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DEVELOPMENT OF LIGHTWEIGHT REINFORCED PLASTIC LAMINATES FOR SPACECRAFT INTERIOR APPLICATIONS

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16 Abstract

The specific purpose of this program was the development and optimization of lightweight, Kevlar-reinforced laminating systems that are non-burning, generate little smoke in the Space Shuttle environment, and are physically equivalent to the fiber-glass/polyimide system used in the Apollo program for such applications as non-structural cabin panels, racks, etc. A series of ten candidate resin systems representing five generic classes of resins were screened as matrices for Kevlar 49 reinforced laminates. Of the systems evaluated, the polyimides were the most promising with the phenolics a close second. Skybond 703 was selected as the most promising resin candidate. With the exception of compression strength, all program goals of physical and mechanical properties were exceeded. Several prototype Space Shuttle mobility and translation handrail segments were manufactured using Kevlar/epoxy and Kevlar-graphite/epoxy designs. This application shows a significant weight savings potential over the baseline aluminum configuration used previously on Apollo. The hybrid Kevlar-graphite/epoxy is the more suitable approach from a processing standpoint.

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PREFACE

A series of ten candidate resin systems representing five generic classes of resins were screened as matrices for Kevlar 49 reinforced laminates. The objective of the program was the timely development of laminates physically equivalent to current fiberglass/polyimide systems but 30 percent lighter in weight, that would be suitable for replacing aluminum in spacecraft interior applications. The specific purpose was the development and optimization of lightweight, Kevlar-reinforced laminating systems that are non-burning, generate little smoke in the Space Shuttle environment, and are physically equivalent to the fiberglass/polyimide system used in the Apollo program using commercially available resin systems. Of the systems evaluated, the polyimides were the most promising with the phenolics a close second. Skybond 703 was selected as the most promising resin candidate. Prepreg and laminate processing were optimized for the Skybond 703/Kevlar 49 system using a "no-postcure" ground rule to minimize costs. With the exception of compression strength, all program goals of physical and mechanical properties were met or exceeded. Since the end applications were non-structural in nature, the low compression strength obtained with the Skybond 703/Kevlar 49 was not of immense concern.

There is an apparent interface problem with Kevlar 49/polyimide laminates that exceeds the known problem with Kevlar/epoxy laminates. This results in low strengths in such properties as flexure, shear, and edge compression. This same problem prevents proper failures when testing Charpy impact specimens and causes delamination to occur.

For structural developments, recently developed highly modified epoxies, such as Hexcel's F-164, look promising for interior cabin applications. For exterior applications where volatile condensable material (VCM) testing is also a criterion, the non-postcured Skybond 703 and Hexcel F-164 both exceed the total weight loss allowable limit. Both systems meet the VCM requirements, and postcured Skybond 703 met the total weight loss requirement.

As part of this program, several prototype Space Shuttle mobility and translation handrail segments were manufactured using two Convair designs, i.e., (1) an all Kevlar/epoxy design, and (2) a Kevlar-graphite/epoxy design. Both met weight and dimensional requirements. The Kevlar-graphite/epoxy design is more suitable from a processing standpoint. This application appears very promising for Space Shuttle for saving weight over the aluminum type handrails previously used on Apollo. Continued development leading to full-scale production is recommended. This would include in-depth analysis, subcomponent testing, joint evaluation, production tooling tryout, and cost analysis.

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INTRODUCTION

NASA has for several years been involved in the development and end-item application of fire-resistant reinforced plastics for essentially non-structural spacecraft interior applications for Mercury, Apollo, and Skylab programs. The oxygen-rich environments of the previous spacecraft limited the use of reinforced plastics to polyimide matrices reinforced with glass fibers. The Space Shuttle environment is expected to be more benign than those of previous spacecraft, with the worst case expected to be 31% oxygen and 69% nitrogen at 70 KPa(10 psia) total pressure. Therefore, other resins may provide the required flame resistance.

The objective of this program was the timely development of laminate systems physically equivalent to current fiberglass/polyimide systems, but 30% lighter in weight. This weight savings could theoretically be accomplished by the direct substitution of Kevlar-49 reinforcement for the fiberglass reinforcement used in previous NASA spacecraft interior applications. Kevlar-49 is an aramid fiber developed several years ago by E.I. DuPont De Nemours and Company, Inc. It exhibits strength and elongation properties similar to glass fiber at a density of approximately 57% of the commonly used "E"-glass (1.45 versus 2.54 gm/cc). The availability of this fiber raised the prospect of very lightweight, flame-resistant laminates for applications where fiberglass/polyimides were used in prior spacecraft, as well as replacements for aluminum in such applications as floor surfaces, ceiling and wall panels, equipment storage racks, lavatories, etc. Substitution of Kevlar-49 for "E"-glass represents a potential savings of 30%, based on composite densities for laminates with similar fiber content (percentage by weight).

The potential use of other resin systems, such as epoxies, polyesters, and phenolics, instead of the polyimide resin used in earlier space programs, has several potential benefits. It can result in the use of lower-cost and lower-density resins, as well as minimizing the curing problems sometimes encountered with the condensation reaction polyimides used in the past.

The specific objective, therefore, was the development and optimization of lightweight, Kevlar-reinforced laminating systems that are non-burning, generate little smoke in the Space Shuttle environment, and are physically equivalent to the fiberglass/polyimide used on the Apollo program. Although the English system of units (ft, lb, sec) has been used for all measurements and calculations, in this report the SI system of units is shown as the primary system with English units following in parentheses.

The program was organized into four primary work areas:

Phase I - Selection and Screening of Organic Matrix Materials
Phase II - Optimization of Prepreg Laminating System
Phase III - Panel Fabrication and Data Generation
Phase IV - Documentation

The first phase selected a candidate resin based on screening tests conducted on representative resins from each of the following families: condensation reaction polyimides, addition reaction polyimides, epoxies, polyesters, and phenolics. Phase II combined the selected resin with Style 181 Kevlar-49 fabric, and optimized the resulting prepreg. In Phase III, prepregs were obtained using the knowledge gained in Phase II, and the resulting materials were processed into a series of deliverable panels. These panels contained combinations of Kevlar-49 reinforcement in various forms in conjunction with the selected resin from Phase I. The cured configurations were also subjected to an extensive characterization program.

As an additional task to this program, Convair was required to fabricate two prototype Space Shuttle mobility and translation handrail segments using Kevlar as the principal fiber reinforcement. In contrast to the general non-structural applications for spacecraft interiors, the handrails are considered structural.

SECTION 2

DISCUSSION

2.1 Phase I: Selection and Screening of Organic Matrix Materials

The objective of this phase of the program was to select candidate organic matrix materials of five specific families and to screen these candidates in combination with Kevlar by testing both physical and mechanical characteristics. Cost was another important factor in the selection and development of Space Shuttle materials. Laminate costs were considered on an overall basis. Therefore, consideration was given not only to the costs of the raw materials, but also to such items as molding costs. The latter is dictated by such things as mold tooling costs and hence the resin curing temperature, processing costs including storage requirements, handling costs, and cure profiles and durations. Towards this end, the program was directed towards process parameters which are compatible with vacuum bag molding techniques.

Phase I was initiated with a brief literature survey and contact of basic resin manufacturers and users. E.I. DuPont de Nemours & Company, Saugus, California was selected as the supplier for all Kevlar reinforced resin systems (prepregs) for the following reasons:

1. Geographically, the Saugus facility is close to Convair, which results in ease of communication and minimal shipping times for the prepregs.

2. The DuPont Saugus facility had prepregged more Kevlar than most other prepreggers, and this experience would be extremely valuable for this type of program.

3. DuPont is the manufacturer of Kevlar fiber and Kevlar fabrics and, as such, all pertinent reinforcement information on new woven or non-woven fabrics would be more readily obtainable by the Saugus facility.

4. It is more cost-effective to do multiple small quantity runs since it minimizes setup charges; therefore, a single prepregger results in lower costs.

5. The DuPont facility can accommodate small prepreg runs on production coating equipment.

6. DuPont is an experienced prepregger of polyimides.

Prime resin suppliers to DuPont's Saugus facility are as follows:

1. Addition reaction polyimides: Rhodia, Ciba-Geigy
2. Condensation reaction polyimides: DuPont, Monsanto
3. Phenolics: Ironsides Resins
4. Epoxies: Shell, Dow, Ciba-Geigy
5. Polyesters: Hooker

Prior to program initiation, ten resins had been tentatively selected based on survey of the literature, conversations with resin manufacturers, and conversations with DuPont (on the assumption that DuPont would be the sole prepregger). These resins are detailed below.

Condensation Reaction Polyimides

DuPont PI-4701 - This resin system was extensively evaluated by Rockwell International, and was the basic resin used in the Apollo program. Known initially as the 2501 resin system, it was evaluated extensively for flammability, smoke generation, outgassing, mechanical properties, etc.. PI-4701 is presently designated Pyralin polyimide 3001. The resin consists of a polyimide precursor in an NMP solvent and yields a polyimide when subjected to heat. The polyimide is the reaction product of an aromatic dianhydride and an aromatic diamine. Convair had evaluated the 4701 in the past with graphite reinforcements under NASA Contract NAS8-26198.

Monsanto Skybond 703 - This resin is similar in description to the DuPont PI-4701. Convair had evaluated it extensively with both glass and quartz fabric reinforcements, and has been producing production Standard ARM radomes with the Skybond 703 system for many years.

Addition or Modified Addition Reaction Polyimides

Ciba Geigy Kerimid 601 - This is an addition reaction, thermosetting polyimide resin that yields low-void polyimide parts when reinforced with fibers such as glass, graphite, etc. This system is presently finding extensive use in circuit boards. Postcuring at 480F is recommended for optimum properties. Kerimid 601 belongs to the so-called "bis malimide" class of resins.

DuPont Pyralin Polyimide 3003 - An aromatic, modifed addition reaction polyimide precursor that, under normal cure conditions, yields polyimides having void contents below 5%. As opposed to straight condensation reaction polyimides, the 3003 gives excellent properties after postcuring at 480 to 500F. Preliminary data indicated zero smoke generation in a 31% oxygen atmosphere.

Self-Extinguishing Grade Epoxies

DuPont Corlar Epoxy 5105 - This is a 350F curing, modified epoxy-novolac resin formulation that yields high mechanical strengths and excellent fire-resistant properties.

DuPont Corlar Epoxy 5134 - This bisphenol-A formulated epoxy system cures at 250F. In addition to good retention of properties at 160F, the system exhibits good flame resistance. The aspect of curing at 250F goes along with the stress on cost reduction techniques.

Self-Extinguishing Grade Polyesters

DuPont Corlar Polyester 4101 - This is a formulated system that uses chlorinated isophthalate polyester resin as a base. The system conforms to the requirements of MIL-R-7575B, Grade B, Class 4, and has good flame resistance.

DuPont Corlar Polyester 4102 - This is an improved flame-resistant version of 4101 and also uses a chlorinated isophthalate polyester resin as the base.

Phenolics

Ironside 101 - This is an unformulated phenol-formaldehyde phenolic resin which is qualified to MIL-R-9299A and exhibits good fire and smoke resistance. This system is particularly amenable to vacuum bag processing.

DuPont Corlar Phenolic 6113 - This is a phenol-formaldehyde based phenolic that has been specifically formulated with organo-metallics to yield systems having outstanding fire and smoke resistance. Preliminary testing by NASA (using Kevlar-49 reinforcement) substantiated the claim for excellent fire resistance and low smoke generation.

After program initiation, discussions were held with representatives of Shell Chemical Company, Dow Chemical Company, Ciba-Geigy, Hooker Chemical/ Durez Division, and Ironsides Resins in regard to their products which would best meet the goals of minimal flammability, smoke generation and offgassing. The following information was obtained:

1. Shell Chemical Company: They have discontinued their brominated epoxies and do not produce epoxy-novolacs. They have no rating on their resins for flammability and smoke generation. Since the prepreggers and laminators modify the resins to a great extent, Shell leaves the rating of resin systems to the prepreggers or end users.

2. Dow Chemical Company: They produce a line of epoxy-novolacs, but do not rate their systems for flammability and smoke generation.

3. Ciba-Geigy: They have no data on relative flame resistance of their epoxies. They produce brominated epoxies, epoxy-novolacs, and epoxy-cresol novolacs. Their 20% brominated epoxy is approximately 10% heavier than their standard DGEBA epoxy.

4. Hooker Chemical/Durez Division: They do not have data on flammability and smoke generation properties of their polyesters. Their Hetron 17 and Hetron 19, which they sell to DuPont, are highly chlorinated products, but final fire resistance is dependent on processing and formulating of prepregger.

5. Ironsides Resins: All phenolics have minimum flammability and smoke generation. Their best system is their 24-2 phenolic, because it has a minimum of volatiles. They have sold the 24-2 to DuPont in the past, and it has a UL SEO rating.

Discussions with DuPont revealed that the 24-2 phenolic is very boardy, has no drape, is extremely difficult to prepreg, and is approximately ten times the cost of standard phenolics. Therefore, the Ironsides 24-2 was dropped from further consideration.

