 260°C (500°F) after step-wise heat-up to 260°C (500°F)	504.0 (73.1)	--
				Post-cure 8 hr at 232°C (450°F) No step-wise heat-up	537.1 (77.9)	--

ORIGINAL PAGE IS  
OF POOR QUALITY

ORIGINAL PAGE IS  
 OF POOR QUALITY

TABLE 3 - TEST OUTLINE - OPTIMIZATION OF GRAPHITE/POLYIMIDE LAMINATES (Concluded)

Resin	Thornel 300 $\Delta$ Fiber Batch	Reinforcement		Processing Variables	Flexure Str. Results MPa (Ksi.)	
		Uni Tape	Woven Fabric		RT	260°C (500°F)
F178	Lot 055-21 1K filament/ tow (for use in cure webs)	X		Post-cure 3 hr at 232°C (450°F) after step-wise heat-up to 232°C (450°F)	1675.5 (243)	390.9 (56.7) 382.0 (55.4) Avg. = 386.8 (56.1)
			X	Post-cure 3 hr at 232°C (450°F) after step-wise heat-up to 232°C (450°F)	395.1 (57.3)	65.5 (9.5) 107.6 (15.6) -avg. = 86.9 (12.6)
HX-588 (modified F-178)	Lot 65-2 3K filament/ tow (current batch)		X	Post-cure 3 hr at 232°C (450°F) after step- wise heat-up to 232°C (450°F)	211.0 (30.6)	69.6 (10.1) 73.1 (10.6) Avg. = 71.7 (10.4)
			X	Post-cure 3 hr at 232°C (450°F) after step-wise heat-up to (450°F)	344.1 (49.9)	25.5 (3.7) 20.0 (2.9) Avg. = 22.8 (3.3)
Second Modification of F-178	Current batch 3K filament/ tow (three separate specimens)		X	No post-cure	635.7 (92.2) 655.0 (95) Avg. = 645.4 (93.6)	--
				Post-cure at 260°C (500°F) after step- wise heat-up to 260°C (500°F)	758.5 (110) 724.0 (105) 774.3 (112.3) Avg. = 752.2 (109.1)	173.1 (25.1)
NR-150A2/B2	Lot 65-2 3K filament/ tow (current batch)		X	Cured at 399°C (750°F) for 2 hr at 2758 kPa (400 psi). No post-cure No MEK extraction	546.8 (79.3)	321.3 (46.6)
				Same cure after MEK extraction	481.2 (69.8)	230.3 (33.4)

$\Delta$  The current batch of Thornel 300 consists of several packages of Lot 65-2. After weaving, individual packages cannot be differentiated



TABLE 4 - NR-150A2:B2 PROCESSING DEVELOPMENT


Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties				
				Resin Content % by Wt.	Density g/cc.	Voids % (calc.)	Thickness Per Ply (mils)	Tg °C (°F)
NR-150A2:B2 Fiberglass	10 ply 0° 35.6 cm by 30.5 cm (14 in. by 12 in.) 1.8 mm (0.070 in.) thick	Initial trial to verify platen. Vendor recommended cure.	<ol style="list-style-type: none"> <li>1) Apply full vacuum, autoclave heat to 177°C (350°F) at 5.6-8.3°C (10-15°F)/minute.</li> <li>2) Heat platen to 200°C (392°F) - 35 minutes required.</li> <li>3) Dwell at 200°C (392°F) for 30 minutes. Apply 344.75 kPa (50 psi), maintain vacuum.</li> <li>4) Heat platen to 300°C (572°F) (25 min. req'd). Dwell 30 min.</li> <li>5) Heat platen to 400°C (752°F) for 120 min. Apply 1379 kPa (200 psi).</li> <li>6) Cure 120 minutes at 1379 kPa (200 psi).</li> <li>7) Cool to 82°C (180°F) under pressure.</li> </ol>	--	--	--	--	Laminate visually observed to be resin starved
NR-150A2:B2 1300 Graphite 34% resin content by wt., 23.6% voids.	5 ply 0° 15.24 cm by 15.24 cm (6 in. by 6 in.)	Partial vacuum to reduce flow	<p>Same cycle as above, except 254-381 mm Hg (5-10 in. Hg) held through dwell at 200°C (392°F), then full vacuum and 344.75 kPa (50 psi) applied.</p> <ol style="list-style-type: none"> <li>1) Apply full vacuum heat autoclave to 177°C (350°F) at 5.6-8.3°C (10-15°F)/min.</li> <li>2) heat platen to 200°C (392°F). Dwell 30 min.</li> <li>3) Apply 344.75 kPa (50 psi) maintain vacuum.</li> <li>4) Dwell 60 min. additional at 200°C (392°F)</li> <li>5) Heat platen to 300°C (572°F). Dwell 30 min.</li> <li>6) Heat platen to 350°C (662°F). Dwell 30 min.</li> </ol>	--	--	--	--	Laminate visually observed to be resin starved.
		Same as 1st cycle, but 662°F dwell added, 392°F dwell extended. DESIGNATED AS STANDARD NR-150A2:B2 CYCLE 		--	--	--	--	Laminate visually observed to be resin starved.

TABLE 4 - NR-150A2:B2 PROCESSING DEVELOPMENT (Concluded)

Prepreg Description	Laminate Description	Processing Variable	Cure and $\Delta$ Post-cure Cycle	Cured Laminate Properties					
				Resin Content % by wt.	Density g/cc.	Voids % (calc.)	Thickness Per Ply (mils)	Ig oc (OF)	Visual Observation
NR-150A2:B2 1300 graphite 34% resin content by wt., reprocessed to 11.7% vol.	5 ply 0° 15.24 cm by 15.24 cm (6 in. by 6 in.)	--	7) Heat platen to 400°C (752°F). Increase pressure to 1379 kPa (200 psi). 8) Cure at 400°C (752°F) 60 min. 9) Cool to 79°C (175°F) under pressure.  STANDARD NR-150A2:B2 CYCLE	26.7%	--	--	13.4	--	Satisfactory appearance. After 1 hr. at 288°C (550°F), 0.8% wt. loss.

$\Delta$  Typical heat-up times:

- RT - 200°C (392°F) 45 min.
- 200°C (392°F) - 300°C (572°F) 37 min.
- 300°C (572°F) - 350°C (662°F) 18 min.
- 350°C (662°F) - 400°C (752°F) 30 min.

$\Delta$  Bleeder:

- Teflon coated glass (1 layer) on each surface
- 1 ply 120 glass next to top surface, plus
- 161 glass bleeder on top in ratio of 1 ply
- 181 to 2 plies prepreg.

ORIGINAL PAGE IS  
OF POOR QUALITY



TABLE 5 - NR-150A2:B2/T300 PRE-CURED SKINS AND TEST LAMINATES

Panel Identification	Dimension No. plies cm (in.)	Lay-up Orientation	Thickness cm (in.)	Thickness per ply mm (mils)	Resin Content (% by wt.)	Density (g/cm <sup>3</sup> )	Fiber Vol. %	Void Vol. %	Remarks
1M2 747	15.24 x 30.5 (6 x 12) 10 plies	0°	0.332 (0.131)	0.332 (13.1)	28.5	1.5686	64.5	4.5	Slight warp. Araldite release cloth omitted leaving roughened blender pattern on one face. Kaptan film bonded to other face and was removed by sanding
1M2 752	15.24 x 30.5 (6 x 12) 10 plies	(0°, +45°, 90°, -45°, 0°) <sub>2</sub>	0.340 (0.134)	0.340 (13.4)	29.4	1.5696	63.7	4.3	Slight warp - to greater extent than 0° warp panel
1M2 757	40.6 x 35.6 (16 x 14) 5 plies	0°	0.173 (0.068)	0.346 (13.6)	--	--	--	--	Slight warp
1M2 761	40.6 x 35.6 (16 x 14) 5 plies	0°	0.168 (0.066)	0.336 (13.2)	--	--	--	--	Slight warp
1M2 764	30.5 x 30.5 (12 x 12) 5 plies	0°	0.170 (0.067)	0.335 (13.4)	28.9	1.5615	63.8	4.9	Slight warp. Some adhesion of Araldite, which was sanded off prior to bonding
1M2 766	30.5 x 30.5 (12 x 12) 5 plies	0°	0.173 (0.068)	0.346 (13.6)	27.8	1.5708	65.2	4.5	Slight warp. All 5 ply laminates can be flattened with hand pressure
1M2 769	30.5 x 30.5 (12 x 12) 5 plies	0°	0.170 (0.067)	0.340 (13.4)	27.4	1.5623	65.2	5.1	Slight warp
1M2 773	30.5 x 30.5 (12 x 12) 5 plies	(0°, +45°, 90°, +45°, 0°)	0.170 (0.067)	0.340 (13.4)	28.2	1.5580	64.3	5.2	Slight warp
1M2 842	40.6 x 35.6 (16 x 14) 5 plies	0°	0.165 (0.065)	0.330 (13.0)	--	--	--	--	Slight warp
1M2 845	40.6 x 35.6 (16 x 14) 5 plies	0°	0.173 (0.068)	0.346 (13.6)	--	--	--	--	Slight warp

