REPORT NO. GDC-DBG70-005

DEVELOPMENT OF DESIGN DATA FOR GRAPHITE REINFORCED EPOXY AND POLYIMIDE COMPOSITES

FINAL REPORT

W. G. Scheck

May 1974

Prepared Under
Contract NAS8-26198
for
GEORGE C. MARSHALL SPACE FLIGHT CENTER
ASTRONAUTICS LABORATORY
MATERIALS DIVISION
NONMETALLICS BRANCH

Prepared by
GENERAL DYNAMICS CONVAIR AEROSPACE DIVISION
San Diego, California

FOREWORD

This report was prepared by Convair Aerospace Division of General Dynamics, San Diego, California under Contract NAS8-26198, entitled "Development of Design Data for Graphite Reinforced Epoxy and Polyimide Composites," for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. This work was administered under the technical direction of the Nonmetallics Branch, Materials Division, Astronautics Laboratory of the George C. Marshall Space Flight Center, with Dr. James M. Stuckey as the original project manager and Mr. Leldon M. Thompson acting as project manager for the last year of the study.

The contributions of Mr. Jerald Fleming of Fiberite Corporation for the resin characterization work and the assistance of J. Hertz, M. Maximovich, R. May, and M. Varlas of Convair Aerospace are gratefully acknolwedged.

TABLE OF CONTENTS

Section		Page
1	INTRODUCTION	1-1
2	GRAPHITE/EPOXY COMPOSITES	2-1
2.1	GRAPHITE FIBER AND EPOXY RESIN SELECTION	2-1
2.2	COMPOSITE CHARACTERIZATION	2-4
2.2.1	Resin Cure Data	2-7
2.2.2	Panel Fabrication	2-7
2.2.3	Mechanical Property Tests	2-23
2.3	EPOXY RESIN CHARACTERIZATION	2-27
2.3.1	Chemistry of the X-904 Epoxide System	2-28
2.3.2	Polymer Reaction Rates	2-29
2.3.3	Refractive Index Studies, X-904	2-31
2.3.4	Quality Control Procedures	2-31
2.3.5	Specific Gravity of Cast Resin	2-35
2.4	EPOXY/GRAPHITE PREPREG CHARACTERIZATION	2-35
2.4.1	Prepreg Gel Times	2-35
2.4.2	Prepreg Storage Stability Tests	2-39
2.4.3	Prepreg Parameters	2-40
2.4.4	Volatile Content	2-41
2.5	GRAPHITE-EPOXY PROCESS OPTIMIZATION	2-42
2.5.1	Vacuum-Pressure Augmented Cure Study (Press)	2-42
2.5.2	Press Cure Test Results	2-45
2.5.3	Vacuum Bag Cure Study	2-53
2.5.4	Vacuum Bag Test Results	2-55
2.5.5	Autoclave Cure and Postcure Study	2-63
2.5.6	Autoclave Test Data	2-70
2.6	ELEVATED TEMPERATURE STABILITY OF EPOXY-MATRIX COMPOSITES	2-72
3	HIGH-STRENGTH GRAPHITE/POLYIMIDE COMPOSITES	3-1
3.1	GRAPHITE FIBER SELECTION	3-1
3.2	POLYIMIDE RESIN SELECTION	3-1
3.3	GRAPHITE/POLYIMIDE CHARACTERIZATION	3-10
3.3.1	Panel Fabrication	3-11
3.3.2	Mechanical Property Tests	3-13
3.3.3	Graphite/Polyimide Systems	3-13
3.4	POLYIMIDE RESIN CHARACTERIZATION	3-15
3.4.1	Composition of Skybond 710 Polyimide Resin	3-15

TABLE OF CONTENTS, Contd

Section			Page
	3.4.2	Solvents Employed in Skybond 710 Polyimide Varnish	3-16
	3.4.3	Inorganic Components of Skybond 710 Polyimide Varnish	3-17
	3.4.4	Reaction Kinetics for Skybond 710	3-17
	3.4.5	Quality Control Aspects of 710	3-30
	3.4.6	Rheological Aspects of 710	3-31
	3.5	POLYIMIDE/GRAPHITE PREPREG CHARACTERIZATION	3-31
	3.5.1	Prepreg Gel and Flow as a Function of Temperature	3-32
	3.5.2	Staging of HT-S/710 Prepreg	3-32
	3.5.3	HT-S/710 Prepreg Storage Stability	3-37
	3.6	GRAPHITE/POLYIMIDE PROCESS DEVELOPMENT	3-38
	3.6.1	Vacuum-Pressure Augmented Cure Study (Press)	3-38
	3.6.2	Vacuum Bag Cure Study	3-48
	3.6.3	Graphite Fiber Finish Study	3-50
	3.6.4	Material Supplier Evaluation	3-54
	3.6.5	Bleed and Vacuum Process Studies	3-55
	3.6.6	Graphite/Polyimide Solvent Study	3-65
	3.6.7	Graphite/Polyimide Precompaction Study	3-69
	3.6.8	Graphite/Polyimide (HT-S/710) Cure Cycle	3-69
	3.7	GRAPHITE/POLYIMIDE (HT-S/710) DESIGN DATA	3-72
	3.7.1	Static Properties of HT-S/710 Graphite/Polyimide	3-74
	3.7.2	Thick Laminate Fabrication and Testing	3-81
	3.7.3	Graphite/Polyimide Biaxial Testing	3-96
	3.7.4	Creep Testing of HT-S/710 Composites	3-100
	3.7.5	Ambient and High Temperature Aging	3-103
	3.7.6	Thermal Expansion Testing of HT-S/710 Composites	3-103
4	HIGH MODULUS GRAPHITE/POLYIMIDE COMPOSITES		4-1
	4.1	Preliminary Composite Evaluation	4-1
	4.2	HM-S/710 Graphite/Polyimide Design Data	4-10
	4.2.1	Static Mechanical Properties HM-S/710 Graphite/Polyimide	4-12
	4.2.2	Thermal Aging, Moisture Resistance, and Fatigue Characteristics of HM-S/710	4-12
5	HIGH TEMPERATURE COMPOSITE SYSTEMS		5-1
	5.1	HT-S/HT-424 GRAPHITE/EPOXY-PHENOLIC COMPOSITES	5-1

TABLE OF CONTENTS, Contd

Section			Page
	5.2	P105A GRAPHITE/POLYIMIDE EVALUATION STUDY	5-2
6		FABRICATION DEMONSTRATION ARTICLES	6-1
	6.1	CONVAIR AEROSPACE FRAME SEGMENT	6-1
	6.2	CONVAIR AEROSPACE ADAPTER SKIN FRAME SEGMENT	6-4
	6.3	PRELIMINARY MANUFACTURING INSPECTION SPECIFICATION FOR HT-S/710 POLYIMIDE ADAPTER SKIN FRAME SEGMENT	6-6
7		GRAPHITE/POLYIMIDE (HT-S/710) STRINGER TEST ARTICLES	7-1
	7.1	ORIGINAL ANALYSIS - GRAPHITE/POLYIMIDE STRINGER	7-1
	7.2	FABRICATION AND TOOLING DEVELOPMENT	7-6
	7.3	REVISED HAT STRINGER ANALYSIS	7-9
	7.4	GRAPHITE/POLYIMIDE STRINGER TEST RESULTS	7-14
8		BORON/POLYIMIDE COMPOSITE DEVELOPMENT	8-1
	8.1	FABRICATION DEVELOPMENT	8-1
9		CONCLUSIONS AND RECOMMENDATIONS	9-1
10		REFERENCES	10-1
Appendix			
A		GRAPHITE/EPOXY PROCESS DATA HT-S/X-904	A-1
B		HT-S/710 PROCESS TEST DATA	B-1
C		EPOXY RESIN SPECIFICATION	C-1
D		IMPREGNATING POLYIMIDE RESIN SPECIFICATION	D-1
E		GRAPHITE/EPOXY PREPREG SPECIFICATION	E-1
F		GRAPHITE/EPOXY PREPREG SPECIFICATION	F-1
G		GRAPHITE/POLYIMIDE PREPREG SPECIFICATION	G-1

LIST OF ILLUSTRATIONS

Figure		Page
2-1	Thermogravimetric and Differential Thermal Analysis of MODMOR II/1004	2-9
2-2	Thermogravimetric and Differential Thermal Analysis of HT-S/BXP-2401	2-10
2-3	Thermogravimetric and Differential Analysis of hy-E-1311-B	2-11
2-4	Thermogravimetric and Differential Thermal Analysis of Celanese GRT-70-350A	2-12
2-5	Thermogravimetric Analysis of Hercules HT-S/3002	2-13
2-6	Time/Temperature/Viscosity Relationships of the Fiberite X-904 Resin System	2-14
2-7	Time/Temperature/Viscosity Relationships of the Celanese R-350A Resin System	2-15
2-8	Time/Temperature/Viscosity Relationship of the Whittaker 1004 Resin System	2-16
2-9	Typical Graphite/Epoxy Cure Layup	2-16
2-10	Cutting Diagram	2-24
2-11	Isothermal Aging Data at 450°K (350°F) for HT-S/X-904 Tested at 450°K (350°F)	2-27
2-12	Elevated Temperature Test Data for HT-S/X-904	2-29
2-13	Isothermal Reaction Rate Curves for X-904 Resin System	2-30
2-14	X-904 Resin Gel Time Versus Reaction Temperature	2-32
2-15	Time/Viscosity Refractive Index Relationships for the X-904 Resin System at 353°K (176°F) at 100% Resin Solids	2-33
2-16	Refractive Indices of X-904 Resin Solutions (MEK Solvent)	2-34
2-17	Storage Stability of X-904 Master Batches at 85% Resin Solids (No BDMA)	2-36
2-18	Stability at 298°K (78°F) of X-904 Premix at 85% Resin Solids (with BDMA Catalyst Added)	2-37

LIST OF ILLUSTRATIONS, Contd

Figure		Page
2-19	X-904/HT-S Prepreg Gel Time at Several Temperatures	2-38
2-20	Panel Description for Cure and Postcure Studies (HT-S/X-904)	2-42
2-21	Dielectric Monitor Cure Curve for HT-S/X-904	2-71
2-22	Longitudinal Flexural Strength Versus Temperature for HT-S/X-904	2-75
2-23	Longitudinal Flexural Strength at 450°K (350°F) for HT-S/X-904	2-77
3-1	Effect of Void Content on Thermal Degradation of PI Glass Laminate at 589°K (600°F)	3-2
3-2	Typical Graphite/Polyimide Cure Layup	3-11
3-3	Skybond 710 Component Cure	3-18
3-4	Conversion Rate of 710 Polyamic Acid at 373°K (212°F)	3-22
3-5	Conversion Rate of 710 Polyamic Acid at 398°K (257°F)	3-22
3-6	Conversion Rate of 710 Polyamic Acid at 436°K (325°F)	3-23
3-7	Conversion Rates of 710 Polyamic Acid at Several Temperatures	3-23
3-8	Activation Energy for Conversion of 710 Polyamic Acid to Polyimide	3-24
3-9	Titration of Skybond 710 in DMF With 0.1000N KOH (Alcoholic)	3-25
3-10	Some Possible Species in Skybond 710 of the Anhydride Functional Group	3-27
3-11	Hydrolysis of the Amic Ester	3-28
3-12	Hydrolysis of the Amic Acid	3-29
3-13	Rheological Aspects of 710 Polyimide Resin	3-33
3-14	Gel Time and Resin Flow of HT-S/710 Prepreg at Several Temperatures (Prepreg Resin Solids 42.9%, Volatile Content 20.8%)	3-34
3-15	HT-S/710 Prepreg Volatile Loss at Several Staging Temperatures (Prepreg Resin Solids 45.5%, Volatile Content 19.8%)	3-34

LIST OF ILLUSTRATIONS, Contd

Figure		Page
3-16	HT-S/710 Prepreg Volatile Content Versus Staging Time at Several Temperatures (Prepreg Resin Solids 46.2%, Volatile Content 21.5%)	3-35
3-17	HT-S/710 Prepreg Softening and Flow Point and Volatile Content as a Function of Staging at 353°K (176° F)	3-35
3-18	Panel Description for Cure and Postcure Studies (HT-S/710)	3-38
3-19	Graphite/Polyimide Process Optimization Flow Chart	3-39
3-20	Schematic of Graphite/Polyimide Cure Layup	3-72
3-21	Graphite/Polyimide HT-S/710 Design Data Laminate	3-75
3-22	Test Specimen for Longitudinal Tension, Creep, and Fatigue Testing	3-78
3-23	Transverse Tensile Test Specimen	3-79
3-24	Longitudinal and Transverse Compression Specimen	3-79
3-25	Longitudinal Flexure (0°) Specimen	3-80
3-26	Transverse Flexure (90°) Specimen	3-80
3-27	Short Beam Shear Test Specimen	3-81
3-28	Tensile Strength (HT-S/710) as a Function of Temperature and Laminate Orientation	3-82
3-29	Compression Strength (HT-S/710) as a Function of Temperature and Laminate Orientation	3-83
3-30	Tensile Modulus of HT-S/710 as a Function of Temperature and Laminate Orientation	3-84
3-31	Compression Modulus of HT-S/710 as a Function of Temperature and Laminate Orientation	3-85
3-32	Tensile Strain to Failure (HT-S/710) as a Function of Temperature and Laminate Orientation	3-86
3-33	Compression Strain to Failure (HT-S/710) as a Function of Temperature and Laminate Orientation	3-87
3-34	HT-S/710 Graphite Polyimide Specimen - 2.54 cm (1.00 in.) Laminate	3-95

Figures		Page
3-35	HT-S/710 Graphite/Polyimide - Thick Laminate Test Specimen	3-95
3-36	HT-S/710 Graphite/Polyimide - Thick Laminate Data	3-96
3-37	Biaxial Tension Specimen	3-97
3-38	Biaxial Test Results of (± 45°) HT S/710 Composite Tubes	3-98
3-39	Tested Biaxial Graphite/Polyimide HT-S/710 Tubes	3-99
3-40	Modified Leitz Dilatometer	3-106
3-41	Details of Dilatometer Mechanism	3-106
3-42	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen	3-109
3-43	Total Linear Thermal Expansion of Unidirectional Layup, 0° Specimen	3-111
3-44	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen	3-112
3-45	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen	3-113
3-46	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen	3-114
3-47	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen	3-115
3-48	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen	3-116
3-49	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen	3-117
3-50	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen	3-118
3-51	Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen	3-119
6-1	Graphite/Polyimide Frame Segment Tooling	6-2
6-2	Web Section Patterns	6-3
6-3	Layup Sequence	6-3

LIST OF ILLUSTRATIONS, Contd

Figure		Page
6-4	Graphite/Polyimide (RS-6234) Frame Segment and Adapter Skin Frame Segment	6-4
6-5	Skin Segment Tools	6-4
6-6	Tooling and Manufacturing Feasibility Specimen (All Dimensions in Inches Only for Clarification)	6-5
6-7	Layup Sequence	6-7
7-1	HT-S/710 Graphite/Polyimide Stringer Test Component	7-1
7-2	Graphite/Polyimide (HT-S/710) Stringer and Tooling	7-6
7-3	HT-S/710 Graphite/Polyimide Stringer Section Data	7-9
7-4	Revised HT-S/710 Graphite/Polyimide Stringer Test Component	7-14
7-5	Strain Gage Locations for Graphite/Polyimide Test Stringers	7-15

LIST OF TABLES

Table		Page
2-1	Typical Strength Properties and Relative Merits of Graphite Filaments	2-2
2-2	Comparison of Composites Made With Domestic Graphite Fibers	2-3
2-3	Comparisons of Composites Made With English High-Strength Graphite Fibers	2-3
2-4	Comparison of Composites Made With High-Modulus Graphite Fibers	2-5
2-5	Comparison of 450°K (450°F) Resin Systems With Graphite Fibers	2-5
2-6	Evaluation of High-Modulus Graphite-Epoxy Systems	2-6
2-7	Evaluation of High-Strength Graphite-Epoxy Systems	2-6
2-8	Materials Flow Chart	2-7
2-9	Graphite/Epoxy Prepreg Test Results	2-8
2-10	Evaluation of High-Strength Graphite/Epoxy Systems	2-25
2-11	Evaluation of High Modulus Graphite/Epoxy System	2-26
2-12	Elevated Temperature Test Data for HT-S/X-904	2-29
2-13	Prepreg Stability Study of HT-S/X-904 Prepreg at 297°K (75°F)	2-39
2-14	HT-S/X-904 Prepreg Stability Study - Storage at 278°K (40°F)	2-40
2-15	HT-S/X-904 Prepreg Stability Study - Storage at 255°K (0°F)	2-41
2-16	HT-S/X-904 Cure Study - Press Cure No. 1	2-46
2-17	HT-S/X-904 Cure Study - Press Cure No. 2	2-47
2-18	HT-S/X-904 Cure Study - Press Cure No. 3	2-48
2-19	HT-S/X-904 Cure Study - Postcure Cure No. 1	2-49
2-20	HT-S/X-904 Cure Study - Postcure Cure No. 2	2-50
2-21	HT-S/X-904 Cure Study - Postcure Cure No. 3	2-51
2-22	HT-S/X-904 Cure Study - Postcure Cure No. 4	2-52

LIST OF TABLES, Contd

Table		Page
2-23	HT-S/X-904 Cure Study - Vacuum Bag Cure No. 1	2-56
2-24	HT-S/X-904 Cure Study - Vacuum Bag Cure No. 2	2-57
2-25	HT-S/X-904 Cure Study - Vacuum Bag Cure No. 3	2-58
2-26	HT-S/X-904 Cure Study - Postcure No. 1	2-59
2-27	HT-S/X-904 Cure Study - Postcure No. 2	2-60
2-28	HT-S/X-904 Cure Study - Postcure No. 3	2-61
2-29	HT-S/X-904 Cure Study - Postcure No. 4	2-62
2-30	Autoclave Cure Cycle Test Results (HT-S/X-904)	2-64
2-31	Modified Autoclave Cure Cycle Test Results (HT-S/X-904)	2-65
2-32	Longitudinal Flexure Properties (Panels 14390 and 14391)	2-73
2-33	Longitudinal Flexural Strength of HT-S/X-904, Panel OC-16-1-15	2-74
2-34	Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing X-904 Resin	2-76
2-35	Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing 3002 Resin	2-76
2-36	Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing 1004 Resin	2-76
2-37	Horizontal Shear Strength at 450°K (350°F)	2-78
2-38	Longitudinal Flexural Strength at Temperature as a Function of RT Aging	2-78
2-39	Effects of Post Cure Treatments on 450°K (350°F) Longitudinal Flexural Strength	2-78
3-1	Comparison of Hercules HT-S/P13N Composites Made With Various Laminating Pressures	3-3
3-2	Comparison of Test Panel Results on Quartz /703 Polyimide as a Function of Cure Technique (Room Temperature Data)	3-4

LIST OF TABLES, Contd

Table		Page
3-3	Properties of Boron/X222-84 Polyimide Composite	3-5
3-4	Aging Studies on Glass-Reinforced Skybond 709 Polyimide	3-6
3-5	Summary of Preliminary Graphite/Polyimide-Composite Results	3-7
3-6	562°K (550°F) Tensile Results for 450°K (350°F) Cured Graphite/Polyimide Laminates	3-8
3-7	Initial 589°K (600°F) Tensile Results for High-Fiber-Volume Graphite Polyimide Laminates	3-8
3-8	Preliminary Tensile-Properties of Convair Aerospace-Developed, Graphite/Polyimide Laminates	3-9
3-9	Materials Flow Chart	3-10
3-10	Graphite/Polyimide Prepreg Test Results	3-10
3-11	Evaluation of High-Strength Graphite Polyimide/Systems	3-14
3-12	Evaluation of High Modulus Graphite/Polyimide Systems	3-14
3-13	Reduced Pressure Distillation of Skybond 710	3-17
3-14	Reaction Rate Constants for 710 Polyimide Varnish	3-21
3-15	Reaction Rate Constants for Conventional Polyamic Acids	3-24
3-16	Comparison of Two Different Lots of Skybond 710	3-26
3-17	710, Prepreg Characteristics as a Function of Staging Conditions	3-32
3-18	HT-S/710 Prepreg Storage Stability at 298°K (78°F) Room Temperature	3-36
3-19	HT-S/710 Prepreg Storage Stability at 278°K (40°F)	3-36
3-20	HT-S/710 Prepreg Storage Stability at 255°K (0°F)	3-37
3-21	Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 1	3-44
3-22	Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 2	3-44
3-23	Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 3	3-45

LIST OF TABLES, Contd

Table		Page
3-24	Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 4	3-45
3-25	Postcure Study of Press Cure HT-S/710 Graphite/ Polyimide Press Cure Cycle No. 1	3-46
3-26	Postcure Study of Press Cured HT-S/710 Graphite/ Polyimide. Press Cure Cycle No. 2	3-46
3-27	Postcure Study of Press Cured HT-S/710 Graphite/ Polyimide. Press Cure Cycle No. 3	3-47
3-28	Postcure Study of Press Cured HT-S/710 Graphite/ Polyimide. Press Cure Cycle No. 4	3-47
3-29	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Postcure Cycle No. 1	3-51
3-30	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Postcure Cycle No. 2	3-51
3-31	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Postcure Cycle No. 3	3-52
3-32	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Postcure Cycle No. 4	3-52
3-33	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Cure Cycle No. 1	3-53
3-34	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide. Cure Cycle No. 2	3-53
3-35	Press Cure Study of HT-S/710 Graphite/Polyimide With a P13N Finish. Cure Cycle No. 3	3-54
3-36	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide With a P13N Finish, Cure Cycle No. 1	3-55
3-37	Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide With a P13N Finish. Cure Cycle No. 2	3-55
3-38	Prepreg Properties, HT-S/710	3-58
3-39	Vacuum-Bag Cure Study of HT-S/710 Composites	3-59
3-40	Press-Cure Study of HT-S/710 Composites	3-59
3-41	Press-Cure Study of HT-S/710 Composites	3-60

LIST OF TABLES,Contd

Table		Page
3-42	Press-Cure Study of HT-S/710 Composites	3-60
3-43	Autoclave-Cure Study of HT-S/710 Composites	3-61
3-44	Autoclave-Cure Study of HT-S/710 Composites	3-61
3-45	Bleeder Study of HT-S/710 Graphite/Polyimide	3-63
3-46	Vacuum Study of HT-S/710 Graphite/Polyimide	3-64
3-47	Graphite/Polyimide HT-S/710 Prepreg Properties (Solvent Study)	3-66
3-48	Mechanical Properties of HT-S/710 Graphite Composites. Unit I - Solvent Study (Ethanol); Autoclave Cure	3-66
3-49	Mechanical Properties of HT-S/710 Graphite Composites. Unit II - Solvent Study (NMP); Vacuum Bag Cure	3-67
3-50	Mechanical Properties of HT-S/710 Graphite Composites. Unit II - Solvent Study (NMP); Autoclate Cure	3-67
3-51	Mechanical Properties of HT-S/710 Graphite Composites. Unit III - Solvent Study (NMP - Xylene 2 to 1)	3-68
3-52	Mechanical Properties of HT-S/710 Graphite Composites. Unit IV - Solvent Study (NMP - Xylene 1 to 1)	3-68
3-53	Precompaction Study, HT-S/ 710	3-70
3-54	Flexural Strength of Graphite/Polyimide (HT-S/710) Composite System as a Function of Time at 589°K (600°F) Prior to Testing at 589°K (600°F)	3-73
3-55	Flexural Strength of Graphite/Polyimide (HT-S/710) Composite System as a Function of Temperature	3-73
3-56	Tensile Strength of HT-S/710 Graphite/Polyimide as a Function of Temperature	3-74
3-57	Design Data Tests for HT-S/710 Graphite/Polyimide	3-75
3-58	Graphite/Polyimide (HT-S/710) Design Data Laminates - Physical Properties	3-76
3-59	Design Properties of HT-S/710 Composites - 0°	3-88
3-60	Design Properties of HT-S/710 Composites - 45°	3-89
3-61	Design Properties of HT-S/710 Composites - 90°	3-90

LIST OF TABLES, Contd

Table		Page
3-62	Design Properties of HT-S/710 Composites - (± 60°)	3-91
3-63	Design Properties of HT-S/710 Composites - (± 45°)	3-92
3-64	Design Properties of HT-S/710 Composites - (± 45°)	3-93
3-65	Design Properties of HT-S/710 Composites - (0, ± 45°, 90°)	3-94
3-66	297°K (75° F) Thick Laminate Compression Test Results of HT-S/710 Graphite/Polyimide Composites	3-97
3-67	Biaxial Tube Testing of (± 45) HT-S/710 Graphite/ Polyimide Composites at 297° K (75° F)	3-98
3-68	Theoretical Principal Stress and Strain Test Results of Biaxial Tube Testing of (2 45°) HT-S/710 Composites	3-100
3-69	Stress Rupture and Residual Tensile Strength of Unidirectional HT-S/710 Composites	3-101
3-70	Stress Rupture and Residual Tensile Strength of $(\pm 45°)_S$ HT-S/710 Composites	3-102
3-71	Average Creep Test Results of (± 45°) HT-S/710 Composites	3-102
3-72	Graphite Composite Aging Data (HT-S/710)	3-104
3-73	Graphite/Polyimide 589°K (600° F) Heat-Aging Test Results	3-105
4-1	HM-S/710 Graphite/Polyimide Composite Data	4-2
4-2	Modmor I/710 Graphite/Polyimide CompositeData	4-3
4-3	GY-70/710 Graphite/Polyimide Composite Data	4-4
4-4	HM-S/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing	4-6
4-5	Modmor I/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing	4-7
4-6	GY-70/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing	4-8
4-7	GY-70-HM-S/710 Graphite/Polyimide Composite Data	4-9
4-8	Preliminary Design Data HM-S/710 Graphite/Polyimide Composites	4-10

LIST OF TABLES, Contd

Table		Page
4-9	Preliminary Design Data GY-70/710 Graphite/ Polyimide Composite	4-11
4-10	Design Properties of HM-S/710 Graphite/Polyimide Composites - 0°	4-13
4-11	Design Properties of HM-S/710 Graphite/Polyimide Composites - 90°	4-14
4-12	Design Properties of HM-S/710 Graphite/Polyimide Composites - (± 45°)	4-15
4-13	Physical Properties of HM-S/710 Design Data Laminates	4-16
4-14	Environmental Aging Data HM-S/710 Graphite/Polyimide Composites	4-17
4-15	Room Temperature Fatigue Testing R = 0.1 of 0° HM-S/710 Graphite/Polyimide Composites	4-18
4-16	Room Temperature Fatigue Testing R = 0.1 of (± 45°) HM-S/710 Graphite/Polyimide Composites	4-19
5-1	Mechanical Properties of HT-S/HT-424 Graphite Composites (Vacuum Bag Cure)	5-3
5-2	Mechanical Properties of HT-S/HT-424 Graphite Composite (Press Cure)	5-4
5-3	Mechanical Properties of HT-S/HT-424 Graphite Composites (Autoclave Cure)	5-5
5-4	Graphite Composite Aging Data, HT-S/HT-424	5-6
5-5	Preliminary Design Data, HT-S/P105A Graphite/ Polyimide Composite	5-7
5-6	Preliminary Design Data, HM-S/P105A Graphite/ Polyimide Composite	5-8
7-1	Precompaction and Cure Layups for HT-S/710 Graphite/ Polyimide Stringer	7-8
7-2	Lap Shear Strengths of Graphite/Polyimide (HT-S/710) Adhesively Bonded With HT-424	7-8
7-3	Test Results of Graphite/Polyimide (HT-S/710) Skin Stringer Test Components	7-15

LIST OF TABLES, Contd

Table		Page
8-1	Boron/Polyimide (B/703) Fiber Evaluation Study	8-1
8-2	Cure Pressure and Bleeder Evaluation of B/703 Polyimide Composites (Room Temperature)	8-3
8-3	Cure Pressure and Bleeder Evaluation of B/P105A Polyimide Composites (Room Temperature)	8-4
8-4	Mechanical Properties for Boron/Polyimide B/P105A Composites	8-5

SUMMARY

The primary objective of this program was to characterize processing techniques and design data for a graphite/epoxy composite system that is useful from 75°K (-320°F) to 450°K (350°F) and a graphite/polyimide composite system that is useful from 75°K (-320°F) to 589°K (600°F). With the identification of the graphite/epoxy high temperature strength problem caused by moisture this program was reconstructed to concentrate on the development of polyimide composite systems.

The Monsanto 710 (RS 6234) polyimide resin was selected as the resin to be characterized and used with the graphite fiber reinforcement. The basic resin was fully characterized and a resin specification was written and has been used in purchasing resin and prepreg materials. Detailed processing techniques were developed for the HT-S/710 graphite/polyimide system based on the resin characterization work and a prepreg specification was developed.

Material was purchased using the prepreg specification for the design data generation for both the HT-S/710 and HM-S/710 graphite/polyimide composite systems. Lamina and laminate properties were determined at 75°K, 297°K, and 589°K (-320°F, 75°F, and 600°F). Processing techniques that were developed on this program were proven to be completely applicable to fabricating large complicated parts by the fabrication of several demonstration articles. Laminates up to 5.08 cm (2.0 inches) thick and test skin-stringer components were fabricated. The successful test results obtained on the skin-stringer components proved that graphite/polyimide composites can be reliably designed and analyzed much like graphite/epoxy composites.

The design data generated on this program includes the standard static mechanical properties, biaxial strain data, creep, fatigue, aging, and thick laminate data. All of the design data test specimens were taken from laminates that were at least 61 cm by 61 cm (2 feet by 2 feet) in size. This was done in order that realistic design properties would be generated that would be typical of the strength and stiffness properties expected in real complicated parts.

SECTION 1

INTRODUCTION

The initiation of the Space Shuttle program by NASA in 1969, coupled with the potential use of advanced composite systems for structural applications, required the development of design data applicable to reusable space launch vehicles and spacecraft. Previous programs conducted by other government agencies and industry for the development of design data were aimed at aircraft applications. The reusability criteria and immense size required that new epoxy and polyimide resin systems be evaluated for use as matrices for advanced composite systems.

The overall program objective was to develop processing techniques and develop design data for both epoxy and polyimide reinforced composites. In order to meet this primary goal, the following guidelines were used in conducting this program.

a. Develop commercially available graphite/epoxy, graphite/polyimide, and boron/polyimide prepregs that were not proprietary.

b. Develop processing techniques applicable to making large structural components by normal manufacturing techniques.

c. Develop preliminary design data for high-strength graphite/epoxy and graphite/polyimide composite systems and for high modulus graphite/polyimide composite systems.

d. Develop preliminary processing techniques and a prepreg with tack and drape for use in making complex boron/polyimide composite parts.

e. All composite systems would have to withstand at least 400 hours at 450°K (350°F) or 589°K (600°F) with less than 50 percent loss of initial strength at either room or elevated temperatures.

f. Fabricate and test selected graphite/polyimide structural components using the processing techniques developed for flat laminates.

g. Develop detailed material and process specifications for the graphite composite systems developed on this program.

The following approach was taken to meet these technical objections. Based on Convair's advanced composite experience and the available literature, several resin systems, graphite fibers, and boron fibers were selected for initial characterization. After this characterization one high-strength graphite/epoxy, one high-strength and

one high-modulus graphite/polyimide, and one boron/polyimide system were selected for detailed development of processing techniques, resin characterization, and part fabrication. Processing was optimized, design property laminates were fabricated, and a detailed test program was conducted. Also, fabrication demonstration items were manufactured to demonstrate that the processing techniques developed were applicable to complex parts. Structural sheet-stringer specimens were designed, fabricated, and tested out of the selected high strength graphite/polyimide system.

Although the English system of units (ft, lb, sec) has been used for all measurements and calculations, in this report the SI system of units is shown as the primary system, with English units following in parentheses.

This report has been organized so that summary technical data is presented in the main body of the report and the detailed test data and specifications are presented as separate appendixes. This report is divided into the following major subsections:

1. Graphite/Epoxy Composites.

2. High-Strength Graphite/Polyimide Composites.

3. High-Modulus Graphite/Polyimide Composites.

4. New Polyimide Resins.

5. Boron/Polyimide Composites.

6. Graphite/Polyimide Demonstration Parts.

7. Graphite/Polyimide Structural Element Testing.

8. Appendixes

It was during the process development phase of the program for the HT-S/X-904 system that the effects of moisture on epoxy resins was discovered. The program at that point was redirected to concentrate on polyimide systems. A separate program, sponsored by NASA-MSFC Contract NAS8-27435 "Investigation Into the High-Temperature Strength Degradation of Fiber Reinforced Resin Composite During Ambient Aging," investigated this problem in depth.

Table 2-1. Typical Strength Properties and Relative Merits of Graphite Filaments

Properties	Graphite Fibers							
	HMG-50	Thornel 50-S	Morganite Type I	Morganite Type II	Hercules HM-S	Hercules HT-S	Celanese Fiber	Thornel 75
Modulus GN/m^2 (10^6 psi)	346 (50)	346 (50)	416 (60)	276 (40)	346 (50)	220 (32)	521 (75)	521 (75)
Specific Modulus 10^6 m (10^6 in.)	20.8 (819)	20.6 (814)	21.2 (833)	16.1 (635)	18.3 (721)	12.8 (504)	26.1 (1027)	29.2 (1150)
Tensile Strength MN/m^2 (10^3 psi)	1979 (287)	1931 (280)	1724 (250)	2413 (350)	1724 (250)	2069 (300)	2069 (300)	2413 (350)
Specific Tensile 10^5 m (10^6 in.)	1.20 (4.70)	1.22 (4.75)	0.88 (3.47)	1.41 (5.55)	0.88 (3.63)	1.20 (4.70)	1.04 (4.11)	1.25 (4.93)
Density gm/cc ($lb/in.^3$)	1.70 (0.061)	1.63 (0.059)	1.94 (0.072)	1.75 (0.063)	1.90 (0.069)	1.76 (0.063)	1.95 (0.070)	1.86 (0.067)
Cost ($/lb) Dry Filament 1970 Prices	300	300	380	380	250	250	325	450
Relative Merits	Continuous Specific Moduli & Strength	Continuous Specific Moduli & Strength	Fiber Surface Specific Modulus	Fiber Surface Specific Strength	Continuous Cost	Continuous Cost Specific Strength	Continuous High Modulus High Strength	Continuous High Modulus High Strength

SECTION 2

GRAPHITE/EPOXY COMPOSITES

In the selection of the graphite fibers and epoxy resin systems to be evaluated for potential characterization and development of detailed design data, two important criteria were applied to any candidate system. These criteria were that both the graphite fiber and resin were commercially available from multiple sources and that the resin system could be vacuum-bag cured, press cured, and autoclave cured. Using these guidelines, the four graphite/epoxy composite systems were selected.

2.1 GRAPHITE FIBER AND EPOXY RESIN SELECTION

An initial requirement of the characterization portion of the program was to evaluate a high-strength fiber 2413 MN/m^2 (~350,000 psi) and a high-modulus fiber 416 GN/m^2 (~60,000,000 psi). Throughout the remainder of this report, the terms high-strength fiber and high-modulus fiber will be used per these definitions.

There are two distinct types of fibers: high-strength fiber where modulus is a secondary consideration, and high-modulus fiber where strength is a secondary consideration. Table 2-1 shows the fibers that were considered. HMG-60 (an uprated version of HMG-50) was still in the experimental stage and was considered only as a possible back-up for the high-modulus fiber selected.

An extensive survey was conducted of all available data on graphite/epoxy composites made with the fibers listed in Table 2-1. Prime emphasis was placed on data generated in two major AFML programs (References 2-1 and 2-2), and that generated in Convair IRAD programs (References 2-3 and 2-4). Vendor data was reviewed but was used generally as a check rather than an influence on final material selection.

Tables 2-2 and 2-3 summarize the pertinent data, available at the time of fiber selection (1970), on high-strength systems made with both domestic and English fibers. It is obvious on comparison of these two tables that the English fibers gave consistently higher unidirectional tensile strength when tested in a composite. The English fibers are wetted better by epoxy resins as demonstrated by higher transverse tensile and horizontal shear data. The two English fibers, Courtaulds HT-S and Morganite II, are both made from a polyacrylonitrile (PAN) precursor, and both give similar results when evaluated as reinforcements in graphite-epoxy composites. Convair chose to use the HT-S and Morganite II fibers as interchangeable under the general heading of Type II fiber, and selected the Type II fiber as the high-strength reinforcement.

Table 2-2. Comparison of Composites Made With Domestic Graphite Fibers**

Fiber	Epoxy Resin	0° Tension σ, MN/m^2 (ksi)	0° Tension E, GN/m^2 (msi)	90° Tension σ, MN/m^2 (ksi)	90° Tension E, GN/m^2 (msi)	0° Flexure σ, MN/m^2 (ksi)	Horizontal Shear, MN/m^2 (ksi)	Reference 2-
Thornel 50*	Narmco 2387	910 (132)	166 (24.0)	47 (6.8)	11 (1.6)	-	-	1
HMG-50*	E-787	647 (93)	134 (19.4)	42 (6.1)	9.0 (1.3)	-	-	1
Thornel-50S*	Narmco 2387	924 (134)	215 (31.2)	-	-	-	-	1
HMG-50	4617	855 (124)	208 (30.1)	17 (2.5)	7.6 (1.1)	690 (100)	36 (5.2)	2
HMG-50	E-715	745 (108)	172 (24.9)	12 (1.7)	5.6 (0.8)	-	35 (5.1)	3
HMG-50	BP-907	924 (134)	196 (28.5)	-	-	-	46 (6.6)	2
Thornel 50	ERL-2256	703 (102)	159 (23.0)	18 (2.6)	5.6 (0.8)	-	21 (3.0)	6
Thornel 50	E-798	814 (118)	155 (22.5)	6 (0.9)	4.9 (0.7)	-	-	2
HMG-50	X-05	896 (130)	158 (22.9)	-	-	-	-	2

*Sandwich data.

**Reference 2-5.

Table 2-3. Comparison of Composites Made With English High-Strength Graphite Fibers***

Fiber	Epoxy Resin	0° Tension σ, MN/m^2 (ksi)	0° Tension E, GN/m^2 (msi)	90° Tension σ, MN/m^2 (ksi)	90° Tension E, GN/m^2 (msi)	0° Flexure σ, MN/m^2 (ksi)	90° Flexure σ, MN/m^2 (ksi)	Horizontal Shear MN/m^2 (ksi)	Ref. 2-
HTS	Fiberite X-904	1248 (181)	175 (25.5)	21 (3.0)	7.6 (1.1)	-	-	-	4
Morganite II	Fiberite X-904	1193 (173)	141 (20.5)	21 (3.0)	7.6 (1.1)	-	-	-	4
Morganite II*	Narmco 2387	1269 (184)	147 (21.3)	-	-	-	-	-	1
Morganite II*	F&H 4617	1724 (250)	169 (24.5)	93.1 (13.5)	23 (3.4)	-	-	-	1
Morganite II	BXP 2401	682 (98)	132 (19.1)	60 (8.6)	9.0 (1.3)	1083 (157)	-	99.3 (14.4)	2
Morganite II	Narmco 1004	-	-	-	-	1862 (270)	-	113 (16.5)	2
Morganite II	Narmco 2387	-	-	-	-	1014 (147)	128 (18.7)	113 (16.5)	2
Morganite II	Narmco 1004	1531 (222)	-	33 (4.7)	10 (1.5)	-	-	-	4
Morganite II	Fiberite X-05	1393 (202)	160 (23.2)	-	-	-	-	-	2
HTS	Fiberite X-05	972 (141)	124 (18.0)	-	-	-	-	-	2
HTS	Fiberite X-903	-	-	-	-	1441 (209)	-	96.5 (14.0)	2
HTS	Fiberite X-904	786 (114)	140 (20.3)	44 (6.3)	7.6 (1.1)	1041 (151	-	87.6 (12.7)	2
Type II*	F&H 4617	-	-	-	-	1800 (261)	117 (17.0)	105 (15.3)	1
Type II*	Fiberite 4617	-	-	-	-	1469 (213)	75.2 (10.9)	94.5 (13.7)	1
Type II*	Ciba 95	-	-	-	-	1351 (196)	107 (15.5)	111 (16.1)	1

*Sandwich beam data.

** Type II indicates that HTS and Morganite II are used interchangeably.

***Reference 2-5.

Table 2-4 is a summary of the most recent and pertinent data on high-modulus systems available at the time of fiber selection. The GY-70/epoxy composite has a significantly higher modulus than systems made with the high-modulus English fibers, Courtaulds HM-S and Morganite I. Table 2-4 shows that the GY-70/epoxy composite is also considerably better from a modulus standpont that the domestic fiber systems. No data was included on Thornel 75 systems since at the time of the survey none was available, and extensive efforts to obtain the Thronel 75 fiber for evaluation were fruitless.

In general, the strength of a Celanese-epoxy system is comparable to that obtained with composites made of Morganite I, HM-S, Thornel 50-S, and HMG-50. However, because the composite modulus of GY-70/epoxy systems is so much higher than that of all other graphite-epoxy systems, the total strain capability is significantly reduced. Convair selected the Celanese GY-70 fiber for its high-modulus system because modulus is the prime consideration, and total strength and total strain are of secondary importance.

The criteria used in selecting the epoxy resin systems for each of the two types of fibers were:

a. Useful temperature range, 21 to 450°K (-423 to 350°F).

b. All three types of curing techniques, vacuum bag, press, and autoclave, must be applicable for selected resins.

c. Resin prepregs must be commercially available.

A cursory literature survey was conducted to determine the available 450°K (350°F) epoxy resin systems. The results of this survey are shown in Table 2-5. Also, concurrent work at Convair (Reference 2-5) gave further insight into the characteristics of several 450°K (350°F) epoxy resin systems. The data available on these systems are shown in Tables 2-6 and 2-7. Based on the literature survey and the concurrent work being conducted at Convair Aerospace, the Fiberite X-904 and Hercules 3002 epoxy resin systems were selected for initial characterization with the two types of graphite fibers. The X-904 resin system was selected based on its superior cryogenic properties and the 3002 on its superior high-temperature properties.

2.2 COMPOSITE CHARACTERIZATION

One pound of each of the four graphite/epoxy prepregs was ordered during July 1970 and received in August. Table 2-8 summarizes the material designations, suppliers, batch numbers, etc. for the various prepregs. Table 2-9 summarizes the prepreg testing in support of the resin-evaluation program. These results are included for the purpose of comparing the mechanical properties obtained on the various laminates reported later in the body of this report. In general, the graphite/epoxy prepreg/

Table 2-4. Comparison of Composites Made with High-Modulus Graphite Fibers***

Fiber	Epoxy Resin	0° Tension σ, MN/m² (ksi)	0° Tension E, GN/m² (msi)	90° Tension σ, MN/m² (ksi)	90° Tension E, GN/m² (msi)	0° Flexure σ, MN/m² (ksi)	90° Flexure E, GN/m² (ksi)	Horizontal Shear, MN/m² (ksi)	Reference 2-
Celanese	Epi-Rez 508	834 (121)	327 (47.5)	35 (5.0)	6.9 (1.0)	855 (124)	46 (6.7)	68 (9.7)	1
Celanese	Fiberite X-904	807 (117)	318 (46.1)	11 (1.6)	7.6 (1.1)	-	-	-	4
Celanese*	Epi-Rez 508	647 (93)	238 (34.5)	58 (8.4)	9.6 (1.4)	-	-	-	3
Celanese	Epi-Rez 508	889 (129)	324 (47.0)	-	-	-	-	58 (8.3)	7
Celanese	Celanese R-350A	807 (117)	303 (44.0)	29 (4.2)	6.0 (0.8)	-	-	-	7
Celanese	Celanese R-350A	556 (80)	28 (10.7)**	23 (3.3)	6.0 (0.8)	661 (95)	-	62 (8.9)	2
HMS	Hercules 3002	703 (102)	176 (25.6)	32 (4.7)	7.6 (1.1)	848 (123)	-	69 (10.0)	2
Morganite I	Narmco 1004	528 (76)	163 (23.7)	40 (5.8)	6.3 (0.9)	765 (111)	-	56 (8.1)	2
HMS	Fiberite X-904	514 (74)	194 (28.2)	18 (2.6)	6.0 (0.8)	772 (112)	-	68 (9.7)	2
HMS	3M's PR-287	724 (105)	192 (27.9)	35 (5.0)	7.6 (1.1)	896 (130)	-	70 (10.2)	2
HMS	ERLA 4617	786 (114)	193 (28.0)	31 (4.5)	9.0 (1.3)	-	-	79 (11.4)	2
Morganite I	ERLA 4617	869 (126)	176 (25.5)	41 (5.9)	8.3 (1.2)	972 (141)	70.3 (10.2)	60 (8.6)	2
Morganite I	Narmco XHB178	731 (106)	226 (32.8)	28 (4.0)	6.3 (0.9)	661 (95)	36 (5.2)	49 (7.1)	2
Morganite I	Narmco 2387	661 (95)	221 (32.0)	28 (4.0)	6.9 (1.0)	668 (96)	40 (5.7)	50 (7.2)	2
Celanese	Celanese R-350A	-	-	-	-	862 (125)	42 (6.0)	59 (8.5)	8

*45 v/o Celanese plus asbestos carrier. **50.4 msi in compression. ***Reference 2-5.

Table 2-5. Comparison of 450° K (450° F) Resin Systems with Graphite Fibers***

Epoxy Resin	Fiber	0° Tensile Strength RT, MN/m² (ksi)	0° Tensile Strength % Ret. @ 450°K (350F)	90° Tensile Strength RT, MN/m² (ksi)	90° Tensile Strength % Ret. @ 450°K (350F)	0° Flexural Strength RT, MN/m² (ksi)	0° Flexural Strength % Ret. @ 480°K (350F)	90° Flexural Strength RT, MN/m² (ksi)	90° Flexural Strength % Ret. @ 450°K (350F)	Horizontal Shear RT, MN/m² (ksi)	Horizontal Shear % Ret. @ 450°K (350F)	Reference
F&H 4617	Type II	-	-	-	-	1793 (261)	55.1*	117 (17.0)	39.4*	105 (15.3)	39.3*	1
Fiberite 4617	Type II	-	-	-	-	1469 (213)	85.9*	75 (10.9)	-	945 (13.7)	43.1*	1
3M's PR-287	Type II	-	-	-	-	1027 (149)	100.0*	76 (11.0)	81.8*	883 (12.8)	68.8*	1
Ciba 95	Type II	-	-	-	-	1351 (196)	86.4*	107 (15.5)	63.2*	111 (16.1)	60.3*	1
Fiberite X-904	HTS	1248 (181)	87.6	-	-	-	-	-	-	101 (14.6)	36.0	4
Fiberite X-904	Celanese	807 (117)	94.0	-	-	-	-	-	-	38 (5.5)	68.0	4
Fiberite X-904	Morganite II	1193 (173)	103.4	-	-	-	-	-	-	83 (12.0)	44.4	4
Ferro E-293**	Morganite II	765 (111)	90.5	22 (3.2)	25.0	-	-	-	-	-	-	4
BXP-2401**	Morganite II	745 (108)	98.7	17 (2.5)	117	-	-	-	-	-	-	4
Narmco 2387**	Morganite II	710 (103)	120.6	32 (4.7)	61.4	-	-	-	-	-	-	4
Narmco 1004**	Morganite II	862 (125)	101.4	48 (6.9)	63.6	-	-	-	-	81 (11.7)	6.1	4
Fiberite X-904	HMS	-	-	-	-	772 (112)	65.5	-	-	68 (9.7)	54.7	2
3M's PR-287	HMS	-	-	-	-	896 (130)	82.0	-	-	70 (10.2)	50.0	2
Narmco 1004	Morganite I	-	-	-	-	765 (111)	57.6	-	-	56 (8.1)	59.4	2
Hercules 3002	HMS	-	-	-	-	848 (123)	70.2	-	-	69 (10.0)	72.0	2
Fiberite X-904	HTS	-	-	-	-	1041 (151)	81.9	-	-	88 (12.7)	53.5	2
BXP 2401	Morganite II	-	-	-	-	1083 (157)	70.0	-	-	99 (14.4)	43.8	2
Celanese R350A	Celanese	-	-	-	-	661 (95)	72.0	-	-	62 (8.9)	59.5	2
Epon 1031/828/CPDA	HMG-50	-	-	-	-	627 (90)	56.2	-	-	-	-	2
Fiberite X-903	HMS	-	-	-	-	689 (99)	90.0	-	-	36 (5.2)	67.4	2
Fiberite X-904	HTS	-	-	-	-	1124 (163)	73.7	-	-	81 (11.7)	56.5	2
Fiberite X-904	HMS	-	-	-	-	858 (124)	83.3	-	-	48 (6.9)	79.8	2
Narmco 2387	Morganite II	-	-	-	-	1058 (152)	-	-	-	86 (12.4)	47.0	2

*High-temperature tests conducted at 300F. **Made with some very early continuous Morganite II. ***Collection of data first appeared in Reference 2-5.

Table 2-6. Evaluation of High-Modulus Graphite-Epoxy Systems

	Hybrid HT-S/X-904 & Celanese GY-70/X-904	Celanese GY-70/R-350A (GRT-70-350A)	Celanese GY-70/1004
Longitudinal Flexure Strength MN/m^2 (ksi)			
77°K (-320F)	703 (102)	592 (85)	814 (118)
297°K (RT)	821 (119)	772 (112)	827 (120)
450°K (350F)	668 (96)	848 (123)	731 (106)
Transverse Flexure Strength MN/m^2 (ksi)			
77°K (-320F)	31.1 (4.47)	35.1 (5.07)	36.7 (5.31)
297°K (RT)	31.4 (4.53)	38.8 (5.59)	33.7 (4.87)
450°K (350F)	21.0 (3.06)	26.7 (3.87)	21.9 (3.29)
Interfiber Shear Strength MN/m^2 (ksi)			
77°K (-320F)	38.3 (5.53)	42.1 (6.09)	47.4 (6.83)
297°K (RT)	39.0 (5.63)	49.1 (7.07)	54.1 (7.79)
450°K (350F)	24.9 (3.61)	25.0 (3.62)	33.4 (4.83)
Specific Gravity	1.719	1.621	1.679
Resin Content, %	25.57	31.91	28.18
Fiber Volume, %	65.1	57.6	62.0

Table 2-7. Evaluation of High-Strength Graphite-Epoxy Systems

	HT-S/X-904 (hy-E-1311-B)	Modmor II/ 1004	HT-S/ BXP-2401	HT-S/3002 (Hercules 3002T)
Longitudinal Flexure Strength MN/m^2 (ksi)				
77°K (-320°F)	1338 (194)	1062 (154)	772 (112)	855 (124)
297°K (RT)	1620 (235)	1207 (175)	1089 (158)	1255 (182)
450°K (350°F)	855 (124)	1110 (161)	855 (124)	1276 (185)
Transverse Flexure Strength MN/m^2 (ksi)				
77°K (-320°F)	104 (15.1)	123 (17.8)	87.6 (12.7)	65 (9.4)
297°K (RT)	51 (7.3)	85.5 (12.4)	91.0 (13.2)	94.5 (13.7)
450°K (350°F)	33 (4.8)	53 (7.6)	60 (8.6)	57 (8.2)
Interfiber Shear Strength MN/m^2 (ksi)				
77°K (-320F)	77.9 (11.3)	64 (9.2)	65 (9.3)	66 (9.5)
297°K (RT)	69.6 (10.1)	73.8 (10.7)	70.3 (10.2)	62 (8.9)
450°K (350°F)	37 (5.4)	38 (5.5)	44 (6.3)	29 (4.2)
Specific Gravity	1.536			
Resin Content, %	34.61			
Fiber Volume, %	57.3			

*Failed as extension of notch, rather than in shear.

Note:
Modmor II/1004 is meter length
HT-S/X-904 is short length
HT-S/BXP-2401 is continuous
HR-S/3002T is continuous

Table 2-8. Materials Flow Chart

Material	Material Designation	Supplier	Quantity (pounds)	Batch No.	Date of Receipt
Graphite/Epoxy	hy-E-1311-B	Fiberite	1	OB66	7/30/70
	hy-E-1511	Fiberite	1	OB65	8/10/70
	Hercules 3002T	Hercules	1	C500	8/19/70
	GRT-70-350A	Celanese	1	99-1-1	5/14/70
	hy-E-1511	Fiberite	1	OA80	5/19/70
	hy-E-1311-B	Fiberite	1	OA81	5/15/70
	Celanese 70/1004	Whittaker	1	0019-1	5/19/70
	Modmor II/1004	Whittaker	1	0019-2	5/19/70
	HT-S/BXP-2401	American Cyanamid	1	W-50	5/21/70
	Hercules 3002T	Hercules	1	X201-40	5/13/70

materials obtained in this program compare very favorably with the materials received on similar programs. The procedures for obtaining volatile content, resin content, percent flow, and for running the process gel test are summarized in Appendix E, Volume II.

2.2.1 RESIN CURE DATA. One of the requirements of this program was to develop the technology of fabricating laminates up to two inches in thickness. To be able to accurately fabricate this type of laminate, a great amount of knowledge is required about the various candidate resin systems. Differential thermal analysis (DTA), thermogravimetric analysis (TGA), and time/temperature/viscosity/relationships are all of importance in solving the problems incurred in the curing and fabrication of thick laminates. Convair Aerospace under government sponsorship collected or developed this data on some of the potential high temperature epoxy resins currently being used in graphite prepregs (Reference 2-5). This data is shown in Figures 2-1 through 2-8. Details of the techniques used to obtain this data are given in Reference 2-5.

2.2.2 PANEL FABRICATION. Unidirectional panels 25.4 cm by 15.2 cm by 10 ply (10- by 6-inch by 10-ply laminates) were fabricated for each of the systems selected for the characterization study. Vendor-specified cure cycles for vacuum-bag, press, and autoclave curing of the composites were used in this initial study. Typical layup for the graphite/epoxy system is shown in Figure 2-9.

Table 2-9. Graphite/Epoxy Prepreg Test Results

Material Designation	Batch No.	Fiber	Resin	Avg. % Volatiles	Avg. % Resin	Avg. % Flow	Process Gel Test Results	Observations
hy-E-1311-B	OB66	HT-S	X-904	7.8	33.6	14.9	Gelled at 393°K (250°F) between 15	Uniform, good tack
hy-E-1511	OB65	GY-70	X-904	5.4	33.8	15.8	Gelled at 393°K (250°F) between 0 and 15 minutes	Gaps, good tack
Hercules 3002T	C500	HT-S	3002T	5.3	33.8	12.7	Gelled at 433°K (320°) between 5 and 10 minutes	Very uniform, good tack
GRT-70-350A	99-1-1	Celanese GY-70	R-350A	1.0	41.0	20.7	Gelled between 393°K (250°F) and 450°K (350°F)	Very tacky, some gaps
hy-E-1511	OA80	Celanese GY-70	X-904	5.5	35.1	26.8	Gelled at 393°K (250°F) between 15 and 30 minutes	Slight tack, a few gaps, transverse compaction marks
Celanese 70/1004	0019-1	Celanese CY-70	1004	0.30	41.7	25.0	Gelled at 450°K (350°F) between 15 and 30 minutes	Very tacky, difficult to remove Mylar, some fiber fraying and gaps
Modmor II/1004	0019-2	Modmor II	1004	0.73	42.3	24.3	Gelled at 450°K (350°F) between 15 and 30 minutes	Dry on one side, some tack on other side, some fiber disarrangement
hy-E-1311-B	OA81	HT-S	X-904	5.9	45.4	24.1	Gelled at 393°K (250°F) between 0 and 15 minutes	Dry, some gaps, transverse compaction marks
HT-S/BXP-2401	W-50	HT-S	BXP-2401	0.13	41.0	12.1	Gelled at 455°K (360°F) between 0 and 15 minutes	Prepreg is dry between tacky layer of resin on both sides
Hercules 3002T	X201-40	HT-S	3002	8.1	40.3	22.6	Gelled at 393°K (250°F) between 60 and 75 minutes	Uniform, good tack

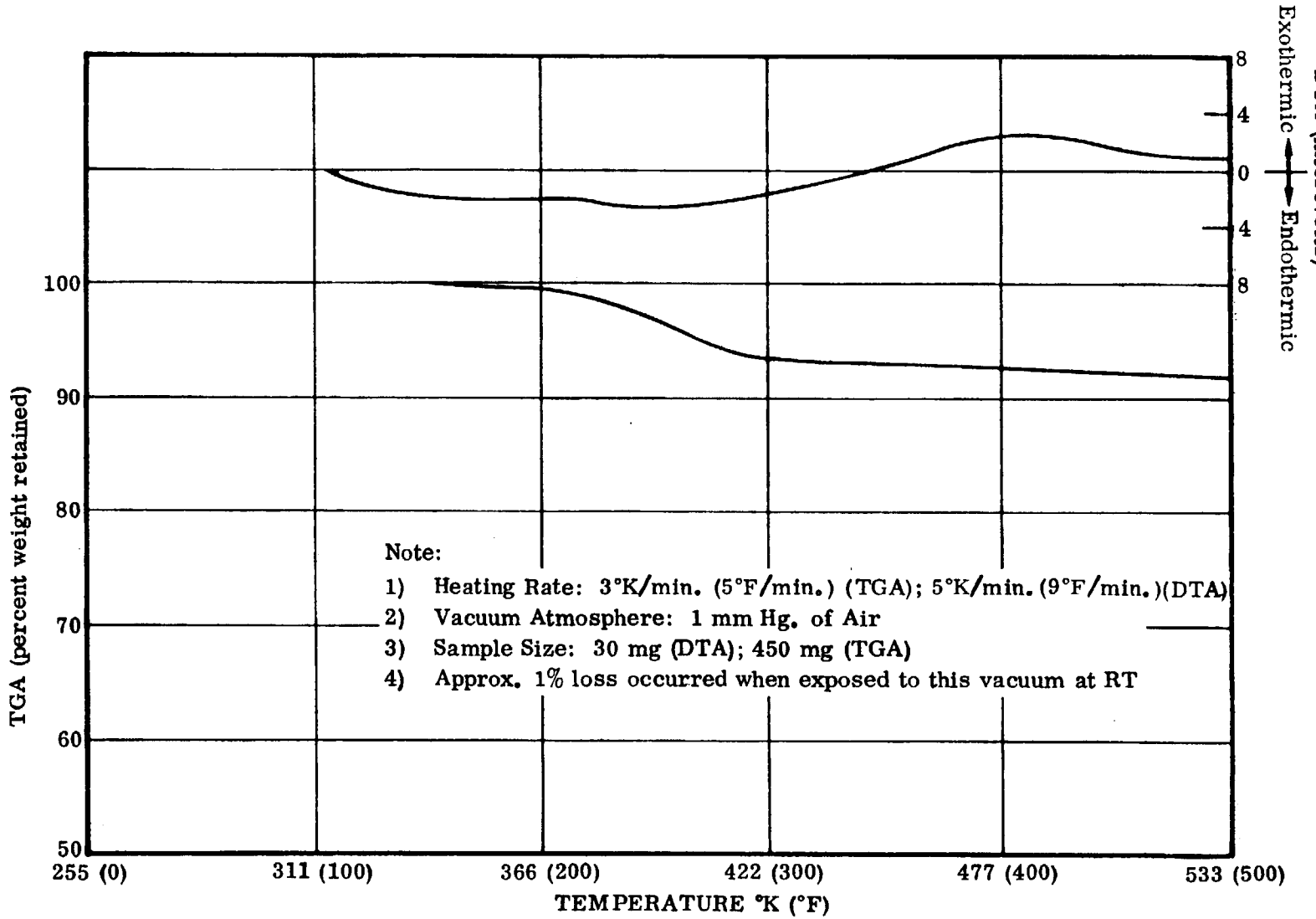


Figure 2-1. Thermogravimetric and Differential Thermal Analysis of MODMOR II/1004

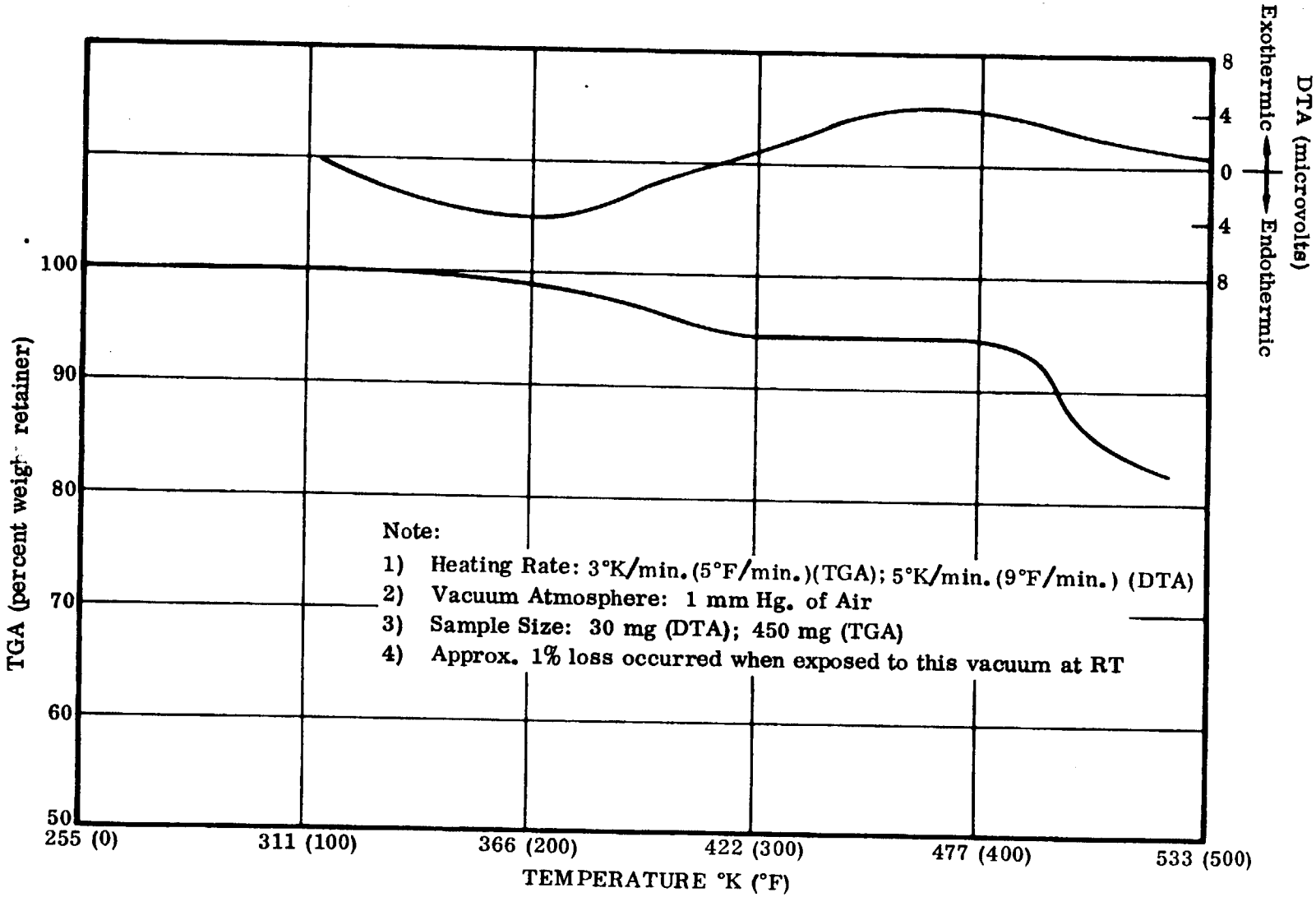


Figure 2-2. Thermogravimetric and Differential Thermal Analysis of HT-S/BXP-2401

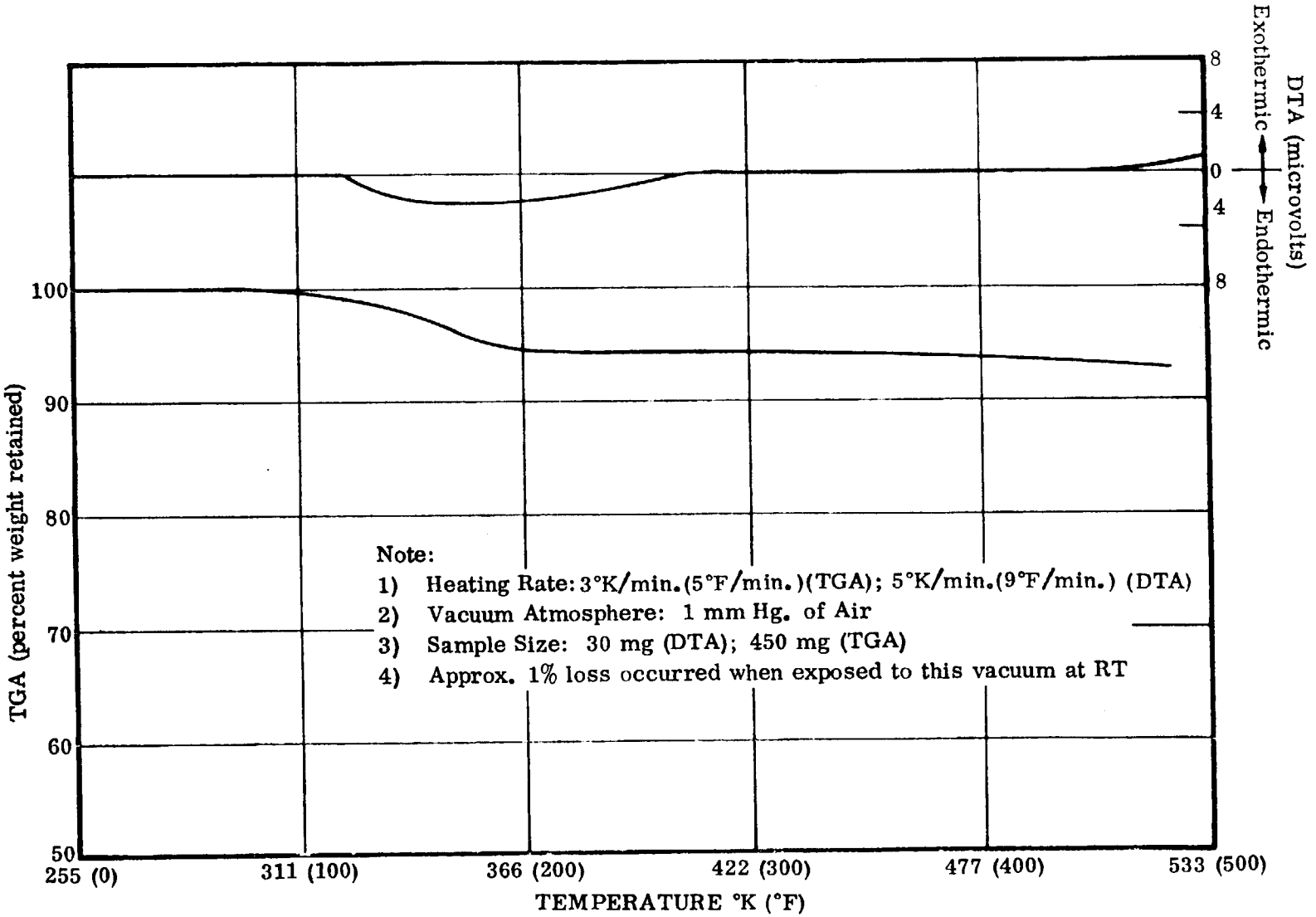


Figure 2-3. Thermogravimetric and Differential Analysis of hy-E-1311-B

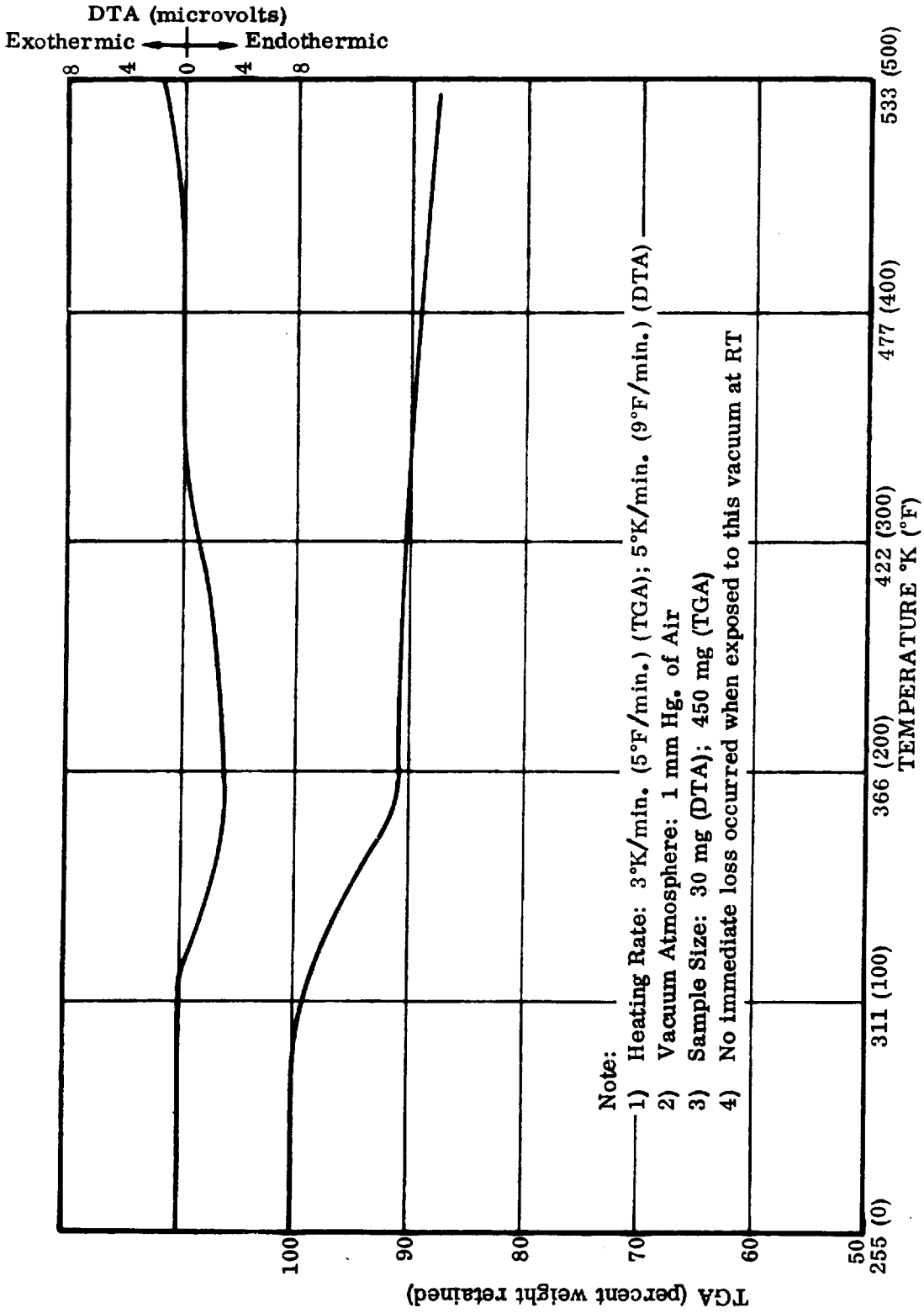


Figure 2-4. Thermogravimetric and Differential Thermal Analysis of Celanese GRT-70-350A

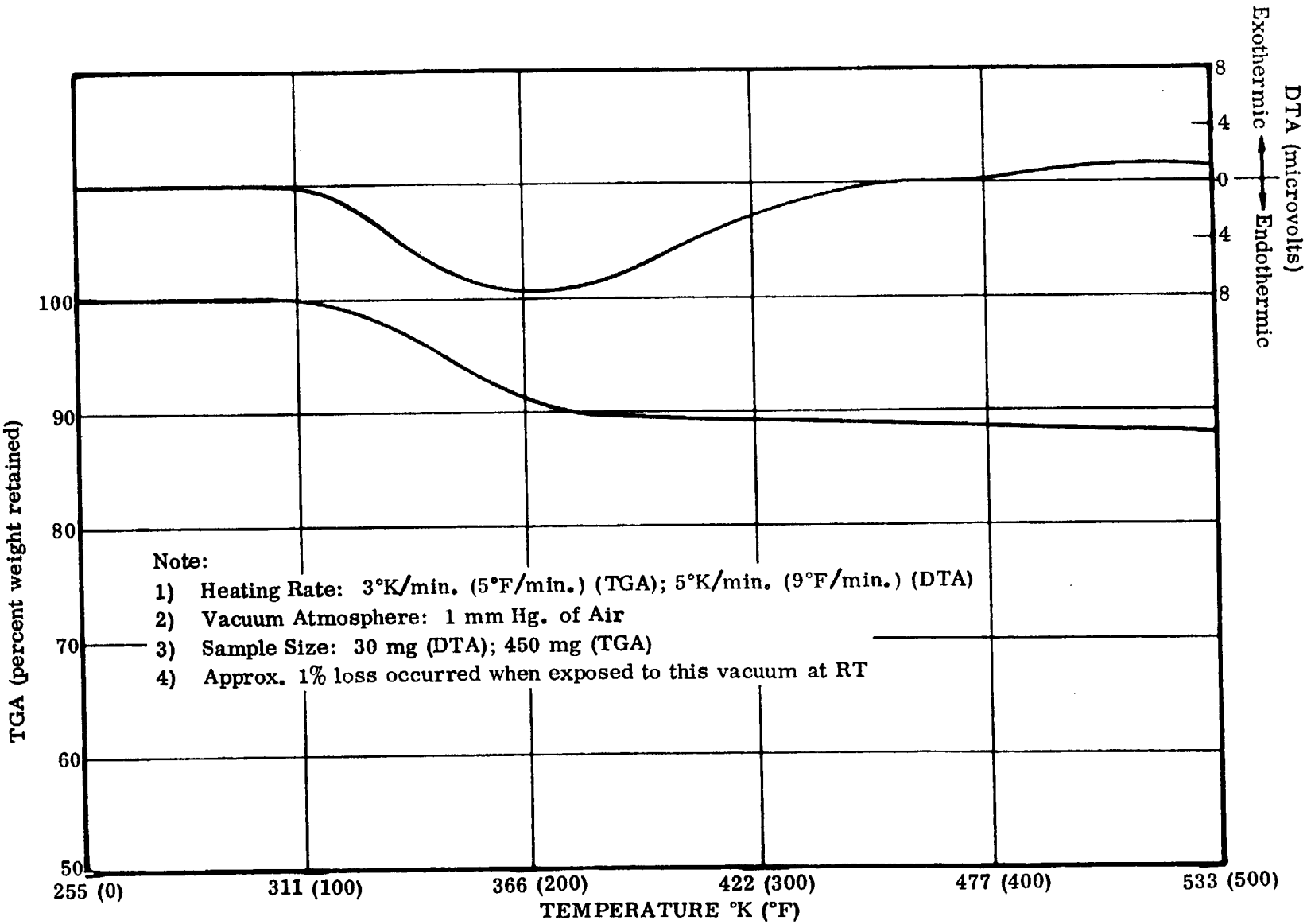


Figure 2-5. Thermogravimetric Analysis of Hercules HT-S/3002

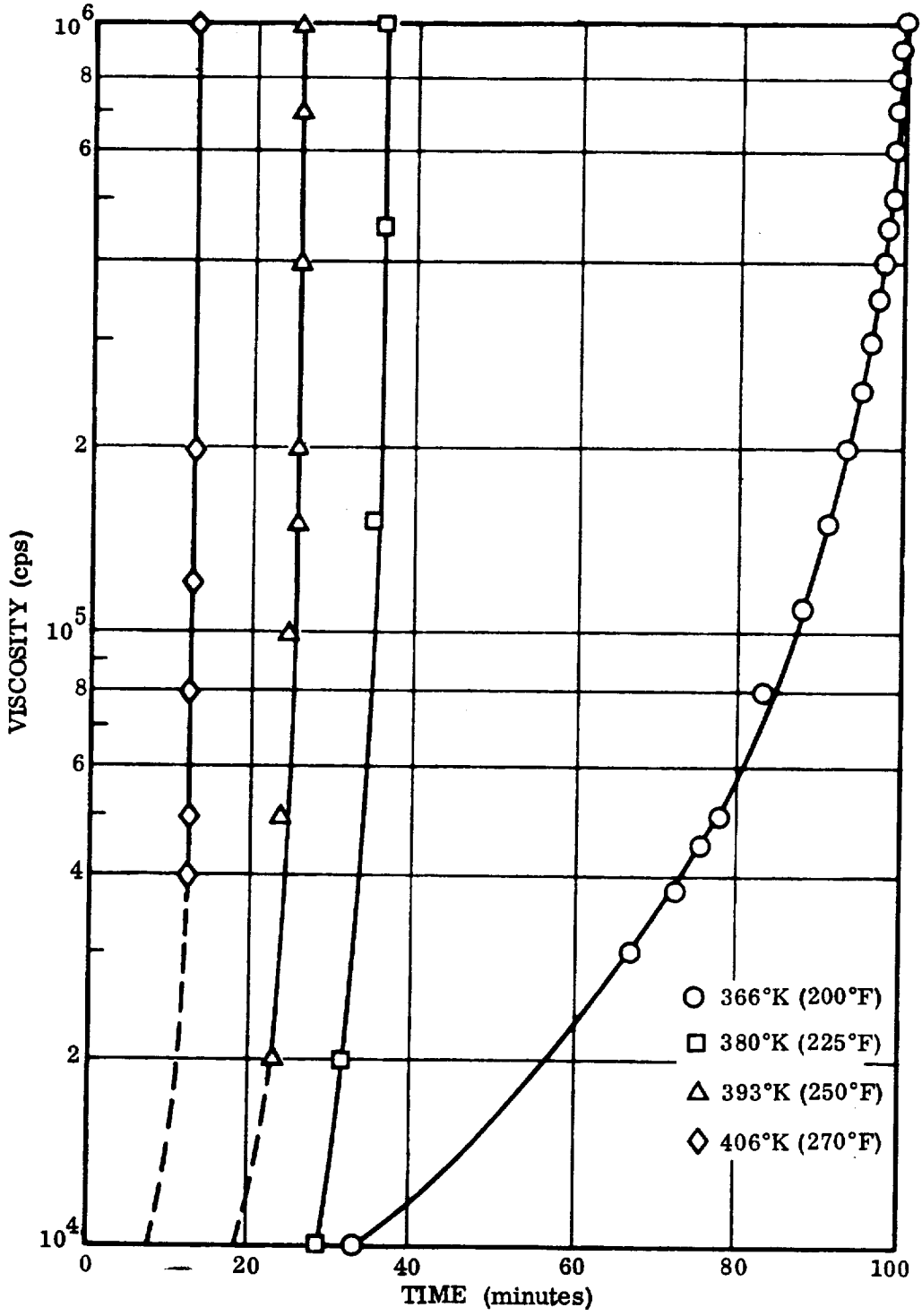


Figure 2-6. Time/Temperature/Viscosity Relationships of the Fiberite X-904 Resin System

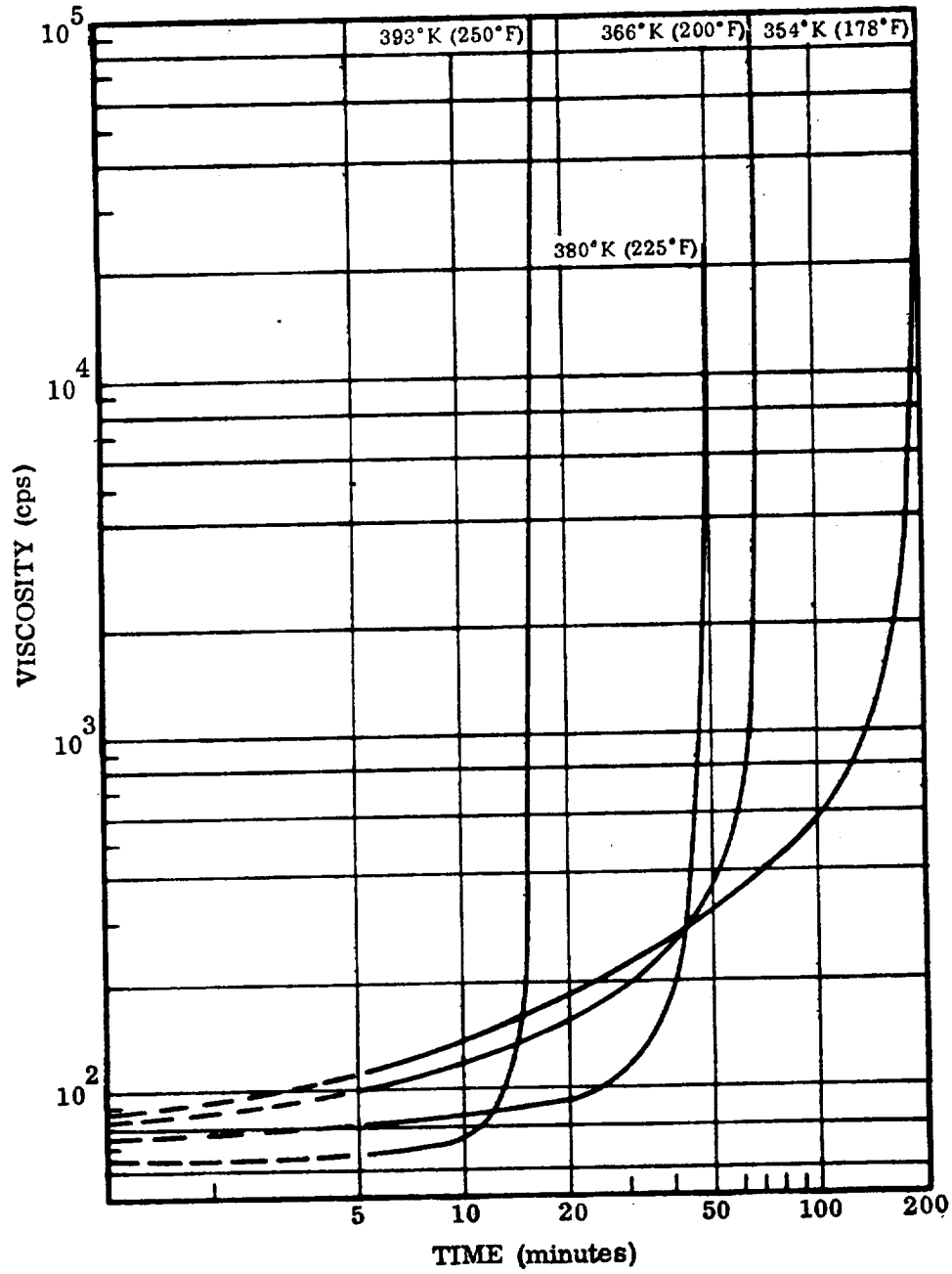


Figure 2-7. Time/Temperature/Viscosity Relationships of the Celanese R-350A Resin System

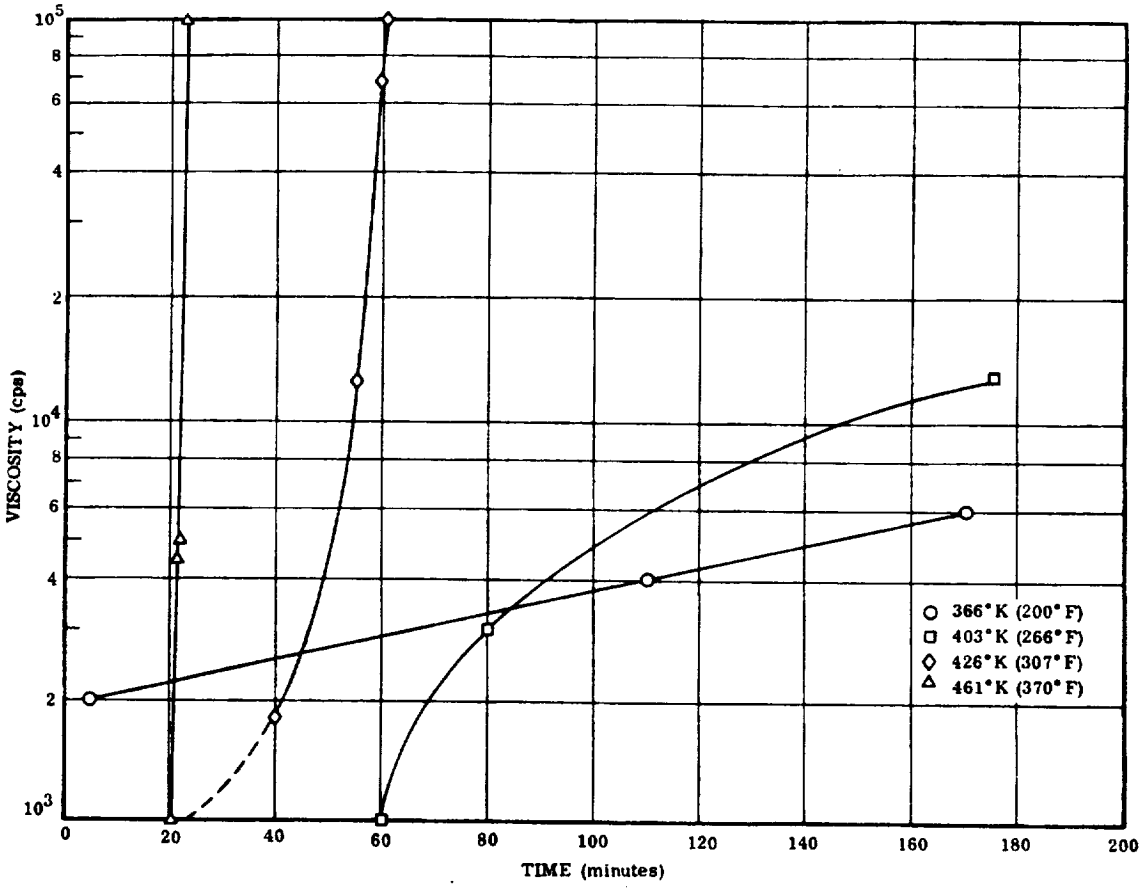


Figure 2-8. Time/Temperature/Viscosity Relationship of the Whittaker 1004 Resin System

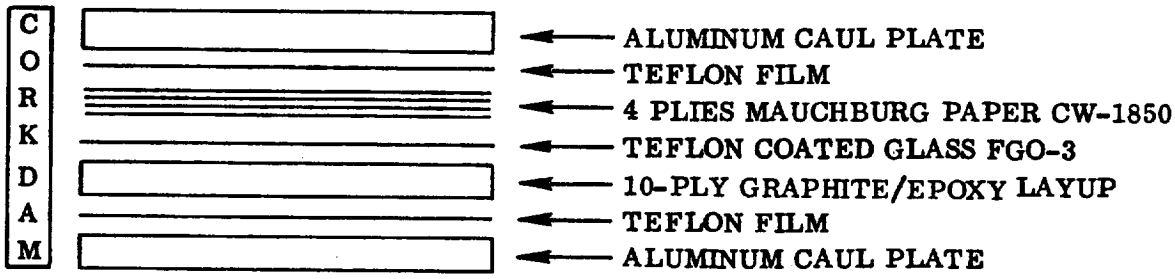


Figure 2-9. Typical Graphite/Epoxy Cure Layup

Each lot of prepreg is checked per the tests outlined on the Graphite Prepreg Tests sheet, Form 512-2-MLR-1 (see page 2-18). On completion of these tests, laminate fabrication is initiated. Process control of the fabricated panels is greatly improved by the use of Form 512-2-MLR-2 (see page 2-19), Laminate Fabrication and Test Instructions. By using these forms a detailed account of prepreg lot, prepreg sheet, laminate number, and test results is tabulated. Each graphite/epoxy panel was subjected to an identical post cure of 1/2 hour at 297°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F), and 16 hours at 464°K (375°F). Vacuum-bag, press, and autoclave cures were attempted with each graphite/resin system.

I. EPOXY PREPREGS

A. Material Designation: hy-E-1511-B
Fiber Type: GY-70 (continuous)
Material Form: 30.5-cm by 114-cm (12- by 45-inch) sheet
Batch No.: OB65
Resin: X-904
Manufacturer: Fiberite

1. Fabrication Procedure: (Autoclave Cure)

Mold Release: Teflon film
Layup: 10 plies unidirectional, 25.4 by 15.2 cm (10 by 6 inches)
Separator Film: Teflon-coated glass cloth, FG0-3
Bleeder: 4 plies Mauchburg paper CW-1850
Special Instruction: A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 25.4 by 15.2 by 0.64 cm (10- by 6- by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
Cure Pressure: 760 mm (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) autoclave pressure was applied at RT and maintained through entire cure cycle and cool down to below 344°K (160°F).
Cure Cycle: Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool to below 344°K (160°F).

512-2-MLR-1

GRAPHITE PREPREG TESTS

Requestor: _____ WAP No. _____ Prepreg No. _____

Source:

P.A. No.:

Reinforcement: Type _____ UTS MN/m^2 (KSI) ____ MODULUS ____ GN/m^2 (10^6 psi)

Diameter _____ Form _____.

Resin: Type _____ Designation _____

Prepreg: Qty. Shipped _____ Lot No. _____ Size In _____

Resin Solids _____ % Volatile Content _____ %

Laminate Flow, @ _____ psi _____ °K (°F) _____ Min ____ %

GDC Tests

Resin Solids _____ % Volatile Content _____ %

Laminate Flow, @ ___ kN/m^2 (psig) _____ °K (°F) ___ Min _____ %

Process Gel Test Instructions: Heat-up Rate °K/min (°F/min.)

Other:

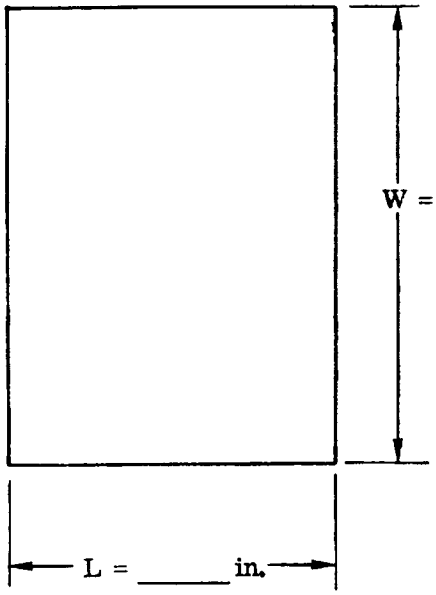
IR Conformation ☐ Yes ☐ No

DTA

TGA

LAMINATE FABRICATION AND TEST INSTRUCTIONS

Requestor:_____ WAP No._____ Laminate No. _____



Prepreg No.:

Reinforcement:

Resin:

Carrier:

No. of Plies:

Ply Orientation:

Ply Order:

Fabrication

Mold Release:

Special Layup Instructions:

Separator Film:

Bleeder System:

Cure Cycle:

Cure Pressure:

Observations: Flow_____% Other _____

Post Cure Cycle:

Physical Properties: Sp. Gr. (g/cc)_____ Avg Thns. (cm)_____ R.C._____%
v/v %

NDT Results

Specimen Preparation

Special Test Instructions

Sketch Nos.:

Other:

	Post Cure:	1/2 hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F) and 16 hours at 464°K (375°F)
2.	Fabrication Procedure: (Press Cure)	Same as fabrication procedure I. A. 1.
	Cure Pressure:	690 kN/m^2 (100 psi) was applied at room temperature and maintained throughout cure cycle — no vacuum.
	Post Cure:	Same as I. A. 1.
3.	Fabrication Procedure: (Vacuum-Bag Cure)	Same as fabrication procedure I. A. 1.
	Cure Pressure:	Full vacuum 760 mm (29 in.) Hg was applied at room temperature and maintained throughout cure cycle.
	Post Cure:	Same as I. A. 1.
B.	Material Designation:	hy-E-1311-B
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114 cm (12- by 45-inch) sheet
	Batch No.:	OB66
	Resin:	X-904
1.	Fabrication Procedure: (Autoclave)	Same as hy-E-1511-B fabrication procedure I. A. 1.
	Cure Pressure:	Same as hy-E-1511-B I. A. 1.
	Post Cure:	Same as hy-E-1511-B I. A. 1.
2.	Fabrication Procedure: (Press)	Same as hy-E-1511-B fabrication procedure I. A. 1.
	Cure Pressure:	Same as hy-E-1511-B I. A. 1.
	Post Cure:	Same as hy-E-1511-B I. A. 1.
3.	Fabrication Procedure: (Vacuum Bag)	Same as hy-E-1511-B fabrication procedure I. A. 1.
	Cure Pressure:	Same as hy-E-1511-B I. A. 1.
	Post Cure:	Same as hy-E-1511-B I. A. 1.
C.	Material Designation:	HT-S/3002T
	Fiber Type:	HT-S (continuous)
	Material Form:	30.5 by 30.5 cm (12- by 12-inch) sheet
	Batch No.:	X201-40
	Resin:	3002
	Manufacturer:	Hercules
1.	Fabrication Procedure: (Autoclave)	
	Mold Release:	Teflon film.

	Layup:	10 plies unidirectional, 25.4 by 15.2 cm (10- by 6-inches).
	Separator Film:	Teflon-coated glass cloth, FGO-3, 0.0028-inch thick.
	Bleeder:	4 plies Mauchburg paper CW-1850.
	Special Instruction:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply of Teflon film (nonperforated) was used over the bleeder. A 25.4 by 15.2 by 0.64 cm (10- by 6- by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	103 kN/m^2 (15 psi) autoclave pressure is applied at room temperature and maintained until 30 minutes at 393°K (250°F) when pressure is increased to 172 KN/m^2 (25 psi). After an additional 25 minutes at 393°K (250°F). the pressure is increased to 690 kN/m^2 (100 psi) was applied at through entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 393°K (250°F) at 3 to 6°K/minute (5 to 10°F/minute), hold 90 minutes at 393°K (250°F); heat to 450°K (350°F) at 3 to 6°K/minute (5 to 10°F/minute) and hold for 2 hours; cool to below 344°K (160°F).
	Post Cure:	One-half hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F), and 16 hours at 464°K (375°F).
2.	Fabrication Procedure: (Press Cure)	Same as fabrication procedure I.C.1.
	Cure Pressure:	Same as I.C.1 except hydraulic press instead of autoclave pressure.
	Post Cure:	Same as I.C.1.

3.	Fabrication Procedure: (Vacuum Bag)	Same as fabrication procedure I. C. 1.
	Cure Pressure:	Full vacuum applied at room temperature and maintained to 378°K (220°F); vacuum pressure reduced to slight positive pressure, 26 to 78 mm (1 to 3 in.) Hg; after 60 minutes at 393°K (250°F) apply full vacuum and maintain throughout the remainder of the cure.
	Cure Cycle:	Heat to 378°K (220°F) at 3 to 6°K/minute (5 to 10°F/minute), hold 10 minutes; heat to 393°K (250°F) and hold for 90 minutes; raise temperature to 450°K (350°F) at 3 to 6°K/minute (5 to 10°F/minute) and hold for 2 hours; cool to below 344°K (160°F).
	Post Cure:	Same as I. C. 1.
D.	Material Designation:	GY-70/3002
	Fiber Type:	GY-70 (continuous)
	Material Form:	7.6 cm (3 in.) tape
	Batch No.:	X203-10
	Resin:	3002
	Manufacturer:	Hercules
1.	Fabrication Procedure: (Press)	Same as fabrication procedure I. C. 1.
	Cure Pressure:	Same as I. C. 1 except hydraulic press instead of autoclave pressure.
	Post Cure:	Same as I. C. 1.
2.	Fabrication Procedure: (Vacuum Bag)	Same as fabrication procedure I. C. 3.
	Cure Pressure:	Same as I. C. 3.
	Post Cure:	Same as I. C. 1.

2.2.3 MECHANICAL PROPERTY TESTS. Test specimens were obtained from each panel per the cutting diagram shown in Figure 2-10. Flexural tests both longitudinal and transverse, were conducted at 77, 297, and 450°K (-320, 75, and 350°F). The longitudinal flexure tests were conducted with a three-point loading system, while the transverse flexure tests were conducted with a four-point loading system. A span-to-depth ratio of 32:1 was used for the longitudinal flexure tests. A notched-interfiber-shear specimen was used instead of a short-beam-shear specimen. All of the other interlaminar shear data in this report was obtained from the short-beam-shear specimen because the majority of interlaminar shear data that is currently available uses this type of specimen. The 77°K (-320°F) tests were conducted after a five-minute soak, while the 450°K (350°F) specimens were exposed to a 10-minute temperature soak before testing. Laminate resin contents were determined using an H_2SO_4/H_2O_2 digestion method as detailed in Appendix C. Specific gravity was determined for each panel per Federal Test Method Standard No. 4-6, Method 5011.

A summary of the data obtained on the graphite/epoxy systems is listed in Tables 2-10 and 2-11. The major difference in the data is the higher strengths obtained from the HT-S composites compared to the GY-70 composites. This result was as expected, because there is a major difference in the modulus and strengths of the two fibers. The major attribute of the GY-70 composite is its high composite modulus, normally over 276 GN/m^2 (40×10^6 psi), as compared to the HT-S composite, 110 to 138 GN/m^2 (16 to 20×10^6 psi). During this cursory evaluation the shear and transverse strengths could not be significantly improved, and the HT-S fiber was selected over the GY-70 fiber. Also, the HT-S fiber is more highly characterized and thus more data is available on this type of fibrous composite.

The selection of the epoxy resin system was a difficult problem in that both the X-904 and 3002 systems have excellent properties depending on the particular application. Also complicating the selection was the substandard lot of HT-S/X-904 prepreg initially received by Convair. This problem was resolved, and good quality prepreg was obtained for this program. The selection criterion used by Convair was two-fold in that the selected graphite/resin system had to be usable over a temperature range from 77 to 450°K (-320 to 350°F), and useful strength properties were required whether it was vacuum bag, press, or autoclave cured. The 3002T system shows excellent properties at 450°K (350°F), but it shows a significant dropoff in strength at cryogenic temperatures and also a smaller reduction in vacuum bag laminate properties. The poor data at 450°K (350°F) for the HT-S/X-904 is attributed to the inferior lot of prepreg. Therefore, based on the data developed on this program and related data on other contractual efforts, the HT-S/X-904 system was selected for further process refinement and design data development.

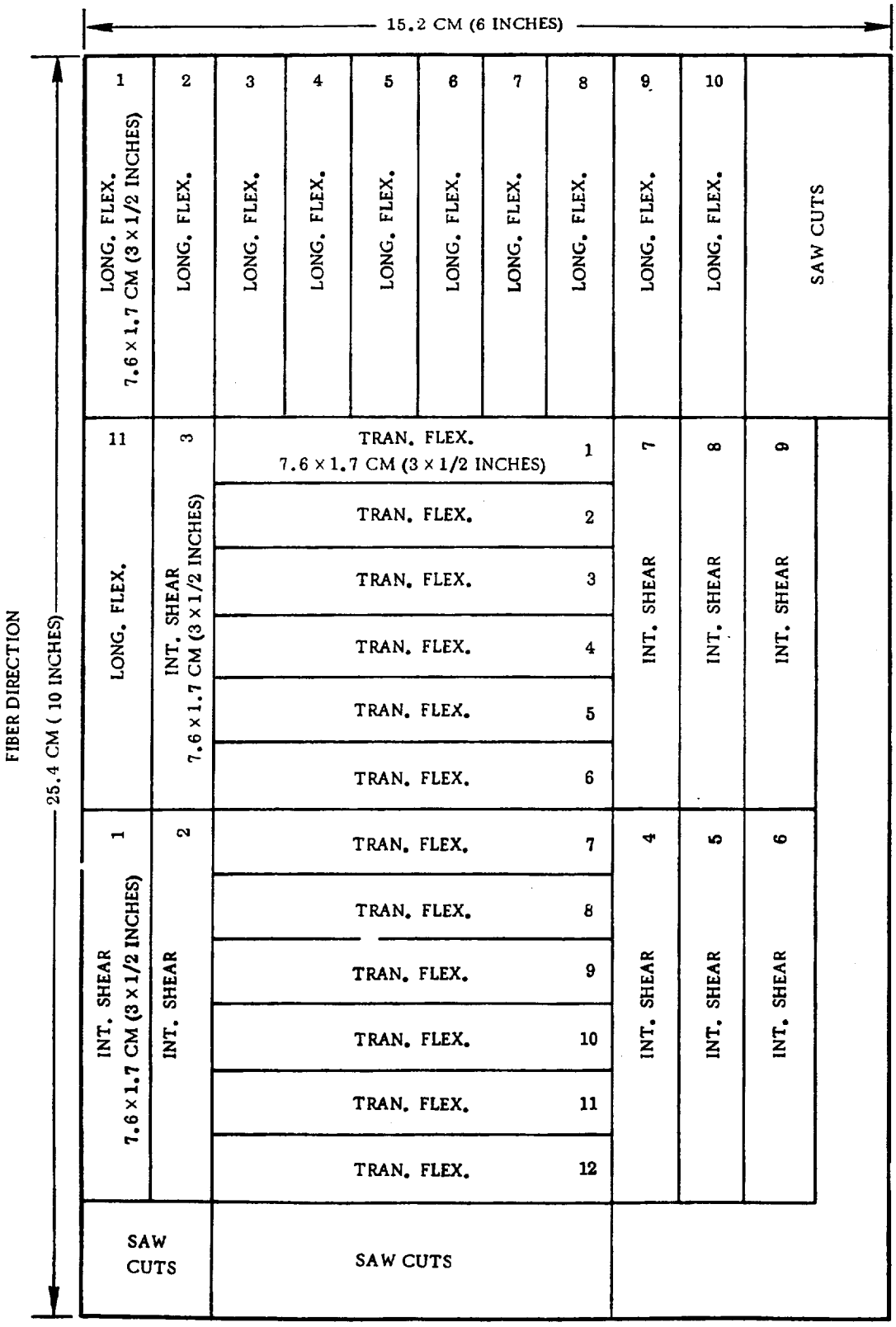


Figure 2-10. Cutting Diagram

Table 2-10. Evaluation of High-Strength Graphite/Epoxy Systems

	HT-S/X-904 Cure Cycle*						HT-S/3002T Cure Cycle*					
	1		2		3		1		2		3	
Longitudinal Flexure Strength MN/m^2 (ksi)												
77°K (-320°F)	1055	(152.7)	1138	(165.4)	-	-	585	(83.6)	1083	(157.3)	1110	(160.8)
297°K (75°F)	1000	(145.0)	1241	(179.6)	1393	(202.8)	640	(92.8)	1593	(230.6)	1682	(243.9)
450°K (350°F)	606	(87.2)	465	(67.2)	535	(77.4)	500	(72.1)	1089	(158.1)	1089	(158.4)
Transverse Flexure Strength MN/m^2 (ksi)												
77°K (-320°F)	40	(5.8)	88	(12.8)	-	-	43	(6.2)	49	(7.0)	60	(8.6)
297°K (75°F)	28	(4.0)	62	(8.9)	46	(6.6)	27	(3.9)	76	(11.0)	69	(10.0)
450°K (350°K)	17	(2.4)	17	(2.4)	14	(2.0)	24	(3.5)	34	(4.9)	37	(5.3)
Interfiber Shear Strength MN/m^2 (ksi)												
77°K (-320°F)	65	(9.4)	75	(10.9)	-	-	58	(8.3)	56	(8.0)	50	(7.2)
297°K (75°F)	58	(8.4)	72	(10.5)	-	-	60	(8.6)	67	(9.6)	54	(7.8)
450°K (350°K)	-	-	-	-	-	-	-	-	-	-	-	-
Specific Gravity	1.47		1.54		1.56		1.51		1.59		1.60	
Resin Contant (%)	31.0		31.6		30.0		32.0		28.0		21.8	
Fiber Volume (%)	61.3		60.6		62.3		59.0		62.5		63.0	
Calculated Void Content (%)	6.0		1.3		0.6		1.3		0.0		0.0	

*Cure Cycle 1: Vacuum Bag
Cure Cycle 2: Press
Cure Cycle 3: Autoclave

Table 2-11. Evaluation of High Modulus Graphite/Epoxy System

	GY-70/X-904 Cure Cycle						GY-70/3002 Cure Cycle			
	1		2		3		1		2	
Longitudinal Flexure Strength MN/m^2 (ksi)										
77°K (-320°F)	896	(129.9)	834	(121.0)	772	(111.7)	640	(92.3)	703	(102.3)
297°K (75°F)	793	(115.1)	682	(98.0)	855	(123.5)	738	(107.0)	834	(121.2)
450°K (350°F)	613	(88.5)	535	(77.3)	690	(99.5)	549	(78.6)	627	(90.4)
Transverse Flexure Strength MN/m^2 (ksi)										
77°K (-320°F)	20	(2.9)	16	(2.4)	35	(5.0)	-	-	-	-
297°K (75°F)	29	(4.2)	17	(2.5)	-	-	11	(1.6)	32	(4.6)
450°K (350°F)	14	(2.0)	17	(2.5)	15	(2.2)	-	-	-	-
Interfiber Shear Strength MN/m^2 (ksi)										
77°K (-320°F)	37	(5.4)	35	(5.0)	44	(6.4)	-	-	-	-
297°K (75°F)	36	(5.3)	24	(3.5)	-	-	-	-	-	-
450°K (350°F)	21	(3.0)	19	(2.8)	25	(3.6)	-	-	-	-
Specific Gravity		1.74		1.64		1.69		-		-
Resin Content (%)		24.0		30.6		28.3		-		-
Fiber Volume (%)		66.9		59.4		61.8		-		-
Calculated Void Content (%)		0.0		1.6		0.5		-		-

2.3 EPOXY RESIN CHARACTERIZATION

Several high-temperature epoxide resin systems were screened at the initiation of this program by Convair. These included Whittaker's 1004, Hercules' 3002, and Fiberite's X-904 resin systems. The Fiberite X-904 resin system was chosen for this program because of its broad temperature range capabilities, 77° K to 450° K (-320° F to 350° F). The X-904 epoxy resin system was specifically formulated for use as a graphite fiber matrix in composites subjected to high-temperature environments for extended periods of time. Figure 2-11 presents some isothermal aging data on an HT-S composite after 1000 hours aging at 450° K (350° F). Figure 2-12 presents high-temperature strength retention to 533° K (500° F) of the same system.