Discussions with NASA revealed that the Lyndon B. Johnson Space Center was evaluating polyethersulfone (PES) because of its low flammability and smoke generation characteristics for other Space Shuttle applications. Review of the ICI data on polyethersulfone resulted in the substitution of the PES for one of the polyesters (DuPont's 4101). With concurrence of the NASA technical monitor, the following prepregs were ordered and obtained from DuPont's Saugus facility:

1. Style 181 Kevlar 49 reinforced polyimide PI 4701 (DuPont's 3001).
2. Style 181 Kevlar 49 reinforced polyimide (Monsanto's Skybond 703).
3. Style 181 Kevlar 49 reinforced polyimide (Rhodia's Kerimid 601).
4. Style 181 Kevlar 49 reinforced polyimide (DuPont's 3003).
5. Style 181 Kevlar 49 reinforced epoxy (DuPont's 5105).
6. Style 181 Kevlar 49 reinforced epoxy (DuPont's 5134).
7. Style 181 Kevlar 49 reinforced phenolic (DuPont's 6113).
8. Style 181 Kevlar 49 reinforced phenolic (Ironsides' 101).
9. Style 181 Kevlar 49 reinforced polyester (DuPont's 4102).
10. Style 181 Kevlar 49 reinforced polyethersulfone (ICI's 300P).

2.1.1 Tooling

In order to meet the low cost goals of this program, metal tooling was considered unacceptable. High temperature resistant plaster or silicone rubber were considered acceptable tooling materials. The first attempt to produce large flat tools utilized high temperature resistant plaster. Two one-inch thick hydroperm plaster tools, one 91.4 by 91.4 cm (36 by 36 inches), and the other 76.2 by 50.1 cm (30 by 20 inches), were prepared using the following procedure:

1. Prepare surface table by coating table with light oil or stearic acid solution.
2. Pour hydroperm into dammed area and rake smooth.
3. When slab is hard, remove dams and put slab in oven on flat plate.
4. Remove moisture from plaster tool by slowly following a stepped heat cycle:
   - 8 hours at 339K (150F)
   - 2 hours at 366K (200F)
   - 2 hours at 422K (300F)
   - 4 hours at 450K (350F)
5. Cool slowly in oven until tool is at room temperature.

The first Kevlar/epoxy panels were prepared on the 76.2 by 50.1 cm (30 by 20 in.) hydroperm tool. In order to seal during vacuum bagging, a thin layer of FEP Teflon was placed on the tool prior to layup of the panels. During cure, the Teflon wrinkled and caused some wrinkles in the panels. An attempt was then made to seal the hydroperm with a Dow Corning silicone release coating, R-671. During cool down on the curing cycle for the R-671, the hydroperm tool cracked. More flexible seal coatings were investigated for the larger hydroperm tool. The larger tool was coated with Airtech International Inc.'s Airseal 476, but it too suffered cracking problems during the cooling portion of a cure cycle.

A 76.2 by 40.6 cm (30 by 16 in.) cast silicone rubber tool was prepared from Dapcocast 38-3, a product of the Aircraft Products Company, Anaheim, California. The material is a two-part system which is outgassed in vacuum after mixing and prior to pouring. The cure cycle consisted of 24 hours at room temperature, two hours at 355K (180F), and four hours at 477K (400F).

The first attempt to bag a Kevlar/epoxy layup to the silicone rubber resulted in a warped panel. This results from the shrinkage of the bagging material during cure. Subsequent cures using envelope bagging and the silicone rubber tool overcame the warpage problem. A second silicone rubber tool of the same dimensions was prepared, and all the test panels evaluated in this program were made on the two rubber tools.

2.1.2 Prepreg Properties

Volatile contents, resin contents, and resin flow on all Phase I prepregs was conducted by DuPont using the test methods described below. The resin contents for the two epoxy prepregs and the polyethersulfone prepreg were calculated both as percent wet resin (percent resin content) and percent dry resin (percent resin solids). All the other systems were calculated for percent dry resin. The test data is summarized in Table 2-I. Degree of tack was a qualitative (judgement) evaluation conducted at Convair.

2.1.2.1 Volatile Content: Cut three specimens, 10.2 by 10.2 cm (4.0 by 4.0 in.), using steel template, and weigh collectively to the nearest 0.001 gm. Attach "S" shaped hook to corner and place in circulating air oven for required time and temperature as noted below. Suspend specimens individually, allowing for free air circulation between each. Do not allow oven door to remain open longer than 10 seconds while loading specimens. If dripping of resin occurs, repeat, hanging specimens by diagonal corners.

Prepreg Type	*Temperature, °K (°F)	*Time, Minutes
High temperature epoxy	436 (325)	10
General purpose epoxy	394 (250)	10
Polyimide	672 (750)	2.5
Phenolic	411 (280)	10
Polyester	394 (250)	10
Polyethersulfone	589 (600)	20

* All temperature tolerances ± 1.7 K (± 3F); all time tolerances ± 0.25 minutes.

Remove samples from oven, desiccate for five minutes, and reweigh collectively to the nearest 0.001 gm.

Calculate volatile content as follows:

$$\% \text{ Volatiles} = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 = original weight, gm

W_2 = final weight, gm

Table 2-I. Prepreg Properties of Phase I Materials

Material	Dry Resin Wt. %	Wet Resin Wt. %	Resin Flow * Wt. %	Resin Flow * Test Temp.	Volatile Content Wt. %	Volatile Content Test Conditions	Degree of Tack
Corlar 5105/Kevlar 49	51.4	50.9	16.9	436K (325F)	1.06	10 min. at 436K (325F)	Very dry
Corlar 5134/Kevlar 49	55.0	54.5	9.8	394K (250F)	0.99	10 min. at 394K (250F)	Slight tack
Skybond 703/Kevlar 49	36.7	-	37.4	505K (450F)	20.7	2.5 min. at 672K (750F)	Very dry
Pyralin 3001/Kevlar 49	42.6	-	51.8	505K (450F)	27.0	2.5 min. at 672K (750F)	Slight tack
Kerimid 601/Kevlar 49	46.3	-	34.3	505K (450F)	14.84	2.5 min. at 672K (750F)	Very dry
Pyralin 3003/Kevlar 49	33.5	-	23.7	505K (450F)	19.3	2.5 min. at 672K (750F)	Very dry
Ironsides' 101/Kevlar 49	46.9	-	35.0	411K (280F)	1.00	10 min. at 436K (325F)	Tacky
Corlar 6113/Kevlar 49	57.8	-	12.7	411K (280F)	0.65	10 min. at 436K (325F)	Slight tack
Corlar 4102/Kevlar 49	49.7	-	8.9	394K (250F)	3.56	10 min. at 408K (275F)	Tacky
300P/Kevlar 49	42.8	41.2	5.2	589K (600F)	2.80	20 min. at 450K (350F)	Very dry

* All resin flow tests run at 103.5 KPa (15 psi) for 10 minutes except 300P/Kevlar 49 system, which was run for 15 minutes using 689.5 KN/m^2 (100 psi).

2.1.2.2 Resin Content: Cut 10.2 by 10.2 cm. (4.0 by 4.0 in.) prepreg specimen and weigh to the nearest 0.001 gm (W_1). Subtract weight of volatiles as determined by test method above. The resultant is known as devolatilized weight (W_2). Subtract weight of dry Kevlar fabric (W_3) having same surface area as prepreg sample to give resin weight.

Calculate dry resin content (resin solids) for prepregs with high volatile contents using the following formula:

$$\% \text{ dry resin content} = \% \text{ resin solids} = \frac{W_2 - W_3}{W_2} \times 100$$

For prepregs with very low volatile contents such as epoxies, calculate wet resin content using the following formula:

$$\% \text{ wet resin content} = \% \text{ resin content} = \frac{W_2 - W_3}{W_1} \times 100$$

For prepregs with very low volatile contents, the differences between percent dry resin content and percent wet resin content are very small.

2.1.2.3 Resin Flow: Weigh proper number of specimens to 0.001 gm. Properly align specimens between release film or release agent coated caul plates. If using release film, place specimens and film between aluminum caul plates. Position specimen(s) in the center of a press (preconditioned to specified temperature) and close to specified pressure as rapidly as possible. Release at end of time interval. Remove laminate from press and cool. Scrape off flash down to original size with dull knife, being careful to avoid removing any reinforcement from original dimensions. Reweigh laminate to 0.001 gm. Calculate flow as follows:

$$\% \text{ flow} = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 = original weight, gm

W_2 = final weight, gm

For the different prepreg systems, the following press temperatures are used:

High temperature epoxies	436K (325F)
General purpose epoxies	394K (250F)
Polyimides	505K (450F)
Phenolics	411K (280F)
Polyesters	394K (250F)
Polyethersulfones	589K (600F)

All flow tests are run for ten minutes at 103 KPa (15 psi).

2.1.3 Test Panels

Phase I test panels were prepared from each of the prepregs using the standard vacuum bag curing technique and cure and postcure cycles as recommended by the prepreg manufacturer. The silicone rubber tooling was used for all panels in conjunction with envelope bagging. The separator film used was a porous Teflon coated glass cloth having a thickness of approximately 0.0071 cm. (0.0028 in.). Bleeder cloth was style 7581 E-glass fabric. Ratio of bleeder plies to prepreg plies was per recommendation of the prepreg supplier. The vacuum bagging material was a high temperature nylon film, Vac-Pak H.S. -8171 obtained from Richmond, a Division of Pak-Well. A summary of the processing parameters is given in Table 2-II.

In general, test panel sizes for each material were as follows: two panels 45.8 by 22.9 cm. (18 by 9 in.), two panels 30.5 by 12.7 cm. (12 by 5 in.), one panel 38.1 by 38.1 cm. (15 by 15 in.), and one panel 15.2 by 15.2 cm. (6 by 6 in.). The latter panel was used for flexure and density determinations, while resin content and void content was calculated on all panels. The 15.2 by 15.2 cm. (6 by 6 in.) panels were 12-ply layups, while the other panels were 6-ply layups. All the panels with the exception of the 15.2 by 15.2 cm. (6 by 6 in.) panels were sent to NASA's Lyndon B. Johnson Space Center for flammability, smoke, and offgassing evaluations.

2.1.4 Test Methods

Flexural strength testing was conducted at room temperature (RT) per ASTM D790-71. Specimen dimensions were 7.62 by 2.54 cm. (3 by 1 in.). The span-to-depth ratio was 16 to 1, loading head and supports were 0.635 cm. (0.25 in.) diameter rods, and head speed was 0.127 cm. (0.05 in.) per minute.

Densities were obtained per ASTM D792-66. All densities were obtained on 12-ply laminates, and specimen dimensions were 2.54 by 2.54 cm. (1 by 1 in.). Resin contents (percent by weight) were obtained on all laminates by weighing the laminates and subtracting out the weight of style 181 Kevlar fabric. The weight per unit of area of Kevlar was obtained by sampling known size samples of prepreg, washing out the resin with acetone or MEK, drying, and weighing. The weight per unit area obtained in this manner was 0.0168 gm/cm^2. This is in excellent agreement with the value of 0.017 gm/cm^2 reported in DuPont's Kevlar Design Manual. Void content calculations were conducted using the following formula:

$$\% \text{ Void Content} = 100 - (\text{s.g.})_c \left[\frac{(\text{wt.}\% \text{ resin})}{(\text{s.g.})_r} + \frac{(\text{wt. } \% \text{ fiber})}{(\text{s.g.})_f} \right]$$

Table 2-II. Cure Cycles for Kevlar 49 Reinforced Resin Systems

Material	Bleeder Ratio Prepreg/7581 Glass Cloth	Cure Cycle	Post Cure Cycle
Corlar 5134/Kevlar 49-181 fabric	3 to 1	Apply full vacuum at R.T. Heat at 1.7 to 2.8K (3 to 5F) per minute to 394K. (250F). Hold 2 hours and cool to below 352K (175F) before removal from vacuum bag.	None.
Pyralin 3003/Kevlar 49-181 fabric	1 to 1	Apply full vacuum at R.T. Heat at 1.4K (2.5F) per minute to 450K (350F). Hold 1 hour. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 450K (350F), and hold for 16 hours. Heat to 477K (400F) and hold for 16 hours. Heat to 505K (450F), and hold for 16 hours. Heat to 533K (500F) and hold for 16 hours. Cool to R.T.
Kerimid 601/Kevlar 49-181 fabric	1 to 1	Apply full vacuum at R.T. Heat at 2.8K (5F) per minute to 394K (250F). Hold 1 hour. Raise temperature to 450K (350F) at 2.8K (5F) per minute. Hold 1 hour. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 464K (375F) and hold for 2 hours. Heat to 477K (400F), and hold for 2 hours. Heat to 505K (450F) and hold for 2 hours. Heat to 522K (480F) and hold for 16 hours. Cool to R.T.
Skybond 703/Kevlar 49-181 fabric	1 to 1	Apply Full vacuum at R.T. Heat to 389K (240F) at 1.7K (3F) per minute. Hold 30 minutes. Raise temperature at 0.6K (1F) per minute to 405K (270F). Hold 30 minutes. Heat to 450K (350F) at 0.6K (1F) per min., and hold for 1 hour. Cool to below 352K (185F) before removal from vacuum bag.	Heat to 450K (350F) and hold 1 hour. Heat to 477K (400F) and hold 2 hours. Heat to 505K (450F) and hold 2 hours. Heat to 533K (500F) and hold 2 hours. Heat to 561K (550F) and hold 4 hours. Heat to 589K (600F) and hold 4 hours. Cool to R.T.

