N O T I C E

THIS DOCUMENT HAS BEEN REPRODUCED FROM MICROFICHE. ALTHOUGH IT IS RECOGNIZED THAT CERTAIN PORTIONS ARE ILLEGIBLE, IT IS BEING RELEASED IN THE INTEREST OF MAKING AVAILABLE AS MUCH INFORMATION AS POSSIBLE

(NASA-CR-165339) HYBRIDIZED POLYMER MATRIX COMPOSITES Contractor Final Report, Jun. 1978 - Jun. 1980 (Grumman Aerospace Corp.) 113 p HC A06/MF A01 CSCL 11G

N81-31361

Unclas

G3/27 27382

CONTENTS

Section		Page
1	SUMMARY	1
2	INTRODUCTION	3
3	TECHNICAL DISCUSSION	5
	3.1 Concept Definition and Analysis (Task I)	5
	3.1.1 Technical Approach	5
	3.1.2 Material Considerations	5
	3.1.3 Material Selection	6
	3.1.4 Design and Analysis Considerations	8
	3.1.5 Laminate Concept Selection	9
	3.2 Concept Fabrication and Evaluation (Task II)	41
	3.2.1 Materials	41
	3.2.2 Laminate Fabrication	41
	3.2.3 Pilot Laminate Examination	44
	3.2.4 Concept/Laminate Characterization	44
	3.2.4.1 Unconditioned Laminate Characterization	45
	3.2.4.1.1 Ultrasonic Inspection	45
	3.2.4.1.2 Photomicrographic Integrity	45
	3.2.4.1.3 Specific Gravity	47
	3.2.4.1.4 Volume Fraction and Void Content	47
	3.2.4.1.5 Flexural Strength and Modulus	48
	3.2.4.1.6 Interlaminar Shear Strength	54
	3.2.4.1.7 Heat Distortion Temperature	55
	3.2.4.1.8 Isothermal Gravimetric Analysis	56
	3.2.4.2 Environmental Conditioning	58
	3.2.4.2.1 Thermo-Oxidative Conditioning	58
	3.2.4.2.2 Moisture Conditioning	58

PRECEDING PAGE BLANK NOT FILMED

CONTENTS (Cont)

Section		Page
3.2.4.3	Conditioned Laminate Characterization	60
3.2.4.3.1	Specific Gravity	60
3.2.4.3.2	Volume Fraction and Void Content	61
3.2.4.3.3	Flexural Strength and Modulus	61
3.2.4.3.4	Interlaminar Shear Strength	63
3.2.4.3.5	Heat Distortion Temperature Measurements	64
3.2.4.4	Laboratory Burn Tests	66
3.2.4.4.1	Flame Spreading Tests (Downward Vertical Burning Rate)	67
3.2.4.4.2	Limiting Oxygen Index Tests	72
3.2.4.4.3	Controlled Burn and Particulate Collection	74
4	CONCLUSIONS	87
Appendix		
A	Preliminary Burn Test Observations	89
B	Edge Photomicrographs of Candidate Laminate Concepts	95
C	List of Symbols	113
REFERENCES		115

ILLUSTRATIONS

Figure		Page
1	Concept Fabrication and Evaluation Task Flow Plan	42
2	Schematic Diagram of Controlled Burn Apparatus	75
3	Controlled Burn Apparatus	76
4	Close-up View of Specimen Being Tested in Controlled Burn Apparatus	77
5	Photomicrographs of Thin Laminates (100 X Mag)	97
6	Photomicrographs of Thick Laminates (100 X Mag)	104

TABLES

Number		
1	Summary of Recommendations	1
2	Unidirectional Per-Ply Properties of Composite Materials (Layer Properties)	10
3	Fiber Release Prevention Reporting Terminology	11
4	Mechanical Properties of Candidate Laminates (Calculated)	12
5	Thin Control Panel (Laminate No. 1)	13
6	Aluminum Foil Coated (Laminate No. 2)	14
7	Boron Outer Plies (Laminate No. 3)	15
8	Woven Fiberglass Outer Plies (Laminate No. 4)	16
9	Woven Glass Outer Plies, PI Matrix (Laminate No. 5)	17
10	Woven Graphite Outer Plies (Laminate No. 6)	18
11	Polyimide NR-150-B2 Sized Graphite (Laminate No. 7)	19
12	Kimbar Flame Barrier Surface (Laminate No. 8)	20
13	Sodium Silicate Treated Woven Graphite Outer Plies (Laminate No. 9)	22
14	Thick Control Panel (Laminate No. 10)	24
15	Boron Outer and Internal Ply Bands (Laminate No. 11)	25
16	Woven Glass Outer and Internal Ply Bands, Polyimide Matrix (Laminate No. 12)	26

TABLES (Cont)

Number		Page
17	Intumescent Coating with Woven Quartz Outer Layers (Laminate No. 13)	27
18	Woven Fiberglass Outer and Internal Plies (Laminate No. 14)	29
19	Woven Graphite-Glass Outer and Internal Plies (Laminate No. 15)	30
20	NR-150-B2 Sized with Woven Graphite Internal Plies (Laminate No. 16)	32
21	Fire-Retardant Epoxy Woven Glass Internal Plies (Laminate No. 17)	34
22	Sodium Silicate Glass Scrim Alternating Plies (Laminate No. 18)	36
23	6% Boron Powder Between Graphite/Epoxy Plies (Laminate No. 5A)	38
24	Sodium Borate Glass Scrim Alternating Plies (Laminate No. 18A)	39
25	Materials	43
26	Preliminary Burning Test Conditions	44
27	Inspection Results; Hybridized Polymer Matrix Composite Pilot Laminates	45
28	Test Results; Ultrasonic Examination, Photomicrograph Integrity and Specific Gravity	46
29	Test Results; Constituent Volume Fraction, Void Content and Related Properties of Unconditioned Specimens	49
30	Test Results; Flexural Strength and Modulus, Unconditioned Specimens	52
31	Ranked Flexure Test Results and In-plane Predictions	53
32	Test Results; Interlaminar (Horizontal) Shear Strength, Unconditioned Specimens	54
33	Test Results; Heat Distortion Temperature (Tg), Unconditioned Specimens	56
34	Test Results; Isothermal Gravimetric Analysis, Unconditioned Specimens	57
35	Moisture Absorption Data for Thin Laminates	59
36	Moisture Absorption Data for Thick Laminates	60
37	Specific Gravity of Conditioned Laminates	61
38	Test Results; Flexural Strength and Modulus, Thin Laminates After 16 Days at 60°C/98% R.H.	62

TABLES (Cont)

Number		Page
39	Test Results; Flexural Strength and Modulus, Thick Laminates After 21 Days at 60°C/98% R.H.	62
40	Test Results; Interlaminar (Horizontal) Shear Strength, Thin Laminates After 16 Days at 60°C/98% R.H.	63
41	Test Results; Interlaminar (Horizontal) Shear Strength, Thick Laminates After 21 Days at 60°C/98% R.H.	64
42	Test Results; Tg (HDT) Of Conditioned Laminates	65
43	Test Results; Elevation of Tg (HDT) Due to Thermal Exposure	66
44	Test Results; Flame Spread (DVBR)	69
45	Thin Laminate Burning Performance Ranking	71
46	Thick Laminate Burning Performance Ranking	71
47	Limiting Oxygen Index Test Results and Ranking	73
48	Controlled Burn/Particulate Collection Procedure	78
49	Observations of Burning and Charred Specimens	79
50	Observations of Particulate Matter	82
51	Char Characteristic and Particulate Matter Ratings	85
52	Normalized Physical, Mechanical and Burn Test Ratings	88
53	Preliminary Burn Test Observations	91

Section 1

SUMMARY

This study program has defined design approaches and materials from which can be fabricated pyrostatic graphite/epoxy (Gr/Ep) laminates that show improved retention of graphite particulates when subjected to burning. Sixteen hybridized plus two standard Gr/Ep laminates were designed, fabricated, and tested in an effort to eliminate the release of carbon (graphite) fiber particles from burned/burning, mechanically disturbed samples. (The term pyrostatic is defined as meaning mechanically intact in the presence of fire.) The main thrust of this program was aimed at the formulation of graphite particulate-retentive laminates whose constituent materials, cost of fabrication, and physical and mechanical properties were not significantly different from existing Gr/Ep composites. Therefore, all but one laminate (a Celion graphite/bismaleimide polyimide) were based on an off-the-shelf Gr/Ep, the AS-1/3501-5A system. Of the 16 candidates studied, four thin (10-ply) and four thick (50-ply) hybridized composites are recommended. These are presented in Table 1. Panels of the selected laminates were delivered to the NASA-Lewis Research Center, the sponsor of this program (Contract NAS 3-21382; Dr. T. T. Serafini, Project Manager).

TABLE 1 SUMMARY OF RECOMMENDATIONS

TYPE OF LAMINATE	RANK	LAMINATE NO.	HYBRIDIZING FEATURE
THIN	1	5A	BORON POWDER IN MATRIX
	2	3	BORON FACES
	3	6	WOVEN GRAPHITE FACES
	4	4	WOVEN FIBERGLASS FACES
THICK	1	13	INTUMESCENT COATING
	2	17	FIRE-RETARDANT EPOXY
	3	15	WOVEN GR/GL PLIES AND FACES
	4	11	BORON PLIES AND FACES

R81-0911-001D

Section 2

INTRODUCTION

The advantages and practicality of using graphite/epoxy (Gr/Ep) laminates as a structural material have been confirmed. Graphite (carbon) composites save weight; they produce structures as strong and stiff as those built from traditional materials but are much lighter and more fatigue resistant. This weight saving translates directly into fuel savings in transportation vehicles of any type. Thus, application of these materials in the transportation industry is increasing.

Graphite fiber materials are now available as unidirectional tape, woven fabric, chopped fiber, paper stock, and sheet molding compounds. Graphite fibers can be impregnated with thermoplastic and thermosetting resins, and composites can be formed by hot processing, pultrusion, or automatic forming rollers. Composites can be made with co-laminated metal skins, such as aluminum or stainless steel foils, or they can be metallized or painted after molding. Graphite fibers can even be produced from annually renewable agricultural raw materials such as rayons based on vegetable cellulose. In short, graphite fiber-reinforced polymer matrix composites have definitely begun to impact our life style.

At the start of this study, the future use of graphite fiber-reinforced composites was threatened because carbon and graphite fibers are extremely good conductors of electricity. It was thought that free carbon fibers produced by intentional or accidental incineration of graphite composites could become airborne and settle on, and short-out, electrical and electronic circuits.

This potential hazard was of sufficient concern to NASA that it undertook risk analysis and materials modification programs. The purpose of this study was to hybridize Gr/Ep laminates through alterations of the polymer binder matrix and/or the advanced composite reinforcement in order to improve the quantity and strength of the char formed when the polymer matrix burns, because it was believed that char formation would minimize the release of free carbon fibers. In summary, this program defined design approaches and materials for the fabrication of hybridized Gr/Ep laminates which showed improved retention of graphite particulates when subjected to burning.

PAGE 2 INTENTIONALLY BLANK

Section 3

TECHNICAL DISCUSSION

3.1 CONCEPT DEFINITION AND ANALYSIS (TASK 1)

In this task, applicable off-the-shelf fibers, resins, and ancillary materials were listed to establish their potential graphite particulate retention characteristics. From this evaluation, 16 of the most promising hybrid combinations were selected for fabrication in the subsequent task and predictions of the potential laminate properties made.

3.1.1 Technical Approach

The technical approach of the study was to provide graphite particulate retention of a selected Gr/Ep baseline by hybridization. Hybridization was to be accomplished with as little modification as possible to current graphite fiber composites technology, since the advantages of graphite fiber composites could be easily lost if severe cost and weight penalties were incurred. Therefore, the most practical materials and their physical arrangements were emphasized. The basic Gr/Ep used for the study was Hercules AS-1/3501-5A unidirectional prepreg tape, for which a large bank of data has been compiled at Grumman. Consideration was given to the effects of char formation tendencies, heat resistance, melting characteristics, burning characteristics, laminate mechanical properties, and laminate weight, cost, and availability. Consideration was also given to the effects of interaction between material components. Literature research and vendor contacts helped to narrow the choice of candidate laminate concepts.

3.1.2 Material Considerations

Based upon material considerations (Ref. 1-8), the following concepts were selected:

- Use of high-char-yield phenolic and polyimide (PI) resins which can almost eliminate fiber release
- Use of metallic coatings for oxidation resistance, fire protection and weathering

PAGE 4 INTENTIONALLY BLANK

- Bis-maleimide polyimide (BMI) resins for stable char to 800°C, with char yield of 40 to 60% and 0.17 char/fiber weight ratio
- Optimum cure cycle to develop full cross-linking in epoxy (Ep) matrices for reduced fiber release
- Hybrid reinforcement with Kevlar-SiC, Kevlar-alumina and Kevlar-glass in PMR (Polymerization Monomeric Reactant) PI and epoxy matrices
- Polymer blends of PI's, epoxies, and polyesters with silicones
- Sizing or "double" sizing of AS-1 graphite with NR-150B2 PI (as done for Celion fibers) to provide a protective char former at the surface of the graphite
- Sodium silicate or sodium borate fiber treatments to promote fiber clumping
- Blending of epoxy and BMI in graphite laminates, cocuring through aromatic diamine (DDS)
- Intumescent paint (non-structural) on thick panels for minimum weight penalty
- Hybrid composites and fiber coatings for near-term solution to the problem
- Hybrid tape and fabric, different weave and tape combinations, supplemental coatings, use of existing Gr/Ep prepregs
- Boron/PI (B/PI) or boron/epoxy (B/Ep) outer plies
- Epoxy-novolac plus milled quartz fibers to yield tough char under ablating conditions.

3.1.3 Material Selection

Baseline laminates were fabricated from (Hercules) AS-1/3501-5A Gr/Ep unidirectional prepreg tape, for which a large bank of data has been compiled at Grumman. Additional materials considered for inclusion in the laminates included the following:

- Perforated aluminum foil
- AVCO 5505-4 B/Ep unidirectional prepreg
- Woven fiberglass/epoxy (Gl/Ep)

- NR-150B 2-sized AS-1/3501-5A unidirectional prepreg
- T-300 and Celion 6000/F-178 Gr/BMI unidirectional prepreg
- Woven Celion 6000 or T-300/BMI prepreg fabric
- Woven Style 581 quartz/BMI prepreg fabric
- Aluminum pressed powder adhesive-bonded coating
- Kimbar (Schweitzer) novoloid phenolic flame-barrier paper either alone or in combination with Celion 6000 or T-300/BMI unitape
- Kimbar flame-barrier paper in combination with Style 581 woven fiberglass cloth
- AVCO 5505-4 B/Ep unitape in combination with Celion 3000 or T-300 Gr/BMI unitape
- Hybrid Celion 3000 or T-300 graphite (warp), S-2 fiberglass (fill) woven fabric, in combination with Celion or T-300/BMI or AS-1/3501-5A unitape
- Sodium silicate or sodium borate treated Style 104 glass scrim cloth
- Milled quartz fiber, 5%, in 3501-5A or F-178 (Hexcel) BMI resin
- Kimbar flame-barrier paper in combination with milled quartz fiber/resin mixture
- Woven Style 581 quartz/epoxy (Qu/Ep) cloth prepreg
- Woven Gr/Ep cloth prepreg
- AVCO "Flamarest 1600 B" thermal insulating coating (intumescent and ablative)
- Aluminum-coated "Thorstrand" woven Gl/Ep prepreg
- Flame-retardant (Tetrabromobisphenol) epoxy/woven fiberglass cloth prepreg.

Producibility guidelines eliminated the use of precured high-temperature materials such as graphite or 7781 glass-reinforced PMR-15 PI (and similar PI candidates), silicone, and fire-retardant polyester prepregs. Because two cure cycles, as a minimum, would be required, the production of contoured parts would be

difficult. Examination of the properites of non-woven mats, as produced by the Pellon Corp., indicated no advantages (except surface finish) in using these materials; woven quartz because of its high cost was later ruled out in favor of woven fiberglass, except for one specimen.

Mechanical reinforcement (stitching) was also considered but is limited because only Kevlar thread works well, and it burns. Fiberglass thread was found to break too easily, especially when penetrating thick laminates. Also, the quilting pattern would have to be tight in order to retain small fibers; this would add too much weight to the laminates. The use of metal staples is also not recommended, after further study of the problem.

Resin fillers or fillers applied between plies of graphite prepreg, either in powder or microballoon form, were intially ruled out because their effects on mechanical properties, other than an anticipated reduction in interlaminar shear strength, were difficult to predict. However, boron powder (-325 mesh) was later tried, after discussion with the NASA-Lewis Project Manager.

Intralaminar mixing within a single ply can be used to place selected prepregs at specific locations. This approach was not selected because of the following complications:

- Manufacturing costs would be high for large panels if separate side-by-side tapes or woven prepregs were butted, either by hand or by tape-laying machine
- Application of lamination theory would be very difficult because per-ply properties would be variable
- Problems are anticipated with in-plane differing coefficients of thermal expansion and matching in-plane cured thicknesses of adjacent plies.

3.1.4 Design and Analysis Considerations

The candidate laminates chosen represent a balance between retention of engineering properties and significant reduction of the fiber release hazard. Because current graphite fiber laminates have been optimized for efficient performance, any changes would tend to decrease performance characteristics. Increased density reduces potential weight savings; unusual materials or processes increase cost or reduce strength and stiffness.

Limits were established, beyond which no proposed solution would be considered satisfactory. These limits will vary greatly with the potential application but, in general, they can be used as guidelines. At present, Gr/Ep structural aircraft components are being designed to an ultimate strain of 4,000 μin./in. and a corresponding weight savings of 30% over comparable metal structures. In this study, only those hybridized advanced fiber polymer matrix composites which calculations showed did not initially reduce the specific mechanical properties of composites by more than 25% were considered. Similarly, only those concept/material combinations that showed projected cost increases of not more than 20% and/or producibility increases of not more than 25% were considered. The cost increase can also be assumed to be a function of the total amount of material utilization. An initial cost increase may, therefore, disappear if a large volume of graphite hybridizing material is used.

An analytical study to predict the engineering properties of laminates was performed, using the concept/material combinations given above. The analysis considered the constituent material properties of each laminate ply, the volume ratios of the materials in each concept/materials combination, the ply-stacking sequence, and the cured materials' strength and stiffness. Predictions were made using the STIFFNESS-5 computer program, which computes in-plane, bending matrices and engineering constraints for hybrid laminates. Layer properties for each ply material were first calculated (Table 2); the symbols are defined in Appendix C.

3.1.5 Laminate Concept Selection

The selected laminate concepts and calculated properties are summarized in Tables 3 and 4, respectively. They are subdivided by thickness, i.e., thin or thick, with the thin laminates being of a basic $(\pm 45/0_2/90)_s$ configuration [10 plies, 1.0 to 1.5 mm (0.040 to 0.060 in.)] and the thick laminates being of a basic $(\pm 45/90_2/\pm 45/0_7/\pm 45/90)_s$ configuration [50 plies, 6.25 mm (0.250 in.)]. The laminate designation number/laminate description/reporting terminology relationship reported in Table 3 is used throughout this report.

The laminates are more fully described in Tables 5 through 24. Initially, it was conceived that woven Qu/F-178 BMI would be a viable material system for incorporation as outer and internal ply bands (see Table 16). However, its cost was deemed too high and Gl/F-178 BMI was substituted.

During fabrication, Laminate No. 5 delaminated. Therefore, Laminate No. 5A (Table 23) containing 6% boron powder between the Gr/Ep unitape plies was substituted.

TABLE 2 UNIDIRECTIONAL PER PLY PROPERTIES OF COMPOSITE MATERIALS (LAYER PROPERTIES)

MATERIAL	E_L	E_T	G_{LT}	ν_{LT}	t'	F_L^t	F_L^c	F_T^t	F_T^c
	MPa x 10^3 (MSI)	MPa x 10^3 (MSI)	MPa x 10^3 (MSI)		MM IN.	Pa x 10^7 (KSI)	Pa x 10^7 (KSI)	Pa x 10^7 (KSI)	Pa x 10^7 (KSI)
GR/EP, AS-1/3501-5A, HERCULES	127.6 (18.5)	11.0 (1.6)	4.5 (0.65)	0.25	0.13 (0.00525)	145.5 (211)	140.0 (203)	5.2 (7.5)	21.4 (31)
B/EP, AV 5505-4, AVCO	214.4 (31.1)	19.3 (2.8)	5.0 (0.72)	0.25	0.13 (0.00525)	142.0 (206)	413.7 (600)	9.0 (13.1)	34.5 (50)
GL/EP, 7781/F-161, HEXCEL	25.5 (3.7)	24.1 (3.5)	7.6 (1.10)	0.10	0.25 (0.010)	38.3 (55.5)	38.0 (55)	33.7 (48.9)	32.4 (47)
QU/EP, 581/F161, HEXCEL	23.4 (3.4)	22.1 (3.2)	6.9 (1.01)	0.10	0.25 (0.010)	44.1 (64)	39.3 (57)	38.5 (66)	33.6 (49)
QU/PI, 581/F-178, HEXCEL	28.3 (4.1)	26.9 (3.9)	8.3 (1.21)	0.10	0.28 (0.011)	41.4 (60)	43.4 (63)	— —	— —
GR/PI, T-300/F-178, HEXCEL	139.2 (20.2)	11.7 (1.7)	4.9 (0.71)	0.25	0.13 (0.005)	151.7 (220)	144.8 (210)	5.4 (7.8)	22.1 (32)
GR/EP, T-300/F-263, HEXCEL	71.7 (10.4)	67.6 (9.8)	10.3 (1.50)	0.10	0.36 (0.0.	47.4 (68.8)	44.8 (65)	46.9 (68)	46.2 (67)
GR/EP, T-300/X934, FIBERITE	151.7 (22.0)	10.3 (1.5)	3.6 (0.52)	0.25	0.13 (0.005)	145.5 (211)	121.4 (176)	3.7 (5.4)	17.2 (25)
GR-GL/EP, HMF-721/34, FIBERITE	53.1 (7.7)	51.7 (7.5)	7.6 (1.10)	0.10	0.20 (0.008)	48.3 (70)	48.3 (70)	38.6 (56)	38.6 (56)
AL-GL/EP, AL COATED 7781 WOVEN GLASS/F-166, HEXCEL	23.4 (3.4)	23.4 (3.4)	7.6 (1.10)	0.10	0.25 (0.010)	41.4 (60)	42.1 (61)	41.4 (60)	42.1 (61)
GR/EP, WOVEN A 370-8H/3501-5A, HERCULES	72.4 (10.5)	72.4 (0.5)	10.3 (1.50)	0.10	0.33 (0.013)	62.1 (90)	55.8 (81)	62.1 (90)	55.8 (81)
GL/EP, FIRE RETARDANT 7781/F-164, HEXCEL	22.8 (3.3)	22.8 (3.3)	6.1 (0.88)	0.10	0.25 (0.010)	46.9 (68)	33.5 (49)	46.9 (68)	33.5 (49)
GL/EP, 104 SCRIM WITH 5% SODIUM SILICATE	4.0 (0.6)	4.0 (0.6)	1.3 (0.19)	0.30	0.03 (0.001)	8.3 (12)	8.3 (12)	8.3 (12)	8.3 (12)
6061 AL	68.3 (9.9)	68.3 (9.9)	26.2 (3.80)	0.30	0.05 (0.002)	29.0 (42)	24.8 (36)	29.0 (42)	24.8 (36)

R81-0911-002D

TABLE 3 FIBER RELEASE PREVENTION REPORTING TERMINOLOGY

TYPE OF LAMINATE	LAMINATE DESIGNATION NO.	NUMBER OF PLIES	FIBER RELEASE PREVENTION FEATURE	REPORTING TERMINOLOGY
THIN	1	10	GR/EP AS-1/3501-5A UNIDIRECTIONAL UNI-TAPE	CONTROL
	2	10 + 2	ALUMINUM FOIL COATED, AS-1/3501-5A UNI-TAPE	ALUMINUM FACED
	3	10	B/EP OUTER PLIES, AS-1/3501-5A UNI-TAPE	B/EP FACED
	4	8	WOVEN GL/EP OUTER PLIES, AS-1/3501-5A UNI TAPE	WOVEN GL/EP FACED
	5	8	WOVEN GL/PI OUTER PLIES, C-6K/F-178 UNI-TAPE (1)	WOVEN GL/PI FACED, GR/PI
	5A	8	BORON POWDER BETWEEN PLIES, AS-1/3501-5A UNI-TAPE (2)	BORON POWDER
	6	8	WOVEN GR/EP OUTER PLIES, AS-1/3501-5A UNI-TAPE	WOVEN GR/EP FACED
	7	10	NR-150B2-SIZED FIBERS, AS-1/3501-5A UNI-TAPE	PI SIZED
	8	10	KIMBAR FLAME BARRIER SURFACE, AS-1/3501-5A UNI-TAPE	KIMBAR FACED
	9	8	SODIUM SILICATE TREATED WOVEN GR/EP OUTER PLIES, AS-1/3501-5A UNI-TAPE	SODIUM SILICATE TREATED
THICK	10	50	GR/EP AS-1/3501-5A UNDIRECTIONAL UNI-TAPE	CONTROL
	11	50	B/EP OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE	B/EP PLIES
	12	44	WOVEN GL/PI OUTER AND INTERNAL PLY BANDS, C-6K/F-178 UNI-TAPE (1)	WOVEN GL/PI PLIES
	13	50	INTUMESCENT COATING ON WOVEN QU/EP OUTER PLIES, AS-1/3501-5A UNI-TAPE (3)	INTUMESCENT COATED
	14	44	WOVEN GL/EP OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE	WOVEN GL/EP PLIES
	15	44	WOVEN GL-GR/EP OUTER AND INTERNAL PLY BANDS, AS 1/3501 5A UNI-TAPE	WOVEN GL/GR/ EP PLIES
	16	44	NR-150B2-SIZED WOVEN GR/EP OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE	PI SIZED GR
	17	46	WOVEN FIRE-RETARDANT GL/EP OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE	FIRE RETARDANT EPOXY
	18	75	SODIUM SILICATE-GLASS SCRIM OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE	SODIUM SILICATE TREATED
	18A	75	SODIUM BORATE-GLASS SCRIM OUTER AND INTERNAL PLY BANDS, AS-1/3501-5A UNI-TAPE (4)	SODIUM BORATE TREATED

R81-0911-003D

NOTES:

(1) C-6K IS THE ABBREVIATION FOR CELION 6000 GRAPHITE FIBER.

(2) LAMINATE NO. 5A WAS USED FOR ALL MECHANICAL AND THERMAL TESTING

(3) LAMINATE NO. 13 WAS USED W/O INTUMESCENT COATING FOR TESTS WHERE THE COATING WOULD HAVE INTERFERED; THE COATING ADDS NON-STRUCTURAL THICKNESS AND DECOMPOSES SLOWLY AT 200°C.

(4) LAMINATE NO. 18A WAS USED FOR ALL MECHANICAL AND THERMAL TESTING.

TABLE 4 MECHANICAL PROPERTIES OF CANDIDATE LAMINATES (CALCULATED)

TYPE OF LAMINATE	LAMINATE NO.	E_X	E_Y	G_{XY}	ν_{XY}	F_X^{TU}
		MPA x 10^3 (MSI)	MPA x 10^3 (MSI)	MPA x 10^3 (MSI)		PA x 10^7 (KSI)
THIN	1*	65.5 (9.5)	43.4 (6.3)	15.9 (2.3)	0.31	74.7 (108.2)
	2	65.5 (9.5)	45.5 (6.6)	16.5 (2.4)	0.31	74.6 (108.2)
	3	69.6 (10.1)	49.0 (7.1)	25.5 (3.7)	0.43	79.8 (115.7)
	4	62.7 (9.1)	38.6 (5.6)	6.9 (1.0)	0.10	70.9 (102.9)
	5	65.5 (9.5)	41.4 (6.0)	8.3 (1.2)	0.11	71.2 (103.3)
	6	65.5 (9.5)	46.2 (6.7)	17.2 (2.5)	0.30	75.2 (109.0)
	7	65.5 (9.5)	43.4 (6.3)	15.9 (2.3)	0.31	74.6 (108.2)
	8	65.5 (9.5)	43.4 (6.3)	15.9 (2.3)	0.31	74.6 (108.2)
	9	74.5 (10.8)	51.0 (7.4)	16.5 (2.4)	0.27	71.6 (103.8)
THICK	10*	82.1 (11.9)	33.1 (4.8)	13.8 (2.0)	0.35	93.7 (135.9)
	11	84.8 (12.3)	39.3 (5.7)	17.2 (2.5)	0.38	96.6 (140.1)
	12	86.2 (12.5)	31.7 (4.6)	9.7 (1.4)	0.20	93.8 (136.1)
	13	82.1 (11.9)	31.7 (4.6)	11.7 (1.7)	0.30	93.3 (135.3)
	14	81.4 (11.8)	29.6 (4.3)	8.3 (1.2)	0.20	92.3 (133.9)
	15	85.5 (12.4)	32.4 (4.7)	11.0 (1.6)	0.26	97.9 (142.0)
	16	82.1 (11.9)	35.9 (5.2)	14.5 (2.1)	0.33	94.0 (136.4)
	17	81.4 (11.8)	30.3 (4.4)	10.3 (1.5)	0.26	92.4 (134.1)
	18	75.2 (10.9)	30.3 (4.4)	12.4 (1.8)	0.35	86.0 (124.7)

R81-0911-004D

*CONTROL

TABLE 5 THIN CONTROL PANEL, LAMINATE NO. 1

PLY NO.	PLY CONFIG, °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
2	135	↓	↓
3	0	↓	↓
4	0	↓	↓
5	90	↓	↓
6	90	↓	↓
7	0	↓	↓
8	0	↓	↓
9	135	↓	↓
10	45	↓	↓

Volume Ratio of Materials in Panel, Calculated

- Graphite: 55 to 60% (generally normalized to 60%)
- Epoxy: 40 to 45%.

Material and Processing Costs

- Gr/Ep: Hercules AS-1/3501-5A; 3-in. tape costs $45/lb; 12-in-wide tape costs $42/lb
- Processing, which includes lay-up, compaction, bag and bleeder application, cure, post-cure (assume piggy-back runs), and trim, takes approximately 3 hr/lb.

Enivronmental Stability

The overall chemical stability of cured AS-1/3501-5A laminates is very good. The combination of heat and moisture cause swelling and plasticization of the laminate with subsequent loss of strength at temperature, although dry heat alone causes little strength drop-off, up to 260 to 270°F. This effect is attributed to the resin component of the laminate (Ref. 6).

Rationale for Choice

1. Selected PAN-based graphite because of lower conductivity in low modulus range; also present pitch-base graphite has low strain to failure.
2. AS-1/3501-5A has better char-forming resin (DDS-cure) than other types (TETA, MPDA).
3. Cross-plied laminates show particulate problem more than unidirectional.
4. Grumman has considerable experience with Hercules AS-1/3501-5A.

R81-0911-005D

TABLE 6 ALUMINIUM FOIL COATED, LAMINATE NO 2.

PLY NO.	PLY CONFIG, °	MATERIAL	TYPE
1	N.A.	ALUMINUM	PERFORATED FOIL, 2 MILS THICK
2	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	135	↓	↓
4	0		
5	0		
6	90		
7	90		
8	0		
9	0		
10	135		
11	45		
12	N.A.	ALUMINUM	PERFORATED FOIL, 2 MILS THICK

Volume Ratio of Materials in Panel, Calculated

- Graphite: approximately 53%
- Aluminum perforated foil: 7%
- Epoxy: approximately 40%.

Material and Processing Costs

- Gr/Ep: same as for control panel
- Aluminum foil plus Dexter-Hysol 9628 film adhesive: perforated, as for use in honeycomb core fabrication, plus adhesive, cost approximately $9 to 10/lb
- Processing cost: approximately 3 hr/lb, slightly more than for control panel.

Environmental Stability

Aluminum foil coatings have shown the best environmental protection of any system in tests at Grumman (report in progress). The thermal/moisture strength reduction effect is effectively reduced by almost 100%. Chemical resistance is excellent, except for caustic bases or acids which could attack the aluminum surface. The effect of galvanic corrosion potential is reduced by the adhesive layer between the foil and laminate.

Rationale for Choice

1. Co-cured aluminum foil, perforated to allow resin bleed-out during cure and (possible) gas escape upon burning; is useful on flat or gently curved surfaces only.