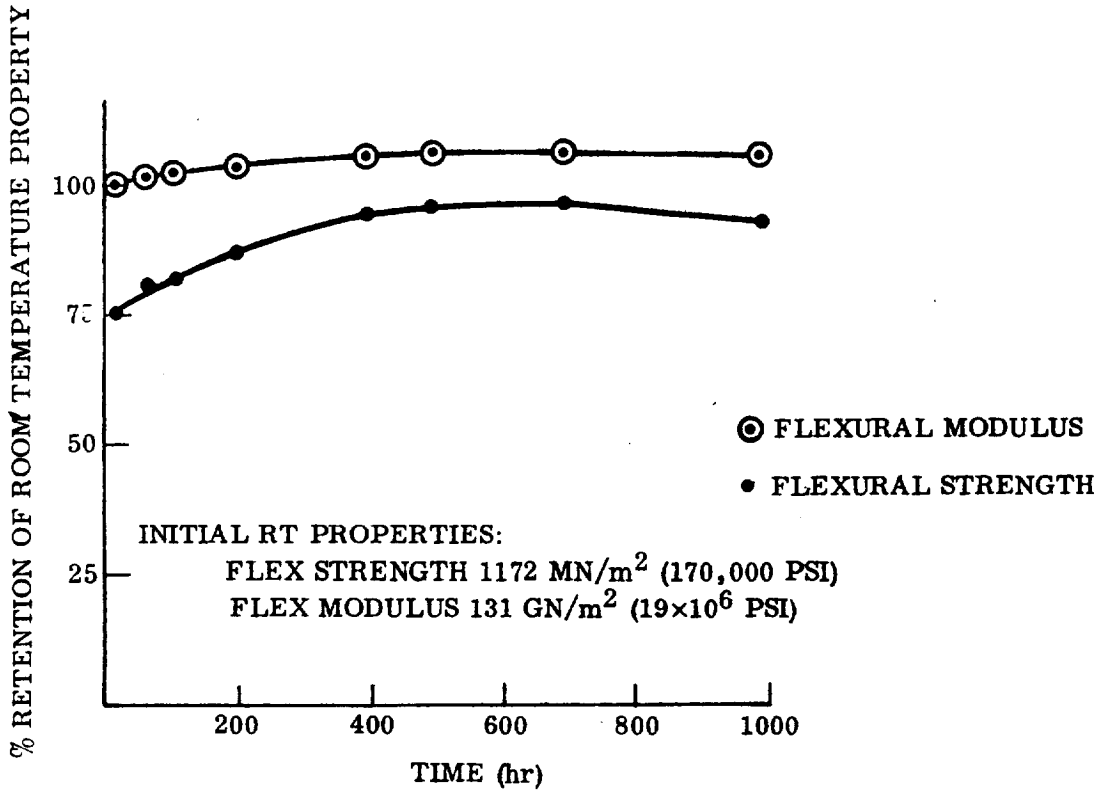
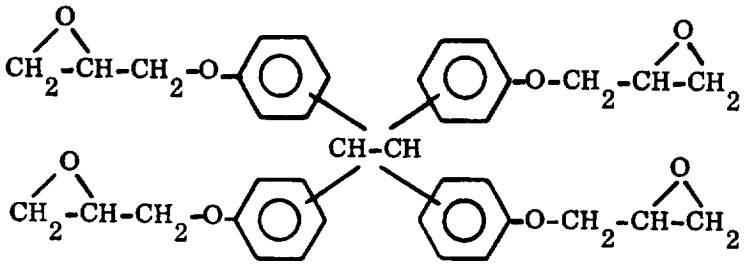
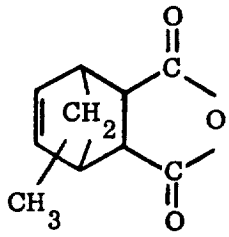


Figure 2-11. Isothermal Aging Data at 450° K (350° F) for HT-S/X-904 Tested at 450° K (350° F)

2.3.1 CHEMISTRY OF THE X-904 EPOXIDE SYSTEM. The X-904 chemically consists of an aromatic polyglycidyl ether (ERRA-0163), cured with a monoanhydride (nadic methyl anhydride), and catalized with benzyldimethylamine (BDMA). The resin and curing agent have the following idealized structures:

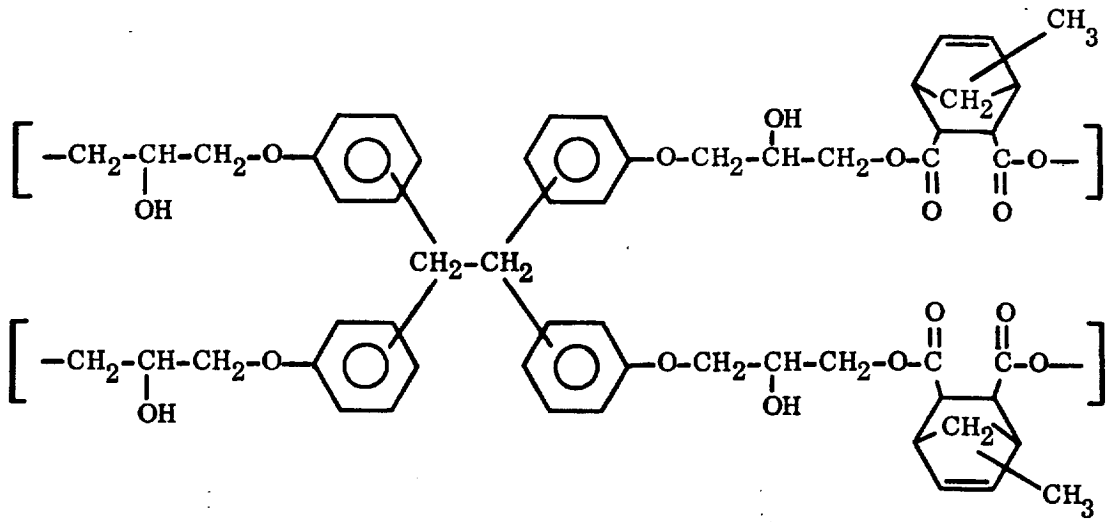


ERRA-0163



NMA

The stoichiometry of the anhydride is 85% of theoretical. This allows for 15% etherification reaction. Reaction rate theory and practice substantiates that the highest Tg is obtained using 85% stoichiometry with this system. The cured structure for X-904 is given below in its idealized form:



Anhydride cured systems such as the X-904 have consistently shown superior isothermal aging characteristics when compared to amine or catalytically cured systems. The reason for this is the inherent thermal stability of the resultant ester linkage over the amine or ether linkage produced by other cure mechanisms.

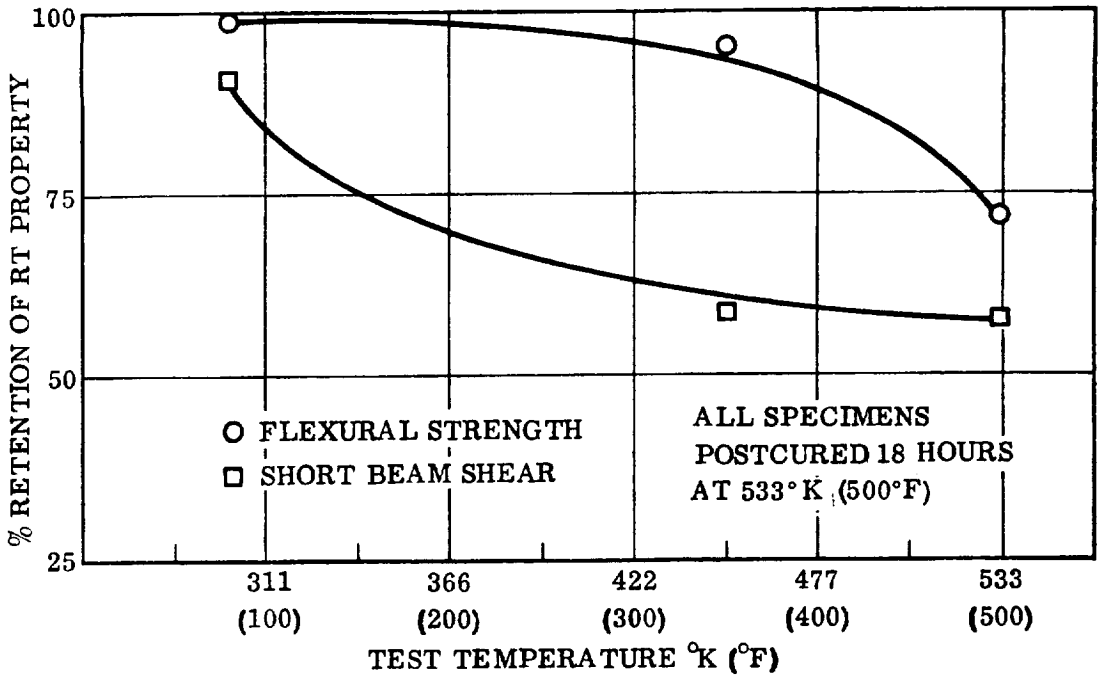


Figure 2-12. Elevated Temperature Test Data for HT-S/X-904

The formulation as a whole is somewhat insensitive (± 5%) to NMA concentrations since the etherification reaction will complete the cure if insufficient NMA is present. However, the absence of BDMA or similar tertiary amine catalyst will have a pronounced effect upon the resin system as will the presence of water (1% or greater). Table 2-12 shows data concerning the effects of catalyst loss or omission as well as the effect of moisture.

Table 2-12. X-904 Resin Gel Times as a Function of Catalyst and Water Content

Formulation	A	B	C
ERRA-0163	100	100	100
NMA	70	70	70
BDMA	—	0.5	0.5
H_2O	—	—	2.0
Gel Time at 395°K (251°F) in minutes	185.8	12.5	17.1

2.3.2 POLYMER REACTION RATES. The reaction rate of this resin system at 353°K (175°F), 366°K (200°F), and 406°K (270°F) was measured and plotted as time-versus-viscosity curves and is shown in Figure 2-13. These measurements were made with a Brookfield viscometer. The resin system was placed in an isothermal bath and the viscosity (monitored continuously) was recorded with time.

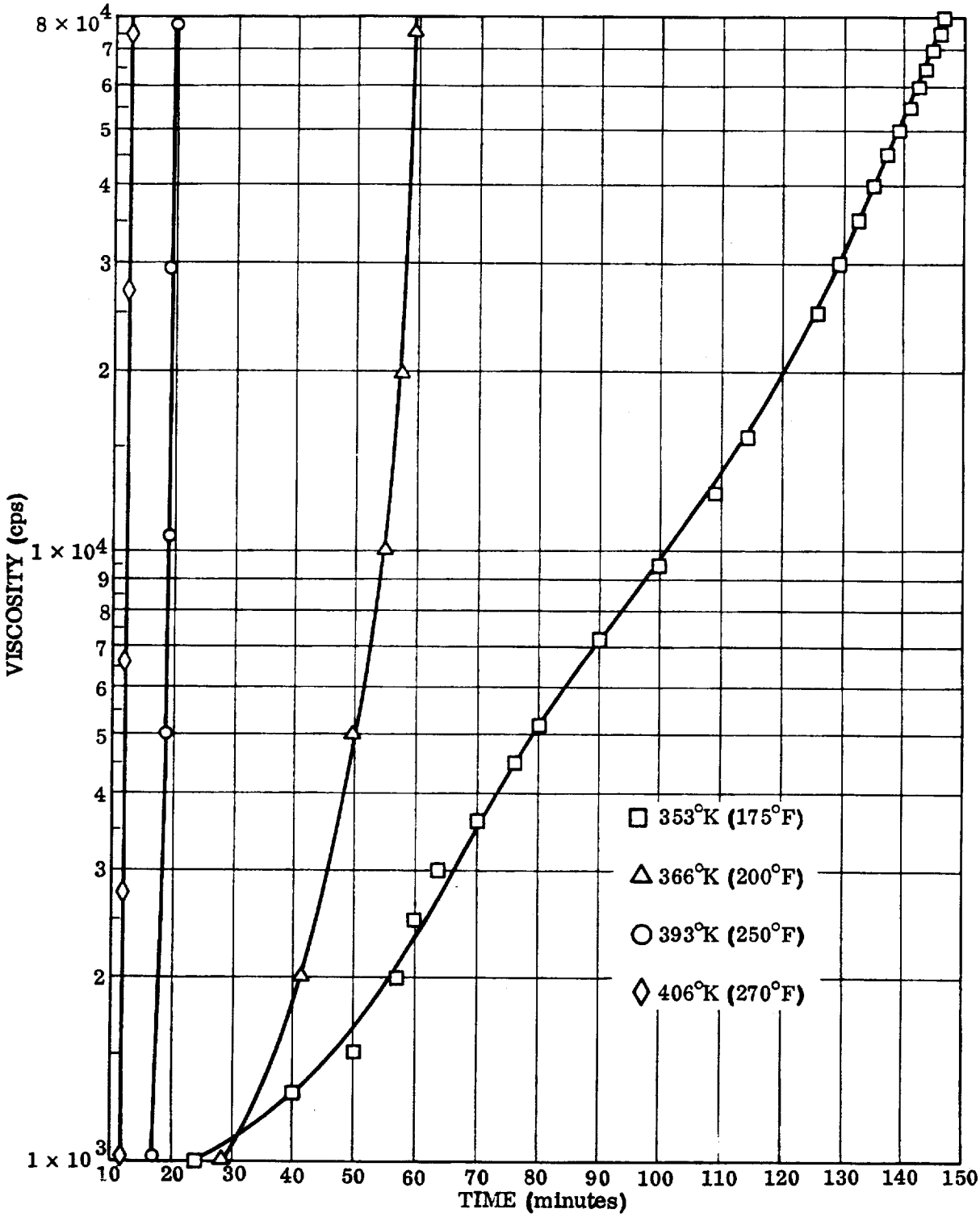


Figure 2-13. Isothermal Reaction Rate Curves for X-904 Resin System

The isothermal reaction curves show an interesting phenomenon of this resin system. The resin viscosity at the higher temperatures stays quite low (below 1000 cps) for a major portion of the time to gel. This low viscosity allows entrapped air and residual solvent volatile to escape via the bleeder to give very low void content composites. This zero or extremely low composite void content contributes to the excellent isothermal aging performance of this system.

Figure 2-14 is a semi-logarithmic plot of the gel time versus reaction temperatures for the X-904 system. Gel times at any given temperature can be derived from this plot.

2.3.3 REFRACTIVE INDEX STUDIES, X-904. A study was conducted on the increase of refractive index with a reaction of the X-904 at 353°K (176°F). Data is presented in Figure 2-15. The refractive index increases almost linearly with time at 353°K (176°F) and should provide a good quality control procedure for determining resin advancement. The initial nonlinear appearance of the viscosity curve is probably due to a greater extent of chain extension as opposed to branching. Chain branching would result in a greater increase in viscosity with reaction as indicated at the 60- to 90-minute portion of the curve.

Figure 2-16 presents data on the refractive index of X-904 resin shipments. Again the relationship between refractive index and percent resin solids is fairly linear over the range investigated. Hence, this appears to be a useful quality control procedure.

2.3.4 QUALITY CONTROL PROCEDURES. A number of quality control procedures should be run on the incoming epoxy resin and curing agent as well as master batches of this mixed system. They are as follows:

Resin: ERRA-0163	Acceptable Values
Epoxide Equivalent Weight	200 - 210
% Hydrolyzable Chlorine	0 - 0.4%
Total Chlorine	0 - 0.6%
Total Volatiles, 3 hours at 436°K (325°F)	1.5%
Softening Point	317 to 429°K (111 to 133°F)
Infrared Spectrum	Structure Correlation

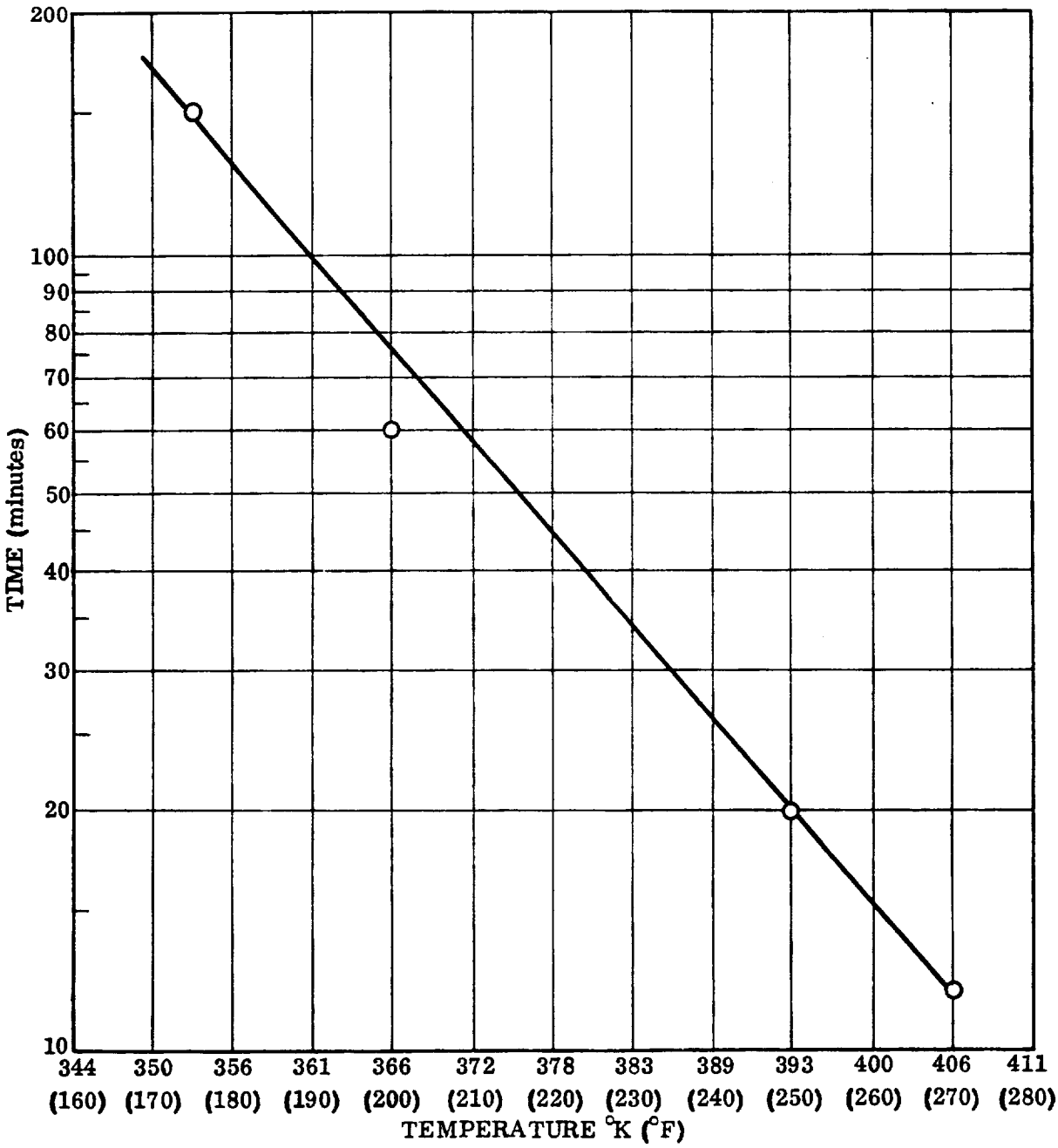


Figure 2-14. X-904 Resin Gel Time Versus Reaction Temperature

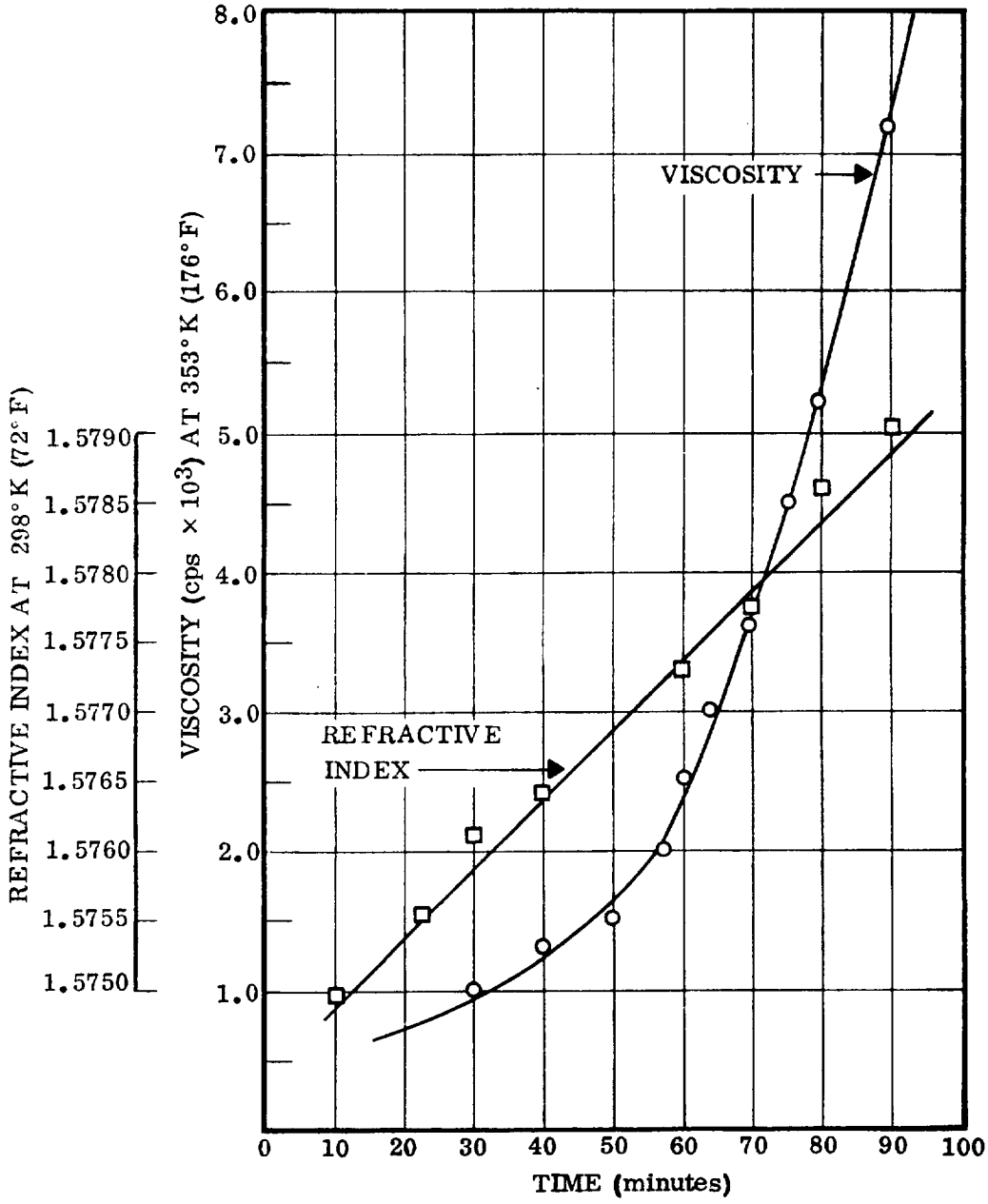


Figure 2-15. Time/Viscosity Refractive Index Relationships for the X-904 Resin System at 353°K (176°F) at 100% Resin Solids

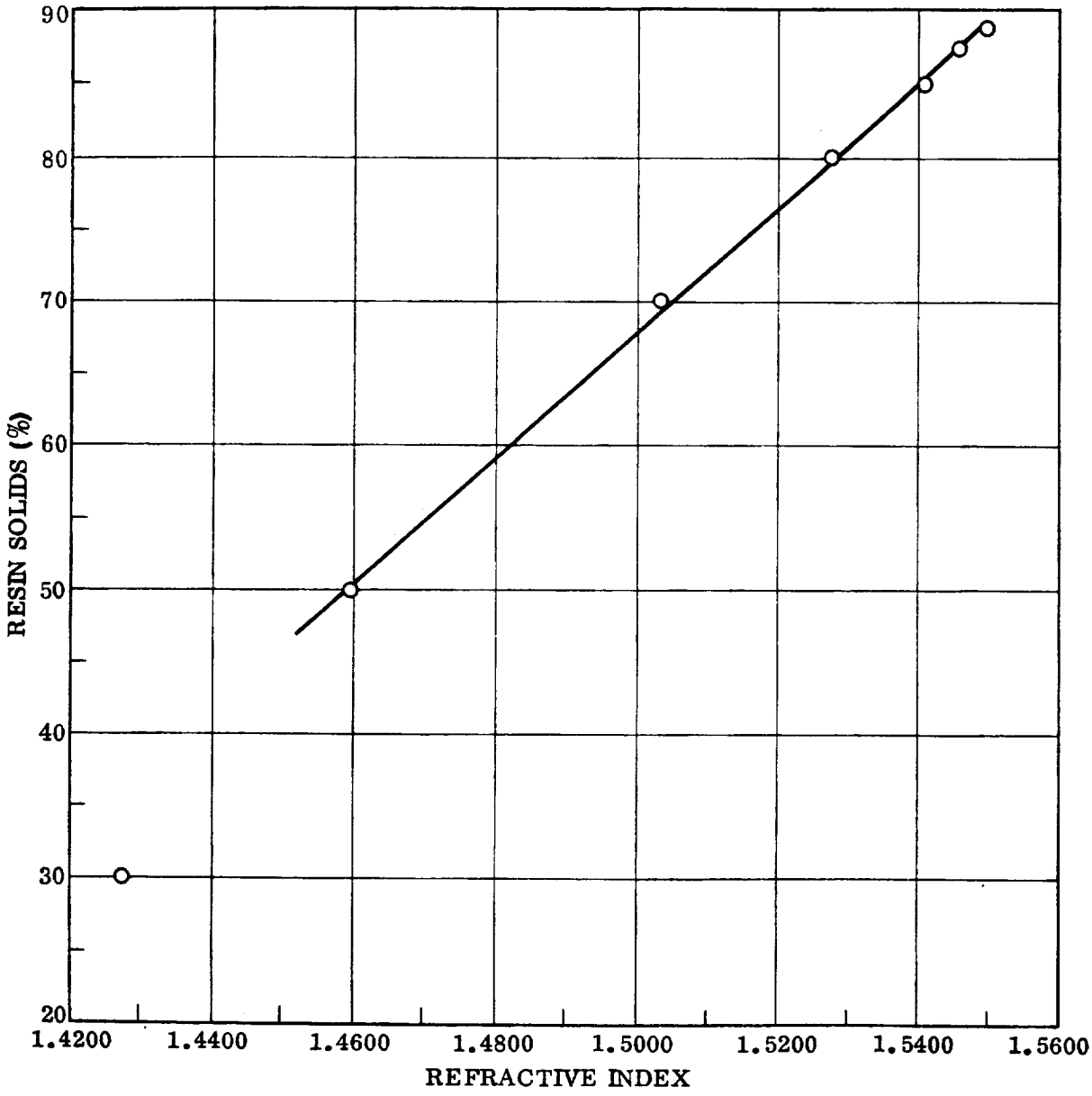


Figure 2-16. Refractive Indices of X-904 Resin Solutions (MEK Solvent)

Curing Agent - NMA	Acceptable Values
Refractive Index	1.5048 ± 0.0005
Viscosity	204 ± 5 cps
Acid Equivalent	175 ± 5 grams/eq.
Mixed Master Batch	
Viscosity	460 ± 30 cps
Gel Time at 395°K (251°F)	10 to 15 minutes
Infrared Spectrum	Structure Correlation

Master batches of the resin and curing agent may be mixed and stored (without the BDMA catalyst) at 85% resin solids or less for several weeks. The importance of storing at 278°K (40°F) or lower is clearly shown in Figure 2-17. After an autocatalytic period of 5 to 6 days, the resin started advancing rapidly at room temperature. No appreciable change was noted after two weeks at 278°K (40°F).

Figure 2-18 shows the rapid increase in viscosity and gel time resulting from room temperature storage of catalyzed X-904.

2.3.5 SPECIFIC GRAVITY OF CAST RESIN. The specific gravity of X-904 cast resin is 1.26. This value is reproducible using the following cure cycle:

1 hour at 366°K (200°F)
1 hour at 422°K (300°F)
4 hours at 464°K (375°F)
Postcure 16 hours at 464°K (375°F)

2.4 EPOXY/GRAPHITE PREPREG CHARACTERIZATION

The major portion of the epoxy/graphite prepreg characterization work in this program was performed with HT-S/X-904 prepreg. The nominal properties were: resins solids 40%, volatile content 6%, and resin flow of 23% unless specifically noted otherwise.

2.4.1 PREPREG GEL TIMES. Prepreg gel times for HT-S/X-904 are presented in Figure 2-19. The semi-logarithmic plot is a straight line as it should be for a gel time plot (reaction rate curve).

The measurements were made on small pieces of prepreg using a Fisher-Johns melting point apparatus held isothermally at the specified temperatures. The gel time of the prepreg below 360°K (200°F) increases rapidly and is in accord with the time-temperature-viscosity curves.

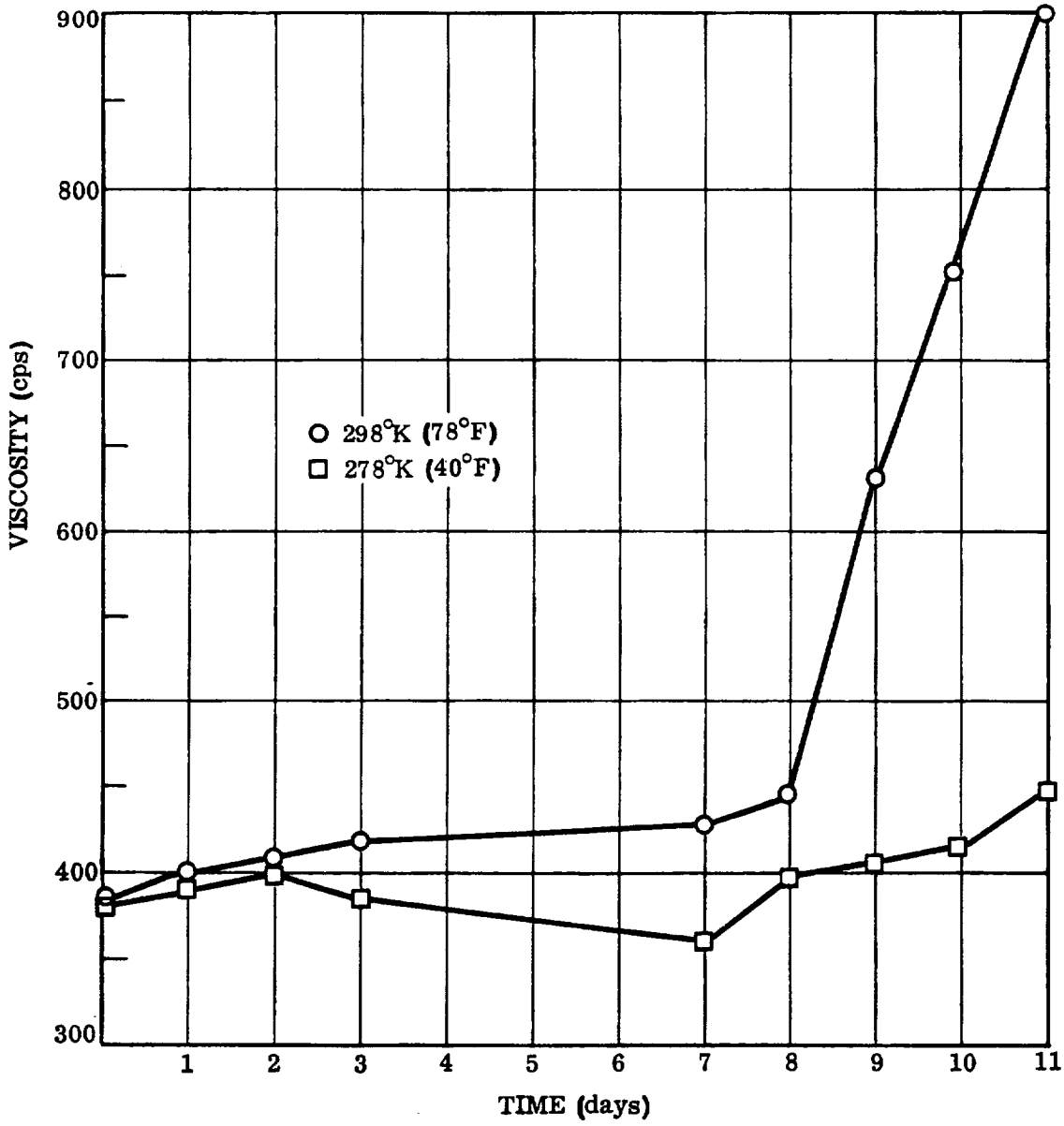


Figure 2-17. Storage Stability of X-904 Master Batches at 85% Resin Solids (No BDMA)

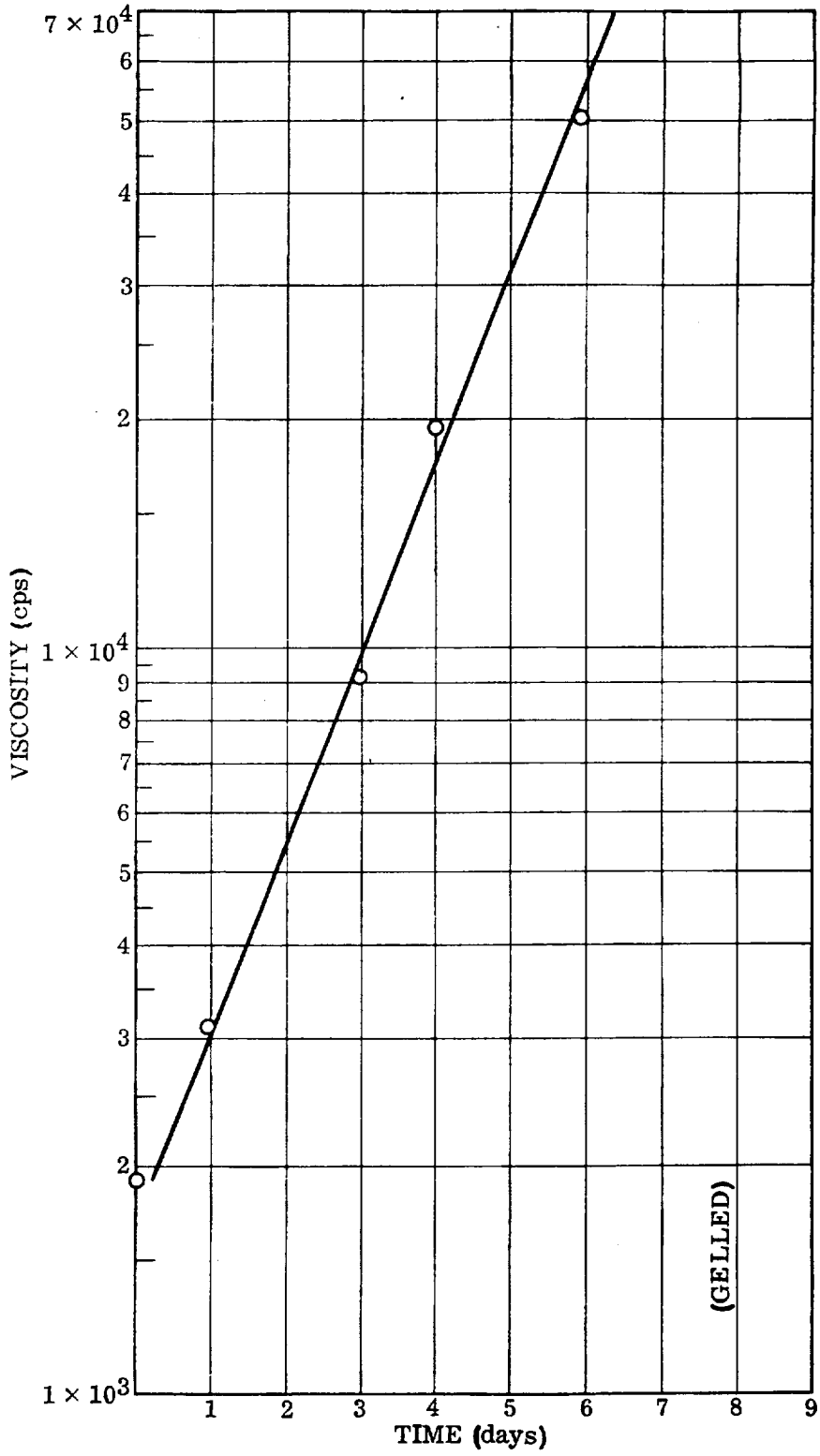


Figure 2-18. Stability at 298°K (78°F) of X-904 Premix at 85% Resin Solids (with BDMA Catalyst Added)

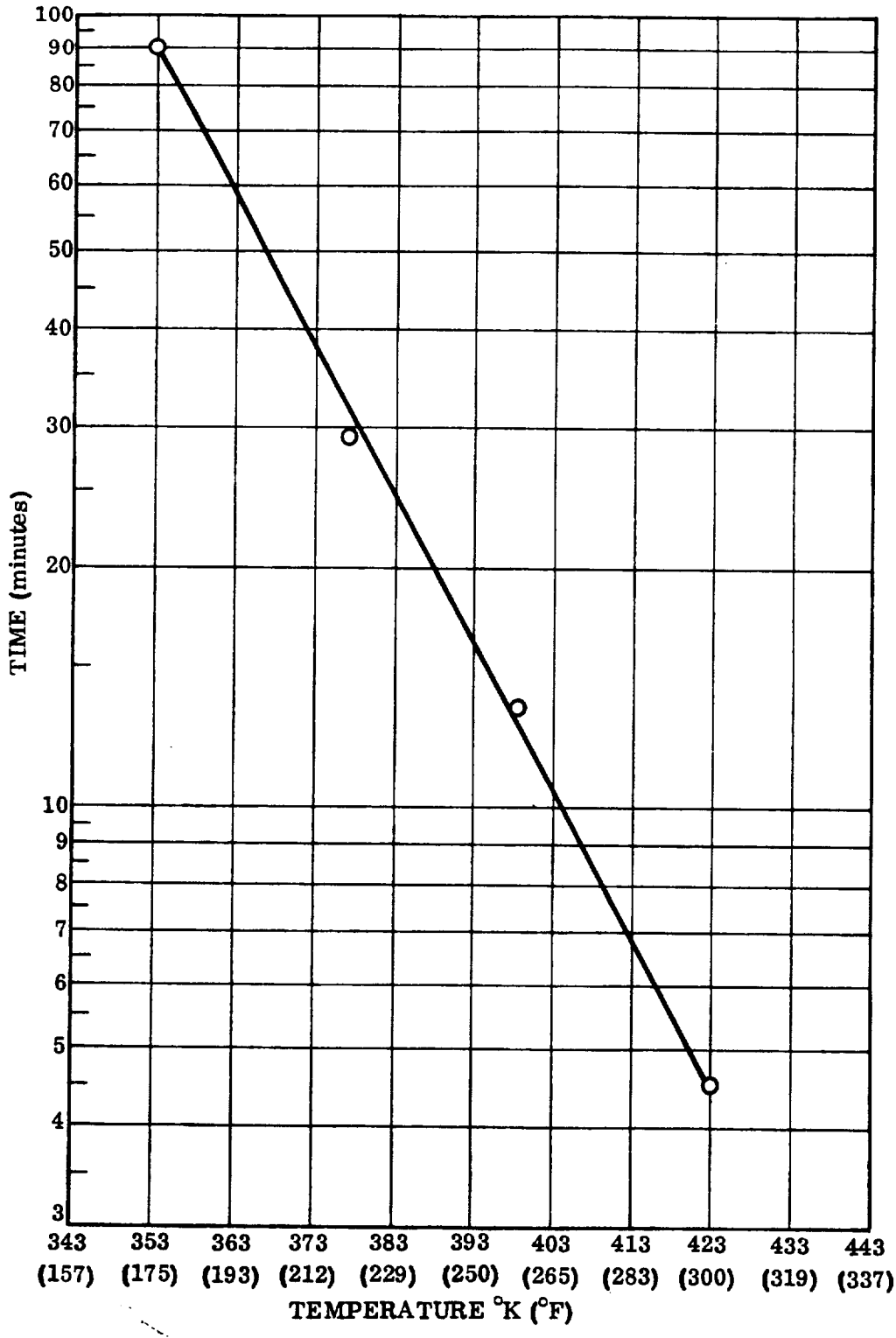


Figure 2-19. X-904/HT-S Prepreg Gel Time at Several Temperatures

2.4.2 PREPREG STORAGE STABILITY TESTS. Studies were conducted to determine the storage stability of X-904 prepreg at 297° K (75° F), 278° K (40° F), and 255° K (0° F). Drape, tack, resin flow, and prepreg gel time were measured as a function of aging. The 297° K (75° F) prepreg was stored in a dust-free but unsealed condition to simulate actual working out-time. The 278° K (40° F) and 255° K (0° F) stability tests were conducted on prepreg in sealed polyethylene bags.

The prepreg was removed from storage and allowed to warm to 297°K (75°F) before a sample was taken. Data generated in this study is presented in Tables 2-13, 2-14, and 2-15.

The 297°K (75°F) stability study indicates that a 3 to 4 day working life in regard to tack and drape is to be expected. However, the prepreg material is still moldable after one-month storage at 297°K (75°F) as indicated by the flow and gel measurements.

The three-month storage stability data for HT-S/X-904 is presented in Table 2-14. The only noticeable change is a small decrease in the percent resin flow. The tack shows some signs of changing in the last month of aging, but this could be caused by the numerous warm-up and cool-down cycles required for test sampling.

The 255°K (0°F) refrigerated storage study data is shown in Table 2-15. Again, as for the 278°K (40°F) stability study, the only real differences noted are in the tack and drape of the material.

Table 2-13. Prepreg Stability Study of HT-S/X-904 Prepreg at 297°K (75°F)

Time (days)	Resin Flow (%)	Gel Time at 443°K (337° F), minutes	Drape	Tack
1	22.5	3.3	Good	Good
4	19.8	2.8	Fair	Fair
6	19.5	2.3	Boardy	Fair-Poor
8	18.2	2	Boardy	Poor
15	19.5	1.8	Boardy	Poor
18	18.7	2.0	Boardy	Poor
20	15.8	2.0	Boardy	Poor
22	16.5	1.8	Boardy	Poor
29	15.4	1.5	Boardy	Poor
36	15.2	1.5	Boardy	Poor

Table 2-14. HT-S/X-904 Prepreg* Stability Study - Storage at 278°K (40°F)

Time	Resin Flow	Gel Time at 443°K (337°F), minutes	Drape	Tack
3 days	24.2	3.0	Good	Good
5 days	23.8	3.0	Good	Good
1 week	24.5	3.0	Good	Good
2 weeks	23.4	2.8	Good	Good
3 weeks	22.9	2.7	Good	Good
4 weeks	23.7	2.9	Good	Good
5 weeks	22.6	2.8	Good	Good
6 weeks	21.5	3.0	Good	Good
7 weeks	19.5	2.5	Good	Fair-Good
8 weeks	20.8	2.5	Good	Fair-Good
9 weeks	19.9	2.5	Good	Fair-Good
10 weeks	19.2	2.5	Good	Fair-Good
11 weeks	20.4	2.6	Good	Fair-Good
12 weeks	20.7	2.8	Good	Fair-Good

*Initial prepreg properties: Resin Solids 41.2%, Volatile Content 5.2%, Resin Flow 24.1%

2.4.3 PREPREG PARAMETERS. The following are nominal prepreg parameters for HT-S/X-904 prepreg:

Resin Solids	40 ± 3%
Volatile Content	5 ± 2%
Resin Flow	18 to 28%
Gel Time at 395°K (250°F)	11 to 16 minutes

Prepreg having these parameters and using the following cycle will routinely produce graphite composites with 30 ± 2% resin content, and 0 to 1% void content.

1. Vacuum bag using 1 ply Mauchberg bleeder per 3 ply of 0.15 mm (6 mil) prepreg.
2. Apply vacuum - heat to 366°K (200°F) at 2 to 3°K (3 to 5°F)/minute.
3. Apply 690 kN/m^2 (100 psi) - vent bag - hold one hour at 366°K (200°F).
4. Heat to 422°K (300°F) at 2 to 3°K (3 to 5°F)/minute - hold one hour.
5. Heat to 464°K (375°F) at 2 to 3°K (3 to 5°F)/minute - hold three hours.
6. Cool below 366°K (200°F) under pressure.

Table 2-15. HT-S/X-904 Prepreg Stability Study - Storage at 255°K (0°F)

Time	Resin Flow	Gel Time at 443°K (337°F), minutes	Drape	Tack
1 day	25.1	3.0	Good	Good
2 days	24.3	3.1	Good	Good
1 week	24.8	3.0	Good	Good
2 weeks	24.1	2.8	Good	Good
3 weeks	24.9	3.0	Good	Good
4 weeks	23.8	3.1	Good	Good
5 weeks	25.2	3.0	Good	Good
6 weeks	24.4	2.9	Good	Good
7 weeks	24.2	2.9	Good	Good
8 weeks	23.9	3.0	Good	Good
9 weeks	24.8	2.8	Good	Good
10 weeks	23.1	3.0	Good-Fair	Good
11 weeks	24.7	2.8	Good-Fair	Good
12 weeks	24.1	2.8	Good-Fair	Good-Fair
13 weeks	22.8	2.8	Fair	Fair
14 weeks	23.1	2.5	Fair	Fair
15 weeks	24.2	2.8	Fair-Poor	Fair-Poor
16 weeks	22.2	2.5	Fair-Poor	Poor
17 weeks	23.8	2.5	Poor	Poor
18 weeks	24.1	2.6	Poor	Poor

*Initial prepreg properties: Resin Solids 43.4%, Volatile Content 5.8%, Resin Flow 25.1%

7. Postcure 16 hours at 464°K (375°F).

The prepreg itself (HT-S/X-904) has tack and drape at room temperature. Very low resin viscosities are obtained on the resin above 343°K (157°F) and hence there is relatively high flow above this temperature. Even at room temperature, some flow will be experienced by the application of pressure. However, the determination of percent resin flow and gel time does not represent a good test for determining when the X-904 prepreg has lost its tack and drape.

2.4.4 VOLATILE CONTENT. The major prepreg volatile content arises from the NMA curing agent, which is liquid at room temperature. The resin system is specifically formulated to give the correct prepreg tack and drape without "B" staging. This was felt desirable so as to eliminate prepreg variance because of "B" stage nonuniformity.

The percent volatile obtained in a given test is quite sensitive to two parameters:

a. Surface area of prepreg exposed.

b. Concentration of catalyst.

The NMA constitutes approximately 65 to 85% of the 6% prepreg volatiles normally found in HT-S/X-904 prepreg. Using the normal composite autoclave cure, less than 1% of this "volatile" is observed. Omission of the catalyst will result in prepreg volatiles of 9 to 13% when evaluated at 436°K (325°F) for 15 minutes. This can be correlated with gel times at 395°K (251°F), which will also greatly increase with a decrease in catalyst (BDMA) concentration.

2.5 GRAPHITE-EPOXY PROCESS OPTIMIZATION

One of the major objectives of this program was to develop vacuum bag and vacuum pressure augmented cure cycles for the HT-S/X-904 graphite epoxy composite system. To meet this objective 30.5 by 30.5 cm (12 by 12 in.) by 12 ply laminates were fabricated utilizing different cure cycles. Each of these panels was then cut into 15.2 by 15.2 cm (6 by 6 in.) panels as shown in Figure 2-20 and underwent four different postcure cycles. Flexural and short beam shear tests were conducted on the post-cured panels. Also resin content, fiber volume, and specific gravity of the individual panels were determined.

2.5.1 VACUUM-PRESSURE AUGMENTED CURE STUDY (PRESS). Convair Aerospace had conducted extensive autoclave curing studies on the HT-S/X-904 system under USAF funding (Reference 2-5). Thus the primary effort investigated in this program for pressure augmented cures was aimed at press curing. The following is a list of processing techniques and cure procedures for each cure evaluated:

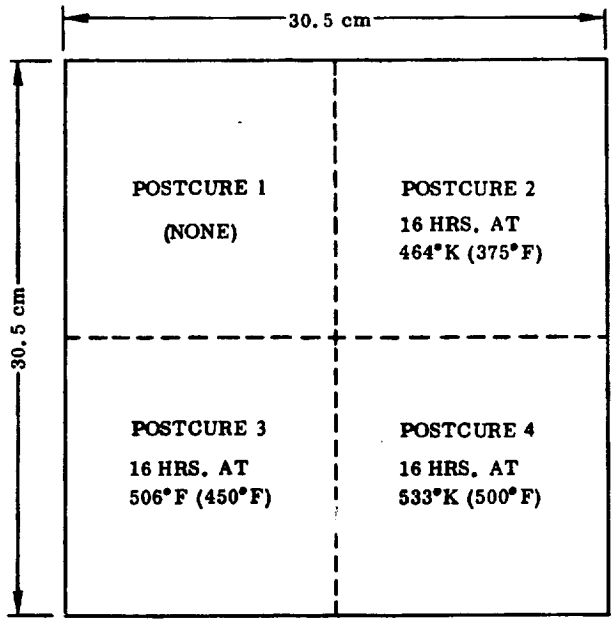


Figure 2-20. Panel Description for Cure and Postcure Studies (HT-S/X-904)

a. Material Designation: (hy-E-1311-B)
Fiber Type: HT-S (staple)
Material Form: 30.5 by 114.5 cm (12 by 45 in.) sheet
Batch No.: OC-19
Resin: X-904
Manufacturer: Fiberite

b. Fabrication Procedure: (Press Cure No. 1)
Mold Release: Teflon Film
Layup: 12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
Separator Film: Teflon-coated glass cloth, FGO-3
Bleeder: 4 plies Mauchburg paper CW-1850
Special Instructions: A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
Cure Pressure: 760 mm. (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) press pressure was applied at room temperature and maintained through the entire cure cycle and cool down below 344°K (160°F).
Cure Cycle: Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
Post Cure: Various ones.

c. Material Designation: (hy-E-1311-B)
Fiber Type: HT-S (staple)
Material Form: 30.5 by 114.5 cm (12 by 45 in.) sheet
Batch No: OC-19
Resin: X-904
Manufacturer: Fiberite

d. Fabrication Procedure: (Press Cure No. 2)
Mold Release: Teflon Film
Layup: 12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
Separator Film: Teflon-coated glass cloth, FGO-3
Bleeder: 4 plies Mauchburg paper CW-1850
Special Instructions: A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was

		used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum plus 69.0 kN/m^2 (10 psi) press pressure was applied at room temperature and maintained to 358°K (185°F), and additional 630 kN/m^2 (90 psi) was applied and maintained through the entire cure cycle and cool down below 344°K (160°F).
	Cure Cycle:	Heat to 358°K (185°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 30 minutes, heat to 393°F (250°F), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	Various ones.
e.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite
f.	Fabrication Procedure:	(Press Cure No. 3)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) press pressure was applied at room temperature and maintained through the entire cure cycle and cool down below 344°K (160°F)
	Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 464°K (375°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	Various ones.

The following four postcure cycles were evaluated:

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Each of the panels was slowly heated to 450°K (350°F) and remained at this temperature for a period of two hours prior to raising the temperature to the higher postcure temperatures. Heat raises were in increments of 28°K (50°F) with dwell times of 2 hours at each increment. Postcures 2, 3, and 4 all concluded with 16 hours at the selected postcure temperatures. The panels were all restrained during the postcure cycles either by vacuum bag pressure, weights or plates clamped together.

Longitudinal flexure, transverse flexure, and short beam shear specimens were machined from each of the panels. A minimum of three specimens were tested at each of the three test temperatures 77°K (-320°F), 297°K (75°F), and 450°K (350°F). Duplicate tests were conducted to determine resin content (percent by weight), fiber volume, and specific gravity.

2.5.2 PRESS CURE TEST RESULTS. The evaluation of the test results for the press cure study was complicated severely by the apparent degradations of graphite/epoxy systems at 450°K (350°F) due to room temperature aging. The subject of this degradation is covered in a later section in this report. As such, all the test results at 450°K (350°F) have to be disgarded, since the degradation phenomena is time dependent, the selection of the cure and postcure cycle was done by considering only the 77°K (-320°F) and 297°K (75°F) mechanical test results. Since all the panels were close in resin content and fiber volume it was decided not to normalize the data to 60% fiber volume. The press cure test data is summarized in Tables 2-16 through 2-18 as a function of cure cycle and in Tables 2-19 through 2-22 as a function of postcure. The detailed test data is included in this report as Appendix A.

The method used in evaluating the cure cycles was to rank the flexural and short beam shear strengths on the basis of 3, 2, 1, where a rank of 3 is given for the highest strength. The data obtained at 77°K (-320°F) and 297°K (75°F) was utilized in this evaluation. Using this system of ranking, cure cycle No. 2 was found to be far superior than either cure cycles No. 1 or No. 3 . The scores achieved were 53, 34-1/2, and 32-1/2 respectively. It is believed that the test results do indicate significant differences in the cure cycles evaluated. Based on this data, it is recommended that cure cycle No. 2 be used as the press cure cycle for HT-S/X-904.

The evaluation of the postcure cycle for press cured HT-S/X-904 cycles was made by evaluating the data in Tables 2-19 through 2-22 on a 4, 3, 2, 1 ranking basis. . Postcure

Table 2-16. HT-S/X-904 Cure Study — Press Cure No. 1

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)	No. 4 MN/m^2	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1147	(166.3)	1183	(171.6)	818	(118.6)	787	(113.9)
	297	(75)	1480	(214.6)	1486	(215.6)	1031	(149.5)	964	(139.8)
	450	(350)	313	(45.3)	322	(46.7)	1069	(155.0)	964	(139.8)
Transverse Flexure	297	75	81	(11.8)	82	(11.9)	51	(7.4)	43	(6.2)
Short Beam Shear	77	(-320)	113	(16.4)	74	(10.7)	43	(6.2)	34	(4.9)
	297	(75)	62	(9.0)	77	(11.1)	74	(10.7)	57	(8.3)
	450	(350)	13	(1.9)	15	(2.2)	35	(5.1)	35	(5.1)
Fiber Volume, %			64.2		62.0		60.0		63.4	
Resin Content, %			28.6		30.4		32.3		29.0	
Specific Gravity			1.58		1.56		1.54		1.56	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-17. HT-S/X-904 Cure Study — Press Cure No. 2

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)	No. 4 MN/m^2	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1211	(175.6)	1223	(177.4)	1300	(188.7)	1398	(202.7)
	297	(77)	1838	(266.6)	1672	(242.4)	1851	(268.4)	1666	(241.5)
	450	(350)	250	(36.3)	310	(45.0)	399	(57.7)	166	(24.0)
Transverse Flexure	297	(75)	66	(9.6)	86	(12.4)	61	(8.9)	44	(6.4)
Short Beam Shear	77	(-320)	101	(14.6)	84	(12.2)	61	(8.9)	42	(6.1)
	297	(75)	73	(10.6)	92	(13.3)	93	(13.5)	59	(8.6)
	450	(350)	12	(1.7)	17	(2.4)	15	(2.2)	14	(2.0)
Fiber Volume, %			64.0		60.6		65.1		67.1	
Resin Content, %			28.5		31.8		27.6		25.1	
Specific Gravity			1.59		1.53		1.58		1.56	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-18. HT-S/X-904 Cure Study Press Cure No. 3

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)	No. 4 MN/m^2	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1510	(219.0)	1560	(226.3)	1100	(159.2)	1109	(160.9)
	297	(75)	1573	(228.1)	1719	(249.2)	1552	(225.2)	1571	(227.8)
	450	(350)	274	(39.8)	316	(45.9)	939	(136.2)	1113	(161.4)
Transverse Flexure	297	(75)	65	(9.4)	63	(9.2)	48	(6.9)	34	(5.0)
Short Beam Shear	77	(-320)	70	(10.1)	70	(10.2)	21	(3.0)	52	(7.5)
	297	(75)	52	(7.6)	70	(10.1)	70	(10.2)	57	(8.3)
	450	(350)	15	(2.1)	14	(2.0)	33	(4.8)	36	(5.2)
Fiber Volume, %			65.5		63.9		63.8		65.6	
Resin Content, %			27.4		28.7		28.9		27.2	
Specific Gravity			1.58		1.57		1.54		1.57	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-19. HT-S/X-904 Cure Study — Postcure Cure No. 1

Type of Test	Test Temperature °K	Test Temperature (°F)	Press Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	1147	(116.3)	1211	(175.6)	1510	(219.0)
	297	(75)	1480	(214.6)	1838	(266.6)	1573	(228.1)
	450	(350)	312	(45.3)	250	(36.3)	275	(39.8)
Transverse Flexure	297	(75)	81	(11.8)	66	(9.6)	65	(9.4)
Short Beam Shear	77	(-320)	113	(16.4)	101	(14.6)	70	(10.1)
	297	(75)	62	(9.0)	73	(10.6)	52	(7.6)
	450	(350)	13	(1.9)	12	(1.7)	14	(2.1)
Fiber Volume, %			64.2		64.0		65.5	
Resin Content, %			28.6		28.5		27.4	
Specific Gravity			1.58		1.59		1.59	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-20. HT-S/X-904 Cure Study — Postcure Cure No. 2

Type of Test	Test Temperature °K	Test Temperature (°F)	Press Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	1183	(171.6)	1223	(177.4)	1560	(226.3)
	297	(75)	1486	(215.6)	1672	(242.4)	1718	(249.2)
	450	(350)	322	(46.7)	310	(45.0)	316	(45.9)
Transverse Flexure	297	(75)	82	(11.9)	86	(12.4)	63	(9.2)
Short Beam Shear	77	(-320)	74	(10.7)	84	(12.2)	70	(10.2)
	297	(75)	77	(11.1)	92	(13.3)	70	(10.1)
	450	(350)	15	(2.2)	17	(2.4)	14	(2.0)
Fiber Volume, %			62.0		60.6		63.9	
Resin Content, %			30.4		31.8		28.7	
Specific Gravity			1.56		1.53		1.57	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-21. HT-S/X-904 Cure Study — Postcure Cure No. 3

Type of Test	Test Temperature °K	Test Temperature (°F)	Press Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	818	(118.6)	1290	(188.7)	1097	(159.2)
	297	(75)	1031	(149.5)	1851	(268.4)	1535	(225.2)
	450	(350)	1069	(155.0)	398	(57.7)	939	(136.2)
Transverse Flexure	297	(75)	51	(7.4)	61	(8.9)	48	(6.9)
Short Beam Shear	77	(-320)	43	(6.2)	61	(8.8)	21	(3.0)
	297	(75)	74	(10.7)	93	(13.5)	70	(10.2)
	450	(350)	35	(5.1)	15	(2.1)	33	(4.8)
Fiber Volume, %			60.0		65.1		63.8	
Resin Content, %			32.3		27.6		28.9	
Specific Gravity			1.54		1.58		1.54	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-22. HT-S/X-904 Cure Study — Postcure Cure No. 4

Type of Test	Test Temperature °K	Test Temperature (°F)	Press Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	785	(113.9)	1398	(202.7)	1109	(160.9)
	297	(75)	964	(139.8)	1666	(241.5)	1571	(227.8)
	450	(350)	964	(139.8)	166	(24.0)	1113	(161.4)
Transverse Flexure	297	(75)	43	(6.2)	44	(6.4)	34	(5.0)
Short Beam Shear	77	(-320)	34	(4.9)	42	(6.1)	52	(7.5)
	297	(75)	57	(8.3)	59	(8.6)	57	(8.3)
	450	(350)	35	(5.1)	14	(2.0)	36	(5.2)
Fiber Volume, %			63.4		67.1		65.6	
Resin Content, %			29.0		25.1		27.2	
Specific Gravity			1.56		1.56		1.57	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

cycle No. 2 was clearly superior to any of the other three postcure cycles. As the postcure temperature was increased, the 77°K (-320°F) and 297°K (75°F) strength properties were severely reduced. This might be caused by oxidation occurring during the postcure or perhaps further crosslinking embrittling the polymer. The panels that were not postcured showed good strength at room and cryogenic temperatures, but it is obvious from the data in this report that an elevated postcure is required if the panels are to be used at an elevated temperature.

When the epoxy degradation problem is solved the selected cure and postcure cycles will need to be evaluated to insure that good elevated temperature results are obtained using these processing techniques. No further work was accomplished under this program in evaluating HT-S/X-904 press cure and postcure cycles.

2.5.3 VACUUM BAG CURE STUDY. The same procedure used in evaluating press cures and postcures was utilized in evaluating potential processing techniques for vacuum bag curing of HT-S/X-904. Two cure cycles with one variation in bleeder material and four postcure cycles were evaluated. The cure cycles used in this evaluation were as follows:

a.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite
b.	Fabrication Procedure:	(Vacuum Bag Cure)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3,
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum pressure was applied at room temperature and maintained through the entire cure cycle and cool down below 344°K (160°F).

	Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	Various ones.
c.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite
d.	Fabrication Procedure:	(Vacuum Bag Cure)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3,
	Bleeder:	4 plies Style 181 glass cloth
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum pressure was applied at room temperature and maintained through the entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 358°K (185°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 30 minutes, heat to 393°F (250°F), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	Various ones.
e.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite

f. Fabrication Procedure: (Vacuum Bag Cure No. 3)

Mold Release:	Teflon Film
Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
Separator Film:	Teflon-coated glass cloth, FGO-3,
Bleeder:	4 plies Mauchburg paper CW-1850
Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
Cure Pressure:	380 mm. (14.5 in.) Hg. vacuum pressure was applied at room temperature, an additional 380 mm (14.5 in.) Hg vacuum pressure was applied at 393°K (250°F) and maintained through the entire cure cycle and cool down to below 344°K (160°F).
Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
Post Cure:	Various ones.

2.5.4 VACUUM BAG TEST RESULTS. Longitudinal flexure, transverse flexure, and short beam shear tests were conducted in order to evaluate the various vacuum bag cure and postcure cycles. Here again the high-temperature degradation effect in epoxy composites made all of the 450°K (350°F) test data questionable. This was due to the length of the time between when the panel was postcured and when the tests were actually conducted. Therefore, in the evaluations of these test results only the cryogenic and room temperature data is utilized. The vacuum bag test results are summarized in Tables 2-23 through 2-25 as a function of cure cycle and in Tables 2-26 through 2-29 as a function of postcure cycle. The detailed test data is presented in Appendix A.

In evaluating the test results as a function of cure cycle, the cycle No. 2 was clearly superior to cure cycles No. 1 and No. 3. The only difference between cure cycles No. 1 and No. 2 is that the bleeder in cycle No. 2 is style 181 glass cloth and in cure cycle No. 1 the bleeder was Mauchburg paper CW-1850. Cure cycle No. 2 is the recommended vacuum bag cure and is also the same as the recommended press cure. In general the strength values achieved with the vacuum bag cures were 20 to 30% lower than the values obtained from the press cured panels. An interesting point is that the

Table 2-23. HT-S/X-904 Cure Study — Vacuum Bag Cure No. 1

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m²	No. 1 (ksi)	No. 2 MN/m²	No. 2 (ksi)	No. 3 MN/m²	No. 3 (ksi)	No. 4 MN/m²	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1427	(207.0)	1474	(213.7)	874	(126.7)	919	(133.3)
	297	(75)	988	(143.3)	1014	(147.0)	856	(124.1)	749	(108.6)
	450	(350)	533	(77.3)	650	(94.2)	439	(63.7)	408	(59.2)
Transverse Flexure	297	(75)	37	(5.3)	35	(5.1)	28	(4.0)	22	(3.2)
Short Beam Shear	77	(-320)	42	(6.1)	53	(7.7)	44	(6.4)	34	(5.0)
	297	(75)	36	(5.2)	40	(5.8)	32	(4.7)	30	(4.3)
	450	(350)	19	(2.7)	22	(3.2)	25	(3.6)	21	(3.1)
Fiber Volume, %			62.1		59.5		61.6		62.1	
Resin Content, %			30.2		32.7		30.7		30.7	
Specific Gravity			1.49		1.49		1.47		1.46	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-24. HT-S/X-904 Cure Study — Vacuum-Bag Cure No. 2

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)	No. 4 MN/m^2	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1378	(199.8)	1057	(153.3)	1148	(166.4)	1318	(191.2)
	297	(75)	1249	(181.1)	1352	(196.1)	1241	(180.0)	1490	(216.1)
	450	(350)	372	(53.9)	542	(78.5)	754	(109.3)	990	(143.5)
Transverse Flexure	297	(75)	28	(4.1)	22	(3.2)	26	(3.7)	24	(3.5)
Short Beam Shear	77	(-320)	48	(6.9)	43	(6.2)	51	(7.4)	40	(5.8)
	297	(75)	48	(6.9)	43	(6.2)	39	(5.7)	46	(6.6)
	450	(350)	17	(2.4)	20	(2.9)	30	(4.3)	30	(4.4)
Fiber Volume, %			58.8		60.0		59.3		61.7	
Resin Content, %			33.2		32.0		32.7		30.6	
Specific Gravity			1.48		1.49		1.49		1.50	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-25. HT-S/X-904 Cure Study — Vacuum Bag Cure No. 3

Type of Test	Test Temperature °K	Test Temperature (°F)	Postcure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)	No. 4 MN/m^2	No. 4 (ksi)
Longitudinal Flexure	77	(-320)	1374	(199.3)	1144	(165.9)	1078	(156.3)	986	(143.0)
	297	(75)	1353	(196.3)	1140	(165.3)	1333	(193.3)	1221	(177.2)
	450	(350)	626	(90.8)	613	(88.9)	730	(105.9)	843	(122.4)
Transverse Flexure	297	(75)	28	(4.0)	25	(3.6)	23	(3.4)	23	(3.4)
Short Beam Shear	77	(-320)	42	(6.1)	41	(6.0)	50	(7.3)	46	(6.6)
	297	(75)	46	(6.6)	39	(5.6)	41	(5.9)	45	(6.5)
	450	(350)	25	(3.6)	26	(3.7)	30	(4.3)	30	(4.4)
Fiber Volume, %			56.5		57.5		58.3		57.6	
Resin Content, %			35.1		34.4		33.7		34.5	
Specific Gravity			1.44		1.47		1.46		1.47	

Postcure Cycles

1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)
4. 16 hours at 533°K (500°F)

Table 2-26. HT-S/X-904 Cure Study — Postcure No. 1

Type of Test	Test Temperature °K	Test Temperature (°F)	Vacuum-Bag Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	1427	(207.0)	1376	(199.8)	1374	(199.3)
	297	(75)	988	(143.3)	1249	(181.1)	1353	(196.3)
	450	(350)	533	(77.3)	371	(53.9)	626	(90.8)
Transverse Flexure	297	(75)	37	(5.3)	28	(4.1)	28	(4.0)
Short Beam Shear	77	(-320)	42	(6.1)	48	(6.9)	42	(6.1)
	297	(75)	36	(5.2)	48	(6.9)	46	(6.6)
	450	(350)	19	(2.7)	17	(2.4)	25	(3.6)
Fiber Volume, %			62.1		58.8		56.5	
Resin Content, %			30.2		33.2		35.1	
Specific Gravity			1.49		1.48		1.44	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-27. HT-S/X-904 Cure Study — Postcure No. 2

Type of Test	Test Temperature °K	Test Temperature (°F)	Vacuum-Bag Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	1474	(213.7)	1055	(153.3)	1144	(165.9)
	297	(75)	1014	(147.0)	1352	(196.1)	1140	(165.3)
	450	(350)	650	(94.2)	541	(78.5)	612	(88.9)
Transverse Flexure	297	(75)	35	(5.1)	22	(3.2)	25	(3.6)
Short Beam Shear	77	(-320)	53	(7.7)	43	(6.2)	41	(6.0)
	297	(75)	40	(5.8)	43	(6.2)	39	(5.6)
	450	(350)	22	(3.2)	20	(2.9)	26	(3.7)
Fiber Volume, %			59.5		60.0		57.5	
Resin Content, %			32.7		32.0		34.4	
Specific Gravity			1.49		1.49		1.47	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-28. HT-S/X-904 Cure Study — Postcure No. 3

Type of Test	Test Temperature °K	Test Temperature (°F)	Vacuum-Bag Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	874	(126.7)	1148	(166.4)	1078	(156.3)
	297	(75)	856	(124.1)	1241	(180.0)	1333	(193.3)
	450	(350)	439	(63.7)	754	(109.3)	730	(105.9)
Transverse Flexure	297	(75)	28	(4.0)	26	(3.7)	23	(3.4)
Short Beam Shear	77	(-320)	44	(6.4)	51	(7.4)	50	(7.3)
	297	(75)	32	(4.7)	39	(5.7)	41	(5.9)
	450	(350)	25	(3.6)	28	(4.0)	30	(4.3)
Fiber Volume, %			61.6		59.3		58.3	
Resin Content, %			30.7		32.7		33.7	
Specific Gravity			1.47		1.49		1.46	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

Table 2-29. HT-S/X-904 Cure Study — Postcure No. 4

Type of Test	Test Temperature °K	Test Temperature (°F)	Vacuum-Bag Cure Cycles No. 1 MN/m^2	No. 1 (ksi)	No. 2 MN/m^2	No. 2 (ksi)	No. 3 MN/m^2	No. 3 (ksi)
Longitudinal Flexure	77	(-320)	919	(133.3)	1318	(191.2)	986	(143.0)
	297	(75)	749	(108.6)	1490	(216.1)	1221	(177.2)
	450	(350)	408	(59.2)	990	(143.5)	843	(122.4)
Transverse Flexure	297	(75)	22	(3,2)	24	(3.5)	23	(3.4)
Short Beam Shear	77	(-320)	34	(5.0)	40	(5.8)	46	(6.6)
	297	(75)	30	(4.3)	46	(6.6)	45	(6.5)
	450	(350)	21	(3.1)	30	(4.4)	30	(4.4)
Fiber Volume, %			62.1		61.7		57.6	
Resin Content, %			30.7		30.6		34.5	
Specific Gravity			1.46		1.50		1.47	

Postcure Cycles
1. No postcure
2. 16 hours at 464°K (375°F)
3. 16 hours at 506°K (450°F)

composites cured by the two different techniques were found to be equivalent in load-carrying capability. The reason that the stress levels are significantly different is that the press cured panels were significantly thinner.

Postcures No. 1 and No. 2 were found to give higher test results than either postcures No. 3 and No. 4. Postcure No. 2 is recommended as the postcure cycle since high temperature properties, 450°K (350°F), will be required when the epoxy degradation problem is solved. As the postcure temperature was increased both the cryogenic and room temperature strengths decreased. As noted earlier this is probably due to oxidation of the composite or cross linking of the epoxy resin. The recommended postcure is also identical to the postcure recommended for press cured panels.

2.5.5 AUTOCLAVE CURE AND POSTCURE STUDY. Autoclave curing studies on the HT-S/X-904 graphite epoxy systems had previously been conducted by Convair Aerospace (Reference 2-5) and as such this portion of the cure and postcure study was not as extensive as the press and vacuum studies. The test data developed on previously sponsored work (Reference 2-5) is presented in Table 2-30 and new data generated on this program is tabulated in Table 2-31. A limitation of four days between the time a panel was postcured and the high temperature testing of specimens from these panels was utilized for the data generated on the autoclave study on this program. This minimizes the epoxy degradation effect and accounts for the higher test numbers at 450°K (350°F). The cure and postcure cycles utilized for fabricating the panels are as follows:

a.
Material Designation:	(hy-E-1311-B)
Fiber Type:	HT-S (staple)
Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
Batch No.:	OB 59
Resin:	X-904
Manufacturer:	Fiberite

b.
Fabrication Procedure:	Autoclave Cure OB 59-1
Mold Release:	Teflon Film
Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
Separator Film:	Teflon-coated glass cloth, FGO-3
Bleeder:	4 plies Mauchburg paper CW-1850
Special Instructions:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as s pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.

Table 2-30. Autoclave Cure Cycle Test Results (HT-S/X-904)

Type of Test	Test Temperature °K	Test Temperature (°F)	Panel Number OA81 MN/m^2	OA81 (ksi)	OB59-1 MN/m^2	OB59-1 (ksi)	OB59-2 MN/m^2	OB59-2 (ksi)
Longitudinal Flexure	77	(-320)	1338	(193.9)	1448	(210.3)	1613	(234.2)
	297	(75)	1620	(235.0)	1724	(249.5)	1744	(253.1)
	450	(350)	855	(123.5)	876	(127.3)	758	(110.3)
Transverse Flexure	77	(-320)	104	(15.1)	93	(13.5)	127	(18.4)
	450	(350)	33	(4.8)	18	(2.6)	18	(2.6)
Interfiber Shear	77	(-320)	78	(11.3)	88	(12.8)	83	(12.0)
	450	(350)	37	(5.4)	42	(6.1)	53	(7.7)
Specific Gravity			1.54		1.56		1.55	
Resin Content, %			34.6		33.3		35.0	
Fiber Volume, %			57.3		58.7		56.9	

Table 2-31. Modified Autoclave Cure Cycle Test Results (HT-S/X-904)

Type of Test	Test Temperature °K (°F)	Panel No. C-1 MN/m^2 (ksi)	MB-1 MN/m^2 (ksi)	A-1NP MN/m^2 (ksi)	A-1P MN/m^2 (ksi)	A-2 MN/m^2 (ksi)	OC16-1-15 MN/m^2 (ksi)
Long. Flexure	77 (-320)	— —	— —	— —	— —	1572 (228.2)	1441 (209.1)
	297 (75)	1400 (202.8)	1358 (197.1)	1786 (259.1)	1731 (251.3)	1613 (234.3)	1572 (228.4)
	393 (250)	— —	— —	— —	— —	1303 (189.1)	1117 (162.2)
	450 (350)	534 (77.4)	724 (104.7)	678 (98.4)	965 (104.3)	1027 (148.7)	793 (115.1)
Specific Gravity	—	1.56	—	1.57	1.57	1.57	—
Resin Content, %	—	30.0	—	33.4	31.4	30.5	—
Fiber Volume, %	—	62.3	—	58.9	61.0	61.2	—

	Cure Pressure:	760 mm. (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) press pressure was applied at room temperature and maintained through the entire cure cycle and cool down below 344°K (160°F).
	Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	None
c.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No:	OA-81
	Resin:	X-904
	Manufacturer:	Fiberite
d.	Fabrication Procedure:	Autoclave Cure OA-81
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGC-3
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting) and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum plus 69.0 kN/m^2 (10 psi) press pressure was applied at room temperature and maintained to 358°K (185°F), and additional 630 kN/m^2 (90 psi) was applied and maintained through the entire cure cycle and cool down below 344°K (160°F).
	Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool to below 344°K (160°F) under pressure.
	Postcure Cycle:	One-half hour at 393°K (250°F), 1/2 hour at 422°K (300°F), 1/2 hour at 450°K (350°F) and 16 hours at 464°K (375°F).

e.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OB-59
	Resin:	X-904
	Manufacturer:	Fiberite
f.	Fabrication Procedure:	Autoclave Cure OB 59-2
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum pressure was applied at room temperature, an additional 690 kN/m^2 (100 psi) pressure was applied at 362°K (195°F) after a 1/2 hour hold at this temperature and maintained through the entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 362°K (195°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, heat to 436°K (325°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1/2 hour, heat to 464°K (375°F) and hold for 4-1/2 hours.
	Postcure Cycle:	None.
g.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OB-66
	Resin:	X-904
	Manufacturer:	Fiberite
h.	Fabrication Procedure:	Autoclave Cure Panel C-1
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	4 plies Mauchburg paper CW-1850

	Special Instructions:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) press pressure was applied at room temperature and maintained through the entire cure cycle and cool down below 344°K (160°F).
	Cure Cycle:	Heat to 393°K (250°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, then heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to to below 344°K (160°F) under pressure.
	Post Cure:	One-half hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F) and 16 hours at 464°K (375°F).
i.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite
j.	Fabrication Procedure:	Autoclave Cure MB-1 Dielectric Monitor
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3,
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg. vacuum was applied at room temperature, an additional 690 kN/m^2 (100 psi) pressure was applied at 407°K (275°F) and maintained through the entire cure cycle and cool down to below 344°K (160°F).

	Cure Cycle:	Heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 2 hours, and cool down to below 344°K (160°F) under pressure.
	Post Cure:	One-half hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F) and 16 hours at 464°K (375°F).
k.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OC-19
	Resin:	X-904
	Manufacturer:	Fiberite
l.	Fabrication Procedure:	Autoclave Cure A-1, A-2
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg. vacuum pressure was applied at room temperature, an additional 690 kN/m^2 (100 psi) pressure was applied at 366°K (200°F) and maintained through the entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 366°K (200°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 20 minutes, heat to 422°K (300°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, heat to 464°K (375°F) and hold for 4 hours.
	Post Cure:	One-half hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F) and 20 hours at 464°K (375°F).
m.	Material Designation:	(hy-E-1311-B)
	Fiber Type:	HT-S (continuous)
	Material Form:	30.5 by 114.5 cm (12 by 45 in.) sheet
	Batch No.:	OC-16
	Resin:	X-904
	Manufacturer:	Fiberite

n.	Fabrication Procedure:	Autoclave Cure OC16-1-15
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	4 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5 by 30.5 by 0.64 cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and was enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum pressure was applied at room temperature, an additional 690 kN/m^2 (100 psi) pressure was applied at 366°K (200°F) and maintained through the entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 366°K (200°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 20 minutes, heat to 422°K (300°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, heat to 464°K (375°F) and hold for 4 hours.
	Post Cure:	One-half hour at 393°K (250°F), 1 hour at 422°K (300°F), 1 hour at 450°K (350°F) and 20 hours at 464°K (375°F).

Cure cycle MB-1 is a dielectric monitor cure in which the complex electrical properties of the epoxy matrix was monitored. By monitoring these changes gellation of the resin can be established and the proper point for pressure to be applied for a particular panel can be readily identified. Figure 2-21 is the actual curve obtained from the dielectric monitor recorder which shows that the pressure was applied at 407°K (275°F). This is a very effective tool in developing new cure cycles or checking the quality of a particular batch of material but finds limited use in a production situation.

2.5.6 AUTOCLAVE TEST DATA. The data developed previously by Convair Aerospace (Table 2-30) indicated that the HT-S/X-904 graphite/epoxy system was fairly insensitive to the initial cure cycle, but as expected significant increases in oriented properties such as transverse flexure at 450°K (350°F) were obtained by postcuring. The HT-S/X-904 system also showed that this particular system retains a high percentage of its room temperature strength at cryogenic temperatures unlike other high temperature epoxy resin systems. The revised cure cycle utilized on panels A-1P, A-2, and OC16-1-15 is the recommended cure and postcure cycle. The primary difference between the recommended cure cycle and the others evaluated is that the initial cure temperature reaches 464°K (375°F) as compared to 450°K (350°F) for the other cure cycles. This probably increases the heat distortion temperature of the cured composite, thus giving higher strength properties at 450°K (350°F).

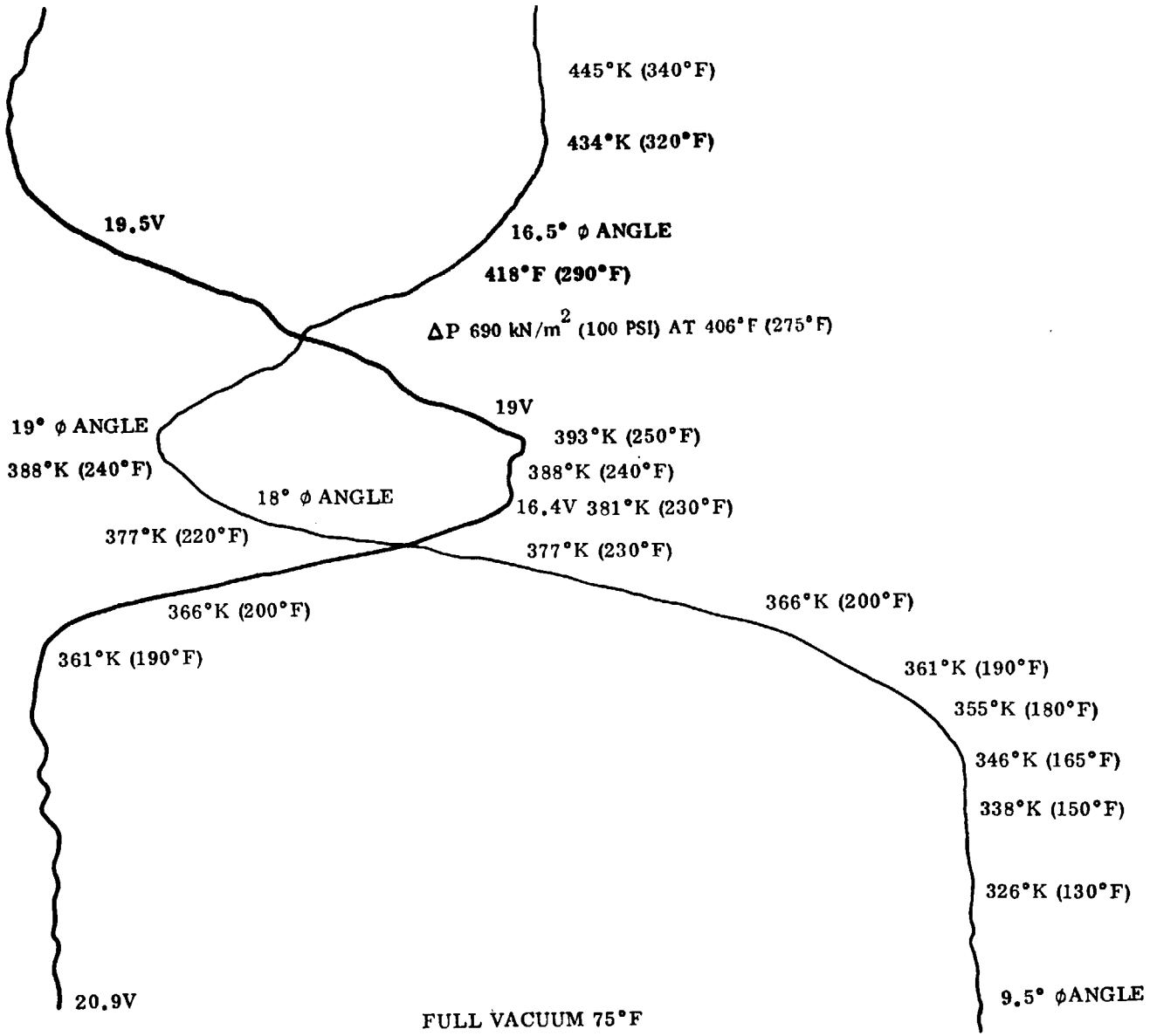


Figure 2-21. Dielectric Monitor Cure Curve for HT-S/X-904

2.6 ELEVATED TEMPERATURE STABILITY OF EPOXY-MATRIX COMPOSITES

Investigation of problems associated with the high-temperature resistance of Fiberite's X-904 epoxy resin has led to discovery of a general elevated temperature stability problem associated with epoxy-matrix composites. The discovery was made jointly by Convair Aerospace and Fiberite in their attempts to resolve difficulties with HT-S/X-904 prepreg being used in support of NAS8-26198 and F33615-70-C-1442.

The problem manifested itself initially by unusually low longitudinal flexural strength at 450°K (350°F) as compared to data obtained earlier in a screen program. This was found to be the case in tests conducted at both Convair Aerospace and Fiberite.

Whereas in the past longitudinal flexure strength of HT-S/X-904 often approached or exceeded 1034 MN/m^2 (150 ksi) when tested after either 10 minutes or 1/2 hour at 450°K (350°F), recent batches failed below 600 kN/m^2 (100 ksi). It was initially believed that the poorer thermal stability resulted from some inherent change in the basic resin.

From a historical standpoint, the X-904 formula consisted of Union Carbide's ERRA 0153 (aromatic polyglycidylether), NMA, BDMA, and MEK. Several months prior to discovery of the problem, Union Carbide went from a small-batch process to a large-scale process and changed the number of the resin from ERRA 0153 to ERRA 0163. Fiberite was assured that the resin would remain identical.

When the thermal stability problem was first discovered, analysis of the resins showed that the ERRA 0153 had epoxide values in the order of 202 to 205, while the early batches of ERRA 0163 had epoxide values of approximately 225. In additon, the batches of ERRA 0163 had significantly higher chlorine contents. It was felt that these changes would result in lower crosslink density due to greater spacing of functional groups, which in turn would result in a cured resin having a lower thermal distortion temperature.

To check this theory, laminates were prepared and tested. However, it was found that a new batch received from Union Carbide having a low epoxide value and relatively low chlorine content also had inherently poor thermal stability. Two new panels (14390 and 14391) made from batch C-2 of ERRA 0163 gave the longitudinal flexure properties shown in Table 2-32.

The C-2 batch of resin had been used earlier for some of the prepreg that gave low 450°K (350°F) longitudinal flexure properties, and therefore other variables were evaluated such as the purity of NMA and BDMA. However, no apparent reason could be determined for the earlier poor results.

Table 2-32. Longitudinal Flexure Properties (Panels 14390 and 14391)

Panel No.	Test Temperature °K	Test Temperature °F	Longitudinal Flexure Strength MN/m^2	Longitudinal Flexure Strength (ksi)	Horizontal Shear Strength MN/m^2	Horizontal Shear Strength (ksi)
14390	297	(75)	1496	(217.2)	84	(12.2)
	450	(350)	945	(137.2)	46	(6.7)
14391	297	(75)	1593	(231.0)	88	(12.7)
	450	(350)	1000	(145.2)	50	(7.2)

Late in October 1970, low values again were observed on the 450°K (350°F) longitudinal flexure strength of HT-S/X-904. Fiberite was contacted, and their feeling was that the problem could be remedied by changing the cure cycle to give a maximum cure temperature of 464°K (375°F) rather than the standard 450°K (350°F) cure. This cure cycle is identical to the recommended autoclave cure cycle. Two panels were fabricated: OC-19-A-2 and OC-16-1-15. The OC-19-A-2 panel used short HT-S fibers and was fabricated on Contract NAS8-26198 and the OC-16-1-15 panel used continuous HT-S fibers and was fabricated on AF Contract F33615-70-C-1442. Specimens cut from these two panels were tested at 77°K (-320°F), 200°K (-100°F), 297°K (75°F), 366°K (200°F), 393°K (250°F), 422°K (300°F), 450°K (350°F), and 477°K (400°F). The data is tabulated in Table 2-33 and plotted in Figure 2-22.

Although the test data for panels OC-19-A-2 and OC-16-1-15 were adequate at 450°K (350°F) for both programs, the strength-versus-temperature plot was disturbing. Since at the high-temperature end of the plot the strength drops rapidly with increase in temperature, an accurate measurement of the test temperature is extremely important. A 10 degree error at 450°K (350°F) could result in a 5 percent deviation in strength. It was therefore decided to rerun some of the panels which had given low flexural strengths at 450°K (350°K). Rerun of test panels confirmed previously low test values. Rerun of some early test panels, which had given some acceptable test values, resulted in greatly reduced values. Early panels made with some other resins and fibers were also rerun. Tables 2-34 through 2-36 summarize the data obtained on the X-904, 3002, and 1004 high-temperature epoxy resin systems (Reference 2-9). Included are data obtained at Convair Aerospace, Fiberite, Hercules, and Whittaker. All data is on longitudinal flexure strength of unidirectional laminates.

Figure 2-23 is a plot of the HT-S/X-904 data presented in Table 2-34 (Reference 2-9). Similar plots could be prepared for HT-S/3002 and HT-S/1004 aging data, although there was not as much data on the latter two systems. The general trend shows an approximate reduction in strength of 20 to 50 percent. The shape of the data plot can be very misleading since only two data points are available for each test laminate. When plotted on semilog paper the data in Figure 2-23 appears as a series of straight lines having

Table 2-33. Longitudinal Flexural Strength of HT-S/X-904, Panel OC-16-1-15

Test Temperature		Longitudinal Flexure Strength*	
°K	°F	MN/m^2	(ksi)
77	(-320)	1441	(209.1)
200	(-100)	1462	(211.7)
297	(75)	1572	(228.4)
366	(200)	1220	(176.8
393	(250)	1117	(162.2)
422	(300)	1000	(145.4)
450	(350)	793	(115.1)
477	(400)	444	(63.7)
*Average of 3 tests.			

approximately the same negative slope. Aging time at RT, humidity during the RT aging period, and the time period from initial panel manufacture to initial testing can all have significant influence on the data obtained and the shape of the resulting plot. Normal scatter in 450°K (350°F) flexural strength testing can also be a problem.

After much test procedure evaluation and retesting of old panels, the problem was recognized as a loss of flexural strength at 450°K (350°F) resulting from RT aging under laboratory environments. Confirmation from other test sources such as Fiberite, Hercules, and Whittaker helped to define the problem sooner. Horizontal shear strength at 450°K (350°F) appears to be affected in a similar manner. Table 2-37 summarizes some of the early data that was available (Reference 2-9).

Additional flexural data is available on other epoxy resin systems, both high-temperature resistant and general purpose. This data is summarized in Table 2-38 (Reference 2-9). There is too little data available on general-purpose epoxy systems and their retension of 352°K (180°F) properties after RT aging.

Several possible mechanisms have been proposed by various investigators for the unstable behavior of the epoxy matrix composites. The most likely mechanisms are thermal relaxation (creep of resin, fiber finish, etc.), hydrolysis effects, oxidative degradation, and fiber surface effects.

Some brief experiments with degraded panels indicate that the 450°K (350°F) flexural strength can be partially or completely regenerated by heating or by exposure to vacuum.

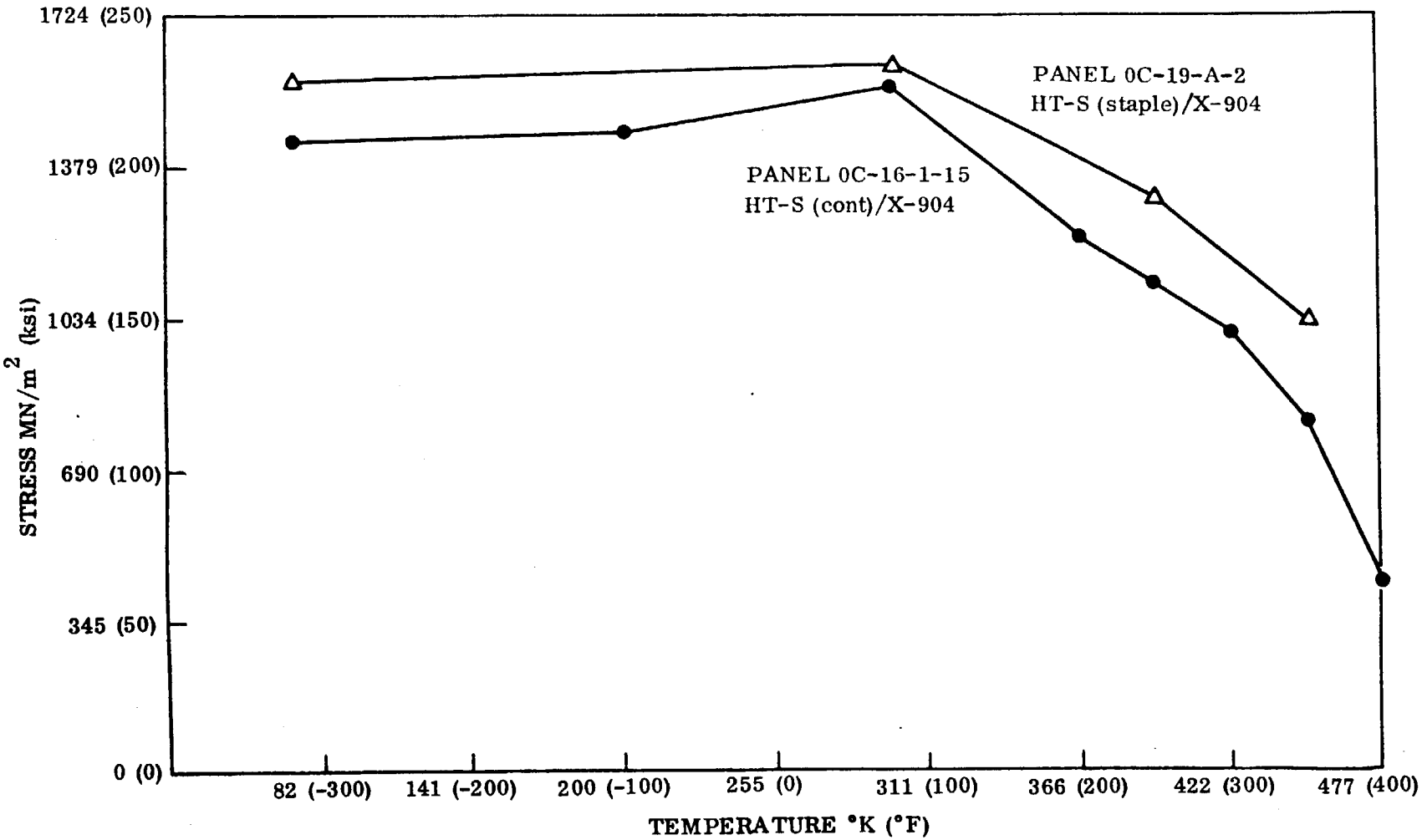


Figure 2-22. Longitudinal Flexural Strength Versus Temperature for HT-S/X-904

Table 2-34. Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing X-904 Resin

Fabricator	Fiber	Mfg. Date	Panel No.	Initial Test Date	Initial Strength MN/m^2	(ksi)	Retest Date	Retest Strength MN/m^2	(ksi)
Convair	HT-S (staple)	5-20-70	OA-81	6-10-70	855	(124)	10-28-70	486	(69.7)
Convair	HT-S (staple)	7-28-70	OB-59-1*	8-11-70	876	(127)	10-28-70	647	(93)
Convair	HT-S (staple)	7-30-70	OB-59-2*	8-13-70	758	(110)	10-28-70	556	(79.8)
Convair	HT-S (staple)	10-15-70	OC-19-1-MB	10-21-70	724	(105)	11-4-70	444	(63.7)
Convair	HT-S (cont.)	10-22-70	OC-16-1-15	10-26-70	793	(115)	11-2-70	807	(117)
Fiberite	HT-S (staple)	9-21-70	OC-19	9-23-70	1069	(155)	10-27-70	779	(113)
Fiberite	HT-S (staple)	9-10-70	14398-1	9-18-70	876	(127)	10-27-70	717	(104)
Fiberite	HT-S (staple)	9-15-70	14000E	9-15-70	883	(128)	10-29-70	542	(78)
Convair	GY-70 (cont.)	7-22-70	OB55-1*	8-14-70	690	(99.5)	10-30-70	731	(105)
Convair	GY-70 (cont.)	7-23-70	OA80-1-1	8-14-70	479	(69.2)	10-30-70	339	(48.6)

* No post cure.

Table 2-35. Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing 3002 Resin

Fabricator	Fiber	Mfg. Date	Panel No.	Initial Test Date	Initial Strength MN/m^2	(ksi)	Retest Date	Retest Strength MN/m^2	(ksi)
Convair	HT-S (cont.)	6-11-70	X-201-40	6-18-70	1276	(185)	10-29-70	889	(129)
Convair	HT-S (cont.)	8-25-70	T-2	9-17-70	1089	(158)	10-27-70	690	(100)
Convair	HT-S (cont.)	8-25-70	T-3	9-17-70	1089	(158)	10-27-70	1110	(161)
Fiberite	HT-S (cont.)	8-14-70	14386A	8-15-70	1158	(168)	10-29-70	834	(121)
Fiberite	HT-S (cont.)	8-14-70	R-3**	8-15-70	187	(120)	10-29-70	633	(91)
Hercules	HT-S (cont.)	6-1-70	X-201-60C-1	6-2-70	1027	(149)	11-4-70	528	(76)
Hercules	HT-S (cont.)	6-1-70	X173-83-B	6-2-70	1131	(164)	11-4-70	690	(100)
Hercules	HT-S (cont.)	6-1-70	X201-60-B	6-2-70	862	(125)	11-4-70	654	(94)

* One specimen only available for retest.
** No post cure.

Table 2-36. Longitudinal Flexural Strength at 450°K (350°F) as a Function of Room Temperature Aging for Composites Containing 1004 Resin

Fabricator	Fiber	Mfg. Date	Panel No.	Initial Test Date	Initial Strength MN/m^2	(ksi)	Retest Date	Retest Strength MN/m^2	(ksi)
Convair	HT-S (cont.)	6-26-70	1	7-9-70	1048	(152)	10-29-70	827	(120)
Convair	HT-S (cont.)	7-28-70	0042-2	8-17-70	599	(85.7)	10-30-70	556	(80.3)
Convair	HT-S (cont.)	7-27-70	0042-1*	8-17-70	689	(98.6)	10-30-70	472	(67.6)
Convair	M-II (Meter)	5-29-70	0019-2	6-4-70	1110	(161)	10-29-70	807	(117)
Whittaker	GY-70 (cont.)	12-1-69	1	12-3-69	689	(99)	10-30-70	311	(45)
Convair	GY-70 (cont.)	7-21-70	0039-1*	8-7-70	493	(71.4)	10-30-70	430	(62.3)
Convair	GY-70 (cont.)	7-23-70	OA80-1-1	8-14-70	479	(69.2)	10-30-70	339	(48.6)

* No post cure.

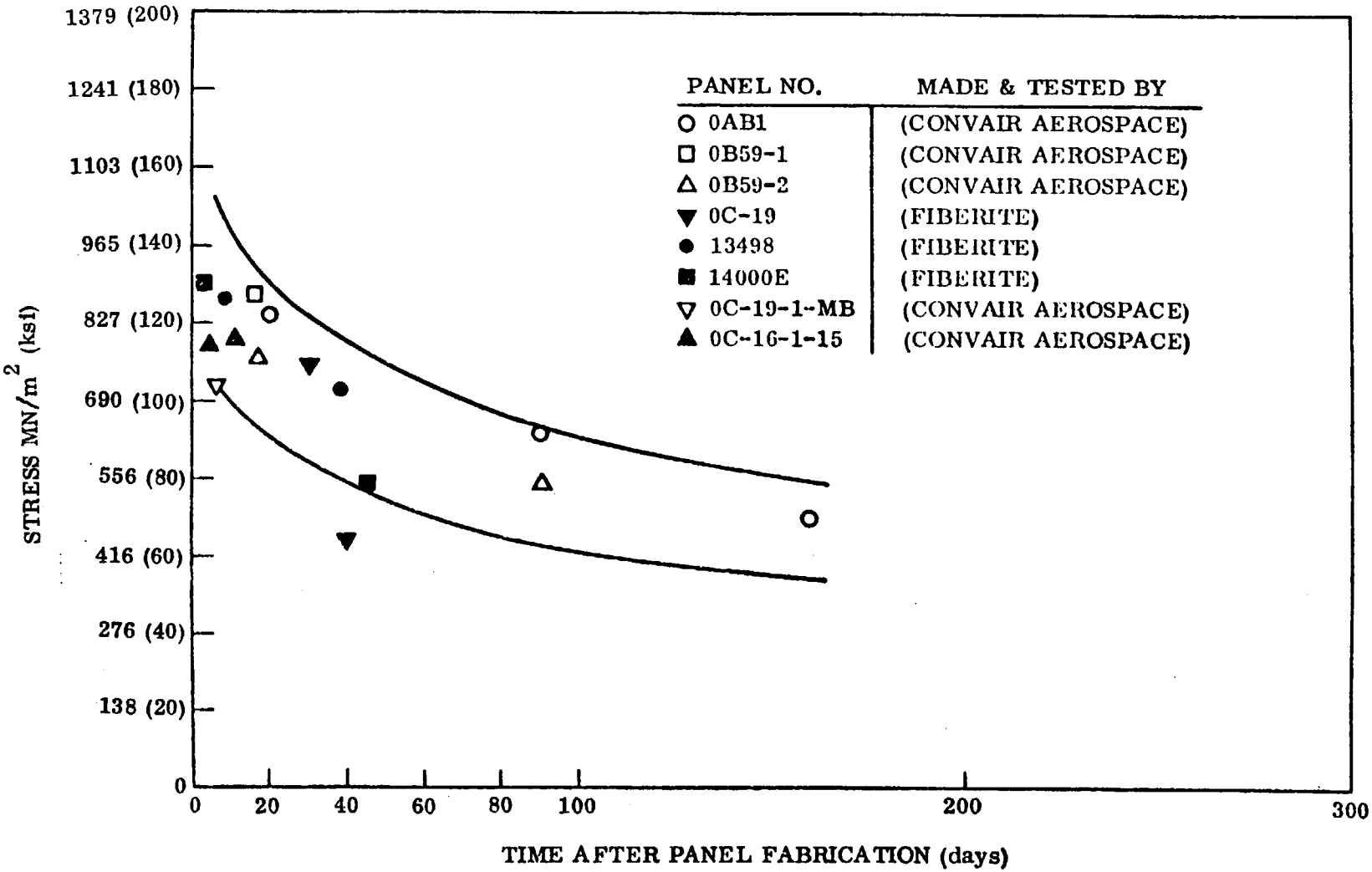


Figure 2-23. Longitudinal Flexural Strength at 450°K (350°F) for HT-S/X-904

Table 2-37. Horizontal Shear Strength at 450°K (350°F)

Fabricator	Resin	Fiber	Mfg. Date	Panel No.	Initial Test Date	Initial Strength MN/m^2 (ksi)	Retest Date	Retest Strength MN/m^2 (ksi)
Hercules	3002	HT-S (cont.)	—	X201-20C-1	6-1-70	46 (6.7)	11-4-70	34 (4.9)
Fiberite	3002	HT-S (cont.)	8-14-70	R-3	8-15-70	63 (9.0)	11-3-70	22 (3.2)
Fiberite	X-904	HT-S (staple)	9-21-70	OC-19	9-23-70	40 (5.7)	11-3-70	32 (4.7)

Table 2-38. Longitudinal Flexural Strength at Temperature as a Function of RT Aging

Fabricator	Resin	Fiber	Mfg. Date	Panel No.	Test Temp. °K	Test Temp. °F	Initial Test Date	Initial Strength MN/m^2 (ksi)	Retest Date	Retest Strength MN/m^2 (ksi)
Fiberite	Experimental*	HT-S (staple)	9-8-70	14397B	450	(350)	9-19-70	1138 (165)	10-27-70	1041 (151)
Fiberite	Experimental*	HT-S (staple)	9-8-70	14397C	450	(350)	9-19-70	1324 (192)	10-26-70	1289 (187)
Fiberite	Experimental*	HT-S (staple)	6-8-70	14351	450	(350)	6-15-70	731 (106)	11-6-70	620 (89)
Hercules	BP-907	HM-S (cont.)	6- -70	37	355	(180)	6-7-70	1069 (155)	11-5-70	896 (130)
Ferro	E-293	MODMOR I (short)	2- -69	—	355	(180)	2- -69	542 (78)	11- -70	606 (87)
Ferro	E-293	MODMOR I (short)	2- -69	—	355	(180)	2- -69	647 (93)	11- -70	585 (84)

* High-temperature-resistant epoxy.

Table 2-39 summarizes these results (Reference 2-5). This type of property regeneration would lend credence to the hydrolysis theory, since both vacuum and heat exposures acted to regenerate the 450°K (350°F) flexural strength. The mechanism of degradation by water penetration could be a result of plasticizing effect, molecular scission, or chemical reaction.

Table 2-39. Effects of Post Cure Treatments on 450°K (350°F) Longitudinal Flexural Strength

Fabricator	Resin	Fiber	Panel No.	Retest Date	Initial Strength MN/m^2 (ksi)	Post Cure Treatment	Test Date	Final Strength MN/m^2 (ksi)
Convair	X-904	HT-S	OB59-2	10-28-70	556 (79.8)	72 hours at $\sim 10^{-5}$ torr vacuum and tested immediately**	11-13-70	668 (95.6)
Convair	3002	HT-S	X201-40	10-29-70	834 (121)	4 hours at 450K (350F)	11-19-70	1020 (148)
Convair	3002	HT-S	X201-40	10-29-70	834 (121)	4 hours at 393K (250F)	11-19-70	979 (142)
Fiberite	X-904	HT-S	14398-1	10-27-70	717 (104)	4 hours at 450K (350F) plus 9 hours at 464K (375F)	11-3-70	1027 (149)
Fiberite	X-904	HT-S	14000E	10-29-70	542 (78)	4 hours at 450K (350F) plus 9 hours at 464K (375F)	11-3-70	668 (96)
Whittaker***	1004	MODMOR II	—	11-3-70	311 (45)	~16 hours at 375F	11-4-70	724 (105)

* See Tables 2-33 to 2-35 for initial data.
** Weight loss in vacuum was 0.13%.
*** Weight loss from post cure was 0.5%.

Fiberite conducted several experiments to determine moisture pickup by typical graphite/epoxy composites. The following weight gains were noted after a one-hour water boil: HT-S/X-904 (0.78%), HT-S-3002 (0.52%), and HT-S/modified X-904 (0.24%). Flexural specimens of HT-S/X-904 that were two months old lost 0.15% by weight as a result of heating for one hour at 450°K (350°F). An eight- by two-inch panel of HT-S/X-904 lost 0.17% by weight when heated for one hour at 450°K (350°F).

Several prepreggers conducted extensive testing on neat epoxy resin samples. They have conducted extensive temperature-hardness tests on 13 neat resin samples that had been stored for one or two years. These samples included ERR 4205, ERLA 4617, ERLA 0510, and DEN 438 resins; MDA, MPDA, and DADPS hardeners; as well as the BF_3 MEA catalyst. Barcol hardness was monitored on the epoxy systems through a heatup to 450°K (350°F), a hold at 450°K (350°F), cooldown to RT, and a second heatup and hold cycle. All samples showed aging through loss of high-temperature hardness and all samples were rejuvenated through soaking at 450°K (350°F). Similar phenomena can be induced in neat resins by exposure to high humidity. Hercules subjected 4205/MDA, 4617/MDA, and 2256/MPDA neat resins to 100% relative humidity at 321°K (120°F) for 13 days. In all cases the resins degraded more from this exposure than from two years of RT aging. Post cure of four hours at 450°K (350°F) brought back the high-temperature properties of the neat resins. Heat-distortion temperature (HDT) of the neat epoxy resins also degrades as a result of exposure to water vapor. Degradations of 10% in HDT have been measured.

As a subcontractor on NAS8-26198, Fiberite conducted some calculations on the lowering of heat distortion temperature as a result of water ingress into graphite/epoxy composites. Water so absorbed could act as a plasticizer. Changes in HDT of up to 5% are readily conceivable. Such changes could account for reductions in high-temperature 450°K (350°F) resistance.

Further exploratory testing of aging behavior was considered beyond the scope of this contract. The program at this point was redirected to concentrate on polyimide composite systems. Since this initial work in October 1970 NASA/MSFC has sponsored a program to investigate this problem with epoxy composites. In the final report on Contract NAS8-27435, "Investigation into the High-Temperature Strength Degradation of Fiber-Reinforced Resin Composite During Ambient Aging," June 1973, it was concluded that moisture was the problem affecting the epoxy resin systems.

SECTION 3

HIGH-STRENGTH GRAPHITE/POLYIMIDE COMPOSITES

The availability of materials and their resistance to temperature were the primary considerations in selecting the graphite fibers and polyimide resin systems for the characterization studies. A guideline or criteria having a significant effect on the selection of the resin was the requirement for (a) oxidative stability for 400 hours at 589°K (600°F), and (b) a resin system that could be vacuum-bag, press, or autoclave cured.

3.1 GRAPHITE FIBER SELECTION

Section 2.1 of this report has an extensive discussion as to the types of graphite fibers available at the initiation of this program. An objective of this program was to look at one high-strength and one high-modulus graphite fiber in combination with two polyimide resin systems. The HT-S and GY-70 fibers were selected, based on selection criteria reported in Section 2.1, so that comparisons could be made between the graphite/epoxy and graphite/polyimide data being developed on this program.

3.2 POLYIMIDE RESIN SELECTION

The selection of the resin systems for initial characteriation depended on the best balance of laminate properties, handling characteristics such as tack and drape, and reasonable process requirements such as time, temperature, and pressure. A program guideline was that a maximum pressure of 793 KN/m^2 (115 psi) and an initial cure temperature of 450°K (350°F) would be allowed to cure the graphite/polyimide composite.

It is difficult to draw accurate conclusions from existing data concerning service life at elevated temperature for laminates based on polyimide resins. This difficulty is due in a large part to the range of quality (void content) normally obtained on laminates made from polyimide resin systems available from different suppliers. Well-known suppliers of polyimide resins include DuPont, Monsanto, TRW, and General Electric. The recommended fabrication process differs for each of these resin systems. In addition, the degree to which the various systems have been optimized from a fabrication process standpoint varies considerably. Aside from the basic thermal stability of the polymer, the one single factor that most affects the service life of a plastic composite at elevated temperature is the surface area of resin available for oxidative attack. Differences in surface area for 30.5 by 30.5 by 0.32-cm (12- by 12- by 1/8-inch) laminates, which have been widely used for thermal aging studies, are functions of the void content of the laminate. These relationships are graphically illustrated in Figure 3-1 (Reference 3-1). Low-void resin systems having substantially lower thermal oxidative resistance may often appear to be superior to a more thermal resistant but porous system when compared directly in thermal aging at certain time-temperature conditions.

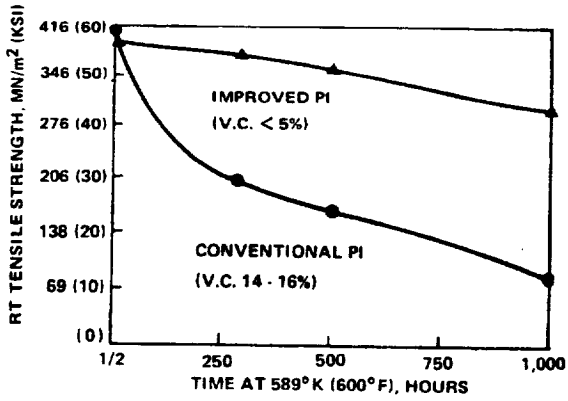


Figure 3-1. Effect of Void Content on Thermal Degradation of PI Glass Laminate at 589° K (600° F)

At the start of this program, there were a number of polyimide resin systems available which potentially could meet program requirements. Various workers had developed low-void PI resins, PI systems, PI processing, PI prepreg, etc. The more significant materials include TRW's P13N resin, Brunswick's BP-373 system, Aerojet's processing of Monsanto's Skybond 700, Skybond 703, and DuPont's low-flow Pyralin X222-84 prepreg. For example, unlike conventional polyimides, P13N cures with only minimum evolution of volatile matter. The low-molecular-weight precursors of P13N are much more stable than those used to form other high-temperature resins. In addition, the TRW system requires no post-cure. This advantage results in considerable time-saving in comparison to the other polyimides. There are two types of prepreg that can be used to fabricate components: a tack-free, fully imidized prepreg, or a tacky, partially advanced prepreg. The first approach produces laminates that evolve no volatiles. However, this system is dry, quicker to cure, fairly stiff, and therefore, useful only for simple curvature or flat shapes. The tacky version can be draped over fairly complex contours, but it requires a separate imidization step to ensure low-void products. The processing advantages of P13N also lead to improved composite properties (Reference 3-2). The strength retention at 562° K (550° F) after 1000 hours was even greater than 60% of the original 562° K (550° F) strength.

Although the P13N resin system has several advantages, it also possesses a number of disadvantages. The type of components that can be fabricated is limited by the cure cycle. Very little processing information exists on this resin cured below 533° K (550° F). Extensive data has not yet been obtained to fully determine its heat-resistant capabilities. A final important consideration is cost. The price of P13N is two to three times that of the other commercially available polyimides.

A limited amount of work has been conducted using the P13N resin with boron and graphite compoites. An example of the types of properties obtainable with high-pressure lamination of graphite/P13N prepreg is shown in Table 3-1 (Reference 3-2).

Based on the data available and the experience of Convair Aerospace personnel and other investigators with the P13N resin system, it was dropped from consideration as a candidate system for the characterization study.

