To AEC-NASA Space Nuclear Propulsion Office

THE FABRICATION AND PROPERTIES
OF THE FIBROUS REINFORCED
GRAPHITE COMPOSITE, AGCarb-101

RN-S-0549

March 1970

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AEROJET NUCLEAR SYSTEMS COMPANY

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THE FABRICATION AND PROPERTIES
OF THE FIBROUS REINFORCED
GRAPHITE COMPOSITE, AGCarb-101

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ABSTRACT

The fabrication characteristics for the production of a fibrous-graphite-reinforced composite, AGCarb-101, are described. Plates and cones were fabricated to determine the following physical and mechanical properties: tensile strength in three directions, compression strength, flexure strength, interlaminar shear strength, fatigue strength, fracture toughness, dynamic modulus, damping decrement, porosity, permeability, hardness, electrical resistivity, thermal expansion and thermal conductivity. The data obtained from the plates are to be used as baseline data for design purposes. The data from the cones were to be used to determine the effect of the configuration on the properties; however, defects (wrinkles) in the cones prevented correlation of data from plates and cones. Additional work is required to improve the fabrication characteristics of the cones.

FOREWORD

The work described in this report was performed by the personnel in Materials Engineering Group, Aerojet Liquid Rocket Company. R. J. Kotfila, as Project Engineer, was responsible for the physical and mechanical property determinations; H. O. Davis was cognizant engineer for the fabrication of plates and cones; W. Schleicher assisted in property determination. The work was monitored by H. J. Bronner and M. Takemori, Skirt Extension Group, Thrust Chamber Department, and I. M. Rehn, Materials and Processes, Aerojet Nuclear Systems Company.

TABLE OF CONTENTS

	<u>Page</u>
I. Introduction	1
II. Summary	2
III. Fabrication	7
A. General	7
B. Documentation of Fabrication History for the Cones and Plates	9
1. Raw Materials	9
2. Material Distribution	13
3. Material Cure and Postcure	17
4. Carbonization	22
5. Graphitization and Impregnation	23
C. Analysis of In-process and Final Component Measurements	24
1. Shrinkage	25
2. Weight Change During Processing	31
3. Density	34
IV. Physical and Mechanical Properties	36
A. General	36
B. Plates	36
1. Tensile Strength	36
2. Elevated-Temperature Tensile Strength	45
3. Block Tensile Strength	49
4. Fill Tensile Strength	54
5. Bias Tensile Strength	56
6. Interlaminar Shear Strength	60
7. Compression Strength	63
8. Fatigue Life	68
9. Fracture Toughness	73
10. Flexure Strength	78
11. Dynamic Modulus	84
12. Damping Decrement	86
13. Thermal Conductivity	88

TABLE OF CONTENTS (cont.)

	<u>Page</u>
14. Thermal Expansion	97
15. Density	101
16. Porosity	101
17. Permeability	108
18. Hardness	109
19. Electrical Resistivity	113
C. Cones	118
1. Hoop Tensile Strength	118
2. Tensile Strength	138
3. Block Tensile Strength	143
4. Compressive Strength	144
5. Interlaminar Shear Strength	144
6. Hardness	148
7. Electrical Resistivity	148
8. Porosity	148
V. Conclusions	150
VI. Recommendations	152

APPENDIX

A	Location and Identification of Test Specimens from 1/4- and 1-in. Plates
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LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Summary of the Physical and Mechanical Properties of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	4
2	Plate and Cone Identification and Description	8
3	Properties of WCA Graphite Cloth	10
4	Typical Physical Properties of Cast USP 39 Resin	12
5	Comparison of Ovalness and Shrinkage of Cones as Measured on Minimum and Maximum ID After Complete Processing	15
6	Average Dimensional Change in in./in. for Plates (Thickness)	26
7	Average Dimensional Change in in./in. for Plates (Length and Width)	30
8	Shrinkage, in./in., for Plates with Different Processing	32
9	Room-Temperature Tensile Test Results of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (Flat Specimens)	39
10	Room-Temperature Tensile Test Results of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (Round Specimens, 1-in.-thick plate)	40
11	Room-Temperature Tensile Test Results of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (round specimens, 1/4-in.-thick plate)	41
12	Elevated-Temperature Tensile Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	46
13	Tensile Modulus of a Fibrous-Reinforced-Graphite Composite	51
14	Block Tensile Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	53
15	Room-Temperature Fill-Direction Tensile Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	55
16	Tensile Strength of Bias Cut (45 Deg) Fibrous-Reinforced-Graphite Composite, AGCarb-101	59
17	Interlaminar Shear Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	62
18	Compressive Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (1/4-in.-thick plate)	65

LIST OF TABLES (cont.)

<u>Table</u>		<u>Page</u>
19	Compressive Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (1-in.-thick plate)	66
20	Rotating-Beam Fatigue Test Results of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	71
21	Fracture Toughness of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	76
22	Flexural Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	82
23	Density of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	102
24	Vendor's Certification for Plates of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	103
25	Porosity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	107
26	Permeability of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	110
27	Rockwell R Hardness Test Results of a Fibrous Reinforced-Graphite Composite, AGCarb-101 (1/4-in.-thick plate)	111
28	Rockwell R Hardness of a Fibrous-Reinforced-Graphite Composite, AGCarb-101 (1-in.-thick plate)	112
29	Electrical Resistivity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	116
30	Number and Amplitude of Wrinkles in AGCarb-101, Fibrous-Reinforced Graphite, Cones	119
31	Hoop Tensile Strength of AGCarb-101, Fibrous-Reinforced Graphite, Cones	134
32	Room-Temperature Tensile Test Results of AGCarb-101, Fibrous-Reinforced Graphite, Cones	141
33	Elevated-Temperature Tensile Test Results of AGCarb-101, Fibrous-Reinforced Graphite, Cones	142
34	Block Tensile Strength of AGCarb-101, Fibrous-Reinforced Graphite, Cones	145
35	Compressive Strength of AGCarb-101, Fibrous-Reinforced Graphite, Cones	146

LIST OF TABLES (cont.)

<u>Table</u>		<u>Page</u>
36	Interlaminar Shear Strength of AGCarb-101, Fibrous-Reinforced Graphite, Cones	147
37	Hardness, Electrical Resistivity, and Porosity of AGCarb-101, Fibrous-Reinforced Graphite, Cones	149

FIGURE LIST

<u>Figure</u>		<u>Page</u>
1	Dimensions of AGCarb-101 Cone 5A1; Bias Tape, No Mandrel	18
2	Dimensions of AGCarb-101 Cone 5A2; Bias Tape, Graphite Mandrel	19
3	Dimensions of AGCarb-101 Cone 5B1; Warp, No Mandrel	20
4	Dimensions of AGCarb-101 Cone 5B2; Warp, Graphite Mandrel	21
5	Average Shrinkage in./in. (Thickness Perpendicular to Plies) of AGCarb-101 (Fibrous-Reinforced Graphite) Plates	28
6	Average Percent Weight Gain and Loss and Average Density for Each Type of Plate of AGCarb-101	33
7	Flat Tensile Specimen, 1-in.-thick plate	37
8	Round Tensile Specimen	37
9	Microstructure of a Fibrous-Graphite-Reinforced Composite (AGCarb-101) Showing Density Effects	44
10	Tensile Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	50
11	Block Tensile Coupon, 0.250-in.-thick plate	52
12	Block Tensile Coupon, 1-in.-thick plate	52
13	Macrostructure of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	57
14	Microstructure of a Fibrous-Reinforced-Graphite Composite (AGCarb-101) Illustrating Difference of Orientation	58
15	Interlaminar Shear Specimen, 0.250-in.-thick plate	61
16	Interlaminar Shear Specimen, 1-in.-thick plate	61
17	Compression Specimen	64
18	Rotating-Beam Fatigue Specimen, 0.250-in.-thick plate	69
19	Rotating-Beam Fatigue Specimen, 1-in. thick plate	70
20	Microstructure of Fatigue Failure of AGCarb-101 Specimen	74
21	WOL Fracture-Toughness Specimen	75
22	Flexure Specimen, 1.75-in. Span	79
23	Flexure Specimen, 3.2-in. Span	80

FIGURE LIST (cont.)

<u>Figure</u>		<u>Page</u>
24	Thermal Diffusivity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	93
25	Calculated Thermal Conductivity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	94
26	Thermal Conductivity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101, Thermal Gradient Technique	95
27	Thermal Conductivity of Fibrous-Graphite Composites	96
28	Thermal-Expansion Specimens	98
29	Thermal Expansion of a Fibrous-Reinforced-Graphite Composite, AGCarb-101	100
30	Porosity Specimen	105
31	Electrical-Resistivity Specimen	114
32	Electrical-Resistivity Setup	115
33	Hoop Tensile Specimen HT-1 Cut from Bias-Tape Cone 5A1, Showing Wrinkles	120
34	Hoop Tensile Specimen HT-2 Cut from Bias-Tape Cone 5A1, No Wrinkles Noted	121
35	Hoop Tensile Specimen HT-3 Cut from Bias-Tape Cone 5A1, No Wrinkles Noted	122
36	Hoop Tensile Specimen HT-1 Cut from Bias-Tape Cone 5A2, No Wrinkles Noted	123
37	Hoop Tensile Specimen HT-2 Cut from Bias-Tape Cone 5A2, No Wrinkles Noted	124
38	Hoop Tensile Specimen HT-3 Cut from Bias-Tape Cone 5A2, No Wrinkles Noted	125
39	Hoop Tensile Specimen HT-1 Cut from Straight-Tape Cone 5B1, Showing Wrinkles	126
40	Hoop Tensile Specimen HT-2 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	127
41	Hoop Tensile Specimen HT-3 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	128
42	Hoop Tensile Specimen HT-4 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	129
43	Hoop Tensile Specimen HT-1 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	130

FIGURE LIST (cont.)

<u>Figure</u>		<u>Page</u>
44	Hoop Tensile Specimen HT-2 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	131
45	Hoop Tensile Specimen HT-3 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	132
46	Hoop Tensile Specimen HT-4 Cut from Straight-Tape Cone 5B2, Showing Wrinkles	133
47	Fractures of Hoop Tensile Specimens Showing Effect of Wrinkles	136
48	Microstructure of a Fibrous-Graphite-Reinforced Composite (AGCarb-101) Straight-Tape-Wrapped Cone	137
49	Fracture of Hoop Tensile Specimen in Bias-Wrapped Cone with No Wrinkles	139
50	Microstructure of a Fibrous-Reinforced-Graphite Composite (AGCarb-101) Bias- (45 Deg) Tape-Wrapped Cone	140

I. INTRODUCTION

Major advancements have been made in the development and use of fibrous-reinforced materials for structural purposes. These advances have occurred concurrently in the field of raw materials and fabrication, permitting the selection of these materials for structural components in advanced aerospace vehicles and rocket engines. Of the many and varied fibrous-reinforced-graphite composites available, AGCarb-101 has established a record of reliable performance in rocket engine service. AGCarb-101 is a graphite-fabric reinforcement in a graphite matrix. Components made from this material were designed with very limited physical- and mechanical-property data. For efficient and reliable structural applications of this material in high-temperature structural application, the physical and mechanical properties must be sufficiently defined to determine the full potential of the material and to establish its limitations.

The objective of this program is to provide baseline data for the physical and mechanical properties of AGCarb-101 made to a prescribed fabrication plan. These properties are to be compared with those obtained on a structural component (cones) to determine the extent of component-property relationship and to establish the limitations of the material, the fabrication process, and the inspection procedures. From these findings, improvements in raw-materials processing, and/or design should be obtained to gain the required reliability of a structural component for man-rated service application.

II. SUMMARY

To determine the potential of the limitations of graphite-reinforced composites for applications requiring structural integrity, a program was conducted to evaluate the fabrication characteristics and to determine the physical and mechanical properties of AGCarb-101, a composite consisting of graphite-fabric reinforcement in a graphite matrix. Four cones and four plates of two thicknesses (1/4 and 1 in.) were fabricated by using the procedures developed in previous programs. The plates were used to determine the physical and mechanical property data; the cones were used to evaluate the effect of the configuration on fabrication characteristics and properties.

The plates were made with a hand lay-up of straight and 45-deg-bias-cut fabric in a picture-frame fixture. The plates were cured, post-cured, carbonized, graphitized, impregnated, graphitized, re-impregnated, and re-graphitized, to an established processing schedule. All fabrication up to and including carbonization was performed by the San Rafael Plastics Co.; impregnation and graphitization were performed by the Union Carbide Corp. The final density range of the plates ranged from 1.485 to 1.505 gms/cc. The maximum shrinkage occurred in the perpendicular to ply direction. No measurable shrinkage occurred in the warp and fill direction during the postcure and carbonization cycles; all shrinkage occurred during the graphitization cycles.

The cones were wrapped with straight and 45 degree bias tape. Steel mandrels were used for two cones (one each of straight and bias tape) and bulk-graphite mandrels were used for the other two cones. The graphite mandrels were used as support fixtures for the cones during subsequent processing, and the cones wrapped on steel mandrels were processed without support fixtures. The sequence of operations was the same as that for the plates; however, the second impregnation and the third graphitization were not required to achieve the specified minimum density of 1.40 g/cc.

The cones wrapped with straight tape had as many as seven wrinkles around the circumference with a maximum amplitude of 0.080 in. The wrinkles were a direct result of the low as-wrapped density (less than 90% of cured theoretical density). Only one bias-wrapped cone was free of wrinkles; one cone had a delamination that was detected by X-ray.

A summary of the physical and mechanical properties of the AGCarb-101 plates and cones is shown in Table 1. The 1/4-in.-thick plates generally had higher mechanical properties. The plates are characterized by gross anisotropy in tensile strength with the highest being in the warp direction and the lowest being in the perpendicular to ply direction.

The wrinkle and tape-orientation effects prevented correlation of cone properties with those of the plates. The wrinkles even of small amplitude seriously reduced the hoop strength of the cones. Other properties were not as seriously affected.

Improvements in the fabrication process are required to obtain conical or cylindrical components that have the required structural integrity necessary for maximum reliability.

TABLE 1

SUMMARY OF PHYSICAL AND MECHANICAL
PROPERTIES OF A FIBROUS REINFORCED GRAPHITE
COMPOSITE, AGCarb-101

A. Plates	Temperature, °F		
	RT	2000°	3000°
Tensile Strength, psi			
1/4-in. thick	11,140	11,750	13,467
1-in. thick	10,450	11,529	12,782
Tensile Modulus, psi x 10 ⁶			
1-in. thick	2.31		1.74
Block Tensile Strength, psi			
1/4-in. thick	638		
1-in. thick	644		
Fill Tensile Strength, psi			
1-in. thick	4,563		
Bias (45 deg) Tensile Strength, psi			
1/4-in. thick	4,015		
1-in. thick	4,432		
Interlaminar Shear Strength, psi			
1/4-in. thick	1,300		
1-in. thick	1,350		
Compression Strength, psi			
1/4-in. thick	9,500	9,160	9,073
1-in. thick	9,400	8,720	9,125
Fatigue Strength at 10 ⁷ cycles, psi			
1/4-in. thick	9,700		
1-in. thick	8,500		
Fracture Toughness K _{IC} , ksi√in.			
1-in. thick warp	8.5		
fill	6.55		
Flexure Strength, psi			
1/4-in. thick, parallel to ply	19,980	-	23,485
1/4-in. thick, perpendicular to ply	17,595		20,630
1-in. thick, parallel to ply	17,514	-	19,950
1-in. thick, perpendicular to ply	16,254	-	19,650
Dynamic Modulus, psi x 10 ⁶			
1/4-in. thick	2.94		
1-in. thick	2.86		

TABLE 1 (cont.)

A. Plates (cont.)		Temperature, °F			
		RT 5-7 x 10 ⁻³	1000°	2000°	3000°
Damping Log Decrement					
1/4-in. thick					
Thermal Conductivity, Btu/hr/ft ² /°F/ft					
1-in. thick, parallel to ply		-	19	15	14
perpendicular to ply		-	12	11	10
Thermal Expansion, in./in. x 10 ⁻³					
1-in. thick, parallel to ply		-	1.6	3.2	5.5
perpendicular to ply		-	2.6	5.3	7.8
Density, gm/cc		1.45 ± 0.05			
Total Porosity, vol %					
1/4-in. thick		8.7			
1-in. thick		9.7			
Permeability, Darcys x 10 ⁻⁴					
1/4-in. thick, 35 psig N ₂		0.715			
1-in. thick, 59 psig N ₂		0.33			
Hardness, Rockwell R					
1/4-in. thick		97/104			
1-in. thick		93/105			
Electrical Resistivity, ohm-cm x 10 ⁻⁴					
1-in. thick		12.7			
1-in. thick		15.45			
B. Cones		RT	2000°	3000°	
Hoop Strength, psi					
Straight-Tape Wrapped	No. 1	2,494			
	No. 2	3,078			
45-deg-Bias-Tape Wrapped	No. 1	2,994			
	No. 2	4,076			
Tensile Strength, psi					
Straight-Tape Wrapped	No. 1	2,375	-	-	
	No. 2	2,800	2,650	3,430	
45-deg-Bias-Tape Wrapped	No. 1	2,313	-	-	
	No. 2	3,425	3,500	3,970	
Block Tensile Strength, psi					
Straight-Tape Wrapped	No. 1	767			
	No. 2	703			
45-deg-Bias-Tape Wrapped	No. 1	763			
	No. 2	696			

TABLE 1 (cont.)