TABLE 5 - NR-150A2:B2/T300 PRE-CURED SKINS AND TEST LAMINATES  $\Delta$  (Concluded)

Panel Identification	Dimension No. plies cm (in.)	Lay-up Orientation $\Delta$	Thickness cm (in.)	Thickness per ply mm (mil)	Resin Content (% by Wt.)	Density (gm <sup>3</sup> )	Fiber Vol. %	Void Vol. % $\Delta$	Remarks
2M2 845	40.6 x 17.8 (16 x 7) 5 plies	0°	0.173 (0.068)	0.346 (13.6)	--	--	--	--	Slight warp
1M2 847	40.6 x 35.6 (16 x 14) 5 plies	0°	0.173 (0.068)	0.346 (13.6)	--	--	--	--	Slight warp

$\Delta$  Bi-directional graphite fabric reinforcement. 0° refers to direction of warp fibers and is parallel to specimen length

$\Delta$  Calculated from density and resin content

$\Delta$  Panel #'s 747, 752, 764, 766, 769, 773 supplied to MSA, Panels 757, 761, 842, 845, 847 used for panel skins.

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 6 - NR-150A2:B2 HONEYCOMB PANEL BONDING DEVELOPMENT

Surface Preparation and Bonding Procedures	Cure Cycle $\Delta$	Flatwise Tensile kPa (psi)	Remarks
Pre-cured NR-150A2:B2/ fiberglass skins were hand sanded and solvent wiped. One ply NR-150A2:B2/ fiberglass prepreg cocured against core. NR-150A2:B2 glass core coated with NR-150A2:B2 resin was used.	<ol style="list-style-type: none"> <li>1) Apply full vacuum</li> <li>2) Heat to 200°C (392°F), rate not critical.</li> <li>3) Dwell at 200°C (392°F), for 30 min.</li> <li>4) Heat to 300°C (572°F), rate not critical.</li> <li>5) Apply 69 kPa (10 psi) plus full vacuum</li> <li>6) Cure 2 hours at 300°C (572°F)</li> </ol>	<ol style="list-style-type: none"> <li>1) 1303 (189)</li> <li>2) 1882 (273)</li> <li>3) 1579 (229)</li> <li>4) 1476 (214)</li> </ol> Avg. = 1558 (226)	Glass core subsequently observed to have thinner, more uneven coat than NR-150 graphite core. All skin-core failures.
Pre-cured NR-150A2:B2/ skins hand sanded and solvent wiped as above. One ply NR-150A2:B2/ graphite prepreg which had not been reprocessed to lower volatiles was cocured against core. NR-150A2:B2 graphite core coated with NR-150A2:B2 resin was used.		<ol style="list-style-type: none"> <li>1) 2137 (310)</li> <li>2) 2213 (321)</li> <li>3) 2131 (309)</li> <li>4) 2641 (383)</li> </ol> Avg. = 2282 (331)	Skin-core failure

$\Delta$  Typical heat-up times: RT - 200°C (392°F), 40 min.  
200°C (392°F) - 300°C (572°F), 35 min.

TABLE 7 - PMR-15 PROCESSING DEVELOPMENT

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (mils)	Tg °C (°F)	
PMR-15/ 181 fiberglass 30.3% resin content by wt. 10.8% volatiles	6 ply 0° 35.6 cm x 31.75 cm (14 in. x 12-1/2 in.)	Vendor recommended cycle	<p>Oven pre-stage:</p> <ol style="list-style-type: none"> <li>1) Apply 50.8 mm - 76.2 mm (2-3 in.) Hg.</li> <li>2) Heat to 204°C (400°F) at 1.7-2.8°C (3-5°F)/min.</li> <li>3) Hold 1 hr.</li> </ol> <p>Cure:</p> <ol style="list-style-type: none"> <li>1) Heat to 254°C (490°F) at 4.45°C (8°F)/min.</li> <li>2) Apply full vacuum + 1034 kPa (150 psi).</li> <li>3) Continue heat to 288°C (550°F).</li> <li>4) Hold 2 hr.</li> <li>5) Cool to 82°C (180°F) under pressure. (Bleeder 1 ply 120)</li> </ol> <p>Post-cure:</p> <ol style="list-style-type: none"> <li>1) Place between caul plates. Heat to 316°C (600°F). Hold 16 hr.</li> </ol>	22.98	2.40	Negligible	0.191 (7.5)	320 (608)	Satisfactory appearance but starved
	6 ply 0° 35.6 cm x 31.75 cm (14 in. x 12-1/2 in.)	Vendor recommended cycle	<ol style="list-style-type: none"> <li>1) Heat to 260°C (510°F) at 2.2-2.8°C (4-5°F)/min.</li> <li>2) Apply 103.4 kPa (15 psi) vacuum plus 1379 kPa (200 psi) pressure.</li> <li>3) Heat to 274°C (525°F).</li> <li>4) Hold 4 hr.</li> <li>5) Cool to 79°C (175°F) under pressure. (Bleeder - 1 ply 120)</li> </ol> <p>Post-cure:</p> <ol style="list-style-type: none"> <li>1) Heat to 316°C (600°F) at 1.7 - 2.8°C (3-5°F)/min.</li> <li>2) Hold 24 hr at 316°C (600°F).</li> </ol>	27.41	2.02	4.5	0.206 (8.1)	315 (599)	Satisfactory appearance, but excessive voids

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 7 - PMR-15 PROCESSING DEVELOPMENT (Continued)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (mils)	Tg °C (°F)	
PMR-15/ 181 fiberglass	6 ply 0° 35.6 cm x 31.75 cm (14 in. x 12-1/2 in.)	Slight modification of first vendors' cycle per recommendation. Principal variable is temperature at which full pressure is applied	1) Apply 50.8 mm - 76.2 mm (2-3 in.) Hg. 2) Heat to 204°C (400°F) at 2.2-2.8°C (4-5°F)/min. Hold 3 hrs. 3) Heat to 249°C (480°F) at 2.2-2.8°C (4-5°F)/min. 4) Apply full vac. plus 862 kPa (125 psi). 5) Heat to 288°C (550°F) at 2.2-2.8°C (4-5°F)/min. Hold 1 hr. 6) Cool to 82°C (180°F) under pressure. (Bleeder 1 ply 120)	26.5	1.92	9.6	0.208 (8.2)	331 (628)	Considerable surface porosity
PMR-15/ IM-S graphite fabric 36.6% resin content 12.0% volatiles, 21.6% flow @ 100 psi	6 ply 0° 15.2 cm x 15.2 cm (6 in. x 6 in.)	Further slight modification of first vendors' cycle, with principal variable temp. at which pressure applied. Used with graphite prepreg	Post-cure: 16 hr at 316°C (600°F) 1) Apply 50.8 mm - 76.2 mm (2-3 in.) Hg. 2) Heat to 204°C (400°F) at 1.7-2.8°C (3-5°F)/min. Dwell 60 min. 3) Heat to 251°C (490°F) at 4.45°C (8°F)/min. 4) Apply full vac. plus 1034 kPa (150 psi). 5) Continue heating to 288°C (550°F). 6) Hold 2 hr. 7) Cool to 82°C (180°F) under pressure. (No bleeder except Armalon)	27.24	1.62	0.11	0.325 (12.8)	390 (734)	Satisfactory appearance Acceptable
PMR-15/ IM-S graphite fabric	5 plies 38.1 cm x 30.4 cm (15 in. x 12 in.)	DESIGNATED AS STANDARD PMR-15 CYCLE Second trial of optimum standard cycle. Used for test laminates	Post-cure 16 hr at 316°C (600°F). Standard PMR-15 cycle. (See above)	29.57	1.602	0.17	0.340 (13.4)	397 (747)	Satisfactory appearance. Used for test laminate. Photomicrograph showed transverse resin cracks through fiber layers (Fig. 6)