Table 3-1. Comparison of Hercules HT-S/P13N Composites Made With Various Laminating Pressures

Test Temp. °K (°F)	Mechanical Property	Laminating Pressure					
		2069 KN/m^2 (300 psi)		1380 KN/m^2 (200 psi)		690 KN/m^2 (100 psi)	
297 (75)	Flex. Strength MN/m^2 (ksi)	1538	(223)	1462	(212)	1572	(228)
	Flex Modulus GN/m^2 (10^6 psi)	148	(21.5)	145	(21.0)	161	(23.3)
	Short Beam Shear MN/m^2 (ksi)	84.8	(12.3)	98.6	(14.3)	97.9	(14.2)
589 (600)	Flex. Strength MN/m^2 (ksi)	896	(130)	834	(121)	758	(110)
	Flex Modulus GN/m^2 (10^6 psi)	146	(21.2)	143	(20.8)	152	(22.1)
	Short Beam Shear MN/m^2 (ksi)	54.2	(7.82)	59.9	(8.55)	54.9	(7.87)

The Brunswick BPI-373 system exhibits low void content and service life at 589°K (600°F) in excess of 2000 hours. At present it is a proprietary system, and available only in the form of a glass or quartz prepreg or a fabricated part.

B-staging and fabrication processes, developed by Aerojet for Skybond 700 and Skybond 703, have yielded low-void content laminates having physical properties equivalent to epoxy systems along with good elevated temperature strength and stability. However, the processing is lengthy and has proven difficult to reproduce by other workers.

Convair Aerospace has conducted extensive evaluations and processing R&D using modifications of the Aerojet cycle with both glass/polyimide and quartz/polyimide. With a 346 kN/m^2 (50-psi) autoclave cure, Convair Aerospace found no difference in advancement (modified Aerojet cycle) techniques, conventional techniques using dielectric monitoring, or a combination of advancement and dielectric monitoring techniques. Table 3-2 is a summary of the data obtained by Convair Aerospace from 30.4 by 30.5-cm (12- by 12-inch) laminates.

The DuPont Pyralin X222-84 "low-flow" polyimide is available only in a preimpregnated fabric form. It is apparently based on one of their earlier resins systems, and is a large improvement over the previous DuPont prepreg. Glass-reinforced composites having void content of 10% or less may be fabricated with this material.

Table 3-2. Comparison of Test Panel Results on Quartz/703 Polyimide as a Function of Cure Technique (Room Temperature Data)

Technique	Flexural Strength MN/m^2 (ksi)	Flexural Modulus GN/m^2 (10^6 psi)	Tensile Strength MN/m^2 (ksi)	Tensile Modulus GN/m^2 (10^6 psi)	Void Content (%)
Advancement					
1	592 (85.4)	22.3 (3.23)	528 (75.5)	25.8 (3.74)	13.2
2	549 (79.4)	20.5 (2.97)	514 (73.5)	25.9 (3.76)	13.4
3	556 (79.8)	20.3 (2.95)	507 (72.8)	25.7 (3.73)	13.1
4	613 (88.1)	22.2 (3.22)	514 (74.0)	26.1 (3.79)	14.2
Avg.	577 (83.2)	21.3 (3.09)	514 (74.0)	25.9 (3.76)	13.5
Dielectric Monitoring					
1	592 (84.7)	23.4 (3.40)	479 (68.8)	26.6 (3.85)	12.0
2	620 (88.8)	24.0 (3.48)	479 (69.4)	26.3 (3.81)	11.4
3	599 (86.3)	23.2 (3.37)	479 (68.9)	24.6 (3.57)	12.0
4	563 (81.2)	21.6 (3.13)	535 (76.8)	25.7 (3.73)	10.0
Avg.	592 (85.3)	23.1 (3.35)	493 (71.0)	25.8 (3.74)	11.4
Combination					
1	549 (78.8)	21.1 (3.06)	514 (74.2)	23.8 (3.45)	12.2
2	627 (90.3)	20.8 (3.02)	535 (76.7)	25.2 (3.65)	10.7
3	620 (88.9)	22.6 (3.27)	514 (73.5)	28.0 (4.07)	—
4	599 (86.0)	21.2 (3.08)	521 (75.3)	24.8 (3.59)	—
Avg.	599 (86.0)	21.4 (3.11)	521 (74.9)	25.4 (3.69)	11.5

From a thermal stability standpoint, the DuPont polyimides appeared to have an advantage over most of the other available resins. However, the development of low-void, fiber reinforced composites made with DuPont polyimides had not progressed as far as those made with other systems such as Skybond 703. Boeing had reported data on boron composites made with this resin up to 505° K (450° F) (Table 3-3). A low-flow resin, DuPont 4707, prepregged by U.S. Polymeric on Modmor II and boron gave low-void laminates having very high room-temperature properties.

General Dynamics had worked with two low-flow resins developed by Monsanto: Skybond 709 (a low-flow version of Skybond 700) and Skybond 710 (a low-flow version of Skybond 700). Convair Aerospace evaluated the Skybond 709 resin system for use at 616° K (650° F), and Table 3-4 shows variation of properties with cure cycle. This preliminary information showed that the long-term heat resistance may not be as good as that reported for the BPI-373 (see data for improved PI - Figure 3-1). However, the handling characteristics of the Skybond 709 prepregs are better than those of the BPI-373, and the blend of mechanical properties and handling characteristics was the goal of this program.

Table 3-3. Properties of Boron/X222-84 Polyimide Composite

Boron Source	Hamilton Standard
Resin Matrix:	DuPont, type X222-84
Volume Fraction of Boron Filaments:	50% ±5
Modulus of Elasticity 297°K and 505°K (77°F and 450°F):	206 GN/m^2 (30×10^6 psi)
Ultimate Tensile and Compressive Strength at 279°K (75°F):	1379 GN/m^2 (200 ksi)
Ultimate Tensile and Compressive Strength at 505°K (450°F):	1007 GN/m^2 (146 ksi)

Convair Aerospace initiated the evaluation of graphite-reinforced-polyimide composite materials in 1967. Initial laminates using Thornel 50 graphite yarns (heat cleaned) and Skybond 703 polyimide resins (Monsanto Company) were of poor quality. Convair Aerospace contacted the various polyimide-resin manufacturers to obtain more processable systems and selected the Skybond 710 polyimide resin. Typical properties of this polyimide system are listed here with a rating of importance for each.

Property	Range	Rating
Solids	53 to 57%	Medium
Viscosity	2000 to 9000 cps	High
Specific Gravity	1.09 to 1.14	Low
Gel Time	5 to 20 minutes	High
pH	4.0 to 3.8	High

Laminates were made with both Modmor II and Courtaulds HTS graphite filaments, and the Skybond 710 polyimide resin using vacuum-bag only, or autoclave-augmented cure pressures. The typical cure schedule developed was based on simple viscosity versus time and temperature tests.

A summary of typical physical and mechanical properties measured for graphite-reinforced-polyimide laminates is given in Tables 3-5 through 3-7. The main points of interest are as follows.

a. Postcure may be minimized for specific graphite/polyimide composites used for 562°K (550°F) temperature applications.

Table 3-4. Aging Studies on Glass-Reinforced Skybond 709 Polyimide

Panel No.	Void Content (%)	RT Flexural Strength MN/m^2 (ksi)	RT Flexural Modulus GN/m^2 (10^6 psi)	RT Flexural Properties After 100 Hours at 616°K (650°F)		RT Flexural Strength After 200 Hours at 616°K (650°F) MN/m^2 (ksi)	RT Flexural Strength After 300 Hours at 616°K (650°F) MN/m^2 (ksi)	Cure Cycle
				Strength MN/m^2 (ksi)	Modulus GN/m^2 (10^6 psi)			
105		479 (69.0)	23 (3.3)	283 (41.0)	21 (3.1)	200 (29.0)	117 (17.0)	Standard 450°K (350°F) cure, 345 kN/m^2 (50 psi) at 405°K (270°F)
106	7.0	458 (66.0)	19 (2.8)	192 (28.0)	14 (2.1)	145 (21.0)	110 (16.0)	Standard 450°K (350°F) cure, 690 kN/m^2 (100 psi) at 405°K (270°F)
110	5.0	507 (73.0)	23 (3.3)	262 (38.0)	23 (3.3)	172 (25.0)	103 (15.0)	H_2O hydrated, 375°K (215°F) for 15 min., 690 KN/m^2 (100 psi) at 416°K (290°F)
111	3.0	360 (52.0)	23 (3.3)	311 (45.0)	23 (3.3)	234 (34.0)	124 (18.0)	Standard 450°K (350°F) cure, 690 kN/m^2 (100 psi) at 405°K (270°F)
112	6.6	556 (80.0)	26 (3.7)	283 (41.0)	23 (3.3)	227 (33.0)	124 (18.0)	Standard 450°K (350°F) cure, 690 kN/m^2 (100 psi) at 422°K (300°F)

Table 3-5. Summary of Preliminary Graphite/ Polyimide-Composite Results

Material System	Graphite Vol. (%)	Construction Orientation/ Plies	Property	Test Temp. °K (°F)	Ultimate MN/m^2 (ksi)
Courtaulds HTS/Polyimide (710)	70	$[0]_6$	Tension (L)	297 (75)	668 (96)
Courtaulds HTS/Polyimide (710)	70	$[0]_6$	Tension (L)	589 (600)	690 (100)
Courtaulds HTS (P13N Finish)/ Polyimide (710)	70	$[0]_6$	Tension (L)	297 (75)	654 (94)
Courtaulds HTS (P13N Finish)/ Polyimide (710)	70	$[0]_6$	Tension (L)	589 (600) (NPC)*	668 (96)
Courtaulds HTS-Asbestos/ Polyimide (710)	45	$[(HTS_0/A90)_3]_S$	Tension (L)	297 (75)	367 (53)
Courtaulds HTS-Asbestos/ Polyimide (710)	45	$[(HTS_0/A90)_3]_S$	Tension (T)	297 (75)	54 (7.8)
Courtaulds HTS-Asbestos/ Polyimide (710)	45	$[(HTS_0/A90)_3]_S$	Tension (T)	297 (75) (NPC)	53 (7.7)
Courtaulds HTS/Polyimide (710)	70	$[0]_6$	Shear (L)	297 (75)	26 (3.7)
Courtaulds HTS (P13N Finish)/ Polyimide (710)	70	$[0]_6$	Shear (L)	297 (75)	54 (7.8)
Modmor II/Polyimide (710)	48	$[0]_6$	Tension (T)	297 (75) (NPC)	1145 (166)
Modmor II/Polyimide (710)	48	$[0]_6$	Tension (T)	297 (75) (NPC)	20 (2.9)
Modmor II/Polyimide (703)	56	$[0]_6$	Tension (L)	297 (75) (NPC)	1089 (158)
Modmor II/Polyimide (703)	56	$[0]_6$	Tension (T)	297 (75) (NPC)	35 (5.1)
Modmor II/Polyimide (703)	65	$[0]_6$	Tension (L)	297 (75) (NPC)	1214 (176)
Modmor II/Polyimide (710)	52	$[0]_6$	Tension (L)	297 (75) (NPC)	1324 (192)

* NPC indicates final cure temperature was 450°K (350°F),

Table 3-6. 562°K (550°F) Tensile Results for 450°K (350°F) Cured Graphite/Polyimide Laminates

Laminate Material System	Cure Pressure	Density (gm/cc)	Calc. Graphite (Vol. %)	Ultimate MN/m^2 (ksi)	Average % Retention of RT Strength
Modmor II/710	Vacuum Bag	1.51	47		
Specimen No. 1				606 (87.3)	
Specimen No. 2				585 (84.5)	
Specimen No. 3				738 (106.6)	
Specimen No. 4				606 (87.1)	
				Avg. 640 (91.6)	59
Modmor II/710	Vacuum Bag - 521 kN/m^2 (75 psig)	1.59	52		
Specimen No. 1				924 (134.1)	
Specimen No. 2				841 (122.4)	
Specimen No. 3				758 (110.2)	
Specimen No. 4				979 (141.8)	
				Avg. 876 (127.1)	66
Modmor II/703	Vacuum Bag (Only)	1.48	56		
Specimen No. 1				640 (92.5)	
Specimen No. 2				724 (105.4)	
Specimen No. 3				786 (113.9)	
Specimen No. 4				717 (104.3)	
				Avg. 717 (104.1)	66
Modmor II-Asbestos/ 703	Vacuum Bag (Only)	1.50	47		
Specimen No. 1				633 (91.3)	
Specimen No. 2				710 (103.2)	
Specimen No. 3				633 (91.0)	
Specimen No. 4				592 (84.0)	
				Avg. 647 (92.6)	No RT Control

Table 3-7. Initial 589°K (600°F) Tensile Results for High-Fiber-Volume Graphite Polyimide Laminates

Material System	Graphite (Vol. %)	Construction Orientation Plies	Property	Test Temp. °K (°F)	Ultimate MN/m^2 (ksi)
Courtaulds HTS/Polyimide (710)	70	0_6	Tension (L)	297 (75)	668 (96)
Courtaulds HTS/Polyimide (710)	70	0_6	Tension (L)	589 (600)	690 (100)
Courtaulds HTS (P13N Finish)/ Polyimide (710)	70	0_6	Tension (L)	297 (75)	654 (94)
Courtaulds HTS (P13N Finish)/Polyimide (700)	70	0_6	Tension (L)	589 (600)	668 (96)

b. P13N polyimide finish on graphite fiber may improve composite interlaminar-shear-strength properties.

c. Graphite/polyimide laminates having less than 10% voids can be made with commercial polyimide resins using low-pressure cure techniques.

d. Graphite/polyimide laminates having tensile-strength properties competitive with graphite/epoxy laminates have been developed.

Under Convair Aerospace-sponsored IRAD studies (Reference 2-4), preliminary investigations were conducted using HT-S graphite fiber and Modmore Type II graphite fiber preimpregnated with Monsanto's 710 polyimide resin. The data obtained from these studies is shown in Table 3-8.

Table 3-8. Preliminary Tensile-Properties of Convair Aerospace-Developed, Graphite/Polyimide Laminates

Laminate Material System	Cure Pressure	Specific Gravity (gm/cc)	Calc. Graphite (Vol. %)	Ultimate Strength (Long.) MN/m^2	(ksi)	Modulus GN/m^2	(msi)
Modmor II/703	Vacuum Bag only (VB)						
Specimen No. 1				1110	(160.8)	123	(17.8)
Specimen No. 2		Avg.	Avg.	965	(140.1)	136	(19.8)
Specimen No. 3		1.48	58	1083	(157.0)	126	(18.2)
Specimen No. 4				1207	(175.4)	125	(18.1)
				Avg. 1089	(158.4)	Avg. 128	(18.5)
Modmor II/710							
Specimen No. 1				869	(125.8)	133	(19.3)
Specimen No. 2		Avg.	Avg.	1248	(181.1)	141	(20.4)
Specimen No. 3		1.51	48	1172	(170.4)	152	(22.1)
Specimen No. 4				1020	(148.1)	126	(18.3)
				Avg. 1076	(156.4)	Avg. 138	(20.0)
Modmor II/703	VB/521 kN/m^2 (75 psig)						
Specimen No. 1		Avg.	Avg.	1069	(154.8)	140	(20.3)
Specimen No. 2		1.56	65	1351	(195.7)	128	(18.6)
				Avg. 1207	(175.3)	Avg. 134	(19.5)
Modmor II/710	VB/521 kN/m^2 (75 psig)						
Specimen No. 1				1351	(195.5)	129	(18.7)
Specimen No. 2		Avg.	Avg.	1386	(200.9)	135	(19.6)
Specimen No. 3		1.59	52	1276	(185.3)	166	(24.1)
Specimen No. 4				1296	(187.5)	149	(21.6)
				Avg. 1324	(192.3)	Avg. 145	(21.0)

Based on the data obtained from the literature, data generated at Convair Aerospace and discussions held at NASA-MSFC, the Monsanto 710 and DuPont 4707 resins were selected for evaluation in the characterization study. These resins were selected for the following reasons:

a. Commercially available — several prepreg companies had experience with these systems.

b. Prepregs using these resins had good tack and drape.

c. Processing requirements are amenable to the fabrication of large parts.

3.3 GRAPHITE/POLYIMIDE CHARACTERIZATION

One pound of each of the four graphite/polyimide resin prepregs was obtained from Fiberite Corporation. Table 3-9 summarizes the material designations, supplier, batch numbers, etc., for the various prepregs. Table 3-10 summarizes the prepreg testing in support of the resin-evaluation program. The procedures for obtaining volatile content, resin content, percent flow, and for running the process gel test are summarized in Appendix G.

Table 3-9. Materials Flow Chart

Material	Material Designation	Supplier	Quantity (pounds)	Batch No.	Date of Receipt
Graphite/Polyimide	hy-E-1512	Fiberite	1	OB68	8-5-70
	hy-E-1312-B	Fiberite	1	OB69	8-5-70
	hy-E-1313-B	Fiberite	1	OB70	8-5-70
	hy-E-1513	Fiberite	1	OB67	8-10-70

Table 3-10. Graphite/Polyimide Prepreg Test Results

Material Designation	Batch No.	Fiber	Resin	Avg. % Volatiles	Avg. % Resin	Avg. % Flow	Process Gel Test Results	Observations
hy-E-1513-B	OB67	GY-70	4707	14.8	35.4	29.3	Gelled at 436°K (325°F) after 3 minutes	Very tacky
hy-E-1512	OB68	GY-70	710	13.9	28.3	21.2	Gelled at 436°K (325°F) between 0 and 2 minutes	Slight tack, small number of gaps
hy-E-1312-B	OB69	HT-S	710	14.1	23.9	20.5	Gelled at 436°K (325°F) after 3 minutes	Fairly dry, low flow into bleeder
hy-E-1313-B	OB70	HT-S	4707	14.8	31.8	22.9	Gelled at 436°K (325°F) after 3 minutes	Very tacky, fully wet 4 plies bleeder

3.3.1 PANEL FABRICATION. Ten-ply unidirectional panels 25.4- by 15.2 cm (10- by 6-inch) were fabricated for each of the systems selected for the characterization study. Material-supplier-specified cure cycles for vacuum-bag, press, and autoclave curing of the composites were used in this initial study. The lay-up used in this study is shown in Figure 3-2. Prepreg sheets as well as Process Control Sheets were used for each of the four lots of prepreg and twelve laminates (see Section 2.2 of this report). All graphite/polyimide panels received a staged post-cure cycle of 1 hour at 450°K (350°F), 4 hours at 505°K (450°F), 16 hours at 533°K (500°F), 4 hours at 589°K (600°F) and 4 hours at 616°K (650°F). Vacuum bag, press, and autoclave cures were attempted with each system.

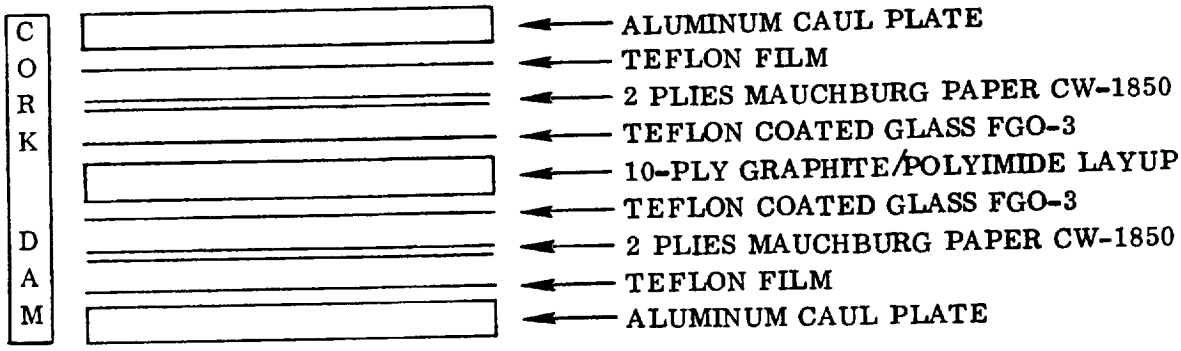


Figure 3-2. Typical Graphite/Polyimide Cure Lay-up

I. POLYIMIDE PREPREGS

A. Material Designation: hy-E-1513
Fiber Type: GY-70 (continuous)
Material Form: 30.5- by 91.5-cm (12- by 36-inch) sheet
Batch No.: OB67
Resin: DuPont 4707
Manufacturer: Fiberite

1. Fabrication Procedure (Autoclave Cure)

Mold Release	Teflon Film
Layup:	10 plies unidirectional, (25.4 by 15.2 cm) (10 by 6 inch)
Separator Film:	Teflon-coated glass cloth, FGO-3, 0.0028-inch thick
Bleeder	4 plies Mauchburg paper CW-1850

	Special Instruction:	A Corprene 0.95 cm (3/8 in.) dam was used around the periphery of layup. Bleeder material was placed on both sides of the prepreg (2 plies each). One-ply Teflon film (nonperforated) was used over the bleeder. A 25.4- by 15.2- by 0.64-cm (10- by 6- by 1/4-inch) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg. vacuum plus 690 kN/m^2 (100 psi) autoclave pressure. Vacuum bag was applied at room temperature, and 690 kN/m^2 (100 psig) was applied at 450°K (350°F) and maintained through entire cure cycle and cool down to below 344°K (160°F).
	Cure Cycle:	Heat to 450°K (350°F) at 1 to 3°K/min (3 to 5° F/min), hold for 1 hour, and cool to below 344°K (160°F).
	Post Cure:	1 hour at 450°K (350°F), 4 hours at 505°K (450°F), 16 hours at 533°K (500°F), 4 hours at 589°K (600°F), 4 hours at 616°K (650°F).
2.	Fabrication Procedure: (Press Cure)	Same as fabrication procedure I.A.1.
	Cure Pressure:	Same as I.A.1.
	Cure Cycle:	Same as I.A.1.
	Post Cure:	Same as I.A.1.
3.	Fabrication Procedure: (Vacuum Bag)	Same as fabrication procedure I.A.1.
	Cure Pressure:	Vacuum bag applied at room temperature and held throughout the cure cycle.
	Cure Cycle:	Same as I.A.1 except 2 hours at 450°K (350°F).
	Post Cure:	Same as I.A.1.
B.	Material Designations:	hy-E-1512
	Fiber Type:	GY-70 (continuous)
	Material Form:	30.5- by 91.5-cm (12- by 36-inch) sheet
	Batch No.:	OB68
	Resin:	Monsanto 710
	Manufacturer:	Fiberite

All fabrication procedures, cure cycles, and postcure cycles are identical to II.A.1, 2, and 3.

C. Material Designation: hy-E-1312B
Fiber Type: HT-S (staple)
Material Form: 30.5- by 114-cm (12- by 45-inch) sheet
Batch No.: OB69
Resin: Monsanto 710
Manufacturer: Fiberite

All fabrication procedures, cure cycles, and postcure cycles are identical to II.A.1, 2, and 3.

D. Material Designation: hy-E-1313B
Fiber Type: HT-S (staple)
Material Form: 30.5- by 114-cm (12- by 45-inch) sheet
Batch No.: OB70
Resin: DuPont 4707
Manufacturer: Fiberite

All fabrication procedures, cure cycles, and postcure cycles are identical to I.A.1, 2, and 3.

3.3.2 MECHANICAL PROPERTY TESTS. Test specimens were obtained from each panel per the cutting diagram shown in Figure 2-10. Flexural tests, both longitudinal and transverse, were conducted at 77°, 297° and 589° K (-320, 75 and 600° F). The longitudinal flexure tests were conducted with a three-point loading system, and the transverse flexure tests were conducted with a four-point loading system. A span-to-depth ratio of 32:1 was used for the longitudinal flexure tests. A notched-interfiber-shear specimen was used initially because it is believed by Convair Aerospace that this type of specimen evaluates the shear capability of the advanced composite system under test to a much higher degree than the short-beam-shear specimen. However, short-beam-shear specimens were tested throughout the remainder of the program, because the majority of interlaminar shear data currently available were obtained on this type of specimen. The 77° K (-320° F) tests were conducted after a five-minute soak, while the 589° K (600° F) specimens were exposed to a 10-minute temperature soak before testing. The test specimens used in this program are illustrated in Volume II. Laminate resin contents were determined using a H_2SO_4/H_2O_2 digestion method, as detailed in Volume II. Specific gravity was determined for each panel per Federal Test Method Standard No. 406, Method 5011.

3.3.3 GRAPHITE/POLYIMIDE SYSTEMS. A summary of the data obtained on the graphite-polyimide systems is presented in Tables 3-11 and 3-12. Again the major difference between the two fiber systems was the higher strength of the HT-S composites compared to the GY-70 composites. Also, the lower shear strength and transverse

Table 3-11. Evaluation of High-Strength Graphite Polyimide/Systems

	HT-S/710 Cure Cycle*			HT-S/4707 Cure Cycle*		
	1	2	3	1	2	3
Longitudinal Flexure Strength MN/m^2 (ksi)						
77°K (-320°F)	958 (139.0)	1131 (163.9)	869 (126.4)	814 (117.6)	-	958 (138.9)
297°K (75°F)	717 (104.3)	1048 (152.3)	1089 (157.8)	577 (82.7)	-	945 (137.3)
589°K (600°F)	339 (49.1)	675 (97.4)	430 (61.8)	606 (87.4)	-	458 (66.2)
Longitudinal Flexure Strength MN/m^2 (ksi) after 200 hours at 589°K (600°F)						
297°K (75°F)	800 (116.5)	779 (113.1)	1145 (165.7)	758 (109.9)	-	731 (106.2)
Transverse Flexure Strength MN/m^2 (ksi)						
297°K (75°F)	18 (2.6)	10 (1.4)	30 (4.4)	14 (2.0)	-	24 (3.5)
589°K (600°F)	21 (3.1)	11 (1.6)	23 (3.3)	15 (2.1)	-	15 (2.1)
Specific Gravity	1.44	1.54	1.42	1.45	-	1.46
Resin Content (%)	41.3	35.0	34.0	29.0	-	26.2
Fiber Volume (%)	53.1	59.6	60.8	66.0	-	69.1
Calculated Void Content (%)	9.5	4.6	12.4	11.5	-	11.4

* Cure Cycle 1 — Vacuum Bag; Cure Cycle 2 — Press; Cure Cycle 3 — Autoclave

Table 3-12. Evaluation of High Modulus Graphite/Polyimide Systems

	GY-70/710 Cure Cycle*			GY-70/4707 Cure Cycle*		
	1	2	3	1	2	3
Longitudinal Flexure Strength MN/m^2 (ksi)						
77°K (-320°F)	592 (85.2)	(126.1)	690 (99.8)	682 (97.7)	613 (88.2)	765 (111.0)
297°K (75°F)	563 (80.5)	(108.2)	599 (86.2)	577 (83.0)	549 (78.8)	599 (86.3)
589°K (600°F)	444 (63.8)	640 (91.5)	592 (85.4)	577 (82.6)	521 (75.0)	493 (70.6)
Longitudinal Flexure Strength MN/m^2 (ksi) after 200 hours at 589°K (600°F)						
297°K (75°F)	563 (80.7)	690 (99.7)	570 (82.3)	549 (79.2)	570 (81.9)	592 (85.0)
Transverse Flexure Strength MN/m^2 (ksi)						
297°K (75°F)	7.6 (1.1)	-	10 (1.5)	-	-	-
589°K(600°F)	-	-	-	-	-	-
Specific Gravity	1.52	1.64	1.52	1.45	1.45	1.46
Resin Content (%)	24.6	26.9	34.7	29.0	32.1	26.2
Fiber Volume (%)	68.6	66.1	57.5	66.0	62.6	69.1
Valculated Void Content (%)	14.6	6.9	11.4	11.5	7.9	11.4

* Cure Cycle 1 — Vacuum Bag; Cure Cycle 2 — Press; Cure Cycle 3 — Autoclave

strength of the GY-70 composites became critical. Several of the GY-70 fabricated panels had longitudinal cracks, and transverse flexural strength was very low or non-existent. The data from specimens aged at 589° K (600° F) for 200 hours and then tested at room temperature indicated nearly a 100% retention of the initial strength. Calculated void content for the GY-70 graphite-polyimide laminates in general ran slightly higher than for the HT-S composite laminates. Resin content for the GY-70 laminates was found to be below the desired value of 30% in four out of six laminates. Therefore, based on this data, and the fact that the HT-S fiber was selected for use with the epoxy-resin systems, it was decided that the HT-S fiber would be used for the remainder of the polyimide-resin portion of the program.

The two polyimide-resin systems, 710 and 4707, were initially selected for evaluation because of their controlled flow characteristics and because both systems could be cured by all three processing techniques: vacuum bag, press, and autoclave. Some difficulty was encountered in curing the HT-S/4707 laminates in that volatiles were easily entrapped and the polyimide resin precipitated to the bottom of the laminate. Void contents were higher and resin contents lower with the HT-S/4707 laminates than with the HT-S/710 laminates. Both resin systems showed increases in strength at 77° K (-320° F) compared with room-temperature strength values. The strength values achieved at 589° K (600° F) were far below those expected and probably were caused by volatile entrapment.

Fiberite Corporation was contacted as to the prepregging characteristics of the two polyimide-resin systems. They stated that the 710 resin system was easier to handle from the prepregging standpoint. Also, problems incurred by other investigators in using 4707 resin with either graphite or boron fibers were influential in selection of the 710 resin system for further process optimization.

3.4 POLYIMIDE RESIN CHARACTERIZATION

The Monsanto polyimide varnish Skybond 710 was selected for development in this program. Early screening of a number of polyimide resin systems such as Dupont 4707, Skybond 703, and P13N indicated that the Skybond 710 resin system possessed the best compromise of an autoclave curable system [690 kN/m^2 (100 psi) and 450° K (350° F), maximum] having high-temperature properties.

3.4.1 COMPOSITION OF SKYBOND 710 POLYIMIDE RESIN. Skybond 710 is speculated to be made from approximately the following formulation.

BTDA (benzophenone dianhydride)	31 parts
Aromatic Diamine*	19 parts
Solvent [(Et OH/Xylene/NMP (5/3/1)]	50 parts

Inorganic Ash	4 parts
Amine Catalyst	Trace

* MDA was assumed in the calculation of this recipe and agrees well with the expected stoichiometry determined experimentally.

It is felt from the studies that the BTDA in Skybond 710 has been reacted with technical grade ethanol (approximately 5% water = 12 mol % water) to yield an acid ester. The speculated ratio of acid groups to ester groups is about 1.1 to 1.0. The actual solution composition of Skybond 710 as it is used is felt to have the following composition:

Acid Esters of BTDA	38 parts
Aromatic Diamine	19 parts
Solvent [(Et OH/Xylene/NMP (3/3/1)]	43 parts
Inorganics	4 parts

3.4.2 SOLVENTS EMPLOYED IN SKYBOND 710 POLYIMIDE VARNISH. The volatile liquids present in Skybond 710 include ethanol, xylene, NMP (N-methyl - 2-pyrrolidone) and water. Since the resin components are completely soluble in such solvents as methyl ethyl ketone, the complex mixture used obviously serves a function beyond that of a solvent. All the solvents used will form azeotropes with water and may therefore be present to help remove reactive volatiles (water); however, it seems more likely the primary reason for the choice is to aid in processability by preventing precipitation of the polymer before molecular weight buildup can occur.

The normal sequence of solvent evolution depending on the heating cycle is an initial loss of ethanol present as solvent along with the water present initially. The percent xylene evolved increases with time and temperature, and as reaction begins the percent water increases and reactive ethanol volatile is present. Finally, NMP, boiling point 475°K (396°F), begins to evolve. No firm statements can be made about solvent evolution because it, of course, varies with temperature and heat rate. Table 3-13 shows the solvent evolution for a vacuum strip followed by heating under vacuum.

The Skybond 710 was distilled under both ambient pressure and reduced pressure, approximately 665 N/m^2 (5 mm Hg). Various fractions were taken at several different temperatures. The temperature was raised as the rate of distillation at any given temperature was greatly reduced. The fractions collected were then analyzed both qualitatively and quantitatively with the use of an F & M Model 810 analytical gas chromatograph. A silicone gum rubber column was employed for component separation. The chromatograph is equipped with a flame ionization detector. This detector is extremely sensitive for most compounds (can detect parts per million), but is insensitive to water (a possible reactive volatile).

Table 3-13. Reduced Pressure Distillation of Skybond 710

Sample	Temp. Range of Cut	Pressure Range of Cut	% of Total Vol. in Cut	% Residue at End of Cut	% EtOH[a] in Cut	% Xylene[a] in Cut	% NMP[a] in Cut	% H_2O[b] in Cut
Cut 1	298°K (78°F)	Reduced to maintain boil in pot	56.7	—	36	19		1.7
Cut 1B	298°K to 338°K (78°F to 149°F)	5 mm	8.6	69	2.7[c] 3.3[d]	1.4[c] 0.9[d]		0.4[c] <0.1[d]
Cut 2	338°K to 443°K (149°F to 338°F)	5 mm	34.7	51.9	9.26	10.2	10.1	5.6
TOTALS			100		51.3	31.5	10.1	7.8
						100%		

[a]Determined by VPC, [b]Determined by Karl Fisher Titration, [c]Upper Phase, [d]Lower Phase

Some important facts about the solvent system are as follows:

a. Ethanol is present both as a solvent and as a reactive volatile.

b. Because four major volatiles are present (ethanol, water, xylene, NMP), a change in cure cycle can drastically change the composition of solvent evolution at a given time in the reaction and therby change the product.

c. NMP boils at a very high temperature 475°K (396°F) and care must be taken that its removal does not cause void problems.

d. Xylene (according to Monsanto) is essential for flow control. Since xylene is preferentially lost through polyethylene bags, aluminum foil or similar nonporous material is recommended for storage and shipment of prepregs.

3.4.3 INORGANIC COMPONENTS OF SKYBOND 710 POLYIMIDE VARNISH. Approximately four parts by weight of inorganic material, based on total resin solution, were found following a burnoff at 1075°K (1475°F). This was presumably Cab-O-Sil* or similar material for flow control.

Figure 3-3 is a graph of vacuum strip at ambient temperature, followed by heating to 436°K (325°F) under vacuum, and finally a 1075°K (1475°F) burnoff.

3.4.4 REACTION KINETICS FOR SKYBOND 710. A concentrated effort was made to obtain reaction rate data on the Monsanto Skybond 710 polyimide varnish. Initially, it was planned to conduct studies on cast, self-supporting polyamic acid films (from Skybond 710). These films would then be aged at three different temperatures. An infrared spectrum would then be taken at certain time intervals. The increase in absorption of the imide band would then be plotted versus time to obtain the desired rate constants (k values). In essence, this is what was done. However, since the Skybond 710 varnish

* Cabot Corporation Trade Mark

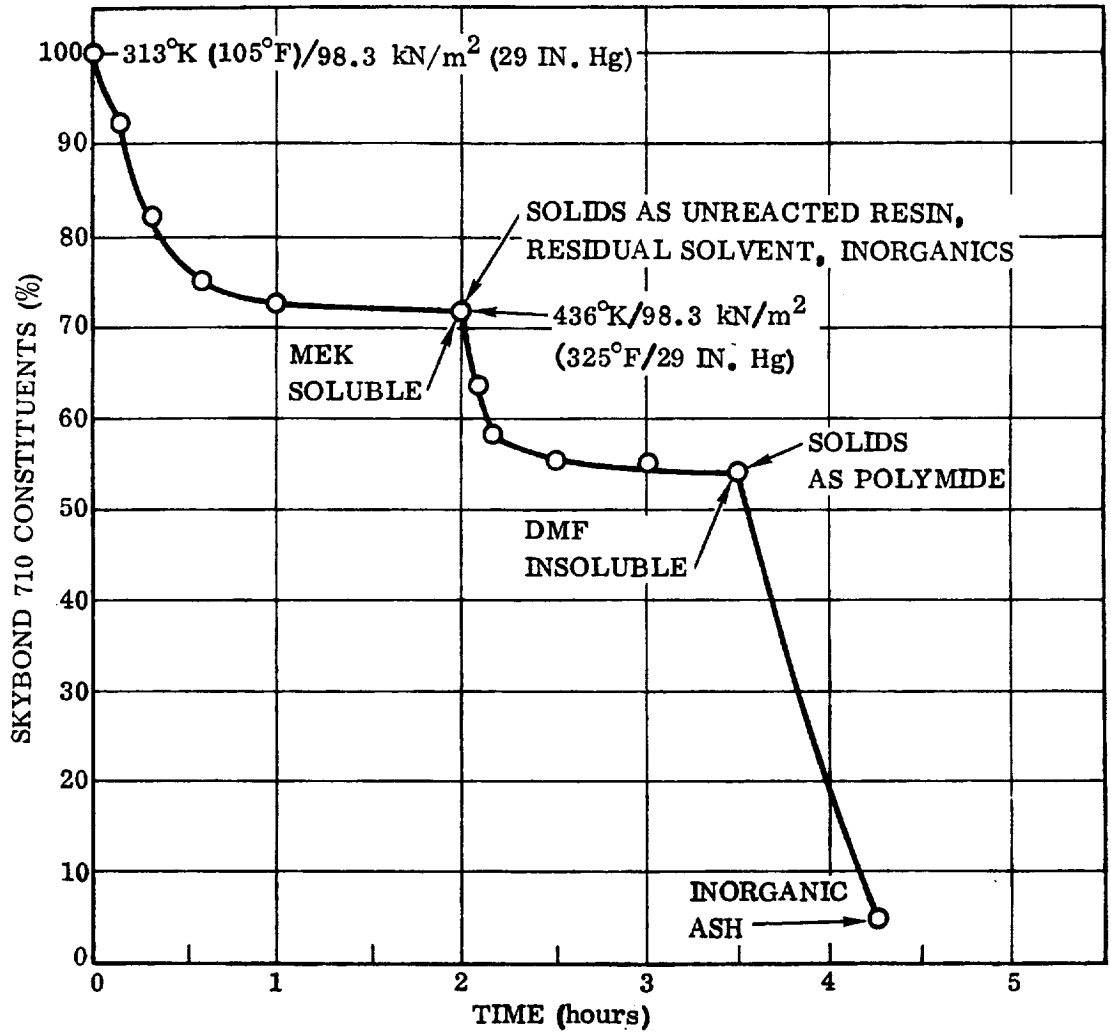
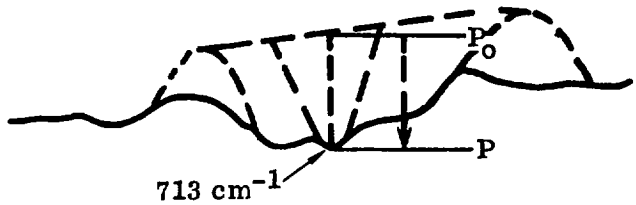


Figure 3-3. Skybond 710 Component Cure

does not lend itself to film formation (presumably due to a low molecular weight precursor), KBr pellets had to be made for every determination. However, it was observed that KBr accelerates the conversion reaction rates of polyamic acid to polyimide. Therefore, the fabrication of a new KBr pellet for each IR determination at a given time for a given temperature was made necessary. Experimental details of the procedure used are described below.

The imidization rate of Skybond 710 was determined by following the appearance of an absorption band at 713 cm^{-1}. There were many absorption bands in the neighborhood of 713 cm^{-1}. The absorption at 713 cm^{-1} was therefore determined by assuming the neighboring bands to be symmetrical (Lorentzian). A base line of



P_o was then determined (see sketch). The baseline method was then used to determine P_o, and absorbance was found from

$$A = \log \frac{P_o}{P}$$

To avoid a negative log, log 1-A = log P/P_o was in fact determined.

Since the spectrum was determined in a KBr pellet and a new pellet was prepared for each determination, an internal reference was needed. The band at 1340 cm^{-1} was chosen for this purpose because it appeared to be least affected during the imidization reaction. A method similar to that mentioned earlier for the 713 cm^{-1} band was used to determine P_o for the 1340 cm^{-1} band.

From the equation

$$\frac{P}{P_o} = 10^{-abc}$$

where

A = absorbance
P_o = incident power
P = transmitted power
P/P_o = transmittance
a = absorbtivity constant
b = cell width
c = concentration

then log

$$\frac{P}{P_o} = -abc$$

and

$$\frac{\left[\log \frac{P}{P_o}\right]_{1340\ cm^{-1}}}{\left[\log \frac{P}{P_o}\right]_{713\ cm^{-1}}} = \frac{-a_{1340\ cm^{-1}}\ (b)_{1340}\ c_{1340\ cm^{-1}}}{-a_{713\ cm^{-1}}\ (b)_{713}\ c_{713\ cm^{-1}}}$$

since $b_{1340} = b_{713}$ for a given pellet, and $^{-a}1340\ cm^{-1}/a_{713\ cm^{-1}}$ = constant for this system = K, then if we set $c_{1340} = 1$

$$\frac{\left[\log \frac{P}{P_o}\right]_{1340\ cm^{-1}}}{\left[\log \frac{P}{P_o}\right]_{713\ cm^{-1}}} = \frac{K}{c_{713}} \approx K \times \frac{1}{\text{Mole fraction of material causing absorption at 713}}$$

The rate constant for the reaction at a given temperature can now be determined by plotting

$$\log \left[\frac{\left(\log \frac{P}{P_o}\right)_{1340}}{\left(\log \frac{P}{P_o}\right)_{713}}\right] \text{ versus time and multiplying slope by 2.303}$$

$$\frac{dc}{dt} = kc \text{ and } \frac{dc}{c} = Kdt$$

$$\int \frac{dc}{c} = \int kdt = \ln c = kt$$

by substitution

$$\ln \left[\frac{K}{\frac{\left(\log \frac{P}{P_o}\right)_{1340\ cm^{-1}}}{\left(\log \frac{P}{P_o}\right)_{713\ cm^{-1}}}}\right] = kt$$

$$-2.303 \log \left[\frac{\left(\log \frac{P}{P_o}\right)_{1340\ cm^{-1}}}{\left(\log \frac{P}{P_o}\right)_{713\ cm^{-1}}}\right] = kt - \ln K$$

From the form y = mx+b, if the reaction is first order and therefore the $\Delta \log c/t$ = constant then ln K should = y intercept.

$$\ln K = \frac{a_{1340\ cm^{-1}}}{a_{713\ cm^{-1}}}$$

The reaction rate curves for 710 are presented in Figures 3-4, 3-5, and 3-6 for 373, 398, and 436°K (212, 257, and 325°F) respectively. Figure 3-7 presents these curves on one plot and Figure 3-8 shows a plot of $1/T \times 10^3$ versus log 10 k to obtain the activation energy (-E*) for the reaction.

On examination of the reaction rate curves (Figures 3-2 to 3-7) it is apparent that the reaction is quite slow at 373°K (212°F), about 10 to 12 hours for more than 90% conversion, while at 436°K (325°F) it is quite rapid - about 10 minutes for more than 90% conversion. The k values (reaction rate constants) and relative rate of reaction (k_x/k 100° C) are given in Table 3-14.

The reaction rate of the 710 varnish is quite similar to the amine catalyzed conventional polyamic acid but quite different from the uncatalyzed polyamic acid. This would lead one to believe that the Monsanto 710 polyamic acid does indeed have a catalyst constituent possibly in the form of an amine salt of the polyamic acid.

Table 3-14. Reaction Rate Constants for 710 Polyimide Varnish

Temperature, °K (°F)	k (min^{-1})	k_x/k, 373°K (121°F)
373 (212)	14.4×10^{-4}	1
398 (257)	5.0×10^{-3}	3.4
436 (325)	17.3×10^{-2}	180

The rate of reaction at 436°K (325° F) is 180 times that at 373°K (212°F). Of special interest are the reaction rates determined by Kreuz* et al for conventional polyamic acid and tertiary amine catalyzed polyamic acids. These values are given in Table 3-15 along with the k determination for Skybond 710 at 436°K (325°F).

The activation energy for the conversion of 710 from polyamic acid to polyimide appears to be about 25 kcal/mole ± 1 kcal/mole. This plot is shown in Figure 3-8. The three points give a nice straight line as they should. However, additional points would be required for a more accurate determination.

*J. A. Kreuz et al, J. Polymer Sci., A-1, 4, 2607 (1966)

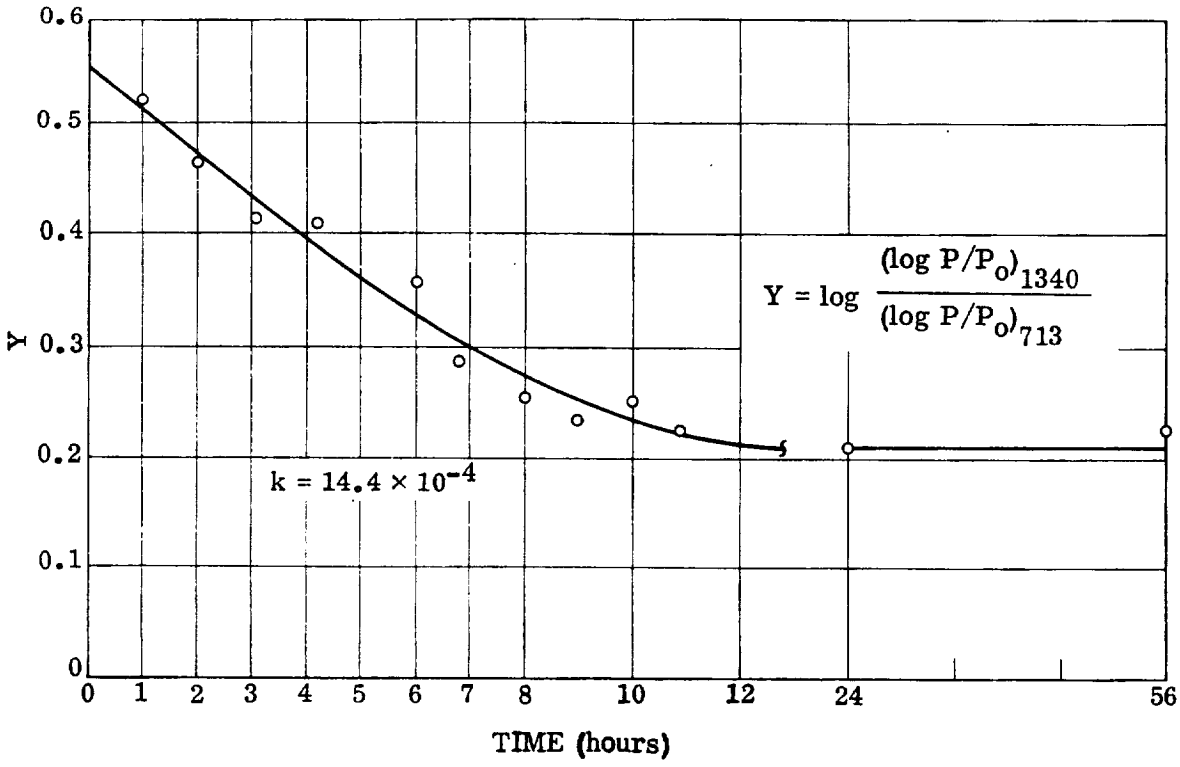


Figure 3-4. Conversion Rate of 710 Polyamic Acid at 373°K (212°F)

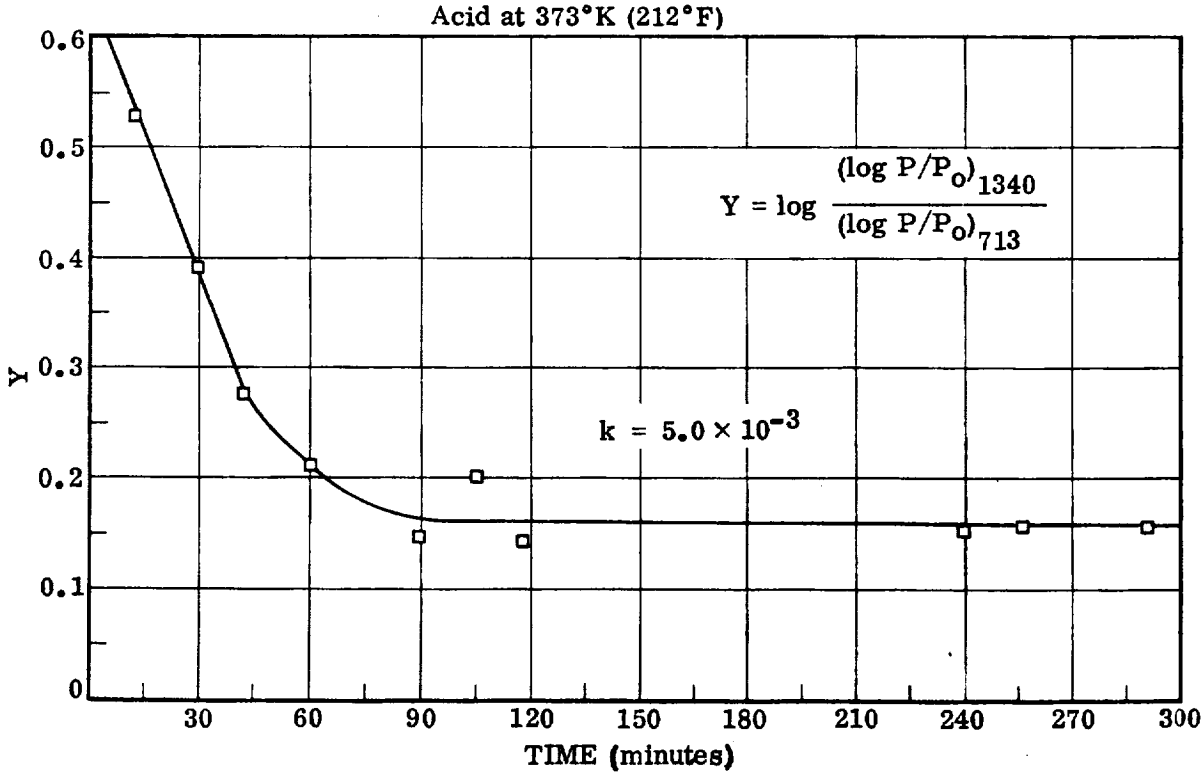


Figure 3-5. Conversion Rate of 710 Polyamic Acid at 398°K (257°F)

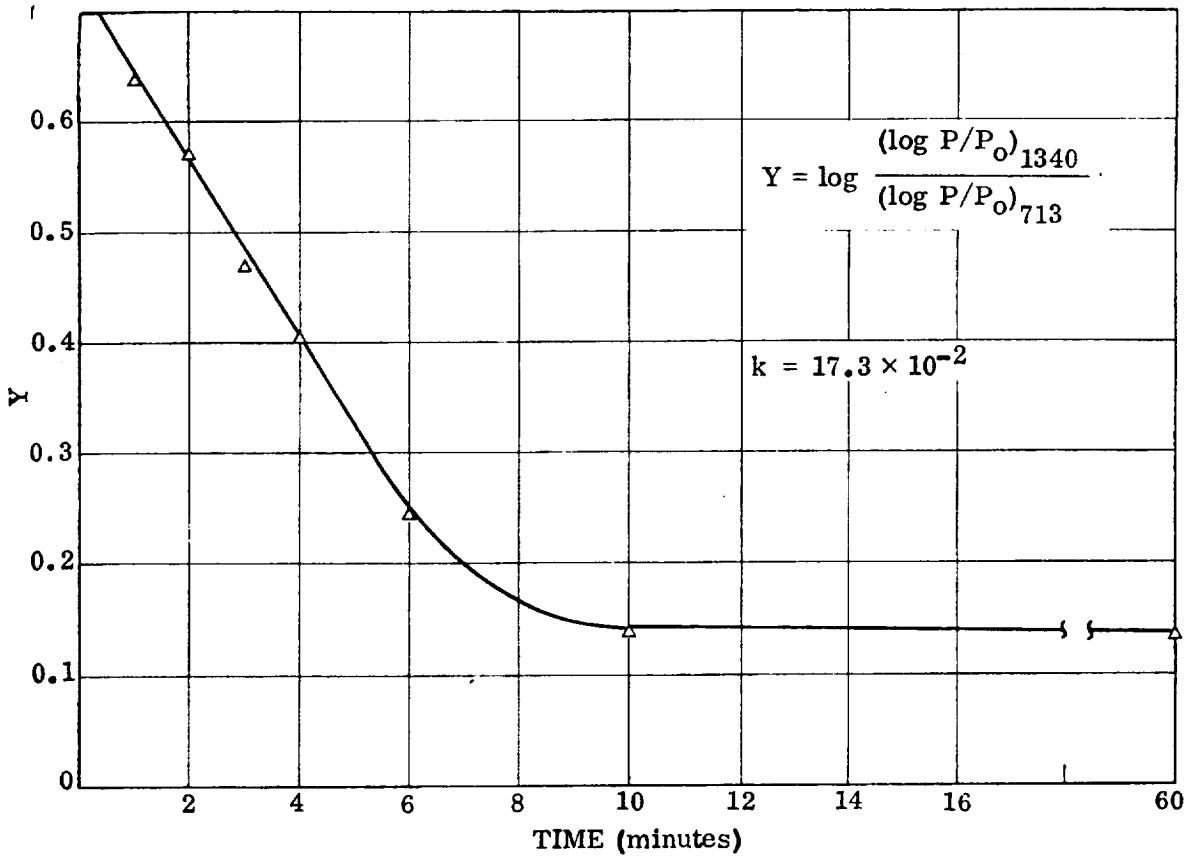


Figure 3-6. Conversion Rate of 710 Polyamic Acid at 436°K (325°F)

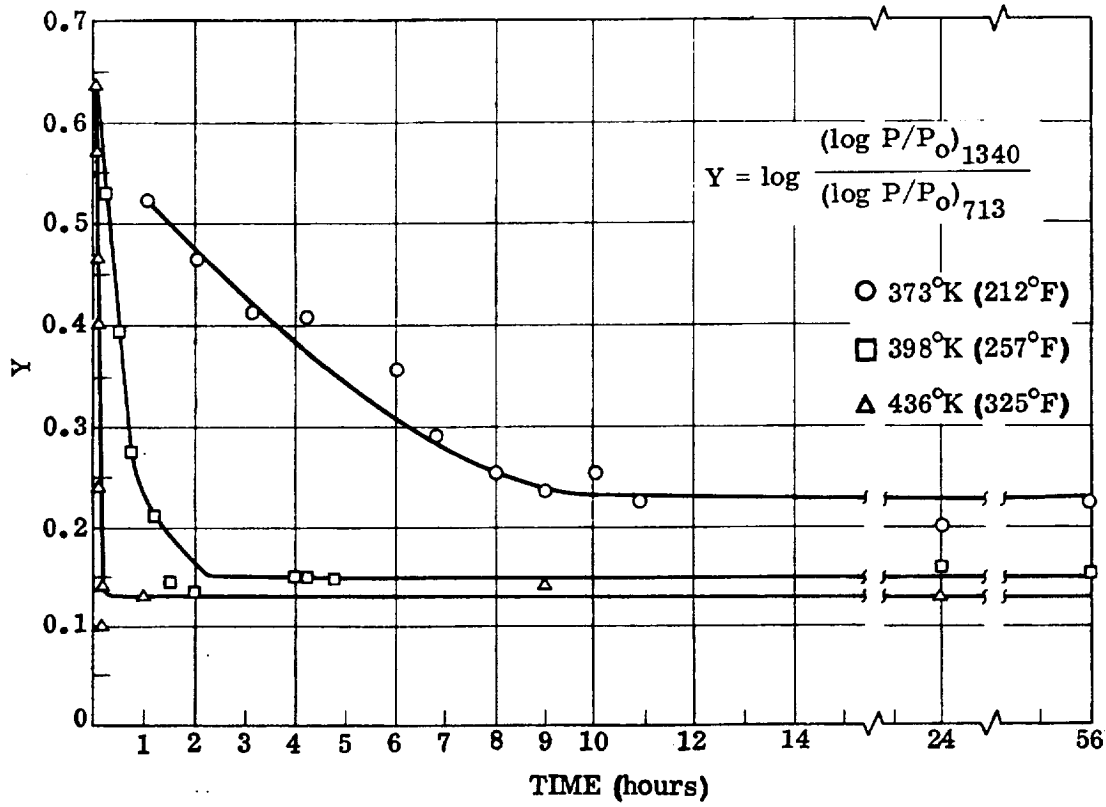


Figure 3-7. Conversion Rates of 710 Polyamic Acid at Several Temperatures

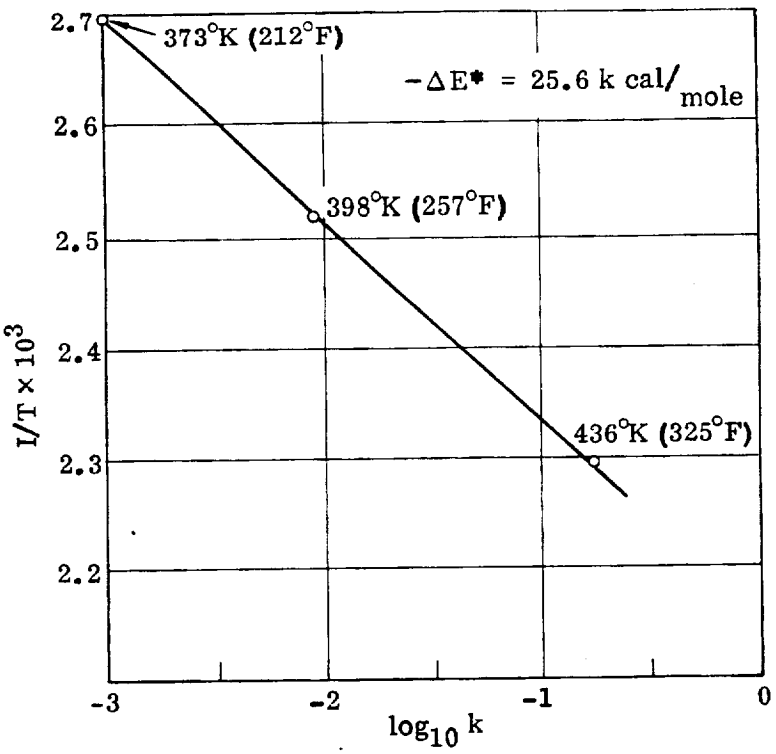


Figure 3-8. Activation Energy for Conversion of 710 Polyamic Acid to Polyimide

Table 3-15. Reaction Rate Constants for Conventional Polyamic Acids

Temperature °K (°F)	k (min^{-1})	Polyamic Acid
434 (322)	1.95×10^{-2}	4,4'-diaminodiphenyl ether with pyromellitic dianhydride
434 (322)	2.06×10^{-1}	4,4'-diaminodiphenyl ether with pyromellitic dianhydride plus tertiary amine catalyst
436 (325)	1.73×10^{-1}	Skybond 710

It became apparent during the study that an aging problem was occurring. There was, however, no significant change in the viscosity or the infrared spectrum of the resin. Titration for acid equivalence showed a rise with aging. The storage life of the resin is about one month at room temperature and three months at 278°K (40°F). However, it is apparent that the acid number should be checked during this time.

The acid equivalent of the polyimide varnish (total free acid) is determined potentiometrically by dissolving approximately 2.0 gm of the Skybond 710 in 60 ml of DMF and titrating in 2 ml aliquots with 0.1000N alcoholic KOH. A graph is then plotted as shown in Figure 3-9 in the appendix. The number of grams per equivalent of KOH is found from:

$$\frac{\text{grams of sample}}{\text{ml of 0.1N KOH}} \times 10^4 = \text{gms/equivalent}$$

For the example in Figure 3-9, the acid equivalent is calculated as follows:

$$\frac{2.1225\text{g}}{39.8} \times 10^4 = 0.0534 \times 10^4$$

$$= 534 \text{ gm/equivalent}$$

Several comparisons of two batches of Skybond 710 are shown in Table 3-16.

The critical acid number is probably in the neighborhood of 530 to 540. That is not to say that the system will not mold when the varnish has an acid equivalent above this, but only that the cure cycle would have to be changed.

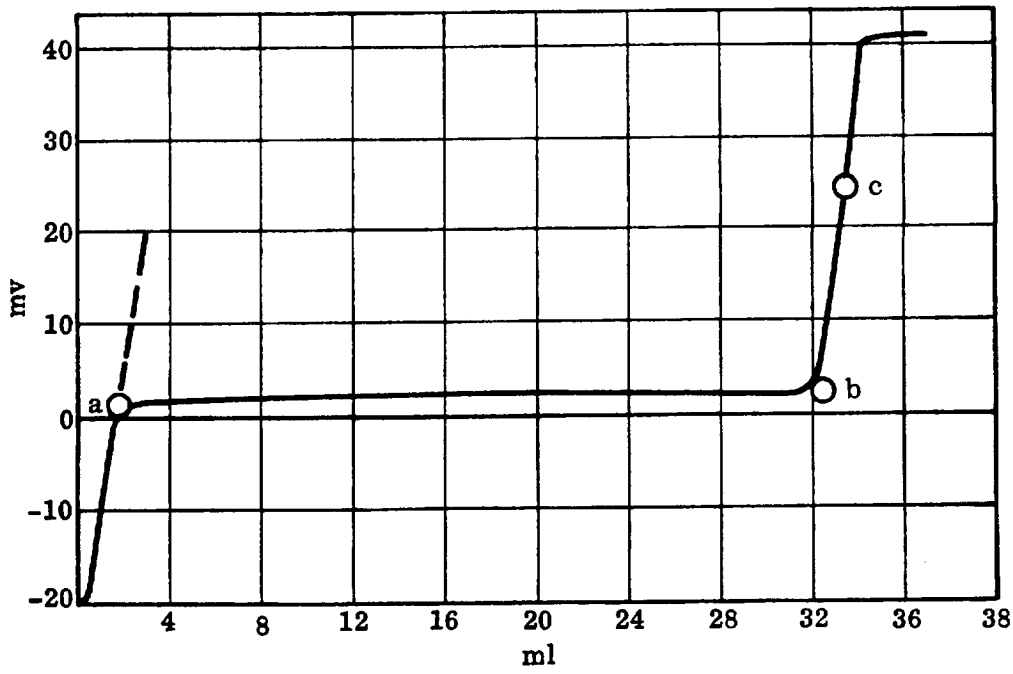


Figure 3-9. Titration of Skybond 710 in DMF with 0.1000N KOH (Alcoholic)

Table 3-16. Comparison of Two Different Lots of Skybond 710

Lot No.	2994	2994	3190	3190
Remarks	3 months storage at 283-288°K (50-60°F)	3 months storage at 283-288°K (50-60°F) + 2 weeks at 299°K (80°F)	As received	1 month at room temperature
Color	Red-Black	Red-Black	Orange/Red/ Brown	Red-Black
Acid Equivalent[a]	584.6	610.3	535.9	597
% Organic	3.8	3.8	4.2	4.2

[a]Based on total varnish

In attempting to interpret the significance of acid equivalence numbers, the following points were considered. See forms A, B, C, D, E, F, G Figure 3-10.

a. Forms A and C of BTDA are tetrafunctional units. The assumption is that if these species predominate they would predict acid equivalent for the resin solution of 260 gm/equivalent.

b. Forms B and D if predominant would be difunctional and would predict an acid equivalent of 520 gm/equivalent.

c. Forms E, F, and G possess no functionality and would raise the number of grams of resin solution/equivalent.

Method I - To eliminate the effect of all but a single acid species, only the volume from point a to point b (31 ml) was included in this calculation. The lowest value obtained by this method was 520 gm/equivalent based on total resin solution.

Method II - To make endpoint titration feasible without recording the potential curve, the total volume to point c (33 ml) was used for calculation. The lowest value obtained by this method was 460 gm/equivalent.

Titration of Skybond 710 yields the potential curve shown in Figure 3-9.

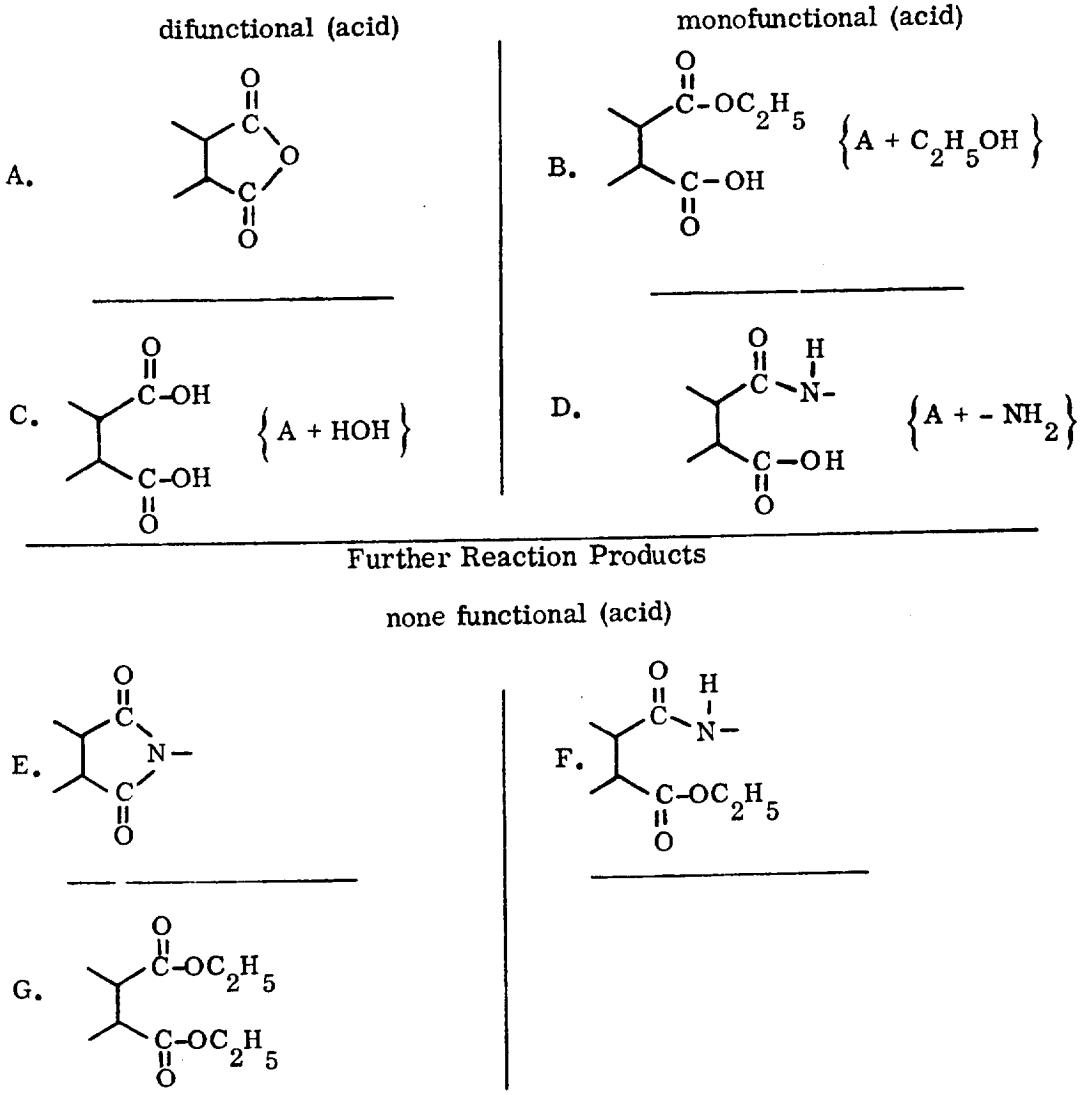


Figure 3-10. Some Possible Species in Skybond 710 of the Anhydride Functional Group

The values 520 gm/equivalent (Method I) and 460 gm/equivalent (Method II) were the most frequent values obtained for newly received lots of Skybond 710. This would imply that either form B or D predominated, or that there was a mixture of A or C/E, F, or G. Based on volatile and viscosity considerations, it was concluded that the resin as received is approximately 90% form B and 10% form C. It is felt Method I acid titration determines the acid from form B plus the second acid group on form C, while Method II determines both acid groups of C plus the acid group of B.

It is also felt the primary cause for the rise in acid number upon aging of the resin is the formation of form F.

In addition, hydrolysis of the amic acid or the amic ester species would result in a rise in the acid number. This is clearly shown in Figure 3-11 and 3-12. Note that such hydrolysis has two observable consequences.

a. The reaction is a chain stopper. High molecular weight is never achieved and laminate properties are therefore low.

b. Salt formation results in the common precipitate found in poor polyimide resin panels. In the case of salt formation, high temperature post cure can set up equilibrium between the salt and the hydrolyzed resin. This is observed by a disappearance of some precipitate. Some improvement in room temperature properties will be observed, but strengths will always be low. Note that good polyimide resin panels show little change of room temperature properties after a high temperature post cure.

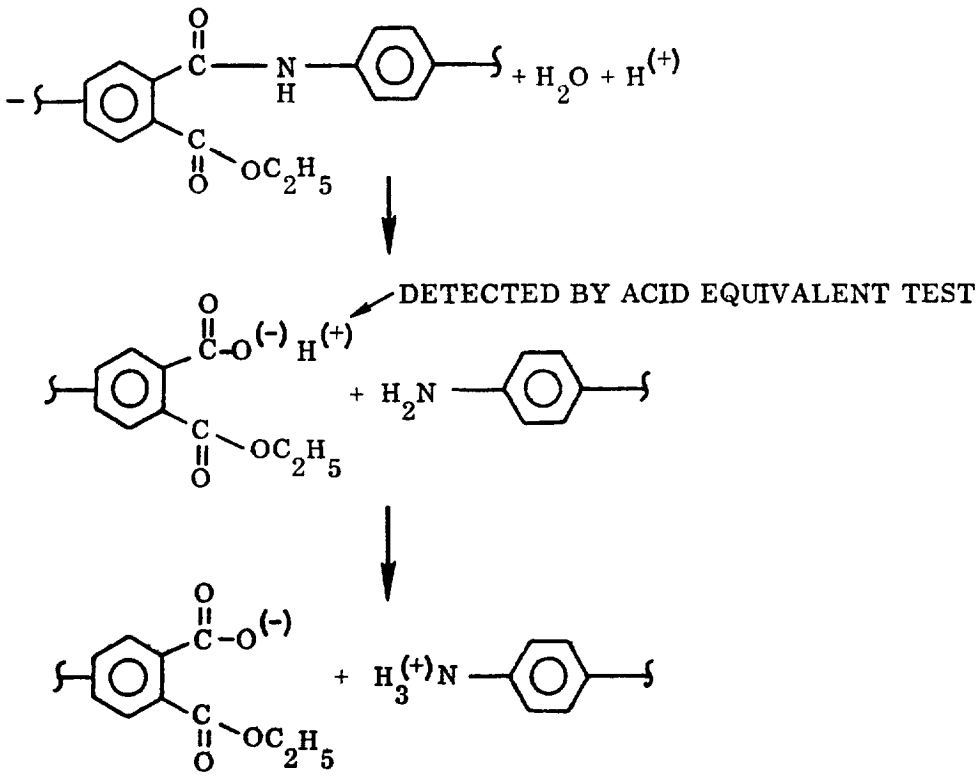


Figure 3-11. Hydrolysis of the Amic Ester

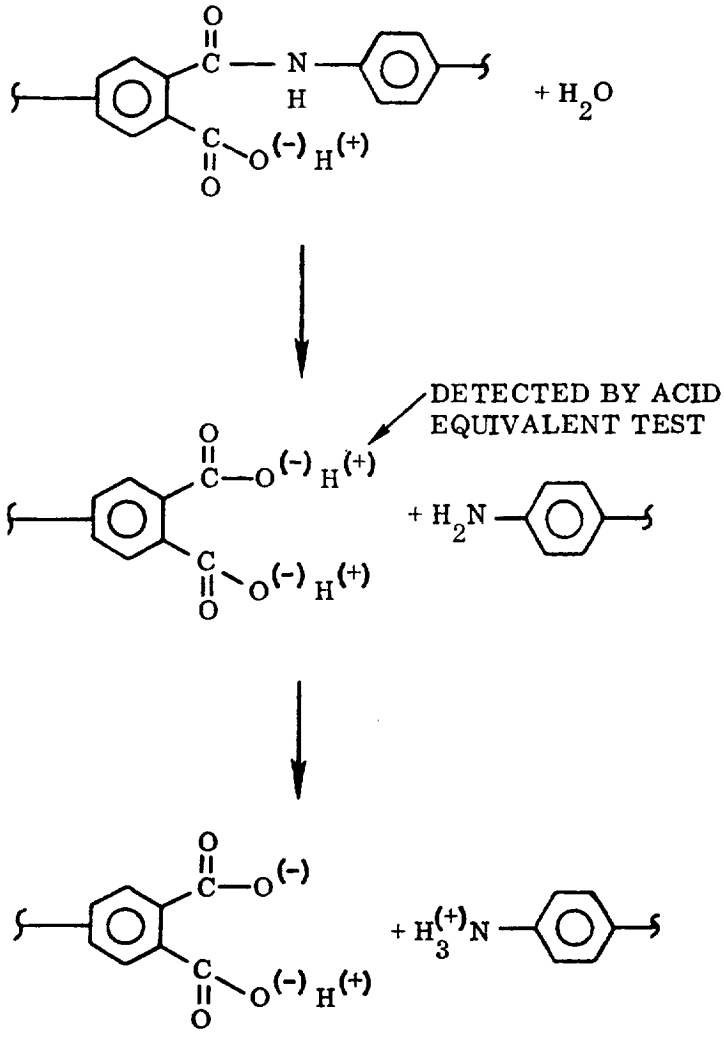


Figure 3-12. Hydrolysis of the Amic Acid

3.4.5 QUALITY CONTROL ASPECTS OF 710. Based on the speculated composition of 710 and on laboratory experiments, the following tests seem to be pertinent to quality control.

3.4.5.1 Resin Solids. Because of the multicomponent nature of the solvent, the objective of the resin solids test must be clearly defined before the test is run.

a. Non-Reactive Volatile. - Fairly consistent results (68 to 72% residue) were obtained with a vacuum strip for 24 hours at 200°K (100°F) and 532 N/m^2 (4 mm Hg). This test is not recommended for routine use because moderate variations in procedure may substantially alter the results. The composition of the residue under these conditions has been determined to be essentially all the original NMP, some xylene, inorganics, the aromatic diamine and BTDA derivatives.

b. Reactive and Non-Reactive Volatile. - A more reproducible method of determining residue is to heat a 2-gm sample one hour at 533°K (500°F). The residue at this point is polyimide and inorganics. The reproducibility of this method on the same lot of material is approximately ±1%, and the range of values obtained has been 51 ± 2%.

3.4.5.2 Inorganics. Two-gram samples were held in air at 810°K (1000°F) for three days. The resulting residue is inorganic, probably a flow control agent such as Cab-O-Sil. The residue obtained by this method is approximately 4% ± 0.5 based on the total initial resin solution. The precision of this method is ± 0.2% on the same lot of material.

3.4.5.3 Recommended QC Procedures - Resin Solids, Total Volatile, Ash. The procedure in running resin solids was to place approximately 2 gm (of Skybond 710) in a preweighed (±0.1 mg), heat cleaned 922°K (1200°F) crucible. The sample is then placedin a muffle furnace at room temperature (to prevent boilover and sample loss), heated to 533°K (500°F), and held one hour. The samples are then removed, cooled in a desiccator, reweighed, and replaced in the muffle furnace. The temperature is reset to 810°K (1000°F) and held for three days. The samples are removed and again cooled in a desiccator and reweighed. Resin solids - 100% = (% volatile + ash), and values range from 46 to 50%.

3.4.5.4 Acid Equivalence for 710. Based on the previous discussion , an acid equivalence number should be determined. For ease of calculation, the number reported is the number of grams of Skybond solution/equivalent. It should be remembered that the number reported in this manner is a function of resin solids. When using Method I to determine acid equivalence, the value should not exceed 540 gm/equivalent. Resin should be closely checked if the value exceeds 520 gm/eq when using Method I. When using Method II, these values become 506 gm/eq and 460 /eq respectively.

3.4.5.5 Miscellaneous QC Procedures for 710. Other quality control methods used include refractive index, Sunshine gel at 404°K (266°F), and Brookfield viscosity. Acceptable value ranges are presented below:

Brookfield Viscosity (spindle #2 at 12 rpm)	2000 to 2600 cps
Gel Time [Sunshine Gel Meter @ 404°K (266°F)]	27 to 33 minutes
Refractive Index	1.5210 to 1.5250

3.4.5.6 Summary of 710 QC Values.

Brookfield Viscosity	2000 to 2600 cps
Refractive Index	1.5210 to 1.5250
Gel Time	30 minutes ± 3
Residue 533°K (500°F), 1 hour	51% ± 2
Residue 810°K (1000°F), 72 hours	4.0% ± 0.5
Acid Equivalence (Method II)	460 ± 25

3.4.6 RHEOLOGICAL ASPECTS OF 710. 710 was vacuum stripped for 15 hours at 298°K (78°F) to remove the majority of the solvent. Various rheological characteristics of the residue were determined at three different heating rates: 1, 3, and 5°K (2, 5, and 9°F) minute using a Fisher-Johns melting point apparatus. Five different characteristics of the 710 resin was determined (Figure 3-13). They are 1) melting point, 2) initial flow, 3) good flow, 4) starting to gel, 5) gellation. Points 1, 4, and 5 are accurate within ± 3°K (5°F). Points 2 and 3 are accurate within ± 7°K (13°F) since they required a much more subjective decision by the instrument operator. In general, the initial melting point and flow temperature are independent of heating rate, whereas the temperature required for good flow and gellation is dependent on heating rate. While this study was performed on the resin, no great differences would be expected with graphite/710 prepreg because of the high thermal conductivity of the graphite.

3.5 POLYIMIDE/GRAPHITE PREPREG CHARACTERIZATION

The following are nominal prepreg values for HT-S/710 graphite prepreg. Using the Convair Aerospace developed cure cycle, optimum composite properties will be obtained from prepreg having the following values:

Resin Solids	40% ± 5
Volatile	15% ± 5
Flow	20% ± 5
Gel at 450°K (350°F)	1 to 3 minutes

3.5.1 PREPREG GEL AND FLOW AS A FUNCTION OF TEMPERATURE. Figure 3-14 presents data on HT-S/710 prepreg gel time and flow at 393, 422, and 450°K (250, 300, and 350°F). The gel time decreased quite markedly with an increased temperature as expected. The flow, however, remained constant.

3.5.2 STAGING OF HT-S/710 PREPREG. Table 3-17 presents prepreg volatile, tack, drape, gel time, and flow as a function of staging. The gel time test at 436°K (325°F) (Fisher-Johns) appears to be a poor test for this type of resin system since resin flow can still be accomplished by pressure after the prepreg has apparently gelled by the Fisher-Johns method.

Volatile loss versus time at temperature for HT-S/710 prepreg is given in Figure 3-15 for 323 and 348°K (122 and 167°F). In both cases, the prepreg had become boardy after the first half-hour. A similar experiment was run for total volatiles at 450°K (350°F), and the data is plotted versus staging time in Figure 3-10. Again, the prepreg became boardy after the first half-hour, indicating that 15% volatile is the minimum volatile content at which drape and tack can be retained at 45% resin solids.

Table 3-17. 710, Prepreg Characteristics as a Function of Staging Conditions

Staging Conditions	Volatile Remaining (%)	Tack Top	Tack Back	Drape	Gel Time 436°K (325°F)	% Flow 436°K (325°F)	Comments
Initial	20	Good	Good	Good	2 min.	20.5	Prepreg hard to handle
10 minutes at 353°K (176°F)	12.6	Poor	Good	Fair	1 min.	17.5	15 minutes at 353°K (176°F) gives good workable prepreg
30 minutes at 353°K (176°F)	10.5	None	Fair	Poor	45 sec.	17.3	
120 minutes at 353°K (176°F)	8.8	None	None	None	gelled	9.0	
5 minutes at 433°K (320°F)	5.3	None	None	None	gelled	1.1	
15 minutes at 433°K (320°F)	2.0	None	None	None	gelled	0.1	Very poor bond between flow pieces
60 minutes at 433°K (320°F)	0.5	None	None	None	gelled	0	No bond between flow pieces

The break in the 348°K (167°F) curve after 24 hours indicates a small amount of reaction volatile. At 323°K (122°F), there seems to be a negligible amount of this reaction.

Figure 3-17 presents data on HT-S/710 prepreg in regard to the temperature at which the prepreg softens and flows as a function of staging time at 353°K (176°F). This data was obtained using a Fisher-Johns melting point apparatus.

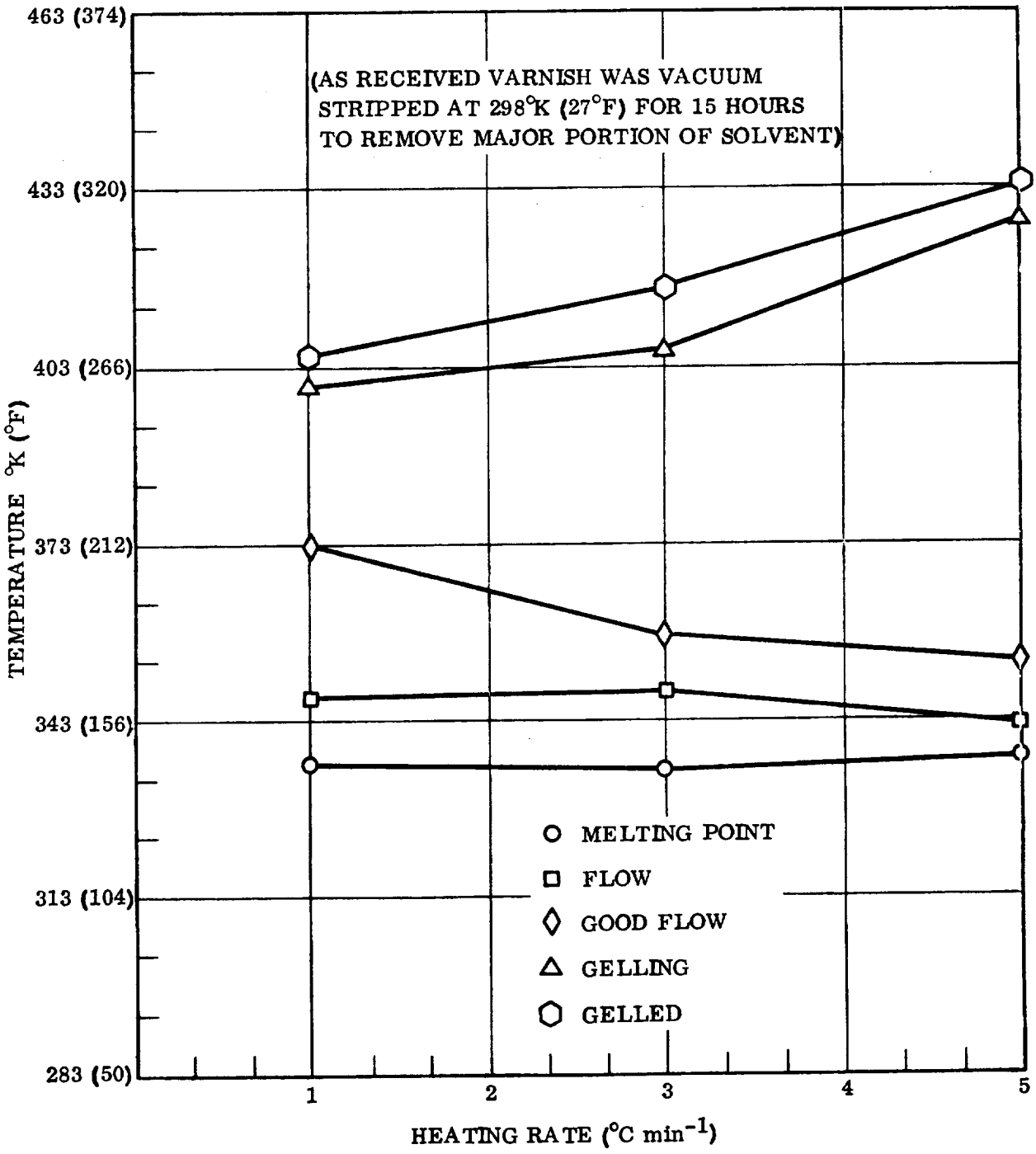


Figure 3-13. Rheological Aspects of 710 Polyimide Resin

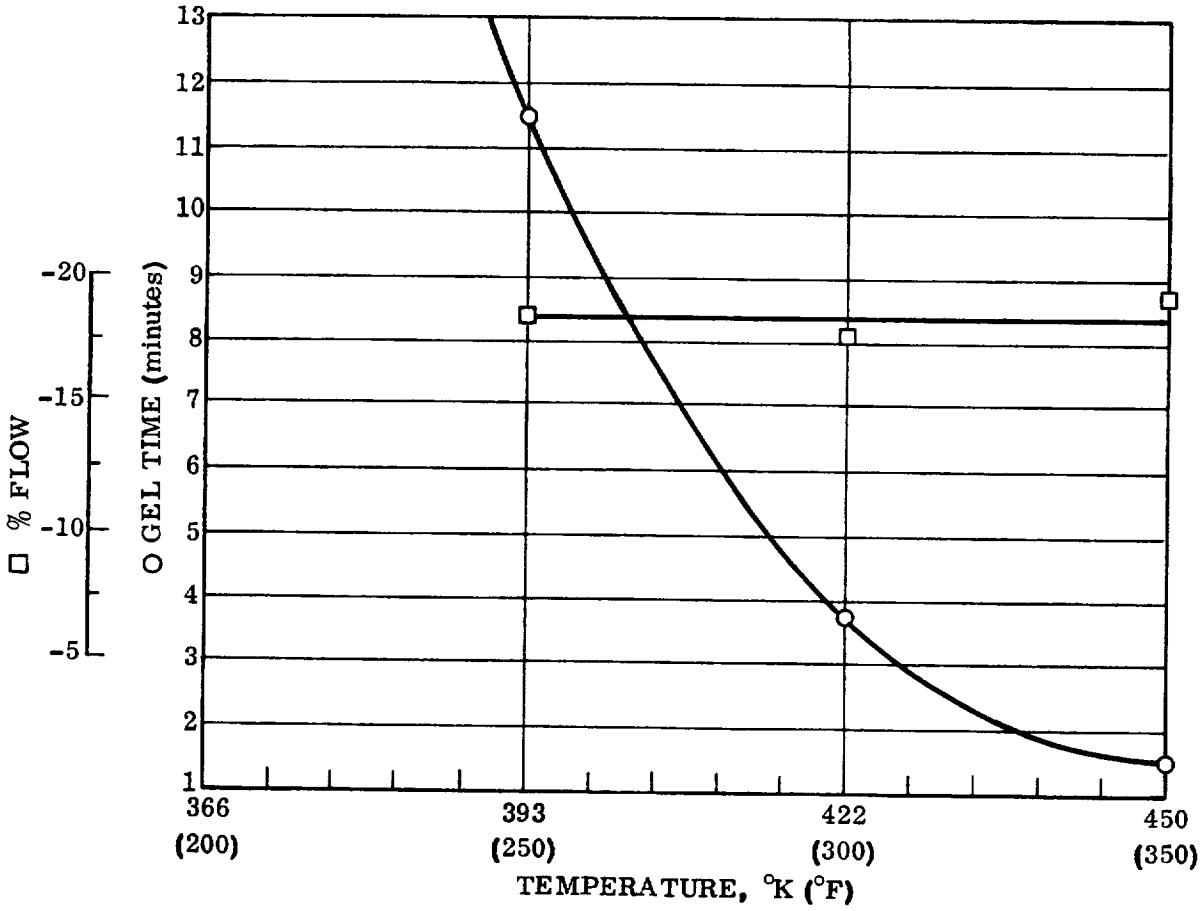


Figure 3-14. Gel Time and Resin Flow of HT-S/710 Prepreg at Several Temperatures (Prepreg Resin Solids 42.9%, Volatile Content 20.8%)

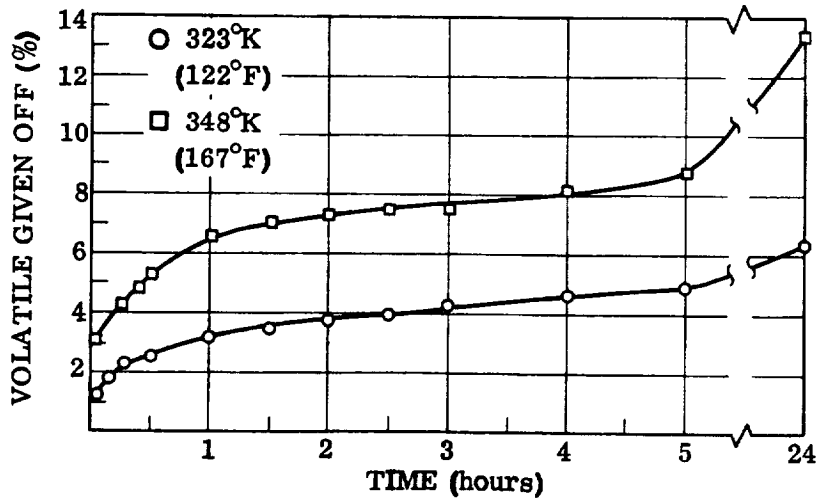


Figure 3-15. HT-S/710 Prepreg Volatile Loss at Several Staging Temperatures (Prepreg Resin Solids 45.5%, Volatile Content 19.8%)

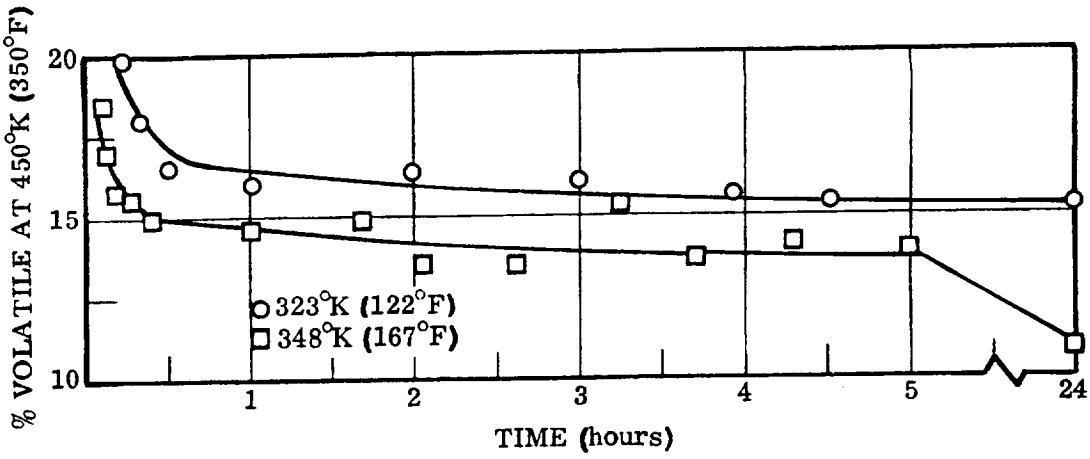


Figure 3-16. HT-S/710 Prepreg Volatile Content Versus Staging Time at Several Temperatures (Prepreg Resin Solids 46.2%, Volatile Content 21.5%)

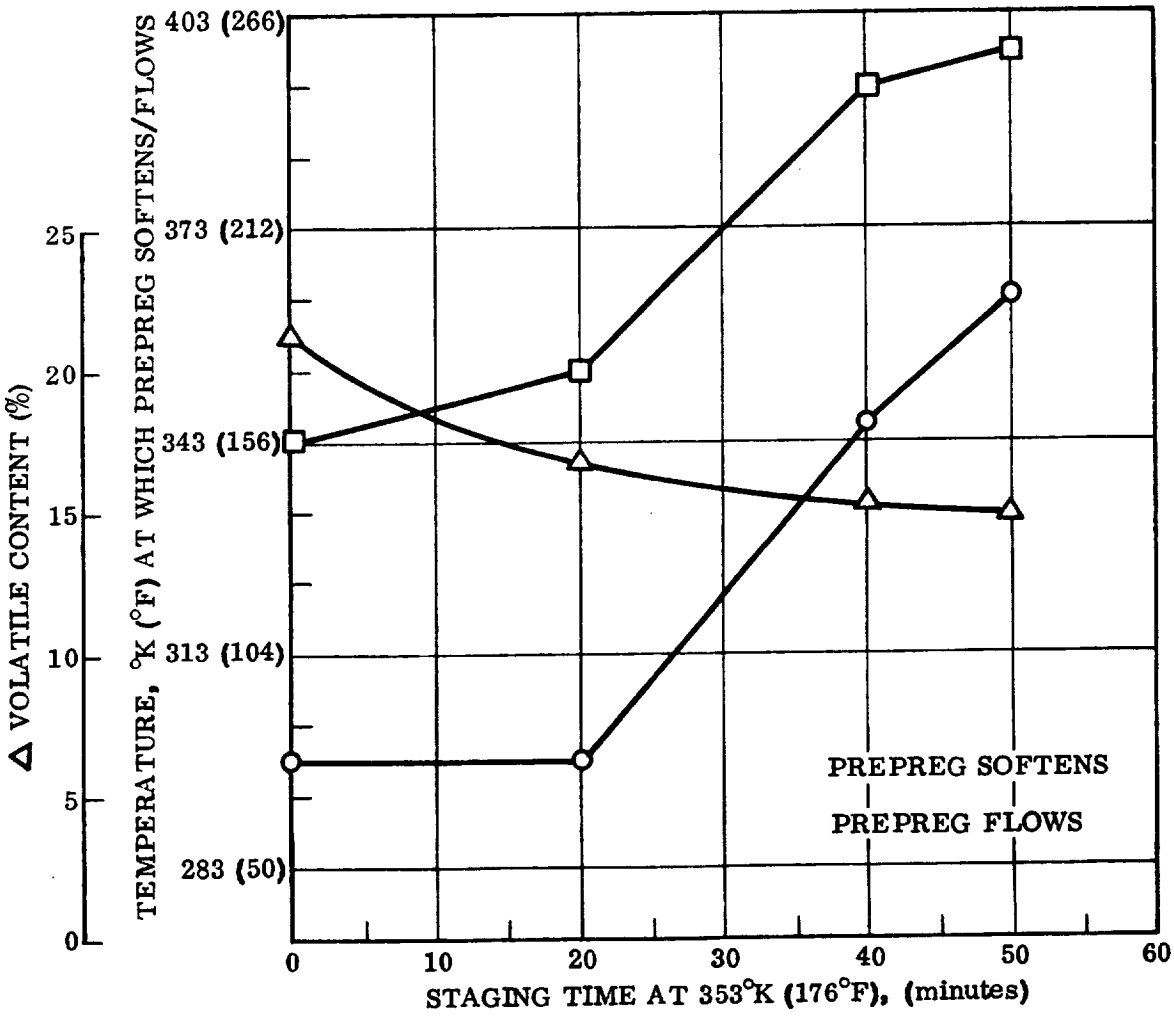


Figure 3-17. HT-S/710 Prepreg Softening and Flow Point and Volatile Content as a Function of Staging at 353°K (176°F)

Table 3-18. HT-S/710 Prepreg* Storage Stability at 298°K (78°F) Room Temperature

Time	Volatile[a] Content	% Resin[a] Flow	Gel Time[a] (minutes)	Tack	Drape
1 day	21.0	22.6	1.8	Good	Pliable
3 days	17.9	19.0	1.5	Fair	Pliable
1 week	19.2	15.0	1.4	Fair	Pliable
2 weeks	14.8	19.1	1.0	Poor	Boardy
3 weeks	15.4	20.0	1.0	Poor	Boardy
4 weeks	16.9	18.6	1.0	Poor	Boardy
5 weeks	16.2	10.8	1.0	Poor	Boardy
6 weeks	16.7	16.3	1.0	Poor	Brittle
7 weeks	16.7	17.5	1.0	Poor	Brittle
8 weeks	16.0	17.8	1.0	Poor	Brittle
9 weeks	15.8	18.5	1.0	Poor	Brittle
10 weeks	14.3	17.8	1.0	Poor	Brittle
11 weeks	15.6	17.8	1.0	Poor	Brittle

Table 3-19. HT-S/710 Prepreg* Storage Stability at 278°K (40°F)

Time	Volatile[a] Content	% Resin[a] Flow	Gel Time[a] (minutes)	Tack	Drape
1 day	21.2	22.2	1.7	Good	Pliable
3 days	20.2	21.8	1.5	Good	Pliable
1 week	19.7	20.9	1.5	Good	Pliable
2 weeks	15.3	23.8	1.5	Good	Pliable
3 weeks	14.0	24.2	1.5	Good	Pliable
4 weeks	18.5	21.9	1.6	Good	Pliable
5 weeks	19.4	22.7	1.6	Good	Pliable
6 weeks	18.3	20.8	2.0	Good	Pliable
7 weeks	18.5	19.9	1.8	Good	Pliable
8 weeks	21.0	16.6	1.5	Good	Pliable
9 weeks	16.1	20.0	1.5	Good	Pliable
10 weeks	21.6	20.2	1.7	Good	Pliable
11 weeks	16.3	20.8	1.7	Good	Pliable
12 weeks	20.3	18.2	1.6	Good	Pliable
13 weeks	17.8	21.0	1.5	Good	Pliable
14 weeks	19.5	22.3	1.6	Good	Pliable
15 weeks	16.2	20.6	1.2	Fair	Boardy

*Initial Prepreg Properties. Resin Solids 46.5%, Volatile Content 20.1%

[a]Measured at 450°K (350°F)

Table 3-20. HT-S/710 Prepreg* Storage Stability at 255°K (0°F)

Time	Volatile[a] Content	% Resin[a] Flow	Gel Time[a] (minutes)	Tack	Drape
1 day	21.2	22.8	1.6	Good	Pliable
3 days	20.6	23.0	1.8	Good	Pliable
1 week	19.4	18.9	1.6	Good	Pliable
2 weeks	18.5	22.3	1.6	Good	Pliable
3 weeks	18.5	22.0	1.5	Good	Pliable
4 weeks	21.4	21.0	1.7	Good	Pliable
5 weeks	22.2	20.8	1.7	Good	Pliable
6 weeks	21.9	18.1	1.8	Good	Pliable
7 weeks	17.5	19.2	1.6	Good	Pliable
8 weeks	18.9	19.5	1.8	Good	Pliable
9 weeks	19.1	21.6	1.5	Good	Pliable
10 weeks	18.9	20.7	1.4	Good	Pliable
11 weeks	21.3	17.1	1.4	Good	Pliable
12 weeks	20.1	17.5	1.5	Good	Pliable
13 weeks	19.9	18.0	1.5	Good	Pliable
14 weeks	17.8	20.2	1.4	Good	Pliable
15 weeks	18.0	19.7	1.4	Fair	Pliable

*Initial Prepreg Properties: Resin Solids 46.5%, Volatile Content 20.1%

[a]Measured at 450°K (350°F)

3.5.3 HT-S/710 PREPREG STORAGE STABILITY. Tables 3-18, 3-19 and 3-20 present data on volatile content, % resin flow, gel time, tack, and drape as a function of aging for HT-S/710 prepreg. Table 3-18 shows storage at room temperature, and Tables 3-19 and 3-20 are storage at 278°K (40°F) and 255°K (0°F) respectively. At room temperature, properties show a dramatic falloff at one week. A corresponding falloff is not observed until about 14 weeks when storage is at 278° K (40° F). After 15 weeks at 255° K (0° F), the prepreg was still pliable and retained fair tack.

3.6 GRAPHITE/POLYIMIDE PROCESS DEVELOPMENT

One of the major objectives of this program was to develop vacuum bag, press, and autoclave cure cycles for graphite/polyimide composites. To meet this objective 30.5 by 30.5-cm (12 by 12-in.) by 12-ply laminates were fabricated using various cure cycles. Each of these panels was then cut into 15.2 by 15.2-cm (6 by 6-in.) panels, as shown in Figure 3-18. Flexural and short-beam shear tests were conducted on the postcured panels. Also, resin content, fiber volume, and specific gravity of the individual panels were determined.

The graphite/polyimide process optimization plan followed during these studies is shown in Figure 3-19. This work, combined with the resin development and evaluation, was used in fully developing the cure cycle used for the fabrication of the design laminates, thick laminates, tubes, and structural test elements. Each of the individual parts of the process development study is discussed in the following paragraphs.

3.6.1 VACUUM-PRESSURE AUGMENTED CURE STUDY (PRESS). Based on previous Convair-Aerospace-sponsored studies, four cure cycles, where pressure, heating

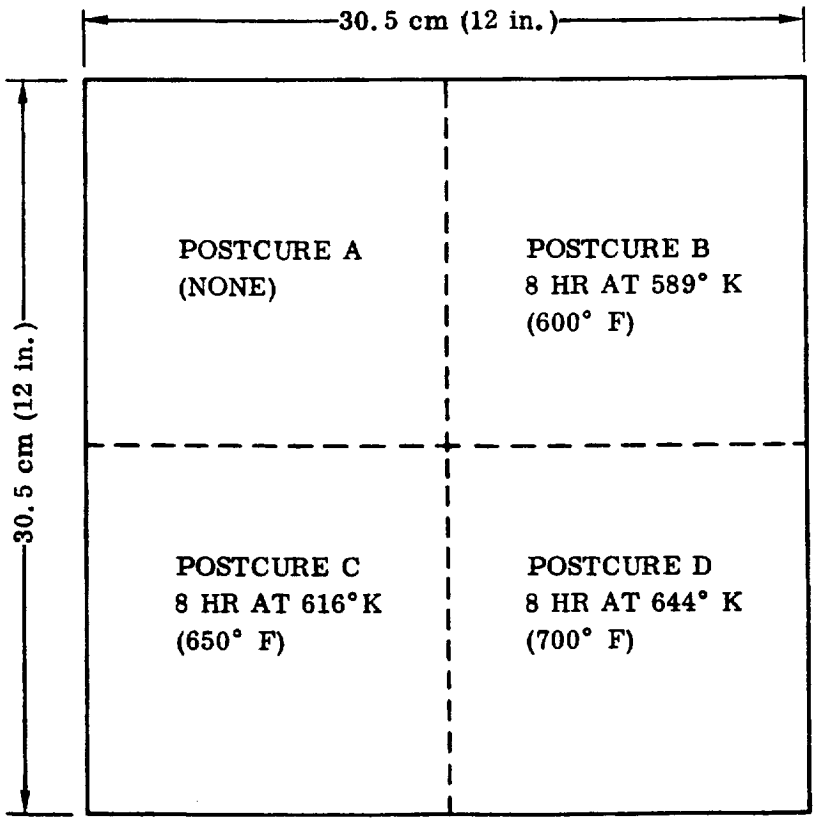


Figure 3-18. Panel Description for Cure and Postcure Studies (HT-S/710)

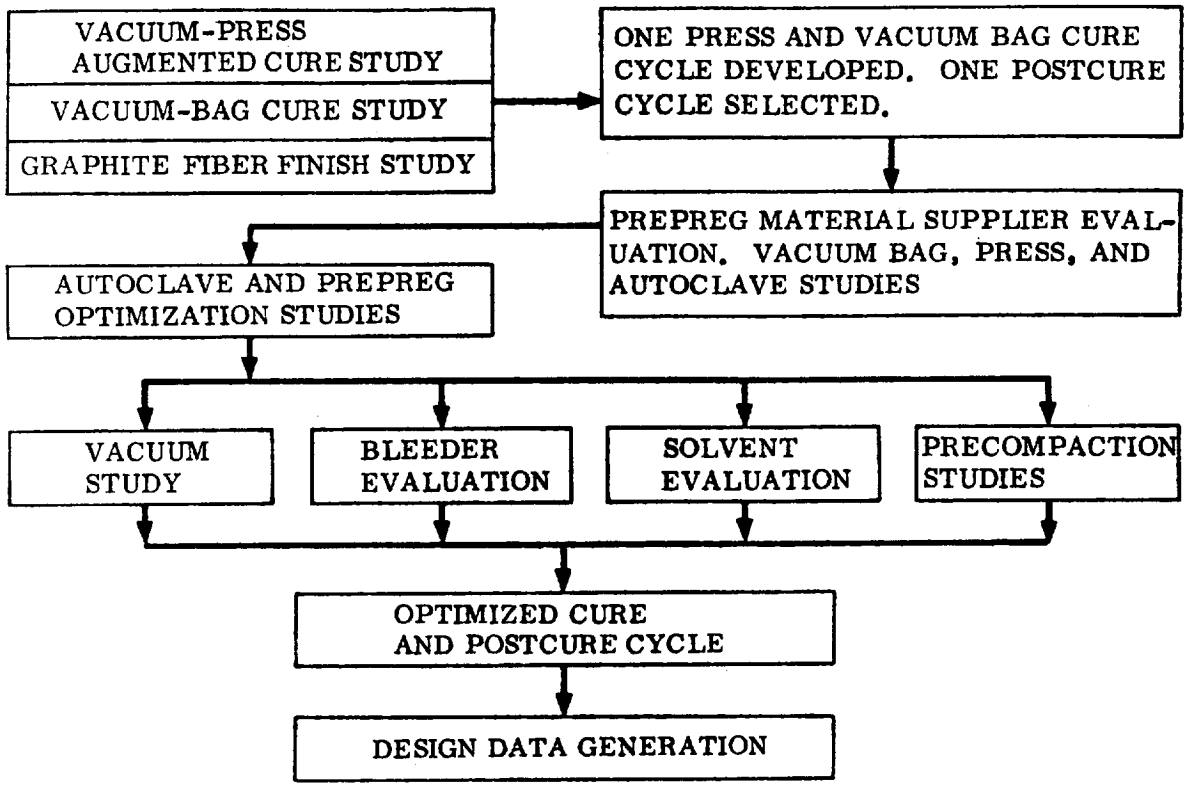


Figure 3-19. Graphite/Polyimide Process Optimization Flow Chart

rates, and bleeder were varied, and four postcure cycles were evaluated. In all press cures, the laminate layup was continuously under vacuum. The following is a list of processing techniques and cure procedures for each cure evaluated:

a.	Material Designation:	(hy-E-1312-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5-cm (12 by 45-in.) sheet
	Batch No.:	OC-28
	Resin:	710
	Manufacturer:	Fiberite
b.	Fabrication Procedure:	(Press Cure No. 1)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5 cm (12 by 12 in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	5 plies Mauchburg paper CW-1850; 2 plies on the bottom and 3 plies on the top.

	Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg vacuum was applied at room temperature and maintained throughout the entire cure cyle and cool down to below 352° K (175° F). 690 kN/m^2 (100 psi) press pressure was applied at 431°K (315°F) and maintained throughout the remainder of the cure.
	Cure Cycle:	Heat to 450°F (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, and cool at no greater than 1° K/minute (3° F/minute) to 352° K (175° F) under pressure.
	Postcure:	Various ones.
c.	Material Designation:	(hy-E-1312-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5-cm (12- by 45-in.) sheet
	Batch No.:	OC-28
	Resin:	710
	Manufacturer:	Fiberite
d.	Fabrication Procedure:	(Press Cure No. 2)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5-cm (12- by 12-in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	5 plies Mauchburg paper CW-1850; 2 plies on the bottom and 3 plies on the top.
	Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.

Cure Pressure:	760 mm (29 in.) Hg vacuum was applied at room temperature and maintained throughout the entire cure cycle and cool to below 352° K (175° F). 690 kN/m^2 (100 psi) press pressure was applied at 431°K (315°F) and maintained throughout the remainder of the cure.
Cure Cycle:	Heat to 355°K (180°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 408°K (275°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 1 hour and cool at no greater than 1° K/minute (3° F/minute) to 352°K (175°F) under pressure.
Postcure:	Various ones.

e.

Material Designation:	(hy-E-1312-B)
Fiber Type:	HT-S (staple)
Material Form:	30.5 by 114.5-cm (12- by 45-in.) sheet
Batch No.:	OC-28
Resin:	710
Manufacturer:	Fiberite

f.

Fabrication Procedure:	(Press Cure No. 3)
Mold Release:	Teflon Film
Layup:	12 plies unidirectional, 30.5 by 30.5-cm (12- by 12-in.)
Separator Film:	Teflon-coated glass cloth, FGO-3
Bleeder:	1 ply style 104 glass cloth and 2 plies of style 181 glass cloth on both sides of the layup.
Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
Cure Pressure:	760 mm (29 in.) Hg. vacuum was applied at room temperature and maintained throughout the entire cure cycle and cool down to below 352° K (175° F). 690 kN/m^2 (100 psi) press pressure was applied at 431° K (315° F) and maintained throughout the remainder of the cure.
Postcure:	Various ones

g.

Material Designation:	(hy-E-1312-B)
Fiber Type:	HT-S (staple)
Material Form:	30.5 by 114.5-cm (12- by 45-in.) sheet
Batch No.:	OC-28
Resin:	710
Manufacturer:	Fiberite

h.

Fabrication Procedure:	(Press Cure No. 4)
Mold Release:	Teflon Film
Layup:	12 plies unidirectional, 30.5 by 30.5-cm (12- by 12-in.)
Separator Film:	Teflon-coated glass cloth, FGO-3
Bleeder:	1 ply style 104 glass cloth and 2 plies of style 181 glass cloth on both sides of the layup.
Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4 in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
Cure Pressure:	760 mm (29 in.) Hg vacuum was applied at room temperature and maintained throughout the entire cure cycle and cool down to 352° K (175° F). 690 kN/m^2 (100 psi) press pressure was applied at 431° K (315° F) and maintained throughout the remainder of the cure.
Cure Cycle:	Heat to 355°K (180°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 408°K (275°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 450°K (350°F) at 1 to 3°K/minute, hold 1 hour and cool at no greater than 1° K/minute (3° F/minute) to 352° K (175° F) under pressure.
Postcure:	Various ones.