B. Cones (cont.)		RT	2000°	3000°
<hr/>				
Compressive Strength, psi				
Straight-Tape Wrapped	No. 1	4,206		
	No. 2	4,182		
45-deg-Bias-Tape Wrapped	No. 1	4,795		
	No. 2	5,033		
Hardness, Rockwell R				
Straight-Tape Wrapped	No. 1	74-75		
	No. 2	75-76		
45-deg-Bias-Tape Wrapped	No. 1	65-69		
	No. 2	86-87		
Electrical Resistivity, ohm-cm x 10 ⁻⁴				
Straight-Taped Wrapped	No. 1	19.2		
	No. 2	16.5		
45-deg-Bias-Tape Wrapped	No. 1	18.0		
	No. 2	16.2		
Total Porosity, Volume %				
Straight-Tape Wrapped	No. 1	13.5		
	No. 2	15.1		
45-deg-Bias-Tape Wrapped	No. 1	15.7		
	No. 2	9.9		

III. FABRICATION

A. GENERAL

To better characterize AGCarb-101, (a graphite-fabric reinforcement in a graphitic matrix) four cones and four types of molded plates were fabricated. The identification and description of the cones and plates are shown in Table 2. Fabrication was performed by both the San Rafael Plastics Co. and the Union Carbide Corp. Processing techniques that had previously been developed were used with some changes in the mandrel material and in the impregnation of the graphitized components. The fabrication of AGCarb-101 in any configuration is basically a step process with each step being independent of the previous step. However, the end product of each step affects the final end product particularly in relation to density and structural integrity. The steps are identified as:

1. Raw-materials selection (reinforcement and resin)
2. Material distribution (placement of material and fabric orientation)
3. Cure and postcure
4. Carbonization
5. Graphitization and impregnation

The result of the five steps listed above with the necessary controls at each step is AGCarb-101. The chemistry of the starting resin is considered proprietary to the U.S. Polymeric Chemicals, Inc., and the Code 88 pitch and graphitization cycle is considered proprietary to Union Carbide Corp. However, the directions for combining the materials and processes to form the final fibrous-graphite material were first given by the Aerojet-General Corp. The details of each individual fabrication step for the cones and plates are discussed in the following sections. These details are based on records from the processing history and the manufacturing plan. The cones and plates are discussed separately where differences in the

TABLE 2

PLATE AND CONE IDENTIFICATION AND DESCRIPTION

<u>Plate⁽¹⁾ No.</u>	<u>Approximate Thickness, in.</u>	<u>Width, in.</u>	<u>Length, in.</u>	<u>Direction of Fabric Parallel to Length</u>
1702-1	1/4	16	12	45-deg bias ⁽²⁾
1702-2	1	24	12	45-deg bias
1702-3	1/4	36	24	Warp ⁽³⁾
1702-4	1	36	24	Warp

<u>Cone No.</u>	<u>Type Tape</u>	<u>Approximate Thickness, in.</u>	<u>Approximate Length, in.</u>	<u>Min. ID, in.</u>	<u>Max. ID, in.</u>	<u>Mandrel Material</u>
5A1	Bias	1/2	13	2.325	9.5	Steel
5A2	Bias	1/2	13	2.325	9.5	Graphite
5B1	Straight (Warp)	1/2	13	2.325	9.5	Steel
5B2	Straight (Warp)	1/2	13	2.325	9.5	Graphite

- (1) After cure, all plates were trimmed and cut into sections, 4.8-in. wide x 10.5-in. long (Warp direction parallel to 10.5-in. length)
- (2) 45-deg bias - Fabric warp direction is 45 deg to length of specimen.
- (3) Warp - Fabric warp direction is parallel to length.

fabrication method exist. All processing details such as time and temperature are not included; however the details for cure, postcure and carbonization are available.

B. DOCUMENTATION OF FABRICATION HISTORY FOR THE CONES AND PLATES

1. Raw Materials

The raw materials used to make the starting resin-impregnated fabric (prepreg) are WCA graphite fabric and USP 39 resin. These materials are combined by the standard impregnation process used for fiberglass and ablative reinforcing fabrics. The WCA graphite fabric is a plain (square) weave fabric with approximately the same number of warp and fill yarns.⁽¹⁾ There are nominally 27 warp yarns and 21 fill yarns/inch. Each yarn bundle has 1440 filaments; the diameter of each filament is 0.0004 in. The gage (thickness) is 0.018 in., and the weight averages 7.4 oz/sq yard. Carbon assay is 99.9 wt%, and surface area averages 3 M²/gram. Density measured in helium is 1.42 gms/cc.

The property measurements reported for the lot (9C8WCA3) of WCA graphite fabric used in the fabrication of the cones and plates are shown in Table 3.

- (1) Warp = yarns that run parallel to length of fabric
Fill = yarns that run the width of the fabric

TABLE 3
 PROPERTIES OF WCA GRAPHITE CLOTH

		(Lot 9C8WCA3)		Lot WCA-2-117 ⁽¹⁾	
		<u>Min.</u>	<u>Max.</u>	<u>Min.</u>	<u>Max.</u>
Tensile Strength, lb/in. width - Warp		37.4	75.4	41	61
	- Fill	15.0	59.4	33	39
Thread Count, threads/in. - Warp		27	28	28	28
	- Fill	21	22	22	22
Electrical Resistance, ohms/ sq. in. - Warp		0.36	0.39	-	-
	- Fill	0.49	0.54	-	-
pH				7.2	
Carbon Assay, wt%		99.2		-	-
Average Weight, oz/sq yd		7.340		7.37	
Density, gms/cc		1.41	1.45	1.392	1.446

(1) Values from a previous lot. Davis, H. O., Lampman, J. A., Warga, J. J., Evaluation of Characteristics Affecting Attainment of Optimum Properties of Ablative Plastics, AFRPL-TR-68-29, Vol. I and II, February 1968.

The only value reported that exceeds the values reported in the Union Carbide Corp. product bulletin is carbon assay. It is assumed that the product bulletin refers to an average. For better control and more reproducible properties in the end product, lower and upper limits should be established for WCA fabric used in fabrication of AGCarb-101.

The values obtained on a previous WCA lot (WCA-2-117) used in a process variable study are also shown in Table 3. The values are listed to show the variation in the WCA graphite fabric. The range in tensile strengths for both warp and fill directions for lot 9C8WCA3 was twice that measured for lot WCA-2-117. A concentration of the low-strength fabric in any area would significantly lower the tensile strength of the end product. The low (minimum value) for the fill breaking strength is believed to be somewhat related to the low tensile strengths in the fill direction for the plates. The values obtained are discussed later in the report.

Much less is known about the resin system. This is generally true for thermosetting resins used in structural and ablative plastics. It is difficult to characterize a resin system which is a group of polymers i.e., primarily characterized by a hydroxyl group attached to a benzene ring in various forms and stages of combination with various molecular weights in an alcohol solvent. The USP 39 resin system used by U.S. Polymeric Chemicals, Inc. to make the AGCarb-101 prepreg (FM 5228) is reported to be a phenyl-aldehyde-condensation resin and more recently identified as a polyphenyl resin. The typical properties of the cast resin are shown in Table 4.

USP 39 resin also contains a carbonaceous filler of which the quantity, type, and particle size are considered proprietary by U.S. Polymeric Chemical, Inc. An analysis has not been conducted at AGC to identify and characterize the filler. However, the filler distribution and particle size can be ascertained with an electron microscope at magnification of 10,000 to 20,000.

TABLE 4

TYPICAL PHYSICAL PROPERTIES OF CAST USP 39 RESIN⁽¹⁾

Density, gr/cc	1.3
Ultimate Tensile Strength, psi	13,000
Elongation, %	1.5
Flexural Modulus, psi	0.5×10^6
Ultimate Flexural Strength, psi	20,000
Compressive Strength, psi	30,000
Barcol Hardness	90
Specific Heat, Btu/lb	0.5
Thermal Conductivity, Btu/in./ft ² -hr-°F	0.78
Thermal Expansion Coefficient, in./in. °F x 10 ⁻⁶ 75°F/400°F	32
Weight Loss after 1/2 hr Exposure to 1500°F, %	18.4 ⁽²⁾

(1) Obtained from U.S. Polymeric Chemical, Inc.

(2) An average of six specimens - processing or testing
must have been uncontrolled - range was 13 to 22%.

The viscosity, solids content, and pH of a typical batch of USP 39 resin were determined in a program conducted by AGC for AFRPL⁽¹⁾. The average viscosity was 223 centipoise. The solids content was 80.4% wt, and the pH was 8.51. In general, the pH and viscosity is normal for phenolic resins, but resin solids are 10 to 25% higher than for standard phenolics used for impregnating glass, silica, carbon, and graphite fabrics.

Another distinguishing characteristic of USP 39 is the high percentage of acetone extractable material after complete cure. The acetone extractable substance has been isolated in the crystalline form but has not been analyzed. The material appears to be thermoplastic. It is not known whether the substance is an unreacted constituent or an additive to facilitate fabrication. It could affect the carbonization process and should be analyzed.

2. Material Distribution (Placement of Material and Fabric Orientation)

Two basic types of material distribution (fabric lay-up) were selected for the study program. They were (1) tape wrapping on a male mandrel and (2) hand lay-up of cut patterns of flat plates. Each technique is standard and readily adaptable to any size and configuration; however, the tape-wrap process is more economical and is generally used for composite cones or cylinders. Four tape-wrapped cones and four flat plates were fabricated using both bias and straight tape for each. The cones were 15-deg half-angle with a minimum OD of 3 in. and a maximum OD of 10-1/4 in. Two flat plates were 1/4-in. and two were 1-in. thick. The dimensions are

(1) Davis, H. O., Lampman, J. A., Warga, J. J., Evaluation of Characteristics Affecting Attainment of Optimum Properties of Ablative Plastics AFRPL TR 68-29 Vol. I and II February 1968.

shown in Table 2. Plate areas ranged from approximately 170 to 900 sq. in. The plates were cut in smaller sections (4.8 x 10.5 in.) after cure.

The cones and plates were fabricated in accordance with the "Manufacturing and Inspection Plan," AGC P.O. 103707-C01 J1702, 30 October 1968. The orientation of the fabric on the cones was parallel to the centerline of the component. The fabric in the plates was laid-up parallel to the molding surface of the steel plates. Both straight and bias-cut tape were used for molding plates and wrapping cones. The bias fabric was cut at a 45-deg angle to the warp and fill yarns. This angle does change slightly because of "neck down" during wrapping. In the straight-tape-wrapped cones, the warp yarns are positioned in a circumferential direction around the cone. The fill yarns are in the axial direction parallel to centerline.

Two different materials, ATJ graphite and steel, were used for the wrapping mandrels. Graphite was used as a mandrel material to determine if it could be utilized as a holding fixture during the carbonization and first graphitization cycle to prevent out-of-roundness due to warpage.

The comparative effects of the steel and graphite mandrels on the shrinkage and ovality are shown in Table 5. The cones from the graphite mandrels had less ovality and diameter shrinkage. The steel mandrels were removed after the first graphitization cycle.

The general parameters for tape wrapping the cones were included in the manufacturing plan; however, the exact tape-wrapping pressure was not reported in the certifications. If the 100-lb/in width was used (as specified in the manufacturing plan), it was inadequate for the straight tape. Extensive wrinkling occurred and some of the wrinkles had an amplitude of 0.080. These wrinkles significantly affected the hoop tensile strength of the straight-tape cones, which is discussed in a later section of this report.

TABLE 5

COMPARISON OF OVALNESS AND SHRINKAGE OF CONES AS MEASURED ON MINIMUM
AND MAXIMUM INSIDE DIAMETERS AFTER COMPLETE PROCESSING

<u>Cone No.</u>	<u>Type Mandrel</u>	<u>Type Tape</u>	<u>Ovalness</u>		<u>Shrinkage</u>	
			<u>Min. ID in.</u>	<u>Max. ID in.</u>	<u>Min. ID in.</u>	<u>Max. ID in.</u>
5A1	Steel (None after cure)	Bias	0.001	0.013	0.085	0.150
5A2	Graphite (through 1st Graphitization)	Bias	0.005	0.002	0.033	0.020
5B1	Steel (None after cure)	ST	0.001	0.060	0.071	0.175
5B2	Graphite (through 1st Graphitization)	ST	0.010	0.003	0.019	0.059

The interrelation of tape-wrapping parameters has been studied in detail.⁽¹⁾ A minimum debulk of 90% as-wrapped density can be readily achieved for parallel to centerline wrapping. The as-wrapped density is based on the percentage of theoretical (average cured) density achieved during the wrapping process. The necessary wrapping controls (for the resin content and degree of polymerization on the lot of FM 5228 prepreg) were not achieved as evidenced by the excess wrinkles. The factors that must be controlled were in the manufacturing plan, but the combination of the limits used on (1) wrap speed, (2) tape temperature, (3) billet temperature, and (4) roller pressure did not produce the desired as-wrapped density of 90% or greater.

The as-wrapped density was not determined, even though it was added to the initial manufacturing plan. A rough calculation indicated that less than 85% as-wrapped density was achieved.

Observation of the cones after wrapping also indicated a lack of sufficient control in the traverse of the roller as tape was applied. A uniform feed based on a known as-wrapped ply thickness will produce a smooth OD surface. A constant "neck down" must be maintained for the bias tape. This was not achieved; however, there are no known effects on the finished-composite properties related to the uneven OD surface.

(1) Davis, H. O., Lampman, J. A. Warga, J. J., Evaluation of Characteristics Affecting Attainment of Optimum Properties of Ablative Plastics AFRPL TR 68-29 Vol. I and II, February 1968.

The tape width and wrapping conditions were sufficient to produce a 0.500-in. wall thickness throughout each cone with the exception of some local areas. The cone identified as 5B1 was the only cone with no areas below 0.500-in. in thickness after the processing was completed. The thicknesses for cones 5A1, 5A2, 5B1, and 5B2 are shown in Figures 1, 2, 3, and 4 respectively. Measurements were taken at 1.5-in. intervals axially.

3. Material Cure and Postcure

The plates were cured on steel plates with the laminates surrounded by "frames" to reduce edge effects from the pressure bag. A flexible bagging material was used over bleeder cloth. Vacuum was pulled on the laid-up plates and a 1000-psi gas pressure was applied in an autoclave. The cycle used was per specifications in the manufacturing plan. A flexible bag (clave cure) was utilized to better simulate future fabrication techniques for cones and/or cylinders. Platen presses (generally used to make flat laminates) cannot be used for cones or cylinders of laid-up fabric.

The wrapped cones were bagged and cured in the same manner as the plates. No bag leakage problems were recorded.

The plates and cones were postcured in accordance with the cycle specified in the manufacturing plan. No delaminations or cracks were reported after postcure. The weight and dimensional changes between cure and postcure were recorded. The values for each plate and cone are shown in the Certification of Conformance, dated 19 and 26 March 1969. Other properties reported in the Certification of Conformance both before and after postcure were Barcol hardness and density. The components were also alcohol penetrant and radiographic inspected after cure and postcure.