2. Serves as lightning protection as well as humidity protection barrier.

R81-0911-006D

TABLE 7 BORON OUTER PLIES, LAMINATE NO. 3

PLY NO.	PLY CONFIG, °	MATERIAL	TYPE
1	45	B/EP	AV 5505-4, AVCO UNI-TAPE
2	135	B/EP	AV 5505-4, AVCO UNI-TAPE
3	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
4	0	↓	↓
5	90	↓	↓
6	90	↓	↓
7	0	↓	↓
8	0	↓	↓
9	135	B/EP	AV 5505-4, AVCO UNI-TAPE
10	45	B/EP	AV 5505-4, AVCO UNI-TAPE

Volume Ratio of Materials in Panel, Calculated

- Graphite: approximately 36%
- Boron (including F/G scrim): 20%
- Epoxy: 44%.

Material and Processing Costs

- Gr/Ep: material cost same as for control panel
- B/Ep: currently costs $2.87/linear ft of 3-in.-wide tape = $192/lb
- Processing cost: approximately 3 hr/lb, same as for control panel; if specimens require drilling, costs go up.

Environmental Stability

Stability of this laminate is similar to that of the control Gr/Ep laminate.

Rationale for Choice

1. The boron fibers are intended to provide high temperature, high strength mechanical entrapment at the surface of the laminate.
2. Boron will also improve the mechanical properties of the panel, at a small cost in weight.

R81-0911-007D

TABLE 8 WOVEN FIBERGLASS OUTER PLIES, LAMINATE NO. 4

PLY NO.	PLY CONFIG.,°	MATERIAL	TYPE
1	45,135	GL/EP	7781/F-161 WOVEN CLOTH, HEXCEL
2	0	GR/EP	AS-1/3501-5A HERCULES UNI-TAPE
3	0	↓	↓
4	90		
5	90		
6	0		
7	0		
8	45, 135	GL/EP	7781/F-161 WOVEN CLOTH, HEXCEL

Volume Ratio of Materials in Panel, Calculated

- Graphite: 36%
- Fiberglass: 22%
- Epoxy: 42% .

Material and Processing Costs

- Gr/Ep: same material cost as for control panel
- Gl/Ep: F-161 currently costs about $3.50/yd of 38-in.-wide woven prepreg; each yard of prepreg weighs about 13 oz the figure of $2.85 is used for the per pound cost
- Processing Cost: somewhat less than 3 hr/lb.

Environmental Stability

The stability of this panel is similar to that of the control panel. The outer layers of glass will prevent possible galvanic corrosion in the presence of moisture if the laminate is fastened to metallic structure. A long history of fiberglass usage on aircraft has revealed very little deterioration due to environmental factors.

Rationale for Choice

1. Mechanical entrapment at lower temperatures is provided by the woven glass, which will melt at higher temperatures and possibly bind graphite particles.
2. The 3501-5A and F-161 epoxy resin systems are chemically compatable matrices.

R81-0911-008D

TABLE 9 WOVEN GLASS OUTER PLIES, POLYIMIDE MATRIX, LAMINATE NO. 5

PLY NO.	ORIENTATION, °	MATERIAL	TYPE
1	45, 135	GL/PI	STYLE 7781 GL/F-178, HEXCEL (.011)
2	0	GR/PI	CELION 6000/F-178, HEXCEL UNI-TAPE
3	0	↓	↓
4	90		
5	90		
6	0		
7	0		
8	45, 135	GL/PI	STYLE 7781-GL/F-178, HEXCEL (.011)

Volume Ratio of Material in Panels, Calculated

- Graphite: 30%
- Glass: 30%
- Polyimide: 31%

Material and Processing Costs

- Gr/PI: $80/lb (uni-tape)
- Gl/PI: $40 to $45/lb for 7781/F-178 Hexel prepreg 38-in.-wide woven fabric, depending on quantity
- Processing cost: About 4 hr/lb due to longer post-cure cycle.

Environmental Stability

F-178 PI has good temperature resistance for long term exposures up to 475°F and is rated non-flammable. Resistance to moisture and chemicals has not been fully determined but should be similar to expoxies. Since it has a higher operating temperature range, a fall-off in properties due to moisture absorption would still allow 350°F use. F-178 PI has good char-forming properties upon thermal oxidation.

Rationale for Choice

This PI system has a cure cycle similar to that for epoxies and does not require 600-650° processing to produce good laminates. The glass outer layer is to overcome the brittleness tendency of pure Gr/F-178, for high temperature mechanical entrapment and for contribution to a strong char. Cost of raw materials is, however, high.

R81-0911-009D

TABLE 10 WOVEN GRAPHITE OUTER PLIES, LAMINATE NO. 6

PLY NO.	ORIENTATION, °	MATERIAL	TYPE
1	45, 135	GR/EP	HMF 134/34 WOVEN PREPREG, FIBERITE AS-1/3501-5A, HERCULES UNI-TAPE
2	0	GR/EP	
3	0		
4	90		
5	90		
6	0		
7	0		
8	135, 45	GR/EP	HMF 134/34 WOVEN PREPREG, FIBERITE

Volume Ratio of Materials in Panel, Calculated

- Graphite (uni): 32%
- Graphite (woven): 30%
- Epoxy resin: 38%

Material and Processing Costs

- Gr/Ep: uni-tape, $43 to 45/lb
- Gr/Ep: woven cloth, 8 H-S, 24 x 23 weave/epoxy prepreg, approximately $80 to 85/lb
- Processing cost: about 3 hr/lb, a little less than the control because one ply of woven replaces two plies of uni-tape.

Environmental Stability

Should be very similar to the control panel in resistance to moisture, chemical, and thermal resistance.

Rationale for Choice

1. Woven graphite fabric outer plies act to entrap particles, although impact may cause more fiber release after fire (Ref. 7).
2. Constituent properties are well defined and a minimum weight and cost penalty would be taken. Hercules 3501-5A and Hexcel F-166 woven prepregs, if available, would blend better than the F-263 resin with the base laminate.

R81-0911-010D

TABLE 11 POLYIMIDE NR-150-B2 SIZED GRAPHITE, LAMINATE NO. 7

PLY NO.	ORIENTATION, °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A UNI-TAPE, NR-150 B2 SIZED
2	135	↓	↓
3	0	↓	↓
4	0	↓	↓
5	90	↓	↓
6	90	↓	↓
7	0	↓	↓
8	0	↓	↓
9	135	↓	↓
10	45	↓	↓

Volume Ratio of Materials in Panel, Calculated

- Graphite: 55 to 60% (generally normalized to 60%)
- Epoxy: 38 to 43%
- NR-150 B2: 1 to 2%.

Material and Processing Costs

- Gr/Ep: uni-tape AS-1/3501-5A, fiber sized with DuPont NR-150B2 PI by Hercules before epoxy impregnation, cost approximately $45/lb for 3-in. tape in production quantities, higher for pilot quantities
- Processing cost: same as for control panel, about 3 hr/lb

Environmental Properties

Should be identical to control panel.

Rationale for Choice

1. NR-150B2 PI has been found (Ref. 1) to have good char forming properties upon burning, which causes clumping of graphite fibers.
2. Celion graphite used for PI prepregs are regularly sized with NR-150B2, so that there should be no problem with AS-1 fibers. This treatment places the NR-150B2 directly onto the graphite, under the epoxy, and should be effective at no cost/weight penalty.

R81-0911-011D

TABLE 12 KIMBAR FLAME BARRIER SURFACE, LAMINATE NO. 8 (SHEET 1 OF 2)

PLY NO.	ORIENT-ATION, °	MATERIAL	TYPE
1	MD*	KIMBAR	KYNOL NOVOLOID FLAME BARRIER, 3 MIL PAPER, SCHWEITZER
2	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	135	↓	↓
4	0		
5	0		
6	90		
7	90		
8	0		
9	0		
10	135		
11	45		
12	MD*	KIMBAR	KYNOL NOVOLOID FLAME BARRIER, 3-MIL PAPER, SCHWEITZER

*MACHINE DIRECTION OF PAPER WILL BE 0°.

Volume Ratio of Materials in Panel, Calculated

- Graphite: 54%
- Epoxy: 36%
- Kimbar: 10%.

Material and Processing Costs

- Gr/Ep: uni-tape AS-1/3501-5A, Hercules, $42 to45/lb
- Kimbar: flame barrier paper, Schweitzer Div of Kimberly-Clark Corp, costs $.08 to .11/ft^2, equivalent to $8 to 9/lb
- Processing cost: about 3 hr/lb, just slightly more than for the control since the paper will be co-cured with the laminate and only the additional layup time is needed.

Environmental Stability

Resistance to chemicals, heat and moisture should be very similar to that for the control panel, since the Kimbar paper will become saturated with epoxy resin during cure. The paper is made from novoloid phenolic fibers which begin to char at approximately 150 to 180°C; the char stays inert. The paper can be initially treated with other materials, such as intumescents, before laminating with Gr/Ep.

R81-0911-012D

TABLE 12 KIMBAR FLAME BARRIER SURFACE, LAMINATE NO. 8
(Sheet 2 of 2)

Rationale for Choice
1. Kimbar is an effective barrier to the propagation of flame, to 1000°F. It leaves an intact, high volume char which still functions against flame propagation and does not melt. 2. During combustion the porous Kimbar will allow smoke to escape while acting as a mechanical entrapment barrier for the short carbon fibers.

R81-0911-012D

TABLE 13 SODIUM SILICATE TREATED WOVEN GRAPHITE OUTER PLIES, LAMINATE NO. 9 (SHEET 1 OF 2)

PLY NO.	ORIENTATION, °	MATERIAL	TYPE
1	45, 135	GR/EP	WOVEN T-300 GRAPHITE, STYLE W-134, (FIBERITE) TREATED TO CONTAIN 2% SODIUM SILICATE
2	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	0	↓	↓
4	90	↓	↓
5	90	↓	↓
6	0	↓	↓
7	0	↓	↓
8	135, 90	GR/EP	STYLE W-134 WOVEN GRAPHITE (FIBERITE) TREATED TO CONTAIN 2% SODIUM SILICATE

Volume Ratio of Materials in Panel, Calculated

- Graphite: uni-tape, 32%
- Graphite: W-134 woven, 30%
- Epoxy: 36 to 37%
- Sodium silicate: 1 to 2%

Material and Processing Costs

- Gr/Ep: uni-tape AS-1/3501-5A, Hercules, $42 to 45/lb, 3-in. or 12-in. wide
- Gr/Ep: W-134 style woven T-300 cloth, Fiberite; this material, a dry cloth, costs approximately $75/lb and is available in 42-in. width; it molds out to .007 m ply and will contain 3501/5A resin absorbed from the uni-tape
- Processing cost: molding will take about 3 hr/lb to which must be added the cost of sodium silicate treatment (immersion of the graphite cloth in sodium silicate solution, drying, 250°F baking and sealing in plastic till ready for molding).

Environmental Stability

Chemical and thermal properties should be similar to the Gr/Ep control, but the presence of sodium silicate in and on the graphite fibers may adversely affect moisture resistance.

R81-0911-013D

TABLE 13 SODIUM SILICATE TREATED WOVEN GRAPHITE OUTER PLIES, LAMINATE NO. 9 (SHEET 2 OF 2)

Rationale for Choice
1. Ref (1) states that 2% sodium silicate fiber treatment caused graphite fibers to fall down in bundles, causing no short circuits in an electrical test for more than 90 seconds.
2. Resin saturation of the silicate-treated graphite cloth will take place during the cure cycle, by bleeder ply reduction.
R81-0911-013D

TABLE 14 THICK CONTROL PANEL, LAMINATE NO. 10

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
2	135	↓	↓
3	90		
4	90		
5	135		
6	45		
7	0		
8	0		
9	0		
10	0		
11	0		
12	0		
13	0		
14	45		
15	135		
16	0		
17	0		
18	0		
19	0		
20	0		
21	0		
22	0		
23	135		
24	45		
25	90		
			SYM

Volume Ratio of Materials in Panel, Calculated

- Graphite: 55 to 60% (generally normalized to 60%)
- Epoxy: 40 to 45%.

NOTE: The material and processing costs, environmental stability and rationale for choice of this laminate are the same as for Laminate No. 1, the thin laminate control panel.

R81-0911-014D

TABLE 15 BORON OUTER AND INTERNAL PLY BANDS, LAMINATE NO. 11

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45	B/EP	AV 5505-4, AVCO UNI-TAPE
2	135	B/EP	AV 5505-4, AVCO UNI-TAPE
3	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
4	90	↓	↓
5	135	↓	↓
6	45	↓	↓
7	0	↓	↓
8	0	↓	↓
9	0	↓	↓
10	0	↓	↓
11	0	↓	↓
12	0	↓	↓
13	0	↓	↓
14	45	B/EP	AV 5505-4, AVCO UNI-TAPE
15	135	B/EP	AV 5505-4, AVCO UNI-TAPE
16	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
17	0	↓	↓
18	0	↓	↓
19	0	↓	↓
20	0	↓	↓
21	0	↓	↓
22	0	↓	↓
23	135	↓	↓
24	45	↓	↓
25	90	B/EP	AV 5505-4, AVCO UNI-TAPE
— — —	— — —	— — —	— — — SYM

Volume Ratio of Materials in Panel, Calculated

- Graphite: 48%

  Boron (including F/G scrim): 10%

  Epoxy: 42%.

NOTE: The material and processing costs and environmental stability of this laminate are similar to the controls, No. 1 and 10, and Laminate No. 3, the boron/epoxy-faced thin laminate. As in Laminate No. 3, the rationale for choice is that the boron fibers in the 45 and 135° plies are intended to provide high temperature, high strength mechanical entrapment of graphite fibers at the surface and within the body of this laminate. The effect on stiffness is enhanced by the boron, although a small weight penalty must be taken.

R81-0911-015D

TABLE 16 WOVEN GLASS OUTER AND INTERNAL PLY BANDS, POLYIMIDE MATRIX, LAMINATE NO. 12

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45,135	GL/PI	STYLE 7781 GL/F-178, HEXCEL WOVEN CLOTH PREPREG (.011)
2	90	GR/PI	CELION 6000/F-178, HEXCEL UNI-TAPE
3	90	↓	↓
4	135		
5	45		
6	0		
7	0		
8	0		
9	0		
10	0		
11	0		
12	0		
13	45,135	GL/PI	STYLE 7781 GL/F-178, HEXCEL WOVEN CLOTH PREPREG (.011)
14	0	GR/PI	CELION 6000/F-178, HEXCEL UNI-TAPE
15	0	↓	↓
16	0		
17	0		
18	0		
19	0		
20	0		
21	135,45	GL/PI	STYLE 7781 GL/F-178, HEXCEL WOVEN CLOTH PREPREG (.011)
22	90	GR/PI	CELION 6000/F-178, HEXCEL UNI-TAPE
			SYM

Volume Ratio of Materials in Panel, Calculated

- Graphite: 46%
- Glass: 18%
- Polyimide: 36%.

NOTE: The material and processing costs and environmental stability of this laminate would be similar to thin Laminate No. 5, where the glass fabric is located only on the exterior surfaces. The rationale for its choice is the same, with the added benefit of glass layers within the laminate contributing to the char strength and forming a good mechanical barrier for entrapment of short carbon fibers. A weight penalty must be taken for the interlaminar glass layers, but its effect is much smaller than for thin laminates.

R81-0911-016D

TABLE 17 INTUMESCENT COATING WITH WOVEN QUARTZ OUTER LAYERS, LAMINATE NO. 13 (SHEET 1 OF 2)

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	N.A.	INTUMESCENT	THERMAL INSULATION COATING, FLAMAREST 1600B, AVCO,
2	45,135	QU/EP	STYLE 581/F-161 OR F-166, HEXCEL WOVEN CLOTH
3	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
4	90	↓	↓
5	135		
6	45		
7	0		
8	0		
9	0		
10	0		
11	0		
12	0		
13	0		
14	45		
15	135		
16	0		
17	0		
18	0		
19	0		
20	0		
21	0		
22	0		
23	135		
24	45		
25	90		
			SYM

Volume Ratio of Materials in Panel, Calculated

- Graphite: 46%
- Quartz: 5%
- Epoxy: 33%
- Flamarest: 16%.

Material and Processing Costs

The material and processing costs for this panel are higher than for most of the other laminate panels. However, the proportion of Gr/Ep (at $42 to 45/lb) is high and that of Qu/Ep (at $65 to 85/lb) is low, so there is a cost benefit for this thick panel. Also, there is added cost for the 25 mil thick Flamarest coating ($75/gal in the 1 to 10 gal range, $37.50/gal for quantities over 100 gal) and added processing time for application. Total processing cost is estimated at 5 hr/lb.

R81-0911-017D(1/2)

TABLE 17 INTUMESCENT COATING WITH WOVEN QUARTZ OUTER LAYERS, LAMINATE NO. 13 (SHEET 2 OF 2)

Environmental Stability

Environmental stability of the laminate would be excellent with respect to moisture and chemical attack, since Flamarest B is a modified epoxy coating. Thermal resistance should be excellent; the coating intumesces and forms a low-density, high-volume, fairly strong, inert char that insulates the substrate from fire and heat, prolonging structural collapse and thermal delamination of the substrate.

Rationale for Choice

The rationale for choice combines the thermal/mechanical protection of the woven quartz plus the intumescence/ablation of the insulative coating. The weight penalty, which precludes the use of Flamarest in thin panels, is less in thick laminates. Also, the cost benefit would accrue only to thick panels.

NOTE: Two full-size panels of this configuration were built, but one was uncoated. For mechanical property tests where the coating would interfere, the bare panel was cut into specimens and tested; for the burning tests, the coated panel was used.

R81-0911-017D(2/2)

TABLE 18 WOVEN FIBERGLASS OUTER AND INTERNAL PLIES, LAMINATE NO. 14

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45,135	GL/EP	7781/F-161 or F-166, HEXCEL, OR 7781/2054, NARMCO, WOVEN PREPREG
2	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	90	↓	↓
4	135	↓	↓
5	45	↓	↓
6	0	↓	↓
7	0	↓	↓
8	0	↓	↓
9	0	↓	↓
10	0	↓	↓
11	0	↓	↓
12	0	↓	↓
13	45,135	GL/EP	SAME AS PLY NO. 1, WOVEN GLASS PRE PREG
14	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
15	0	↓	↓
16	0	↓	↓
17	0	↓	↓
18	0	↓	↓
19	0	↓	↓
20	0	↓	↓
21	135,45	GL/EP	SAME AS PLY NO. 1
22	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
			SYM

Volume Ratio of Materials in Panel, Calculated

- Graphite: 47%
- Fiberglass: 12%
- Epoxy: 41%.

NOTE: The material and processing costs, environmental stability and rationale for choice are similar to those for thin Laminate No. 4; inclusion of the woven glass cloth within the laminate as well as at the surface should enhance mechnical entrapment of short carbon fibers.

R81-0911-018D

TABLE 19 WOVEN GRAPHITE-GLASS OUTER AND INTERNAL PLIES, LAMINATE NO. 15 (SHEET 1 OF 2)

PLY NO.	CON-FIG.,°	MATERIAL	TYPE
1	45-135	GR-GL/EP	WOVEN GRAPHITE-GLASS (50-50)/EPOXY PREPREG, FIBERITE OR HEXCEL
2	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	90	↓	↓
4	135		
5	45		
6	0		
7	0		
8	0		
9	0		
10	0		
11	0		
12	0		
13	45-135	GR-GL/EP	SAME AS PLY NO. 1, FIBERITE HMF-721/34 OR HEXCEL F-6C-742/F-558
14	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
15	0	↓	↓
16	0		
17	0		
18	0		
19	0		
20	0		
21	135-45	GR-GL/EP	SAME AS PLY NO. 1
22	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
—	—	—	SYM.

Volume Ratio of Materials in Panel, Calculated

- Graphite: 48%
- Gr/Gl (woven): 11% (half each Gr and Gl)
- Epoxy: 41%.

Material and Processing Costs

Material costs will be higher than for the control panel because of the woven graphite-glass prepreg. This is a relatively new material and is quoted at $58 to $65 per pound of prepreg. Typical is Gr T-300/Gl, 12 x 10 weave, 8.5 mils thick, 5.9 oz./yd. + 40% of F-166 compatible resin. Processing costs will be somewhat lower, since each ply of woven goods replaces two angle plies of unidirectional graphite.

R81-0911-019D

TABLE 19 WOVEN GRAPHITE-GLASS OUTER AND INTERNAL PLIES, LAMINATE NO. 15 (SHEET 2 OF 2)

Environmental Stability

Environmental stability, moisture and chemical resistance of this panel should be very similar to the control panel; thermal stability should be improved. The rationale for choice of the graphite-glass woven interlaminar reinforcement is to provide additional mechanical strength in the warp direction of the fabric which is made from T-300(6K) graphite yarn. The above Hexcel material or the similar Fiberite HMF-721/34, which is made from an 8 x 8 plain weave fabric (similar to their all-graphite W-321), weighs less than the equivalent thickness of all-glass fabric. The plain (square) weave should provide a tight mechanical lock for prevention of escape of carbon fibers.

R81-0911-019D

TABLE 20 NR-150B2 SIZED WITH WOVEN GRAPHITE OUTER AND INTERNAL PLIES, LAMINATE NO. 16 (SHEET 1 OF 2)

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45, 135	GR/EP	P.I. NR-150B2 SIZED WOVEN GRAPHITE A 370-8H/3501-5A, HERCULES
2	90		AS-1/3501-5A, HERCULES UNI-TAPE
3	90		
4	135		
5	45		
6	0		
7	0		
8	0		
9	0		
10	0		
11	0		
12	0		
13	45, 135		SAME AS PLY NO. 1
14	0		AS-1/3501-5A, HERCULES UNI-TAPE
15	0		
16	0		
17	0		
18	0		
19	0		
20	0		
21	135, 45		SAME AS PLY NO. 1
22	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
			SYM.

Volume Ratio of Materials in Panel

Graphite: 55 to 60% (uni and woven)

Epoxy: 38 to 43%

NR-150B2: 1 to 2%

Material and Processing Costs

Material and processing costs will be in the same range as those for thin Laminate No. 7; the higher cost of the NR-150B2 sized woven Gr/Ep prepreg will be somewhat offset by reduced layup time. Each ply of cloth replaces two of angle-piled unidirectional tape.

R81-0911-020D

TABLE 20 NR-150B2 SIZED WITH WOVEN GRAPHITE OUTER AND INTERNAL PLIES, LAMINATE NO. 16 (SHEET 2 OF 2)

Environmental Stability Moisture and chemical resistance properties should be identical to the control panel, but thermal resistance is improved. The rationale for choice of sizing the woven graphite with NR-150B2 is that the cost of sizing uni-tape in relatively small quantities may be prohibitive. But with woven cloth this sizing can be more readily applied before epoxy prepregging. It has little effect on thick laminate mechanical properties and will promote good char formation. R81-0911-020D(2/2)

TABLE 21 FIRE-RETARDANT EPOXY WITH WOVEN GLASS INTERNAL PLIES, LAMINATE NO. 17 (SHEET 1 OF 2)

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45, 135	GL/EP	F-164/7781 FIRE RETARDANT WOVEN GLASS PREPREG, HEXCEL
2	90	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
3	90	GR/EP	SAME AS PLY NO. 2
4	135, 45	GL/EP	SAME AS PLY NO. 1
5	0	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
6	0	↓	↓
7	0		
8	0		
9	0		
10	0		
11	0		
12	45		
13	135		
14	0		
15	0		
16	0		
17	0		
18	0		
19	0		
20	0		
21	135		
22	45		
23	90		
			SYM.

Volume Ratio of Materials in Panel, Calculated

- Graphite: 51%
- Glass: 11%
- Epoxy: 34%
- Fire-ratardant epoxy: 4%.

Material and Processing Costs

The material and processing costs for this laminate should be slightly less than for the control panel. Eight angle plies of unidirectional Gr/Ep are replaced by four plies of woven cloth 7781/F-164 (Hexcel) prepreg, so layup time and material costs are reduced.

R81-0911-021D

TABLE 21 FIRE-RETARDANT EPOXY WITH WOVEN GLASS INTERNAL PLIES, LAMINATE NO. 17 (Sheet 2 of 2)

Environmental Stability
The effects of moisture and of chemicals will be similar to that of the control panel, but the thermal resistance should be superior. The brominated novolac-epoxy matrix will confer fire retardance to the surface of the panel and in combination with the fiberglass provide a combination char-mechanical barrier to prevent escape of carbon fibers. In this rationale the fact that fire-retardant epoxy takes a 20% reduction in matrix dominated properties precludes its use throughout the laminate.

R81-0911-021D

TABLE 22 SODIUM SILICATE GLASS SCRIM ALTERNATING PLIES, LAMINATE NO. 18 (SHEET 1 OF 2)

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
2	•	GL/SOD. SIL.	104 FIBERGLASS SCRIM + 5% SODIUM SILICATE
3	135	GR/EP	SAME AS PLY NO. 1
4	90	GR/EP	SAME AS PLY NO. 1
5	•	GL/SOD. SIL.	SAME AS PLY NO. 2
6	90	GR/EP	LIKE PLY 1
7	135	GR/EP	LIKE PLY 1
8	•	GL/SOD. SIL.	LIKE PLY 2
9	45	GR/EP	
10	0	GR/EP	
11	•	GL/SOD. SIL.	
12	0	GR/EP	
13	0	GR/EP	
14	•	GL/SOD.SIL.	
15	0	GR/EP	ALTERNATING TWO PLIES AS-1/3501A UNI-TAPE WITH ONE PLY OF GL/SODIUM SILICATE
16	0	GR/EP	
17	•	GL/SOD. SIL.	
18	0	GR/EP	
19	0	GR/EP	
20	•	GL/SOD. SIL.	
21	45	GR/EP	
22	135	GR/EP	
23	•	GL/SOD. SIL.	
24	0	GR/EP	
25	0	GR/EP	
26	•	GL/SOD. SIL.	
27	0	GR/EP	
28	0	GR/EP	
29	•	GL/SOD. SIL.	
30	0	GR/EP	
31	0	GR/EP	
32	•	GL/SOD. SIL.	
33	0	GR/EP	
34	135	GR/EP	
35	•	GL/SOD. SIL.	
36	45	GR/EP	
37	90	GR/EP	

R81-0911-022D(1/2)

• SYM; IS ALSO A PLY OF GL/SODIUM SIL

TABLE 22 SODIUM SILICATE GLASS SCRIM ALTERNATING PLIES, LAMINATE NO. 18 (SHEET 2 OF 2)

Volume Ratio of Materials in Panel, Calculated

- Graphite: uni-tape 55%
- Epoxy: 36%
- Glass: 7 to 8%
- Sodium silicate: 1 to 2%.

Material and Processing Costs

Material and processing costs will be higher than for the control panel; the same amount of Gr/Ep unidirectional tape will be used but additional style 104 scrim will be treated with sodium silicate, dried and incorporated into the layup. Material costs will be higher by $2 to 3/lb, and layup time will increase by about 1/2 hr/lb. Comments for thin panel No. 9 are appropriate here.

Environmental Stability

The environmental stability will be similar to the control thermally and chemically but the sodium silicate may adversely affect moisture resistance. However, the rationale for choosing this additive is given in Ref. (1) and backed up by the preliminary test described earlier. It should be noted that addition of the 104 glass scrim/sodium silicate plies as shown (each ply affecting two graphite plies) will yield a 5 to 6% weight penalty, and the panel will be about 0.64 mm (0.025 in.) thicker than an all Gr/Ep panel. These extra plies will act like the glass scrim in boron laminates; the addition of sodium silicate will cause clumping of the graphite fibers and prevent their release.

R81-0911-022D(2/2)

TABLE 23. 6% BORON POWDER BETWEEN GR/EP PLIES, LAMINATE NO. 5A

PLY NO.	PLY CONFIG, °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
2	135		
3	0		
4	0		
5	90		BORON POWDER (325 MESH) INTERSPERSED BETWEEN EACH GR/EP PLY
6	90		
7	0		
8	0		
9	135		
10	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE

R81-0911-023D

Volume Ratio of Materials in Panel, Calculated

- Graphite: 55 to 60% (generally normalized to 60%)
- Epoxy: 38 to 40%
- Boron Powder: 6% by weight.

Material and Processing Costs

- Gr/Ep: Hercules AS-1/3501-5A: 3-in. tape costs $45/lb; 12-in.-wide tape costs $42/lb
- Boron: -325 mesh powder, Alfa Division, Ventron Corp., $45/100 gm
- Processing, which includes lay-up, compaction, bag and bleeder application, cure, post-cure (assume piggy-back runs) and trim, takes approximately three hours per pound.

Environmental Stability

The overall chemical stability of cured AS-1/3501-5A laminates is very good. The combination of heat and moisture cause swelling and plasticization of the laminate with subsquent loss of strength at temperature, although dry heat alone causes little strength drop-off, up to 260 to 270°F. This effect is attributed to the resin component of the laminate (Ref. 6, Subsection 1.1.1). Inclusion of fine boron powder can cause some reduction in interlaminar shear strength.

Rationale for Choice

1. Selected PAN-based graphite because of lower conductivity in low modulus range; also present pitch-base graphite has low strain to failure.
2. AS-1/3501-5A has better char-forming resin (DDS-cure) than other types (TETA, MPDA).
3. Cross-plied laminates show particulate problem more than unidirectional.
4. Grumman has high experience factor with Hercules AS-1/3501-5A.
5. Boron powder has been reported by NASA-Lewis to promote char formation and reduce fly-off of graphite fragments during combustion.

R81-0911-023D

TABLE 24 SODIUM BORATE-GLASS SCRIM ALTERNATING PLIES, LAMINATE NO. 18A (Sheet 1 of 2)

PLY NO.	CON-FIG., °	MATERIAL	TYPE
1	45	GR/EP	AS-1/3501-5A, HERCULES UNI-TAPE
2	–	GL/SOD.BOR.	104 FIBERGLASS SCRIM + 5% SODIUM BORATE
3	135	GR/EP	SAME AS PLY NO.1
4	90	GR/EP	SAME AS PLY NO.1
5	–	GL/SOD.BOR.	SAME AS PLY NO.2
6	90	GR/EP	LIKE PLY 1
7	135	GR/EP	LIKE PLY 1
8	–	GL/SOD.BOR.	LIKE PLY 2
9	45	GR/EP	
10	0	GR/EP	
11	–	GL/SOD.BOR.	
12	0	GR/EP	
13	0	GR/EP	
14	–	GL/SOD.BOR.	
15	0	GR/EP	ALTERNATING TWO PLIES AS-1/3501-5A UNI-TAPE
16	0	GR/EP	WITH ONE PLY OF GL/SODIUM BORATE
17	–	GL/SOD.BOR.	
18	0	GR/EP	
19	0	GR/EP	
20	–	GL/SOD.BOR	
21	45	GR/EP	
22	135	GR/EP	
23	–	GL/SOD.BOR.	
24	0	GR/EP	
25	0	GR/EP	
26	–	GL/SOD.BOR.	
27	0	GR/EP	
28	0	GR/EP	
29	–	GL/SOD.BOR.	
30	0	GR/EP	
31	0	GR/EP	
32	–	GL/SOD.BOR.	
33	0	GR/EP	
34	135	GR/EP	
35	–	GL/SOD.BOR.	
36	45	GR/EP	
37	90	GR/EP	
—	—	— — —	— — — SYM.; IS ALSO A PLY OF GL/SOD. BORATE

R81-0911-024D

TABLE 24 SODIUM BORATE-GLASS SCRIM ALTERNATING PLIES LAMINATE NO. 18A (Sheet 2 of 2)

Volume Ratio of Materials in Panel, Calculated

Graphite: Uni-tape, 55%
Epoxy: 36%
Glass: 7 to 8%
Sodium Borate: 1 to 2%

Material and Processing Costs

Material and processing costs will be higher than for the control panel; the same amount of Gr/Ep unidirectional tape will be used but additional style 104 scrim will be treated with sodium borate, dried and incorporated into the layup. Material costs will be higher by $2 to 3/lb, and layup time will increase by about 1/2 hr/lb. Comments for thin panel No. 9 are appropriate here.

Environmental Stability

The environmental stability will be similar to the control (thermally and chemically) but the sodium borate may adversely affect moisture resistance. It should be noted that addition of the 104 glass scrim/sodium borate plies as shown (each ply affecting two graphite plies) will yield a 5 to 6% weight penalty, and the panel will be about 0.64 mm (0.025 in.) thicker than an all-Gr/Ep panel. These extra plies will act like the glass scrim in boron laminates; the addition of sodium borate will cause clumping of the graphite fibers and prevent their release.

R81-0911-024D

Laminate No. 13 was built twice: one panel was painted with intumescent coating and the second left bare. The uncoated panel was used for tests where the coating would have interfered with the test; the intumescent paint decomposes at temperatures above 200°C (293°F) and is degraded during thermal exposure tests via a non-intumescent process. Laminate No. 18 was relaminated after initial testing showed that the sodium silicate treatment was too severe for the glass scrim cloth. Sodium borate was substituted (Table 24).