The following four postcure cycles were evaluated:

1. No postcure
2. 8 hours at 589°K (600°F)
3. 8 hours at 616°K (650°F)
4. 8 hours at 644°K (700°F)

Each of the panels was slowly heated to 450°K (350°F) and remained at this temperature for two hours prior to raising the temperature to the higher postcure temperatures. Temperature increases were in increments of 28° K (50° F) with dwell times of 2 hours at each increment. Postcures 2, 3, and 4 all concluded with 8 hours at the selected postcure temperatures. The panels were all unrestrained during the postcure cycles.

Longitudinal and transverse flexural and short-beam shear specimens were machined from each of the panels. A minimum of three specimens were tested at each of the three test temperatures 77°K (-320°F), 297°K (75°F), and 589°K (600°F). Duplicate tests were conducted to determine resin content (percent by weight), fiber volume, and specific gravity.

The data developed during this study is reported in Tables 3-21 through 3-24. The method used in evaluating the cure cycles was to rank the flexural and short-beam shear strengths on the basis of 4, 3, 2, 1 where a rank of 4 is given for the highest strength. Using this system of ranking, cure cycle No. 2 was found to be far superior than any of the other cycles evaluated in this study. The selected press cure cycle is amenable to the fabrication of large parts in that the heating rates are reasonable, there are various hold times, and the pressure required for curing the laminate is only 690 kN/m^2 (100 psi).

The data developed during the press cure study aimed at selecting the best postcure cycle is presented in Tables 3-25 through 3-28. The same method of ranking was used to evaluate the postcure cycles as in evaluating the cure cycles. Postcure cycle D was found to be superior for overall properties for the temperature range of 77°K (-320°F) to 589°K (600°F). However, if the temperature range for a particular application is from 77°K (-320°F) to 450°K (350°F), postcure cycle A is the recommended cycle. This is not too surprising in that a more thorough cure and a higher temperature postcure closes more rings and extends polymer chain length. The high temperature postcure in air also causes some crosslinking in the polyimide resin. This crosslinking increases strength, particularly at high temperatures and reduces the sites available for the initiation of oxidative and thermal degradation.

This same process also reduces the mobility of the polymer chain and increase the steric hindrance in the cured system. This leads to an increase in polymer brittleness and potential premature failure at low cryogenic temperatures. However, even though

Table 3-21. Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 1

Property and Test Temperature	PRESS CURE CYCLES -OC-28								OC-31	
	1		2		3		4		3	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure										
77°K (-320°F)	1284	(185)	1336	(194)	1233	(179)	1049	(152)	955	(138)
297°K (75°F)	1336	(194)	1243	(180)	1255	(182)	984	(143)	1184	(172)
589°K (600°F)	174	(25)	186	(27)	195	(28)	148	(21)	181	(26)
90° Flexure										
297°K (75°F)	23	(3.3)	15	(2.2)	23	(3.4)	32	(4.6)	25	(3.6)
Short Beam Shear										
77°K (-320°F)	38	(5.5)	29	(4.2)	24	(3.5)	20	(2.9)	30	(4.4)
297°K (75°F)	44	(6.4)	34	(5.0)	31	(4.5)	28	(4.1)	40	(5.8)
589°K (600°F)	26	(3.8)	27	(3.9)	26	(3.7)	26	(3.7)	22	(3.2)
% Fiber Vol.	51.7		55.0		58.3		57.6		59.0	
% Resin	43.4		38.7		36.5		37.1		35.9	
Specific Gravity	1.53		1.48		1.49		1.43		1.48	

Table 3-22. Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 2

Property and Test Temperature	PRESS CURE CYCLES OC-28								OC-31	
	1		2		3		4		3	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure										
77°K (-320°F)	1051	(152)	1059	(154)	923	(134)	980	(142)	849	(123)
297°K (75°F)	842	(122)	1257	(182)	1076	(156)	1068	(155)	750	(109)
589°K (600°F)	547	(79)	664	(96)	384	(56)	416	(60)	381	(55)
90° Flexure										
297°K (75°F)	5	(0.7)	16	(2.3)	24	(3.5)	19	(2.7)	23	(3.3)
Short Beam Shear										
77°K (-320°F)	37	(5.3)	36	(5.2)	30	(4.4)	33	(4.8)	20	(2.9)
297°K (75°F)	47	(6.8)	56	(8.1)	49	(7.1)	44	(6.4)	30	(4.4)
589°K (600°F)	28	(4.1)	34	(4.9)	27	(3.9)	27	(3.9)	21	(3.0)
% Fiber Vol.	58.0		66.3		59.9		61.8		64.9	
% Resin	37.0		28.7		35.3		34.1		30.1	
Specific Gravity	1.54		1.49		1.46		1.48		1.50	

Table 3-23. Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 3

Property and Test Temperature	PRESS CURE OC-28				OC-31
	1	2	3	4	3
	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)
0° Flexure					
77°K (-320°F)	848 (123)	1093 (158)	888 (129)	741 (107)	825 (120)
297°K (75°F)	1215 (176)	1155 (168)	1123 (163)	971 (141)	950 (138)
589°K (600°F)	702 (102)	747 (108)	565 (80)	559 (80)	625 (89)
90° Flexure					
297°K (75°F)	23 (3.4)	23 (3.3)	23 (3.4)	25 (3.6)	17 (2.4)
Short Beam Shear					
77°K (-320°F)	47 (6.8)	47 (6.8)	37 (5.4)	30 (4.3)	28 (4.1)
297°K (75°F)	46 (6.6)	55 (8.0)	42 (6.1)	37 (5.4)	39 (5.7)
589°K (600°F)	30 (4.3)	34 (4.9)	27 (3.9)	26 (3.8)	30 (4.3)
% Fiber Vol.	57.0	57.4	62.1	57.9	61.9
% Resin	38.0	37.6	32.8	37.1	33.1
Specific Gravity	1.50	1.51	1.46	1.44	1.44

Table 3-24. Press Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 4

Property and Test Temperature	PRESS CURE CYCLES OC-28				OC-31
	1	2	3	4	3
	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)	MN/m^2 (ksi)
0° Flexure					
77°K (-320°F)	885 (128)	821 (119)	835 (121)	652 (95)	830 (120)
297°K (75°F)	1061 (154)	1192 (173)	1169 (170)	1061 (154)	953 (138)
589°K (600°F)	727 (105)	848 (123)	757 (110)	652 (98)	582 (84)
90° Flexure 297°K (75°F)	25 (3.6)		28 (4.1)	23 (3.3)	
Short Beam Shear					
77°K (-320°F)	45 (6.5)	49 (7.1)	45 (6.5)	44 (6.4)	28 (4.1)
297°K (75°F)	46 (6.6)	53 (7.7)	48 (6.9)	49 (7.1)	36 (5.2)
589°K (600°F)	29 (4.2)	34 (5.0)	35 (5.1)	35 (5.1)	28 (4.1)
% Fiber Vol.	63.1	63.1	62.5	63.2	63.2
% Resin	31.8	31.8	32.6	31.9	31.8
Specific Gravity	1.47	1.48	1.48	1.44	1.49

Table 3-25. Postcure Study of Press Cured HT-S/710 Graphite/Polyimide. Press Cure Cycle No. 1

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1284	(185)	1050	(152)	848	(123)	883	(128)
297°K (75°F)	1336	(194)	841	(122)	1210	(176)	1061	(154)
589°K (600°F)	174	(25)	547	(79)	703	(102)	727	(105)
90° Flexure 297°K (75°F)	23	(3.3)	5	(0.7)		-	25	(3.6)
Short Beam Shear								
77°K (-320°F)	38	(5.5)	37	(5.3)	47	(6.8)	45	(6.5)
297°K (75°F)	44	(6.4)	47	(6.8)	46	(6.7)	46	(6.6)
589°K (600°F)	26	(3.8)	28	(4.1)	30	(4.3)	29	(4.2)
% Fiber Vol.		51.3		58.0		56.9		64.2
% Resin		43.4		37.0		38.0		31.8
Specific Gravity		1.53		1.54		1.50		1.47

Table 3-26. Postcure Study of Press Cured HT-S/710 Graphite/Polyimide. Press Cure Cycle No. 2

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1339	(194)	1058	(154)	1092	(158)	821	(119)
297°K (75°F)	1243	(180)	1257	(182)	1154	(168)	1192	(173)
589°K (600°F)	186	(27)	676	(96)	747	(108)	848	(123)
90° Flexure 297°K (75°F)	15	(2.2)	16	(2.3)	23	(3.3)		-
Short Beam Shear								
77°K (-320°F)	29	(4.2)	36	(5.2)	47	(6.8)	49	(7.1)
297°K (75°F)	35	(5.0)	56	(8.1)	55	(8.0)	53	(7.7)
589°K (600°F)	27	(3.9)	34	(4.9)	34	(4.9)	35	(5.0)
% Fiber Vol.		56.2		66.3		57.2		63.3
% Resin		38.7		28.5		37.6		31.8
Specific Gravity		1.48		1.49		1.51		1.48

Table 3-27. Postcure Study of Press Cured HT-S/710 Graphite/Polyimide. Press Cure Cycle No. 3

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1233	(179)	923	(134)	888	(129)	835	(121)
297°K (75°F)	1255	(182)	1076	(156)	1123	(163)	1169	(170)
589°K (600°F)	195	(28)	383	(56)	555	(80)	757	(110)
90° Flexure								
297°K (75°F)	23	(3.4)	24	(3.5)	23	(3.4)	28	(4.1)
Short Beam Shear								
77°K (-320°F)	24	(3.5)	30	(4.4)	37	(5.4)	45	(6.5)
297°K (75°F)	31	(4.5)	49	(7.1)	42	(6.1)	48	(6.9)
589°K (600°F)	25	(3.7)	27	(3.9)	27	(3.9)	35	(5.1)
% Fiber Vol.		58.5		59.8		62.4		62.8
% Resin		36.5		35.3		32.8		32.6
Specific Gravity		1.49		1.46		1.46		1.48

Table 3-28. Postcure Study of Press Cured HT-S/710 Graphite/Polyimide. Press Cure Cycle No. 4

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1049	(152)	980	(142)	741	(107)	656	(95)
297°K (75°F)	984	(143)	1068	(155)	971	(141)	1061	(154)
589°K (600°F)	148	(21)	416	(60)	554	(80)	683	(98)
90° Flexure								
297°K (75°F)	32	(4.6)	19	(2.7)	25	(3.6)	23	(3.3)
Short Beam Shear								
77°K (-320°F)	20	(2.9)	33	(4.8)	30	(4.3)	44	(6.4)
297°K (75°F)	28	(4.1)	44	(6.4)	37	(5.4)	49	(7.1)
589°K (600°F)	26	(3.7)	27	(3.9)	26	(3.8)	35	(5.1)
% Fiber Vol.		57.8		60.8		57.8		63.1
% Resin		37.1		34.1		37.1		31.9
Specific Gravity		1.43		1.48		1.44		1.44

the flexural properties were slightly lower at 77°K (-320°F) compared to properties at 297°K (75°F), it is believed that the HT-S/710 graphite/polyimide system can be used over the entire temperature range.

Based on the studies conducted in this portion of the program, press cure cycle No. 2 with a 644°K (700°F) postcure cycle is recommended for this particular graphite/polyimide composite system. In later sections of this report, the initial cure cycle is changed slightly to more nearly approximate the cycles used in the resin characterization and autoclave cure studies.

3.6.2 VACUUM BAG CURE STUDY. The same procedure used in evaluating press cures and postcures was used in evaluating potential processing techniques for vacuum bag curing of HT-S/710. Two cure cycles and four postcure cycles were evaluated.

The cure cycles used in this evaluation are as follows:

a.	Material Designation:	(hy-E-1312-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5-cm (12- by 45-in.) sheet
	Batch No.:	OC-28
	Resin:	710
	Manufacturer:	Fiberite
b.	Fabrication Procedure:	(Vacuum Bag Cure No. 1)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5-cm (12- by 12-in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	5 plies Mauchburg paper CW-1850; 2 plies on the bottom and 3 plies on the top.
	Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg vacuum pressure was applied at room temperature and maintained throughout the entire cure cycle and cool to below 352° K (175° F).

	Cure Cycle:	Heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold for 1 hour, and cool to below 352° K (175° F) under pressure.
	Postcure:	Various ones.
c.	Material Designation:	(hy-E-1312-B)
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5-cm (12- by 45-in.) sheet
	Batch No.:	OC-28
	Resin:	710
	Manufacturer:	Fiberite
d.	Fabrication Procedure:	(Vacuum Bag Cure No. 2)
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5 by 30.5-cm (12- by 12-in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	5 plies Mauchburg paper CW-1850; 2 plies on the bottom and 3 plies on the top.
	Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
	Cure Pressure:	760 mm (29 in.) Hg vacuum pressure was applied at room temperature and maintained throughout the entire cure cycle and cool down to below 352° K (175° F).
	Cure Cycle:	Heat to 355°K (180°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 408°K (275°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 1 hour and cool at no greater than 1° K/minute (3°F/minute) to 352°K (175°F) under pressure.
	Postcure:	Various ones.

Historically, it has been difficult to fabricate dense, moderately low void laminates from polyimide resins without the use of high-pressure processing techniques. One of the primary objectives of this program was to develop a low-pressure cure cycle so that large parts could be fabricated in an oven with only heat and vacuum bag pressure. The 710 polyimide resin is unique in that the three solvents present form azeotropes with water, and under proper conditions form a ternary azeotrope of their own, which allows removal of the solvent at 352° K (175° F) while only under vacuum bag pressure.

Longitudinal and transverse flexural and short-beam shear tests were conducted at 77°K (-320°F), 297°K (75°F), and 589°K (600°F) to evaluate the various vacuum-bag cure and postcure cycles. The vacuum-bag test results are summarized in Tables 3-29 through 3-32 as a function of cure cycle and in Tables 3-33 and 3-34 as a function of postcure cycle.

In evaluating the test results as a function of cure cycle, the No. 2 cycle was clearly superior to cure cycle No. 1. The primary difference between the two cure cycles was the 30-minute holds at 355°K (180°F) and at 408°K (275°F) for cure cycle No. 2. The holds obviously gave more time for volatile removal before the polymer began to gel.

In evaluating the test results as a function of postcure cycle, cycle 3 was superior to cycles 2 and 4 and they, in turn, were superior to cycle 1. It appears that any type of a high-temperature postcure coupled with a vacuum-bag cure cycle will give acceptable mechanical properties at elevated temperatures.

An interesting point was that the press-cured and vacuum-bag cured laminates both contained 12 plies, and when tested in flexure and shear, both carried the same total load. However, when calculating the stress, the press-cure results were generally higher. The press-cured parts were merely thinner than the vacuum bag parts.

The recommended vacuum-bag cure is the No. 2 cycle, with a high-temperature postcure that reaches at least 589° K (600° F), but preferably 616° K (650° F). This vacuum-bag cure cycle was later slightly modified in that the holds were changed from 355° K (180° F) to 352° K (175° F) and from 408° K (275° F) to 400° K (260° F). These changes were made based on the resin characterization work conducted on the 710 resin in parallel with these initial cure studies.

3.6.3 GRAPHITE FIBER FINISH STUDY. Several graphite fibers, most notable the Thornel fibers, have an epoxy sizing on the fiber to improve the interlaminar shear strength of the composite. In an attempt to increase the shear strength of the graphite/polyimide composite, material was purchased from Fiberite that had been prepared with a P13N polyimide resin sizing on the graphite fiber. After the P13N resin had been applied to the fiber and B-staged, the prepreg was coated with the 710 polyimide resin. Convair Aerospace fabricated test panels using press cure 3 and vacuum-bag cures 1 and 2. Flexural and short-beam shear tests were conducted on each of the

Table 3-29. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 1

Property and Test Temperature	CURE CYCLES OC-28				CURE CYCLES OC-31			
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1051	(152)	1279	(186)	1146	(166)	208	(132)
297°K (75°F)	839	(122)	1151	(167)	1130	(164)	993	(144)
589°K (600°F)	130	(19)	197	(28)	165	(24)	155	(22)
90° Flexure								
297°K (75°F)	17	(2.4)	31	(4.5)	27	(3.9)	32	(4.7)
Short Beam Shear								
77°K (-320°F)	34	(5.0)	37	(5.4)	40	(5.8)	32	(4.6)
297°K (75°F)	41	(6.0)	42	(6.1)	31	(4.5)	44	(6.4)
589°K (600°F)		-		-		-		-
% Fiber Vol.		57.0		56.5		55.0		57.1
% Resin		38.0		38.4		39.8		37.8
Specific Gravity		1.38		1.46		1.48		1.49

Table 3-30. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 2

Property and Test Temperature	CURE CYCLES OC-28			
	1		2	
	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure				
77°K (-320°F)	1054	(153)	1059	(154)
297°K (75°F)	1147	(166)	1104	(160)
589°K (600°F)	294	(43)	578	(84)
90° Flexure				
297°K (75°F)	26	(3.8)	38	(5.5)
Short Beam Shear				
77°K (-320°F)	43	(6.2)	37	(5.3)
297°K (75°F)	61	(8.9)	43	(6.2)
589°K (600°F)	32	(4.6)	32	(4.6)
% Fiber Vol.		59.6		59.5
% Resin		35.3		35.5
Specific Gravity		1.41		1.47

Table 3-31. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 3

Property and Test Temperature	CURE CYCLES OC-28			
	1		2	
	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure				
77°K (-320°F)	930	(135)	1095	(159)
297°K (75°F)	1198	(174)	1218	(177)
589°K (600°F)	619	(90)	680	(99)
90° Flexure				
297°K (75°F)	34	(5.0)	33	(4.8)
Short Beam Shear				
77°K (-320°F)				
297°K (75°F)	48	(6.9)	52	(7.5)
589°K (600°F)				
% Fiber Vol.		59.9		59.0
% Resin		35.1		36.0
Specific Gravity		1.34		1.46

Table 3-32. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/Polyimide. Postcure Cycle No. 4

Property and Test Temperature	CURE CYCLES OC-28				CURE CYCLES OC-31			
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	726	(105)	978	(142)	1001	(145)	849	(123)
297°K (75°F)	1056	(153)	1040	(151)	1141	(165)	1004	(146)
589°K (600°F)	630	(91)	685	(99)	560	(81)	686	(99)
90° Flexure								
297°K (75°F)	36	(5.2)	32	(4.6)		-		-
Short Beam Shear								
77°K (-320°F)	41	(6.0)	49	(7.1)	34	(4.9)	37	(5.3)
297°K (75°F)	43	(6.2)	57	(8.3)	47	(6.8)	45	(6.5)
589°K (600°F)	26	(3.7)	34	(4.9)	31	(4.5)	31	(4.5)
% Fiber Vol.		64.0		59.2		56.2		60.6
% Resin		31.0		35.7		38.7		34.3
Specific Gravity		1.48		1.47		1.47		1.50

Table 3-33. Vacuum Bag Cure Cycle Study of HT-S/170 Graphite/Polyimide. Cure Cycle No. 1

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1051	(152)	1054	(153)	930	(135)	1726	(105)
297°K (75°F)	839	(122)	1147	(166)	1198	(174)	1056	(153)
589°K (600°F)	130	(19)	983	(43)	618	(90)	630	(91)
90° Flexure								
297°K (75°F)	17	(2.4)	26	(3.8)	34	(5.0)	36	(5.2)
Short Beam Shear								
77°K (-320°F)	34	(5.0)	43	(6.2)	50	(7.2)	41	(6.0)
297°K (75°F)	41	(6.0)	61	(8.9)	48	(6.9)	43	(6.2)
589°K (600°F)	-	-	32	(4.6)	30	(4.3)	26	(3.7)
% Fiber Vol.		57.0		59.6		59.9		64.0
% Resin		38.0		35.3		35.1		31.0
Specific Gravity		1.38		1.41		1.34		1.48

Table 3-34. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/Polyimide. Cure Cycle No. 2

Property and Test Temperature	POSTCURE CYCLES OC-28							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	1279	(186)	1059	(154)	1095	(159)	978	(142)
297°K (75°F)	1151	(167)	1104	(160)	1218	(177)	1040	(151)
589°K (600°F)	197	(28)	578	(84)	680	(99)	685	(99)
90° Flexure								
297°K (75°F)	31	(4.5)	38	(5.5)	33	(4.8)	32	(4.6)
Short Beam Shear								
77°K (-320°F)	37	(5.4)	37	(5.3)	52	(7.5)	49	(7.1)
297°K (75°F)	42	(6.1)	43	(6.2)	52	(7.5)	57	(8.3)
589°K (600°F)					31	(4.5)	34	(4.9)
% Fiber Vol.		56.5		59.5		59.0		59.2
% Resin		38.4		35.5		36.0		35.7
Specific Gravity		1.47		1.47		1.46		1.47

panels at the three test temperatures. The test results obtained are summarized in Tables 3-35 through 3-37 and are shown for comparison with data in Tables 3-29 through 3-34. The material lot with the P13N finish is identified as lot OC-31 and the material without the finish as OC-28.

It was believed that the low molecular weight, low volatile P13N system would increase the wetting and bonding to the graphite fibers. This would then increase both the shear strength and the transverse flexure strength. The test results obtained indicate that the addition of the P13N polyimide resin as a finish to the HT-S/710 composite system lowered rather than increased the mechanical properties. It is believed that either the high-temperature postcure caused the P13N resin to oxidize, or that the P13N resin was never fully cured until the postcure cycle. If the latter occurred, the small amount of volatiles given off by the P13N system could not have escaped from the composite because the 710 resin was cured at this point. Escaping volatiles would have caused micro cracks in the resin-fiber interface. No further effort was directed toward finding a finish that would improve the strength characteristics of the graphite/polyimide composite; instead work was directed at further optimizing the 710 polyimide resin cure cycle.

3.6.4 MATERIAL SUPPLIER EVALUATION. One of the objectives of the program was to develop a graphite/polyimide prepreg system that would be available from at least two material suppliers. After completing the initial processing studies

Table 3-35. Press Cure Study of HT-S/710 Graphite/ Polyimide With a P13N Finish, Cure Cycle No. 3

Property and Test Temperature	POSTCURE CYCLES OC-31							
	1		2		3		4	
	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure								
77°K (-320°F)	955	(139)	849	(123)	825	(120)	831	(120)
297°K (75°F)	1184	(172)	750	(109)	950	(138)	946	(138)
589°K (600°F)	181	(26)	381	(55)	613	(89)	582	(84)
90° Flexure								
297°K (75°F)	25	(3.6)	23	(3.3)	17	(2.4)		-
Short Beam Shear								
77°K (-320°F)	30	(4.4)	20	(2.9)	32	(4.6)	28	(4.1)
297°K (75°F)	40	(5.8)	30	(4.4)	39	(5.7)	36	(5.2)
589°K (600°F)	22	(3.2)	21	(3.0)	30	(4.3)	28	(4.1)
% Fiber Vol.		59.0		64.9		61.9		63.2
% Resin		35.9		30.1		33.1		31.8
Specific Gravity		1.48		1.50		1.44		1.49

Table 3-36. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide With a P13N Finish, Cure Cycle No. 1

Property and Test Temperature	POSTCURE CYCLES OC-31			
	1		2	
	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure				
77°K (-320°F)	1146	(166)	1001	(145)
297°K (75°F)	1130	(164)	1141	(165)
589°K (600°F)	165	(24)	560	(81)
90° Flexure				
297°K (75°F)	30	(4.4)		-
Short Beam Shear				
77°K (-320°F)	40	(5.8)	34	(4.9)
297°K (75°F)	31	(4.5)	47	(6.8)
589°K (600°F)	37	(5.4)	31	(4.5)
% Fiber Vol.		55.0		56.2
% Resin		39.8		38.7
Specific Gravity		1.49		1.47

Table 3-37. Vacuum Bag Cure Cycle Study of HT-S/710 Graphite/ Polyimide With a P13N Finish, Cure Cycle No. 2

Property and Test Temperature	POSTCURE CYCLES OC-31			
	1		2	
	MN/m^2	(ksi)	MN/m^2	(ksi)
0° Flexure				
77°K (-320°F)	908	(132)	849	(123)
297°K (75°F)	993	(144)	1004	(146)
589°K (600°F)	155	(22)	686	(99)
90° Flexure				
297°K (75°F)	27	(3.9)	36	(4.7)
Short Beam Shear				
77°K (-320°F)	32	(4.6)	37	(5.3)
297°K (75°F)	44	(6.4)	45	(6.5)
589°K (600°F)		-	31	(4.5)
% Fiber Vol.		57.1		60.6
% Resin		37.8		34.3
Specific Gravity		1.49		1.50

and resin characterization work, a preliminary materials specification was prepared and material was purchased from three different suppliers (Fiberite, Ferro, and Whittaker R&D). Two lots of material were purchased, four weeks apart, from each supplier. Extensive evaluation of the prepreg, including tack, drape, and physical properties was made before the initial laminates were fabricated.

Comments concerning the appearance, handling characteristics, and quality of the material received is reported in the following paragraphs.

Fiberite Corporation

1. Material was improperly packaged (no aluminum liner).

2. Prepreg material had very slight tack initially and upon opening the material a second time, it had no tack at all. (Material would not stick to itself.)

3. Resin was spread over any one sheet of material uniformly and had very few gaps. However, upon handling, the prepreg material split apart longitudinally in various spots.

Whittaker R&D

1. Prepreg material was properly packaged, sealed in aluminum foil with aluminized tape, and then sealed in polyethylene.

2. Prepreg material had very slight tack initially. It became extremely dry after one opening. The material fell apart, had no tack, and resin dust flew around as the prepreg was cut.

3. All average prepreg properties were within the requested range.

4. Prepreg material was resin rich on one side and there were minor gaps in the prepreg.

Ferro Corporation

1. Prepreg material was packaged in an excellent manner using MIL-SPEC materials.

2. Prepreg material had excellent tack initially and retained this tack throughout the four periods during which the material was used.

3. All prepreg properties were within requested range.

4. Resin appeared to be spread evenly over the entire sheet; the material had good handling qualitites but had some minor fiber misalignment.

Table 3-38 lists the prepreg properties determined by Convair Aerospace on five random sheets from each of the prepreg lots. Considering that at this time the material was prepregged by hand and that the 710 resin system is not a hot melt coated resin system, the variation in physical properties is not excessive.

3.6.4.1 Laminate Fabrication Techniques. Laminates were cured using vacuum bag, press, and autoclave processing techniques. All laminates were 30.5- by 30.5-cm (12- by 12-in.) 12-ply laminates. For both the vacuum bag and autoclave cures, the laminates made from each material supplier were cured at the same time. Laminates made in the press were cured individually because of platen size. One cure cycle and three postcure cycles were used in evaluating the laminates made from material purchased from the three material suppliers. Flexure and short-beam shear tests were conducted whenever possible at all three test temperatures. The cure and postcure cycles used in evaluating this material were as follows:

a.	Material Designation:	hy-E-1312-B, F-5036, WRD-710
	Manufacturer:	Fiberite, Ferro, Whittaker R&D
	Fiber Type:	HT-S (staple)
	Material Form:	30.5 by 114.5-cm (12 by 45-in.) sheet
	Resin:	710
b.	Fabrication Procedure:	Vacuum Bag, Press, or Autoclave
	Mold Release:	Teflon Film
	Layup:	12 plies unidirectional, 30.5- by 30.5-cm (12- by 12-in.)
	Separator Film:	Teflon-coated glass cloth, FGO-3
	Bleeder:	5 plies Mauchburg paper CW-1850
	Special Instructions:	A Corprene 0.95-cm (3/8-in.) dam was used around the periphery of layup, separated from prepreg by no more than 1.5 mm (0.06 in.). One-ply Teflon film (nonperforated) was used over the bleeder. A 30.5- by 30.5- by 0.64-cm (12 by 12 by 1/4-in.) aluminum plate was used as a pressure plate over the Teflon film, was covered in turn by 3 plies of 181 style glass cloth (for venting), and enclosed by a vacuum bag.
	Cure Pressure:	760 mm. (29 in.) Hg. vacuum pressure was applied at room temperature and maintained throughout the entire cure cycle and cool down to below 352° K (175° F). For the press or autoclave cures 689 kN/m^2 (100 psi) was applied after the 30 minute hold at 400° K (260° F).
	Cure Cycle:	Heat to 352°K (175°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 400°K (260°F) at 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 450°K (350°F) at 1 to 3°K/minute (3 to 5°F/minute),

Table 3-38. Prepreg Properties, HT-S/710

Vendor	Sheet No.	Volatiles* (%)	Flow* (%)	Fiber Content (%)	Resin* Content (%)
Fiberite	1B	22.6	33.7	25.7	51.7
	5B	19.3	23.6	39.9	40.8
	7A	19.0	23.4	34.3	46.7
	16A	22.7	35.0	32.1	45.2
	18A	20.8	29.5	31.9	47.3
	Ave.	20.9	29.0	32.8	46.3
Whittaker	1	16.0	24.3	37.9	44.6
	5	16.0	27.8	38.0	44.5
	10	16.2	26.0	39.2	43.1
	15	17.3	25.5	37.2	44.0
	19	11.1	19.6	43.3	44.1
	Ave.	15.3	24.6	39.1	44.1
Ferro	1	13.4	12.6	48.4	38.2
	3	15.8	15.1	46.5	37.7
	4	15.8	23.6	38.1	46.1
	6	15.6	19.7	38.8	45.6
	8	19.5	25.7	41.0	39.5
	Ave.	16.2	19.3	42.6	41.4

*Requested Range

1. Volatiles 10 to 20%
2. Flow 15 to 25%
3. Resin 40 to 50% except Fiberite - 43 to 52%

hold 1 hour and cool at no greater than 1° K/minute (3° F/minute) to 352° K (175° F) under pressure.

Postcure: No postcure
8 hours at 644°K (700°F) in air
8 hours at 644°K (700°F) in N_2

3.6.4.2 Laminate Test Results. An interesting process investigated with the laminates fabricated in this portion of the program was to postcure part of each of the laminates in N_2 gas up to 644°K (700°F). This postcure cycle was investigated to improve initial properties at 77°K (-320°F) and 297°K (75°F). The test results obtained are presented in Tables 3-39 through 3-44. In all cases the highest results at 77°K (-320°F) and 297°K (75°F) were obtained on panels postcured in N_2 to 644°K (700°F). When tested at 589°K (600°F) the test results for the N_2 postcured panels showed approximately a 10 percent decrease in strength compared to the air postcured panels. This difference in properties is believed to be caused by not allowing the oxidative crosslinking reaction to occur in the N_2 postcure. The crosslinking of polyimide resins in general causes the resin to become more brittle, hence the lower properties at room and cryogenic temperatures. On the other hand, the crosslinking improves thermal stability and increases elevated temperature properties.

Table 3-39. Vacuum-Bag Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	(°F)	Postcure Cycles: No Postcure		644°K (700°F) in Air		644°K (700°F) in N_2	
Ferro Corporation	Flex. Strength MN/m^2 (ksi)	77	(-320)	-	-	1062	(154)	1179	(171)
		297	(75)	1179	(171)	1083	(157)	1165	(169)
		589	(600)	-	-	647	(93)	556	(80)
	Specific Gravity			1.42		1.41		1.40	
	Resin Cont. %			38.2		34.3		33.5	
	Fiber Vol. %			57.8		60.7		61.4	
Whittaker R&D	Flex. Strength MN/m^2 (ksi)	77	(-320)	-	-	-	-	-	-
		297	(75)	1062	(154)	1138	(165)	1117	(162)
		589	(600)	-	-	682	(98)	585	(84)
	Specific Gravity			1.41		1.41		1.40	
	Resin Cont. %			48.4		39.9		31.9	
	Fiber Vol. %			45.9		55.0		63.2	
Fiberite Corporation	Flex. Strength MN/m^2 (ksi)	77	(-320)	-	-	758	(110)	917	(133)
		297	(75)	834	(121)	627	(90)	862	(125)
		589	(600)	-	-	542	(78)	437	(63)
	Specific Gravity			1.41		1.38		1.38	
	Resin Cont. %			40.2		37.7		38.9	
	Fiber Vol. %			54.5		57.2		56.1	

Table 3-40. Press-Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	(°F)	Postcure Cycles: No Postcure		644°K (700°F) in Air		644°K (700°F) in N_2	
Ferro Lam. No. 1	Flex. Strength MN/m^2 (ksi)	77	(-320)	1200	(174)	958	(139)	1365	(198)
		297	(75)	1331	(193)	1186	(172)	1379	(200)
		589	(600)	-	-	869	(126)	793	(115)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	-	-	-	-	70	(10.1)
		297	(75)	-	-	-	-	75	(10.9)
		589	(600)	-	-	-	-	39	(5.6)
	Specific Gravity			1.44		1.46		1.46	
	Resin Cont. %			42.5		36.3		35.3	
	Fiber Vol. %			52.2		58.7		59.8	
Ferro Lam. No. 2	Flex. Strength MN/m^2 (ksi)	77	(-320)	1220	(177)	1048	(152)	1358	(197)
		297	(75)	1276	(185)	1269	(184)	1317	(191)
		589	(600)	-	-	931	(135)	945	(137)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	-	-	77	(11.2)	84	(12.2)
		297	(75)	-	-	83	(12.0)	88	(12.8)
		589	(600)			47	(6.8)	50	(7.2)
	Specific Gravity			1.51		1.48		1.52	
	Resin Cont. %			35.4		31.2		33.3	
	Fiber Vol. %			59.5		63.9		61.8	

Table 3-41. Press-Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	(°F)	Postcure Cycles: No Postcure		644°K (700°F) in Air		644°K (700°F) in N_2	
Fiberite P-1	Flex. Strength MN/m^2 (ksi)	77	(-320)	1269	(184)	-	-	-	-
		297	(75)	1200	(174)	1324	(192)	1083	(157)
		589	(600)	-	-	869	(126)	814	(118)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	-	-	-	-	-	-
		297	(75)	-	-	-	-	-	-
		589	(600)	-	-	-	-	-	-
	Specific Gravity			1.52		-		-	
	Resin Cont. %			48.4		-		-	
	Fiber Vol. %			45.6		-		-	
Fiberite P-2	Flex. Strength MN/m^2 (ksi)	77	(-320)	910	(132)	1579	(229)	1462	(212)
		297	(75)	1096	(159)	1317	(191)	1227	(178)
		589	(600)	-	-	793	(115)	807	(117)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	-	-	71	(10.3)	42	(6.1)*
		297	(75)	-	-	81	(11.7)	44	(6.3)*
		589	(600)	-	-	48	(6.9)	30	(4.4)
	Specific Gravity			1.49		1.51		1.48	
	Resin Cont. %			38.4		35.9		43.3	
	Fiber Vol. %			56.4		59.0		51.6	

*Specimens bottomed out in bending - no shear failure.

Table 3-42. Press-Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	(°F)	Postcure Cycles: No Postcure		644°K (700°F) in Air		644°K (700°F) in N_2	
Whittaker Lam. P-1	Flex. Strength MN/m^2 (ksi)	77	(-320)	1186	(172)	952	(138)	1172	(170)
		297	(75)	1255	(182)	1000	(145)	834	(121)
		589	(600)	-	-	779	(113)	889	(129)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	-	-	-	-	-	-
		297	(75)	-	-	-	-	-	-
		589	(600)	-	-	-	-	-	-
	Specific Gravity			1.46		1.50		1.50	
	Resin Cont. %			45.7		40.1		42.4	
	Fiber Vol. %			49.3		54.8		53.3	

Table 3-43. Autoclave-Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	Temperature (°F)	Postcure Cycles: No Postcure	Postcure Cycles: 644°K (700°F) in Air	Postcure Cycles: 644°K (700°F) in N_2
Ferro Lam A-6	Flex. Strength MN/m^2 (ksi)	77	(-320)	1269 (184)	986 (143)	1193 (173)
		297	(75)	1407 (204)	1234 (179)	1351 (196)
		589	(600)	- -	1007 (146)	903 (131)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	- -	65 (9.4)	79 (11.5)
		297	(75)	- -	79 (11.4)	77 (11.2)
		589	(600)	- -	55 (7.9)	41 (5.9)
	Specific Gravity			1.49	1.50	1.49
	Resin Cont. %			37.5	33.4	34.8
	Fiber Vol. %			57.3	61.7	60.2
Ferro Lam A-7	Flex. Strength MN/m^2 (ksi)	77	(-320)	1303 (189)	883 (128)	1103 (160)
		297	(75)	1317 (191)	993 (144)	1331 (193)
		589	(600)	-	1096 (159)	910 (132)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	- -	54 (7.8)	77 (11.2)
		297	(75)	- -	47 (6.8)	85 (12.3)
		589	(600)	- -	35 (5.1)	50 (7.2)
	Specific Gravity			1.49	1.50	1.48
	Resin Cont. %			40.0	34.0	37.5
	Fiber Vol. %			54.9	61.0	57.2

Table 3-44. Autoclave-Cure Study of HT-S/710 Composites

Material Supplier	Type of Test	Temperature °K	Temperature (°F)	Postcure Cycles: No Postcure	Postcure Cycles: 644°K (700°F) in Air	Postcure Cycles: 644°K (700°F) in N_2
Fiberite Lam. A-3	Flex. Strength MN/m^2 (ksi)	77	(-320)	1158 (168)	- -	- -
		297	(75)	1200 (174)	1062 (154)	1276 (185)
		589	(600)	- -	917 (133)	841 (122)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	- -	- -	- -
		297	(75)	- -	- -	- -
		589	(600)	- -	- -	- -
	Specific Gravity			1.45	-	-
	Resin Cont. %			37.9	-	-
	Fiber Vol. %			56.8	-	-
Fiberite Lam. A-4	Flex. Strength MN/m^2 (ksi)	77	(-320)	1282 (186)	1014 (147)	1386 (201)
		297	(75)	1200 (174)	1048 (152)	1248 (181)
		589	(600)	- -	848 (123)	869 (126)
	Short Beam Shear MN/m^2 (ksi)	77	(-320)	- -	64 (9.2)	62 (8.9)
		297	(75)	- -	82 (11.8)	61 (8.7)
		589	(600)	- -	48 (6.9)	48 (6.9)
	Specific Gravity			1.50	1.49	1.51
	Resin Cont. %			37.8	43.1	46.8
	Fiber Vol. %			56.9	52.6	48.2

All panels made from material supplied by Ferro showed no evidence of precipitation while 10 percent of the Fiberite panels showed some precipitation, and 80 percent of the Whittaker panels showed heavy precipitation. The precipitation that occurs in polyimide composites is caused by the entrapment of water or other volatiles at the time the polymer begins to gel. This is originally caused in the 710 polyimide resin by using it with an acid equivalent outside the recommended range of 520 to 540.

In fabricating graphite/polyimide laminates, the resin uniformity throughout the prepreg is extremely important, because lack of resin or resin-rich portions in a laminate will cause the panel to warp. This is a much more serious problem than with glass/polyimide laminates because of the negative expansion characteristics of the graphite fiber.

Based on mechanical property data, prepreg quality, prepreg reproducibility, and handling ease, Fiberite and Ferro were selected as approved material suppliers. The material used throughout the remainder of the program was purchased on a competitive basis from these two firms.

3.6.5 BLEEDER AND VACUUM PROCESS STUDIES. Studies were made to evaluate placement of bleeder materials and methods of applying a vacuum to the part to be cured. All previous laminates had been made with top and bottom caul plates and with the bleeder materials on both sides of the prepreg layup. Three different lots of material were used in this evaluation, and the cure cycle for all panels is the autoclave cycle reported in Section 3.6.4. All laminates fabricated for this particular study were 15.2- by 15.2-cm (6- by 6-in.) by 12-ply laminates.

The purpose of the bleeder study was to determine the effect of bleeder placement on the fabrication of large complex parts. It was found that the difficulty in making such parts can be reduced if all the bleeder is placed on one rather than both sides of the layup. The data in Table 3-45 shows clearly that higher mechanical properties can be attained by placing the bleeder on one side only; in this case, the bleeder was always placed on the top of the layup. Placing the bleeder in this manner tends to give directionality to volatile release and causes the panel to cure from the bottom up.

From this study, Convair Aerospace believes that graphite/polyimide HT-S/710 can be successfully cured with the bleeder on one side only. One disadvantage to this method is that flat panels tend to have a slight warp caused by the resin-rich surface on one side of the panel. The panels are flat when removed from the initial cure, but during the postcure cycle they warp because of the large thermal expansion differential between resin and graphite fiber. For flat panels is is therefore recommended that the bleeder be placed on both sides.

It was also found during this study that either (a) the volatiles released during the cure, or (b) the 710 resin itself reacted with the CW-1850 Mauchberg paper sealing the

Table 3-45. Bleeder Study of HT-S/710 Graphite/Polyimide

Laminate No.	Bleeder* Placement	Test Temperature °K	Test Temperature (°F)	Flexural Strength MN/m^2 ksi — Postcure Cycles: NPC		644°K (700°F) In Air		644°K (700°F) In N_2		Laminate Physical Properties	
Oc-28-1	Bleeder Both Sides - 2 Plies Top, 3 Plies Bottom	77	(-320)	-	-	1103	(160)	-	-	S.G.	1.49
		297	(75)	1407	(204)	1269	(184)	1345	(195)	R.C.	33.0%
		589	(600)	-	-	965	(140)	731	(106)	F.V.	62.0%
OC-28-2	Bleeder One Side Only, 5 Plies	77	(-320)	-	-	-	-	-	-	S.G.	1.50
		297	(75)	1510	(219)	1476	(214)	1517	(220)	R.C.	38.1%
		589	(600)	-	-	986	(143)	703	(102)	F.V.	56.8%
OC-31-1	Bleeder Both Sides - 2 Plies Top, 3 Plies Bottom	77	(-320)	-	-	910	(132)	-	-	S.G.	1.48
		297	(75)	1131	(164)	1145	(166)	1200	(174)	R.C.	39.4%
		589	(600)	-	-	827	(120)	724	(105)	F.V.	55.4%
Oc-31-2	Bleeder One Side Only, 5 Plies	77	(-320)	-	-	1248	(181)	-	-	S.G.	1.53
		297	(75)	1510	(219)	1289	(187)	1510	(219)	R.C.	36.5%
		589	(600)	-	-	841	(122)	765	(111)	F.V.	58.3%
A-3	Bleeder Both Sides - 2 Plies Top, 3 Plies Bottom	297	(75)	1317	(191)	-	-	-	-	S.G.	1.55
										R.C.	35.9%
										F.V.	59.0%
A-9	Bleeder Both Sides - 2 Plies Top, 3 Plies Bottom	297	(75)	1393	(202)	-	-	-	-	S.G.	1.52
										R.C.	39.6%
										F.V.	55.2%
A-10	Bleeder One Side Only, 5 Plies	297	(75)	1379	(200)	-	-	-	-	S.G.	1.53
										R.C.	37.0%
										F.V.	57.9%
A-11	Bleeder One Side Only, 5 Plies	297	(75)	1524	(221)	-	-	-	-	S.G.	1.55
										R.C.	31.0%
										F.V.	64.1%
A-14	Bleeder Both Sides - 2 Plies Top, 3 Plies Bottom	297	(75)	1427	(207)	-	-	-	-	S.G.	1.54
										R.C.	58.9%
										F.V.	64.0%
A-15	Bleeder One Side Only, 5 Plies	297	(75)	1620	(235)	-	-	-	-	S.G.	1.52
										R.C.	34.8%
										F.V.	60.1%

laminate off from the vacuum. No studies were made to investigate this problem; instead the Mauchberg paper was replaced with 104 glass cloth as the bleeder material.

The vacuum study was conducted because the reliability of producing good autoclave cured graphite/polyimide parts was not as high as it was for producing vacuum bag or press cured laminates. A small study was made to determine the effect of varying the method by which the vacuum reached the part. Test results are presented in Table 3-46. Essentially no difference occurred between the properties reported for each of the four methods. At first the results were difficult to understand, but after reviewing each layup the problem was resolved as being caused by the venting material rather than bleeder arrangement and vacuum placement.

In previous autoclave cycles, the venting material consisted of three plies of style 181 glass; in this study the venting material was a combination of glass cloth and glass mat, and when pressure was applied the 181 glass nested. Because of the large quantity of volatiles and resin given off during the cure, the nested glass sealed the vacuum so that it could not reach the part. In press cures, the pressure is normal to the part, whereas in an autoclave the part is surrounded on all sides by pressure; thus, for graphite/polyimides, the same venting arrangement will not work for both curing techniques. (Such is not the case for epoxy parts because of the lower volatiles in the prepreg.)

Table 3-46. Vacuum Study of HT-S/710 Graphite/Polyimide

Laminate No.	Vacuum Arrangement	Test Temperature °K (°F)	Flexural Strength MN/m^2 ksi — Postcure Cycles: NPC	644°K (700°F) In Air	644°K (700°F) In N_2	Laminate Physical Properties
A-2	Bleeder Top and Bottom - Dams on Fiber Length Only	297 (75)	1331 (193)	1379 (200)	1482 (215)	S.G. 1.49 R.C. 38.4% F.V. 56.2 %
A-3	Bleeder Top and Bottom - Picture Frame Dam	297 (75)	1324 (192)	1248 (181)	1324 (192)	S.G. 1.51 R.C. 32.6% F.V. 62.6%
A-4	Bleeder Top and Bottom - No Dams	297 (75)	1434 (208)	1289 (187)	1310 (190)	S.G. 1.50 R.C. 37.2% F.V. 57.6%
A-5	Bleeder Top and Bottom - Vacuum Line to Top Caul Plate	297 (75)	1317 (191)	- -	1365 (198)	S.G. 1.51% R.C. 36.4% F.V. 58.5%

Based on this study Convair Aerospace has standarized on the following venting material arrangement for autoclave-cured HT-S/710 laminates: 1 ply 181 glass cloth, 1 layer glass mat, 1 ply 1534 glass cloth, 1 layer glass mat, and 1 ply 181 glass cloth.

3.6.6 GRAPHITE/POLYIMIDE SOLVENT STUDY. One of the problems associated with curing polyimide composites is that if the volatiles, either from the initial solvent or as a by-product of the curing reaction, are present when the polymer begins to gel, the resin will precipitate out of the composite. Based on the resin characterization work, it was determined that the solvent system in the 710 resin system is a mixture of ethanol, NMP, and Xylene. In an effort to reduce the possibility of precipitation, four lots of graphite/polyimide prepreg were purchased in which the solvent system was modified by changing the concentrations of the various components.

The four lots of prepreg were made from the same resin batch, but were diluted with different prepregging solvents. The solvents were:

a. 5% additional ethanol.

b. 5% additional NMP.

c. 5% solution of NMP – Xylene (2 to 1).

d. 5% solution of NMP – Xylene (1 to 1)

A significant problem encountered in prepregging the graphite fiber was difficulty in retaining the resin. Therefore, several prepreg lots had low resin content. The prepreg properties that were determined are presented in Table 3-47. Resin contents are on the low side of the requested range, and the percent flow in two cases was below the target values.

Probably the most important difference in these four lots of prepreg is that the gel time at 400°K (260°F) is considerably longer than that for the normal graphite/polyimide prepreg lots. The extension in gel time is probably caused by upsetting the solvent concentrations, so that the ternary aezotrope that normally forms and releases all of the solvent volatiles did not form or react to completion. As a result some of the solvent volatiles were still present at 400°K (260°F), which impeded polymer gelation.

Vacuum bag and autoclave laminates were fabricated using the cure cycle described in Section 3.6.4. Longitudinal flexure and short-beam shear tests were conducted at 77, 297, and 589°K (-320, 75, and 600°F). Specific gravity, resin contents, and fiber volumes were determined for each laminate. As expected, the fiber volumes were higher than normal because of low resin content in the prepreg; thus short-beam shear strengths were low. The data is presented in Tables 3-48 through 3-52. In some cases where material was available, several laminates were made by the same processing technique for the same lot of material. No significant increases in strength at either 297°K (75°F) or 589°K (600°F) were obtained for the modified systems as

Table 3-47. Graphite/Polyimide HT-S/710 Prepreg Properties (Solvent Study)

Material Lot No.	Volatile* Content (%)	Resin* Content (%)	% Flow*	Fiber Content (%)	Process* Gel at 400°K (260°F)(Min)
UNIT I (Ethanol)	12.0	35.0	8.1	53.0	35
UNIT II (NMP)	20.5	36.3	19.0	43.2	45
UNIT III (NMP/Zylene 2 to 1)	17.3	36.3	15.6	46.4	55
UNIT IV (NMP/Zylene 1 to 1)	16.0	29.7	7.7	54.3	55

*Target Values

Volatiles	10 to 20%
Resin Content	35 to 45%
% Flow	15 to 25%
Gel	15 to 25 minutes

Table 3-48. Mechanical Properties of HT-S/710 Graphite Composites. Unit I — Solvent Study (Ethanol); Autoclave Cure

Type of Test	Test Temperature °K	(°F)	Laminate No. 129 MN/m^2	(ksi)	Laminate No. 139 MN/m^2	(ksi)	Laminate No. 153 MN/m^2	(ksi)	Laminate No. 154 MN/m^2	(ksi)
Flexure	77	(-320)	1269	(183.9)	1214	(175.8)	1062	(153.7)	1338	(194.1)
	297	(75)	1482	(215.1)	1324	(191.5)	1303	(188.8)	1489	(215.9)
	589	(600)	834	(121.0)	675	(97.9)	889	(129.4)	507	(72.8)
Short Beam Shear	77	(-320)		-	99	(14.3)	83	(12.0)	64	(9.2)
	297	(75)		-	84	(12.2)	40	(5.8)	59	(8.6)
	589	(600)		-	40	(5.8)	39	(5.7)	40	(5.8)
Fiber Volume %			64.8		66.6		65.4		72.9	
Resin Content %			30.7		28.5		29.7		23.7	
Specific Gravity			1.52		1.52		1.52		1.49	

Table 3-49. Mechanical Properties of HT-S/710 Graphite Composites. Unit II — Solvent Study (NMP); Vacuum Bag Cure

Type of Test	Test Temperature °K	(°F)	Laminate No. 105 MN/m^2	(ksi)	Laminate No. 137 MN/m^2	(ksi)
Flexure	77	(-320)	1207	(175.1)	938	(135.7)
	297	(75)	1179	(171.0)	952	(137.9)
	589	(600)	575	(83.5)	575	(83.4)
Short Beam Shear	77	(-320)	52	(7.5)	36	(5.2)
	297	(75)	46	(6.7)	37	(5.3)
	589	(600)	-	-	20	(2.9)
Fiber Volume %			69.8		70.3	
Resin Content %			26.6		25.1	
Specific Gravity			1.47		1.48	

Table 3-50. Mechanical Properties of HT-S/710 Graphite Composites. Unit II — Solvent Study (NMP); Autoclave Cure

Type of Test	Test Temperature °K	(°F)	Laminate No. 103 MN/m^2	(ksi)	Laminate No. 132 MN/m^2	(ksi)
Flexure	77	(-320)	1386	(200.8)	1200	(173.8)
	297	(75)	1193	(172.7)	1165	(168.7)
	589	(600)	696	(100.6)	752	(109.2)
Short Beam Shear	77	(-320)	49	(7.2)	43	(6.2)
	297	(75)	49	(7.2)	43	(6.2)
	589	(600)	33	(4.8)	29	(4.2)
Fiber Volume %			68.7		70.8	
Resin Content %			26.6		24.2	
Specific Gravity			1.47		1.56	

Table 3-51. Mechanical Properties of HT-S/710 Graphite Composites. Unit III — Solvent Study (NMP - Xylene 2 to 1)

Type of Test	Test Temperature °K	(°F)	Laminate No. 106[1] MN/m²	(ksi)	Laminate No. 110[3] MN/m²	(ksi)
Flexure	77	(-320)	1448	(210.4)	896	(129.8)
	297	(75)	1531	(221.8)	1131	(164.4)
	589	(600)	855	(124.2)	772	(111.8)
Short Beam Shear	77	(-320)	65	(9.5)	46	(6.7)
	297	(75)	68	(9.9)	56	(8.1)
	589	(600)	69	(10.2)	36	(5.2)
Fiber Volume %			65.1		65.1	
Resin Content %			30.0		30.0	
Specific Gravity			1.48		1.46	
1. Autoclave Cure 2. Vacuum Bag						

Table 3-52. Mechanical Properties of HT-S/710 Graphite Composites. Unit IV — Solvent Study (NMP - Xylene 1 to 1)

Type of Test	Test Temperature °K	(°F)	Laminate No. 107[1] MN/m²	(ksi)	Laminate No. 109[2] MN/m²	(ksi)	Laminate No. 111[3] MN/m²	(ksi)
Flexure	77	(-320)	1131	(164.3)	595	(86.4)	635	(92.2)
	297	(75)	1069	(154.8)	800	(115.7)	910	(131.9)
	589	(600)	745	(107.8)	827	(120.3)	696	(100.8)
Short Beam Shear	77	(-320)	55	(7.9)	45	(6.5)	35	(5.1)
	297	(75)	58	(8.4)	52	(7.6)	41	(5.9)
	589	(600)	696	(10.1)	50	(7.3)	37	(4.7)
Fiber Volume %			64.3		64.9		66.9	
Resin Content %			30.8		30.3		28.3	
Specific Gravity			1.49		1.50		1.51	
1. Autoclave Cure 2. Press Cure 3. Vacuum Bag								

compared to the standard system. In fact, in most cases the measured properties were lower than those obtained for the nonmodified graphite/polyimide composite system. Certainly some of the test results, primarily shear strength, were obscured by the unusually high fiber volumes, but the flexural strengths should have been quite high if the solvent modifications improved the composite. Since the test results indicated no improvement, it is recommended that the 710 polyimide resin be used as received, and no modifications should be made to the resin.

3.6.7 GRAPHITE/POLYIMIDE PRECOMPACTION STUDY. In the fabrication of large complex parts or thick composite parts, it is beneficial to debulk plies of material prior to the final cure. A study was made to investigate this debulking or precompaction processing technique for the graphite/polyimide prepreg material.

Based on the 710 polyimide resin characterization studies, a precompaction temperature of 352°K (175°F) was selected, because at this temperature nearly 100 percent of the solvents in the 710 polyimide resin escape. Previous graphite/epoxy studies at Convair Aerospace showed that a 6- to 8-ply modular buildup was preferred for handling and forming the precompacted material. These studies also showed that the precompacted material outer plies had less residual resin than the inner plies. The graphite/polyimide precompaction study was conducted with 6-ply modular layups. Wet or nonprecompacted insert plies were also evaluated in an effort to replace some of the resin that is preferentially drawn from the outer plies. Precompaction pressures included 760 mm (29 in.) Hg-vacuum bag only; 172 kN/m^2 (25 psi) plus vacuum; 345 kN/m^2 (50 psi) plus vacuum; and 521 kN/m^2 (75 psi) plus vacuum. After precompaction all panels were autoclave-cured at the same time per the cure cycle reported in Section 3.6.4.

Flexural and short-beam shear strengths were determined for each of the laminates (Table 3-53). Specific gravity, resin content, and fiber volumes were also determined for each laminate. It is quite apparent upon reviewing both the mechanical and physical properties of the various laminates that the proper precompaction pressure is either vacuum or vacuum plus 172 kN/m^2 (25 psi); also, that a one-ply unprecompacted insert gives more uniform and higher test results than the other methods of fabrication. More important is the fact that the flexural specimens for the panels with the inserts and the lower precompaction pressures failed in tension, whereas all the other specimens failed by delamination. Based on this test data, Convair Aerospace selected a precompaction pressure of vacuum plus 172 kN/m^2 (25 psi).

3.6.8 GRAPHITE/POLIMIDE (HT-S/710) CURE CYCLE. Upon completing all process studies on the graphite/polyimide (HT-S/710) composite system, Convair Aerospace reviewed the data and processing techniques and recommended the following cure cycle, which is completely adaptable to vacuum-bag, press, or autoclave-curing techniques simply by modification of the pressure. It is insensitive to heating rates up to 6° K/minute (10F/minute). This cure cycle was used in making the design-property

Table 3-53. Precompaction Study, HT-S/710

Laminate Number	Flexural Strength 297°K(75°F) MN/m^2(ksi)		Flexural Strength 589°K(600°F) MN/m^2(ksi)		Short Beam Shear 297°K(75°F) MN/m^2(ksi)		Short Beam Shear 589°K(600°F) MN/m^2(ksi)		Specific Gravity	Resin Content (%)	Fiber Volume (%)	Precompaction Pressure	Insert Plies	Thickness per Ply (mils)
1	1282	(185.7)	758	(110.1)	54.6	(7.94)	36.5	(5.29)	1.57	35.5	59.2	0	0	6.7
2	1448	(210.3)	663	(96.1)	53.9	(7.81)	34.8	(5.05)	1.54	28.7	66.3	Vac	0	5.5
3	1420	(206.5)	1000	(144.9)	61.2	(8.88)	42.2	(6.12)	1.53	30.3	64.5	Vac	1	5.5
4	1434	(208.3)	1000	(144.9)	68.0	(9.88)	43.7	(6.33)	1.54	28.7	66.3	Vac	2	5.3
5	1276	(184.8)	793	(114.9)	62.6	(9.08)	41.6	(6.04)	1.48	26.1	68.7	Vac + 25 PSI	0	5.3
6	1517	(220.4)	958	(138.8)	62.2	(9.02)	35.2	(5.10)	1.52	27.5	67.5	Vac + 25 PSI	1	5.0
7	1517	(219.7)	765	(110.5)	62.0	(9.00)	38.2	(5.54)	1.55	29.8	65.2	Vac + 25 PSI	2	5.4
8	1131	(163.5)	848	(123.2)	56.0	(8.11)	35.6	(5.16)	1.48	26.4	68.6	Vac + 50 PSI	0	5.2
9	772	(112.4)	696	(100.6)	55.0	(7.98)	37.7	(5.47)	1.48	24.6	70.3	Vac + 50 PSI	1	5.1
10	807	(117.0)	584	(84.7)	56.3	(8.16)	36.6	(5.30)	1.48	27.6	67.5	Vac + 50 PSI	2	5.1
11	731	(106.3)	525	(76.1)	46.5	(6.74)	32.4	(4.70)	1.48	24.1	70.7	Vac + 75 PSI	0	5.4
12	657	(95.4)	518	(75.2)	48.3	(6.99)	33.1	(4.80)	1.47	25.6	68.6	Vac + 75 PSI	1	5.5
13	1200	(173.8)	731	(105.7)	51.9	(7.53)	41.3	(5.99)	1.48	26.0	68.4	Vac + 75 PSI	2	5.4

laminates, hardware-demonstration items, and the sheet-stringer test components.

The example precompaction and cure cycle is presented for a 12-ply laminate. For laminates having a different number of plies, the bleeder material requires adjustment on the basis of one ply of style 104 glass per ply of graphite/polyimide for precompaction and 1 ply of style 104 glass per 2 plies of graphite/polyimide for curing.

Precompaction Cycle

1. Lay up a separator cloth and 6 plies of 120 glass cloth bleeder on both sides of the graphite/polyimide 12-ply layup.

2. Seal top and bottom with suitable film material such as Mylar or Teflon.

3. Vacuum bag the layup, apply full vacuum 760 mm (29 in.) Hg, and place the part in the autoclave.

4. Heat the part to 352°K (175°F) at a rate of 1 to 3°K/minute (3 to 5°F/minute), apply 172 KN/m^2 (25 psi), hold 15 minutes, and cool to 297°K (75°F). Remove bleeder and initiate the cure cycle layup.

Cure Cycle

1. Place 3 plies of 104 glass cloth on each side of the precompacted layup and complete the bagging and venting material as shown in Figure 3-20.

2. Apply full vacuum 760 mm (29 in.) Hg, heat to 352°K (175°F) at a rate of 1 to 3°K/minute (3 to 5°F/minute), hold 30 minutes, heat to 400°K (260°F) at a rate of 1 to 3°K/minute (3 to 5°F/minute), hold 25 minutes, apply 690 kN/m^2 (100 psi), heat to 450°K (350°F) at a rate of 1 to 3°K/minute (3 to 5°F/minute), hold 2 hours, cool under pressure to 352° K (175° F) or lower at a rate no greater than 1° K/minute (3°F/minute).

Postcure Cycle

1. Place the part, unrestrained, in a room-temperature oven, heat to 450°K (350°F), and initiate the following postcure cycle:

2 hours at 450°K (350°F)	2 hours at 562°K (550°F)
2 hours at 477°K (400°F)	2 hours at 589°K (600°F)
2 hours at 505°K (450°F)	4 hours at 616°K (650°F)
2 hours at 533°K (500°F)	8 hours at 644°K (700°F)

Cool to 352°K (175°F) or lower and remove from the oven.

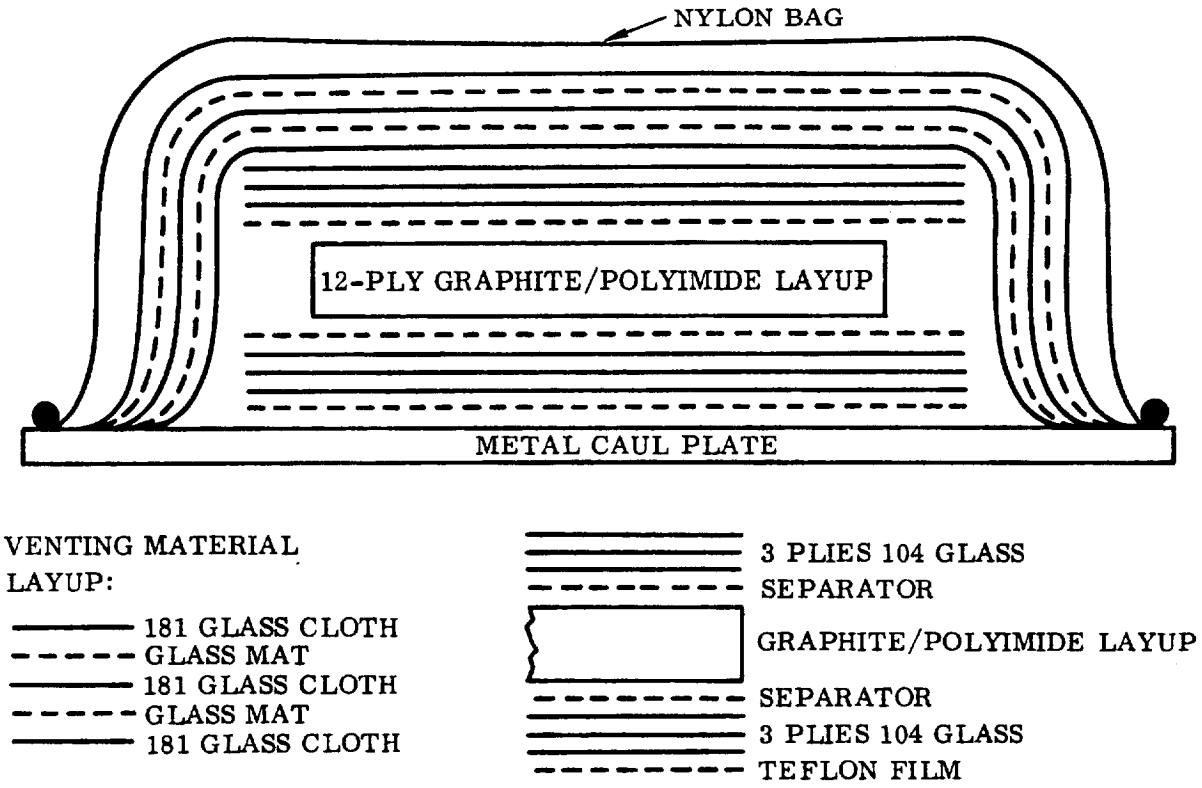


Figure 3-20. Schematic of Graphite/Polyimide Cure Layup

3.7 GRAPHITE/POLYIMIDE (HT-S/710) DESIGN DATA

In the development of composite design data, the laminate fabrication procedure, test specimen geometry, and testing procedure must be carefully selected and strictly followed. Based on the process studies reported in earlier sections of this report, the autoclave cure cycle reported in Section 3.6.8 along with a 644°K (700°F) air or nitrogen postcure cycle was selected as the processing technique for panel fabrication.

To check out testing procedures and test specimen geometry, tabs, etc., a minimal study was conducted prior to the design data generation. Flexure and tensile data on HT-S/710 composites is reported in Tables 3-54 through 3-56. A study was conducted to investigate the short time at temperature problem associated with polyimide composites. To do this, a single panel was cut into flexure specimens and tests were conducted at 589°K (600°F) with various exposure times prior to testing. Tests were conducted at 0, 5, 7, 9, 11, 15, 20, 30, and 60 minutes after reaching a test temperature of 589°K (600°F). The data in Table 3-54 clearly shows that there is no dip in strength; rather, strength increases steadily with increased exposure time. Between 7 and 11 minutes, very little change in strength was noted. Based on this information, a time of 10 minutes at temperature prior to test initiation was selected for all elevated temperature testing. Higher strengths could be developed by leaving the specimen at temperature longer, but this would increase testing costs and for many applications the

Table 3-54. Flexural Strength of Graphite/Polyimide (HT-S/710) Composite System as a Function of Time at 589°K (600° F) Prior to Testing at 589°K (600° F)

Time Prior to Test at 589°K (600°F) Minutes	Flexural Strength	
	MN/m^2	(ksi)
0	775.	(110.2)
5	710.	(101.0)
7	819.	(116.5)
9	821.	(116.8)
11	831.	(118.2)
15	915.	(130.1)
20	980.	(139.4)
30	965.	(137.2)
60	1067.	(151.7)

Fiber Volume (%) 59.0
Resin Content (%) 36.0
Specific Gravity 1.54

Table 3-55. Flexural Strength of Graphite/Polyimide (HT-S/710) Composite System as a Function of Temperature

Test Temperature		Flexural Strength	
°K	(°F)	MN/m^2	(ksi)
77	(-320)	1396	(198.5)
297	(75)	1462.	(207.9)
450	(350)	1438.	(204.5)
533	(500)	1286.	(182.9)
589	(600)	831.	(118.2)
616	(650)	699.	(99.4)
644	(700)	470.	(66.8)
700	(800)	268.	(38.1)

Fiber Volume (%) 59.0
Resin Content (%) 36.0
Specific Gravity 1.54

Table 3-56. Tensile Strength of HT-S/710 Graphite/Polyimide as a Function of Temperature

Test Temperature		Longitudinal Tensile Strength		Tensile Modulus	
°K	(°F)	MN/m^2	(ksi)	GN/m^2	(psi $\times 10^6$)
297	(75)	1193	(173.2)	148	(21.5)
450	(350)	1117	(161.7)	-	-
533	(500)	1110	(160.9)	-	-
589	(600)	1115	(161.9)	149	(21.6)
616	(650)	1041	(151.1)	148	(21.5)
644	(700)	1000	(145.4)	146	(21.1)
700	(800)	690	(99.8)*	-	-

Fiber Volume (%) 62.3
Resin Content (%) 30.7
Specific Gravity 1.52

*Failed in grip area

temperature requirements on a structural part are rapid. Therefore, the strength at temperature after a short time is important. High-temperature, long-term aging tests were also conducted to determine the aging characteristics of the HT-S/710 composite system. This is reported in a later section of this report.

To further evaluate testing procedures, flexural and tensile tests were conducted as functions of temperature as reported in Tables 3-55 and 3-56. The test results indicated that the procedures and specimen configuration were acceptable for conducting tests over the temperature range of 77 to 589° K (-320 to 600° F). Design data tests for the high-strength HT-S/710 composite system are shown in Table 3-57 and discussed in Sections 3.7.1 through 3.7.5.

3.7.1 STATIC PROPERTIES OF HT-S/710 GRAPHITE/POLYIMIDE. To more fully simulate the strength and physical properties obtained in fabrication of large structural parts, the design data laminates were all fabricated as 122 by 61 cm (4 by 2 ft) laminates. A typical laminate is shown in Figure 3-21. The laminates were then cut and given either an air or nitrogen postcure to 644°K (700°F). Fiber volumes, resin contents, and specific gravity were determined for each laminate after completion of the postcure. Laminate physical property data is summarized in Table 3-58. Fiber volumes for the individual laminates were all at 62 ±3%, except for the 8 ply unidirectional and (±45°) laminates. Fiber volumes for these laminates were higher because it is extremely difficult to keep the polyimide resin in the composite when fabricating unidirectional, large thin laminates.

Table 3-57. Design Data Tests for HT-S/710 Graphite/Polyimide

Tests	Test Temperatures	Laminate Orientations
1. Static Tension, Compression, Flexure, and Short Beam Shear	77°K (-320°F) 297°K (75°F) 589°K (600°F)	0°, 45°, 90°, (±45°), (±60°), (0° ±45°), and (0° ±45°, 90°)
2. Biaxial Strain Tube Tests	297°K (75°F)	(±45°)
3. Creep	200 hours at 589°K (600°F)	0° and (±45°)
4. Thick Laminate Compression Tests 1.27 cm (0.50 in.), 2.54 cm (1.00 in.), and 3.81 cm (1.50 in.) Laminates	297°K (75°F)	0 and 90°
5. Ambient and High Temperature Aging Tests	297°K (75°F) and 589°K (600°F)	0° (Ambient) 0 and 90° (High Temperature)

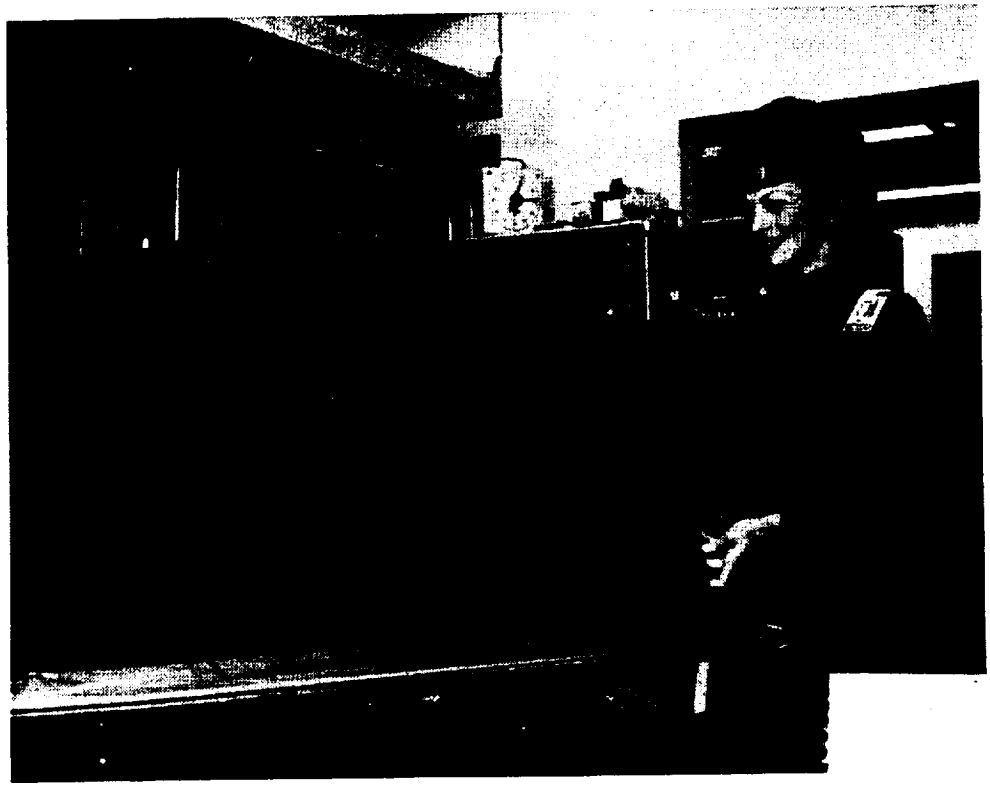


Figure 3-21. Graphite/Polyimide HT-S/710 Design Data Laminate

Table 3-58. Graphite/Polyimide (HT-S/710) Design Data Laminates — Physical Properties

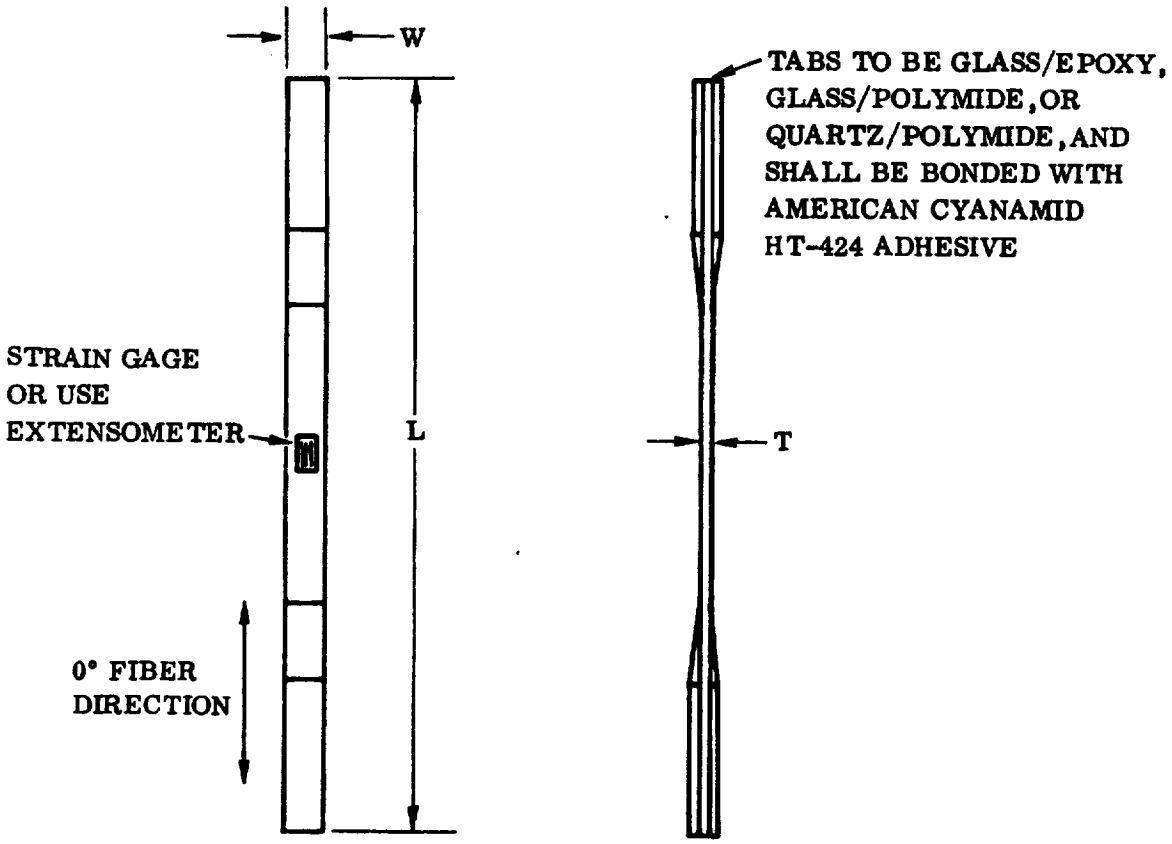
Laminate No.	No. of Plies	Laminate Lay-up	Fiber Volume (%)	Resin Content (%)	Specific Gravity
RD-1	8	0°	68.3	26.4	1.48
RD-2	8	0°	68.6	26.2	1.48
RD-3	12	0°	62.7	32.2	1.53
RD-4	12	0°	59.9	35.1	1.52
RD-5	12	$(0°, \pm45°)_{2s}$	62.2	32.7	1.55
RD-6	12	$(0°, \pm45°)_{2s}$	63.4	31.9	1.50
RD-7	8	$(\pm45°)_{2s}$	64.8	30.3	1.49
RD-8	12	$(\pm45°)_{3s}$	61.1	33.6	1.58
RD-9	12	$(\pm45°)_{3s}$	64.8	30.2	1.52
RD-10	12	$(\pm45°)_{3s}$	62.0	33.0	1.56
RD-11	12	$(\pm60°)_{3s}$	62.8	32.2	1.52
RD-12	12	$(\pm60°)_{3s}$	59.5	35.3	1.57
RD-13	12	$(\pm60°)_{3s}$	62.0	33.0	1.51
RD-14	12	$(\pm60°)_{3s}$	58.6	36.1	1.51
RD-15	8	$(0°, \pm45°, 90°)_{s}$	64.4	30.6	1.50
RD-16	16	$(0°, \pm45°, 90)_{4s}$	62.7	32.3	1.54
RD-17	16	$(0°, \pm45°, 90)_{2s}$	64.0	31.1	1.53
RD-18	16	$(0°, \pm45°, 90)_{2s}$	62.3	32.5	1.54
RD-19	8	0°	67.1	27.9	1.50
RD-21	8	0°	67.5	27.8	1.50
RD-21A	8	$(\pm45°)_{2s}$	68.2	26.3	1.47
RD-22	12	0°	65.6	29.3	1.48
RD-23	12	$(\pm45°)_{3s}$	68.5	26.4	1.44

The tension, compression, flexure, and short beam shear specimens machined from the test laminates are shown in Figure 3-22 through 3-27. All test specimens are laminate-type, since large polyimide sandwich structures cannot be made reliably at this time. Thus, sandwich beams were not used for tension and compression tests. The test specimens conform to the requirements of the Structural Design Guide for Advanced Composite Applications (Reference 3-4), with the exception of the transverse tension specimen. The transverse tension specimen used in this program was an 8 ply, 0.10 cm (0.040 in.) thick specimen instead of the suggested 16 ply, 0.20 cm (0.080 in.) thick specimen. Six specimens were tested at each condition noted in Table 3-57, with the strength, modulus, and strain-to-failure determined for the tension and compression tests. Strength and modulus were determined for the flexure tests and strength only for the short beam shear tests. The data is summarized in Figures 3-28 through 3-33 and presented in tabular form in Tables 3-59 through 3-65. Testing difficulties encountered included unidirectional tension tests at 77°K (-320°F) and unidirectional laminate compression testing at all test temperatures. Postcure cycle 1 was conducted in air, and postcure cycle 2 in nitrogen.

The tensile strength of the various laminate orientations varied very little with temperature as long as there were fibers in the direction of the applied load. The 25% reduction in unidirectional strength at 77°K (-320°F) is attributed primarily to a testing problem and partially to possible embrittlement of the resin. All unidirectional tension failures at 77°K (-320°F) occurred at or within the doublers. Glass/epoxy and quartz/polyimide doublers as well as epoxy, polyimide, and polyurethane adhesives were evaluated in an attempt to solve this problem. Regardless of the combination of doubler and adhesive, the failures occurred in the same spot.

When the tensile strength at 0° on (±45°) and (±60°) laminates was evaluated, the effect of cryogenic temperature was found to be minor, indicating the resin is not affected significantly because the strength in these laminates is more resin-dependent. Tension tests conducted at 589°K (600°F) had no effect on the 0°, (0° ± 45°) or the (0° ±45°, 90°) laminates, but did show a strength decrease of about 25% for the (±45°) and $(\pm 60°)_s$ laminates when tested in the 0° direction. Tension modulus and strain-to-failure both remained relatively constant (±5%) for the entire test temperature range, except for the (±45°) and (±60°) laminates. These laminates showed significant increases in strain-to-failure and lower modulus values when tested at 589°K (600°F). This indicates some softening of the resin.

The Norair laminate compression specimen was selected for this study because it had previously been used over a temperature range of 77°K (-320°F) to 450°K (350°F) by a number of different investigators. The sandwich beam test specimen was not used because of its higher cost in both material and labor and because of the thermal strains that would be induced in the specimen at the test temperature extremes. Test data generated from a compression test using the Norair specimen is considered conservative for strength values, but representative of modulus and strain-to-failure data. Compression strength for all laminate orientations decreased significantly at 589°K (600°F) as compared to room or cryogenic test values. This again indicates that the resin was softening.



Specimen Dimensions:

Length (L) = 20.32 cm (8.00 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.076 to 0.152 cm (0.030 to 0.060 in.)

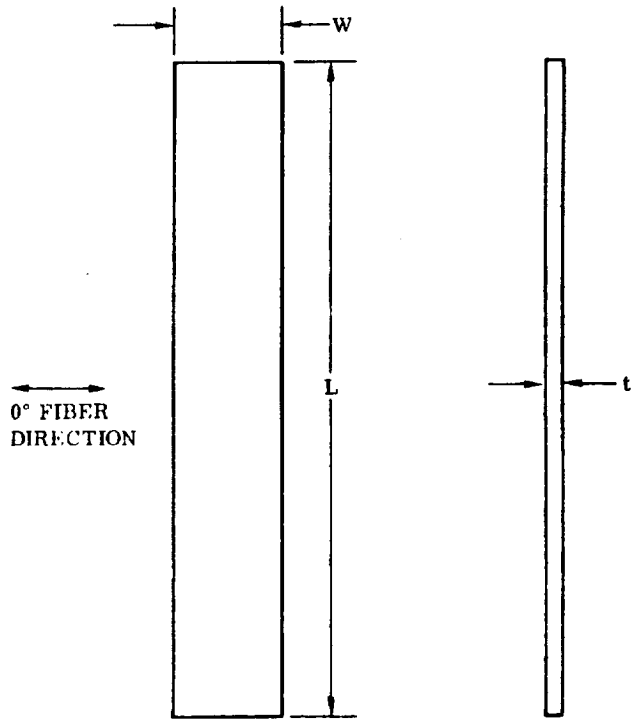
Total Tab Length = 6.35 cm (2.50 in.)

Tab Chamfer Length = 1.91 cm (0.75 in.)

Tab Thickness = 0.076 to 0.152 cm (0.030 to 0.060 in.)

Specimen edges shall be parallel to 0.0076 cm (0.003 in.)

Figure 3-22. Test Specimen for Longitudinal Tension, Creep, and Fatigue Testing



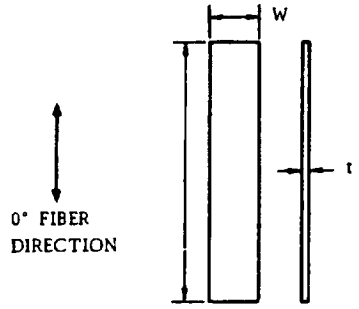
Specimen Dimensions:

Length (L) = 11.430 cm (4.500 in.)

Width (W) = 1.908 cm (0.750 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Figure 3-23. Transverse Tensile Test Specimen



Specimen Dimensions:

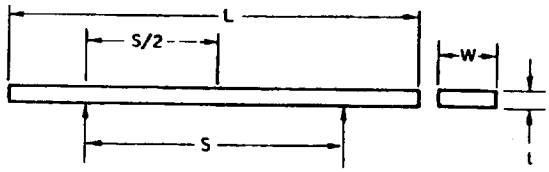
Length (L) = 11.430 cm (4.500 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Top and bottom surfaces shall be parallel and flat to 0.0013 cm (0.0005 in.)
Longitudinal edges shall be parallel to 0.0076 cm (0.003 in.)

Figure 3-24. Longitudinal and Transverse Compression Specimen



Specimen Dimensions:

Length (L) = 7.6 to 10.2 cm (3.0 to 4.0 in.)

Width (W) = 1.270 cm (0.500 in.)

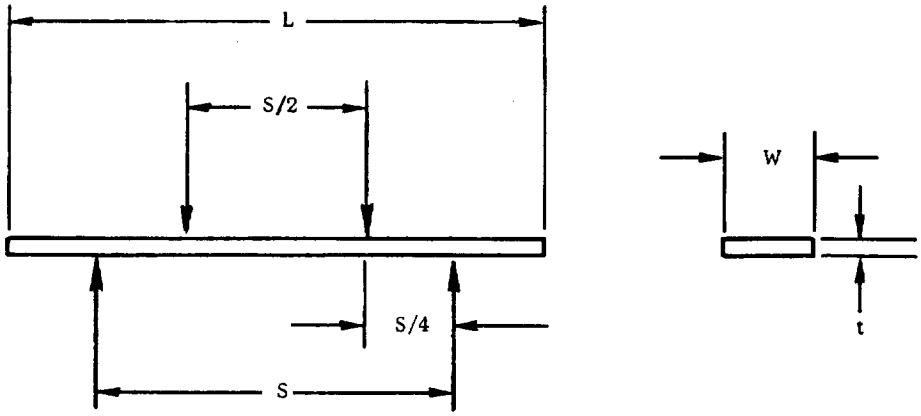
Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 32 to 1

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 3-25. Longitudinal Flexure (0°) Specimen



SPECIMEN DIMENSIONS

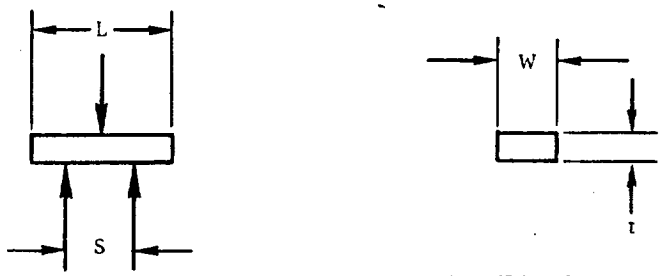
LENGTH (L) . 7.62 cm (3.0 in.)
WIDTH (W) 1.27 cm (0.500 in.)
THICKNESS 0.152-0.228 cm (0.060-0.090 in.)

LOAD METHOD

FOR t = 0.152-0.228 cm (0.060-0.090 in.)
S = 5.08 cm (2.00 in.)

LOAD AND REACTION SUPPORTS SHALL BE 1/8 IN. RADIUS STEEL ROD.
ALL FILAMENTS SHALL BE 90° TO THE L DIMENSION.
LOAD RATE SHALL BE 0.05 IN/MIN.

Figure 3-26. Transverse Flexure (90°) Specimen



SPECIMEN DIMENSIONS

LENGTH (L)	= 152 CM (0.60 IN.)
WIDTH (W)	= 0.635 CM (0.250 IN.)
THICKNESS (t)	= 0.152 CM (0.060 IN.)

LOAD METHOD

S = 4 TO 1
OVERHANG MUST BE SAME OVER EACH END.

LOAD & REACTION SUPPORTS SHALL BE 1/8 IN. RADIUS STEEL ROD.
ALL FILAMENTS TO BE 0° TO THE L DIMENSION.

Figure 3-27. Short Beam Shear Test Specimen

Modulus values decreased except for the 0 and 90° laminate as the test temperature was increased to 589°K (600°F). Strain-to-failure remained constant from room temperature to 589°K (600°F) for all laminates except the (0 ±45°) and (0, ±45°, 90) laminates. This is expected, since strength and modulus decreased significantly for the same change in temperature. The compression strengths reported in Tables 3-59 through 3-65 are believed to be conservative, but the modulus data is believed representative of the HT-S/710 graphite/polyimide composite system.

Flexural and short beam shear tests were conducted on each of the laminate orientations at all three test temperatures. These tests were conducted primarily as a quality control measure for comparison with tag ends from actual structural parts.

3.7.2 THICK LAMINATE FABRICATION AND TESTING. Structural applications often require composite laminates much thicker than the normal 0.254 cm (0.100 in.) laminates. Studies were conducted to determine if thick graphite/polyimide parts could be fabricated and what effect the increased thickness had on mechanical properties. Process development studies were conducted; and it was determined that by slight modification of the cure cycle reported in Section 3.6.8, thick laminates could be fabricated successfully. The only change to the cure cycle was that the heat-up rate was decreased to 0.3°K/min (1°F/min) from the normal 1 to 3°K/min (3 to 5°F/min). Total postcure time was doubled because the hold time at each temperature was doubled. This extra time allowed any volatiles remaining in the composite to migrate to the surface and escape. Two typical 2.54 cm (1.00 in.) laminates are shown in Figure 3-34. Laminates were fabricated and machined into block compression specimens, shown in Figure 3-35, for testing at 297°K (75°F).

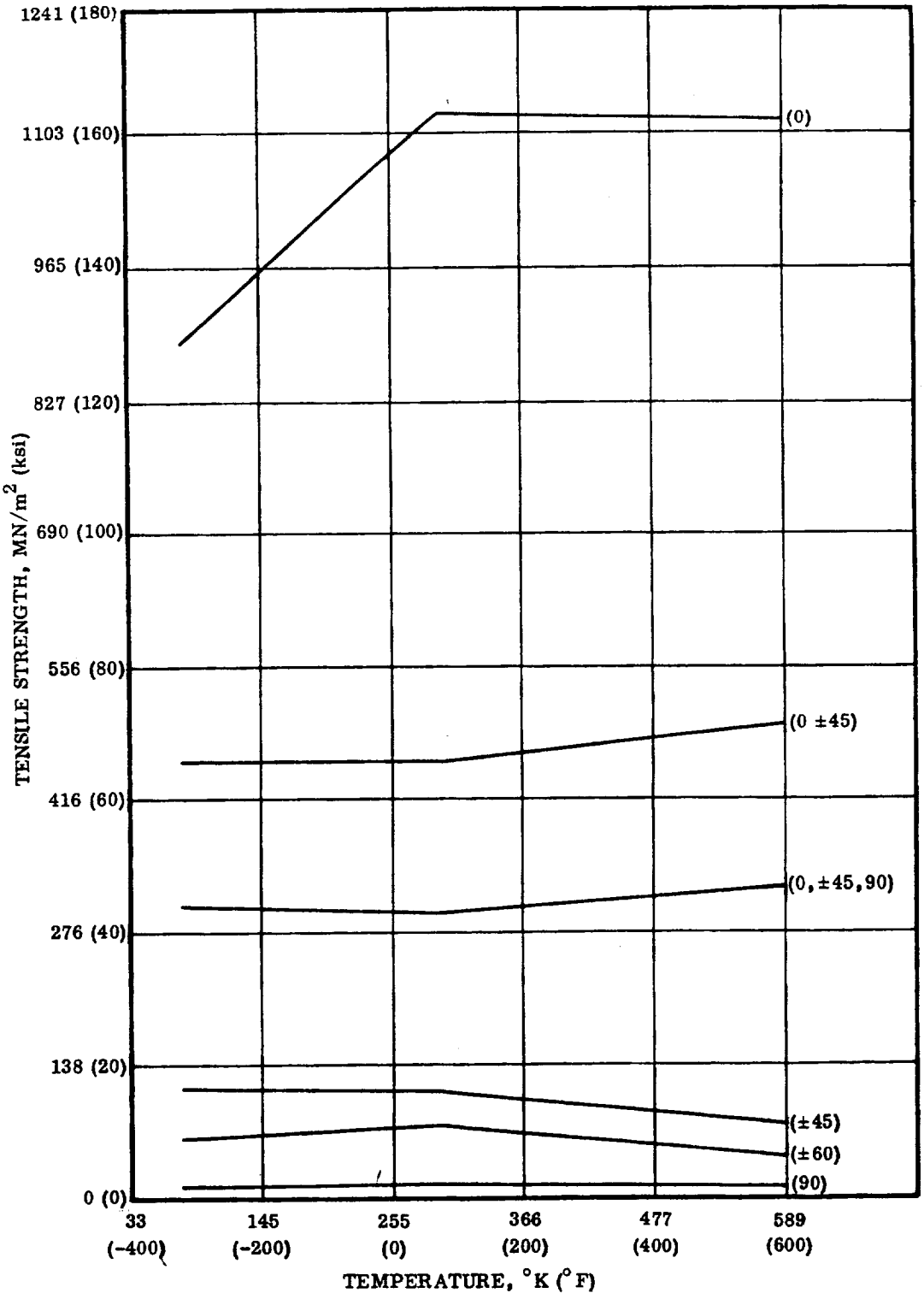


Figure 3-28. Tensile Strength (HT-S/710) as a Function of Temperature and Laminate Orientation

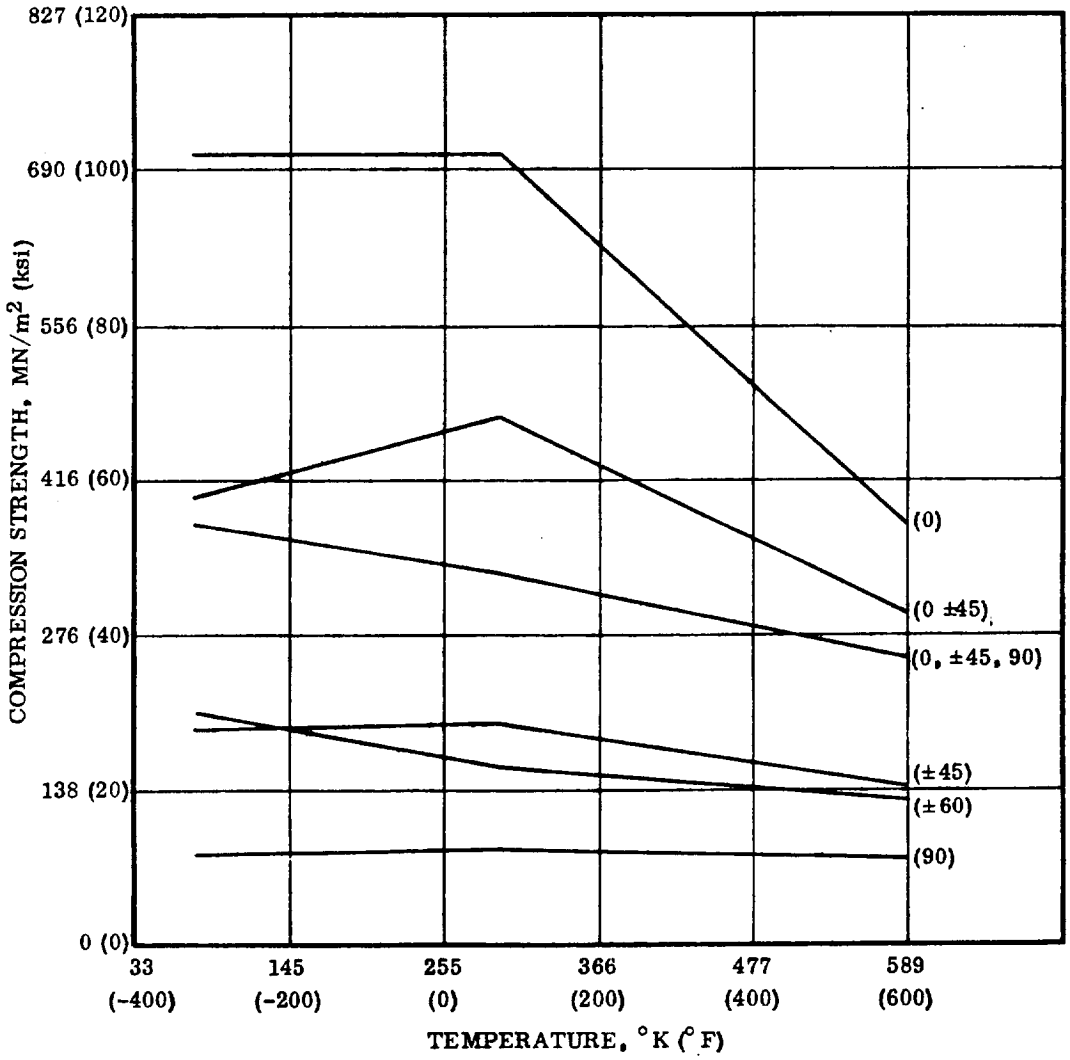


Figure 3-29. Compression Strength (HT-S/710) as a Function of Temperature and Laminate Orientation

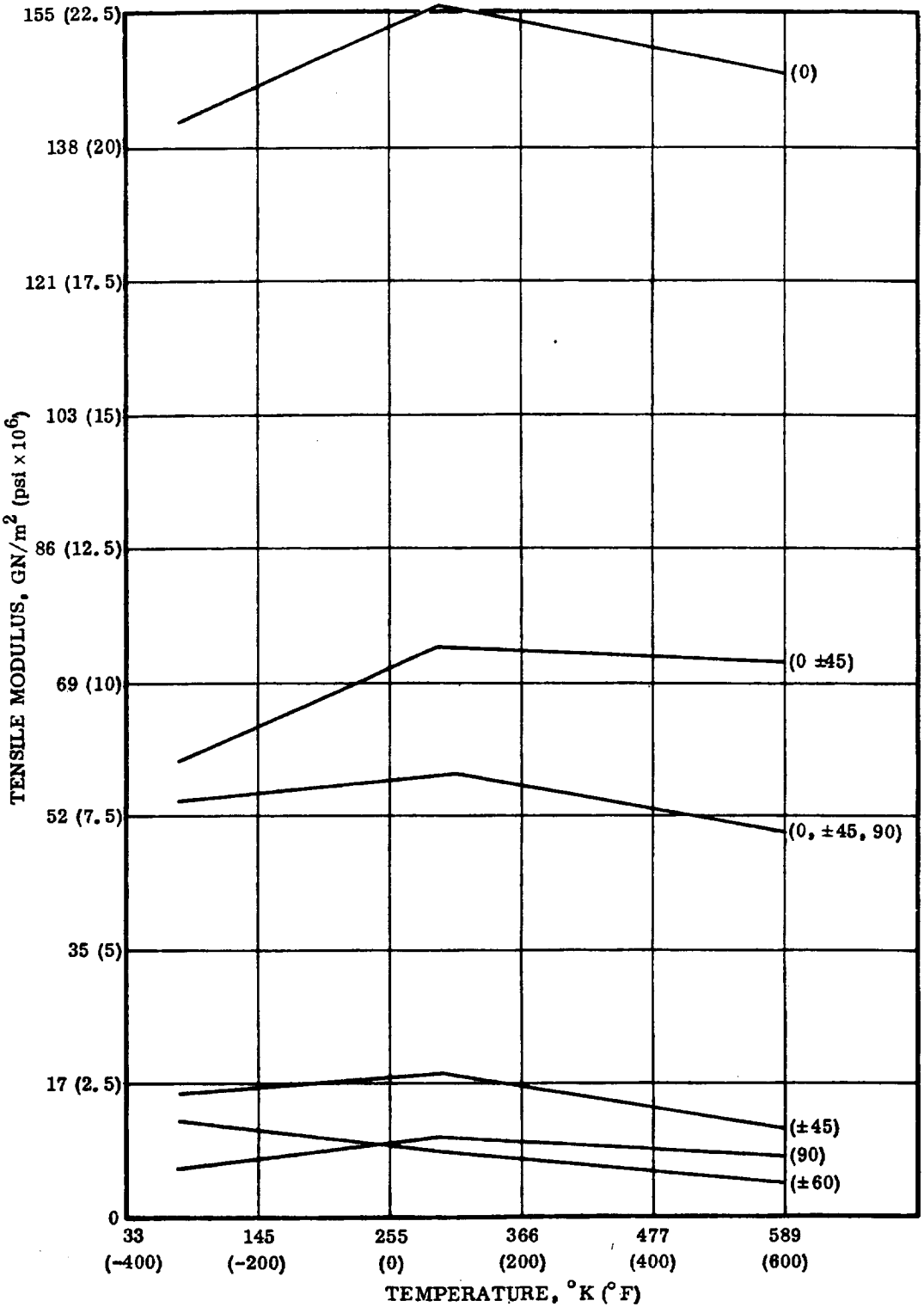


Figure 3-30. Tensile Modulus of HT-S/710 as a Function of Temperature and Laminate Orientation

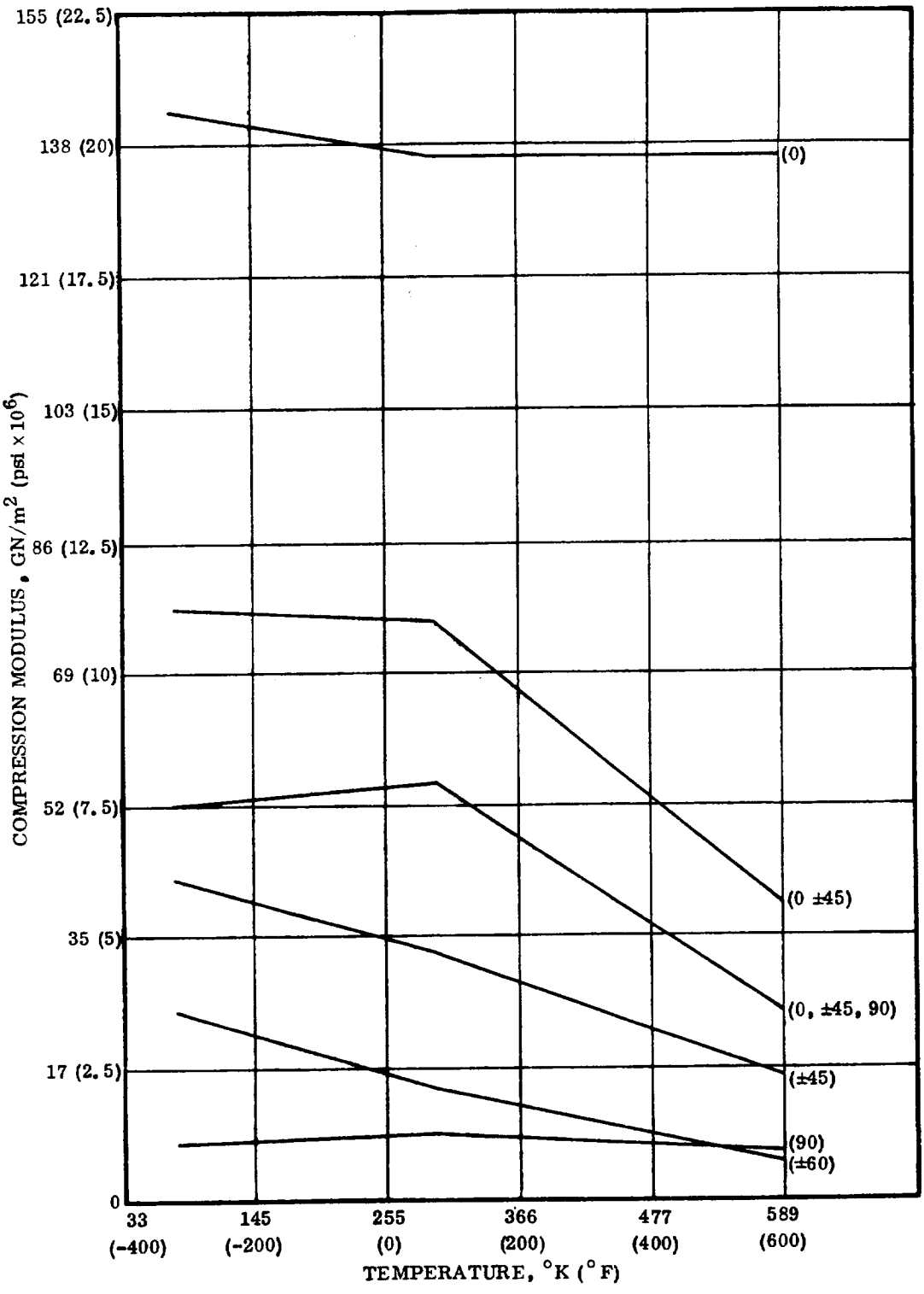


Figure 3-31. Compression Modulus of HT-S/710 as a Function of Temperature and Laminate Orientation

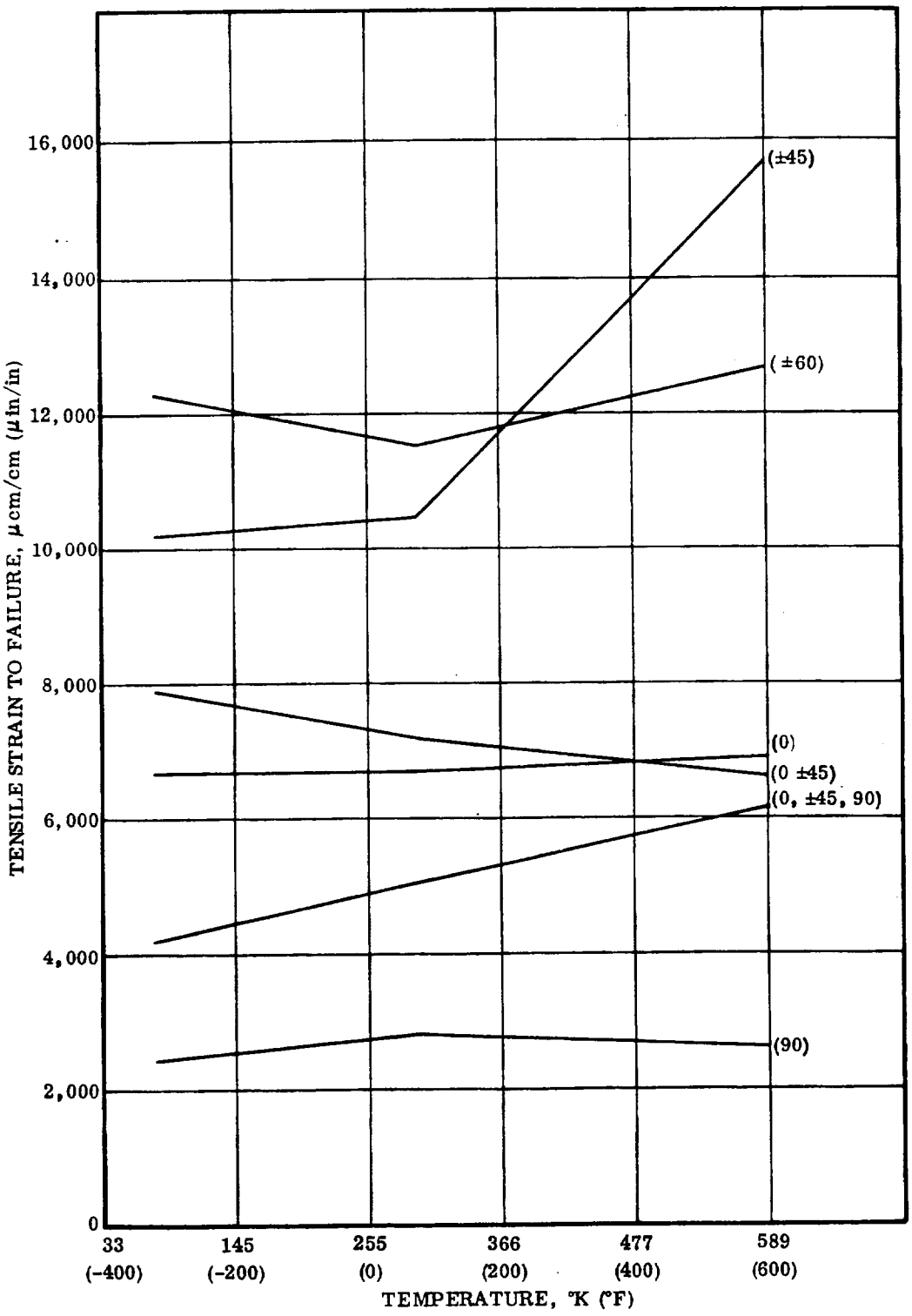


Figure 3-32. Tensile Strain to Failure (HT-S/710) as a Function of Temperature and Laminate Orientation

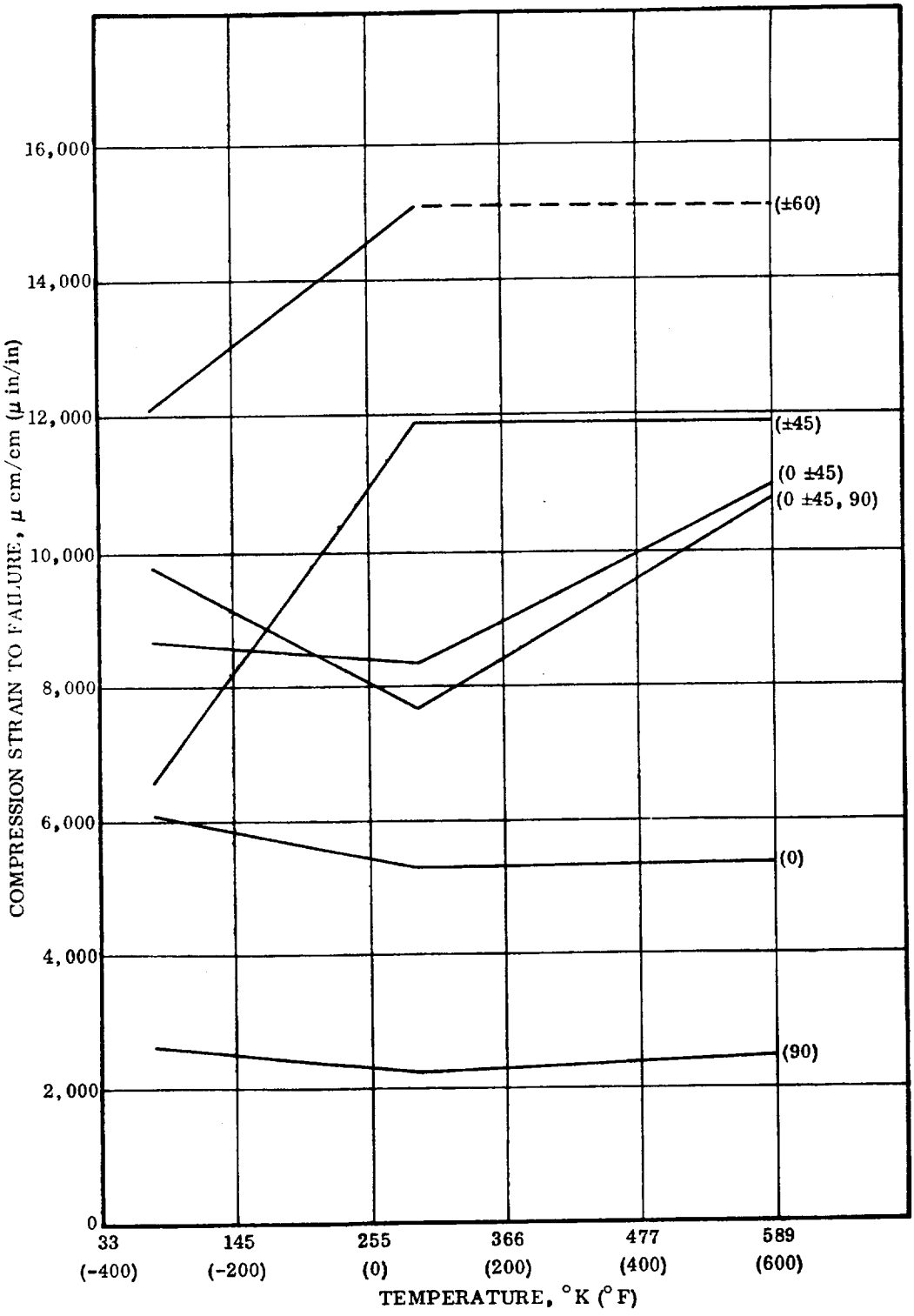


Figure 3-33. Compression Strain to Failure (HT-S/710) as a Function of Temperature and Laminate Orientation