THICKNESS AT 1.5 INCH INTERVALS		
STA.	0°	90°
1	.524	.524
2	.524	.523
3	.524	.524
4	.523	.523
5	.475	.511
6	.523	.526
7	.511	.516
8	.521	.526
MAX.VAR.	.049	.015

STA	180°	270°
1	.522	.523
2	.523	.524
3	.523	.525
4	.524	.525
5	.523	.524
6	.526	.525
7	.527	.496
8	.522	.519
MAX.VAR.	.005	.029

	D1	D2
OVALITY	.001	.013
SHRINK.	.085	.150

Figure 1 - Dimensions of AGCarb-101 Cone 5A1; Bias Tape, No Mandrel

THICKNESS AT 1.5 INCH INTERVALS		
STA	0°	90°
1	.496	.504
2	.496	.505
3	.501	.506
4	.506	.507
5	.500	.493
6	.485	.476
7	.479	.492
8	.501	.501
MAX.VAR.	.027	.031

STA	180°	270°
1	.498	.494
2	.499	.496
3	.501	.499
4	.503	.501
5	.491	.499
6	.477	.495
7	.479	.485
8	.509	.508
MAX.VAR.	.032	.023

	D1	D2
OVALITY	.005	.002
SHRINK.	.033	.020

Figure 2 - Dimensions of AGCarb-101 Cone 5A2; Bias Tape, Graphite Mandrel

THICKNESS AT 1.5 INCH INTERVALS		
STA	0°	90°
1	.535	.537
2	.537	.538
3	.536	.539
4	.537	.538
5	.539	.538
6	.540	.539
7	.541	.541
8	.542	.540
MAX. VAR.	.007	.004

STA	180°	270°
1	.535	.530
2	.536	.533
3	.537	.536
4	.537	.537
5	.537	.537
6	.536	.539
7	.538	.538
8	.539	.538
MAX. VAR.	.004	.009

	D1	D2
OVALITY	.001	.060
SHRINK.	.071	.175

Figure 3 - Dimensions of AGCarb-101 Cone 5Bl; Warp, No Mandrel

THICKNESS AT 1.5 INCH INTERVALS		
STA	0°	90°
1	.525	.525
2	.525	.525
3	.525	.524
4	.525	.523
5	.514	.511
6	.502	.486
7	.505	.503
8	.527	.521
MAX.VAR.	.025	.039

STA	180°	270°
1	.523	.522
2	.523	.523
3	.523	.523
4	.515	.523
5	.500	.511
6	.494	.500
7	.509	.513
8	.509	.524
MAX.VAR.	.029	.024

	D1	D2
OVALITY	.010	.003
SHRINK.	.019	.059

Figure 4 - Dimensions of AGCarb-101 Cone 5B2; Warp, Graphite Mandrel

The loss in weight and the dimensional change were significant during the postcure cycle. A loss of residual volatiles, decomposition of resin, and stress relief occur during the postcure. The range for percent weight loss during postcure was approximately 5 to 9 percent.

A range of this magnitude was not anticipated because of the identical raw material and processing steps. On the basis of thermogravimetric analysis of USP 39 resin, a weight loss of less than 2 percent excluding volatiles would be expected at 500°F. A volatile content of approximately 2 percent would be expected; therefore, a total weight loss of approximately 4 to 5 percent would be normal. It appears that the USP 39 resin decomposes during extended postcure at 500°F in air and/or the thermoplastic constituent is evolved.

The zero Barcol readings on the surface of the flat plates after postcure at 500°F is indicative of severe resin degradation. A study of the gases evolved and of the weight loss at various temperatures could contribute to the development of techniques or processing cycles for reducing the weight loss.

There were no observable defects as determined by radiographs after postcure.

4. Carbonization

The plates and cones were carbonized in accordance with the cycle shown in the manufacturing plan with the exception of the temperature at the opening of the furnace. The plan specifies cooling below 400°F before removing from the furnace. The exact temperature of the components is not known; however, several of the plates were burning (oxidizing). The furnace was open approximately five minutes to the ambient atmosphere. After the burning was observed, the furnace was closed immediately and the nitrogen

flow through the furnace was started again. The furnace was reopened after approximately four additional hours of cooling. The surface temperature of the components (after reopening) was approximately 400°F as measured with a surface pyrometer. Plates with observable surface oxidation were identified as:

- (1) 1702-3 SN 2
- (2) 1702-3 SN 6
- (3) 1702-3 SN 11
- (4) 1702-2 SN 1
- (5) 1702-2 SN 3
- (6) 1702-4 SN 5
- (7) 1702-4 SN 13
- (8) 1702-4 SN 14
- (9) 1702-4 SN 7

The areas of oxidation were mapped, and each component was identified. The information is included with the Certification of Conformance. Inspection and the test results after completion indicated no measurable effects on component properties as a result of the surface oxidation.

5. Graphitization and Impregnation

The final processing of the AGCarb-101 material generally includes: (1) a graphitization cycle, (2) an impregnation with a Code 88 treatment, and (3) a regraphitization cycle.

Cones were processed in this manner; however, plates received an additional impregnation and graphitization cycle. The plates were stacked and held with graphite blocks and bolts during the postcure, carbonization, and initial graphitization, impregnation, and regraphitization cycles. The total thickness of the "stack" apparently reduced the amount of Code 88 liquid that penetrated the plates. Therefore, the density was low,

approximately 1.36 gm/cc average. A second impregnation and graphitization cycle had been used previously on AGCarb-101 to increase the density without deleterious effects; and, therefore, the plates were reprocessed through these two steps. The density was increased to 1.48 gms/cc or above for all plates so reprocessed.

The time and temperature cycles for the plates and cones during graphitization were not reported in the manufacturing plan, or Certification of Conformance; however, the components were at 5000°F for a minimum of four hours during each graphitization cycle.

C. ANALYSIS OF IN-PROCESS AND FINAL COMPONENT MEASUREMENTS

There were five basic parameters measured and/or observed during the processing of the plates and cones. They were dimensional change (shrinkage), weight change, density, physical integrity, and hardness. The measurements of weight and volume were recorded after each step in the processing with the exception of the first graphitization cycle. Density was determined by using the weight and volume measurements. Individual components were checked for delamination or cracks by visual observation and radiographic inspection. One small delamination was detected in cone 5A1 bias wrapped; otherwise, the cones and plates were satisfactory. The shrinkage, weight change, and density change are discussed in the following section. The measurements taken on the cones were generally not sufficiently discriminating to permit any positive conclusions except for the lower shrinkage and less ovality obtained with the graphite mandrels.

Barcol hardness values ranged from 22 to 30 for the as-cured plates. The cause for the large variation is not known. Zero readings were recorded after postcure through and including the last graphitization cycle.

1. Shrinkage

Volatile loss, stress relief, and decomposition of the resin matrix produce thermal dilation of the fibrous-graphite composite. Shrinkage (and/or expansion) occurs at each step in the processing. Changes in dimension of the individual plates occurred after, (1) postcure, (2) carbonization, (3) graphitization-impregnation-regraphitization, and (4) re-impregnation-graphitization. Of particular significant in evaluating the data obtained was the handling of each plate for a particular process. Except for three plates (discussed later), the plates were supported during the first three steps of the process. The plates were unsupported during the last impregnation and graphitization cycles.

All of the dimensional changes were converted to in./in. for comparison and evaluation. Those measurements less than 0.001 in./in. were considered negligible and were not utilized in the evaluation. In general, the change in dimension for the warp and fill direction and 45 deg to warp and fill was negligible after the postcure and carbonization steps. However, the dimensional change perpendicular to the plies was significant for both of these steps.

The average dimensional change for the plates perpendicular to the plies after postcure, carbonization, first graphitization, impregnation, regraphitization, reimpregnation, and regraphitization is shown in Table 6.

TABLE 6

AVERAGE DIMENSIONAL CHANGE IN IN./IN. FOR PLATES (THICKNESS)

<u>Plate No.</u>	<u>Tape Lay-up</u>	<u>Approx. Thickness of Plate</u>	<u>Ave. Dimen. Change after Post Cure in./in.</u>	<u>Ave. Dimen. Change after Carbonization in./in.</u>	<u>Ave. Dimen. Change 1st Graph. Process in./in.</u>	<u>Ave. Dimen. Change 2nd Graph. Process in./in.</u>	<u>Total Shrinkage Based on Average Shrinkage</u>
1702-1	Bias	1/4 in.	-0.0102	-0.0032	-0.014	-0.049	0.0764
1702-2	Bias	1 in.	-0.010	-0.001	-0.0146	-0.0126	0.0382
1702-3	Straight	1/4 in.	-0.0125	+0.0001	-0.013	-0.0392	0.0646
1702-4	Straight	1 in.	-0.0085	+0.001	-0.0104	-0.0198	0.0386

In general, a high shrinkage would have been anticipated during carbonization on the basis of the coefficient of thermal expansion for carbonaceous-reinforced phenolics above 500°F.⁽¹⁾ However, the shrinkage occurred during the extended postcure at 500°F. The resin decomposes during a 500°F exposure; therefore, the shrinkage that results from decomposition of the resin occurred during the postcure cycle rather than the carbonization cycle.

The dimensional change during carbonization varied from -0.0032 to +0.001 in. The bias plates shrank slightly, and the straight plates expanded slightly.

A bar graph of the average shrinkage for each of the manufacturing steps after the cure cycle is shown in Figure 5. The 1/4-in.-thick blocks had the greatest total shrinkage. The majority of the shrinkage occurred during the final impregnation and graphitization cycle.

The difference in total shrinkage across the plies between the 1/4- and the 1-in.-thick plates is significant. Since it occurred for both bias and the straight tape, the thickness of the component will have to be one of the primary factors in predicting shrinkage in future components.

The results documented to-date indicate correlation between thickness and shrinkage, and configuration and shrinkage. Trends have been shown, but the data are insufficient to make predictions for sizes other than those already fabricated.

(1) Davis, H. O., Lampman, J. A., Warga, J. J., Evaluation of Characteristics Affecting Attainment of Optimum Properties of Ablative Plastics AFRPL TR 68-29 Vol. I and II, February 1968.

Figure 5 - Average Shrinkage in./in. (Thickness Perpendicular to Plies) of AGCarb-101 (Fibrous-Reinforced Graphite) Plates.

Shrinkage in the length and width direction was significantly less than in the thickness direction. Practically no shrinkage occurred during postcure and carbonization, and up to 0.005 in./in. of shrinkage occurred during graphitization. The average shrinkage (width and length) values for the four types of flat plates are shown in Table 7.

The maximum average shrinkage occurred in both the length and width for a 1/4-in. straight-tape plate, S/N 1702-3. The shrinkage for a 1/4-in. bias-taped plate, S/N 1702-1 was almost equivalent to that recorded for the 1702-3 plate. In general, the differences in shrinkage were small and there appears to be no significant differences in shrinkage between the bias and the straight-taped plates.

The additional shrinkage (length and width) which occurred during the reimpregnation and last graphitization cycle was equivalent to that which occurred during the first graphitization cycle. The shrinkage resulted from the removal of the support fixtures during the last cycle. (Ramifications of the apparent dimensional instability of a component graphitized with supports have not been ascertained.)

The shrinkage which occurred perpendicular to the plies during the last graphitization (see Figure 5 and Table 7) contributed to the increase in density. It is evident that greater shrinkage occurs in the unrestrained condition because of the dimensional change for all plates on the second impregnation and graphitization process.

TABLE 7

AVERAGE DIMENSIONAL CHANGE IN IN./IN. FOR PLATES
(Length and Width)

Plate No.	Direction of Fabric in Relation to Length	Thickness, in.	Length ⁽¹⁾					Width ⁽¹⁾				
			1	2	3	4	Total	1	2	3	4	Total
1702-1	Bias	1/4	-	-	0.0038	0.0046	0.0084	-	-	0.0036	0.0048	0.0084
1702-2	Bias	1	-	-	0.0033	0.003	0.0063	-	-	0.0033	0.005	0.0083
1702-3	Straight Tape	1/4	-	-	0.005	0.003	0.008	+0.0005	-	0.0048	0.0055	0.0103
1702-4	Straight Tape	1	-	-	0.0036	0.002	0.0056	-	-	0.0036	0.0043	0.0079

- (1) 1 After Postcure
 2 After Carbonization
 3 After 1st Graphitization Impregnation and Regraphitization
 4 After Reimpregnation and Regraphitization

Shrinkage for the three plates that were processed somewhat differently is shown in Table 8. The total shrinkage was approximately the same with the exception of 1702-4 SN 1. The total shrinkage across the plies was 0.0525 in./in. for plate 1702-4 SN 1 compared to an average of 0.038 in./in. for the remainder of the 1702-4 plates. The difference in processing is listed in Table 8. The primary difference between 1702-4 SN-1 and the remainder of the 1702-4 plates was the support during postcure, carbonization and graphitization. Plate 1702-4 SN 1 was not supported or restrained during any of the processing. The greater shrinkage plus other unexplained variations in the impregnation resulted in a significantly higher density for 1702-4 SN 1.

2. Weight Change During Processing

The percent weight change after carbonization and all impregnation and graphitization cycles is shown in Figure 6. The average percent weight loss for each of the four groups of plates ranged from 16.44 to 17.65 percent after carbonization. With the exception of the three plates of 1702-2, there was very little variation between plates of a specific group. The cause for the range of 16.84 to 18.69 percent for the 1702-2 plates is not known.

The average percent weight gain for the four groups of plates is also shown in Figure 6. The average varied from 17.35 to 20.79 percent. The low percent weight gain for plate 1702-3 (17.35%) is significant because the 1702-3 plates had the highest density and also the greatest amount of shrinkage. If weight gain had been equivalent to the other plates, the density would have been even higher. (It is postulated that the greater shrinkage reduced the porosity; therefore, the amount of impregnant picked up was reduced.

TABLE 8

SHRINKAGE IN./IN. FOR PLATES WITH DIFFERENT PROCESSING

<u>Plate No.</u>	<u>Thickness⁽¹⁾</u>			<u>Total</u>	<u>Length⁽¹⁾</u>			<u>Width⁽¹⁾</u>		
	<u>1</u>	<u>2</u>	<u>3</u>		<u>1</u>	<u>2</u>	<u>3</u>	<u>1</u>	<u>2</u>	<u>3</u>
1702-3 SN 1 ⁽²⁾	0.011	0.0018	0.0205	0.0495	-	-	0.022	-	-	0.020
1702-4 SN 1 ⁽²⁾⁽³⁾	0.012	-	0.0405	0.0525	-	-	0.023	-	-	0.026
1702-4 SN 7 ⁽⁴⁾	0.0125	-	0.013	0.0255	-	--	0.005	-	-	0.005

- (1) 1. After Postcure
 2. After Carbonization
 3. After Graphitization, Impregnation and Regraphitization
- (2) No support during postcure, carbonization, and graphitization.
- (3) Component received two impregnations and three graphitization cycles.
- (4) Component had support during postcure, carbonization, and graphization cycle.

- (1) Based on original weight after cure and the total weight loss during post cure and carbonization (weight after first graphitization was not recorded).
- (2) Based on weight after carbonization and represents weight pick-up for both impregnation steps.

Figure 6 - Average Percent Weight Gain and Loss and Average Density for Each Type of Plate of AGCarb-101

The percentage weight difference (starting vs final) was also lower for the 1702-3 plates. The percentage difference for the 1702-3 plates and all other plates is shown in Figure 6. There were no definite patterns established between weight loss and shrinkage. The weight loss during the initial graphitization was not recorded for any of the plates.

The weight change for the cones has not been evaluated because the weight of the graphite mandrels was not obtained.

3. Density

The average density for the plates is shown in Figure 6. Average values ranged from 1.485 to 1.505. The 1702-3 plates had the 1.505 average density; however, the range was also greater for the 1702-3 plates. Values ranged from 1.48 to 1.53. Range for the other three groups was a minimum of 1.48 and maximum of 1.49.

Three separately processed plates (see Table 8) had the following density variation:

<u>Plate No.</u>	<u>Density gr/cc</u>
1702-3 SN 1	1.44
1702-4 SN 1	1.585
1702-4 SN 7	1.365

Plate 1702-3 SN 1 and 1702-4 SN 7 received only one impregnation; therefore, the density would be expected to be lower. The low density for 1702-4 SN 7 is attributed to the stacking and support of the plates during the first impregnation. The stacked plates reduced the surface area in contact with the pitch during impregnation. The length of the path that the pitch had to move was also greater because most of the

impregnant had to enter the sides of the plates. The 1.365 value represents the approximate density for the remainder of the plates after the first graphitization, impregnation, and regraphitization. Since the density was below the 1.40 minimum, the plates were reprocessed.

The high density for 1702-4 SN 1 (1.585 gr/cc) is a result of high shrinkage across the plies and the two impregnation cycles. The variation in shrinkage indicates that one side of the plate was under pressure during one or more of the graphitization cycles.

IV. PHYSICAL AND MECHANICAL PROPERTIES

A. GENERAL

All plates and cones were serialized as noted in Table 2. Because the plates are limited in size, plates of the two thickness were treated, for analysis purposes, as two homogeneous lots where identical processing steps were used in their fabrication. Plates processed with the cones were separated and not included in this population (except for tensile test results where the F and T tests permitted data inclusion). A test-specimen layout was made for each plate, and layouts are contained in Appendix A of this report. A similar procedure was used for the cones.

The test procedures were those that are commonly used for laminated-plastic composites. The procedures are described in Section IV,B, Plates, and were not, therefore, repeated in Section IV,C, Cones.