TABLE 7 - PMR-15 PROCESSING DEVELOPMENT (Concluded)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cc.	Voids % (Calc)	Thickness Per Ply mm (mil)	Tg °C (°F)	
PMR-15/HM-S graphite fabric	5 ply uni. 15.2 cm x 15.2 cm (6 in. x 6 in.)	Standard cycle with controlled cure and post-cure heat-up and cool-down cycles. Modified post-cure eliminating 650°F	Cure per standard PMR-15 (see above), except cool-down of 0.6°C (1°F)/min used in autoclave cycle. Post-cure: Press, under vacuum. Heat RT to 316°C (600°F) at 0.6°C (1°F)/min. Hold 16 hr. Cool to RT at 0.6°C (1°F)/min. Cool-down rates controlled by programmed cam.						Satisfactory appearance. Photomicrographs showed transverse cracks through fiber layers. (Fig. 6)
			Cure per Standard PMR-15 cycle: (see above), except 0.6°C (1°F)/min cool-down in autoclave cycle. Post-cure: Press, under vacuum. Heat to 260°C (500°F) at 0.6°C (1°F)/min. Hold 3 hr. Heat to 288°C (550°F) at 1.7°C (3°F)/min. Hold 3 hr. Heat to 316°C (600°F) at 1.7°C (3°F)/min. Hold 3 hr. Heat to 329°C (625°F) at 1.7°C (3°F)/min. Hold 1 hr. Cool to RT at 1.7°C (3°F)/min.					ORIGINAL PAGE IS OF POOR QUALITY	Satisfactory appearance. Photomicrographs showed transverse cracks through fiber layers. (Fig. 6)
PMR-15/HM-S graphite fabric	5 ply uni. 15.2 cm x 15.2 cm (6 in. x 6 in.)	Standard cycle with cure and post-cure heat-up and cool-down cycles. Modified step-wise post-cure.							ORIGINAL PAGE IS OF POOR QUALITY



TABLE 8 - PMR-15/HM-S PRE-CURED SKIN AND TEST LAMINATES

Lay-up Orientation	Thickness Per Ply mm (Mils)	Resin Content % by Wt.	Density g/cm <sup>3</sup>	% Voids (Calc.)	Tg °C (°F)
5 Ply 0°	0.356 (14.0)	29.57	1.6021	1.86	397 (747)
5 Ply 0°	0.363 (14.3)	32.74	1.5660	3.00	
5 Ply 0°	0.361 (14.2)	32.36	1.5728	2.72	
5 Ply (0°, +45°, 90°, +45°, 0°)	0.353 (13.9)	32.49	1.5734	2.64	
10 Ply 0°	0.353 (13.9)	30.61	1.5882	2.38	
10 Ply (0°, +45°, 90°, -45°, 0°) <sub>B</sub>	0.363 (14.3)	28.47	1.5853	3.30	

TABLE 9 - MR-150B2 PROCESSING DEVELOPMENT

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (in.)	Tg °C (°F)	
MR-150B2/ 181 fiberglass 32.6% resin content by wt.; 10.2% volatiles at 200°C (400°F)	6 ply 0° 35.6 cm x 31.75 cm (14 in. x 12-1/2 in.)	Vendors' recommended cycle on fiberglass prepreg	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 132-143°C (270-290°F) at 1.7-2.2°C (3-4°F)/min.</li> <li>3) Apply 1379 kPa (200 psi). Dwell 15 min more.</li> <li>4) Heat to 160°C (320°F) at 1.7-3.3°C (3-6°F)/minute. Dwell 45 min.</li> <li>5) Reduce pressure by 345 kPa (50 psi). Heat to 200°C (392°F) at 1.7-3.3°C (3-6°F)/min.</li> <li>6) Reduce pressure to 137.9 kPa (20 psi). Cure 4 hr at 200°C (392°F).</li> <li>7) Cool to 79°C (175°F) under pressure. (Bleeder 1 ply 120, 1 ply 181)</li> </ol> Post-cure: (heating rates 1.1-1.7°C (2-3°F)/min. <ol style="list-style-type: none"> <li>1) Heat to 204°C (400°F). Hold 2 hr.</li> <li>2) Hold 3 hr at 232°C (450°F).</li> <li>3) Hold 16 hr at 260°C (500°F).</li> <li>4) Hold 4 hr at 288°C (550°F).</li> <li>5) Hold 2 hr at 316°C (600°F).</li> <li>6) Hold 2 hr at 343°C (650°F).</li> </ol>	26.74	2.14	Negligible	0.23-0.24 (9.0-9.6)	330 (626)	Satisfactory
MR-150B2/MH-S graphite fabric 36% resin content by wt.; 16.1% volatiles at 200°C (400°F)	6 ply 0° 15.2 cm x 15.2 cm (6 in. x 6 in.)	Vendors' recommended cycle on MH-S graphite fabric prepreg	Same as above cycle.	26.84	1.56	7.87	0.34 (13.5)	341 (640)	Poor quality Surface porosity

ORIGINAL TEST  
OF POOR QUALITY

TABLE 9 - NR-150B2 PROCESSING DEVELOPMENT (Continued)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (mils)	Tg °C (°F)	
NR-150B2/HM-S Graphite fabric	4 ply 0° 15.2 cm x 15.2 cm (6 in. x 6 in.)	Full pressure applied at higher temperature to allow more escape of volatiles	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 132-143°C (270-290°F) at 1.7-2.2°C (3-4°F)/min. Hold 30 min.</li> <li>3) Heat to 160°C (320°F) at 1.7-2.2°C (3-4°F)/min. Hold 15 min.</li> <li>4) Apply 1379 kPa (200 psi). Hold additional 45 min at 160°C (320°F).</li> <li>5) Reduce pressure to 138 kPa (20 psi). Cure 4 hr at 200°C (492°F).</li> <li>6) Cool to 79°C (175°F) under pressure. (Bleeder 1 ply 181)</li> </ol>	24.36	1.4665	13.9	0.35 (13.8)	--	Poor quality. Surface porosity
	6 ply 0° 15.2 cm x 15.2 cm (6 in. x 6 in.)	Prestage to remove prepreg volatiles prior to cure	<p>Prestage:</p> <p>Each individual ply presteaged 15 min at 107°C (225°F).</p> <p>Cure same as above.</p> <p>Reduced vols. from 16.14% (30 min. @ 371°C (700°F)) to 11.98% based on prior test.</p>	31.91	1.4332	14.0	0.39 (15.5)	--	Poor quality. Surface porosity
	5 plies uni. 15.2 cm x 15.2 cm (6 in. x 6 in.)	Variation in temperature and time at which 200 psi applied	<ol style="list-style-type: none"> <li>1) Apply 50.8-101.6 mm (2-4 in.) Hg vacuum.</li> <li>2) Heat to 104-110°C (220-230°F) at 2.8-5.6°C (5-10°F)/min. Dwell 15 min.</li> <li>3) Apply full vacuum. Heat to 118-124°C (245-255°F) at 1.7-2.2°C (3-4°F)/min. Dwell 30 min.</li> <li>4) Apply 1379 kPa (200 psi). Dwell additional 30 min.</li> <li>5) Heat to 160°C (320°F) at 1.7-3.3°C (3-6°F)/min. Dwell 45 min.</li> <li>6) Reduce pressure to 345 kPa (50 psi).</li> </ol>	--	--	--	0.35 (13.8)	--	Poor quality. Surface porosity, but less than previous trials.

TABLE 9 - NR-150B2 PROCESSING DEVELOPMENT (Continued)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (mils)	Tg °C (°F)	
NR-150B2/HM-S Graphite fabric	5 plies uni. 15.2 cm x 15.2 cm. (6 in. x 6 in.)	Earliest application of full pressure	<ol style="list-style-type: none"> <li>7) Heat to 200°C (392°F) at 1.7-3.3°C (3-6°F)/min.</li> <li>8) Reduce pressure to 138 kPa (20 psi).</li> <li>9) Cure 4 hr.</li> <li>10) Cool to 79°C (175°F) under pressure.</li> </ol>	--	--	--	0.38 (15.0)	--	Complete absence of interlaminar bond.
	5 plies uni. 15.2 cm x 15.2 cm. (6 in. x 6 in.)	Variation in temperature and time at which 1379 kPa (200 psi) applied, plus pre-drying	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 110°C (230°F) at 1.7-2.2°C (3-4°F)/min. Dwell 15 min.</li> <li>3) Apply 1379 kPa (200 psi) and dwell additional 15 min.</li> <li>4) Heat to 160°C at 1.7-3.3°C (3-6°F)/min. Dwell 45 min.</li> <li>5) Reduce pressure to 345 kPa (50 psi).</li> <li>6) Heat to 200°C (392°F) at 1.7-3.3°C (3-6°F)/min.</li> <li>7) Reduce pressure to 138 kPa (20 psi).</li> <li>8) Cure 4 hr.</li> <li>9) Cool to 79°C (175°F) under pressure. Individual plies pre-dried 15 min in oven at 107°C (225°F).</li> </ol>	--	--	--	0.35 (15)	--	Poor quality surface porosity