Table 2-II. - continued

Material	Bleeder Ratio Prepreg/7581 Glass Cloth	Cure Cycle	
Corlar 5105/Kevlar 49-181 fabric	4 to 1	Apply full vacuum at R. T. Heat to 450K (350F) at 1.7 to 2.8K (3 to 5F) per minute. Hold for 1 hour. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 450K (350F) and hold for 3 hours. Cool to R. T.
Corlar 6113/Kevlar 49-181 fabric	2 to 1	Apply full vacuum at R. T. Heat to 422K (300F) at 1.7 to 2.8K (3 to 5F) per minute. Hold for 2 hours. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 436K (325F) and hold for 2 hours. Cool to R. T.
* Corlar 6101-2/Kevlar 49-181 fabric	2 to 1	Apply full vacuum at R. T. Heat to 366K (200F) at 1.7 to 2.8K (3 to 5F) per minute. Hold 30 minutes. Heat to 394K (250F) at same rate, and hold 30 minutes. Heat to 422K (300F) at same rate, and hold 1 hour. Heat to 450K (350F) at same rate, and hold 1 hour. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 450K (350F) and hold for 2 hours. Cool to R. T.
Pyralin 3001 (4701)/Kevlar 49-181 fabric	1 to 1	Apply full vacuum at R. T. Heat to 405K (270F) at 1.7 to 2.8K (3 to 5F) per minute. Hold for 30 minutes. Heat to 450K (350F) at 2.8K (5F) per minute, maximum. Hold for 2 hours. Cool to below 352K (175F) before removal from vacuum bag.	Heat to 477K (400F) and hold 2 hours. Heat to 505K (450F) and hold 2 hours. Heat to 433K (500F) and hold 4 hours. Heat to 561K (550F) and hold 4 hours. Heat to 589K (600F) and hold 4 hours. Cool to R. T.
Corlar 4102/Kevlar 49-181 fabric	4 to 1	Apply full vacuum at R. T. Heat to 394K (250F) at 1.7 to 2.8K (3 to 5F) per minute. Hold 2 hours at 394K (250F). Cool to below 352K (175F) before removal from vacuum bag.	None.

* DuPont designation for Ironsides 101 resin

The specific gravities used in the above formula for the various resins and for Kevlar were obtained from DuPont, and are listed in Table 2 III. The composite specific gravities used in the void content calculations were those obtained on the 12-ply laminates.

Flammability, offgassing, and odor testing were conducted per NHB8060.1, "Flammability, Odor and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion." The flammability testing was per NHB8060.1 test number 1, upward propagation. The offgassing test method was NHB8060.1 test number 7, classification 5, C, 1, 69 KPa (10.0 psia) air atmosphere. The test method for optical smoke density was per ASTM Special Technical Publication 422 (propane flame heat source), 103 KPa (14.7 psia) air atmosphere. Odor was determined per NHB 8060.1 test number 6, classification 5(b), 103 KPa (14.7 psia) air atmosphere. Volatile condensible material (VCM) determinations were performed on the Phase I materials per the test method described in NASA's SP-R-0022, "Specification Vacuum Stability Requirements of Polymeric Material for Spacecraft Application."

?.1.5 Cured Laminate Test Results

Mechanical and physical property test results for the cured laminates of Phase I are summarized in Table 2-IV. Flammability, optical smoke density, offgassing, odor, and volatile condensible material testing was conducted on the panels by NASA. The results of this testing are summarized in Tables 2-V to 2-VI.

Flexure strengths for the Kevlar reinforced laminates were highest for the polyethersulfone resin and decreased in the following order: epoxies, phenolics and polyesters, and polyimides. Within any generic family, the individual resin systems gave similar flexure strengths. Each value reported in Table 2-IV is an average of three individual tests. The strengths obtained were in good agreement with data reported in DuPont's "Kevler" 49 Data Manual, i.e., the vacuum bag epoxy laminates prepared in this program had an average flexure strength of 327.9 MPa (47.56 ksi) versus a reported value of 345. MPa (50.0 ksi) for a vacuum augmented pressure cure.

Average density and resin content values for the cured laminates are summarized in Table 2-III, and show the resin contents of the Kevlar/polyimide laminates to be significantly lower than those of the other systems evaluated. This can be attributed chiefly to the lower dry resin content in the polyimide prepregs than in most of the other prepregs. Reported densities are averages of three specimens, and resin contents are averages of a single measurement from each of six panels. A lot of air bubbles were encountered in the specific gravity determinations on the 101, 6113 and 601 matrix materials. This would lead to greater errors in the measurement of specific gravity for those materials. The accuracy of the void calculations is highly dependent on the accuracy of the literature values for the specific gravities of the resin matrices.

Table 2-III. Specific Gravities of Phase I Raw Materials

Resin	Resin Type	Resin Specific Gravity	Reinforcement	Reinforcement Form	Reinforcement Specific Gravity
DuPont's Corlar 5105	Epoxy	1.25	Kevlar-49	Style 181 Fabric	1.45
DuPont's Corlar 5134	Epoxy	1.37	↓	↓	↓
Monsanto's Skybond 703	Condensation Polyimide	1.33			
DuPont's Pyralin 3001 (4701)	Condensation Polyimide	1.39			
Rhodia's Kerimid 601	Addition Polyimide	1.32			
DuPont's Pyralin 3003	Modified Condensation Polyimide	1.36			
Ironsides' 101	Phenolic	1.29			
DuPont's Corlar 6113	Phenolic	1.26			
DuPont's Corlar 4102	Polyester	1.41			
ICI's 300P	Polyethersulfone	1.37			

Table 2-IV. RT Mechanical and Physical Properties of Vacuum Bagged Laminates of Phase I Materials

Material	Average Flexure Strength MPa	(ksi)	Average Density g/cc	Average Resin Content wt. %	Average Void Content vol. %
Corlar 5105/Kevlar 49	318.3	(46.17)	1.225	45.02	9.43
Corlar 5134/Kevlar 49	337.4	(48.94)	1.255	54.86	10.68
Skybond 703/Kevlar 49	224.9	(32.62)	1.207	27.49	14.69
Pyralin 3001/Kevlar 49	201.0	(29.15)	1.201	26.06	16.24
Kerimid 601/Kevlar 49	223.7	(32.45)	1.295	35.20	7.60
Pyralin 3003/Kevlar 49	230.4	(33.41)	1.282	32.55	9.57
Ironsides' 101/Kevlar 49	280.3	(40.66)	1.163	35.15	16.30
Corlar 6113/Kevlar 49	301.4	(43.72)	1.192	40.87	12.73
Corlar 4102/Kevlar 49	286.5	(41.56)	1.356	44.41	5.31
300P/Kevlar 49	345.2	(50.07)	1.328	38.80	6.45
Skybond 703/Kevlar 49 *	216.0	(31.32)	1.208	-	-

* No postcure

Table 2-V. Results of Flammability Testing *

Material Trade Name	Panel Thickness cm (in)	Open Flame Time	Total Time of Test	Distance to Self Extinguish, cm (in.)	Burning Rate, cm/sec (in/sec)	Remarks
Skybond 703/Kevlar 49	0.119 (0.047)	55 sec	2 min. 54 sec.	1.27 (0.5)	-	small clean flame, little smoke
Corlar 6113/Kevlar 49	0.160 (0.063)	-	5 min. 45 sec.	8.89 (3.5)	-	small orange flame, emitted strong odor, very light smoke
Kerimid 601/Kevlar 49	0.264 (0.104)	-	3 min. 4 sec.	1.27 (0.5)	-	small clean flame, little visible smoke, glowed for about 1 min. after flame extinguished
Corlar 5105/Kevlar 49	0.160 (0.063)	-	1 min. 1 sec.	sample consumed	0.20 (0.08)	burned with black sooty smoke, large orange flame
Corlar 5134/Kevlar 49	0.191 (0.075)	-	45 sec.	2.54 (1.0) charred 12.7 (5.0) discolored	-	small orange flame, heavy smoke, sooty
Corlar 5134/Kevlar 49	0.107 (0.042)	-	37 sec.	sample consumed	0.33 (.13)	orange flame, sooty, heavy smoke
Corlar 6101-2/Kevlar 49	0.142 (0.056)	-	2 min. 50 sec.	3.81 (1.5)	-	orange flame, very little smoke, clean burning
Pyralin 3003/Kevlar 49	0.119 (0.047)	2 min. 10 sec.	3 min. 19 sec.	2.54 (1.0)	-	clean orange flame, very light smoke
Pyralin 3001/Kevlar 49	0.119 (0.047)	about 1 min.	2 min. 58 sec.	1.27 (0.5)	-	small orange flame, very light smoke
Corlar 4102/Kevlar 49	0.142 (0.056)	-	39 sec.	sample consumed	0.30 (0.12)	large orange flame, heavy smoke
Skybond 703/Kevlar 49**	0.130 (0.051)	-	4 min.	1.27 (0.5)	-	very light smoke
Skybond 703/Kevlar 49**	0.066 (0.026)	-	4 min. 33 sec.	11.4 (4.5)	-	very light smoke

* Upward propagation test, clenweld ignitor, 69.0 KPa (10 psia) pressure, 31% O_2/69% N_2, sample size: 6.35 by 12.7 cm (2.5 by 5 in.)

** No postcure.

Table 2-VI. Offgassing Test Results *

Material Trade Name	Material Sample Weight, grams	Weight Loss, grams/gram	Carbon Monoxide Mgm./gm.	Total Organics Mgm./gm.
Corlar 6101-2/Kevlar 49	20.0913	0.0011	2.0	1.0
Skybond 703/Kevlar 49 **	20.4765	0.0013	0.4	3.0
Corlar 5105/Kevlar 49	20.7214	0.0006	1.0	2.0
Corlar 5134/Kevlar 49	20.1260	0.0011	0.4	2.0
Corlar 4102/Kevlar 49	20.1134	0.0017	1.0	56.0
Skybond 703/Kevlar 49	20.5252	0.0029	0.4	0.7
Pyralin 3003/Kevlar 49	20.3687	0.0024	1.0	0.8
Pyralin 3001/Kevlar 49	20.9211	0.0023	0.2	0.4
Corlar 6113/Kevlar 49	20.2314	0.0012	1.0	1.0
Kerimid 601/Kevlar 49	20.7024	0.0021	1.0	1.0

* Test chamber size was 4 liters and soak time was 72 hours at 49C (120F). Sample was withdrawn at 81.4 to 82.7 KPa (11.8 to 12.0 psia).

** No postcure.

Table 2-VII. Odor Test Results

Material Trade Name	Test Chamber Volume, liters	Material per Liter of Test Chamber Volume, grams	Odor Sample Concentration: 1 part to 29 parts O_2	Odor Sample Concentration: 1 part to 9 parts O_2	No Dilution
Corlar 6101-2/Kevlar 49	2	5.0	0.4	0.0	0.4
Skybond 703/Kevlar 49**	2	5.0	0.6	1.0	1.4
Corlar 5105/Kevlar 49	2	5.2	0.2	0.2	1.2
Corlar 5134/Kevlar 49	2	5.0	0.4	0.4	1.2
Corlar 4102/Kevlar 49	2	4.8	0.2	0.6	1.2
Skybond 703/Kevlar 49	2	5.0	0.2	0.3	0.5
Pyralin 3003/Kevlar 49	2	5.0	1.4	1.6	1.8
Pyralin 3001/Kevlar 49	2	5.0	0.0	0.2	0.6
Corlar 6113/Kevlar 49	2	5.2	0.4	0.2	0.6
Kerimid 601/Kevlar 49	2	5.0	0.2	0.0	0.0

* Total soak was 72 hours at 49C (120F). Sample withdrawn at 84.8 KPa (12.3 psia).

** No postcure.

Table 2-VIII. Optical Smoke Density (DSM) Test Results

Material Trade Name	Maximum Specific Optical Density (DSM)	Time to Develop 90% DSM	Time to Develop Critical Specific Optical Density (DSC)
Corlar 6113/Kevlar 49	12 at 20 min.	19.0 min.	>20.0 min.
Kerimid 601/Kevlar 49	15 at 15.5 min.	11.5 min.	>15.5 min.
Corlar 5105/Kevlar 49	74 at 11.5 min.	7.0 min.	>11.5 min.
Corlar 5134/Kevlar 49	519 at 3.5 min.	>3.0 min.	< .5 min.
Ironsides' 101/Kevlar 49	26 at 20 min.	17.5 min.	>20.0 min.
Skybond 703/Kevlar 49	21 at 20 min.	17.5 min.	>20.0 min.
Skybond 703/Kevlar 49*	9 at 20 min.	>18 min.	>20.0 min.
ICI's 300P/Kevlar 49	16 at 20 min.	14 min.	>20.0 min.
	20 at 20 min.	<12.5 min.	>20.0 min.
Pyralin 3003/Kevlar 49	11 at 20 min.	18.5 min.	>20.0 min.
Pyralin 3001/Kevlar 49	28 at 20 min.	17 min.	>20.0 min.
Corlar 4102/Kevlar 49	494 at 9 min.	<7 min.	< 2 min.

* No postcure

In the manufacture of the 300P/Kevlar 49 laminates, a pungent odor was encountered somewhat reminiscent of hydrogen sulfide and sulfur dioxide. The panels were not acceptable for Space Shuttle cabin applications because of this odor, and therefore, no further testing of this system was conducted.

The flammability requirement states that a laminate subjected to NHB 8060.1 test number 1 (upward propagation) shall self-extinguish within six inches in a 31% oxygen, 69% nitrogen, atmosphere at 69.0 ± 0.35 KPa (10.0 ± 0.5 psia). Testing was conducted by NASA, and the results are summarized in Table 2-V. The epoxy and polyester materials were severely damaged or completely consumed. The phenolics passed the test, however the polyimides were significantly more flame resistant. Several of the systems were evaluated in different laminate thicknesses. As the thickness was decreased, the distance to self-extinguish increased markedly.

The offgassing requirement states that a laminate shall not offgas more than 100 micrograms per gram of laminate of total organics, or more than 25 micrograms per gram of laminate of carbon monoxide when tested per NHB 8060.1, test number 7, classification 5, C, 1 in a 103 KPa (14.7 psia) air atmosphere. Testing was conducted by NASA, and the results are summarized in Table 2-VI. All of the materials evaluated met the above requirements, however, the polyester laminate's offgassing of total organics was almost twenty times higher than the next highest system.