B. PLATES

1. Tensile Strength

a. Test Procedure

Two types of tensile specimens, a flat and a round, were initially tested to determine the effect of specimen size on strength; Specimen configurations are shown in Figures 7 and 8. Specimens were tested in a standard Instron tester at a strain rate of 0.05 in./min. Universal joints in the load train of the Instron assured self-alignment of the specimen during testing.

A compensating clip-on extensometer was used in all room-temperature tests to obtain a stress-strain curve.

Figure 7 - Flat Tensile Specimen, 1-in.-thick Plate

Figure 8 - Round Tensile Specimen

In addition to tensile-strength design data, tensile data were also used to determine the variation among (1) plates of identical size, (2) plates of different sizes, and (3) plates processed in a different manner.

b. Test Results

Test results of all room-temperature tensile tests are listed in Tables 9, 10, and 11. These data were analyzed to determine the effects of (1) processing, (2) plate thickness, (3) specimen size, and (4) density.

(1) Effect of Processing

Tensile data from two plates of the same thickness for each of the thicknesses were compared using the F and T tests. These tests determine whether there are any differences in their (1) variability (F test) and (2) average strength (T test). Round-tensile-specimen data were used. Statistical tests at a confidence level of 0.95 indicate that there were no differences in variability and average strength between two plates of a given size. On this basis, all plates of a given size processed at the same time were considered a homogeneous batch.

(2) Effect of Plate Size

The tensile data from the 1/4- and 1-in.-thick plates were compared using the F and T tests. The means and variation for each size are as follows:

TABLE 9

ROOM-TEMPERATURE TENSILE TEST RESULTS OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

(Flat specimens machined from a 1-in.-thick plate
with warp parallel to tensile axis)

<u>Specimen Code</u>		<u>Tensile Test No.</u>	<u>Tensile Strength, psi</u>
<u>Dash No.</u>	<u>Plate S/N</u>		
-4	3	1	11,700
	3	2	11,500
	3	3	12,400
	3	4	11,200
	3	5	11,800
	2	6	11,160
	2	7	11,155
	2	8	10,710
	2	9	11,800
	3	10	10,200
	4	11	10,400
	5	12	11,900
	6	13	11,700
	8	14	11,600
	9	15	10,900
	10	16	12,200
	11	17	11,100
	12	18	10,100
	13	19	10,300
	14	20	11,500

$\bar{x} = 11,266 \text{ psi}$

$s = 665 \text{ psi}$

TABLE 10

ROOM-TEMPERATURE TENSILE TEST RESULTS OF A FIBROUS-REINFORCED-
GRAPHITE COMPOSITE, AGCarb-101

(Round Specimens machined from 1-in.-thick plate with
warp parallel to tensile axis)

<u>Specimen Code</u>		<u>Tensile Test No.</u>	<u>Tensile Strength, psi</u>	<u>Specimen Code</u>		<u>Tensile Test No.</u>	<u>Tensile Strength, psi</u>
<u>Dash No.</u>	<u>Plate S/N</u>			<u>Dash No.</u>	<u>Plate S/N</u>		
-4	2	1	9,900	-4	14	22	9,930
		2	10,200			23	10,500
		3	9,700			24	10,030
		4	9,700			25	11,330
		5	10,300			26	10,460
		6	10,200			27	10,800
		7	10,400			28	10,100
		8	10,100			29	11,200
		9	10,200			30	10,070
		10	11,100			31	9,170
		11	10,400			32	11,070
		12	12,100			33	12,500
		13	9,900			34	10,310
		14	11,400			35	10,520
		15	11,600			36	10,000
		16	10,900			37	10,450
		17	9,100			38	10,800
		18	9,000		7	39	9,600
	1	19	11,000		7	40	11,100
	1	20	10,200		7	41	11,400
	1	21	9,600				

$$\bar{x} = 10,450 \text{ psi}$$

$$s = 765 \text{ psi}$$

TABLE 11

ROOM-TEMPERATURE TENSILE TEST RESULTS OF A FIBROUS-REINFORCED-
GRAPHITE COMPOSITE, AGCarb-101(Round specimen machined from 1/4-in.-thick plate with
warp parallel to tensile axis)

Specimen Code		Tensile Test No.	Tensile Strength, psi	Specimen Code		Tensile Test No.	Tensile Strength, psi
Dash No.	Plate S/N			Dash No.	Plate S/N		
-3	2	1	11,900	-3	3	23	13,400
		2	10,800			24	9,400
		3	12,100			25	11,600
		4	11,300			26	11,100
		5	11,800			27	10,800
		6	10,600			28	11,400
		7	12,200			29	11,830
		8	11,400			30	12,100
		9	10,900			31	9,600
		10	10,700			32	12,500
		11	9,700			33	10,600
		12	10,300			34	10,800
		13	11,300			35	12,100
		14	8,100			36	11,300
		15	11,900			37	11,400
		16	10,200			38	10,900
		17	10,100			39	9,700
	2	18	11,200		10	40	10,300
	3	19	10,500				
		20	11,300				
		21	12,700				
		22	13,800				
-3	3						

 $\bar{x} = 11,140 \text{ psi}$ $s = 1,090 \text{ psi}$

<u>Plate Thickness, in.</u>	<u>$\bar{X}$, psi</u>	<u>S, psi</u>
1/4	11,140	1,090
1	10,450	765

At a confidence level of 0.95, the F and T tests show that there is a significant difference between the 1/4- and 1-in.-thick plates. Sufficient background on the thickness-processing relation is not available to establish the cause for the difference between the two plate sizes; however, this may be related to the greater shrinkage of the 1/4-in. plate in the "perpendicular to the plies" direction.

(3) Effect of Specimen Sizes

The tensile data from round and flat specimens machined from 1-in.-thick plates are listed in Tables 9 and 10. The means and deviations are as follows:

Round Specimen	$\bar{X} = 10,450$ psi	$S = 765$ psi
Flat Specimen	$\bar{X} = 11,270$ psi	$S = 665$ psi

The F tests at a confidence level of 0.95 indicate that there is no statistical difference in their variability. The T tests, however, at the same confidence level, indicate that there is a statistical difference in their average strength. Therefore, the data from any one plate size will show the same amount of scatter but at a different average-strength level which may be attributed to specimen size. This may be due to a difference in stress distribution in the specimen because of the difference in radius between the shoulders and gage length or to the method of gripping the specimen. A controlled experiment would be required to establish the exact cause for this observed variation in strength.

Despite these findings, the round specimens were used for all remaining tensile tests primarily to conserve test material.

(4) Effect of Density

Plates No. 1 and 7 (1-in. thickness) and plate No. 1 (1/4-in. thickness) were inadvertently processed in a different manner. The outstanding difference in the plates was their density which was at the two extremes of the density spectrum of fibrous-reinforced graphites. Tensile specimens (three each) were machined from a plate of high density (1.59 gm/cc) and from a plate of low density (1.36 gm/cc). The results are shown in Table 10. The mean and deviations are as follows:

<u>Plate No.</u>	<u>Density</u>	<u>$\bar{X}$</u>	<u>S</u>
1	1.59 gm/cc	10,267	645
7	1.36 gm/cc	10,700	950

The F and T tests at the 0.95 confidence level indicate that there is no significant difference in the average strengths and variability of the two plates. If density were the only difference between the two plates, it may be concluded that density has no effect on the tensile properties. However, the plates were not processed at the same time, and density is not the only variable to be considered. Further, although no statistical differences are noted in the tensile strength, differences would be noted in other properties such as thermal expansion, thermal conductivity, fatigue, and modulus of elasticity.

The microstructures of a low-, intermediate-, and high-density plate are shown in Figure 9. The effects of density are readily apparent; the size and frequency of voids decrease as density increases.

Plate
Serial No. 7
Density 1.36 gm/cc

Plate
Serial No. 8
Density 1.48 gm/cc

Plate
Serial No. 1
Density 1.59 gm/cc

*Warp direction in plane of paper

Magnification, 100X

Figure 9 - Microstructure* of a Fibrous-Graphite-Reinforced Composite
(AGCarb-101 Showing Density Effects)

2. Elevated-Temperature Tensile Strength

a. Test Procedure

The test equipment and procedure were the same as for room temperature with the modifications described here. The specimens were direct resistance heated in an enclosure containing argon. A forepump was used to obtain a vacuum of less than 1 psi and then the enclosure was back filled with argon. A positive pressure of argon was maintained at a flow rate of approximately 5 cu-ft/hr until test completion. Temperature was measured with an L & N optical pyrometer. About one minute was required to stabilize the temperature.

An optical extensometer (Optron Model 800) was used to obtain stress-strain data. The Optron was sighted on flat fiducial markers made of fibrous graphite attached to the specimen near the shoulders. The output of the Optron and the tensile tester was fed into an X-Y recorder to obtain stress-strain curves.

Specimen location is shown in the plate layout, Appendix A. For all elevated temperature tests, the round specimen was used.

b. Test Results

The results of the tensile tests at 2000 and 3000°F are shown in Table 12. The mean and the deviation of the samples tested are as follows:

TABLE 12

ELEVATED-TEMPERATURE TENSILE STRENGTH
OF A FIBROUS-REINFORCED-GRAPHITE COMPOSITE,

<u>Thickness Plate, in.</u>	<u>Specimen No.</u>	<u>Test Temp, °F</u>	<u>Warp Direction Tensile Strength, psi</u>	<u>Remarks</u>
1/4	1	2000	13,300	
1/4	2		12,900	
1/4	3		12,100	
1/4	4		11,700	
1/4	5		12,700	
1/4	6		12,200	
1/4	7		10,400	
1/4	8		11,200	
1/4	9		12,000	
1/4	10	2000	11,100	
1/4	11	3000	12,900	
1/4	12		11,900	
1/4	13		12,900	
1/4	14		14,100	
1/4	15		15,500	
1/4	16		13,100	
1/4	17		14,600	
1/4	18		14,100	
1/4	19		14,600	
1/4	20		14,300	
1	21	2000	7,665	No Protective atmosphere
	22		11,115	
	23		12,862	
1	24		11,394	

TABLE 12. (cont.)

Thickness Plate, in.	Specimen No.	Test Temp, °F	Warp Direction Tensile Strength , psi	Remarks
1	25	2000	11,972	
	26		11,620	
	27		11,324	
	28		10,033	
	29		10,836	
	30	2000	12,606	
	31	3000	13,690	
	32	13,659		
	33	11,972		
	34	12,100		
	35	12,500		
	36	*	Not tested	
	37	*		
	38	*		
	39	*		
	40	*		

<u>Test Brightness Temp., °F</u>	<u>Block Thickness, in.</u>	<u>$\bar{x}$</u>	<u>s psi</u>
2000	1/4	11,750	890
	1	11,529	798
3000	1/4	13,467	1,070
	1	12,782	840

The results show rising strength with increasing temperature which is characteristic of graphite. As in the room-temperature tests, the specimens cut from the 1/4-in.-thick plate have the highest average strength and the greatest variability.

The attachment of graphite-composite fiduciary markers presented a problem. The markers acted as a heat sink drawing heat away from the gage-length portion of the specimen. Because of the rising trend in strength with temperature, failure occurred near the fiducial markers where the temperature was lower. Measurement indicated that the temperature was a maximum of 150°F lower. The temperature used during testing was the brightness temperature. A correction for emissivity (assumed to be 0.9 for the composite) raised the test brightness temperatures of 2000 and 3000°F to true temperatures 2015 and 3032°F, respectively. Correction raised the temperature of the entire specimen including the value at the point of failure. A small, positive slope of the strength-temperature line indicates that a relatively small error is introduced. This error will be on the conservative side and will be well within the limits of deviation noted for the elevated-temperature tests. For future tests, if direct resistance heating is to be used, the specimen should be redesigned to include a longer gage length and sharper transition between the shoulders and gage length. Insulating materials, such as reinforced pyrolytic graphite, should be used for fiduciary markers.

The tensile strength of the blocks is plotted as a function of temperature in Figure 10.

Tensile moduli calculated from the stress-strain curves are contained in Table 13. Only the flat-specimen room-temperature data are reported.

Stress-strain curves plotted using the Optron optical extensometer were very erratic and much of the data are not included. Tests at 3000°F produced curves with some degree of reproducibility from which tensile moduli could be calculated, and the data are also contained in Table 13.

3. Block Tensile Strength

a. Test Procedure

Block tensile is a measure of the tensile strength of the composite in the perpendicular to ply direction. The limited thickness of the plates precluded machining of tensile specimens. A special test specimen is fabricated by adhesive bonding extensions to the cylindrical section coupons shown in Figures 11 and 12. A strain rate of 0.05 in./minute^{*} is used. Because of adhesive limitations, only room-temperature tests are made.

b. Test Results

Block-tensile test results are listed in Table 14. The mean and deviation of the data are as follows:

<u>Plate Thickness, in.</u>	<u>$\bar{x}$, psi</u>	<u>s, psi</u>
1/4	638	64
1	644	130

* Strain Rate measured by Crosshead Speed - No extensionmeter

Figure 10 - Tensile Strength of a Fibrous-Reinforced-Graphite Composite, AGCarb-101

TABLE 13

TENSILE MODULUS OF A
FIBROUS-REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

			1-in.-Thick Plate	
<u>Extensometer Type</u>	<u>Specimen Type</u>	<u>Specimen No.</u>	<u>Test Temp.</u> <u>°F</u>	<u>Tensile Modulus</u> <u>psi x 10⁶</u>
Clip-on	Flat	1	75	2.10
		2		2.05
		3		2.04
		4		2.12
		5		2.10
		6		2.18
		7		2.22
		8		2.33
		9		2.62
		10		2.46
		11		2.37
		12		2.39
		13		2.22
		14		2.39
		15		2.29
		16		2.68
		17		2.47
Clip-on	Flat	18	75	2.52
		$\bar{X} = 2.31 \times 10^6 \text{ psi}$ $S = 0.18 \times 10^6 \text{ psi}$		
Optron 800	Round	1	3000	2.26
		2		1.92
		3		1.62
		4		1.59
Optron 800	Round	5	3000	1.29
		$\bar{X} = 2.31 \times 10^6 \text{ psi}$ $S = 0.34 \times 10^6 \text{ psi}$

Figure 11 - Block Tensile Coupon, 0.250-in.-thick plate

Figure 12 - Block Tensile Coupon, 1-in.-thick plate

TABLE 14

BLOCK TENSILE STRENGTH OF A FIBROUS-REINFORCED-GRAPHITE COMPOSITE,
AGCarb-101

<u>Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Block Tensile Strength, psi</u>	
1/4	1	714	
	2	619	
	3	647	
	4	716	
	5	547	
	6	620	
	7	621	
	8	524	
	9	635	
1/4	10	689	
		$\bar{x} = 638 \text{ psi}$	$s = 64 \text{ psi}$
1	1	707	
	2	807	
	3	732	
	4	475	
	5	520	
	6	682	
	7	723	
	8	699	
	9	403	
1	10	692	
		$\bar{x} = 644 \text{ psi}$	$s = 130 \text{ psi}$

Although the block tensile strengths are about equal, the 1-inch plate has the biggest variability.

The low "through-the-ply" strength of the cloth reinforcement is the major weakness of the bidirectional composites. This strength probably can be increased by use of other resins or cloth weaves; however, the perpendicular to the ply strength will not approach the strength in the warp or fill direction. Where strengths are needed in the three principal directions, three dimensional reinforcements are required.

4. Fill Tensile Strength

a. Test Procedure

Except for specimen orientation, the procedures and specimen configuration for determination of fill tensile strength are the same as for the warp tensile strength.

b. Test Results

Fill direction tensile strength results are shown in Table 15. The mean is 4,563 psi and deviation is 325 psi.

The lower strength in the fill direction as compared to the warp direction may be attributed to the lower number of yarns/inch in the fill direction (26.9, warp, 22.7, fill) of the cloth reinforcement and consequently lower tensile strength (warp, 27 lb/in.; fill, 24 lb/in.). Because of the larger number of yarns, the amplitude of the crossover of fill over the warp is greater. The large amplitude of crossover results in a greater departure of the fill yarns from a parallel orientation with the tensile axis. This is readily apparent in the macro and microstructures taken in these two

TABLE 15

ROOM-TEMPERATURE FILL DIRECTION TENSILE STRENGTH OF A
FIBROUS -REINFORCED -GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness,</u> <u>in.</u>	<u>Specimen No.</u>	<u>Tensile</u> <u>Strength, psi</u>
1	1	4,000
	2	4,500
	3	4,870
	4	5,020
	5	4,375
	6	4,570
	7	4,435
	8	4,755
$\bar{x} = 4,563 \text{ psi}$		$s = 325 \text{ psi}$

directions, which are shown in Figures 13 and 14. To obtain equal strengths in both directions in a flat laminate, the prepreg must be indexed 90 deg.