In Tables 5 through 24, the first column describes the ply-stacking sequence, the second column the ply configuration, the third column the generic materials

selected and the fourth column the specific type of material. These tables also report estimates of the volume ratios of the constituents, material and processing costs, a statement of the environmental stability, and the rationale for choosing the hybridizing constituents.

3.2 CONCEPT FABRICATION AND EVALUATION (TASK II)

This task involved procurement of selected materials; conversion of the prepregs into laminates; and testing to determine their quality, physical and mechanical properties both before and after environmental conditioning, and graphite particulate retention characteristics. The test results were analyzed and eight of the candidate laminates selected (four thin, four thick) for submittal to NASA. The task flow plan is presented in Fig. 1.

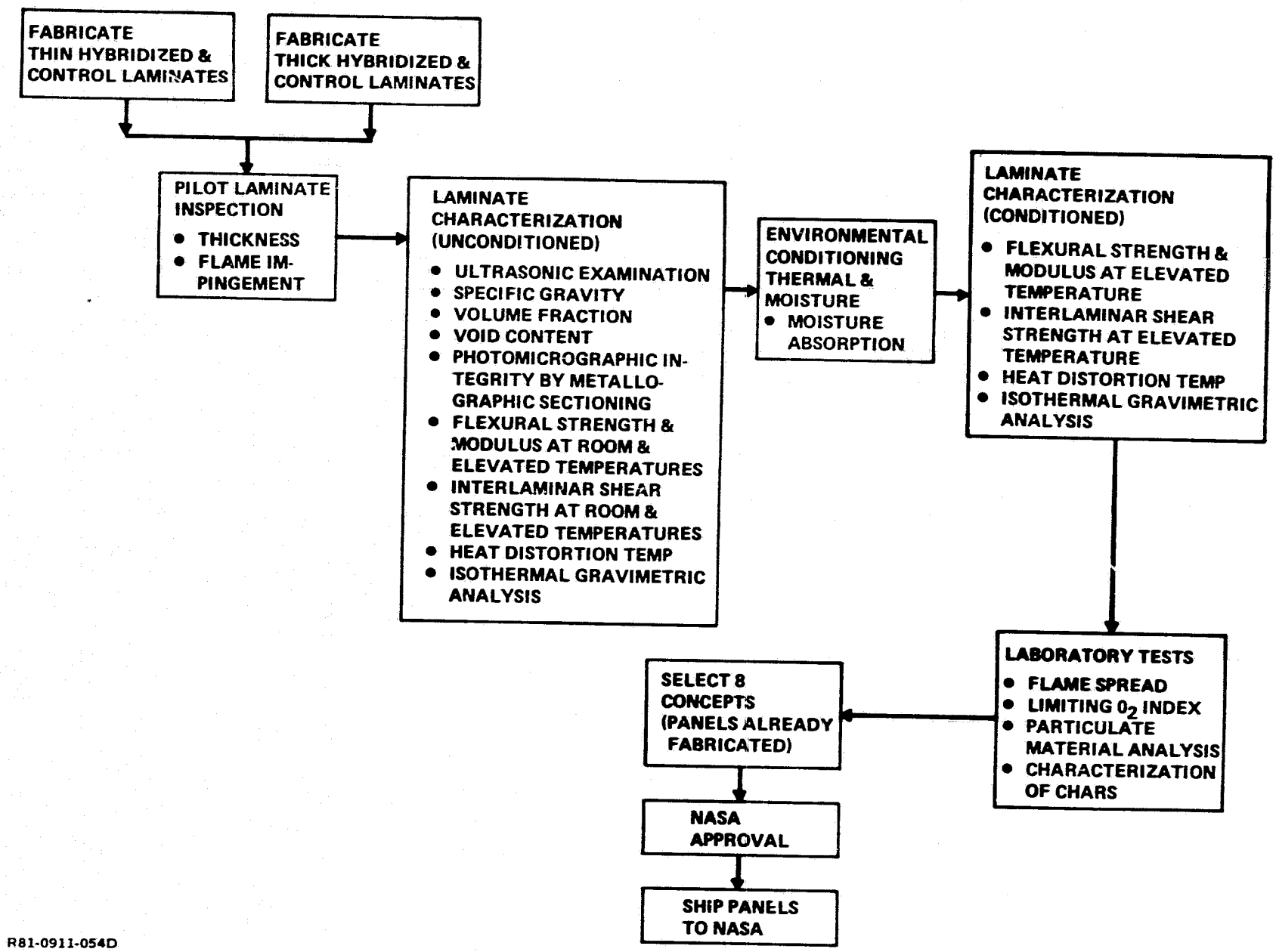
3.2.1 Materials

Table 25 lists the primary materials used in the study. The sodium silicate- and sodium borate-treated Style 104 fiberglass scrim cloth and boron powder/epoxy resin "paint" were made in Grumman's Laboratories. Since woven graphite fabric sized with NR-150B2 could not be made by the prepreggers, it also was prepared by Grumman using NR-150 B2 resin solution from Hercules. The boron powder was made into a paint with 3501-5A epoxy resin and MEK solvent, and brushed onto each Gr/Ep unitape layer and on the outer surfaces. Ideally, the boron powder should have been dispersed in the resin before the graphite was prepregged; failure to accomplish this resulted in some lowering of unconditioned flexural strength.

3.2.2 Laminate Fabrication

All laminates were layed up by hand, autoclave cured at 177°C/586 Pa/1 hr (350°F/85 psi/1 hr) and oven post-cured at 177°C/4 hr (350°F/4 hr).

Initially, pilot laminates, 15.2 x 15.2 cm (6 x 6 in.), of each concept/material/thickness combination were molded to confirm material compatibility predictions and for preliminary burning tests (propane torch). Then, 45.7 x 66 x 0.13-cm (18 x 26 x 0.050-in.) panels were molded for the thin specimens, and 45.7 x 78.7 x 0.64 cm (18 x 31 x 0.250 in.) panels for the thick specimens. These large panels included coupons for property tests plus 20.3 x 20.3-cm (8 x 8-in.) panels, the eight best of which were delivered to NASA-Lewis. A 15-ply undirectional process control panel was fabricated from baseline Gr/Ep unitape prepreg. These panels were 7.6 x 25.4-cm (3 x 10 in.) in size; they were cut into specimens ans tested in



R81-0911-054D

Fig. 1 Concept Fabrication and Evaluation Task Flow Plan

TABLE 25 MATERIALS

MATERIAL	PHYSICAL FORM	MANUFACTURER	PANEL NO.
GR/EP, AS-1/3501-5A	3- AND 12-IN.-WIDE UNI-DIRECTIONAL TAPE PREPREG	HERCULES	ALL EXCEPT 5 AND 12
PERFORATED AL, 5052 ALLOY	0.002 IN. FOIL	HEXCEL	2
EA9628 ADHESIVE	0.002 IN. FILM	DEXTER-HYSOL	2
B/EP, AV 5505-4	3-IN.-WIDE UNIDIRECTIONAL TAPE PREPREG	AVCO	3, 11
GL/EP, 7781/F-161	38-IN.-WIDE WOVEN CLOTH PREPREG	HEXCEL	4, 14
GL/P1, 7781/F-178	38-IN.-WIDE WOVEN CLOTH PREPREG	HEXCEL	5,12
GR/PI (F-178)	CELION 6000 12-IN.-WIDE TAPE, NR-150B2 SIZED, UNIDIRECTIONAL	HEXCEL	5, 12
GR/EP, HMF 134/34	42-IN.-WIDE WOVEN CLOTH PREPREG	FIBERITE	6
GR/EP (PI SIZED), AS-1/3501-5A	3-IN.-WIDE UNIDIRECTIONAL TAPE PREPREG, SIZED WITH NR-150B2 PI	HERCULES	7, 16
KIMBAR 814-54-1	KYNOL NOVOLOID PAPER	SCHWEITZER	8
GRAPHITE CLOTH, SODIUM SILICATE TREATED	STYLE W-134 WOVEN CLOTH	FIBERITE AND GRUMMAN LAB.	9
FLAMAREST 1600B	INTUMESCENT COATING	AVCO	13
QU/EP, 581/F-161	38-IN.-WIDE WOVEN QUARTZ CLOTH PREPREG	HEXCEL	13
GR-GL/EP, 7781/F-558	38-IN.-WIDE GRAPHITE-GLASS WOVEN CLOTH PREPREG	HEXCEL	15
GR/EP, PI SIZED AS-1/3501-5A	STYLE W-134 GRAPHITE 42-IN.-WIDE WOVEN CLOTH, NR-150B2-SIZED	FIBERITE AND GRUMMAN LAB	16
GL/EP, 7781/F-164	38-IN.-WIDE WOVEN CLOTH FIRE RETARDANT PREPREG	HEXCEL	17
GL SCRIM, SODIUM SILICATE OR SODIUM BORATE TREATED	STYLE 104 ONE MIL SCRIM CLOTH	CLARK-SCHWEBEL & GAC LAB	18
BORON	–325 MESH POWDER	ALFA-VENTRON	5A
EPOXY RESIN, 3501-5A	SOLID LUMPS	HERCULES	5A
POLYIMIDE RESIN, NR-150 B2-S2X	60% SOLUTION IN ETHANOL	HERCULES/ DUPONT	16
SODIUM SILICATE	TECH GRADE, SOLUBLE POWDER	FISHER	9
SODIUM BORATE (BORAX)	ANAL. REAGENT, SOLUBLE CRYSTALS	MALLINKRODT	18

R81-0911-025D

flexure at room temperature and 177°C (350°F). The flexural strengths and moduli exceeded Grumman's process control requirements.

3.2.3 Pilot Laminate Examination

The thickness of the 15.2 x 15.2-cm (6 x 6-in.) pilot laminates was measured at various locations. Then, the laminates were machined into 2.54-cm- (1.0-in.-) wide strips oriented such that the zero-degree plies were in the longitudinal direction. These strips (coupons) were used for preliminary burning tests (Table 26 and Table A-1) for an initial determination of graphite fiber retention after laminate exposure to severe thermo-oxidative exposure. Visual inspection of the pilot laminates using microscopic procedures was also used as a preliminary criteria for sound hybridized laminates. All pilot laminates were found acceptable per the above preliminary screening (Table 27) and fabrication of the larger laminates was, therefore, initiated.

TABLE 26 PRELIMINARY BURNING TEST CONDITIONS

HEAT SOURCE:	PROPANE TORCH, T = 954°C (1750°F) SPECIMENS HELD 2.54 CM (1.0 IN.) FROM NOZZLE AND ROTATED, TAPPED AND SHAKEN DURING COMBUSTION.
BURN TIME:	THREE MINUTES FOR 10-PLY SPECIMENS IN HOOD WITH AIR CIRCULATING PAST SPECIMEN DURING BURN, SIMULATING GOOD BREEZE; 5 MINUTES FOR 50-PLY SPECIMENS.
TESTING:	SPECIMENS WERE 2.54 CM (1.0 IN.) WIDE; ONE END, ABOUT 2.54 CM (1.0 IN.) LONG, WAS HELD IN FLAME. THE SPECIMENS WERE NOT SLIT. IT WAS FELT THAT IN THIS WIDTH THE EDGE AND END EFFECTS WERE AS EFFECTIVE AS SLITTING WIDER SPECIMENS. THE PROPANE FLAME WAS PLAYED ON THE END AND BOTH EDGES OF THE SPECIMENS AS WELL AS ON THE FACES. THE SPECIMENS WERE CONSTANTLY ROTATED AND TAPPED DURING IGNITION.

R81-0911-026D

3.2.4 Concept/Laminate Characterization

The laminates were characterized by measurement of physical, mechanical, chemical and thermal properties before and after thermal and moisture conditioning. Characterization testing included ultrasonic examination; photomicrographic integrity by metallurgical sectioning; measurement of specific gravity, constituent volume fraction and void contents, flexural strength and modulus, shear strength, and heat distortion temperature; and isothermal gravimetric analysis. In addition, flame spread, limiting oxygen index and particulate material analysis tests were conducted.

TABLE 27 INSPECTION RESULTS; HYBRIDIZED POLYMER MATRIX COMPOSITE PILOT LAMINATES

LAMINATE NO.	PANEL NO.	NO. OF PLIES	MEASURED THICKNESS, mm (in.)	FLAME TEST	
				CHAR FORMATION	FIBER RETENTION
1	1-13	10	1.4 (0.055)	MINIMAL	POOR
2	2-13	12	1.3 (0.050)	MINIMAL	POOR
3	3-13	10	1.4 (0.054)	MINIMAL	GOOD
4	4-13	8	1.2 (0.049)	MINIMAL	GOOD
5A	5A-13	10	1.3 (0.050)	FAIR	GOOD
6	6-13	8	1.2 (0.049)	MINIMAL	EXCELLENT
7	7-12	10	1.4 (0.054)	MINIMAL	POOR
8	8-13	12	1.7 (0.066)	MINIMAL	POOR
9	9-13	8	1.3 (0.050)	FAIR	EXCELLENT
10	10-13	50	5.7 (0.223)	GOOD	GOOD
11	11-13	50	6.0 (0.236)	VERY GOOD	EXCELLENT
12	12-13	44	6.4 (0.251) (1)	EXCELLENT	VERY GOOD
13	13-13	50	5.4 (0.213)	EXCELLENT	EXCELLENT
14	14-13	44	5.7 (0.223)	VERY GOOD	VERY GOOD
15	15-13	44	6.3 (0.249)	GOOD	GOOD
16	16-13	44	6.3 (0.249)	EXCELLENT	VERY GOOD
17	17-13	46	6.9 (0.271)	VERY GOOD	VERY GOOD
18	18-13	74	7.3 (0.287)	GOOD	EXCELLENT

NOTE: (1). UNCOATED; WHEN COATED WITH FLAMAREST 1600B THE PANEL WAS 6.1 mm (0.239 in.) THICK

R81-0911-027D

3.2.4.1 Unconditioned Laminate Characterization

3.2.4.1.1 Ultrasonic Inspection. The full-size laminates were ultrasonically inspected by the pulso-echo reflector plate technique, more commonly known as ultrasonic "C"-scan. The thin panels (Laminates No. 1 to 9) were generally satisfactory, except for the Gr/F-178 PI panel (Laminate No. 5). This panel appeared to be resin-starved and delaminated during cure/post-cure. The thick panels (Laminates No. 10-18) were less satisfactory, showing various degrees of voids, a condition sometimes seen when thick, multi-ply, unitape and woven-graphite prepregs are interlayered. These results are summarized in Table 28.

3.2.4.1.2 Photomicrographic Integrity. Laminates were inspected by edge photomicrographic analysis to determine the presence of voids, cracks, and fiber orienta-

TABLE 28 TEST RESULTS; ULTRASONIC EXAMINATION, PHOTOMICROGRAPHIC INTEGRITY, AND SPECIFIC GRAVITY

LAMINATE NO.	PANEL NO.	NO. OF PLIES	MEASURED THICKNESS, MM(IN.)(1)	ULTRASONIC EXAMINATION	PHOTOMICROGRAPHIC INTEGRITY	SPECIFIC GRAVITY GM/CM^3(LB/IN^3)
1	1-11	10	1.4(0.055)	SATISFACTORY	SATISFACTORY	1.583(0.057)
2	2-11	12	1.6(0.064)	SOME VOIDS BETWEEN LAMINATE AND FOIL	SOME VOIDS BETWEEN LAMINATE AND FOIL	1.609(0.058)
3	3-11	8	1.3(0.052)	SATISFACTORY	SATISFACTORY	1.777(0.064)
4	4-11	8	1.3(0.052)	SATISFACTORY	SATISFACTORY, WOVEN GL/EP IDENTIFIED UNDER POLARIZED LIGHT	1.714(0.062)
5	5-11	8	1.4(0.057)	VISUAL DELAMINATION	VISUAL DELAMINATION	— —
5A	5A-11	10	1.5(0.059)	SATISFACTORY	SATISFACTORY	1.552(0.056)
6	6-11	8	1.2(0.049)	SATISFACTORY	SATISFACTORY	1.514(0.055)
7	7-11	10	1.3(0.053)	SATISFACTORY	SATISFACTORY	1.548(0.056)
8	8-11	12	1.8(0.069)	SATISFACTORY	SATISFACTORY	1.531(0.055)
9	9-11	8	1.3(0.052)	SATISFACTORY	SATISFACTORY	1.482(0.054)
10	10-11	50	6.9(0.273)	SOME VOIDS	SATISFACTORY	1.555(0.056)
11	11-11	50	6.9(0.270)	SOME VOIDS	SATISFACTORY	1.629(0.059)
12	12-11	44	6.7(0.265)	NUMEROUS VOIDS, ONE CORNER DELAMINATED	SATISFACTORY IN AREA EXAMINED, VOIDS ELSEWHERE	1.627(0.059)
13	13-11	50	7.2(0.284)	NUMEROUS VOIDS	NOT EXAMINED	— —
13A	13A-11	50	7.7(0.304)(2)	SATISFACTORY	SATISFACTORY	1.541(0.056)
14	14-11	44	7.1(0.279)	SOME VOIDS	SOME VOIDS IN OUTER GR/EP PLY UNDER POLARIZED LIGHT	1.614(0.058)
15	15-11	44	6.9(0.271)	NUMEROUS VOIDS	NUMEROUS VOIDS	1.543(0.056)
16	16-11	44	6.9(0.271)	SATISFACTORY	SATISFACTORY, SOME RESIN STARVED AREAS	1.557(0.056)
17	17-11	46	6.9(0.273)	NUMEROUS VOIDS	UNSATISFACTORY, VOIDS UNDER POLARIZED LIGHT	1.591(0.057)
18	18-11	74	8.0(0.314)	NUMEROUS VOIDS	UNSATISFACTORY, VOIDS	1.527(0.057)

NOTES: (1) LAMINATES HAVE EXTERIOR PEEL PLY ON EACH SIDE EXCEPT FOR NO. 2, 8 AND 13A
(2) PEEL PLIES REMOVED AND LAMINATE COATED WITH FLAMAREST 1600B.

R81-0911-028D

tion anomalies. This procedure is useful in determining the compatibility of dissimilar materials in hybrid laminates. Photomicrographs (Appendix B) were taken at a magnification of 100X; some were taken under polarized light in addition to normal light. Comments on the integrity of the specimens are given in Table 28. Peel-ply (Miltex nylon tricot, Style 3921) was left on the specimens, except where non-composite surface-specimens were examined, i.e., aluminum foil-coated. This is shown on top of Specimen 1 (Fig. B-1A), but not in the other photographs. Most of the laminates appeared satisfactory, except for Laminates No. 15, 17, and 18. The photomicrographs of these laminates (Fig. B-2) revealed excessive porosity.

3.2.4.1.3 Specific Gravity. Machined specimens [2.54 x 2.54-cm (1 x 1-in.)] were used to measure specific gravity, volume fraction of constituent materials, and void content. Specific gravity was measured by Method A-1 of ANSI/ASTM D-792-66, "Specific Gravity of Plastics by Displacement." This method involves weighing a one-piece specimen in water; a Sartorius Model 2652 analytical balance was used for these determinations. Average specific gravity values are listed in Table 28.

3.2.4.1.4 Volume Fraction and Void Content. These tests were performed per ANSI/ASTM D-792 and ASTM D-2734 on the same specimens used to measure specific gravity. Due to hybridization of the graphite/epoxy laminates with different reinforcements and resins, the volume fraction wet analyses (and the dependent void-content calculations) were difficult to perform in several cases, as noted.

The general technique for volume fraction determination was to initially determine the weight percentages of resin and fiber by a resin digestion technique; digestive media were chosen which did not attack the reinforcement fibers. Depending on the fibers used in each laminate specimen, either nitric acid, sulfuric acid plus hydrogen peroxide, or ethylene glycol plus potassium hydroxide was used. Several small pieces (approximately 0.5 to 1.0 g total) of each specimen were weighed on an analytical balance. The sample was completely dissolved in the hot digestive medium and the fibers collected in tared 30-ml, coarse-porosity, glass Gooch crucibles. After drying, the weight of the collected fiber was obtained, and the weight percentages of resin and fiber were directly calculated.

Volume fractions of each constituent were obtained using the formula found in the D-3171 procedure. The previously determined (in-house) value of laminate specific gravity and vendor-supplied values for resin and fiber density were also required for this calculation.

Void contents of the composite specimens were determined per Method B of ASTM D-2734, "Void Content of Reinforced Plastics." This calculation required the previously determined values of laminate specific gravity, resin weight percentage, fiber weight percentage, and vendor-supplied data for resin and fiber densities.

The analytical and calculated values for volume and weight fraction of specimen constituents, and for void content, are presented in Table 29. The resultant values for "percent fiber volume" were all acceptable based on a program requirement range of 50 to 60 percent.

3.2.4.1.5 Flexural Strength and Modulus. The flexural tests were performed on universal testing machines (UTM's) of the constant-rate-of-head-movement type; these machines are verified semi-annually per ASTM E-4. Test loads were applied at a crosshead rate of 0.05 in./min; tests were conducted at 23°C (27°F)/50% R.H. and at 125°C (260°F). Elevated-test temperatures were provided by large-volume circulating-air environmental chambers which mate with the UTM's. Thermocouples were attached to the surface of the specimens and monitored throughout the elevated temperature tests with potentiometers.

The flexure specimens were uniform, rectangular-cross-section, center-loaded, simply supported beams tested at span-to-depth ratios of 32:1. They were tested to failure with their center deflection autographically recorded as a function of load application. Unconditioned flexural strength and modulus values are reported in Table 30. The test results show that most of the thin laminates were equivalent or superior to the Gr/Ep control, Laminate No. 1, both at room and elevated temperatures, with the exception of Laminate No. 8. This panel showed approximately half the elevated temperature strength and modulus of the control panel. Laminate No. 8 was made with outer layers of Kimbar flame-barrier surfacing material. Several thin laminates were superior to the Gr/Ep standard, namely Laminates No. 2, 3, and 4, the boron-faced, aluminum foil-faced and woven fiberglass-faced specimens. These laminates had excellent room-and elevated-temperature flexural strength and modulus.

Two of the thick laminates, No. 12 and 18A, had flexural strength and modulus values below those for the control (Laminate No. 10, all Gr/Ep). These laminates were hybridized with PI matrix and sodium borate-treated scrim cloth, respectively. Laminate No. 14, fabricated with woven fiberglass as outer and internal ply bands, and Laminate No. 17, hybridized with flame-retardant epoxy impregnated woven fiberglass outer and internal ply bands, both had superior flexural strength and modulus values compared to the all-Gr/Ep control.

TABLE 29 TEST RESULTS: CONSTITUENT VOLUME FRACTION, VOID CONTENT, AND RELATED PROPERTIES OF UNCONDITIONED SPECIMENS (SHEET 1 OF 3)

PROPERTY	THIN LAMINATES								
	NO. 1	NO. 2	NO. 3	NO. 4	NO. 5(A)	NO. 6	NO. 7	NO. 8	NO. 9
RESIN	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A
WT %	38.8 ± 0.3	32.8 ± 0.8	28.4 ± 0.4	38.0 ± 0.5	36.1 ± 2.0	40.7 ± 0.3	41.3 ± 1.3	45.1 ± 0.3	39.7 ± 0.2
VOL %	42.2 ± 0.4	41.5 ± 0.8	40.0 ± 0.6	51.3 ± 0.3	45.5 ± 2.4	48.5 ± 0.2	49.9 ± 2.2	54.4 ± 0.3	46.4 ± 0.2
SP G	1.27	1.27	1.26	1.27	1.27	1.27	1.27	1.27	1.27
RESIN	—	—	Ep 5505	Ep F-161	—	Ep 934	—	—	—
WT %	—	—	(40% OF	(40% OF	—	(40% OF	—	—	—
VOL %	—	—	ABOVE	ABOVE	—	ABOVE	—	—	—
SP G	—	—	FIGURES)	FIGURES)	—	FIGURES)	—	—	—
FIBER	Gr AS-1	Gr AS-1	Gr AS-1	Gr AS-1	Gr AS-1	AS-1+W-134	Gr AS-1	Gr AS-1	Gr AS-1
WT %	66.2 ± 0.3	55.8 ± 0.7	38.9 ± 1.3	42.6 ± 1.4	61.6 ± 2.1	59.3 ± 0.3	58.7 ± 1.3	54.3 ± 0.3	60.3 ± 0.2
VOL %	58.2 ± 0.2	49.9 ± 0.9	38.4 ± 1.2	40.6 ± 1.7	54.8 ± 2.0	49.9 ± 0.4	50.0 ± 0.7	46.7 ± 0.3	49.6 ± 0.1
SP G	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80
FIBER OR FILLER	—	—	B AVCO	—	B POWDER	Gr W 134	—	—	—
WT %	—	—	29.7 ± 1.2	—	2.3 ± 0.1	23.7 ± 0.3	—	—	—
VOL %	—	—	20.3 ± 0.9	—	1.5 ± 0.1	20.0 ± 0.4	—	—	—
SP G	—	—	2.60	—	2.46	1.69	—	—	—
FIBER	—	—	GI (SCRIM)	GI (SCRIM)	—	Gr AS-1	—	—	—
WT %	—	—	3.1 ± 0.2	19.3 ± 1.0	—	35.6 ± 0.3	—	—	—
VOL %	—	—	2.2 ± 0.1	13.0 ± 0.6	—	30.0 ± 0.4	—	—	—
SP G	—	—	2.54	2.55	—	1.80	—	—	—
LAMINATE									
SP G	1.58 ± 0.00	1.61 ± 0.01	1.78 ± 0.00	1.71 ± 0.01	1.60 ± 0.00	1.51 ± 0.00	1.53 ± 0.02	1.53 ± 0.00	1.48 ± 0.00
VOIDS, V/O	−4. ± 0.2	1.8 ± 0.3	0.8 ± 0.4	−5.0 ± 0.8	−1.8 ± 0.5	1.7 ± 0.2	0.1 ± 1.8	−1.1 ± 0.1	3.9 ± 0.1
COATING	—	AL FOIL	—	—	—	—	—	KIMBAR	—
WT %	—	11.5 ± 0.1	—	—	—	—	—	0.6 APPROX.	—
VOL %	—	6.9 ± 0.1	—	—	—	—	—	—	—
SP G	—	2.7	—	—	—	—	—	—	—

R81-0911-029D(1/3)

TABLE 29 TEST RESULTS; CONSTITUENT VOLUME FRACTION, VOID CONTENT, AND RELATED PROPERTIES OF UNCONDITIONED SPECIMENS (SHEET 2 OF 3)

PROPERTY				THICK LAMINATES					
	NO. 10	NO. 11	NO. 12	NO. 13(A)	NO. 14	NO. 15	NO. 16	NO. 17	NO. 18
RESIN	Ep 3501-5A	Ep 3501-5A	PI F-178	Ep 3501-5A	Ep 3501-5A.	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A	Ep 3501-5A
WT %	35.3 ± 1.8	36.1 ± 1.9(1)	46.1 ± 1.2	42.9 ± 0.1	42.3 ± 1.1(1)	40.9 ± 0.8	38.5 ± 0.8	44.4 ± 0.7	39.7 ± 0.8
VOL %	43.3 ± 1.2	46.6 ± 2.1(1)	57.8 ± 1.4	52.4 ± 0.3	53.8 ± 1.0(1)	49.6 ± 0.8	47.2 ± 0.8	55.7 ± 0.7	49.2 ± 0.7
SP G	1.27	1.26(2)	1.30	1.27(6)	1.27(2)	1.27	1.27	1.27	1.27
RESIN	—	Ep 5505	—	—	Ep F-161	Ep F-558	Ep 934	Ep F-164	—
WT %	—	SEE NOTES	—	—	SEE NOTES	SEE NOTES	(24% OF	(16% OF	—
VOL %	—	(1) & (2)	—	—	(1) & (2)	(1) & (2)	ABOVE	ABOVE	—
SP G	—		—	—			FIGURES)	FIGURES)	—
FIBERS, COMB.	Gr AS-1	Gr + B	Gr + Gl	Gr + Gl	Gr + Gl	Gr+Gl/Gr	Gr UNI + WOVEN	Gr + Gl	Gr + Gl
WT %	64.7 ± 1.2	63.9 ± 1.9(3)	53.9 ± 1.2(5)	57.1 ± 0.3	57.7 ± 0.9	59.1 ± 0.8	61.5 ± 0.8	55.6 ± 0.7	60.2 ± 0.7
VOL %	55.8 ± 1.4	53.3 ± 2.0(3)	47.0 ± 1.1(5)	47.8 ± 0.3	48.9 ± 1.1	49.4 ± 0.8	53.2 ± 0.9	47.4 ± 0.8	51.9 ± 0.8
SP G	1.80	1.96 (4)	1.87	—	—	1.85	1.80	1.87	—
FIBER OR FILLER	—	B AVCO	Gl 7781	Gl 7781	Gl 7781	Gl	Gr W-134	Gl 7781	Gl 104
WT %	—	12.5 CALC	7.5 CALC	5.5 ± 0.3	10.9 ± 0.3	(8)	14.8 ± 0.8	8.9 ± 0.7	3.3 ± 0.1
VOL %	—	10.4 CALC	6.6 CALC	3.3 ± 0.2	6.9 ± 0.2	(8)	12.8 ± 0.9	7.6 ± 0.8	2.1 ± 0.1
SP G	—	2.60	2.55	2.55	2.55	—	1.69	2.55	2.55
FIBER	—	Gr AS-1	Gr CELION	Gr AS-1	Gr AS-1	Gr	Gr AS-1	Gr AS-1	Gr AS-1
WT %	—	51.4 CALC	46.4 CALC	51.6 ± 0.2	46.8 ± 0.9	(8)	46.7 ± 0.8	46.7 ± 0.7	56.9 ± 0.7
VOL %	—	42.9 CALC	40.4 CALC	44.5 ± 0.3	42.0 ± 1.1	(8)	40.4 ± 0.9	39.8 ± 0.8	49.8 ± 0.8
SP G	—	1.80	1.76	1.80	1.80	1.80	1.80	1.80	1.80
LAMINATE									
SP G	1.56 ± 0.0	1.63 ± 0.0	1.63 ± 0.0	1.54 ± 0.0	1.61 ± 0.0	1.54 ± 0.0	1.56 ± 0.0	1.59 ± 0.0	1.57 ± 0.0
VOIDS, V/O	0.9 ± 0.5	0.1 ± 0.3	−4.8 ± 0.3	−0.2 ± 0.5	−2.6 ± 0.3	1.0 ± 0.1	−0.4 ± 0.1	−3.1 ± 0.3	−1.1 ± 0.2
COATING	—	—	—	FLAMAREST 16008	—	—	—		—
WT %	—	—	—	4.9 CALC(7)	—	—	—	—	—
VOL %	—	—	—	—	—	—	—	—	—
SP G	—	—	—	—	—	—	—	—	—

R81-0911-029D

TABLE 29 TEST RESULTS; CONSTITUENT VOLUME FRACTION, VOID CONTENT, AND RELATED PROPERTIES OF UNCONDITIONED SPECIMENS (SHEET 3 OF 3)

NOTES:

(1) COMBINED AVERAGE OF THE 3501-5A, 5505-4, F-161, F-558, and F-164 EP RESINS IN THE LAMINATES.

(2) WEIGHTED AVERAGE OF THE TWO RESINS.

(3) COMBINED AVERAGE OF THE GR AND B FIBERS IN THE LAMINATE. THE WEIGHTED FIBER AVERAGE SP. G. = 1.96 WHICH CALCULATES OUT TO 80.5% GR AND 19.5% B.

(4) WEIGHTED AVERAGE OF THE TWO FIBERS.

(5) COMBINED AVERAGE OF THE GR AND GL FIBERS IN THIS LAMINATE. THE WEIGHTED FIBER AVERAGE SP. G. = 1.87 WHICH CALCULATES OUT TO 86% GR AND 14% FIBERGLASS.

(6) SP. G. OF 3501-5A, EP RESIN PLUS THE FLAMAREST COATING.

(7) CALCULATED BY DIFFERENCE BETWEEN AVERAGE EP RESIN CONTENT FOR NORMAL PANELS AND ADDITIONAL WEIGHT FOR THIS PANEL.

(8) NOT DETERMINED ANALYTICALLY DUE TO SEPARATION PROBLEM WITH WOVEN GR/GL.