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 9 - NR-150B2 PROCESSING DEVELOPMENT (Continued)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					Visual Observation
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply (mm) (mils)	Tg °C (°F)	
NR-150B2/HR-S graphite fabric	5 plies uni. 15.2 cm x 15.2 cm. (6 in. x 6 in.)	Applicable of full pressure at higher temperature without intermediate dwell	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 100°C (320°F) at 1.7-2.2°C (3-4°F)/min. Dwell 30 min.</li> <li>3) Apply 1034 kPa (150 psi). Dwell additional 45 min.</li> <li>4) Reduce pressure to 345 kPa (50 psi).</li> <li>5) Heat to 200°C (392°F) at 1.7-2.2°C (3-4°F)/min.</li> <li>6) Reduce pressure to 138 kPa (20 psi).</li> <li>7) Cure 4 hr.</li> <li>8) Cool to 79°C (175°F) under pressure. Not predried.</li> </ol>	29.34	1.4336	14.5	0.40 (16.0)	--	Satisfactory appearance
	5 plies uni. 15.2 cm x 15.2 cm. (6 in. x 6 in.)	Same as above, but increased pressure to 1379 kPa (200 psi)	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 110°C (320°F) at 0.6-1.1°C (1-2°F)/min. Dwell 30 min.</li> <li>3) Apply 1379 kPa (200 psi). Dwell 30 min.</li> <li>4) Reduce pressure to 345 kPa (50 psi) maintain vacuum.</li> <li>5) Heat to 200°C (392°F) at 1.7-2.2°C (3-4°F)/min.</li> <li>6) Reduce pressure to 138 kPa (20 psi).</li> <li>7) Cure 4 hr.</li> <li>8) Cool to 79°C (175°F) under pressure.</li> </ol>	28.76	1.4713	12.5	0.335 (14.8)	--	Satisfactory appearance
	5 plies uni. 15.2 cm x 15.2 cm. (6 in. x 6 in.)	Additional trial of 1st cure cycle (vendor recommended)	<ol style="list-style-type: none"> <li>1) Apply full vacuum.</li> <li>2) Heat to 138°C (280°F) at 1.7-2.2°C (3-4°F)/min. Dwell 15 min.</li> <li>3) Apply 1379 kPa (200 psi). Dwell 15 min.</li> <li>4) Heat to 160°C (320°F). Dwell 45 min.</li> </ol>						Discolored surface appearance

TABLE 9 - NR-150B2 PROCESSING DEVELOPMENT (Concluded)

Prepreg Description	Laminate Description	Processing Variable	Cure and Post-cure Cycle	Cured Laminate Properties					
				Resin Content % by Wt.	Density g/cm <sup>3</sup>	Voids % (Calc)	Thickness Per Ply mm (mils)	Tg °C (°F)	Visual Observation
NR-150B2/IM-S Graphite fabric			5) Reduce pressure to 345 kPa (50 psi). 6) Heat to 200°C (392°F) at 1.7-3.3°C (3-6°F)/min. 7) Reduce pressure to 138 kPa (20 psi). 8) Cure 4 hr. 9) Cool to 79°C (175°F) under pressure.						

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 10 - LARC-13 BONDING DEVELOPMENT

Surface Preparation and Bonding Procedures	Cure Cycle	Flatwise Tensile kPa (psi)	Remarks
PMR-15/fiberglass skins sanded and solvent wiped. Bonded to uncoated PMR-15/graphite core. LARC-13 supported film adhesive applied to core.	<ol style="list-style-type: none"> <li>1) Apply full vacuum</li> <li>2) Heat to 316°C (600°F) at 2.8°C (5°F)/min.</li> <li>3) Cure 1 hour at 316°C (600°F)</li> <li>4) Cool to 79°C (175°F) under vacuum</li> </ol>	<ol style="list-style-type: none"> <li>1) 2192.6 (316)</li> <li>2) 1935.3 (288)</li> <li>3) 1820.3 (264)</li> <li>Avg.: 1999.55 (290)</li> </ol>	<ol style="list-style-type: none"> <li>1) Skin-core adhesive failure</li> <li>2) Loading block failure</li> <li>3) Loading block failure</li> </ol>
NR-150B2/fiberglass skins sanded and solvent wiped. Bonded to NR-150B2 core coated with cell edge coating of unstaged NR-150B2 resin. LARC-13 supported film adhesive applied to core.	<ol style="list-style-type: none"> <li>1) Apply full vacuum</li> <li>2) Heat to 160°C (320°F) at 1.7-2.8°C (3-5°F)/min.</li> <li>3) Dwell at 160°C (320°F) for 30 min.</li> <li>4) Apply 69 kPa (10 psi) maintaining vacuum</li> <li>5) Heat to 200°C (392°F) at 1.7-2.8°C (3-5°F)/min.</li> <li>6) Dwell at 200°C (392°F) for 1 hour</li> <li>7) Heat to 316°C (600°F) at 1.7-2.8°C (3-5°F)/min.</li> <li>8) Cure at 316°C (600°F) for 4 hours</li> <li>9) Cool to 93°C (200°F) under pressure.</li> </ol>	<ol style="list-style-type: none"> <li>1) 2826.95 (410)</li> <li>2) 2971.75 (431)</li> <li>Avg. = 2899.35 (420.5)</li> </ol>	All skin-core failures
NR-150B2/fiberglass skins sanded and solvent wiped. Bonded to NR-150B2 core coated with cell edge coating of unstaged NR-150B2 resin. Cocured layer of NR-150B2/fiberglass adjacent to the core		<ol style="list-style-type: none"> <li>1) 2592.5 (376)</li> <li>2) 3068.3 (445)</li> <li>Avg. = 2830.4 (410.5)</li> </ol>	All skin-core failures

TABLE 10 - LARC-13 BONDING DEVELOPMENT (Concluded)

Surface Preparation and Bonding Procedures	Cure Cycle	Flatwise Tensile kPa (psi)	Remarks
NR-150B2/fiberglass skins and NR-150B2/graphite skins for test panels. Surface preparation and lay-up as described above for NR-150 skins and LARC-13 adhesive.	Same cycle as described above for PMR-15 skins bonded with LARC-13, except cure time at 316°C (600°F) extended to 4 hours.	See Table 13	Satisfactory appearance, with good filletting of adhesive around cells.

ORIGINAL PAGE IS  
OF POOR QUALITY



TABLE 11 - TEST RESULTS - NR-150A2:B2/T300 SANDWICH PANEL SPECIMENS

Test	Test Method MIL-STD 401, Para. No.	Specimen $\frac{L}{2}$ Dimensions cm (in.)	Test Temp	Conditioning	Test Results				Remarks, Failure Modes
					Ult. Strength kPa (psi)	Yield Strength kPa (psi)	Modulus MPa (psi)	Ult. Deflection (in.)	
Short-beam flexure "L" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.25(.500)	RT	---	1951 (263)	2131 (309)	217.9 (31,600)	0.076 (0.030)	Skin-core debond at one end of specimen due to cleavage loads on 1st & 3rd specimens. Second specimen had no visible debond. Shear buckling of core presumably oc- curred, but could not be detected visually.
					2656 (420)	2144 (311)	342.7 (49,700)	0.081 (0.032)	
					1972 (286)	2110 (306)	222.0 (32,200)	0.077 (0.030)	
					AVG. = 2275 (330)	AVG. = 2131 (309)	AVG. = 260.9 (37,633)		
Short-beam flexure "W" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.27(.500)	288°C (550°F)	---	2386 (346)	2131 (309)	A Δ ---	0.036 (0.014)	No skin-core debond. Shear buckling of core could not be detected visually.
					2551 (370)	2144 (311)		0.033 (0.013)	
					2613 (379)	2110 (306)	0.053 (0.021)		
					AVG. = 2517 (365)	AVG. = 2131 (309)			
Short-beam flexure "W" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.27(.500)	RT	500 hr at 288°C (550°F)	1834 (266)	986 (143)	655.7 (95,100)	0.043 (0.017)	No skin-core debond. Shear buckling of core could not be detected visually. A distinct yield point, signifi- cantly below ultimate, was noted.
					2130 (309)	1531 (222)	285.5 (41,400)	0.061 (0.024)	
					1620 (235)	1276 (185)	408.0 (58,600)	0.046 (0.018)	
					AVG. = 1751 (254)	AVG. = 1262 (183)	AVG. = 446.2 (65,033)		
Short-beam flexure "W" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.27(.500)	RT	---	2075 (301)	186.4 (27,100)	186.4 (27,100)	0.089 (0.035)	Skin-core debond at one end of specimen due to cleavage loads on 1st & 3rd specimens. Second specimen had no visible debond. Shear buckling at core presumably oc- curred, but could not be detected visually.
					2296 (333)	159.3 (23,100)	159.3 (23,100)	0.109 (0.043)	
					1827 (265)	131.7 (19,100)	0.099 (0.039)		
					AVG. = 2069 (300)	AVG. = 159.3 (23,100)			
Short-beam flexure "W" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.27(.500)	288°C (500°F)	---	1930.6 (280)	344.5 (195)	381.3 (55,300)	0.056 (0.022)	No skin core debond. Shear buckling of core could not be detected visually. A distinct yield point, signifi- cantly below ultimate was noted.
					2130.6 (309)	1385.9 (201)	395.8 (57,400)	0.061 (0.024)	
					1930.6 (280)	1475.5 (214)	348.2 (50,500)	0.058 (0.023)	
					AVG. = 1999.6 (290)	AVG. = 1399.7 (203)	AVG. = 375.1 (54,400)		
Short-beam flexure "W" direction	5.2.4	17.6(7)L x 5.1(2)W x 1.27(.500)	RT	500 hr at 288°C (550°F)	1089.4 (158)	730.9 (106)	237.9 (34,500)		Identical failure mode to unaged "W" shears. All showed distinct yield well below ultimate.
					910.1 (132)	813.6 (118)	246.2 (35,700)		
					1041.2 (151)	820.5 (115)	196.5 (28,500)		
					AVG. = 1013.6 (147)	AVG. = 786.0 (114)	AVG. = 226.8 (32,900)		