5. Bias Tensile Strength

a. Test Procedure

Except for reinforcement orientation, the test procedure for determination of the bias tensile strength is identical to that described for the warp tensile strength. Specimens were machined from a laminate made with the 45-deg-bias-cut cloth and the warp direction in the specimen is 45 deg to the longitudinal axis of the specimen.

b. Test Results

Bias tensile-strength results are listed in Table 16. The mean and deviation are as follows:

<u>Block Thickness, in.</u>	<u>$\bar{x}$, psi</u>	<u>s, psi</u>
1/4	4,015	158
1	4,432	144

The bias tensile can be normalized to a 0 deg orientation by the equation:

$$\sigma_{45^\circ} = \sigma_{0^\circ} \cos^2 \theta$$

where

σ_{45° = tensile strength at 45° orientation

σ_{0° = tensile at 0° orientation

θ = angle of principal fiber to tensile

Warp*

Fill*

Magnification, 6X

* Views indicated are in plane of paper.

Figure 13 - Macrostructure of a Fibrous-Reinforced-Graphite Composite,
AGCarb-101

Warp*

Fill*

Magnification, 100X

* Views indicated are in plane of paper.

Figure 14 - Microstructure* of a Fibrous-Reinforced-Graphite Composite (AGCarb-101) Illustrating Difference of Orientation

TABLE 16

TENSILE STRENGTH OF BIAS CUT (45 DEG) FIBROUS-REINFORCED-
GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness,</u> <u>in.</u>	<u>Specimen No.</u>	<u>Tensile</u> <u>Strength, psi</u>	
1/4	1	4170	
	2	3780	
	3	4150	
	4	3990	
1/4	5	3970	
		$\bar{x} = 4015 \text{ psi}$	$s = 158 \text{ psi}$
1	6	4520	
	7	4230	
	8	4550	
	9	4530	
1	10	4330	
		$\bar{x} = 4432 \text{ psi}$	$s = 144 \text{ psi}$

The calculated strengths are 8030 and 8864 psi, respectively for 1/4- and 1-in. plates. This is significantly lower than the warp tensile strength obtained in a test where the warp is oriented parallel to the tensile axis indicating that some deterioration of strength by factors other than bias.

The fracture faces of the bias tensile specimens were generally at about an angle of 45 deg to the tensile axis.

6. Interlaminar Shear Strength

a. Test Procedure

The test specimen configuration is shown in Figures 15 and 16.

The test procedure is essentially the same as for tensile strength. A strain rate* of 0.05 in./min was used for all tests. Only room-temperature tests were performed.

b. Test Results

Results of the interlaminar shear tests are listed in Table 17. The mean and deviation of the results are as follows:

<u>Block Thickness, in.</u>	<u>$\bar{x}$, psi</u>	<u>s, psi</u>
1/4	1,300	67
1	1,350	106

Although the interlaminar shear strength of the 1-in.-thick plates is greater, its variability is also greater.

* Strain Rate measured by Crosshead Speed no extensometer

Figure 15 - Interlaminar Shear Specimen, 0.250-in.-thick plate

Figure 16 - Interlaminar Shear Specimen, 1-in.-thick plate

TABLE 17

INTERLAMINAR SHEAR STRENGTH OF A FIBROUS-GRAPHITE-REINFORCED
COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Interlaminar Shear Strength, psi</u>
1/4	1	1295
	2	1203
	3	1208
	4	1286
	5	1271
	6	1422
	7	1375
	8	1273
	9	1322
	10	1320
		$\bar{x} = 1298 \text{ psi} \quad s = 67 \text{ psi}$
1	1	1256
	2	1216
	3	1304
	4	1306
	5	1375
	6	1215
	7	1523
	8	1423
	9	1472
	10	1394
		$\bar{x} = 1348 \text{ psi} \quad s = 160 \text{ psi}$

The interlaminar shear strength is another measure of the bonding between plies. The strength between plies is due solely to the carbon-carbon bond formed during carbonization and graphitization. Increases in interlaminar shear strength may be achieved by use of other resins or weaves, but a major increase can only be obtained by the use of three-dimensional reinforcements.

7. Compression Strength

a. Test Procedure

The specimen configuration used for the compression test is shown in Figure 17. The warp fiber direction was parallel to the longitudinal axis of specimen. A compression tool that consisted of heavy, steel plates (ground flat and parallel) was used for testing. The method of attachment of the load ram to the specimen maintained the load perpendicular to the bases of the specimens, which were also ground flat and parallel.

For elevated-temperature tests, a hard-carbon load ram and base plate were used. Specimens were radiant heated in a graphite-tube furnace with power being supplied to the furnace by a saturable core reactor controlled by a 31-kw ac transformer. Specimen temperatures were measured with an L&N optical pyrometer. A deflectionometer was used to monitor crosshead movement. Helium was used as a protective atmosphere.

b. Test Results

(1) The results for the 1/4- and 1-in.-thick plates are listed in Tables 18 and 19, respectively. The mean and deviation of the compression test are as follows:

Figure 17 - Compression Specimen

TABLE 18

COMPRESSIVE STRENGTH OF A FIBROUS-REINFORCED-GRAPHITE
COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Test Temp, °F</u>	<u>Compressive Strength, psi</u>
1/4	1	RT	9030
	2		10430
	3		10510
	4		10600
	5		9640
	6		9820
	7		6272*
	8		9269
	9		8328
	10		9027
			RT tests $\bar{x}$ = 9506 psi
			s = 702 psi
	11	2000	8990
	12		9820
	13		7670
	14		8600
	15		9000
	16		8740
	17		9690
	18		8845
	19		9710
	20		10520
			2000°F tests $\bar{x}$ = 9159 psi
			s = 477 psi
1/4	21	3000	6830*
	22		7350*
	23		3890*
	24		9950
	25		8350
	26		8300
	27		8640*
	28		9200
	29		9540
	30		9100
			3000°F tests $\bar{x}$ = 9073 psi
			s = 650 psi

*Tested before Instron recalibrated; data not included in population.

TABLE 19

COMPRESSIVE STRENGTH OF A FIBROUS-REINFORCED-GRAPHITE
COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Test Temp, °F</u>	<u>Compressive Strength, psi</u>
1	1	RT	9686
	2		7726
	3		9812
	4		9279
	5		9843
	6		9498
	7		9404
	8		10,500
	9		8527
	10		9718
		RT tests $\bar{x}$ = 9400 psi	
		s = 760 psi	
	11	2000	7743
	12		2973 (machining defect)
	13		8790
	14		9045
	15		8352
	16		7742
	17		8952
	18		7760
	19		10,252
	20		9858
		2000°F tests $\bar{x}$ = 8722 psi	
		s = 921 psi	
	21	3000	9180
	22		9000
	23		9900
	24		8100
	25		9100
	26		8200
	27		9500
	28		9800
	29		9100
	30		9370
		3000°F tests $\bar{x}$ = 9125 psi	
		s = 594 psi	

<u>Plate Thickness,</u> <u>in.</u>	<u>Test</u> <u>Temperature, °F</u>	<u>$\bar{x}$, psi</u>	<u>s, psi</u>
1	RT	9,400	760
	2000	8,720	920
	3000	9,125	594
1/4	RT	9,500	700
	2000	9,160	477
	3000	9,073	650

Examination of these data shows two anomalies: (1) compressive strength lower than tensile strength and (2) decreasing strength with increasing temperature. The lower compressive-strength values are attributed to orientation during test. With the warp parallel to the compression axis, the fibrous-reinforced graphites have lower compressive strengths than tensile strength; with the compression axis perpendicular to the plies, the compressive strengths are normally higher. All compression test specimens in this program were tested with the compression axis parallel to the ply direction. Failure normally occurred by ply separation and then fracture. Additional tests are required to determine the compressive strength in the two directions: parallel to fill and perpendicular to plies. It is anticipated that the parallel to fill orientation will be the weakest in compression as postulated from tensile-strength data.

(2) The elevated-temperature compression tests were conducted with a different test setup, and the lower values are attributed primarily to test setup differences. For most graphites, the strength rise from room temperature to 3000°F is small as shown by the tensile-strength data. Therefore, use of the room-temperature value of compressive strength for all temperatures in this range will introduce little error, and the data would be on the conservative side.

8. Fatigue Life

a. Test Procedure

Fatigue tests were conducted on a Tatnall-Krouse rotating-beam fatigue-testing machine. All tests were conducted at 5000 rpm (except where indicated) using specimen configurations shown in Figures 18 and 19.

The stress to determine the load to be applied is calculated by using the following formula.

$$S = \frac{P}{0.01227 d^3}$$

where:

S = stress

P = load

d = diameter of specimen

b. Test Results

The rotating-beam fatigue results are listed in Table 20. The scatter in results is partially attributed to the sensitivity of the balance of the machine. Because of the low modulus and high stress, the deflection in the test setup is large. This combined with high-speed rotation makes the specimen very sensitive to any factor which would tend to unbalance the machine. If the machine is unbalanced, failure of the specimen occurs.

Observation of the specimen at or near the failure point indicated that the failure originated on the compression side of the specimen.

NOTES:

- 1 Diameters .125 and .250 to be concentric within .001 inch.
- 2 No tool marks or circumferential marks in .750 inch gage length.
- 3 Finish with 3/0 EMERY PAPER in longitudinal direction.
- 4 Surface finish - 16 RMS max over .750 inch gage length.

Figure 18 - Rotating-Beam Fatigue Specimen; 0 .250-in.-thick plate, Code FT, SK521

NOTE:

- 1 Diameters 0.250 and 0.500
to be concentric within 0.001 inch.
- 2 No tool marks or circumferential
marks in reduced gage length.
- 3 Finish with 3/0 Emery paper in
longitudinal direction.
- 4 Finish 16 RMS max over gage length.

Figure 19 - Rotating-Beam Fatigue Specimen; 1-in. thick plate, Code FRB, SK417

TABLE 20

ROTATING-BEAM FATIGUE
TEST RESULTS OF A FIBROUS-REINFORCED-
GRAPHITE COMPOSITE, AGCarb-101

<u>Test Plate Thickness , in.</u>	<u>Specimen No.</u>	<u>Stress at Outer Fiber, psi</u>	<u>Cycles to Failure</u>	<u>Remarks</u>
1/4	1	9,000	-	Setup Failure
	2	9,000	-	Setup Failure
	3	9,500	2.7×10^7	Test stopped
	4	9,600	1.3×10^7	Test stopped
	5	9,700	3.06×10^7	Test stopped
	6	10,000	60,000	
	7	9,700	-	Setup Failure
	8	9,700	6.1×10^6	
	9	9,700	6.4×10^6	
	10	-	-	Specimen out-of-round
	11	10,000	450,000	
	12	10,000	853,000	
	13	9,500	5.0×10^7	Test stopped
	14	-	-	Not tested
	15	-	-	Not tested
	16	-	-	Not tested
1	1	5,000	4.86×10^7	Test stopped
	2	9,000	72,000	Test ran at 8000 rpm to determine max. rotational speed.
	3	9,500	20,000	
	4	9,500	950,000	
	5	9,500	42,900	
	6	9,250	-	Setup Failure
1	7	9,250	136,000	

TABLE 20 (cont.)

<u>Test Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Stress at Outer Fiber, psi</u>	<u>Cycles to Failure</u>	<u>Remarks</u>
1	8	9,250	-	Setup Failure
	9	9,000	7.73×10^6	
	10	8,750	1.3×10^7	
	11	8,750	391,800	
	12	9,700	10,000	
	13	9,000	1.9×10^7	Test stopped
	14	9,000	286,000	
	15	9,000	2,100	Considered test setup failure
	16	9,000	4.7×10^6	

A slight lump (probably delamination) appeared in the top center of the specimen. When this protuberance appeared, the machine would soon go out of balance and lead to specimen failure.

The microstructure of the failed specimen shown in Figure 20 indicates that the crack ran between and through the plies. Because this specimen did not fail completely (test stopped before complete fracture occurred), it may be assumed that this type of failure is typical in fatigue. Failure due solely to crack propagation between plies, as reported by other investigators, was not noted here.

9. Fracture Toughness

a. Test Procedure

Fracture toughness was measured using the WOL specimen shown in Figure 21. Specimens were machined with the notch perpendicular to the plies in the warp and fill directions. To sharpen the notch, a razor-blade cut was made at the root. The specimens were tensile-tensile fatigued on the Materials Research test machine. Crack initiation and growth were observed visually. The specimen was cycled at a load for a period of 20 minutes at 300 cycles/minute, and the load was increased every 20 minutes until a crack was initiated.

b. Test Results

The fracture-toughness results obtained by the above techniques are shown in Table 21. The data are nonconservative as initial crack length was based on measurements on the surface. Surface measurements of the fatigue-crack length are inaccurate as generally the crack is shortest at this location. Accurate measurements of the fatigue-crack length can be

Fracture Near Outer Surface

Fracture Near Center
Showing Displacement of Fracture Line

Figure 20 - Microstructure of Fatigue Failure of AGCarb-101 Specimen

Notch perpendicular to warp or fill

Figure 21 - WOL Fracture-Toughness Specimen

TABLE 21

FRACTURE TOUGHNESS OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>Specimen No.</u> <u>CH 1</u>	<u>Failure Load,</u> <u>pounds</u>	<u>Notch Direction</u> <u>Normal to</u>	<u>a_o in.**</u>	<u>K_{Ic}</u> <u>ksi - in^{1/2}</u>
1	550	Warp	1.05	8.5
2	510	Warp	1.10	8.5
3	90*	Warp	1.20	1.7
4	415	Fill	1.05	6.4
5	415	Fill	1.09	6.7

* Defective specimen

** Initial notch plus crack depth measured from center
line of load pin hole to bottom of fatigue induced crack.

made by using the crack-opening-displacement gage. Calibration curves would be required from which the crack length could be determined. The calibration curve would be valid for all WOL specimens providing specimen dimensions are proportional to that of the specimen used to develop the curve. Such a calibration curve was not made as the optimum method for determination of fracture toughness in the three orthogonal axes has not been developed. The WOL specimens must be modified for measurement of fracture toughness between plies which is the most critical orientation. No fibers are present to arrest propagation of any crack and intuitively it is surmised that the between-ply fracture toughness would be extremely low.

The values listed in Table 21 indicate that fracture toughness of AGCarb-101 in the two directions tested is good by using the criterion that the ratio of fracture toughness to tensile strength should approach unity.

10. Flexure Strength

a. Test Procedure

The flexure-strength specimens are shown in Figures 22 and 23. Flexural strength was measured at ambient and elevated temperatures by single-point loading using two setups. For room-temperature tests, a polished metal pin 1/8-in. dia was used in the load ram. The fixture utilized two 0.25-in.-dia polished metal rollers as the span support. The specimens and the fixture were positioned to assure normal loading on the axial centerline. Two spans were used: 1.75 and 3.20 in. Because the elevated-temperature test furnace could only accommodate a 1.75-in. test specimen, the span-depth ratio was 7, whereas the preferred ratio is $16 \pm \frac{4}{2}$. The larger span had a span-depth ratio of 14.75.

For all elevated-temperature flexural tests, the span was 1.75 inches. The specimens were radiant heated in a resistance-heated cylindrical-graphite-tube furnace. The test fixture is positioned on a water-cooled copper ram that transmits the load to a compression load cell. A bushing in the upper lid of the furnace provides added alignment to the hard-carbon load ram containing a 1/8-in.-dia hard-carbon roller. The specimen is positioned in the load train on two 0.25-in.-dia polished hard-carbon rollers.

Temperature was monitored with an L&N disappearing-filament optical pyrometer. All temperatures reported are brightness temperatures. Test specimens were tested in both the perpendicular and the parallel to ply orientations.

SK 428

Figure 22 - Flexure Specimen, 1.75-in. Span

SK 572

Figure 23 - Flexure Specimen, 3.2-in. Span

b. Test Results

Test results at room- and elevated-temperature flexural tests are listed in Table 22.

The mean and the variation for all flexure tests are as follows:

Specimen Orientation	Test Temp., °F	$\bar{x}$ psi	s psi			
Plate Thickness Parallel to Ply	RT	1/4 in. 19,980	1 in. 17,514	1/4 in. 626	1 in. 597	
	3000	23,485	19,950	1,780	1,908	
Perpendicular to Ply	RT	17,595	16,254	1,006	900	
	3000	20,630	19,650	895	640	

The results show the rising trend in strength with temperature typical of graphites. The parallel to ply orientation consistently showed higher strengths as expected. The large deviations noted are attributed to the small number of samples; some data had to be discarded because of test-machine malfunction.

The data from both span-to-depth ratios were included in the above tabulation even though the test indicated that there is a difference in average strength. Again, the low number of samples tested may have influenced the results.