R81-0911-029D

TABLE 30 TEST RESULTS; FLEXURAL STRENGTH AND MODULUS, UNCONDITIONED SPECIMENS

TYPE OF LAMINATE	LAMINATE NO.	ROOM TEMP. DATA, 24°C (75°F)		ELEVATED TEMP. DATA, 127°F (260°F)	
		FLEX. STRENGTH	FLEX. MODULUS	FLEX. STRENGTH	FLEX. MODULUS
		MPA (KSI)	GPA (MSI)	MPA (KSI)	GPA (MSI)
THIN	1	919.1 (133.3)	37.7 (5.47)	821.2 (119.1)	34.0 (4.93)
	2	937.0 (135.9)	39.3 (5.70)	949.4 (137.7)	41.4 (6.00)
	3	998.4 (144.8)	46.5 (6.75)	908.8 (131.8)	41.0 (5.94)
	4	1001.2 (145.2)	41.7 (6.05)	911.5 (132.2)	35.6 (5.17)
	5A	717.8 (104.1)	38.3 (5.56)	627.4 (91.0)	33.9 (4.92)
	6	1046.0 (151.7)	45.2 (6.55)	747.4 (108.4)	35.9 (5.20)
	7	922.6 (133.8)	36.8 (5.33)	760.5 (110.3)	34.7 (5.03)
	8	568.1 (82.4)	20.3 (2.95)	435.8 (63.2)	19.5 (2.83)
	9	1007.4 (146.1)	43.7 (6.34)	749.5 (108.7)	39.3 (5.70)
THICK	10	1044.6 (151.5)	57.2 (8.29)	933.5 (135.4)	52.8 (7.66)
	11	1006.7 (146.0)	56.5 (8.19)	942.5 (136.7)	56.0 (8.12)
	12	758.5 (110.0)	54.3 (7.87)	771.6 (111.9)	52.7 (7.64)
	13A	977.7 (141.8)	50.1 (7.27)	955.6 (138.6)	47.4 (6.87)
	14	1073.6 (155.7)	51.4 (7.45)	966.7 (140.2)	50.8 (7.37)
	15	1057.0 (153.3)	52.2 (7.57)	961.9 (139.5)	50.5 (7.32)
	16	1040.5 (150.9)	52.8 (7.66)	919.8 (133.4)	53.0 (7.68)
	17	1092.2 (158.4)	50.3 (7.30)	1043.9 (151.4)	48.5 (7.03)
	18A	901.9 (130.8)	49.8 (7.22)	960.5 (139.3)	49.4 (7.17)
R81-0911-030D					

NOTES:

(1) ALL DATA ARE THE AVERAGE OF THREE TESTED SPECIMENS.

(2) ALL SPECIMENS WERE CORRECTED FOR 0.006-IN. PEEL-PLY, EXCEPT FOR LAMINATE NO. 2 (AL FOIL-COATED) AND LAMINATE NO. 8 (KIMBAR FLAME BARRIER-COATED).

The room-temperature flexural strength and modulus values were interpreted and compared with the predicted values; in-plane strength and modulus are also compared and ranked in Table 31. Comparisons can only be made qualitatively because the test measurements are of flexural strength and the predictions are based on in-plane lamination theory.

TABLE 31 RANKED FLEXURE TEST RESULTS AND IN-PLANE PREDICTIONS

LAMINATE NO.	TEST (FLEXURE)		PREDICTION (IN-PLANE)	
	F_x^b	E_{xb}	F_x^{tu}	E_x
	MPA (KSI)	MPA (KSI)	MPA (KSI)	MPA (KSI)
6	1046.0 (151.7)	45.2 (6.55)	751.6 (109.0)	65.6 (9.5)
9	1007.4 (146.1)	43.7 (6.34)	715.7 (103.8)	74.5 (10.8)
4	1001.2 (145.2)	41.7 (6.05)	709.5 (102.9)	62.8 (9.1)
3	998.4 (144.8)	46.5 (6.75)	797.8 (115.7)	69.7 (10.1)
2	937.0 (135.9)	39.3 (5.70)	746.0 (108.2)	65.6 (9.5)
7	922.6 (133.8)	36.8 (5.33)	746.0 (108.2)	65.6 (9.5)
1	919.1 (133.3)	37.7 (5.47)	746.0 (108.2)	65.6 (9.5)
5A	717.8 (104.1)	38.3 (5.56)	746.7 (108.3)	65.6 (9.5)
8	568.1 (82.4)	20.3 (2.95)	746.0 (108.2)	65.6 (9.5)
17	1092.2 (158.4)	50.3 (7.30)	924.0 (134.1)	81.4 (11.8)
14	1073.6 (155.7)	51.4 (7.45)	923.2 (133.9)	81.4 (11.8)
15	1057.0 (153.3)	52.2 (7.57)	979.1 (142.0)	85.6 (12.4)
10	1044.6 (151.5)	57.2 (8.29)	937.0 (135.9)	82.1 (11.9)
16	1040.5 (150.9)	52.8 (7.66)	940.5 (136.4)	82.1 (11.9)
11	1006.7 (146.0)	56.5 (8.19)	966.0 (140.1)	84.9 (12.3)
13	977.7 (141.8)	50.1 (7.27)	934.3 (135.3)	82.1 (11.9)
18A	901.9 (130.8)	49.8 (7.22)	859.8 (124.7)	75.2 (10.9)
12	758.5 (110.0)	54.3 (7.87)	938.4 (136.1)	86.3 (12.5)

R81-0911-031D

The flexural strength (F_x^b) is measured on the outermost ply strains, which are ±45° layers and can take much higher strains than the 0° layers. However, the in-plane prediction of strength (F_x^{tu}) is based upon lamination theory, i.e., the average strain of the specimen. Therefore, the test values of F_x^b are usually higher than those of in-plane predicted values of F_x^{tu}.

In flexural tests, the transverse shear deformation increases the deflection of the test specimens, resulting in values of bending moduli, E_x^b, lower than predicted.

Test results and in-plane predictions are in qualitative agreement, in spite of the above mentioned discrepancies.

3.2.4.1.6 Interlaminar Shear Strength. Interlaminar (horizontal) shear strength tests were performed on the candidate laminates using equipment and procedures described for the flexure tests. Specimens were tested to failure at a span-to-depth ratio of 5:1. Shear strengths are reported in Table 32.

TABLE 32 TEST RESULTS; INTERLAMINAR (HORIZONTAL) SHEAR STRENGTH, UNCONDITIONED SPECIMENS

TYPE OF LAMINATE	LAMINATE NO.	ROOM TEMP., 24°C (75°F) SHEAR STRESS MPA (KSI)	ELEVATED TEMP., 260°F (127°C) SHEAR STRESS MPA (KSI)
THIN	1	44.8 (6.50)	40.0 (5.80)
	2	46.5 (6.75)	49.0 (7.11)
	3	41.1 (5.96)	34.7 (5.03)
	4	35.9 (5.21)	33.9 (4.91)
	5A	40.4 (5.86)	44.5 (6.46)
	6	32.5 (4.72)	29.6 (4.03)
	7	41.9 (6.07)	31.3 (4.54)
	8	33.6 (4.87)	29.6 (4.30)
	9	39.9 (5.79)	26.6 (3.86)
THICK	10	42.4 (6.15)	39.2 (5.69)
	11	50.7 (7.35)	41.2 (5.97)
	12	26.8 (3.88)	26.7 (3.87)
	13	38.3 (5.55)	34.3 (4.97)
	13A	39.5 (5.73)	36.5 (5.30)
	14	43.2 (6.27)	39.7 (5.76)
	15	42.5 (6.17)	40.4 (5.86)
	16	35.5 (5.15)	35.4 (5.13)
	17	53.9 (7.82)	43.8 (6.35)
	18A	51.9 (7.53)	46.7 (6.77)

NOTE: DATA ARE THE AVERAGE OF THREE TESTED SPECIMENS.
R81-0911-032D

The test results indicate that only two of the thin laminates were equivalent to or superior than the Gr/Ep control (Laminate No. 10) at room temperature; these were Laminates No. 2 and 7, the aluminum foil-coated and the NR-150B 2-sized panels. Most of the laminates had interlaminar shear strengths at ambient temperature within 12% of the control.

Elevated-temperature test results on the thin specimens showed that two candidates, Laminates No. 2 and 5A (the aluminum foil-coated and the boron powder/matrix), had increased shear strength and were 22% and 11% stronger, respectively, than the Gr/Ep control. The rest of the specimens showed the more usual decrease in shear strength, ranging from 13% to 33% (for Laminate No. 9).

Among the thick laminates, five candidates were equal to or exceeded the measured interlaminar shear strength of the Gr/Ep control at room temperature. These were Laminates No. 11, 14, 15, 17, and 18A. Laminate No. 17, hybridized with fire-retardant resin on woven fiberglass internal plies, exceeded the strength of the control by 27%. The poorest laminate was No. 12, made with a PI matrix, which had properties 37% lower than those for the control.

At elevated temperatures, all test laminates showed a reduction (or equivalence) in interlaminar shear strength, which is the normal mode of behavior. The same five laminates discussed above were again superior to the Gr/Ep control; Laminate No. 18, which contained sodium borate-treated fiberglass scrim cloth alternating plies, exceeded the strength of the control by 19%. Again, Laminate No. 12 was poorest, with a reduction of 32% in shear strength.

3.2.4.1.7 Heat Distortion Temperature. It was originally intended to measure the heat distortion temperature (HDT) of the candidate laminates by Standard Test Method ANSI/ASTM D648-72, "Deflection Temperature of Plastics Under Flexural Load." However, the standard HDT test apparatus did not give reliable measurements in the temperature range of 200°C (392°F) and above. Therefore, measurements were made by thermomechanical analyses (TMA) using a Perkin-Elmer Model TMS-1 Thermomechanical Analyzer. This machine measures the Tg (glass transition temperature, a second-order transition in polymers manifested by a change in the rate of expansion as a function of a steady change in the rate of sample heating) by sensing sample expansion via a probe assembly, converting the motion into an electrical signal and displaying (recording) the signal potentiometrically.

The data obtained are reported in Table 33. Most of the laminates show a Tg at or near 200°C (392°F), or higher. Laminate No. 12, fabricated from unidirectional Gr/PI with interlaminar and surface woven Gl/PI layers, showed the highest Tg, 255°C (491°F). This is due to the inherently higher second-order transition temperature associated with PI polymers. The single low Tg, for Laminate No. 13A, which was coated with an intumescent epoxy paint, reflected the low transition temperature of the coating, not that for the laminate on which it was applied.

TABLE 33 TEST RESULTS; HEAT DISTORTION TEMPERATURE (T_g), UNCONDITIONED SPECIMENS

TYPE OF LAMINATE	LAMINATE NO.	TG (HDT) BY TMA °C (°F)
THIN	1	206 ± 0 (403 ± 0)
	2	211 ± 2 (411 ± 5)
	3	207 ± 3 (405 ± 5)
	4	215 ± 5 (420 ± 9)
	5A	200 ± 4 (392 ± 7)
	6	204 ± 2 (398 ± 3)
	7	198 ± 1 (388 ± 2)
	8	201 ± 2 (394 ± 4)
	9	202 ± 2 (394 ± 4)
THICK		
	10	206 ± 1 (403 ± 2)
	11	199 ± 2 (390 ± 3)
	12	255 ± 1 (491 ± 2)
	13	208 ± 1 (407 ± 1)
	13A	183 ± 1 (361 ± 2)
	14	212 ± 1 (414 ± 2)
	15	213 ± 1 (415 ± 1)
	16	200 ± 1 (342 ± 2)
	17	208 ± 2 (407 ± 4)
	18A	203 ± 2 (397 ± 3)

R81-0911-033D

3.2.4.1.8 Isothermal Gravimetric Analysis (ITGA). This test provides a determination of the response of laminate materials to a thermo-oxidative medium.

The tests were performed in a standard laboratory thermogravimetric analyzer utilizing an air atmosphere. The ITGA test provides the magnitude of laminate weight loss verus time, when the laminate is held at a temperature equivalent to the previously determined heat distortion or second-order transition temperature. The thermogravimetric analyzer also provides a determination of the rate at which a given laminate loses weight in the thermo-oxidative medium.

The ITGA tests were performed for 30 days at 200°C (392°F) for Laminates No. 5A, 7, 8, 9, 11, 16, and 18; at 206°C (403°F) for Laminates No. 1, 3, 6, 10, 13 and 17; at 213°C (415°F) for Laminates No. 2, 4, 14 and 15; and at 225°C (437°F) for

Laminate No. 12. Test results are presented in Table 34. Several conclusions can be drawn from these test results for the unconditioned laminates. Generally, thick laminates were more stable than the thin laminates. Of the thin laminates, the most stable was Laminate No. 2, the aluminum foil-coated Gr/Ep specimen; this laminate lost weight at half the rate of the uncoated Gr/Ep control. The Kimbar-faced, thin laminate was about 14% more effective than the control.

TABLE 34 TEST RESULTS; ISOTHERMAL GRAVIMETRIC ANALYSIS, UNCONDITIONED SPECIMENS

TYPE OF LAMINATE	LAMINATE NO.	WEIGHT LOSS, %	DEVIATION, %	TEST TEMPERATURE °C (°F)	TEST DURATION, DAYS
THIN	1	5.95	±0.14	206 (403)	30
	2	3.17	±0.34	213 (415)	30
	3	5.29	±0.25	206 (403)	30
	4	6.33	±0.30	213 (415)	30
	5	5.47	±0.03	200 (392)	30
	6	6.62	±0.08	206 (403)	30
	7	6.21	±0.14	200 (392)	30
	8	5.12	±0.10	200 (392)	30
	9	5.40	±0.05	200 (392)	30
THICK	10	2.94	±0.17	206 (403)	30
	11	2.69	±0.12	200 (392)	30
	12	16.30	±0.80	255 (491)	30
	13	3.38	±0.07	206 (403)	30
	13A (1)	–	–	–	–
	14	3.57	±0.18	213 (415)	30
	15	4.21	±0.21	213 (415)	30
	16	3.63	±0.09	200 (392)	30
	17	2.96	±0.18	206 (403)	30
	18	2.67	±0.08	200 (392)	30

R81-0911-034D

(1) THIS SPECIMEN WAS COATED WITH INTUMESCENT PAINT; THE COATING FROTHED AND DECOMPOSED, RENDERING LAMINATE MEASUREMENTS USELESS. DECOMPOSITION OF THIS COATING AT 200°C (392°F) IS NORMAL, ALTHOUGH IT TAKES PLACE SLOWLY. THE MANUFACTURER STATES THAT DECOMPOSITION BEGINS AT 260° C (500°F). GRUMMAN OBSERVED THAT DECOMPOSITION BEGINS AT THE LOWER 200°C TEMPERATURE.

Among the thick laminates, No. 12, made with a PI matrix, was the highest in its weight loss rate, almost six times greater than the Gr/Ep control. Another general

trend observed was that laminates containing woven internal and surface plies lost weight faster than the standard. Only the boron and sodium-borate-treated interlaminar glass scrim panels lost weight more slowly than the Gr/Ep control.

3.2.4.2 Environmental Conditioning

Sections of the candidate laminates were conditioned in thermal and moisture environments such that comparisons of the laminate properties before, during and after conditioning would permit assessment of the potential utility of the candidate concepts with respect to anticipated commercial aircraft usage. For these tests, the laminates were cut to final specimen configuration.

3.2.4.2.1 Thermo-Oxidative Conditioning. The specimens were thermally conditioned in a circulating-air oven for 200 hr at 204°C (400°F) with the exception of specimens from Laminates No. 12 and 13. Laminate No. 12 had a PI matrix and was accordingly conditioned at 254°C (498°F) for 200 hr. Laminate No. 13 was treated without its intumescent coating, which degrades in this type of environment.

3.2.4.2.2 Moisture Conditioning. Test specimens were conditioned in a temperature/humidity chamber set to provide and maintain 95 to 98% relative humidity at 60°C (140°F). The thin laminates were exposed for 16 days with the exception of the specimens from Laminates No. 5 and 7 which were inadvertently conditioned for 21 days. The 16-day exposure period for the thin laminates was the conditioning required at 60°C/98% RH to achieve moisture absorption levels of 1.2% by weight. The thick panel specimens for Laminates No. 10 through 18 were also conditioned for 21 days at 60°C/98% RH to achieve a moisture content of 0.50% by weight. Moisture absorption data for the thin and thick specimens are presented in Tables 35 and 36, respectively.

Of the thin laminates, the specimens from Laminates No. 2 and 4 absorbed significantly less moisture than the control laminate (Laminate No. 1). Laminate No. 3 specimens absorbed approximately the same amount of moisture as the control laminate specimens. Specimens from Laminates No. 6, 8 and 9 absorbed significantly more moisture than the control laminate specimen. Specimens from Laminates No. 5A and 7 (those exposed for 21 days) absorbed significant amounts of water, probably due to their over-exposure.

Laminate No. 2 specimens had a cocured aluminum foil protective coating while Laminate No. 4 specimens had woven fiberglass outer plies. Laminate No. 3 specimens with the B/Ep outer plies were essentially equivalent to the control laminate and, not

TABLE 35 MOISTURE ABSORPTION DATA FOR THIN LAMINATES

ELAPSED TIME, DAYS	LAMINATE MOISTURE PICKUP, % (NOTE 1)								
	NO. 1	NO. 2	NO. 3	NO. 4	NO. 5A (NOTE 2)	NO. 6	NO. 7 (NOTE 2)	NO. 8	NO. 9
0	(3.6439)	(3.9441)	(3.5902)	(3.4271)	(3.7451)	(3.1070)	(3.8070)	(3.9279)	(3.0052)
2	0.49	0.07	0.52	0.49	0.82	0.64	0.69	1.78	.95
4	0.67	0.13	0.70	0.64	–	0.86	–	1.86	1.61
7	0.84	0.15	0.83	0.84	1.17	0.99	0.95	2.01	1.66
10	0.96	0.28	0.85	0.98	1.23	1.15	1.00	2.02	1.83
14	1.06	0.30	0.89	0.94	1.46	1.17	1.10	1.90	1.83
16	1.08 (3.6832)	0.39 (3.9593)	0.91 (3.6230)	0.95 (3.4595)	– –	1.22 (3.1449)	– –	1.86 (4.0010)	1.85 (3.0609)
21	– –	– –	– –	– –	1.60 (3.8051)	– –	1.20 (3.8527)	– –	– –

NOTES: (1) NUMBERS IN PARENTHESES ARE LAMINATE PANEL WEIGHTS IN GRAMS.

(2) LAMINATES NO. 5A AND 7 WERE TESTED AT A LATER DATE THAN THE REST OF THE SPECIMENS. THEIR EXPOSURE WAS CARRIED OUT FOR A LONGER TIME PERIOD BECAUSE THICK LAMINATES WERE INCLUDED IN THE TEST BATCHES.

R81-0911-035D

unexpectedly, absorbed moisture at approximately the same rate. The specimens from Laminates No. 6 (woven Gr/Ep outer plies), No. 8 (Kimbar flame barrier) and No. 9 (silicate-treated woven graphite outer plies) absorbed excessive moisture. This moisture may have been retained primarily by the protective surface layers of the respective laminates.

With respect to moisture absorption, Laminates No. 5A, 8, and 9 were judged unacceptable, while Laminates No. 6 and 7 were judged marginal.

Although the 21-day exposure of the thick laminates did not result in significant moisture pickup relative to that of the thin laminates, it is obvious that Laminates No. 12 (woven glass outer plies - PI matrix), No. 16 (woven-PI treated-graphite outer plies), and No. 18 (borate-treated fiberglass scrim outer plies) picked up excessive moisture (relative to the control panel).

TABLE 36 MOISTURE ABSORPTION DATA FOR THICK LAMINATES

ELAPSED TIME, DAYS	LAMINATE MOISTURE PICKUP, % (NOTE 1)									
	NO. 10	NO.11	NO. 12	NO. 13	NO. 13A (NOTE 2)	NO. 14	NO. 15	NO. 16 (NOTE 2)	NO. 17	NO. 18A (NOTE 2)
0	(16.4451)	(16.7883)	(15.5186)	(16.8199)	(18.4932)	(17.0892)	(15.2791)	(15.4597)	(16.8894)	(18.005)
2	0.15	0.19	0.55	0.14	0.40	0.14	0.13	0.29	0.14	0.28
4	0.22	0.27	0.74	0.21	–	0.20	0.21	–	0.21	–
7	0.29	0.32	0.77	0.27	0.26	0.26	0.27	0.44	0.28	0.46
10	0.35	0.39	0.80	0.34	0.25	0.34	0.33	0.51	0.35	0.53
14	0.42	0.44	0.82	0.41	0.22	0.37	0.39	0.58	0.43	0.63
16	0.43	0.47	0.80	0.43	–	0.43	0.41	–	0.46	–
18	0.47	0.48	0.82	0.46	–	0.46	0.45	–	0.51	–
21	0.51 (16.5386)	0.50 (16.8718)	0.83 (15.6477)	0.51 (16.9057)	0.14 (18.5190)	0.49 (17.1729)	0.49 (15.3539)	0.71 (15.5704)	0.55 (16.9820)	0.83 (18.1542)

NOTE: (1) NUMBERS IN PARENTHESES ARE LAMINATE PANEL WEIGHTS IN GRAMS.

(2) LAMINATES NO. 13A, 16 AND 18A WERE TESTED AT A LATER DATE THAN THE REST OF THE SPECIMENS. OVERALL DURATION OF EXPOSURE WAS THE SAME AS FOR THE REST OF THE THICK LAMINATES.

R81-0911-036D

3.2.4.3 Conditioned Laminate Characterization

Characterization of the conditioned laminate specimens included measurement of specific gravity, constituent volume fraction and void content, flexural strength and modulus, shear strength, and heat distortion temperature. The procedures and specimens used were the same as those used on the unconditioned specimens and described in Subsection 3.2.4.1.

3.2.4.3.1 Specific Gravity. The specific gravity values of the moisture and thermally conditioned candidate laminate specimens are presented in Table 37. The specific gravity values of the unconditioned specimens are included for reference. With respect to moisture conditioning, the change in specific gravity resulting from moisturization was minimal for both the thin and thick laminate concepts. There were also no significant differences in specific gravity following thermal conditioning.

TABLE 37 SPECIFIC GRAVITY OF CONDITIONED LAMINATES

LAMINATE NO.	SPECIFIC GRAVITY		
	UNCONDITIONED	MOISTURE CONDITIONED	THERMAL CONDITIONED
	GM/CC (LB/IN.^{3})	GM/CC (LB/IN.^{3})	GM/CC (LB/IN.^{3})
1	1.583 ± 0.002 (0.057)	1.543 ± 0.003 (0.056)	1.545 ± 0.002 (0.056)
2	1.609 ± 0.009 (0.058)	1.601 ± 0.008 (0.058)	1.620 ± 0.018 (0.058)
3	1.777 ± 0.002 (0.064)	1.704 ± 0.001 (0.062)	1.714 ± 0.002 (0.062)
4	1.714 ± 0.012 (0.062)	1.649 ± 0.002 (0.060)	1.651 ± 0.002 (0.060)
5A	1.552 ± 0.003 (0.056)	1.551 ± 0.000 (0.056)	1.554 ± 0.004 (0.056)
6	1.514 ± 0.004 (0.055)	1.512 ± 0.001 (0.055)	1.515 ± 0.007 (0.055)
7	1.548 ± 0.005 (0.056)	1.536 ± 0.005 (0.055)	1.545 ± 0.002 (0.056)
8	1.531 ± 0.003 (0.055)	1.524 ± 0.004 (0.055)	1.534 ± 0.006 (0.055)
9	1.482 ± 0.000 (0.054)	1.479 ± 0.003 (0.053)	1.495 ± 0.003 (0.054)
10	1.555 ± 0.011 (0.056)	1.576 ± 0.001 (0.057)	1.562 ± 0.006 (0.056)
11	1.629 ± 0.015 (0.059)	1.648 ± 0.010 (0.060)	1.639 ± 0.016 (0.059)
12	1.627 ± 0.003 (0.059)	1.634 ± 0.008 (0.059)	1.636 ± 0.003 (0.059)
13A	1.541 ± 0.006 (0.056)	1.523 ± 0.007 (0.055)	(1)
14	1.614 ± 0.013 (0.058)	1.618 ± 0.012 (0.058)	1.619 ± 0.010 (0.058)
15	1.543 ± 0.006 (0.056)	1.551 ± 0.007 (0.056)	1.537 ± 0.000 (0.055)
16	1.557 ± 0.006 (0.056)	1.556 ± 0.006 (0.056)	1.552 ± 0.002 (0.056)
17	1.591 ± 0.007 (0.057)	1.605 ± 0.003 (0.058)	1.598 ± 0.004 (0.058)
18A	1.572 ± 0.010 (0.057)	1.571 ± 0.010 (0.057)	1.561 ± 0.002 (0.056)

(1) SPECIMEN COATING DECOMPOSED DURING CONDITIONING; DETERMINATION NOT MADE.

R81-0911-037D

3.2.4.3.2 Volume Fraction and Void Content. Volume fraction and void content determinations after environmental conditioning were not performed. The minimal changes in specific gravity after moisturizing and thermal conditioning (refer to Table 37) indicated that these tests would be meaningless.

3.2.4.3.3 Flexural Strength and Modulus. Tables 38 and 39 summarize the flexural strength and modulus data at 127°C (260°F) for the moisturized laminates. Percentage changes in these values as a result of the conditioning are also tabulated.

With respect to thin-laminate flexural strength (relative to Laminate No. 1), specimens from Laminates No. 6 (woven graphite outer plies), No. 8 (Kimbar flame barrier), and No. 9 (silicate-treated woven graphite outer plies) exhibited excessive

TABLE 38 TEST RESULTS: FLEXURAL STRENGTH AND MODULUS, THIN LAMINATES AFTER 16 DAYS AT 60°C/98% R.H.

LAMINATE NO.	ELEVATED TEMPERATURE DATA, 127°C (260°F)		CHANGE IN FLEXURAL PROPS.	
	FLEXURAL STRENGTH MPA (KSI)	FLEXURAL MODULUS GPA (MSI)	STRENGTH %	MODULUS %
1	612.3 (88.8)	30.4 (4.41)	−33.4	−19.4
2	928.8 (134.7)	40.3 (5.84)	0.9	+2.5
3	673.0 (97.6)	35.3 (5.12)	−33.2	−24.2
4	764.7 (110.9)	35.1 (5.09)	−23.6	−15.9
5A (2)	592.3 (85.9)	32.7 (4.74)	−17.5	−14.7
6	547.5 (79.4)	35.1 (5.09)	−46.6	−22.3
7 (2)	473.7 (68.7)	28.8 (4.17)	−48.7	−21.8
8	389.6 (56.5)	15.6 (2.26)	−31.4	−23.4
9	395.8 (57.4)	30.4 (4.41)	−60.7	−30.0
R81-0911-038D				

NOTE: (1) SEE TABLE 30 FOR PROPERTIES BEFORE CONDITIONING
(2) CONDITIONED FOR 21 DAYS, ALL OTHERS 16 DAYS

TABLE 39 TEST RESULTS; FLEXURAL STRENGTH AND MODULUS, THICK LAMINATES AFTER 21 DAYS AT 60°C/98% R.H.

LAMINATE NO.	ELEVATED TEMPERATURE DATA, 127°C (260°F)		CHANGE IN FLEXURAL PROPS.	
	FLEXURAL STRENGTH MPA (KSI)	FLEXURAL MODULUS GPA (MSI)	STRENGTH %	MODULUS %
10	1021.1 (148.1)	59.2 (8.59)	−9.3	−13.2
11	918.4 (133.2)	66.2 (9.60)	−2.6	+19.5
12	903.2 (131.0)	59.3 (8.60)	+17.1	+12.6
13	952.9 (138.2)	55.7 (8.08)	−0.1	+17.6
13A	729.5 (105.8)	51.2 (7.42)	—	—
14	895.0 (129.8)	55.0 (7.97)	−7.4	+8.1
15	902.6 (130.9)	52.4 (7.60)	−6.7	+3.8
16	613.0 (88.9)	54.2 (7.86)	−33.3	+2.3
17	908.8 (131.8)	52.3 (7.59)	−12.3	−0.5
18A	698.5 (101.3)	56.3 (8.16)	−27.3	+14.0
R81-0911-039D				

NOTE: SEE TABLE 30 FOR PROPERTIES BEFORE CONDITIONING

loss in flexural strength. Only the specimens from Laminate No. 9 developed unsatisfactory modulus values following environmental conditioning.

With respect to the flexural strength of thick laminates (relative to control Laminate No. 10), only the specimens from Laminates No. 16 (PI-coated graphite outer plies) and No. 18A (borate-treated fiberglass scrim outer plies) exhibited excessive reductions in flexural strength at 127°C (260°F).

Surprisingly, all of the laminate concepts with the exception of Laminate No. 17 (woven fiberglass outer plies with fire-retardant resin coating) exhibited increased flexural modulus values at 127°C following moisturizing. Since the baseline laminate lost 13.2% of the unconditioned modulus value at 127°C, the positive changes cannot be readily explained and may be due to a test anomaly.

3.2.4.3.4 Interlaminar Shear Strength. Table 40 summarizes the horizontal shear strength values for the thin laminates at 127°C after 16 days of moisturizing. Percentage changes as a result of the moisture exposure are also presented. Of the laminates tested, the specimens from Laminate No.6 (woven Gr/Ep outer plies) exhibited the least reduction in horizontal shear strength relative to control Laminate No. 1.

TABLE 40 TEST RESULTS; INTERLAMINAR (HORIZONTAL) SHEAR STRENGTH, THIN LAMINATES AFTER 16 DAYS AT 60°C/98% R.H.

LAMINATE NO.	ELEVATED TEMPERATURE 127°C (260°F)		CHANGE, %
	UNCONDITIONED MPA (KSI)	CONDITIONED MPA (KSI)	
1	40.0 (5.80)	30.5 (4.43)	−23.6
2	49.0 (7.11)	42.9 (6.22)	−12.6
3	34.7 (5.03)	26.4 (3.83)	−23.9
4	33.9 (4.91)	25.6 (3.72)	−24.2
5A	44.5 (6.46)	33.5 (4.86)	−24.8
6	29.6 (4.03)	25.4 (3.68)	−8.6
7	31.3 (4.54)	26.6 (3.86)	−15.0
8	29.6 (4.30)	24.8 (3.60)	−16.2
9	26.6 (3.86)	20.5 (2.98)	−22.7

R81-0911-040D

NOTE: SEE TABLE 32 FOR PROPERTIES BEFORE CONDITIONING.

With respect to the thick laminates, the specimens from Laminate No. 18A (sodium borate-treated) exhibited excessive loss of interlaminar (horizontal) shear strength at 127°C (relative to the control configuration) following humidity conditioning (Table 41).

TABLE 41 TEST RESULTS; INTERLAMINAR SHEAR (HORIZONTAL) SHEAR STRENGTH, THICK LAMINATES AFTER 21 DAYS AT 60°C/98% R.H.

LAMINATE NO.	ELEVATED TEMPERATURE, 127°C (260°F)		CHANGE %
	UNCONDITIONED	CONDITIONED	
	MPA (KSI)	MPA (KSI)	
10	39.2 (5.69)	40.3 (5.84)	+2.6
11	41.2 (5.97)	33.0 (4.78)	−19.9
12	26.7 (3.87)	25.3 (3.67)	−5.2
13	34.3 (4.97)	42.5 (6.17)	+24.1
13A	36.5 (5.30)	37.2 (5.39)	+1.6
14	39.7 (5.76)	38.9 (5.64)	−2.1
15	40.4 (5.86)	36.8 (5.33)	−9.0
16	35.4 (5.13)	30.8 (4.47)	−12.8
17	43.8 (6.35)	39.9 (5.78)	−9.0
18A	46.7 (6.77)	27.4 (3.98)	−41.2

R81-0911-041D

NOTE: SEE TABLE 32 FOR PROPERTIES BEFORE CONDITIONING.

3.2.4.3.5 Heat Distortion Temperature Measurements. Recent environmental testing performed by Grumman with epoxy matrix advanced composites has demonstrated that the heat distortion temperature of many of these polymer systems decreases with increasing moisture absorption. The candidate hybridized polymer matrix composite concepts proved to be no different. Intrusion of water into the composite laminates consistently lowered the Tg, as shown in Table 42. In those cases where the outermost plies of the thin laminates were protected by aluminum foil (Laminate No. 2), boron fibers (Laminate No. 3), fiberglass (Laminate No. 4) or Kimbar flame barrier (Laminate No. 8), the lowering of the Tg was not as pronounced. The first three of these hybridizers probably acted by physically slowing down moisture intrusion (as evidenced by the weight gain data); the latter probably acted by absorbing most of the moisture, thereby slowing down moisture penetration into the interior of the laminate.