Table 3-59. Design Properties of HT-S/710 Composites - 0°

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
	Air	0	0	910 (132.4)	134 (19.4)	(6780)	665 (9615)	139 (20.2)	(6200)	1220 (177.3)	148 (21.4)	889 (12.90)
				965 (139.9)	126 (18.3)	(6580)	656 (95.2)	136 (19.8)	(6100)	1324 (192.4)	126 (18.3)	827 (12.00)
				993 (144.4)	152 (22.1)	(6980)	660 (95.9)	142 (20.6)	(5800)	1386 (201.3)	146 (21.1)	834 (12.10)
				924 (134.2)	137 (19.9)	(6120)	841 (122.1)	131 (19.0)	(6400)	1255 (182.0)	149 (21.6)	972 (14.08)
				738 (106.8)	145 (20.9)	(7320)	724 (105.4)	160 (23.2)	-	1310 (190.4)	152 (22.0)	1020 (14.79)
				814 (118.4)	152 (22.1)	(6280)	690 (100.1)	- -	(5760)	1289 (186.8)	126 (18.2)	958 (13.86)
			Average	891 (129.4)	141 (20.5)	(6676)	706 (102.5)	142 (20.6)	(6052)	1297 (188.3)	141 (20.4)	917 (13.29)
297 (75)	Air	0	0	1172 (169.6)	154 (22.4)	(6600)	765 (111.3)	128 (19.1)	-	1448 (210.0)	156 (22.6)	710 (10.30)
				1186 (171.8)	155 (22.5)	(7400)	772 (112.1)	137 (19.9)	(6100)	1386 (200.8)	154 (22.4)	772 (11.18)
				1062 (154.2)	164 (23.8)	(6450)	639 (92.6)	124 (18.0)	(5000)	1503 (217.8)	169 (24.5)	752 (10.90)
				1145 (166.4)	162 (23.5)	(6600)	674 (97.6)	150 (21.7)	(5260)	1393 (201.8)	148 (21.4)	821 (11.88)
				1124 (162.6)	158 (22.9)	(7100)	655 (95.0)	131 (19.0)	(5000)	1469 (212.8)	159 (23.1)	910 (13.21)
				1048 (152.2)	147 (21.3)	(6300)	731 (106.3)	141 (20.4)	(5220)	1593 (230.6)	155 (22.5)	889 (12.85)
			Average	1123 (162.8)	157 (22.7)	(6741)	706 (102.5)	135 (19.7)	(5316)	1465 (212.3)	157 (22.8)	809 (11.72)
589 / (600)	Air	0	0	1131 (164.3)	148 (21.5)	(6100)	364 (52.9)	- -	-	986 (142.7)	126 (18.3)	492 (7.13)
				1131 (164.2)	- -	-	386 (56.3)	- -	-	945 (136.9)	125 (18.1)	460 (6.67)
				1096 (158.9)	- -	-	415 (60.3)	- -	-	896 (129.5)	126 (18.2)	476 (6.91)
				1138 (165.4)	160 (23.2)	-	376 (54.6)	136 (19.7)	(5560)	993 (144.4)	132 (19.2)	445 (6.45)
				1076 (156.4)	139 (20.2)	(7700)	317 (46.0)	143 (20.7)	(4400)	827 (120.5)	132 (19.2)	475 (6.90)
				1117 (162.0)	- -	-	391 (56.7)	126 (18.3)	(6220)	938 (136.2)	136 (19.7)	466 (6.76)
			Average	1115 (161.9)	149 (21.6)	(6900)	375 (54.5)	135 (19.6)	(5394)	931 (135.0)	130 (18.8)	469 (6.80)
297 (75)	GN_2	0	0	1117 (162.0)	154 (22.3)	(7200)	745 (107.7)	135 (19.6)	(5780)	1482 (214.9)	148 (21.5)	650 (9.43)
				931 (134.7)	158 (22.8)	(5750)	827 (119.8)	141 (20.5)	(6660)	1420 (206.0)	136 (19.7)	680 (9.86)
				1034 (150.4)	152 (22.1)	(6450)	738 (106.6)	131 (19.0)	(5780)	1427 (206.8)	139 (20.1)	731 (10.65)
				1076 (156.0)	158 (22.9)	(6650)	661 (94.7)	124 (18.0)	(6600)	1372 (198.6)	- -	793 (11.51)
				986 (143.3)	154 (22.4)	(6400)	675 (96.5)	151 (21.9)	(5830)	1338 (193.8)	139 (20.1)	772 (11.19)
				1007 (146.5)	154 (22.3)	(6700)	682 (98.5)	132 (19.1)	(5830)	1386 (201.2)	133 (19.3)	841 (12.15)
			Average	1025 (148.8)	155 (22.5)	(6525)	721 (104.0)	135 (19.7)	(6080)	1404 (203.6)	139 (20.1)	752 (10.91)
												746 (10.81)

Table 3-60. Design Properties of HT-S/710 Composites - 45°

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	0	45	22.7 (3.30)	- -	-	148 (21.4)	17.2 (2.50)	-	47.9 (6.94)	- -	25.5 (3.73)
				13.1 (1.92)	- -	-	138 (20.0)	12.3 (1.78)	-	43.0 (6.19)	- -	20.1 (2.92)
				17.2 (2.46)	- -	-	121 (17.6)	13.6 (1.98)	-	45.8 (6.63)	- -	24.8 (3.63)
				41.5 (6.02)	12.5 (1.82)	(3300)	172 (17.0)	9.1 (1.32)	-	32.5 (4.73)	12.8 (1.86)	17.1 (2.48)
				42.2 (6.13)	- -	-	110 (15.1)	7.6 (1.10)	-	35.3 (5.10)	11.2 (1.62)	12.0 (1.74)
				41.9 (6.08)	12.7 (1.84)	(3300)	122 (17.7)	9.8 (1.42)	-	52.8 (7.63)	13.0 (1.89)	14.5 (2.10)
			Average	29.8 (4.31)	12.6 (1.83)	(3300)	135 (18.3)	11.6 (1.68)		42.9 (6.20)	12.3 (1.79)	19.0 (2.77)
297 (75)	Air	0	45	23.2 (3.50)	12.4 (1.80)	(2000)	164 (23.8)	7.9 (1.15)	-	50.0 (7.24)	12.1 (1.76)	16.6 (2.40)
				27.2 (3.95)	11.3 (1.64)	(2840)	147 (21.3)	12.2 (1.77)	-	43.0 (6.20)	10.8 (1.56)	20.6 (2.99)
				27.6 (4.01)	- -	-	138 (20.0)	16.3 (2.37)	-	38.8 (5.62)	11.4 (1.65)	20.3 (2.94)
				41.2 (5.96)	12.5 (1.82)	(3280)	140 (20.3)	- -	-	39.5 (5.67)	9.5 (1.38)	18.1 (2.63)
				38.7 (5.62)	11.0 (1.59)	(3600)	129 (18.7)	9.3 (1.35)		51.4 (7.35)	11.7 (1.70)	20.0 (2.90)
				31.7 (4.60)	15.7 (2.28)	(2680)	121 (17.6)	6.8 (0.98)		37.4 (5.45)	- -	22.0 (3.22)
			Average	31.6 (4.60)	12.6 (1.83)	(2880)	140 (20.3)	10.5 (1.52)		43.4 (6.26)	11.1 (1.61)	19.6 (2.85)
589 (600)	Air	0	45	17.2 (2.50)	5.0 (0.73)	(5000)	126 (18.3)	3.7 (0.53)	-	38.1 (5.53)	- -	12.3 (1.78)
				18.1 (2.62)	6.1 (0.88)	(3800)	101 (14.6)	4.0 (0.57)	-	46.5 (6.66)	- -	11.6 (1.68)
				9.0 (1.33)	7.6 (1.07)	-	101 (14.7)	2.9 (0.42)	-	49.3 (7.09)	8.5 (1.23)	12.3 (1.78)
				18.1 (2.63)	- -	-				38.8 (5.61)	12.1 (1.76)	15.6 (2.27)
				23.4 (3.39)	8.3 (1.21)	-				40.2 (5.80)	10.8 (1.57)	19.3 (2.80)
				22.9 (3.32)	6.0 (0.87)	(5300)				44.4 (6.37)	13.4 (1.95)	29.0 (4.15)
			Average	18.1 (2.63)	6.6 (0.95)	(4700)	109 (15.9)	3.5 (0.51)		42.9 (6.17)	11.2 (1.63)	16.7 (2.41)
297 (75)	GN_2		45	20.6 (2.99)	14.3 (2.07)	(1750)	125 (18.1)	12.3 (1.79)	-	55.6 (8.01)	11.6 (1.69)	14.6 (2.11)
				20.6 (2.99)	17.4 (2.52)	(1900)	101 (14.7)	9.6 (1.40)	-	40.2 (5.81)	13.1 (1.90)	13.8 (2.00)
				22.1 (3.21)	15.7 (2.28)	(1500)	127 (18.4)	10.3 (1.49)	-	39.5 (5.74)	12.7 (1.84)	10.8 (1.56)
				20.1 (2.92)	16.1 (2.33)	(1650)	99 (14.3)	11.4 (1.66)	(4125)			14.9 (2.16)
				17.7 (2.57)	17.6 (2.55)	(1260)	105 (15.2)	11.4 (1.65)	(4375)			11.6 (1.69)
				20.0 (2.92)	6.9 (1.02)	(2900)	101 (14.6)	11.7 (1.70)	(4250)			15.0 (2.18)
			Average	20.2 (2.93)	14.7 (2.13)	(1826)	110 (15.9)	11.1 (1.61)	4250	45.1 (6.52)	12.5 (1.81)	13.4 (1.95)

Table 3-61. Design Properties of HT-S/170 Composites - 90°

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	0	90	11.5 (1.66)	5.17 (0.75)	(2300)	80.0 (11.6)	8.48 (1.23)	(2765)	13.0 (1.89)		4.7 (0.68)
				16.0 (2.32)	7.86 (1.14)	(2250)	80.0 (11.6)	7.17 (1.04)	(2450)	9.2 (1.34)		4.5 (0.65)
				19.9 (2.88)	5.99 (0.87)	(3000)	77.9 (11.3)	8.07 (1.17)	(2830)	16.5 (2.39)		1.9 (0.28)
				9.2 (1.31)	4.61 (0.67)	–	79.3 (11.5)	– –	–	13.3 (1.93)		1.5 (0.22)
				12.0 (1.74)	– –	–	89.6 (13.0)	8.07 (1.17)	(2560)	12.5 (1.81)		1.9 (0.27)
				11.8 (1.71)	6.48 (0.94)	(2250)	– –	– –	–	14.1 (2.05)		1.4 (0.20)
			Average	13.4 (1.94)	6.02 (0.87)	(2450)	81.4 (11.8)	7.95 (1.15)	(2662)	13.1 (1.90)		2.6 (0.38)
297 (75)	Air	0	90	12.3 (1.78)	– –	–	104.1 (15.1)	10.14 (1.47)	(1905)	11.4 (1.65)		2.5 (0.36)
				12.8 (1.85)	9.58 (1.39)	(2720)	76.5 (14.0)	7.93 (1.15)	(2930)	10.4 (1.51)		4.7 (0.67)
				13.3 (1.93)	8.83 (1.28)	(3000)	75.8 (11.0)	7.31 (1.06)	–	12.2 (1.77)		3.0 (0.43)
				17.9 (2.59)	9.17 (1.33)	(2650)	78.6 (11.4)	– –	–	13.9 (1.97)		2.5 (0.36)
				20.3 (2.95)	8.83 (1.28)	(3375)	64.0 (9.2)	6.90 (1.00)	–	14.5 (2.10)		2.1 (0.30)
				19.3 (2.80)	10.69 (1.55)	(2550)	93.1 (13.5)	– (1.40)	(2020)	13.5 (1.96)		3.8 (0.53)
			Average	16.0 (2.32)	9.42 (1.37)	(2859)	85.5 (12.4)	8.07 (1.22)	(2285)	12.6 (1.83)		3.0 (0.44)
589 (600)	Air	0	90	11.1 (1.61)	– –	–	80.7 (11.7)			13.8 (2.00)		2.6 (0.37)
				10.8 (1.56)	8.14 (1.18)	(2700)	82.7 (12.0)			15.2 (2.20)		2.1 (0.31)
				12.5 (1.82)	8.96 (1.30)	(2100)	73.1 (10.6)		(0.99) (2500)	15.5 (2.25)		1.9 (0.28)
				12.7 (1.84)	7.65 (1.11)	(3100)				14.6 (2.12)		2.0 (0.29)
				10.3 (1.50)	– –	–				14.7 (2.13)		1.8 (0.26)
				13.7 (1.99)	7.58 (1.10)	(2600)				16.1 (2.33)		2.3 (0.34)
			Average	11.8 (1.72)	8.08 (1.17)	(2625)	78.6 (11.4)	6.83 (0.99)	(2500)	15.0 (2.17)		2.1 (0.31)
297 (75)	GN_2	0	90	8.8 (1.28)	– –	(2425)	80.7 (11.7)	7.31 (1.06)	(2135)	10.5 (1.52)		– –
				10.1 (1.46)	6.48 (0.94)	(1700)	79.3 (11.5)	7.38 (1.07)	(2270)	10.1 (1.46)		5.9 (0.84)
				10.1 (1.46)	7.03 (1.02)	(1650)	75.8 (11.0)	8.48 (1.23)	–	10.1 (1.46)		4.9 (0.71)
				15.7 (2.28)	10.83 (1.57)	(2200)	80.0 (11.6)	8.55 (1.24)	–	12.8 (1.85)		4.6 (0.66)
				11.0 (1.60)	9.03 (1.31)	(1650)	73.7 (10.7)	7.45 (1.08)	(2200)	16.9 (2.45)		3.5 (0.51)
				12.8 (1.85)	8.00 (1.16)	(2000)	79.3 (11.5)	8.48 (1.23)	–	16.5 (2.39)		4.65 (0.67)
			Average	11.5 (1.66)	8.27 (1.20)	(1938)	78.1 (11.3)	7.94 (1.15)	(2202)	12.8 (1.85)		4.7 (0.68)

Table 3-62. Design Properties of HT-S/710 Composites - (±60°)

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	(±60)	0°	63 (9.12)	5.7 (.83)	(11200)	200 (28.6)	26.7 (3.88)	(7800)	138 (20.1)	8.1 (1.18)	18.6 (2.69)
				76 (10.91)	35.1 (5.11)	(12400)	206 (30.1)	20.1 (2.91)	(10500)	180 (25.9)	11.0 (1.59)	21.2 (3.07)
				65 (9.48)	10.8 (1.50)	(11900)	255 (37.4)	24.3 (3.53)	(12100)	158 (23.4)	9.7 (1.41)	20.8 (3.02)
				63 (9.20)	6.7 (.97)	(13400)	152 (22.1)	31.7 (4.60)	(9300)	131 (18.8)	8.1 (1.17)	18.1 (2.62)
				60 (8.70)	5.8 (.84)	(12600)	220 (31.8)	21.9 (3.18)	(16000)	124 (17.5)	8.7 (1.26)	19.1 (2.77)
				61 (8.90)	6.1 (.88)	(12500)	206 (29.9)	23.9 (3.46)	(16450)	138 (19.5)	10.1 (1.46)	17.1 (2.48)
			Average	64.7 (9.39)	12.4 (1.80)	(12300)	207 (30.0)	24.8 (3.59)	(12100)	145 (20.9)	9.3 (1.35)	192 (2.78)
297 (75)	Air	(±60)	0	69 (10.14)	8.9 (1.29)	(11800)	166 (23.9)	17.6 (2.55)	(12600)	158 (22.7)	8.7 (1.26)	26.6 (3.85)
				83 (11.51)	10.5 (1.52)	(13200)	180 (26.2)	13.2 (1.91)	(17700)	138 (20.3)	7.7 (1.11)	21.0 (3.05)
				76 (10.68)	11.6 (1.68)	(9800)	166 (24.4)	16.9 (2.45)	(15700)	138 (19.9)	8.4 (1.22)	21.3 (3.09)
				76 (10.30)	9.4 (1.37)	-	166 (24.3)	11.0 (1.60)	-	124 (17.9)	7.7 (1.12)	23.0 (3.33)
				76 (10.74)	6.1 (0.89)	-	145 (20.9)	12.9 (1.87)	(15300)	131 (18.8)	7.8 (1.13)	20.4 (2.96)
				76 (10.76)	4.9 (0.71)	-	131 (19.0)	12.1 (1.76)	(14000)	131 (19.0)	7.1 (1.03)	19.9 (2.89)
			Average	76.0 (10.69)	8.6 (1.24)	(11600)	159 (23.1)	13.9 (2.02	(15100)	137 (19.8)	7.9 (1.15)	22.0 (3.20)
589 (600)	Air	(±60)	0	42.4 (6.15)	3.7 (.53)	(12400)	117 (17.0)	3.79 (.55)		69 (10.0)	2.6 (.38)	14.9 (2.16)
				46.4 (6.73)	5.0 (.73)	(11600)	117 (17.4)	1.86 (.27)		68 (9.9)	3.0 (.43)	17.0 (2.46)
				48.4 (7.02)	4.8 (.70)	(11700)	124 (17.8)	1.86 (.27)		76 (10.8)	3.1 (.45)	14.7 (2.13)
				45.2 (6.56)	5.0 (.72)	(15000)	138 (19.6)	7.72 (1.12)		55 (8.0)	2.9 (.42)	18.2 (2.64)
				35.4 (5.14)	3.2 (.46)	(10800)	180 (25.9)	6.21 (1.21)		57 (8.2)	2.7 (.39)	16.1 (2.33)
				36.3 (5.26)	4.1 (.59)	(14400)	124 (17.6)	7.03 (1.16)		62 (9.0)	2.7 (.39)	18.2 (2.64)
			Average	42.4 (6.14)	4.3 (.62)	(12700)	133 (19.2)	5.24 (.76)		65 (9.3)	2.8 (.41)	16.5 (2.39)
297 (75)	GN_2	(±60)	0	69 (10.4)	10.8 (1.57)	(10200)	192 (27.7)	15.4 (2.23)	(17800)	110 (16.2)	10.9 (1.58)	23.0 (3.33)
				76 (11.0)	12.3 (1.79)	(10700)	166 (24.0)	12.9 (1.87)	-	131 (19.4)	11.8 (1.71)	21.9 (3.18)
				76 (10.8)	11.0 (1.59)	(14000)	172 (24.6)	14.0 (2.03)	-	158 (22.6)	11.9 (1.73)	21.6 (3.13)
				68 (9.8)	6.1 (0.89)	-	172 (24.7)	12.4 (1.80)	(16900)	138 (20.4)	14.9 (2.16)	29.9 (4.34)
				60 (8.7)	6.5 (0.94)	(13800)	158 (23.3)	9.8 (1.42)	(24000)	131 (19.3)	16.3 (2.37)	32.1 (4.65)
				62 (9.2)	6.8 (0.98)	(10800)	138 (20.2)	11.0 (1.59)	(23100)	110 (16.3)	12.3 (1.78)	31.9 (4.63)
			Average	68 (9.98)	8.9 (1.29)	(11900)	166 (24.1)	12.6 (1.82)	(20500)	130 (19.0)	1310 (1.89)	26.7 (3.88)

Table 3-63. Design Properties of HT-S/710 Composites - (± 45°)

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	$(\pm 45)_{2s}$	0	124 (18.0)	17.5 (2.53)	(8100)	186 (26.7)	47.7 (6.92)	(5900)	339 (49.3)	26.2 (3.8)	30.4 (4.41)
				138 (19.6)	19.0 (2.76)	(-)	200 (29.2)	- -	-	311 (44.7)	22.8 (3.3)	25.9 (3.75)
				117 (16.9)		(-)	172 (25.2)	45.4 (6.59)	(5000)	304 (43.5)	27.6 (4.0)	33.9 (4.91)
				110 (15.8)	15.7 (2.28)	(11200)	186 (27.0)	37.0 (5.37)	(8300)	290 (42.0)	20.7 (3.0)	23.5 (3.41)
				103 (15.4)	17.5 (2.54)	(10800)	213 (31.4)	42.6 (6.18)	(6450)	269 (38.9)	20.0 (2.9)	24.3 (3.53)
				103 (14.9)	11.4 (1.65)	(10600)	192 (27.6)	36.2 (5.25)	(7120)	234 (34.2)	18.3 (2.7)	21.9 (3.17)
			Average	116 (16.8)	16.2 (2.35)	(10200)	192 (27.9)	41.8 (6.06)	(6550)	291 (42.1)	22.8 (3.3)	26.7 (3.86)
297 (75)	Air	$(\pm 45)_{2s}$	0	145 (20.9)	17.2 (2.5)	(14000)	172 (25.1)	29.8 (4.32)	(11100)	283 (41.0)	17.2 (2.5)	38.2 (5.54)
				131 (18.7)	15.2 (2.2)	(13200)	166 (24.1)	27.2 (3.94)	(13800)	276 (39.9)	13.8 (2.0)	32.1 (4.65)
				96 (14.3)	13.8 (2.0)	(8800)	206 (30.4)	24.7 (3.58)	(11100)	276 (39.8)	14.5 (2.1)	32.9 (4.77)
				96 (14.3)	14.5 (2.1)	(12300)	206 (29.5)	34.2 (4.96)	(7700)	248 (35.8)	15.9 (2.3)	32.1 (4.66)
				103 (14.9)	26.2 (3.8)	(7930)	234 (33.5)	41.1 (5.96)	(14000)	248 (36.4)	15.9 (2.3)	27.8 (4.03)
				96 (13.9)	18.6 (2.8)	(6560)	200 (28.5)	34.8 (5.04)	(13600)	255 (37.4)	16.6 (2.4)	27.4 (3.97)
			Average	111 (16.2)	17.9 (2.6)	(10500)	197 (28.5)	31.9 (4.63)	(11900)	264 (36.7)	15.9 (2.3)	31.8 (4.60)
589 (600)	Air	$(\pm 45)_{2s}$	0	76 (11.2)	10.3 (1.5)	(13600)	152 (21.7)	14.6 2.12	-	124 (17.8)	11.7 (1.7)	25.4 (3.69)
				76 (11.0)	13.8 (2.0)	(13200)	152 (22.3)	13.8 2.00		138 (19.9)	- -	21.2 (3.08)
				76 (10.6)	15.2 (2.2)	(13100)	186 (26.7)	14.8 2.15	-	192 (27.6)	12.4 (1.8)	22.8 (3.30)
				69 (9.9)	12.1 (1.76)	(7200)	124 (17.9)	17.2 (2.49)	(9900)	158 (23.3)	10.3 (1.5)	13.9 (2.02)
				76 (11.2)	9.4 (1.36)	(21000)	110 (15.9)	18.5 (2.69)	-	145 (20.8)	7.6 (1.1)	24.1 (3.49)
				76 (10.6)	7.0 (1.02)	(26000)	124 (18.4)	11.8 (1.71)	(13900)	152 (21.5)	6.9 (1.0)	23.7 (3.44)
			Average	74.8 (10.8)	11.7 (1.7)	(15700)	141 (20.5)	15.1 (2.19)	(11900)	151 (21.9)	9.7 (1.4)	21.9 (3.17)
297 (75)	GN_2	$(\pm 45)_{2s}$	0	117 (16.9)	- -	-	158 (22.9)	24.8 (3.60)	(8660)	283 (41.2)	19.3 (2.80)	39.3 (5.70)
				110 (16.2)	11.4 (1.65)	-	158 (23.2)	26.2 (3.80)	(10000)	276 (40.4)	15.2 (2.20)	32.6 (4.73)
				117 (17.1)	19.4 (2.82)	-	186 (26.9)	26.8 (3.88)	(8110)	255 (36.8)	14.5 (2.10)	22.6 (3.27)
				90 (13.2)	16.6 (2.40)	(8700)	213 (30.5)	20.7 (3.00)	(7200)	269 (38.8)	16.1 (2.34)	21.2 (3.07)
				103 (15.4)	19.7 (2.85)	(8300)	172 (25.2)	30.8 (4.47)	(7670)	227 (33.4)	16.8 (2.44)	20.9 (3.03)
				96 (13.8)	14.1 (2.04)	(8200)	172 (25.3)	35.3 (5.11)	(6500)	227 (32.7)	14.8 (2.14)	20.2 (2.93)
			Average	106 (15.4)	16.2 (2.35)	(8400)	177 (25.7)	27.5 (3.98)	(8200)	256 (37.2)	16.1 (2.37)	26.1 (3.79)

Table 3-64. Design Properties of HT-S/710 Composites (0 ± 45°)

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	$(0, \pm 45)_{2s}$	0	444 (64.4)	67.8 (9.84)	(8040)	367 (52.9)	78.7 (11.4)	(9100)	311 (45.4)	80.0 (11.6)	38.3 (5.56)
				423 (61.0)	53.8 (7.80)	(8730)	339 (48.6)	88.4 (12.8)	(8600)	318 (46.1)	75.2 (10.9)	43.2 (6.27)
				430 (61.8)	56.3 (8.17)	(7610)	395 (57.4)	52.7 (7.64)	(14300)	332 (47.9)	66.1 (9.6)	39.7 (5.76)
				472 (68.4)	55.4 (8.04)	(7200)	444 (64.3)	78.7 (11.4)	(4700)	318 (45.6)	- -	41.2 (5.98)
				- -	- -	-	465 (67.4)	73.1 (10.6)	(9300)	416 (59.5)	- -	36.8 (5.33)
				507 (73.3)	57.4 (8.33)	-	395 (56.6)	80.3 (13.1)	(6200)	255 (36.5)	- -	36.6 (5.31)
			Average	455 (65.8)	59.0 (8.56)	(7900)	401 (57.9)	77.2 (11.2)	(8700)	281 (46.8)	73.8 (10.7)	39.3 (5.80)
297 (75)	Air	$(0\pm45)_{2s}$	0	479 (69.4)	78.6 (11.4)	(8400)	458 (65.8)	63.8 (9.4)	(9100)	402 (57.5)		39.7 (5.75)
				430 (62.1)	74.6 (10.8)	(7200)	465 (67.2)	69.0 (10.0)	(8200)	325 (46.7)		38.4 (5.57)
				374 (53.8)	86.2 (12.5)	(6400)	472 (67.9)	69.0 (10.0)	(8400)	381 (55.2)		36.0 (5.22)
				514 (74.2)	66.3 (9.62)	(6400)	472 (67.6)	84.8 (12.3)	(8100)	297 (42.8)		46.0 (6.67)
				493 (71.0)	70.8 (10.27)	(6420)	514 (74.1)	76.5 (11.1)	(8200)	346 (49.7)		51.6) (7.49)
				465 (67.0)	69.3 (10.06)	(5560)	479 (69.4)	92.4 (13.4)	(8200)	339 (49.2)		42.9 (6.21)
			Average	459 (66.3)	73.8 (10.7)	(7100)	477 (68.7)	75.8 (11.0)	(8400)	348 (50.2)		42.4 (6.15)
589 (600)	Air	$(0\pm45)_{2s}$	0	367 (53.4)	77.8 (11.3)	(3620)	311 (45.2)	38.2 (5.54)	(11800)	304 (44.2)		27.4 (3.98)
				353 (51.4)	76.5 (11.1)	(3420)	262 (38.1)	48.8 (7.07)	(11000)	304 (43.5)		29.7 (4.31)
				- -	- -	-	248 (36.2)	35.7 (5.18)	10500)	276 (39.8)		28.9 (4.19)
				500 (72.3)	63.5 (9.21)	(7750)	339 (49.0)	36.0 (5.22)	10600)	318 (45.6)		28.9 (4.19)
				458 (66.3)	74.5 (10.8)	(9600)	283 (41.4)	36.1 (5.24)	(11100)	318 (45.8)		26.8 (3.89)
				479 (69.4)	66.0 (9.57)	(6650)	332 (48.1)	33.5 (4.86)	(11000)	283 (40.8)		27.7 (4.02)
			Average	432 (62.6)	71.7 (10.4)	(5400)	295 (43.0)	38.1 (5.52)	(11000)	301 (43.3)		28.2 (4.10)
297 (75)	GN_2	$(0\pm45)_{2s}$	0	451 (64.5)	63.2 (9.14)	(8300)	409 (59.4)	60.0 (8.7)	(8000)	437 (63.1)		31.2 (4.52)
				472 (68.0)	63.2 (9.18)	(8600)	423 (61.8)	70.4 (10.2)	(8140)	465 (66.5)		30.9 (4.48)
				423 (60.5)	63.2 (9.17)	(9870)	374 (53.7)	78.2 (11.3)	(7130)	318 (46.2)		32.3 (4.68)
				479 (69.2)	106.2 (15.4)	(9200)	542 (78.0)	70.4 (10.2)	(10200)	381 (55.4)		41.4 (6.00)
				535 (76.8)	86.2 (12.5)	(5700)	514 (73.4)	69.0 (10.0)	(8010)	444 (64.2)		39.7 (5.75)
				444 (63.6)	74.5 (10.8)	(6600)	507 (73.4)	70.3 (11.5)	(8220)	451 (64.5)		37.0 (5.37)
			Average	467 (67.1)	75.8 (11.0)	(6200)	462 (66.5)	71.1 10.3	(8300)	416 (60.0)		35.4 (5.13)

C-3

Table 3-65. Design Properties of HT-S/710 Composites - (0, ±45°, 90)

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi × 10⁶)	Strain to Failure (in./in. × 10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi × 10⁶)	Strain to Failure (in./in. × 10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi × 10⁶)	Short Beam Shear Strength MN/m² (ksi)
77 (-320)	Air	$(0, \pm45, 90)_{2s}$	0	346 (50.4)	33 (4.8)	(4200)	444 (63.9)	56.7 (8.22)	(8900)	353 (50.6)	29.4 (4.26)	25.5 (3.70)
				290 (42.0)	59 (8.5)	-	423 (61.4)	55.3 (8.02)	(9600)	367 (53.2)	27.9 (4.04)	30.5 (4.42)
				311 (45.0)	68 (9.9)	-	367 (53.2)	46.4 (6.73)	-	388 (56.3)	30.1 (4.36)	33.5 (4.86)
				304 (43.8)	66 (9.6)	-	325 (47.0)	34.9 (5.07)	(10500)	304 (44.1)	- -	20.8 (3.02)
				262 (38.4)	47 (6.8)	-	339 (49.2)	61.2 (8.88)	(9800)	339 (48.9)	- -	24.4 (3.54)
				290 (41.7)	43 (6.3)	-	360 (52.2)	56.6 (8.20)	(10400)	367 (52.6)	28.8 (4.18)	23.2 (3.36)
			Average	301 (43.6)	53 (7.7)	(4200)	376 (54.5)	51.8 (7.52)	(9800)	353 (51.0)	29.1 (4.22)	26.3 (3.82)
297 (75)	Air	$(0 \pm45, 90)_{2s}$	0	276 (39.6)	53 (7.7)	-	374 (53.7)	60.0 (8.70)	(6700)	458 (65.5)	25.2 (3.66)	23.2 (3.37)
				283 (40.9)	59 (8.5)	(3140)	353 (51.4)	60.2 (8.73)	(7600)	388 (55.7)	24.1 (3.50)	24.5 (3.55)
				297 (43.2)	59 (8.6)	(5200)	360 (51.8)	53.5 (7.76)	(7700)	367 (52.9)	24.8 (3.59)	24.7 (3.58)
				339 (48.9)	66 (9.6)	(5320)	304 (43.7)	59.2 (8.58)	(7900)	353 (50.9)	24.5 (3.55)	19.7 (2.86)
				311 (44.7)	50 (7.2)	(7660)	311 (45.1)	46.5 (6.74)	(8200)	353 (51.3)	24.0 (3.48)	19.7 (2.85)
				283 (41.4)	57 (8.2)	(4200)	283 (41.0)	48.8 (7.07)	(7800)	402 (58.4)	26.2 (3.80)	19.3 (2.80)
			Average	298 (43.1)	57 (8.3)	(5100)	331 (47.8)	54.7 (7.93)	(7700)	387 (55.8)	24.8 (3.60)	21.9 (3.17)
589 (600)	Air	$(0, \pm45, 90)_{2s}$	0	374 (53.5)	48 (7.0)	(6620)	283 (41.1)	24.6 (3.57)	(11600)	283 (41.0)	17.7 (2.56)	20.7 (3.00)
				325 (46.9)	46 (6.6)	(6600)	255 (37.2)	24.3 (3.53)	(9300)	227 (33.1)	16.1 (2.34)	21.7 (3.14)
				318 (46.3)	46 (6.6)	(5950)	234 (34.4)	24.8 (3.60)	(9700)	255 (37.0)	15.5 (2.25)	21.1 (3.06)
				325 (47.2)	42 (6.1)	(7320)	241 (35.0)	25.4 (3.68)	(10200)	255 (36.6)	17.6 (2.55)	23.9 (3.46)
				290 (42.3)	66 (9.5)	(5040)	262 (37.5)	22.9 (3.33)	(12100)	311 (44.5)	- -	22.3 (3.24)
				304 (43.9)	48 (6.9)	(5660)	255 (37.2)	21.2 (3.04)	(11600)	290 (42.4)	- -	21.6 (3.13)
			Average	323 (46.7)	49 (7.1)	(6200)	255 (37.1)	23.9 (3.46)	(10800)	270 (39.1)	16.8 (2.43)	21.9 (3.17)
297 (75)	GN_2	$(0, \pm45, 90)_{2s}$	0	332 (48.4)	70 (10.2)	(4400)	360 (51.9)	66 (9.6)	(7890)	318 (46.3)	32.4 (4.70)	22.3 (3.24)
				290 (42.1)	50 (7.2)	(4800)	395 (57.0)	55 (8.0)	(10000)	297 (43.1)	28.3 (4.10)	26.1 (3.78)
				339 (49.3)	74 (10.8)	(4800)	332 (48.0)	58 (8.4)	(8220)	290 (41.5)	26.9 (3.90)	24.6 (3.56)
				269 (39.2)	51 (7.4)	(5900)	332 (47.5)	54 (7.9)	(10000)	276 (39.8)	22.3 (3.23)	31.5 (4.57)
				283 (41.4)	66 (9.6)	(6100)	332 (47.9)	41.1 (6.0)	(11550)	234 (34.1)	23.4 (3.40)	37.7 (5.47)
				339 (49.3)	48 (6.9)	(4000)	381 (54.6)	43.3 (6.3)	(11100)	283 (41.0)	21.0 (3.05)	35.6 (5.16)
			Average	309 (45.0)	60 (8.7)	(5000)	355 (51.2)	53 (7.7)	(11300)	283 (41.0)	25.7 (3.73)	29.6 (4.30)

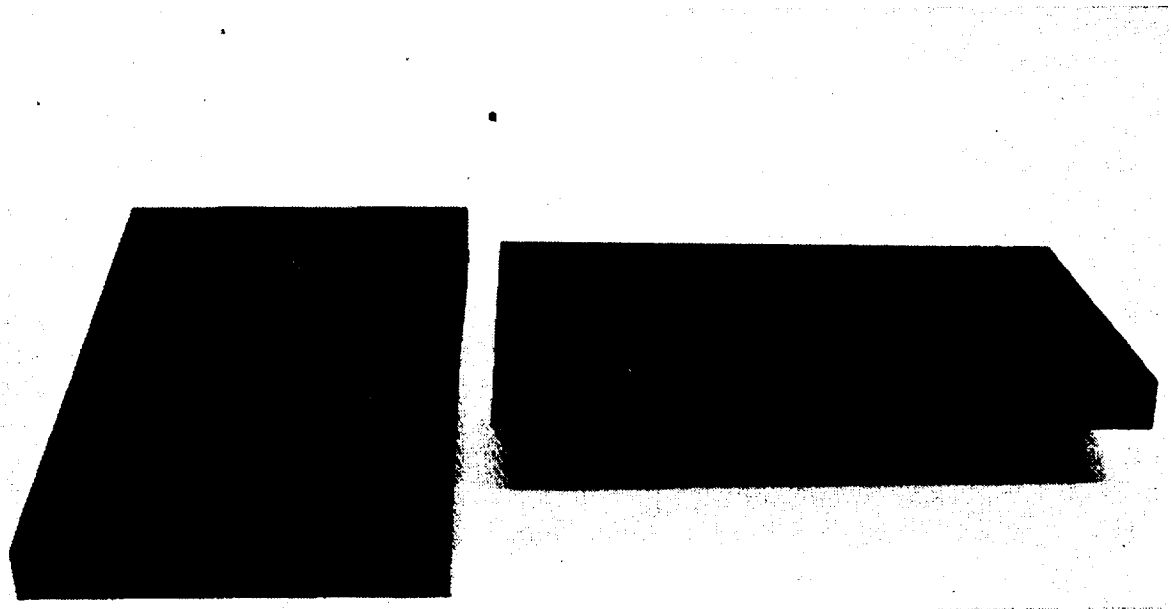
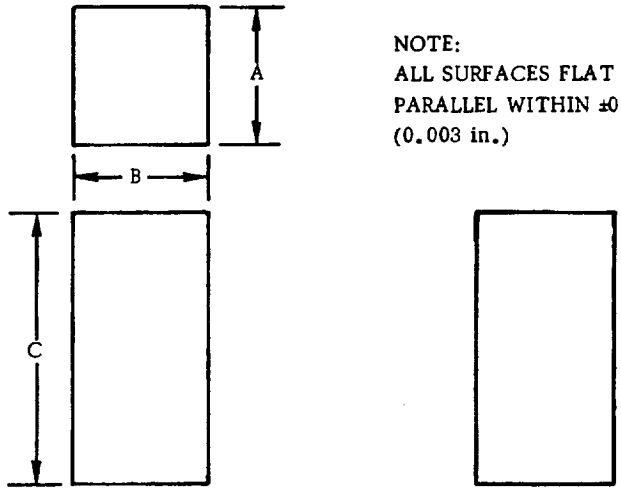


Figure 3-34. HT-S/710 Graphite Polyimide Specimen – 2.54 cm (1.00 in.) Laminate



LAMINATE THICKNESS cm	(in.)	A cm	(in.)	B cm	(in.)	C cm	(in.)
1.28	(0.50)	1.28	(0.50)1	1.28	(0.50)	2.54	(1.00)
2.54	(1.00)	1.92	(0.75)	1.92	(0.75)	2.54	(1.00)
3.81	(1.50)	3.81	(1.50)	3.81	(1.50)	5.08	(2.00)

Figure 3-35. HT-S/710 Graphite/Polyimide - Thick Laminate Test Specimen

Longitudinal and transverse specimens were machined from nominal 1.27 cm (0.50 in.), 2.54 cm (1.0 in.), and 3.81 cm (1.50 in.) laminates. These thicknesses represent 100-, 200-, and 300-ply laminates. The test results obtained are presented in Table 3-66 and compared to thin laminate data in Figure 3-36. Longitudinal and transverse modulus values were relatively unaffected by the increase in thickness. The transverse strength showed only a 10% decrease as the laminate thickness approached 3.81 cm (1.50 in.). The longitudinal strength did show decreases in strength of 20, 35, and 41% as compared to compression strengths generated on 0.25 cm (0.10 in.) laminates. This decrease in strength was probably caused by the inner plies not remaining perfectly straight, slight errors in machining, and perhaps insufficient curing of the inner plies during the initial cure. The latter two reasons are unlikely, because the transverse strength would also have shown a significant decrease. The straightness of the plies would have no major effect on the transverse strength, since this is primarily resin-dependent. By examining a cross section of the laminate, it was determined that the plies did not remain perfectly straight and this probably caused the decrease in longitudinal strength. Further process development work is required in this area.

3.7.3 GRAPHITE/POLYIMIDE BIAXIAL TESTING. One of the many tests conducted during the design property determination portion of the program was for biaxial strain. To conduct these tests, high-quality tubes of the HT-S/710 system had to be made. The prepreg material was precompacted flat before being rolled onto an aluminum mandrel by Convair's special tube rolling machine. The tube was rolled, then cured, using the autoclave cure cycle discussed in this report. However, the hold at 400° K

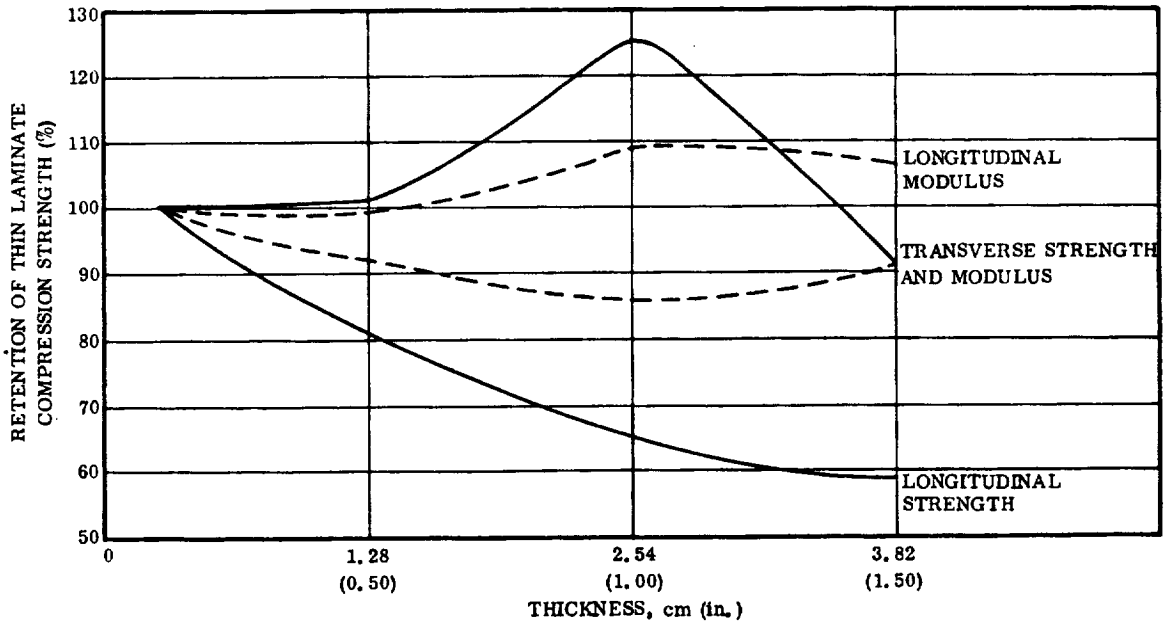


Figure 3-36. HT-S/710 Graphite/Polyimide – Thick Laminate Data

Table 3-66. 297°K (75°F) Thick Laminate Compression Test Results of HT-S/710 Graphite/Polyimide Composites

Laminate Thickness cm (in.)	Longitudinal Strength MN/m^2	(ksi)	Longitudinal Modulus GN/m^2	($psi \times 10^6$)	Transverse Strength MN/m^2	(ksi)	Transverse Modulus GN/m^2	($psi \times 10^6$)
1.27 (0.5)	556	(79.8)	121	(17.5)	70.3	(10.2)	6.82	(0.98)
	577	(83.1)	142	(20.6)	71.7	(10.4)	7.58	(1.10)
	592	(84.7)	143	(20.8)	115.8	(16.8)	8.96	(1.30)
Average	575	(82.5)	135	(19.6)	85.9	(12.5)	7.79	(1.13)
2.54 (1.0)	472	(67.5)	116	(16.9)	91.7	(13.3)	6.13	(0.88)
	451	(65.1)	163	(23.6)	110.3	(16.0)	7.79	(1.13)
	472	(67.7)	163	(23.6)	124.8	(18.1)	7.86	(1.14)
Average	465	(66.8)	147	(21.4)	108.9	(15.8)	7.26	(1.05)
3.81 (1.5)	416	(59.6)	-	-	71.0	(10.3)	6.89	(0.99)
	437	(63.1)	142	(20.6)	77.9	(11.3)	7.45	(1.08)
	409	(58.7)	147	(21.4)	86.2	(12.5)	8.62	(1.25)
Average	421	(60.5)	144	(21.0)	78.4	(11.3)	7.65	(1.11)

(260°F) was only 10 minutes, rather than the normal 25 minutes, before the pressure was applied. The tubes were postcured unrestrained in air to 644°K (700°F). Tubes of 5.08 cm (2 in.) diameter, 50.8 cm (20 in.) long were made and later machined into three test specimens. Aluminum doublers and caps were bonded with a room-temperature adhesive. Figure 3-37 shows the test specimen configuration for the

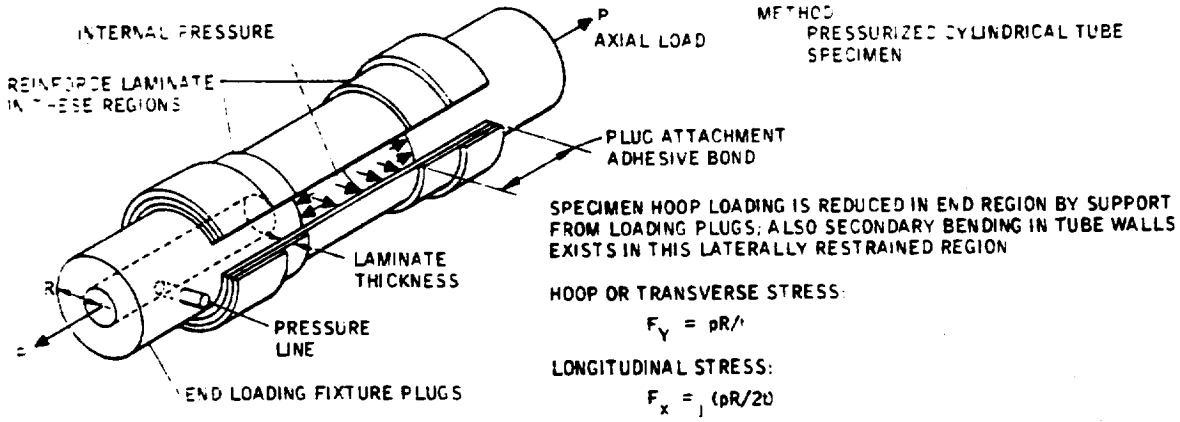


Figure 3-37. Biaxial Tension Specimen

biaxial tension tests. A water-oil mixture was used as the internal pressurizing medium. This fluid was kept inside the tube by a thin RTV bladder on the inside of the tube. Two sets of rosette strain gages were placed on each of the test specimens.

The measured test results are reported in Table 3-67 and graphically reproduced in Figure 3-38. The failed test specimens are shown in Figure 3-39. By the use of fingered doublers, the stress concentrations caused by the doublers were greatly reduced. Biaxial strain testing of composite tubes has some inherent difficulties that

Table 3-67. Biaxial Tube Testing of [±45] HT-S/710 Graphite/ Polyimide Composites at 297° K (75° F)

Spec. No.	Applied Stress Axial	Hoop	Measured Strain Axially μcm/μcm (μin./μin.)	Hoop μcm/μcm (μin./μin.)	Maximum Stress MN/m^2	(ksi)
10	1	0	14,000	-12,200	141	(20.5)
7	1	1	4,000	3,050	297	(43.2)
11	1	1	5,000	2,800	325	(46.8)
8	2	1	15,500	-11,600	297	(43.2)
12	1	2	-10,100	13,700	304	(44.4)

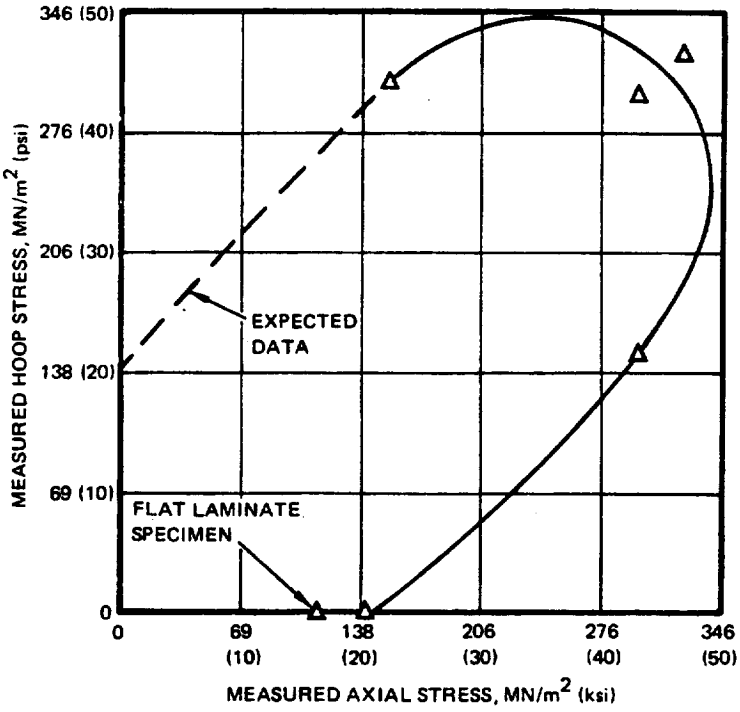


Figure 3-38. Biaxial Test Results of (±45°) HT-S/710 Composite Tubes

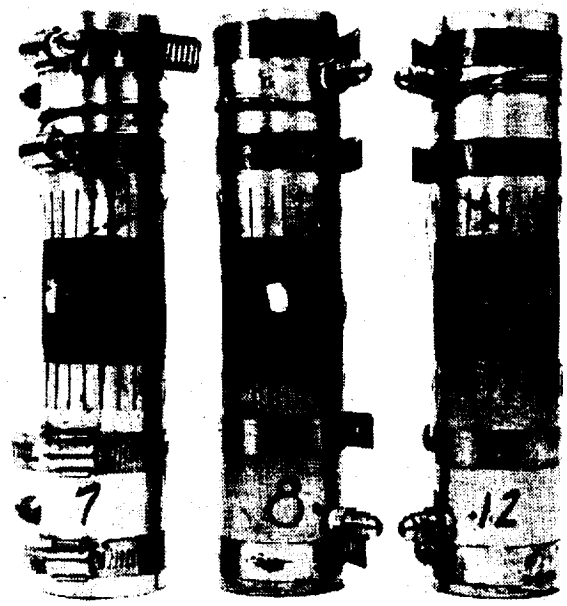


Figure 3-39. Tested Biaxial Graphite/Polyimide HT-S/710 Tubes

cause errors which would mask the test results. Some of the potential sources of error in order of decreasing probable significance are:

a. Doubler stress concentrations.

b. Misalignment of the fibers (i.e., a ±45° (±α) laminate rather than a true ±45° laminate).

c. Error in applying the rosette strain gage exactly in the longitudinal and transverse directions. Errors on the order of 1 to 2° seem quite possible.

d. Changes in nominal thickness of the material. (Normally ±2 mils).

The theoretical principal stresses and strains were calculated using a computer program (SQ-5) that translates measured data into principal strains by use of a stiffness matrix. After obtaining the principal strains, the principal stresses are calculated. An elastic modulus of 12.4 GN/m^2 (18.3×10^6 psi) and a shear modulus of 3.1 GN/m^2 (0.45×10^6 psi) were used in calculating the strains rather than the initial modulus values reported earlier in this section of the report. These moduli values are the secant moduli for the HT-S/710 composite system. The calculated theoretical principal stresses and strains are tabulated in Table 3-68. These results

Table 3-68. Theoretical Principal Stress and Strain Test Results of Biaxial Tube Testing of (±45°) HT-S/710 Composites

Specimen No.	σ_1 MN/m^2	(ksi)	σ_2 MN/m^2	(ksi)	τ_{12} MN/m^2	(ksi)	$\epsilon_1 = \epsilon_2$	γ_{12}
10	124	(18.2)	11	(1.6)	69	(9.9)	969	21930
7	528	(76.4)	46	(6.7)	0	(0)	4078	0
11	577	(82.8)	50	(7.2)	0	(0)	4417	0
8	395	(57.3)	35	(5.0)	72	(10.4)	3058	23080
12	409	(58.9)	36	(5.1)	74	(10.7)	3143	23720

clearly show that there was a significant stress concentration present in all the test specimens. None of the test specimens obtained the ultimate stress or strain values achieved for flat laminate specimens. The stress concentration was caused either by improper fabrication of the tube, end doublers, or specimen dimensions. Tube number 10 was tested in uniaxial tension and compares in strength quite well with flat laminate test data. However, the strain values are approximately 1/10 of those obtained for the flat laminate specimens. In this case, the doublers were restraining the fibers and acting as the stress concentration. In the cases of the other four tubes, a major stress concentration is present (low strengths), but it is not clear as to the cause. The test results reported in this section should be considered extremely conservative. More work is required to develop a specimen configuration and testing procedure for biaxial tube testing.

3.7.4 CREEP TESTING OF HT-S/710 COMPOSITES. Creep tests were conducted on (0°) and (±45°) HT-S/710 graphite/polyimide composites at 589° K (600° F) for periods up to 200 hours. Both the (0°) and (±45°) laminates were tested in the 0° fiber direction. The standard tensile specimen was used for the creep testing, and the creep and static tensile specimens were machined from the same laminate. Optical extensometers were used to determine the strain rates.

The unidirectional test specimens showed essentially no creep, as expected. The test program for the unidirectional specimens was then modified more toward a stress-rupture program. The test results obtained are tabulated in Table 3-69. The upper applied stress level is equivalent to 69% of the average static strength. Any specimens that did not rupture during the test were then statically tested at room temperature. The average residual tensile strength of the higher stress level specimens was approximately 70% of the static strength. This reduction in strength could have been caused by specimens damaged during the initial creep testing, oxidation of the matrix, or creep. By conducting unloaded aging tests at 589° K (600° F), it was

Table 3-69. Stress Rupture and Residual Tensile Strength of Unidirectional HT-S/710 Composites

Specimen No.	Test Temp °K	(°F)	Stress Level MN/m^2	(ksi)	Test Time (Hrs)	Test Results	297° K (75° F) Residual Tensile Strength MN/m^2	(ksi)
U-1	589	(600)	675	(97)	207	No Failure	938	(136)
-2	589	(600)	675	(97)	65	Rupture	-	-
-3	589	(600)	627	(90)	204	No Failure	1158	(168)
-4	589	(600)	675	(97)	202	No Failure	772	(112)
-5	589	(600)	675	(97)	232	No Failure	675	(97)
-7	589	(600)	627	(90)	200	No Failure	1034	(150)
-8	589	(600)	627	(90)	203	No Failure	758	(110)
-10	589	(600)	758	(110)	163	Rupture	-	-
-11	589	(600)	758	(110)	0.4	Rupture	-	-
-12	589	(600)	724	(105)	209	No Failure	821	(119)
-13	589	(600)	724	(105)	201	No Failure	821	(119)
-14	589	(600)	758	(110)	217	No Failure	779	(113)
-15	589	(600)	724	(105)	212	No Failure	979	(141)

determined that the matrix does not oxidize at this temperature for this length of time. The residual strength test results for the (±45°) composites showed no dropoff in strength due to the creep testing. From post test analysis of the test setup and specimen failure surface, it is believed that the specimens were damaged during loading into the creep machine. A slight twisting of a (0°) specimen can severely damage the specimen.

The stress rupture, residual tensile strength, and creep test results for the (±45°) composites are presented in Tables 3-70 and 3-71. The secondary creep rate, K, was determined for four different stress levels. The residual tensile strength of the specimens that did not fail was approximately 10% higher than the static ultimate strength.

The tensile results were also very consistent compared with results obtained for the (0°) composites. A (±45°) specimen would not be as easily damaged by twisting as a unidirectional test specimen. If the direction of loading has fibers in that direction, there will be essentially no creep unless the temperature is such that the matrix begins to oxidize.

Table 3-70. Stress Rupture and Residual Tensile Strength of $(\pm 45°)_s$ HT-S/710 Composites

Specimen No.	Test Temp °K	(°F)	Stress Level MN/m^2	(ksi)	Test Time (Hrs)	Test Results	297°K (75°F) Residual Tensile Strength MN/m^2	(ksi)
45-1	589	(600)	56	(8.0)	74	Rupture	-	-
-2	589	(600)	56	(8.0)	10	Rupture	-	-
-3	589	(600)	35	(5.0)	209	No Failure	112	(16.2)
-4	589	(600)	35	(5.0)	202	No Failure	118	(17.1)
-5	589	(600)	20	(3.0)	201	No Failure	119	(17.3)
-6	589	(600)	22	(3.2)	200	No Failure	123	(17.8)
-7	589	(600)	21	(3.1)	202	No Failure	127	(18.4)
-10	589	(600)	42	(6.0)	228	No Failure	119	(17.3)
-11	589	(600)	42	(6.0)	214	No Failure	123	(17.9)
-14	589	(600)	28	(4.0)	209	No Failure	118	(17.1)
-15	589	(600)	42	(6.0)	209	No Failure	120	(17.4)
-16	589	(600)	35	(5.0)	201	No Failure	132	(19.2)
-17	589	(600)	28	(4.0)	200	No Failure	123	(17.8)
-18	589	(600)	28	(4.0)	202	No Failure	119	(17.2)

Table 3-71. Average Creep Test Results of $(\pm 45°)$ HT-S/710 Composites

Applied Stress at 589°K (600°F) MN/m^2	(ksi)	% Ultimate	Maximum Strain cm/cm (in/in)	Secondary Creep Strain Rate, k cm/cm/hr (in/in/hr)
21	(3.0)	28	0.0087	0.65×10^{-6}
28	(4.0)	37	0.0083	0.70×10^{-6}
35	(5.0)	46	0.0065	0.70×10^{-6}
42	(6.0)	56	0.0126	1.35×10^{-6}
56	(8.0)	74	Rupture	-

3.7.5 AMBIENT AND HIGH TEMPERATURE AGING. Ambient and high temperature 589° K (600° F) aging exposures were conducted on the HT-S/710 graphite/polyimide composites. Two laminates were fabricated, vacuum bag and autoclave cured, and machined into flexure and shear specimens. Two-hour and 24-hour H_2O boil tests, 3 week and 6 week humidity, and 20 week laboratory exposures were conducted. None of the exposures showed any significant effect on the properties of the composite at any of the test temperatures. The test results are presented in Table 3-72. In fact, at the end of the 20-week laboratory exposure, the flexural strength at all test temperatures had increased significantly. The short beam shear strength remained approximately the same after all of the exposures.

Longitudinal flexure and tensile and transverse flexure and tensile specimens were machined from two of the static design property laminates for 589° K (600° F) heat aging. The specimens were aged as test specimens in an air-circulating oven for periods up to 400 hours. The test data is shown in Table 3-73. No significant changes in strength were found for the HT-S/710 composite system as a function of heat aging up through 400 hours. In fact, there is so little change that a much more refined design allowable for the tensile and flexural strength of this system can be determined by using all the data regardless of aging times. The HT-S/710 graphite/polyimide composite system is stable at 589° K (600° F) for periods up to 400 hours, even with 5% void present in the laminate.

3.7.6 THERMAL EXPANSION TESTING OF HT-S/710 COMPOSITES. Nine thermal expansion specimens were tested over the temperature range of 77° K to 589° K (-320F to 600F). Three specimens had their test axis parallel to the 0° direction of the laminate, three specimens were at 45° to this, and three specimens were at 90°.

Measurements were made using the modified Leitz dilatometer shown in Figures 3-40 and 3-41. In this apparatus, the specimen is contacted at each end by and supported by concentric fused silica tubes. The relative length is indicated by relative positions of the tubes. Movements of one with respect to the other causes movement of a prism, which results in vertical deflection of a light beam projected on a ground-glass plate. Specimen length changes are magnified by a factor as large as 800. Horizontal deflection of the light beam is controlled by a thermocouple mounted on the specimen.

The specimen is maintained in a dry helium atmosphere. Temperature of the specimen is varied by varying the power setting of the furnace. The furnace used for low temperatures is shown in detail in Figure 3-41. This operates in a LN_2 environment, and has a range of 77° K to 477° K (-320F to 400F). When necessary to go above 477° K (400F), the furnace shown is replaced with an ambient to 1310° K (1900F) furnace when ambient is crossed.

Power changes necessary to provide the desired temperature change are made, and the specimen is permitted to reach thermal and structural equilibrium. In this investigation,

Table 3-72. Graphite Composite Aging Data (HT-S/710)

LAM. NO.	TYPE OF TEST	TEST TEMPERATURE K	(F)	CONTROL MN/m^2	(ksi)	2-HOUR H_2O BOIL MN/m^2	(ksi)	24 HOUR H_2O BOIL MN/m^2	(ksi)	3 WEEK HUMIDITY MN/m^2	(ksi)	6 WEEK HUMIDITY MN/m^2	(ksi)	2 WEEK AGING MN/m^2	(ksi)	5 WEEK AGING MN/m^2	(ksi)	10 WEEK AGING MN/m^2	(ksi)	20 WEEK AGING MN/m^2	(ksi)
139	Flex	77	(-320)	1,214	(175.8)	–	–	–	–	–	–	–	–	1,186	(171.9)	1,317	(190.5)	1,145	(165.7)	1,310	(190.4)
		297	(75)	1,324	(191.5)	1,193	(173.4)	1,110	(161.4)	1,117	(161.9)	1,262	(183.4)	1,303	(188.8)	1,489	(215.6)	1,434	(208.5)	1,510	(218.9)
		406	(275)													1,476	(214.2)				
		450	(350)	1,303	(189.2)									1,069	(154.5)			1,331	(193.3)	1,613	(233.5)
		505	(450)	1,303	(188.8)					1,000	(145.2)	1,083	(156.7)			1,420	(205.6)				
		589	(600)	675	(97.9)	690	(100.2)	487	(70.6)	590	(85.9)	1,098	(159.1)	565	(82.1)			738	(106.6)	1,207	(175.0)
	Shear	77	(-320)	98.6	(14.3)	–	–	–	–	–	–	–	–	76.7	(10.4)	74.5	(10.8)	87.6	(12.7)	91.7	(13.33)
		297	(75)	84.1	(12.2)	47	(6.8)	49	(7.1)	50	(7.2)	51	(7.4)	65	(9.4)	64	(9.3)	79.3	(11.5)	84.8	(12.32)
		450	(350)	57	(8.3)	–	–	–	–	–	–	–	–	57	(8.3)	56	(8.1)	69.0	(10.0)	75.2	(10.87)
		589	(600)	40	(5.8)	41	(5.9)	37	(5.4)	35	(5.1)	65	(9.2)	40	(5.8)	46	(6.7)	48	(7.0)	46	(6.62)
153	Flex	77	(-320)	1,062	(153.7)	–	–	–	–	–	–	–	–	–	–	–	–	1,103	(160.1)	1,289	(186.7)
		297	(75)	1,303	(188.8)	1,234	(177.8)	1,193	(173.2)	1,372	(199.4)	1,151	(167.0)	–	–	1,276	(185.0)	1,296	(188.1)	1,489	(216.1)
		406	(275)	1,303	(188.8)	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
		450	(350)	1,207	(175.0)	1,280	(185.7)	1,160	(168.0)									1,207	(175.0)	1,427	(206.7)
		505	(450)	1,179	(171.0)	1,155	(167.2)	1,155	(167.0)	1,158	(167.9)	1,020	(148.4)	–	–	1,165	(169.0)				
		589	(600)	889	(129.4)	9.24	(134.3)	800	(116.0)	840	(122.1)	1,159	(167.7)					827	(120.4)	1,117	(161.6)
	Shear	77	(-320)	82.7	(12.0)	–	–	–	–	–	–	–	–	–	–	–	–	66	(9.6)	77.2	(11.17)
		297	(75)	40	(5.8)	55	(8.0)	43	(6.2)	52	(7.5)	50	(7.3)	–	–	64	(9.2)	65	(9.5)	73.1	(10.58)
		450	(350)	55	(8.0)	–	–	–	–	–	–	–	–	–	–	–	–	54	(7.8)	57.2	(8.31)
		589	(600)	39	(5.7)	39	(5.7)	39	(5.6)	40	(5.8)	40	(5.8)	–	–	37	(5.3)	41	(6.0)	43.5	(6.30)

	LAM 139	LAM 153
Fiber Volume	65.3	63.3
Resin Content	28.5	29.7
Specific Gravity	1.52	1.52

Table 3-73. Graphite/Polyimide 589° K (600° F) Heat-Aging Test Results

Hours at 589° K (600° F)	Test Temperature °K	(°F)	Long. Flexure MN/m²	(ksi)	Trans. Flexure MN/m²	(ksi)	Long. Tensile MN/m²	(ksi)	Trans. Tensile MN/m²	(ksi)
0	297	(75)	1448	(210.0)	11.4	(1.65)	1172	(169.6)	12.3	(1.78)
			1386	(200.8)	10.4	(1.51)	1186	(171.8)	12.8	(1.85)
			1503	(217.8)	12.2	(1.77)	1062	(154.2)	13.3	(1.93)
			1393	(201.8)	13.9	(1.97)	1145	(166.4)	17.9	(2.59)
			1469	(212.8)	14.5	(2.10)	1124	(162.6)	20.3	(2.95)
			1593	(230.6)	13.5	(1.96)	1048	(152.2)	19.3	(2.80)
0	Ave		1465	(212.3)	12.6	(1.83)	1123	(162.8)	16.0	(2.32)
	589	(600)	986	(142.7)	13.8	(2.00)	1131	(164.3)	11.1	(1.61)
			945	(136.9)	15.2	(2.20)	1131	(164.2)	10.8	(1.56)
			896	(129.5)	15.5	(2.25)	1096	(158.9)	12.5	(1.82)
			993	(144.4)	14.6	(2.12)	1138	(165.4)	12.7	(1.84)
			827	(120.5)	14.7	(2.13)	1076	(156.4)	10.3	(1.50)
			938	(136.2)	16.1	(2.33)	1117	(162.0)	13.7	(1.99)
	Ave		931	(135.0)	15.0	(2.17)	1115	(161.9)	11.8	(1.72)
100	297	(75)	1503	(217.7)	23.4	(3.40)	1124	(163.2)	22.1	(3.21)
			1662	(240.9)	22.1	(3.20)	1220	(177.4)	19.7	(2.86)
			1345	(195.1)	24.1	(3.50)	1069	(155.2)	18.5	(2.68)
			1379	(199.5)	16.3	(2.37)	1089	(157.7)	23.9	(3.46)
			1613	(234.5)	21.2	(3.07)	1089	(158.1)	21.0	(3.05)
			-	-	24.6	(3.56)	1241	(179.5)	25.4	(3.69)
	Ave		1503	(217.5)	21.9	(3.18)	1138	(165.2)	21.8	(3.16)
	589	(600)	1103	(159.5)	16.0	(2.32)	1062	(154.5)	13.7	(1.99)
			1048	(152.1)	15.2	(2.21)	1241	(179.5)	12.7	(1.84)
			1062	(153.5)	13.0	(1.89)	1131	(164.2)	12.6	(1.82)
			993	(143.5)	15.6	(2.26)	1096	(158.9)	-	-
			979	(141.8)	12.6	(1.82)	1117	(162.0)	-	-
			993	(143.6)	23.4	(3.39)	1172	(170.3)	-	-
	Ave		1027	(148.9)	15.9	(2.31)	1138	(164.9)	13.0	(1.88)
200	297	(75)	1413	(205.3)	9.1	(1.32)	1158	(168.4)	7.9	(1.15)
			1462	(212.0)	9.6	(1.40)	958	(138.9)	15.0	(2.18)
			1407	(203.7)	10.4	(1.51)	1014	(146.9)	14.5	(2.10)
			1310	(190.3)	10.1	(1.46)	1172	(170.2)	21.0	(3.04)
			1413	(204.8)	8.6	(1.24)	1020	(148.5)	10.7	(1.55)
			1455	(210.6)	9.6	(1.40)	-	-	-	-
	Ave		1407	(204.4)	9.6	(1.39)	1069	(154.6)	13.8	(2.00)
	589	(600)	903	(130.9)	13.3	(1.93)	1007	(145.5)	13.1	(1.90)
			1055	(153.0)	15.4	(2.24)	1089	(158.0)	7.1	(1.03)
			1041	(151.3)	16.1	(2.33)	1055	(153.0)	11.1	(1.61)
			1193	(172.9)	16.8	(2.43)	1055	(152.7)	9.0	(1.31)
			1007	(145.6)	13.2	(1.91)	1089	(158.0)	-	-
			1055	(153.1)	14.2	(2.06)	-	-	-	-
	Ave		1041	(151.1)	14.8	(2.15)	1055	(153.4)	10.1	(1.46)
400	297	(75)	1551	(225.0)	12.5	(1.82)	1172	(170.0)	12.6	(1.82)
			1379	(199.9)	10.8	(1.57)	1020	(148.5)	8.3	(1.21)
			1655	(239.8)	10.4	(1.51)	1110	(161.4)	-	-
			1386	(200.6)	11.2	(1.62)	1296	(187.8)	-	-
			1503	(217.9)	14.5	(2.10)	1186	(171.6)	-	-
			1365	(197.9)	13.0	(1.89)	1179	(171.3)	-	-
	Ave		1476	(213.5)	12.1	(1.75)	1158	(168.4)	10.5	(1.52)
	589	(600)	862	(125.0)	9.9	(1.43)	1117	(161.9)		
			786	(114.3)	12.6	(1.83)	1055	(152.8)		
			876	(126.6)	11.3	(1.64)	1103	(160.4)		
			814	(118.5)	11.4	(1.66)	1000	(145.1)		
			883	(128.1)	10.4	(1.51)	938	(136.2)		
			800	(115.8)	-	-	993	(143.9)		
	Ave		834	(121.4)	11.1	(1.61)	1034	(150.1)		

	Flexure Laminate	Tensile Laminate
Fiber Vol. %	62.7	67.5
Resin Cont. %	32.2	27.8
Specific Gravity	1.53	1.50

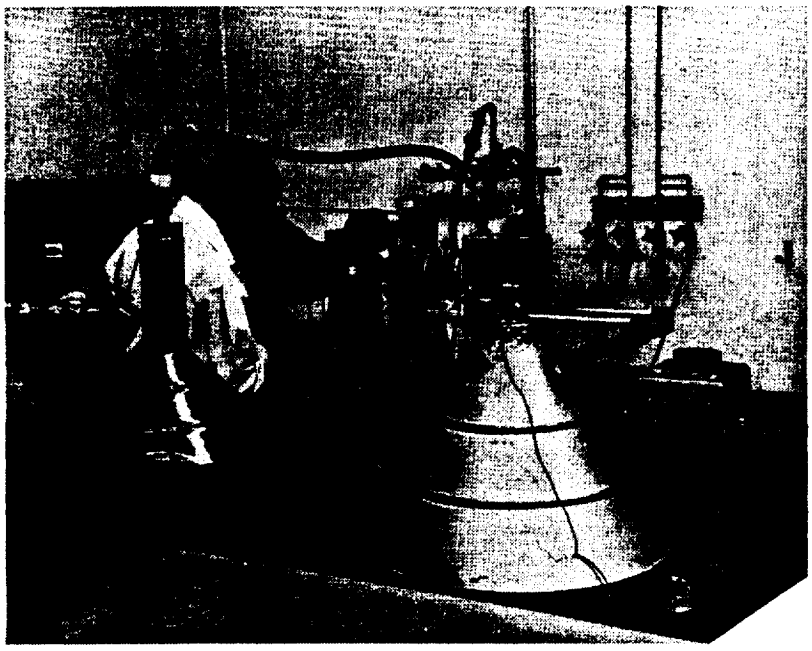


Figure 3-40. Modified Leitz Dilatometer

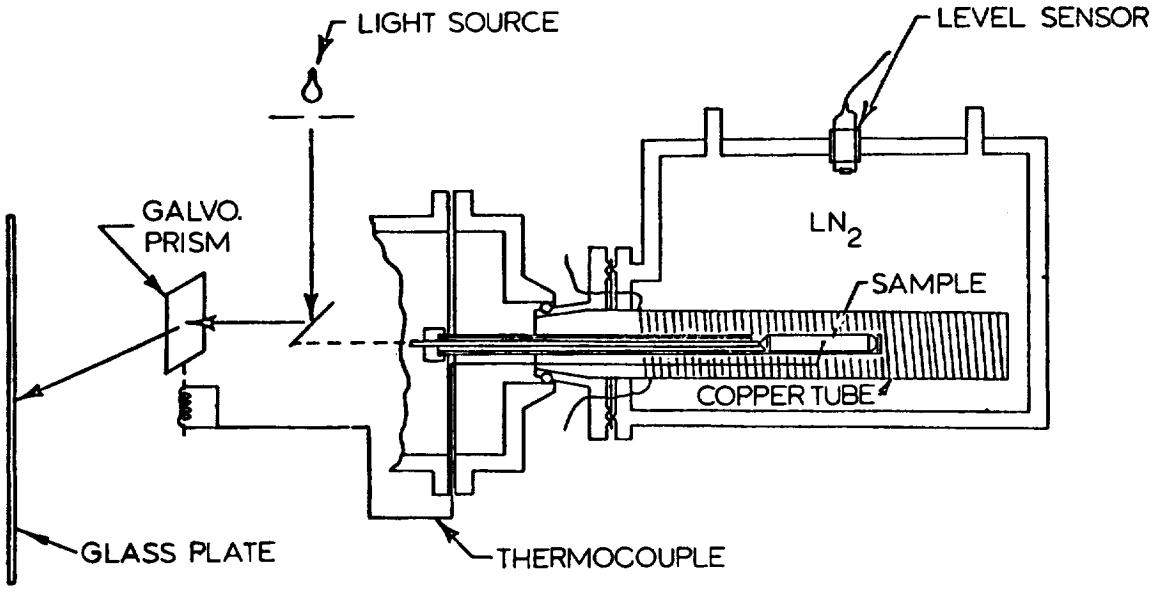


Figure 3-41. Details of Dilatometer Mechanism

changes of approximately 41° K (75F) were made in approximately 10 to 20 minutes. When equilibrium is reached, vertical displacement of the light beam and specimen temperature are recorded, and the power is set for the next desired temperature. Displacement is hand marked on the glass plate.

At the conclusion of a run, the specimen displacement data is read using a traveling telescope, converted to $\Delta L/L$ with respect to the original reference length at ambient temperature, and plotted as a function of temperature.

The apparatus is calibrated routinely in two steps. The magnification factor is first computed by deflecting the system with a precision micrometer. Errors due to temperature gradients within the dilatometer are then determined by analyzing the results of measurements made on a standard quartz specimen calibrated by NBS.

For maximum accuracy, the apparatus requires a two-hour stabilization period after the LN_2 environment is established in the low temperature furnace. Considerable technique in recording and reading raw data is also required to maintain high accuracy. Under the best conditions, an uncertainty of less than $\pm .07 \times 10^{-4}$ $\Delta L/L$ can be maintained. Approximately one-half of this figure is due to uncertainties in the data for the quartz standard.

Because of the large temperature range of interest in this investigation, it was necessary to obtain the data during two independent runs. The first, using the low temperature furnace, covered the range 77 to 450° K (-320 to 350° F). The second overlapped the first by 153° K (275° F), starting at 297° K (75° F) and ending at 589° K (600° F).

As organic matrix composites are thermally cycled, they generally undergo changes in length not directly related to thermal expansion. These can be attributed to such things as moisture and volatile losses, structural changes, and stress relieving. Since strains of these origins can be comparable to, or greater than, the thermal expansion, the test sequences must be chosen to differentiate between them. For this reason, the low temperature range - where the smallest changes would be expected - was evaluated first. Following specimen installation, the system was cooled to 77° K (-320° F) and held for the required stabilization period. Specimen length was then recorded and the temperature increased to 450° K (350° F). This temperature was held until no changes were seen (minimum of 1/2 hour). Data points were then taken at regular intervals from 450 to 77° K (350 to -320° F).

The specimen was then returned to 297° K (75° F) and removed for storage in a chemical dessicator prior to the high temperature run. Points were taken from 297 to 589° K (75 to 600° F). Following the point at 589° K (600° F), the specimen was slowly cooled back to 297° K (75° F). Several points were taken on the way down to evaluate changes due to the 589° K (600° F) exposure.

As suspected, the strain data was rather complex. Although the same general effects were seen in the 0°, 45°, and 90° runs, they were much more obvious in the 0° direction. Results for one of the 0° specimens will be used as an example.

Figure 3-42 shows the complete results for 0° specimen number L0947, run numbers N0848 and N0868. Following stabilization at 77° K (-320° F), the length was recorded and the temperature raised to 450° K (350° F). The first point was recorded when the temperature had stabilized. Approximately 1 hour later, the 5.08 cm (2 in.) specimen had lost approximately 1.01×10^{-4} cm ($.4 \times 10^{-4}$ in.) of length, and had shown no further change for 1/2 hour. As data was taken on the return cycle to 77° K (-320° F), a "J"-shaped curve was formed. The length at 77° K (-320° F) was somewhat greater than $.3 \times 10^{-4}$ in./in. less than originally, indicating that the thermal cycle and/or the exposure to elevated temperatures had caused a permanent change in the material.

At this point, the low-temperature run was terminated and the specimen was warmed, removed, and stored in a chemical dessicator. Fourteen days later, it was installed in the high-temperature furnace and data was taken from 297° K (75° F) to 589° K (600° F). Between 297° K (75° F) and 450° K (350° F), the slope of the curve was not the same as in the low temperature run, indicating the presence of a change in the material. (Since the length measurements made are relative rather than absolute, it was assumed that the reference length at 297° K (75° F) was the same as that at the completion of the previous run. A change would result in a vertical displacement of each point on the graph, but would not affect the slope of the curve.)

At about 505° K (450° F), the curve reversed slope to 589° K (600° F). There was no hold at 589° K (600° F) other than that required to stabilize the temperature. On the return to 297° K (75° F), the slope was different and the final length was $.8 \times 10^{-4}$ in./in. less at 297° K (75° F).

It is obvious from the data presented that the material underwent several changes during the thermal cycling it experienced, and that the length change over a particular temperature range depends on previous history. In an attempt to determine the cause of the instability in the material, several reruns were made on the specimen.

On the second run, there was little change to 450° K (350° F) in the low-temperature run. During the high-temperature portion, the length to 450° K (350° F) was generally reproduced. Between 422° K (300° F) and 477° K (400° F) the length abruptly changed approximately -1.0×10^{-4} in./in. Additional length was lost between 477° K (400° F) and 589° K (600° F), resulting in a net change in length on return to ambient of approximately -1.3×10^{-4} in./in. This was a significant change, but the results to 450° K (350° F) indicated that the material was considerably more stable than during the first run.

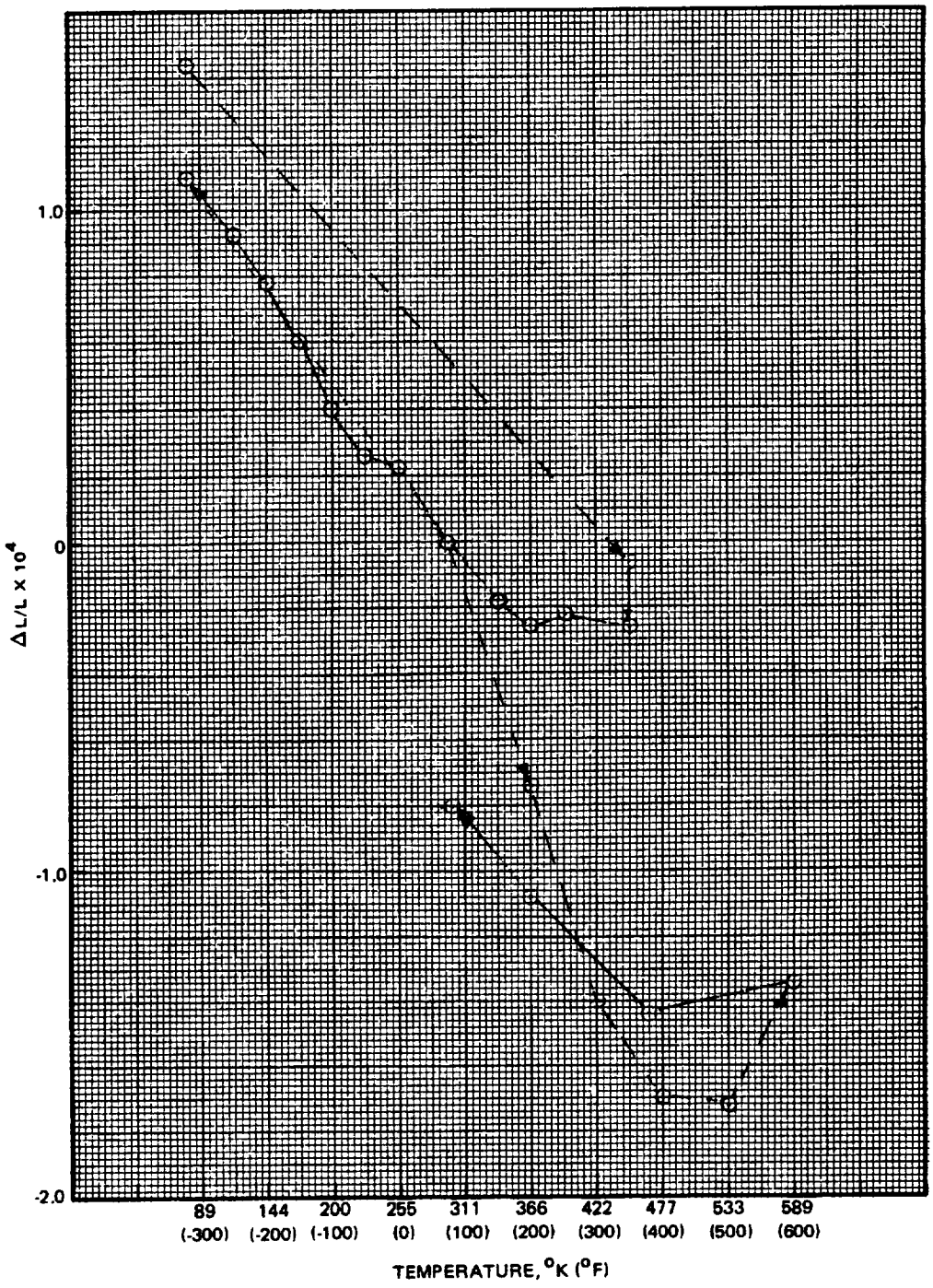


Figure 3-42. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen

Several days later, after dessicator storage, the specimen was rerun from ambient to 589° K (600° F). There was no permanent length change. These results, presented in Figure 3-43, were repeated the next day.

Review of weight measurements made on the specimen indicated no significant changes from run-to-run. The length changes could not, therefore, be attributed to loss of moisture or volatiles. The material is susceptible to a structural change when oxidized at high temperatures. However, changes occurred although the specimen was in a high purity helium atmosphere.

Elimination of these possibilities suggest the changes are attributable to incomplete curing of the material, which was eventually taken to completion through exposure to elevated temperatures during the expansion runs. The incomplete postcure does not have any effect on the structural properties of the composite, but definitely changes the length of the specimen. These small differences in length could only be determined by running a test such as thermal expansion.

Since the expansion data acquired on eight of the nine specimens in this program is not representative of the stabilized material, the data was somewhat rearranged to construct the presentations in Figures 3-44 through 3-51. These were constructed from the "down" halves of the low- and high-temperature runs, with the final 297° K (75° F) point of the high temperature run shifted to correspond with the 297° K (75° F) point of the low-temperature run. (Figure 3-43, Specimen L0947, was constructed from the last data obtained.) It is felt that the slopes of the curves thus obtained are the best approximations of those for the stable material.

Averaging all the test data, it was determined that the average thermal expansion coefficients for the HT-S/710 graphite/polyimide composites from 77 to 589° K (-320 to 600° F) was -0.31×10^{-6} cm/cm/° K (-0.17×10^{-6} in./in./° F) for 0°, 8.12×10^{-6} cm/cm/° K (4.51×10^{-6} in./in./° F) for a 45° test direction, and 17×10^{-6} cm/cm/° K (9.45×10^{-6} in./in./° F) for a 90° test direction. Test directions are all denoted relative to the fiber direction.

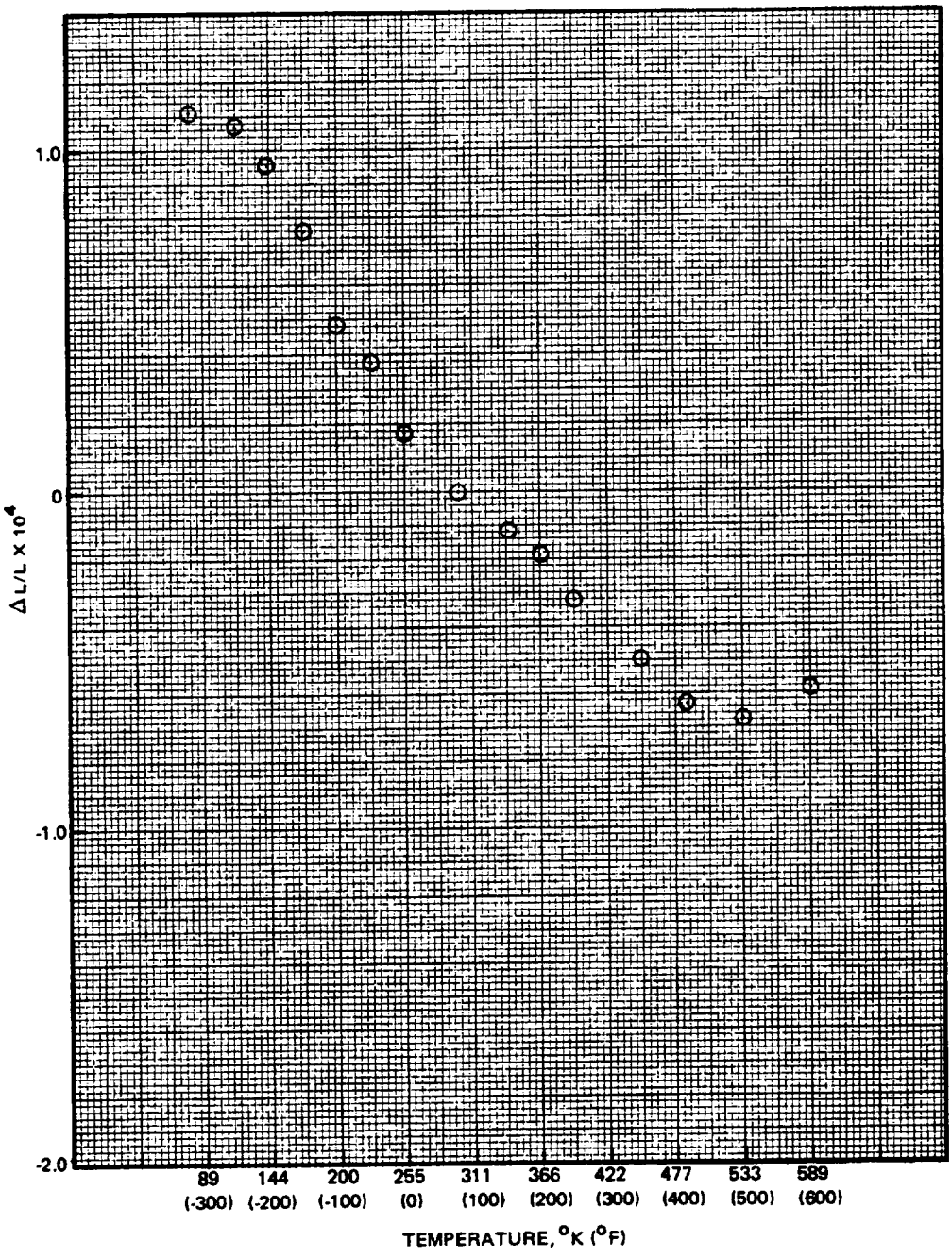


Figure 3-43. Total Linear Thermal Expansion of Unidirectional Layup, 0° Specimen

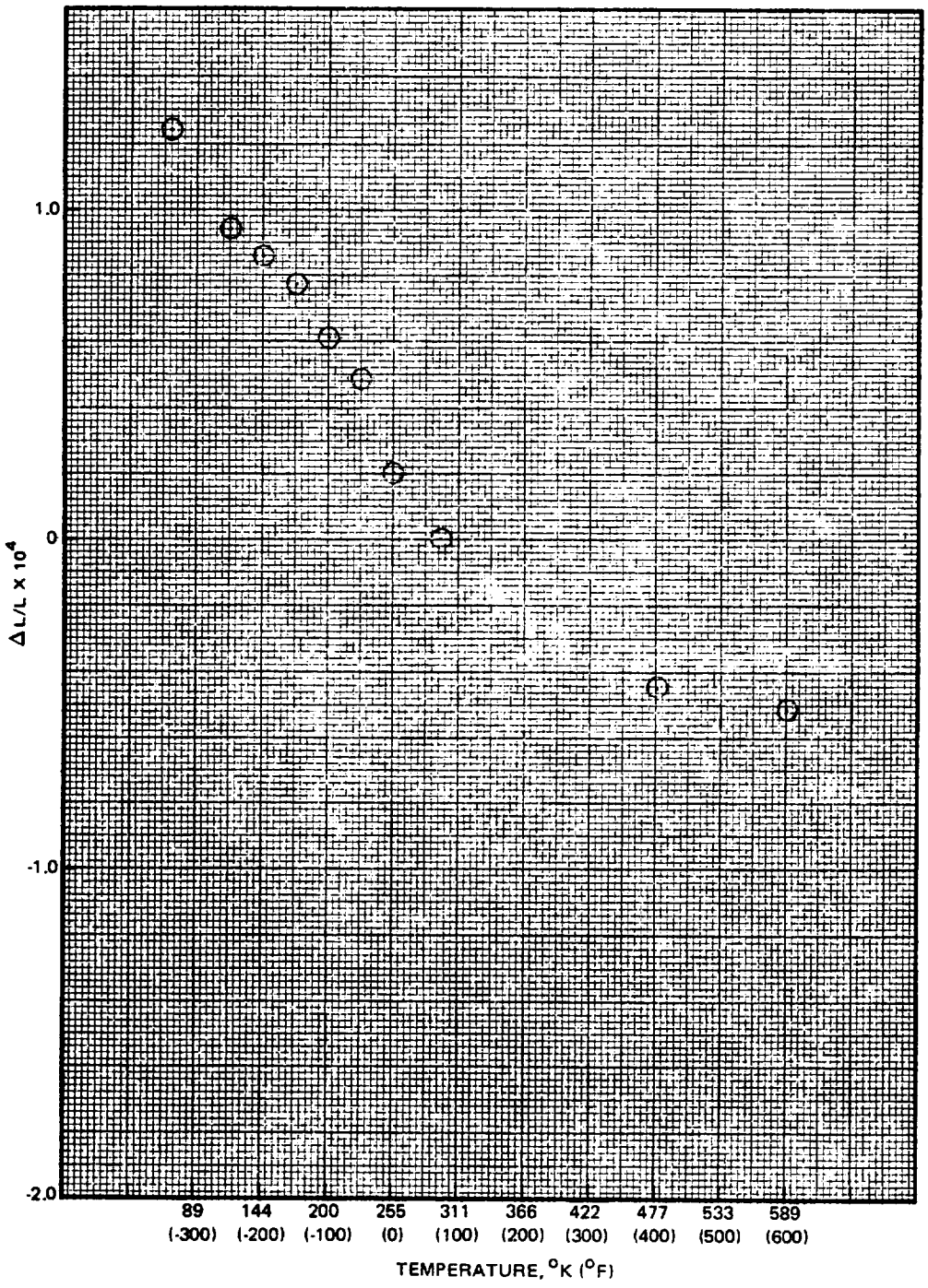


Figure 3-44. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen

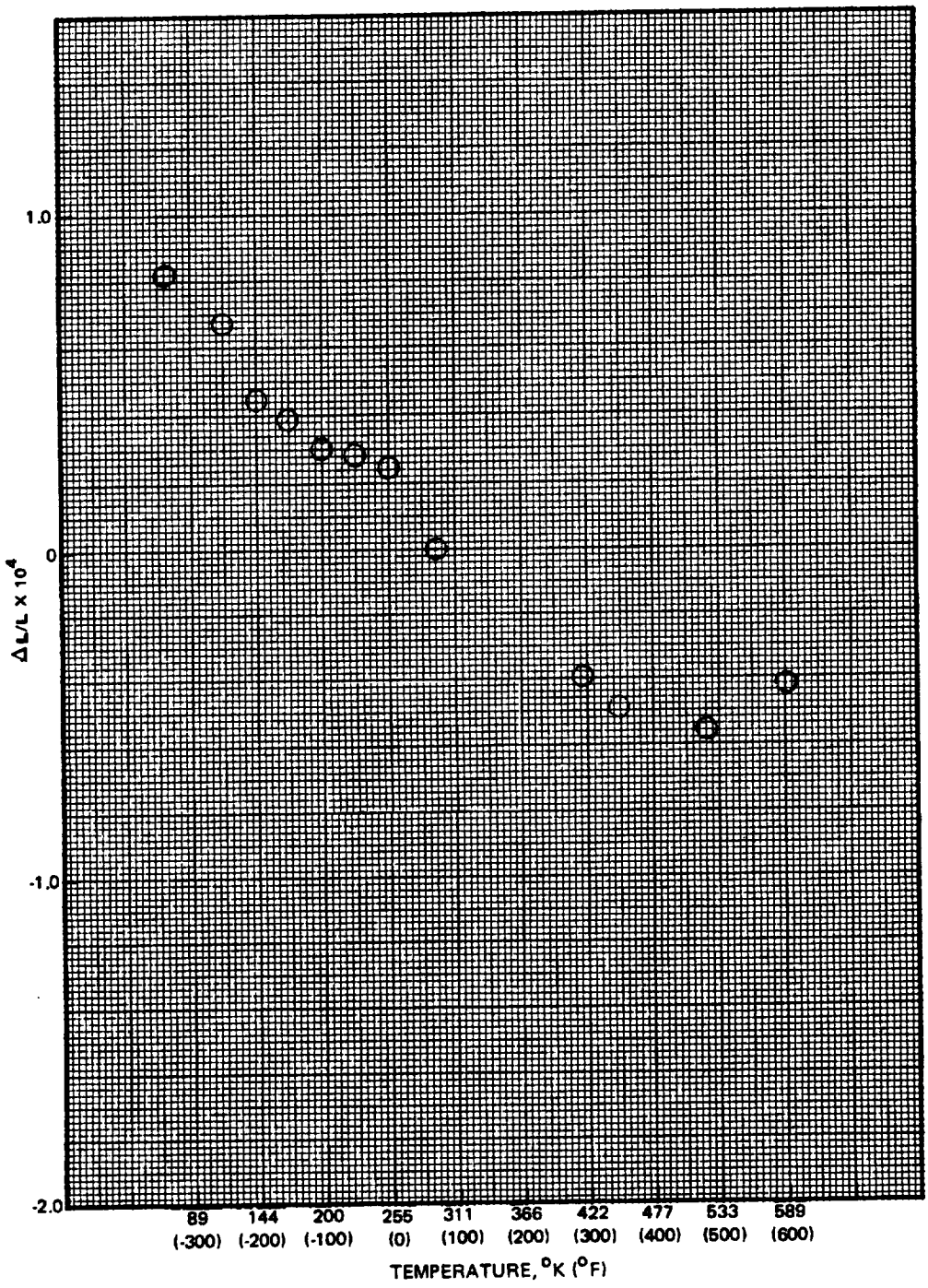


Figure 3-45. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 0° Specimen

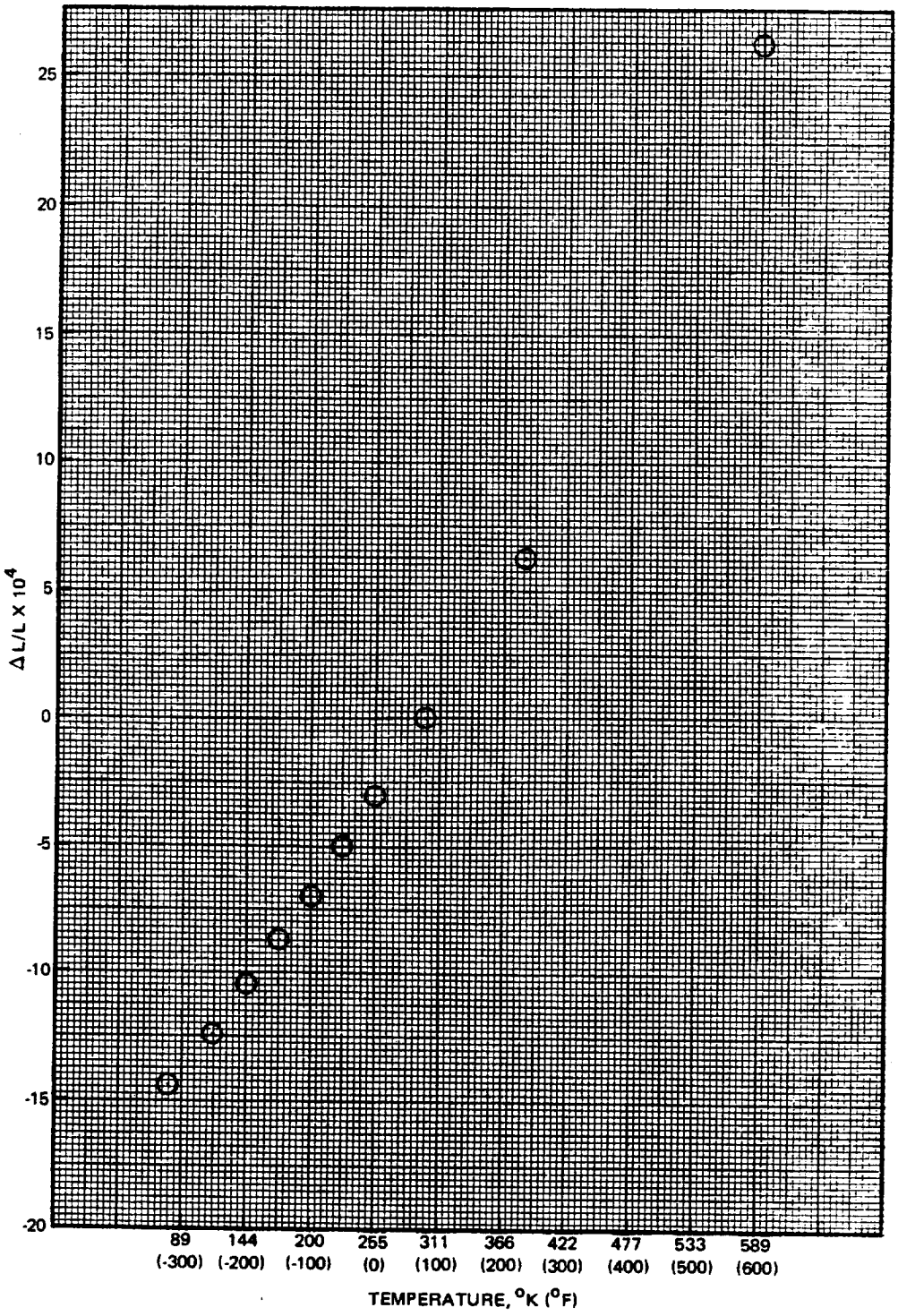


Figure 3-46. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen

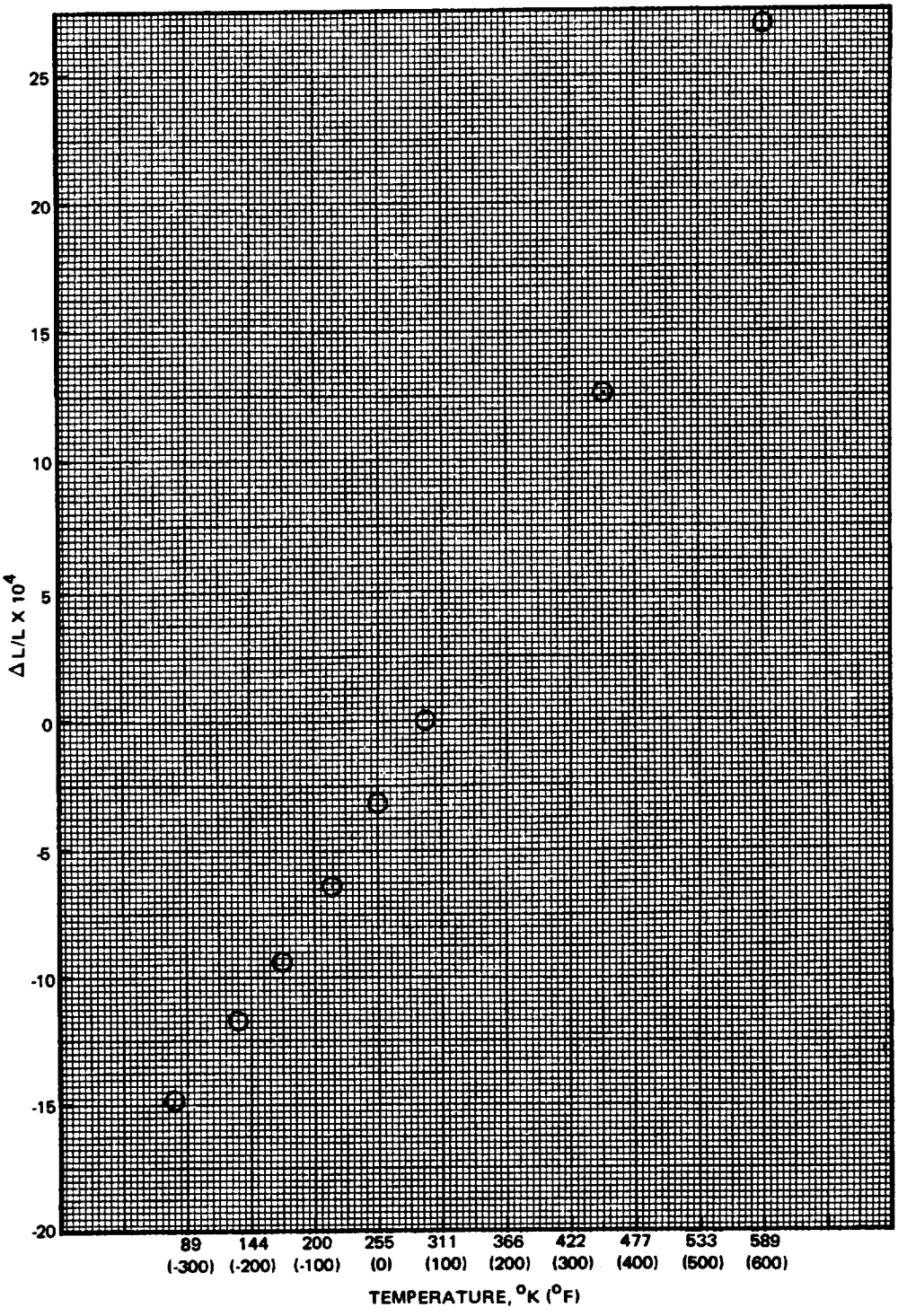


Figure 3-47. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen

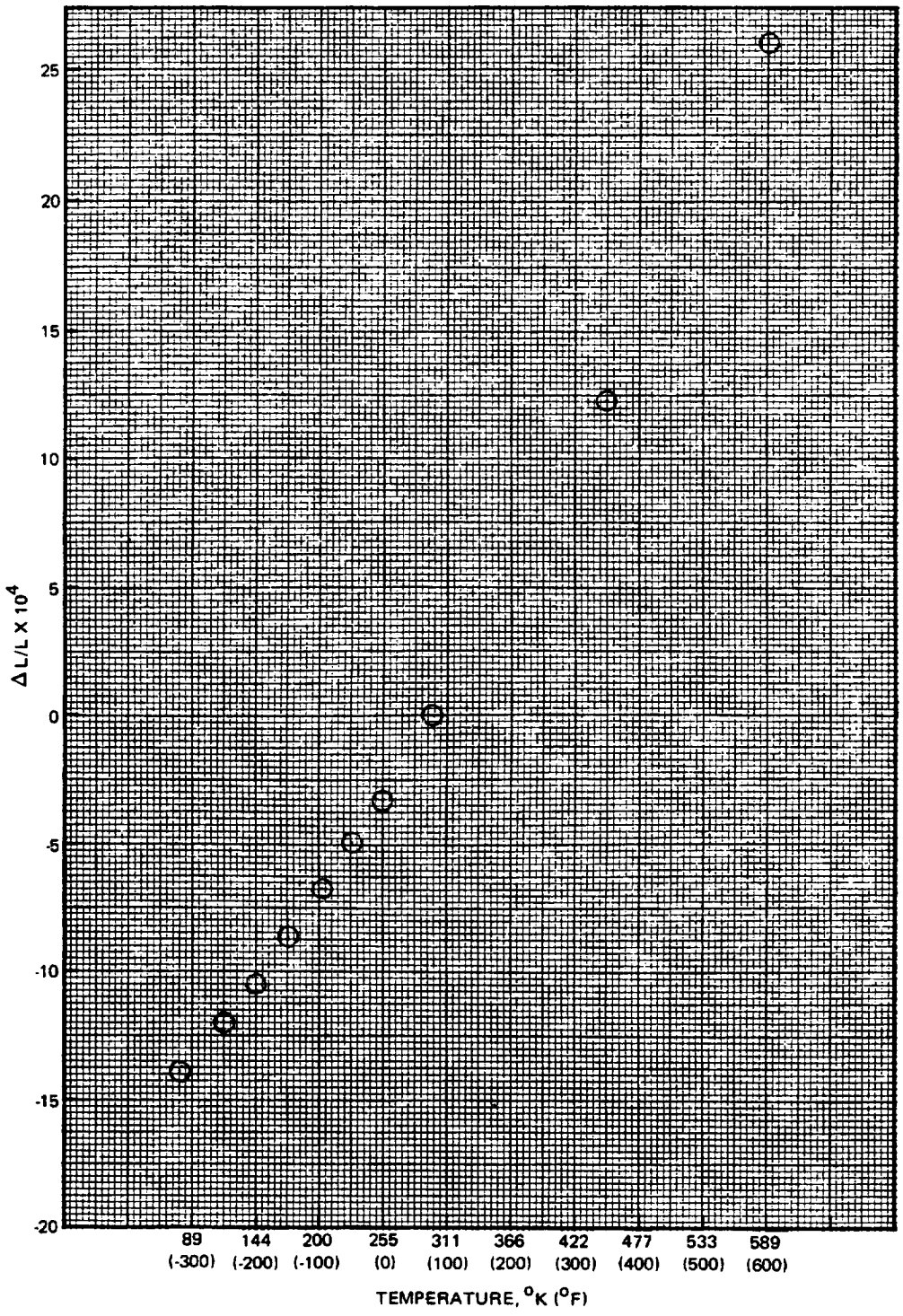


Figure 3-48. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 45° Specimen

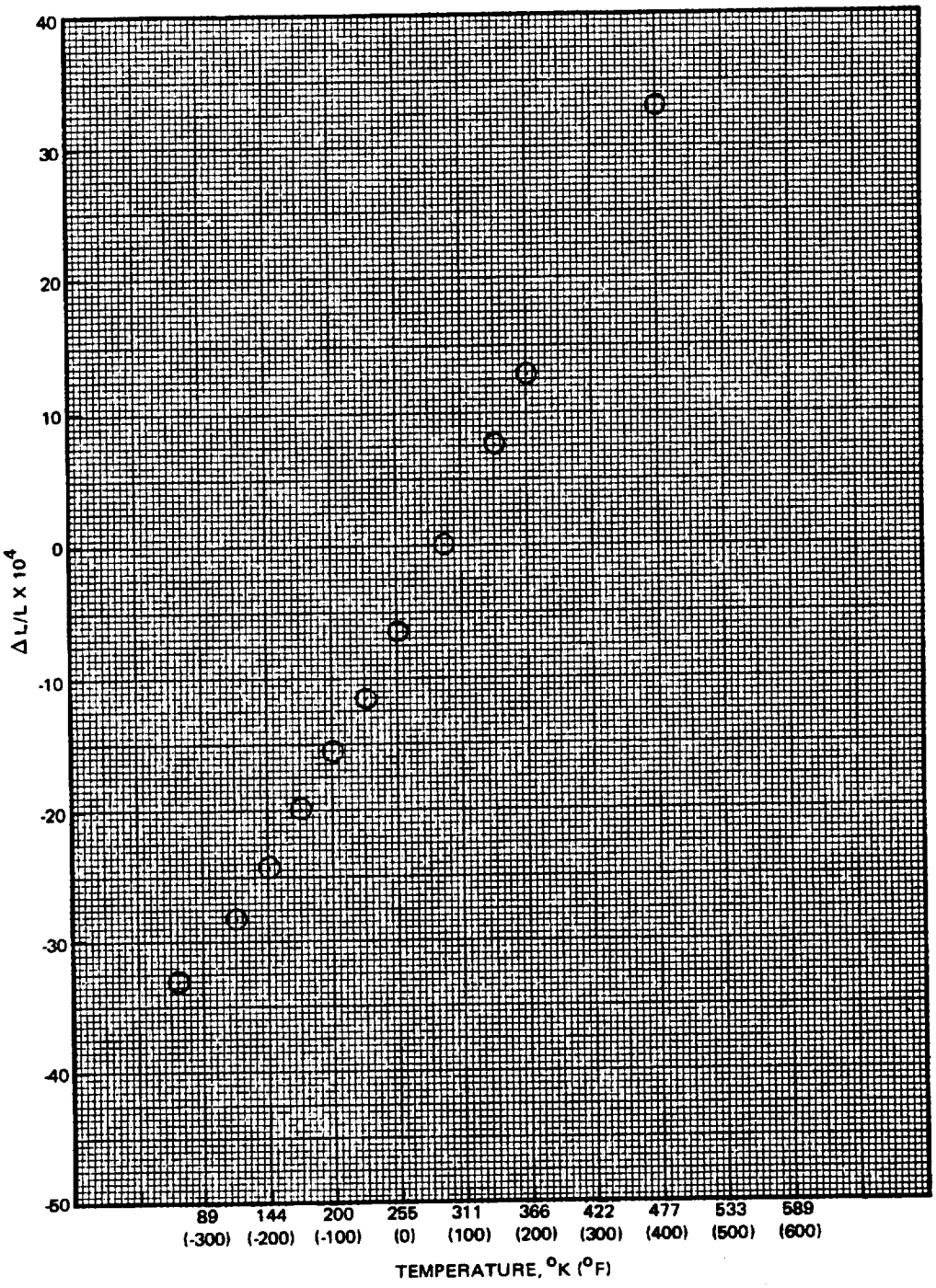


Figure 3-49. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen

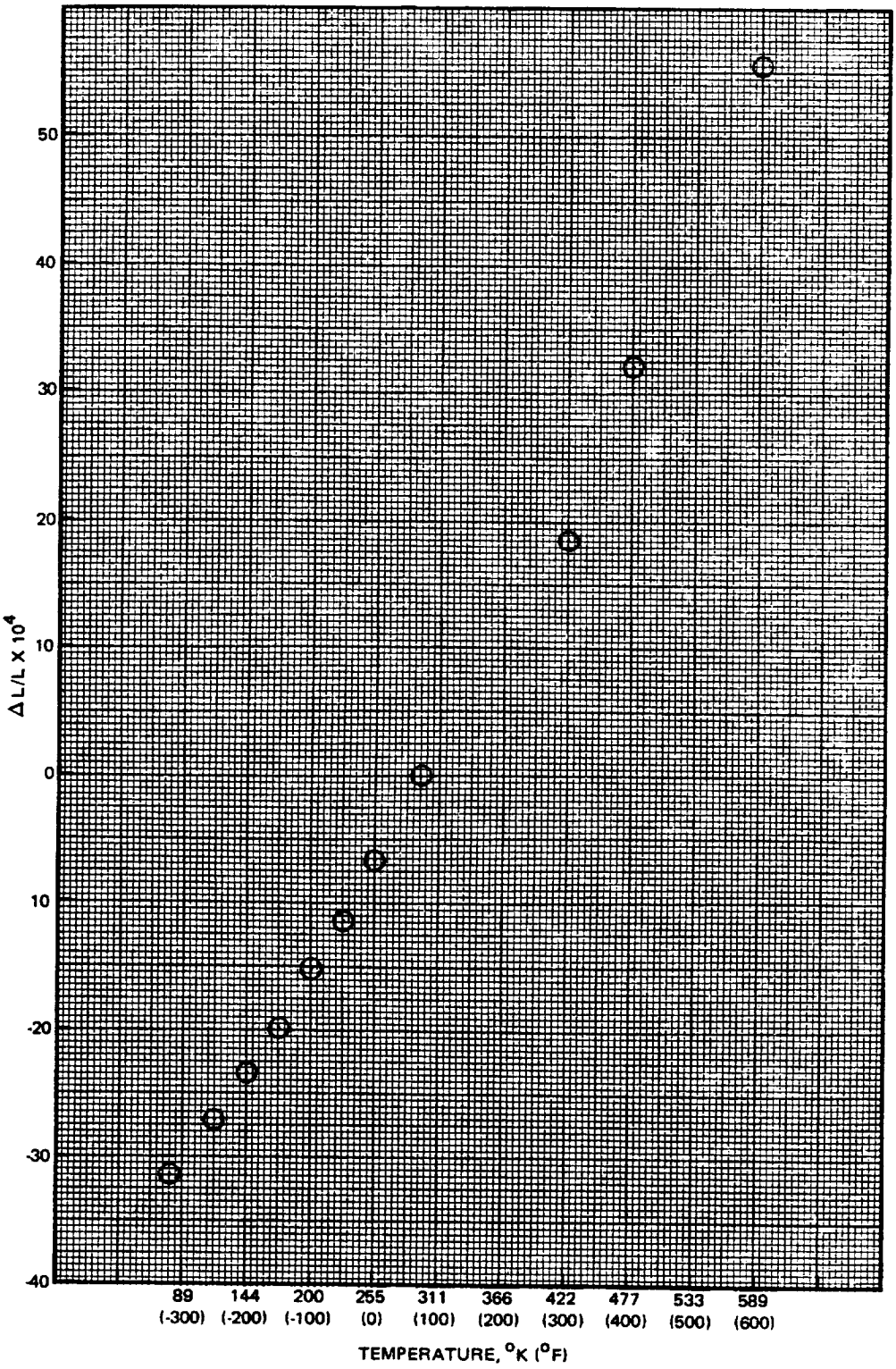


Figure 3-50. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen

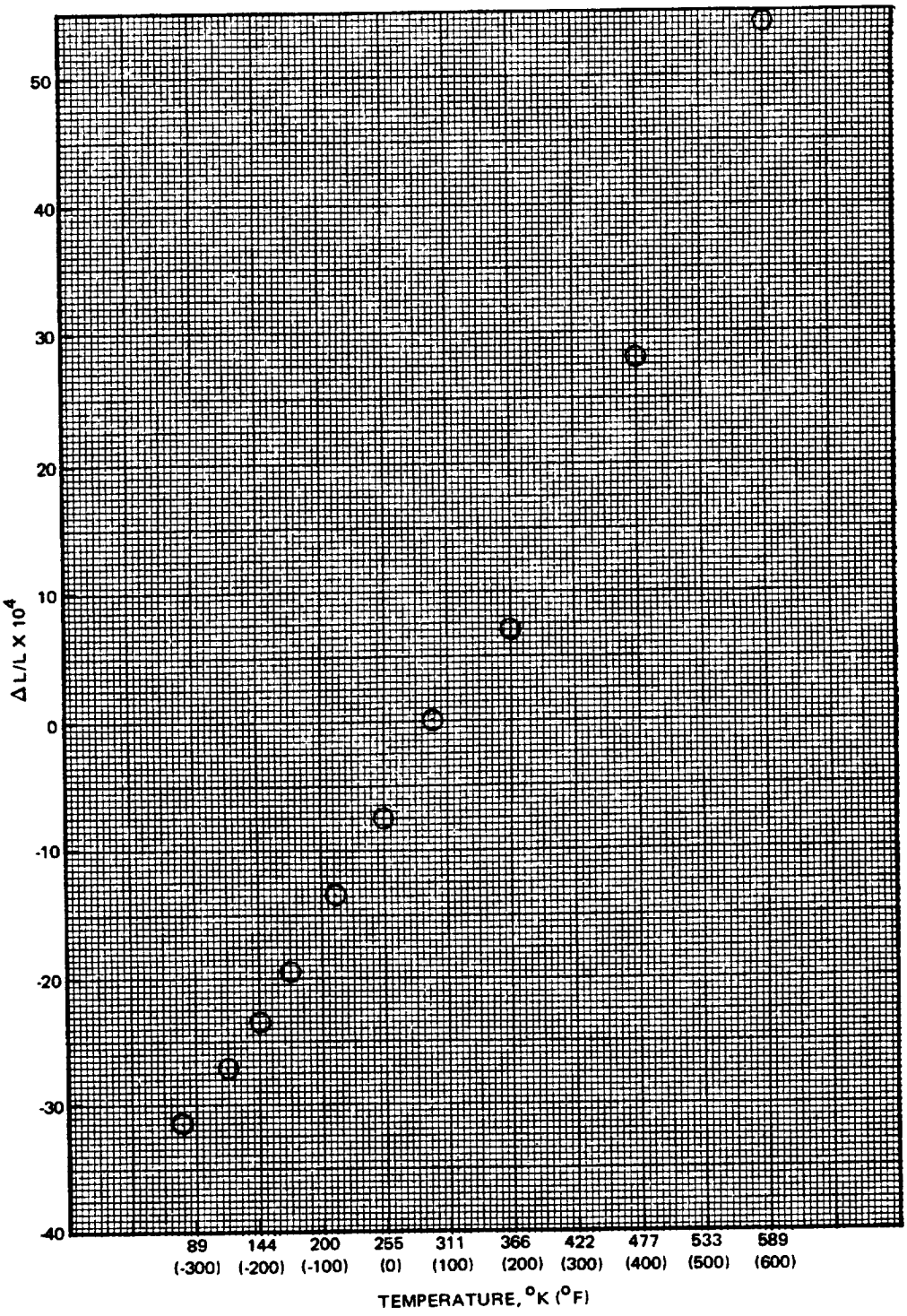


Figure 3-51. Total Linear Thermal Expansion of HT-S/710 Unidirectional Layup, 90° Specimen

SECTION 4

HIGH MODULUS GRAPHITE/POLYIMIDE COMPOSITES

In many space-vehicle systems certain structures are stiffness critical. Unlike aircraft, where fatigue and strain to failure are important design criteria, high-modulus composites can be used for space applications because of the differences in design criteria. Graphite composites are divided into two groups: those having high modulus fibers and those having ultra-high-modulus fibers. A high-modulus composite is one that is reinforced with a fiber that has a modulus greater than 346 GN/m^2 (50×10^6 psi). An ultra-high-modulus fiber is one that has a modulus greater than 486 GN/m^2 (75×10^6 psi). Typical fibers of each class are HM-S and Modmor I high-modulus fibers and GY-70 and Thornel 75 ultra-high-modulus fibers. Typically these fibers have a lower strain to failure than the high-strength graphite fibers. The interlaminar shear strength and the transverse strength of graphite composites using these fibers are low compared to high-strength graphite composites.