The odor requirement for this program was that the laminates shall not have an offensive odor when tested per NHB 8060.1, test number 6, classification 5(b), in a 103 KPa (14.7 psia) air atmosphere. With the exception of the PES/Kevlar 49 laminates, all other systems met the odor requirements. Table 2-VII gives a summary of the quantitative data gathered on the test laminates.

The optical smoke density (DSM) requirement for this program was that the laminates were not to have (DSM) greater than 25 when tested per ASTM Special Technical Publication 422 (propane flame heat source) in a 103 KPa (14.7 psia) air atmosphere. The results obtained by NASA are summarized in Table 2-VIII. As shown, values obtained for the epoxy systems greatly exceeded the requirement.

Volatile condensible material (VCM) determinations were not an initial consideration for this program, since VCM testing is not performed on materials to be used inside the pressurized area of a spacecraft or inside any hermetically sealed container. The VCM testing was accomplished by NASA per SP-R-0022 in order to more fully characterize the candidate materials in the event that their potential usage might extend beyond the initial space cabin application. The VCM test is used as a means of selecting materials for applications near or adjacent to optical equipment that are exposed to the thermal vacuum atmosphere of space. The requirements for polymeric materials are that they have a maximum VCM of 0.1 percent and a total weight loss of 1.0 percent when tested in accordance with

SP-R-0022. General test requirements are a pressure of 10^{-6} Torr or less, a specimen temperature of 398 $\pm$ 1K (257 $\pm$ 2F), a cold surface temperature of 298K $\pm$ 1K (77F $\pm$ 2F), and a vacuum exposure time of 24 hours. The results obtained by NASA on the Phase I phenolic and polyimide materials are summarized in Table 2-IX. The other systems were not evaluated because of their failure to meet some other screening tests such as flammability, odor, etc. All of the tested materials met the VCM requirement, although there was considerable scatter between individual results. The two phenolic systems and the Kerimid 601 system exceeded the 1.0 percent total weight loss requirement. The Skybond 703 system passed the total weight loss requirement when fully post-cured, but exceeded the 1.0 percent maximum requirement when evaluated without postcure.

2.1.6 Material Costs

Costs on the various Phase I candidate materials were obtained from DuPont and are summarized in Table 2-X. Also included in Table 2-X are costs on the non-proprietary resin systems used in some of the prepregs. The costs were one of the considerations in the final selection for the material to be used throughout the remainder of the program.

2.1.7 Material Selection

The output of Phase I was the selection of a prepreg material which would then be optimized during Phase II for prepreg properties and cure cycle. The Phase II material selection was accomplished by a joint review of the Phase I data by Convair and NASA's technical monitor.

The polyethersulfone prepreg was eliminated from further consideration because of its failure to meet the odor requirements. It is believed that this is not an inherent problem with the polyethersulfone, but instead is one that was caused by the improper selection of solvent by the prepregger. Additional work, beyond the scope of this program could resolve that problem, and the polyethersulfone would be a fine candidate for further research on fire retardant Kevlar laminates.

The epoxy prepregs in this program were eliminated from further consideration because of their high burning rates and the large quantities of smoke produced by their burning. The polyester prepreg was eliminated for the same reasons as the epoxies. The phenolics and polyimides performed the best of all candidates, with the polyimides having somewhat better flammability characteristics. The Pyralin 3001 and Skybond 703 systems performed slightly better in the flammability testing than did the Kerimid 601 and Pyralin 3003. Although the polyimides were better than the phenolics, they are more costly prepregs and require more lengthy processing. In order to obtain a better comparison,

Table 2-IX. VCM Test Results

Material Trade Name	Resin	Reinforcement	Total Weight Loss, % Sample #1	Total Weight Loss, % Sample #2	Total Weight Loss, % Average	VCM, % Sample #1	VCM, % Sample #2	VCM, % Average
Corlar 6101-2/Kevlar 49	Ironsides 101 phenolic	Kevlar 49-181 style cloth	1.2175	1.0686	1.1430	0.0064	0.0140	*
Skybond 703/Kevlar 49**	Skybond 703 polyimide	Kevlar 49-181 style cloth	2.3333	2.3819	2.3576	0.0007	0.0051	0.0029
Skybond 703/Kevlar 49	Skybond 703 polyimide	Kevlar 49-181 style cloth	0.8356	0.8572	0.8464	-	-	-
Pyralin 3003/Kevlar 49	Pyralin 3003 polyimide	Kevlar 49-181 style cloth	0.7486	0.6905	0.7195	0.0163	0.0031	*
Pyralin 3001/Kevlar 49	Pyralin 4701 polyimide	Kevlar 49-181 style cloth	0.5621	0.5857	0.5739	0.0174	0.0047	*
Corlar 6113/Kevlar 49	Corlar 6113 phenolic	Kevlar 49-181 style cloth	1.2616	1.2035	1.2325	-	-	-
Kerimid 601/Kevlar 49	Kerimid 601 polyimide	Kevlar 49-181 style cloth	1.0700	1.0172	1.0436	0.0158	0.0083	*

* Due to wide variation of results, an average value of VCM percent is not applicable.

** No postcure.

Table 2-X. Cost Data - Phase I Candidate Materials*

Prepreg Material	Style 181 Fabric 97 cm (38 in.) $/m	($/yd.)	Style 120 Fabric 97 cm (38 in.) $/m	($/yd.)
ICI 300P/Kevlar 49	27.35	25.00	23.52	21.50
Pyralin 3001/Kevlar 49	27.19	24.85	22.97	21.00
Kerimid 601/Kevlar 49	28.88	26.40	24.89	22.75
Pyralin 3003/Kevlar 49	26.91	24.60	23.63	21.60
Corlar 5105/Kevlar 49	19.97	18.25	17.50	16.00
Corlar 6113/Kevlar 49	20.57	18.80	17.78	16.25
Ironsides' 101/Kevlar 49	20.57	18.80	17.78	16.25
Corlar 4102/Kevlar 49	19.97	18.25	17.50	16.00
Skybond 703/Kevlar 49	27.24	24.90	23.96	21.90
Corlar 5134/Kevlar 49	20.29	18.55	17.67	16.15

* For less than 457 meter (500 yd) orders, add the following:

1 roll (59 meters, 65 yards):	$2.30/m. ($2.10/yd.)
2 rolls (119 meters, 130 yards):	$1.15/m. ($1.05/yd.)
3 rolls (178 meters, 195 yards):	$.77/m. ($.70/yd.)
4 rolls (238 meters, 260 yards):	$.58/m. ($.53/yd.)
5 rolls (297 meters, 325 yards):	$.42/m. ($.46/yd.)
6 rolls (357 meters, 390 yards):	$.38/m. ($.35/yd.)
7 rolls (416 meters, 455 yards):	$.33/m. ($.30/yd.)

Raw Material

Kevlar 49-181 style fabric - 97 cm. (38 in.)	$12.58/m. ($11.50/yd.)
Kevlar 49-120 style fabric - 97 cm. (38 in.)	$7.88/m. ($7.20/yd.)
Skybond 703, 18.9 liter (5 gal.) pail	$9.39/Kg. ($4.26/lb.) plus freight
208 liter (55 gal.) drum	$8.24/Kg. ($3.82/lb.) plus freight
ICI 300P	$12.13/Kg. ($5.50/lb.) plus freight
Pyralin 4701	$22.46/liter ($85.00/gal.)

a laminate was prepared of Skybond 703/Kevlar 49 using the cure cycle of Table 2-II, but without the subsequent lengthy postcure. This panel was evaluated for flexural strength, density, flammability, offgassing, odor, and VCM. The non-postcured material passed all the tests with the exception of the total weight loss portion of the VCM test. Since VCM testing was beyond the basic scope of the program, the failure to meet the total weight loss limitation was not considered a hindrance to the selection of non-postcured Skybond 703/Kevlar 49 as the system to be evaluated for the remainder of the program. The Skybond 703 had some advantages in that it can be prepregged by numerous material suppliers and it is presently in use on production programs with other reinforcements. The non-postcured laminate had approximately the same strength as the postcured lamainate. Other considerations, such as solvent and moisture resistance and long term heat stability were beyond the scope of this program.

As a result of the above considerations, the Skybond 703/Kevlar 49 system was selected for further evaluation. Emphasis in Phases II and III were to be placed on non-postcured laminates.

2.2 Phase II: Optimization of Prepreg Laminating System

Phase II consisted of processing studies directed at optimization of the prepreg laminating system. Three batches of Skybond 703/Kevlar 49 (style 181 cloth) were obtained representing various stages of advancement and differing percentages of resin solids. Prepreg properties were evaluated, two variations were selected, and then panels were prepared to evaluate effects of cure cycle (time, temperature, heating and/or cooling rates), bleeder material configuration (type, form, amount), and postcure. Emphasis was placed on optimizing mechanical properties and minimizing processing costs. Tensile strength, flexure strength, resin content, void content, and density were used as the basis of optimization.

2.2.1 Prepreg Properties

Three yards of three different variations of Skybond 703/Kevlar 49 prepreg were obtained from DuPont on a best-efforts basis. The goals for these prepregs were as follows:

1. Similar to prepreg evaluated in Phase I, i.e., resin solids = 36.7%, flow = 37.4%, volatiles = 20.7%.
2. Resin solids = 45 $\pm$ 3%, flow $\leq$30%, volatiles = 15 to 25%.
3. Resin solids = 41 $\pm$ 2%, flow $\leq$ 25%, volatiles = 15 to 25%.

DuPont ran the prepreg on their production line and had difficulty in successfully varying the properties every three yards. The three rolls received had the following properties:

Lot 5826: Dry Resin Solids = 37.5%, flow = 43.6%, volatiles = 24.1%
Lot 5825: Dry Resin Solids = 45.8%, flow = 42.7%, volatiles = 24.0%
Lot 5824: Dry Resin Solids = 46.5%, flow = 52.1%, volatiles = 27.2%

The first roll was acceptable, but the percent flow on the other two rolls were excessively high. An attempt was made at Convair to reduce the flow and volatiles of the two rolls by additional advancement using DuPont's recommended cycle of 5 to 8 minutes at 383K (230F). Pieces of each roll were advanced per the above cycle, and the following results were obtained.

	Volatile Content, %		Flow, %	
Lot No.	Initial	Advanced	Initial	Advanced
5824	27.2	16.7	52.1	32.6
5825	24.0	13.9	42.7	34.9

The results were in the range of desired properties, therefore, the two rolls of prepreg were cut into sheets, hung in an oven and advanced as described above. All three lots of prepreg were then evaluated for resin solids, volatile content, resin flow, tack, drape, and gel time. The results obtained are summarized in Table 2-XI.

Lot 5825 was advanced excessively as noted by very low gel time and difficulty in washing resin out of prepreg during resin solids determination. This is attributed to slight temperature variations in the oven used for prepreg advancement. After prepreg evaluations showed slightly greater advancement than initial checks on small pieces, an oven temperature profile was performed. It showed temperature variations of 383 to 389K (230 to 240F).

Prepreg testing methods used at Convair are in some cases different than those used at DuPont (see 2.1.2). The Convair methods are described below.

2.2.2 Prepreg Test Methods

2.2.2.1 Volatile Content: Convair test method is essentially the same as the DuPont test method. However, polyimide prepregs are generally evaluated by testing 10 minutes at 450K (350F). It was felt that the DuPont test of 2.5 minutes at 672K (750F) may drive off portions of the resin or effect the weight of the Kevlar. No volatile content test method gives an absolute value for the volatiles

Table 2-XI. Phase II Prepreg Test Results on Skybond 703/Kevlar 49

Material Lot No.	Prepreg Condition	Test Lab	Volatile Content, wt.% 2.5 min. at 672K(750F)	Volatile Content, wt.% 10 min. at 450K(350F)	Dry Resin Content, wt.%	Wet Resin Content, wt.%	*Resin Flow, wt. % 10 min. at 505K(450F)	*Resin Flow, wt. % 15 min. at 450K(350F)	Gel Time sec.	Drape	Tack
5824	As-made	DuPont	27.2	-	46.5	-	52.1	-	-	-	-
5824	Advanced	Convair	-	13.8	-	42.1	-	28.5	187	pass	fail
5825	As-made	DuPont	24.0	-	45.8	-	42.7	-	-	-	-
5825	Advanced	Convair	-	11.9	-	48.0	-	30.9	114	pass	fail
5826	As-made	DuPont	24.1	-	37.5	-	43.6	-	-	-	-
5826	As-made	Convair	-	17.2	-	38.6	-	28.9	160	pass	pass

* 103 KPa (15 psi)

in a prepreg unless a simulated cure cycle is followed. The test acts as a qualitycontrol standard to evaluate batch-to-batch consistency in a product. The 450K (350F) test probably gives values closer to the amount of solvent evolved during cure than the 672K (750F) temperature used by DuPont, since the laminates were not going to be postcured in this program.

2.2.2.2 Resin Content: Three 10.2 by 10.2 cm. (4.0 by 4.0 in.) samples of prepreg shall be cut and weighed to the nearest 0.001 gm (W_1). Each sample shall be placed in a 30 ml tall-form beaker and shall be washed in clean, boiling methylethylketone (MEK) for two minutes, starting from the time the MEK starts to boil. The sample shall be washed three times in this manner and then forced air dried at 436K (325F) for 15 minutes and reweighed to the nearest 0.001 gm (W_2). The nonfiber content shall be calculated as follows:

$$\text{Nonfiber content} = \frac{W_1 - W_2}{W_1} \times 100$$

Wet resin content = nonfiber content - volatile content (from 2.1.3.1).