Exposure to elevated temperatures [204°C (400°F) for 100 hr] consistently caused a rise in Tg (Table 43), with only two exceptions. One exception was Laminate No. 9 (silicate-treated, woven graphite outer plies) wherein the combination of prolonged elevated temperature exposure and silicate treatment lowered the Tg by 11°C (20°F) or -5.4%, probably by alkaline attack on the matrix resin. The second exception was Laminate No. 12 (PI matrix) which had already been subjected to a

TABLE 42 TEST RESULTS; T_G (HDT) OF CONDITIONED LAMINATES

TYPE OF LAMINATE	LAMINATE NO.	T_G (HDT) BY TMA		
		UNCONDITIONED	MOISTURE CONDITIONED	THERMAL CONDITIONED
		°C (°F)	°C (°F)	°C (°F)
THIN	1	206 ±0 (403 + 0)	122 ± 2 (251 ± 4)	228 ± 4 (443± 8)
	2	211 ± 2 (411 ± 5)	157 ± 10 (315 ± 18)	224 ± 7 (436 ± 12)
	3	207 ± 3 (405 ± 5)	151 ± 6 (304 ± 10)	250 ± 3 (482 ± 5)
	4	215 ± 5 (420 ± 9)	150 ± 8 (302 ± 16)	240 ± 2 (463 ± 3)
	5A	200 ± 4 (392 ± 7)	103 ± 1 (218 ± 2)	240 ± 1 (465 ± 1)
	6	204 ± 2 (398 ± 3)	109 ± 3 (228 ± 5)	238 ± 2 (460 ± 3)
	7	198 ± 1 (388 ± 2)	105 ± 1 (220 ± 1)	238 ± 1 (460 ± 2)
	8	201 ± 2 (394 ± 4)	134 ± 4 (273 ± 7)	225 ± 7 (437 ± 13)
	9	202 ± 2 (394 ± 4)	111 ± 4 (232 ± 6)	191 ± 3 (376 ± 5)
THICK	10	206 ± 1 (403 ±2)	91 ± 2 (195 ± 5)	237 ± 1 (458 ± 2)
	11	199 ± 2 (390 ± 3)	91 ± 3 (195 ± 6)	225 ± 2 (436 ± 3)
	12	255 ± 1 (481 ± 2)	104 ± 6 (220 ± 10)	253 ± 3 (488 ± 5) (2)
	13	208 ± 1 (407 ± 1)	93 ± 2 (200 ± 3)	234 ± 1 (453 ± 2)
	13A	183 ± 1 (361 ± 2)	129 (1) (264)	(1)
	14	212 ± 1 (414 ± 2)	91 ± 2 (196 ± 3)	228 ± 2 (442 ± 4)
	15	213 ± 1 (415 ± 1)	88 ± 5 (191 ± 8)	234 ± 3 (453 ± 5)
	16	200 ± 1 (342 ± 2)	101 ± 1 (215 ± 2)	232 ± 1 (450 ± 1)
	17	208 ± 2 (407 ± 4)	92 ± 1 (198 ± 1)	230 ± 1 (446 ± 2)
	18A	203 ± 2 (397 ± 3)	106 ± 1 (222 ± 1)	230 ± 1 (446 ± 2)

NOTES: (1) COATING DECOMPOSES

(2) THESE SPECIMENS (POLYIMIDE MATRIX) WERE ALSO EXPOSED AT 254°C (490°F) FOR 200 HOURS, YIELDING A T_G OF 263° ± 8°C (505° ± 14°F).

R81-0911-042D

post-cure temperature of 246°C (475°F); prolonged exposure at 200°C (392°F) caused only negligible change in Tg of -0.8%. Exposure at 254°C (490°F) for 200 hr, however, raised the Tg by 8°C (14°F), or 3.1%.

The general trend to higher Tg's caused by elevated-temperature exposure (Table 43) is most probably a result of further polymer cross-linking, causing reductions in vibrational and/or rotational degrees of freedom with a concomitant rise in second-order transition temperature. Laminates No. 3, 5A and 7 showed a 20% rise in Tg; for Laminates No. 3 and 5A, the inclusion of boron (reinforcement or matrix), which serves as a stiffening agent, may be responsible. For Laminate No. 7, the

TABLE 43 TEST RESULTS; ELEVATION OF T_G (HDT) DUE TO THERMAL EXPOSURE

LAMINATE NO.	CHANGE IN T_G °C(°F)	CHANGE, %
1	22(40)	10.7
2	13(23)	6.2
3	43(77)	20.8
4	25(45)	11.6
5A	40(72)	20.0
6	34(61)	16.7
7	40(72)	20.2
8	24(43)	11.9
9	–11(–20)	–5.4
10	31(60)	15.0
11	26(47)	13.1
12	–2(–4)	–0.8
13	26(47)	12.5
13A	SEE NOTE	–
14	16(29)	7.5
15	21(38)	9.9
16	32(48)	16.0
17	22(40)	10.6
18A	7(13)	13.3
R81-0911-043D		

NOTE: THERMAL EXPOSURE OF LAMINATE 13A CAUSED DECOMPOSITION AND SWELLING OF THE COATING AND INABILITY TO MEASURE HDT.

graphite was sized with NR-150B2 polyimide; this treatment evidently resulted in thermal stabilization of the graphite fibers.

3.2.4.4 Laboratory Burn Tests

The ability of each candidate hybridization concept to retain graphite fiber particulates in a severe thermo-oxidative environment was determined in a series of laboratory burn tests. The tests were selected to establish each candidate's flame-resistance and char-forming characteristics. The tests selected to quantify these characteristics were:

- Flame Spread
- Limiting Oxygen Index
- Controlled Burn
- Particulate Collection.

3.2.4.4.1 Flame Spreading Tests (Downward Vertical Burning Rate). The flame-spreading characteristics for each of the candidate laminate systems was determined using a bench-top apparatus based on the downward vertical burning rate (DVBR) work of E. R. Larsen.[9] Basically, the DVBR test involves replacing the sample holder normally used in the oxygen index (OI) test with a sample holder which holds a 1.9 x 9.6-cm (3/4 x 3-in.) specimen so that only a single surface is exposed. The specimen is clamped in place by means of a thin brass sheet which is pressed firmly against the back of the specimen by means of thumb screws. Scribe marks are made on the thin knife edges against which the specimen is pressed. These marks are located 0.64, 3.17, and 5.17-cm (1/4, 1-1/4, and 2 1/4-in.) from the top of the holder. When the sample is properly in place, only a single surface is exposed, and both side edges and back of the sample are covered.

The mounted specimen is placed in the OI apparatus and the oxygen level adjusted to give the desired atmosphere. After a one-minute flush of the chamber, the sample is ignited by passing a small acetylene flame along the top edge. The time required for the flame to spread from the 0.64-cm (1/4-in.) mark to the 5.17-cm (2 1/4-in.) mark is measured using a stop watch. The time is also noted as the flame passes the 3.17-cm (1-1/4-in.) mark as a check on the adequacy of the oxygen flow to maintain the flame so that it progresses at an even rate.

It is generally accepted that the ASTM E-84 7.3-m (24-ft) tunnel test predicts the relative performance of fire-retardant systems in a majority of cases. It is also generally true that with respect to flame spread tests, the slower the burning rate and the higher the oxygen level, the greater the probability that the material in question will have a low E-84 FSC rating (tendency toward non-burning).

The flame spread tests were performed on unconditioned (as-cured), moisturized and thermally conditioned laminates. Testing was performed at three oxygen levels (50%, 72%, and 100%) in accordance with the conventional procedure used for Gl/Ep materials. Test data generated at the 100% level were used to establish the relative fire resistance of a given candidate laminate. This convention is in accordance with the work of Larsen.

Samples were run in duplicate at 50%, 72%, and 100% oxygen levels at a gas flow rate of about 4-cm/sec (1.57-in./sec). The results presented in Table 44 include burning time and the length of specimen burned. Where applicable, i.e., where the specimen continued to burn along the entire test (gage) length, the rate of flame spread is calculated. Average data is presented for each combination of laminate and test condition (oxygen level).

The burning rate of the unconditioned laminates was used to establish the relative performance of the candidate laminates. Corresponding data recorded for the moisturized and thermally exposed specimens were used to provide a measure of the service durability of the proposed fiber retention system.

With respect to the thin laminates, Laminates No. 2 (aluminum faced) and No. 8 (Kimbar faces) were adjudged NP -- no propagation of the surface flame; this is explained by the ability of the aluminum foil and the Kynol phenolic outer layers to resist the surface burn propagation of this test and prevent underlying structure from being oxidized. These specimens burned differently, in this test, than the others and the results, in Grumman's judgement, are atypical. (These test specimens burned to a relatively minor degree (max. 1.9-cm (3/4-in.) at 72% oxygen). The remaining specimens all burned the 5.1-cm (2-in.) test length and are ranked in Table 45 from the most fire resistant by test to the least. Moisturizing and thermal conditioning had no consistent effect on the flame-spread rating of the thin candidate laminate systems.

The flame-spread characteristics of the unconditioned, thick-laminate specimens in 100% oxygen were somewhat multi-modal. In some instances, namely Laminates No. 10 14, 15 and 16, the laminates burned for a period of time and then self-extinguished before burning the entire test length as did the remaining laminates. The concepts are ranked in Table 46 with the laminate supporting burning for the shortest length ranked highest and the laminate exhibiting the highest burning rate ranked lowest.

Like the thin laminates, moisturized and thermally conditioned thick laminates generally showed no consistent trend with respect to fire resistance compared to the unconditioned laminates. For the tests conducted at the 100% oxygen level the rate of burning of the moisturized specimens from Laminates No. 10, 11, 12, 13, 13A, 14, 15, 16, and 17 decreased; only those from Laminate No. 18A exhibited a higher burn rate.

Thermally conditioned specimens from Laminates No. 11, 12, 14, 15, and 16 tested at the 100% oxygen level exhibited faster burn rates while those from Laminates

TABLE 44 TEST RESULTS; FLAME SPREAD (DVBR) (SHEET 1 OF 2)

LAMINATE NO.	UNIT OF MEASURE	SPECIMEN CONDITIONING								
		UNCONDITIONED			MOISTURE			THERMAL		
		OXYGEN LEVEL, %								
		50	72	100	50	72	100	50	72	100
1	TIME, sec	18.9	24.1	25.0	10.7	25.4	24.9	3.1	15.2	22.2
	LENGTH, cm	1.6	5.1	5.1	0.6	2.5	5.1	0.6	1.3	1.3
	RATE, cm/sec	(2)	0.21	0.20	(2)	(2)	0.20	(2)	(2)	(2)
2	TIME, sec	2.7	4.1	6.2	3.0	6.7	5.2	3.8	3.2	10.4
	LENGTH, cm	0	0	0	0	0	0	0	0	0
	RATE, cm/sec	NP	NP	NP	NP	NP	NP	NP	NP	NP
3	TIME, sec	5.0	36.5	22.6	17.2	21.2	32.0	7.5	17.1	42.4
	LENGTH, cm	1.2	5.1	5.1	1.7	5.1	5.1	0.6	0.9	1.3
	RATE, cm/sec	(2)	0.14	0.22	(2)	0.24	0.16	(2)	(2)	(2)
4	TIME, sec	26.4	60.2	33.0	16.2	27.0	28.4	14.5	18.2	71.5
	LENGTH, cm	1.9	5.1	5.1	1.1	5.1	5.1	0.6	1.1	5.1
	RATE, cm/sec	(2)	0.14	0.22	(2)	0.24	0.16	(2)	(2)	(2)
5A	TIME, sec	6.4	45.8	30.8	4.5	8.8	24.6	4.6	16.2	35.0
	LENGTH, cm	0	5.1	5.1	0.3	0	5.1	0.6	0.8	2.5
	RATE, cm/sec	NP	0.11	0.16	(2)	NP	0.21	(2)	(2)	(2)
6	TIME, sec	6.3	42.6	20.6	4.2	8.0	42.0	5.3	21.8	24.3
	LENGTH, cm	0.6	5.1	5.1	0.2	0	5.1	0.6	1.3	4.1
	RATE, cm/sec	(2)	0.12	0.25	(2)	NP	0.12	(2)	(2)	0.21
7	TIME, sec	33.8	40.4	19.4	30.6	7.0	29.1	6.2	14.6	28.6
	LENGTH, cm	3.3	5.1	5.1	2.2	0	5.1	0.6	1.3	1.3
	RATE, cm/sec	(2)	0.13	0.26	(2)	NP	0.17	(2)	(2)	(2)
8	TIME, sec	7.5	14.7	30.8	7.2	6.7	32.0	6.8	20.8	21.4
	LENGTH, cm	0.8	0	0	0	0	0	0.6	1.8	0.6
	RATE, cm/sec	(2)	NP	NP	NP	NP	NP	(2)	(2)	(2)
9	TIME, sec	14.1	38.4	31.0	18.9	8.9	29.4	6.5	17.5	22.2
	LENGTH, cm	1.1	5.1	5.1	0.8	0	5.1	0.6	1.3	5.1
	RATE, cm/sec	(2)	0.13	0.16	(2)	NP	0.17	(2)	(2)	0.23
10	TIME, sec	5.0	57.9	28.8	8.0	6.0	28.6	7.7	14.6	51.2
	LENGTH, cm	0.6	5.1	3.8	0	0	5.1	0.6	1.3	2.5
	RATE, cm/sec	(2)	0.09	(2)	NP	NP	0.18	(2)	(2)	(2)
11	TIME, sec	13.5	28.3	35.1	4.2	10.5	31.1	6.5	19.8	24.6
	LENGTH, cm	1.3	1.3	5.1	0	0	4.1	0.6	0.6	0.6
	RATE, cm/sec	(2)	(2)	0.14	NP	NP	0.16	(2)	(2)	(2)
12	TIME, sec	2.0	10.0	70.1	2.4	12.3	44.4	7.0	11.7	29.4
	LENGTH, cm	0	0	5.1	0	0	5.1	0.6	0.6	1.9
	RATE, cm/sec	NP	NP	0.07	NP	NP	0.09	(2)	(2)	(2)
13	TIME, sec	21.7	30.2	32.4	13.6	45.1	27.4	15.2	21.9	67.8
	LENGTH, cm	1.3	5.1	5.1	1.1	5.1	5.1	0.9	1.3	3.8
	RATE, cm/sec	(2)	0.17	0.16	(2)	0.11	0.19	(2)	(2)	(2)
13A	TIME, sec	4.2	8.7	15.8	1.4	5.5	5.7	—	—	—
	LENGTH, cm	0	5.1	5.1	0	0	0.6	—	—	—
	RATE, cm/sec	NP	0.59	0.32	NP	NP	(2)	—	—	—

R81-0911-044D(1/2)

TABLE 44 TEST RESULTS: FLAME SPREAD (DVBR) (SHEET 2 OF 2)

LAMINATE NO.	UNIT OF MEASURE	SPECIMEN CONDITIONING								
		UNCONDITIONED			MOISTURE			THERMAL		
		OXYGEN LEVEL, %								
		50	72	100	50	72	100	50	72	100
14	TIME, sec	16.9	15.7	50.1	12.2	48.4	35.1	4.8	68.7	27.6
	LENGTH, cm	0.9	0.9	0.9	0.8	5.1	5.1	0	5.1	5.1
	RATE, cm/sec	(2)	(2)	(2)	(2)	0.10	0.14	NP	0.07	0.18
15	TIME, sec	9.0	25.3	38.1	27.9	31.5	37.2	3.0	20.0	24.5
	LENGTH, cm	0.6	1.3	2.5	1.3	1.3	1.3	0.3	1.9	5.1
	RATE, cm/sec	1(2)	(2)	(2)	(2)	(2)	(2)	(2)	(2)	(2)
16	TIME, sec	11.2	14.0	54.6	16.3	65.5	36.5	1.2	51.4	25.8
	LENGTH, cm	0.9	0.6	3.8	1.3	5.1	5.1	0	5.1	5.1
	RATE, cm/sec	(2)	(2)	(2)	(2)	0.08	0.14	NP	0.10	0.20
17	TIME, sec	10.4	6.6	41.0	1.8	29.2	27.6	13.5	23.1	62.4
	LENGTH, cm	1.1	0	5.1	0	2.5	5.1	0.9	1.3	5.1
	RATE, cm/sec	(2)	NP	0.12	NP	(2)	0.18	(2)	(2)	0.08
18A	TIME, sec	11.9	5.6	24.1	1.4	62.9	29.7	9.8	16.8	46.9
	LENGTH, cm	0.8	0	5.1	0	5.1	5.1	0.6	0.9	1.9
	RATE, cm/sec	(2)	NP	0.21	NP	0.08	0.17	(2)	(2)	(2)

NOTES: (1) NP DENOTES "NO PROPAGATION", I.E., ONCE THE FLAME WAS REMOVED, THE SPECIMEN CEASED BURNING.

(2) THE SPECIMEN CONTINUED TO BURN AFTER THE FLAME WAS REMOVED FOR THE REPORTED PERIOD (TIME AND LENGTH) AND STOPPED BURNING BETWEEN THE GAGE MARKS.

R81-0911-044D(2/2)

TABLE 45 THIN LAMINATE BURNING PERFORMANCE RANKING

LAMINATE	FIBER RETENTION FEATURE	BURNING RATE, CM/SEC(1)	RANKING
1	CONTROL	0.20	4
2	ALUMINUM FACED	(2)	1
3	B/EP FACED	0.22	5
4	WOVEN GL/EP FACED	0.15	2
5A	BORON POWDER	0.16	3
6	WOVEN GR/EP FACED	0.25	6
7	PI SIZED	0.26	7
8	KIMBAR FACED	(2)	1
9	SODIUM SILICATE TREATED	0.16	3
R81-0911-045D			

NOTES: (1) UNCONDITIONED, 100% OXYGEN LEVEL
(2) NO PROPAGATION

TABLE 46 THICK LAMINATE BURNING PERFORMANCE RANKING

LAMINATE NO.	FIBER RETENTION FEATURE	BURN LENGTH, CM	RATE OF BURNING CM/SEC(1)	RANKING
10	CONTROL	3.8	–	3
11	B/EP PLIES	–	0.14	6
12	WOVEN GL/PI PLIES	–	0.07	4
13 (2)	QU/EP FACED	–	0.16	7
13A	INTUMESCENT COATED	–	0.32	9
14	WOVEN GL/EP PLIES	0.9	–	1
15	WOVEN GL-GR/EP PLIES	2.5	–	2
16	PI SIZED GR	3.8	–	3
17	FIRE RETARDANT EPOXY	–	0.12	5
18A	SODIUM BORATE TREATED	–	0.21	8
R81-0911-046D				

NOTES: (1) UNCONDITIONED, 100% OXYGEN LEVEL.
(2) LAMINATE NO. 13 IS AN UNCOATED PORTION OF LAMINATE NO. 13A.

No. 10, 13, 17, and 18A developed slower burn rates. (Laminate No. 13 is an uncoated Laminate No. 13A.) Because the intumescent coating on Laminate No. 13A decomposed as a result of thermal conditioning, no coupons from this category were tested.

3.2.4.4.2 Limiting Oxygen Index Tests. The limiting oxygen index of each hybrid laminate was determined by ASTM Test Method D 2863-76, "Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)." This method describes a procedure for measuring the minimum concentration of oxygen, in a flowing mixture of oxygen and nitrogen, that will just support flaming combustion; oxygen index is given as "n," in percent, by the formula:

$$n\ (\%) = (100 \times O_2) / (O_2 + N_2)$$

The test column, flow-controlling devices and ignition source were assembled in accordance with the standard method. A deviation was required, however, with regard to sample thicknesses. The method calls for samples approximately 2-mm (0.120-in.) thick whereas the hybrid laminates prepared in this study fell into two groupings: one approximately 1.3-mm (0.050-in.) thick, the other approximately 6.3-mm (0.250-in.) thick. Comparisons of limiting oxygen indices can only be validly made between specimens having the same thickness; the data so obtained will undoubtedly differ from data that would be obtained if the tests were made with 3-mm (0.120-in.) thick specimens.

For this study, the alternate test column described in the test method was used. Since this column has a restricted upper opening (50-mm), it was felt to be advantageous for sampling the effluent of this study for quantity and nature of emitted particulate materials.

These tests provide comparative data with respect to oxygen levels required to support combustion of the candidate materials; the higher the oxygen index, the better the sample. In addition, this test procedure provides a method of determining the effect of additives and other composite modifications on the flame resistance of the candidate systems.

Oxygen index determinations were made on unconditioned and moisturized specimens from each of the candidate systems. Tests were not performed on thermally conditioned specimens, although the related flame spread tests indicated that there were some differences in the burning characteristics of moisturized and thermally conditioned specimens.

The results of the limiting oxygen index tests are reported in Table 47. The candidate concepts are also rated in accordance with their burn resistance (i.e., the

TABLE 47 LIMITING OXYGEN INDEX TEST RESULTS AND RANKING

TYPE OF LAMINATE	LAMINATE NO.	FIBER RETENTION FEATURE	LIMITING OXYGEN INDEX, %		RANKING		
			UNCOND-ITIONED	MOISTURE CONDITIONED	UNCOND-ITIONED	MOISTURE CONDITIONED	AVG OF UNCON. AND MOIST. COND.
THIN	1	CONTROL	28	28	4	5	7
	2	ALUMINUM-FACED	44	47	1	1	1
	3	B/EP-FACED	29	33	3	4	6
	4	WOVEN GL/EP-FACED	32	33	2	4	5
	5A	BORON POWDER	29	46	3	2	3
	6	WOVEN GR/EP-FACED	26	26	5	6	8
	7	POLYIMIDE-SIZED GR	28	46	4	2	4
	8	KIMBAR FACES	44	44	1	3	2
	9	SODIUM SILICATE-TREATED	26	26	5	6	8
THICK	10	CONTROL	44	47	4	3	4
	11	B/EP PLIES	44	48	4	2	3
	12	PI MATRIX	60	44	2	5	2
	13A	INTUMESCENT COATED	70	70	1	1	1
	14	WOVEN GL/EP PLIES	41	40	6	7	7
	15	WOVEN GL-GR/EP PLIES	40	42	7	6	6
	16	PI-SIZED GR	36	46	8	4	6
	17	FIRE-RETARDANT EPOXY	48	44	3	5	6
	18A	SODIUM BORATE-TREATED	43	46	5	4	5

R81-0911-047D

higher the index, the more resistance to burning). The ranking is established for each condition (unconditioned and moisturized) as well as for the average of the two rankings.

Regardless of which of the ranking systems was used, the aluminum-faced thin laminate concept (Laminate No. 2) offers the most resistance to burning within its thickness category. Similarly, Laminate No. 13A, which incorporated the intumescent coating, offers the most resistance to burning of the candidate thick-laminate concepts. It should be noted, however, that within each thickness/condition grouping the OI's of several laminates are essentially equivalent, e.g., thin/unconditioned Laminates No. 1, 3, 4, 5A, 6, 7, and 9.

3.2.4.4.3 Controlled Burn and Particulate Collection. Particulate materials emitted as a result of the Limiting Oxygen Index test were collected using an Aerosol Monitoring kit (Millipore Corp, No. XX7303700). A vacuum pump was used to pull the airborne products of the combustion through an aerosol adapter containing a preweighed membrane filter. The mass of airborne particles resulting from the combustion of the candidate laminate was determined by differential weighing of the millipore filter. However, the relatively high oxygen content (compared with air) of the gas mixture used to burn the specimens oxidized the combustion products almost completely to gases, so that very little material was collected on the filter. Therefore, the apparatus sketched in Fig. 2 was assembled to produce controlled burn (oxygen and nitrogen gas flow rate controlled by flow meters) and mechanical shaking of the specimen (vibrator attached to specimen holding device). It featured a millipore collector and an acetylene torch to provide a temperature range of 1050° to 1100°C (1922 to 2012°F). Figure 3 shows the controlled burn apparatus; Fig. 4 shows a close-up view of a specimen being tested.

The test procedure is described in Table 48. It was noted that combustion temperatures, measured at the point of impingement of the acetylene torch, reached a maximum of 2500 ± 20°F while the specimens were burning in the oxygen-enriched atmosphere during the main burn time period (Step 5).

During the burning, observations were made of the specimens, the charred specimens after burning, and the particulate matter collected on the millipore filter. The observations included the following considerations:

- Burning Specimens: Flame conditions noted during preburn and main burn, changes in the burning laminate such as

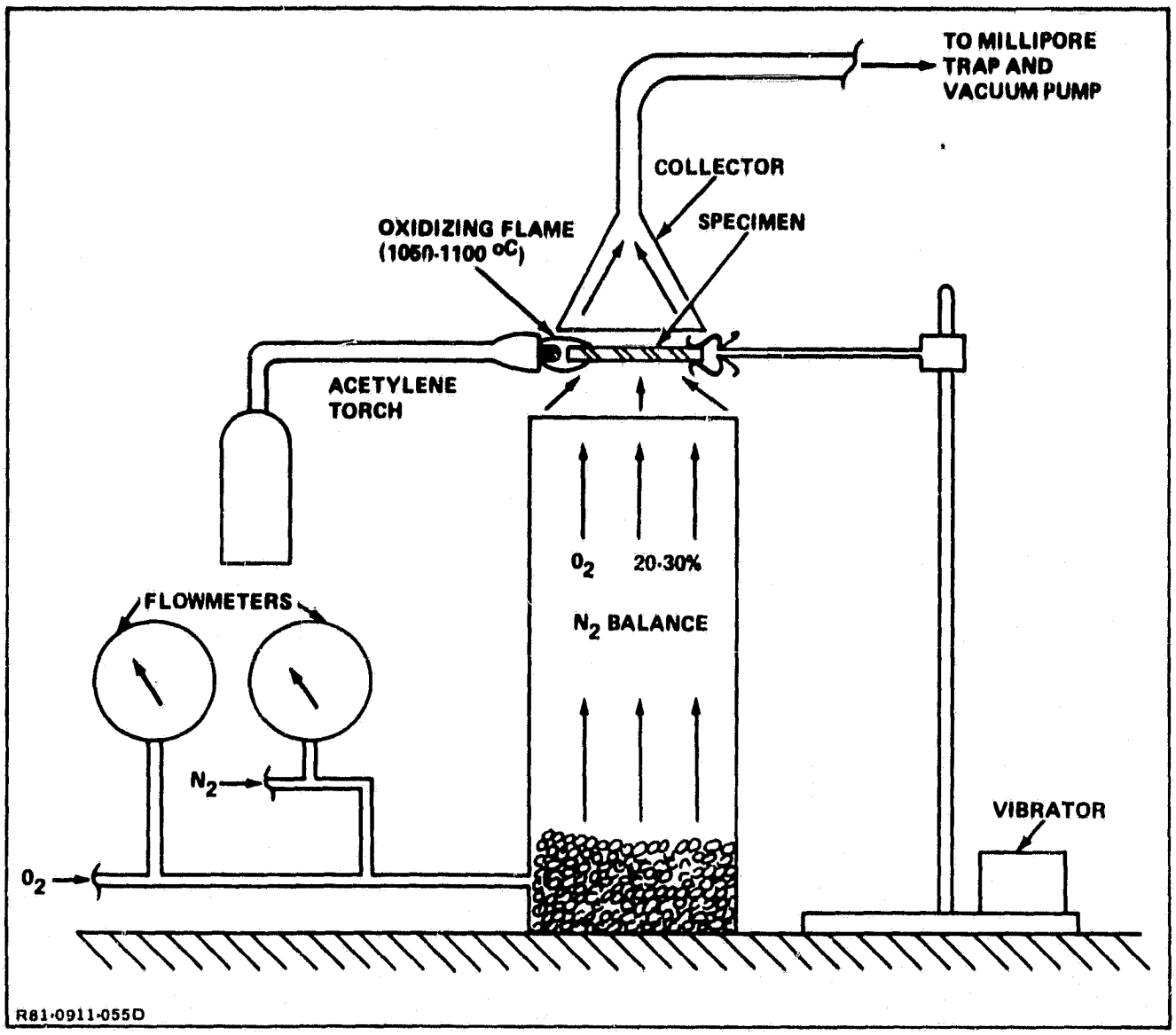
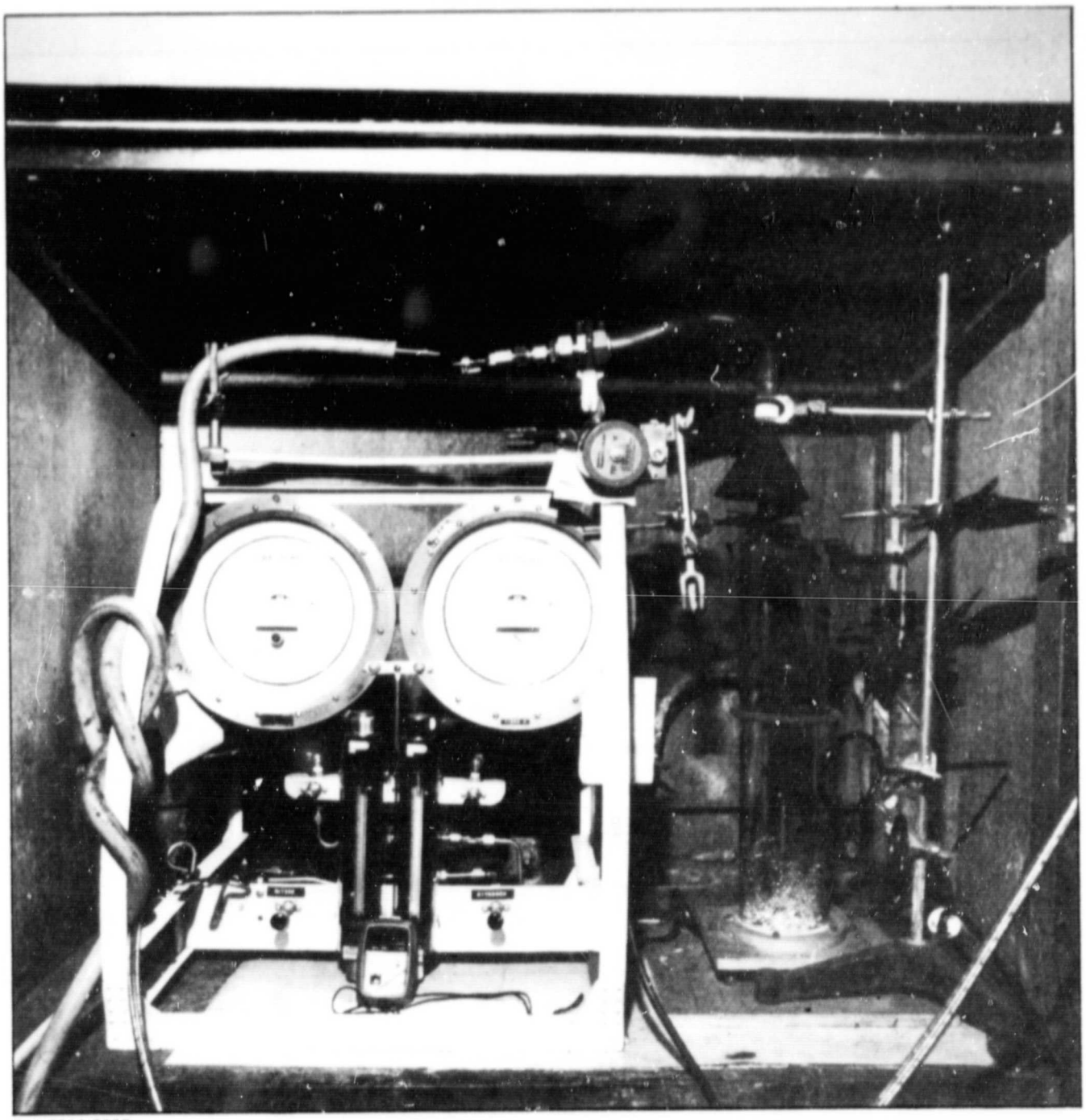


Fig. 2 Schematic Diagram of Controlled Burn Apparatus

ash or char formation, layer separations or peel back, particulate release (airborne or drop-off) and specimen changes such as discolorations and swelling

- Charred Specimens: Appearance of specimens after removal from the controlled burn apparatus, apparent structural integrity, char formation, condition of ply layers (separated or intact), laminate separation mode, erosion and brooming

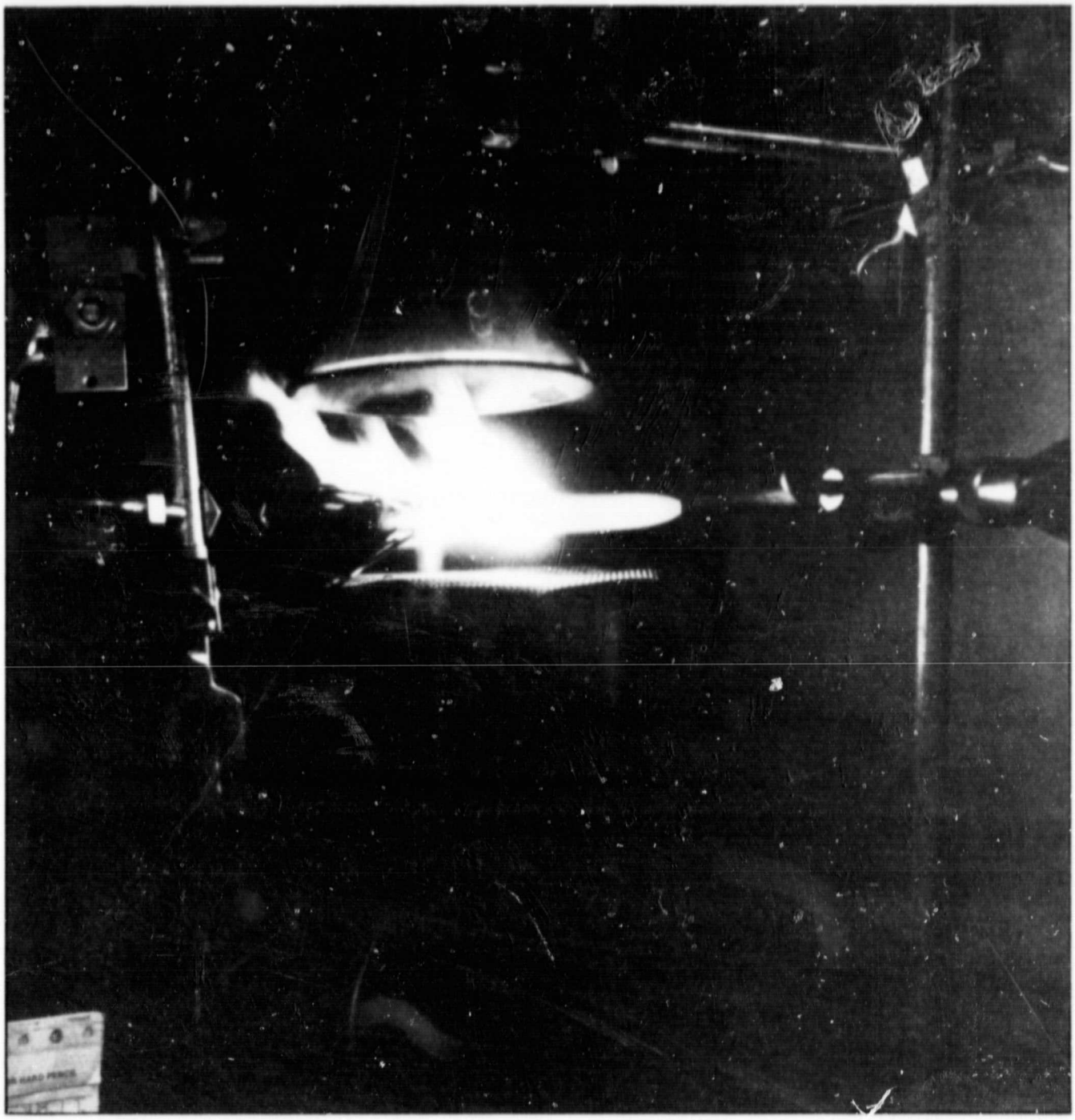


R81-0911-056D

Fig. 3 Controlled Burn Apparatus

- Particulate Matter: Analysis of material collected on millipore filter, weight of material collected, and visual and microscopic (45X magnification) inspection of particulate matter with emphasis on presence or absence of graphite material and its physical form.