Three graphite fibers, HM-S, Modmor I, and GY-70, were selected for this program based on cost and availability. A fiber finish study and a thin interlayer study were conducted to improve the interlaminar shear and transverse strengths of the composites. One graphite fiber, HM-S was selected after the initial evaluations. Design and environmental test data were developed. A specification covering the material requirements and processing techniques is presented in the Appendix of this report.

4.1 PRELIMINARY COMPOSITE EVALUATION

Laminates were fabricated using the 710 polyimide resin cure cycle, reported in Section 3 of this report, and the staple length HM-S, meter length Modmore I, and continuous GY-70 graphite fibers. All prepreg was purchased to the prepreg requirements denoted in the specification and all resin came from the same batch. Longitudinal and transverse flexural strengths, interlaminar shear strength, and room-temperature compression strengths were determined for each of the composites. Data obtained is presented in Tables 4-1 through 4-3. For the GY-70 composites, transverse flexural strength could not be determined because the specimens failed upon machining them from the laminate. The HM-S/710 and Modmor I/710 laminates had higher flexural strengths and in general higher interlaminar shear strengths than the GY-70/710 laminates. The HM-S/710 composite system had 10 to 40 per cent higher strength than the Modmor I/710 system and was rated easier to handle by the material suppliers.

As discussed earlier, the interlaminar shear strength of high-modulus graphite/polyimide composites is very low. In an attempt to improve the strength of the

Table 4-1. HM-S/710 Graphite/Polyimide Composite Data

Test Temperature °K	(°F)	Laminate 215 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Compression Strength MN/m²	(ksi)	Laminate 225 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Compression Strength MN/m²	(ksi)
77	(-320)	1007	(146)			47.2	(6.76)			1014	(147)			42.3	(6.08)		
		896	(130)			44.4	(6.42)			834	(121)			38.8	(5.60)		
		869	(126)			46.5	(6.73)			1103	(160)			41.6	(6.01)		
	Ave	924	(134)			45.8	(6.64)			979	(142)			40.9	(5.90)		
297	(75)	883	(128)	17.2	(2.50)	49.3	(7.09)	465	(66.7)	862	(125)	20.6	(2.99)	38.8	(5.64)		
		924	(134)	20.8	(3.01)	50.0	(7.16)	395	(57.2)	1007	(146)	21.3	(3.09)	45.1	(6.48)		
		883	(128)	19.9	(2.88)	52.1	(7.47)	444	(64.3)	896	(130)	20.3	(2.94)	44.4	(6.43)		
	Ave	896	(130)	19.3	(2.80)	50.0	(7.24)	437	(62.7)	924	(134)	20.8	(3.01)	43.0	(6.18)		
589	(600)	758	(110)			29.0	(4.25)			765	(111)			33.9	(4.89)		
		640	(92)			33.2	(4.81)			821	(119)			36.7	(5.33)		
		786	(114)			31.1	(4.47)			834	(121)			36.7	(5.26)		
	Ave	731	(106)			31.1	(4.51)			807	(117)			36.0	(5.16)		
Specific Gravity		1.58								1.61							
% Resin Content		28.6								27.7							
% Fiber Volume		64.8								66.2							

Table 4-2. Modmor I/710 Graphite/Polyimide Composite Data

Test Temperature °K	(°F)	Laminate 207 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Compression Strength MN/m²	(ksi)	Laminate 224 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Compression Strength MN/m²	(ksi)
77	(-320)	577	(83)			31.1	(4.51)			827	(120)			47.2	(6.80)		
		710	(103)			32.5	(4.74)			752	(109)			46.5	(6.70)		
		549	(79)			45.1	(6.54)			633	(91)			50.7	(7.26)		
	Ave	613	(88)			36.7	(5.26)			731	(106)			47.9	(6.92)		
297	(75)	855	(124)	18.4	(2.67)	50.0	(7.16)	437	(62.8)	765	(111)	9.45	(1.37)	50.7	(7.31)	388	(56.0)
		689	(99)	15.0	(2.18)	47.9	(6.89)	381	(55.4)	765	(111)	7.17	(1.04)	43.0	(6.20)	318	(46.4)
		876	(127)	18.8	(2.73)	51.4	(7.41)	353	(50.6)	807	(117)	8.00	(1.16)	40.2	(5.85)	325	(46.6)
	Ave	807	(117)	17.4	(2.53)	50.0	(7.15)	388	(56.3)	779	(113)	8.21	(1.19)	44.4	(6.45)	346	(49.7)
589	(600)	689	(99)			29.0	(4.23)			772	(112)			37.4	(5.42)		
		807	(117)			31.8	(4.57)			800	(116)			40.2	(5.84)		
		654	(94)			29.0	(4.16)			786	(114)			36.7	(5.30)		
	Ave	710	(103)			29.7	(4.32)			786	(114)			38.1	(5.52)		
Specific Gravity		1.58								1.63							
% Resin Content		29.3								27.1							
% Fiber Volume		63.9								66.3							

Table 4-3. GY-70/710 Graphite/Polyimide Composite Data

Test Temperature °K	(°F)	Laminate 156 Long Flex Strength MN/m²	(ksi)	Laminate 156 Short Beam Shear Strength MN/m²	(ksi)	Laminate 156 Compression Strength MN/m²	(ksi)	Laminate 208 Long Flex Strength MN/m²	(ksi)	Laminate 208 Short Beam Shear Strength MN/m²	(ksi)	Laminate 208 Compression Strength MN/m²	(ksi)
77	(-320)	724	(105.1)	40.2	(5.82)			542	(77.9)	34.6	(5.00)		
		661	(95.0)	44.4	(6.38)			514	(73.8)	31.1	(4.51)		
		640	(91.6)	40.2	(5.84)			606	(86.8)	34.6	(4.99)		
	Ave	675	(97.2)	41.6	(6.01)			556	(79.5)	33.2	(4.83)		
297	(75)	640	(91.6)	40.2	(5.83)	332	(48.5)	690	(100.0)	33.9	(4.93)	318	(46.2)
		690	(100.4)	40.2	(5.85)	283	(40.5)	738	(106.6)	37.4	(5.37)	311	(45.4)
		682	(97.8)	41.6	(5.95)	290	(42.0)	613	(88.4)	36.7	(5.31)	381	(55.4)
	Ave	675	(96.6)	40.9	(5.87)	304	(43.7)	682	(98.3)	36.0	(5.20)	339	(49.0)
589	(600)	620	(89.0)	53.5	(7.66)			675	(97.0)	29.7	(4.30)		
		654	(94.3)	52.1	(7.46)			682	(98.0)	29.0	(4.21)		
		620	(89.1)	52.8	(7.57)			606	(87.2)	28.3	(4.14)		
	Ave	633	(90.8)	52.8	(7.56)			654	(94.1)	29.0	(4.22)		
Specific Gravity		1.65						1.62					
% Resin Content		26.5						27.0					
% Fiber Volume		65.1						66.1					

composite, a polyphenylquixoline (PPQ) sizing was applied to the graphite fibers first before the polyimide resin. The PPQ sizing was cured at 644° K (700° F) for 1 hour prior to the polyimide prepregging operation. Two laminates were fabricated for each of three composite systems. Flexural and short beam shear strengths were determined and are reported in Tables 4-4 through 4-6. For the HM-S/710 and Modmor I/710 composite systems, the PPQ sizing did not improve the interlaminar shear or transverse strength. In fact, a slight decrease was noted for the elevated temperature interlaminar shear strength of these composites when compared with data obtained for laminates without the PPQ fiber finish. The PPQ fiber finish had no effect on the longitudinal flexural strength, and since it was an added cost to the graphite/polyimide prepreg, it was decided that no further studies would be conducted with laminates fabricated from systems which had a PPQ sizing on the graphite fiber.

The use of a thin graphite interlayer with GY-70 composites had been shown to be feasible and improved the transverse strength for GY-70/epoxy laminates. Thin HM-S/710 graphite/polyimide prepreg was purchased and GY-70 laminates were fabricated. The first laminate had a 90° ply of HM-S/710 for every two 0° plies of GY-70/710, and the second laminate had a 90° ply of HM-S/710 for every 0° ply of GY-70/710. The test results obtained are presented in Table 4-7. Significant increases in transverse flexural strength were obtained along with a major reduction in longitudinal strength. This was unexpected, since the GY-70/HM-S/epoxy composites showed only a 10 per cent reduction in longitudinal strength. No significant differences were found for the interlaminar shear strengths of these laminates when compared with GY-70/710 laminates. The effects of the number of thin HM-S/710 plies present in the composite laminate were dramatic. By having a thin 90° HM-S/710 ply for every ply of 0° GY-70/710, the transverse strength was doubled over the other laminate and was 3500 per cent better than a conventional GY-70 laminate. No decrease in longitudinal modulus was determined; thus, for strictly a stiffness critical application such as stringers on adapters, the GY-70/HM-S/710 composite system is very attractive.

Before proceeding to the development of the design data, preliminary tensile, flexural, and compression tests were conducted on the HM-S/710 composite laminates. These tests were conducted to ensure that the flexural and interlaminar shear tests were not masking any problems with this type of composite. The test data is presented in Tables 4-8 and 4-9 and compares well with epoxy composite data for staple length HM-S and continuous GY-70. Based on the data developed on this program and reviewing potential applications, the HM-S/710 composite system was selected.

Table 4-4. HM-S/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing

Test Temperature °K	(°F)	Laminate 228 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Laminate 233 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)
77	(-320)	876	(127)			45.8	(6.59)	800	(116)			41.6	(5.95)
		986	(143)			41.6	(5.99)	731	(106)			41.6	(6.01)
		1034	(150)			45.1	(6.49)	690	(100)			45.8	(6.61)
	Ave	965	(140)			44.4	(6.36)	738	(107)			43.0	(6.19)
297	(75)	841	(122)	27.3	(3.96)	49.3	(7.06)	827	(120)	11.0	(1.59)	41.6	(6.04)
		910	(132)	21.0	(3.05)	46.5	(6.70)	883	(128)	14.8	(2.14)	40.9	(5.87)
		903	(131)	29.0	(4.18)	45.8	(6.61)	917	(133)	11.6	(1.68)	43.0	(6.21)
	Ave	883	(128)	25.7	(3.73)	47.2	(6.79)	876	(127)	12.4	(1.80)	41.6	(6.04)
589	(600)	549	(79.1)			27.2	(3.94)	855	(124)			31.1	(4.54)
		570	(81.5)			27.6	(4.03)	745	(108)			31.8	(4.57)
		542	(78.4)			25.9	(3.75)	903	(131)			29.7	(4.34)
	Ave	556	(79.7)			27.0	(3.91)	834	(121)			31.1	(4.48)
Specific Gravity		1.56						1.63					
% Resin Content		27.9											
% Fiber Volume		65.9											

Table 4-5. Modmor I/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing

Test Temperature °K	(°F)	Laminate 230 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Laminate 234 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)
77	(-320)	807	(117)			30.4	(4.44)	814	(118)			35.3	(5.09)
		758	(110)			36.7	(5.28)	1062	(154)			39.5	(5.68)
		772	(112)			35.3	(5.10)	855	(124)			34.6	(4.97)
	Ave	779	(113)			33.9	(4.94)	910	(132)			36.0	(5.25)
297	(75)	821	(119)	25.8	(3.74)	33.2	(4.79)	855	(124)	11.0	(1.59)	31.8	(4.63)
		855	(124)	26.3	(3.81)	32.5	(4.68)	765	(111)	10.4	(1.51)	33.9	(4.88)
		800	(116)	20.4	(2.96)	30.4	(4.41)	827	(120)	10.5	(1.52)	31.1	(4.51)
	Ave	827	(120)	24.1	(3.50)	31.8	(4.63)	821	(119)	10.6	(1.54)	32.5	(4.67)
589	(600)	563	(80.7)			25.3	(3.67)	689	(99)			29.7	(4.30)
		570	(81.7)			22.5	(3.26)	883	(128)			27.6	(4.03)
		535	(77.0)			23.4	(3.40)	855	(124)			29.0	(4.16)
	Ave	556	(79.8)			23.7	(3.44)	807	(117)			29.0	(4.16)
Specific Gravity		1.52						1.67					
% Resin Content		28.6											
% Fiber Volume		64.5											

Table 4-6. GY-70/710 Graphite/Polyimide Composite Data PPQ Fiber Sizing

Test Temperature °K	(°F)	Laminate 229 Long Flex Strength MN/m²	(ksi)	Laminate 229 Tran Flex Strength MN/m²	(ksi)	Laminate 229 Short Beam Shear Strength MN/m²	(ksi)	Laminate 235 Long Flex Strength MN/m²	(ksi)	Laminate 235 Tran Flex Strength MN/m²	(ksi)	Laminate 235 Short Beam Shear Strength MN/m²	(ksi)
77	(-320)	479	(68.9)			45.8	(6.65)	521	(75.2)			37.4	(5.45)
		409	(59.4)			33.9	(4.86	696	(100.5)			40.9	(5.89)
		599	(86.0)			31.8	(4.58)	627	(90.2)			40.2	(5.83)
	Ave	493	(71.4)			37.4	(5.36)	620	(88.6)			39.5	(5.72)
297	(75)	654	(93.8)	16.1	(2.33)	68.9	(9.88)	696	(101.0)	4.65	(0.67)	36.0	(5.19)
		507	(72.8)	17.4	(2.52)	65.4	(9.41)	668	(96.5)	6.89	(0.99)	36.0	(5.17)
		690	(100.4)	15.9	(2.30)	-	-	696	(101.2)	5.56	(0.80)	36.0	(5.15)
	Ave	620	(89.0)	15.2	(2.21)	66.8	(9.65)	690	(99.5)	5.70	(0.82)	36.0	(5.17)
589	(600)	592	(85.2)			25.7	(3.73)	675	(96.9)			31.1	(4.47)
		599	(86.4)			29.7	(4.29)	752	(109.1)			28.3	(4.13)
		627	(90.5)			28.3	(4.06)	640	(91.9)			32.5	(4.70)
	Ave	606	(87.4)			27.6	(4.03)	689	(99.4)			30.4	(4.43)
Specific Gravity		1.58						1.67					
% Resin Content		27.2											
% Fiber Volume		66.1											

Table 4-7. GY-70-HM-S/710 Graphite/Polyimide Composite Data

Test Temperature °K	(°F)	Laminate 218 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)	Laminate 219 Long Flex Strength MN/m²	(ksi)	Tran Flex Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)
77	(-320)	528	(76.4)			30.4	(4.37)	542	(78.4)			25.9	(3.75)
		549	(78.6)			33.9	(4.86)	570	(82.0)			24.6	(3.56)
		585	(84.5)			31.1	(4.53)	465	(67.2)			27.6	(4.04)
	Ave	556	(79.9)			31.8	(4.59)	528	(75.9)			26.1	(3.78)
297	(75)	535	(77.2)	166	(23.7)	27.6	(4.04)	479	(68.6)	248	(35.9)	32.5	(4.70)
		563	(81.0)	124	(18.3)	36.7	(5.31)	479	(69.1)	226	(32.8)	27.0	(3.92)
		549	(78.8)	103	(15.1)	35.3	(5.14)	458	(66.2)	263	(38.2)	25.0	(3.63)
	Ave	549	(79.0)	131	(19.0)	33.2	(4.83)	472	(68.0)	246	(35.6)	28.3	(4.08)
589	(600)	479	(68.9)			22.7	(3.32)	486	(69.9)			18.8	(2.73)
		430	(62.5)			22.0	(3.23)	465	(66.9)			27.2	(3.22)
		304	(44.4)			20.6	(2.98)	444	(64.2)			21.4	(3.11)
	Ave	409	(58.6)			22.0	(3.18)	465	(67.0)			20.8	(3.02)
Specific Gravity		1.58						1.56					
% Resin Content		32.3						28.7					
% Fiber Volume		60.5						64.2					

Table 4-8. Preliminary Design Data HM-S/710
Graphite/Polyimide Composites

Test Temperature °K	(°F)	Longitudinal Flexural Strength* MN/m^2	(ksi)	Longitudinal Tensile Strength MN/m^2	(ksi)
297	(75)	1020	(148)	745	(108)
		1062	(154)	855	(124)
		1110	(161)	731	(106)
		-	-	807	(117)
		-	-	834	(121)
		-	-	758	(110)
	Ave.	1062	(154)	786	(114
589	(600)	696	(101)	827	(120
		738	(107)	765	(111)
		758	(110)	772	(112)
		-	-	876	(127)
		-	-	855	(124)
		-	-	786	(114)
	Ave.	731	(106)	814	(118)
Specific Gravity		1.60			
% Resin Content		28.9			
% Fiber Volume		64.8			
*Determined on 8 ply tensile laminate					

4.2 HM-S/710 GRAPHITE/POLYIMIDE DESIGN DATA

Up to this point in the program, meter or staple length fiber had been used because of cost and availability. Continuous fiber became available and the difference in cost between staple or continuous became very small. It was decided that continuous HM-S fiber would be used for the design data development portion of this program. Tensile, compressive, flexural, and interlaminar shear strength (0° only) were determined for 0°, 90°, and ±45° layups. Tension, compression, and flexural modulus were also determined at 297° K, and 589° K (75° F, and 600° F). Thermal aging, fatigue, and resistance to moisture characteristics were determined for the HM-S/710 graphite/polyimide composite system.

Table 4-9. Preliminary Design Data GY-70/710 Graphite/Polyimide Composite

Test Temperature °K	(°F)	Long. Flex. Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)	Long. Tensile Strength MN/m^2	(ksi)	Tensile Modulus GN/m^2	(10^6 psi)	Compression Strength MN/m^2	(ksi)
297	(75)	627	(89.7)	30.4	(4.45)	731	(106)	276	(40.2)		
		627	(89.9)	32.5	(4.74)	772	(112)	272	(39.5)		
		556	(79.9)	29.0	(4.16)	841	(122)	283	(41.4)		
	Ave.	606	(86.5)	30.4	(4.45)	779	(113)	276	(40.4)		
589	(600)	613	(88.0)	26.3	(3.81)	620	(89.3)	240	(34.8	332	(48.5)
		633	(91.2)	26.3	(3.81)	627	(90.4)	271	(39.3)	276	(40.5)
		640	(92.1)	26.8	(3.88)	633	(91.2)	-	-	290	(42.0)
	Ave.	627	(90.4)	26.4)	(3.83)	627	(90.3)	255	(37.0)	304	(43.7)
Specific Gravity		1.64				1.62					
% Resin Content		28.8				27.8					
% Fiber Volume		64.2				65.3					

4.2.1 STATIC MECHANICAL PROPERTIES HM-S/710 GRAPHITE/POLYIMIDE. The design data obtained on this program is presented in Tables 4-10 through 4-12. The unidirectional tensile strengths were approximately 10 per cent higher than those achieved from staple length graphite fiber composites. However, the compression strength remained unchanged, and both the flexural and short beam shear strength decreased by as much as 30 per cent from that obtained on staple length composite laminates. The physical properties of the design laminates are presented in Table 4-13. The specific gravity is lower and the fiber volume is less than that formerly obtained for HM-S/710 composite laminates. Calculated void contents were also found to be as high as 10 to 12 per cent. This is approximately twice the normal void content achieved with 710 composite laminates and explains the lower flexural and interlaminar shear strengths.

The high temperature flexural tests failed primarily by delaminating again, which was probably caused by the higher void content. The ±45° laminates retained more resin than the 0° laminates, as did the HT-S/710 laminates. The 90° compression strength was 4 times that of either the 90° tension or flexural strength. Temperature seemed to have little effect on the 90° HM-S/710 mechanical properties. The strain to failure, as expected, was very low. The tension and compression properties of the ±45° laminates when tested in the 0° direction showed a decrease in strength of approximately 20 per cent as the temperature was increased from 297° K to 589° K (75° F to 600° F). The strain to failure also increased substantially as the test temperature was increased.

Problems encountered during the design data testing included the normal problem associated with compression testing, machining of the test specimens, and handling of the transverse specimens. The machining of the HM-S/710 specimens was more difficult in that the edges of the specimens chipped if diamond cutting tools were not used in machining the specimens. This was not the case when machining HT-S/710 specimens. The low transverse strength of the HM-S/710 composite made machining and handling the specimens extremely difficult.

The design data presented in this report for the HM-S/710 graphite/polyimide composite system is believed to be conservative, especially the compression data. The modulus data and the strain-to-failure data for the unidirectional specimens is believed to be higher than reported since extensometers were used instead of strain gages. The Norair compression specimen was used, since it was previously used in this program, instead of the sandwich beam or Celanese compression specimen. Both these latter specimens give higher compression test values.

4.2.2 THERMAL AGING, MOISTURE RESISTANCE, AND FATIGUE CHARACTERISTICS OF HM-S/710. Tension, compression, flexural, and short beam shear specimens were aged for 200 hours at 589° K (600° F) in an air circulating oven. The specimens after aging were then tested at 297° K (75° F) and 589° K (600° F). The

Table 4-10. Design Properties of HM-S/710 Graphite/Polyimide Composites - 0°

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)	Short Beam Shear Strength MN/m² (ksi)
297 (75)	1	0° (Initial)	0°	921 (134)	165 (24.0)	(5500)	359 (52.1)	186 (27.0)	(3000)	836 (121)	174 (25.2)	25.4 (3.69)
				855 (124)	172 (25.0)	(4900)	394 (57.2)	193 (28.0)	-	733 (106)	170 (24.6)	27.7 (4.02)
				936 (136)	162 (23.5)	(5750)	347 (50.3)	181 (26.2)	-	911 (132)	188 (27.3)	27.9 (4.04)
				1010 (146)	216 (31.3)	-	337 (48.9)	- -	-	776 (113)	196 (28.4)	32.1 (4.65)
				821 (119)	185 (26.8)	-	324 (47.0)	- -	-	808 (117)	195 (28.3)	31.4 (4.55)
				889 (129)	174 (25.2)	-	353 (51.2)	193 (28.0)	(2900)	763 (111)	201 (29.2)	30.8 (4.47)
			Ave.	905 (131)	179 (26.0)	(5380)	352 (51.1)	188 (27.3)	(2950)	805 (117)	188 (27.2)	29.2 (4.23)
589 (600)	1	0° (Initial)	0°	858 (124)	186 (27.0)	(4450)	216 (31.3)			574 (83.3)	179 (25.9)	23.9 (3.46)
				905 (131)	198 (28.7)	(4600)	249 (36.1)			633 (91.8)	142 (20.6)	23.7 (3.44)
				917 (133)	192 (27.9)	(3700)	273 (39.6)			619 (89.8)	161 (23.4)	24.0 (3.48)
				872 (126)	170 (24.7)	-	290 (42.0)			539 (78.2)	137 (19.9)	26.0 (3.77)
				826 (120)	164 (23.8)	(5010)	- -			586 (85.0)	177 (25.6)	26.5 (3.84)
				944 (137)	150 (21.8)	(4900)	- -			592 (85.8)	150 (21.8)	26.1 (3.78)
			Ave.	887 (129)	177 (25.6)	(4532)	257 (37.2)			590 (85.6)	158 (22.9)	25.0 (3.63)
297 (75)	1	0° [200 hrs at 589°K(600°F)]	0°	945 (137)	161 (23.4)	(8650)	346 (50.2)			747 (108)		28.0 (4.06)
				849 (123)	159 (23.1)	(6000)	290 (42.0)			927 (134)		31.2 (4.52)
				868 (126)	169 (24.5)	(9900)	- -			806 (117)		29.4 (4.26)
			Ave.	887 (129)	163 (23.7)	(8180)	318 (46.1)			827 (120)		29.5 (4.28)
589 (600)	1	0° [200 hrs at 589°K(600°F)]	0°	903 (131)	194 (28.1)	(4250)	217 (31.5)			579 (84.0)		24.5 (3.56)
				752 (109)	190 (27.5)	(5500)	284 (41.2)			587 (85.2)		27.2 (3.94)
				891 (129)	208 (30.1)	(5300)	264 (38.3)			671 (97.3)		25.8 (3.74)
			Ave.	849 (123)	197 (28.6)	(4875)	255 (37.0)			612 (88.8)		25.9 (3.75)

Table 4-11. Design Properties of HM-S/710 Graphite/Polyimide Composites - 90°

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Strain to Failure (in./in.×10⁻⁶)	Flexural Strength MN/m² (ksi)	Flexural Modulus GN/m² (psi×10⁶)
297 (75)	1	0	90	9.4 (1.37)	10.0 (1.44)	(1920)	53.6 (7.77)	18.7 (2.21)		16.6 (2.41)	10.8 (1.57)
				10.3 (1.49)	10.2 (1.48)	(2030)	67.4 (9.78)	12.1 (1.76)		22.5 (3.26)	10.2 (1.48)
				14.1 (2.04)	10.8 (1.56)	(2600)	61.4 (8.91)	13.2 (1.92)		20.5 (2.97)	10.7 (1.55)
				12.3 (1.78)	10.2 (1.48)	(2500)	59.0 (8.55)	14.6 (2.12)		31.8 (4.61)	13.9 (2.02)
				14.1 (2.05)	9.6 (1.38)	(2700)	66.8 (9.69)	14.3 (2.08)		18.6 (2.70)	10.3 (1.50)
				12.8 (1.85)	9.0 (1.30)	(2750)	- -	- -		28.5 (4.10)	10.2 (1.48)
			Ave.	12.2 (1.77)	10.0 (1.44)	(2420)	61.6 (8.94)	13.9 (2.02)		23.0 (3.34)	11.0 (1.60)
589 (600)	1	0	90	11.0 (1.60)	8.0 (1.16)	(2600)	49.8 (7.22)	5.9 (0.85)	(1650)	16.0 (2.32)	9.4 (1.36)
				11.4 (1.65)	10.0 (1.44)	(2950)	52.1 (7.49)	9.6 (1.40)	-	18.4 (2.67)	9.7 (1.41)
				11.9 (1.72)	12.0 (1.74)	(2000)	52.5 (7.61)	5.2 (0.76)	(2050)	21.1 (3.06)	7.6 (1.10)
				11.7 (1.70)	8.8 (1.28)	(3100)	50.0 (7.25)	7.9 (1.15)	(1540)	19.7 (2.86)	6.8 (0.99)
				10.6 (1.54)	9.1 (1.32)	(2450)	49.2 (7.13)	5.4 (0.78)	(1540)	21.9 (3.17)	8.4 (1.22)
				13.9 (2.02)	12.0 (1.74)	(3000)	51.4 (7.40)	8.3 (1.20)	-	18.3 (2.66)	6.3 (0.91)
			Ave.	11.7 (1.70)	10.0 (1.44)	(2680)	51.2 (7.35)	7.0 (1.02)	(1695)	19.2 (2.79)	8.0 (1.16

Table 4-12. Design Properties of HM-S/710 Graphite/Polyimide Composites - (±45°)

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m^2 (ksi)	Tensile Modulus GN/m^2 ($psi \times 10^6$)	Strain to Failure ($in./in. \times 10^{-6}$)	Compression Strength MN/m^2 (ksi)	Compression Modulus GN/m^2 ($psi \times 10^6$)	Strain to Failure ($in./in. \times 10^{-6}$)
297 (75)	1	(±45°)	0°	75 (10.9)	15.9 (2.30)	(8800)	100 (14.5)	29.6 (4.30)	(8910)
				72 (10.5)	13.7 (1.99)	(9200)	106 (15.4)	19.0 (2.76)	(10200)
				62 (9.9)	13.6 (1.97)	(6100)	101 (14.7)	31.0 (4.50)	(9900)
				77 (11.2)	13.9 (2.02)	(6900)	94 (13.7)	25.2 (3.65)	-
				74 (10.7)	13.4 (1.95)	(8400)	120 (17.4)	26.3 (3.81)	-
				74 (10.7)	14.5 (2.10)	-	102 (14.8)	25.0 (3.62)	-
			Ave.	74 (10.7)	13.8 (2.00)	(7880)	104 (15.1)	26.0 (3.77)	(9670)
589 (600)	1	(±45°)	0°	59 (8.5)	14 (2.1)	-	66 (9.6)	22.1 (3.20)	(18000)
				55 (8.0)	12 (1.8)	(12300)	88 (12.8)	15.4 (2.23)	(12100)
				58 (8.4)	13 (1.9)	-	75 (10.9)	12.3 (1.78)	(11000)
				53 (7.7)	10 (1.4)	(14400)	78 (11.3)	17.2 (2.49)	(13750)
				59 (8.5)	12 (1.8)	(11700)	82 (11.9)	19.7 (2.86)	-
				58 (8.4)	9 (1.3)	(12800)	92 (13.3)	15.5 (2.25)	-
			Ave.	57 (8.3)	12 (1.7)	(12800)	80 (11.6)	17.0 (2.47)	(13712)

Table 4-13. Physical Properties of HM-S/710 Design Data Laminates

Laminate Number	Number of Plies	Degrees Laminate Orientation	Specific Gravity GMS/CC	Per Cent Resin Content	Per Cent Fiber Volume
D-1	8	0	1.49	31.3	62.1
D-2	12	0	1.49	33.2	60.0
D-3	8	±45	1.53	37.6	55.4
D-4	8	0	1.48	34.8	58.5
D-5	12	±45	1.53	36.3	56.7
D-6	18	0	1.50	31.8	61.6
D-7	18	±45	1.53	35.3	57.9

test results are shown in Table 4-10. The 200 hour aging had no effect on the mechanical properties of the HM-S/710 graphite/polyimide composite. This was as expected, since the HT-S/710 composites had shown relatively no change over a similar 400-hour thermal aging at 589° K (600° F). Even with the higher than expected void content in the HM-S/710 composite laminates, the mechanical properties were not affected.

With the discovery that moisture has an effect on epoxy composite high temperature strength, Convair Aerospace checks each new composite system for its resistance to moisture. Unidirectional flexure and short beam shear specimens were tested after being exposed to 24 hour H_2O boil, 3-week humidity [323° K (120° F) 95-100 per cent humidity] and 6-week humidity exposure. The test results are presented in Table 4-14. No effect on the room- or high-temperature mechanical properties was observed for the HM-S/710 graphite/polyimide system. Similar results had been obtained for the HT-S/710 composites.

Fatigue tests R = 0.1 were conducted on 0° and ±45° HM-S/710 laminates at room temperature. The test results are shown in Tables 4-15 and 4-16. The fatigue specimens were 8 ply thick and identical in dimensions to the standard composite tensile specimen. Residual strength was determined for the specimens that did not fail during the fatigue testing. A safe runout fatigue value (i.e., 10,000,000 cycles) is approximately 60 ±5 per cent of the static strength of the composite. The ±45° test specimens were tested at a higher percentage of static ultimate strength than the unidirectional specimens. At least for this type of fatigue loading, the voids present in the composite did not seem to have an effect on the fatigue properties of the HM-S/710 system, when compared with other composite systems. The residual strength and modulus of the composites were relatively unchanged after the fatigue testing.

Table 4-14. Environmental Aging Data HM-S/710 Graphite/Polyimide Composites

Exposure	Test Temperature °K	(°F)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)
Controls	297	(75)	836	(121)	27.7	(4.02)
			733	(106)	27.9	(4.04)
			911	(132)	32.1	(4.65)
			776	(113)	31.4	(4.55)
			808	(117)	30.8	(4.47)
			763	(111)	25.4	(3.69)
		Ave.	805	(117)	29.2	(4.23)
	589	(600)	574	(83.3)	23.9	(3.46)
			633	(91.8)	23.7	(3.44)
			619	(89.8)	24.0	(3.48)
			539	(78.2)	26.0	(3.77)
			586	(85.0)	26.5	(3.84)
			592	(85.8)	26.1	(3.78)
		Ave.	590	(85.6)	25.0	(3.63)
24 Hours H_2O Boil	297	(75)	850	(123)	32.3	(4.69)
			716	(104)	33.2	(4.81)
			784	(114)	31.9	(4.63)
		Ave.	783	(114)	32.5	(4.71
	589	(600)	650	(94.2)	23.6	(3.43)
			647	(93.9)	23.3	(3.38)
			583	(84.5)	26.0	(3.77)
		Ave.	627	(91.0)	24.3	(3.53)
3 Weeks 323° K (120° F) 95-100% Relative Humidity	297	(75)	718	(104)	31.6	(4.59)
			765	(111)	29.7	(4.31)
			675	(98)	31.3	(4.54)
		Ave.	720	(104)	30.9	(4.48)
	589	(600)	563	(81.6)	21.2	(3.08)
			600	(87.0)	24.2	(3.51)
			656	(95.2)	22.1	(3.20)
		Ave.	606	(87.9)	22.5	(3.26)
6 Weeks 323° K (120° F) 95-100% Relative Humidity	297	(75)	716	(104)	29.4	(4.26)
			713	(103)	28.3	(4.10)
			692	(100)	28.9	(4.19)
		Ave.	707	(102)	28.8	(4.18)
	589	(600)	613	(88.9)	26.9	(3.90)
			620	(89.9)	25.1	(3.64)
			565	(81.9)	24.1	(3.50)
		Ave.	599	(86.9)	25.4	(3.68

Table 4-15. Room Temperature Fatigue Testing R = 0.1 of 0°
HM-S/710 Graphite/Polyimide Composites

Specimen Number	Stress Level MN/m^2	(ksi)	Cycles to Failure	Residual Tensile Strength MN/m^2	(ksi)	Tensile Modulus GN/m^2	($psi \times 10^6$)
1	486	(70)	10,000,000	703	(102)	169	(24.5)
2	592	(85)	64,000	-	-	-	-
3	592	(85)	<1,000	-	-	-	-
4	627	(90)	6,555,000	-	-	-	-
5	627	(90)	<1,000	-	-	-	-
6	627	(90)	1,000	-	-	-	-
7	627	(90)	9,665,000	1055	(153)	176	(25.6)
8	661	(95)	<1,000	-	-	-	-
9	661	(95)	14,916,000	779	(113)	186	(27.0)
10	690	(100)	<1,000	-	-	-	-
11	690	(100)	1,000	-	-	-	-
12	690	(100)	2,000	-	-	-	-
13	724	(105)	8,161,000	986	(143)	177	(25.7)
14	724	(105)	<1,000	-	-	-	-
15	758	(110)	10,110,000	814	(118)	188	(27.2)
16	758	(110)	<1,000	-	-	-	-
			Average	869	(126)	179	(26.0)
Average static strength = 905 MN/m^2 (131 ksi)							

Table 4-16. Room Temperature Fatigue Testing R = 0.1 of (±45°) HM-S/710 Graphite/Polyimide Composites

Specimen Number	Stress Level MN/m^2	(ksi)	Cycles to Failure	Residual Tensile Strength MN/m^2	(ksi)
1	28	(4.0)	12,542,000	75.2	(10.9)
2	42	(6.0)	3,007,000	71.7	(10.4)
3	49	(7.0)	4,461,000	69.0	(10.0)
4	52	(7.5)	751,000	-	-
5	52	(7.5)	4,330,000	-	-
6	52	(7.5)	3,000,000	73.1	(10.6)
7	54	(7.8)	387,000	-	-
8	54	(7.8)	4,352,000	66.8	(9.6)
9	54	(7.8)	12,925,000	56.3	(8.1)
10	56	(8.0)	<1,000	-	-
11	56	(8.0)	<1,000	-	-
12	56	(8.0)	520,000	-	-
13	56	(8.0)	2,226,000	-	-
14	56	(8.0)	1,109,000	-	-
15	58	(8.3)	662,000	-	-
16	59	(8.5)	58,000	-	-
			Average	68.9	(9.9)
Average static strength = 74 MN/m^2 (10.7 ksi)					

SECTION 5

HIGH TEMPERATURE COMPOSITE SYSTEMS

An epoxy-phenolic and an additional type polyimide resin were evaluated as potential high-temperature resin systems. The epoxy-phenolic resin was a modification of the HT-424 adhesive system. This resin system was evaluated as a potential replacement resin for epoxies when the moisture problem was discovered. The additional polyimide resin system was the P105A system that had been developed by TRW and is available from Ciba Geigy. Data in the literature was conflicting as to the upper temperature/time capability of the P105A resin. Both HT-S/P105A and HM-S/P105A prepreg were obtained for this limited evaluation study.

5.1 HT-S/HT-424 GRAPHITE/EPOXY-PHENOLIC COMPOSITES

Two laminates each were fabricated using the HT-S/HT-424 system by vacuum bag, press, and autoclave curing techniques. Initial laminates fabricated by each processing technique were intended for checking out cure cycle parameters. The cure cycle used for all three fabrication methods is as follows:

> Full vacuum of 760 mm (29 in.) Hg at room temperature was applied and the part was heated to 352° K (175° F) at a rate of 1 to 3° K/min (3 to 5° F/min) and held for 4 hours. After the 4-hour hold in the press and autoclave cures, 690 KN/m^2 (100 psi) pressure was applied. The part was heated to 450° K (350° F) at a rate of 1 to 3° K/min (3 to 5° F/min) and held for 2 hours. The part was then cooled to below 344° K (160° F) under pressure and removed from the oven, press, or autoclave.

The initial flexural and short beam shear strengths of the six HT-S/HT-424 laminates are reported in Tables 5-1 through 5-3. One laminate fabricated for each of the processing techniques was evaluated for its resistance to moisture. The strength retention data after various types of exposure to moisture is presented in Table 5-4. At elevated test temperatures, the flexural strength of the HT-S/HT-424 composite decreased approximately 10 per cent, while the short beam shear strength decreased as much as 70 per cent after being exposed to moisture. As expected, the room temperature strengths were unaffected by exposure to moisture. Based on this limited evaluation study, the HT-424 epoxy-phenolic resin did not offer any improvement over epoxy resin systems. Actually this system has some disadvantages over epoxy systems, such as longer cure cycles and the larger amount of volatiles to be removed during the cure.

5.2 P105A GRAPHITE/POLYIMIDE EVALUATION STUDY

HT-S/P105A and HM-S/P105A graphite/polyimide laminates were fabricated, and mechanical properties of these composite systems were determined at 297° K (75° F) and 589° K (600° F) initially and after 200 hours at 589° K (600° F). The laminates were autoclaved cured to the following cycle:

> Full vacuum 760 mm (29 in.) Hg was applied at room temperature and the laminates were heated to 456 (375° F) at 1 to 3° K/min (3 to 5° F/min) and held for 2 hours. After the 2 hour hold 690 KN/m^2 (100 psi) was applied and the part was heated to 562° K (550° F) and held for 2 hours. The part was then cooled to below 344° K (160° F) under pressure and removed from the autoclave.

The only differences in curing this polyimide system than for other polyimide or epoxy systems is that the bagging material is H-film (polyimide) instead of nylon film, and the putty is silicone instead of zinc chromate.

The test data from this evaluation study is presented in Tables 5-5 and 5-6. Both the HT-S/P105A and the HM-S/P105A composite systems have excellent strength properties initially at room temperature and 589° K (600° F). However, after 200 hours at 589° K (600° F), the strength properties decreased as much as 70 per cent, especially the compression strength. It appears from this study that the P105A polyimide resin system can be used for short times at 589° K (600° F) but not for any extended period of time. This resin system, however, might be usable at 533° K (500° F) for extended periods of time. No difficulties were encountered in curing the P105A polyimide resin system. For lower temperature applications this is a potential resin system, and a more detailed study should be conducted.

Table 5-1. Mechanical Properties of HT-S/HT-424 Graphite Composites (Vacuum Bag Cure)

Type of Test	Test Temperature °K	(°F)	Laminate No. 121 MN/m^2	(ksi)	Laminate No. 136 MN/m^2	(ksi)
Flexure	77	(-320)	-	-	-	-
	297	(75)	1083	(157.1)	834	(120.9)
	450	(350)	577	(83.6)	685	(99.4)
	505	(450)	-	-	548	(79.5)
Short Beam Shear	297	(75)	-	-	50	(7.3)
	505	(450)	-	-	27	(3.9)
% Fiber Volume			50.5		54.5	
% Resin Content			41.1		39.2	
Specific Gravity			1.32		1.38	

Table 5-2. Mechanical Properties of HT-S/HT-424 Graphite Composite (Press Cure)

Type of Test	Test Temperature °K	(°F)	Laminate No. 120 MN/m^2	(ksi)	Laminate No. 142 MN/m^2	(ksi)
Flexure	77	(-320)			-	-
	297	(75)	1241	(180.3)	1276	(185.0)
	450	(350)	636	(93.6)	588	(85.3)
	505	(450)	-	-	464	(67.3)
Short Beam Shear	297	(75)	-	-	80	(11.6)
	505	(450)	-	-	47	(6.9)
% Fiber Volume			56.0		52.3	
% Resin Content			37.3		41.6	
Specific Gravity			1.44		1.51	

Table 5-3. Mechanical Properties of HT-S/HT-424 Graphite Composites (Autoclave Cure)

Type of Test	Test Temperature °K	(°F)	Laminate No. 127 MN/m^2	(ksi)	Laminate No. 145 MN/m^2	(ksi)
Flexure	77	(-320)	1131	(164.3)	-	-
	297	(75)	1462	(212.3)	1296	(188.2)
	450	(350)	876	(126.9)	631	(91.6)
	505	(450)	562	(81.5)	489	(70.9)
Short Beam Shear	297	(75)	-	-	88	(12.9)
	505	(450)	-	-	69	(10.0)
% Fiber Volume			54.0		58.8	
% Resin Content			37.1		34.4	
Specific Gravity			1.48		1.50	

Table 5-4. Graphite Composite Aging Data, HT-S/HT-424

Laminate Number	Type of Test	Test Temperature °K	Test Temperature (°F)	Control MN/m²	Control (ksi)	2 Hr. H_2O Boil MN/m²	2 Hr. H_2O Boil (ksi)	24 Hr. H_2O Boil MN/m²	24 Hr. H_2O Boil (ksi)	3 Week Humidity Exposure MN/m²	3 Week Humidity Exposure (ksi)	6 Week Humidity Exposure MN/m²	6 Week Humidity Exposure (ksi)
136	Flexural	297	(75)	834	(120.9)	602	(87.3)	821	(119.4)	443	(64.3)	903	(131.0)
		450	(350)	685	(99.4)	622	(90.3)	585	(84.8)	587	(85.2)	630	(91.5)
		505	(450)	548	(79.5)	426	(61.8)	538	(77.9)	487	(70.9)	645	(93.5)
	Short Beam Shear	297	(75)	50	(7.3)	32	(4.7)	29	(4.2)	30	(4.3)	30	(4.3)
		505	(450)	27	(3.9)	28	(4.1)	28	(4.0)	27	(3.9)	27	(3.9)
142	Flexural	297	(75)	1276	(185.0)	1365	(197.9)	1193	(172.6)	1227	(177.8)	1186	(172.0)
		450	(350)	588	(85.3)	595	(86.2)	505	(73.2)	471	(68.4)	489	(70.9)
		505	(450)	464	(67.3)	486	(70.5)	420	(60.9)	390	(56.8)	491	(71.4)
	Short Beam Shear	297	(75)	80	(11.6)	76	(11.7)	64	(9.3)	57	(8.3)	68	(9.9)
		505	(450)	47	(6.9)	24	(3.5)	21	(3.0)	18	(2.6)	20	(2.9)
145	Flexural	297	(75)	1296	(188.2)	1317	(191.0)	1214	(176.0)	1234	(178.8)	1248	(180.5)
		450	(350)	631	(91.6)	543	(78.8)	470	(68.3)	448	(65.0)	458	(66.5)
		505	(450)	490	(70.9)	499	(72.4)	402	(58.2)	375	(54.5)	458	(66.3)
	Short Beam Shear	297	(75)	88	(12.9)	83	(12.1)	73	(10.6)	75	(10.9)	69	(10.1)
		505	(450)	69	(10.0)	23	(3.3)	19	(2.7)	19	(2.7)	19	(2.7)

	Lam. 136 Vacuum Bag Cure	Lam. 142 Press Cure	Lam. 145 Autoclave Cure
% Fiber Volume	54.5	52.3	58.8
% Resin Content	39.2	41.6	34.4
Specific Gravity	1.38	1.51	1.50

Table 5-5. Preliminary Design Data, HT-S/P105A Graphite/Polyimide Composite

Test Temperature °K	(°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m²	(ksi)	Tensile Modulus GN/m²	(psi × 10⁶)	Compression Strength MN/m²	(ksi)	Compression Modulus GN/m²	(psi × 10⁶)	Flexural Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)
297	(75)	1	0°	0°	1151	(167)	134	(19.4)	592	(85)	127	(18.4)	1282	(186)	62.7	(8.95)
					1000	(145)	135	(19.5)	779	(113)	130	(18.9)	1482	(215)	65.4	(9.40)
					1110	(161)	137	(19.9)	758	(110)	117	(17.0)	1482	(215)	61.3	(8.78)
			Ave.		1089	(158)	135	(19.6)	710	(103)	125	(18.1)	1413	(205)	62.7	(9.04)
589	(600)				1255	(182)	161	(23.3)	507	(73)	126	(18.2)	1055	(153)	38.8	(5.63)
					1172	(170)	150	(21.8)	549	(79)	121	(17.5)	952	(138)	38.1	(5.52)
					1179	(171)	159	(23.0)	528	(76)	128	(18.6)	1117	(162)	38.8	(5.56)
			Ave.		1200	(174)	156	(22.7)	528	(76)	125	(18.1)	1041	(151)	38.8	(5.57)
297	(75)	1	0°	0°	606	(87)	129	(18.7)	158	(23)	-	-	613	(88)	8.76	(1.27)
			After 200 Hrs.		717	(104)	138	(20.0)	166	(24)	-	-	374	(54)	9.17	(1.33)
			at 589°K (600°F)		675	(97)	136	(19.7)	206	(30)	-	-	521	(75)	8.83	(1.28)
					668	(96)	134	(19.5)	180	(26)			500	(72)	8.89	(1.29)
589	(600)	1			710	(103)	123	(17.9)	166	(24)	-	-	549	(79)	5.21	(0.75)
					717	(104)	172	(24.9)	145	(21)	162	(23.4)	549	(79)	6.20	(0.89)
					883	(128)	-	-	152	(22)	148	(21.4)	479	(69)	5.49	(0.79)
			Ave.		772	(112)	148	(21.4)	152	(22)	154	(22.4)	500	(72)	5.63	(0.81)

Table 5-6. Preliminary Design Data, HM-S/P105A Graphite/Polyimide Composite

Test Temperature °K (°F)	Post-cure Cycle	Laminate Orientation (deg)	Test Orientation (deg)	Tensile Strength MN/m² (ksi)	Tensile Modulus GN/m² (psi×10⁶)	Compression Strength MN/m² (ksi)	Compression Modulus GN/m² (psi×10⁶)	Flexural Strength MN/m² (ksi)	Short Beam Shear Strength MN/m² (ksi)
297 (75)	1	0	0	395 (57.3)	209 (30.3)	374 (53.8)	172 (24.9)	703 (102.2)	48.6 (7.02)
				437 (63.4)	207 (30.0)	493 (71.3)	198 (28.7)	500 (72.0)	45.8 (6.58)
				- -	- -	507 (73.2)	198 (28.6)	689 (98.6)	50.0 (7.13)
		Ave.		416 (60.2)	208 (30.2)	458 (66.1)	189 (27.4)	633 (90.9)	47.9 (6.91)
(589) (600)				724 (105.3)	- -	311 (45.9)	- -	752 (108.9)	36.0 (5.16)
				738 (107.2)	- -	353 (50.8)	- -	606 (86.8)	35.3 (5.10)
				910 (131.6)	- -	304 (43.8)	- -	563 (80.7)	36.0 (5.23)
		Ave.		793 (115.4)		325 (46.8)		640 (92.1)	36.0 (5.16)
297 (75)	1	0°	0	430 (62.3)	207 (30.0)	180 (26.1)	- -	416 (60.1)	35.3 (5.14)
		After 200 hr at		542 (78.0)	222 (32.2)	193 (28.0)	- -	556 (79.8)	33.9 (4.87)
		589°K (600°F)		521 (75.4)	221 (32.1)	218 (31.6)	- -	592 (84.7)	33.2 (4.80)
		Ave.		500 (71.9)	216 (31.4)	197 (28.6)	- -	521 (74.9)	33.9 (4.93)
589 (600)				627 (90.3)	- -	176 (25.5)	- -	549 (78.7)	- -
				690 (100.4)	- -	219 (31.7)	- -	395 (56.9)	- -
				745 (107.3)	- -	156 (22.7)	- -	521 (75.2)	- -
		Ave.		689 (99.3)		183 (26.6)		486 (70.3)	

SECTION 6

FABRICATION DEMONSTRATION ARTICLES

One of the most important objectives of this program was that the graphite/polyimide system selected for generating the design data must be completely usable for making large, complex structures. In order to demonstrate that the composite system selected (HT-S/710) met this objective, several demonstration articles were fabricated. Tooling was available at Convair Aerospace that had been used to fabricate a full-scale graphite/epoxy space adapter. Several different and very complex parts were fabricated successfully, the first time. The fabrication procedures used to make these parts are discussed in the following paragraphs.

All the graphite/polyimide articles were considered advanced state-of-the-art components. Steel tooling was used in all cases. The steel tooling was designed to control composite thickness by having all tool mating surfaces bottom out at specific depths. The general fabrication outline used to make these articles consisted of five basic steps:

a. Mylar templates to control fiber orientation in individual ply layup.

b. Preplying several plies at a time, either flat and/or on part tool using vacuum bag pressure at room temperature.

c. Precompacting several plies at a time, either flat and/or on part tool using heat 353° K (175° F) and pressure vacuum plus 172 KN/m^2 (25 psig).

d. 120 style glass cloth bleeder system with a minimum of two layers of 1534 style glass cloth and glass mat as venting system.

e. Autoclave cure vacuum 760 mm (29 in. Hg minimum) pressure plus autoclave augmented pressure of 690 KN/m^2 (100 psi) maximum and 450°K (350° F) maximum cure temperature.

6.1 CONVAIR AEROSPACE FRAME SEGMENT

The steel tooling (excluding aluminum skin) shown in Figure 6-1 was used to assemble and cure the graphite/polyimide frame segment. Each half of the web section consisted of alternate layers of 5-mil HT-S graphite/710 polyimide (three layers each) with the HM-S oriented 90 deg to the HT-S. They were precompacted at 353° K (175° F) under vacuum bag plus 172 KN/m^2 (25 psi) pressure before assembly.

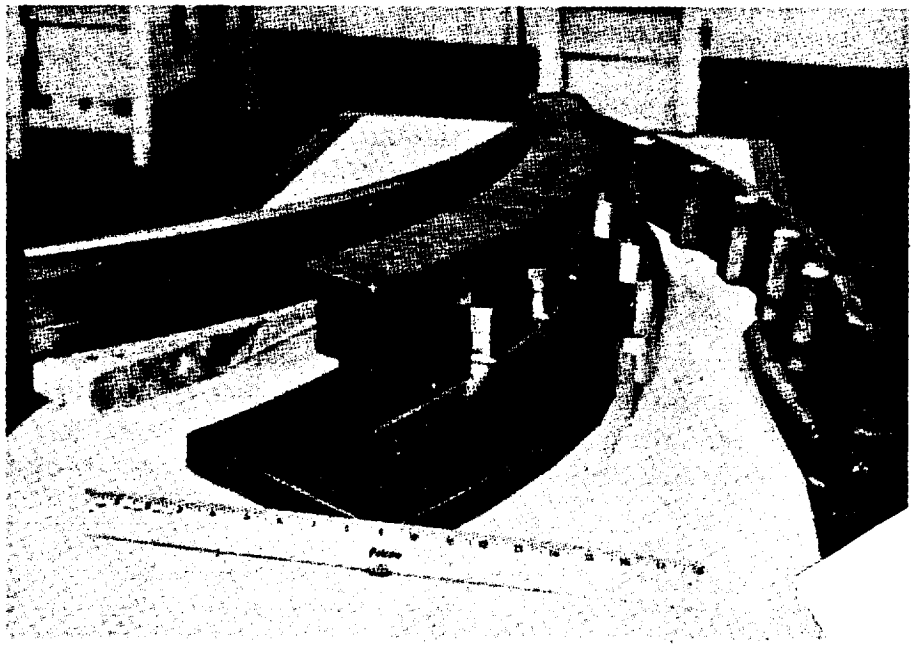
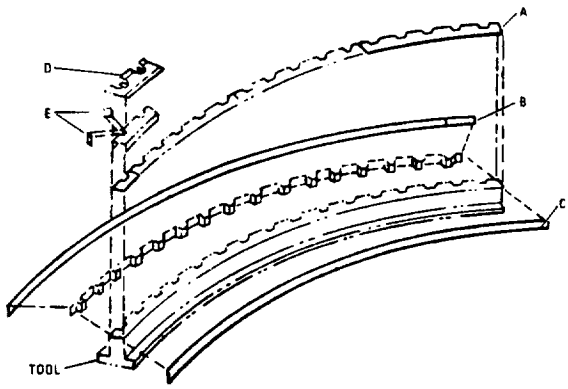


Figure 6-1. Graphite/Polyimide Frame Segment Tooling

The frame caps consisted of two layers of 5-mil GY-70 graphite/polyimide prepreg with one layer of 2-mil HM-S/710 sandwiched between the GY-70/710 layers and oriented 90 deg to them. The web section patterns and layup sequence are shown in Figures 6-2 and 6-3.

GY-70 HM-S/GY-70/710 cap strips were precompacted and located over the web flange sections, over which one layer of porous Armalon plus two layers of 120 style glass cloth were laid up before bolting together the tooling side plates. A combination of 1534 style glass cloth (cut on the bias to ensure minimum bridging of bagging material) and glass mat was used as venting material. The part was cured in an autoclave using a heat-up rate of 1 to 3° K/min (3 to 5° F) per minute, vacuum plus 690 KN/m^2 (100 psi) pressure with the pressure being applied after staging at 400° K (260° F) for 40 to 45 minutes, and final curing at 450° K (350° F) for two hours. The part was cooled under pressure to below 353° K (175° F). A stepwise postcure, terminating after eight hours at 644° K (700° F), was run in air in an oven.

The finished part represented a significant advancement in the state of the art, not only in polyimide composite uniformity, but in the shape complexity of the part and is shown in Figure 6-4.



A – 5-MIL HT-S/710 CENTER WEB SECTION
B – 5-MIL HT-S/710 OUTER WEB FLANGE SECTION
C – 5-MIL HT-S/710 INNER WEB FLANGE SECTION
D – 2-MIL HM-S/710 TRANSVERSE WEB SECTION
E – 2-MIL HM-S/710 TRANSVERSE WEB FILLERS

Figure 6-2. Web Section Patterns

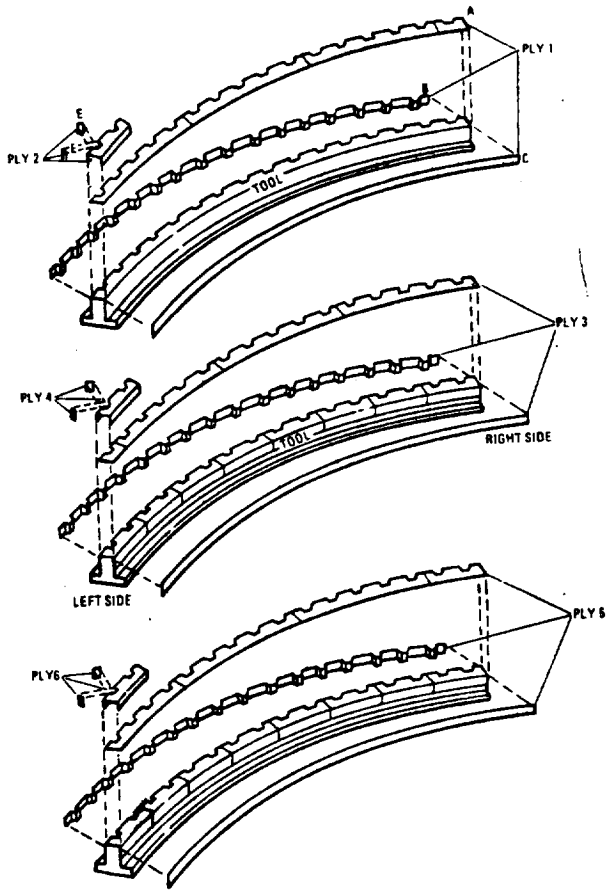


Figure 6-3. Layup Sequence

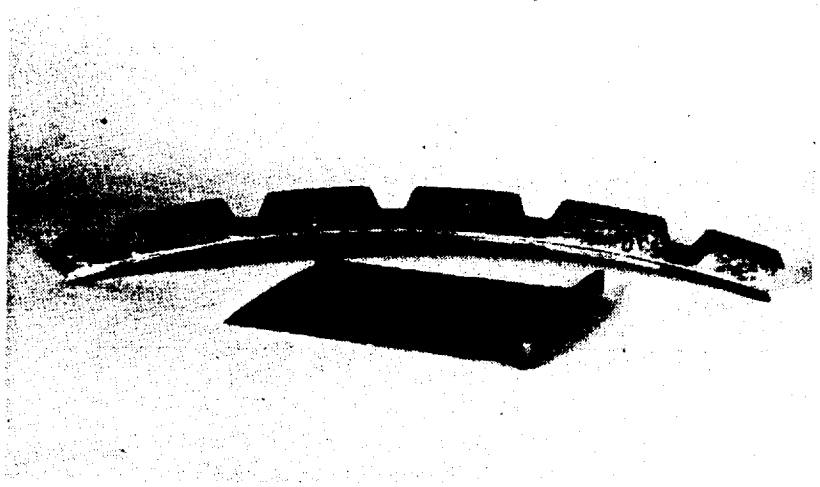


Figure 6-4. Graphite/Polyimide (RS-6234) Frame Segment and Adapter Skin Frame Segment

6.2 CONVAIR AEROSPACE ADAPTER SKIN FRAME SEGMENT

Both the frame segment and the adapter skin frame segment employed the same tooling used to make prototype graphite/epoxy process test articles. Mylar templates and tooling are shown in Figure 6-5. This skin frame segment was made using 5-mil HT-S graphite/710 polyimide prepreg oriented (0/±45) deg and consisted of a total of 32 plies plus a filler block that was precured and consisted of 40 plies oriented 0/90 deg. The HT-S/710 polyimide filler was made using the same basic fabrication steps as the parts being discussed. Figure 6-6 is a design drawing showing number of layers, location of filler, and final dimensions. The following preliminary manufacturing inspection specification was used to fabricate the adapter skin frame segment.

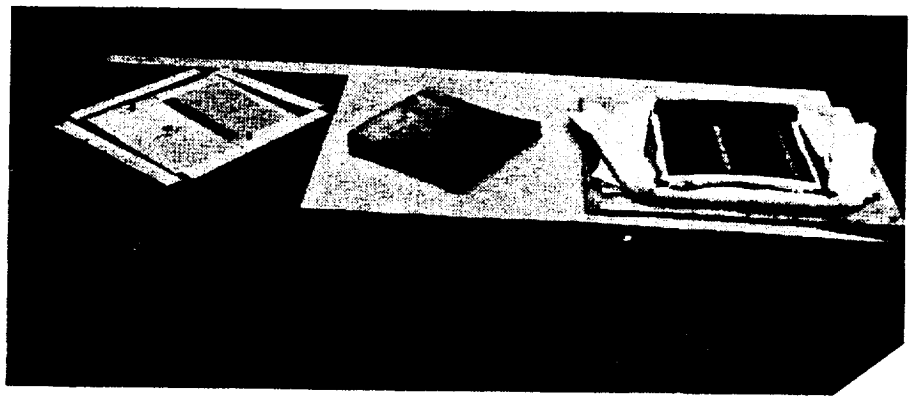


Figure 6-5. Skin Segment Tools

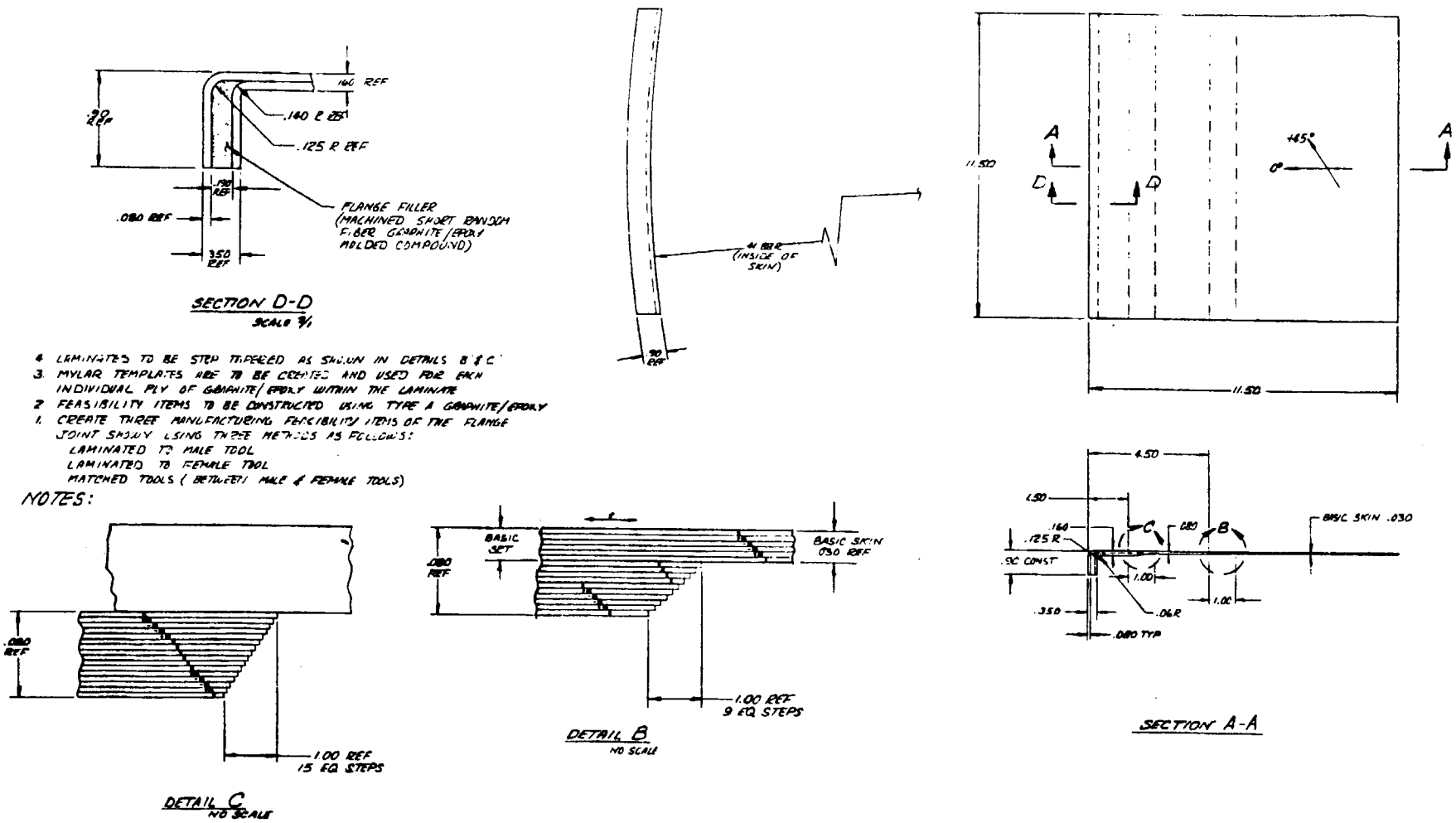


Figure 6-6. Tooling and Manufacturing Feasibility Specimen (All Dimensions in Inches Only for Clarification)

6.3 PRELIMINARY MANUFACTURING INSPECTION SPECIFICATION FOR HT-S/710 POLYIMIDE ADAPTER SKIN FRAME SEGMENT

The following specification is presented as an example of the type of planning that accompanies advanced composite parts during the fabrication cycle. This manufacturing Inspection Specification is for fabrication of one feasibility study part to be layed up and cured, by autoclave-vacuum bag method, on tool OV1-1102-400 PDMOP using HT-S/710 graphite/polyimide prepreg. Changes are to be written in ink and signed or initialed.

1. Clean mold and apply two coats of Frekote 33 mold release to working surfaces of the tools. Bake each coat at least 30 minutes at 464° K (375° F)(tool temperature).

2. Cut one kit of HT-S/710. The kit shall consist of 32 plies. Each individual ply of prepreg shall be positioned on its Mylar template, which shows fiber orientation by the direction of the arrows and is numbered as to the layup sequence; e.g., No. 1 (red) will be laid on the tool followed by No. 2 (blue), No. 3 (black), as shown in Figure 6-7, etc. Lay Teflon film between the Mylar template and the prepreg approximately 2.54 cm (1.00 inches) from the edge of part on the flange and extending beyond the edge of the Mylar (to provide easy removal of the Teflon film so that it will not be left in the laminate during subsequent layup).

   Templates with Red 1 are for zero (0-deg) orientation.
   Templates with Blue 2 are for plus (±45-deg) orientation.
   Templates with Black 3 are for minus (-45 deg) orientation

   Check off ply on sequence chart as it is laid up.

3. Position ply No. 1 on preplying tool. Carefully remove Mylar template and Teflon film, then set the template aside. Position No. 2 ply and template onto No. 1, remove the template and Teflon film, then place it on top of template No. 1.

   Position No. 3 ply and template onto No. 2; leave template No. 3 and Teflon film in position during RT preplying. Check templates to make sure layup is correct.

4. Position preplied layers (1, 2, and 3) on OV1-1102-500 PDMOP tool. Peel off Mylar, leaving the Teflon film in place and locate insert block OV1-1102-501 using locating pins and clamp it into place. Roll out.

5. Continue preplying (4, 5, and 6). Remove templates and Teflon film, except that for the No. 6 ply and stack them as before so that the layup can be checked for sequence and orientation.

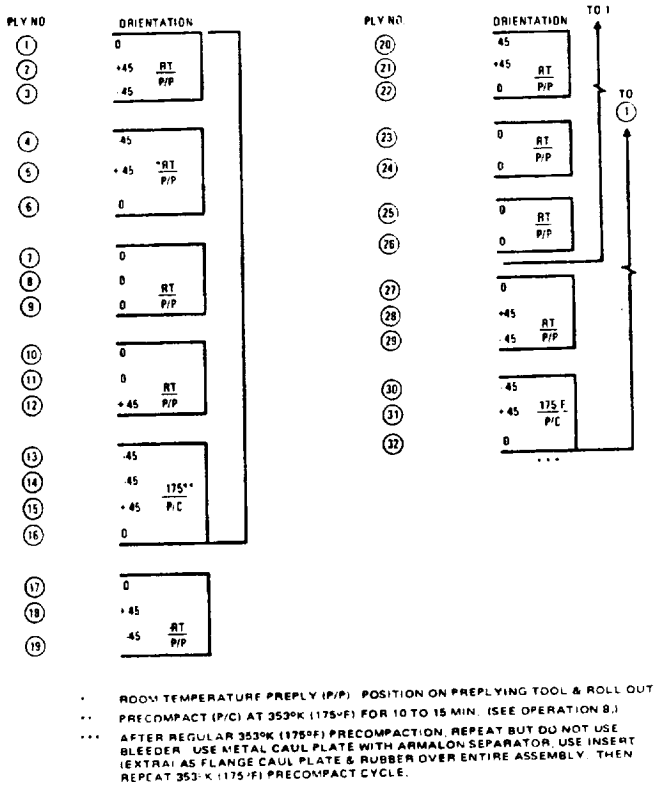


Figure 6-7. Layup Sequence.

6. Remove Template No. 3 and Teflon film, then continue layup on tool by positioning preplied layers (4, 5, and 6). Carefully remove Mylar template No. 6; leave the Teflon film in place. Recheck this six-ply layup and orientation using the color code and directional arrows on the Mylar templates OV1-1102-400 PDMOP. Use OV1-1102-401 insert block again as a forming tool by clamping it into place.

   Templates with Red 1 are for zero (0-deg) orientation.
   Templates with Blue 2 are for plus 45 (+45-deg) orientation.
   Templates with Black 3 are for minus 45 (-45 deg) orientation.

7. Remove insert block OV1-1102-501 and Teflon film and insert block.

8. Position the next preplied layers (7, 8, and 9) on ply No. 6. Carefully remove Mylar template, leaving the Teflon film in place. Stack the Mylar templates, as before, for inspection purposes.

9. Remove No. 9 template and Teflon film, then continue preplying and layup of the next three plies (10, 11, and 12).

10. Preply the next four plies (13, 14, 15, and 16). Remove Teflon film (No. 12 ply) then make layup (preplied 13, 14, 15, and 16).

    This step immediately following operation No. 12.

11. Precompact the 16 plies of prepreg using heat 353° K (175° F) vacuum pressure and autoclave pressure of 690 KN/m^2 (100 psi). Pressure is to be applied when temperature hits 344° K (160° F).

    a. Cut one ply of separator (3-mil porous Armalon) and bleeder (10 plies of style 120 and 2 plies of style 181 glass fabric) on the bias. Place the separator next to the laminate, the 120 fabric over the separator, then the 181 on top of the 120 plies. Place a 1/16 in. piece of sheet silicone rubber over the bleeder and tape it into place. Use locating pins where possible. Use sufficient vent material around layup to ensure removal of volatiles.

    b. Position one thermocouple between the tool and the layup.

    c. Bag with green Nylon Vac Pak rubber bag or equivalent using high-temperature extruded sealer and check for leaks.

    d. Place assembly in autoclave and set chamber temperature at 372° K (210° F) maximum; heat at 1 to 3° K (3 to 5° F) per minute to 353° K (175° F), as measured on the thermocouple located between the laminate and the tool. Automatically record thermocouple temperatures. Hold at 353° K (175° F) for 10 to 15 minutes.

    e. Remove from oven and cool, under vacuum pressure to room temperature. Remove bleeder and separator and retain along with heat cycle temperature chart and vacuum record.

12. Preply and lay up plies 17, 18, and 19. Check layup and orientation using the Mylar templates and color coding. Remove Teflon film from the laminate and peel both plies from the insert. Position OV1-1102-501 on the locating pins and continue layup.

13. Preply and lay up plies 17, 18, and 19 over OV1-1102-501 insert block and ply 16. Clamp insert block template over the layup as a forming tool.

14. Preply and lay up plies 20, 21, and 22 and layup on ply No. 19 after removing Teflon film.

15. Preply plies 23 and 24 and lay up on ply 22 after removing Teflon film

16. Preply plies 25 and 26 and lay up on ply 24 after removing Teflon film.

17. Preply plies 27, 28, and 29 and lay up on ply 26 after removing Teflon film.

18. Preply plies 30, 31, and 32 and lay up on ply 29 after removing Teflon film.

19. Apply separator and bleeder and precompact as in Operation 11.

20. Cut Armalon on the bias and cover layup.

21. Using Teflon separator, apply parting agent to metal caul plate and position it over the peel ply.

22. Locate on thermocouple between the laminate and the tool and one insulated on the aluminum plate

23. Provide sufficient cushioning around the tool to ensure that sharp corners do not puncture vacuum bag. Use Style 1534 glass fabric or equivalent and glass mat (between plies of 1534) for vent.

24. Use only vacuum hoses and fittings that have been inspected and approved for autoclave use.

25. Bag the part and check for leaks at 690 KN/m^2 (100 psi).

26. Cure in autoclave. Note: Chamber temperature shall not exceed 473° K (390° F).

    a. Vacuum = pressure complete cure. Apply 69 KN/m^2 (10 psi) immediately.

    b. Increase heat to 1 to 3° K (3 to 5° F) per minute to 380° K (225° F). Hold at temperature for 40 to 45 minutes then apply 690 KN/m^2 (100 psi) immediately.

    c. Increase heat to 450° F (350° F) and hold for 2 hours.

    d. Cool, while under pressure, to (160° F) or lower.

    e. Remove, de-bag and deliver part to Engineering. Save all cure charts (temperature, pressure, and vacuum) and bleeder for Engineering.

The fabrication of the HT-S/710 graphite/polyimide demonstration articles represents a major step forward for polyimide advance composites. The demonstration articles prove that complex structural parts can be fabricated from graphite/polyimide and are no longer limited to simple flat or slightly contoured parts.

SECTION 7

GRAPHITE/POLYIMIDE (HT-S/710) STRINGER TEST ARTICLES

Upon completing the fabrication demonstration articles, it was decided to fabricate and test 101.7 cm (40 in.) and 38.2 cm (15 in.) hat-shaped stringers. NASA/MSFC had in-house previously analyzed, fabricated, and tested some boron/epoxy stringers to the same set of load conditions. Both stringers were analyzed for the same loading conditions. The expected failure modes were column buckling for the longer stringers and crippling for the shorter stringers. Fabrication studies were conducted, tooling developed, and the stringers were fabricated and tested successfully.

The SQ-5 computer program was used to optimize the fiber orientation in both the facing and the hat-shaped stringer. It was found, after the first stringer was fabricated and tested, that a revised analysis was required to increase the adhesive bond shear strength. Both the original and revised analyses are presented in this report. The analyses are in standard engineering units only to reduce complexity. Figure 7-1 is a sketch of the stringer test article.

7.1 ORIGINAL ANALYSIS – GRAPHITE/POLYIMIDE STRINGER

1. HT-S/710 material required - 36 lbs.
2. For testing: Pot ends solid for 2 in. with Epon 934. Grind flat and parallel to +0.001 in. Test per NASA MSFC report, except skin edges to have 1-1/2 in. diameter split steel tubes clamped on.
3. Stringer designed to meet or exceed 42 kip load on a 40-in. long Euler column. Skin between stringer uprights buckles at about limit load (30 kips).

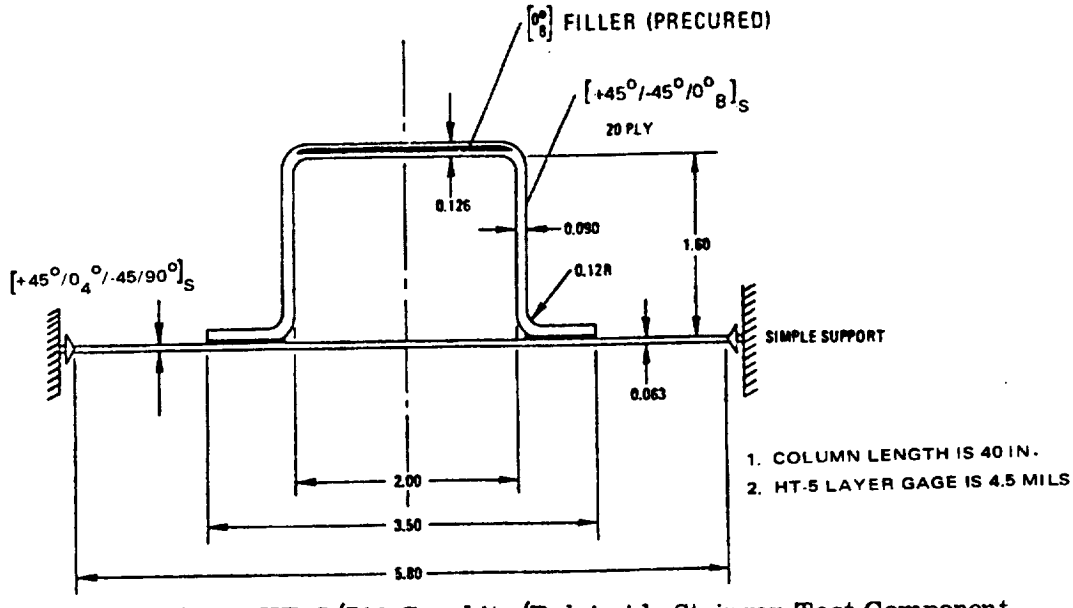
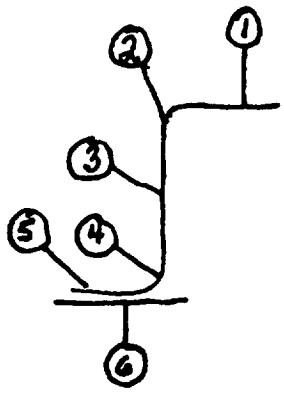


Figure 7-1. HT-S/710 Graphite/Polyimide Stringer Test Component

Graphite/ PI Column Behavior (HT-S)



Element	t	W #	r	Area	y	A · y	d	d^2	A · d^2	I_o
1	.126	.925	-	.1166	1.663	.194	.829	.686	.0800	-
2	.103	-	.170	.0288	1.560	.045	.726	.526	.0151	-
3	.090	1.134	-	.1021	.782	.080	.052	.002	.0002	.0110
4	.090	-	.170	.0240	.130	.003	.704	.495	.0119	-
5	.090	.535	-	.0482	.045	.002	.789	.622	.0300	-
6	.054	1.315	-	.0710	-.032	-.003	.879	.771	.0496	-
				.3907		.321			.1868	
									.0110	
									.1978	

Equivalent actual ① = .875, ② = 1.34

$I = .3956\ in^2$

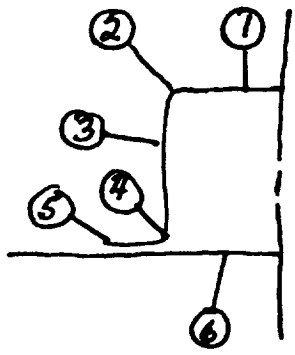
$$\bar{y} = \frac{.321}{.391} = .820$$

$$EI = 17.8\ (.39\ \ )\ 10^6 = 6.95 \times 10^6$$

$$Pcr = \frac{9.89\ (6.95)\ 10^6}{1600} = 42,800\#$$

$$\text{Ult MS} = \frac{42.8}{41.8} - 1 = {}^{+}.02$$

$$\sigma_{cu} = \frac{42,800}{.781} = 55,500\ PSI$$



Element	t	Radius	Length	Area Element
1	.126	-	.875	.1100
2	.090	.188	-	.0266
3	.090	-	1.134	.1020
4	.090	.170	-	.0241
5	.090	-	.535	.0481
6	.063	-	2.900	.1827
				.4935

x2 = .9870 in^2 length

Unit Weight = .987 .054 = .053# in/length

Δ wt (from Boron/epoxy) = .010# 16%

Skin: $E_x = 13.0 \times 10^6$
$E_g = 5.0 \times 10^6$
$G_{xy} = 4.0 \times 10^6$

Stringer Body: $17.8 \times 10^6 = E_x$
$2.0 \times 10^6 = E_g$
$3.4 \times 10^6 = G_{xy}$

Top: $E_x = 18.9 \times 10^6$
$E_g = 1.7 \times 10^6$
$G_{xy} = 2.8 \times 10^6$

Section Local Crippling - Graphite/ PI

For an orthotropic plate element with all edges simply supported:

$$\sigma_{cc} = \frac{2\pi^2}{t^3} \left(\frac{t}{b}\right)^2 \left(\sqrt{D_{11} - D_{22}} + D_{12} + 2D_{66}\right) = \frac{2\pi^2}{t^3} \left(\frac{t}{b}\right)^2 \bar{D}$$

$$= \frac{19.78}{t^3} \bar{D} \left(\frac{t}{b}\right)^2$$

Appropriate "D" values are:

Skin layup: $D_{11} = 795$, $D_{22} = 364$, $D_{12} = 116$, $D_{66} = 145$

$$\bar{D} = 944 \times \frac{000250}{000729} = 343$$

Basic Stringer Body: $D_{11} = 861$, $D_{22} = 219$, $D_{12} = 156$, $D_{66} = 185$

$$\bar{D} = 971$$

Stringer Cap: $D_{11} = 2660$, $D_{22} = 491$, $D_{12} = 334$, $D_{66} = 413$

$$\bar{D} = 2302$$

(For element code -see column check)

Element	b*	t	b/t	$(b/t)^2$	$\bar{D}$	t^3	σ_{cc} (ksi)
1	2.00	.126	15.9	252	2302	.00200	Hi -
2	-	-					-
3	1.134	.090	12.6	159	971	.000729	Hi -
4	-	-					-
5(1)	.535	.090	6.0	36	971	.000729	55.5
6a	1.150	.063	18.3	335	343	.000250	Hi -
6b	2.20	.063	35.0	1220	343	.000250	22.2

$\Sigma A \cdot E$: $.110\ (18.9)\ 10^6 = 2.08$

$+ \quad .200\ (17.8)\ 10^6 = 3.56$

$+ \quad .1827\ (13.0) 10^6 = \underline{2.32}$

8.02

% load in upper cap: 26.0 %

% load in body: 44.5 %

% load in skin: 29.5 %

At P = 42,800 #,

$P_{skin} = 42,800\ (.295) = 12,610$

Total skin area = (.3654)

Gross $\sigma_{skin} = 12,610 / .3654 = 35,000$ psi

1. Center Section Skin will buckle

at $\frac{22.2}{35} = 63\%$ of Ult. Load

or at $\frac{22.2}{35/1.4} = 90\%$ Limit Load

7.2 FABRICATION AND TOOLING DEVELOPMENT

A matched metal tool was designed and fabricated to make both the 101.7 cm (40 in.) and 38.2 cm (15 in.) hat-shaped stringers. The tooling is a four-piece matched steel tool that closes to stops, thus giving a part of expected thickness and performance. The cavity in the tool was calculated not only for the thickness of the part, but also for the inner and outer bleeder and separators. By using matched metal tooling, no thinning of the upper hat radius or wrinkling of the lower hat radius occurs. It is possible to make graphite/epoxy parts on male tooling only by the use of thin metal inserts between the layers of the prepreg, and because the volatiles released during the cure of the epoxy part is low, 3 to 5 per cent. However, in fabricating a polyimide part, approximately 20 per cent volatile is released, and the flow of the polyimide resin is generally higher than that of the epoxy resin systems. Thus matched steel tooling was selected over other alternative tooling concepts.

A 45.7 cm (18 in.) tool proofing test stringer was fabricated to check out both the tooling and fabrication procedures as shown in Figure 7-2. The graphite/polyimide part looked quite good, except that it was thicker than expected, denoting that the tool did not close. Upon flexing the hat-shaped stringer, two things became apparent. In the upper radius, a crack was initiated starting at the precured insert and extending down and around the radius. The second thing that happened was that the part started to delaminate at five places on the base of the stringer.

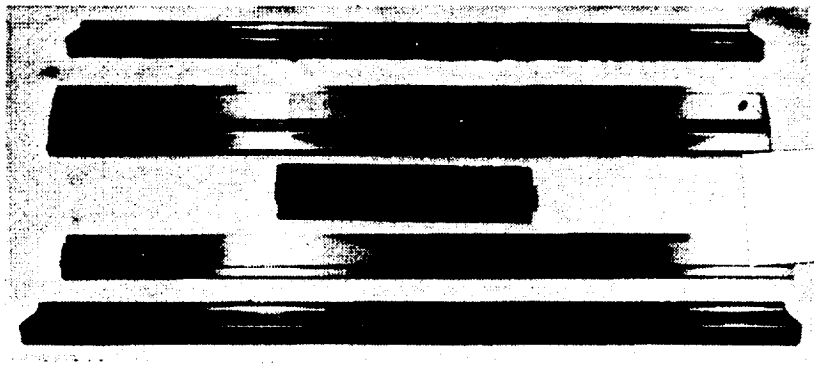


Figure 7-2. Graphite/Polyimide (HT-S/710) Stringer and Tooling

It was determined that the crack at the upper radius insert area was caused by the insert not being beveled along each edge. This caused a major stress concentration, and upon flexing the stringer, a crack was initiated. There are two ways to alleviate this problem: 1) bevel the precured insert, and 2) do not use a precured graphite/polyimide insert. Convair Aerospace selected the second method, since the part is not that thick.

The second item that was readily apparent was that the delaminations at the base of the hat-shaped stringer occurred right between each of the precompacted layers and between the inner ±45° plies and the unidirectional plies. The delamination at the intersection of the ±45° plies and the major 0° plies is caused by a thermal stress setup in the part during cure. This problem is easily eliminated by going to both an inner and outer ply orientation of +45°, -45°, -45°, +45° instead of +45°, -45°.

The other delaminations, however, were not as easy to understand and alleviate. As mentioned earlier, these delaminations occurred between each of the precompacted layers. All of the graphite/polyimide laminates made to date had either been precompacted as a total layup, or not precompacted at all. Based on previous Convair Aerospace graphite/epoxy experience and knowledge of the characteristics of the 710 resin, it was decided that precompacting several groups of plies of material was advantageous over placing large amounts of bleeder in the tool. The more bleeder used, the greater the chance of the bleeder to wrinkle, thus causing wrinkles in the part.

The test part was cut up and resin contents were determined for various locations on the stringer. Samples were taken from the base, side, and top of the hat. The resin content was found to be very uniform, 25 ±2 per cent. This is 3 to 7 percent below the resin content normally obtained in previous flat laminates. In examining the bleeder from the part, it was obvious that very little resin had flowed into the bleeder. The resin had been removed during the precompaction cycle. However, since a smaller number of plies (5 instead of 12) were being precompacted, more resin was being removed even though the precompaction bleeder had been changed to account for the fewer plies being precompacted at one time. Also, it seemed possible that resin was being preferentially removed from the plies next to the bleeder. Thus, when the various precompacted groups were laid up and cured, a resin-starved area could possibly be present at each of these layers. The lower the resin content, the lower the interlaminar shear strength will be, and thus delaminations could occur with little or no load applied to the part. Since there was no apparent quick method to determine the resin content at each of the delaminations, a precompaction study was developed. In this study, the effects of precompaction pressure and the use of wet inserts were investigated.

The details of the precompaction study have been previously discussed in Section 3 of this report and will not be repeated in this section. The precompaction and cure layups selected for the first test stringer are shown in Table 7-1. The first stringer to be fabricated and tested was a 101.7 cm (40 in.) article and failed at a load considerably below the design load. The failure was an adhesive bond/interlaminar shear failure between the 45° plies of both the sheet and the stringer. The adhesive being used was HT-424, a high-temperature epoxy-phenolic system.

Table 7-1. Precompaction and Cure Layups for HT-S/710 Graphite/ Polyimide Stringer

Precompaction Layup		Cure Layup
+45°		
-45°		Group I
-45°	Group I	2 ply insert
+45°		
$[0°]_4$		Group II
		2 ply insert
	Group II	Group III
$[0°]_6$		
$[0°]_4$		
+45°		
-45°	Group III	
-45°		
+45°		

Before proceeding any further with the fabrication of the remaining test stringers, a limited study was conducted to determine the best ply combinations that would give the highest lap shear adhesive strength.

Flat laminates with 0° plies and 45° plies were fabricated and lap shear specimens were tested. The test results are shown in Table 7-2. It is obvious from these

Table 7-2. Lap Shear Strengths of Graphite/Polyimide (HT-S/710) Adhesively Bonded with HT-424

Adherend Orientation	0° to 0°		0° to 45°		45° to 45°	
	KN/m^2	(psi)	KN/m^2	(psi)	KN/m^2	(psi)
Lap Shear Strength	2069	3000	724	1050	745	1080
	1793	2600	827	1200	633	910
	1896	2750	627	900	556	800
	1841	2670	627	900	535	770
	1965	2850	800	1160	647	928
	1913	2775	721	1042	623	898

results that the design of the stringer and sheet had to be revised such that there would be 0° plies on the outside of both the stringer and the sheet. A revised analysis was conducted, and the remaining five test articles were fabricated in accordance with the new configuration. The revised analysis is presented in the next section of this report.

7.3 REVISED HAT STRINGER ANALYSIS

The final hat stringer-skin configuration is analyzed for the design requirement of 42,800 pounds and a column length of 40 inches.

The cross section elements are checked for local stability and the overall section for Euler Column strength. The ultimate strength is calculated assuming any buckled panel (element) is inactive.

SECTION DATA (Figure 7-3)

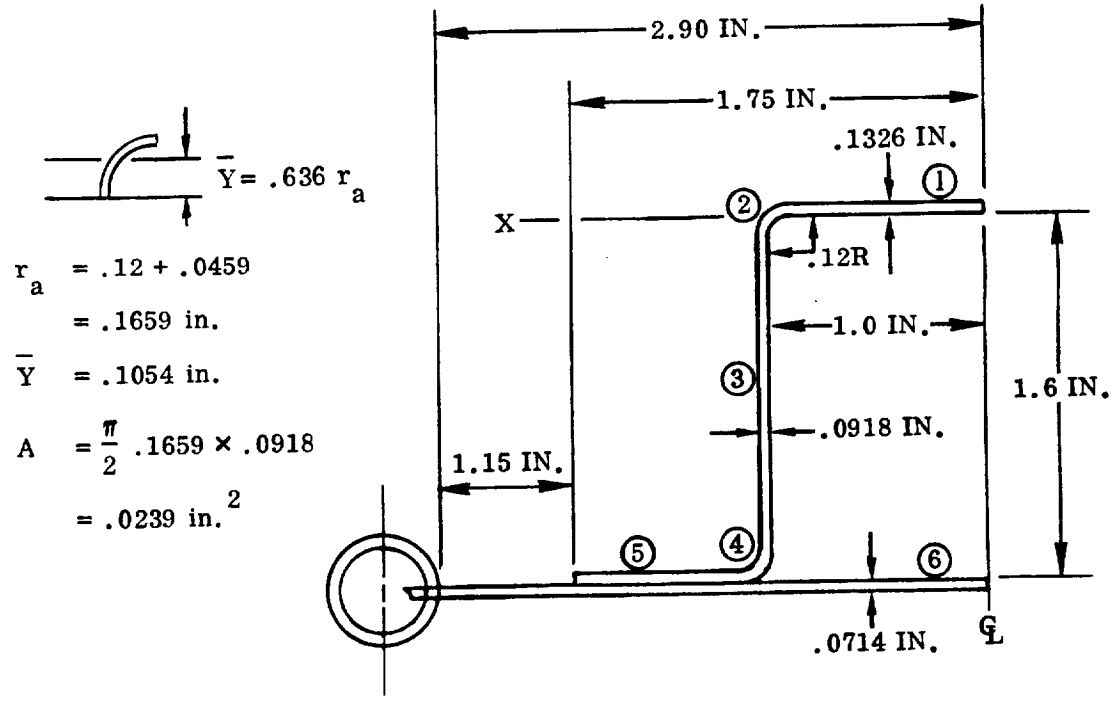


Figure 7-3. HT-S/710 Graphite/Polyimide Stringer Section Data

Element Area	E/106	η	A_e	Y	$A_e Y$	$A_e Y^2$	I_o
1. .1326 x .8 = .11668	15.448	1.197	.13965	-.0613	-.00856	.00052	
2. = .02390	12.904	1.0	.02390	.0146	.00034		
3. .0918 x 1.2682 = .11642	12.904	1.0	.11642	.7541	.08779	.06620	.0156
4. = .02390	12.904	1.0	.02390	1.5936	.03808	.06068	
5. .0918 x .6382 = .05858	12.904	1.0	.05858	1.5541	.09103	.14146	
6. .0714 x 2.90 = .20706	13.479	1.0446	.21629	1.6357	.35378	.57867	
.54654			.57874	.97186	.56246	.86313	.0156

$$I_o = \frac{.0918 \times \overline{1.2682}^3}{12} = .0156 \text{ in.}^4$$

$$I = .0156 + .86313 - .56246 \times .97186 = .33210/\text{half in.}^4$$

$$I_{tot} = .6642 \text{ in}^4 \qquad A_e = 1.15748 \text{ in}^2 \qquad \rho = \sqrt{\frac{.3321}{.57874}} = .7575 \text{ in.}$$

Element Material Properties are Calculated by Elastic Standard Lamination Theory

Graphite/Polyimide Hat Stringer Panels

$E_1 = 21 \times 10^6$ psi

$E_2 = 1.0 \times 10^6$ psi

$\nu = .25$

$G = .7 \times 10^5$ psi

Skin $\left[0°/45°/0_3/-45/90\right]_s$ 14 ply @ .005

θ	t		
+45	.0102	E_x	13.479×10^6
-45	.0102	E_y	5.235×10^6
0	.0408	ν	.295
90	.0102	G_{xy}	2.0478×10^6

$t = 14 \times .0051 = .0714$ in.

Hat Sides and Flange $\left[0/\pm45°/0_4/\pm45°\right]_s$ 18 ply

θ	t		
+45	.0204	E_x	12.904×10^6
-45	.0204	E_y	3.000×10^6
0	.0510	ν	.692
		G_{xy}	2.7849×10^6

$t = 18 \times .0051 = .0918$ in.

Hat Top $\left[0/\pm45°/0_8/\pm45°\right]_s$ 26 ply

θ	t		
+45	.0204	E_x	15.448×10^6
-45	.0204	E_y	2.472×10^6
0	.0918	ν	.640
		G_{xy}	2.1434×10^6

$t = 26 \times .0051 = .1326$ in.

Panel	Skin	Hat Sides	Hat Crown
D_{11}	462.673	571.574	181.180
D_{22}	95.463	349.828	887.959
D_{12}	63.196	266.733	657.714
D_{16} 26	53.219	5.322	5.322
D_{66}	76.823	295.696	744.999

Orthotropic Simply Supported Plates (Reference Design Guide Eq. 4.3.2.9)

$$F_{cr} = \frac{2\pi^2}{t\,b^2}\left[\sqrt{D_{11}D_{22}} + D_{12} + 2D_{66}\right]$$

Skin Panels

1. t = .0714 in; b = 1.15 in.

$$F_{cr} = 2\pi^2 \left(\frac{1}{.0714 \times 1.15^2}\right)\left[(462.673 \times 95.463)^{1/2} + 63.196 + 2 \times 76.823\right]$$

$$19.739(10.5902)(427.004) = 89261 \text{ psi}$$

2. t = .0714 in; b = 2.0918 inches

$$F_{cr} = 89,261 \left(\frac{1.15}{2 \times 1.0459}\right)^2 = 26978 \text{ psi}$$

Hat Crown t = .1326 in; b = .88 in.

$$F_{cr} = (19.739)\left(\frac{1}{.1326 \times .88^2}\right)\left[(2181.18 \times 887.959)^{1/2} + 657.714 + 2 \times 744.999\right]$$

$$= (19.739)(9.7385)\left[3539.4010\right] = 680373 \text{ psi}$$

Hat Side Panels $t = .0918$ in; $b = 1.268$ in.

$$F_{cr} = (19.739)\left(\frac{1}{.0918 \times \overline{1.268}^{2}}\right)\left((571.574 \times 349.828)^{1/2} + 266.713 \div 295.696\right)$$

$$= (19.739)(6.775)(1305.2656) = 174555 \text{ psi}$$

The skin center panel buckles at 26,978 psi and will be neglected for the ultimate strength check.

	A_e	Y	AY	AY^2	I_o
Full Section	.57874		.56246	.86313	.0156
(46) - 1.2118 x .0714 x 1.044	.08980	1.6357	-.14692	-.24032	
	.48894	.84988	.41554	.62281	.0156

Equivalent Area $A_e = 2 \times .4884 = .97788 \text{ in}^2$

$I = .0156 + .62281 - .41554 \times .89988$

$= .285252$/side

$I_{tot} = 2 \times .28525 = .57050 \text{ in}^4$

At ultimate design load

$$F_c = 42,800/(2 \times .48894) = 43,768 \text{ psi}$$

Skin Buckles at:

$$P_{cr} = 26978 \times 2 \times .57874 = 31227 \text{ lb.}$$

$$P_{cr} = \frac{31227 \times 1.4}{1.044 \times 42800} = 97.8\% \text{ limit}$$

With buckled skin

$$\rho = (.5705/.97788)^{1/2} = .583 \text{ inches}$$

For 40-inch Euler Column (ℓ)

$$F_{col} = \frac{\pi^2 EI}{\ell^2} \qquad \frac{\pi^2 \times 12.904 \times .5705}{\overline{40}^{2}}$$

$$= 45,400 \text{ psi}$$

Design is critical for column

$$M.S. = \frac{45400}{43768} - 1 = \underline{+.035}$$

The column strength is critical with a margin of +0.035. The most critical buckling element is the skin panel on $\mathbb{Q}_L$. The $\mathbb{Q}_L$ skin panel will buckle at 97.8 per cent of limit load without any benefit from the available fixity of continuity over the hat support point. The revised configuration is shown in Figure 7-4.

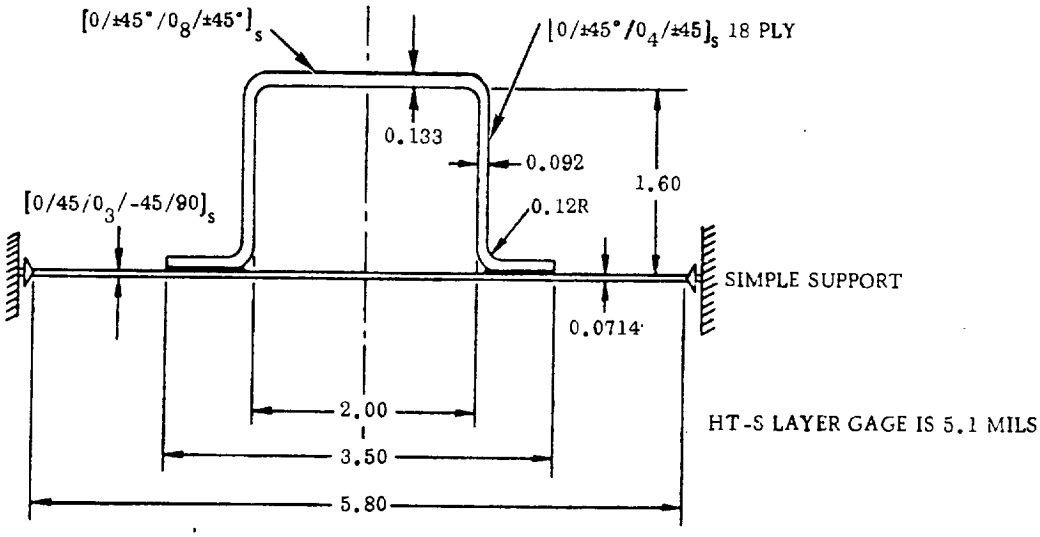
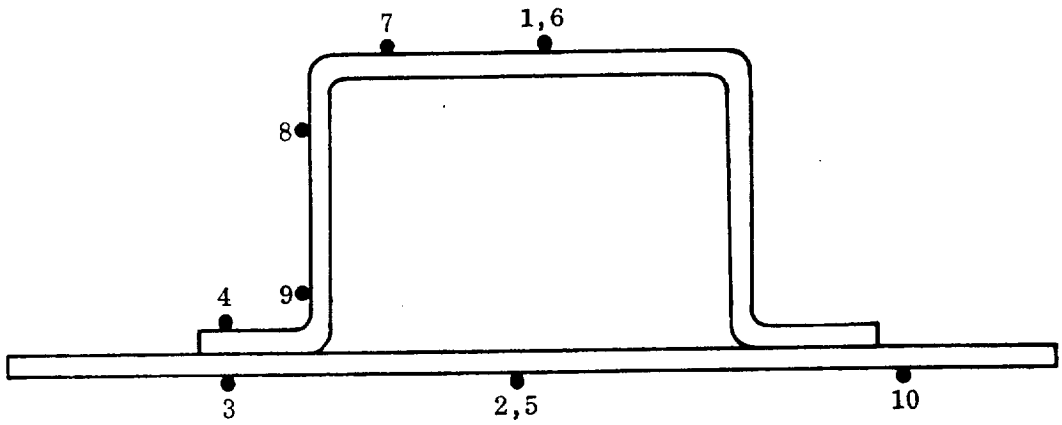


Figure 7-4. Revised HT-S/710 Graphite/Polyimide Stringer Test Component

7.4 GRAPHITE/POLYIMIDE STRINGER TEST RESULTS

Each of the remaining five test stringers were potted at both ends with an epoxy compound, and then the potting compound was machined flat and parallel to ±0.013 cm (±0.005 in.). Split tubes were placed on the edge of the skin to eliminate localized buckling. Ten strain gages as shown in Figure 7-5 were placed on each of the test articles at the midpoint. The test results obtained are presented in Table 7-3 and show that the analysis was slightly conservative in that the ultimate load for the 101.7 cm (40 in.) stringers was exceeded by 5 and 15 percent. The test results obtained on the shorter specimens were excellent in that the three test specimens, cured separately, gave very reproducible results. Failure modes were as expected, and by changing the outer ply orientation, no further adhesive or delamination failures occurred.