TABLE 11 - TEST RESULTS - NR-150A2:B2/T300 SANDWICH PANEL SPECIMENS (Concluded)

Test	Test Method MIL-STD 401, Para. No.	Specimen Dimensions cm (in.)	Test Temp	Conditioning	Test Results				Remarks, Failure Modes
					Ult. Strength kPa (psi)	Yield Strength kPa (psi)	Modulus MPa (psi)	Ult. Deflection cm (in.)	
Flatwise Compression	5.2.1a	7.6(3) x 7.6(3) x 1.27(.500)	RT	---	6123 (888)				Buckling failure of cell walls.
					6212 (901) 6461 (937) AVG. = 6268 (909)				
Flatwise Tensile	5.2.3	6.45(2) x 6.45(2) x 1.27(.500)	228°C (550°F)	---	3468 (503)				Buckling failure of cell walls.
					3151 (457) 3068 (445) AVG. = 468 (327)				
					3185 (462) 2551 (370) 2468 (358) 2813 (408) 2751 (399) AVG. = 2751 (399)				

ORIGINAL PAGE IS  
OF POOR QUALITY

△ Modulus could not be calculated due to excessively low deflection value.

△ L = parallel to core ribbon direction  
W = 90° to core ribbon direction

TABLE 12 - TEST RESULTS OF NR-150A2:B2 SPECIMENS AND  
LOADING BLOCKS RE-BONDED WITH LARC-13  $\Delta$

Test	Panel Description	Specimen $\Delta$ Dimensions	MIL-STD-401 Ref.	Test Temp.	Cell Strength kPa(psi)	Failure Mode
Flatwise Tensile	NR-150A2:B2/T300 skins bonded to	5.1 cm (2 in.) x 5.1 cm (2 in.) x 1.27 cm (0.500 in.)	5.2.3	288°C (550°F)	744.7 (108)	Adhesive failure skins to core. Loading block failure Loading block failure
	NR-150A2:B2/T300 core with				108.3 (15.7)	
Plate Shear	NR-150A2:B2 cell edge adhesive. Loading blocks re-bonded to graphite skins with LARC-13 adhesive.	15.2 cm (6 in.)L x 5.1 cm (2 in.)W x 1.27 cm (0.500 in.)	5.1.5	RT	1383.1 (200.6)	Primarily skin-core failure. Primarily skin-core failure.
	NR-150A2:B2/T300 skins bonded to			288°C (550°F)	1118.4 (162.2)	
	NR-150A2:B2/T300 core with				992.2 (143.9)	
	NR-150A2:B2 cell edge adhesive. Plates re-bonded to panels with LARC-13 adhesive.				AVG. = 1054.9 (153.0)	

$\Delta$  Surface preparation as follows: Steel plates were cleaned with steel wool, followed by MEK rinse and alcohol rinse. Aluminum loading blocks were cleaned with Pasajell chromic acid paste. LARC-13 liquid primer was applied to all adherends prior to lay-up.

$\Delta$  L = parallel to core ribbon

W = 90° to core ribbon

TABLE 13 - PMR-15/HM-S SANDWICH PANEL TEST RESULTS

Test	MIL-STD-401 para No.	Specimen (2) Dimensions	Test temp.	Conditioning	PMR-15 Test Results			Failure Mode
					Ult. Strength kva (psi)	Retention of RT Strength	Ult. Defl. cm (in.)	
Short-beam Shear "L" direction	5.2.4	17.8 cm (7 in.) L x 5.1 cm (2 in.) W x 1.3 cm (0.5 in.)	RT	--	1503.6 (224.7)	--	0.150 (0.059)	All beam flexures failed in core shear buckling, which could not be visually detected. No skin or adhesive failures.
					1306.0 (201.3)		0.135 (0.053)	
					1679.6 (243.6)		0.152 (0.060)	
			288°C (550°F)	--	1204.6 (174.7)	6d.0%	0.091 (0.036)	
					663.2 (125.2)		0.122 (0.048)	
					Avg. = 1034.25 (150.0)			
Short-beam Shear "W" direction	5.2.4	17.8 cm (7 in.) W x 5.1 cm (2 in.) L x 1.3 cm (0.5 in.)	RT	500 hr at 288°C (550°F)	166.2 (24.1)	8.2%	0.335 (0.132)	
					85.5 (12.4)		0.554 (0.218)	
					120.0 (17.4)		0.533 (0.210)	
					Avg. = 124.1 (18.0)			
					671.5 (126.4)		0.112 (0.044)	
					872.2 (126.5)		0.100 (0.0395)	
					895.7 (129.9)		0.104 (0.041)	
					919.0 (133.4)(1)		0.244 (0.096)	
					Avg. = 890.1 (129.1)			
Flatwise Compression	5.2.1(a)	7.6 cm (3 in.) x 7.6 cm (3 in.) x 1.3 cm (0.5 in.)	200°C (550°F)	--	570.2 (82.7)	74.4%	0.236 (0.093)	
					754.3 (109.4)		0.127 (0.050)	
					Avg. = 662.6 (96.1)			
					176.5 (25.6)		0.246 (0.0975)	
					171.0 (24.8)		0.242 (0.111)	
					160.0 (26.1)		0.266 (0.1125)	
					Avg. = 175.8 (25.5)			
					1766.6 (256.5)		--	crushing all specimens.
					1767.2 (259.2)			
					1769.3 (256.6)			
					Avg. = 1774.6 (257.4)			
					1434.85 (206.1)			
					1431.4 (207.6)			
					Avg. = 1433.1 (207.65)			

ORIGINAL PAGE IS  
OF POOR QUALITY

TABLE 13 - PMR-15/HM-S SANDWICH PANEL TEST RESULTS (Concluded)

Test	MIL-STD-401 Para No.	Specimen Dimensions	Test Temp.	Conditioning	PMR-15 Test Results			Failure Mode
					Ult. Strength kPa (psi)	Retention of RT Strength	Ult. Defl. cm (in.)	
Flatwise Tensile	5.2.3	5.1 cm (2 in.) x 5.1 cm (2 in.) x 1.3 cm (0.5 in.) Loading blocks bonded to panel	RT	---	2275.35 (330)		--	Skin-core failure - all specimens
					2365.7 (346)			
					AVG. = 2330.5 (338)			
Plate Shear	5.1.5	15.2 cm (6 in.) L x 5.1 cm (2 in.) W x 1.3 cm (0.5 cm) Plates bonded to panel	266°C (550°F)	500 hrs. at 288°C (550°F)	1214.2 (176.1)	53.1%	--	Some core shear failure, but primarily skin-core failure.
					1283.2 (186.1)			
					1214.2 (176.1)			
					AVG. = 1237.0 (179.4)			
					62.05 (11.9)	2.8%		
					50.0 (7.25)			
					AVG. = 66.0 (9.575)			
					1625.8 (235.8)	41.2%		
					1532.1 (222.2)			
					2671.8 (387.5)*			
					787.4 (114.2)*			
					AVG. = 1654.1 (239.9)			
					650.9 (94.4)	41.2%		
					666.7 (96.7)*			
					908.1 (131.7)*			
					AVG. = 741.9 (107.6)			

\* Re-tests. Plates bonded using same procedure, but in separate operation.