TABLE 22

FLEXURAL STRENGTH OF A FIBROUS-REINFORCED-
GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Specimen No.</u>	<u>Specimen Orientation</u>	<u>Test Temp, °F</u>	<u>Flexure Strength, psi</u>	<u>Remarks</u>
1/4	1	Parallel to Ply	RT	20,526	Block 7
	2			20,000	Block 7
	3			20,385	Block 7
	4			20,513	Block 7
	5			20,385	3.20 span
	6			20,220	
	7			20,076	
	8			20,124	
	9			18,600	
	10			18,964	
	11	Perpendicular to Ply	3000	19,048	
	12			17,425	
	13			16,726	
	14			17,711	
	15			17,987	
	16			18,295	
	17			18,440	
	18			17,813	
	19			15,440	
	20			17,067	
21	Parallel to Ply	3000	25,487		
22			23,330		
23			21,176		

TABLE 22 (cont.)

Plate Thickness, in.	Specimen No.	Specimen Orientation	Test Temp, °F	Flexure Strength, psi	Remarks	
1/4	24	Parallel to Ply	3000	23,955		
	25			--		
	26	Perpendicular		19,872		
	27			20,796		
	28			20,159		
	29			22,000		
	30			20,332		
1	31	Parallel	RT	17,063		
32	17,997					
33	17,974					
34	17,840					
35	16,697					
36	Perpendicular	15,802				
37		15,212				
38		15,981				
39		17,530				
40		16,746				
1	41	Parallel	3000	21,300		
	42			18,600		
	43			15,300*	Test machine malfunction	
	44	15,200*				
	45	Perpendicular		18,800		
	46			20,100		
	47			20,200		
	48			19,500		

* Data suspect as subsequent tests (compressive) indicated machine went out of calibration; tests were then suspended pending repair and recalibration of machine. Data not included in mean and deviation calculation.

11. Dynamic Modulus

a. Test Procedure

Dynamic modulus was determined by using a composite piezo-electric resonator consisting of a driver crystal, a gage crystal, and the specimen. The specimen is 1/4-in. square by approximately 4-in. long. The length of the specimen is determined by matching its resonant frequency with that of the driver. For a multi-component resonator, the resonant frequency of a component is calculated using the following equations:

$$f_s = \frac{(M_t f_t - M_R f_R)}{M_s}$$

where:

M_s , M_t , and M_R = mass of the sample

total resonator assembly, and resonator assembly
minus the sample, respectively.

f_s , f_t , and f_R = the corresponding resonant frequencies.

The modulus is calculated from the following equation:

$$E_s = \rho (2l_s f_s)^2$$

where:

ρ = specimen density

l_s = specimen length

f_s = specimen resonant frequency

The sample is mounted on a sapphire rod with a small amount of Sauereisen cement. This assembly is then mounted with cement on the piezo-electric resonator. The specimen with part of the sapphire tube extends into a tungsten-wire-wound-tube furnace. An oscillator with amplifier is used to drive the crystal. A frequency counter continuously monitors frequency, and the resonant frequency is determined by peak voltage on a vacuum tube voltmeter. A vacuum of approximately 500 microns is maintained using a forepump only. Temperature is raised incrementally and is monitored using a chromel-alumel thermocouple located adjacent to the specimen. Readings are taken approximately every 100 degrees. The resonant frequency is calculated and a best-fit curve is plotted. Data from the curve are then used to calculate modulus.

b. The Results

The results of the room-temperature dynamic-modulus tests were 2.94×10^6 psi for the 1/4-in. blocks and 2.86×10^6 psi for the 1-in. plates. A spurious secondary peak was detected at temperatures above 1200°F , and the data above this temperature are suspect and are not reported. The moduli data at 1000°F are 2.96×10^6 psi for the 1/4-in. plates and 2.90×10^6 psi for the 1-in. plates.

The dynamic modulus and the tensile (static) modulus of most engineering materials differ by a small percentage. This can be attributed to the very small strains ($10^{-4}/10^{-5}$ in.) developed for very short times in the dynamic technique. The dynamic modulus is more accurate; however, for engineering purposes, the tensile modulus is used because stress-strain data are normally required. At elevated temperatures, the disparity between dynamic and tensile modulus is usually greater because of creep. Other time dependent mechanisms are not manifested to any great degree in the dynamic technique.

12. Damping Decrement

a. Test Procedure

The test setup for determination of the damping decrement is the same as for determination of dynamic modulus except for the experimental procedure and the data acquired.

The log decrement of the quartz is determined by measuring driver-gage voltage relationship. The specimen is mounted on the quartz to form a three-component assembly. The length of the 1/4-in.-sq specimen is adjusted (by sanding) until the assembly resonates at the same frequency as the quartz driver. The log decrement of the system is determined from the following equation:

$$\delta_T = \frac{A}{M_T} \frac{V_D}{V_g}$$

δ_t = system log decrement

A = a constant

M_t = total mass of the system

V_D = driver voltage

V_g = gage voltage

The specimen decrement is then obtained by the following relation:

$$\delta_s = \frac{M_T \delta_T - M_q \delta_q}{M_s}$$

δ_s = log decrement of the specimen
 M_q = mass of the quartz
 δ_q = log decrement of the quartz
 M_s = mass of the specimen

The vibrational strain is calculated by the following relation:

$\epsilon_g = B V_g$
 $\epsilon_s = V_g l_g / l_s$
 ϵ_g = gage strain
 B = a constant
 V_g = gage voltage
 ϵ_s = specimen strain
 l_g = length of gage
 l_s = length of specimen

b. Test Results

In the strain range of 10^{-5} to 10^{-4} in., AGCarb-101 has a log decrement of 4×10^{-3} to 7×10^{-3} . At these low strains, the log decrement was strain dependent. The stress in the specimens at these low strains is about 30 to 300 psi, which is not in the range of engineering interest. Therefore, a different test technique is required to obtain information regarding the damping decrement at higher strains.

The log decrement of 347 stainless steel determined by the same test technique and in the same range of strain was 2.8×10^{-4} . The specific value of the log decrement though not of engineering interest indicates in a qualitative manner that AGCarb-101 will absorb 18 to 25 times more energy than 347 stainless steel.

13. Thermal Conductivity

a. Test Procedure

Thermal diffusivity was determined up to 1500°F and thermal conductivity from RT to 3000°F. The purpose of using two methods of conductivity determinations was to obtain a cross check of the results. The diffusivity method can be used to temperatures of 4000°F; however, equipment was not available at Aerojet for the high-temperature measurements. The standard thermal gradient technique was used for measuring thermal conductivity.

Thermal diffusivity was determined by the Xenon lamp flash method using a specimen 0.108-in. thick and 0.8-in. dia. The method consists essentially of absorption of a very short pulse of radiant energy on the front surface of the specimen and the determination of the resultant thermal history of the back surface.

For a perfectly insulated specimen with a constant absorptivity across its surface, uniformly irradiated with a pulse of thermal energy (pulse short compared to the time required for heat to flow through the specimen), the back-surface temperature is given by the following equation:

$$\frac{T(L,t)}{T_M} = 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp \left(-n^2 \pi^2 \frac{\alpha t}{L^2} \right)$$

where:

$T_{(L,t)}$ = instantaneous back-surface temperature rise at
time t seconds

T_M = maximum back-surface temperature rise

α = thermal diffusivity, cm^2/sec

L = specimen thickness, cm

n = successive integers

At time $t_{1/2}$ where $T_{(L,t)} = 0.5 T_M$, the above equation reduces to

$$\alpha = \frac{0.139 L^2}{t_{1/2}}$$

Thus, the thermal diffusivity can be determined from the specimen thickness and the back-surface temperature to reach one-half of its maximum value.

Back-surface rise time is a function of specimen thickness (L). The specimen thickness, therefore, is restricted by two conditions:

(1) Backface rise time must be long compared with pulse duration.

(2) The amount of the backface temperature rise (T_M) should be high because of the limited input energy.

These two restrictions are met by using specimens one to three millimeters in thickness. For these experiments, the specimen thickness is 2.75 millimeters.

Convective and conductive heat losses were minimized by mounting the specimen on an insulator with a small area. Heat losses are usually negligible because of the short times involved by the flash technique.

The specimen is heated in a furnace having Nichrome heating elements. The furnace is purged with atomic-grade helium, and a positive pressure of helium is maintained to prevent oxidation of the sample. Specimen temperature is monitored by a chromel-alumel spring loaded against the specimen. The backface temperature rise is also monitored by thermocouple. The output of this thermocouple is measured by a Tektronix Type D High Gain DC Amplifier Module which fits into a Tektronix Type 535 oscilloscope. The output or trace of the specimen-backface thermal history is recorded by synchronizing a camera with the thermal input pulse. A print is obtained for each reading. The time, $t_{1/2}$, is obtained with a reading device that divides the distance between the steady-state temperature (T_0) and the maximum temperature (T_M) in half and that has a calibrated scale to read the distance from the time the lamp is fired (firing point shown as a transient peak on the print).

Thermal conductivity from room temperature to 1500°F is calculated from thermal-diffusivity measurements. The relation between diffusivity and conductivity is as follows:

$$\alpha = \frac{K}{\rho C_p}$$

where

- α = thermal diffusivity
- K = thermal conductivity
- C_p = specific heat
- ρ = density

A thermal-resistance furnace was utilized for thermal conductivity measurements. A 1-in.-dia. specimen is placed inside a pyrofelt insulator and a molybdenum standard is placed in series between the specimen and a water calorimeter. A cylindrical-graphite heat applicator heated by radiation

from the furnace elements is located on the end of the specimen. A graphite radiation shield is placed around the insulated specimen to aid in attaining one-dimensional heat flow through the specimen to the calorimeter. Thermocouples are located on the specimen and the molybdenum standard. Specimen thermal conductivity is determined by measuring (1) the temperature across the specimen and the standard, and (2) the heat flow to the calorimeter. The thermal conductivity of the standard is accurately known and, therefore, is a check of the heat flow through the specimen. The thermocouple leads to the specimen and standard are W-5 Re/W-26 Re.

Slots cut in the graphite heater aid in producing linear heat flow by varying the electrical resistance of the heater. A greater number of slots opposite the applicator increases the electrical resistance so that more heat is radiated to the applicator. A smaller number of slots in the heater opposite the insulated specimen reduces the radiant heat flow to the side of the specimen and ensures that heat input is conducted to the specimen from the heat applicator.

Thermal conductivity is calculated from the following formula, using values of A, T, and Q measured in the test setup:

$$\text{Thermal conductivity} = \frac{Q/A}{\Delta T/\Delta L}$$

where:

Q = Heat transferred to calorimeter and molybdenum standard

ΔL = Distance between thermocouple locations

A = Cross-section area of specimen

ΔT = Temperature gradient between thermocouple locations

b. Test Results

Thermal diffusivity data up to 1500°F are plotted in Figure 24, and the conductivity calculated from these data are plotted in Figure 25. The specific heat values used in these calculations were those listed in Table 5B.05.01, "Specific Heat For Manufactured Graphites," in the Industrial Engineering Graphite Handbook.

Actual diffusivity measurements were made to temperatures of 1500°F (500°F increments); because the diffusivity curves of the graphites are linear with a very small slope above 1500°F, the curve was extended to 3000°F. The conductivity from 1500° to 3000°F was calculated from these extrapolated values.

The thermal conductivity measured by using the thermal gradient method is shown in Figure 26. The values obtained are inordinately high and approximate the conductivity values of bulk graphites that have higher densities. The data are not in agreement with the conductivity data obtained from diffusivity measurements. For comparative purposes, the conductivity of another fibrous-graphite-reinforced composite, Carb-I-Tex 700, is plotted in Figure 27. These latter data are of the same order of magnitude as the conductivity of AGCarb-101 obtained by using the diffusivity technique. Some difference in absolute values is expected as the raw materials and impregnant are different. Since the thermal gradient method as conducted in this program is not a proven method, the use of the conductivity obtained by the diffusivity technique is recommended.

Additional checks on conductivity are being conducted at another source and the results will be used to amend the data presented here.

Figure 24 - Thermal Diffusivity of a Fibrous-Reinforced-Graphite Composite, AGCarb-101

* Calculated from thermal diffusivity

Figure 25 - Calculated Thermal Conductivity of a Fibrous-Reinforced Graphite Composite, AGCarb-101

Figure 26 - Thermal Conductivity of a Fibrous Reinforced Graphite Composite, AGCarb-101 Thermal Gradient Technique

Figure 27 - Thermal Conductivity, (K), of Fibrous Graphite Composites

14. Thermal Expansion

a. Test Procedure

Thermal expansion was determined parallel and perpendicular to the plies. The specimen configuration is shown in Figure 28. To make this specimen from the perpendicular to the plies orientation, three 1-in.-thick cylinders were stacked. The 1/4-in.-thick plates were not tested in the perpendicular to the plies orientation because of possible errors introduced by stacking the larger number of cylinders.

The test method used for thermal-expansion measurements is the optical technique where differences between reference edges of a horizontal specimen are measured at various steady-state temperatures by a pair of filar optical telescopes. Fiducial marks are ground in the specimen to provide a gage distance of 2 in. in a 1/4-in.-dia rod, 2-1/2-in. long. The specimen is heated by thermal radiation from a 6-in. hollow, cylindrical, graphite heating element. A 10 psig helium gas atmosphere is maintained around the specimen and heating element during the test. The specimen is mounted horizontally on two 1/4-in.-dia tantalum-carbide rods positioned on a carbon support fixture. The carbide supports are positioned so that each support is spaced one-third the specimen length. The "stacked" specimen (perpendicular to ply orientation) is placed in a loosely fitted graphite tube approximately 1-3/4-in. long and compressed at one end by two stacked 1/4-in.-dia tungsten pins. The cross hairs of the telescopes are fixed on the fiducial markers through two, filtered view ports in the furnace. The expansion of the specimen is manually tracked with readings taken at various steady-state temperatures. The telescopes have an accuracy of two microinches. The specimen temperature is determined with an automatic-recording, optical-brightness pyrometer.

Figure 28 - Thermal-Expansion Specimens

Data correction factors are applied to the temperature measurements, absorptivity of the quartz window, and for refraction caused by the gas column. All of the corrections are applied to the measured expansion data in an IBM 1130 computer program that derives a least-square-fit curve for the corrected data according to the following equation:

$$\Delta L = A_1 (T_1 - T_{RT}) + A_2 (T_1 - T_{RT})^2 = \frac{L_{T1} - L_{RT}}{L_{RT}}$$

where

- ΔL = thermal expansion
- A_1, A_2 = constants
- L_{T1} = length between fiducial markers at $T^\circ\text{C}$
- L_{RT} = length at room temperature in $^\circ\text{C}$
- T_1 = temperature, $^\circ\text{C}$
- T_{RT} = room temperature, $^\circ\text{C}$

b. Test Results

Thermal expansion of AGCarb-101 parallel and perpendicular to the plies is plotted as a function of temperature in Figure 29. The scatter is greatest at the higher temperatures (above 3500°F) where the data are extrapolated on the basis of best-fit curves. Differences in expansion were noticed in the plates of the two sizes.

Stacking to build up a perpendicular-to-ply specimen of 2-1/2-in. length, which was required because of the thickness (1-in. max), presented no problems. The specimen maintained alignment throughout the test as evidenced by a "zero" reading on cooldown to ambient temperature.

Figure 29 - Thermal Expansion of a Fibrous-Reinforced-Graphite Composite, AGCarb-101

15. Density

a. Test Procedure

Density was measured by using two techniques: bulk measurements and water immersion. In the bulk measurement technique, the rectangular, machined specimen is measured accurately to within 0.0001 in. and the volume calculated. The specimen is weighed on an analytical balance capable of 00.1 mg readout. The density is then calculated.

In the water immersion technique, the specimen is weighed on an analytical balance, covered with a thin plastic coating, weighed to determine the weight of the plastic, and then weighed in water. The density is then calculated.

b. Test Results

Results of the density measurements are listed in Table 23. The water-immersion technique gave slightly higher values than the bulk technique. The difference is within ± 00.01 gm/cc of the average reading.

The densities measured agree with vendor certification data, and the density of each plate is listed in Table 24.