NOTES:

1. Gages 1 & 6 and 2 & 5 are combined in order to determine Poisson's Ratio.
2. Gages 1, 2, 3, 4, 7, 8, 9, and 10 are placed along the length of the test article.
3. Gages 5 & 6 are transverse.

Figure 7-5. Strain Gage Locations for Graphite/Polyimide Test Stringers

Table 7-3. Test Results of Graphite/Polyimide (HT-S/710) Skin Stringer Test Components

Specimen No.	Stringer Length cms.	(in.)	Failure Load 10^3 kg	(10^3 lbs)	Failure Mode
1	38.2	(15)	25.3	(55.7)	Crippling
2	38.2	(15)	26.1	(57.5)	Crippling
3	38.2	(15)	26.8	(59.0)	Crippling
4	101.7	(40)	10.7	(23.8)*	-
5	101.7	(40)	20.4	(44.9)	Column Buckling
6	101.7	(40)	22.4	(49.3)	Column Buckling

Design Ultimate Buckling Load 19440 kg. (42,800 lb)
*Failure due to ply orientation

SECTION 8

BORON/POLYIMIDE COMPOSITE DEVELOPMENT

Boron/polyimide composite systems are applicable for structures that require high strength and stiffness with high temperature capability. Limited work has been done toward developing a usable boron/polyimide system for making large complex parts. In this study, both 5.6- and 4.0-mil-diameter boron fiber were evaluated with both the 703 and P105A polyimide resin systems. Processing studies and some preliminary test data were developed for each of the two resin systems.

8.1 FABRICATION DEVELOPMENT

Two boron/polyimide prepreg systems, B/703 and B/P105A, were obtained with the 5.6-mil boron fiber. It was determined that cure cycles previously used with glass and graphite fiber reinforcements would not work for the B/703 system. The fabricated laminates were so low in resin content (less than 22 percent) that they fell apart when attempts were made to machine test specimens. Buchfield and Kollmansberger (Reference 8-1) had previously developed a cure cycle for B/703 composites that was applicable to making flat panels. All their work had been done using the 4.0-mil boron fiber.

A laminate was made using their recommended cure cycle using 5.6-mil B/703 prepreg. The test results obtained are shown in Table 8-1 with data from the literature

Table 8-1. Boron/Polyimide (B/703) Fiber Evaluation Study

Test Temperature		4.0-Mil Boron*				5.6-Mil Boron			
		Flex. Strength		Sbs Str.		Flex. Strength		Sbs Str	
°K	(°F)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
297	(75)	1786	(259)	138	(20.0)	1979	(287)	79	(11.4)
	(550)	1420	(206)	70	(10.2)	1503	(218)	56	(8.1)
589	(600)	-	-			986	(143)	57	(8.2)
*Ref. 8-1									

for 4.0-mil B/703. The flexural data compares very well; however, the short-beam shear data for the composite having 5.6-mil reinforcement was significantly lower. The resin content was also lower for the 5.6-mil boron fiber composite, which probably caused the lower short-beam shear strength. It was decided at this point

that boron prepreg with the 4.0-mil fiber would be used for the remainder of the program, since the resin content and laminate shear properties were higher for this type of composite.

Boron/polyimide prepreg with 4.0-mil boron fiber and the P105A and 703 polyimide resins were purchased. A bleeder study was conducted in which the bleeder arrangement and amount were varied. The autoclave curing pressure was also varied. Flexural and short-beam shear tests were then conducted to evaluate the cured laminates. Test results for the B/703 composite laminates and for the B/P105A system are presented in Tables 8-2 and 8-3. The data show that the B/P105A composite system was significantly better than the B/703 composite system. The B/P105A composite system was selected for further evaluation.

Eight- and 10-ply unidirectional and 8-ply ±45° B/P105A laminates were made using the B/P105A system. Tensile strengths and modulus were determined on the 8-ply laminates; flexural and interlaminar shear strengths were determined on the 10-ply laminate at 297° K (75° F) and 589° K (600° F). The cure cycle was the same as that reported earlier for the graphite/P105A system. The boron prepreg had substantial tack and drape, indicating that material and processing conditions are applicable for making large complex parts. Test results are presented in Table 8-4.

Based on this cursory evaluation, it is believed that the 4.0-mil B/P105A composite system is one of engineering interest and should be further characterized. The number of fibers per inch, resin content, and cure cycle optimization should be investigated before detailed design data is established.

Table 8-2. Cure Pressure and Bleeder Evaluation of B/703 Polyimide Composites (Room Temperature)

Cure Pressure		Flexural Strength		Short Beam Shear Strength		Flexural Strength		Short Beam Shear Strength		Flexural Strength		Short Beam Shear Strength		Flexural Strength		Short Beam Shear Strength		Flexural Strength		Short Beam Shear Strength	
KN/m^2	(psi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)	MN/m^2	(ksi)
200	(29)	1131	(164)	69	(9.9)	1269	(184)	63	(9.0)	1255	(182)	57	(8.2)	1110	(161)	61	(8.8)	1310	(190)	59	(8.5)
		1289	(187)	68	(9.8)	1393	(202)	61	(8.8)	1186	(172)	56	(8.1)	1165	(169)	63	(9.0)	1289	(187)	69	(10.0)
		1338	(194)	71	(10.3)	1379	(200)	61	(8.8)	1062	(154)	60	(8.6)	1165	(169)	61	(8.7)	1193	(173)	65	(9.4)
		1255	(182)	69	(10.0)	1345	(195)	62	(8.9)	1172	(170)	58	(8.3)	1145	(166)	62	(8.8)	1262	(183)	65	(9.3)
690	(100)	444	(64)	43	(6.2)	479	(69)	41	(5.9)	381	(55)	42	(6.0)	381	(55)	42	(6.1)				
		402	(58)	41	(5.9)	381	(55)	49	(7.1)	367	(53)	44	(6.3)	374	(54)	47	(6.8)				
		430	(62)	40	(5.7)	493	(71)	39	(5.6)	458	(66)	51	(7.4)	423	(61)	53	(7.6)				
		423	(61)	41	(5.9)	451	(65)	43	(6.2)	402	(58)	46	(6.6)	395	(57)	48	(6.9)				
Bleeder* Arrangement		**4/181/T**				**4/181/T 2/181/B**				**6/181/T**				**5/181/T 1/181/B**				**4/181/T 1/181/B**			

*Example 4/181/T = 4 Plies 181 Glass Cloth on Top of Lay Up, No Bleeder on the Bottom.

Table 8-3. Cure Pressure and Bleeder Evaluation of B/P105A Polyimide Composites (Room Temperature)

Cure Pressure KN/m^2	(psi)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)	Flexural Strength MN/m^2	(ksi)	Short Beam Shear Strength MN/m^2	(ksi)
200	(29)	1903	(276)	54	(7.7)	1600	(232)	53	(7.6)	2000	(290)	49	(7.1)	1572	(228)	44	(6.3)	1531	(222)	42	(6.1)
		1779	(258)	49	(7.0)	1544	(224)	58	(8.4)	1613	(234)	50	(7.2)	1110	(161)	46	(6.6)	1386	(201)	44	(6.4)
		1517	(220)	53	(7.6)	1551	(225)	58	(8.4)	1296	(188)	49	(7.0)	1462	(212)	49	(7.0)	1110	(161)	44	(6.3)
		1731	(251)	52	(7.5)	1565	(227)	56	(8.1)	1634	(237)	49	(7.1)	1379	(200)	46	(6.6)	1345	(195)	44	(6.3)
Specific Gravity		2.05				1.96				1.99				1.98				1.97			
% Resin Content		19.3				22.1				28.2				23.6				27.2			
690	(100)	1855	(269)	70	(10.1)	1765	(256)	85	(12.3)	1993	(289)	74	(10.8)	1779	(258)	77	(11.2)	1958	(284)	78	(11.3)
		1793	(260)	72	(10.5)	1965	(285)	85	(12.3)	1931	(280)	76	(11.1)	2006	(291)	80	(11.6)	2131	(309)	86	(12.5)
		1910	(277)	73	(10.6)	2096	(304)	77	(11.2)	1627	(236)	74	(10.7)	1827	(265)	81	(11.7)	1875	(272)	79	(11.5)
		1855	(269)	72	(10.4)	1944	(282)	83	(12.0)	1848	(268)	75	(10.9)	1869	(271)	79	(11.5)	1986	(288)	81	(11.7)
Specific Gravity		2.00				1.99				2.02				2.01				1.99			
% Resin Content		20.4				18.8				24.6				22.5				19.3			
1380	(200)					1896	(275)	56	(8.1)	2179	(316)	61	(8.8)								
						1772	(257)	54	(7.7)	1882	(273)	61	(8.8)								
						1765	(256)	61	(8.7)	2069	(300)	60	(8.7)								
						1813	(263)	57	(8.2)	2041	(296)	61	(8.8)								
Specific Gravity						1.94				1.94											
% Resin Content						25.4				29.4											
Bleeder* Arrangement		4/181/T				4/181/T 2/181/B				6/181/T				5/181/T 1/181/B				4/181/T 1/181/B			

*Example 4/181/T = 4 Plies 181 Glass Cloth on Top of Lay up, No Bleeder on the Bottom.

Table 8-4. Mechanical Properties for Boron/Polyimide B/P105A Composites

Test Temperature °K	(°F)	Laminate Orientation °	Test Direction °	Tensile Strength MN/m²	(ksi)	Tensile Modulus GN/m²	(10⁶ psi)	Flexural Strength MN/m²	(ksi)	Short Beam Shear Strength MN/m²	(ksi)
297	(75)	0°	0°	1427	(207)	185	(26.8)	1956	(284)	83.4	(12.1)
				1489	(216)	167	(24.2)	1979	(287)	81.4	(11.8)
				1634	(237)	175	(25.4)	1937	(281)	78.6	(11.4)
			Ave	1517	(220)	176	(25.5)	1958	(284)	81.4	(11.8)
589	(600)			1207	(175)	212	(30.7)	1655	(240)	51.4	(7.4)
				1345	(195)	-	-	1669	(242)	49.3	(7.1)
				1379	(200)	223	(32.4)	1682	(244)	49.3	(7.1)
				1214	(176)	196	(28.4)	-	-	-	-
			Ave	1282	(186)	210	(30.5)	1669	(242)	50.0	(7.2)
297	(75)	±45	0°	80.7	(11.7)	91.7	(1.33)				
				77.9	(11.3)	86.2	(1.25)				
				80.0	(11.6)	95.2	(1.38)				
			Ave	79.3	(11.5)	91.0	(1.32)				
589	(600)				(9.8)	107.6	(1.56)				
				80.0	(11.6)	89.6	(1.30)				
				73.8	(10.7)	104.1	(1.51)				
			Ave	73.8	(10.7)	100.7	(1.46)				
Laminate			0° (Ten)	±45°		0° (Flex. & Shear)					
Specific Gravity			2.05	1.99		2.00					
Resin Content			26.4	29.2		28.2					

SECTION 9

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations have been reached based on the work conducted on this program as well as related programs.

1. A serious high temperature 450°K (350°F) strength problem caused by moisture exists with graphite/epoxy composite systems.

2. The 710 polyimide resin was found to be useful over the temperature range of 75°K (-320°F) to 589°K (600°F) for periods up to 400 hours.

3. Low void laminates 5 - 10% were fabricated from the various types of graphite/710 prepregs by vacuum-bag, press, and autoclave curing.

4. The acid equivalent of the starting 710 polyimide resin should be between 520 and 540 for the cure cycle developed on this program to work.

5. The graphite/710 polyimide composite system is one of a few commercially available systems that can be used for long times at 589°K (600°F).

6. The high temperature strength properties of the graphite/polyimide (710) composite system are not affected by moisture.

7. The graphite/polyimide (710) composite system and processing is compatible with all common graphite fibers except the ultra high strength fibers.

8. Fatigue and creep properties of the graphite/polyimide (710) laminates were not affected by the voids present in the laminate.

9. Thick laminates up to 5.08 cm (2 inches) thick were fabricated from the graphite/polyimide (710) system at one time. Highly complicated parts were fabricated using a number of different graphite fibers and the 710 resin.

The following technical problems still exist and require further study.

1. High temperature joint data should be developed between composite to composite and metallic to composite substrates.

2. High temperature graphite/polyimide crippling data should be evaluated.

3. The effects of thermal cycling on graphite/polyimide composites need to be determined.

4. Tension-compression and compression-compression fatigue data at both room and elevated temperatures should be developed for graphite/polyimide composites.

5. Modification of the 710 polyimide resin needs to be studied so that there is less dependence on the acid equivalent number.

6. A usable high-temperature boron/polyimide composite system needs to be developed.

SECTION 10

REFERENCES

2-1 Advanced Composite Technology Fuselage Program, AFML Contract F33615-69-C-1494, Convair Aerospace Division of General Dynamics, dated April 1969.

2-2 Flightworthy Graphite Reinforced Aircraft Primary Structural Assemblies, AFML Contract F33615-69-C-1490, Northrop Corporation Aircraft Division, started April 1969.

2-3 M. Varlas, Development of Epoxy-Graphite/Asbestos Laminates, GDC-ERR-1422, Convair Aerospace Division of General Dynamics, 1969.

2-4 M. Varlas, Advanced Graphite Reinforced Resin Composites, IRAD 111-7006-111, Convair Aerospace Division of General Dynamics, 1970.

2-5 Advanced Composite Applications for Spacecraft and Missiles, AFML Contract F33615-70-C-1442, Convair Aerospace Division of General Dynamics, started April 1970.

2-6 Development of Carbon Composite Structural Elements for Missile Interstage Application, AFML Contract F33615-67-C-1641, The Boeing Company.

2-7 Celanese Corporation, published literature, 1970.

2-8 Private communication with Dr. T. P. Airhart, Convair Aerospace Division of General Dynamics, 14 April 1970.

2-9 Advanced Composite Applications for Spacecraft and Missiles, AFML Contract F33615-70-C-1442 Convair Aerospace Division of General Dynamics Quarterly Report #2 October 1970.

3-1 D. R. Beeler, and V. A. Chase, "Advanced Polyimide Composites," SAE Conference, October 1968.

3-2 R. W. Vaughn, "Glass Reinforced P13N Polyimide Laminates Having Improved Processing Characteristics," Fifteenth National SAMPE Conference, April 1969.

3-3 Fiberite Corporation, published data, 1970.

3-4 "Structural Design Guide for Advanced Composite Applications," 1969, AFML Contract F33615-69-C-1368.

8-1 E. B. Birchfield and R. Kollmansberger, Develop Fabrication/Processing Techniques for High Temperature Advanced Composites for Use in Aircraft Structures, Air Force Technical Report AFML-TR-72-91, July 1972, McDonnell Aircraft Company.

APPENDIX A

GRAPHITE/EPOXY PROCESS DATA

HT-S/X-904

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE	POST CURE CYCLE 1	2	3	4
1	179.7	185.8	137.3	148.6
	232.5	227.1	166.8	128.2
	231.7	233.8	143.8	142.5
	214.6	215.6	149.5	139.8
2	281.6	249.2	281.7	245.8
	263.1	222.3	269.9	244.3
	255.2	255.8	253.5	234.3
	266.6	242.4	268.4	241.5
3	211.0	249.9	219.4	218.3
	239.2	250.2	213.4	218.0
	234.1	247.4	242.8	247.2
	228.1	249.2	225.2	227.8
4				

Preceding page blank

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77°K (-320°F)

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	118.0 200.0 181.4 166.3	164.1 176.7 173.9 171.6	109.8 110.0 136.1 118.6	102.1 119.7 119.9 113.9
2	166.7 133.3 227.0 175.6	155.6 156.6 219.9 177.4	180.1 197.1 188.8 188.7	210.1 200.5 197.6 202.7
3	198.8 215.4 242.8 219.0	216.2 245.6 217.0 226.3	123.3 170.1 184.4 159.2	144.2 205.4 133.0 160.9
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 450°K (350°F)

CURE CYCLE	POST CURE CYCLE 1	2	3	4
1	41.3	53.9	167.2	142.3
	52.9	48.7	141.9	139.8
	41.8	37.6	156.0	137.3
	45.3	46.7	155.0	139.8
2	41.0	59.5	62.0	26.0
	35.2	39.9	53.6	22.4
	32.8	35.5	57.6	23.4
	36.3	45.0	57.7	24.0
3	39.2	47.8	119.8	155.3
	40.2	51.9	131.0	161.9
	40.0	37.9	157.9	167.2
	39.8	45.9	136.2	161.4
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Transverse Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	12.4 12.3 10.7 11.8	13.8 10.7 11.3 11.9	7.3 8.4 6.6 7.4	6.4 6.4 5.9 6.2
2	8.8 9.0 11.2 9.6	13.0 11.8 12.3 12.4	9.0 8.9 8.7 8.9	7.4 5.9 6.0 6.4
3	9.3 9.8 9.1 9.4	8.8 8.8 10.0 9.2	6.4 7.0 7.1 6.9	4.4 5.2 5.2 5.0
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	9.6 9.4 8.1 9.0	10.6 11.4 11.3 11.1	10.8 11.1 10.1 10.7	7.0 8.7 9.2 8.3
2	11.6 10.1 10.0 10.6	12.7 13.6 13.6 13.3	11.8 14.3 14.4 13.5	8.4 10.0 7.4 8.6
3	6.9 8.3 7.7 7.6	10.3 9.1 10.8 10.1	10.0 10.1 10.6 10.2	8.4 8.9 7.5 8.3
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE 77°K (-320°F)

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	14.6 20.3 14.3 16.4	9.9 11.2 10.8 10.7	5.1 6.6 6.9 6.2	5.2 4.4 5.2 4.9
2	16.5 16.7 10.7 14.6	9.4 15.7 11.6 12.2	9.4 8.8 8.3 8.8	6.6 5.4 6.2 6.1
3	9.0 9.9 11.3 10.1	10.0 10.4 10.1 10.2	2.9 3.5 2.7 3.0	7.5 7.4 7.7 7.5
4				

PRESS CURE STUDY

HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE 450°K (350°F)

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	1.9 2.0 1.9 1.9	2.1 2.1 2.2 2.1	5.3 5.3 4.7 5.1	5.1 5.3 4.9 5.1
2	1.7 1.8 1.7 1.7	2.6 2.2 2.4 2.4	2.4 2.0 2.3 2.2	1.8 2.0 2.2 2.0
3	2.6 1.8 1.8 2.1	1.9 2.0 2.0 2.0	4.8 5.2 4.4 4.8	5.4 5.6 4.4 5.2
4				

PRESS CURE STUDY

HT-S/X-904

TYPE OF TEST - Specific Gravity, Fiber Volume, Resin Content, Void Content

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	1.582 64.2 % 28.6 % 0. %	1.556 62.0 % 30.4 % 0.8 %	1.54 60.0 % 32.3 % 1.1 %	1.56 63.4 % 29.0 % 0.4 %
2	1.588 64.0 % 28.5 % 0 %	1.526 60.6 % 31.8 % 1.4 %	1.58 65.1 % 27.6 % 0 %	1.56 67.1 % 25.1 % 1.6 %
3	1.578 65.5 % 27.4 % 0 %	1.572 63.9 % 28.7 % 0 %	1.54 63.8 % 28.9 % 1.9 %	1.57 65.6 % 27.2 % 1.2 %
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	152.7 147.9 129.2 143.3	126.0 157.4 157.7 147.0	108.9 136.5 127.0 124.1	77.8 101.9 146.1 108.6
2	185.3 197.7 160.3 181.1	204.8 185.7 197.9 196.1	173.1 176.4 189.8 180.0	220.4 215.3 212.8 216.1
3	195.9 207.6 185.5 196.3	158.6 164.8 172.5 165.3	210.4 182.8 186.6 193.3	167.7 204.7 159.1 177.2
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE OF 77°K (-320°F)

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	212.3 211.4 197.2 207.0	218.4 201.2 221.4 213.7	117.2 134.7 128.3 126.7	149.3 149.4 101.0 133.3
2	213.8 183.2 202.5 199.8	159.0 148.5 152.3 153.3	181.1 169.0 149.0 166.4	198.4 188.8 186.3 191.2
3	197.0 205.8 195.0 199.3	183.4 195.6 118.6 165.9	166.3 150.9 151.8 156.3	134.7 144.7 149.5 143.0
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 450°K (350°F)

CURE CYCLE	POST CURE CYCLE 1	2	3	4
1	81.4	111.7	74.3	79.7
	84.5	88.4	63.1	49.5
	66.0	82.5	53.8	48.4
	77.3	94.2	63.7	59.2
2	68.4	66.9	107.2	153.2
	50.4	86.8	107.0	137.1
	42.8	81.8	113.6	140.0
	53.9	78.5	109.3	143.5
3	86.6	95.2	103.2	134.5
	86.5	85.6	102.9	117.7
	99.3	85.8	111.6	115.2
	90.8	88.9	105.9	122.4
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Transverse Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	4.9 5.1 5.0 5.3	5.6 4.4 5.2 5.1	3.9 3.6 4.5 4.0	3.1 3.4 3.2 3.2
2	3.8 4.6 3.8 4.1	2.9 3.3 3.4 3.2	3.8 3.9 3.5 3.7	3.2 3.8 3.4 3.5
3	3.4 4.3 4.3 4.0	3.8 3.5 3.4 3.6	3.6 3.4 3.3 3.4	3.3 3.2 3.7 3.4
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE Room Temperature

CURE CYCLE	POST CURE CYCLE 1	2	3	4
1	5.3 4.9 5.4 5.2	5.8 5.8 5.9 5.8	4.6 4.3 5.2 4.7	4.0 4.6 4.3 4.3
2	6.9 6.5 7.2 6.9	6.1 6.4 6.1 6.2	5.4 6.2 5.5 5.7	6.4 6.5 7.0 6.6
3	6.3 6.9 6.5 6.6	5.9 5.7 5.3 5.6	5.8 6.0 5.9 5.9	6.4 6.8 6.3 6.5
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE 77°K (-320°F)

CURE CYCLE	POST CURE CYCLE 1	2	3	4
1	5.5 6.2 6.6 6.1	6.6 8.2 8.4 7.7	6.2 6.8 6.1 6.4	5.3 4.7 5.1 5.0
2	6.9 6.5 7.2 6.9	6.1 6.4 6.1 6.2	7.4 6.8 8.0 7.4	5.0 6.3 6.2 5.8
3	6.7 6.2 5.4 6.1	6.3 5.4 6.2 6.0	6.5 7.3 8.2 7.3	6.5 6.6 6.6 6.6

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Short Beam Shear, ksi

TEST TEMPERATURE 450°K (350°F)

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	2.7 2.8 2.7 2.7	3.2 3.1 3.3 3.2	3.7 3.6 3.4 3.6	3.5 2.9 3.0 3.1
2	2.4 2.4 2.5 2.4	2.7 3.0 2.9 2.9	4.4 3.8 3.9 4.0	4.6 4.3 4.3 4.4
3	3.8 3.5 3.4 3.6	3.7 3.5 4.0 3.7	4.5 4.1 4.2 4.3	4.4 4.5 4.2 4.4
4				

PRESS CURE STUDY
HT-S/X-904

TYPE OF TEST - Specific Gravity, Fiber Volume, Resin Content, Void Content

TEST TEMPERATURE Room Temperature

CURE CYCLE \ POST CURE CYCLE	1	2	3	4
1	1.486 62.1 % 30.2 % 4.5 %	1.487 59.5 % 32.7 % 3.4 %	1.47 61.6 % 30.7 % 5.7 %	1.46 62.1 % 30.7 % 5.9 %
2	1.48 58.8 % 33.2 % 4.6 %	1.49 60.0 % 32.0 % 4.3 %	1.49 59.3 % 32.7 % 4.0 %	1.50 61.7 % 30.6 % 3.9 %
3	1.44 56.5 % 35.1 % 6.6 %	1.47 57.5 % 34.4 % 4.7 %	1.46 58.3 % 33.7 % 5.6 %	1.47 57.6 % 34.5 % 4.4 %
4				

APPENDIX B

HT-S/710

PROCESS TEST DATA

VACUUM-PRESS-AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 297° K (75° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1(MP)	191.1 186.5 203.6 193.7	141.2 98.9 126.2 122.1	166.3 175.3 187.1 176.2	163.5 159.5 138.7 153.9
	2 (MP)	158.8 185.3 196.8 180.3	188.5 176.5 182.0 182.3	190.8 142.4 169.7 167.6	173.9 166.3 178.2 172.8
	3(G)	181.3 170.2 194.6 182.0	131.5 153.2 183.3 156.0	180.8 157.0 150.4 162.8	173.7 172.9 162.2 169.6
	4(G)	165.4 110.5 152.1 142.7	171.2 140.7 152.8 154.9	180.1 138.6 103.6 140.8	161.3 148.2 152.0 153.8
OC-31	3 (G)	177.4 177.4 160.3 171.7	96.4 104.2 125.4 108.7	153.4 174.3 85.4 137.7	117.6 122.1 174.8 138.2

MP Mauchberg Paper
G Glass

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

Preceding page blank

VACUUM-PRESS AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength ksi

TEST TEMPERATURE 77° K (-320° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	185.4 205.0 165.4 185.3	120.0 183.8 153.3 152.4	131.1 145.4 82.5 123.0	117.5 152.0 115.3 128.3
	2	221.1 182.2 179.2 194.2	147.7 161.7 151.2 153.5	129.5 179.8 166.3 158.5	115.2 128.4 113.5 119.0
	3	177.0 187.7 171.9 178.9	140.9 132.1 128.8 133.9	141.5 122.7 122.6 128.9	149.4 144.0 70.2 121.2
	4	187.7 116.9 151.6 152.1	146.9 133.5 146.3 142.2	115.3 112.7 94.3 107.4	80.1 90.7 113.1 94.6
OC-31	3	174.9 146.2 94.3 138.4	133.4 157.3 79.3 123.2	131.1 145.4 82.5 119.7	151.8 114.5 95.3 120.5

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM-PRESS AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589° K (600° F)

	POST CURE CYCLE	A*	B*	C*	D*
	CURE CYCLE				
OC-28	1	25.9	73.6	100.9	-
		25.9	80.3	93.4	75.4
		23.7	84.0	111.5	135.3
		25.2	79.3	101.9	105.4
	2	26.7	88.4	101.6	134.6
		26.2	98.2	104.6	107.8
		28.0	102.1	118.7	126.6
		27.0	96.2	108.3	123.0
	3	25.2	57.3	85.8	111.1
		28.6	55.1	71.2	102.0
		31.2	54.5	84.1	116.7
		28.3	55.6	80.4	109.9
	4	22.2	71.1	79.7	93.9
		22.6	55.2	80.6	99.4
		19.4	54.6	78.2	101.6
		21.4	60.3	79.5	98.3
OC-31	3	25.2	48.5	89.5	95.3
		26.8	58.4	90.5	73.3
		26.6	58.6	86.9	84.7
		26.2	55.2	88.9	84.4

*A No post cure
B 589° K (600°F) in air
C 616° K (650°F) in air
D 644° K (700°F) in air

VACUUM-PRESS AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Short Beam Shear Strength - ksi

TEST TEMPERATURE 297° K (75° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	6.4 6.4 6.5 6.4	7.8 6.3 6.2 6.8	6.1 7.9 6.0 6.6	6.4 6.5 7.0 6.6
	2	4.9 5.1 4.9 5.0	7.1 9.4 7.7 8.1	8.3 8.3 7.4 8.0	8.5 7.2 7.5 7.7
	3	4.9 4.3 4.3 4.5	6.6 7.3 7.4 7.1	6.4 6.1 5.6 6.1	6.8 7.0 7.0 6.9
	4	4.0 4.1 4.2 4.1	5.9 7.1 6.2 6.4	5.2 5.3 5.6 5.4	7.0 7.6 6.6 7.1
OC-31	3	5.8 5.7 6.1 5.8	4.8 3.9 — 4.4	4.9 5.8 6.5 5.7	4.4 6.1 5.1 5.2

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM-PRESS-AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 77° K(-320° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	5.7 5.9 4.9 5.5	5.6 4.7 5.5 5.3	7.1 6.4 7.1 6.8	7.3 5.5 6.6 6.5
	2	4.5 4.2 3.9 4.2	5.6 5.2 4.7 5.2	7.8 6.1 6.6 6.8	6.8 7.2 7.3 7.1
	3	3.6 3.5 3.4 3.5	3.7 5.0 4.4 4.4	6.3 5.0 5.0 5.4	6.7 6.7 6.1 6.5
	4	2.4 3.1 3.3 2.9	4.6 4.9 4.8 4.8	4.2 4.2 4.6 4.3	7.1 5.9 6.1 6.4
OC-31	3	4.4 4.7 4.1 4.4	3.2 3.2 2.3 2.9	4.8 4.3 4.7 4.6	4.9 4.4 3.0 4.1

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM-PRESS AUGMENTED CURE STUDY
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 589° K (600° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	3.8 3.8	4.6 4.5 3.2 4.1	4.3 4.3	4.2 4.2
	2	3.9 3.9	5.1 4.9 4.6 4.9	4.9 4.9	5.0 5.0
	3	3.8 3.6 3.8 3.7	3.8 3.7 4.1 3.9	3.9 3.9	5.1 5.1
	4	3.8 3.7 3.7 3.7	3.9 3.7 4.2 3.9	3.9 3.8 3.8 3.8	5.1 5.1
OC-31	3	3.7 3.1 2.9 3.2	2.5 3.0 3.4 3.0	4.3 4.3	4.1 4.1

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM-PRESS-AUGMENTED CURE STUDY

HT-S/710

TYPE OF TEST - Transverse Flexure Strength, ksi

TEST TEMPERATURE 297° K (75° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	3.4 3.6 3.0 3.3	0.8 0.7 0.7 0.7		3.5 4.2 3.0 3.6
	2	2.1 2.3 2.1 2.2	2.4 2.6 2.0 2.3	3.5 3.0 3.5 3.3	
	3	3.4 3.3 3.6 3.4	3.6 3.5 3.5 3.5	3.5 3.0 3.5 3.4	4.5 3.9 3.9 4.1
	4	4.8 4.2 4.8 4.6	2.7 2.5 2.9 2.7	3.5 4.2 3.0 3.6	3.5 3.1 3.3 3.3
OC-31	3	3.9 3.2 3.6 3.6	3.0 3.2 3.6 3.3	2.4 2.4 - 2.4	

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM-PRESS-AUGMENTED CURE STUDY

HT-S/710

TYPE OF TEST - Specific Gravity, Fiber Volume, Resin Content

	CURE CYCLE	POST CURE CYCLE A*	B*	C*	D*
OC-28	1	1.53 51.7 43.4	1.54 58.0 37.0	1.50 57.0 38.0	1.47 63.1 31.8
	2	1.48 55.0 38.7	1.49 66.3 28.7	1.51 57.4 37.6	1.48 63.1 31.8
	3	1.49 58.3 36.5	1.46 59.9 35.3	1.46 62.1 32.8	1.48 62.5 32.6
	4	1.43 57.6 37.1	1.48 61.8 34.1	1.44 57.9 37.1	1.44 63.2 31.9
OC-31	3	1.48 59.0 35.9	1.50 64.9 30.1	1.44 61.9 33.1	1.49 63.2 31.8

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE STUDY
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 297°K (75°F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	85.0 206.3 73.9 121.7	189.2 180.6 129.0 166.3	178.2 178.9 164.1 173.7	172.9 155.5 130.9 153.1
	2	182.4 160.3 158.2 167.0	178.0 162.2 140.1 160.1	171.9 172.0 185.9 176.6	161.0 136.1 155.5 150.9
OC-31	1	154.2 178.5 158.8 163.9			189.1 169.2 137.9 165.4
	2	141.6 144.2 146.2 144.0			117.0 148.0 171.8 145.6

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE STUDY
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77° K (-320° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	199.6 188.0 79.5 152.4	172.2 159.4 127.3 152.9	155.9 108.8 139.6 134.8	69.3 111.9 134.8 105.3
	2	192.3 200.7 163.4 185.5	124.4 166.4 169.9 153.6	165.5 182.1 129.1 158.9	119.1 165.4 141.3 141.9
OC-31	1	185.5 170.9 142.0 166.1			135.0 159.4 141.3 145.2
	2	107.0 131.8 156.2 131.7			153.4 106.0 109.9 123.1

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE

HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589° K (600° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	21.3 19.2 16.0 18.8	45.3 32.1 50.4 42.6	 89.7	83.8 103.3 87.2 91.4
	2	27.0 29.1 29.3 28.5	70.2 98.6 82.1 83.7	97.9 96.3 101.5 98.6	95.1 98.9 103.9 99.3
OC-31	1	24.0 22.4 25.3 23.9			105.8 70.0 ** 67.8 ** 81.2
	2	24.0 18.9 24.3 22.4			120.4 91.6 86.3 99.4

**Delaminated

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 297°K (75°F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	5.5 6.2 6.2 6.0	9.3 8.8 8.6 8.9	7.6 6.2 6.9 6.9	6.6 6.1 6.0 6.2
	2	5.9 6.4 6.0 6.1	6.1 6.4 6.1 6.2	7.8 7.7 7.0 7.5	7.4 9.1 8.4 8.3
OC-31	1	1.9 5.5 6.1 4.5			7.0 7.2 6.3 6.8
	2	5.9 6.4 6.8 6.4			7.0 6.0 6.4 6.5

*A No post cure
B 589°K (611°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE STUDY
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 77° K (-320° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	5.2 4.6 5.1 5.0	6.1 7.0 5.7 6.2	6.9 7.1 7.7 7.2	6.0 6.2 5.8 6.0
	2	4.7 5.7 5.9 5.4	5.2 5.0 5.7 5.3	8.2 6.3 7.9 7.5	6.8 8.3 6.1 7.1
OC-31	1	5.8 5.8 5.7 5.8			6.2 4.5 4.0 4.9
	2	4.7 4.3 4.8 4.6			4.7 5.3 6.0 5.3

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 466°K (700°F) in air

VACUUM BAG CURE STUDY
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 589° K (600° F)

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1		4.2 4.8 4.8 4.6	4.3 4.2 4.3 4.3	3.7 3.8 3.6 3.7
	2		4.6 4.6 4.6 4.6	4.2 4.5 4.7 4.5	4.8 5.2 4.7 4.9
OC-31	1				4.2 4.7 4.7 4.5
	2				4.2 4.5 4.8 4.5

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG CURE
HT-S/710

TYPE OF TEST - Transverse Flexure Strength, ksi

TEST TEMPERATURE 297° K (75° F)

POST CURE CYCLE		A*	B*	C*	D*
OC-28	CURE CYCLE 1	2.4 2.9 2.0 2.4	4.0 3.7 3.6 3.8	5.0 5.2 4.7 5.0	5.8 5.3 4.7 5.2
	2	4.8 4.6 3.9 4.5	5.6 5.7 5.1 5.5	4.5 5.0 5.0 4.8	3.8 5.0 4.9 4.6
OC-31	1				4.3 4.1 4.9 4.4
	2	3.1 4.4 4.1 3.9			4.4 5.0 4.8 4.7

* A No post cure
B 589° K (600° F) in air
C 616° K (650° F) in air
D 644° K (700° F) in air

VACUUM BAG CURE STUDY
HT-S/710

TYPE OF TEST - Specific Gravity, Fiber Volume, Resin Content
TEST TEMPERATURE Room Temperature

	POST CURE CYCLE	A*	B*	C*	D*
OC-28	CURE CYCLE 1	1.38 57.0 38.0	1.41 59.6 35.3	1.34 59.9 35.1	1.48 64.0 31.0
	2	1.47 56.5 38.4	1.47 59.5 35.5	1.46 59.0 36.0	1.47 59.2 35.7
OC-31	1	1.48 55.0 39.8			1.47 56.2 38.7
	2	1.49 57.1 37.8			1.50 60.6 34.3

*A No post cure
B 589°K (600°F) in air
C 616°K (650°F) in air
D 644°K (700°F) in air

VACUUM BAG STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Stregnth, ksi

TEST TEMPERATURE Room Temperature

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO -	162,0	150.2	172.2	
	189.1	163.5	165.3	
	162.7	-	-	
	171.3	156.9	168.8	
WRD -	149.1	162.7	162.7	
	152.0	167.1	161.3	
	159.5	-	-	
	153.5	164.9	162.0	
FIBERITE -	115.6	-	-	
	121.7	70.6	99.7	
	125.8	109.7	151.1	
	121.0	90.2	125.4	

VACUUM BAG SUTDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77°K (-320°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
FERRO - CURE CYCLE		136.2 173.0 - 154.5	189.7 152.4 - 171.1	
WRD -				
FIBERITE -		114.2 105.0 - 109.6	151.4 114.2 - 132.8	

VACUUM BAG STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589°K (600°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO -		94.1 92.3 - 93.2	84.9 76.0 - 80.5	
WRD -		96.1 100.1 - 98.1	71.8 95.6 - 83.7	
FIBERITE -		97.7 57.8 - 77.8	65.2 61.4 - 63.3	

VACUUM BAG STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Specific Gravity, Resin Content, Fiber Volume

POST CURE CYCLE	NPC	644°K (700°F) in Air	644°K (700°F) in N2	
CURE CYCLE FERRO -	1.42 38.2 % 57.8 %	1.41 34.3% 60.7%	1.40 33.5% 61.4%	
WRD -	1.41 48.4 % 45.9 %	1.41 39.9% 55.0%	1.40 31.9% 63.2%	
FIBERITE -	1.41 40.2 % 54.5 %	1.38 37.7% 57.2%	1.38 38.9% 56.1%	

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644° (700°F) IN N_2	
FERRO P-1 CURE CYCLE	212.0 189.2 176.6 186.7 192.4 202.6 193.2	178.9 164.0 173.7 172.2	203.7 203.3 193.1 200.0	
WRD P-2	185.0 187.1 173.4 181.8	161.3 146.2 126.4 144.6	100.6 127.0 135.1 120.9	
FIBERITE P-1	164.4 196.5 160.8 173.9	194.0 189.7 192.0 191.9	168.1 141.1 161.4 156.9	
FERRO - 2	179.1 183.2 192.9 185.1	187.6 188.9 176.1 184.5	189.5 203.2 180.4 191.0	
FIBERITE - P-2	164.5 152.8 143.6 176.0 159.2	- 183.0 191.3 197.9 190.7	- 172.6 183.4 177.5 177.8	

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE - 77°K (-320°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO P-1	165.2			
	162.2	164.3	192.8	
	187.2	114.0	158.5	
	180.0	138.3	241.8	
	173.7	138.9	197.7	
WRD P-2	169.1			
	172.8	148.2	163.8	
	175.5	135.0	183.8	
	171.3	130.5	163.1	
	172.2	137.9	170.2	
FIBERITE P-1	169.9			
	171.4			
	183.8			
	208.5			
	183.4			
FERRO - 2	163.6	161.0	159.1	
	173.4	166.2	205.7	
	195.8	129.7	225.0	
	175.2			
	177.0	152.3	196.6	
FIBERITE - 2	110.7	268.0	202.0	
	154.3	213.7	225.3	
	129.4	205.2	207.3	
	131.5	229.0	211.5	

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589°K (600°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN AIR	
CURE CYCLE FERRO P-1		121.3 133.9 122.7 126.0	128.7 107.1 110.2 115.3	
WRD P-2		83.1 124.6 131.1 112.9	124.3 133.9 - 129.1	
FIBERITE P-1		135.4 119.6 124.4 126.5	124.8 120.0 197.7 117.5	
FERRO P-2		140.1 130.2 134.1 134.8	145.4 127.7 138.0 137.0	
FIBERITE		130.9 123.6 118.4 115.3	125.5 106.3 118.9 116.9	

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST- Short Beam Shear Strength, ksi

TEST TEMPERATURE 297° K (75° F)

POST CURE CYCLE	NPC	644° K(700° F) In Air	644° K(700° F) In N_2	
CURE CYCLE FERRO P-1			11.6 10.2 10.8 10.9	
WRD P-2				
FIBERITE P-1				
FERRO P-2		11.8 12.2 12.1 12.0	12.4 13.1 12.8 12.8	
FIBERITE P-2		11.1 12.4 11.6 11.7	6.2* 6.6* 6.2* 6.3	

* Specimens bottomed out in bending - no shear failure

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST- Short Beam Shear Strength, ksi

TEST TEMPERATURE 77° K (-320° F)

POST CURE CYCLE	NPC	644° K(700° F) In Air	644°K(700° F) In N_2	
CURE CYCLE FERRO P-1			10.0 10.2 9.2 10.1	
WRD P-2				
FIBERITE P-1				
FERRO P-2		11.4 12.5 9.6 11.2	13.6 11.1 11.8 12.2	
FIBERITE P-2		10.8 9.3 10.7 10.3	5.8* 6.4* 6.1* 6.1	

* Specimens bottomed out in bending - no shear failure

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 589° K (600° F)

POST CURE CYCLE	NPC	644° K(700° F) In Air	644° K(700° F) In N_2	
CURE CYCLE FERRO P-1			5.7 5.7 5.6 5.6	
WRD P-2				
FIBERITE P-1				
FERRO P-2		6.9 7.0 6.5 6.8	7.4 7.3 7.0 7.2	
FIBERITE P-2		7.4 6.6 6.8 6.9	4.3 4.5 4.3 4.4	

PRESS STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Specific Gravity, Resin Content, Fiber Volume

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FEERO P-1	1.44 42.5 % 52.2 %	1.46 36.3% 58.7%	1.46 35.3% 59.8%	
WRD P-2	1.46 45.7 % 49.3 %	1.50 40.1% 54.8%	1.50 42.4% 53.3%	
FIBERITE P-1	1.52 48.4 % 45.6 %	—	—	
FERRO - 2	1.51 35.4 % 59.5 %	1.48 31.2% 63.9%	1.52 33.3% 61.8%	
FIBERITE - 2	1.49 38.4 % 56.4 %	1.51 35.9% 59.0%	1.48 43.3% 51.6%	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO A-6	207.1 175.1 215.4 203.7 195.0 228.0 204.0	172.7 178.4 186.2 179.1	197.0 193.7 197.2 195.9	
A-7	193.3 179.2 193.4 190.6	166.7 165.6 114.1 143.8	187.2 196.2 194.7 192.7	
FIBERITE A-3	188.0 190.8 160.0 173.5	179.9 148.2 134.7 154.3	202.0 172.3 181.4 185.2	
A-4	162.6 185.6 185.4 174.2	160.7 146.0 148.7 151.8	149.7 200.5 191.6 180.6	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77°K (-320°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO A-6	173.2	130.2	191.0	
	181.7	150.4	166.2	
	166.6	149.6	160.4	
	212.8			
	183.6	143.4	172.5	
A-7	189.5	137.4	161.1	
	177.4	120.3	162.7	
	196.4	125.2	155.4	
	192.9			
	189.1	127.6	159.7	
FIBERITE A-3	202.2			
	118.1			
	183.5			
	167.9			
A-4	195.6	141.8	206.0	
	186.9	162.2	215.9	
	174.9	138.5	180.0	
	185.8	147.5	200.6	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589°K (600°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO A-6		154.2 141.8 141.1 145.7	132.4 127.4 133.7 131.2	
A-7		146.7 155.8 173.8 158.8	136.0 126.5 132.1 131.5	
FIBERITE A-3		134.6 130.2 132.6 132.5	105.7 118.1 141.2 121.7	
A-4		132.6 120.0 114.9 122.5	121.5 131.0 124.9 125.8	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 297° K (75° F)

POST CURE CYCLE	NPC	644° K(700° F) in Air	644° K(700° F) in N_2	
CURE CYCLE FERRO A-6		11.4 11.2 11.7 11.4	11.3 11.2 11.2 11.2	
FERRO A-7		6.6 6.7 7.0 6.8	12.5 11.9 12.5 12.3	
FIBERITE A-3				
FIBERITE A-4		12.4 12.0 10.9 11.8	8.8 8.3 9.0 8.7	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 77° K (-320° F)

POST CURE CYCLE	NPC	644° K(700° F) In Air	644° K(700° F) In N_2	
CURE CYCLE FERRO A-6	-	7.1 10.6 10.5 9.4	12.2 10.3 11.9 11.5	
FERRO A-7	-	7.3 8.0 8.2 7.8	11.6 10.7 11.2 11.2	
FIBERITE A-3	-			
FIBERITE A-4	-	10.5 8.2 9.0 9.2	9.0 7.6 10.0 8.9	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Short Beam Shear Strength, ksi

TEST TEMPERATURE 589° K (600° F)

POST CURE CYCLE	NPC	644° K(700° F) in Air	644° K(700° F) in N_2	
CURE CYCLE FERRO A-6		8.6 7.8 7.3 7.9	7.3 5.3 5.0 5.9	
FERRO A-7		5.4 5.2 4.8 5.1	7.2 7.4 6.9 7.2	
FIBERITE A-3		-	-	
FIBERITE A-4		6.8 6.7 7.1 6.9	7.3 6.6 6.7 6.9	

AUTOCLAVE STUDY
VENDOR EVALUATION
HT-S/710

TYPE OF TEST - Specific Gravity, Resin Content, Fiber Volume

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE FERRO A-6	1.49 37.5 % 57.3 %	1.50 33.4% 61.7%	1.49 34.8% 60.2%	
A-7	1.49 40.0 % 54.9 %	1.50 34.0% 61.0%	1.48 37.5% 57.2%	
FIBERITE A-3	1.45 37.9 % 56.8 %	—	—	
A-4	1.50 37.8 % 56.9 %	1.49 43.1% 52.6%	1.51 46.8% 48.2%	

AUTOCLAVE STUDY - BLEEDER EVALUATION

HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE OC-28 BBS*	186.2	-	196.8	
	193.8	193.5	211.1	
	231.6	175.0	177.4	
	203.9	184.3	195.1	
BOS**	221.0	-	-	
	225.8	209.3	225.7	
	210.8	218.0	214.1	
	219.2	213.7	219.9	
OC-31 BBS	143.5	-	199.5	
	172.9	185.9	158.5	
	177.1	146.2	164.0	
	164.5	166.1	173.9	
BOS	234.4	-	218.0	
	211.4	187.4	214.1	
	212.4	187.3	224.4	
	219.4	187.4	218.8	

*BBS — Bleeder both sides

**BOS — Bleeder one side

AUTOCLAVE STUDY - BLEEDER EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77°K (-320°F)

POST CURE CYCLE / CURE CYCLE	NPC	644°K (700°F) IN AIR		
OC-28 BBS		170.6 149.7 - 160.2		
BOS				
OC-31 BBS		134.4 129.7 - 132.1		
BOS		176.4 185.2 - 180.8		

AUTOCLAVE STUDY - BLEEDER EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589°K (600°F)

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°F (700°F) IN N_2	
CURE CYCLE OC-28 BBS		138.9 141.7 - 140.3	105.8 96.1 115.2 105.7	
BOS		136.4 149.0 - 142.7	108.6 94.7 - 101.7	
OC-31 BBS		121.4 118.7 - 120.1	108.6 98.2 106.9 104.6	
BOS		127.2 115.7 - 121.5	117.2 111.2 104.0 110.8	

AUTOCLAVE STUDY - BLEEDER EVALUATION
HT-S/710

TYPE OF TEST - Specific Gravity, Resin Content, Fiber Volume

CURE CYCLE \ POST CURE CYCLE				
OC-28 BBS	1.49 33.0 % 62.0 %			
BOS	1.50 38.1 % 56.8 %			
OC-31 BBS	1.48 39.4 % 55.4 %			
BOS	1.53 36.5 % 58.3 %			

AUTOCLAVE STUDY - VACUUM EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE Room Temperature

POST CURE CYCLE	NPC	644°K (700°F) IN AIR	644°K (700°F) IN N_2	
CURE CYCLE 1 EDGES ONLY	179.6	194.4	232.2	
	206.5	206.5	197.9	
	-	-	-	
	193.0	200.4	215.0	
PICTURE FRAME 2	205.4	180.9	200.2	
	184.0	181.0	182.8	
	185.7	-	-	
	191.7	181.0	191.5	
NO DAM 3	212.9	-	198.9	
	178.9	188.4	205.1	
	231.7	185.4	166.2	
	207.8	186.9	190.1	
VACUUM LINE 4	182.6	128.5	201.5	
	191.5	170.8	194.8	
	199.4	-	-	
	191.2	149.7	198.2	

AUTOCLAVE STUDY - VACUUM EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 77°K (-320°F)

POST CURE CYCLE / CURE CYCLE	A NPC	B 644°K (700°F) IN AIR	C 644°K (700°F) IN AIR	D
A-2	NO DATA			
A-3	NO DATA	154.9 153.9 - 154.4	178.9 185.8 - 182.4	
A-4	NO DATA		135.4 - - 135.4	
A-5	NO DATA	135.5 143.6 - 139.6	156.7 183.8 - 170.3	

AUTOCLAVE STUDY - VACUUM EVALUATION
HT-S/710

TYPE OF TEST - Longitudinal Flexure Strength, ksi

TEST TEMPERATURE 589°K (600°F)

POST CURE CYCLE	A NPC	B 644°K (700°F) IN AIR	C 644°K (700°F) IN AIR	D
CURE CYCLE A-2	NO DATA	151.0 155.0 - 153.0	118.3 102.6 - 110.5	
A-3	NO DATA	140.9 148.8 - 144.9	142.6 127.3 - 134.9	
A-4	NO DATA	137.6 137.4 - 137.5	141.9 132.7 - 137.0	
A-5	NO DATA	124.2 129.3 - 126.8	136.8 141.1 - 139.0	

AUTOCLAVE STUDY - VACUUM EVALUATION
HT-S/710

TYPE OF TEST - Specific Gravity, Resin Content, Fiber Volume

POST CURE CYCLE / CURE CYCLE	A	B	C	D
EDGES ONLY 1	1.49 38.4 % 56.4 %			
PICTURE FRAME 2	1.51 32.6 % 62.4 %			
NO DAM 3	1.50 37.2 % 57.7 %			
VACUUM LINE 4	1.51 36.4 % 59.6 %			

APPENDIX C

EPOXY RESIN SPECIFICATION

GENERAL DYNAMICS *Convair Division* CODE IDENT NO. 14170	SPECIFICATION NO. 0-06061	REV A
	DATE	
	CONTRACT NO.	

NO. OF PAGES _____

RESIN, EPOXY, IMPREGNATING, SPECIFICATION FOR

PREPARED BY _____
H. R. Stone

APPROVED BY _____
H. H. Rosenbaum

CHECKED BY _____

Preceding page blank

SPECIFICATION CHANGE NOTICES—INCORPORATED IN SPEC REVISIONS

SCN NO(S)	INCORP BY	CHECK BY	APPROVED BY	DATE	SPEC REV	REVISION PACKAGE NO.	RELEASE

INITIAL PACKAGE NO.	RELEASE	DOC TYPE C-3	DISTR CODE

1. SCOPE

1.1 Scope. This specification establishes the requirements for a heat reactive epoxy resin which can be cured to a crosslinked epoxy system.

1.2 Classification. The material covered by this specification shall be of one type and identified as 0-06061-1.

2. APPLICABLE DOCUMENTS

2.1 Unless otherwise identified below, the following documents of the issue in effect on date of Convair's request for quotation form a part of this specification to the extent specified.

STANDARDS

Federal

Federal Test Method Std. No. 406	Plastics, Methods of Testing

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes

American Society For Testing and Materials

Standard D808	Chlorine in New and Used Petroleum Products (Bomb Method)

PUBLICATIONS

General Industry Safety Orders, State of California Department of Industrial Relations, Article 85, "Labeling of Injurious Substances".

Manufacturing Chemists Association, Labels and Precautionary Information Committee, "Guide to Precautionary Labeling of Hazardous Chemicals". (Manual L1.)

3. REQUIREMENTS

3.1 Chemical structure of polymer.

3.1.1 Epoxy resin. The resin shall consist of an aromatic polyglycidyl ether cured with a monoanhydride (nadic methyl anhydride) and catalyzed with benzyldimethylamine to form the epoxy resin system. The resin and curing agent shall have the idealized structures shown in Figure 1.

3.1.2 Cured polymer. The cured resin system shall have the generalized structure shown in Figure 2.

3.2 Toxic or hazardous formulations. If injurious formulations cannot be avoided, all containers of injurious substances shall be labeled as required by Article 85 of the California General Industry Safety Orders or Manual L1 of the Manufacturing Chemists Association.

3.3 Performance characteristics.

3.3.1 Storage stability. The resin, in the containers as supplied, unopened, and stored at a temperature of 297K ± 6 (75F ± 10) shall be capable of meeting the requirements of Table I for not less than 90 days from date of receipt of shipment.

3.3.2 Physical and chemical properties of epoxy resin. The physical and chemical properties of the uncatalyzed, uncured resin shall meet the requirements of Table I.

Table I

Property Requirements of Resin

Property	Requirement	Test Paragraph
Epoxide Equivalent Weight	200-210	4.6.2
Hydrolyzable Chlorine, %	0.0 - 0.4	4.6.3
Total Chlorine, % max.	0.6	4.6.4
Total Volatiles, 3 hr at 436K (325F)	1.5% max.	4.6.5
Softening Point	313-333K (104-140F)	4.6.6

3.3.3 Resin system reaction rates. The reaction rates of the resin system as determined by time versus viscosity measurements shall be nominally as shown in Figure 3.

3.3.4 Resin system flow and softening points. The neat resin system, as mixed and at room temperature, is soft and will undergo viscous flow. "Good" flow shall be obtained in the temperature range of 323 to 333K (122 to 140F) and higher.

3.3.5 Reaction products. Cure of the resin system shall be accomplished by an addition reaction. Therefore, there shall be no reaction products generated during cure. Liberation of residual solvent, and excess nadic methyl anhydride curing agent, shall be acceptable.

3.3.6 Specific gravity of cured resin system. When cured for 24 hours at 463K (375F) the cured resin system shall have a specific gravity of 1.26 ± 0.01.

3.3.7 Gel time. The gel time of the resin system shall be 12 minutes ± 3.

3.3.8 Workmanship. The resin shall be free of foreign or gelled material and any other contaminants detrimental to its performance.

3.4 Product markings. Product markings shall be in accordance with the preparation for delivery section of this specification.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or order, the resin supplier is responsible for the performance of all inspections and test requirements as specified herein. Except as otherwise specified, the resin supplier may use his own facilities or any commercial laboratory acceptable to Convair. Convair reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.3 Classification of examinations and tests. The examinations and tests of the material shall be classified as follows:

a. Qualification verification
b. Acceptance verification
c. Receiving inspection.

4.3.1 Qualification verification. Qualification verification shall consist of all the examinations and tests specified herein.

4.3.2 Acceptance verification. Acceptance verification shall be performed on representative samples of each batch of material, and shall consist of the following:

a. Epoxide equivalent weight
b. Total volatiles, percent
c. Softening point.

4.3.3 Receiving inspection (for Convair or Convair's preimpregnated material suppliers use only). Receiving inspection shall consist of an examination of the material and such sampling and verification of test data as deemed necessary.

4.4 Sampling plan. The material, as offered for acceptance by Convair or Convair's preimpregnated material suppliers, shall meet the requirements herein at an Acceptance Quality Level of 4.0 (normal inspection) when a batch is sampled per MIL-STD-105 at Inspection Level II.

4.4.1 Batch. A batch shall consist of all material of the same type manufactured in one continuous, unchanged production run.

4.5 Test conditions.

4.5.1 Room temperature. Unless otherwise specified, all tests shall be conducted at a temperature of 298K ± 3 (77F ± 5), and 50% ± 10 relative humidity.

4.6 Test methods.

4.6.1 Examination of product. The material shall be examined to verify that its markings, packaging, and visual physical characteristics conform to the requirements of this specification.

4.6.2 Epoxide equivalent weight. The preparation and procedure for determining the epoxide equivalent weight shall be as follows:

4.6.2.1 Reagents.

a. Acetic acid, glacial, reagent grade.

b. Hydrobromic acid (30 to 32% in acetic acid).

c. Crystal violet indicator solution. (Prepare a 0.1% solution in glacial acetic acid.)

d. Sodium carbonate, primary standard grade, dried to constant weight at 393 to 413K (248 to 284F).

e. Chlorobenzene, reagent grade.

f. Hydrogen bromide in acetic acid, 0.1 N maximum. Normality of HBr shall not exceed 0.1. Weigh 52g hydrobromic acid (30 to 32% in acetic acid) into a 2-liter container fitted with a glass stopper. Fill to 2 liters with glacial acetic acid and mix thoroughly. Standardize the HBr-HAc solution daily. Standardize against 0.1g anhydrous $Na_2 CO_3$ dissolved in 10.0ml glacial HAc (accurately measured by pipet) and titrate to the blue-green end point of the crystal violet indicator.

4.6.2.2 Procedure.

a. Using that quantity of sample which contains 0.001 to 0.002 equivalent of epoxide (approximately 0.25 to 0.50g), weigh the amount of sample desired (to the nearest milligram) into a 125-ml flask. Add 10.0 ml chlorobenzene to the sample. Stir with a magnetic stirrer until sample is dissolved.

b. Add 4 to 6 drops of crystal violet indicator solution. Place the rubber stopper in position and lower the buret tip to a point just above the solution. Titrate the solution with 0.1 N HBr in HAc to the blue-green end of the crystal violet indicator. The titration rate shall be slowed near the end point to allow ample time for the reaction to go to completion. An effort should be made to obtain the same color at the end point as that obtained during standardization of the reagent.

c. Determine the reagent blank, using the same reagents and above procedure, but omitting the sample.

4.6.2.3 Calculation. The average value of two determinations calculated as follows shall be reported.

Standardization of HBr in HAc:

$$\text{N of HBr} = \frac{(\text{wt. of } Na_2CO_3)\ (1,000)}{(53)\ (\text{ml of HBr})}$$

Epoxide equivalent:

$$\text{Weight per epoxide equivalent} = \frac{(W)\ (1,000)}{(S-B)\ (N)}$$

W = weight of sample
S = milliliters of HBr used in titrating sample
B = milliliters of HBr used in titrating blank
N = normality of HBr

4.6.3 Percent hydrolyzable chlorine. The preparation and procedure for determining the percent of hydrolyzable chlorine shall be as follows:

4.6.3.1 Reagents.

a. 0.1 N alcoholic potassium hydroxide solution. (Dissolve 5.61g of reagent grade KOH in 1 liter of methyl alcohol. No standardization is necessary.)

b. 0.1 N hydrochloric acid solution, standardized.

c. Toluene, reagent grade.

d. Methyl alcohol, reagent grade.

e. Phenolphthalein indicator solution.

4.6.3.2 Procedure.

a. Introduce 6 to 8g of sample to be tested into each of two Erlenmeyer flasks.

b. Pipet 50 ml of the 0.1 alcoholic KOH into each sample and into two blanks.

c. Add 15 ml of toluene from a graduated cylinder into each flask. Stopper and swirl until resin is in solution.

d. Add 3 or 4 glass beads. Connect flasks to reflux condensers, and place on hot plate.

e. Reflux gently for 15 minutes and cool to room temperature with condensers in place. (When cool, wash condensers down with 5 ml methyl alcohol.)

f. Add 3 drops of phenolphthalein indicator and titrate with 0.1 N hydrochloric acid to the first disappearance of the pink color.

4.6.3.3 Calculation. The average value of two determinations calculated as follows shall be reported.

$$\text{Percent hydrolyzable chlorine} = \frac{(B-A) \times N \times 3.55}{\text{wt.}}$$

A = milliliters of acid required to titrate sample
B = milliliters of acid required to titrate blank
N = normality of hydrochloric acid solution
wt. = weight of sample used

4.6.4 Total chlorine content. The total chlorine content shall be determined in accordance with ASTM Method D808.

4.6.5 Volatile content. Volatile content shall be determined as follows:

a. Place a 10g sample of resin in a 2-inch tared aluminum weighing dish.

b. Place dish and resin in a 436K ± 3 (325F ± 5) air circulating oven for 3 hours ± 0.1.

c. Calculate volatile content as follows:

$$\text{Percent volatile content} = \frac{W_3 - W_2}{W_2 - W_1} \times 100$$

W_1 = weight of empty dish

W_2 = weight of resin and dish before heating

W_3 = weight of resin and dish after heating

The average of three determinations shall be reported.

4.6.6 Softening point. The softening point of the resin shall be determined by use of a Fisher-Johns melting point apparatus. Heating rate of the solid resin shall be approximately 2K (3.6F) per minute. The average of three determinations shall be reported.

4.6.7 Specific gravity. The specific gravity of the cured resin system shall be determined in accordance with FTMS No. 406, Method 5011. The average of three determinations shall be reported.

4.6.8 Gel time. The gel time of the resin system shall be determined by use of a Sunshine gel meter and a bath temperature of 403K ± 1 (266F ± 2). The average of three determinations shall be reported.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. All material furnished under this specification shall be in suitable containers in quantitites as specified on the purchase order. The material shall be packaged to ensure protection from physical damage during handling, shipping, and storage.

5.2 Packing. The material shall be packed in shipping containers of a type which will ensure acceptance by common carrier at lowest rates, and will ensure protection of the unit containers during shipment.

5.3 Marking for shipment. Each unit and shipping container shall be legibly identified with label, tag, or markings which include the following data.

a. 0-06061-1
b. Purchase order number
c. Manufacturer's material description and identifying designation
d. Manufacturer's name and address
e. Quantity (shipping container only) and unit size
f. Batch number and date of manufacture
g. Date of shipping
h. Manufacturer's recommended storage conditions and temperature
i. Hazardous warnings and handling markings as applicable.

6. NOTES

6.1 Intended use. The material covered by this specification is intended for impregnating graphite fiber used in the manufacture of missile and spacecraft laminated structural parts subject to temperatures from 20K (-423F) to 450K (350F). Use is not limited to these applications.

6.2 Ordering information. The following information together with the requirements of 6.2.1 and 6.2.2 should be included on the purchase order.

a. Number, title, and date of this specification
b. Material name and quantity

6.2.1 Rejection and retest. In the event of failure of a sample to meet any requirements of this specification, a second sample of resin may be submitted for retest. If the retest sample fails to meet the requirements of the specification, the batch represented by the sample shall be rejected.

6.2.2 Reports. Unless otherwise specified, the supplier shall furnish with each batch three copies of the reports showing the results of tests made on each batch in the shipment to determine conformance of the material to the specification. The report shall include the requirements of Table I. The reports shall also include the the purchase order number, the material specification number, suppliers material designation, quantity, batch number(s), and date(s) of manufacture.

6.3 Approved sources. The approved sources for the material described in this specification are as follows:

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
0-06061-1	ERRA-0163	Union Carbide Corp. Thermosetting Resins and Compounds 270 Park Avenue New York, New York 10017

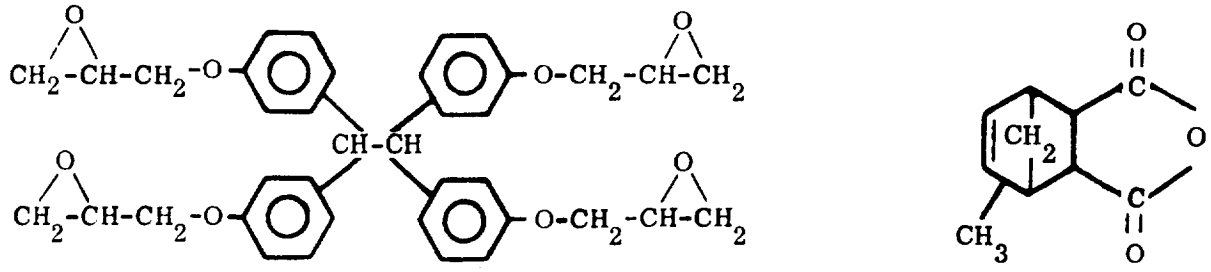


Figure 1. Idealized Structures. Resin and Curing Agent

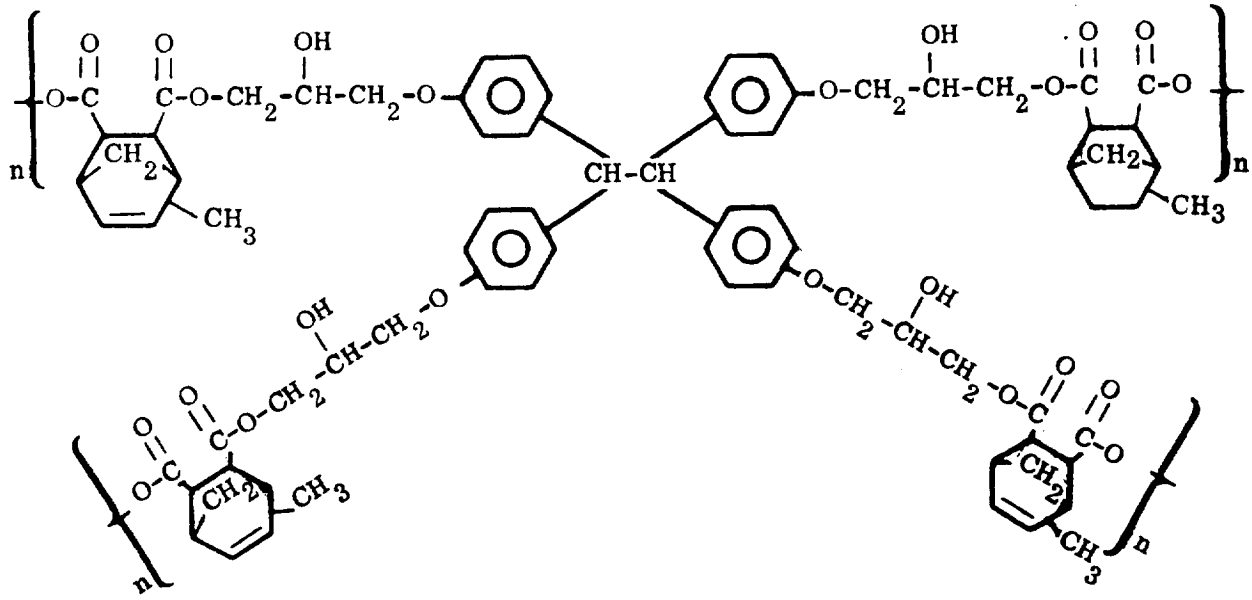


Figure 2. General Structure, Cured Polymer

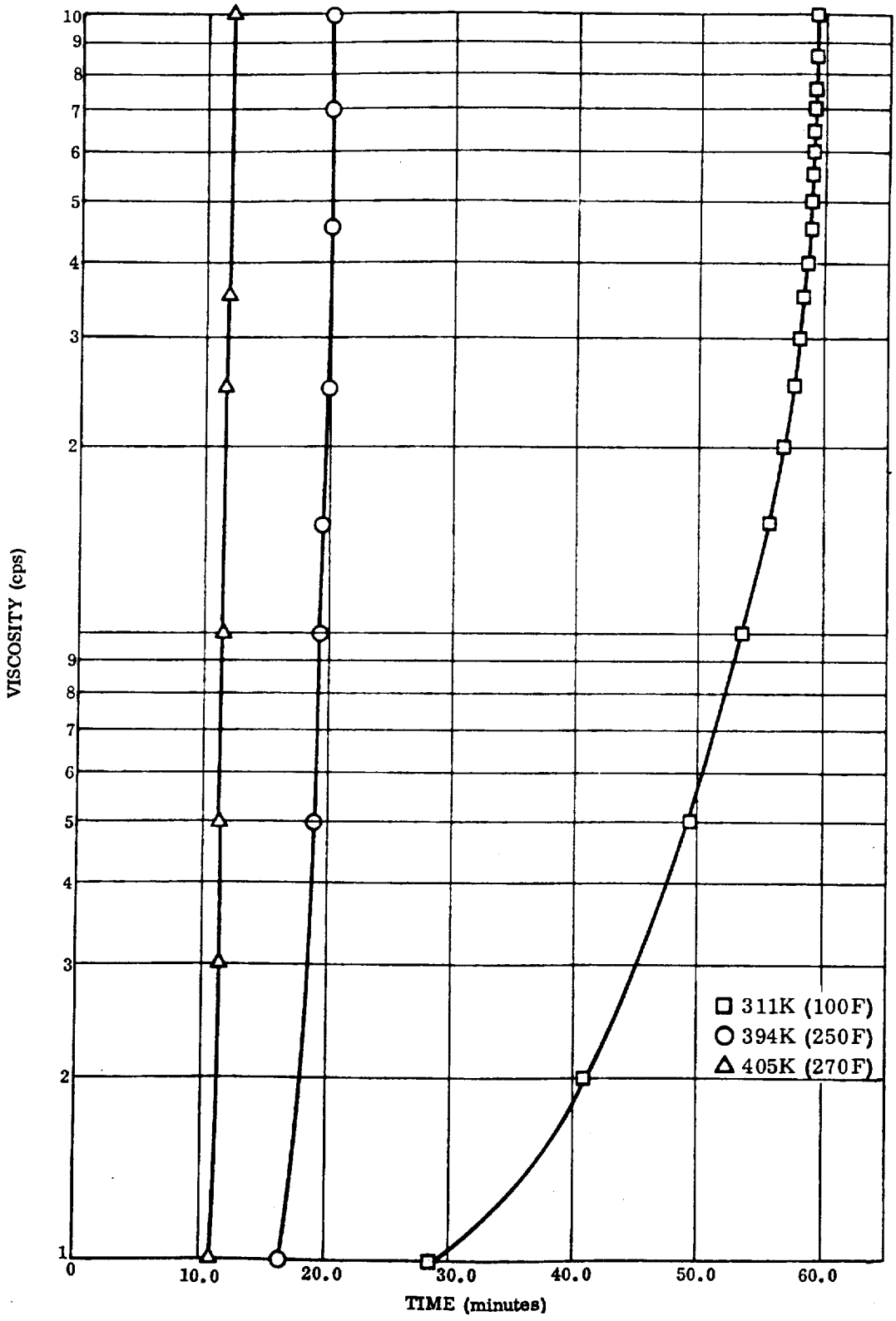


Figure 3. Time Versus Viscosity Measurements for Epoxy Resin System

C-16

APPENDIX D

IMPREGNATING POLYIMIDE RESIN SPECIFICATION

GENERAL DYNAMICS *Convair Division* CODE IDENT NO. 14170	SPECIFICATION NO. 0-06052	REV
	DATE	
	CONTRACT NO.	

NO. OF PAGES _____

RESIN, POLYIMIDE, IMPREGNATING, SPECIFICATION FOR

PREPARED BY _____

CHECKED BY _____

APPROVED BY _____
H. H. Rosenbaum

Preceding page blank

SPECIFICATION CHANGE NOTICES—INCORPORATED IN SPEC REVISIONS

SCN NO(S)	INCORP BY	CHECK BY	APPROVED BY	DATE	SPEC REV	REVISION PACKAGE NO.	RELEASE

INITIAL PACKAGE NO.	RELEASE	DOC TYPE D-3	DISTR CODE

1. SCOPE

1.1 Scope. This material specification establishes the requirements for a heat reactive aromatic resin system which can be thermally cured to a linear polyimide.

1.2 Classification. The material covered by this specification shall be one type and identified as 0-06052-1.

2. APPLICABLE DOCUMENTS

2.1 Unless otherwise specified below, the following documents of the issue in effect on date of Convair's request for quotation form a part of this specification to the extent specified.

STANDARDS

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes

PUBLICATIONS

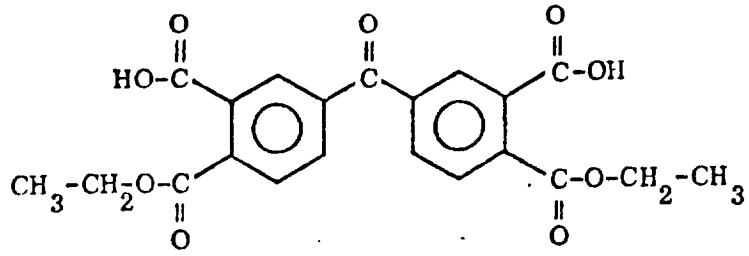
General Industry Safety Orders, State of California, Department of Industrial Relations, Article 85 "Labeling of Injurious Substances".

Manufacturing Chemists Association, Labels and Precautionary Information Committee, "Guide to Precautionary Labeling of Hazardous Chemicals". (Manual L1.)

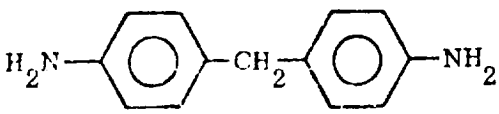
3. REQUIREMENTS

3.1 Chemical structure of polymer.

3.1.1 Varnish. The polyimide varnish (see 6.3.1) shall be chiefly comprised of monomers and low molecular weight polyamic esters. The main components shall be those shown below, or similar ones which will react to form cured polyimides capable of meeting the requirements of this specification.

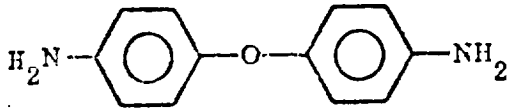


diethylester of benzophenone tetracarboxylic dianhydride



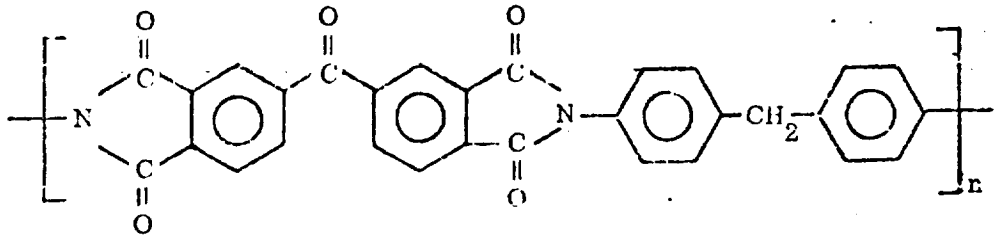
methylene dianiline

or



diamino-diphenylether

3.1.2 Cured resin. The cured resin system shall have the generalized structure shown below. Light crosslinking of the structure is acceptable.



3.2 Resin filler. The use of a silica filler to provide control flow properties to the resin is a requirement. The filler content shall be 2 to 4% by weight.

3.2.1 Foreign material. There shall be no foreign or gelled material present in the filled polyimide varnish except for the standard silica for controlling flow, 3.2.

3.3 Physical properties. The polyimide varnish shall meet the physical property requirements of Table I.

TABLE I

Physical Properties of Polyimide Varnish

Property	Requirements	Test Paragraph
Resin Solids, percent	53 to 57	4.6.2
Viscosity, cps	2000 to 9000	4.6.3
Gel Time, minutes	5 to 20	4.6.4
Specific Gravity	1.09 to 1.14	4.6.5

3.4 Reaction products. As a result of cure, reaction products of ethanol and water shall be released. Other volatiles that shall be released are the initial solvents: NMP, xylene, and additional ethanol. Nominal weight percents of reaction products shall be as follows:

Ethanol - 13%
Water - 1%

3.5 Resin flow and softening points. Nominal temperatures at which the varnish softens and flows is dependent on staging and shall be as shown in Figure 1. The melting point, start of gellation, and gellation points shall agree with those shown in Figure 1 to ± 5K (± 9F), while the flow initiation and "good" flow temperatures shall agree within ± 8K (± 14F).

3.6 Polymer reaction rates. The reaction rates are dependent on temperature and nominal reaction rates shall be similar to those shown in Figure 2. The reaction rate constants (k values) shall be as shown in Table II.

TABLE II

Polymer Reaction Rate Constants

Temperature	Reaction Rate Constant, k ($min.^{-1}$)
373K (212F)	$14.4 \times 10^{-4} \pm 1.4 \times 10^{-4}$
398K (257F)	$5.0 \times 10^{-3} \pm 0.5 \times 10^{-3}$
436K (325F)	$17.3 \times 10^{-2} \pm 1.7 \times 10^{-2}$

3.7 Shelf life. The varnish shall meet the requirements of 3.3 when stored for a minimum of 90 days at 255K (0F).

3.8 Workmanship. The material shall be free of contaminants detrimental to the finished product.

3.9 Toxic or hazardous formulations. If injurious formulations cannot be avoided, all containers of injurious substances shall be labeled as required by Article 85 of California General Industry Safety Orders, or Manual L1 of the Manufacturing Chemist Association.

3.10 Product markings. Product markings shall be in accordance with the preparation for delivery section of this specification. See 5.2.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or order, the varnish supplier is responsible for the performance of all inspections and test requirements as specified herein. Except as otherwise specified, the varnish supplier may use his own facilities or any commercial laboratory acceptable to Convair. Convair, through its preimpregnated material suppliers, reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material furnished or to be furnished conforms to the prescribed requirements.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.3 Classification of examinations and tests. The examinations and tests of the material shall be classified as follows:

a. Qualification verification
b. Acceptance verification
c. Receiving inspection

4.3.1 Qualification verification. Qualification verification shall consist of all the examinations and tests specified herein.

4.3.2 Acceptance verification. Acceptance verification shall be performed on representative samples of each batch of material, and shall consist of the following:

a. Examination of product
b. Resin solids content
c. Viscosity
d. Gel time
e. Specific gravity

4.3.3 Receiving inspection (for Convair or Convair's preimpregnated material suppliers use only). Receiving inspection shall consist of an examination of the material and such sampling and verification of test data as deemed necessary.

4.4 Sampling plan. The material, as offered for acceptance by Convair or Convair's preimpregnated material suppliers, shall meet the requirements herein at an Acceptance Quality Level of 4.0 (normal inspection) when a batch is sampled per MIL-STD-105 at Inspection Level II.

4.4.1 Batch. A batch shall consist of all material of the same type manufactured in one continuous, unchanged production run.

4.5 Test conditions.

4.5.1 Room temperature. Unless otherwise specified, all tests shall be conducted at a temperature of 298K ± 3 (77F ± 5), and 50% ± 10 relative humidity.

4.6 Test methods.

4.6.1 Examination of product. The material shall be examined to verify that its markings, packaging, and visual physical characteristics conform to the requirements of this specification.

4.6.2 Filler content determinations.

4.6.2.1 Preparation of specimens. Preheat three marked drying dishes in the oven at 408K ± 3 (275F ± 5) and weigh to the nearest 0.1 mg (W_1). Weigh a 3-gram ± 0.1 sample (the varnish must be thoroughly mixed) to the nearest 0.1 mg into each of the tared, dried dishes by difference from a weighing tube or syringe (W_2). Place the samples in a muffle furnace and pyrolyze a minimum of 1 hour at 1173K ± 28 (1650F ± 50). Transfer the specimens to a desiccator and cool to room temperature. Reweigh the samples to the nearest 0.1 mg.

4.6.2.2 Calculation. The mean value of three filler content determinations calculated as follows shall be reported.

$$\text{Filler Content, weight percent} = \frac{W_3 - W_1}{W_2} \times 100$$

where W_1 = Weight of drying dish

W_2 = Weight of varnish initially

W_3 = Weight of drying dish plus filler

4.6.3 Solids content.

4.6.3.1 Preparation of specimens. Preheat three marked drying dishes in the oven at 408K ± 3 (275F ± 5) for a minimum of 3 hours. Cool the dishes in a desiccator to room temperature and weigh to the nearest 0.1 mg (W_1). Weigh a 3 gram ± 0.1 sample to the nearest 0.1 mg into each of the tared, dried dishes by difference from a weighing tube or syringe (W_2). Place the samples on a grid tray in a gravity convection type drying oven preheated to 408K ± 3 (275F ± 5) so that the samples are at the same level as the thermometer bulb and grouped around the bulb. Heat for 3 hours ± 3 minutes at 408K ± 3 (275F ± 5), and then transfer the samples to a desiccator and cool to room temperature. Reweigh the samples to the nearest 0.1 mg.

4.6.3.2 Calculation. The mean value of three resin content determinations calculated as follows shall be reported.

$$\text{Resin Content, weight percent} = \frac{W_3 - W_1}{W_2} \times 100$$

where W_1 = Weight of drying dish

W_2 = Weight of varnish initially

W_3 = Weight of drying dish plus specimen after volatile removal

4.6.4 Viscosity. Determine the viscosity of the varnish using a Brookfield Synchro-Lectric Viscometer, Model LVF. Testing shall be conducted at 294K ± 1 (70F ± 2). Place solution to be tested in a jar at least 6.89 cm (2.75 inches) in diameter, cover, and place in a constant temperature bath accurate to ± 0.2K (± 0.4F). Allow material to reach the proper temperature before making viscosity measurement. Check with an accurate thermometer. Select the speed and spindle that will most closely give a reading on the upper end of the dial. The mean value of three viscosity determinations, each made on a separate sample, shall be reported.

4.6.5 Gel time. Gel time of the varnish shall be conducted by use of a General Electric gel meter. Sample weight shall be 5.0 ± 0.1 gm and it shall be held in a clean 18 by 150 mm test tube. The bath temperature shall be 408K ± 1 (275F ± 2). The mean value of three gel time readings, each made on a separate sample, shall be reported.

4.6.6 Specific gravity. The specific gravity of the varnish shall be determined at 298K ± 0.1 (77F ± 0.2) using a Westphal balance. The mean value of three specific gravity readings, each made on a separate sample, shall be reported.

4.6.7 Reaction products. All quantitative determinations of solvent reaction products shall be made using a vapor-phase chromatograph. Determination of water shall be accomplished using the Karl Fisher analytical method. The amounts of ethanol and water in the original varnish shall be determined on volatiles removed by vacuum stripping at 298K ± 3 (77F ± 5), and $<$ 50 mm Hg pressure for a minimum of 15 hours.

4.6.8 Resin flow and softening points. Resin flow and softening points shall be determined on the vacuum stripped polyimide varnish at three different heating rates: 1, 3, and 5K per minute (1.8, 5.4, and 9F per minute) using a Fisher-Johns melting point apparatus. The polyamic acid type residue shall be virtually free of all solvent prior to flow and softening point determinations. Vacuum stripping of solvents shall be conducted at 298K ± 3 (77F ± 5) and $<$50 mm Hg pressure for a minimum of 15 hours. Five different characteristics of the residue shall be determined: (1) melting point, (2) initial flow, (3) good flow, (4) start of gellation, and (5) gellation. Points (2) and (3) are more subjective and shall be more dependent on interpretation by the instrument operator.

4.6.9 Polymer reaction rates. Infrared analysis shall be used to determine polymer reaction rates. The increase in absorption of the imide band shall be plotted versus time to obtain rate constants. A KBr pellet shall be made for every determination for each given time interval at each given temperature. Reaction rate curves shall be obtained at 373K (212F), 398K (257F) and 436K (325F). Conversion rates of polyamic acid to polyimide shall be calculated using the following formula:

$$Y = \log \frac{(\log P/P_o)_{1340}}{(\log P/P_o)_{713}}$$

where P = Transmitted power

P_o = Incident power

P/P_o = Transmittance

subscripts 713 and 1340 = Particular wave lengths

Y = Conversion rate

The rate constant for the reaction at a given temperature shall be determined by plotting

$$\log \left[\frac{\left(\log \frac{P}{P_o}\right)_{1340}}{\left(\log \frac{P}{P_o}\right)_{713}} \right]$$ versus time and multiplying slope by 2.303

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. All material furnished under this specification shall be in suitable containers in quantities as specified on the purchase order. The material shall be packaged to ensure protection from physical damage during handling, shipping, and storage.

5.2 Packing. The material shall be packed in shipping containers of a type which will ensure acceptance by common carrier at lowest rates and will ensure protection of the unit containers during shipment.

5.3 Marking for shipment. Each unit and shipping container shall be legibly identified with label, tag, or markings which include the following data.

a. 0-06052
b. Purchase order number
c. Manufacturer's material description and identifying designation
d. Manufacturer's name and address
e. Quantity (shipping container only) and unit size
f. Batch number and date of manufacture
g. Date of shipping
h. Manufacturer's recommended storage conditions and temperature
i. Hazardous warnings and handling markings as applicable

6. NOTES

6.1 Intended use. The material covered by this specification is intended for impregnating graphite fiber used in the manufacture of missile and spacecraft laminated structural parts subject to temperatures from 20K (-423F) to 589K (600F). Use is not limited to these applications.

6.2 Ordering information. The following information together with the requirements of 6.2.1 and 6.2.2 should be included on the purchase order

a. Number, title, and date of this specification
b. Material name and quantity

6.2.1 Rejection and retest. In the event of failure of a sample to meet any requirements of this specification, a second sample of varnish may be submitted for retest. If the retest sample fails to meet the requirements of the specification, the batch represented by the sample shall be rejected.

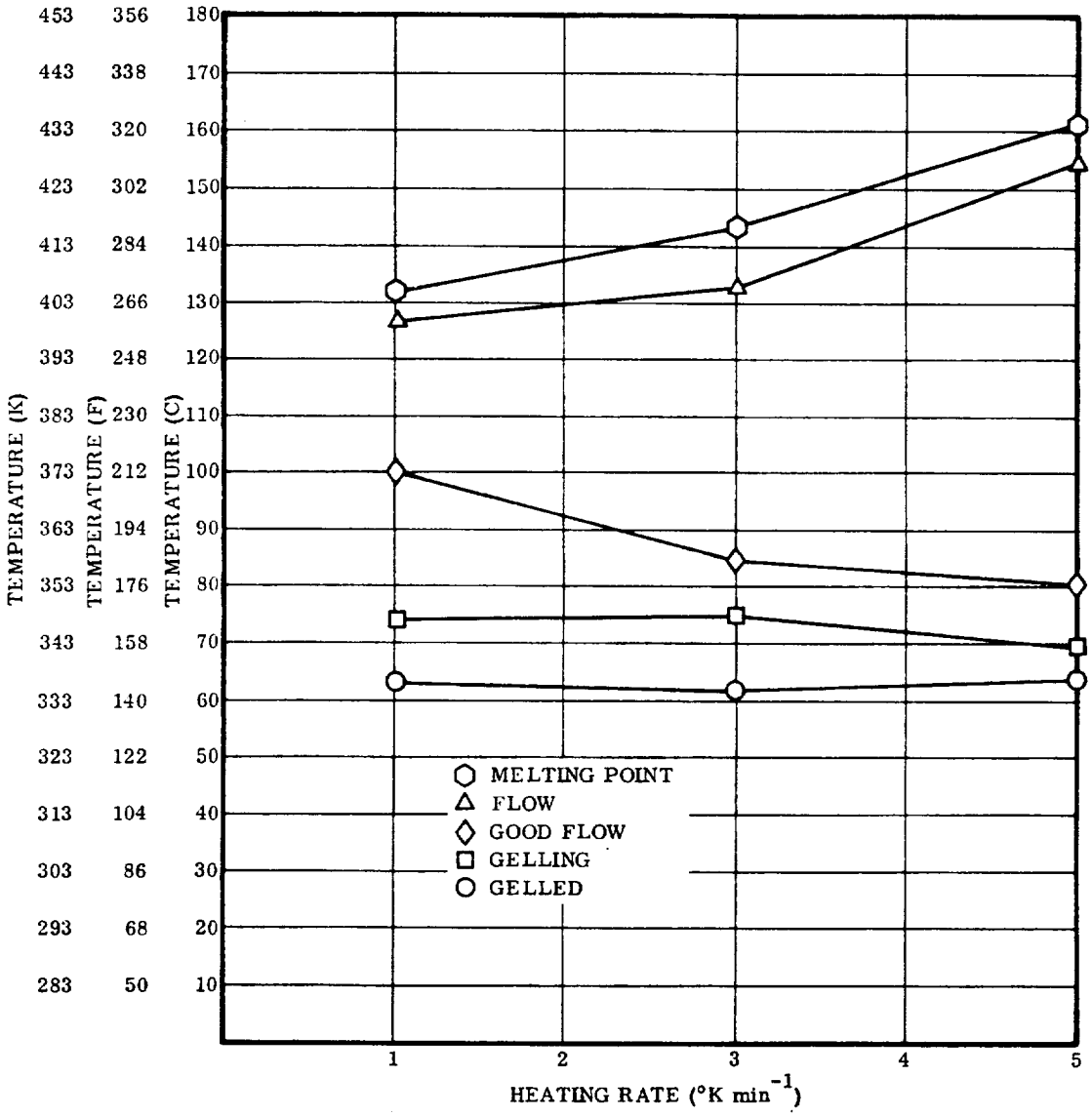
6.2.2 Reports. Unless otherwise specified the supplier shall furnish with each batch three copies of the reports showing the results of tests made on each batch in the shipment to determine conformance of the material to the specification. The report shall include resin solids content, viscosity, gel time, and specific gravity. The reports shall also include the purchase order number, the material specification number, supplier's material designation, quantity, batch number(s), and date(s) of manufacture.

6.3 Definitions.

6.3.1 Varnish. For the purposes of this specification, varnish is defined as a composition of resin and liquid carrier.

6.4 Approved sources. The approved sources for the material described in this specification are as follows:

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
	Skybond 710 polyimide varnish	Monsanto Company P. O. Box 2130 Springfield, Mass. 91101



*AS-RECEIVED VARNISH VACUUM STRIPPED AT 398K (257F) FOR 15 HOURS TO REMOVE MAJORITY OF SOLVENT

Figure 1. Skybond 710 Polyimide Resin Characteristics at Several Heating Rates*

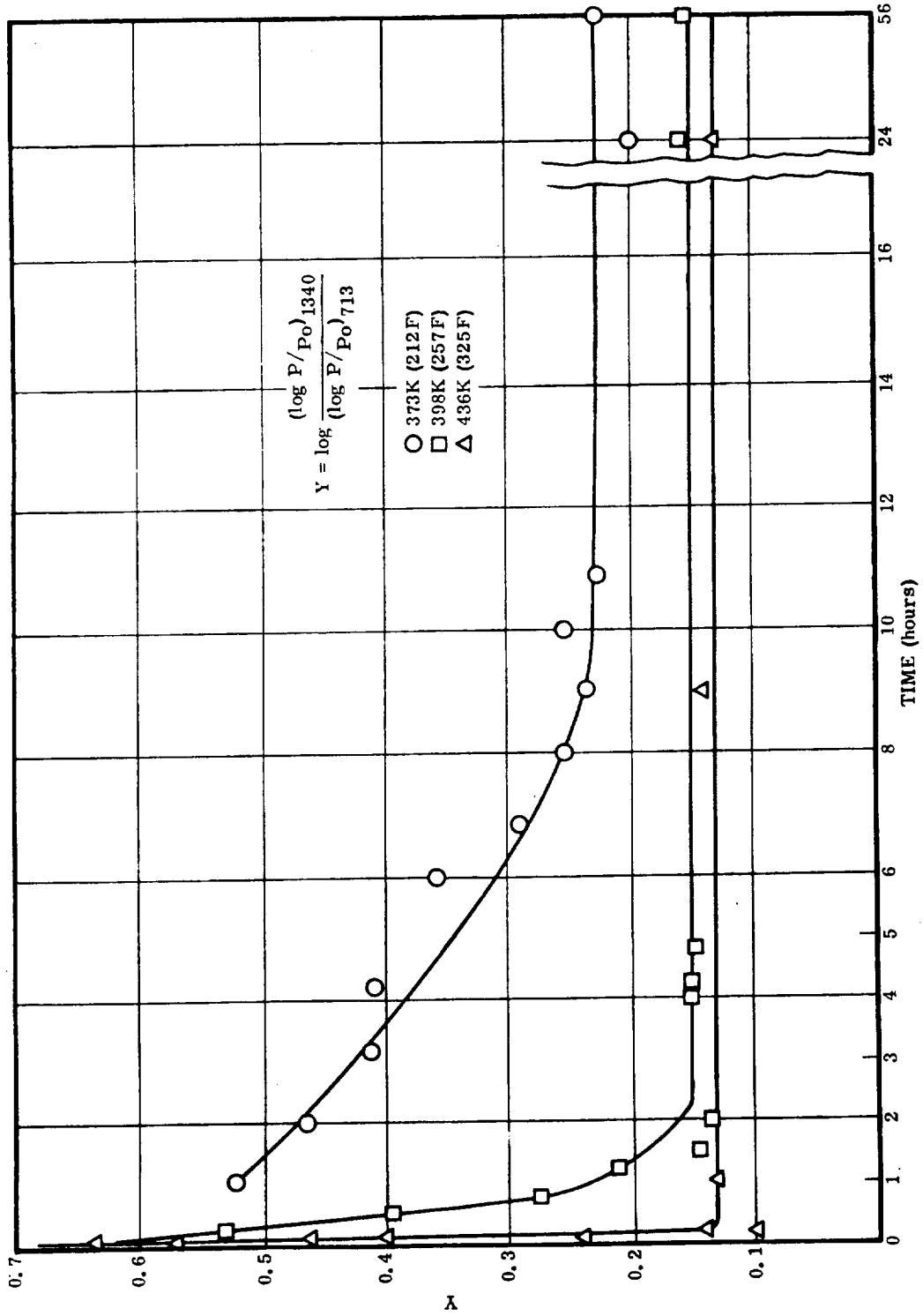


Figure 2. Conversion Rates of Monsanto Skybond 710 Polyamic Acid at Various Temperatures

APPENDIX E

GRAPHITE/EPOXY PREPREG SPECIFICATION

GENERAL DYNAMICS *Convair Division* CODE IDENT NO. 14170	SPECIFICATION NO. 0-06050	REV A
	DATE 8/1/73	
	CONTRACT NO.	

NO. OF PAGES 27

GRAPHITE FLAT SHEET.

EPOXY RESIN IMPREGNATED

TOW, SPECIFICATION FOR

PREPARED BY _____
H. R. Stone

CHECKED BY _____
W. C. Braley

APPROVED BY _____
H. H. Rosenbaum

Preceding page blank

SPECIFICATION CHANGE NOTICES—INCORPORATED IN SPEC REVISIONS

SCN NO(S)	INCORP BY	CHECK BY	APPROVED BY	DATE	SPEC REV	REVISION PACKAGE NO.	RELEASE

INITIAL PACKAGE NO.	RELEASE	DOC TYPE	DISTR CODE
6151 643-4	E-3	20	20-12

1. SCOPE

1.1 Scope. This specification establishes the requirements for graphite flat sheet composed of epoxy resin impregnated tow.

1.2 Classification. The material covered by this specification shall be of one type and identified as 0-06050-1.

2. APPLICABLE DOCUMENTS

2.1 Unless otherwise specified below, the following documents of the issue in effect on date of Convair's request for quotation form a part of this specification to the extent specified.

SPECIFICATIONS

Military

MIL-B-131	Barrier Material: Water Vaporproof, Flexible

Convair

0-00096	Epoxy Compound, 2 Parts Adhesive Bonding, Potting, Sealing and Coating
0-06045	Fabric, Quartz, Polyimide Resin Impregnated (B Stage)
0-73011	Dry Film Structural Adhesive (Epoxy-Phenolic) for Use in Sandwich Construction

Hercules

HD-SG-25001	Test Methods for Determining Physical Properties of Carbon and Graphite Tows.

STANDARDS

Federal

FED-STD-406 Plastics, Methods of Testing

Military

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

PUBLICATIONS

General Industry Safety Orders, State of California Department of Industrial Relations, Article 85, "Labeling of Injurious Substances."

Manufacturing Chemists Association, Labels and Precautionary Information Committee, "Guide to Precautionary Labeling of Hazardous Chemical." (Manual L1.)

3. REQUIREMENTS

3.1 Material. The material shall consist of high strength, high modulus graphite fibers collimated into multifilament tows and impregnated with an epoxy resin.

3.1.1 Graphite fibers. The graphite fibers shall consist of continuous collimated multifilament tow having a minimum modulus of 221 GN/m^2 (32 msi) and a minimum ultimate tensile strength of 2413 MN/m^2 (350 ksi) when tested in accordance with Hercules Specification HD-SG-25001. The fibers shall be compatible with the impregnating resin.

3.1.2 Resin. The resin shall be a high temperature resistant, thermosetting epoxy suitable for both vacuum bag and vacuum augmented autoclave laminating of structural parts.

3.1.3 Handling characteristics. The material, as supplied, shall be suitable for autoclave laminating of contoured parts.

3.1.4 Construction. Unless otherwise specified on the purchase order, the material shall be in the form of broadgood sheets with a nominal width of 30.5 cm (12 in.). When laminated at 690 kN/m^2 (100 psig) the material shall have sufficient tows to produce a nominal 0.0013 cm (0.005 in.) per ply thickness with 60 percent fiber volume. Sheets shall be produced in such manner that a minimal length of 305 cm (10 ft) can be obtained.

3.2 Properties of uncured material.

3.2.1 Physical properties. The uncured material shall meet the requirements of Table I.

TABLE I

Physical Properties of Uncured Material

Property	Requirement	Test Paragraph
Volatile Content, weight percent	3 to 8	4.6.2
Resin Solids Content, weight percent*	36 to 42	4.6.3
Resin Flow, weight percent	15 to 25	4.6.4
Tack	Shall adhere for 30 minutes minimum	4.6.5

*Based on readings from five sheets selected at random. No individual reading shall be less than 34 percent nor greater than 44 percent.

3.2.2 Shelf life. When stored under the conditions noted below, the material shall be capable of meeting the requirements of 3.2.1. Storage time is to be computed from date of receipt of shipment.

a. 90 days at 255K (0 F)

b. 3 days at room temperature

3.2.3 Working life. The material shall possess sufficient room temperature working life that the requirements of 3.2.1 are met after a cumulative exposure of 72 hours in a sealed package and 24 hours unprotected exposure to a temperature of 291 to

300K (65 to 80F) and a relative humidity less than 50%. When material has exceeded its working life, retesting to 3.2.1 is permitted to determine material acceptability.

3.2.4 Workmanship. The material shall be free of contaminants detrimental to the finished product, and shall meet the following requirements for uniformity. In any one sheet, contamination shall be no greater than a total area of 6.45 cm^2 (1 in^2) and this shall be a sum of no more than five individual contaminated spots.

3.2.4.1 Resin. Resin-rich or resin-starved areas, defined as having a resin content greater then forty-five percent or less than thirty-four percent shall not exceed 1.0 percent of the total surface area.

3.2.4.2 Tows. The tows shall be completely wetted. The number of misaligned or overlaid tows occurring in a 7740 cm^2 (1200 in^2) sheet shall not exceed 4 tows per sheet. Misalignment shall not exceed 2 degrees.

3.2.4.3 Gaps. There shall be no fiber gaps wider than 0.025 cm (0.010 in.) in the material with the exception that in a nominal 30.5 by 114 cm (12 by 45 in.) sheet up to 6 gaps are allowed, each having a maximum width of 0.076 cm (0.030 in.) and a maximum length of 7.6 cm (3 in.).

3.2.4.4 Release film. The material shall be protected from self-adhesion by use of a non-migratory crease-free release paper of a contrasting color.

3.2.4.5 Toxic or hazardous formulations. If injurious formulations cannot be avoided, all containers of injurious substances shall be labeled as required by Article 85 of the California General Industry Safety Orders or Manual L1 of the Manufacturing Chemists Association.

3.2.6 Storage of material. Immediately upon receipt by Convair the material, in its original sealed bag, shall be placed in storage at a maintained temperature of 255K (0 F) or below. The material shall be stored horizontally. Material shall never be stored in a vertical position. When material is removed from storage for production or test, the sealed material shall be brought to within 5K (10 F) of room temperature prior to opening the sealed bag. The amount required for use during the remainder of that work shift shall be cut from the sheet and the unused portion shall be replaced in its original bag, the bag resealed by heat application, and the material returned to storage without delay. Bags shall not remain open longer than 1 hour at any one time,

and total elapsed open time shall not be more than 24 hours. Total elapsed time out of storage for any one sealed unit shall never exceed 3 days. At no time shall material which is to be returned to storage be subjected to environmental temperature greater than 302K (85 F).

3.2.7 Storage history. A log of the storage history shall be kept on each unit of material. Material that has exceeded the 4-day (3 days sealed, 1 day open) out-of-storage time or exceeded the 90-day in-storage time shall be retested for conformance to the flow, volatile content, and the flexural requirements of Tables I and II before being used for fabrication of structural parts. Allowable open time shall be considered as in-storage time.

3.2.8 Product markings. Product markings shall be in accordance with the preparation for delivery section of this specification. See 5.3.

3.3 Properties of cured laminates.

3.3.1 Mechanical and physical properties. The cured laminates shall meet the mechanical and physical property requirements of Table II.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or order, the supplier is reponsible for the performance of all inspections and test requirements as specified herein. Except as otherwise specified, the supplier may use his own facilities or any commercial laboratory acceptable to Convair. Convair reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.3 Classification of examinations and tests. The examination and tests of the material shall be classified as follows:

a. Qualification verification

b. Acceptance verification

c. Receiving inspection

TABLE II

Mechanical and Physical Properties of Cured Laminates

Property	Augmented Autoclave Cure RT	Augmented Autoclave Cure 450K(350F)	Test Para.
Longitudinal Tensile Strength, MN/m^2 (ksi), minimum	1172(170)	1034(150)	4.6.7
Longitudinal Tensile Modulus, GN/m^2 (msi), minimum	131(19)	131(19)	4.6.7
Longitudinal Compressive Strength, MN/m^2 (ksi), minimum	896(130)	—	4.6.8
Longitudinal Compressive Modulus, GN/m^2 (msi), minimum	131(19)	131(19)	4.6.8
Longitudinal Flexural Strength, MN/m^2 (ksi), minimum	1448(210)	758(110)	4.6.9
Transverse Flexural Strength, MN/m^2 (ksi), minimum	41(6.0)	21(3.0)	4.6.10
Short Beam Shear Strength, MN/m^2 (ksi), minimum	83(12.0)	41(6.0)	4.6.11
Specific Gravity, minimum	1.550	—	4.6.12
Resin Content, percent by weight, range	28 to 33	—	4.6.13
Fiber Volume, percent, minimum	62 ± 3	—	4.6.14
Water Resistance	No blisters	—	4.6.14

Note:

Test at temperature after 10^{+1}_{-0} minutes exposure.

4.3.1 Qualification verification. Qualification verification shall consist of all the examinations and tests specified herein.

4.3.2 Acceptance verification. Acceptance verification shall be performed on representative samples of each batch of material, and shall consist of the following:

a. Examination of product

b. Volatile content

c. Resin solids content

d. Flow

e. Tack

f. Longitudinal flexural strength at room temperature and 450K(350F)

g. Short beam shear strength at room temperature and 450K(350F)

4.3.3 Receiving inspection (for Convair use only). Receiving inspection shall consist of an examination of the material and such sampling and verification of test data as deemed necessary.

4.4 Sampling plan. The material, as offered for acceptance by Convair, shall meet the requirements specified herein at an Acceptance Quality Level of 2.5 (normal inspection) when a batch is sampled per MIL-STD-105 at Inspection Level II for Items 4.3.2a through 4.3.2e. Items 4.3.2f and 4.3.2g shall be inspected by random selection of one sealed unit of material per batch.

4.4.1 Batch. A batch shall consist of all material of the same type manufactured in one continuous, unchanged production run.

4.4.2 Unit of product. A unit of product shall consist of one sealed unit of material, and shall contain no more than 20 sheets.

4.5 Test conditions.

4.5.1 Room temperature. Unless otherwise specified, all tests shall be conducted at a temperature of 298K±3 (77F±5) and 50 percent ±10 relative humidity.

4.5.2 Exposure at 450K(350F). Test panels shall be raised to 450K±6(350F±10) within 10 minutes in a test chamber previously heated to temperature, held at temperature for 10^{+1}_{-0} minutes, and tested immediately.

4.6 Test methods.

4.6.1 Exmaination of product. The material shall be examined to verify that its markings, packaging and all visual physical characteristics conform to the requirements of this specification.

4.6.2 Volatile content.

4.6.2.1 Preparation of specimens. Cut a minimum of six speciments of the material, each approximately 5.1 by 5.1 cm (2 by 2 in.), from an area of sample away from the edges. The specimen may be any shape that will fit into a pre-weighed and fired crucible or a disposable aluminum foil continaer, but may not be rolled up or more than one-ply lamina. Half of the specimens are to be processed for volatile content determinations, while the others are to be processed for resin solids content determinations.

4.6.2.2 Drying and weighing. Weigh a crucible on an analytical balance to nearest 0.1 mg (W_2). Place the test specimen in the crucible and record the total weight to the nearest 0.1 mg (W_1). Place the crucible containing the specimen in a preheated 436K±3(325F±5) forced air oven for 15^{+1}_{-0} minutes, cool to room temperature, and weigh to the nearest 0.1 mg (W_3).

4.6.2.3 Calculation. The mean value of three volatile content determinations calculated as follows shall be reported.

$$\text{Voltaile content, weight percent} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

where

W_1 = weight of crucible plus specimen before volatile removal

W_2 = weight of crucible

W_3 = weight of cruicible plus specimen after volatile removal.

4.6.3 Resin solids content.

4.6.3.1 Test procedure. Half of the specimens cut per 4.6.2.1 are used for resin solids content determinations. Weigh a specimen to the nearest 0.1 mg (W_4). Place the specimen in a 30 ml tall-form beaker and add 24 ml of dimethyl formamide (DMF), technical grade. Place the beaker on a hot plate and heat to boiling for 10 to 15 minutes. Remove the beaker from the hot plate and allow to cool to room temperature. Pour off the DMF, being careful not to lose any graphite fibers. Rinse the graphite fiber residue two or three times in acetone. After removing most of the acetone, place the graphite fibers in a tared aluminum cup (throwaway type) and dry for 30 minutes minimum in an air oven at 436K 3(325F 5). Determine the weight of graphite fibers (W_5) to the nearest 0.1 mg and record.

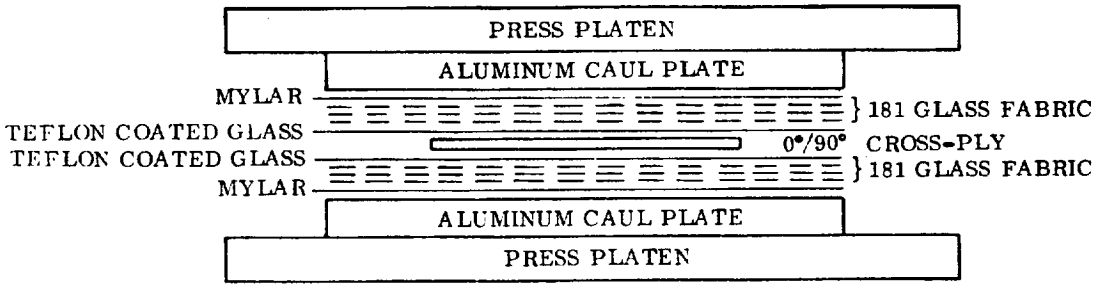
4.6.3.2 Calculation. The mean value of three resin solids content determinations calculated as follows shall be reported.

$$100 - \left[\frac{W_5}{W_4} \times 100\right] = \text{percent by weight of (resin plus volatiles)}$$

$$100 - \left[\frac{W_5}{W_4} \times 100\right] - [\text{percent volatile content (from 4.6.2.3)}] = \text{resin solids content (percent by weight)}$$

4.6.4 Resin flow.

4.6.4.1 Preparation of specimens. Cut six pieces of style 181 glass fabric 10.2 by 10.2 cm (4 by 4 in.) square for bleeder and two squares of Teflon coated glass fabric of equal size for separators. Weigh the above to the nearest 0.1 mg on an analytical balance. Cut two pieces of the material to be tested, 5.1 by 5.1 cm (2 by 2 in.) and weigh, plus the separator and bleeder, to the nearest 0.1 mg. Cross ply the two specimens and assemble, as shown below, in a preheated 436K±3(325F±5) press. Cure for 15^{+1}_{-0} minutes at 436K(325F) under 690kN/m^2(100 psig). Remove the cross-ply test specimens from the separator and bleeder. Weigh the separator plus bleeder to the nearest 0.1 mg.



4.6.4.2 Calculations. Calculate the flow according to the following equation:

$$\text{Percent Flow} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where

W_1 = weight of glass fabric plus Teflon coated glass fabric

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

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

The mean value for three determinations shall be reported.

4.6.5 Tack.

4.6.5.1 Procedure. A corrosion resistant steel sheet, alloy 302 or equivalent with a commercial 2D finish, any thickness by 10.2 by 20.3 cm (4 by 8 in.) shall be cleaned to a water-break-free condition with chlorine-free scouring powder and distilled water, then air dried at a temperature below 340K(150F). A specimen cut to 2.5 by 7.6 cm (1 by 3 in.) shall be attached to the plate with light pressure applied by squeegee or roller over the backing film. The backing film shall then be removed. A second strip of the material, cut to the same dimensions, shall be attached in the same manner to the first strip applied and the backing film removed. The plate shall be maintained in a vertical position for 30 minutes minimum while at 291 to 300K(65 to 80F).