(1) Specimen tested with correct 1/4 span loading as defined in MIL-STD-401. All 550°F specimens tested with correct loading. Other RT tests run with incorrect loading, which complicated modulus calculations but gave apparently consistent strength values.

(2) L = parallel to core ribbon  
W = 90° to core ribbon

TABLE 14 - NR-150B2/HM-S SANDWICH PANEL TEST RESULTS

Test	MIL-STD-401 Para No.	Specimen $\Delta$ Dimensions	Test Temp.	Conditioning	Test Results		Failure Mode
					Ult. Strength kPa (psi)	Ult. Defl. cm (in.)	
Short-beam Shear "L" direction	5.2.4	17.8 cm (7 in.) L x 5.1 cm (2 in.) W x 1.3 cm (0.5 in.)	RT	--	1546.55 (224.3)	--	Shear failure with very slight permanent deformation.
					1607.2 (233.1)		
					1674.6 (242.9) AVG. = 1609.3 (233.4)		
Short-beam Shear "W" direction	5.2.4	17.8 cm (7 in.) W x 5.1 cm (2 in.) L x 1.3 cm (0.5 in.)	268°C (550°F)	--	1409.3 (204.4)	83.7%	Shear failure, slight permanent deformation.
					1274.2 (184.0)		
					1361.1 (177.4) AVG. = 1348.0 (195.5)		
Short-beam Shear "W" direction	5.2.4	17.8 cm (7 in.) W x 5.1 cm (2 in.) L x 1.3 cm (0.5 in.)	268°C (550°F)	500 hr at 288°F (500°F)	1277.6 (185.3)	79.6%	Shear failure, with permanent deformation and local top skin failure at loading points.
					1232.1 (178.7)		
					1332.8 (193.3) AVG. = 1281.1 (185.8)		
Short-beam Shear "W" direction	5.2.4	17.8 cm (7 in.) W x 5.1 cm (2 in.) L x 1.3 cm (0.5 in.)	RT	--	1420.4 (206.0)	--	Shear failure with very slight permanent deformation.
					1402.4 (203.4)		
					1425.9 (206.8) AVG. = 1416.2 (205.4)		
Short-beam Shear "W" direction	5.2.4	17.8 cm (7 in.) W x 5.1 cm (2 in.) L x 1.3 cm (0.5 in.)	268°C (550°F)	--	1099.1 (159.4)	74.6%	Shear failure, slight permanent deflection
					975.6 (141.5)		
					1093.5 (156.6) AVG. = 1056.3 (153.2)		
Flatwise Compression	5.2.1(a)	7.6 cm (3 in.) x 7.6 cm (3 in.) x 1.27 cm (0.50 in.)	268°C (550°F)	500 hr at 288°C (550°F)	1021.1 (148.1)	71.6%	Shear failure with permanent deformation and local top skin failure at loading points.
					993.6 (144.1)		
					1024.6 (148.6) AVG. = 1013.6 (147.0)		
Flatwise Compression	5.2.1(a)	7.6 cm (3 in.) x 7.6 cm (3 in.) x 1.27 cm (0.50 in.)	RT	--	1738.9 (252.2)	--	Core crushing all specimens.
					2114.0 (306.6)		
					1954.0 (283.4) AVG. = 1935.4 (280.7)		
Flatwise Compression	5.2.1(a)	7.6 cm (3 in.) x 7.6 cm (3 in.) x 1.27 cm (0.50 in.)	268°C (550°F)	--	1505.2 (214.3)	65.0%	Core crushing all specimens.
					1784.4 (258.8)		
					1645.1 (236.6) AVG. = 1645.1 (236.6)		

ORIGINAL PAGE IS OF POOR QUALITY

TABLE 14 - NR-150B2/HM-S SANDWICH PANEL TEST RESULTS (Concluded)

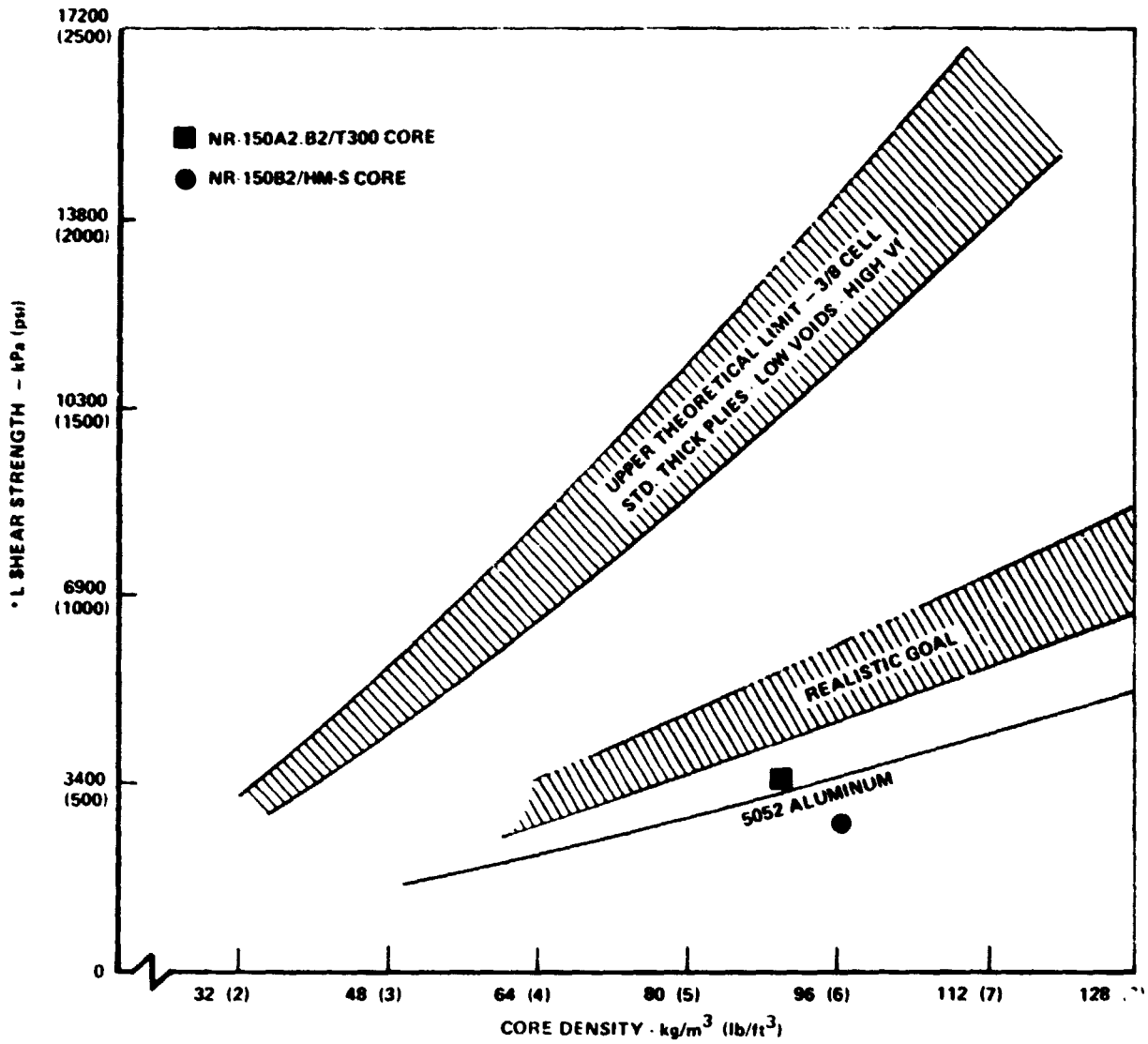
Test	MIL-STD-401 Para. No.	Specimen $\Delta$ Dimensions	Test Temp.	Conditioning	Test Results			Failure Mode
					Ult. Strength kps (psi)	Retention of RT Strength	Ult. Defl. cm (in.)	
Flatwise Tensile $\Delta$	5.2.3	5.1 cm (2 in.) x	RT	--	1453.5 (210.8)	--	--	Skin-core adhesive failure - all specimens.
		5.1 cm (2 in.) x 1.27 cm (0.500 in.) Loading blocks bonded to panels			2737.3 (397.0) 2843.5 (412.4) AVG. = 2345.0 (340.1)			
Plate Shear	5.1.5	15.2 cm (6 in.) x	289°C (550°F)	--	1) 53.1 (7.7) 2) 104.1 (15.1) 3) 165.1 (16.7) AVG. = 91.0 (13.2)	3.9%	1) Loading block bond. 2) Skin-core failure. 3) Loading block bond.	
		5.1 cm (2 in.) x 1.27 cm (0.500 in.) Plates bonded to panel	RT	--	1723.1 (249.9) 1876.1 (272.1) 966.0 (140.1) AVG. = 1799.6 (excl. low value)		Core shear with some skin-core adhesion loss. Specimen with low value primarily adhesive failure.	