16. Porosity

a. Test Procedure

The specimen configuration is shown in Figure 30. Total porosity is determined by using an Aminco-Winslow Porosimeter that uses the

TABLE 23

DENSITY OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness (Id No.)</u>	<u>Serial No.</u>	<u>Bulk Density, gm/cc</u>	<u>Water Immersion Density, gm/cc</u>	<u>Vendor Certification Density, gm/cc</u>
1/4 in. (1702-3)	7	1.463		1.52
		1.482		
		1.481		
		1.485		
		1.482		
	6	1.469		1.49
		1.464		
		1.484		
		1.468		
		1.471		
1/4 in. (1702-3)	1	1.441	1.466	1.44
		1.423	1.445	
1 in. (1702-4)	5	1.460		1.49
		1.490		
		1.500		
		1.465		
		1.477		
	7	1.487		
		1.482		
		1.361	1.368	
		1.361	1.385	
		1.590	1.612	
1 in.- (1702-4)	1	1.590	1.613	1.585

TABLE 24

VENDOR'S CERTIFICATION FOR PLATES OF A
FIBROUS-REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

	<u>Dash No.</u>	<u>Thickness, in.</u>	<u>S/N</u>	<u>After Graphitization Density, gm/cc</u>
1702	-1 Bias	1/4	1	1.49
	-1		2	1.49
	-1		3	1.48
	-1		4	1.485
	-1	1/4	5	1.485
1702	-2 Bias	1	1	1.485
	-2		2	1.485
	-2	1	3	1.48
1702	-3 Straight Wrap	1/4	2	1.49
	-3		3	1.49
	-3		4	1.49
	-3		5	1.48
	-3		6	1.49
	-3		7	1.52
	-3		8	1.52
	-3		9	1.53
	-3		10	1.52
	-3		11	1.49
	-3	1/4	12	1.53
1702	-4 Straight Wrap	1	2	1.48
	-4		3	1.48
	-4		4	1.49
	-4		5	1.49
	-4	1	6	1.49

TABLE 24 (cont)

	<u>Dash No.</u>	<u>Thickness, in.</u>	<u>S/N</u>	<u>After Graphitization Density, gm/cc</u>
1702 (cont.)	-4 Straight Wrap	1	8	1.48
	-4		9	1.49
	-4		10	1.48
	-4		11	1.49
	-4		12	1.49
	-4		13	1.48
	-4	1	14	1.485

0.25 IN. CUBE

MERCURY INTRUSION

CODE P

Figure 30 - Porosity Specimen

mercury intrusion technique. Mercury is forced into the pores at various pressure intervals up to 5000 psi. The total porosity of the specimen is calculated from the following equations.

$$\text{Total Porosity} = \frac{\text{Total Pore Volume}}{\text{Apparent Volume}} \times 100$$

$$\text{Total Pore Volume} = \text{Apparent Volume} - \text{Real Volume}$$

$$\text{Real Volume} = \frac{\text{Specimen Weight}}{\text{Theoretical Density}}$$

$$\text{Apparent Volume} = \frac{\text{Specimen Weight}}{\text{Apparent Density}}$$

b. Test Results

The total porosity of specimens from the 1/4- and 1-in.-thick plates is listed in Table 25. There does not appear to be a relation between density and total porosity on the basis of these data.

A larger spread in measured density is evidently required to obtain any meaningful correlation.

The pore-size spectra as determined by these tests were generally the same for all plates, and its size range was between 0.035 to 3.5 microns in diameter.

TABLE 25

POROSITY OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Total Porosity, Vol %</u>	<u>Density, gm/cc</u>
1/4	9.5	1.485
1/4	9.9 9.85	1.485
1	9.6	1.53
1	7.7	1.53

17. Permeability

a. Test Procedure

Three disc specimens (1.20-in. dia x 0.250-in. thick) were used for measuring nitrogen gas permeability.

The permeability of each disc to nitrogen gas is measured for pressure drops across the discs ranging from 0 to 150 psig at room temperature.

To measure permeability, the discs were sealed with RTV 60 in a fitting across an orifice ID of 0.600-in., which limits the effective flow area of the specimen to 1.825 cm^2 . The fitting with disc installed was placed in a gas line from a pressurized nitrogen-gas bottle. Gas pressure on the upstream face of the disc was measured with a mercury manometer at low pressures (to 760 mm) and with Bourdon gages at higher pressures.

Gas flow through the disc was measured with a Fischer and Porter Co. series 10A2700 Flowrator meter. The meter was connected directly upstream of the test disc with the discharge port of the meter open to the air. For the purpose of calculating permeability coefficients, the pressure on the downstream surface of the disc was assumed to be at 1 atmosphere. The upstream pressure on the disc was increased by small increments from 0 psig to a maximum of 100 psig. Following each pressure increase, the gas flow through the disc was allowed to stabilize for approximately 30 sec before a flowmeter reading was taken. Flowmeter readings were converted to flow rates in cm^3/sec by use of calibration curves for the flowmeter tubes supplied with the instrument.

Gas velocities in cm/sec and permeability coefficients for each specimen were calculated and recorded.

b. Test Results

The permeability as a function of pressure is listed in Table 26. The unit of permeability used is the Darcy, which is defined as follows: a material has a permeability of one Darcy if in a section of 1 cm^2 in area perpendicular to the flow, 1 ml of fluid of unit viscosity flows at the rate of 1 cm/second under a pressure differential of one atmosphere. The permeability for all specimens is low commensurate with the density.

Significantly lower permeabilities are not probable by added impregnations as the impregnant must be forced into the pores as a liquid. Shrinkage due to solidification and graphitization of the impregnant will always result in a minor number of voids.

18. Hardness

a. Test Procedure

Rockwell R hardness was measured using a standard Rockwell hardness tester with 1/2-in.-dia ball and a 60 kg load.

b. Test Results

The Rockwell R hardness test results are listed in Tables 27 and 28. Within a plate, and within a given plate size, the hardness is reasonably uniform. Hardness is usually not a specification requirement because a relation has not been developed between hardness and other mechanical properties.

TABLE 26

PERMEABILITY OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Plate Density gm/cc</u>	<u>Gas Pressure, psi</u>	<u>Permeability Darcy's</u>
1/4	1.49	35	0.715×10^{-4}
		42	0.63×10^{-4}
		51	0.92×10^{-4}
		64	0.91×10^{-4}
		78	0.94×10^{-4}
		88	0.96×10^{-4}
		104	1.01×10^{-4}
		116	1.01×10^{-4}
1/4	1.49	139	1.12×10^{-4}
		147	1.20×10^{-4}
1	1.49	59	0.31×10^{-4}
		70	0.27×10^{-4}
		92	0.34×10^{-4}
		103	0.395×10^{-4}
		124	0.41×10^{-4}
		130	0.475×10^{-4}
		152	0.51×10^{-4}
		56	0.34×10^{-4}
1	1.49	66	0.30×10^{-4}
		81	0.42×10^{-4}
		100	0.42×10^{-4}
		116	0.47×10^{-4}
		128	0.50×10^{-4}
1		148	0.534×10^{-4}

TABLE 27

ROCKWELL R HARDNESS TEST RESULTS
OF A FIBROUS-REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

60 Kg Load 1/2-in. Ball (1/4-in.-thick plates)

<u>Plate S/N</u>	<u>Rockwell R</u>			<u>Average</u>	<u>Density, gm/cc</u>
5	99	101	98	99	1.48
6	102	103	102	102	1.49
5	101	100	102	101	1.48
5	98	98	96	97	1.48
6	103	105	104	104	1.49
2	102	101	102	102	1.49
2	98	98	101	99	
2	104	101	104	103	
2	100	101	101	101	
3	99	97	101	99	1.49
3	100	102	103	102	
3	104	101	103	102	
3	102	102	103	102	
3	103	103	103	103	
2	100	99	100	100	1.49
2	95	98	97	97	
2	102	101	101	101	
2	101	101	100	101	
2	99	102	99	100	

TABLE 28

ROCKWELL R HARDNESS TEST RESULTS OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>1702-4 Plate S/N</u>	<u>Rockwell R</u>			<u>Average</u>	<u>1-in.- thick Plates Density, gms/cc</u>
12	100	100	99	100	1.49
3	104	106	106	105	1.48
13	102	103	102	102	1.48
6	102	100	100	101	1.49
10	102	101	102	102	1.48
8	103	102	102	102	1.48
2	98	97	98	98	1.48
2	98	100	100	99	
2	92	92	95	93	
14	98	99	102	100	1.485
11	104	107	104	105	1.49
4	99	99	100	99	1.49
2	100	100	99	100	1.48
9	101	100	101	101	1.49
5	102	103	103	103	1.49

Hardness has no quantitative correlation with density although a qualitative relation exists. The density of each plate as taken from vendor certifications is listed in Tables 27 and 28 for informational purposes. In addition to density, other factors such as degree of graphitization and matrix-impregnant volume percentages influence hardness.

19. Electrical Resistivity

a. Test Procedure

The test-specimen configuration is shown in Figure 31. A schematic of the test setup is shown in Figure 32. Current in the circuit was adjusted to obtain a known voltage drop through a standard resistor of 0.01 ohms from which the current was calculated. The potentiometer was then switched across the specimen. Connections to the specimen were made by using spring-loaded clamps. The voltage drop across the specimen of known length was measured to determine the resistance of the specimen, and the resistivity was calculated using the following equation:

$$\rho = \frac{A \cdot R}{L}$$

where

ρ = resistivity, ohm cm

A = area of specimen, sq cms

R = resistance of specimen, ohms

L = length of specimen, cms

b. Test Results

Results of the electrical-resistivity tests are listed in Table 29.

	T
CONE	.40
-3 Plate	.20
-4 Plate	.40

CODE ER

Figure 31 - Electrical-Resistivity Specimen

Figure 32 - Electrical-Resistivity Setup

TABLE 29

ELECTRICAL RESISTIVITY OF A FIBROUS-
REINFORCED-GRAPHITE COMPOSITE, AGCarb-101

<u>Plate Thickness, in.</u>	<u>Serial No.</u>	<u>Resistivity ohm. cm x 10⁻⁴</u>	<u>Density gms/cc</u>
1/4	10	12.6	1.52
1/4	10	12.8	1.52
1	10	15.5	1.48
1	10	15.4	1.48

These tests appear to be sensitive to density variations. A basic assumption must be made, however, that the specimens were processed in an identical manner as the degree of graphitization can also affect the results. These tests are primarily used for quality-control purposes.

C. CONES

1. Hoop Tensile Strength

a. Test Procedure

Hoop strength was determined at room temperature by using NOL "D" rings in a Standard Instron test machine at a strain rate of 0.02-in./min. Specimens of the following dimensions were machined from the cones:

2.80-in. ID x 3.40-in. OD x 0.300-in. high
5.75-in. ID x 6.05-in. OD x 0.300-in. high
6.10-in. ID x 6.50-in. OD x 0.300-in. high

The specimens were fitted over machined D rings, and the gap between rings was about 1/8 to 1/4 in.

Prior to testing, all rings were examined visually for defects and flaws. The principal defects were wrinkles, and the number and amplitude are listed in Table 30. The rings and a close-up of the wrinkle pattern are shown in Figures 33 to 46 inclusive. The causes of wrinkles have been discussed in Section III, Fabrication. The greatest number of wrinkles with the highest amplitude occurred in the straight-tape-wrapped cones (5B series). Only one bias-tape-wrapped cone (5A2) was free of wrinkles.

b. Test Results

(1) Straight-Tape-Wrapped Cones (5B Series)

Results of the hoop tests are listed in Table 31. None of the rings failed in the horizontal plane (gap between

TABLE 30

NUMBER AND AMPLITUDE OF WRINKLES IN AGCarb-101
FIBROUS-REINFORCED-GRAPHITE CONES

<u>Cone Specimen Identification</u>	<u>No. of Wrinkles</u>	<u>Amplitude of Wrinkle, in.</u>
5A-1 HT-1 (Bias tape)	3	0.040, 0.060, 0.060
HT-2 (Bias tape)	0	-
HT-3 (Bias tape)	0	-
5A-2 HT-1 (Bias tape)	0	-
HT-2 (Bias tape)	0	-
5B-1 HT-1 (Straight tape)	2	0.032, 0.040
HT-2 (Straight tape)	4	0.060, 0.040, 0.032, 0.060
HT-3 (Straight tape)	7	0.028, 0.028, 0.048, 0.044, 0.024 0.060, 0.024
HT-4 (Straight tape)	7	0.060, 0.028, 0.028, 0.060, 0.024 0.024, 0.024
5B-2 HT-1 (Straight tape)	6	0.048, 0.028, 0.060, 0.060, 0.032 0.040
HT-2 (Straight tape)	4	0.060, 0.040, 0.030, 0.060
HT-3 (Straight tape)	5	0.016, 0.024, 0.020, 0.028, 0.032
HT-4 (Straight tape)	7	0.032, 0.036, 0.032, 0.024, 0.028, 0.024

Figure 33 - Hoop Tensile Specimen HT-1 Cut from Bias-Tape Cone 5A1, Showing Wrinkles

Figure 34 - Hoop Tensile Specimen HT-2 Cut from Bias-Tape Cone 5A1,
No Wrinkles Noted

Figure 35 - Hoop Tensile Specimen HT-3 Cut from Bias-Tape Cone 5A1,
No Wrinkles Noted

Figure 36 - Hoop Tensile Specimen HT-1 Cut from Bias-Tape Cone 5A2,
No Wrinkles Noted

Figure 37 - Hoop Tensile Specimen HT-2 Cut from Bias-Tape Cone 5A2,
No Wrinkles Noted

Figure 38 - Hoop Tensile Specimen HT-3 Cut from Bias-Tape Cone 5A2,
No Wrinkles Noted

Figure 39 - Hoop Tensile Specimen HT-1 Cut from Straight-Tape Cone 5B1, Showing Wrinkles

Figure 40 - Hoop Tensile Specimen HT-2 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

Figure 41 - Hoop Tensile Specimen HT-3 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

Figure 42 - Hoop Tensile Specimen HT-4 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

Figure 43 - Hoop Tensile Specimen HT-1 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

Figure 44 - Hoop Tensile Specimen HT-2 Cut from Straight-Tape Cone 5B2, Showing Wrinkles

Figure 45 - Hoop Tensile Specimen HT-3 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

Figure 46 - Hoop Tensile Specimen HT-4 Cut from Straight-Tape Cone 5B2,
Showing Wrinkles

TABLE 31HOOP TENSILE STRENGTH (RT) OF AGCarb-101 FIBROUS-
REINFORCED-GRAPHITE CONES

<u>Wrap Type</u>	<u>Specimen Ident.</u>	<u>Hoop Tensile Strength, psi</u>	<u>Remarks</u>
Warp	5B-1 HT-1	2860	No mandrel
Warp	HT-2	2016	No mandrel
Warp	HT-3	2794	No mandrel
Warp	HT-4	2306	No mandrel
Warp	5B-2 HT-1	1824	Graphite Mandrel
Warp	HT-2	2851	Graphite Mandrel
Warp	HT-3	3766	Graphite Mandrel
Warp	HT-4	3792	Graphite Mandrel
45° Bias			
Tape	5A-1 HT-1	2204	No mandrel
Tape	HT-2	2919	No mandrel
Tape	HT-3	3878	No mandrel
Tape	5A-2 HT-1	4019	Graphite Mandrel
Tape	HT-2	3689	Graphite Mandrel
Tape	HT-3	4520	Graphite Mandrel

D rings). The location of all ring failures was in a wrinkle area at some angle from the horizontal clearly illustrating the damaging effects of such defects. The amplitude of the wrinkles appeared to govern the failure stress; whereas, the frequency of defects did not appear to influence the test results. Wrinkles of high amplitude in effect reduce the number of plies in the cross-section of the specimen. In machining of a specimen, the apexes of the wrinkle are removed exposing a "between ply" orientation to the specimens surface. The block tensile strength of the material is low (600 psi), and a crack is initiated between the plies and progresses inward until it intersects a ply oriented normal to the direction of propagation. If the amplitude of the wrinkle is 0.080 in., the crack has traversed approximately 27% of the distance through a 0.300 in. thick specimen. This mechanism of failure is illustrated in the macrophotographs shown in Figure 47.

Wrinkles obscure the effects of processing with and without mandrels as wrinkle amplitude differs in the two cones.

There is a difference in the structure of the two cones processed with and without a mandrel as shown in Figure 48. The cone processed without a mandrel has a loose fiber packing and the voids are round; the cone processed with a mandrel has a tight fiber packing and the voids are rectangular in appearance. The rectangular voids are usually associated with wrinkled fabric.

(2) Bias-Wrapped Cones

The hoop-tensile-test results of bias-tape-wrapped cones are also listed in Table 31. The results are commensurate with the absence of defects with one exception. The lowest value is associated with a wrinkle and the remaining values are in line with expected results. Assuming that the bias angle was actually 45 deg, the normalized tensile strength can be calculated by using equation (1).