ORIGINAL PAGE IS
OF POOR QUALITY



R81-0911-057D

Fig. 4 Close-up View of Specimen Being Tested in Controlled Burn Apparatus

The observations made during the burning of the specimens and on the charred specimens are reported in Table 49 and those on the particulate matter in Table 50. These observations were analyzed and the candidate laminates rated with respect to

TABLE 48 CONTROLLED BURN/PARTICULATE COLLECTION PROCEDURE

STEP NO.	OPERATION
1	SAMPLE SIZE, 2.54 CM X 5.08 CM (1 IN. X 2 IN.)
2	2-MIN SOAK IN 50% OXYGEN (O_2)/50% NITROGEN (N_2) AT A FLOW RATE OF 5850 CC/MIN.
3	45 SEC TO 1-MIN PRE-BURN TO REMOVE RESIN, SMOKE NOT COLLECTED.
4	COLLECTOR PUT IN PLACE (FUNNEL ATTACHED TO MILLIPORE FILLER IN TURN ATTACHED TO A VACUUM PUMP) AND SUCTION (VACUUM) TURNED ON.
5	A. THIN LAMINATES: 5-MIN MAIN BURN IN OXIDIZING PORTION OF AIR-ACETYLENE TORCH, FLAME TEMPERATURE 1050 TO 1100°C (1922 TO 2012°F), AS MEASURED BY A DIGITAL DISPLAY POTENTIOMETER. B. THICK LAMINATES: 10-MIN MAIN BURN IN OXIDIZING PORTION OF AIR-ACETYLENE TORCH, FLAME TEMPERATURE 1050 TO 1100°C (1922 TO 2012°F), AS MEASURED BY A DIGITAL DISPLAY POTENTIOMETER.
6	ACTIVATION OF ELECTROMECHANICAL VIBRATOR ATTACHED TO SPECIMEN HOLDER DURING BURN PERIOD (STEP 5).
R81-0911-048D	

char characteristics and the nature of the particulate matter collected. The rankings were based on the following criteria:

- Char Characteristics:
  - Rating 1 (Highest). Abundant char formed, remained intact, minimal drop-off, did not break up or separate as result of light probing with metal probe; overall condition of residue: excellent
  - Rating 2. Good char formation, remained intact, did not break-up or separate as result of light probing with metal probe, minimum of drop-offs; overall condition of residue: very good
  - Rating 3. Fair char formation, separated or delaminated but did not fall apart as result of light probing with metal probe, more drop-off than in Rating 2; overall condition of residue: good
  - Rating 4. Fair char formation, separated or delaminated more than that Rated 3 and started to fall apart as result of light probing with metal probe; overall condition of residue: fair

TABLE 49 OBSERVATIONS OF BURNING AND CHARRED SPECIMENS (SHEET 1 OF 3)

LAMINATE NO.	OBSERVATIONS OF BURNING SPECIMENS	OBSERVATIONS OF CHARRED SPECIMENS (RESIDUE AFTER BURNING)
1	RESIN BURNED OFF WITH SMOKY YELLOW FLAME DURING 1-MIN PRE-BURN. VERY LITTLE ASH FORMED, LAYERS FELL APART, PARTICLES AND CLUMPS BROKE OFF DURING 5-MIN BURN.	SPECIMEN HAD NO STRUCTURAL INTEGRITY AND FELL APART ON TOUCHING. LAYERS SEPARATED. NO CHAR.
2	ALUMINUM FOIL STAYED INTACT DURING RESIN BURNOUT. SMOKY BLACK-YELLOW FLAME EMITTED DURING PRE-BURN. WHEN SUCTION WAS TURNED ON, A PUFF OF HEAVY BLACK SMOKE CAME OFF AND THE RESIN BURNED WITH A SMOKY FLAME AS THE ALUMINUM FOIL PEELED IN SPOTS.	LAYERS SEPARATED BUT REMAINED FAIRLY INTACT. SOME CHAR FORMED. ALUMINUM LAYER OXIDIZED AND BROKE UP INTERMITTENTLY.
3	SMOKY FLAME INITIALLY; NO FLAME DURING 5-MIN BURN, VERY FEW PARTICLES FLY OFF OR BREAK OFF. SPECIMEN REMAINED INTACT; BURNED WITH GREEN TINGE TO FLAME.	FAIRLY GOOD CHAR FORMATION; LAYERS REMAINED INTACT EXCEPT FOR ONE PLANE WHICH SEPARATED COMPLETELY.
4	LIKE NO. 1 IN PRE-BURN. QUIET 5-MIN BURN WITH SOME DROPS.	CHAR FORMED FAIRLY WELL BUT LAYERS SEPARATED INTO THREE SECTIONS (2 PLANES). EXTERIOR LOOKED GOOD.
5A	LIKE NO. 1 IN PRE-BURN. QUIET BURN WITH SPECIMEN REMAINING INTACT.	EXCELLENT CHAR FORMATION. MINIMAL DROP-OFFS. SPECIMEN REMAINED INTACT.
6	PRE-BURN LIKE NO. 1. FAIRLY QUIET BURN, WITH SOME FLARE-UPS AND DROPS OFFS. FLAME STAYED YELLOW IN COLOR.	WOVEN OUTER LAYER ERODED AT EDGE WHERE FLAME IMPACTED. SOME DROP OFFS. CHAR FORMATION GOOD; SPECIMEN MECHANICAL INTEGRITY GOOD.
7	PRE-BURN LIKE NO. 1. FAIRLY QUIET BURN, FALL-OFFS OBSERVED, FLAME STAYED YELLOW.	VERY LITTLE CHAR FORMED. SPECIMEN DELAMINATED AND SEPARATED. NO MECHANICAL INTEGRITY.
8	OBSERVED PEEL-BACK DURING PRE-BURN, OTHERWISE LIKE NO. 1. EXTENSIVE DELAMINATION DURING BURN, WITH FALL-OFFS AND FLY-OFFS.	SOME CHAR ON BUNCHED ZERO-DEGREE LAYERS; OTHERWISE DELAMINATED WITH ANGLE PLY DROP-OFFS. SURFACE LAYER MELTED AND BURNT THROUGH.
9	YELLOW FLAME AND SMOKE AT FIRST, THEN STABLE DURING PRE-BURN. QUIET 5-MIN BURN; SPECIMEN HELD TOGETHER WITH MINIMUM DROPS AND FLY-OFFS SEEN. YELLOW (SODIUM) COLORED FLAME.	WOVEN OUTER LAYER ERODED AT FLAME END. SPECIMEN SEPARATED AT 0/90° PLANE INTO TWO SEGMENTS. VERY LITTLE FLY-OFF COLLECTED; SAME FOR DROP-OFFS. GOOD MECHANICAL INTEGRITY OF TWO SEGMENTS, ALTHOUGH NOT MUCH CHAR WAS OBSERVED.

R81-0911-049D(1/3)

TABLE 49 OBSERVATIONS OF BURNING AND CHARRED SPECIMENS (SHEET 2 OF 3)

LAMINATE NO.	OBSERVATIONS OF BURNING SPECIMENS	OBSERVATIONS OF CHARRED SPECIMENS (RESIDUE AFTER BURNING)
10	YELLOW, SMOKY, CARBONACEOUS FLAME AT FIRST (PRE-BURN). THIS SUBSIDED AT APPROXIMATELY 75 SEC. WITH FUNNEL IN PLACE AND VACUUM TURNED ON, THE FLAME STABILIZED. SPECIMEN ERODED AT FLAME END; FLAME HAD YELLOW SODIUM COLOR. PIECES FELL OFF AND SOME FIBERS FLEW OFF INTO MILLIPORE FILTER.	SPECIMEN DELAMINATED EXCEPT FOR BUNCHED ZERO-DEGREE SECTIONS. NO CHAR OBSERVED IN DELAMINATED LAYERS; SOME CHAR ON INTACT BUNCHED ZEROS. A LOT OF FALL-OFFS.
11	SPECIMEN BURNED WITH WHITE, NON-SMOKY FLAME. AFTER ONE MINUTE, WHEN VACUUM/FUNNEL WAS APPLIED, WHITE SMOKE CAME OFF FOR THREE MINUTES. BURN THEN CONTINUED QUIETLY WITH NO CHANGE IN FLAME COLOR. NO DROP-OFFS; SOME FLY-OFFS. AFTER EIGHT MINUTES, FLAME COLOR TURNED GREEN AND OUTER PLY FELL OFF IN SMALL SEGMENTS.	SPECIMEN DELAMINATED, BUT LESS THAN NO. 10. SOME OF THE OUTER LAYER WAS LOST. GOOD CHAR AND MECHANICAL ATTACHMENT OF INTERNAL LAYERS. EFFECT OF HEAT DAMAGE LATERALLY INTO THE SPECIMEN NOT AS PRONOUNCED AS NO. 10. LESS "BROOMING" EFFECT OBSERVED.
12	SOOTY YELLOW FLAME DURING ONE-MINUTE PRE-BURN. SPECIMEN VERY STABLE DURING 10-MIN OXY-ACETYLENE BURN; GLOWING ORANGE-WHITE BUT NO LOSS OF DROP-OFFS OR FLY-OFFS. NO SMOKE WAS GIVEN OFF DURING BURN.	SPECIMEN REMAINED QUITE INTACT WITH LESS DELAMINATION THAN IN NO. 11. OUTER LAYER TURNED WHITE (GLASS CLOTH). ALMOST NO FALL-OFF OR FLY-OFF OBSERVED.
13A	SPECIMEN IGNITED AND BURNED WITH INTENSE WHITE FLAME. NO SOOT CAME OFF DURING PRE-BURN; CONTINUED TO BURN WITH WHITE FLAME AND SWELLING OF EXTERNAL PAINT. ALMOST NO SMOKE, NO FLY-OFFS OR DROPS; LAMINATE REMAINED INTACT. VERY STABLE BURN AFTER 4.5 MIN.	MINIMAL DELAMINATION OF SPECIMEN. LOTS OF CHAR WHICH HOLDS LAYERS TOGETHER. SWOLLEN SURFACE IS FAIRLY RIGID AND RETAINS OUTER LAYER IN PLACE. NO FALL-OFF OR FLY-OFFS OF SPECIMEN SEEN, BUT SOME FALL-OFF OF INTUMESCENT CHAR WAS OBSERVED.
14	PRE-BURN SHOWED INTENSE WHITE NON-SMOKY FLAME, WHICH CHANGED AFTER ONE MINUTE TO YELLOW. WHEN FUNNEL AND VACUUM WERE PUT IN PLACE, THE LAMINATE PLIES STARTED TO PEEL BACK AS THE LAMINATE CAME APART WITH A SMOKY YELLOW FLAME. THEN, AFTER RESIN BURNED OFF, FLAME TURNED SODIUM YELLOW IN COLOR BUT WAS CLEAR, AND BURN STABILIZED. SOME FALL-OFFS AND PEEL-BACK AFTER FIVE MINUTES.	FALL-OFF COLLECTED; SOME WAS GLASS OUTER LAYER, REST WAS GR PLIES, BOTH WITHOUT CHAR. SPECIMEN DELAMINATED AT ± 45° PLIES; MECHANICALLY HOLDING TOGETHER WITH CHAR FORMATION MINIMAL. BUNCHED ZERO'S HELD TOGETHER WELL.

R81-0911-049D(2/3)

TABLE 49 OBSERVATIONS OF BURNING AND CHARRED SPECIMENS (SHEET 3 OF 3)

LAMINATE NO.	OBSERVATIONS OF BURNING SPECIMENS	OBSERVATIONS OF CHARRED SPECIMENS (RESIDUE AFTER BURNING)
15	YELLOW COLOR PRE-BURN; NOT TOO SMOKY. AFTER TWO MINUTES, TURNED ON VACUUM/ FUNNEL AND GOT A LARGE SMOKY YELLOW FLAME FOR ONE MINUTE. THEN, FLAME STABILIZED TO A CLEAR YELLOW COLOR. SPECIMEN HELD TOGETHER, FORMING CHAR AT INTERLAYERS. EXTERNAL DELAMINATION STARTED AFTER FIVE MINUTES, WITH PEEL BACK AND FALL-OFF.	MEHCANICAL HOLD-TOGETHER BY CHAR FORMED AT BURNED ZERO LAYERS WAS QUITE GOOD. DELAMINATED ALONG ±45° LAYERS; CHAR BURNT AWAY AT EDGES AND END WITH FALL-OFF OF AN OUTER GR/GL LAYER.
16	PRE-BURN STARTED WITH WHITE FLAME AT END FOR 40 SEC, THEN BECAME YELLOW, BIGGER AND SMOKY. DURING TEN MINUTE BURN, THE RESIN STARTED BURNING HEAVILY WITH A YELLOW FLAME, THEN STABILIZED AFTER 1 1/2 MIN TO A QUIET BURN. REMAINED QUIET WITH SPECIMEN STAYING INTACT AFTER SIX MINUTES. AFTER 8-9 MIN, DELAMINATION OCCURRED.	EROSION AT POINT WHERE FLAME HIT SPECIMENS; EXTERNAL PLY FALL-OFF. GOOD CHAR ALONG BUNCHED ZERO'S WITH GOOD MECHANICAL STRENGTH. CHAR POOR WHERE FLAME COULD REACH IT.
17	NORMAL YELLOW, SOMEWHAT SMOKY PRE-BURN. TEN MINUTE BURN WAS VERY SMOKY INITIALLY WITH A YELLOW FLAME. THIS SETTLED TO A STABLE FLAME WITH NO PEEL-BACK. GOOD ADHERENCE OF LAYERS IN BULK OF SPECIMEN. ALMOST NO FLY-OFF OR FALL-OFFS.	OUTER LAYERS TURNED WHITE BUT REMAINED INTACT. GOOD CHAR FORMATION WITH MINIMAL SEPARATION OF LAYERS.
18A	INTENSE WHITE FLAME PRE-BURN WITH GREEN TINGES AND NO SMOKE. FLAME QUIETED DOWN AFTER 1 MIN TO ALMOST NOTHING, THEN PICKED UP AGAIN WHEN VACUUM/FUNNEL WAS PUT IN PLACE AND O_2 TURNED ON; THIS LASTED ONLY WHILE RESIN IN SPECIMEN BURNED. SPECIMEN SWELLED SOMEWHAT AT FLAME IMPINGEMENT END, BUT WAS OTHERWISE STABLE WITH NO FLY-OFF OR DROP-OFFS AFTER 3 MIN. FLAME PICKED UP OCCASIONALLY AS RESIN BURNED FURTHER BACK AND DEEPER INTO SPECIMEN. AFTER 7 MIN, STAYED QUIET WITH NO FLARE-UPS.	SPECIMEN WAS SWOLLEN BUT INTACT WITH GOOD INTERLAMINAR CHAR FORMATION. SOME SEPARATION OF BUNCHED ZERO'S ALONG ±45° PLIES. EXCELLENT MECHANICAL INTEGRITY. WHERE OXIDIZING FLAME HIT LAYERS, THE RESIN CHAR WAS STRIPPED. ELSEWHERE THE CHAR WAS GOOD, ESPECIALLY INTERIOR CHAR.

R81-0911-049D(3/3)

TABLE 50 OBSERVATIONS OF PARTICULATE MATTER (SHEET 1 OF 2)

LAMINATE NO.	EXAMINATION OF PARTICULATE MATERIAL COLLECTED ON MILLIPORE FILTER		
	WEIGHT OF PARTICLES, GM	OVERALL APPEARANCE, NO MAGNIFICATION	APPEARANCE UNDER 45X MAGNIFICATION
1	0.0003	VERY LIGHT PARTICLE COLLECTION, UNIFORM TAN-GRAY IN COLOR	A FEW FINE ROUND PARTICLES SEEN AGAINST A UNIFORM, SLIGHTLY DARKER BACKGROUND; A FEW GR FIBERS COLLECTED.
2	0.0025	UNIFORM BLACK, HEAVY COLLECTION	A VERY FEW BLACK CLUMPS AGAINST THE THICK UNIFORM-APPEARING SOOT-LIKE FINE POWDER. NO GR FIBERS OBSERVED.
3	0.0006	NON-UNIFORM GREEN-GRAY PARTICLE COLLECTION, VERY LIGHT AMOUNT; BLACK IN CENTER WITH SOME BLACK DOTS OVER-ALL.	APPEARED LIKE A COLLECTION OF MINERAL ASH, NOT CARBON. HAD A FEW BLACK DOTS AND A VERY FEW GR CLUMPS. NO GR FIBERS OBSERVED.
4	0.0010	LIGHT-TO-MODERATE COLLECTION OF PARTICLES, DARK GREEN-BLACK IN COLOR.	ALSO APPEARED TOO LIGHT IN COLOR FOR GR PARTICLES. RESIDUE LOOKED LIKE MINERAL OXIDES WITH A FEW GR/EP SPHERES AND CLUMPS. NO GR FIBERS WERE SEEN.
5A	0.0009	PAPER IS VERY CLEAN WITH JUST THE LIGHTEST HAZE OF GRAY SEEN YET.	LIGHT HAZY GRAY ALL OVER, WITH A VERY FEW BLACK PARTICLES. ONE LONG FIBER OF GR WAS OBSERVED, STUCK PERPENDICULAR TO PAPER.
6	0.0007	GREEN (ACTUALLY OLIVE DRAB) COLOR OVER-ALL. FAIRLY LIGHT PARTICLE COLLECTION; SOME BLACK DOTS IN MIDDLE OF FILTER.	UNIFORM LIGHT TAN-GREEN TO TAN-YELLOW COLOR, WITH A FEW BLACK PARTICLES. ONE SMALL GR FIBER WAS SEEN.
7	0.0007	UNIFORM BLACK COATING; MEDIUM COLLECTION.	VERY FINE DARK GREEN PARTICLES MIXED WITH BLACK LARGER PARTICLES, ACTUALLY OVERLAID WITH BLACK; NO GR FIBERS WERE SEEN.
8	0.0010	VERY LIGHT TAN COLLECTION; UNIFORM LAYER OF PARTICLES, SMALL QUANTITY OVER ALL.	SMALL QUANTITY OF BLACK IRREGULARLY SHAPED CLUMPS ON PALE COLORED BACKGROUND; ONE GR FIBER WAS OBSERVED.

R81-0911-050D(1/2)

TABLE 50 OBSERVATIONS OF PARTICULATE MATTER (SHEET 2 OF 2)

LAMINATE NO.	EXAMINATION OF PARTICULATE MATERIAL COLLECTED ON MILLIPORE FILTER		
	WEIGHT OF PARTICLES, GM	OVERALL APPEARANCE, NO MAGNIFICATION	APPEARANCE UNDER 45X MAGNIFICATION
9	0.0008	VERY LIGHT COLLECTION OF FINE PARTICLES, GRAY IN COLOR	VERY SMALL QUANTITY OF TINY BLACK DOTS IN A SHINY, CRYSTALLINE APPEARING LAYER; NO GR FIBERS WERE OBSERVED.
10	0.0017	UNIFORM FAIRLY HEAVY BLACK LAYER COLLECTED ON MILLIPORE FILTER.	THICK, BROWN-BLACK LAYER OF RATHER FINE PARTICLES, WITH LARGER DARK CLUMPS ON TOP. SEVERAL LONG GR FIBERS OBSERVED.
11	0.0020	LIKE NO. 10	SIMILAR TO NO. 10, BUT NO LONG GR FIBERS OBSERVED AND NO CLUMPS ON TOP. APPEARANCE OF RESIDUE ON FILTER IS "FELTED", A COLLECTION OF SHORT FRAGMENT INTERTWINED PARTICULATE MATERIAL.
12	0.0008	LIGHT LAYER OF DARK GRAY POWDER	SIMILAR TO NO. 9, NO GR FIBERS WERE OBSERVED.
13A	0.0013	PALE TAN COLOR, VERY LIGHT LAYER OF PARTICLES.	VERY SMALL NUMBER OF BLACK PARTICLES OBSERVED AGAINST LIGHT COLORED BACKGROUND. TWO SHORT GR FIBERS WERE OBSERVED.
14	0.0028	LIKE NO. 10 BUT SOMEWHAT HEAVIER ACCUMULATION.	LIKE NO. 11, WITH NO GR FIBERS OBSERVED.
15	0.0021	LIKE NO. 10; ABOUT THE SAME TYPE AND QUANTITY OF PARTICULATE MATERIAL COLLECTED.	LIKE NO. 11, WITH NO GR FIBERS OBSERVED.
16	0.0016	RESIDUE ON MILLIPORE FILTER WAS DARKER GRAY/TAN THAN NO. 13; SOMEWHAT MORE PARTICLES COLLECTED.	LARGER AMOUNT OF BLACK IRREGULAR PARTICLES THAN NO. 13. NO LONG GR FIBERS WERE OBSERVED.
17	0.0018	LIGHT LAYER OF BLACK POWDER WITH UNDERLAYMENT, TAN IN COLOR.	LIKE NO. 11 WITH NO LONG GR FIBERS OBSERVED. THINNER MAIN LAYER, WITH "FELTED" APPEARANCE LIKE NO. 11 AND OTHERS.
18A	0.0028	GRAY LAYER, THIN BUT NOT SPARSE, OF COLLECTED PARTICULATES. SIX LARGE CLUMPS OBSERVED; MATERIAL ON CENTER OF MILLIPORE FILTER WAS BLACK.	FAIRLY LARGE CLUMPS ON TOP, OTHERWISE LIKE NO. 9, BUT WITH MORE BLACK DOTS OF IRREGULAR-SHAPED MATERIAL.

R81-0911-050D(2/2)

- Rating 5. Some char formed but less than that rated 4, layers break up easily upon probing; overall condition of residue: minimal
- Rating 6 (Lowest). No char, no structural integrity, layers separated; overall condition of residue: poor

- Particulate Matter:
  - Rating 1 (Highest). No graphite material present as clumps or fibers
  - Rating 2. Very few graphite clumps or fibers observed
  - Rating 3. Clumps (aggregates of graphite) and/or fibers observed, more than that for Rating 2
  - Rating 4. Relatively large amount of collectables observed
  - Rating 5 (Lowest). Abundance of collected clumps and/or individual fibers

The char characteristics and particulate matter ratings for the thin and thick laminates concepts are reported in Table 51. The thick specimens, as a class, produced more collected particulates than the thin specimens. They also produced much more char in the residual specimen.

Correlation between these tests and the earlier flame impingement tests for thin laminates were quite good; all four of the early selections appeared as final selections, with only the rank changing.

For the thick laminates, the correlation was fairly good because of the relatively large amount of collectibles, with three specimens in general agreement but of changed rank.

TABLE 51 CHAR CHARACTERISTIC AND PARTICULATE MATTER RATINGS

TYPE OF LAMINATE	LAMINATE NO.	FIBER RETENTION FEATURE	CHARRED SPECIMEN RESIDUE		COLLECTED PARTICULATES				OVERALL RANK
			CONDITION	RANK	CLUMPS	RANK	FIBERS	RANK	
THIN	1	CONTROL	POOR	6	SOME	4	SOME	4	8
	2	ALUMINUM-FACED	MINIMAL	5	SOME	4	NONE	1	6
	3	B/EP FACED	GOOD	3	SOME	4	NONE	1	4
	4	WOVEN GL/EP-FACED	FAIR	4	SOME	4	NONE	1	5
	5A	BORON POWDER	EXCELLENT	1	NONE	1	ONE	2	1
	6	WOVEN GR/EP-FACED	GOOD	3	NONE	1	ONE	2	2
	7	PI-SIZED GR	POOR	6	MANY	5	NONE	1	7
	8	KIMBAR FACED	POOR	6	SOME	4	ONE	2	7
	9	SODIUM SILICATE-TREATED	GOOD	3	FEW	3	NONE	1	3
THICK	10	CONTROL	POOR	6	MANY	5	SOME	4	5
	11	B/EP PLIES	FAIR TO GOOD	3 1/2	FEW	3	NONE	1	4
	12	WOVEN GL/PI PLIES	VERY GOOD	2	FEW	3	NONE	1	2
	13A	INTUMESCENT COATED	EXCELLENT	1	VERY FEW	2	TWO	2	1
	14	WOVEN GL/EP PLIES	FAIR TO GOOD	3 1/2	FEW	3	NONE	1	4
	15	WOVEN GL-GR/EP PLIES	FAIR TO GOOD	3 1/2	FEW	3	NONE	1	4
	16	PI-SIZED GR	GOOD	3	FEW	3	NONE	1	3
	17	FIRE-RETARDANT EP	VERY GOOD	2	FEW	3	NONE	1	2
	18A	SODIUM BORATE-TREATED	VERY GOOD	2	MANY	5	NONE	1	3

R81-0911-051D

Section 4

CONCLUSIONS

The results of the physical, mechanical, and burn tests were normalized. Data are presented in Table 52. Based on an analysis of these results, the required four selections in both thin and thick laminate categories were made. They are, in decreasing order of rank:

- Thin laminates:

  No. 5A - boron powder in matrix (best)

  No. 3 - boron faces

  No. 6 - woven graphite faces

  No. 4 - woven fiberglass faces

- Thick laminates:

  No. 13 - intumescent coating (best)

  No. 17 - fire-retardant epoxy

  No. 15 - woven Gr/Gl plies and faces

  No. 11 - boron plies and faces

TABLE 52 NORMALIZED PHYSICAL & MECHANICAL TEST DATA & RELATIVE BURN TEST RATINGS

LAM. NO.	FIBER RETENTION FEATURE	THICKNESS	SPEC. GRAVITY	HDT (UNCOND) (T_G)	TGA % WGT. LOSS	UNCOND. AT ELEV TEMP			MOIST. AT ELEV TEMP			LAB. BURN TESTS		DECISION AND COMMENTS, (FOUR SELECTIONS IN EACH CATEGORY)	
						FLEXURE		SHEAR STRENGTH	FLEXURE		SHEAR STRENGTH	OXYGEN INDEX	CHAR & PARTICULATES	RANK	COMMENT
						STRENGTH	MODULUS		STRENGTH	MODULUS					
1	CONTROL	1.0	1.0	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	–	CONTROL
2	ALUMINUM FACED	1.2	1.0	1.02	0.53	1.16	1.22	1.23	1.52	1.32	1.40	1.57	1.29	–	–
3	B/EP FACED	0.9	1.1	1.00	0.89	1.11	1.20	0.87	1.10	1.16	0.86	1.04	1.57	2	GOOD PHYSICAL, EXCELLENT MECHANICAL AND GOOD BURNING PROPERTIES
4	WOVEN GL/EP FACED	0.9	1.1	1.04	1.06	1.11	1.05	0.85	1.25	1.15	0.84	1.14	1.43	4	EXCELLENT PHYSICAL, GOOD MECHANICAL AND FAIRLY GOOD BURNING PROPERTIES
5A	BORON POWDER	1.0	1.0	0.97	0.92	0.76	1.00	1.11	0.97	1.07	1.10	1.04	2.00	1	BEST COMBINATION OF PHYSICAL, MECHANICAL, AND BURNING PROPERTIES
6	WOVEN GR/EP FACED	0.9	1.0	0.99	1.11	0.91	1.05	0.69	0.89	1.15	0.83	0.93	1.86	3	VERY GOOD PHYSICAL, GOOD MECHANICAL AND VERY GOOD BURNING PROPERTIES
7	PI SIZED GR	1.0	1.0	0.96	1.04	0.93	1.02	0.78	0.77	0.95	0.87	1.00	1.14	–	–
8	KIMBAR FACED	1.3	1.0	0.98	0.86	0.53	0.57	0.74	0.64	0.51	0.81	1.57	1.14	–	–
9	SODIUM SILICATE	0.9	0.9	0.98	0.91	0.91	1.16	0.67	0.65	1.00	0.67	0.93	1.71	–	–
10	CONTROL	1.0	1.0	1.00	1.00	1.00	1.00	1.0	1.00	1.00	1.00	1.00	1.43	–	CONTROL FOR THICK SPECIMENS BUT RANKED RELATIVE TO THIN AND THICK
11	B/EP PLIES	1.0	1.1	0.97	0.95	1.01	1.06	1.05	0.90	1.12	0.82	1.00	1.57	4	GOOD PHYSICAL, GOOD MECHANICAL AND MODERATE BURNING PROPERTIES
12	WOVEN GL/PI PLIES	1.0	1.1	1.22	5.73	0.83	1.00	0.68	0.88	1.00	0.63	1.36	1.86	–	–
13/13A	INTUMESCENT COATED	1.1	1.0	0.90	1.13	1.02	0.90	0.87/0.93	0.93/0.71	0.94/0.86	1.06/0.92	1.59	2.00	1	BEST COMBINATION OF PHYSICAL, MECHANICAL AND BURNING PROPERTIES
14	WOVEN GL/EP PLIES	1.0	1.0	1.03	1.26	1.04	0.96	1.01	0.88	0.93	0.97	0.93	1.57	–	GOOD PHYSICAL AND MECHANICAL PROPERTIES
15	WOVEN GL-GR/EP PLIES	1.0	1.0	1.03	1.48	1.03	0.96	1.03	0.88	0.88	0.91	0.91	1.57	3	EXCELLENT PHYSICAL, GOOD MECHANICAL AND MODERATE BURNING PROPERTIES
16	PI SIZED GR/EP	1.0	1.0	0.97	1.28	0.99	1.00	0.90	0.60	0.91	0.76	0.82	1.71	–	–
17	FIRE RETARDANT EP	1.0	1.0	1.01	1.04	1.12	0.92	1.12	0.89	0.88	0.99	1.05	1.86	2	VERY GOOD PHYSICAL, MECHANICAL AND BURNING PROPERTIES
18A	SODIUM BORATE	1.2	1.0	0.90	0.94	1.03	0.94	1.19	0.68	0.95	0.08	1.03	1.71	–	–

R81-0911-052D

APPENDIX A

PRELIMINARY BURN TEST OBSERVATIONS

TABLE 53 PRELIMINARY BURN TEST OBSERVATIONS (SHEET 1 OF 3)

LAMINATE NO.	PANEL NO.	OBSERVATIONS
1	1-13	EP RESIN QUICKLY IGNITED AND BURNED OFF WITH BLACK SMOKE, GR LAYERS SEPARATED, TURNED RED, FINE FIBERS FLEW OFF, NO COHESION TO REMAINS, VERY FRAGILE, SEGMENTS OF LAYERS FELL OFF, MINIMAL CHAR.
2	2-13	EP AND AL VERY QUICKLY IGNITED AND BURNED WITH SOOTY BLACK SMOKE GIVEN OFF; AL OXIDIZED, TURNED GREY/WHITE, SOME FELL OFF, SOME REMAINED; OBSERVATIONS FROM LAMINATE NO. 1 APPLY; NO IMPROVEMENT.
3	3-13	IGNITION OF EP RESIN DELAYED FOR 15-20 SEC, THEN BURNED WITH SMOKY, SOOTY FLAME AS BEFORE; HOWEVER, B/EP OUTER LAYERS REMAINED INTACT; FILAMENTS ARE STRONG BUT LITTLE CHAR FORMED, NO SEGMENTS OF LAYERS FELL OFF, DID NOT OBSERVE INNER GR FIBERS FLOATING OFF, GREEN COLOR TO FLAME NOTICED; INTERNAL LAYERS (GR) APPEARED SOMEWHAT STABILIZED BY MECHANICAL ENTRAPMENT.
4	4-13	IGNITION OF EP RESIN DELAYED FOR 5-10 SEC; SIMILAR BEHAVIOR TO LAMINATES ABOVE; OUTER GLASS LAYERS REMAINED INTACT BUT CURL BACK MUCH MORE THAN BORON, NO. 3 (NO GREEN COLOR); INNER LAYERS NOT STABILIZED LIKE LAMINATE NO. 3; MINIMAL AMOUNT OF FINE GR FIBERS FLEW OFF: MINIMAL CHAR BUT FAIR MECHANICAL ENTRAPMENT.
5A	5A-13	EP RESIN IGNITED WITHIN 5 SEC AND BURNED WITH A YELLOW SMOKY FLAME WHITE SMOKE OBSERVED AS SAMPLE BURNED; A VERY SMALL AMOUNT OF FINE FIBERS FLEW OFF ON TAPPING AS SAMPLE WAS ROTATED; GR FIBERS HAD COHERENCE AND RIGIDITY EVEN THOUGH OUTER LAYERS SEPARATED; INNER LAYERS WERE VERY RIGID.
6	6-13	IGNITION STARTED WITHIN 3-5 SEC, BLACK SMOKE PLUS WHITE SMOKE; ALMOST NO FINE FIBERS FLYING OFF, NONE FROM WOVEN GR OUTER LAYERS; NO LAYERS FELL, MINIMAL DEFORMATION OF OUTER WOVEN LAYERS; ALSO, UNI-INNER LAYERS STAYED COMPACTED; WOVEN LAYERS NOT STIFF, BUT WEAVE HELD THEM INTACT; MECHANICAL ENTRAPMENT GOOD, CHAR MINIMAL.
7	7-13	EPOXY RESIN IGNITED WITHIN 5-10 SEC, BURNED WITH YELLOW SMOKY FLAME; SAMPLE QUICKLY DELAMINATED AND LOST FINE FIBERS PLUS CHUNKS OF PLIES; INTERIOR DOUBLE PLIES STAYED FAIRLY RIGID; PI SIZING DID NOT APPEAR TO HELP AND APPEARED TO BURN OFF.
8	8-13	SIMILAR TO LAMINATE NO. 1, EXCEPT THAT IGNITION WAS DELAYED 8-10 SEC; MOSTLY BLACK SMOKE, LATER SOME WHITE SMOKE; NO IMPROVEMENT; MINIMAL CHAR, NO MECHANICAL ENTRAPMENT.