$\Delta$  Specimen bottomed out under initial loading without picking up additional load.

$\Delta$  Specimens conditioned 500 hours at 289°C (550°F) and complete loss of skin-core adhesion and could not be tested.

$\Delta$  L = parallel to core ribbon  
W = 90° to core ribbon

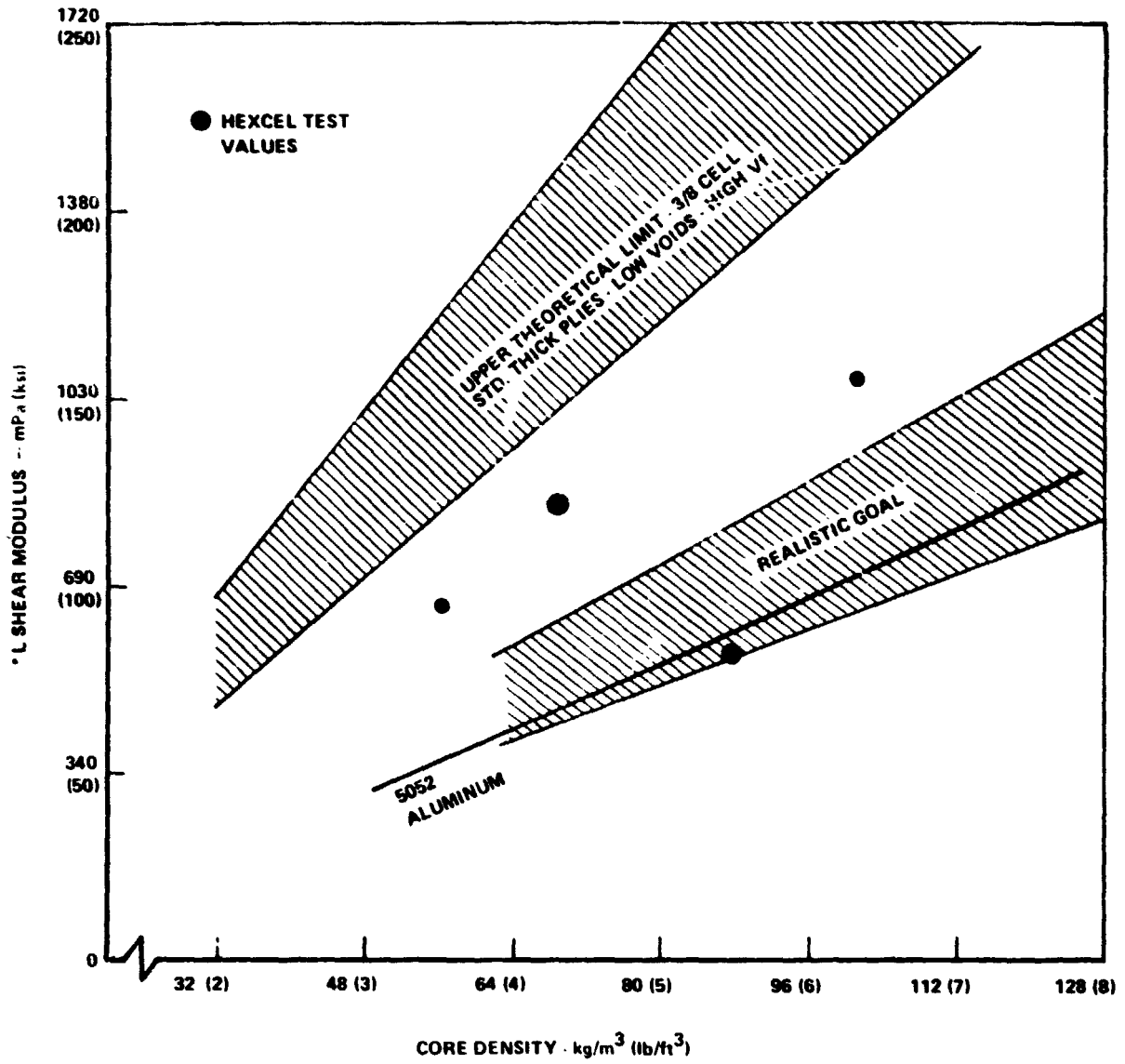
ORIGINAL PAGE IS  
OF POOR QUALITY



\* "L" = Parallel to core ribbon direction

Figure 1. Graphite honeycomb core shear strength (RT).





• "L" = Parallel to core ribbon direction

Figure 2. Graphite honeycomb core shear modulus ( $G_T$ ).

ORIGINAL PAGE IS  
OF POOR QUALITY

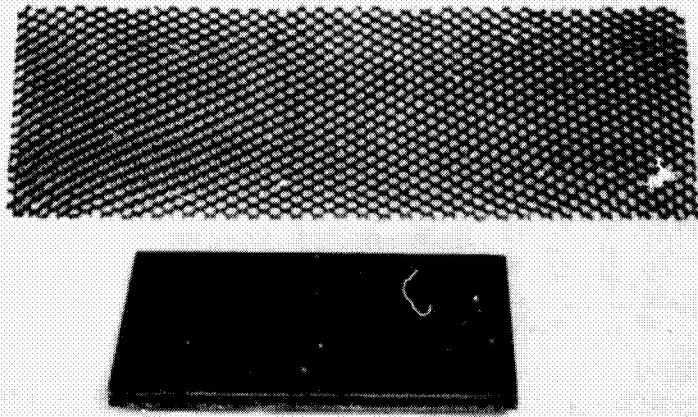


Figure 3. Graphite/polyimide honeycomb core.

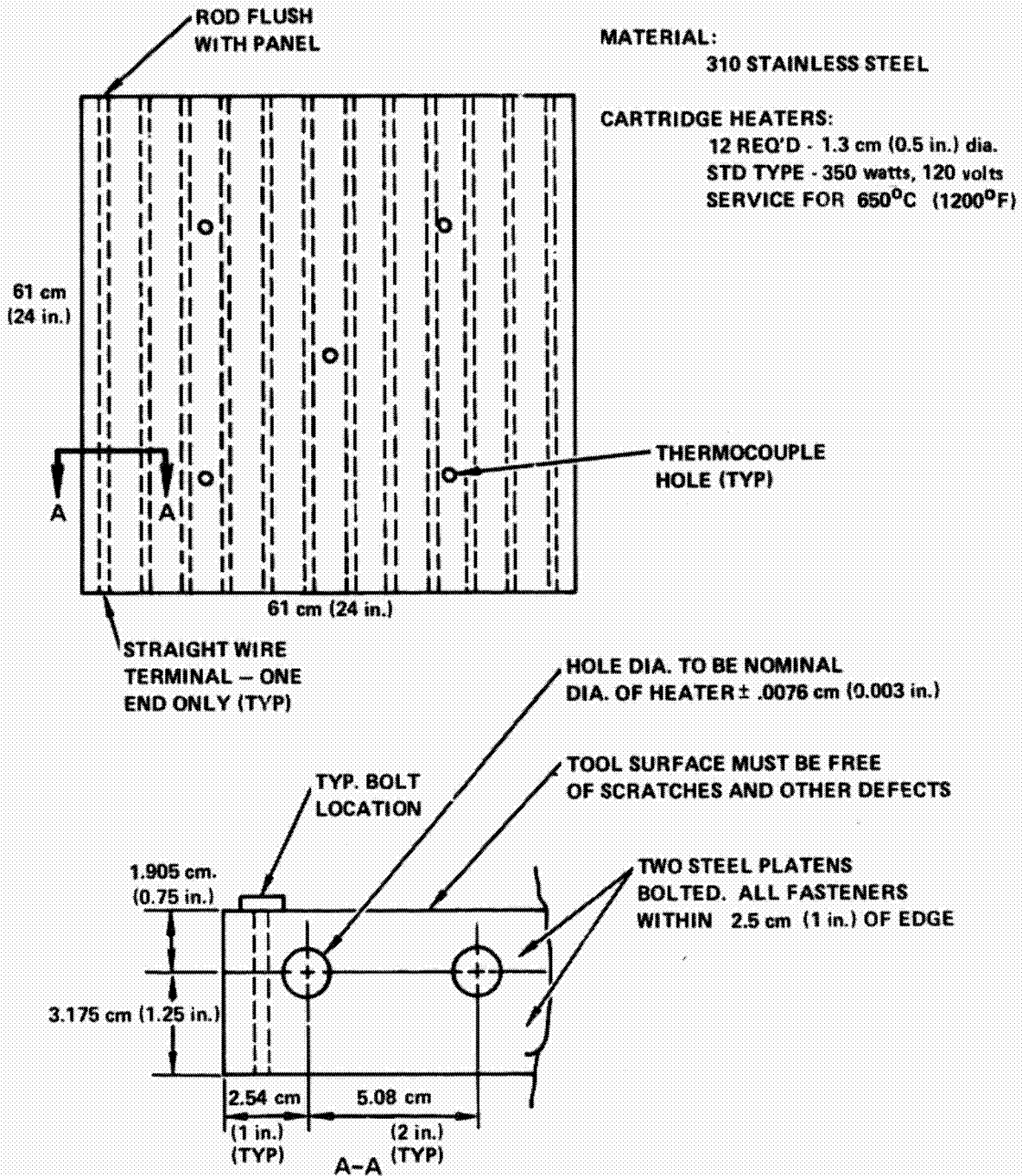


Figure 4. Diagram of heated platen.