The average of three samples will be reported. For polyimides, dry at 450K (350F) for 15 minutes.

2.2.2.3 Resin Flow: Cut six pieces of Style 181 glass fabric 10.2 by 10.2 cm (4.0 by 4.0 in.) square for bleeders and two squares of Teflon coated glass fabric 10.2 by 10.2 cm (4.0 by 4.0 in.) for separators. Cut two pieces of 0.015 cm. (6 mil) Mylar film 10.2 by 10.2 cm (4.0 by 4.0 in.) for caul plate protectors. Weigh the above to the nearest 0.1 mg on an analytical balance. Cut two pieces of the material to be tested 5.1 by 5.1 (2.0 by 2.0 in.) and weigh, plus the separator and bleeder, to the nearest 0.1 mg. Crossply the two specimens and assemble between separators and bleeders in a preheated press. Cure for 15 minutes at temperature under 103 KPa (15 psig). Remove the crossply test specimens from the separator and bleeder. Weigh the separator plus bleeder to the nearest 0.1 mg. Calculate resin flow:

$$\text{Resin flow} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where W_1 = weight of glass fabric plus Teflon-coated glass fabric.

W_2 = weight of glass fabric, Teflon coated glass fabric, and specimens.

W_3 = weight of glass fabric plus Teflon-coated glass fabric after cure.

For polyimides, testing was conducted at 450K (350F).

2.2.2.4 Gel Time: Convair used the following DuPont test method for gel time. Cut at random enough 5.1 by 5.1 cm. (2.0 by 2.0 in.) specimens to make a stack 0.64 cm. (0.25 in.) thick. Insert the stack by means of a spatula onto the center of a preheated press platen. Close press as rapidly as possible applying enough pressure to squeeze resin onto the platen. Start timing as soon as pressure is applied. After 15 seconds, release pressure and open press. Using a thin flexible clean flat wooden stick, work resin back and forth in a rolling motion. Continue until resin gels. Gel point is point where resin ceases to be fluid and reaches a rubbery stage. As the gel point is approached, the resin will become stringy. When the resin is probed and the strings break, the gel point has been reached. For polyimide prepregs, gel test was conducted at 450K (350F).

2.2.2.5 Drape: Convair tested drape on the Skybond 703/Kevlar 49 prepregs per the method described in AMS 3894.

2.2.2.6 Tack: Convair tested tack on the Skybond 703/Kevlar 49 prepregs per the method described in AMS 3894.

2.2.3 Laminate Preparation

Prepreg lots 5824 and 5826 were selected for use in the processing study. The test panels were prepared as shown below using various bleeder and cure cycle variations. All panels consisted of twelve plies of prepreg.

Panel No.	Prepreg Lot	Prepreg To Bleeder Ratio	Cure Cycle*	Post Cure
703k-14	5826	1 to 1	No. 1	None
703k-15	5826	2 to 1	No. 1	None
703k-16	5826	1 to 1	No. 2	None
703k-17	5826	1 to 1	No. 3	None
703k-18	5826	4 to 1	No. 3	None
703k-20	5824	1 to 1	No. 1	Part of panel to be postcured
703k-21	5824	2 to 1	No. 1	Part of panel to be postcured
703k-22	5824	1 to 1	No. 2	Part of panel to be postcured
703k-22	5824	1 to 1	No. 3	Part of panel to be postcured

* Cure cycles detailed below. Post cure cycle same as that used in Phase I.

Cure Cycles:

No. 1: Apply full vacuum at room temperature (RT). Heat at 1.7K (3F) per minute to 389K (240F). Hold 30 min. at 389K (240F). Heat to 450K (350F) at 0.6K (1F) per min. and hold 1 hour. Cool to below 352K (175F) before removal from vacuum bag.

No. 2: Apply full vacuum at RT. Heat at 1.2K (2F) per min. maximum to 450K (350F), and hold 1 hour. Heat at same rate to 477K (400F), and hold 2 hours. Cool to below 352K (175F) before removal from vacuum bag.

No. 3: Apply full vacuum at RT. Heat at 1.7K (3F) per min. to 389K (240F), and hold for 30 min. Heat at 0.6K (1F) per min. to 405K (270F), and hold 30 min. Heat to 450K (350F) at 0.6K (1F) per min., and hold 1 hr. Heat at 0.6K (1F) per min. to 377K (400F), and hold 2 hours. Cool to below 352K (175F) before removal from vacuum bag.

2.2.4 Laminate Properties

Panels prepared from Lots 5824 and 5826 were machined into test specimens and evaluated for the following properties: tensile strength, flexure strength, specific gravity, resin content, and void content. A summary of all the results is presented in Table 2-XII.

The tensile strength specimen configuration and test method was per ASTM D638-71. The flexure specimen and test method was per ASTM D790-71. Specific gravity (or density) was obtained using the displacement method described in ASTM D792-66. Resin content was determined by weighing of the panels, determining weight per unit area, and subtracting out the Kevlar weight. Void contents were calculated using the weight percents of resin and fiber, the reported specific gravities for Kevlar-49 and Skybond 703 cured resin, and the determined average specific gravity for each panel.

A review of the test data in Table 2-XII shows that panels prepared from prepreg lots 5824 and 5826 had approximately the same range of flexure strengths, but varied widely in tensile strengths. The tensile strengths for panels made with lot 5826 were significantly higher than those made from 5824. The panels made from lot 5824 were significantly higher in resin content, slightly thicker, and had significantly lower failure loads. Void contents were approximately the same for all panels. Postcure of the panels made from lot 5824 failed to raise the tensile strengths to the level obtained with lot 5826. The lack of tack in lot 5824 after additional staging at Convair indicates that the prepreg surface of lot 5824 may have been advanced too far.

Therefore, the prepreg condition of lot 5826 would be more desirable for Phase III evaluation. The amount of bleeder was of little significance, at least in the range evaluated. This conclusion is based on comparison of results for Panels 703K-17 and 703K-18 which had prepreg-to-bleeder ratios of 1 to 1 and 4 to 1, respectively. There was no significant differences in tensile strength due to cure cycles, but cycle number 3 resulted in significantly higher flexure strength. This cycle has the slow heatup coupled with a maximum cure temperature of 477K (400F). The 477K (400F) cure is desirable since it allows the high boiling point n-methyl-2-pyrrolidone (NMP) to evolve. This reduces potential long-term aging problems and minimizes toxicity problems.

Table 2-XII. Summary of Phase II Testing

Panel No.	Specific Gravity	Flexural Strength MPa	Flexural Strength (ksi)	Tensile Strength MPa	Tensile Strength (ksi)	Resin Content Wt. %	Resin Content Vol. %	Void Content Vol. %
703K-14	1.216	184	(26.7)	569	(82.5)	26.5	28.2	
	1.198	183	(26.5)	581	(84.2)	26.3	28.0	
	1.204	183	(26.5)	601	(87.2)	26.3	28.0	
Av.	1.206	Av. 183	(26.6)	Av. 583	(84.6)	Av. 26.4	28.1	14.9
703K-15	1.224	199	(28.8)	598	(86.7)	26.9	28.6	
	1.233	210	(30.4)	601	(87.2)	26.7	28.4	
	1.233	202	(29.3)	608	(88.2)	27.1	28.8	
Av.	1.230	Av. 203	(29.5)	Av. 603	(87.4)	26.9	28.6	13.1
703K-16	1.231	196	(28.4)	630	(91.3)	26.7	28.4	
	1.236	194	(28.1)	615	(89.2)	26.1	27.8	
	1.243	210	(30.5)	609	(88.3)	26.4	28.1	
Av.	1.237	Av. 200	(29.0)	Av. 618	(89.6)	Av. 26.4	28.1	12.7
703K-17	1.201	216	(31.3)	625	(90.7)	26.4	28.1	
	1.204	216	(31.3)	632	(91.6)	26.6	28.3	
	1.203	210	(30.4)	552	(80.1)	26.6	28.3	
Av.	1.203	Av. 214	(31.0)	Av. 603	(87.5)	Av. 26.5	28.2	15.1
703K-18	1.201	243	(35.2)	604	(87.6)	26.1	27.8	
	1.204	231	(33.5)	585	(84.8)	26.0	27.7	
	1.204	236	(34.3)	594	(86.2)	25.9	27.6	
Av.	1.203	Av. 236	(34.3)	Av. 594	(86.2)	Av. 26.0	27.7	15.1
703K-20	1.230	223	(32.3)	453	(65.7)	29.9	31.7	
	1.224	219	(31.8)	437	(63.4)	30.0	31.9	
	1.222	225	(32.6)	442	(64.1)	30.0	31.9	
Av.	1.225	Av. 222	(32.2)	Av. 444	(64.4)	Av. 30.0	31.9	13.2

Table 2-XII. - Continued

Panel No.	Specific Gravity	Flexural Strength MPa	Flexural Strength (ksi)	Tensile Strength MPa	Tensile Strength (ksi)	Resin Content Wt. %	Resin Content Vol. %	Void Content Vol. %
703K-21	1.116	142	(20.6)	392	(56.9)	32.8	34.5	
	1.123	138	(20.0)	369	(53.5)	32.8	34.5	
	1.112	143	(20.8)	324	(32.5)	32.8	34.5	
Av.	1.117	Av. 141	(20.5)	Av. 328	(47.6)	Av. 32.8	34.5	20.7
703K-22	1.214	212	(30.8)	328	(47.6)	28.5	30.3	
	1.211	207	(30.0)	-	-	27.9	29.7	
	1.203	197	(28.5)	345	(50.0)	28.4	30.2	
Av.	1.209	Av. 205	(29.8)	Av. 337	(48.8)	Av. 28.3	30.1	14.4
703K-23	1.223	196	(28.4)	212	(30.7)	28.7	30.5	
	1.233	218	(31.6)	189	(27.4)	28.6	30.4	
	1.243	226	(32.8)	195	(28.3)	28.7	30.5	
Av.	1.233	Av. 213	(30.9)	Av. 199	(28.8)	Av. 28.7	30.5	12.8
703K-20 postcured		228	(32.3)	310	(45.0)			
		232	(33.7)					
		263	(38.1)					
		Av. 239	(34.7)					
703K-21 postcured		133	(19.3)	265	(38.4)			
		146	(21.2)					
		141	(20.4)					
		Av. 140	(20.3)					
703K-22 postcured		207	(30.0)	229	(33.2)			
		208	(30.1)					
		194	(28.2)					
		Av. 203	(29.4)					
703K-23 postcured		220	(31.9)	266	(38.6)			
		221	(32.1)					
		215	(31.2)					
		Av. 219	(31.7)					

2.3 Phase III: Panel Fabrication and Data Generation

The objective of Phase III was to fabricate and deliver to NASA a series of panels made from the selected prepreg, Skybond 703/Kevlar 49, and to fully evaluate the mechanical and physical properties of cured laminates prepared from the selected system. The panels were to be fabricated from various combinations of the selected resin prepregged on Kevlar 49 reinforcement of the following forms: style 181 fabric, style 120 fabric, and style 1215 mat.

2.3.1 Prepreg Properties

The following materials were obtained for Phase III:

1. Style 181 Kevlar - 49 fabric reinforced Skybond 703
2. Style 120 Kevlar - 49 fabric reinforced Skybond 703
3. Style 1215 Kevlar - 49 mat reinforced Skybond

The desired properties for the above items were wet resin content of 37 ± 2 percent, resin flow of 40 ± 5 percent [test: 10 min./103 KPa (15 psi)/505K (450F)] volatile content of 21 ± 4 percent [test: 2.5 min. at 672K (750F)], gel time of 150 seconds minimum. For the 120 style fabric, it was anticipated that resin content would be higher. There was no experience in prepregging of style 1215 mat at the initiation of this program.

Phase III prepreg properties are summarized in Table 2-XIII. The resin content of the 1215 mat was very non-uniform. Essentially identical results were obtained at both facilities. when tests were performed using the same test methods. In Phase II, it was found that volatile content measured by the DuPont test were approximately 40 percent higher than when measured by the Convair method. This was confirmed by Phase III testing. Numerically, the dry resin content and wet resin content is approximately the same. There was a little difference in flow depending on the test cycle, but most of the differences noted in the Phase II testing were obviously a result of variation in the prepreg.

The initial concept for using mat was to be able to use a thick layer of prepreg as a core for a part having normal reinforcement acting as the facings. The use of mat would result in lowered production costs, if the properties were not adversely affected. The appearance of style 1215 mat prepreg was like that of a wet chamois. The mat soaked up resin very rapidly, like a sponge, and the weight caused necking of the prepreg on the production coater. The initial width of the mat was 91 cm. (36 in.), and this had necked down to approximately 64 cm. (25 in.). The mat is not considered a suitable production material, and it has recently been learned that the DuPont Textile Division will no longer supply it.

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Table 2-XIII. Phase III Prepreg Test Results on Skybond 703/Kevlar 49

Material Lot No.	Reinforcement Style	Test Lab	Volatile Content, wt. % 2.5 min. at 672K(750F)	Volatile Content, wt. % 10 min. at 450K(350F)	Dry Resin Content, wt. %	Wet Resin Content, wt. %	*Resin Flow, wt. % 10 min. at 505K(450F)	*Resin Flow, wt. % 15 min. at 450K(350F)	Gel Time, sec.
6092	120 fabric	DuPont	32.8	-	44.7	-	44.3	-	-
6094	1215 mat	DuPont	43.8	-	76.6	-	71.0	-	-
6093	181 fabric	DuPont	25.0	-	36.7	-	43.2	-	-
6093	181 fabric	Convair	24.8	18.1	35.8	37.1	46.1	44.7	138

(103 KPa (15 psi)

2.3.2 Test Panels

All panels for Phase III were cured using vacuum bag pressure and the following cure cycle. Panels were not postcured.