R81-0911-053D(1/3)

TABLE 53 PRELIMINARY BURN TEST OBSERVATIONS (SHEET 2 OF 3)

LAMINATE NO.	PANEL NO.	OBSERVATIONS
9	9-13	SURFACE LAYERS OF WOVEN GR WERE STIFFER; WEAVE HELD TOGETHER VERY WELL; GOOD MECHANICAL ENTRAPMENT, NOT MUCH CHAR ON INTERIOR LAYERS; YELLOW COLOR (SODIUM) TO FLAME.
10	10-13	MARKED DIFFERENCE FROM 10-PLY PANELS; THE SURFACE ± 45° PLIES BURNED LIKE LAMINATE NO. 1, BUT THE COUPLED 0° AND 90° PLIES HELD TOGETHER WELL WITH GOOD CHAR; REMAINS OF SPECIMEN SHOWED A STIFF INTERIOR; STABILIZED AT 3 MIN (RESIN BURNED AWAY WITH YELLOW SMOKY FLAME), THEN REMAINED RELATIVELY UNCHANGED (A FEW OUTER PIECES FELL OFF OR FLEW OFF) TILL 5 MIN ELAPSED.
11	11-13	IGNITION DELAYED 30-35 SEC; VERY GOOD CHAR ON 0° AND 90° GR/EP PLY MULTIPLE LAYERS; VERY GOOD MECHANICAL RETENTION OF SURFACE AND INTERNAL B/EP LAYERS; GLASS SCRIM (B/EP PREPREG TAPE SUPPORT) ROLLED BACK ON SURFACE; NO FALL-OFFS, NO FLOATERS, EVEN AFTER BLACK SMOKE/YELLOW FIRE STAGE WHICH LASTED 3 MIN; THEN GREEN TINGE TO FLAME, NO CHANGE FOR NEXT 2 MIN; SIMILAR TO LAMINATE NO. 10, BUT BETTER BECAUSE OF ± 45° B/EP PLIES.
12	12-13	NYLON PEEL PLY COULD NOT BE REMOVED; RESIN IGNITION DELAYED FOR 30-40 SEC; BURNED WITH YELLOW, SMOKY FLAME (BUT LESS SMOKY THAN EPOXY); GLASS OUTER PLY ROLLED BACK A SMALL AMOUNT, NOMINALLY EXPOSING INTERIOR GR; NO DROPS OF INNER PLIES, ALMOST NO FLOATERS; EXCELLENT CHAR FORMATION ON INTERIOR PLIES WHICH APPARENTLY FUSED INTO ONE LARGE MASS.
13	13-13	RESIN IGNITION DELAYED 20-25 SEC, SAMPLE BURNED WITH YELLOW SOOTY FLAME; SURFACE PLY BENT SLIGHTLY (10°-15°) BUT DIDN'T ROLL BACK; SOME CHAR AT INNER MULTIPLE 0° AND 90° LAYERS BUT MOSTLY HELD TOGETHER MECHANICALLY; SMALL RELEASE OF SEGMENTS AND FIBERS.
13	13-13A	INTUMESCENT COATING DISCOLORED, MELTED, BUBBLED AND SWELLED, THEN BURNED, FORMING A STABLE CHAR WHICH PROTECTED THE UNDERLYING LAMINATE, EVEN AT EXPOSED EDGES AND END; SOME BURNING OF LAMINATE AND PLY SPLITTING OCCURED, BUT THIS WAS MINIMAL; FOR THE 5+ MIN THAT THIS SPECIMEN WAS IN THE FIRE ROTATING, TAPPING AND EDGE, END AND SIDE EXPOSURE, THE INTUMESCENT COATING GAVE EXCELLENT PROTECTION.
14	14-13	IGNITION DELAYED 20-25 SEC; VERY GOOD CHAR OF INNER 0° AND 90° GR/EP LAYERS (MULTIPLES); GL SURFACE PLY ROLLED BACK EXPOSING GR WHICH YIELDED FLOATERS BUT NO DROPS; INTERIOR GLASS LAYERS ACTED AS FLAME STOPPERS, SO THAT RESIDUE OF LAMINATE WAS STRONG; NOT AS GOOD AS LAMINATE NO. 11, BUT BETTER THAN 10, ALTHOUGH LESS SEPARATION OF INTERIOR GR AND GL PLIES THAN LAMINATE NO. 11.

R81-0911-053D(2/3)

TABLE 53 PRELIMINARY BURN TEST OBSERVATIONS (SHEET 3 OF 3)

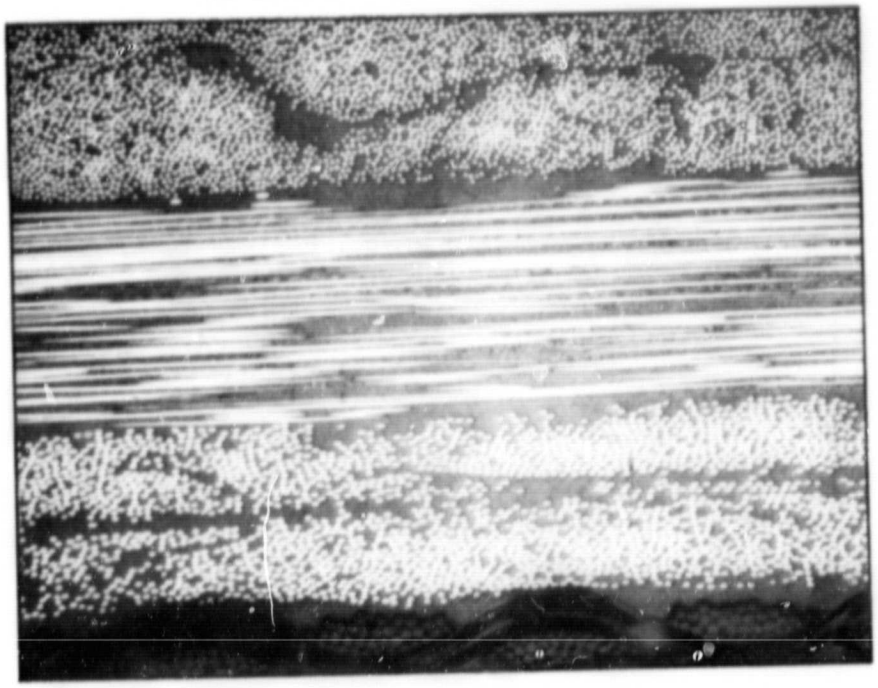
LAMINATE NO.	PANEL NO.	OBSERVATIONS
15	15-13	IGNITION DELAYED FOR 20-25 SEC; EP THEN BURNED OFF WITH YELLOW SMOKY FLAME FOR ABOUT TWO MIN; VERY GOOD CHAR AT INNER MULTIPLE 0° PLIES, BUT SEPARATION OF GR/GL AND 0° AND 90° PLY BUNDLES OCCURRED; SMALL AMOUNTS OF FLOATERS AND DROPS RELEASED; RESIDUE QUITE STRONG - GR/GL PLIES ACTED AS FLAME-STOPPERS.
16	16-13	COMBUSTION STARTED AFTER 25 SEC; WITH YELLOW SMOKY FLAME; BURNING WAS SLOW, CONTINUING OVER 4 MIN; A FEW "DROPS" BUT ALMOST NO "FLOATERS" WERE OBSERVED; EARLY SEPARATION AT THE CENTER PLY REGION TOOK PLACE AND THEN THE REST OF THE LAMINATE SPLIT ALONG STACKED 0° AND 90° INTERFACES; EXCELLENT CHAR FORMED IN LAMINATE, AND "DROPS" WHEN SCRAPED DID NOT SEPARATE AS DID UNPROTECTED GR; STIFFNESS OF RESIDUAL CHARRED LAMINATE WAS GOOD.
17	17-13	COMBUSTION STARTED IN 15 SEC, LASTED ABOUT 3 1/2 MIN, YELLOW SMOKY FLAME, ACRID ODOR (THE RETARDANT?); A FEW FLOATERS BUT NO DROPS WERE OBSERVED; LAMINATE SEPARATED ALONG STACKED 0° AND 90° BANDS; CHAR WAS QUITE GOOD, BUT NOT AS TOUGH AS THE LAMINATE NO. 16 SPECIMEN; THE GLASS (WOVEN) PROVIDED STRENGTH TO THE RESIDUAL LAMINATE (MECHANICAL RETENTION OF CHARRED LAYERS).
18	18-13	PEEL PLY WAS NOT REMOVED: COMBUSTION STARTED AFTER 20-25 SEC, CONTINUED FOR ABOUT 3 MIN, YELLOW SMOKY FLAME; SURFACE PLY "DROPS" WERE OBSERVED, NOT TOO MANY, AND THOSE THAT FELL WERE RIGID AND COHERENT; SPECIMEN SEPARATION BETWEEN BUNDLED 0'S AND 90'S WAS GREATER THAN WHEN A WOVEN REINFORCEMENT WAS USED BUT CHAR FORMATION AT INTERIOR WAS VERY GOOD (CHAR AT EXTERIOR, DOWN 7 TO 8 PLIES WAS LESS, PROBABLY BURNT AWAY); RESIDUAL LAMINATE WAS STIFF, MOSTLY AT INTERIOR WHERE CHAR WAS GOOD, OUTER PLIES WHICH HAD SEPARATED MOST WERE READILY PUSHED BACK IN TOWARDS CENTER.
18A	18A-13	EP RESIN IGNITED WITHIN 4 SEC, BURNED WITH YELLOW SMOKY FLAME. NO FINE FIBERS OBSERVED FLYING OFF, A FEW (3) LOOSE TOWS DROPPED OFF BUT THEY WERE COHERENT; NO CHAR FORMED BUT THE BORATE SIZE HELD THE CLOTH LAYERS INTACT; ALL RESIN BURNT AWAY AND THE LAYERS SEPARATED HOWEVER, RESIDUAL STIFFNESS WAS GOOD; EXCELLENT PERFORMANCE.

R81-0911-053D(3/3)

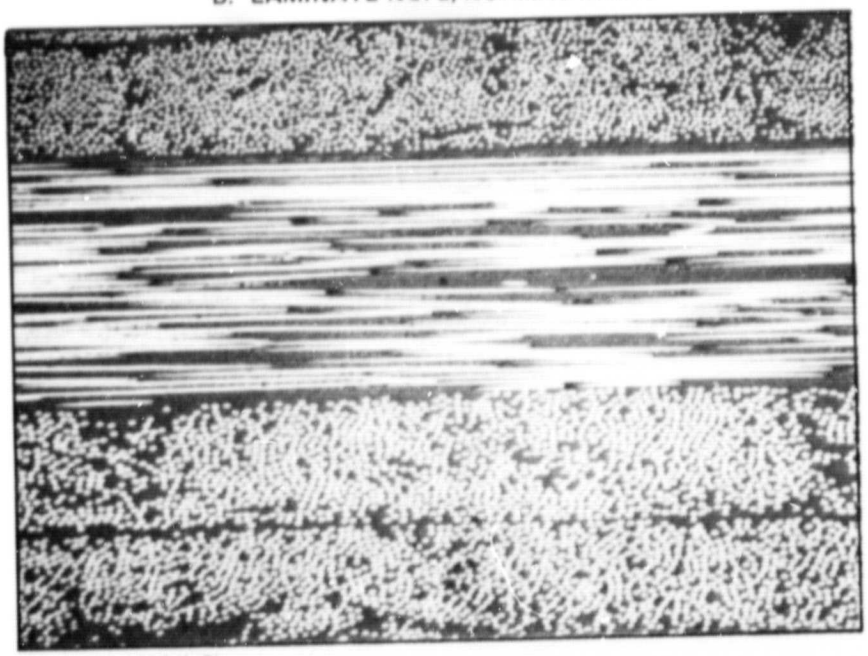
APPENDIX B

EDGE PHOTOMICROGRAPHS OF CANDIDATE LAMINATE CONCEPTS

A. LAMINATE NO. 1, NORMAL LIGHT



B. LAMINATE NO. 2, NORMAL LIGHT



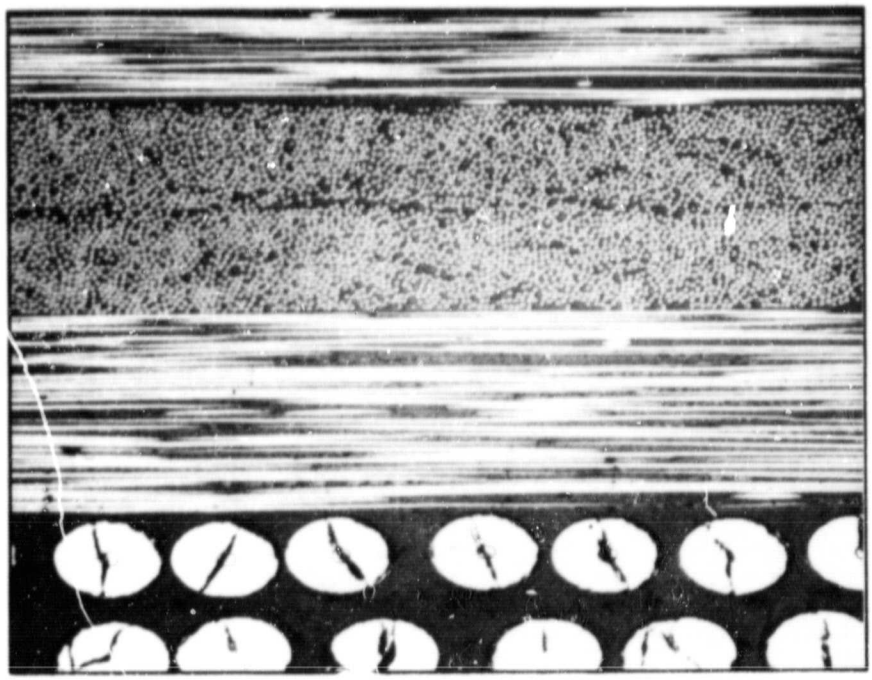
R81-0911-058D(1/7)

Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 1 of 7)

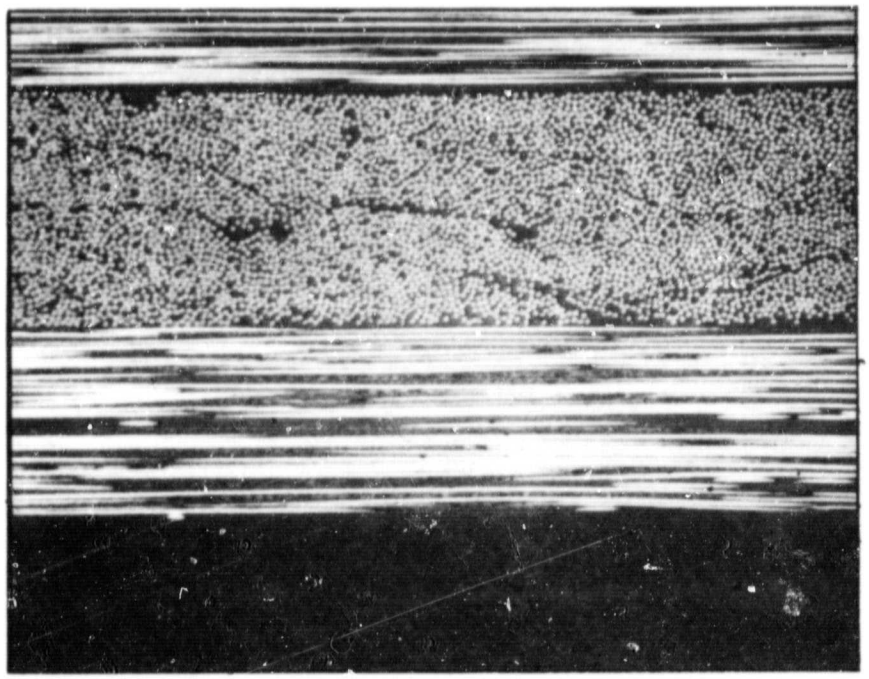
ORIGINAL PAGE IS
OF POOR QUALITY

PAGE 94 INTENTIONALLY BLANK

C. LAMINATE NO. 3, NORMAL LIGHT



D. LAMINATE NO. 4, NORMAL LIGHT



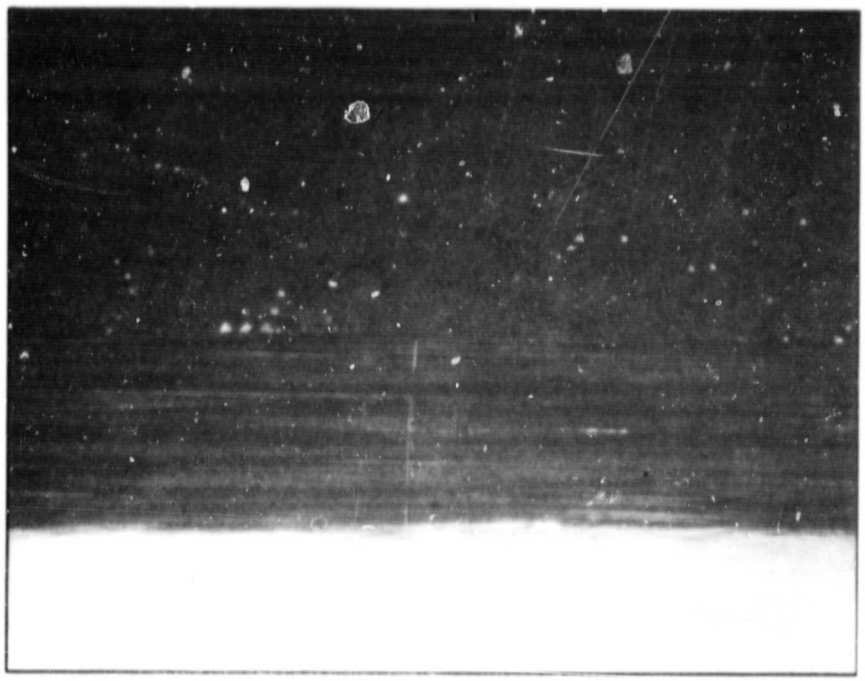
R81-0911-058D(2/7)

Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 2 of 7)

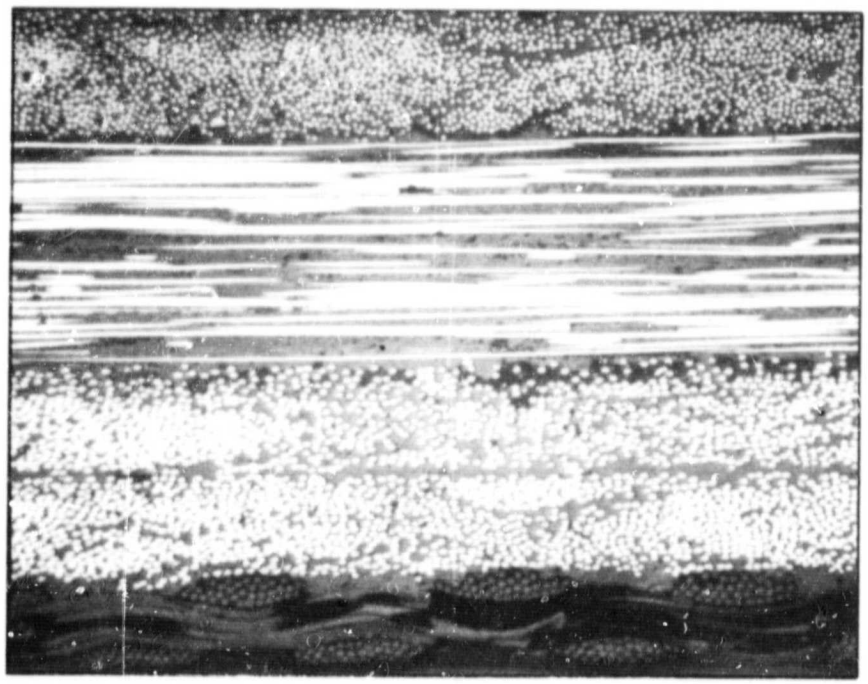
ORIGINAL PAGE IS
OF POOR QUALITY

C-2

E. LAMINATE NO. 4, POLARIZED LIGHT



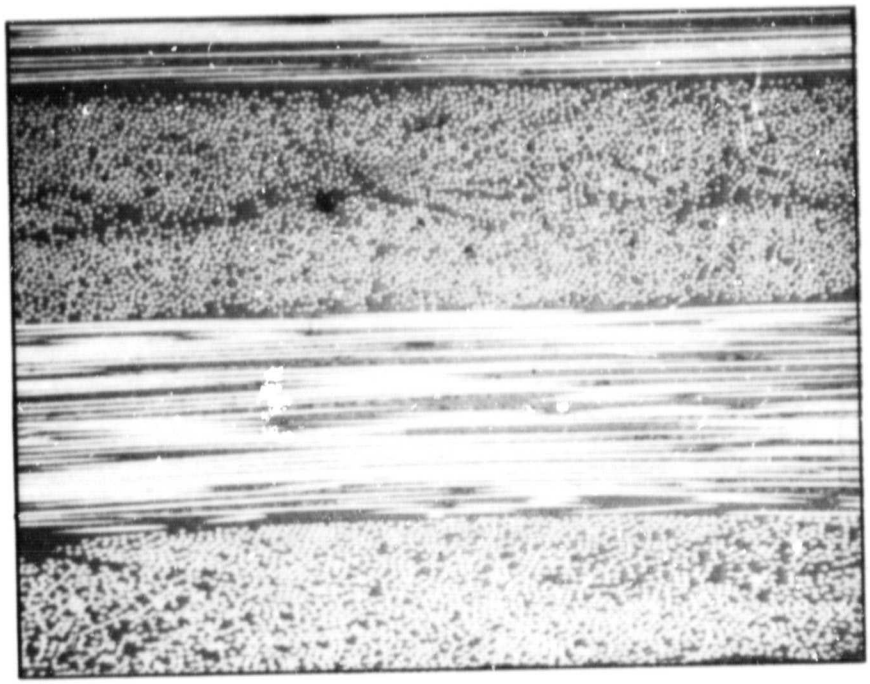
F. LAMINATE NO. 5, NORMAL LIGHT



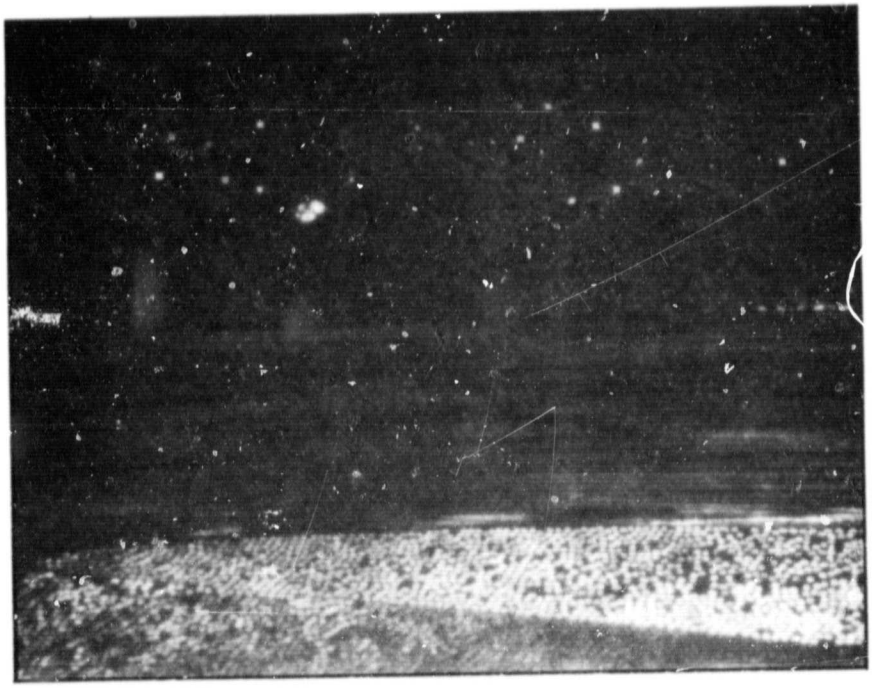
R81-0911-058D(3/7)

Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 3 of 7)

G. LAMINATE NO. 6, NORMAL LIGHT



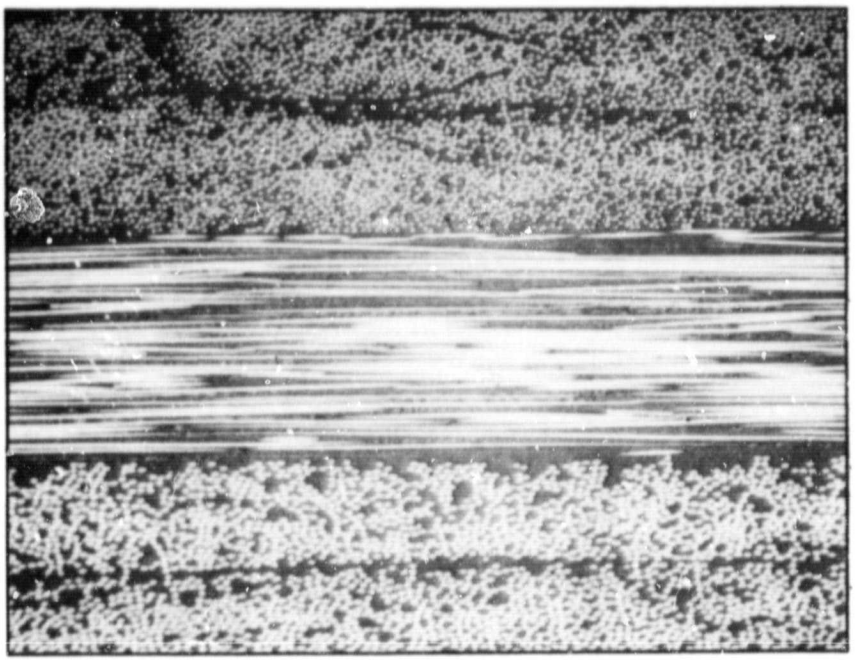
H. LAMINATE NO. 6, POLARIZED LIGHT



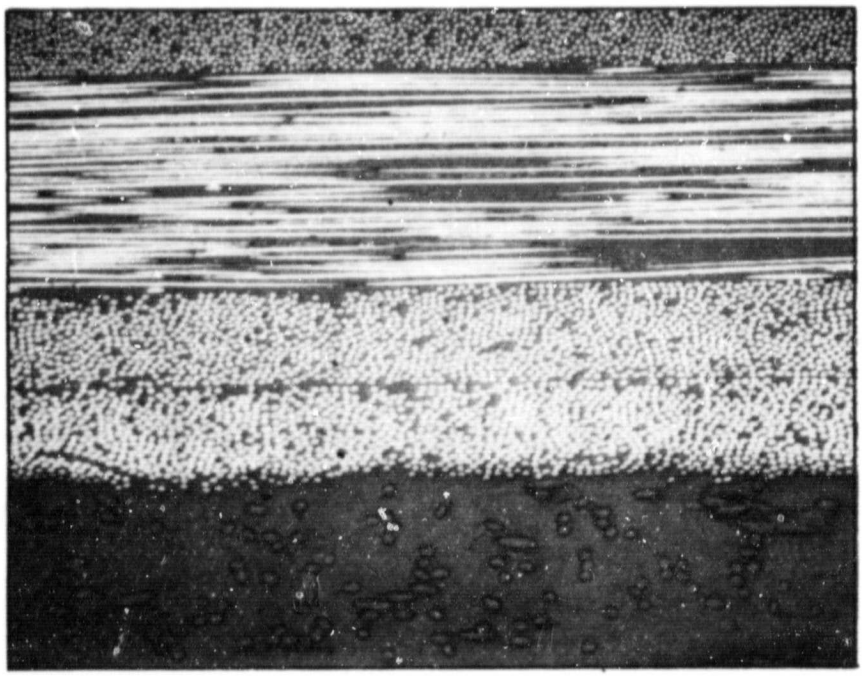
R81-0911-058D(4/7)

Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 4 of 7)