Specimen HT-4 Cone 5B1
Hoop Tensile Strength 2306 psi

Specimen HT-1 Cone 5B2
Hoop Tensile Strength 1824 psi

Figure 47 - Fractures of Hoop Tensile Specimens Showing Effect of Wrinkles

Cone 5 B1
(Processed without
Graphite Mandrel)

Cone 5 B2
(Processed with
Graphite Mandrel)

Magnification 100X

* View of Cross section in axial direction

Figure 48 - Microstructure of a Fibrous-Graphite-Reinforced Composite
(AGCarb-101) Straight-Tape-Wrapped Cone

$$\sigma_{45^\circ} = \sigma_n \cos^2 \theta$$

where:

σ_{45° = strength at 45-deg angle to tensile axis

σ_n = strength parallel to tensile axis

θ = angle of plies to tensile axis

The normalized tensile strength by this calculation is 8150 psi. In reviewing the normalized values, consideration should be given to the bending stresses, which can be as much as 50% of the applied average stress level. Because of scatter, no analysis can be made of ring size effects.

The fracture of bias-tape-wrapped rings is typical in that fracture occurred at a 45-deg angle (measured angle, 40.5 deg) as shown in Figure 49 (Note - ring is tilted and two surfaces, top and OD, are shown in the photo).

There are differences in the microstructure of the cones processed with and without a mandrel as shown in Figure 50. The cone processed without a mandrel shows a tighter fiber packing and large voids; the cone processed with a graphite mandrel has a loose fiber packing and smaller but more numerous voids. This is contrary to what was observed in Figure 48 for the straight-tape cones. Additional structures must be viewed before any major conclusions can be made regarding the effects of processing with and without a mandrel.

2. Tensile Strength

Results of the room- and elevated-temperature tensile tests are listed in Tables 32 and 33. The round tensile specimen (Figure 8) was used for these tests; specimen lengths were oriented in the longitudinal

Figure 49 - Fracture of Hoop Tensile Specimen in Bias-Wrapped Cone with No Wrinkles

5 A1
(Processed without
Graphite Mandrel)

5 A2
(Processed with
Graphite Mandrel)

Magnification, 100X

* View of Cross-section of cone in axial direction

Figure 50 - Microstructure of a Fibrous-Reinforced-Graphite Composite
(AGCarb-101) Bias- (45 Deg) Tape-Wrapped Cone

TABLE 32

ROOM TEMPERATURE TENSILE TEST RESULTS OF
AGCarb-101 FIBROUS-REINFORCED-GRAPHITE CONES

<u>Cone Identification</u>	<u>Specimen Identification</u>	<u>Tensile Strength, psi</u>	<u>Remarks</u>
5A1	1T	1,900	No mandrel
	2T	2,620	No mandrel
	3T	2,580	No mandrel
	4T	1,300	No mandrel
	5T	2,880	No mandrel
	6T	2,930	No mandrel
	7T	2,800	No mandrel
	8T	<u>1,520</u>	No mandrel
		$\bar{x} = 2,312$	
		$s = 649$	
5A2	1T	3,170	Graphite Mandrel
	2T	3,470	Graphite Mandrel
	3T	3,750	Graphite Mandrel
	4T	3,420	Graphite Mandrel
	5T	3,330	Graphite Mandrel
	6T	3,280	Graphite Mandrel
	7T	3,700	Graphite Mandrel
	8T	<u>3,200</u>	Graphite Mandrel
		$\bar{x} = 3,425$	
		$s = 225$	
5B1	1T	2,220	No mandrel
	2T	2,510	No mandrel
	3T	2,730	No mandrel
	4T	<u>2,110</u>	No mandrel
		$\bar{x} = 2,375$	
		$s = 275$	
5B2	1T	2,700	Graphite Mandrel
	2T	2,930	Graphite Mandrel
	3T	3,030	Graphite Mandrel
	4T	<u>2,580</u>	Graphite Mandrel
		$\bar{x} = 2,800$	
		$s = 183$	

TABLE 33

ELEVATED-TEMPERATURE TENSILE-TEST RESULTS OF AGCarb-101
FIBROUS-REINFORCED-GRAPHITE CONES

<u>Cone Identification</u>	<u>Specimen Identification</u>	<u>Test Temp, °F</u>	<u>Tensile Strength, psi</u>	<u>Remarks</u>
5A2	10T	2000	3,300	
	11T	2000	3,500	
	14T	2000	3,500	$\bar{x} = 3,433$
5B2	7T	2000	2,900	
	9T	2000	2,400	$\bar{x} = 2,650$
5A2	9T	3000	4,100	
	12T	3000	3,900	
	13T	3000	3,900	$\bar{x} = 3,970$
5B2	5T	3000	3,200	
	6T	3000	3,700	$\bar{x} = 3,450$

direction of the cone's surface. To analyze the data, it must be recognized that the cone had a 15-deg half angle, and the wrap was parallel to centerline. In effect, the orientation of the principal fiber (warp) reinforcement is in the circumferential orientation, and the fill direction is parallel to centerline. Thus, the fill direction properties of the laminate should be reflected in these tests except for the bias orientation of the fill fiber (15 deg) to the tensile axis. For the bias-deg (45 deg) tape-wrapped cone, the warp and fill is 45 deg to a great circle and 15 deg to the tensile axis.

The straight-tape-wrapped cone processed without a mandrel (5B 1) had slightly higher strength than the biased-tape-wrapped cone processed without a mandrel (5A 1). Conversely, the bias-tape-wrapped cone processed on a graphite mandrel (5A 2) had significantly higher strength than the straight-tape-wrapped cone processed on a graphite mandrel (5B 2). (Caution was exercised to avoid areas with defects when cutting the specimens.)

No correlation can be made with tensile data from the plate because of the complex orientation on effects of the fibrous reinforcement.

No statistical evaluation of the results obtained from the cones was made because of the frequency of defects. Only trends are indicated where noted.

The elevated-temperature tensile strength data are shown in Table 33. Elevated-temperature testing of specimens cut from cones was minimized. The trend of high strength with increasing temperature is evident at 3000°F but not at 2000°F.

3. Block Tensile Strength

The specimens were machined from a radial section of the cone. The surfaces were then ground flat for adhesive bonding to extensions for testing.

The block tensile results are listed in Table 34. The results are higher than those obtained from the plates; this is attributed to the curvature of the plies in the test section, which in effect increases the area between plies.

4. Compressive Strength

The specimens were machined from an axial section parallel to the cone surfaces.

Compression test results are listed in Table 35. In these tests, the fibers were oriented 15 deg to the compression axis. The results are significantly lower than the compressive strength of the blocks. This is attributed to orientation effects, because in the cones, the "fill" is oriented in the longitudinal direction. The block compressive strengths were tested with the "warp" parallel to the compression axis. The compressive strength in the fill direction of the cones is approximately equal to the fill tensile strength of the blocks.

The compressive strength of the cones is higher than the tensile which is contrary to the behavior noted for the plates. No explanation can be made for this difference in behavior.

5. Interlaminar Shear Strength

Interlaminar shear strength results of specimens cut from cones are listed in Table 36. True interlaminar shear was not obtained in this series of tests because of fiber-stress axis orientation. This was reflected in the failure mechanism behavior. Failure was originated by breaking several plies (higher load) and then progressing between plies to failure (low load). In most instances, failure did not occur between

TABLE 34

BLOCK TENSILE STRENGTH OF FIBROUS-REINFORCED-
GRAPHITE COMPOSITE (AGCarb-101) CUT FROM CONES

Cone half angle, 15 Deg

<u>Cone Identification</u>	<u>Wrap Type</u>	<u>Specimen No.</u>	<u>Block Tensile Strength, psi</u>	<u>Remarks</u>
5 A1	45-Deg Bias	1	804	No Mandrel
		2	712	No Mandrel
		3	774	No Mandrel
5 A2	45-Deg Bias	4	700	Graphite Mandrel
		5	698	Graphite Mandrel
		6	690	Graphite Mandrel
5 B1	Straight	7	730	No Mandrel
		8	719	No Mandrel
		9	853	No Mandrel
5 B2	Straight	10	748	Graphite Mandrel
		11	656	Graphite Mandrel
		12	702	Graphite Mandrel

TABLE 35

COMPRESSIVE STRENGTH OF A FIBROUS-REINFORCED-
GRAPHITE COMPOSITE (AGCarb-101) CONES

<u>Cone Identification</u>	<u>Wrap Type</u>	<u>Specimen No.</u>	Cone half angle, 15 Deg	
			<u>Compression Strength, psi</u>	<u>Remarks</u>
5 A1	45-Deg Bias	1	----	No Mandrel
		2	4690	No Mandrel
		3	4900	No Mandrel
5 A2	45-Deg Bias	4	4920	Graphite Mandrel
		5	5050	Graphite Mandrel
		6	5130	Graphite Mandrel
5 B1	Straight Warp	7	3950	No Mandrel
		8	3900	No Mandrel
		9	4770	No Mandrel
5 B2	Straight Warp	10	4180	Graphite Mandrel
		11	4340	Graphite Mandrel
		12	4210	Graphite Mandrel

TABLE 36

INTERLAMINAR SHEAR STRENGTH OF
FIBROUS-REINFORCED-GRAPHITE AGCarb-101 CONES

<u>Cone Identification</u>	<u>Wrap Type</u>	<u>Specimen No.</u>	<u>Interlaminar Shear Strength</u> (psi)	<u>Remarks</u>
5 A1	45-Deg Bias	1	589	No Mandrel
		2	512	No Mandrel
		3	675	No Mandrel
5 A2	45-Deg Bias	4	720	Graphite Mandrel
		5	648	Graphite Mandrel
		6	809	Graphite Mandrel
5 B1	Straight	7	606	No Mandrel
		8	572	No Mandrel
		9	609	No Mandrel
5 B2	Straight	10	589	Graphite Mandrel
		11	682	Graphite Mandrel
		12	589	Graphite Mandrel

the notches. The data are reported but should not be used when true inter-laminar-shear-strength values are required.

6. Hardness

The hardness of the cones is shown in Table 37. The values are significantly lower than those obtained for the plates (95-102 R_B). This indicates that the hardness test can be used as a simple qualifying test.

There is no relation between hardness and density of the cones.

7. Electrical Resistivity

The electrical-resistivity data are also shown in Table 37. These values are higher than those obtained from the plates indicating lower density. However, the electrical resistivity values obtained for the cones cannot be correlated with the density of the cones.

8. Total Porosity

The total porosity of the cones is also shown in Table 37. With the exception of bias-wrapped cone 5A2, the cones had a significantly higher amount of total porosity than the plates (9 to 10%).

There is a relation between hardness and electrical resistivity with total porosity. A relation of hardness, resistivity and total porosity may have been obtained if density checks were performed on each of the specimens used for the tests.

TABLE 37

HARDNESS, ELECTRICAL RESISTIVITY AND POROSITY
OF AGCarb-101, FIBROUS-REINFORCED-GRAPHITE, CONES

<u>Wrapping Mandrel</u>	<u>Hardness, Rockwell R</u>	<u>Electrical Resistivity, Ohm-cm x 10⁻⁴</u>	<u>Total Porosity, Vol. %</u>	<u>Density, gm/cc</u>
Cone 4A, 45-Deg Bias				
No Mandrel	65-69	18.0	15.7	1.43-1.45
Graphite Mandrel	86-87	16.2	9.9	1.41-1.42
Cone 5B, Warp				
No Mandrel	74-75	19.2	13.5	1.40-1.45
Graphite Mandrel	75-76	16.5	15.1	1.45

V. CONCLUSIONS

A. FABRICATION

1. The plates and the cones that were supported during the initial graphitization exhibited additional shrinkage after removal of the supports during subsequent graphitization.

2. Most of the shrinkage in the plates occurred in the perpendicular to the ply direction with the greatest amount of shrinkage occurring in the graphitizing cycle.

3. Impregnation of the plates was incomplete because the stacked plates reduced the surface area directly exposed to the impregnant.

4. The tape-wrapping process controls were not satisfactory. This was at least partly caused by the fabricator deviating from established requirements. The presence of wrinkles in the cones indicated a lack in one or more of the following tape-wrapping controls: (1) wrap speed, (2) tape temperature, (3) billet temperature, and (4) roller pressure.

B. PROPERTIES DETERMINATION

1. Differences in both mechanical and thermal properties of the 1/4- and 1-in.-thick plates were observed, indicating size sensitivity. This is attributed to the degree of shrinkage during processing.

2. On the basis of statistical tests, all plates of a given size can be considered one batch or lot of material.

3. The warp and fill directions differ in tensile strength by a factor of 0.5 to 0.6.

4. The block tensile and the interlaminar shear strengths are low, indicative of relatively poor bonding between plies. This is the principal deficiency of bidirectional reinforced composites.

5. Correlation of property data between plates and cones could not be made because of orientation effects and defects in the cones.

VI. RECOMMENDATIONS

1. Fabrication controls for producing wrinkle-free conical and cylindrical components should be established.
2. The relationship of shrinkage and/or warpage and the method of component support during graphitization should be determined.
3. A development program should be undertaken to improve the block tensile and interlaminar shear properties of AGCarb-101.

APPENDIX A

LOCATION AND IDENTIFICATION OF
TEST SPECIMENS FROM 1/4- AND 1-IN. PLATES

RT - Round Tensile

C - Density

Specimen Location Layout

RT - Round Tensile
T - Flat Tensile

1702-4 S/N 2 1" Thick Plate - 1-18RT, 1-4T
1702-4 S/N 14 1" Thick Plate - 1-33RT, 1T
1702-2 S/N 1 1" Thick Plate Bias - 1-10 RT

Specimen Location Layout

T - Flat Tensile
FRB - Fatigue-Rotating Beam

1702-4
Specimen Plan for Plate S/N 3 - 1-in. Thick

- T - Flat Tensile
- RT - Round Tensile
- C - Compression
- RTF - Round Tensile - Fill Direction
- DF-P - Thermal Diffusivity
Parallel to Plies

1702-4 SN 5

Specimen Location Layout

T - Flat Tensile

IL - Interlaminar Shear

1702-4 S/N 6 1" Thick Plate

Specimen Location Layout

T - Flat Tensile
FRB - Fatigue - Rotating Beam
LXP - Thermal Expansion Parallel to Plies
LXT - Thermal Expansion - Normal to Plies
KN - Thermal Conductivity - Normal to Plies
KP - Thermal Conductivity - Parallel to Plies

Specimen Location Layout

T - Flat Tensile
 IL - Interlaminar Shear
 C - Compression
 BT - Block Tensile

1702-4 SN 9 1" Thick Plate

Specimen Location Layout

- T - Flat Tensile
- RET - Round Tensile (Hi Temp)
- C - Compression
- F - Flexure
- DN - Dynamic Modulus
- ER - Elect. Resistivity

1702 -4 S/N 10 1" Thick Plate

Specimen Location Layout

- FRB - Fatigue - Rotating Beam
- RTF - Round Tensile - Fill Direction
- T - Flat Tensile

1702-4 S/N 11 1" Thick Plate

Specimen Location Layout

WOL - Fracture Toughness
T - Flat Tensile

1702-4 SN 12 1" Thick Plate

Specimen Location Layout

Specimen Location Plan for Plate

S/N 2 $\frac{1}{4}$ " Thick (1702-3) Specimens 1-18
 S/N 3 $\frac{1}{4}$ " Thick (1702-3) Specimens 19-36
 S/N 1 $\frac{1}{4}$ " Thick Bias (1702-1) Specimens 1-10

CODE: RT - Round Tensiles
 ET - Elev. Temp. Round Tensiles

Specimen Location Plan for Plate S/N 4 - 1/4-in. Thick
 (1702-3)

CODE: IL - Interlaminar Shear
 BT - Block Tensile
 ET - Round Tensile
 C - Compression Specimen

Specimen Location Plan for Plate S/N 6 - 1/4-in. Plate 1702-3

CODE: FT - Fracture Toughness Specimen
 C - Compressive Specimen
 F - Flexure Specimen

CODE: IL - Interlaminar Shear

Specimen Location Plan for Plate S/N 8 - 1/4-in. Thick 1702-3

IL - Interlaminar Shear

P - Porosity - Mercury Intrusion

FT - Fatigue - Rotating Beam

Specimen Location Plan for Plate S/N 9 - 1/4-in. Thick 1702-3

FT - Fracture Toughness
 ER - Electrical Resistivity
 LX - Thermal Expansion
 DF - Thermal Diffusivity
 DM - Dynamic Modulus

ET - Round Tensile (Hi Temp)

KN - Thermal Conductivity

F - Flexure

1702-3 S/N 11 1/4" Plate

Specimen Location Layout

F - Flexure
C - Compression

1702-3 SN 12 .250" Thick Plate

Specimen Location Plan

BT - Block Tensile
DF - Thermal Diffusivity

Specimen Location Layout

Bias Plate 1702-2 - S/N 2 - 1.0" Thick

LX - Thermal Expansion

PEB - Gas Permeability

KNB - Thermal Conductivity
Normal to Plies

1702-1 SN 2 $\frac{1}{4}$ " Plate - Bias

Specimen Location Layout