> Cure Cycle: Apply full vacuum at RT. Heat at 1.7K (3F) per minute to 389K (240F) and hold for 30 minutes. Heat at 0.6K (1F) per minute to 405K (270F), and hold for 30 minutes. Heat to 450K (350F) at 0.6K (1F) per minute, and hold for 60 minutes. Heat at 0.6K (1F) per minute to 477K (400F), and hold for 2 hours. Cool to below 352K (175F) before removal from vacuum bag.

Eight panels, each 30.5 by 30.5 cm. (12.0 by 12.0 in.) were prepared per each of the configurations below. Of these, five panels of each were shipped to NASA's Lyndon B. Johnson Space Center, while the other three were evaluated by Convair for mechanical and physical properties. The panel configurations were:

1. Two inner plies of Kevlar 49 style 181 fabric prepreg faced front and back with one ply of Kevlar 49 style 120 prepreg.

2. Five inner plies of Kevlar 49 style 181 fabric prepreg faced front and back with one ply of Kevlar 49 style 120 prepreg.

3. Two inner plies of Kevlar 49 style 1215 mat faced front and back with one ply of Kevlar 49 style 120 fabric prepreg.

4. Four inner plies of Kevlar 49 style 1215 mat faced front and back with one ply of Kevlar 49 style 120 fabric prepreg.

There were no problems in preparing the panels which combined the style 120 and style 181 Kevlar 49 prepregs. However, because of the very high resin content in the style 1215 mat prepreg, there were some problems encountered in making the panels which combined the style 120 fabric with the style 1215 mat. These problems were general poor handling (tendency to distort) and tendency to wrinkle during cure.

Panel evaluations were to include the following properties: tensile strength, tensile modulus, compressive strength, compressive modulus, flexural strength, flexural modulus, interlaminar shear strength, resin content, density, dielectric constant, and Charpy impact strength.

2.3.3 Test Methods

Resin content and density (specific gravity) for Phase III laminates were measured identically to that previously described for Phase I and Phase II panels.

Tensile strength and modulus were determined per ASTM D638-71, and strain measurements were obtained using a linear variable differential transformer (LVDT) type of extensometer. Compressive strength and modulus were determined per ASTM D695-69, and strain measurements were obtained using a LVDT type of compressometer clipped across the specimen edges. Flexural strength and modulus were determined per ASTM D790-71 using a span-to-depth ratio of 25 to 1, 0.635 cm. (0.25 in.) diameter rods for loading head, and supports, and head speed of 0.127 cm. (0.050 in.) per minute. Flexural deflections were obtained using a LVDT type deflectometer, with movement measured directly under the loading head. Interlaminar shear strength was determined per ASTM D2344 using a span-to-depth ratio of 4 to 1, 0.160 cm. (0.063 in.) diameter rods for loading head and supports, and head speed of 0.127 cm. (0.050 in.) per minute. Dielectric constant was measured per ASTM D150-70 using 15.2 cm. (6.00 in.) diameter discs as the test specimens. Charpy impact strength was to be determined per ASTM D256-66. Because of difficulties in test, final impact testing was conducted using a Sonntag Universal Impact Machine, Model SI-1, which is generally used for evaluating metals.

2.3.4 Test Results

Phase III mechanical and physical property test results are shown in Tables 2-14 to 2-20. Table 2-XIV summarizes the measured density (specific gravity) and resin contents for the four different panel configurations. As noted earlier, the 1215 mat prepreg was extremely difficult to handle, was very non-uniform, and was quite wrinkled. The two panel configurations containing plies of 1215 mat were resin rich. This was a result of the very high, non-uniform resin content in the prepreg and the unknown bleeding ability during vacuum bag cure. The resin content calculations were based on a Kevlar weight per unit area of 0.005 gm/cm^2 per ply of 1215 mat, which was obtained by washing the resin from a piece of prepreg, drying, and weighing the resulting Kevlar. However, this number is suspect because of the great distortion of the 1215 during prepregging which may have resulted in non-uniform amounts of Kevlar per unit of area. Therefore, the resin contents reported in Table 2-XIV may be suspect for the configurations containing 1215 mat.

Tensile and flexure strength and modulus data is summarized in Table 2-XV. As anticipated, the thin specimens of this phase of the program were difficult to test, particularly in flexure, short beam shear, compression, and impact. In the case of the flexure specimens, the span-to-depth ratio was raised from 16 to 1 (used earlier in Phase I and II testing) to 25 to 1. For the two configurations containing combinations of Style 120 and Style 181 Kevlar cloth, the tensile strengths were similar to results obtained in Phase II, and are typical of results obtained with high fiber volume, low resin content laminates. The flexure strength data is slightly better than that obtained in Phase II for laminates made only with Style 181 prepreg. Excellent tensile modulus values were

Table 2-XIV. Densities and Resin Contents for Kevlar 49/Skybond 703 Specimens

Panel Configuration		Density gm/cc		Resin Content wt. %
Five layers of style 181 prepreg faced top and bottom with one ply of style 120 prepreg		1.241		25.0
		1.231		25.2
		1.231		28.4
		1.231		25.8
		1.233		25.2
				26.4
				25.2
	Average	1.233	Average	25.9
Two layers of style 181 prepreg faced top and bottom with one ply of style 120 prepreg		1.299		25.2
		1.300		25.3
		1.307		25.4
		1.300		25.4
		1.303		25.4
				23.5
				23.8
	Average	1.302	Average	24.9
Two layers of style 1215 prepreg faced top and bottom with one ply of style 120 prepreg		1.149		53.0
		1.143		59.2
		1.155		58.8
		1.142		59.9
		1.149		56.9
	Average	1.148	Average	57.6
Four layers of style 1215 prepreg faced top and bottom with one ply of style 120 prepreg		1.153		64.1
		1.143		60.8
		1.142		61.9
		1.147		63.6
		1.182		63.9
	Average	1.153	Average	62.5

Table 2-XV. Tensile and Flexure Data on Kevlar 49/Skybond 703 Specimens

Panel Configuration	Tensile Strength		Tensile Modulus		Flexure Strength		Flexure Modulus	
	MPa	(ksi)	GPa	(msi)	MPa	(ksi)	GPa	(msi)
Five layers of style 181	570.	(82.7)	36.	(5.2)	211.	(30.6)	20.	(2.9)
prepreg faced top and bottom	530.	(76.8)	35.	(5.0)	248.	(35.9)	22.	(3.2)
with one ply of style 120	601.	(87.1)	35.	(5.1)	220.	(31.9)	20.	(2.9)
prepreg	559.	(81.1)	32.	(4.7)	232.	(33.6)	22.	(3.2)
	565.	(82.0)	35.	(5.0)	245.	(35.6)	22.	(3.2)
Average	565.	(81.9)	35.	(5.0)	231.	(33.5)	21.	(3.1)
Two layers of 181 style	632.	(91.7)	41.6	(6.03)	259.	(37.5)	-	-
prepreg faced top and bottom	681.	(98.8)	40.3	(5.85)	261.	(37.9)	-	-
with one ply of 120 style	659.	(95.6)	39.5	(5.73)	259.	(37.5)	-	-
prepreg	652.	(94.6)	43.1	(6.25)	285.	(41.3)	13.7	(2.00)
	619.	(89.8)	39.9	(5.78)	259.	(37.5)	12.7	(1.84)
Average	649.	(94.1)	40.9	(5.93)	264.	(38.3)	13.2	(1.92)
Two layers of 1215 style	279.	(40.5)	17.4	(2.53)	207.	(30.0)	13.0	(1.89)
prepreg faced top and bottom	284.	(41.2)	17.4	(2.53)	225.	(32.6)	15.4	(2.23)
with one ply of 120 style	252.	(36.5)	17.9	(2.60)	205.	(29.8)	12.7	(1.84)
prepreg	269.	(39.0)	15.7	(2.27)	177.	(25.6)	26.6	(3.86)
	258.	(37.4)	16.0	(2.32)	178.	(25.8)	21.6	(3.13)
Average	268.	(38.9)	16.9	(2.45)	199.	(28.8)	17.9	(2.59)
Four layers of 1215 style	387.	(56.2)	22.5	(3.26)	170.	(24.6)	14.4	(2.09)
prepreg faced top and bottom	405.	(58.7)	23.0	(3.33)	179.	(26.0)	10.2	(1.48)
with one ply of 120 style	420.	(60.1)	23.0	(3.33)	148.	(21.4)	10.1	(1.46)
prepreg	412.	(59.7)	24.6	(3.57)	156.	(22.6)	11.2	(1.63)
	372.	(53.9)	27.1	(3.93)	170.	(24.6)	10.7	(1.55)
Average	398.	(57.7)	24.0	(3.48)	164.	(23.8)	11.3	(1.64)

obtained, however difficulty was encountered in getting good flexure modulus data for the very thin laminates. This is a result of very high total deformation and large shear deformation contribution. The laminate configurations containing combinations of Style 120 Kevlar cloth and Style 1215 Kevlar mat gave considerably lower tensile and flexure strength and modulus values. This is primarily a result of the excessively high resin content of the latter laminate configurations. Flexure modulus values should be considered suspect for all the laminates.

Compression test results on the four laminate configurations is given in Table 2-XVI. In most cases, failure occurred in edge buckling or brooming rather than compression. In only four specimens did failure occur within the specimen's reduced section, and most of these occurred in the specimen configuration which was anticipated to be the weakest. The compression strengths were very low, and therefore a thicker panel was prepared consisting of three stacked modules each containing five plies of 181 style Kevlar prepreg faced top and bottom with one ply of 120 style Kevlar prepreg. This panel was then machined into test specimens which were then tested at RT. The results are given in Table 2-XVII. Failures occurred properly within the test specimens. The results are significantly greater than those for a single module of the same layup configuration (see Table 2-XVI). However, even the results on the thick laminate gave strength values lower than that normally reported for Kevlar reinforced epoxy laminates, and this is attributed to poor fiber/resin interfacial strength. This is consistent with the high tensile and low flexure values shown in Table 2-XV.

Since there has been a great deal of effort directed to improving the compression and shear properties of Kevlar reinforced epoxies and little or no effort to do the same for Kevlar/polyimides, the values of Table 2-XVII are not reasonable. Data reported in DuPont's "Kevlar" 49 Data Manual gives a typical ultimate compression strength for a 0.318 cm (0.125 in.) thick Kevlar reinforced epoxy laminate as 172.4 MPa (25 ksi). A typical value of 137.9 MPa (20 ksi) is reported for a Kevlar reinforced polyimide laminate. This data is for autoclave cured laminates and would be expected to give higher values than vacuum bag cured laminates, such as those prepared in this program. The DuPont Data Manual reports a compressive modulus of 31.0 GPa (4.5 msi) and 27.6 GPa (4.0 msi) for the epoxy and polyimide laminates, respectively. This agrees very well with the average value of 30.3 GPa (4.4 msi) obtained in this program. The data on the thin laminates is suspect because of the improper mode of failure and the inaccuracy associated in the thickness measurements of such laminates.

Similar to the problems in flexure and compression, short beam shear testing is extremely difficult on very thin specimens. Raising the span-to-depth ratio on the specimens would change the failure mode from shear to flexure and

Table 2-XVI. Compression Strength Results of Kevlar 49/Skybond 703 Specimens

Panel Configuration	Compression Strength MPa	(ksi)	Compression Modulus GPa	(msi)	Remarks
Five layers of 181 style	72.4	(10.5)	25.5	(3.70)	failed by buckling of end
prepreg faced on top and	66.2	(9.6)	25.9	(3.76)	"
bottom with one ply of	71.7	(10.4)	27.2	(3.95)	"
120 style prepreg	62.7	(9.1)	27.3	(3.96)	"
	38.6	(5.6)	21.7	(3.14)	failed in test section
	73.1	(10.6)			
Average	64.1	(9.3)	25.9	(3.75)	
Two layers of 181 style	46.9	(6.8)	22.6	(3.28)	failed by buckling of end
prepreg faced on top and	49.6	(7.2)	23.9	(3.46)	"
bottom with one ply of	35.9	(5.2)	24.5	(3.56)	"
120 style prepreg	59.3	(8.6)	26.5	(3.84)	"
	53.8	(7.8)	17.6	(2.56)	"
	31.7	(4.6)	15.7	(2.28)	"
Average	46.2	(6.7)	21.8	(3.16)	
Two layers of 1215 style	41.4	(6.0)	7.52	(1.09)	failed by buckling of end
prepreg faced on top and	42.7	(6.2)	8.69	(1.26)	"
bottom with one ply of	44.8	(6.5)	8.27	(1.20)	failed in test section
120 style prepreg	39.3	(5.7)	8.21	(1.19)	"
	42.1	(6.1)	8.21	(1.19)	failed by buckling of end
	44.8	(6.5)	8.07	(1.17)	failed in test section
Average	42.7	(6.2)	8.14	(1.18)	
Four layers of 1215 style	38.6	(5.6)	1.15	(1.67)	failed by buckling of end
prepreg faced on top and	34.5	(5.0)	1.14	(1.66)	"
bottom with one ply of	33.8	(4.9)	1.12	(1.62)	"
120 style prepreg	35.9	(5.2)	1.23	(1.79)	"
	31.7	(4.6)	1.08	(1.56)	"
	35.2	(5.1)	1.15	(1.67)	"
Average	35.2	(5.1)	1.14	(1.66)	

Table 2-XVII. Compression Test Results on Thick Skybond 703/Kevlar 49 Specimens

Configuration	Ultimate Compression Strength MPa	(ksi)	Compression Modulus GPa	(msi)
Three parallel stacked modules each containing five layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg	106.6	(13.5)	-	-
	99.5	(12.6)	28.4	(3.6)
	88.4	(11.2)	35.5	(4.5)
	94.7	(12.0)	33.9	(4.3)
	97.9	(12.4)	39.5	(5.0)
Average	97.1	(12.3)	34.7	(4.4)

would defeat the purpose of running short beam shear tests. The approach adopted was to use smaller diameter supports and loading head for the thin specimens. The results obtained are summarized in Table 2-XVIII. The results are lower than the 27.6 to 68.9 MPa (4.0 to 10.0 ksi) reported in DuPont's Data Manual for typical Kevlar/epoxy laminates, but are higher than 15.2 MPa (2.2 ksi) reported for typical Kevlar/polyimide systems.