4.6.5.2 Calculations. A minimum of three determinations, all of which must pass the test, shall be reported.

4.6.6 Preparation of test panels. Flat laminate test panels, as required, shall be fabricated using the processes and procedures described below. Total number of plies shall be dictated by the test specimen thicknesses specified in the following test methods.

4.6.6.1 Panel layup. Flat laminate test panels of appropriate size, but no smaller than 15.2 by 15.2 cm(6 by 6 in.), shall be prepared by a parallel layup of unidirectionally oriented plies of the material. Dams shall be used around the perimeter of the panels to prevent fiber wash out. A release agent shall be used on the caul plate to prevent panel sticking problems. Bleeder shall consist of one ply of Mauchberg paper CW-1850 per every three plies of material, and all the bleeder shall be above the layup. The bleeder shall be separated both top and bottom from the material layup by a ply of perforated Teflon coated glass fabric. A pressure plate of the same size as the basic layup shall be used to minimize surface waviness. This plate shall be separated from the layup by a thin film of Teflon. Figure 1 shows typical layup. Curing shall be conducted by vacuum augmented autoclave cycle.

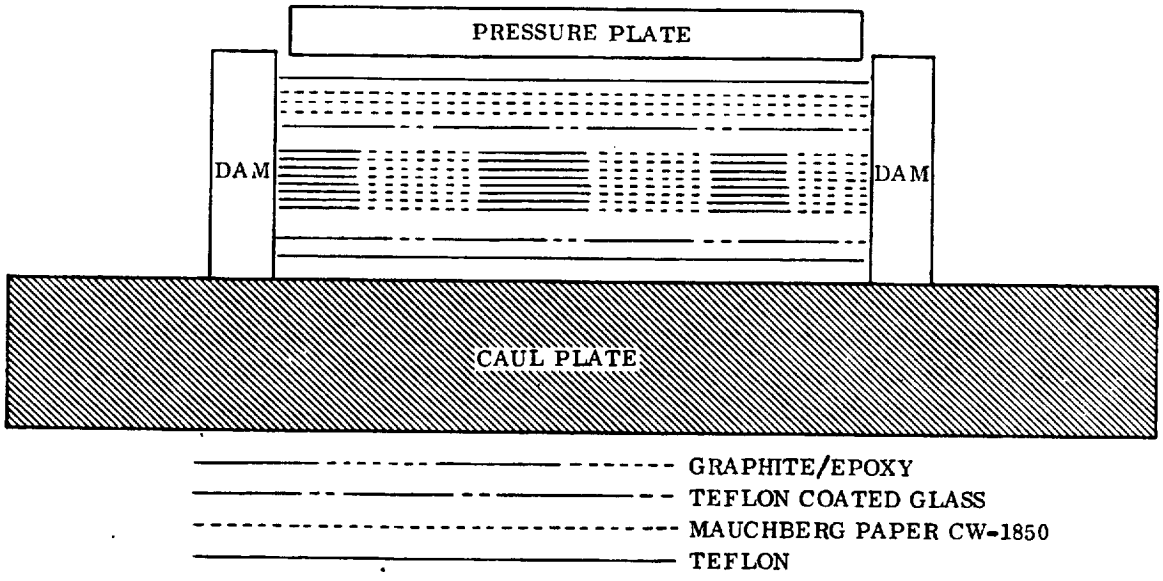


Figure 1. Layup of Unidirectional Graphite/Epoxy Panel

4.6.6.2 Vacuum augmented autoclave cure. The layup as described in 4.6.6.1 shall be vacuum bagged with a film material capable of withstanding long term exposure to 477K(400F). The entire assembly of caul plate, layup, vacuum bag, etc., shall be placed in an autoclave and cured using the following cycle of temperature and pressure.

a. Apply a minimum vacuum of 63.5 cm(25 in.) of mercury on the layup.

b. Raise laminate temperature to 361 to 366K(190 to 200F) at a rate of 1.7 to 2.8K(3 to 5F) per minute and hold temperature for 15^{+1}_{-0} minutes.

c. Apply 690 $kN/m^2 \pm 34$(100 psig±5) autoclave pressure and continue to hold temperature at 361 to 366K(190 to 200F) for an additional 60^{+5}_{-0} minutes.

d. Raise laminate temperature to 422K(300F) and hold at 422K±6(300F±10) for 1 hour ±5 minutes.

e. Raise laminate temperature to 463K(375F) at a rate of 1.7 to 2.8K(3 to 5F) per minute and hold at 463K±6(375F±10) for 4 hours ±15 minutes.

f. Cool laminate under pressure to below 339K(150F) before removal from autoclave.

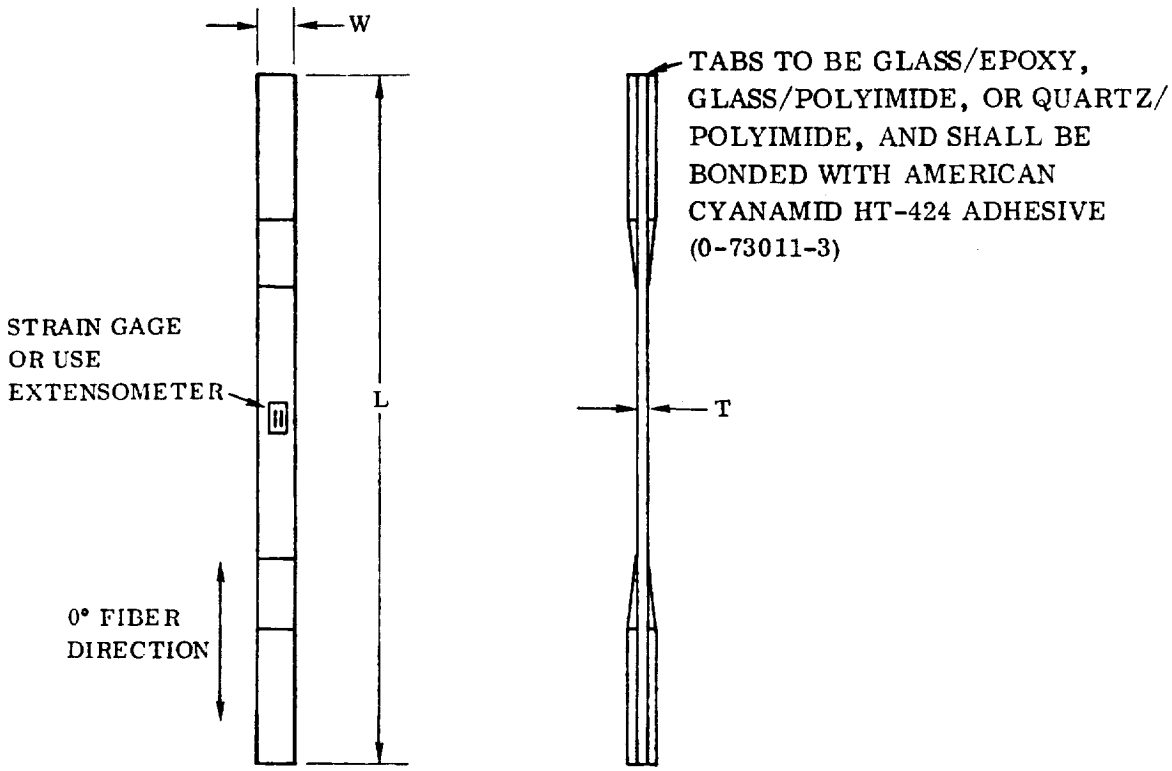
g. Post cure laminate at 463K±8(375±15) for 20 hours ±15 minutes under vacuum bag pressure.

4.6.7 Tensile strength and modulus.

4.6.7.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.7.2 Specimen. The specimen shall be a straight-sided coupon with adhesive-bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be parallel to the longitudinal axis. The tensile specimen configuration is described in Figure 2.

4.6.7.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1011. The specimen shall be loaded to failure at a 0.127 cm±0.013(0.050 in.±0.005) per minute cross head speed in a testing machine using either strain gage or extensometer readings for strain measurements. Test temperatures shall be at room temperature and at 450K±6(350F±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5).



Specimen Dimensions:

Length (L)	=	20.32 cm (8.00 in.)
Width (W)	=	1.270 cm (0.500 in.)
Thickness (t)	=	0.076 to 0.152 cm (0.030 to 0.060 in.)
Total Tab Length	=	6.35 cm (2.50 in.)
Tab Chamfer Length	=	1.91 cm (0.75 in.)
Tab Thickness	=	0.076 to 0.152 cm (0.030 to 0.060 in.)

Specimen edges shall be parallel to 0.0076 cm (0.003 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Tabs for room temperature test specimens may be made of crossplied Scotchply 1002 or Scotchply 1007. Tabs for specimens to be tested at 450K (350 F) are to be made of Style 581 Quartz or 181 Glass fabric impregnated with Monsanto's Skybond 703 resin (0-06045-1). These latter tabs may also be used for room temperature test specimens.

Figure 2. Longitudinal Tensile Test Specimen

4.6.7.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both tensile strength and modulus using the formulas given below.

a. Ultimate tensile strength

$$F_t = \frac{P_t}{A}$$

where

F_t = ultimate tensile strength

P_t = maximum tensile load carried by the specimen

A = specimen cross-sectional area

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straight-line portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain. See Figure 3.

$$E = \frac{F_t}{\epsilon}$$

where

E = modulus of elasticity in tension

F_t = typical tensile stress

ϵ = corresponding strain

4.6.8 Compressive strength and modulus.

4.6.8.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.8.2 Specimen. The specimen shall be a straight-sided sandwich specimen having adhesive bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be parallel to the longitudinal axis. The compression specimen configuration is described in Figure 4.

4.6.8.3 Procedure. The specimen shall be loaded to failure at a 0.102 cm ±0.010 (0.040 in. ±0.005) per minute crosshead speed in a testing machine using strain gage or

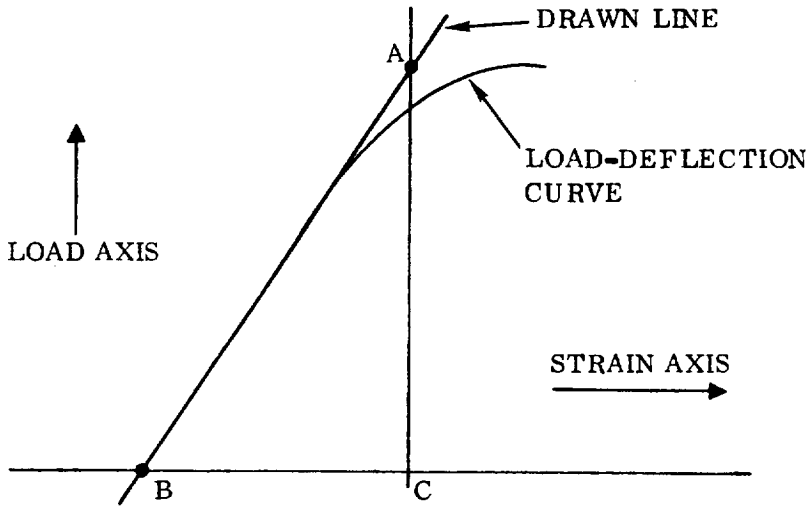


Figure 3. Typical Load-Deflection Curve for Tensile Strength Tests

compressometer readings for strain measurements. Test temperatures shall be at room temperature and at 450K ±6(350F ±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5).

4.6.8.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both compression strength and modulus using the formulas given below.

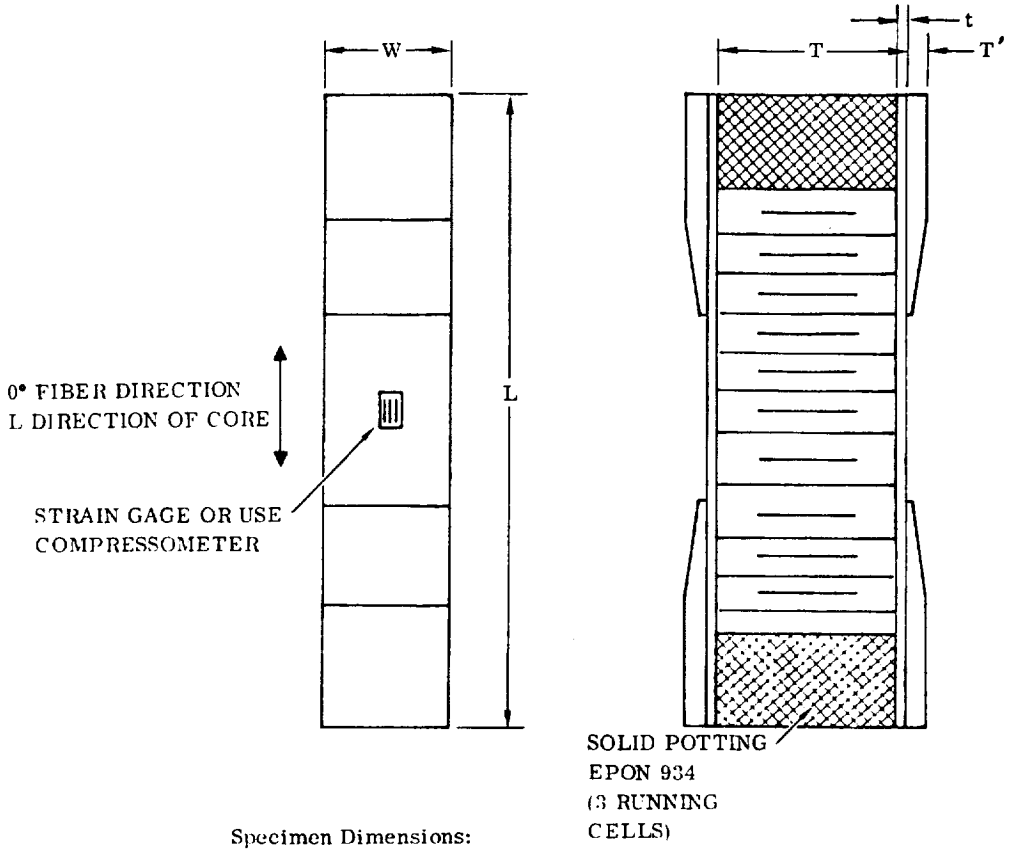
a. Ultimate compression strength

$$F_c = \frac{P_c}{A}$$

where

F_c = ultimate compression strength

P_c = maximum compression load carried by the specimen



Specimen Dimensions:

Length (L) - 8.9 cm (3.5 in.)

Width (W) - 1.270 cm (0.500 in.)

Core Thickness (T) - 2.540 cm (1.000 in.)

Face Thickness (t) = 0.076 to 0.152 cm (0.030 to 0.060 in.)

Tab Thickness (T') = 0.102 to 0.152 cm (0.040 to 0.060 in.)

Total Tab Length = 3.18 cm (1.25 in.)

Tab Chamfer Length = 1.91 cm (0.75 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Core to be 0.318 cm (0.125 in.) cell, 2.8 to 19.2 kg/m^3 (8 to 12 lb/ft^3) aluminum honeycomb.
3. Adhesive to be American Cyanamid HT-424 (0-73011-3).
4. Ends to be potted with Shell Chemical Co. Epon 934 (0-00096-52).
5. Ends to be machined flat and parallel within ±0.0025 cm (±0.001 in.).
6. Specimen sides shall be parallel to 0.0076 cm (0.003 in.) Polish edges after machining.

Figure 4. Longitudinal Compression Test Specimen

A = the sum of the cross-sectional area of both faces

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straight-line portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain.

$$E = \frac{F_{typ}}{\epsilon}$$

where

E = modulus of elasticity in compression

F_{typ} = typical compression stress

ϵ = corresponding strain

4.6.9 Longitudinal flexural strength.

4.6.9.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.9.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 5.

4.6.9.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1031. The specimen shall be loaded to failure at 0.130 cm ±0.013(0.050 in. ±0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 450K±6(350F±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 5 with the smooth side up.

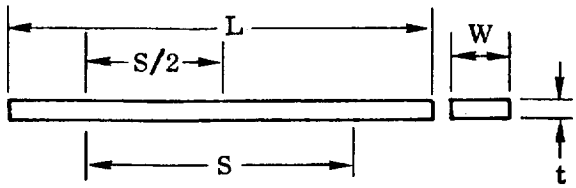
4.6.9.4 Calculation. A mean value based on a minimum of three determinations shall be reported for longitudinal flexural strength using the formula below:

$$F_L = \frac{3PS}{2Wt^2}$$

where

F_L = ultimate longitudinal flexural strength

P = maximum load carried by the specimen



Specimen Dimensions

Length (L) = 7.6 to 10.2 cm (3.0 to 4.0 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 32 to 1

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 5. Longitudinal Flexure Test Specimen

S = span

W = specimen width

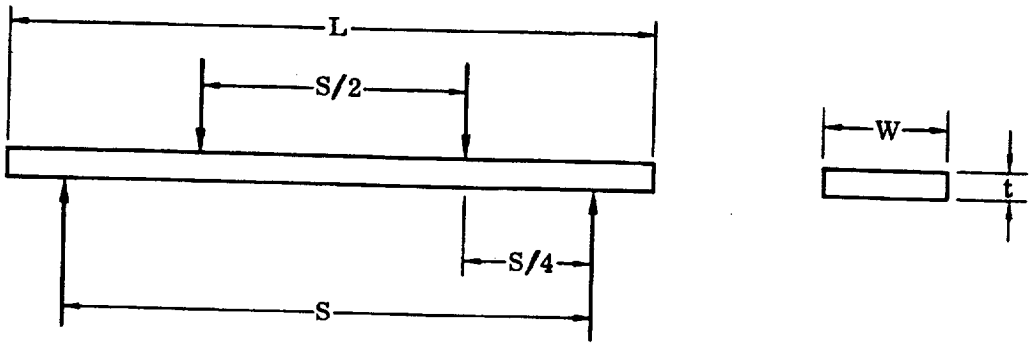
t = specimen thickness

4.6.10 Transverse flexural strength.

4.6.10.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.10.2 Specimen. Fibers are aligned transverse to the longitudinal axis. Specimen configuration is shown in Figure 6.

4.6.10.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1031. The specimen shall be loaded to failure at a 0.130 cm ±0.013(0.050 in. ±0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 450K±6(350F±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 6 with the smooth side up.



Specimen Dimensions:

Length (L) = 7.6 cm (3.0 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span = 5.08 cm (2.00 in.)

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 6. Transverse Flexure Test Specimen

4.6.10.4 Calculation. Mean value based on a minimum of three determinations shall be reported for transverse flexural strength using the formula below:

$$F_t = \frac{3PS}{4Wt^2}$$

where

F_t = ultimate transverse flexural strength

P = maximum load carried by the specimen

S = span

W = specimen width

t = specimen thickness

4.6.11 Short beam shear strength.

4.6.11.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.11.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 7.

4.6.11.3 Procedure. The specimen shall be loaded to failure at a 0.130 cm ±0.013 (0.050 in. ±0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 450K ±6(350F±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 7 with the smooth side up.

Specimen Dimensions:

Length (L) = 1.52 cm (0.060 in.)

Width (W) = 0.635 cm (0.250 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 4 to 1

Loading head and reaction supports are 0.318 cm (0.125 in.) diameter steel rod

Overhang must be the same over each end

Figure 7. Short Beam Shear Specimen

4.6.11.4 Calculation. A mean value based on a minimum of three determinations shall be reported for short beam shear strength, using the formula below:

$$\tau = \frac{3P}{4Wt}$$

where

τ = ultimate short beam shear strength

P = maximum load carried by specimen

W = specimen width

t = specimen thickness

4.6.12 Specific gravity.

4.6.12.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.12.2 Test method. Specimen configuration and test procedure shall be in accordance with Federal Test Method Std. No. 406, Method 5011.

4.6.12.3 Calculation. Specific gravity calculations shall be per Federal Test Method Std. No. 406, Method 5011. A mean value based on a minimum of three determinations shall be reported.

4.6.13 Cured resin content and fiber volume.

4.6.13.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.13.2 Specimen. Test specimens shall be approximately 1.27 by 1.27 cm (0.5 by 0.5 in.) by laminate thickness. (Take specimens from a test panel used in a previous test conducted at room temperature.)

4.6.13.3 Procedure. The cured resin content and fiber volume shall be determined by acid/peroxide digestion as follows:

a. Weigh the test specimen to the nearest 0.1 mg (W_1), place in a 300 ml tall-form beaker, and add 20 ml of concentrated sulfuric acid. Place the beaker on a hot plate and heat the acid until vigorous fuming occurs.

b. When the composite is visibly disintegrated and resin particles and fibers are dispersed throughout the sulfuric solution, carefully add the hydrogen peroxide (50 percent strength) dropwise down the side of the beaker. Rubber gloves and a fume hood with safety glass shield shall be used throughout the addition, and precautions shall be taken as recommended by the applicable safety regulations and procedures for handling hydrogen peroxide.

c. The reaction is considered complete when the hot sulfuric acid solution below the fibers becomes clear and colorless. At this point add two more ml of hydrogen peroxide to the solution, and heat solution to fumes for another 10 minutes to ensure complete decomposition of the polymer. Remove the beaker from the hot plate and allow to cool to 294 to 300K(70 to 80F), and then place in an ice bath.

d. Collect the fibers by vacuum filtration through a medium-porosity, sintered-glass crucible that has been weighed to nearest mg (W_2). After the sulfuric acid has been filtered off, wash the fibers in the crucible thoroughly with 600 ml of distilled water, added a few milliliters at a time. Verify removal of sulfuric acid traces by checking pH of the filtrate drops.

e. Remove the crucible from the filtering system and place in an open beaker. Dry in an oven at 422K(300F) for 45 minutes. After drying, cool the crucible in a desiccator and weight (W_3).

4.6.13.4 Resin fiber content calculation. Calculate the resin and fiber content according to the following equation:

$$\text{Resin content, percent by weight } (W_4) = \frac{W_1 - (W_3 - W_2)}{W_1} \times 100$$

$$\text{Fiber content, percent by weight } (W_5) = \frac{W_3 - W_2}{W_1} \times 100$$

4.6.13.5 Fiber volume calculation. Calculate the fiber volume using the data generated from the resin and fiber content determinations and the following formula.

$$\text{Fiber volume, percent} = \frac{W_6}{W_6 + (W_7 - W_6)\frac{\rho_1}{\rho_2}} \times 100$$

where

ρ_1 = density of fiber

ρ_2 = density of epoxy resin

W_7 = weight of laminate = 1.00

W_6 = weight fraction of dray graphite fiber = $\frac{W_5}{100}$

A mean value based on three determinations shall be reported.

4.6.14 Water resistance. A piece of test panel, 5.08 by 7.62 cm(2 by 3 in.) shall be immersed in boiling water for a period of 24 hours. Upon removal from the boiling water, the panel shall be cooled to room temperature and visually examined for blisters.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. The material shall be packaged with a non-adherent separator applied to both sheet faces. The material within one package shall be of the same length and width to preclude damage to the material during shipment. Packaged material shall be sealed within a moisture-proof plastic bag meeting the requirements of Military Specification MIL-B-131.

5.2 Packing. The packaged material shall be packed in shipping containers of a type which will ensure acceptance by common carrier at lowest rates and will ensure protection of the material during handling, transit, and storage.

5.3 Marking of interior package. Each interior package shall be legibly marked with a label or tag whcih includes the following minimum information.

a. 0-06050-1

b. Manufacturer's material designation

c. Manufacturer's name and address

d. Batch number and sheet number

e. Date of manufacture

f. Weight

g. Recommended storage conditions and temperature range for maximum shelf life

h. Estimated maximum shelf life based on recommended storage conditions and temperature range

i. Hazardous warnings as applicable

5.4 Marking of exterior shipping container. Each exterior shipping container shall be legibly and permanently marked with the following information.

a. 0-06050-1

b. Purchase order number

c. Manufacturer's material designation

d. Manufacturer's name and address

e. Batch number

f. Quantity contained (sheet size and number of sheets)

g. Date of manufacture

h. Date of shipping

i. Manufacturer's recommended storage conditions and temperature range

j. Precautionary and handling markings

In addition, the shipping container shall be identified with a strip of one-inch wide green plastic tape (Scotchlite 3277 or equivalent) completely around the container from top to bottom and approximately 2.54 cm (1 in.) from the side.

6. NOTES

6.1 Intended use. The material covered by this specification is intended for use in the manufacture of missile and spacecraft structural components subject to temperatures from 20K(-423F) to 450K(350F). Use is not restricted to these applications.

6.2 Ordering information. The following information should be included on the purchase order together with the conditions of 6.2.1 and 6.2.2.

a. Number, title, and date of specification

b. Material name and quantity

6.2.1 Rejection and retest. In the event of failure of a sample to meet any of the requirements of this specification, a second sample of uncured material taken adjacent to the first, or a second laminate panel prepared in accordance with 4.6.6 may be submitted for retest. If the retest sample fails to meet the requirements of the specification, the batch represented by the sample shall be rejected.

6.2.2 Reports. Unless otherwise specified, the supplier shall furnish with each shipment three copies of the reports showing the results of tests made on each batch in the shipment to determine conformance of the material to the specification requirements. The report shall include volatile content, resin solids, resin flow, tack and mechanical properties, as applicable. The report shall also include the purchase order number, the material specification number, supplier designation, quantity, batch number, and date of manufacture. The report shall also include fiber properties as reported by the fiber manufacturer, and the batch numbers of fiber used in making each of the sheets of material.

6.3 Approved sources. The approved sources for the material described in this specification are as follows:

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
0-06050-1	hy-E-1311-B(HT-S/X-904)	Fiberite Corporation 515 West Third Street Winona, Minnesota 55987

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
0-06050-1	Ferropreg C-5199	Ferro Corporation 18781 Fiber Glass Road Huntington Beach, California 92646
0-6050-1	WRD373/HT-3	Whittaker Corporation 3540 Aero Court San Diego, California 92112

APPENDIX F

GRAPHITE/EPOXY PREPREG SPECIFICATION

GENERAL DYNAMICS *Convair Division* CODE IDENT NO. 14170	SPECIFICATION NO. 0-06051	REV A
	DATE 8-1-73	
	CONTRACT NO.	

NO. OF PAGES: 25

GRAPHITE FLAT SHEET, EPOXY RESIN IMPREGNATED YARN, SPECIFICATION FOR

PREPARED BY H. R. Stone

APPROVED BY H. H. Rosenbaum

CHECKED BY W. C. Braley

Preceding page blank

SPECIFICATION CHANGE NOTICES—INCORPORATED IN SPEC REVISIONS

SCN NO(S)	INCORP BY	CHECK BY	APPROVED BY	DATE	SPEC REV	REVISION PACKAGE NO.	RELEASE

	INITIAL PACKAGE NO.		RELEASE	DOC TYPE	DISTR CODE
6151	572-4		F-3	20	20-12

1. SCOPE

1.1 Scope. This specification establishes the requirements for graphite flat sheet composed of epoxy resin impregnated yarn.

1.2 Classification. The material covered by this specification shall be of one type and identified as 0-06051-1.

2. APPLICABLE DOCUMENTS

2.1 Unless otherwise specified below, the following documents of the issue in effect on date of Convair's request for quotation form a part of this specification to the extent specified.

SPECIFICATIONS

Military

MIL-B-131	Barrier Material: Water Vaporproof, Flexible

Convair

0-00096	Epoxy Compound, 2 Parts Adhesive Bonding, Potting, Sealing and Coating
0-06045	Fabric, Quartz, Polyimide Resin Impregnated (B Stage)
0-73011	Dry Film Structural Adhesive (Epoxy-Phenolic) For Use in Sandwich Construction

Celanese

SUM-S-108G	Summit Testing Procedure - Standard Method for Tensile Strength of Graphite Fiber

STANDARDS

Federal

FED-STD-406 Plastics, Methods of Testing

Military

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

PUBLICATIONS

General Industry Safety Orders, State of California Department of Industrial Relations, Article 85, "Labeling of Injurious Substances."

Manufacturing Chemists Association, Labels and Precautionary Information Committee, "Guide to Precautionary Labeling of Hazardous Chemicals." (Manual Ll.)

3. REQUIREMENTS

3.1 Material. The material shall consist of high strength, high modulus graphite fibers collimated into multifilament yarns and impregnated with an epoxy resin.

3.1.1 Graphite fibers. The graphite fibers shall consist of continuous collimated multifilament yarn having minimum modulus of 483 GN/m^2 (70 msi) and a minimum tensile strength of 1517 MN/m^2 (220 ksi) when tested in accordance with Celanese Specification SUM-S-108G. The fibers shall be compatible with the impregnating resin.

3.1.2 Resin. The resin shall be a high temperature resistant, thermo-setting epoxy suitable for vacuum augmented autoclave laminating of structural parts.

3.1.3 Handling characteristics. The material, as supplied, shall be suitable for autoclave laminating of contoured parts.

3.1.4 Construction. Unless otherwise specified on the purchase order, the material shall be in the form of broadgood sheets with a nominal width of 30.5 cm (12 in.). The broadgood sheets shall be constructed of butted 7.6 cm (3 in.) wide preimpregnated woven tape. The tape shall have 2 to 3 fill yarns per 2.54 cm (1 in.). When laminated at 690 KN/m^2 (100 psig) the material shall have sufficient yarns to produce a nominal 0.015 cm (0.006 in.) per ply thickness with 60 percent fiber volume. Sheets shall be produced in such a manner that a minimal length of 305 cm (10 ft) can be obtained.

3.2 Properties of uncured material.

3.2.1 Physical properties. The uncured material shall meet the requirements of Table I.

TABLE I

Physical Properties of Uncured Material

Property	Requirement	Test Paragraph
Volatile Content, weight percent*	3 to 8	4.6.2
Resin Solids Content, weight percent*	36 to 42	4.6.3
Resin Flow, weight percent	15 to 25	4.6.4
Tack	Shall adhere for 30 minutes minimum	4.6.5

*Based on readings from five sheets selected at random. No individual reading shall be less than 34 percent nor greater than 44 percent.

3.2.2 Shelf life. When stored under the conditions noted below, the material shall be capable of meeting the requirements of 3.2.1. Storage time to be computed from date of receipt of shipment.

a. 90 days at 255K (0F)
b. 3 days at room temperature

3.2.3 Working life. The material shall possess sufficient room temperature working life that the requirements of 3.2.1 are met after a cumulative exposure of 72 hours in a sealed package and 24 hours unprotected exposure to a temperature of 291 to 300K (65 to 80F) and a relative humidity of less than 50%. When material has exceeded its working life, retesting to 3.2.1 is permitted to determine material acceptability.

3.2.4 Workmanship. The material shall be free of contaminants detrimental to the finished product, and shall meet the following requirements for uniformity. In any one sheet, contamination shall be no greater than a total area of 6.45 cm^2 (1 $in.^2$) and this shall be a sum of no more than five individual contaminated spots.

3.2.4.1 Resin. Resin-rich or resin-starved areas, defined as having a resin content greater than 45 percent or less than 34 percent shall not exceed 1.0 percent of the total surface area.

3.2.4.2 Yarns. The yarns shall be completely wetted. The number of misaligned or overlaid yarns occurring in a nominal 30.5 by 114 cm (12 by 45 in.) sheet shall not exceed 4 yarns per sheet. Misalignment shall not exceed 2 degrees over any 0.305 m (1.0 ft) section. Cumulative misalignment is not permitted. Up to 10 discrepancies in fiber alignment are allowed as long as not more than 4 of these discrepancies are greater than 2.54 cm (1 in.) in length. Fuzzing in areas less than 2.54 cm (1 in.) in diameter are acceptable; fuzzing greater than 2.54 cm (1 in.) in diameter is treated as misaligned fibers.

3.2.4.3 Gaps. There shall be no fiber gaps wider than 0.025 cm (0.010 in.) in the material with the exception that in a nominal 30.5 by 114.3 cm (12 by 45 in.) sheet, up to 6 gaps are allowed, each having a maximum width of 0.076 cm (0.030 in.) and a maximum length of 7.6 cm (3 in.).

3.2.4.4 Release film. The material shall be protected from self-adhesion by use of a non-migratory crease-free release paper of a contrasting color.

3.2.5 Toxic or hazardous formulations. If injurious formulations cannot be avoided, all containers of injurious substances shall be labled as required by Article 85 of the California General Association.

3.2.6 Storage of material. Immediately upon receipt by Convair the material, in its original sealed bag, shall be placed in storage at a maintained temperature of 255K (0F) or below. The material shall be stored horizontally. Material shall never be stored in a vertical position. When material is removed from storage for production or test, the sealed material shall be brought to within 5K (10F) of room temperature prior to opening the sealed bag. The amount required for use during the remainder of that work shift shall be cut from the sheet and the unused portion shall be replaced in its original bag, the bag resealed by heat application, and the material returned to storage without delay. Bags shall not remain open longer than 1 hour at any one time, and total elasped open time shall not be more than 20 hours. Total elapsed time out of storage for any one sealed unit shall never exceed 3 days. At no time shall material which is to be returned to storage be subjected to environmental temperature greater than 302K (85F).

3.2.7 Storage history. A log of the storage history shall be kept on each unit of material. Material that has exceeded the 4-day (3 days sealed, 1 day open) out-of-storage time or exceeded the 90-day in-storage time shall be retested for conformance to the flow, volatile content, and the flexural requirements of Tables I and II before being used for fabrication of structural parts. Allowable open time shall be considered as in-storage time.

3.2.8 Product markings. Product markings shall be in accordance with the preparation for delivery section of this specification. See 5.3.

3.3 Properties of cured laminates.

3.3.1 Mechanical and physical properties. The cured laminates shall meet the mechanical and physical property requirements of Table II.

TABLE II

Mechanical and Physical Properties of Cured Laminates

Property	RT	Augmented Autoclave Cure 450K (350F)	Test Para.
Longitudinal Tensile Strength, MN/m^2 (ksi), min.	586 (85)	414 (60)	4.6.7
Longitudinal Tensile Modulus, GN/m^2 (msi), min.	276 (40)	241 (35)	4.6.7
Longitudinal Compressive Strength, MN/m^2 (ksi), min	483 (70)	345 (50)	4.6.8
Longitudinal Compressive Modulus, GN/m^2 (msi), min.	276 (40)	241 (35)	4.6.8
Longitudinal Flexural Strength, MN/m^2 (ksi), min.	690 (100)	483 (70)	4.6.9
Short Beam Shear Strength, MN/m^2 (ksi), min.	41 (6.0)	28 (4.0)	4.6.10
Specific Gravity, minimum	1.670	-	4.6.11
Resin Content, percent by weight, range	27 to 32	-	4.6.12
Fiber Volume, percent, minimum	60 ± 3		4.6.12

Note: Test at temperature after 10^{+1}_{-0} minutes exposure.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or order, the supplier is responsible for the performance of all inspections and test requirements as specified herein. Except as otherwise specified, the supplier may use his own facilities or any commercial laboratory acceptable to Convair. Convair reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.3 Classification of examinations and tests. The examination and tests of the material shall be classified as follows:

a. Qualification verification
b. Acceptance verification
c. Receiving inspection.

4.3.1 Qualification verification. Qualification verification shall consist of all the examinations and tests specified herein.

4.3.2 Acceptance verification. Acceptance verification shall be performed on representative samples of each batch of material, and shall consist of the following:

a. Examination of product
b. Volatile content
c. Resin solids content
d. Flow
e. Tack
f. Longitudinal flexural strength at room temperature and 450K (350F)
g. Short beam shear strength at room temperature and 450K (350F).

4.3.3 Receiving inspection (for Convair use only). Receiving inspection shall consist of an examination of the material and such sampling and verification of test data as deemed necessary.

4.4 Sampling plan. The material, as offered for acceptance by Convair, shall meet the requirements specified herein at an Acceptance Quality Level of 2.5 (normal inspection) when a batch is sampled per MIL-STD-105 at Inspection Level II for Items 4.3.2.a through 4.3.2.e. Items 4.3.2.f and 4.3.2.g shall be inspected by random selection of one sealed unit of material per batch.

4.4.1 Batch. A batch shall consist of all material of the same type manufactured in one continuous, unchanged production run.

4.4.2 Unit of product. A unit of product shall consist of one sealed unit of material, and shall contain no more than 20 sheets.

4.5 Test conditions.

4.5.1 Room temperature. Unless otherwise specified, all tests shall be conducted at a temperature of 298K ±3 (77F ±5) and 50 percent ±10 relative humidity.

4.5.2 Exposure at 450K (350F). Test panels shall be raised to 450K ±6 (350F ±10) within 10 minutes in a test chamber previously heated to temperature, held at temperature for 10^{+1}_{-0} minutes, and tested immediately.

4.6 Test methods.

4.6.1 Examination of product. The material shall be examined to verify that its markings, packaging and all visual physical characteristics conform to the requirements of this specification.

4.6.2 Volatile content.

4.6.2.1 Preparation of specimens. Cut a minimum of six specimens of the material, each approximately 5.1 by 5.1 cm (2 by 2 in.), from an area of sample away from the edges. The specimen may be any shape that will fit into a pre-weighed and fired crucible or a disposable aluminum foil container, but may not be rolled up or more than one-ply lamina. Half of the specimens are to be processed for volatile content determinations, while the others are to be processed for resin solids content determinations.

4.6.2.2 Drying and weighing. Weigh a crucible or an analytical balance to nearest 0.1 mg (W_2). Place the test specimen in the crucible and record the total weight to the nearest 0.1 mg (W_1). Place the crucible containing the specimen in a preheated 436K ±3 (325F ±5) forced air oven for 25^{+1}_{-0} minutes, cool to room temperature, and weigh to the nearest 0.1 mg (W_3).

4.6.2.3 Calculation. The mean value of three volatile content determinations calculated as follows shall be reported.

$$\text{Volatile Content, Weight percent} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

Where: W_1 = Weight of crucible plus specimen before volatile removal

W_2 = Weight of crucible

W_3 = Weight of crucible plus specimen after volatile removal.

4.6.3 Resin solids content.

4.6.3.1 Test procedure. Half of the specimens cut per 4.6.2.1 are used for resin solids content determinations. Weigh a specimen to the nearest 0.1 mg (W_4). Place the specimen in a 300 ml tall-form beaker and add 25 ml of dimethyl formamide (DMF), technical grade. Place the beaker on a hot plate and heat to boiling for 10 to 15 minutes. Remove the beaker from the hot plate and allow to cool to room temperature. Pour off the DMF, being careful not to lose any graphite fibers. Rinse the graphite fiber residue two or three times in acetone. After removing most of the acetone, place the graphite fibers in a tared aluminum cup (throwaway type) and dry for 30 minutes minimum in an air oven at 436K ±3 (325F ±5). Determine the weight of graphite fibers (W_5) to the nearest 0.1 mg and record.

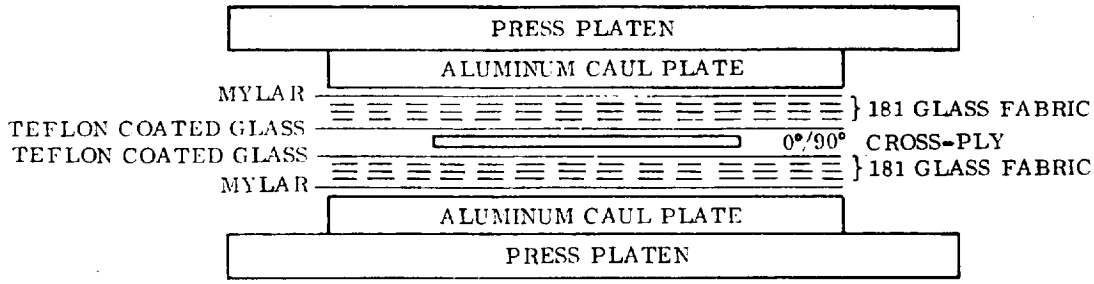
4.6.3.2 Calculation. The mean value of three resin solids content determinations calculated as follows shall be reported.

$$100 - \left[\frac{W_5}{W_4} \times 100\right] = \text{percent by weight of (resin plus volatiles)}$$

$$100 - \left[\frac{W_5}{W_4} \times 100\right] - [\text{percent volatile content (from 4.6.2.3)}] = \text{resin solids content, percent by weight}$$

4.6.4 Resin flow.

4.6.4.1 Preparation of specimens. Cut six pieces of style 181 glass fabric 10.2 by 10.2 cm (4 by 4 in.) for bleeder and two squares of Teflon coated glass fabric of equal size for separator. Weigh the above to the nearest 0.1 mg on an analytical balance. Cut two pieces of the material to be tested 5.1 by 5.1 cm (2 by 2 in.) and weigh, plus the separator and bleeder, to the nearest 0.1 mg. Cross ply the two specimens and assemble as shown below, in a preheated 436K ±3 (325F ±5) press. Cure for 15^{+1}_{-0} minutes at 436K (325F) under 690kN/m^2 (100 psig). Remove the cross-ply test specimens from the separator and bleeder. Weigh the separator plus bleeder to the nearest 0.1 mg.



4.6.4.2 Calculations. Calculate the flow according to the following equation:

$$\text{Percent Flow} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where: W_1 = Weight of glass fabric plus Teflon coated glass fabric

W_2 = Weight of glass fabric, Teflon coated glass fabric, and specimens

W_3 = Weight of glass fabric plus Teflon coated glass fabric after cure.

The mean value for three determinations shall be reported.

4.6.5 Tack.

4.6.5.1 Procedure. A corrosion resistant steel sheet, alloy 302 or equivalent with a commercial 2D finish, any thickness by 10.2 by 20.3 cm (4 by 8 in.), shall be cleaned to a water-break-free condition with chlorine-free scouring poweder and distilled water, then air dried at a temperature below 340K (150F). A specimen cut to 2.54 by 7.6 cm (1 by 3 in.) shall be attached to the plate with light pressure applied by squeegee or roller over the backing film. The backing film shall then be removed. A second strip of the material, cut to the same dimensions, shall be attached in the same manner to the first strip applied and the backing film removed. The plate shall be maintained in a vertical position for 30 minutes minimum while at 291 to 300K (65 to 80F).

4.6.5.2 Calculations. A minimum of three determinations, all of which must pass the test, shall be reported.

4.6.6 Preparation of test panels. Flat laminate test panels, as required, shall be fabricated using the processes and procedures described below. Total number of plies shall be dictated by the test specimen thicknesses specified in the following test methods.

4.6.6.1 Panel layup. Flat laminate test panels of appropriate size, but not smaller than 15.2 by 15.2 cm (6 by 6 in.), shall be prepared by a parallel layup of unidirectionally oriented plies of the material. Dams shall be used around the perimeter of the panels to prevent fiber washout. A release agent shall be used on the caul plate to prevent panel sticking problems. Bleeder shall consist of one ply of Mauchberg paper CW-1850 per every three plies of material, and all the bleeder shall be above the layup. The bleeder shall be separated both top and bottom from the material layup by a ply of perforated Teflon coated glass fabric. A pressure plate of the same size as the basic layup shall be used to minimize surface waviness. This plate shall be separated from the layup by a thin film of Teflon. Figure 1 shows typical layup. Curing shall be conducted by vacuum augmented autoclave cycle.

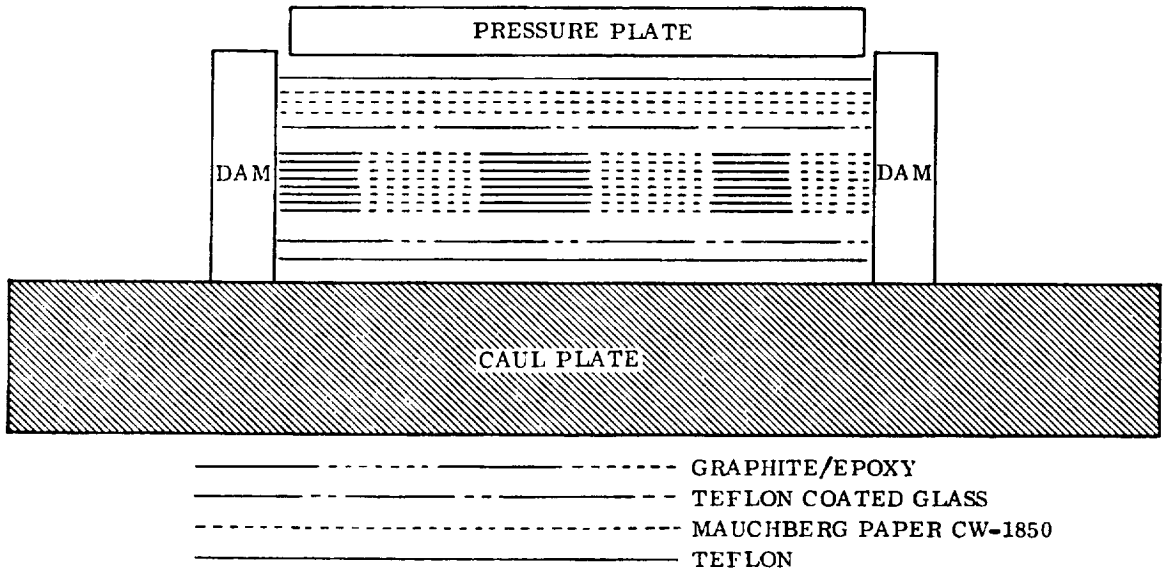


Figure 1. Layup of Unidirectional Graphite/Epoxy Panel

4.6.6.2 Vacuum augmented autoclave cure. The layup as described in 4.6.6.1 shall be vacuum bagged with a film material capable of withstanding long term exposure to 417K (400F). The entire assembly of caul plate, layup, vacuum bag, etc., shall be placed in an autoclave and cured using the following cycle of temperature and pressure.

a. Apply a minimum vacuum of 63.5 cm (25 in.) of mercury on the layup.

b. Raise laminate temperature to 361 to 366K (190 to 200F) at a rate of 1.7 to 2.8K (3 to 5F) per minute and hold temperature for 15^{+1}_{-0} minutes.

c. Apply 690 kN/m^2 ±34 (100 psig ±5) autoclave pressure and continue to hold temperature at 361 to 366K (190 to 200F) for an additional 60 minutes$^{+5}_{-0}$.

d. Raise laminate temperature to 422K (300F) and hold at 422K ±6 (300F ±10) for 1 hour ±5 minutes.

e. Raise laminate temperature to 463K (375F) at at rate of 1.7 to 2.8K (3 to 5F) per minute and hold at 463K ±6 (375F ±10) for 4 hours ±15 minutes.

f. Cool laminate under pressure to below 339K (150K) before removal from autoclave.

g. Post cure laminate at 463K ±8 (375F ±5) for 20 hours ±15 minutes under vacuum bag pressure.

4.6.7 Tensile strength and modulus.

4.6.7.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.7.2 Specimen. The specimen shall be a straight sided coupon with adhesive-bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be paralliel to the longitudinal axis. The tensile specimen configuration is described in Figure 2.

4.6.7.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1011. The specimen shall be loaded to failure at a 0.127 cm ±0.013 (0.050 in. ±0.005) per minute crosshead speed in a testing machine using either strain gage or extensometer readings for strain measurements. Test temperatures shall be at room temperature and at 450K ±3 (350F ±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5).

4.6.7.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both tensile strength and modulus using the formulas given below.

a. Ultimate tensile

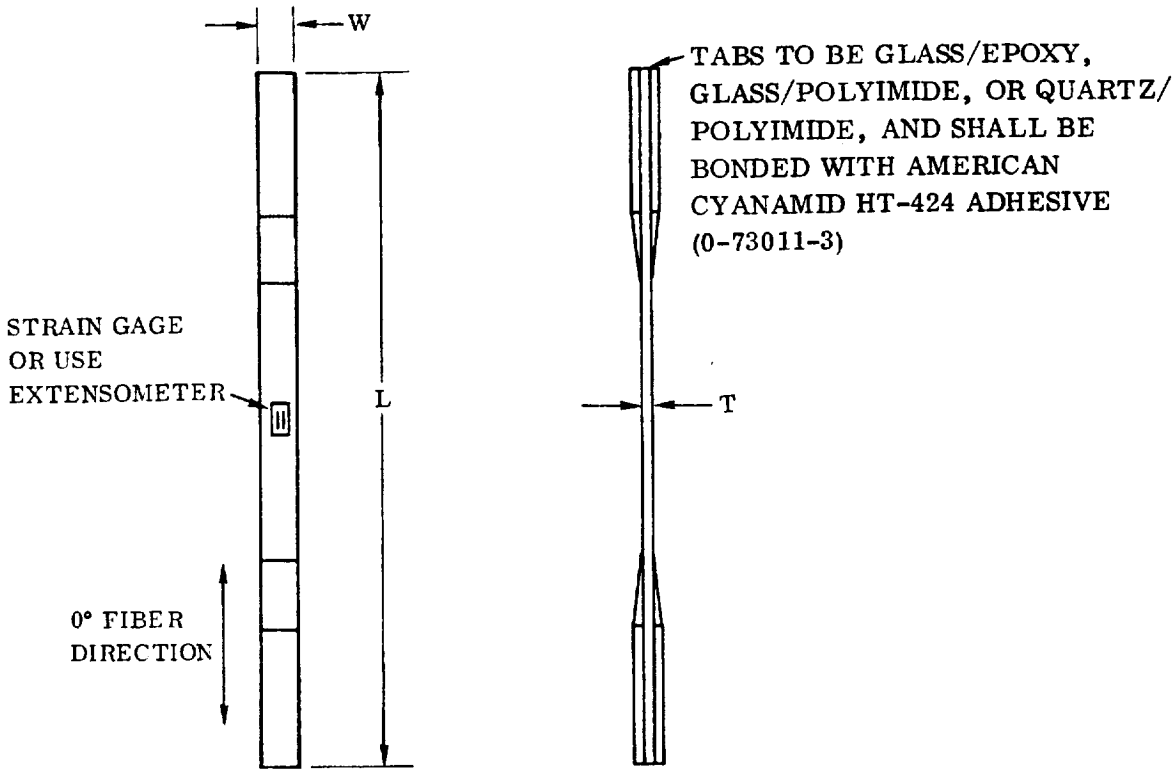
$$F = \frac{P_t}{A}$$

where:

F_t = Ultimate tensile strength

P_t = Maximum tensile load carried by the specimen

A = Specimen cross-sectional area.



Specimen Dimensions:

Length (L)	=	20.32 cm (8.00 in.)
Width (W)	=	1.270 cm (0.500 in.)
Thickness (t)	=	0.076 to 0.152 cm (0.030 to 0.060 in.)
Total Tab Length	=	6.35 cm (2.50 in.)
Tab Chamfer Length	=	1.91 cm (0.75 in.)
Tab Thickness	=	0.076 to 0.152 cm (0.030 to 0.060 in.)

Specimen edges shall be parallel to 0.0076 cm (0.003 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Tabs for room temperature test specimens may be made of crossplied Scotchply 1002 or Scotchply 1007. Tabs for specimens to be tested at 450K (350 F) are to be made of Style 581 Quartz or 181 Glass fabric impregnated with Monsanto's Skybond 703 resin (0-06045-1). These latter tabs may also be used for room temperature test specimens.

Figure 2. Longitudinal Tensile Test Specimen

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straightline portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain. See Figure 3.

$$E = \frac{F_t}{\epsilon}$$

where E = Modulus of elasticity in tension

F_t = Typical tensile stress

ϵ = Corresponding strain

4.6.8 Compressive strength and modulus.

4.6.8.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.8.2 Specimen. The specimen shall be a straight sided sandwich specimen having adhesive bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be parallel to the longitudinal axis. The compression specimen configuration is described in Figure 4.

4.6.8.3 Procedure. The specimen shall be loaded to failure at a 0.102 cm ±0.002 (0.040 in. ±0.005) per minute crosshead speed in a testing machine using strain gage or compressometer readings for strain measurements. Test temperatures shall be at room temperature and at 450K ±6 (350F ±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5).

4.6.8.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both compression strength and modulus using the formulas given below.

a. Ultimate compression strength

$$F_c = \frac{P_c}{A}$$

where F_c = Ultimate compression strength

P_c = Maximum compression load carried by the specimen

A = The sum of the cross-sectional area of both faces.

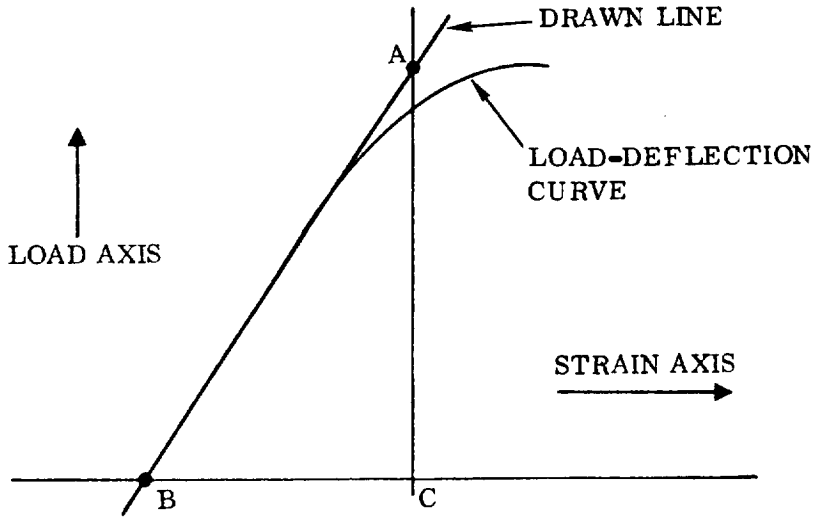


Figure 3. Typical Load-Deflection Curve for Tensile Strength Tests

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straight-line portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain.

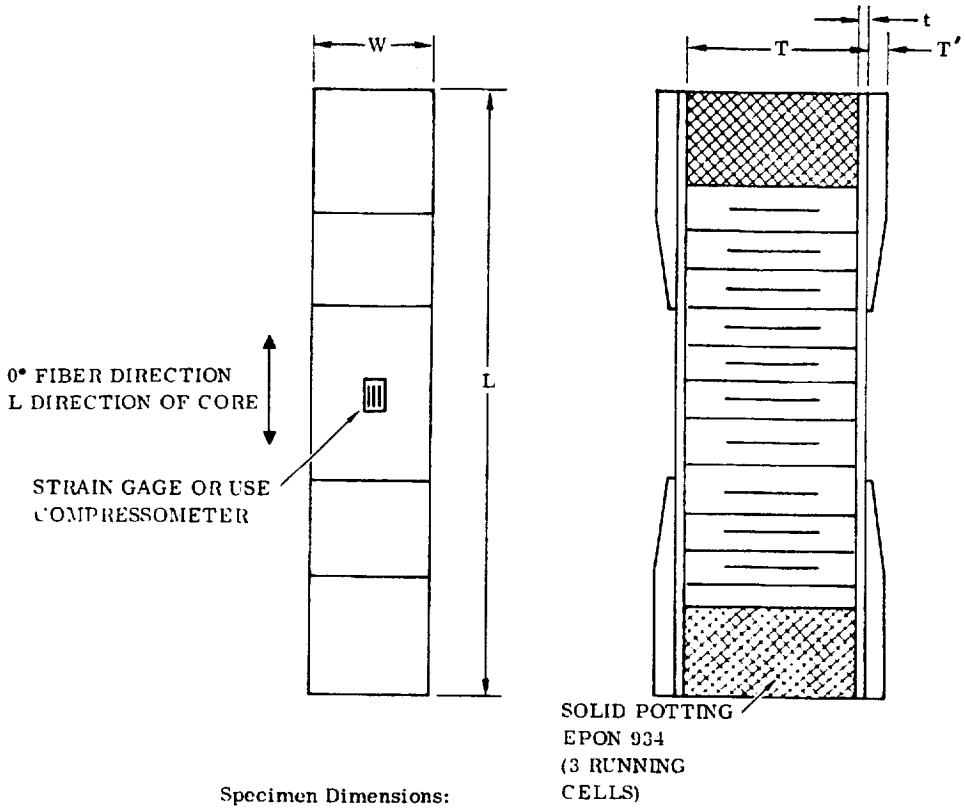
$$E = \frac{F_{typ}}{\epsilon}$$

where: E = Modulus of elasticity in compression

ϵ = Corresponding strain

4.6.9 Longitudinal flexural strength.

4.6.9.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.



Specimen Dimensions:

Length (L) = 8.9 cm (3.5 in.)

Width (W) = 1.270 cm (0.500 in.)

Core Thickness (T) = 2.540 cm (1.000 in.)

Face Thickness (t) = 0.076 to 0.152 cm (0.030 to 0.060 in.)

Tab Thickness (T') = 0.102 to 0.152 cm (0.040 to 0.060 in.)

Total Tab Length = 3.18 cm (1.25 in.)

Tab Chamfer Length = 1.91 cm (0.75 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Core to be 0.318 cm (0.125 in.) cell, 2.8 to 19.2 kg/m^3 (8 to 12 lb/ft^3) aluminum honeycomb.
3. Adhesive to be American Cyanamid HT-424 (0-73011-3).
4. Ends to be potted with Shell Chemical Co. Epon 934 (0-00096-52).
5. Ends to be machined flat and parallel within ±0.0025 cm (±0.001 in.).
6. Specimen sides shall be parallel to 0.0076 cm (0.003 in.) Polish edges after machining.

Figure 4. Longitudinal Compression Test Specimen

4.6.9.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 5.

4.6.9.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1031. The specimen shall be loaded to failure at 0.130 cm ±0.013 (0.050 in. ±0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 450K ±6 (350F ±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 5 with the smooth side up.

4.6.9.4 Calculation. A mean value based on a minimum of three determinations shall be reported for longitudinal flexural strength using the formula below:

$$F_L = \frac{3PS}{2Wt^2}$$

where:

F_L = Ultimate longitudinal flexural strength

P = Maximum load carried by the specimen

S = Span

W = Specimen width

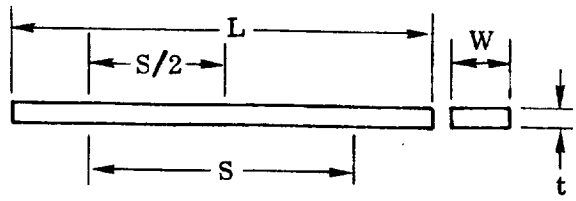
t = Specimen thickness

4.6.10 Short beam shear strength.

4.6.10.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.10.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 6.

4.6.10.3 Procedure. The specimen shall be loaded to failure at a 0.130 cm ± 0.013 (0.050 in. ± 0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 450K ±6 (350F ±10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 6 with the smooth side up.



Specimen Dimensions

Length (L) = 7.6 to 10.2 cm (3.0 to 4.0 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 32 to 1

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 5. Longitudinal Flexure Test Specimen

Specimen Dimensions:

Length (L) = 1.52 cm (0.060 in.)

Width (W) = 0.635 cm (0.250 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 4 to 1

Loading head and reaction supports are 0.318 cm (0.125 in.) diameter steel rod

Overhang must be the same over each end

Figure 6. Short Beam Shear Specimen

4.6.10.4 Calculation. A mean value based on a minimum of three determinations shall be reported for short beam shear strength, using the formula below:

$$\tau = \frac{3P}{4Wt}$$

where: τ = Ultimate short beam shear strength

P = Maximum load carried by specimen

W = Specimen width

t = Specimen thickness

4.6.11 Specific gravity.

4.6.11.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.11.2 Test method. Specimen configuration and test procedure shall be in accordance with Federal Test Method Std. No. 406, Method 5011.

4.6.11.3 Calculation. Specific gravity calculations shall be per Federal Test Method Std. No. 406, Method 5011. A mean value based on a minimum of three determinations shall be reported.

4.6.12 Cured resin content and fiber volume.

4.6.12.1 Laminate preparation. Laminate shall be in accordance with 4.6.6.

4.6.12.2 Specimen. Test specimens shall be approximately 1.27 by 1.27 cm (0.5 by 0.5 in.) by laminate thickness. (Take specimens from a test panel used in a previous test conducted at room temperature.)

4.6.12.3 Procedure. The cured resin content and fiber volume shall be determined by acid/peroxide digestion as follows:

a. Weigh the test specimen to the nearest 0.1 mg (W_1), place in a 300 ml tall-form beaker, and add 20 ml of concentrated sulfuric acid. Place the beaker on a hot plate and heat the acid until vigorous fuming occurs.

b. When the composite is visibly disintegrated and resin particles and fibers are dispersed throughout the sulfuric solution, carefully add the hydrogen peroxide(50 percent strength) dropwise down the side of the beaker. Rubber gloves and and fume hood with appropriate safety glass shield shall be used throughout the addition and precautions shall be taken as recommended by the applicable safety regulations and procedures for handling hydrogen peroxide.

c. The reaction is considered complete when the hot sulfuric acid solution below the fibers becomes clear and colorless. At this point add two more ml of hydrogen peroxide to the solution, and heat solution to fumes for another 10 minutes to ensure complete decomposition of the polymer. Remove the beaker from the hot plate and allow to cool to 294 to 300K (70 to 80F), and then place in an ice bath.

d. Collect the fibers by vacuum filtration through a medium-porosity, sintered-glass crucible that has been weighed to nearest mg (W_2). After the sulfuric acid has been filtered off, wash the fibers in the crucible thoroughly with 600 ml of distilled water, added a few milliliters at at time. Verify removal of sulfuric acid traces by checking pH of the filtrate drops.

e. Remove the crucible from the filtering system and place in an open beaker. Dry in an oven at 422K (300F) for 45 minutes. After drying, cool the crucible in a desiccator and weight (W_3).

4.6.12.4 Resin fiber content calculation. Calculate the resin and fiber content according to the following equation:

$$\text{Resin content, percent by weight } (W_4) = \frac{W_1 - (W_3 - W_2)}{W_1} \times 100$$

$$\text{Fiber content, percent by weight } (W_5) = \frac{W_3 - W_2}{W_1} \times 100$$

4.6.12.5 Fiber volume calculation. Calculate the fiber volume using the data generated from the resin and fiber content determinations and the following formula:

$$\text{Fiber volume, percent} = \frac{W_6}{W_6 + (W_7 - W_6)\ \frac{\rho_1}{\rho_2}} \times 100$$

where:

ρ_1 = Density of fiber

ρ_2 = Density of epoxy resin

W_7 = Weight of laminate = 1.00

W_6 = Weight fraction of dry graphite fiber = $\frac{W_5}{100}$

A mean value based on three determinations shall be reported.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. The material shall be packaged with a non-adherent separator applied to both sheet faces. The material within one package shall be of the same length and width to preclude damage to the material during shipment. Packaged material shall be sealed within a moisture-proof plastic bag meeting the requirements of Military Specification MIL-B-131.

5.2 Packing. The packaged material shall be packed in a shipping container of a type which will ensure acceptance by common carrier at lowest rates and will ensure protection of the material during handling, transit, and storage.

5.3 Marking of interior package. Each interior package shall be legibly marked with a label or tag which includes the following minimum information.

a. 0-06051-1

b. Manufacturer's material designation

c. Manufacturer's name and address

d. Batch number and sheet number

e. Date of manufacture

f. Weight

g. Recommended storage conditions and temperature range for maximum shelf life.

h. Estimated maximum shelf life based on recommended storage conditions and temperature range.

i. Hazardous warnings as applicable.

5.4 Marking of exterior shipping container. Each exterior shipping container shall be legibly and permanently marked with the following information.

a. 0-06051-1

b. Purchase order number

c. Manufacturer's material and designation

d. Manufacturer's name and address

e. Batch number

f. Quantity contained (sheet size and number of sheets).

g. Date of manufacture

h. Date of shipping

i. Manufacturer's recommended storage conditions and temperature range.

j. Precautionary and handling markings

In addition, the shipping container shall be identified with a strip of one-inch wide green plastic tape (Scotchlite 3277 or equvalent) completely around the container from top to bottom and approximately 2.54 cm (1 in.) from the side.

6. NOTES

6.1 Intended use. The material covered by this specification is intended for use in the manufacture of missile and spacecraft structural components subject to temperatures from 20K (-423F) to 450K (350F). Use is not restricted to these applications.

6.2 Ordering information. The following information should be included on the purchase order together with the conditions of 6.2.1 and 6.2.2.

a. Number, title, and date of specification

b. Material name and quantity

6.2.1 Rejection and retest. In the event of failure of a sample to meet any of the requirements of this specification, a second sample of uncured material taken adjacent to the first, or a second laminate panel prepared in accordance with 4.6.6 may be submitted for retest. If the retest sample fails to meet the requirements of the specification, the batch represented by the sample shall be rejected.

6.2.2 Reports. Unless otherwise specified, the supplier shall furnish with each shipment three copies of the reports showing the results of tests made on each batch in the shipment to determine conformance of the material to the specification requirements. The report shall include volatile content, resin solids, resin flow, tack and mechanical properties, as applicable. The report shall also include the purchase order number, the material specification number, supplier designation, quantity, batch number, and date of manufacture. The report shall also include fiber properties as reported by the fiber manufacturer, and the batch numbers of fiber used in making each of the sheets of material.

6.3 Approved sources. The approved sources for the material described in this specification are as follows:

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
0-06051-1	hy-E-1511(GY-70/X-904)	Fiberite Corporation 515-West Third Street Winona, Minn. 55987

APPENDIX G

GRAPHITE/POLYIMIDE PREPREG SPECIFICATION

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification establishes the requirement for graphite flat sheet composed of polyimide resin impregnated tow.

1.2 Classification. The material covered by this specification shall be of two types identified as 0-06053-1 and 0-06053-2.

Class I - High strength graphite reinforced polyimide
Class II - High modulus graphite reinforced polyimide

2. APPLICABLE DOCUMENTS

2.1 Unless otherwise specified below, the following documents of the issue in effect on date of Convair's request for quotation form a part of this specification to the extent specified.

SPECIFICATIONS

Military

MIL-B-131	Barrier Material: Water Vaporproof, Flexible

Convair

0-00096	Epoxy Compound, 2 Parts Adhesive Bonding, Potting, Sealing and Coating
0-06045	Fabric, Quartz, Polyimide Resin Impregnated (B Stage)
0-06052	Resin, Polyimide, Impregnating
0-73011	Dry Film Structural Adhesive (Epoxy-Phenolic) for Use in Sandwich Structure

STANDARDS

Federal

FED-STD-406	Plastics, Methods of Testing

Preceding page blank

Military

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

PUBLICATIONS

General industry Safety Orders, State of California Department of Industrial Relations, Article 85, "Labeling of Injurious Substances".

Manufacturing Chemists Association, Labels and Precautionary Information Committee, "Guide to Precautionary Labeling of Hazardous Chemicals". (Manual L1.)

3. REQUIREMENTS

3.1 Material. The material shall consist of high strength, high modulus graphite fibers collimated into multifilament tows and impregnated with a polyimide resin.

3.1.1 Graphite. Graphite shall consist of continuous collimated multifilament tow having a minimum modulus of 221 GN/m^2 (32 msi) and a minimum ultimate tensile strength of 2,413 MN/m^2 (350 ksi) for Class I and a minimum modulus of 358 GN/m^2 (52 msi) and a minimum ultimate tensile strength of 1,551 MN/m^2 (225 ksi) for Class II. The fibers shall be compatible with the resin.

3.1.2 Fiber finish. Surface treatment of the fiber such as acid etching is acceptable as long as it enhances the fiber-resin compatibility over the full range of service temperatures. Use of organic or inorganic finishes is not permitted.

3.1.3 Resin. The resin shall be a high temperature resistant, thermosetting polyimide meeting the requirements of Specification 0-06052 and suitable for use in the manufacture of structural laminates.

3.1.4 Handling characteristics. The material, as supplied, shall be suitable for both vacuum bag and vacuum augmented autoclave laminating of contoured parts.

3.1.5 Construction. Unless otherwise specified on the purchase order, the material shall be in the form of sheets with a nominal width of 30.5 cm (12 in.). When laminated at 690 kN/m^2 (100 psig) vacuum augmented autoclave pressure, the material shall have sufficient tows to produce a nominal 0.013 cm (0.005 in.) per ply thickness with 60 percent fiber volume. Sheets shall be produced in such a manner that a minimal length of 91.5 cm (36 in.) can be obtained.

3.2 Properties of uncured material.

3.2.1 Physical properties. The material shall meet the physical property requirements of Table I.

TABLE I

Physical Properties of Uncured Material - Both Classes

	Requirements	Test Paragraph
Volatile Content, weight percent	10 to 20	4.6.2
Resin Solids Content, weight percent	38 to 48	4.6.3
Resin Flow, weight percent	15 to 25	4.6.4
Tack	shall adhere for 30 minutes minimum	4.6.5
Gel Time, minutes	1.0 minimum	4.6.6

3.2.2 Shelf life. The shelf life of the prepreg shall be such that the requirements of 3.2.1 are met after storage (computed from date of receipt of shipment) as follows:

a. 90 days at 255K (0F) or
b. 60 days at 277K (40F) or
c. 3 days at 294K (70F)

3.2.3 Working life. The refrigerated material shall possess sufficient room temperature working life that the requirements of 3.2.1 are met after exposure to 291 to 300K (65 to 80F) temperature and relative humidity not in excess of 70 percent for a continuous period of 3 days following removal of material from refrigeration.

3.2.4 Workmanship. The material shall be free of contaminants detrimental to the finished product.

3.2.5 Uniformity. The tows shall be completely wetted. Visual defects shall be maintained at a consistent minimum level.

3.2.5.1 Resin. Resin-rich or resin-starved areas on the material surface shall not exceed 1.0 percent of the total surface area.

3.2.5.2 Tow alignment. The number of misaligned or overlaid tows occurring in a nominal 7740 sq. cm (1,200 sq. in.) sheet shall not exceed 4 tows per sheet. Misalignment shall not exceed 2 degrees.

3.2.6 Toxic or hazardous formulations. If injurious formulations cannot be avoided, all containers of injurious substances shall be labeled as required by Article 85 of California General Industry Safety Orders or Manual L1 of the Manufacturing Chemist Association.

3.2.7 Storage of material. Immediately upon receipt by Convair, the material, in its original sealed bag, shall be placed in storage at a maintained temperature of 255K (0F) or below. The sheets of material shall be stored horizontally. Sheets of material shall never be stored in a vertical position. When material is removed from storage for production use or test, the sealed material shall be brought to within 5K (10F) of room temperature prior to opening the sealed bag. The amount required for use during the remainder of that work shift shall be cut from the sheet and the unused portion shall be replaced in its original bag, the bag resealed by heat application, and the material returned to storage without delay. Bags shall not remain open longer than 1 hour at any one time, and total elapsed open time shall not be more than 24 hours. Total elapsed time out of storage for any one sealed unit shall never exceed 3 days. At no time shall the material which is to be returned to storage be subjected to environmental temperatures greater than 302K (85F).

3.2.8 Storage history. A log of the storage history shall be kept on each unit of material. Material that has exceeded the 4-day out-of-storage (3 days sealed plus 1 day open) time or exceeded the 90-day in-storage time shall be retested for conformance to the flow, volatile content, and the flexural requirements of Tables I and II before being used for fabrication of structural parts. Allowable open time shall be considered as in-storage time.

3.2.9 Product markings. Product markings shall be in accordance with the preparation for delivery section of this specification. See 5.3.

3.3 Properties of cured laminates.

3.3.1 Mechanical and physical properties. The cured laminates shall meet the mechanical and physical property requirements of Table II.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or order, the supplier is responsible for the performance of all inspections and test requirements as specified herein. Except as otherwise specified, the supplier may use his own facilities or any commercial laboratory acceptable to Convair. Convair reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.3 Classification of examinations and tests. The examination and tests of the material shall be classified as follows:

a. Qualification verification
b. Acceptance verification
c. Receiving inspection.

4.3.1 Qualification verification. Qualification verification shall consist of all the examinations and tests specified herein.

4.3.2 Acceptance verification. Acceptance verification shall be performed on representative samples of each unit of material, and shall consist of the following:

a. Examination of product
b. Resin solids content
c. Volatile content
d. Flow

TABLE II

Mechanical and Physical Properties of Cured Laminates - Class I

Property	Vacuum Augmented Autoclave Cure		Vacuum Bag Cure		Test Paragraph
	RT	589K (600F)	RT	589K (600F)	
Longitudinal Tensile Strength, MN/m^2 (ksi), minimum	1034 (150)	827 (120)	896 (130	621 (90)	4.6.8
Longitudinal Tensile Modulus, GN/m^2 (msi) minimum	131 (19)	131 (19)	131 (19)	131 (19)	4.6.8
Longitudinal Compressive Strength, MN/m^2 (ksi), minimum	690 (100)	- -	690 (100)	- -	4.6.9
Longitudinal Compressive Modulus, GN/m^2 (msi), minimum	131 (19)	131 (19)	131 (19)	131 (19)	4.6.9
Longitudinal Flexural Strength, MN/m^2 (ksi), minimum	1276 (185)	827 (120)	1103 (160)	690 (100)	4.6.10
Transverse Flexural Strength, MN/m^2 (ksi), minimum	41 (6.0)	- -	41 (6.0)	- -	4.6.11
Short Beam Shear Strength, MN/m^2 (ksi), minimum	59 (8.5)	34 (5.0)	48 (7.0)	28 (4.0)	4.6.12
Specific Gravity, minimum	1.48	- -	1.42	- -	4.6.13
Resin Content, % by weight, minimum	28	- -	32	- -	4.6.14
Fiber Volume, percent	62 ± 3	- -	62 ± 3	- -	4.6.14
Void Content, percent, maximum	10	- -	12	- -	4.6.15

Note: (1) Test at temperature after 10 $^{+1}_{-0}$ minutes exposure.

TABLE II Continued

Mechanical and Physical Properties of Cured Laminates - Class II

Property	Vacuum Augmented Autoclave Cure		Vacuum Bag Cure		Test Paragraph
	RT	589K (600F)	RT	589K (600F)	
Longitudinal Tensile Strength, MN/m^2 (ksi), minimum	690 (100)	627 (90)	556 (80)	486 (70)	4.6.8
Longitudinal Tensile Modulus, GN/m^2 (msi) minimum	180 (26)	180 (26)	180 (26)	180 (26)	4.6.8
Longitudinal Compressive Strength, MN/m^2 (ksi), minimum	346 (50)	- -	346 (50)	- -	4.6.9
Longitudinal Compressive Modulus, GN/m^2 (msi), minimum	180 (26)	180 (26)	180 (26)	180 (26)	4.6.9
Longitudinal Flexural Strength, MN/m^2 (ksi), minimum	827 (120)	758 (110)	690 (100)	627 (90)	4.6.10
Transverse Flexural Strength, MN/m^2 (ksi), minimum	21 (3.0)	- -	21 (3.0)	- -	4.6.11
Short Beam Shear Strength, MN/m^2 (ksi), minimum	35 (5.0)	24 (3.5)	31 (4.5)	21 (3.0)	4.6.12
Specific Gravity, minimum	1.60	- -	1.58	- -	4.6.13
Resin Content, % by weight, minimum	28	- -	30	- -	4.6.14
Fiber Volume, percent	65 ± 3	- -	65 ± 3	- -	4.6.14
Void Content, percent maximum	10	- -	12	- -	4.6.15

Note: (1) Test at temperature after 10^{+1}_{-0} minutes exposure.

e. Tack
f. Longitudinal flexural strength at room temperature
g. Transverse flexural strength at room temperature
h. Short beam shear strength at room temperature

4.3.3 Receiving inspection (for Convair use only). Receiving inspection shall consist of an examination of the material and such sampling and verification of test data as deemed necessary.

4.4 Sampling plan. The material, as offered for acceptance by Convair, shall meet the requirements specified herein at an Acceptance Quality Level of 2.5 (normal inspection) when a batch is sampled per MIL-STD-105 at Inspection Level II for Items 4.3.2a through 4.3.2e. Items 4.3.2f through 4.3.2h shall be inspected by random selection of one sealed unit of material per batch.

4.4.1 Batch. A batch shall consist of all material of the same type manufactured in one continuous, unchanged production run.

4.4.2 Unit of product. A unit of product shall consist of one sealed unit of material and shall contain no more than 20 sheets.

4.5 Test conditions.

4.5.1 Room temperature. Unless otherwise specified, all tests shall be conducted at a temperature of 298K ± 3 (77F ± 5) and 50 percent ± 10 relative humidity.

4.5.2 Exposure at 589K (600F). Test panels shall be raised to 589K ± 6 (600F ± 10) within 15 minutes in a test chamber previously heated to temperature, held at temperature for $10\ ^{+1}_{-0}$ minutes, and tested immediately.

4.5.3 Work and test area. The relative humidity of the layup and test area shall not exceed 60 percent.

4.6 Test methods.

4.6.1 Examination of product. The material shall be examined to verify that its markings, packaging, and all visual physical characteristics conform to the requirements of this specification.

4.6.2 Volatile content.

4.6.2.1 Preparation of specimens. Cut a minimum of six specimens of the material, each approximately 5.1 by 5.1 cm (2.54 by 2.54 in.), from an area of sample away from the edges. The specimen may be any shape that will fit into a pre-weighed and fired crucible or disposable aluminum dish, but may not be rolled up, or more than one ply lamina. Half of the specimens are to be processed for volatile content determinations, while the others are to be processed for resin solids content determinations.

4.6.2.2 Drying and weighing. Weigh each crucible on an analytical balance to nearest 0.1 mg (W_2). Place the test specimen in the crucible and record the total weight to the nearest 0.1 mg (W_1). Place the crucible containing the specimen in a preheated 450K (350F) air circulating oven for 20 $^{+1}_{-0}$ minutes, cool to room temperature, and weigh to the nearest 0.1 mg (W_3).

4.6.2.3 Calculation. The mean value of the three volatile content determinations calculated as follows shall be reported.

$$\text{Volatile Content, weight percent} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

where W_1 = Weight of crucible plus specimen before volatile removal

W_2 = Weight of crucible

W_3 = Weight of crucible plus specimen after volatile removal.

4.6.3 Resin solids content.

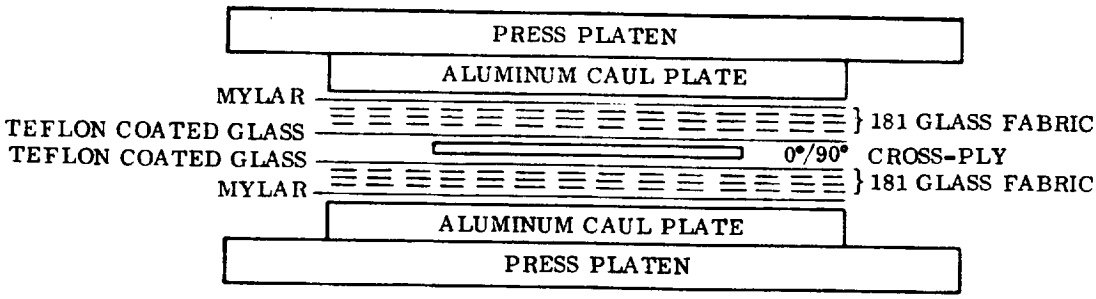
4.6.3.1 Test procedure. Half of the specimens cut per 4.6.2.1 are used for resin solids content determinations. Weigh a specimen to the nearest 0.1 mg (W_4), place in a 300 ml tall-form beaker, and add 25 ml of dimethyl formamide (DMF) technical grade. Place the beaker on a hot plate and allow to cool to room temperature. Pour off the DMF, being careful not to lose any graphite fibers. Rinse the graphite fiber residue two or three times in acetone. After removing most of the acetone, place the graphite fibers in a tared aluminum cup (throwaway type), and dry for 30 minutes minimum in an air oven at 450K (350F). Determine the weight of graphite fibers (W_5) to the nearest 0.1 mg and record.

4.6.3.2 Calculation. The mean value of three solids content determinations calculated as follows shall be reported.

$$100 - \left[\frac{W_5}{W_4} \times 100\right] \text{ percent volatile content from 4.6.2.3} = \text{resin solids content, percent by weight}$$

4.6.4 Flow.

4.6.4.1 Preparation of specimens. Cut six pieces of style 181 glass fabric 10.2 by 10.2 cm (4 by 4 in.) square for bleeder, and two squares of Teflon coated glass fabric of the same size for separator. Weigh the above to the nearest 0.1 mg on an analytical balance. Cut two pieces of the material to be tested 5.1 by 5.1 cm (2 by 2 in.) and weigh the material plus the separator and bleeder to the nearest 0.1 mg. Cross ply the two specimens and assemble as shown below and place in a preheated 450K ± 3 (350F ± 5) press. Cure for 15 $^{+1}_{-0}$ minutes at 450K (350F) under 690 kN/m^2 (100 psig). Remove the cross-ply test specimens from the separator and bleeder. Weigh the separator plus bleeder to the nearest 0.1 mg.



4.6.4.2 Calculations. Calculate the flow according to the following equation:

$$\text{Percent Flow} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where W_1 = Weight of glass fabric plus Teflon coated glass fabric.

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

W_3 = Weight of glass fabric plus Teflon coated glass fabric after cure.

The mean value for three determinations shall be reported.

4.6.5 Tack.

4.6.5.1 Procedure. A corrosion resistant steel sheet, alloy 302 or equivalent with a commercial 2D finish, any thickness by 10.2 by 20.3 cm (4 by 8 in.) shall be cleaned to a water-break-free condition with chlorine-free scouring powder and distilled water, then air dried at a temperature below 340K (150F). A specimen cut to 2.5 by 7.6 cm (1 by 3 in.) shall be attached to the plate with light pressure applied by squeegee or roller over the backing film. The backing film shall then be removed. A second strip of the material, cut to the same dimensions, shall be attached in the same manner to the first strip applied and the backing film removed. The plate shall be maintained in a vertical position for 30 minutes minimum while at 291 to 300K (65 to 80F).

4.6.5.2 Calculations. A minimum of three determinations, all of which must pass the test, shall be reported.

4.6.6 Gel time.

4.6.6.1 Test procedure. Preheat and stabilize a Fisher-Johns Melting Point Apparatus at 450K ± 0.5 (350F ± 1). Place a small piece of newly thawed material, approximately 0.64 by 0.64 cm (0.25 by 0.25 in.), between two glass slides and place the glass slides containing the material on the melting point block. Start the timer. Periodically apply slight pressure to the top glass slide with a small, blunt-end wooden stick to determine visually through the magnifying eye piece if gellation has occurred. The time at which no fiber or resin movement is detected is the gel time.

4.6.6.2 Report. The mean value for three determinations shall be reported to the nearest 0.1 minute.

4.6.7 Preparation of test panels. Flat laminate test panels, as required, shall be fabricated using the processes and procedures described below. Total number of plies shall be dictated by the test specimen thicknesses specified in the following test methods.

4.6.7.1 Panel layup. Flat laminate test panels of appropriate size, but no smaller than 15.2 by 15.2 cm (6 by 6 in.), shall be prepared by a parallel layup of unidirectionally oriented plies of the material. Dams shall be used around the perimeter of the panels to prevent fiber "wash-out". A release agent shall be used on the caul plate to prevent panel sticking problems. Bleeder shall consist of one ply of 181 glass per every two plies of material, and the bleeder shall be divided evenly beneath and above the layup. The bleeder shall be separated both top and bottom from the material layup by a ply of perforated Teflon coated glass fabric. A pressure plate of the same size as the basic layup shall be used to minimize surface waviness. This plate shall be separated from the layup by a thin film of Teflon or Mylar. Figure 1 shows typical layup. Curing shall be conducted, as applicable, by either vacuum bag cycle or vacuum augmented autoclave cycle.

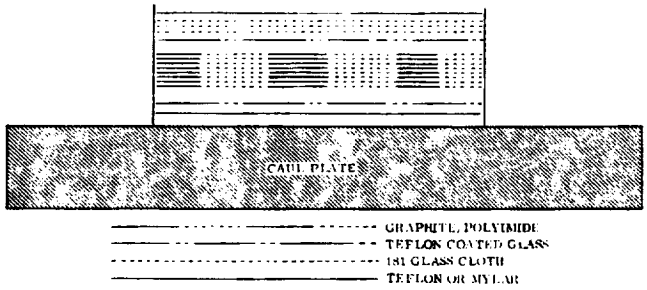


Figure 1. Layup of Unidirectional Graphite/Polyimide Panel

4.6.7.2 Vacuum bag cure. The layup as described in 4.6.7.1 shall be vacuum bagged with a film material capable of withstanding long term exposure to 477K (400F). The entire assembly of caul plate, layup, vacuum bag, etc., shall be placed in an air circulating oven and cured using the following cure cycle and a minimum vacuum of 65 cm (26 in.) of mercury on the layup at all times during cure.

4.6.7.3 Vacuum augmented autoclave cure. The layup as described in 4.6.7.1 shall be vacuum bagged with a film material capable of withstanding long term exposure to 477K (400F). The entire assembly of caul plate, layup, vacuum bag, etc., shall be placed in an autoclave and cured using the following cycle of temperature and pressure.

Cure Cycle.

a. Apply full vacuum at room temperature and keep throughout entire cure cycle. (65 cm, (26 in.) Hg minimum)

b. Raise temperature to 352K ± 2 (175F ± 4) at a rate of 2K ± 0.6 (4F ± 1) per minute and hold for 30 minutes.

c. Raise temperature to 400K ± 3 (260F ± 5) at a rate of 2C ± 0.6 (4F ± 1) per minute and hold for 20 minutes.

d. Apply 690 kN/m^2 (100 psi) autoclave pressure after 20 minute hold at 400K ± 3 (260F ± 5).

e. Raise temperature to 450K ± 3 (350F ± 5) at a rate of 2C ± 0.6 (4F ± 1) per minute and hold for 2 hours.

f. Cool under pressure to 352K (175F).

4.6.7.4 Post cure cycle. Tolerance on all temperatures is ± 5K (± 10F).

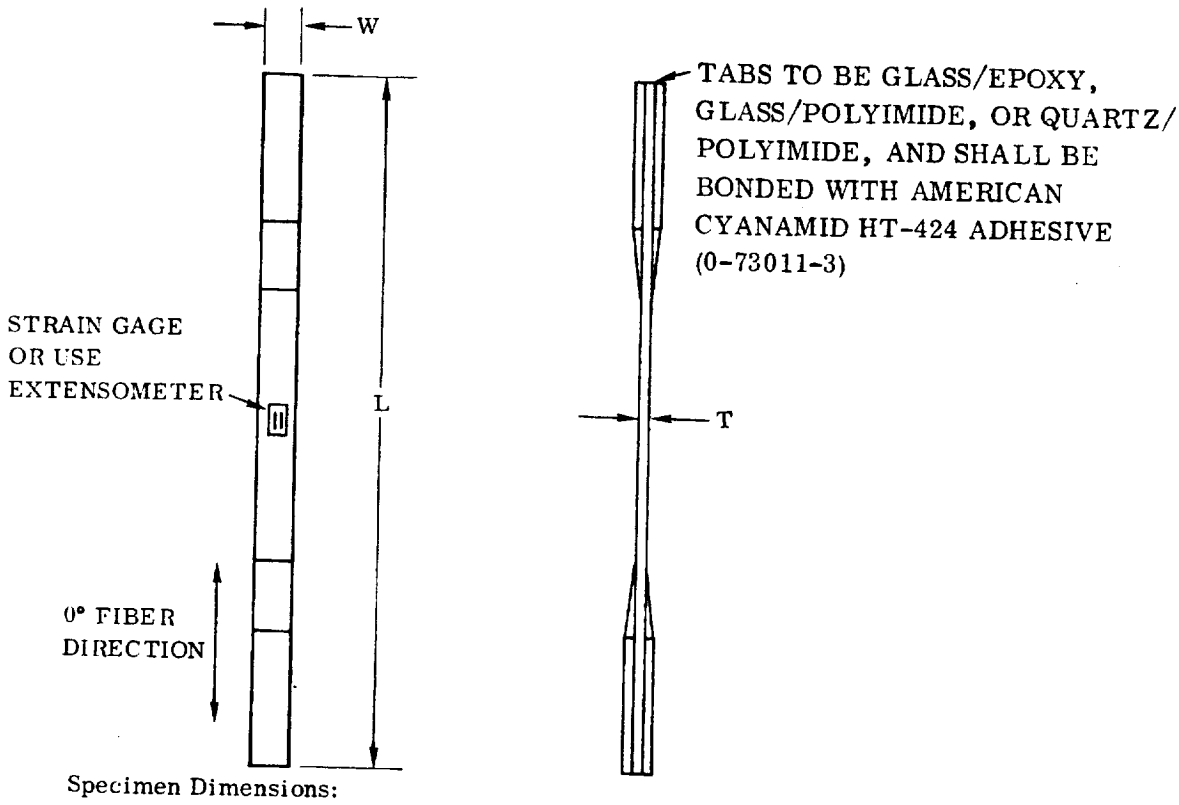
1 hr at 450K (350F)	2 hr at 561K (550F)
1 hr at 477K (400F)	2 hr at 589K (600F)
2 hr at 505K (450F)	2 hr at 616K (650F)
1 hr at 533K (500F)	8 hr at 644K (700F)

4.6.8 Tensile strength and modulus.

4.6.8.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.8.2 Specimen. The specimen shall be a straight sided coupon with adhesive-bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be parallel to the longitudinal axis. The tensile specimen configuration is described in Figure 2.

4.6.8.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1011. The specimen shall be loaded to failure at a 0.127 cm ± 0.013 (0.050 in. ± 0.005) per minute crosshead speed in a testing machine using either strain gage or extensometer readings for strain measurements. Test temperatures shall be at room temperature and at 589K ± 6 (600F ± 10) after a 10 $^{+1}_{-0}$ minute exposure at temperature (see 4.5).



Specimen Dimensions:

Length (L)	=	20.32 cm (8.00 in.)
Width (W)	=	1.270 cm (0.500 in.)
Thickness (t)	=	0.076 to 0.152 cm (0.030 to 0.060 in.)
Total Tab Length	=	6.35 cm (2.50 in.)
Tab Chamfer Length	=	1.91 cm (0.75 in.)
Tab Thickness	=	0.076 to 0.152 cm (0.030 to 0.060 in.)

Specimen edges shall be parallel to 0.0076 cm (0.003 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Tabs for room temperature test specimens may be made of crossplied Scotchply 1002 or Scotchply 1007. Tabs for specimens to be tested at 450K (350 F) are to be made of Style 581 Quartz or 181 Glass fabric impregnated with Monsanto's Skybond 703 resin (0-06045-1). These latter tabs may also be used for room temperature test specimens.

Figure 2. Longitudinal Tensile Test Specimen

4.6.8.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both tensile strength and modulus using the formulas given below.

a. Ultimate tensile strength

$$F_t = \frac{P_t}{A}$$

where:

F_t = Ultimate tensile strength

P_t = Maximum tensile load carried by the specimen

A = Specimen cross-sectional area

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straight-line portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain. See Figure 3.

$$E = \frac{F_t}{\epsilon}$$

where:

E = Modulus of elasticity in tension

F_t = Typical tensile stress

ϵ = Corresponding strain

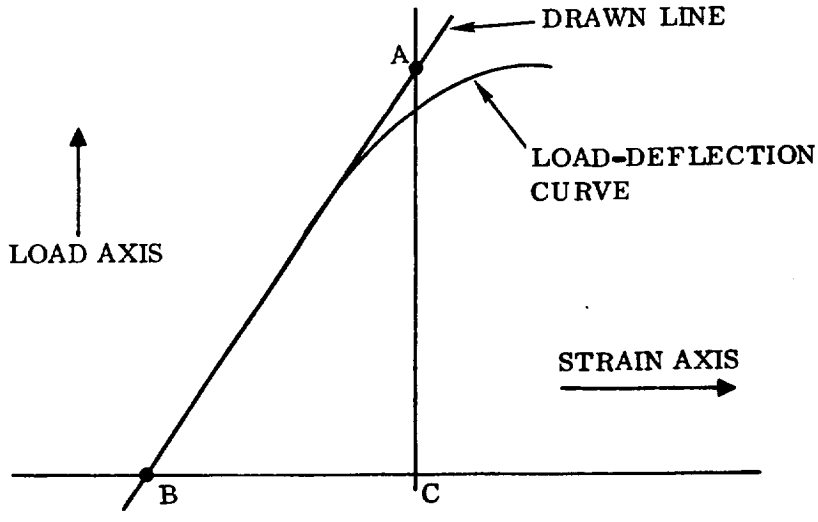


Figure 3. Typical Load-Deflection Curve for Tensile Strength Tests

4.6.9 Compression strength and modulus.

4.6.9.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.9.2 Specimen. The specimen shall be a straight sided sandwich specimen having adhesive bonded tabs. Specimen edges shall be ground to the required length and width dimensions with abrasive finer than 400 grit. The fibers shall be parallel to the longitudinal axis. The compression specimen configuration is described in Figure 4.

4.6.9.3 Procedure. The specimen shall be loaded to failure at a 0.102 cm ±.013 (0.040 in. ±0.005) per minute crosshead speed in a testing machine using strain gage or compressometer readings for strain measurements. Test temperatures shall be at room temperature and at 589K ± 6 (600 F ± 10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5).

4.6.9.4 Calculation. A mean value based on a minimum of three determinations shall be reported for both compression strength and modulus using the formulas given below.

a. Ultimate compression strength

$$F_c = \frac{P_c}{A}$$

where:

F_c = Ultimate compression strength

P_c = Maximum compression load carried by the specimen

A = The sum of the cross-sectional area of both faces.

b. Modulus of elasticity. Obtain the modulus of elasticity by extending the initial straight-line portion of the load-deflection curve and graphically determining the ratio of stress to corresponding strain.

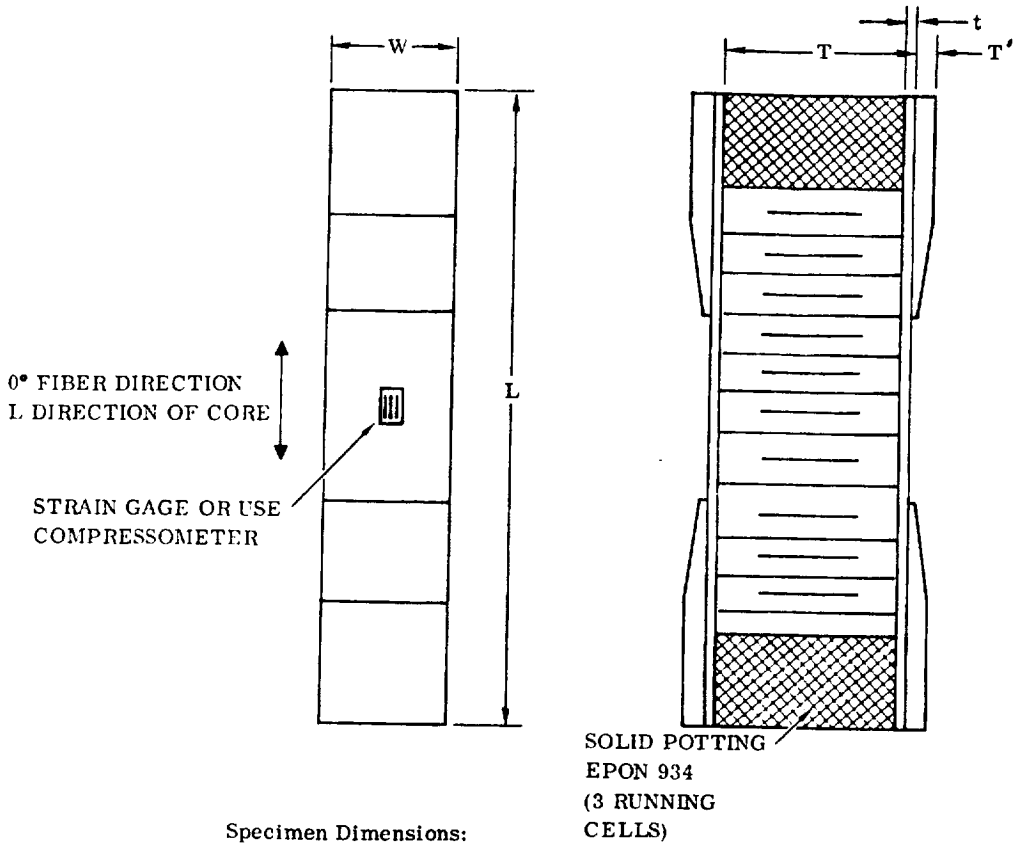
$$E = \frac{F_{typ}}{\epsilon}$$

where:

E = Modulus of elasticity in compression

F_{typ} = Typical compression stress

ϵ = Corresponding strain



Specimen Dimensions:

Length (L) = 8.9 cm (3.5 in.)

Width (W) = 1.270 cm (0.500 in.)

Core Thickness (T) = 2.540 cm (1.000 in.)

Face Thickness (t) = 0.076 to 0.152 cm (0.030 to 0.060 in.)

Tab Thickness (T') = 0.102 to 0.152 cm (0.040 to 0.060 in.)

Total Tab Length = 3.18 cm (1.25 in.)

Tab Chamfer Length = 1.91 cm (0.75 in.)

NOTES:
1. Ply thickness based on 0.015 cm (0.006 in.) per ply.
2. Core to be 0.318 cm (0.125 in.) cell, 2.8 to 19.2 kg/m^3 (8 to 12 lb/ft^3) aluminum honeycomb.
3. Adhesive to be American Cyanamid HT-424 (0-73011-3).
4. Ends to be potted with Shell Chemical Co. Epon 934 (0-00096-52).
5. Ends to be machined flat and parallel within ±0.0025 cm (±0.001 in.).
6. Specimen sides shall be parallel to 0.0076 cm (0.003 in.) Polish edges after machining.

Figure 4. Longitudinal Compression Test Specimen

4.6.10 Longitudinal flexural strength.

4.6.10.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.10.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 5.

4.6.10.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1031. The specimen shall be loaded to failure at a 0.127 cm ± 0.013 (0.050 in. ± 0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 589K ± 6 (600F ± 10) after a $10\ ^{+1}_{-0}$ minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 5 with the smooth side up.

4.6.10.4 Calculation. A mean value based on a minimum of three determinations shall be reported for longitudinal flexural strength using the formula below:

$$F_L = \frac{3PS}{2Wt^2}$$

where:

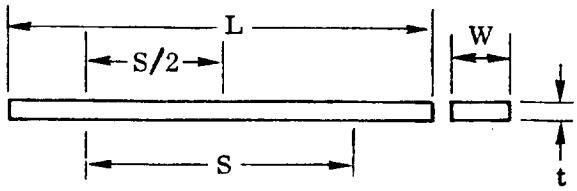
F_L = Ultimate longitudinal flexural strength

P = Maximum load carried by the specimen

S = Span

W = Specimen width

t = Specimen thickness



Specimen Dimensions

Length (L) = 7.6 to 10.2 cm (3.0 to 4.0 in.)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 32 to 1

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 5. Longitudinal Flexure Test Specimen

4.6.11 Transverse flexure strength.

4.6.11.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.11.2 Specimen. Fibers are aligned transverse to the longitudinal axis. Specimen configuration is shown in Figure 6.

4.6.11.3 Procedure. Unless otherwise specified, conditions of the test shall be in accordance with Federal Test Method Standard No. 406, Method 1031. The specimen shall be loaded to failure at a 0.127 cm ± 0.013 (0.050 in. ± 0.005) per minute crosshead speed in a testing machine. Test temperature shall be at room temperature and at 589K ± 6 (600F ± 10) after a $10\ ^{+1}_{-0}$ minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 6 with the smooth side up.

4.6.11.4 Calculation. Mean value based on a minimum of three determinations shall be reported for transverse flexural strength using the formula below:

$$F_t = \frac{3PS}{4Wt^2}$$

where: F_t = Ultimate transverse flexural strength

P = Maximum load carried by the specimen

S = Span

W = Specimen width

t = Specimen thickness

4.6.12 Short beam shear.

4.6.12.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.12.2 Specimen. Fibers are aligned parallel to the longitudinal axis. Specimen configuration is shown in Figure 7.

4.6.12.3 Procedure. The specimen shall be loaded to failure at a 0.127 cm ± 0.013 (0.050 in. ± 0.005) per minute crosshead speed in a testing machine. Test temperatures shall be at room temperature and at 589K ± 6 (600F ± 10) after a 10^{+1}_{-0} minute exposure at temperature (see 4.5). The specimen shall be loaded as shown in Figure 7 with the smooth side up.

4.6.12.4 Calculation. A mean value based on a minimum of three determinations shall be reported for short beam shear strength using the formula below:

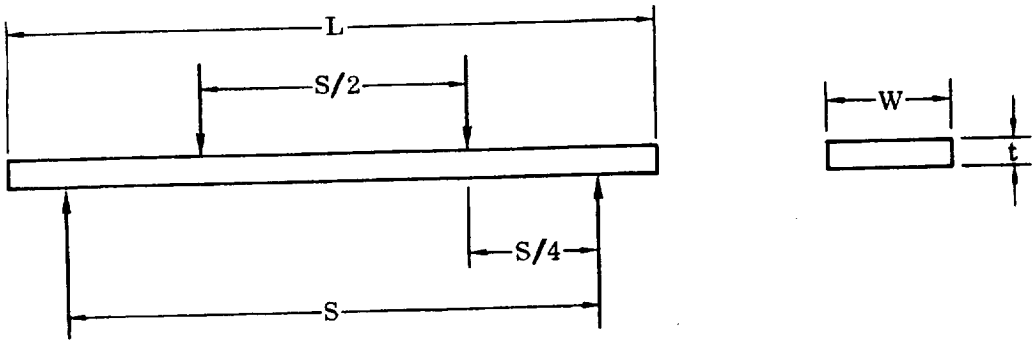
$$\tau = \frac{3P}{4Wt}$$

where: τ = Ultimate short beam shear strength

P = Maximum load carried by the specimen

W = Specimen width

t = Specimen thickness



Specimen Dimensions:

Length (L) = 7.6 cm ±0.25 (3.0 in. ± 0.1)

Width (W) = 1.270 cm (0.500 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span = 5.08 cm (2.00 in.)

Loading head and reaction supports are 0.635 cm (0.250 in.) diameter steel rod

Overhang must be the same over each end

Figure 6. Transverse Flexure Test Specimen

Specimen Dimensions:

Length (L) = 1.52 cm (0.060 in.)

Width (W) = 0.635 cm (0.250 in.)

Thickness (t) = 0.152 to 0.229 cm (0.060 to 0.090 in.)

Span/Thickness Ratio (S/t) = 4 to 1

Loading head and reaction supports are 0.318 cm (0.125 in.) diameter steel rod

Overhang must be the same over each end

Figure 7. Short Beam Shear Test Specimen

4.6.13 Specific gravity.

4.6.13.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.13.2 Test method. Specimen configuration and test procedure shall be in accordance with Federal Test Method Standard No. 406, Method 5011.

4.6.13.3 Calculation. Specific gravity calculations shall be per Federal Test Method Standard No. 406, Method 5011. A mean value based on a minimum of three determinations shall be reported.

4.6.14 Cured resin content and fiber volume.

4.6.14.1 Laminate preparation. Laminate shall be in accordance with 4.6.7.

4.6.14.2 Specimen. Test specimens shall be approximately 1.27 by 1.27 cm (0.5 by 0.5 in.) by laminate thickness. (Take specimens from a test panel used in a previous test conducted at room temperature.)

4.6.14.3 Procedure. The cured resin content and fiber volume shall be determined by acid/peroxide digestion as follows:

a. Weigh the test specimen to the nearest 0.1 mg (W_1), place in a 300 ml tall-form beaker, and add 20 ml of concentrated sulfuric acid. Place the beaker on a hot plate and heat the acid until vigorous fuming occurs.

b. When the composite is visibly disintegrated and resin particles and fibers are dispersed throughout the sulfuric solution, carefully add the hydrogen peroxide (50% strength) dropwise down the side of the beaker. Rubber gloves and a fume hood with appropriate safety glass shield shall be used throughout the addition and precautions shall be taken as recommended by the applicable safety regulations and procedures for handling hydrogen peroxide.

c. The reaction is considered complete when the hot sulfuric acid solution below the fibers becomes clear and colorless. At this point add two more ml of hydrogen peroxide to the solution, and heat the solution to fumes for another 10 minutes to ensure complete decomposition of the polymer. Remove the beaker from the hot plate, and allow to cool to 294 to 300K (70 to 80F) and then place in an ice bath.

d. Collect fibers by vacuum filtration through a medium-porosity, sintered-glass crucible that has been weighed to nearest mg (W_2). After the sulfuric acid has been filtered off, wash the fibers in the crucible thoroughly with 600 ml of distilled water, adding a few milliliters at a time. Verify removal of sulfuric acid traces by checking pH of the filtrate drops.

e. Remove the crucible from the filtering system and place in an open beaker in an oven at 573K (300F) for 45 minutes. After drying, cool the crucible in a desiccator and weigh (W_3).

4.6.14.4 Resin and fiber content calculation. Calculate the resin and fiber content according to the following equation:

$$\text{Resin content, percent by weight } (W_4) = \frac{W_1 - (W_3 - W_2)}{W_1} \times 100$$

$$\text{Fiber content, percent by weight } (W_5) = \frac{W_3 - W_2}{W_1} \times 100$$

4.6.14.5 Fiber volume calculation. Calculate the fiber volume using the data generated from the resin and fiber content determinations and the following formula:

$$\text{Fiber volume, percent} = \frac{W_6}{W_6 + (W_7 - W_6)\dfrac{\rho_1}{\rho_2}} \times 100$$

where: ρ_1 = Density of fiber

ρ_2 = Density of resin

W_7 = Weight of laminate - 1.00

W_6 = Weight fraction of dry graphite fiber $= \frac{W_5}{100}$

A mean value based on three determinations shall be reported.

4.6.15 Void content. The void content is calculated from the resin fiber content determinations. The resin specified in Specification 0-06052 contains 2 to 4 percent of silica as a filler, and this is treated as a separate entity in the void content determinations.

4.6.15.1 Calculation. A mean value based on three determinations shall be reported.

$$V_c = 100 - \frac{W_F \rho_c}{\rho_F} + \frac{W_R \rho_c}{\rho_R} + \frac{W_S \rho_c}{\rho_S}$$

where: V_c = Void content, volume percent

W_F = Weight percent of fiber

W_R = Weight percent of resin (without filler)

W_S = Weight percent of silica filler

ρ_c = Composite density

ρ_R = Density of unfilled resin

ρ_F = Density of fiber

ρ_S = Density of silica

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. The material shall be packaged with a non-adherent separator applied to both sheet faces. The material within one package shall be of the same length and width to preclude damage to the material during shipment. Packaged material shall be sealed within a moisture-proof plastic bag meeting the requirements of Military Specification MIL-B-131.

5.2 Packing. The packaged material shall be packed in shipping containers of a type which will ensure acceptance by common carrier at lowest rates and will ensure protection of the material during handling, transit, and storage.

5.3 Marking of interior package. Each interior package shall be legibly marked with a label or tab which includes the following minimum information:

a. 0-06053-1 or 0-06053-2
b. Manufacturer's material designation
c. Manufacturer's name and address
d. Batch number and sheet number
e. Date of manufacture
f. Weight
g. Recommended storage conditions and temperature range for maximum shelf life.
h. Estimated maximum shelf life based on recommended storage conditions and temperature range.
i. Hazardous warnings as applicable.

5.4 Marking of exterior shipping container. Each exterior shipping container shall be legibly and permanently marked with the following information:

a. 0-06053-1 or 0-06053-2
b. Purchase order number
c. Manufacturer's material designation
d. Manufacturer's name and address
e. Batch number
f. Quantity contained (sheet size and number of sheets)
g. Date of manufacture
h. Date of shipping
i. Manufacturer's recommended storage conditions and temperature range
j. Precautionary and handling markings.

In addition, the shipping container shall be identified with a strip of one-inch wide green plastic tape (Scotchlite 3277 or equivalent) completely around the container from top to bottom and approximately one inch from the side.

6. NOTES

6.1 Intended use. The material covered by this specification is intended for use in the manufacture of missile and spacecraft structural components subject to temperatures from 20K (-423F) to 589K (600F). Use is not restricted to these applications.

6.2 Ordering information. The following information should be included on the purchase order, together with the conditions of 6.2.1 and 6.2.2.

a. Number, title, and date of specification
b. Material name and quantity.

6.2.1 Rejection and retest. In the event of failure of a sample to meet any of the requirements of this specification, a second sample of uncured material taken adjacent to the first, or a second laminate panel prepared in accordance with 4.6.7 may be submitted for retest. If the retest sample fails to meet the requirements of the specification, the batch represented by the sample shall be rejected.

6.2.2 Reports. Unless otherwise specified, the supplier shall furnish with each lot three copies of the reports showing the results of tests made on each batch in the shipment to determine conformance of the material to the specification requirements. The report shall include volatile content, resin solids, resin flow, tack, and mechanical properties, as applicable. The report shall also include the purchase order number, the material specification number, supplier designation, quantity, batch number, and date of manufacture. The report shall also include fiber properties as reported by the fiber manufacturer, and the batch numbers of fiber used in making each of the sheets of material.

6.3 Approved sources. The approved sources for the material described in this specification are as follows:

Convair Designation	Manufacturer's Designation	Manufacturer's Name and Address
0-06053-1	G/710	E. I. DuPont DeNemours & Company Fabrics & Finishes Department Saugus, California 91350
0-06053-1	6002T	Hercules Incorporated Bacchus Works Magna, Utah 84044
0-06053-2	G/710	E. I. DuPont DeNemours & Company Fabrics & Finishes Department Saugus, California 91350
0-06053-2	6002M	Hercules Incorporated Bacchus Works Magna, Utah 84044