ORIGINAL PAGE IS  
OF POOR QUALITY

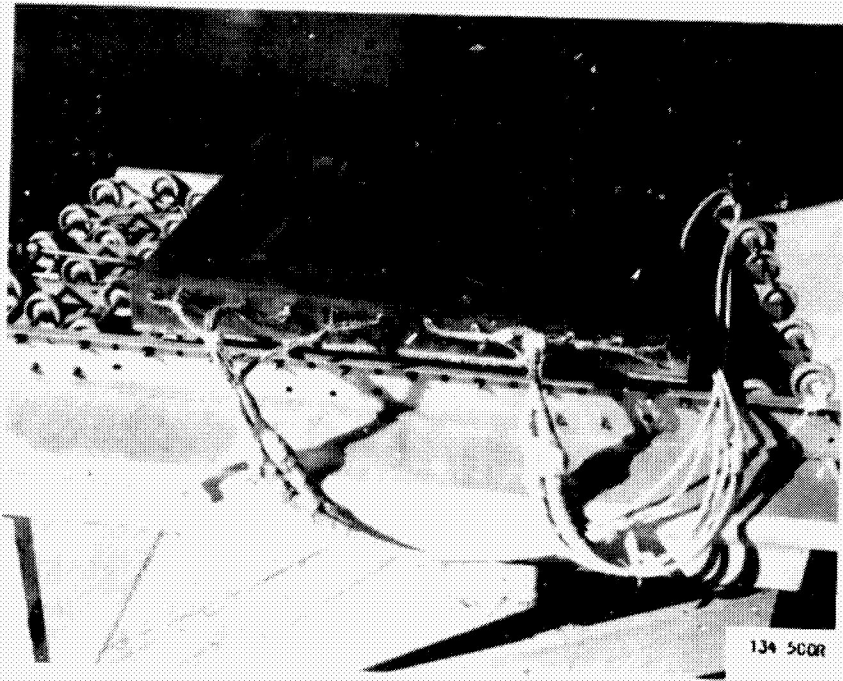
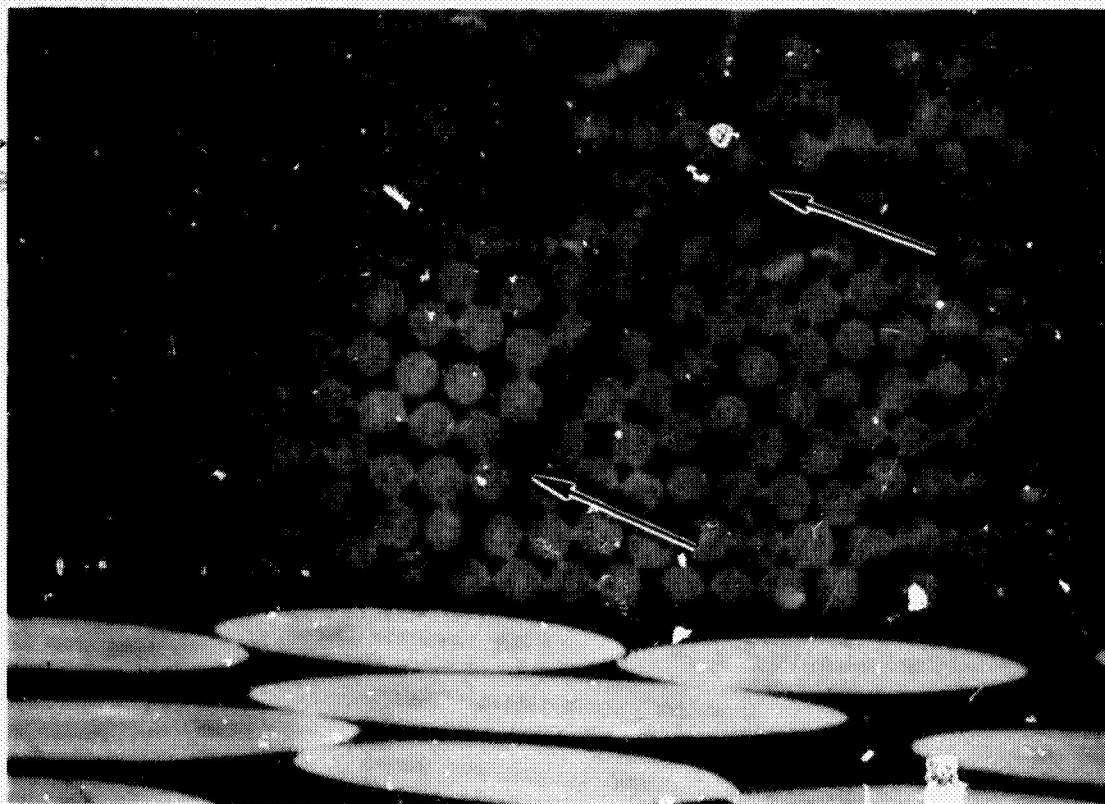


Figure 5. Heated platen with lead wires.

ORIGINAL  
DB PC05



0.025 mm. (0.001 in.)

ORIGINAL PAGE IS  
OF POOR QUALITY.

Figure 6. PMR-15/HM-S laminates.

ORIGINAL PAGE IS  
OF POOR QUALITY

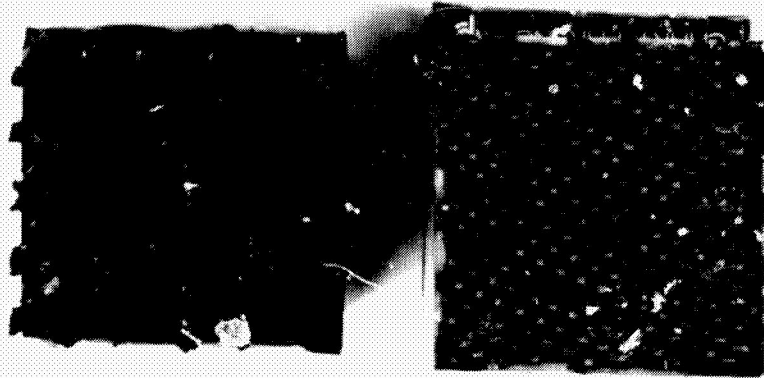


Figure 7. Flatwise tensile specimens bonded with LARC-13 adhesive.

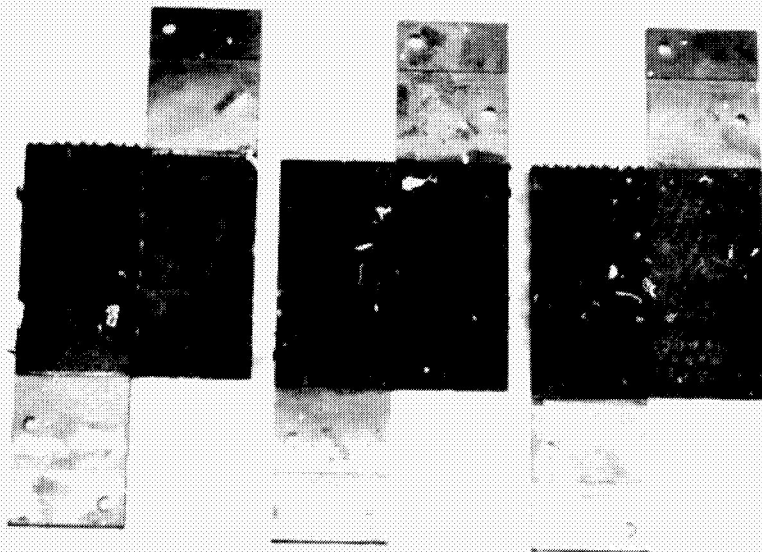


Figure 8. Plate shear specimens showing partial core failures.