Dielectric constant testing was performed on the four laminate configurations using 15 cm (6.0 in.) diameter discs and testing at 10^3 Hz at RT per ASTM D150-70. The results are tabulated in Table 2-XIX. All the results exceeded the initial program goal of 1.9 minimum.

Charpy impact specimens conforming to ASTM D256-66 were machined from each of the four laminate configurations and shipped to Delsen Laboratories for test. The first attempt to fail the specimens was unsuccessful. When the striker hit the specimens, they deformed and were thrown from the test fixture. Another attempt was made to fail the specimens by impacting them after clamping the specimens between Plexiglas strips. This too failed to break the specimens. Convair then prepared impact specimens from the thick laminate prepared for compression testing, consisting of three stacked modules each containing five plies of 181 style Kevlar faced front and back with one ply of 120 Style Kevlar. Delsen's impact tester for plastics has a limited energy range, and therefore the thicker specimens were evaluated at Convair in a Sonntag Universal Impact Machine, Model SI-1. This machine is usually used for evaluating metals, because of the high energy levels associated with the various pendulum settings. The specimens failed to break.

Delamination and distortion occurred as shown in Figure 2-1. The recorded values at which this occurred are given in Table 2-XX. Two different pendulum settings were evaluated, but there was no difference in end result. There is no way of obtaining an impact strength value for the thick laminate using conventional impact specimens. The type of damage which occurred during impact testing confirms the excellent resistance of Kevlar 49 laminates to impact. The mode of failure confirms the poor interlaminar behavior of the Skybond 703/Kevlar 49 system.

A summary is given in Table 2-XXI of all the mechanical and physical properties for the Skybond 703/Kevlar 49 configuration consisting of modules of five plies of style 181 prepreg faced front and back with one ply of style 120 prepreg. Table 2-XXI compares the average test values with the initial program goals. There was no comparison possible on Charpy impact strength because it was not possible to properly fail the Skybond 703/Kevlar 49. The only goal which was not met by the Skybond 703/Kevlar 49 system was compression strength. As noted earlier, low compression strength is one of the problems which have been encountered with Kevlar laminates even when used in conjunction with epoxy resins. Since the program emphasis was on non-structural applications, the low compression strength is not a major concern. Table 2-XXII is a detailed fabrication procedure summary for all of the Phase III laminate configurations.

Table 2-XVIII. Short Beam Shear Strength of Kevlar 49/Skybond 703 Specimens

	Short Beam Shear Strength		
Panel Configuration	MPa	(ksi)	Remarks
Five layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg	16.6	(2.41)	-
	15.1	(2.19)	-
	16.6	(2.41)	-
	14.1	(2.05)	-
	15.7	(2.28)	-
Average	15.7	(2.27)	
Two layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg	24.5	(3.56)	-
	24.8	(3.59)	-
	25.9	(3.75)	-
	19.7	(2.85)	-
	22.1	(3.20)	-
Average	23.4	(3.39)	
Two layers of 1215 style prepreg faced on top and bottom with one ply of 120 style prepreg	28.0	(4.06)	-
	-	-	failed in handling
	29.6	(4.29)	-
	24.8	(3.60)	-
	22.5	(3.26)	
Average	26.2	(3.80)	
Four layers of 1215 style prepreg faced on top and bottom with one ply of 120 style prepreg	19.2	(2.78)	-
	18.4	(2.67)	-
	18.2	(2.64)	-
	20.6	(2.99)	-
	19.7	(2.85)	-
Average	19.2	(2.79)	

Table 2-XIX. Dielectric Constant Measurements of Kevlar 49/Skybond 703 Specimens

Panel Configuration		Dielectric Constant
Five layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg		3.89
		3.65
		3.62
		4.02
		3.94
	Average	3.83
Two layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg		3.25
		3.56
		3.46
		3.46
		3.42
	Average	3.43
Four layers of 1215 style prepreg faced on top and bottom with one ply of 120 style prepreg		3.94
		4.20
		4.03
		3.89
		3.24
	Average	3.86
Two layers of 1215 style prepreg faced on top and bottom with one ply of 120 style prepreg		3.58
		3.28
		Non-uniform thickness; no measurement possible
		3.41
		3.85
	Average	3.53

Table 2-XX. Impact Test Results of Skybond 703/Kevlar 49 Specimens

Configuration	Pendulum Setting	Failure Force	Failure Mode
Three parallel stacked modules each containing five layers of 181 style prepreg faced on top and bottom with one ply of 120 style prepreg	135.6 joules (100 ft-lb)	29.8 joules (22 ft-lb)	improper, delamination
	325.4 joules (240 ft-lb)	29.8 joules (22 ft-lb)	"
	135.6 joules (100 ft-lb)	25.8 joules (19 ft-lb)	"
	135.6 joules (100 ft-lb)	28.5 joules (21 ft-lb)	"
	135.6 joules (100 ft-lb)	25.8 joules (19 ft-lb)	"

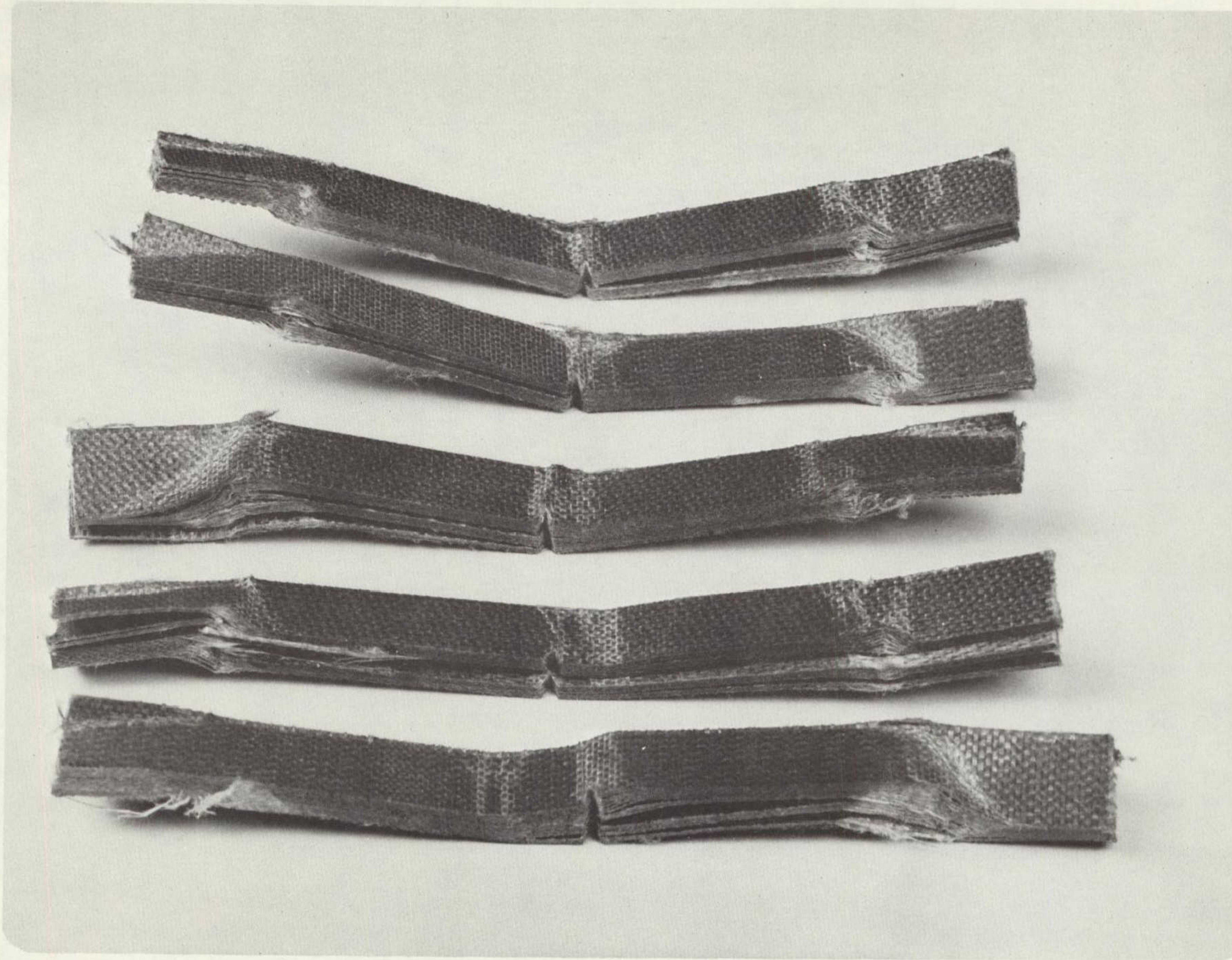

Figure 2-1. Failed Skybond 703/Kevlar 49 Impact Specimens

Table 2-XXI. Program Goals and Attainments-Material: Skybond 703/Kevlar 49*

Property	Program Goal	Average Test Value**	Referenced Table
Flammability	self-extinguish within 15.2 cm (6 in.)	self-extinguishes in 1.27 cm (0.5 in.) for 0.130 cm; (0.051 in.) thick laminate	2-5
Offgassing	100 mgm/gm total organics	3.0 mgm/gm total organics	2-6
	25 mg/g carbon monoxide	0.4 mgm/gm carbon monoxide	2-6
Odor	Shall not be offensive	Not offensive	2-7
Optical Smoke Density	DSM not to exceed 25	9 at 20 min.	2-8
Tensile strength	379 MPa (55 ksi) minimum	565 MPa (81.9 ksi)	2-15
Tensile modulus	21.4 GPa (3.1 msi) minimum	35 GPa (5.0 msi)	2-15
Compressive strength	124 MPa (18 ksi) minimum	97 MPa (12.3 ksi)	2-17
Compressive modulus	21.4 GPa (3.1 msi) minimum	34.7 GPa (4.4 msi)	2-17
Flexural strength	207 MPa (30 ksi) minimum	231 MPa (33.5 ksi)	2-15
Flexural modulus	20.7 GPa (3.0 msi) minimum	21 GPa (31. msi)	2-15
Interlaminar Shear Strength	15.2 MPa (2.2 ksi) minimum	15.7 MPa (2.27 ksi)	2-18
Resin content	Measure and report	25.9% by wt.	2-14
Density	Measure and report	1.233 gm/cc	2-14
Dielectric constant	1.9 minimum	3.83	2-19
Charpy impact strength	21.0 joules/cm^2 (100 ft-lb/in^2)	could not get proper failure	2-20

* No postcure.

**Mechanical and physical properties based on configuration consisting of module(s) with five plies of style 181 Kevlar 49 prepreg faced front and back with one ply of style 120 Kevlar 49 prepreg.

Table 2-XXII. Summary Material and Fabrication Chart

Detailed Information	Five plies 181 prepreg faced top & bottom with one ply 120 prepreg	Two plies 181 prepreg faced top & bottom with one ply 120 prepreg	Two plies of 1215 prepreg faced top & bottom with one ply 120 prepreg	Four plies of 1215 prepreg faced top & bottom with one ply 120 prepreg
Reinforcement	Kevlar 49			
Source of Reinforcement	DuPont de Nemours & Co.			
Resin	Skybond 703			
Resin Source	Monsanto Co.			
Resin Type	Condensation Polyimide			
Mold Material	Dapcocast 38-3 Silicone Rubber			
Mold Cure Cycle	24 hr. RT, 2 hr. 355K (180F), 4 hr. 477K (400F)			
Type of Cure	Vacuum Bag			
Type of Bagging	Envelope			
Bagging Material	High Temperature Resistant Nylon Film			
Cure Cycle	Apply full vacuum at RT. Heat at 1.7K (3F) per minute to 389K (240F) and hold for 30 minutes. Heat at 0.6K (1F) per minute to 405K (270F), and hold for 30 minutes. Heat to 450K (350F) at 0.6K (1F) per minute, and hold for 60 minutes. Heat at 0.6K (1F) per minute to 477K (400F), and hold for 2 hours. Cool to below 352K (175F) before removal from vacuum bag.			
Post Cure Cycle	None			
Bleeder Material	6 plies 7581 style glass fabric	3 plies 7581 style glass fabric	5 plies 7581 style glass fabric	9 plies 7581 style glass fabric
Current Material Costs	See Table 2-10			

2.4 Space Shuttle Hand Rail Specimens

As an additional task to the contract, Convair was required to fabricate two prototype Space Shuttle mobility and translation handrail segments per Figure 2-2. The rails were to use Kevlar as the principal fiber reinforcement and were to weigh no more than 0.15 pounds per foot nominal. The resin was to be flight acceptable per NHB 8060.01A. Maximum design load was 136 kg (300 lb) and ultimate design load was 204 kg (450 lb) where the load was applied centrally at a single point and perpendicular to the rail axis.