I. LAMINATE NO. 7, NORMAL LIGHT



J. LAMINATE NO. 8, NORMAL LIGHT

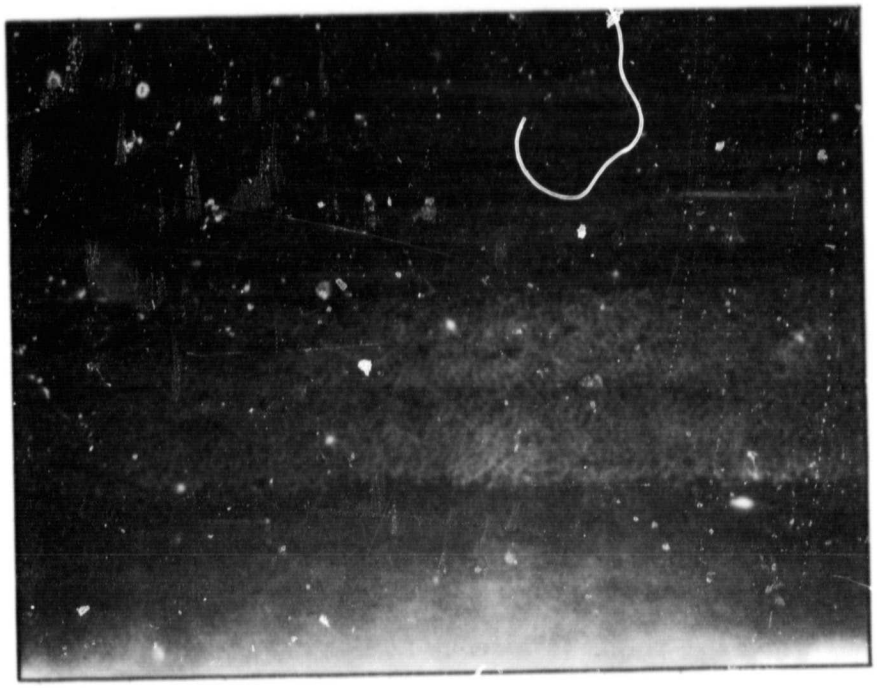


R81-0911-058D(5/7)

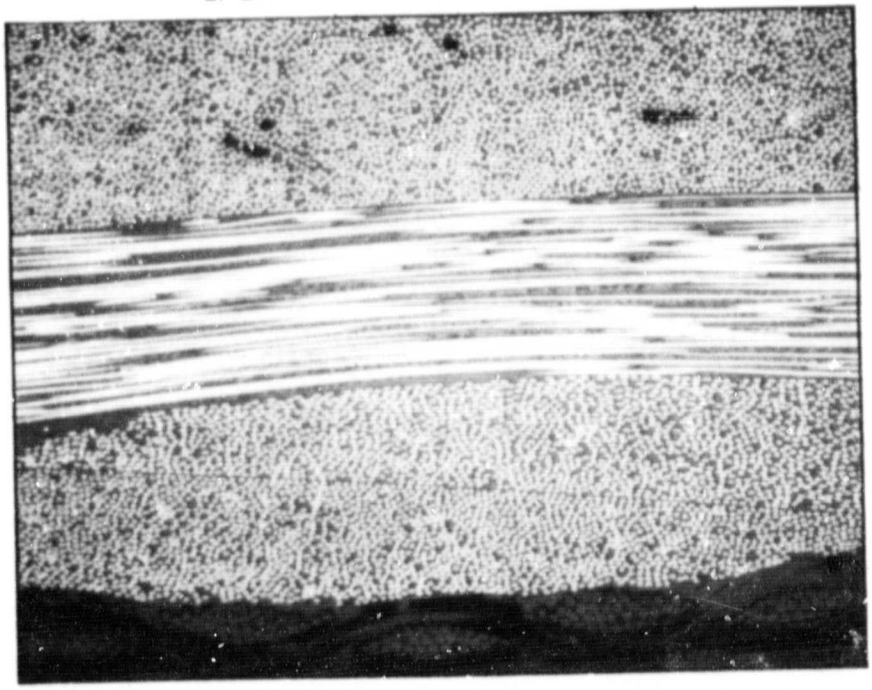
Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 5 of 7)

ORIGINAL PAGE IS
OF POOR QUALITY

K. LAMINATE NO. 8, POLARIZED LIGHT



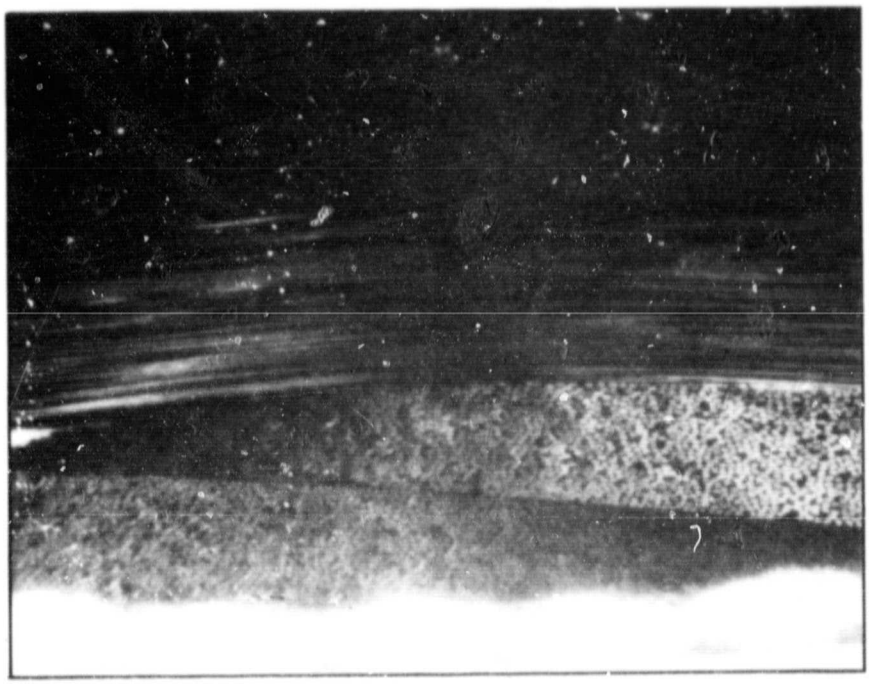
L. LAMINATE NO. 9, NORMAL LIGHT



R81-0911-058D(6/7)

Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 6 of 7)

M. LAMINATE NO. 9, POLARIZED LIGHT

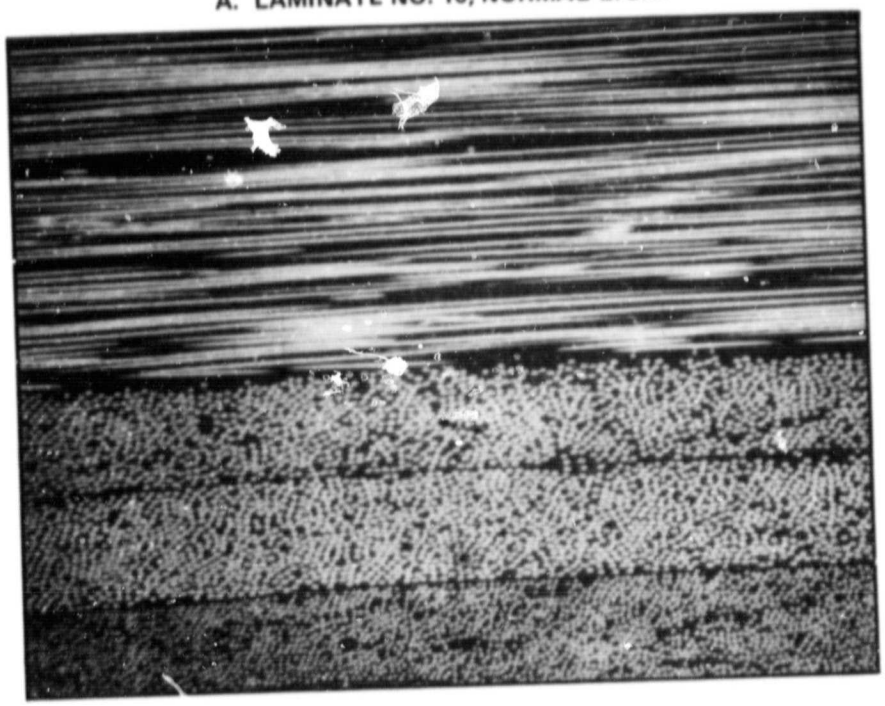


R81-0911-058D(7/7)

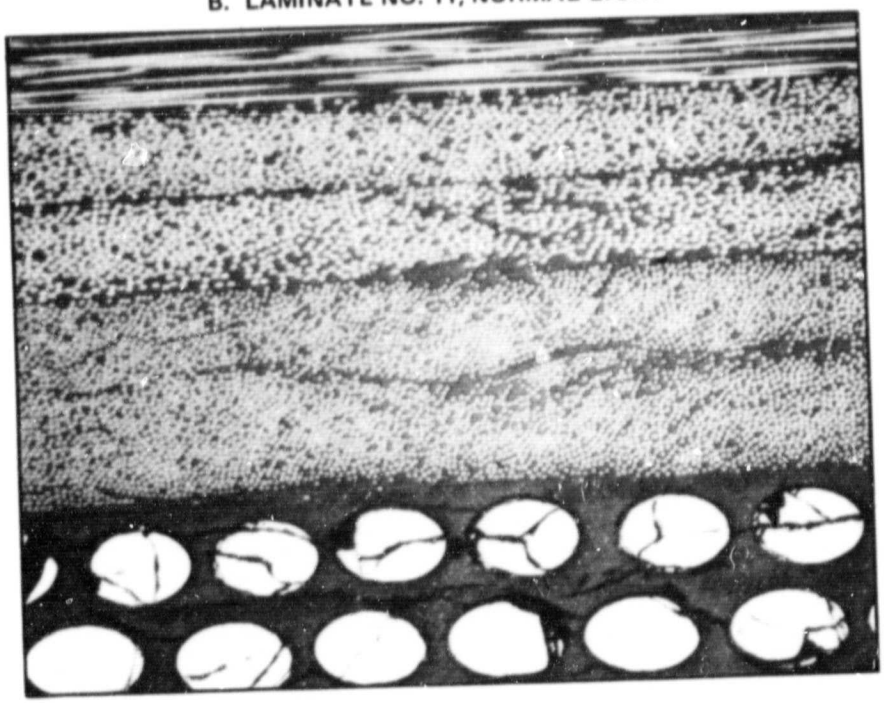
Fig. 5 Photomicrographs of Thin Laminates, 100x Mag (Sheet 7 of 7)

ORIGINAL PAGE IS
OF POOR QUALITY

A. LAMINATE NO. 10, NORMAL LIGHT



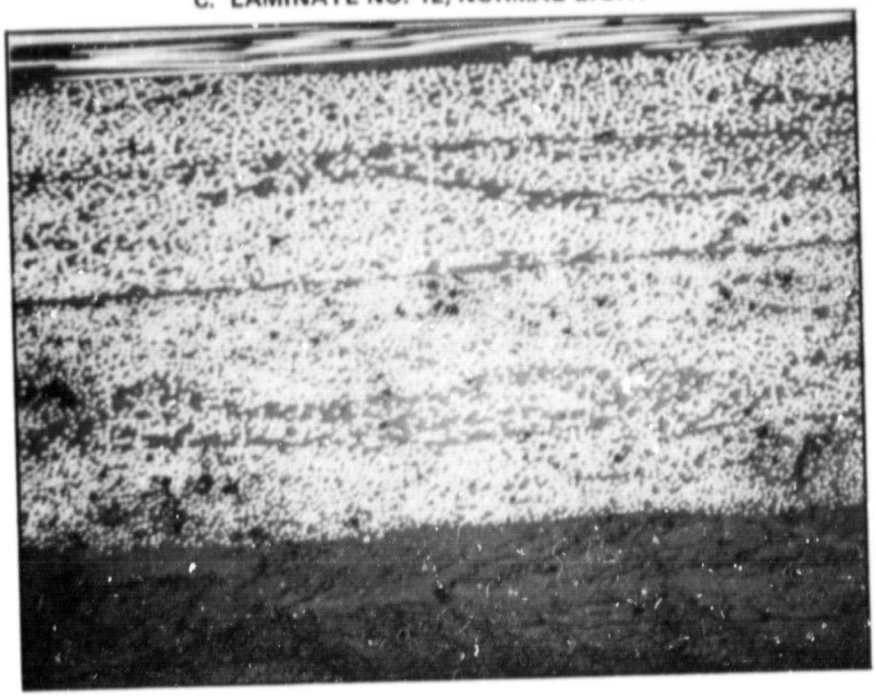
B. LAMINATE NO. 11, NORMAL LIGHT



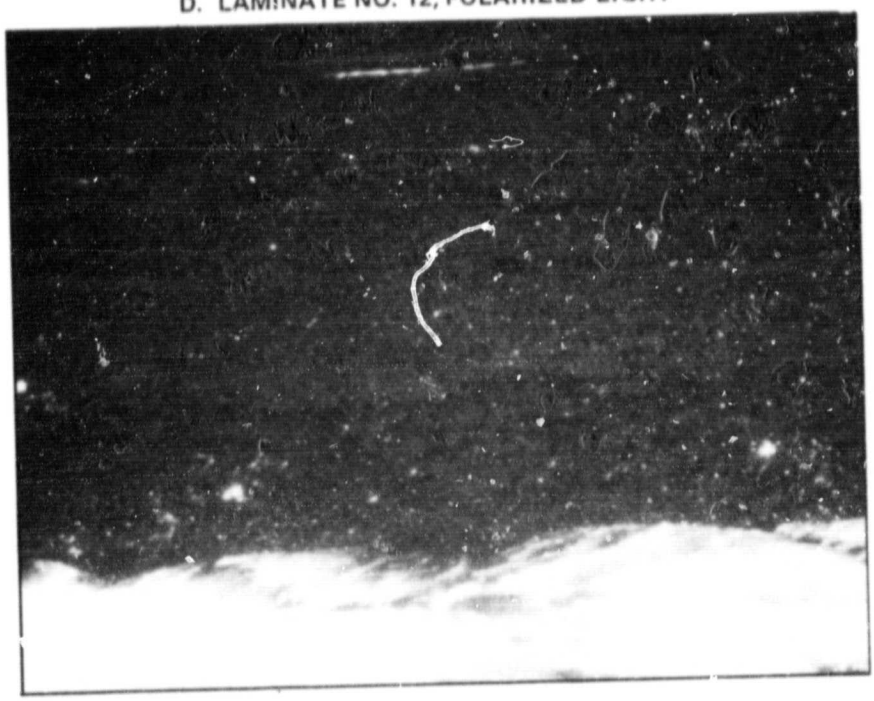
R81-0911-059D(1/8)

Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 1 of 8)

C. LAMINATE NO. 12, NORMAL LIGHT



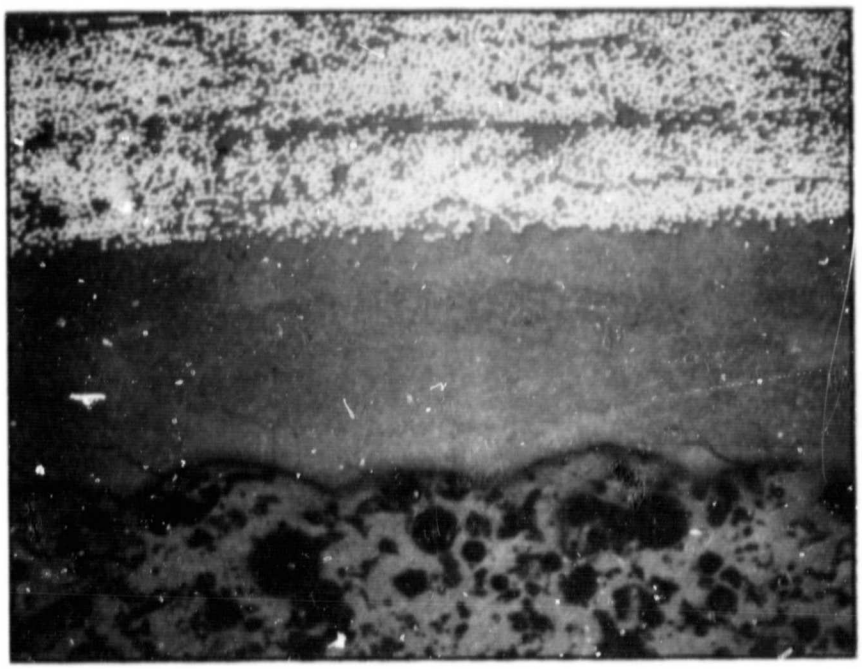
D. LAMINATE NO. 12, POLARIZED LIGHT



R81-0911-059D(2/8)

Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 2 of 8)

E. LAMINATE NO. 13, NORMAL LIGHT

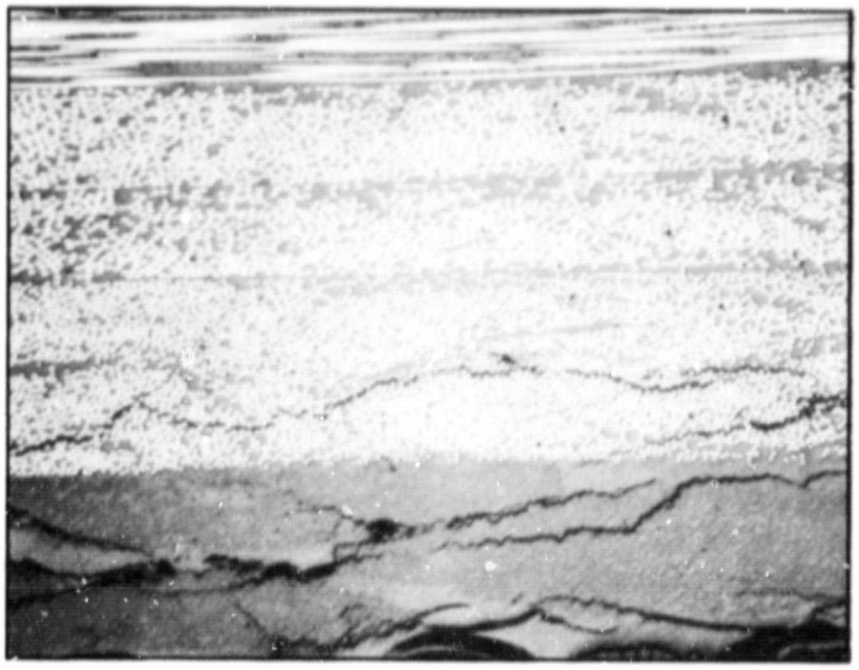


F. LAMINATE NO. 13, POLARIZED LIGHT

R81-0911-059D(3/8)

Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 3 of 8)

G. LAMINATE NO. 14, NORMAL LIGHT



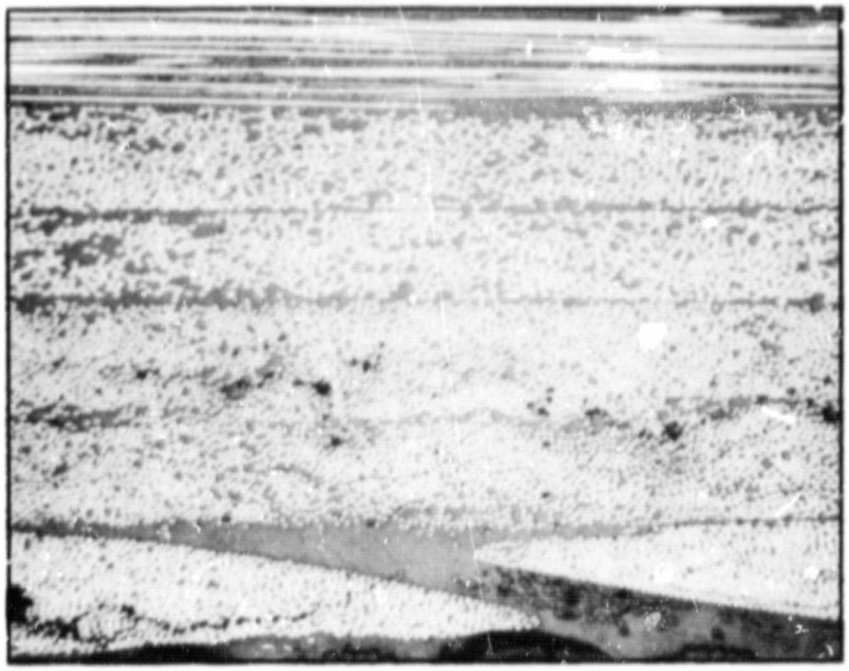
H. LAMINATE NO. 14, POLARIZED LIGHT

R81-0911-059D(4/8)

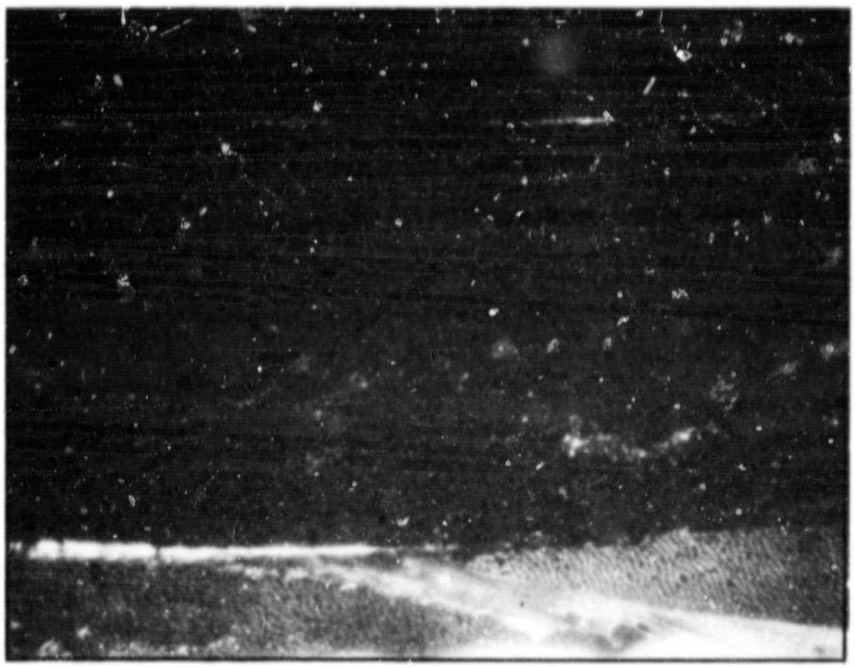
Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 4 of 8)

ORIGINAL PAGE IS
OF POOR QUALITY

I. LAMINATE NO. 15, NORMAL LIGHT



J. LAMINATE NO. 15, POLARIZED LIGHT

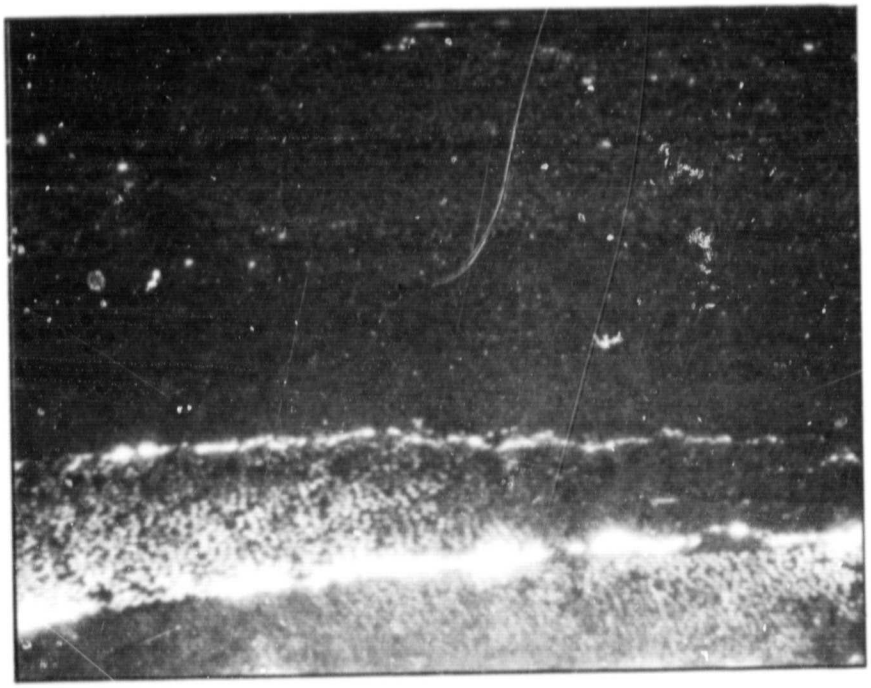


R81-0911-059D(5/8)

Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 5 of 8)

K. LAMINATE NO. 16, NORMAL LIGHT

L. LAMINATE NO. 16, POLARIZED LIGHT

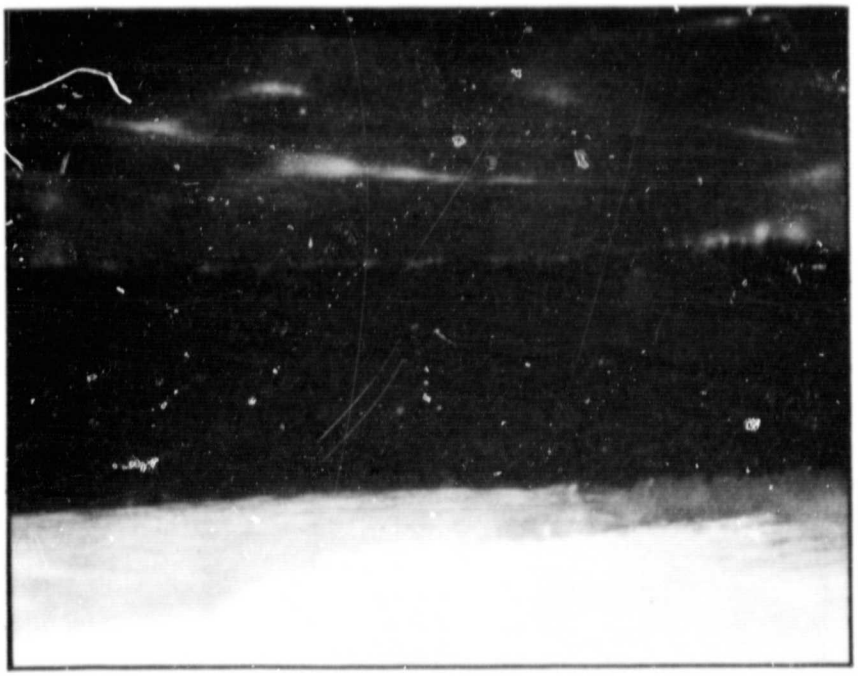


R81-0911-059D(6/8)

Fig. 6 Photomicrographs of Thick Laminates, 100X Mag (Sheet 6 of 8)

M. LAMINATE NO. 17, NORMAL LIGHT

N. LAMINATE NO. 17, POLARIZED LIGHT

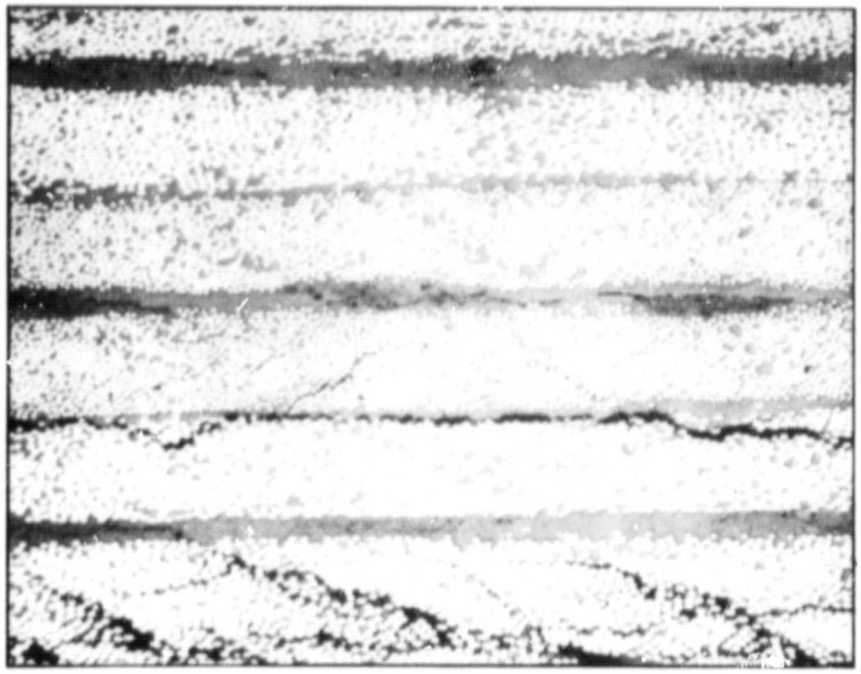


R81-0911-059D(7/8)

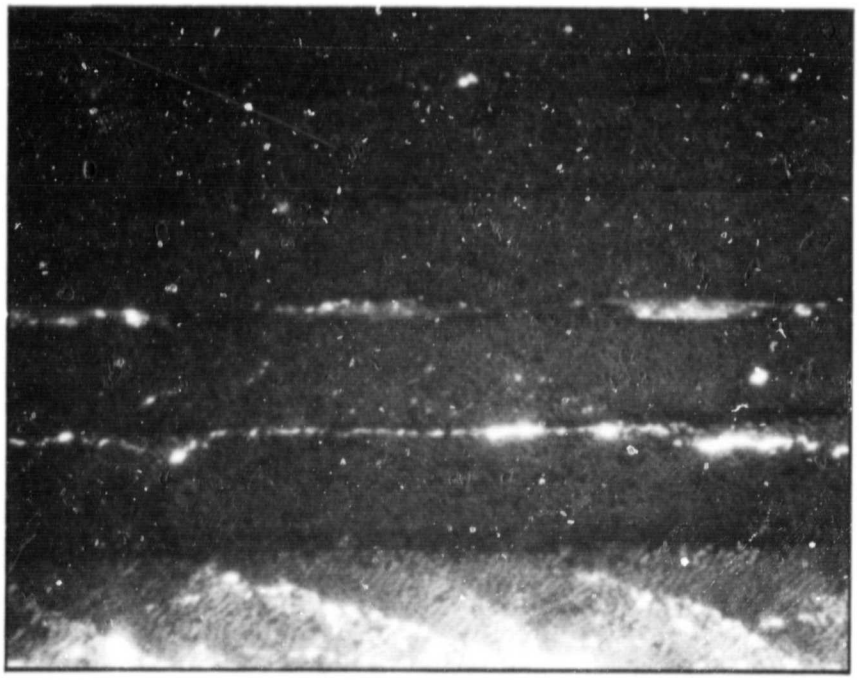
Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 7 of 8)

ORIGINAL PAGE IS
OF POOR QUALITY

O. LAMINATE NO. 18, NORMAL LIGHT



P. LAMINATE NO. 18, POLARIZED LIGHT



R81-0911-059D(8/8)

Fig. 6 Photomicrographs of Thick Laminates, 100x Mag (Sheet 8 of 8)

ORIGINAL PAGE IS
OF POOR QUALITY

APPENDIX C

LIST OF SYMBOLS

E_L = layer average Young's modulus (tension and compression), longitudinal

E_T = layer average Young's modulus (tension and compression), transverse

G_{LT} = layer in-plane shear modulus

ν_{LT} = layer major Poisson's ratio

t' = layer average cured layer thickness

F_L^t = layer average longitudinal tensile strength

F_L^c = layer average longitudinal compression strength

F_T^t = layer average transverse tension strength

F_T^c = layer average transverse compression strength

E_x = laminate average Young's modulus, longitudinal

E_y = laminate average Young's modulus, transverse

G_{xy} = laminate average in-plane shear

ν_{xy} = laminate average major Poisson's ratio

F_x^{tu} = laminate average tensile strength, longitudinal

PAGE 112 INTENTIONALLY BLANK

REFERENCES

1. NASA Technical Memorandum 78761, "Modified Composite Materials Workshop," D. L. Dicus, Compiler, July 1978.

2. Handbook of Fiberglass and Advanced Plastics Composites, G. Lubin, editor, Van Nostrand Reinhold, New York, 1969.

3. NASA SP-5027, "Thermal Insulation Systems," 1967.

4. Ablative Plastics, G. F. D'Alelio and J. A. Parker, editors, Marcel Dekker, Inc, New York, 1971.

5. "Polyimide Resins Chemical and Process Categorization," R. Moulton, Hexcel Corp, 1978, (unpublished paper).

6. Hedrick, J. G. and J. Whiteside, "Effect of Environment on Structural Composites," AIAA Paper 77-463, March 24, 1977.

7. Bell, V., "Carbon Fiber Risk Analysis Conference," NASA-Langley, October 31, 1978.

8. NASA Technical Memorandum 78762, "Preliminary Burn and Impact Tests of Hybrid Polymeric Composites," S. T. Tompkins and W. D. Breyer, July 1978.

9. Larsen, E. R., "DVBR-3, A Small-Scale Test for Evaluating the Surface Flame-Propagating Properties of Polymers," Journal of Fire and Flammability, Volume 9, April 1978.

PAGE 114 INTENTIONALLY BLANK