A preliminary design activity was conducted using a combination of Kevlar fabric and unidirectional Kevlar yarn, as well as a combination of Kevlar fabric and unidirectional T-300 graphite fibers. Two designs resulted, i.e. (1) consisting of six layers of style 181 Kevlar 49 fabric prepreg with a 0.178 cm (0.070 in.) thick layer of unidirectional Kevlar 49 tape prepreg along both sidewalls, sandwiched between the third and fourth wrap of Kevlar fabric, and tapering off at the tangency points, and (2) consisting of six layers of style 181 Kevlar 49 fabric prepreg with a 0.076 cm (0.030 in.) thick layer of unidirectional T-300 graphite prepreg tape along both sidewalls, sandwiched between the third and fourth wrap of Kevlar fabric, and tapering off at the tangency points.

Since the Shuttle's mobility and translation handrails are considered to be structural hardware, the use of an epoxy resin was preferred over the use of Skybond 703 condensation polyimide. Hexcel's F-164 fire retardant epoxy resin meets the requirements of NHB8060.1A, and is being used by Rockwell International for various Space Shuttle applications. Therefore, Kevlar 49/F-164 was selected for use in preparing the prototype handrails. Hexcel had previously prepregged the F-164 on Kevlar fabric, but had no experience with prepregging the material on either unidirectional Kevlar yarn or unidirectional graphite tow. Hexcel has no unidirectional tape prepregging line, so they prepared a small quantity of unidirectional Kevlar prepreg by drum winding 1420 denier, 1000 filament Kevlar yarn. For expediency purposes, the graphite/epoxy prepreg used for one of the prototypes was Narmco's T-300/5208. The 5208 is a high temperature resistant epoxy resin commonly used in advanced composites, but is not qualified to NHB8060.1A.

Three handrail prototypes were fabricated and delivered to NASA's Lyndon B. Johnson Space Center. Tube wall thicknesses were based on predesign hand calculations using typical Kevlar/epoxy and graphite/epoxy properties and load requirements supplied by NASA-JSC. Outer tube dimensions were those supplied on a NASA-JSC (see Figure 2-2). The first design configuration was an all-Kevlar tube prepared using six plies of the Kevlar cloth, 0.13 to 0.14 cm (0.050 to 0.055 in.), and incorporating twelve plies of the unidirectional Kevlar/F-164 tape (approximately 0.132 cm) on the flat sides of the handrail between the third and fourth wrap of the Kevlar cloth. This might be used as a preliminary test article to give a bench mark as to the strength of the two design concepts.

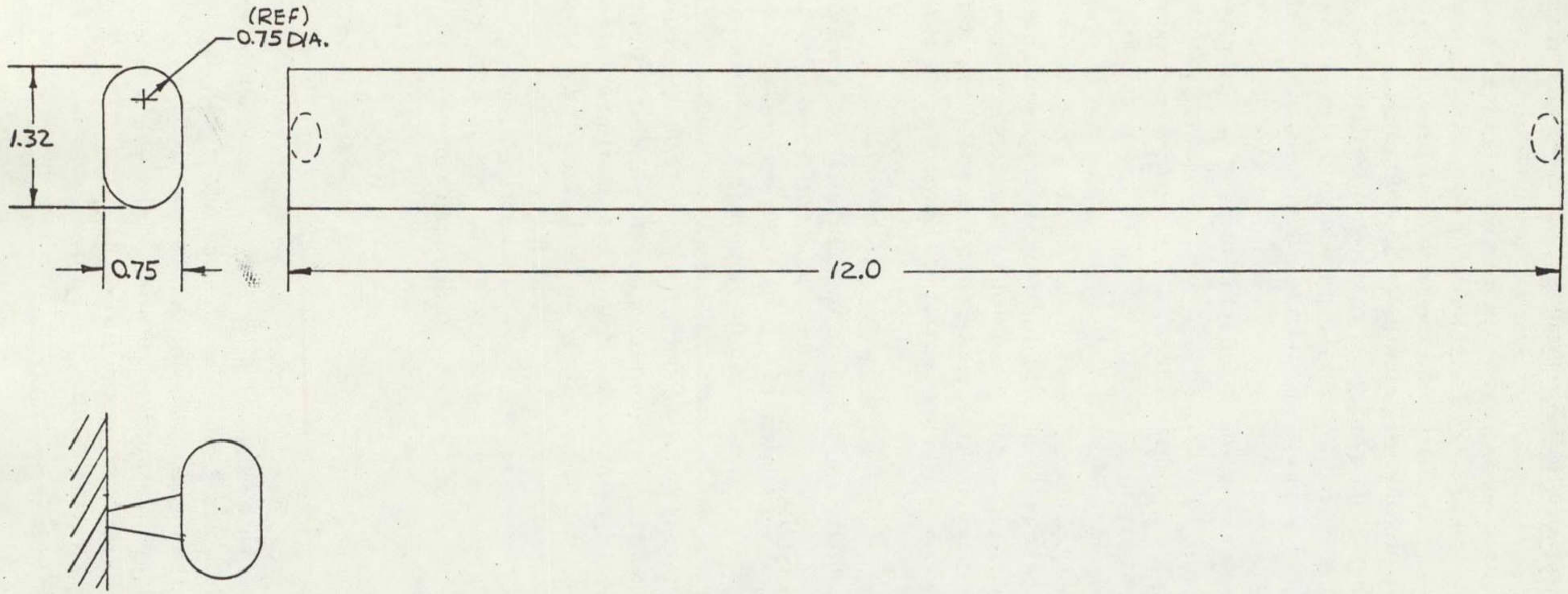

Figure 2-2. Translation and Mobility Manhold Segment

The handrail segments were prepared using Convair's tube making process. A male aluminum mandrel was machined to the outer dimensions of the final handrail segments. This was coated with a mold release agent, a thick epoxy gel coat, and approximately 0.64 cm (0.25 in.) thickness of glass cloth reinforced epoxy. The layup was cured and provided the final female tool. A smaller aluminum mandrel of the proper shape was fabricated, encapsulated with heat shrinkable rubber tubing, and then wrapped with one of the proposed layups. This assembly was then slipped into the fiberglass female tool, sealed, and placed in an autoclave for cure. Pressure application was applied by autoclave gas flowing through several holes in the male mandrel and expanding the rubber tubing. The pressure of 690 KPa (100 psig) was applied at the start of the cycle, and the part was heated 1.7°K (3°F) per minute to 450K (350F) and held for 90 minutes at temperature. Each tube was made by the above process.

Some difficulties were encountered with the female fiberglass tooling. Several voids behind the gel coat collapsed during cure and caused some of the reinforcement to bulge locally. This would not be a problem in a production cycle, since in that case a more expensive split metal female tool would be justified. The appearance of the handrail segments were improved by localized light sanding and subsequent coating of the tubes with epoxy resin. Figure 2-3 shows the three prototype handrail segments prepared by Convair as part of this program.

Weights of the two baseline designs were almost exactly 0.15 pounds per foot. The combination of Kevlar fabric and graphite tape gave a more uniform cross section than did the all Kevlar design. This was a result of smaller amounts of bulk needed between the third and fourth wraps of Kevlar fabric, since unidirectional T-300 has a much higher modulus than unidirectional Kevlar 49. The composite handrail is a very promising application where weight savings might be accomplished on Space Shuttle.

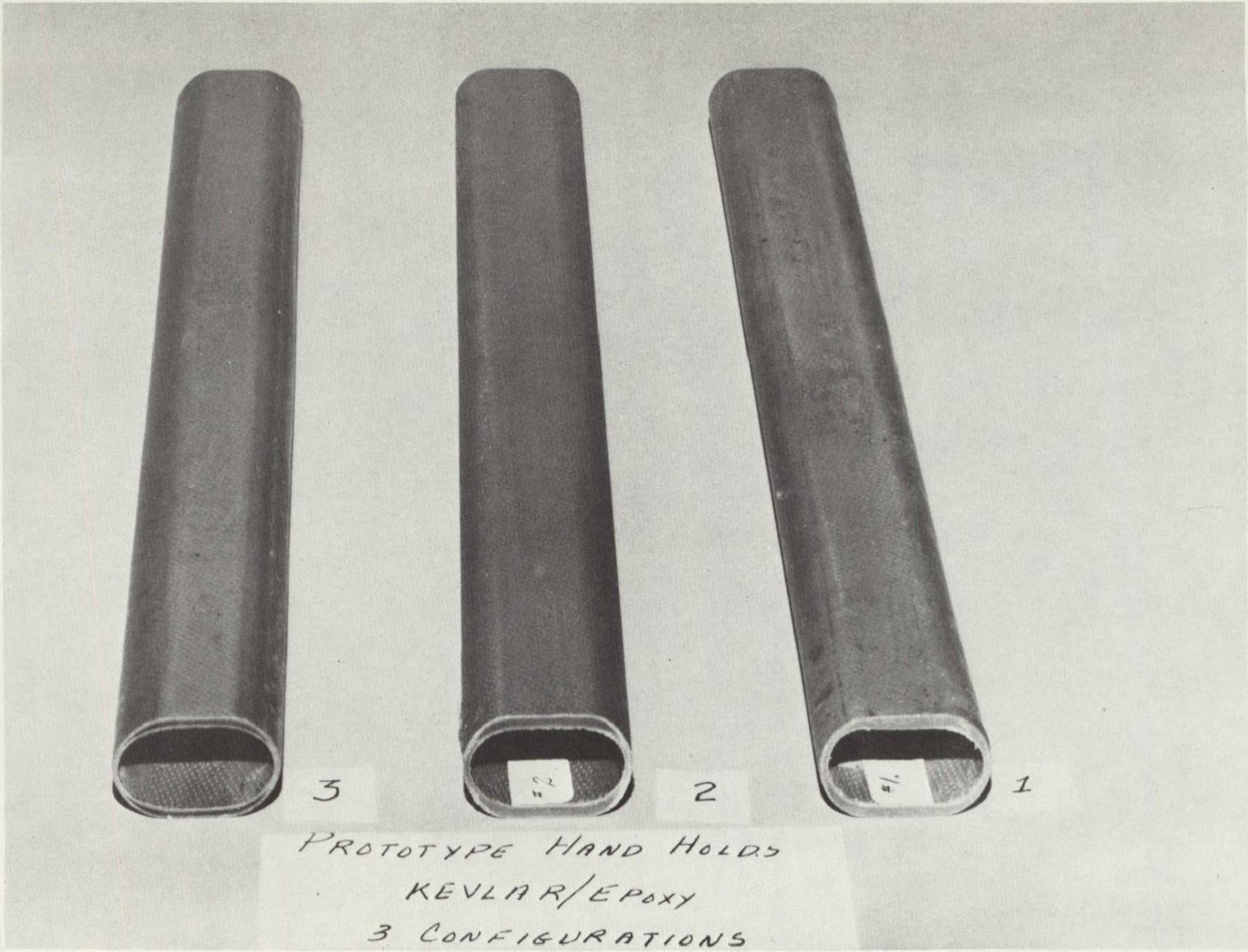

Figure 2-3. Space Shuttle Mobility and Translation Handrail Segments

SECTION 3

NEW TECHNOLOGY

In compliance with the New Technology clause of this contract, personnel assigned to work on the program were advised, and periodically reminded, of their responsibilities in the prompt reporting of items of New Technology. In addition, response was made to all inquiries by the company-appointed New Technology Representative, and copies of reports generated as a result of the contract work were submitted to him for review as a further means of identifying items to be reported. No items of New Technology were found during the performance of work under this contract.

SECTION 4

CONCLUSIONS

1. Of the ten candidate resin matrices representing five generic classes of resins, the Skybond 703 condensation polyimide was the most promising system for meeting all the goals of this program.

2. Kevlar 49 reinforced Skybond 703 laminates can meet all the goals of flammability, offgassing, optical smoke density, odor, etc. even when not postcured.

3. Postcured Kevlar 49/Skybond 703 passes standard VCM testing, however, non-postcured laminates exceed the maximum 1.0 percent total weight loss requirement portion of the VCM test.

4. In very thin Kevlar 49/Skybond 703 laminates, flammability is increased. This is aggravated in condensation polyimide systems by the inherent porosity of the cured laminates.

5. Modified epoxies with large amounts of fire retardants such as Hexcel's F-164 can meet the requirements of NHB8060.1A and would have structural advantages over the Skybond 703 system. However, these modified systems would also have problems passing the VCM test.

6. The Kevlar 49/Skybond 703 system met or exceeded all the initial goals of the program with the exception of compression strength.

7. There is an apparent interface problem with Kevlar 49/polyimide laminates which results in relatively low strengths in such properties as flexure, shear, and edge compression. This same problem prevents proper failures when testing Charpy impact specimens and causes delamination to occur.

8. Prototype Kevlar/epoxy and Kevlar-graphite/epoxy handrail segments were successfully produced, and this promises considerable weight savings for Space Shuttle over conventional aluminum handrails.

SECTION 5

RECOMMENDATIONS

1. Evaluate polyethersulfone/Kevlar combinations to determine cause of strong odors obtained in this program.

2. Evaluate polyethersulfone/Kevlar to fully characterize mechanical and physical properties once odor problem has been resolved.

3. Develop compression forming capabilities of polyethersulfone/Kevlar and polyethersulfone/graphite composites and make typical Space Shuttle interior cabin components using the developed materials and processes.

4. Continue on a logical course for developing full-scale production on the Space Shuttle mobility and translation handrail segments. This would include in-depth analysis, subcomponent testing, joint evaluation, production tooling tryout, and cost analysis.